

Waters GCT Premier Mass Spectrometer Operator's Guide

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Safety information

General

The Waters® GCT™ Premier Mass Spectrometer is designed solely for use as a mass spectrometer; any attempt to use it for any other purpose can damage the instrument, and will invalidate its warranty.

The Waters GCT Premier Mass Spectrometer conforms to European standard EN61010-1:2001, Safety requirements for electrical equipment for measurement, control, and laboratory use - Part 1: General requirements.

The instrument has been designed and tested according to recognized safety standards. If the instrument is used in a manner not specified by the manufacturer, the protection provided to the user may be impaired.

Whenever the safety protection of the instrument has been compromised, disconnect the instrument from all power sources, and secure the instrument against unintended operation.

The instrument must be installed so that the operator can easily isolate the instrument from the mains power supply at all times.

Biological hazard


When you analyze physiological fluids, take all necessary precautions, and treat all specimens as potentially infectious. Precautions are outlined in “CDC Guidelines on Specimen Handling,” *CDC - NIH Manual*, 1984.

Suitable protection against biohazards must be taken during maintenance procedures and cleaning, as parts of the instrument are exposed to potentially infectious samples.

Chemical hazard

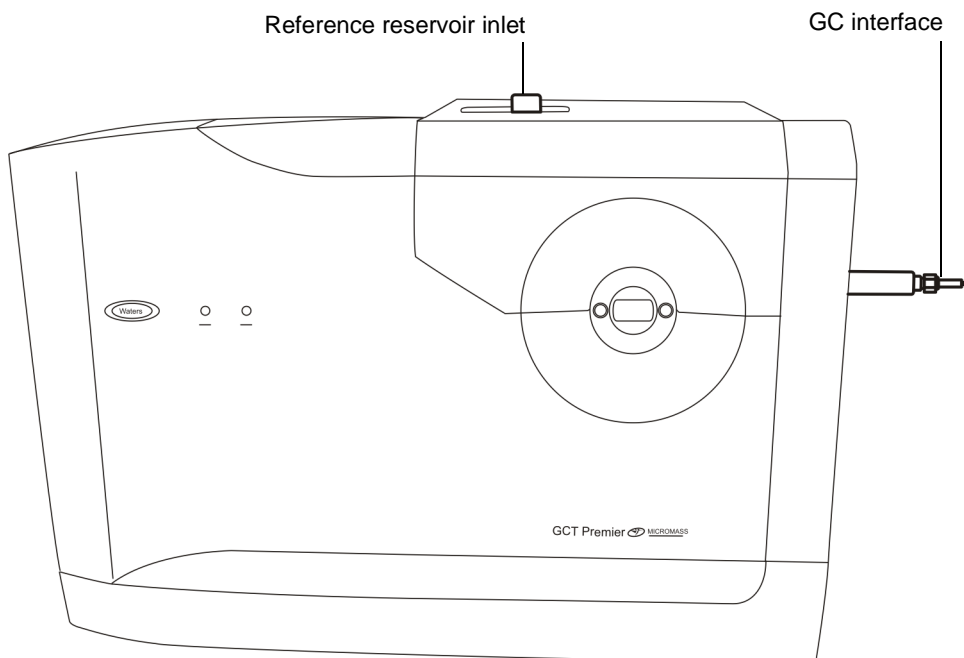
Good Laboratory Practice should be adhered to when using potentially toxic, caustic, or flammable solvents and analytes.


High temperature hazard

 **Warning:** To avoid burn injuries, take care when working with the instrument; the reference reservoir inlet and GC interface can be hot.

The reference reservoir inlet and GC interface shown in [Figure titled “GCT Premier high temperature hazard:” on page 1-iv](#) can be hot.

GCT Premier high temperature hazard:



 **Warning:** To avoid burn injuries, take care when working with the rotary pump; it can be hot.

High voltage hazard



Warning: To avoid electric shock, shut down the instrument if any of the external covers are damaged.

Safety symbols

Warnings in this guide or on the instrument must be observed during all phases of service, repair, installation, and operation of the instrument.

Waters Corporation assumes no liability for the failure of those who install, repair, or operate its instruments to comply with these requirements.

Safety symbols appear in this guide and on the instrument. They accompany warning and caution statements. A Warning statement draws your attention to the risk of injury or death; a Caution statement draws attention to the risk of damaging the instrument.



Warning: General warning indicating a potential health or safety hazard. See the operator's guide for instructions.



Warning: Hazardous voltages.



Warning: Hot surfaces.



Warning: Corrosive substances.



Warning: Toxic substances.



Warning: Flammable substances.



Warning: Biological agents present that may constitute a serious health threat.



Warning: Laser radiation.



Warning: UV radiation.



Caution: Care must be taken to avoid the possibility of damaging the instrument, or affecting its operation.

Consignes de sécurité

Généralités

Le GCT™ Premier de Waters® est destiné exclusivement à être utilisé comme spectromètre de masse. Tout usage détourné du GCT Premier risquerait d'endommager l'instrument et invaliderait sa garantie.

Le spectromètre de masse GCT Premier de Waters est conforme à la norme européenne EN61010-1:2001, Règles de sécurité pour appareils électriques de mesure, de régulation et de laboratoire - Partie 1: Prescriptions générales.

Cet instrument a été conçu et testé conformément aux dispositions des normes de sécurité les plus reconnues. Toute utilisation de l'équipement non conforme aux instructions du fabricant risque de rendre déficiente la protection assurée par l'instrument.

Dans le cas où la sécurité de l'utilisateur se trouverait compromise, débranchez le cordon d'alimentation de l'instrument et assurez-vous qu'il ne pourra être mis en marche par mégarde.

L'instrument doit être installé de façon à faciliter l'accès de l'utilisateur au bloc d'alimentation électrique.

Risques biologiques

Lorsque vous analysez des fluides physiologiques, faites preuve d'une extrême prudence et considérez tous les spécimens comme potentiellement infectieux. Une liste des précautions à prendre figure dans le « CDC Guidelines on Specimen Handling », CDC - NIH Manual, 1984.

Portez des vêtements de protection adéquats lorsque vous procédez à une opération de maintenance ou de nettoyage, car les pièces de l'instrument peuvent comporter des traces des substances infectieuses auxquelles elles ont été exposées.

Risques chimiques

L'usage des solvants et analytes potentiellement toxiques, caustiques ou inflammables doit s'effectuer dans le respect des bonnes pratiques de laboratoire.

Risques de brûlure



Avertissement: Pour éviter des brûlures, prenez garde en travaillant avec l'instrument; le réservoir de référence de l'inlet et l'interface de chromatographie gazeuse peuvent être à température élevée.

Le réservoir de référence de l'inlet et l'interface de chromatographie gazeuse peuvent être à température élevée, comme indiqué dans la figure intitulée "GCT Premier high temperature hazard:" à la page 1-iv.



Avertissement: Pour éviter des brûlures, prenez garde en travaillant avec la pompe qui peut être chaude.

Risques d'électrocution



Avertissement: Pour éviter une décharge électrique, arrêtez l'instrument si l'enveloppe de protection externe est endommagée.

Pictogrammes de sécurité

Les avertissements présents dans le manuel de l'utilisateur ou sur l'instrument-même doivent être scrupuleusement pris en considération, et ce à tout moment, que ce soit pendant l'entretien, la réparation, l'installation ou le fonctionnement de l'instrument. Tout défaut d'application de ces règles de sécurité serait considéré comme une violation des normes de sécurité relatives à la conception et à l'usage prévu de l'instrument.

Waters Corporation ne saurait voir sa responsabilité engagée en cas de manquement de l'utilisateur à respecter les consignes de sécurité.

Les pictogrammes qui suivent apparaissent dans le manuel de l'utilisateur ou sur l'instrument. L'**Avertissement** s'applique à toute instruction destinée à attirer l'attention de l'utilisateur sur l'existence d'un risque de blessure ou de mort. L'**Attention** s'applique à toute instruction destinée à informer l'utilisateur d'une situation qui peut endommager l'instrument.



Avertissement: Symbole d'avertissement indiquant qu'un produit ou composant pose un risque potentiel pour votre santé et sécurité. Consultez le manuel de l'utilisateur.



Avertissement: Tensions dangereuses.



Avertissement: Surfaces chaudes.



Avertissement: Substances corrosives.



Avertissement: Substances toxiques.



Avertissement: Substances inflammables.



Avertissement: Présence d'agents biologiques susceptibles d'être nuisibles à la santé.



Avertissement: Rayonnements laser.



Avertissement: Rayonnements UV.



Caution: Care must be taken to avoid the possibility of damaging the instrument, or affecting its operation.



Attention: Utilisez l'instrument en faisant preuve de beaucoup de précaution pour éviter de l'endommager et ainsi nuire à son fonctionnement.

GCT Premier mass spectrometer information

Intended use

The Waters GCT Premier Mass Spectrometer can be used as a research tool to deliver authenticated exact mass measurement; it is not for use in diagnostic procedures.

Calibration

Follow acceptable methods of calibration with pure standards to calibrate. Use a minimum of five standards to generate a standard curve. The concentration range should cover the entire range of quality-control samples, typical specimens, and atypical specimens.

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1

Instrument Description

This chapter describes the instrument, including its controls, gas, and electrical connections.

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Introduction

The GCT Premier is a high-performance, bench-top, orthogonal acceleration/time-of-flight (oa-TOF) mass spectrometer. To identify volatile sample components, the instrument is coupled to a gas chromatograph (GC). To identify nonvolatile components, it operates as a standalone instrument that receives sample through a direct insertion (DI) or direct chemical ionization (DCI) probe.

GCT Premier mass spectrometer:



Unlike scanning instruments, a TOF instrument performs parallel detection of all masses within the spectrum at high sensitivity and acquisition rates. This is particularly advantageous when the instrument is coupled to fast chromatography, because each spectrum represents the sample composition

at a given moment, regardless of how rapidly the sample composition is changing.

See also: [Appendix G Theory and Principles of Operation](#) for details of the electron ionization (EI), chemical ionization (CI) field ionization (FI), and field desorption ionization (FD) techniques, and the ion optics in the instrument.

Ion source

The outer ion source consists of two assemblies:

- EI/CI/DCI outer source assembly
- FI/FD outer source assembly

The ion source is at ground potential, allowing simple, direct-coupling to the GC inlet. The instrument can operate in positive and negative EI and CI modes and positive FI and FD modes.

Electron ionization/chemical ionization (EI/CI) outer source assembly

You access the outer source assembly from the top of the instrument, removing the outer source lid assembly cover. The outer source assembly comprises, a source heater and focusing and transfer optics. Heaters in the outer source raise the source temperature to ensure sample vaporization and avoid condensation of analytes onto the inner source and lenses.

See also: [Changing the EI/CI outer source assembly on page 2-6.](#)

Field ionization/field desorption ionization (FI/FD) outer source assembly

Field ionization (FI) is a process in which spectra with little or no fragmentation are obtained. It is used to detect and exactly measure molecular ions, which can be weak or absent in EI mode.

The FI/FD outer source assembly is used only with the FI/FD inner source. It comprises the extraction rods, emitter contacts and focussing and transfer optics. It is accessed from the top of the instrument.

See also: [Changing the FI/FD outer source assembly on page 9-3.](#)

Inner source assembly

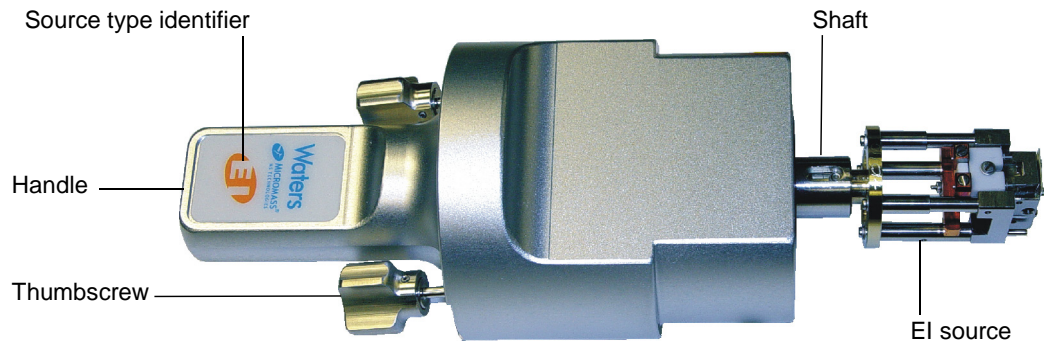
The inner source assembly comprises a handle that is attached to a shaft supporting the source. Separate inner source assemblies are provided for EI, CI, DCI, FI, and FD. The source type is identified on the handle.

The inner source assembly is secured to the outer source by means of two thumbscrews. Two Viton[®] O-rings form a vacuum seal between the inner source mounting flange and the outer source.

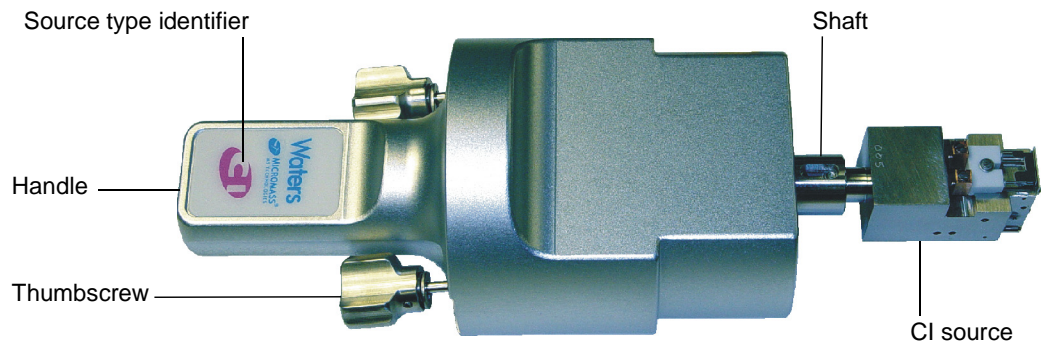
You do not need to vent the analyzer to change the inner source.

See also: [Changing the EI/CI inner source assembly on page 2-2.](#)

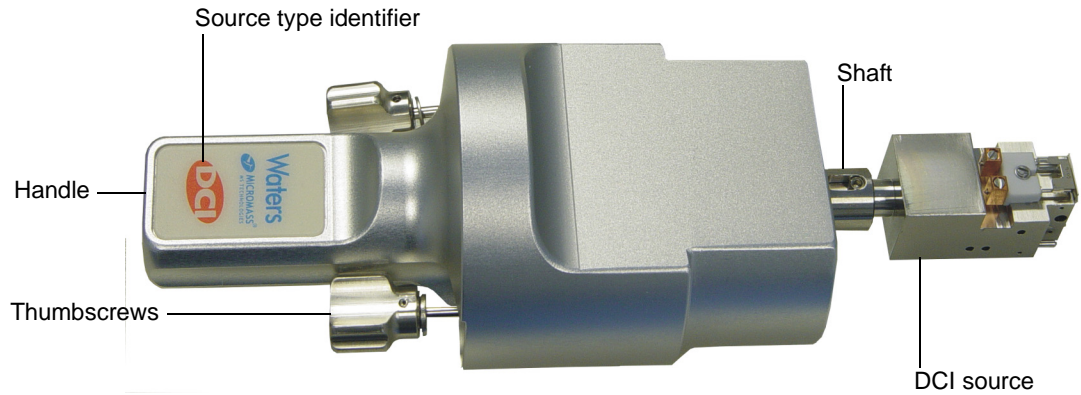
EI inner source assembly:



CI inner source assembly:

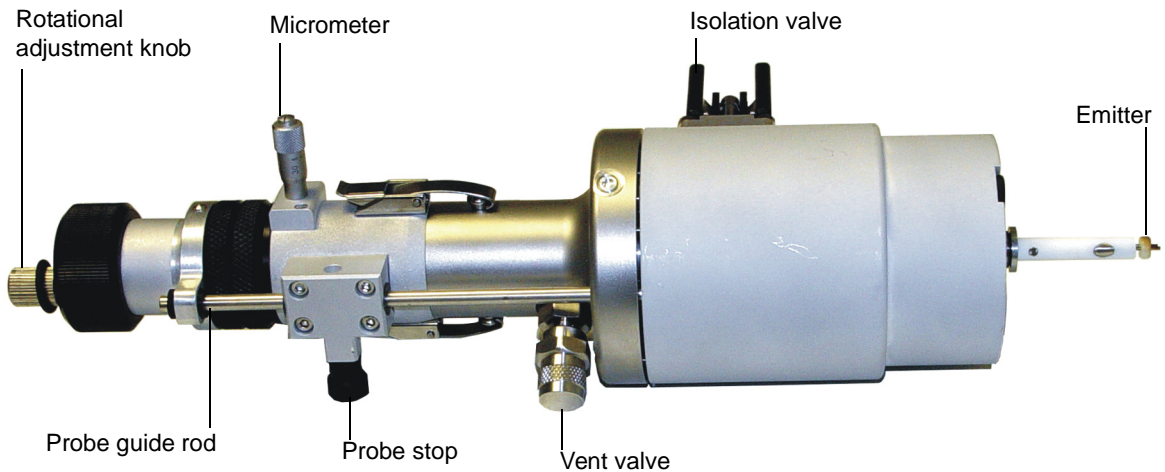


DCI inner source assembly:



The FI/FD inner source consists of a removable probe holding the FI/FD emitter. The emitter consists of a tungsten wire on which there are carbon microneedles.

FI/FD inner source assembly:



Inlets

GC interface for EI/CI

The GC interface provides a heated transfer line between the GC (gas chromatograph) and the ion source. The transfer line ensures even heating which prevents the sample from condensing in the GC column before it reaches the ion source.

See also: [Chapter 6 The GC Interface](#) and the appropriate operator's guide for GC operation details.

GC interface for FI/FD

The GC-FI interface provides a transfer line between the GC (gas chromatograph) and the FI/FD ion source. The end of the column is positioned approximately 2 mm from the emitter wire on the FI/FD inner source.

The sensitivity of analytes introduced via the GC can be reduced if the end of the column is not close to the emitter.

See also: [Chapter 9 FI/FD Operation](#).

Direct insertion (DI) probe

The direct insertion (DI) introduces solid or liquid samples not sufficiently volatile for injection via the reference inlet, into the ion source. The DI probe is inserted into the instrument source via the probe insertion lock, which must be fitted to the instrument prior to using the probe.

See also: [Chapter 7 The Direct Insertion Probe](#).

Direct chemical ionization (DCI) probe

The direct chemical ionization (DCI) probe is used for relatively nonvolatile samples. Like the DI probe, the DCI probe is inserted into the instrument source via the probe insertion lock, which must be fitted to the instrument prior to using the probe. The DCI probe is used with the DCI inner source, rather than the standard CI inner source. The DCI probe can also be used with the EI inner source.

See also: [Chapter 8 The Direct Chemical Ionization \(DCI\) Probe](#).

Reference reservoir inlet

A separate reference reservoir inlet allows the introduction of volatile reference material. The reference inlet introduces reference materials required for calibration and mass measurement.

See also: [Figure titled “Reference reservoir pump valve and reference inlet:” on page 1-11.](#)

Use a septum-piercing syringe to introduce reference liquids into the instrument through the hole in the center of the reference reservoir inlet cap.

A septum in the cap is made of silicon rubber backed with Teflon[®] and is leak-proof (when fitted with the teflon coat facedown). However, the septum can start to leak when you make repeat injections of reference material, use a damaged needle tip, or over-tighten the reference reservoir inlet cap. Renew the septum when required.

See also: [Reference reservoir maintenance on page 10-16.](#)

Vacuum system

The GCT Premier vacuum system incorporates two vacuum systems: fore pumping and fine pumping. Before the fine pumping can be operated, the region to be pumped has to be fore pumped. Fore pumping can achieve a vacuum level of approximately 1e-3 kPa (1e-2 mbar, 1.45e-4 psi) inside the instrument. Fine pumping is able to achieve vacuum levels in the region of 1e-8 kPa (1e-7 mbar, 1.45e-9 psi), which is necessary to achieve the ultimate performance of the mass spectrometer.

The fine pumping is provided by turbomolecular pumps, which are controlled via the MassLynx[™] software. The fore pumping is provided by either an oil-based rotary vane pump (supplied as standard), or an optional oil-free scroll pump. The pump is controlled by a switch on the pump.

There are two main pumping regions in the GCT Premier:

- The source housing.
- The analyzer housing.

The source housing and analyzer housings are fine-pumped respectively by 250 L/s and 70 L/s air-cooled, high-compression turbomolecular pumps. A single oil-cooled rotary fore pump or, optionally, an oil-free scroll fore pump backs both the turbomolecular pumps.

A pneumatically-operated isolation valve allows routine source maintenance to be performed without breaking the analyzer vacuum. This valve is controlled via the MassLynx software.

The source and analyzer vacuums are monitored by Penning gauges, which stop instrument operation if vacuum loss is detected.

See also: [MassLynx control system on page 1-8](#) and [Monitoring the instrument vacuum on page B-9](#).

MassLynx control system

PC based MassLynx software controls the GCT Premier, GC system, and the autosampler. A second PC—the "embedded" PC (EPC)—occupies a place next to the GCT Premier. The EPC acquires and processes data and directly controls the instrument's electronics.

The MassLynx PC and EPC communicates via a network link.

Analog inputs allow data acquisition to occur simultaneously with acquired mass spectrum data from optional conventional GC detectors, such as the flame ionization detector and electrochemical detector.

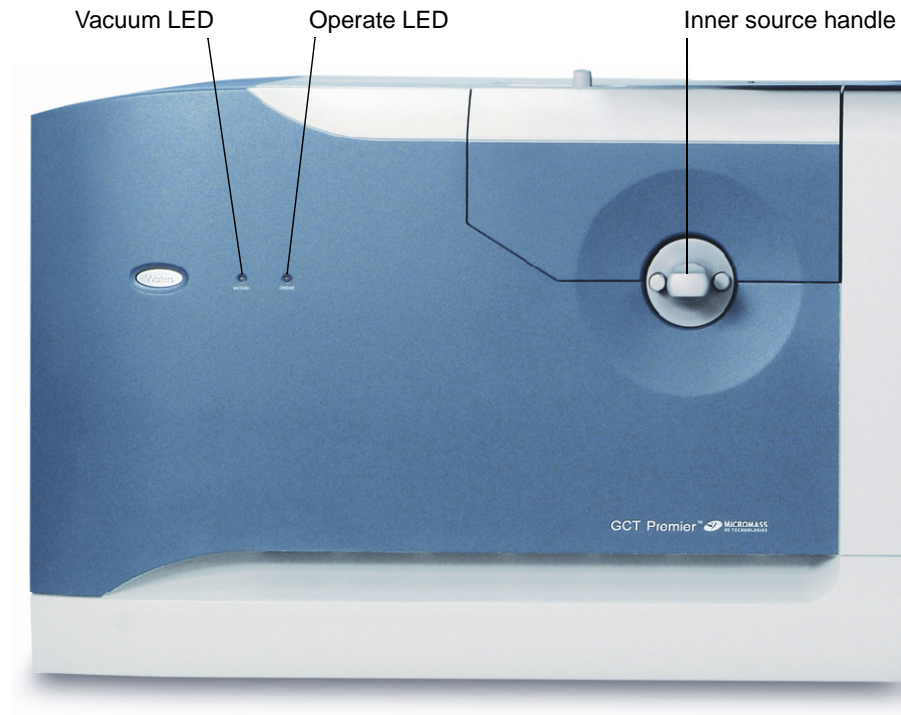
The following processes are performed through the MassLynx software:

- Configuring the GCT Premier.
- Tuning and calibrating the GCT Premier.
- Creating inlet and MS methods that define operating parameters for a run.
- Running samples.
- Monitoring the run.
- Acquiring data.
- Processing data.

See also: The MassLynx User's Guide and MassLynx online help for more information on installing and using the MassLynx software.

Controls and indicators on the instruments' front panel

Front view of the instrument:



Status display

The Vacuum and Operate light-emitting diodes (LEDs) are at the left of the instrument's front panel.

The Vacuum LED indicates the instrument's vacuum status.

See also: [Table titled "Vacuum LED display:" on page 1-10.](#)

The Operate LED indicates the instrument's operational status.

See also: [Table titled "Operate LED display:" on page 1-10](#)

Vacuum LED display:

Status	Vacuum LED indication
Vented	Red
Source vented, analyzer pumped	Yellow
Pumped	Green

Operate LED display:

Status	Operate LED indication
Operate	Green
Standby	Red
Source solvent trip	Yellow

Tip: Any combination of LED indications not shown in these tables can indicate a normal state or fault.

See also:

- [Table titled “Vacuum LED: displays for normal instrument operation” on page E-2.](#)
- [Table titled “Vacuum LED: displays for instrument faults” on page E-5.](#)
- [Contacting Waters on page 11-14.](#)

Controls on the instruments' top panel

The reference reservoir pump valve and reference inlet are located on the top of the instrument.

Reference reservoir pump valve and reference inlet:



Warning: To avoid burn injuries, take care when using the reference inlet; it can be hot.

The reference inlet introduces volatile reference materials for instrument tuning and calibration. Use a septum piercing-syringe to introduce reference material through a septum located in the reference reservoir inlet cap.

See also:

- [Reference reservoir maintenance on page 10-16.](#)
- [Introducing the reference sample on page 3-6.](#)
- [Reference reservoir inlet on page 1-7.](#)

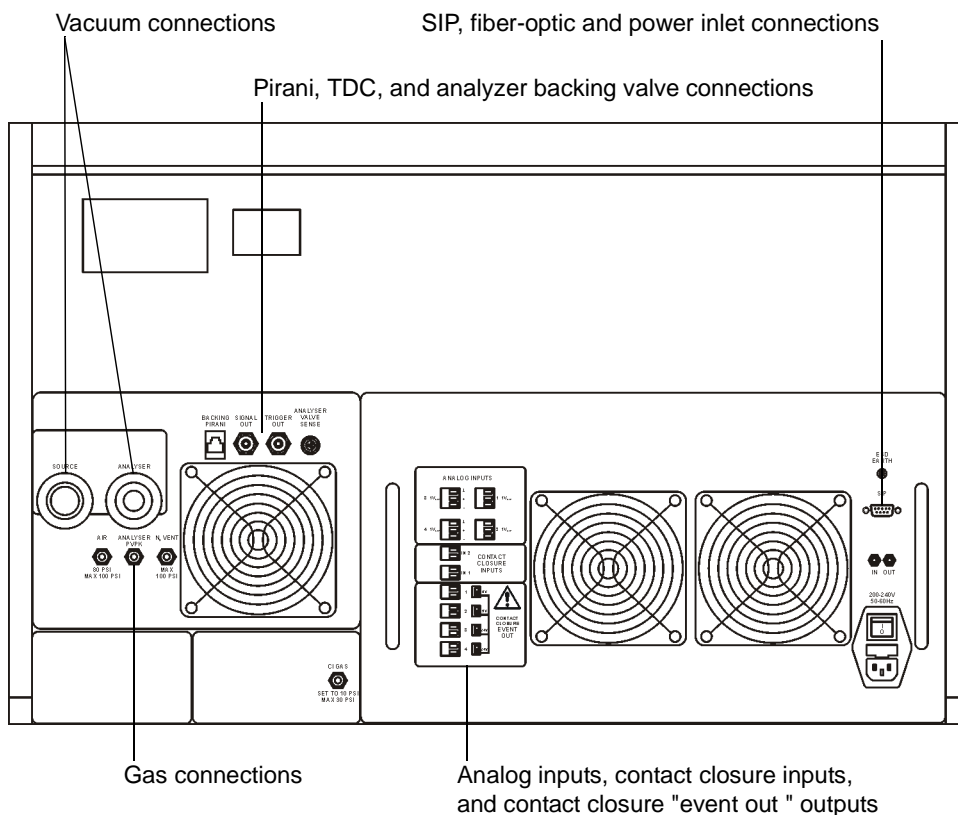
Controls and connectors on instruments' rear panel



Warning: All electrical connections to the rear panel must be separated from hazardous voltages by double or reinforced insulation [safety extra low voltage (SELV)].

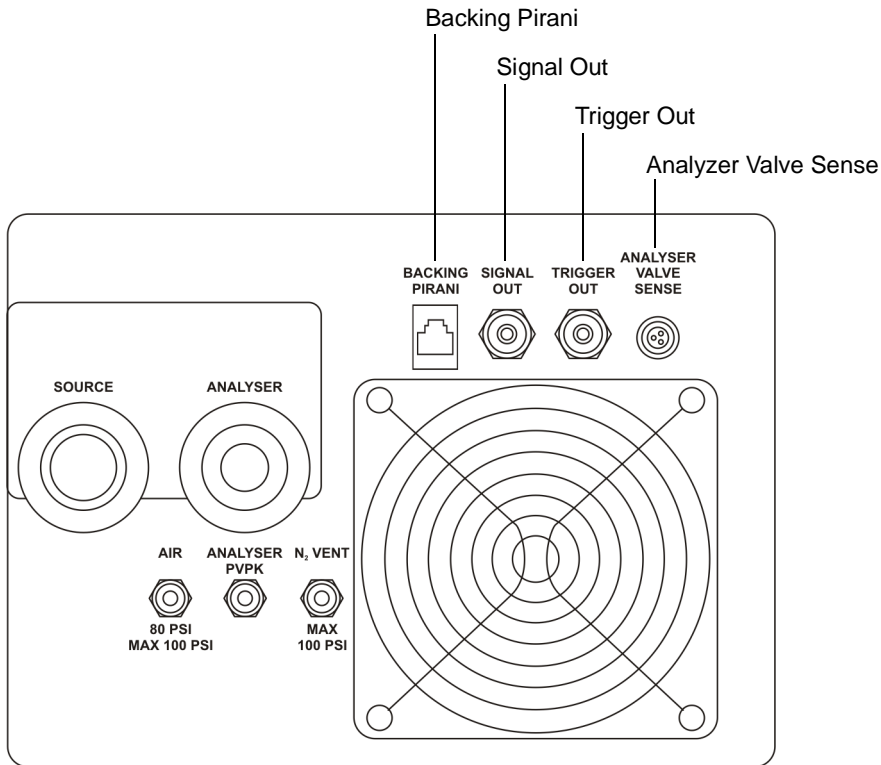
Examples of circuits that are typically SELV include contact closure inputs and outputs for auto-samplers and, FID, TDC, ECD, PID for GC/MS products.

Rear panel:



Signal connections

Pirani, TDC, and analyzer backing valve connectors:



Backing Pirani

Connection to the fore pump Pirani gauge.

Signal Out

Time-to-digital converter (TDC) signal to the EPC.

See also: [Figure titled "GCT Premier system external wiring:" on page D-3.](#)

Trigger Out

TDC trigger signal to the EPC.

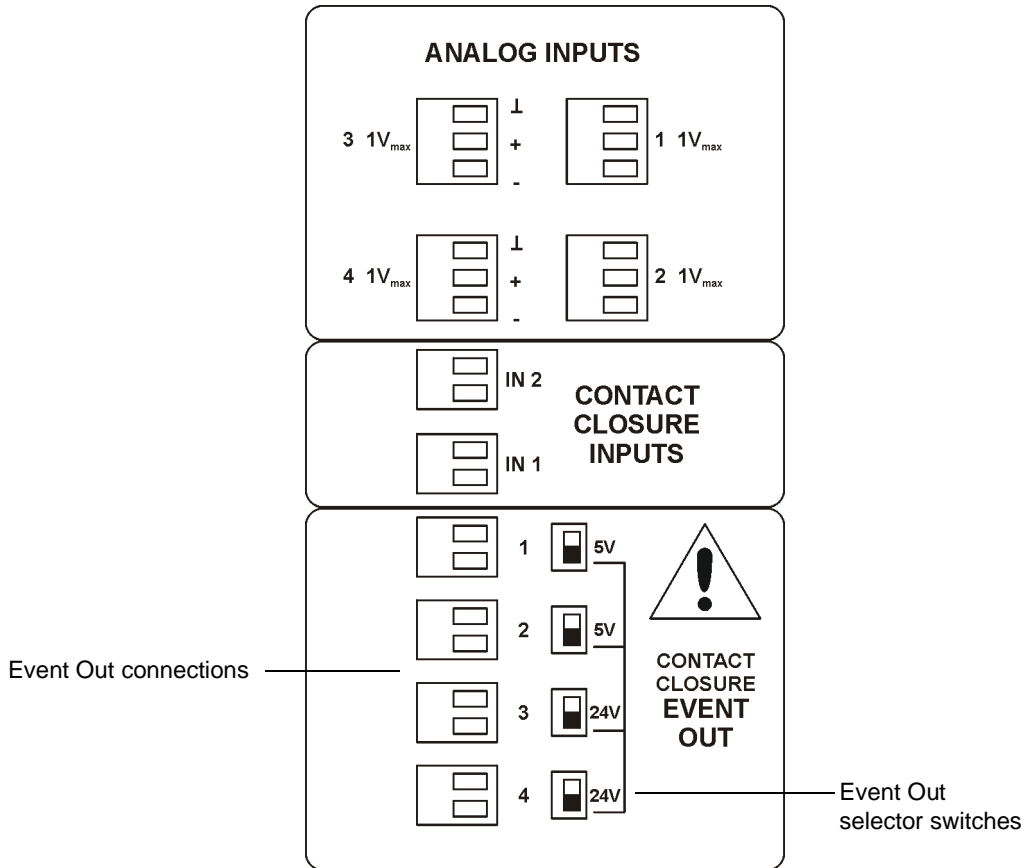
See also: [Figure titled "GCT Premier system external wiring:" on page D-3.](#)

Analyzer Valve Sense

Connection to the PVPK valve on the fore pump.

See also: [GCT Premier system vacuum connections: on page D-4.](#)

Analog Inputs, Contact Closure Inputs, and Contact Closure Event Out outputs:



Analog Inputs



Warning: To avoid electric shock and damage to the instrument, do not apply more than 16 V to any of the analog input connections.

Four analog input channels are available for acquiring simultaneous data, such as a flame ionization detector output. The input differential voltage must not exceed one volt.

See also: [Figure titled “Analog Inputs, Contact Closure Inputs, and Contact Closure Event Out outputs:” on page 1-14](#) and [Acquiring analog data on page 4-10](#).

Contact Closure Inputs

The In1 and In2 inputs allow an external device to start sample acquisition once the device performed its function (typically sample injection).

Normally, the GC is connected to contact closure input In1. When the GC makes an injection it sends a signal via this connection to start the GCT Premier acquiring data.



Warning: To avoid electric shock and damage to the instrument, do not apply more than 5 V to any of the contact closures inputs.

See also: [Figure titled “Analog Inputs, Contact Closure Inputs, and Contact Closure Event Out outputs:” on page 1-14](#).

Contact Closure Event Out



Warning: To avoid electric shock and damage to the instrument, do not apply more than 24 V to any of the Event Out connections.

When the connections are configured as voltage outputs, do not apply any load greater than 250 mA.

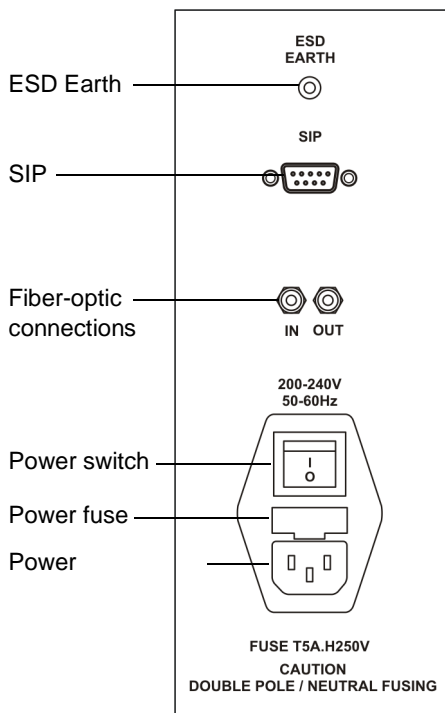
Four Event Out connections, Out1 to Out4, allow various peripherals to be connected to the system.

The Event Out selector switches the outputs, Out1 to Out4, between voltage out and the contact closure. The voltage output of Out1 and Out2 is 5 V, the voltage output of Out3 and Out4 is 24 V.

During a sample run, an event output can be configured to actuate between acquisitions and is used typically to enable an external device to start at the same time the acquisition starts.

See also: [Figure titled “Analog Inputs, Contact Closure Inputs, and Contact Closure Event Out outputs:” on page 1-14.](#)

SIP, fiber-optic and power inlet connections:



ESD Earth

For engineers' use only.

SIP

Scan-in-progress connection to the EPC.

See also: [Figure titled “GCT Premier system external wiring:” on page D-3.](#)

In and Out fiber-optic connections

Blue (In) and gray (Out) fiber optic connections to the EPC.

See also: [Figure titled “GCT Premier system external wiring:” on page D-3.](#)

Power switch

You switch the instrument on and off using this switch.

See also: [Starting-up and Shutting-down the Instrument on page A-1.](#)

Power fuse

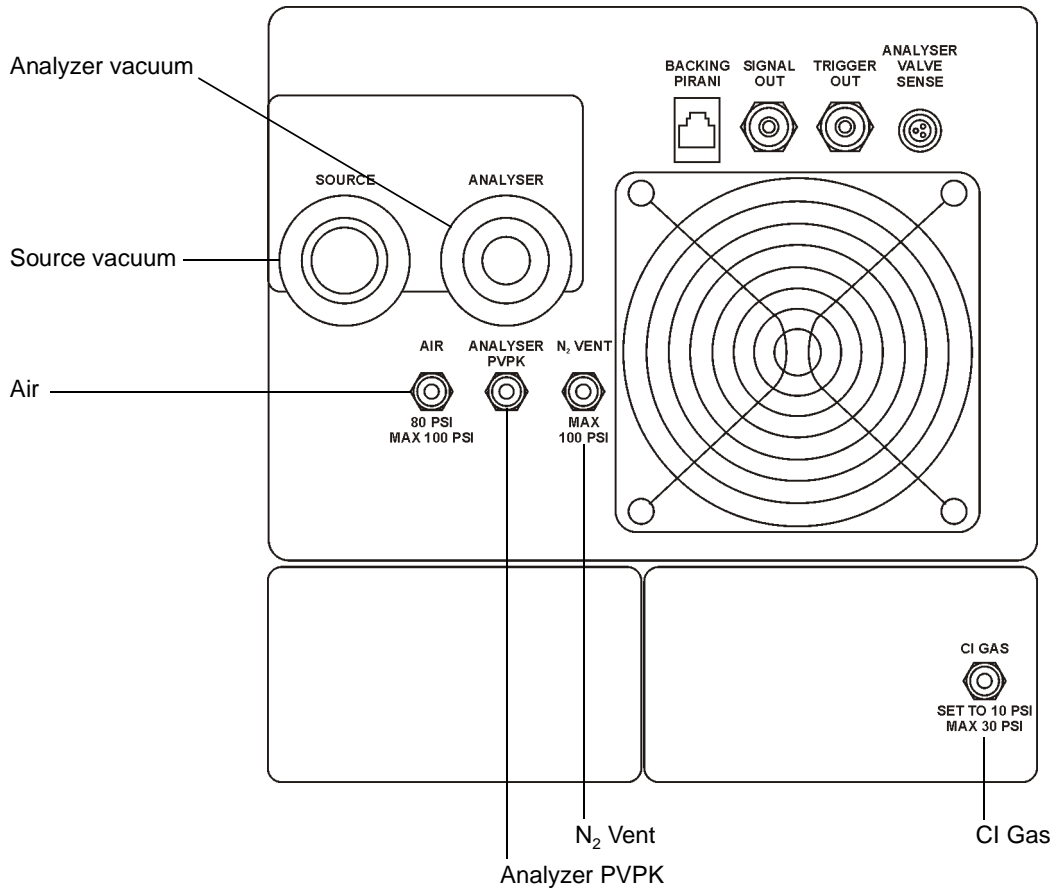
The power fuses are adjacent to the power connection. The fuses are rated at 5 A, 250 V they are time-delay types with high-breaking capacity (T5A.H250V).

Power connection

The power connection for the instrument.

Vacuum and gas inlet connections

Vacuum and gas connections:



Vacuum connections



Warning: To avoid toxic vapors, a suitable exhaust line must be fitted to the fore pump.

There are two vacuum connections to the fore pump; one connection from the analyzer and one from the outer source.

See also: [Figure titled "GCT Premier system vacuum connections:" on page D-4.](#)

Gas inlet connections

CI Gas



Caution: To avoid damage to the instrument, do not exceed a pressure of 30 psi on this connection.

1/8-inch inlet for coupling to CI reagent gas cylinder. Set the CI gas pressure to 10 psi.

Air



Caution: To avoid damage to the instrument, do not exceed a pressure of 100 psi on this connection.

Compressed air inlet for operation of the pneumatic isolation valve and backing line isolation valves. Set the air pressure to 80 psi.

Analyser PVPK

Compressed air connection to the analyzer backing isolation PVPK valve located on the head of the rotary pump.

See also: [Figure titled “GCT Premier system vacuum connections:” on page D-4.](#)

N₂ Vent



Caution: To avoid damage to the instrument, do not exceed a pressure of 100 psi on this connection.

This is an optional connection used to connect a dry inert gas to the instrument. The gas fills the analyzer when the instrument is vented. This prevents water vapor entering the analyzer housing and speeds up subsequent pump down times. Set the inert gas pressure to 5 to 100 psi.

If a suitable dry inert gas is connected to the air inlet connection, this can also be used to supply the gas to the N₂ inlet connection.

See also: [Detector maintenance on page 10-50.](#)

If an inert gas is not connected, the analyzer housing vents to air.

EPC power switch

The EPC power switch is on the front of embedded PC (EPC). The EPC is a separate PC located next to the GCT Premier. The switch is used to power up, power down, and reboot the EPC.

See also: [Figure titled “GCT Premier system external wiring:” on page D-3.](#)

2

Preparing the instrument for EI/CI operation

This chapter describes how to prepare the instrument for operation, including how to install and remove the inner and outer source options for electron ionization (EI) and chemical ionization (CI).

Note: For details about preparing the instrument for FI/FD operation, including how to install and remove the FI/FD inner and outer source options, see [Chapter 9 FI/FD Operation](#).

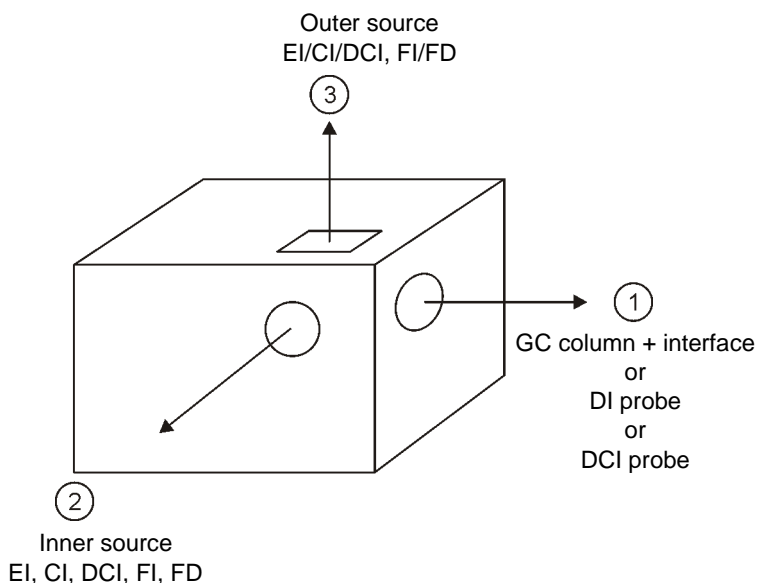
Contents:

Topic	Page
Changing the EI/CI inner source assembly	2-2
Changing the EI/CI outer source assembly	2-6

Changing the EI/CI inner source assembly

Caution: If the GC column is installed, to avoid damaging the GC column and inner source chamber assembly, the source components must be removed in the order shown in the [Figure titled “Removing the source components:”](#) on page 2-2. When installing the source components, they must be installed in the reverse order.

Removing the source components:




Order of fitting or removing source components:

	Fitting	Removal
1	Outer source	GC or, DI probe or, DCI probe
2	Inner source	Inner source
3	GC or, DI probe or, DCI probe	Outer source

Removing the EI/CI inner source

To remove the inner source:

1. In the MassLynx Tune window, click  and confirm that the instrument operate status indicator shows red.

See also: [Figure titled “The MassLynx Tune window:” on page B-3.](#)

2. In the MassLynx Tune window, click Vacuum > Vent Source.
3. Click the Engineer tab.

The Source Turbo value begins to drop steadily. When it drops below 70, you hear the turbo vent valve open. Once the valve opens, the Source Turbo value begins to drop more quickly. Once the source vents, the vacuum status light-emitting diode (LED) shows yellow.

See also: [Status display on page 1-9.](#)



Warning: The GC column and interface are hot. To avoid burn injuries, take care when retracting the GC column.



Caution: To avoid damaging the GC column and inner source chamber assembly, the GC column must be allowed to cool to below 100 °C before retracting. When the GC column has been retracted, remove the inner source assembly.

4. Allow the GC column to cool to below 100 °C.
5. Retract the GC column.

See also: [Installing the GC column on page 6-9.](#)

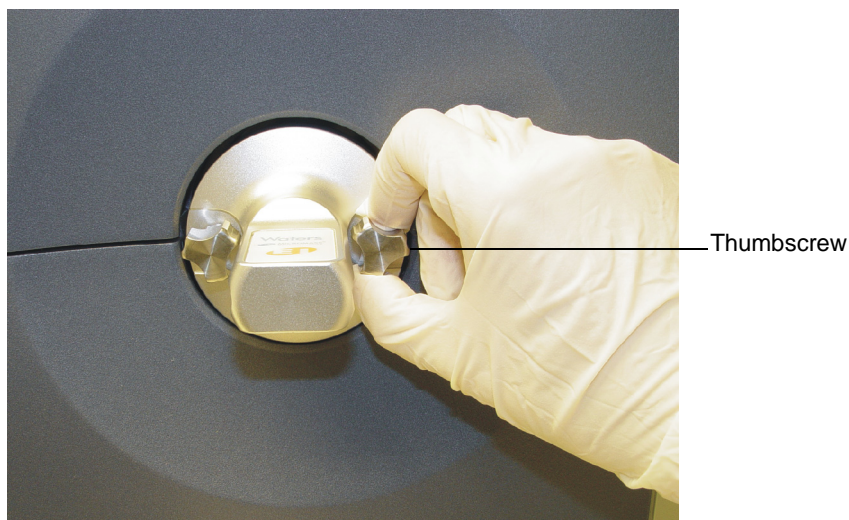
6. In the Tune window Source tab, monitor the Source Temperature readback; allow it to fall below 70°.





Warning: The source components can be contaminated with toxic and biohazardous materials. Wear chemical-resistant, powder-free gloves at all times while handling the components.

7. Unscrew the two thumbscrews that retain the inner source assembly to the outer source.

Inner source:




 **Warning:** The inner source is hot. To avoid burn injuries, take care when withdrawing the inner source assembly.

 **Caution:** When removing the inner source, take great care not to knock the filament or ion chamber assembly, for they can be damaged.

8. Carefully remove the inner source assembly from the outer source.

Installing the EI/CI inner source

 **Caution:** To avoid damaging the GC column and inner source chamber assembly, the GC column must be retracted before installing the inner source assembly.

To install the inner source:

1. Ensure that the GC column is retracted.

See also: [Installing the GC column on page 6-9.](#)

2. Ensure that you are about to install the correct inner source.

See also: [Inner source assembly on page 1-4.](#)

3. On the inner source assembly, verify that the O-rings:

- Sit correctly in the groove.
- Are in good condition.
- Are clean and free from dust.



Caution: To avoid damaging the inner source assembly, be careful not to knock the filament or ion chamber assembly.

4. Carefully fit the inner source assembly on the guide rods and present it centrally to the outer source so that the inner source assembly flange is flush with the outer source cover.

5. Tighten the two thumbscrews that retain the inner source assembly to the outer source.

See also: [Figure titled “Inner source:” on page 2-4.](#)

6. Open the reference reservoir valve.

7. If required, fit the GC column to the instrument.

See also: [Installing the GC column on page 6-9.](#)

8. In the MassLynx Tune window, click Vacuum > Pump Source.
When the pneumatic valve opens, you should hear a small hiss.

9. Ensure that the vacuum LED shows flashing green followed by steady green.

See also: [Status display on page 1-9.](#)

10. When the Source Turbo value in the Turbo Readbacks frame reaches 80 or more, close the reference reservoir valve.

Changing the EI/CI outer source assembly

Removing the EI/CI outer source from the instrument



Warning: The source component can be contaminated with toxic and biohazardous materials. Wear chemical-resistant, powder-free gloves at all times while handling the components.



Warning: The source is hot and can cause burn injuries to the skin. Allow the source to cool before handling it.

To remove the EI/CI outer source:

1. On the MassLynx Tune Window, click Vacuum > Vent Source.
2. Wait for the Vacuum Status LED to display steady yellow.



Warning: To avoid burn injuries, allow the GC column and interface to cool before handling.

3. If fitted, remove the GC column from the instrument.
See also: [Installing the GC interface on page 6-3](#).
4. Move the GC away from the right side of the instrument.
5. Remove the inner source from the outer source.

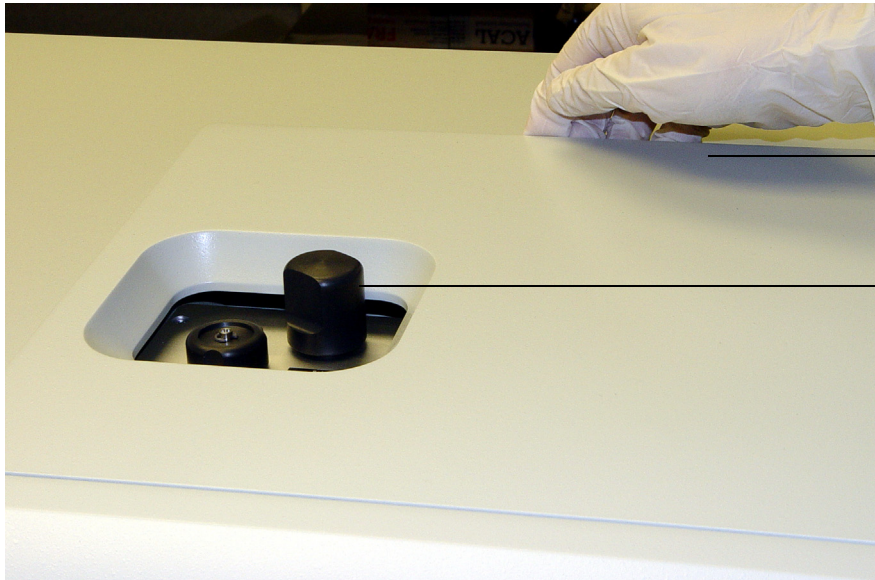
See also: [Removing the EI/CI inner source on page 2-3](#) and [Removing the FI/FD inner source on page 9-13](#).

6. Loosen the two thumbscrews that retains the GC interface, and pull the interface approximately 2 cm back on its support rods.



Warning: To avoid burn injuries, allow the outer source lid assembly to cool before handling it.

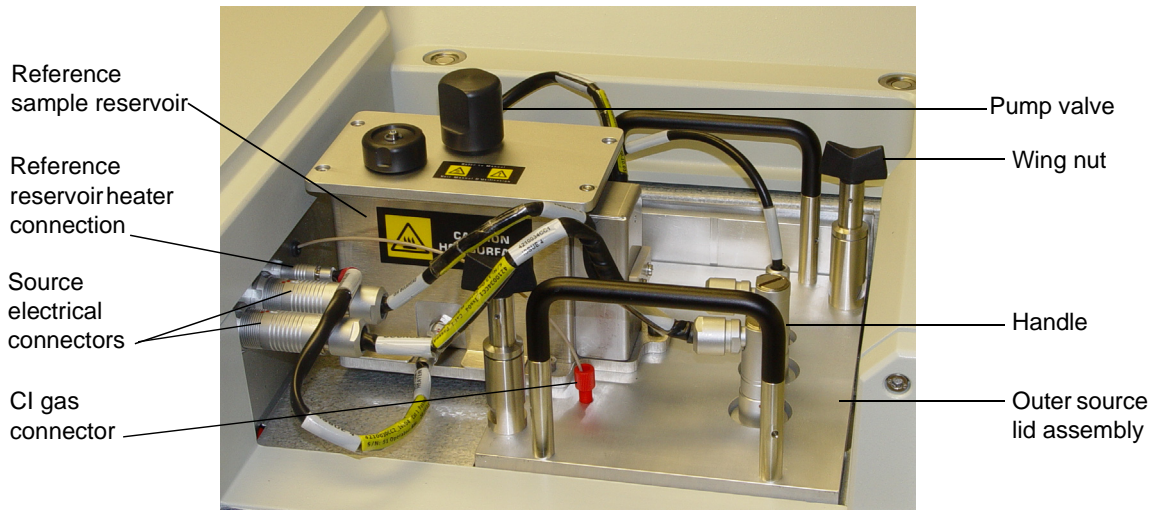
7. Lift the top cover from the back, and pull forward to remove.



Outer source lid assembly cover

Reference reservoir pump valve

Outer source lid assembly:



Tip: A second heater connection is behind the reference reservoir.

8. Disconnect the red CI gas connector.
9. Disconnect the electrical and heater connectors.
10. Unscrew the two wing nuts on the top of the outer source lid assembly.
11. Use the two handles to lift the outer source lid assembly vertically from the outer source enclosure.

Fitting the EI/CI outer source to the instrument

To fit the EI/CI outer source:

1. Carefully lower the outer source lid assembly vertically into the outer source enclosure, using the two studs to ensure correct alignment.
2. Tighten the two wing nuts on the top of the outer source lid assembly.

See also: Figure titled “Outer source lid assembly:” on page 2-8.

3. Connect the red CI gas connector.
4. Connect the heater and electrical connectors.

5. Fit the top cover to the instrument.
6. Fit the inner source.
See also: [Installing the EI/CI inner source on page 2-4.](#)
7. Push the GC interface into place, and tighten the two thumbscrews.
8. Open the reference reservoir valve.
9. If required, fit the GC column to the instrument.
See also: [Installing the GC column on page 6-9.](#)
10. In the MassLynx Tune window, click Vacuum > Pump Source, and confirm that the Vacuum LED shows green.

3

Tuning the Instrument

This chapter describes how to prepare and tune the instrument (that is, adjust the instrument's parameters to achieve maximum performance).

Note:

- Tuning is not the same as instrument calibration, which ensures that the peaks displayed in a spectrum are in their correct locations.
- The figures in this chapter shows indicative parameter values only.

See also: [Chapter 4](#) for details of how to acquire data, [Chapter 5](#) for details of how to calibrate the instrument, and [Appendix B](#) for full details of the Tune window.

Contents:

Topic	Page
Preparing the instrument for operation	3-2
Obtaining an ion beam	3-3
Setting up the instrument automatically	3-8
Tuning the instrument for EI	3-19
Tuning the instrument for best resolution in EI	3-24
Tuning the instrument for positive CI	3-25
Tuning the instrument for negative CI	3-30
Preparing for FI operation	3-33
Managing tune parameters	3-34

Preparing the instrument for operation

Tuning the instrument for operation requires you to perform certain procedures. During the tuning procedure the tuning parameters are optimized so that the optimum resolution and sensitivity can be achieved before acquiring data.

To prepare the instrument for operation:

1. Ensure that the power is connected to the instrument, and the fore pump is turned on.
2. Ensure that the gases are connected to the instrument and do not leak.
3. Ensure the gases are set to the following correct pressures:

Gas pressures:

Gas	Correct pressure for EI	Correct pressure for CI or DCI
Pneumatic gas	80 to 100 psi	80 to 100 psi
Soft vent gas	5 to 100 psi	5 to 100 psi
Helium	90 psi (for GC, if fitted)	90 psi (for GC, if fitted)
CI Gas	Not applicable	10 psi

4. Ensure that the instrument is pumped down (evacuated) with an analyzer pressure of $<3e-6$ mbar.

See also: [Pumping/venting the instrument on page B-8](#) and [Pumping/venting the analyzer on page B-7](#).

5. Ensure that the MCP detector has been conditioned.


See also: [Detector maintenance on page 10-50](#).


6. Ensure that the source is pumped down.

See also: [Pumping/venting the source on page B-8](#).

Obtaining an ion beam


To obtain an ion beam:

1. In the MassLynx Tune window, click .
2. In the TDC Settings dialog box, clear the Low Mass Cut-Off check box.
See also: [Low Mass Cut-Off on page B-16](#).

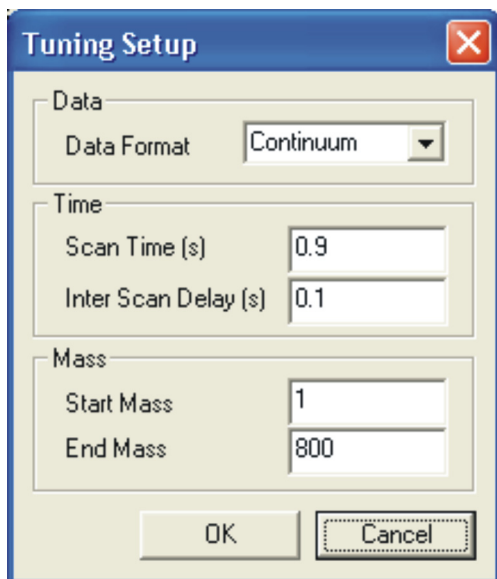
3. In the Tune window, click .
Confirm that the instrument status indicator displays green.
4. In the EI Tune tab, type the recommended starting parameter values shown in the table below, or specify previously saved values.

Recommended Tuning starting values:

Parameter	Recommended EI value	Recommended CI value
MCP Voltage	2700	2700
Source Temperature	200	120 to 150
Electron Energy	70	70
Trap Current	100	Not applicable
Ion Repeller	1	Not applicable
Emission current	Not applicable	250
Beam Steering	0	0
Focus 1	70	70
Focus 2	14	14
Focus 3	40	40

5. Click View > Peak Display 2.
Result: The second peak display appears.
6. Click .

Tuning Setup dialog box:

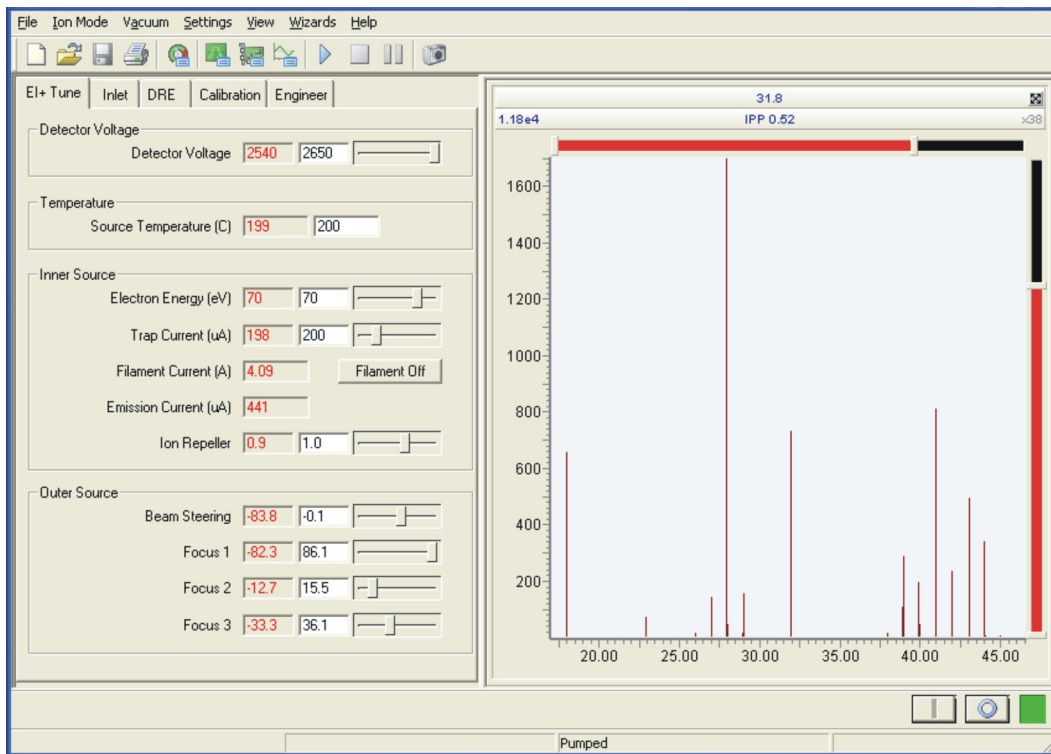


7. In the Tuning Setup dialog box, Time pane, set the Scan Time (s) to 0.9.
8. In the Time pane, set the Inter Scan Delay (s) to 0.1.
9. In the Data Format pane, click Continuum in the Data Format list.
10. Click OK.
11. Right-click in the Peak Display and click Intensity > Normalise Data.

Result: The display is scaled to the value of the intensity of the highest peak.

You should be able to identify some low mass ions on the peak display of the Tune window, such as nitrogen and water, as shown in the following figure.

Tune window showing low mass peaks:



The low mass peaks are displayed as:

- Carbon dioxide = m/z 44
- Nitrogen = m/z 28
- Water = m/z 18

Introducing the reference sample

To introduce the reference sample:

1. Ensure the reference reservoir valve is closed.
2. Use a septum-piercing syringe, inject approximately 0.2 μL of heptacosyl into the reference reservoir inlet cap.

Introducing the reference compound into the reference reservoir inlet cap:

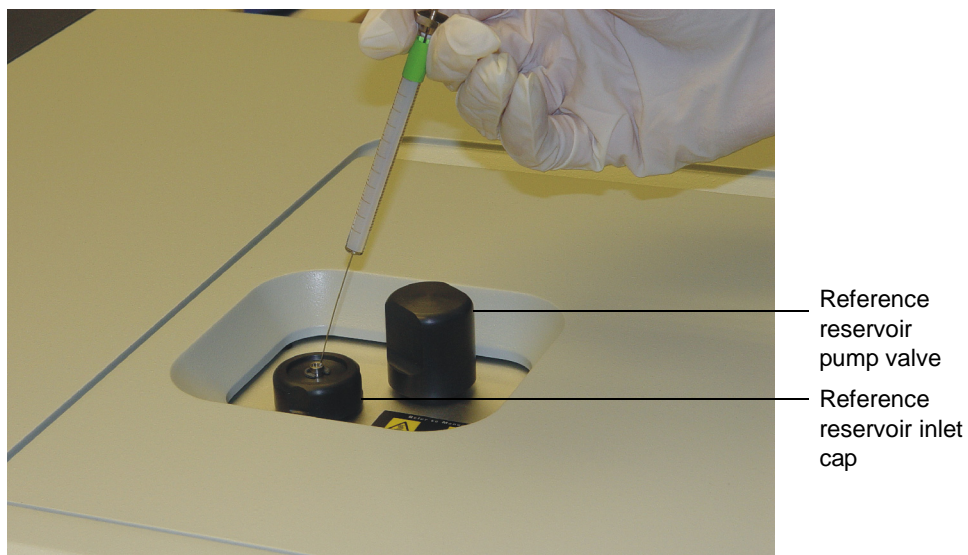
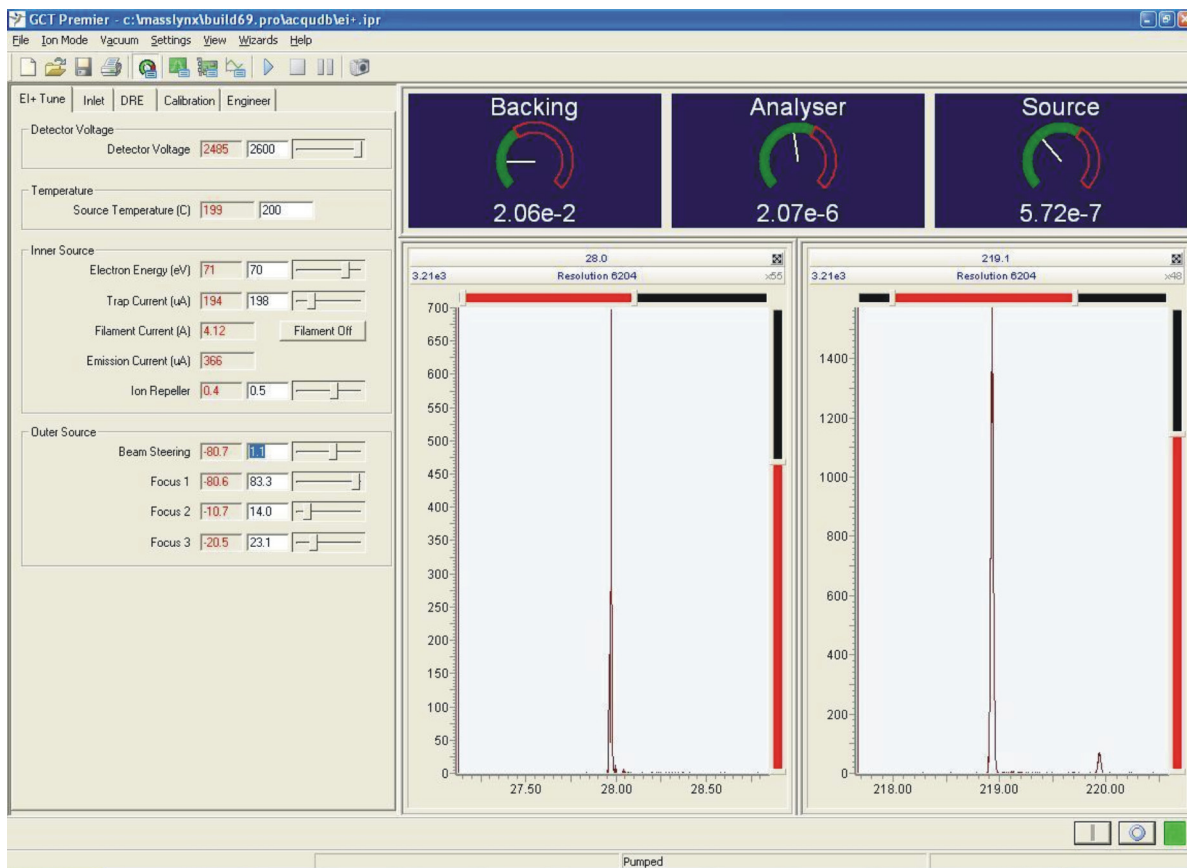


Figure titled “Tune window with peak display:” on page 3-7 shows the expected signal in EI mode from the reference material at m/z 219 for heptacosyl.

Tune window with peak display:



The peak at m/z 28 is air (nitrogen). The peak at m/z 219 is the heptacosane reference sample. The aim of tuning the instrument is to maximize the sample signal while keeping the nitrogen peak as small as possible. To accomplish this, you can increase or decrease the beam steering and focus lens values.

See also: [General considerations for tuning on page G-6.](#)

As tuning improves, the reference mass increases in intensity. Reduce the intensity of the reference sample by slowly opening the reference reservoir valve. Aim to achieve about 1000 ion counts per second for the base peak of the sample in continuum mode on the Tune window. This ensures that the level of the sample peak is below the saturation level of the TDC.

The intensity of the signal is highlighted in the example shown in [Figure titled “Tune window with peak display:” on page 3-7](#).

See also: [Effects of saturation on peak shape on page B-51](#).

Setting up the instrument automatically

Using the Instrument Setup Wizard you can automatically setup the instrument so that it is ready to be tuned for accurate mass data acquisition.




Caution: Ensure that the reagent gas is switched off before running the Instrument Setup wizard in CI positive mode when using metri as the tuning compound. If FC70 is used, then the methane reagent gas can be left at the appropriate pressure.

Required material


The following reference compound solutions are recommended:

- For EI - use heptacosane (218.9856 Da) or FC70 (268.9824 Da)
- For CI positive - use metri (284.9949 Da) or FC70 (268.9824 Da)
- For CI negative - use chloropentafluorobenzene and heptacosane (413.9775 Da)
- For FI and FD - use chloropentafluorobenzene (201.9609 Da)

To run the Instrument Setup Wizard:

1. In the MassLynx window instrument shortcut menu, click  to open the Tune window.
2. In the Tune window menu bar, click Ion Mode and then click the required mode.

Note: MassLynx saves tuning parameters separately for each ion mode.

3. Click , to switch the instrument into operate.
4. Use a septum piercing-syringe to inject a suitable reference compound solution which will produce peaks of a known mass.

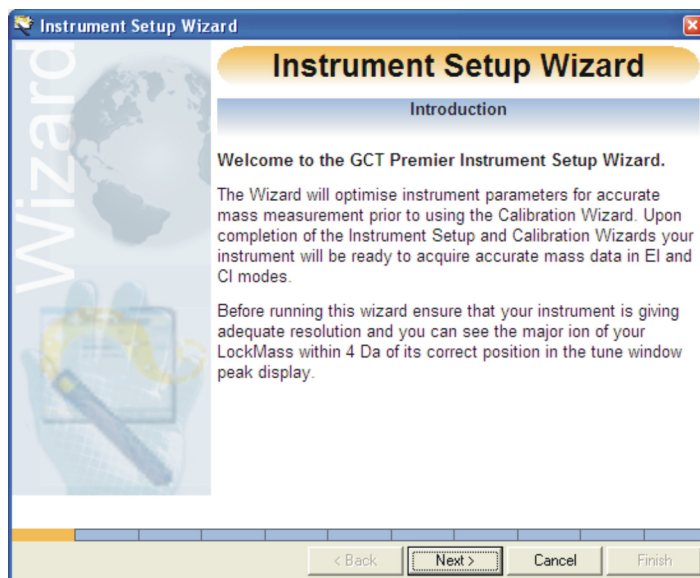
Tip: If there are insufficient ions detected, you may have to inject more reference sample. If there is too much sample and the detector becomes saturated, open the reference reservoir value to release some sample.

Aim to have about 1000 counts per second in continuum.

Note: When the wizard is complete, the infused solution can be used as the lock mass.

5. Aim for a beam with 1000 counts per second in continuum.
6. Ensure you can see a beam in the peak display.
7. Click Wizards > Instrument Wizard Setup.

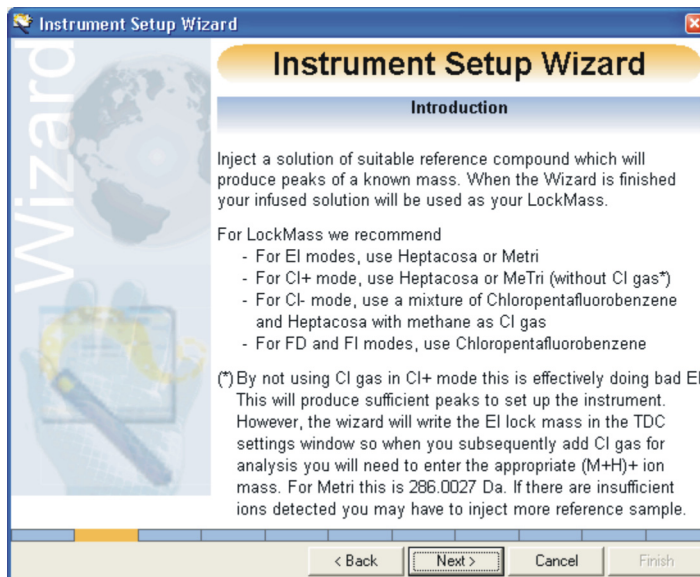
Instrument Setup Wizard Introduction:



8. Click Next.

The Instrument Setup Wizard Introduction window appears.

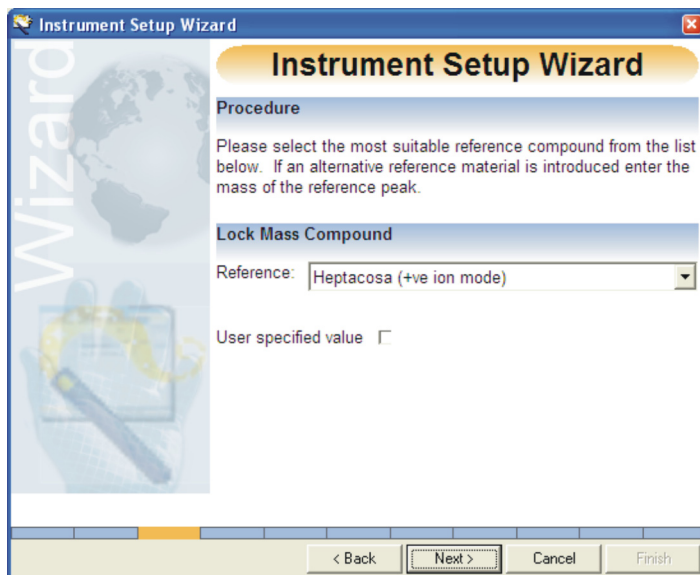
Instrument Setup Wizard Introduction:



9. Click Next.

The Instrument Setup Wizard Lock mass Compound window appears.

Instrument Setup Wizard Lock Mass Compound:



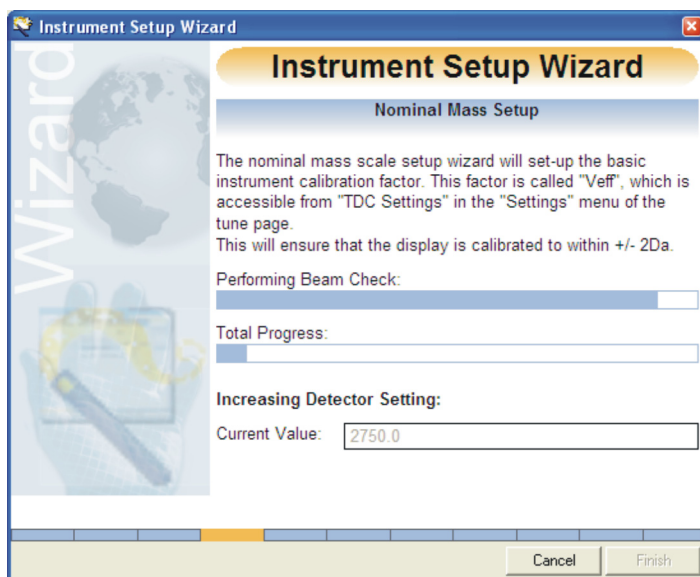
10. From the Reference list, click the reference compound file for the sample that has been injected into the instrument.

Note: Click the User specified value check box to tune using a specific mass value.

11. Click Next.

The Instrument Setup Wizard Nominal Mass window appears.

Instrument Setup Wizard Nominal Mass:



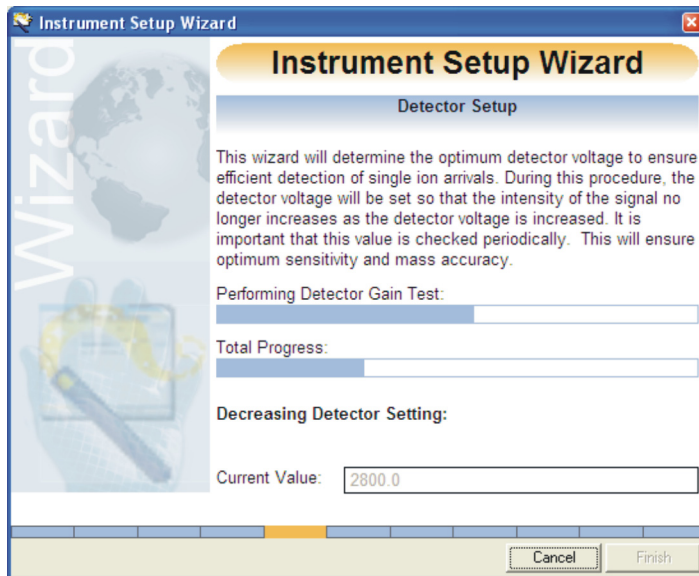
The wizard automatically calculates the Veff setting required to adjust the peak position so that the correct nominal mass is assigned. The Veff value is calculated so that the nominal mass range is set up. For example, the masses are correct to within +/- 0.5 Da.

The Veff factor can also be set-up in the TDC Settings dialog box.

See also: [Configuring the TDC settings on page B-11](#) and [Manually setting the Veff correctly on page B-47](#).

Once assigned the Instrument Setup Wizard Detector Setup window appears.

Instrument Setup Wizard Detector Setup:



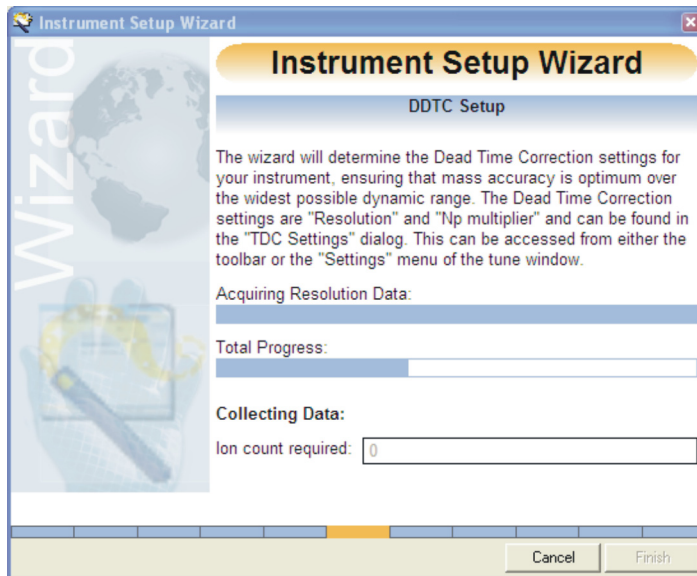
The wizard automatically calculates the detector setting that is required to detect more than 90% of all the ions produced.

During the procedure the detector voltage is set so that the intensity of the signal no longer increases as the detector voltage is increased. To ensure optimum sensitivity and mass accuracy, check this value periodically.

See also: [Setting the optimum gain for the detector on page 10-52.](#)

When completed, the Instrument Setup Wizard DDTC Setup window appears.

Instrument Setup Wizard DDTC Setup:

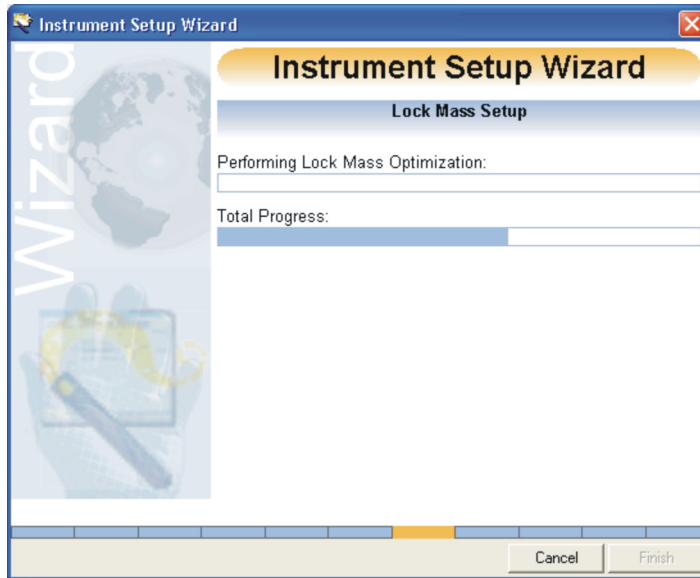


The wizard automatically determines the resolution and sets the Np Multiplier setting to compensate for dead time.

See also: [Setting up resolution for dead time correction manually on page B-48](#) and [Dead time correction on page B-49](#).

When completed the Instrument Setup Wizard Lock mass Setup window appears.

Instrument Setup Wizard Lock Mass Setup:



The wizard automatically sets the Lock Mass by checking a specific mass and intensity. To achieve the best mass accuracy a lock mass is necessary to correct the mass scale for the thermal drift factors.

See also: [Configuring the TDC settings on page B-11](#) and [Lock mass correction on page 5-3](#).

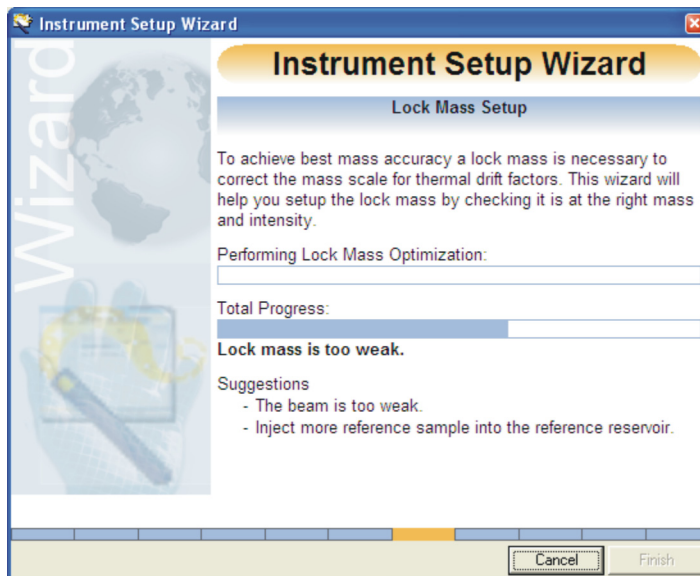
If the Lock Mass intensity is too high, open the reference reservoir valve to reduce the intensity

Lock Mass is too intense:



If the Lock Mass is too low, inject more reference sample into the reference reservoir to increase the intensity of the signal.

Lock Mass is too weak:



The Instrument Setup Wizard Lock Mass check is complete window appears.

Instrument Setup Wizard Lock Mass check is complete:



12. Ensure that the reference reservoir valve is closed.

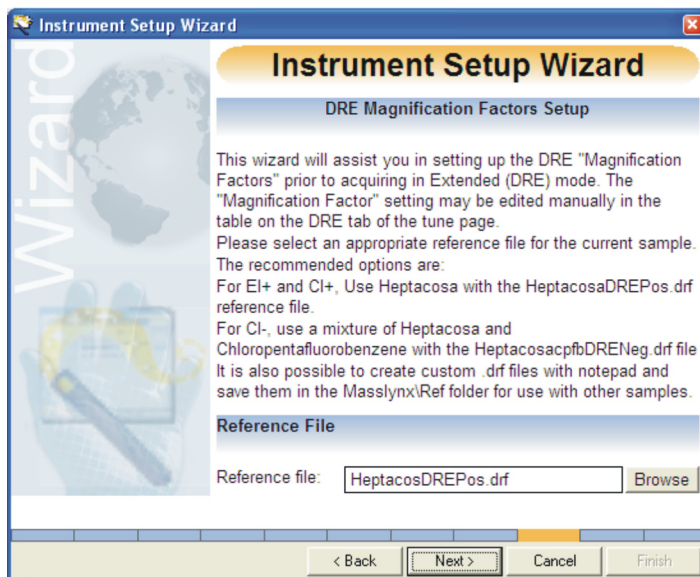
If you have injected more sample or backed sample out using the reference reservoir valve, the signal can overshoot and appear too intense or too weak.

13. Click Back to repeat the Lock Mass intensity check.

14. Click Next.

The Instrument Setup Wizard DRE Magnification Factor Setup window appears.

Instrument Setup DRE Magnification Factor Setup:



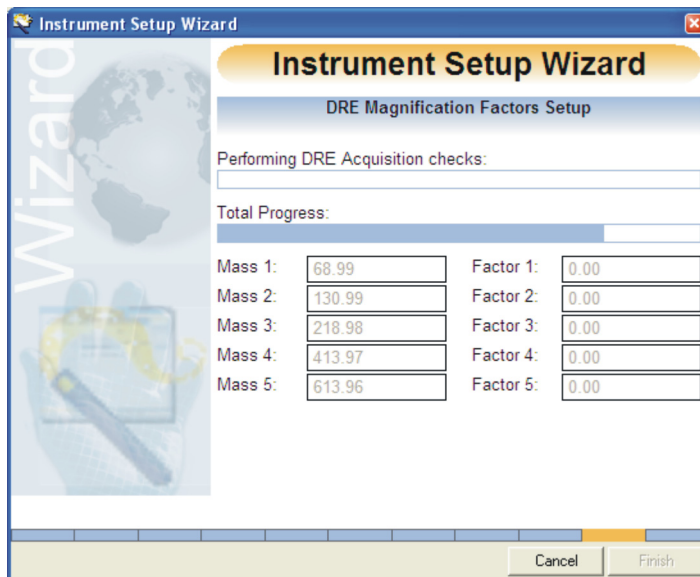
15. From the Reference File list, click the reference compound file for the sample that has been injected into the instrument.

Note: Ensure you are using the correct file for the reference compound sample and the ion mode selected.

16. Click Next.

The Instrument Setup Wizard then sets up the DRE magnification factors.

DRE Magnification Factor:



The screenshot shows a software window titled "Instrument Setup Wizard" with a sub-tab "DRE Magnification Factors Setup". The window displays a progress bar and a table of magnification factors for five different mass values. The magnification factors are all set to 0.00.

Mass	Factor
Mass 1: 68.99	Factor 1: 0.00
Mass 2: 130.99	Factor 2: 0.00
Mass 3: 218.98	Factor 3: 0.00
Mass 4: 413.97	Factor 4: 0.00
Mass 5: 613.96	Factor 5: 0.00

The wizard automatically determines several magnification factors across the mass range required to enable Dynamic Range Enhancement.

The DRE Magnification Factor value can be edited manually on the Tune window DRE tab.

See also: [The DRE tab on page B-43.](#)

Note: If you change any Tune window Source or Engineer parameters, the DRE magnification factors will change and you will have to repeat the Instrument Setup Wizard.

The Instrument Setup Wizard is now complete and the Instrument Setup Complete window appears.

Instrument Setup Complete:



The screenshot shows the 'Instrument Setup Wizard' dialog box with the title 'Instrument Setup Complete'. The window is divided into two main sections: 'Acquisition Settings' and 'Instrument Settings'. Under 'Acquisition Settings', there are two input fields: 'Nominal Mass (Veff):' with the value '5323.1' and 'DDTC (Resolution):' with the value '7000'. Under 'Instrument Settings', there are two input fields: 'Detector:' with the value '2700' and 'Lock Mass:' with the value '502'. At the bottom of the dialog, there are three buttons: '< Back', 'Next >', and 'Finish'. The 'Finish' button is highlighted, indicating it is the next step.

17. Click Finish

Tuning the instrument for EI

Use the following methods to introduce samples in EI mode: DI probe, the DCI probe, the gas chromatograph, or the reference inlet system.

Tip: The example procedure in this chapter specifies tris (trifluoromethyl) triazine as the infused sample. However, you can tune the instrument using a different compound. Different parameter settings may be required from these provided in this chapter.

To tune the instrument for EI operation:

1. In the MassLynx Tune window, click the EI Tune tab.
2. In the TDC Settings dialog box, set the Veff value to display the correct mass in the peak display, unless it has been set already by the Instrument Setup wizard.

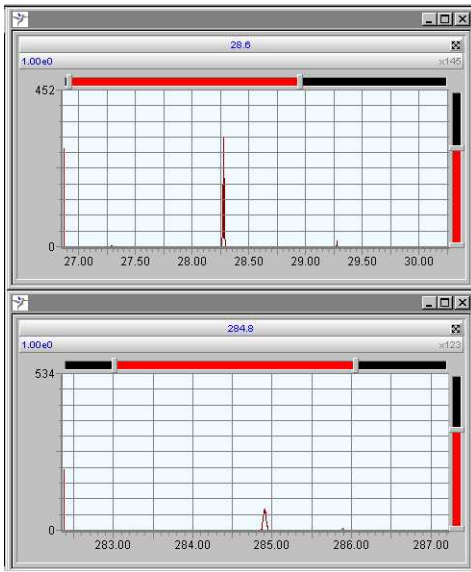
See also: [Manually setting the Veff correctly on page B-47.](#)

3. In the EI Tune tab, adjust beam steering in increments of 0.1 V.

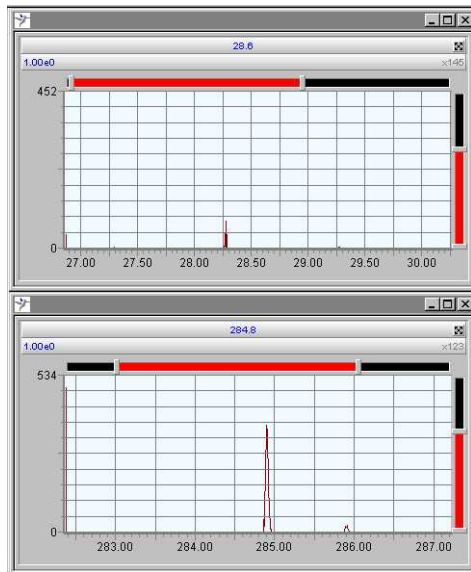
Result: The intensity of the air peak changes.

4. Optimize beam steering so that the peak at m/z 28 is minimized while the peak at m/z 285 is maintained.

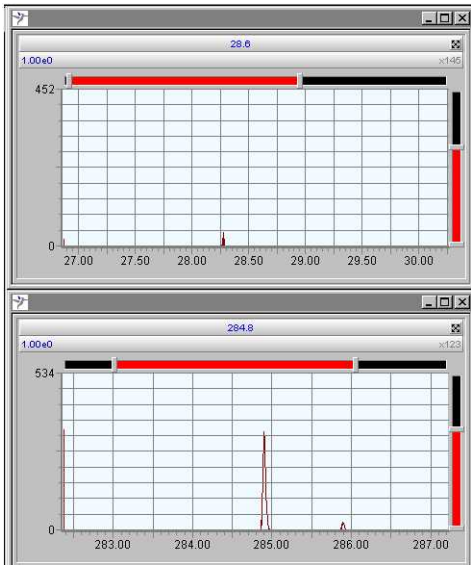
Steering voltage -3.4 V:



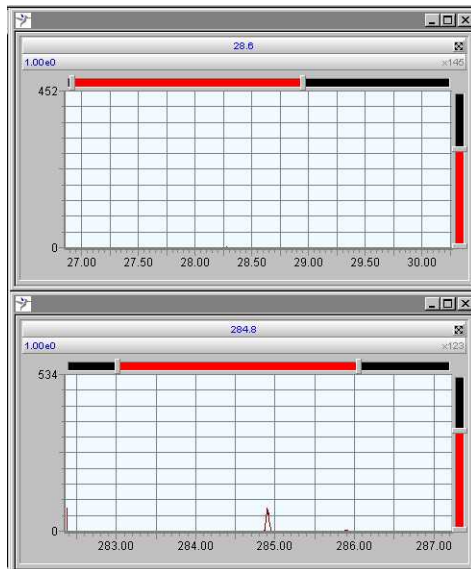
Steering Voltage -1.5 V:



Steering voltage -0.5 V:



Steering Voltage +1.0 V:



As beam steering is increased from -3.4 V, the air peak at m/z 28 drops in intensity. However, the sample peak reaches a maximum at around -0.5 V, and then drops off again as the voltage is increased to +1.0 V. In this example, a steering voltage of about -0.5 V is optimum.

5. Optimize the focus 2 lens to maximize the intensity of the peak at m/z 285.
6. Repeat this for focus 1 and focus 3 lenses.
7. In the EI Tune tab, set the Ion Repeller to optimize the size of peak at m/z 285.

Tips:

- Tuning the focus 1, focus 2, focus 3, and ion repeller can affect the beam steering value.
- If the ion repeller voltage reaches 5 V, then the ion repeller might require cleaning.

See also: [EI/CI outer source maintenance on page 10-35.](#)

8. Continue to tune the instrument by repeating steps 2 through 5 until you maximize the intensity of the signal at m/z 285.

Note: Typically the air signal at m/z 28 should be less than 2000 counts per second for a system with a GC attached and less than 400 counts per second for a system with an insertion lock or a blanking flange fitted.

When you maximize the sensitivity, enable low mass cut-off, which cuts off all masses below a specific mass by preventing them from entering the analyzer.

See also: [Low Mass Cut-Off on page B-16.](#)

It is useful to be able to see the air peak (for example, in the previous tuning step) and particularly useful to check for air leakage. When you are satisfied that there are no leaks, you can use the low mass cut off to remove the air peaks (or CI gas reagent ions at low mass in CI+ mode).

Doing so prolongs the life of the MCP detector as it is not exposed to a constant high ion current. You can repeat [step 3 through 6](#) using the low mass cut-off to further improve the sensitivity for an analysis.

General tips for tuning

A strong beam (more than 2000 counts per second in continuum) can become saturated and distorted due to the dead time effect. Aim for 1000 counts per second in continuum for your lock mass.

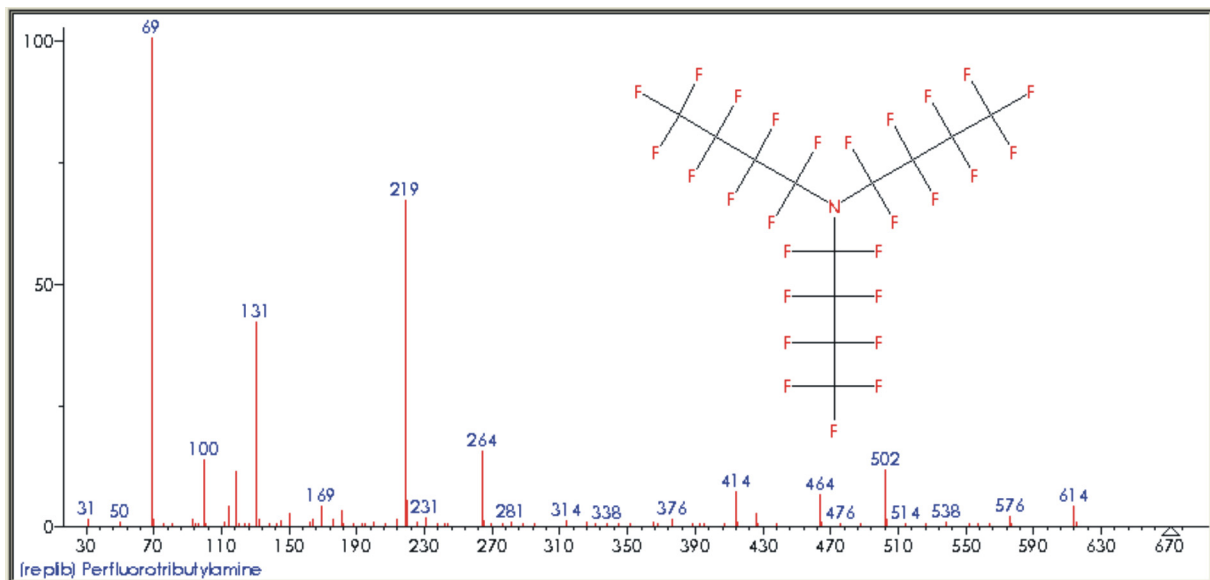
See also:

- [General considerations for tuning on page G-6.](#)
- [Dead time correction on page B-49](#)
- [Effects of saturation on peak shape on page B-51.](#)

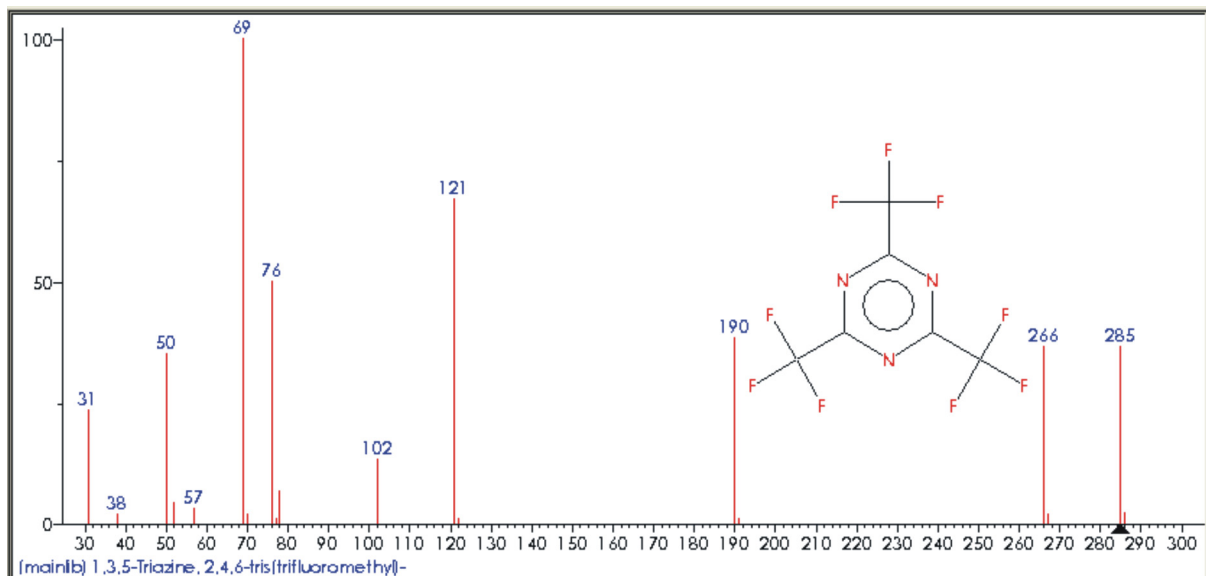
Tip: To improve library matching, adjust the steering parameters until the spectrum of the reference compound closely matches the library spectrum relative intensities at low and high mass.

For example, see [Figure titled “Heptacosyl library spectrum:” on page 3-23](#) for heptacosyl and [Figure titled “Tris\(trifluoromethyl\) triazine library spectrum:” on page 3-24](#) for tri (trifluoromethyl) triazine.

Heptacosyl library spectrum:



Tris(trifluoromethyl) triazine library spectrum:



9. When tuning is complete, calibrate the instrument.

See also: [Calibration on page 5-1](#).

Tuning the instrument for best resolution in EI

Resolution is defined as

$$\text{resolution} = \frac{\text{mass}}{\text{FWHM}}$$

where FWHM is Full Width Half Maximum of the peak. Resolution for the peak at 614 Da of heptacosane should be equal to or more than 7000 counts per second on the GCT Premier.

To tune for best resolution adjust the pusher bias and grid 2 parameters which are found on the Engineers tab.

Note: When using continuum mode, the resolution is shown above the peak display. The resolution shown on the peak display is the resolution of the base peak, even when the base peak is not visible in the peak display window.

Tuning the instrument for positive CI

Use the following methods to introduce samples in CI mode: DI probe, the DCI probe, the gas chromatograph, or the reference inlet system.

In CI mode, you can introduce reference samples in one of the following ways:

- DI probe
- DCI probe
- GC
- Reference inlet

The instrument uses one of the following reagent gases:

- Methane
- Ammonia
- Isobutane



Warning: Methane is extremely flammable. When the instrument operates in chemical ionization mode (CI), which involves using of methane, you must safely vent the effluent from the rotary pump. Verify that all fittings are leak-tight and avoid naked flames.



Warning: Ammonia is a hazardous flammable gas. When the instrument operates in chemical ionization mode (CI), which involves using ammonia, you must safely vent the effluent from the rotary pump. Verify that all fittings are leak-tight.



Warning: Isobutane is extremely flammable. When the instrument operates in chemical ionization mode (CI), which involves using isobutane, you must safely vent the effluent from the rotary pump. Verify all fittings are leak-tight and avoid naked flames.

Setting up and purging the CI gas lines

Before introducing the CI reagent gas, the internal CI reagent gas lines must be purged of air.

To set up and purge the CI gas line:

1. Ensure the CI inner source is correctly installed.
See also: [Installing the EI/CI inner source on page 2-4.](#)
2. Ensure all the CI gas bottle supply connections are free of leaks.
See also: To location the CI gas connection, see [Gas inlet connections on page 1-19.](#)
3. In the Tune window, click Ion Mode > CI+.
4. Ensure a cylinder of methane (or other CI gas) is connected to the CI gas inlet on the rear panel of the instrument.
5. Ensure there are no leaks on the CI gas line connections.
See also: [Inspecting the CI gas line for leaks on page 10-48.](#)
6. Ensure the main outlet valve on the cylinder is closed.
7. In the Tune window Inlet tab, click CI Purge.
The CI line is now be evacuated. If the backing line pressure does not recover, look for leaks in the internal or external CI inlet or pump lines.

Introducing CI reagent gas

To introduce the CI reagent gas:

1. In the Tune window Inlet tab, ensure that the CI Gas check box is cleared so that the CI gas valve is closed.
2. Open the cylinder and set the methane pressure to 10 psi (0.68 bar).
3. In the Tune window Inlet tab click CI Gas.
4. Monitor the source pressure on the Vacuum Monitor menu and increase the CI flow valve slider to achieve a source pressure of 1 to 2 e-4 mbar.
5. In the Engineer tab, reset the solvent trip level.
Result: The filament is turned on at this increased pressure.

Setting up for CI tuning

To setup for CI tuning:

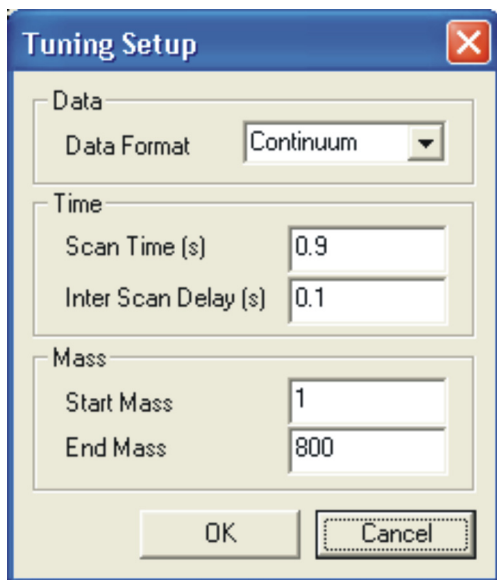
1. Ensure that the instrument is pumped down (evacuated) with an analyser pressure of less than $3\text{e-}6$ mbar ($3\text{e-}7$ kPa, $4.35\text{e-}8$ psi) and that the MCP detector has been conditioned.

See also: [Detector maintenance on page 10-50.](#)

Tip: MassLynx saves tuning parameters separately for each ion mode. From the Tune window, ensure that the correct parameter values appear for Inlet tab, Engineer tab, and TDC Settings dialog box.

2. Ensure you have either a GC interface fitted or a probe is inserted into the source. If not, CI gas will leak out of the hole and you will not be able to optimize even with the maximum CI gas flow.
3. In the Tune window, click Settings > Tuning Setup.

Tuning Setup dialog box:



4. In the Tuning Setup dialog box, specify the parameter values shown in [Figure titled "Tuning Setup dialog box:" on page 3-4.](#)
5. Click OK.

6. Right-click in the peak display and click Intensity > Normalize Data.
Result: The peak display is scaled to the value of the intensity of the highest peak.

Tuning CI gas peaks

To tune CI gas peaks:

1. From the Tune window, clear Low Mass Cut Off check box in the TDC Settings dialog box.
2. In the CI Tune tab, type the recommended starting parameter values for tuning, or specify previously saved parameters.

See also: [Table titled “Recommended Tuning starting values:” on page 3-3.](#)

3. Monitor the CI gas ions.

Tip: For example, if you are using methane, note that methane peaks are viewable at m/z 17, 29 and 41.

4. Increase the flow of CI gas until you achieve the required CI conditions.

Tip: In the peak display, m/z 16 should almost disappear, and the intensities of peaks at m/z 17 and 29 are optimized.

Obtaining a beam in positive CI mode

As a prerequisite to obtaining a beam in positive CI mode, you must perform these tasks:

- Setup and purge CI gas line (see [Setting up and purging the CI gas lines on page 3-26](#)).
- Introduce the reagent gas (see [Introducing CI reagent gas on page 3-26](#)).
- CI tuning setup (see [Setting up for CI tuning on page 3-27](#)).
- Introduce the reference sample (see [Introducing the reference sample on page 3-6](#)).

To obtain a beam in positive CI mode:

1. In the Tune window CI Tune tab, enter the recommended starting parameter values, or specify previously saved parameters.

See also: [Table titled “Recommended Tuning starting values:” on page 3-3.](#)

2. Ensure the Limit Rhenium Filament Current check box is selected.

Tip: The rhenium filament is provided with the instrument. However, if you have fitted a Tungsten filament, then ensure the Limit Rhenium Filament Current check box is cleared.

3. Click .

4. Set the Emission Current to 250 μA .
5. In the TDC Settings dialog box, set the V_{eff} value to display the correct mass in the peak display or run the Instrument Setup wizard for this to be completed automatically for you.

See also:

- [Setting up nominal mass on page B-47.](#)
- [Setting up the instrument automatically on page 3-8.](#)

6. While monitoring the peak from methane C_2H_5^+ at 29 Da, tune the following parameters for maximum intensity for the $(\text{MH})^+$ (m/z 286) ion of tris (trifluoromethyl) triazine.
7. Adjust the beam steering in increments of 0.1 V to minimize the low mass peaks, while maintaining good reference material sensitivity.
8. Optimize the beam steering so that the peak at m/z 29 is minimized while the peak at m/z 286 is retained.
9. Optimize the focus 2 value to maximize the intensity of the peak at m/z 286.
10. Optimize the focus 1 and focus 3 values.

Note: Tuning the focus 1, focus 2, focus 3 can affect the beam steering value.

11. Use the low mass cut-off (LMC) to eliminate the m/z 29 peak and tune for maximum sensitivity on the m/z 286 peak.

See also: [Low Mass Cut-Off on page B-16.](#)

12. Continue to tune the instrument by repeating [step 7 through 10](#) until you maximize the intensity of the peak at m/z 286.

Tips:

- The near-absence of m/z 285 indicates good CI conditions.
- It might be necessary to adjust the level of the reference compound to fall below saturation of the TDC.

See also: [Effects of saturation on peak shape on page B-51.](#)

To adjust the level of reference compound, open the pump-out valve on the reference reservoir slowly. The level of the reference compound should be approximately 1000 counts per second in continuum mode.

See Also: [General considerations for tuning on page G-6.](#)

13. To obtain the best intensity of the 286 ion, retune these parameters in the Tune window, CI Tune tab, Beam Steering, Focus1, Focus2, and Focus3.
14. If required, also adjust the CI gas flow to obtain best sensitivity.

When tuning is complete, calibrate the instrument.

See also:

- [Calibration on page 5-1.](#)
- [Dead time correction on page B-49](#)
- [Effects of saturation on peak shape on page B-51.](#)

Turning off CI gas

To turn off CI gas

In the Tune window Inlet tab, clear the CI Gas check box.

Requirement: At the end of CI operation, the rotary pump must be gas ballasted for 30 minutes.

See also: [Gas-ballasting the rotary pump on page 10-5.](#)

Tuning the instrument for negative CI

Note: MassLynx saves tuning parameters separately for each ion mode. In the Tune window, ensure that the correct parameter values are set for the Inlet tab, Engineer tab, and TDC Settings dialog box.

During electron ionization (EI), negatively charged ions form in addition to positively charged ions. Normally, the negative ions remain undetected because the ion source and focusing potentials allow only the extraction of positive ions.

There are limitations to the analytical usefulness of negative ions formed by electron ionization. For instance, many organic compounds do not form molecular or quasi-molecular anions under EI conditions but instead provide spectra dominated by structurally insignificant low mass ions (such as CN⁻, F⁻, and Cl⁻). Furthermore, the sensitivity for producing negative ions under EI conditions is several orders of magnitude lower than it is for positive ion production.

In contrast to the conditions of conventional electron ionization, low energy electrons are readily captured by many organic compounds without inducing excessive fragmentation. Consequently, the negative CI ion source has been optimized for generating a large population of electrons with near-thermal (low) energy. Introduction of a reagent gas acts as a moderator for the initially energetic electrons. Anionic reagent gas ions can also be generated for ion formation by ion-molecule reactions.

Tuning for negative CI mode operation

To tune for operation in negative CI mode:

1. Install the CI inner source.

See also: [Changing the EI/CI inner source assembly on page 2-2.](#)

2. In the Tune window, click Ion Mode > CI⁻.




Caution: Take care when changing to negative ion mode after using the GCT in positive ion mode for an extended period of time. Perform the following precautionary procedure before operating the instrument in CI⁻ mode.

To configure for tuning in negative CI⁻ mode:

1. In the Tune window, click the Engineer tab.

Note: Record the Detector voltage and the TOF Flight Tube voltage.

2. Set the TOF Flight Tube value to 0.
3. In the CI Tune tab, set the Detector Voltage to 0.

4. In the Tune window, click .
5. Increase the detector and the TOF flight tube voltages to the previously determined operating voltage over a period of 20 seconds. Doing so minimizes the risk of discharge and possible damage to the detector.

Purging the CI gas lines

Before introducing the CI reagent gas, the internal gas lines must be purged of air.

See also: [Setting up and purging the CI gas lines on page 3-26.](#)

Introducing CI reagent gas

The pressure of CI reagent gas in the source is usually somewhat lower for CI negative mode compared to CI positive mode, however this can be compound specific.

Start with a set pressure read from the source Penning gauge of 5 to 8e-5 mbar.

See also: [Introducing CI reagent gas on page 3-26.](#)

Obtaining a beam in negative CI mode

To obtain a beam in negative CI mode:

1. Close the reference reservoir pump valve on the top of the instrument.
2. Introduce 0.2 μl of heptacosane via the reference reservoir inlet cap.
See also: [Introducing the reference compound into the reference reservoir inlet cap: on page 3-6.](#)
3. In the Tune window CI Tune tab, set the Emission Current to 250 μA .
4. Set all other source tuning parameters to those provided in the [Table titled "Recommended Tuning starting values:" on page 3-3.](#) Recall previously stored parameters if available.
5. Set the peak display 1 mass range from 448 to 454 Da, and adjust the gain of the peak display until ions from 451.97 of heptacosane can be detected.

6. In the TDC Settings dialog box Data pane, set the Veff value to display the correct mass in the peak display.
See also: [Setting up nominal mass on page B-47](#).
7. To obtain the best intensity of the m/z 451.97 ion, retune the following parameters in the Tune window, CI Tune tab, Beam Steering, Focus1, Focus2, and, Focus3.
Tip: The CI gas pressure can be optimized as this parameter can often be compound dependant.
Note: There is little low mass background in this technique, and therefore it should not be necessary to consider ions of low mass in terms of saturation while tuning.
8. Continue to tune the instrument until you have maximized the intensity of the peak.
Note: Maintain the intensity of the peak at approximately 1000 counts per second in continuum to avoid saturation.
9. See also:
 - [General considerations for tuning on page G-6](#)
 - [Dead time correction on page B-49](#)
 - [Effects of saturation on peak shape on page B-51](#)
10. Adjust the CI gas pressure if necessary to obtain best sensitivity.

Turning off CI gas

To turn off CI gas

In the Tune window Inlet tab, clear the CI Gas check box.

At the end of CI operation, the rotary pump must be gas ballasted for 30 minutes.


See also: [Gas-ballasting the rotary pump on page 10-5](#).

Preparing for FI operation

For a detailed description of how to tune the instrument for FI operation, see [Chapter 9 Tuning the Instrument](#).

Managing tune parameters

Printing tune information

To print a report of the peak information displayed in the Tune window (including a record of each parameter setting), click .

Restriction: You cannot configure the report.

Storing tune parameters

MassLynx stores tuning parameters with each data file as part of the experimental record. You can view or print the tuning parameters for a particular data file from the MassLynx Data Browser dialog box.

See also: The MassLynx's online help for more information.

Saving and recalling parameter settings

You can save sets of instrument tuning parameters to disk as a named instrument tune parameter file (.ipr). Then, you can recall the parameters at a future date.

The tune parameter files contain the latest source control settings for all supported ionization modes, not just the currently selected ionization mode. They also contain settings for the analyzer and inlets.


To save current tune parameters with the existing file names:

Click  in the Tune window.

To save the current tune parameters with a new file name:

1. Click File > Save As.
2. In the Save As dialog box, specify a new file name, or select an existing file from the list.
3. Click Save.

To recall a saved set of parameters:

1. In the Tune window, click  .
2. Select the required tuning parameter file by typing its name or by selecting it from the displayed list.
3. Click Open.

Result: The *.ipr file opens.

4 Acquiring Data

This chapter describes how to acquire data and set up the function list.

Note: To acquire accurate mass data you must first tune the instrument (see [Chapter 3](#)) and then calibrate it (see [Chapter 5](#)).

Contents:

Topic	Page
Starting data acquisition	4-2
Setting up an MS Method	4-5
Setting up an MS Scan	4-12
Setting up DRE mode	4-15

Starting data acquisition

Requirement: Before acquiring data, you must tune the instrument and prepare it for operation. A beam must be visible in the Tune window's peak display.

See also: [Preparing the instrument for operation on page 3-2.](#)

You can start data acquisition in one of two ways:

- A single sample acquisition from the Tune window. (See [Starting a single sample acquisition on page 4-2.](#))
- A multiple sample acquisition from the MassLynx window. (See [Starting a multiple-sample data acquisition on page 4-5.](#))

Starting a single sample acquisition


The easiest way to acquire data is by doing so directly from the Tune window:

- You can start and stop acquisitions from the Tune window.
- You can control most scanning parameters from the Tune window.

Restriction: With single sample acquisition, you cannot perform the following:

- Use inlet programs.
- Acquire analog data.
- Acquire multiple sample sequences.

To start a single sample acquisition:

1. In the Tune window, click .
2. In the GCT Premier Tune Acquire dialog box, specify the required parameters.

See also: [Table titled "GCT Premier Tune Acquire dialog box parameters:" on page 4-3.](#)

3. Click Start.

Result: The instrument acquires data and saves it to disk.

GCT Premier Tune Acquire dialog box:

GCT Premier Tune Acquire dialog box parameters:

Parameter	Description
File Name	Type the name of the data file to be created in this text box. If the file already exists on the disk, then MassLynx prompts you to rename the file or overwrite the existing one. MassLynx writes the file to the data directory of the current project.
Comment	Type a brief description of the sample in this text box. This description can appear on any output of the acquired data.
Origin	Opens the Sample Origin dialog box, where you add additional information about the sample. For example the submitter, job, task, and conditions.

GCT Premier Tune Acquire dialog box parameters: (Continued)

Parameter	Description
Format	Specifies the data type (Centroid or Continuum) to be collected and stored on disk. Centroid format is recommended for collecting data, except when checking data resolution. See also: Types of data acquisition on page 4-14.
Duration	The length of the acquisition, in minutes.
Scan Time	The length of each scan, in seconds.
Inter Scan	The time between one scan's end and the next scan's start, in seconds. MassLynx does not store data during this time interval. Note: The minimum delay is 10 ms (0.01 s).
Use Probe Ramping Method	When selected, you can type a probe ramping method (.prf) filename and path in the text box, or click Browse to locate the file. See also: <ul style="list-style-type: none"> • Ramping the probe temperature automatically on page 7-11. • Ramping the probe current automatically on page 8-8.
Use DRE	When selected data are acquired in the dynamic range enhancement (DRE) mode. The dynamic range increases to more than four orders of magnitude. Use DRE if using stronger samples that are likely to saturate the detector.
Requirements: <ul style="list-style-type: none"> • You must configure the DRE magnification factors before acquiring data in DRE mode; these factors must also be reconfigured if you change any of the tuning parameters. See also: Setting up DRE mode on page 4-15. <ul style="list-style-type: none"> • When you are acquiring data in DRE mode, scanning at least 5 spectra per second gives better peak shapes and more accurately represents peak intensities. 	
Start Mass	The mass at which the function starts.
End Mass	The mass at which the function stops.

GCT Premier Tune Acquire dialog box parameters: (Continued)

Parameter	Description
Close	Closes the dialog box. Tip: MassLynx does not retain the current settings when the dialog box is next opened.

Starting a multiple-sample data acquisition

The MassLynx window includes a sample list that defines how to acquire multiple samples on the instrument. The samples can be used together to perform a quantitative analysis. A spreadsheet-style editor creates the list of samples which can be tailored to suit individual requirements.

See also: The MassLynx online help for information about using the sample list to analyze multiple-samples.

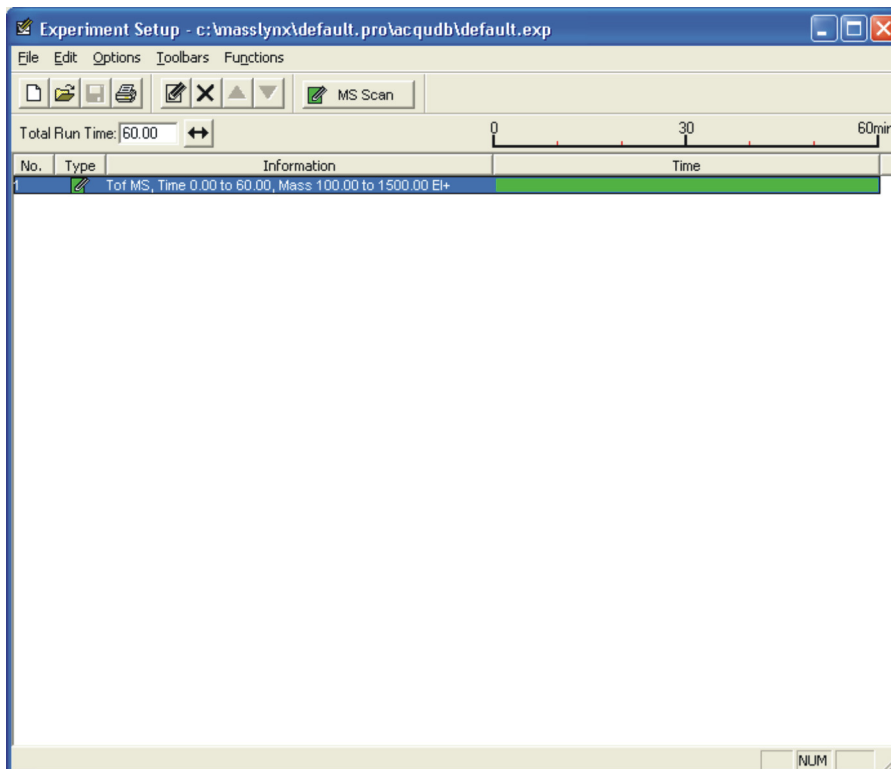
Setting up an MS Method

An MS method is a group of settings defining the scans the mass spectrometer should perform. Once an MS method is saved, these settings can be applied when acquiring data from samples.

In the MS Method editor set up an experiment function list. The functions scan the instrument during an acquisition. You can set up scanning techniques to run either sequentially during an acquisition.

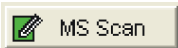
In the MassLynx window, click the MS method to open the Experiment Setup window MS Method editor.

Experiment Setup editor:



Adding a function to the list

To add a function to the list:

1. Click .
2. In the TOF MS Scan Editor, make the required changes to the parameters.

See also: Figure titled “Full Scan Function Editor:” on page 4-12.

3. Click OK.

Result: MassLynx adds the new function to the function list.

See also: Figure titled “Experiment Setup editor:” on page 4-6.

Modifying an existing function in the list

To modify an existing function in the list:

1. In the Function List, select the function.

2. Click .

Alternative: Double-click the function.

3. In the TOF MS Scan Editor, make the required changes to the parameters.
4. Click OK.

Result: MassLynx updates the function list display your changes.

Copying an existing function in the list

To copy an existing function in the list:

1. In the function list, select the function.
2. Click Edit > Copy.
3. Click Edit > Paste.

Result: A copy of the function appears in the function list.

4. Modify the parameters as required.

See also: [Modifying an existing function in the list on page 4-7.](#)

Deleting a function from the list

To delete a function from the list:



1. In the function list, select the function.

2. Click .

Alternative: Click Edit > Delete, or press the Del key.

Changing the order of functions in the list

To change the order of functions in the list:

1. Select the required function.
2. Repeatedly click  or  until the function is in the required position.

Note: Functions appear in ascending order according to their start and end time and the order cannot be changed. For functions with identical start and end times, you can change the order in which MassLynx performs them.

Saving and restoring a function list

To save a function list to file:

1. In the MS Method Editor, click File > Save As.
2. Type a new file name, or select an existing file from the list.
3. Click Save.

Tip: If the file already exists on disk, the system requests confirmation to overwrite the existing information.

4. Click Yes to overwrite the file; click No to select a different name.

Result: When the editor closes, a message appears that prompts you to save any changes made to the function list, if the changes have not been saved.

To restore a saved function list file:

1. In the MS method editor, click File > Open.
2. In the Open dialog box, select the name of the function list file to open by specifying its name or selecting it from the list.
3. Click Open.

Selecting the run time for the experiment

Requirement: The run time, or duration, of the experiment should be long enough to identify all the mass peaks eluting from the chromatographic column.

To select the run time for the experiment:

1. In the Experiment Setup window MS method, specify the run-time value, in the Total Run Time text box.

2. Click .

Result: MassLynx maintains the ratio of defined functions. For example, if two functions are defined, one from 0 to 5 minutes and the other 5 to 10 minutes, then a total run time of 10 minutes appears. If the run-time value is changed to 20, then the first function runs from 0 to 10 minutes and the second from 10 to 20 minutes.

Setting a solvent delay



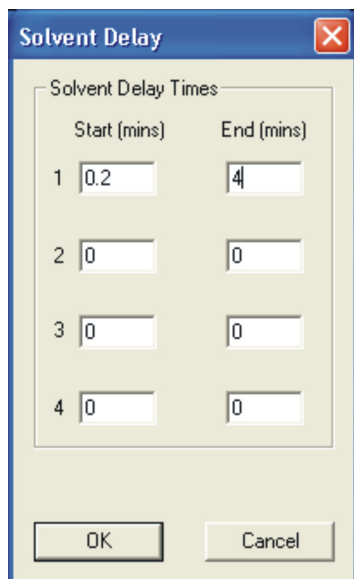
Caution: To avoid damaging the source filament when performing a GC acquisition, to prevent damage to the source filament, always specify a Solvent Delay.

The solvent delay protects the filament from high source pressure in the source, which can cause the filament to burn out. After injecting onto the GC, the low boiling solvent elutes first and solvent evaporating from the sample causes high pressure in the source. Typically, this high pressure lasts up to four minutes, depending on the oven temperature. The solvent delay turns off the filament during this period of high pressure, which prevents the filament from burning out.

To set a solvent delay for a function list:

In the MS Method editor Experiment Setup window, click Options > Solvent Delay.

Solvent Delay dialog box:



MassLynx does not store data during the solvent delay period. Also, MassLynx turns off the source filament during that period. Therefore, solvent peaks that would normally appear as eluting on the TIC (Total Ion Count) chromatogram no longer appear.

The delay time depends on the solvent used. You can set up to four solvent delays.

Note: An initial time is also required in the Start field as a number of software functions require a number of scans of background data. Therefore, the value 0.2 is used in the dialog box.

Acquiring analog data

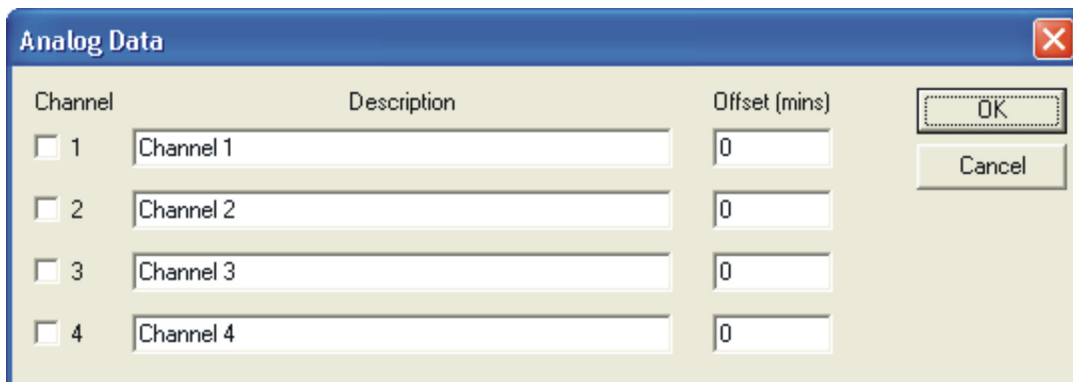
Four analog data channels are available within MassLynx. MassLynx stores the analog data with the data acquired from the mass spectrometer.

Analog channels are typically used to collect data from external units such as flame ionization detectors, which must be connected to the analog channel connectors.

See also: [Figure titled “Analog Inputs, Contact Closure Inputs, and Contact Closure Event Out outputs:” on page 1-14.](#)

The external channel is read at the end of each scan and stored with the data for that scan. The resolution of the chromatography for an analog channel therefore depends on the analog samples.

Analog Data dialog box:



To store data for an analog channel:

1. In the MS Method Editor, click Options > Analog Data.
2. In the Analog Data dialog box, select one or more check boxes for the channels required.
3. Type a description for each selected analog channel.
Tip: MassLynx adopts this description and displays it in the Analog Chromatogram dialog box as the channel description.
See also: The MassLynx User's Guide.
4. Specify an offset value to align the external unit with the mass spectrometer (for example, solvent delay, or time of the first function.) This is the time delay, in minutes, from the start of acquisition, before acquiring through the channel.
5. Click OK.

Setting up an MS Scan

The TOF MS Scan Function Editor sets up centroid and continuum functions.

Full Scan Function Editor:

Function: 2 TOFMS Scan

Mass (m/z)
Start: 1
End: 800
Use DRE:

Method
Ion Mode: EI+
Data: Continuum

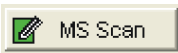
Time (mins)
Start: 0
End: 60

Scan Duration (secs)
Scan Time: 0.9
Inter-Scan Delay: 0.1

Probe Ramp
 Use Probe Ramping Method
Browse

OK Cancel

To set up a full scan function:

1. In the MS Method Editor, click  .
Alternative: Click Functions > MS Scan.
2. In the Full Scan Function Editor, type the required parameters.
3. Click OK.

Full Scan function editor parameters:

Parameter	Description
Start	The mass at which the scan starts.
End	The mass at which the scan stops. Requirement: The Start mass must be less than the End mass.
Use DRE	Acquires data in DRE mode. Doing so increases the dynamic range to over four orders of magnitude. Click Use DRE if using stronger samples which are likely to saturate the detector.
Requirements: <ul style="list-style-type: none">You must configure DRE magnification factors before acquiring data in DRE mode; these factors must also be reconfigured if you change any of the tuning parameters. See also: Setting up DRE mode on page 4-15 .	
Start	The retention time (in minutes) at which the scan starts.

Full Scan function editor parameters: (Continued)

Parameter	Description
End	The retention time (in minutes) at which the scan stops.
Use Probe Ramping Method	<p>When selected, you can enter the name of a Probe Ramp Method file (*.prf) in the adjacent text box; alternatively, click Browse to search for a file.</p> <p>Restriction: Use the probe ramping method only when samples are introduced by Direct Insertion (DI) or Desorption Chemical Ionization (DCI) probe.</p> <p>See also:</p> <ul style="list-style-type: none">• Ramping the probe temperature automatically on page 7-11.• Figure titled “Ramping the probe current automatically” on page 8-8.
Ion Mode	Specifies the ionization mode and polarity to be used during data acquisition.
Data	<p>Specifies the type of data to be collected and stored on disk.</p> <p>See also: Types of data acquisition on page 4-14.</p>
Scan Time	Specifies the duration of each scan, in seconds.
Inter-Scan Delay	Specifies the time between a scan finishing and the next one starting in seconds. No data are stored during this period.

Types of data acquisition

In the Full Scan Function Editor, the Data parameter specifies the types of data to be collected and stored on disk:

- **Centroid data:** MassLynx stores data as centroid intensity data and it stores mass-assigned peaks in a “stick” spectrum. MassLynx stores centroid data for each scan.

- **Continuum:** MassLynx regularly stores the signal received by the interface electronics to provide an analog intensity picture of the acquired data. MassLynx does not display data as centroid peaks, but stores data for each scan as peaks.

Tip: Continuum data files include significantly more data points than centroid data files, so continuum data files are larger.

Setting up DRE mode

Dynamic Range Enhancement (DRE) mode increases the dynamic range of the GCT Premier, permitting the analysis of strong samples that produce intense signals.

In DRE mode, the instrument acquires alternate high and low intensity scan data and then uses the low intensity data to provide the mass and intensity of peaks that saturate in high-intensity data streams.

See also:

- [DRE mode on page G-10](#)
- [Use DRE on page 4-13](#)
- [Use DRE on page 4-4](#)

Before using DRE mode, you must set up the instrument.

See also:

- [Obtaining an ion beam on page 3-3](#)
- [Setting up the instrument automatically on page 3-8.](#)

When you are acquiring data in the DRE mode scan at least 5 spectra per second.



Caution: To ensure accurate data acquisition, you must perform the setup procedure below each time you change any source or analyzer parameter.

To set up DRE mode manually:

1. In the MassLynx Tune window DRE tab, click High Sensitivity.

Rationale: The instrument will operate in high sensitivity mode for the z-focus and the z-lens steering.

2. Set z-focus to 40 and z-lens steering to 0.
3. Tune the instrument in EI with heptacosa or any other reference compound.
4. In the Tuning Setup dialog box, set Data Format to Centroid.
See also: [Tuning the instrument for EI on page 3-19.](#)
5. Record the beam intensity as displayed on the MassLynx Tune window.
6. On the DRE tab, clear High Sensitivity.
Rationale: The instrument will operate in low sensitivity mode for the z-focus and the z-lens steering.
7. Increase the value of z-focus until the beam intensity is approximately 1/40 of the size you noted in [step 5](#).
Note: This would typically occur at a z-focus (V) value of 250, but varies with source tuning.
8. In the DRE tab, select High Sensitivity.
9. Acquire centroid data for 1 minute between 1 and 800 Da (“high intensity” data).
10. On the DRE tab, clear High Sensitivity.
11. Acquire centroid data for 1 minute between 1 and 800 Da (“low intensity” data).
12. For each of the data files acquired in [steps 9 and 11](#), sum the same number of scans and examine the resulting spectra.
Requirements: The peaks must not be saturated in the high intensity data, and there must be at least 1500 counts on each peak of interest in the summed low intensity data.
Tip: A question mark displays above every saturated spectral peak when a single spectrum is viewed.
See also: The MassLynx User’s Guide.
13. Use the following equation to calculate the magnification factor for each of the masses shown in the [Table titled “Mass values and example magnification factor results for heptacosa in EI and CI positive ion mode:” on page 4-17.](#)

$$\text{magnification factor} = \frac{\text{peak intensity in high intensity mode}}{\text{peak intensity in low intensity mode}}$$

Requirements:

- If the instrument was tuned in negative ion mode, use the mass values shown in the [Table titled “Mass values and example magnification factor results for methyltriazine in EI and CI positive ion mode:”](#) on page 4-17.
- The magnification factors for each ion mode are different; you must calculate them separately for EI+, EI-, CI+, and CI-.

Mass values and example magnification factor results for heptacosane in EI and CI positive ion mode:

Mass	Magnification factor
68.99518	34.51
130.9919	23.01
218.9856	23.90
413.9774	23.25
501.9711	27.69

Mass values and example magnification factor results for methyltriazine in EI and CI positive ion mode:

Mass	Magnification factor
68.99521	50.65
121.0013	43.28
189.9965	35.25
265.9964	28.04
284.9949	28.11

14. On the MassLynx Tune window DRE tab, specify the values of Mass and Magnification factor in the table.

Tip: The ideal magnification factor is 40 and you should aim for values between 20 and 60. The magnification factor must always be greater than 10.

Mass values and example magnification factor results for a mixture of chloropentafluorobenzene and heptacosane in CI negative ion mode:

Mass	Magnification factor
201.9609	50.65
451.9743	43.28
513.9711	35.25
594.9663	28.04
632.9632	28.11

5 Calibration

This chapter describes how to calibrate the mass scale of the instrument (that is, to ensure that the peaks displayed in a spectrum are in their correct locations).

Notes: The figures in this chapter show indicative parameter values only.

Contents:

Topic	Page
Calibrating the instrument	5-2
Using the Calibration wizard	5-4
Using calibrations for subsequent acquisitions	5-15
Dynamic External Calibration Temperature Compensation	5-16
Calibrating the instrument in CI	5-18
Manual calibration	5-19

Calibrating the instrument

To obtain exact mass measurements, you must calibrate the instrument's mass scale. When nominal mass is set up, the masses will be correct to within m/z 0.5. Using the calibration routine produces more accurate mass measurements. To obtain exact mass measurement (within 5 ppm RMS), use a lock mass as well as a calibration file. This section describes how to calibrate the mass scale in EI and CI mode using heptacosane as the reference sample.

The Instrument Setup Wizard will perform the following to obtain exact mass measurements:

- Set-up the TDC
- Set-up Nominal Mass
- Set-up Deadtime correction
- Set-up Lock mass correction

See also: [Obtaining an ion beam on page 3-3](#).

Setting up the instrument for calibration

For all accurate mass work, calibrating the TOF analyzer is a critical part of the instrument setup process. MassLynx uses a polynomial equation to calibrate precisely over a wide mass range.

The instrument must be set up correctly and run within certain limits before calibration so it is essential that the setup wizard has been run, or the instrument has been setup manually before you calibrate.

Instrument warm-up

For accurate mass measurements, put the instrument in operate mode for at least one hour before acquiring data or calibrating it to warm up the instrument and stabilize the analyzer voltages.

TOF calibration of the instrument in EI

For all accurate mass work, calibrating the TOF analyzer is a critical part of the instrument setup process. MassLynx uses a polynomial equation to calibrate precisely over a wide mass range.

Instrument Setup wizard

Ensure that the Instrument Setup wizard has been successfully run, or all the appropriate parameters are set up manually.

See also: [Obtaining an ion beam on page 3-3.](#)

Tune Window parameters

Ensure that none of the parameters are changed since the Instrument Setup wizard was run.

Lock mass correction

To produce accurately mass-measured data to within 5 ppm, you must compensate for instrument drift resulting from internal factors including temperature changes.

Compensation is achieved using a single lock mass peak from a reference compound introduced via the reference reservoir. This single peak is used on a spectrum basis to adjust the calibration applied to individual spectra. The result is a complete and accurate mass-measured data set.

For example in EI + operation lock mass 284.9949 from tris (trifluoromethyl) triazine can be used as an internal lock mass.

In the spectrum the lock mass peak is shown in blue, if found, and marked with a star (*).

To specify the lock mass:

1. In the Tune window, click Settings > TDC Settings.
2. In the TDC Settings dialog box, specify the lockmass value (to four decimal places).
3. Set Mass Window +/-, to 0.3 mDa.

Result: The 0.3 Da +/- value is usually sufficient for MassLynx to locate the desired lock mass peak before adjusting the mass measurement if the nominal mass is set up.

See also: [Figure titled “TDC Settings dialog box parameters:” on page B-13.](#)

Lock mass intensity

When using the lock mass in experiments the lock mass compound is continually introduced to the source from the reference reservoir throughout the experiment. Before starting an acquisition using lock mass, make sure that there is a constant signal of lock mass compound at the mass specified in the TDC settings window and that the intensity is appropriate. The lock mass peak should be over 1500 counts in centroid, in each spectrum, for good statistical mass measurements. However, it must not be so strong as to be saturated. Saturated peaks are indicated by a question mark above the peak in a single spectrum view. If saturation is seen on the lock mass peak, then the peak's intensity must be reduced. Open the reference reservoir pump-out valve until the intensity falls to a suitable value.

The Instrument Setup Wizard initially sets up the lock mass intensity.

See also: [Obtaining an ion beam on page 3-3.](#)

Using the Calibration wizard

When you run the calibration wizard, sample is injected into the reference reservoir and data are acquired and compared to data in a reference file containing the correct masses for some of the ions in the reference compound. The instrument then calculates a calibration curve, and a graph with the mean residual value (RMS) displayed.

See also: [Figure titled “Calibration Graph window:” on page 5-10.](#)

The mean residual value indicates the success of the calibration. For example, a mean residual value of less than 1 mDa indicates that the calibration was good. When the acquisition is saved, the calibration is displayed on the Tune window and the file created is applied to all subsequent acquisitions.

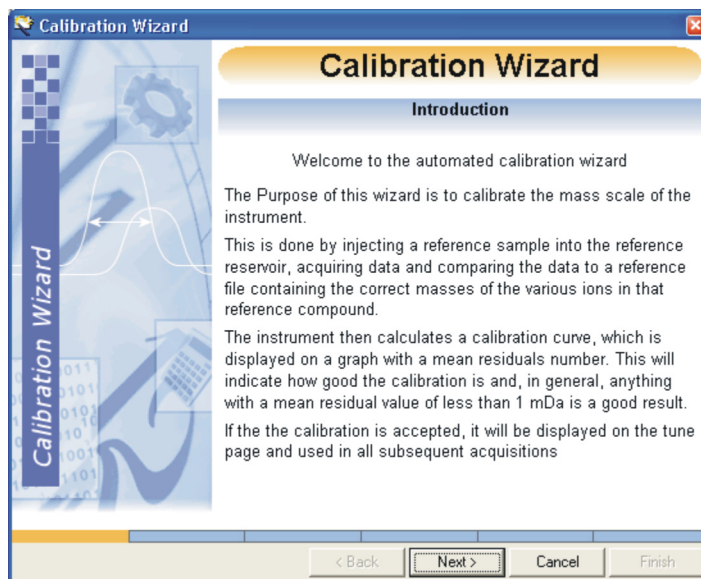
Important: If you click Cancel while running the wizard, the wizard closes. Existing calibrations are no longer valid and you must run them again.

To run the calibration wizard:

1. Use a septum-piercing syringe to introduce the reference sample into the reference reservoir.
2. Adjust the intensity of the reference sample using the reference reservoir pump valve to obtain approximately 1200 counts per second.
3. In the MassLynx Tune window, click Wizard > Calibration Wizard.

The Calibration Wizard Introduction window appears.

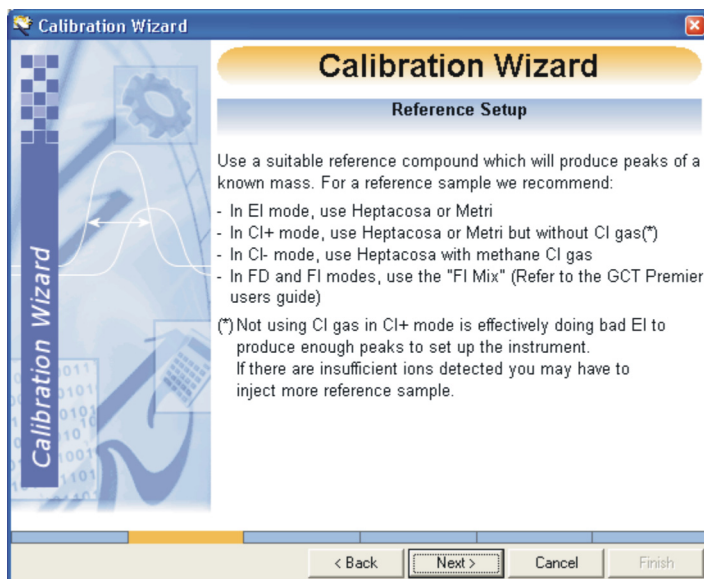
Calibration Wizard Introduction:



4. Click Next.

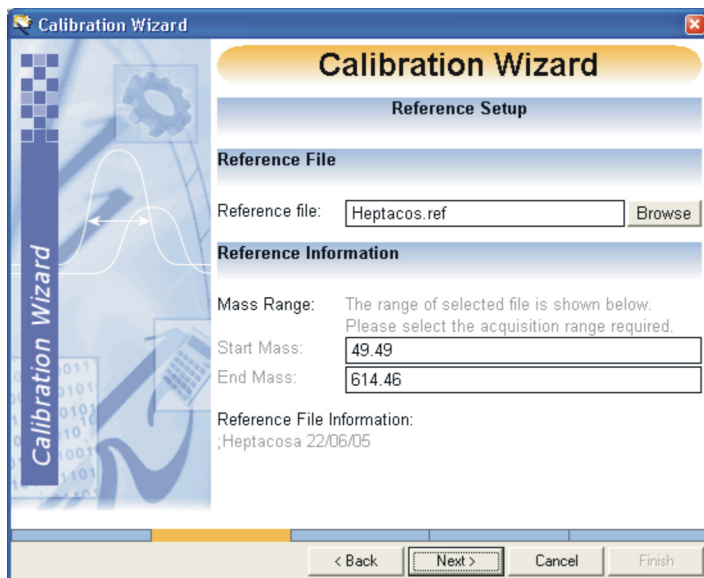
The Calibration Wizard Reference Setup window appears.

Calibration Wizard Reference Setup:



5. Click Next.

Calibration Wizard Reference Setup:



- In the Calibration Wizard Reference Setup page, click Browse, to select the appropriate reference file for the reference sample that you are using for the calibration.

Tip: When the reference file is selected, the reference file information is displayed in the Calibration Wizard Reference Setup window.

- In the Calibration Wizard Acquisition Setup page, specify a Start Mass and an End Mass or use the wizard defaults.
- Click Next.

The Calibration Wizard Acquisition Setup window appears.

Calibration Wizard Acquisition Setup:

Calibration Wizard Acquisition Setup parameters:

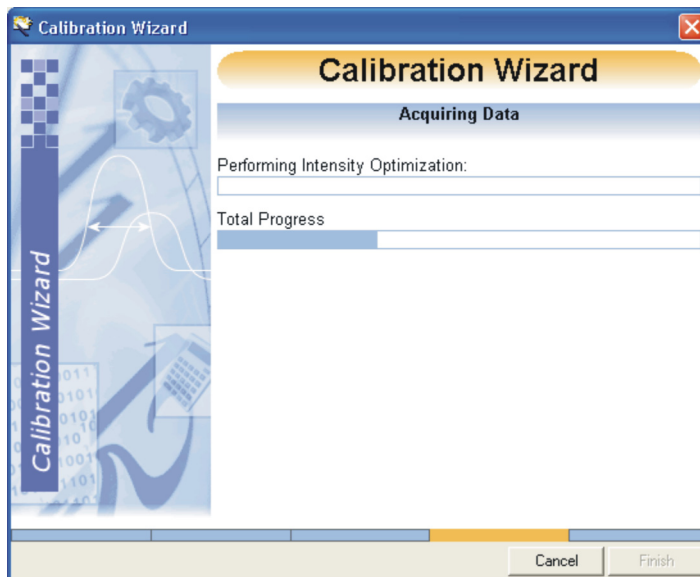
Parameter	Description
Calibration Parameters	
Intensity Threshold	Intensity threshold is a percentage. The calibration wizard discounts small peaks less than the percentage of the base peak when it creates the calibration curve.

Calibration Wizard Acquisition Setup parameters: (Continued)

Parameter	Description
Reference Window	Is the size of the window in Da used to search for the peak in the reference file for initial peak matching.
Auto Polynomial	When selected, the order the wizard attempts to select the polynomial order that fits the data best. Polynomial order of 2 or 1 is normally used for good extrapolation to high mass.
Polynomial Order	Is the order of the curve equation it uses to fit to the data

9. Enter a file name for the calibration data acquired.
Tip: The file name is used as the calibration file name.
10. Specify the scan time and an interscan time or use the wizard defaults.
11. Specify the intensity threshold or use the wizard default.
12. Specify the reference window or use the wizard default.
13. Click Next.
The Calibration Wizard Acquiring Data window appears.

Calibration Wizard Acquiring Data:



The calibration process takes several minutes to acquire the necessary data if the reference sample is weak. An error may occur if the beam is too strong or too weak.

When the calibration wizard finishes the Calibration Graph window appears.

See also: [Figure titled “Calibration Graph window:” on page 5-10.](#)

14. If you are satisfied with the calibration report, accept the calibration.

See also: [Accepting a calibration on page 5-9.](#)

Tip: The instrument automatically uses the calibration for subsequent acquisitions.

See also: [Using calibrations for subsequent acquisitions on page 5-15.](#)

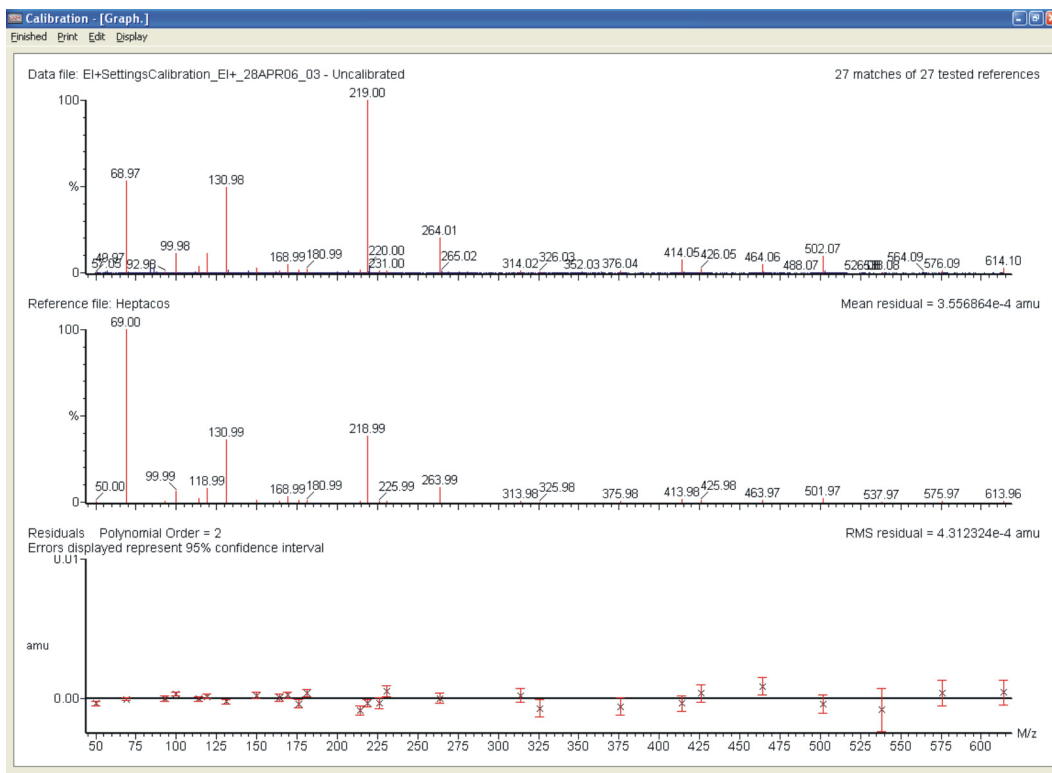
Accepting a calibration

To accept a calibration:

In the Calibration window, click Finish > Accept Calibration.

The calibration is saved as a .cal file and given the file name specified in the Calibration wizard.

Calibration Graph window:



The Calibration report window comprises four sections:

- **Data file:** The data selected for calibration.
- **Reference file:** The spectrum of the selected reference file.
- **Residuals:** The software will have attempted to produce a calibration that corrects the acquired data so that the reference peaks are at the correct expected mass. The residuals graph shows how accurately a calibration corrected the data by showing the mass difference between the data and the calibration line.
- **RMS:** An average of the residuals; a good value is any value less than 0.001.

Manually correcting a calibration

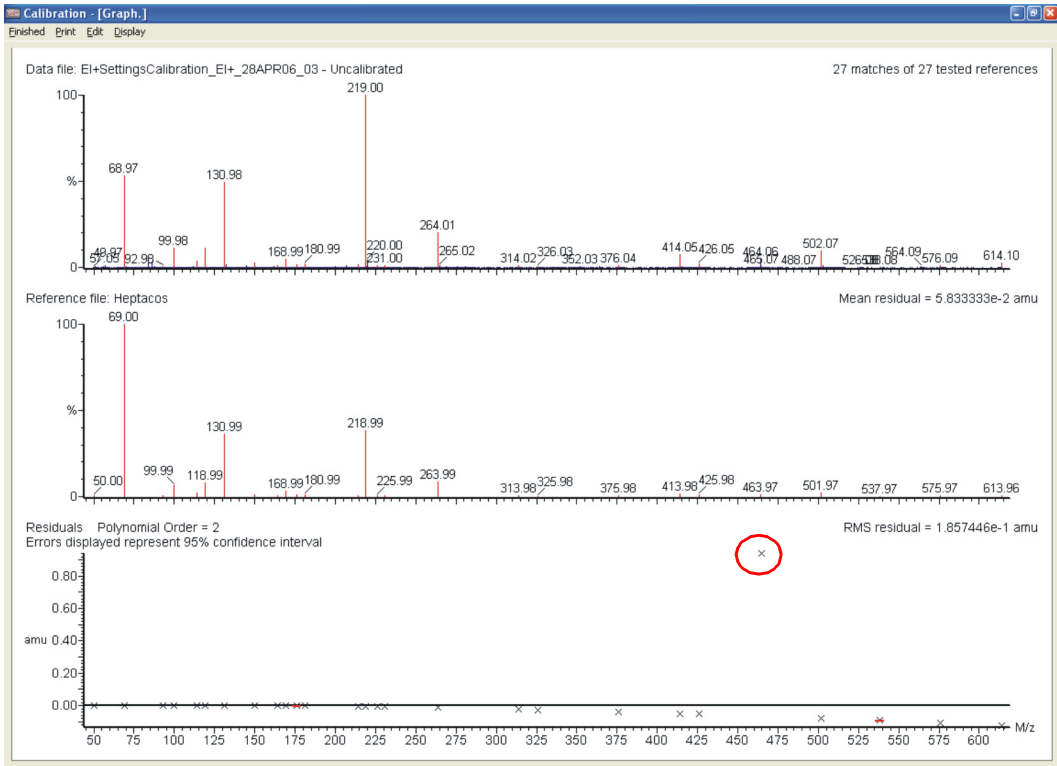


Caution: If the Calibration Parameters dialog box is opened and then closed with the Perform auto peak matching option selected, the spectrum is automatically recalibrated. Any manual corrections are lost.

See also: [Editing the calibration parameters on page 5-13.](#)

You might need to manually adjust some of the matched peaks during the calibration process. Mismatched peaks are usually apparent by the larger than normal residual errors they leave. For example, in the following figure, there is one mismatched peak with larger residuals (highlighted). Such mismatched peaks often occur when the automatic peak assignment function picks the incorrect peak.

Calibration report window with large residual errors:



To manually adjust the large residual error peaks:

1. Left-click and drag across a mass region of a mismatched peak to zoom the mass region.

Tip: Click Display > Default to zoom out.

2. In the reference spectrum pane, right-click the mismatched reference peak.

Tip: Right-click slightly to the right of the peak and below its top.

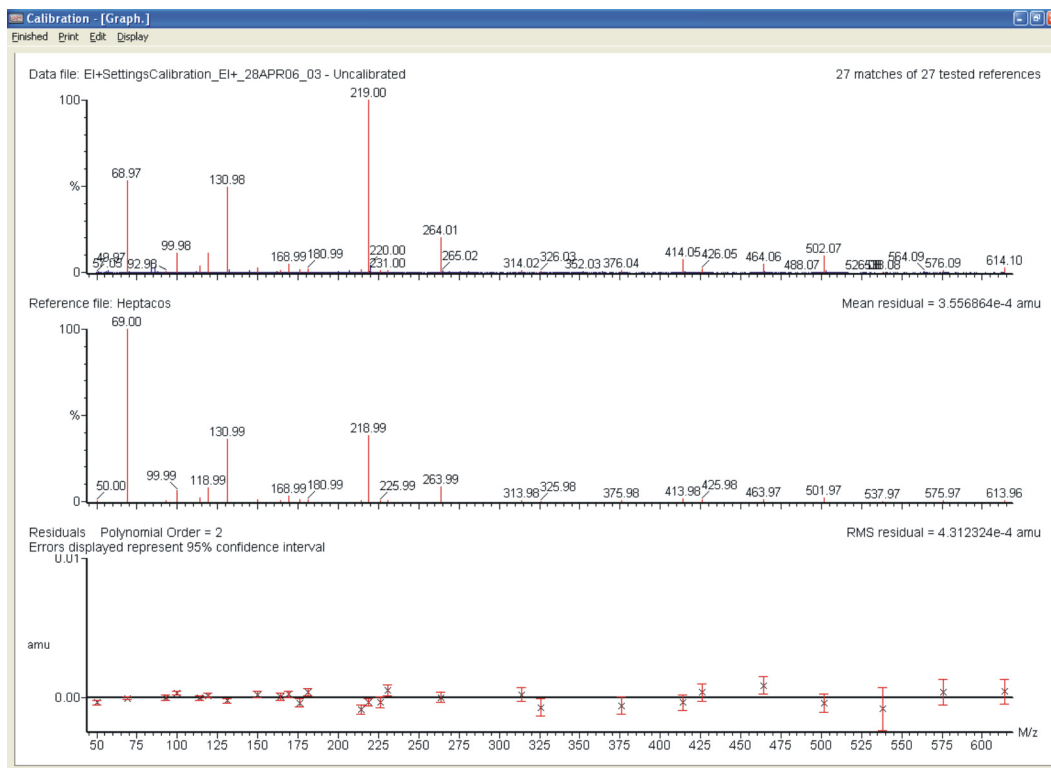
3. Right-click the required peak in the spectrum pane.

Result: MassLynx updates the calibration with the new point.

4. Repeat [steps 1 through 3](#) for other mismatched peaks.

After selecting the correct peaks, the residuals should be reasonably consistent, with no obvious outlying points.

Calibration report window with residual errors corrected:



Editing the calibration parameters

For any type of experiment, you can edit the calibration parameters in the Calibration Parameters dialog box. In the Calibration report window, select Edit > Calibration Parameters.

Calibration Parameters dialog box:

The screenshot shows the 'Calibration Parameters' dialog box with the following settings:

- Peak Match:**
 - Perform auto peak matching
 - Peak window (Da) +/-: 0.50
 - Initial error (Da): 0.50
 - Intensity threshold: 0.10
- Curve Fit:**
 - Automatically restrain polynomial coefficients
 - Polynomial order: 2
 - Intensity weighting
- Display:**
 - Calibrate display

Calibration Parameters dialog box parameters:

Parameter	Description
Perform auto peak matching	When selected, MassLynx automatically calibrates the spectrum. Note: When the option is selected, and the Calibration Parameters dialog box is closed, MassLynx automatically recalibrates the spectrum. Any manual corrections that have been made are lost. See also: Accepting a calibration on page 5-9.

Calibration Parameters dialog box parameters: (Continued)

Parameter	Description
Peak window +/-	<p>Specifies the maximum mass difference between the reference peaks and the expected position of the corresponding peaks in the acquired calibration spectrum.</p> <p>The normal operating range for this parameter is 0.3 to 1.5 Da. Increasing its value increases the chance of incorrect peak matching. The default value is 0.5.</p>
Initial error	<p>Specifies the maximum mass difference between the first reference peak to be matched and the corresponding peak in the acquired calibration spectrum.</p> <p>The normal operating range for this parameter is 0.5 to 2.0 Da. Increasing its value increases the chance of incorrect peak matching. The default value is 2.0.</p>
Intensity threshold	<p>Specifies the intensity threshold value. Any peak in the acquired calibration spectrum with an intensity less than this value (measured as a percentage of the most intense peak in the spectrum) is not used in the calibration procedure.</p> <p>The normal operating range for this parameter is 0 to 5%. The default value is 0.01.</p>
Polynomial order	<p>This parameter defines the number of coefficients to use in the calibration fit equation. The available options are 1 to 5. A typical value is 2.</p> <p>When extrapolating to high masses, use a polynomial order of 1.</p>
Intensity weighting	<p>Weights the curve toward the points representing the more intense acquired peaks. The weight of each point is proportional to the square root of the intensity of the acquired peak.</p>
Calibrate display	<p>This option applies the generated calibration to the data in the Calibration report window Spectrum pane.</p>

Using calibrations for subsequent acquisitions

Using calibrations

When acquiring data via the Tune window or from the Sample List, the last calibration file produced using the calibration wizard or manually produced calibration can be used. The calibration file that is currently being used is displayed on the Tune window Calibration tab. The calibration file should have been acquired using the same Engineer tab parameters and TDC Settings in the Tune window that were used to create the .ipr instrument parameter file used during the MS experiment.

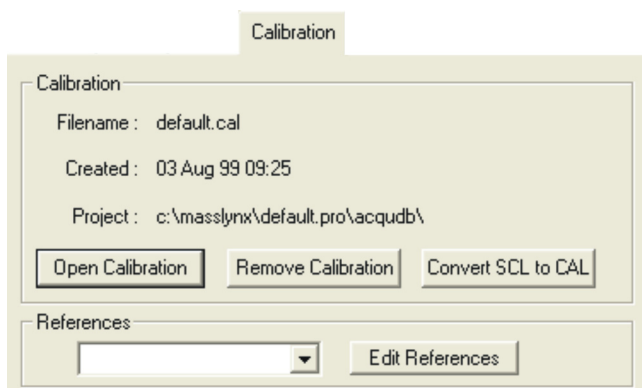
Associating a calibration file with an acquisition

In the Tune window you can select a calibration file that will be used for subsequent acquisitions from both the Tune window and the MS Method window.

To associate a calibration file with an acquisition:

1. In the Tune window, click the Calibration tab.

Calibration tab:



2. Click Open Calibration.
3. In the Open dialog box, select the required calibration file (*.cal).
4. Click Open.

Result: The calibration filename and path appear separately on the Calibration tab.

5. Click OK.

Note: The calibration wizard automatically sets the current calibration file when it finishes.

Removing a calibration file

When a calibration file is created using the Calibration wizard, the file appears in the Tune window and is used in all subsequent acquisitions. You can, however, remove the calibration file to select a different calibration file for another acquisition.

To remove a calibration file:

1. In the Tune window, click the Calibration tab.
2. In the Calibration tab, click Remove Calibration.

Result: The calibration file is replaced with an uncalibrated file (Uncal.cal).

Dynamic External Calibration Temperature Compensation

Dynamic external calibration (DXC™) is a method of compensating for mass drift caused by temperature fluctuations. It is not suitable for accurate mass work, so it is used only in the absence of a suitable lock mass.

To compensate for changes in room temperature, DXC shifts the mass by a specified amount in ppm/degree. Typical values for drift compensation are 40-80 ppm/degree.

Setting the DXC drift value

To set the drift value for DXC:

1. Acquire data from a reference sample such as heptacosane without using DXC, lock mass, or a calibration file.
2. In the Spectrum window, view the data.

3. Double-click at the top of the Spectrum window.
4. In the Header Editor, click Spectrum QTOF in the Group list.
5. Click Temp Correction in the Element list.
6. Click OK.
Result: The current temperature inside the instrument is displayed in Spectrum window.
7. Repeat the same acquisition later in the day under the same conditions and when the room temperature has changed by up to 3°C.
8. Display both spectra.
9. From the difference in mass and the difference in temperature, the drift constant can be calculated in ppm/°C.

Note: The instrument applies the temperature compensation only when the acquisition is performed using a calibration file.

See also:

[Dynamic External Calibration Temperature Compensation on page 5-16](#)

10. Open the TDC Settings window.
11. Click Use DXC Temperature Compensation.
12. Specify the value you calculated in the Drift (PPM/C) field.
13. MassLynx records the temperature at which it was calibrated, and then applies the ppm/degree value according to the measured temperature on each spectrum.

Requirements: Using DXC, a mass accuracy of less than 10 ppm RMS can be generally achieved for less than 3°C temperature drift. If more reliable mass accuracy is required or the instrument is subject to a temperature variation of greater than 3°C, then you have to use a lock mass.

Calibrating the instrument in CI


The instrument can be calibrated in CI ion mode in a similar way as you calibrate EI ion mode. In CI positive ion mode the calibration is performed using a reference sample, such as heptacosane or tris (trifluoromethyl) triazine without CI gas, to reproduce the EI spectrum. In CI positive you can only see the molecular ion and not the lower mass fragments with tris (trifluoromethyl) triazine. Heptacosane does produce some fragmentation when the reagent gas is present.

See also: [Calibrating the instrument in CI- mode on page 5-25.](#)

Note: Check the resolution for deadtime correction, see [Setting up resolution for dead time correction manually on page B-48.](#)

Calibrating the instrument in CI+ mode

To calibrate the instrument in CI+ mode:

1. In the MassLynx Tune window, click .
2. In the TDC Settings dialog box, set the Lock mass to 0.
3. Using a septum-piercing syringe, inject approximately 0.2 μL of tris (trifluoromethyl) triazine into the reference inlet cap.

Tip: Heptacosane or FC70 can also be used.

See also: [Introducing the reference sample on page 3-6.](#)

4. Click , to switch the instrument into operate and obtain a beam.

See also: [Obtaining a beam in positive CI mode on page 3-28.](#)

Tris (trifluoromethyl) triazine predominantly yields the (M+H) protonated molecular ion at 286. The resulting spectrum does not contain enough ions to perform a multi point linear calibration.

Hence two options are available:

- Using tris (trifluoromethyl) triazine the CI gas must be partially or totally removed to produce the classical EI spectrum of the reference compound. You can then perform the calibration using the standard EI calibration procedure.

- Using heptacosane or FC70 the reagent gas can be left at CI+ pressure and calibrated under CI+ conditions.
5. Tune the system in CI mode with CI gas in.
See also: [Tuning the instrument for positive CI on page 3-25.](#)
 6. Set up the nominal mass.
See also: [Setting up nominal mass on page B-47.](#)
 7. Reduce the pressure of CI gas by turning the CI Flow (%) to 0 or by clearing the CI Gas check box on the Inlet window.
 8. Monitor the whole spectrum from 10 to 290 in tune or acquisition mode.
Result: The fragment peak at 69 and other fragments characteristic of tris (trifluoromethyl) triazine in EI+ mode should appear. It may be necessary to introduce more reference material at this point to produce a strong enough signal for calibration.
 9. Perform an EI calibration applying the metri reference file and the reference sample tris (trifluoromethyl) triazine.
See also: [TOF calibration of the instrument in EI on page 5-2.](#)
Note: The C13 isotope of the M+ ion at 286 will be interfered with by the residual (M+H)+ ion at 286. Use metripci.ref as the reference file as the C13 isotope has been removed from this file.
 10. Once MassLynx accepts a calibration, reintroduce CI gas and adjust the level of the reference using the reference reservoir pump out valve. Use the (M+H)+ ion at 286.0027 as the lock mass when using tris (trifluoromethyl) triazine for CI+ accurate mass experiments.
 11. Ensure that the beam is not saturated.
See also: [Effects of saturation on peak shape on page B-51.](#)
 12. Ensure you count all the ions by setting up the MCP and TDC Threshold.

Manual calibration

Scanning acquisitions can be calibrated after they have been acquired. This assumes that the level of error is so small that masses of interest will not fall outside the mass window of the experiment. Hence, it is usually worth

scanning to slightly higher and lower masses than the mass window of interest.

Before manually calibrating the instrument you first need to remove the Lock Mass value in the TDC Settings dialog box and remove any existing calibration file.

Using Dynamic Range Enhancement with calibrations

Dynamic Range Enhancement must not be used when acquiring instrument calibration data. Signal intensities are kept low while calibrating the instrument. Extended and normal dynamic range acquisitions can use the same calibration file.

To turn Dynamic Range Enhancement off when acquiring data for calibration:

1. In the GCT Premier Tune Acquire dialog box, ensure that Use DRE is clear.
2. Click Calibration.

To manually calibrate the instrument:

1. In the TDC Settings dialog box, Centroiding Parameters pane, set Lock Mass to 0.

See also: [Configuring the TDC settings on page B-11.](#)

2. In the Tune window Calibration tab, click Remove Calibration.
3. Using a septum piercing syringe, inject approximately 0.2 μL of heptacosane into the reference reservoir inlet cap.

See also: [Introducing the reference sample on page 3-6.](#)


4. Ensure that the beam is not saturated.

Tip: Ensure the largest peak is approximately 1000 counts per second on the Tune window in continuum mode. The peak is saturated if a question mark (?) is displayed above it, when displayed as a single the spectrum in centroid mode.

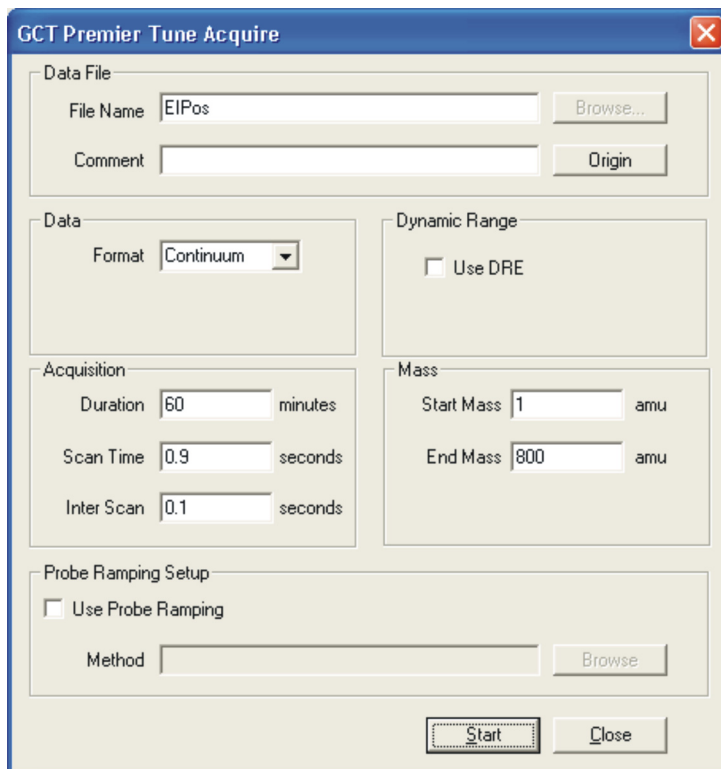
See also: [Effects of saturation on peak shape on page B-51.](#)

5. Ensure you count all the ions by setting-up the MCP and TDC Threshold

See also: TOF calibration of the instrument in EI on page 5-2, Setting the optimum gain for the detector on page 10-52 and Setting up resolution for dead time correction manually on page B-48.

6. In the Tune window, click .

GCT Premier Tune Acquire dialog box:



GCT Premier Tune Acquire dialog box configuration:

- Data File: File Name: EIPos, Comment: (empty), Browse... button, Origin button
- Data: Format: Continuum
- Dynamic Range: Use DRE (unchecked)
- Acquisition: Duration: 60 minutes, Scan Time: 0.9 seconds, Inter Scan: 0.1 seconds
- Mass: Start Mass: 1 amu, End Mass: 800 amu
- Probe Ramping Setup: Use Probe Ramping (unchecked), Method: (empty), Browse button
- Start button, Close button

7. In the GCT Premier Tune Acquire dialog box Data pane Format list, click Centroid.
8. In the Mass pane, set Start Mass to 50 and Set End Mass to 650.
9. In the Dynamic Range pane, ensure that Use DRE is not selected.
10. Click Start.

Result: The instrument starts collecting calibration data.

11. Monitor the acquisition and acquire 3 minutes of data.

See also: [Chapter 4, Acquiring Data](#).

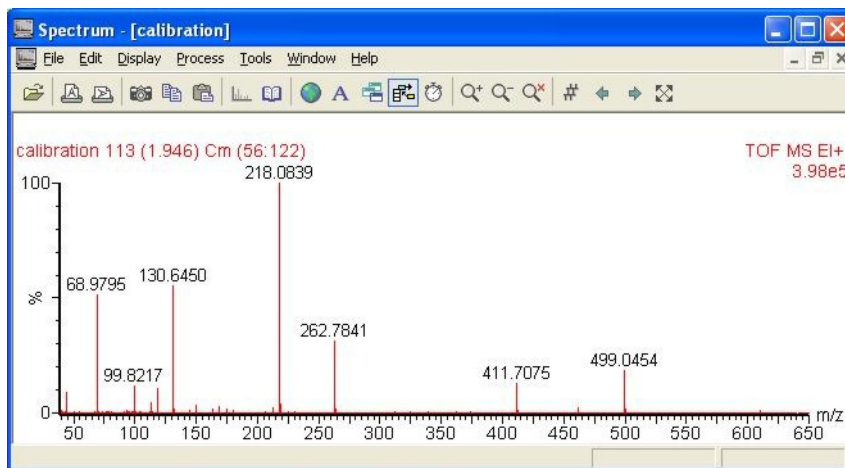
Creating a calibration file

To create a calibration file:

1. In the Chromatogram window, click Process > Combine Spectra.
2. In the Combine Spectrum dialog box, ensure the Peak separation parameter is set to 0.05.
3. In the Chromatogram window, right-click and drag across approximately one minute of data.

Result: The Spectrum Combine progress window appears. When processing is complete, a combined spectrum appears in the Spectrum window.

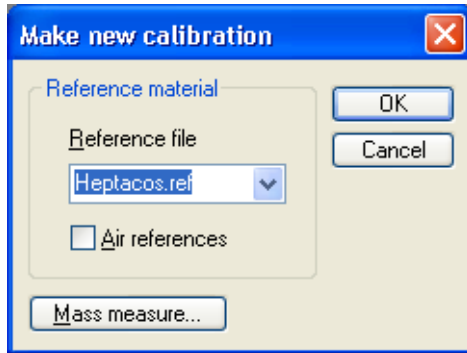
Combined spectrum in the Spectrum window:



4. In the Spectrum window, click Tools > Make Calibration.
5. In the Make new calibration dialog box Reference material pane, Reference file list, select the reference file against which the data is to be calibrated. For example, select Heptacos.ref if heptacos is being used as the reference compound.

See also: [Appendix C, Calibration Reference Information](#).

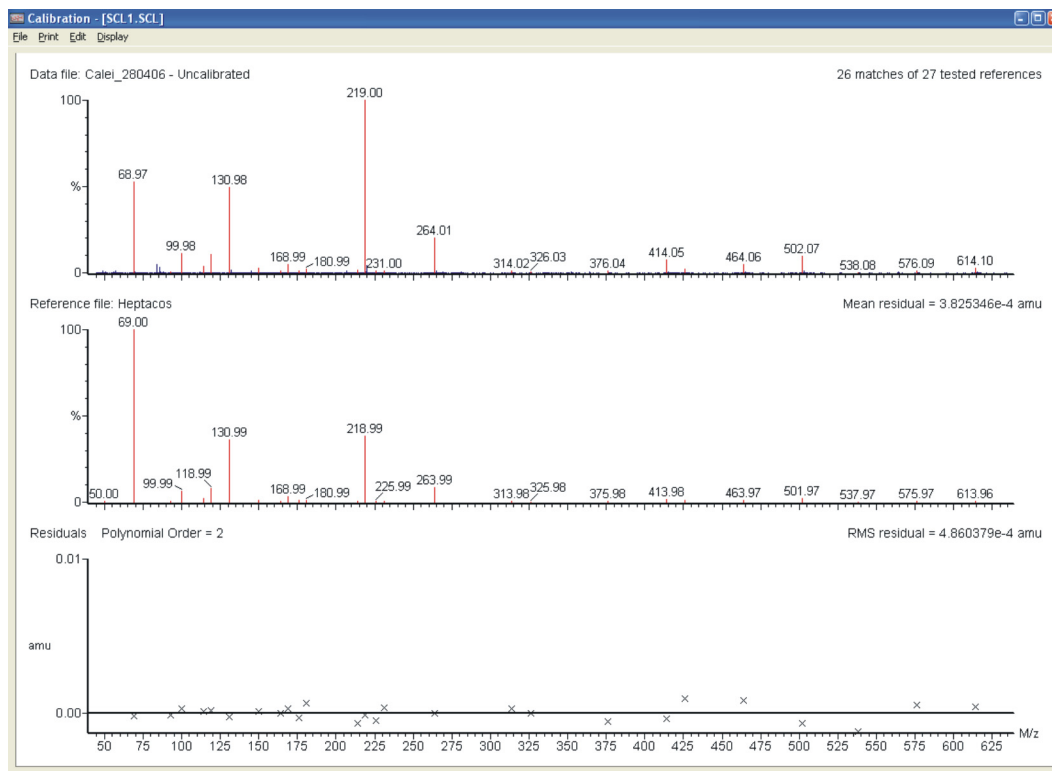
Make new calibration dialog box:



6. Click OK.

Result: When processing is complete, the Calibration report window opens.

Manual calibration report:



To save the calibration:

1. In the Calibration report window, click File > Save As.
2. Save the file with an appropriate name with the extension .scl.

To convert the calibration file:

1. In the Tune window Calibration tab, click Open Calibration.
2. Browse to the calibration file that you have created with the .scl extension.
3. Click Open, to open the calibration file.
4. Click Convert SCL to CAL, to convert the file.

The .cal calibration file is now set in the Tune window and used for subsequent acquisitions.

Calibrating the instrument in CI- mode

The procedure for calibrating in CI- mode is similar to the procedure for calibrating in CI+ mode except use heptacosane as the reference sample with a small amount of CI gas, heptaneg.ref as the reference file and acquire over a mass range up to 700 Da.

Calibrating the instrument in FI/FD mode

The procedure for calibrating in FI and FD mode is explained in detail in [Chapter 9 FI/FD Operation](#).


6 The GC Interface

This chapter describes installing, configuring, and removing of the GC interface.

Contents:

Topic	Page
The GC interface	6-2
Installing the GC interface	6-3
Installing the GC column	6-9
Configuring the GC and autosampler	6-11
Removing the GC interface	6-11

The GC interface

 **Warning:** To avoid burn injuries, take care when working with the GC interface and GC. They operate at high temperatures.

The GC interface provides a heated transfer line between the GC) and the ion source. The transfer line ensures even heating in this region, which prevents the sample condensing in the GC column before it reaches the ion source, reducing chromatographic peak quality.

GC interface with ceramic tip (used in FI):



GC interface with spring tip (used in EI and CI):



See also: [Installing the GC interface on page 6-3.](#)

Installing the GC interface

The GC interface can be heated to a temperature of 350 °C. It incorporates a spring-loaded tip at the source end of the column transfer line. The tip ensures that the interface remains in contact with the outer source block (to maintain reagent gas pressure for CI operation) and accommodates thermal expansion of the transfer line.


Determining the transfer line position

The end of the transfer line must be in the correct position relative to the outer source when fitting the GC interface. If the GC interface is not fitted and the transfer line position not determined. See [Working with the interface fitting tool on page 6-6](#).

If the GC interface has previously been installed and the transfer line position determined, follows the procedure described in [Reinstallation of the GC interface on page 6-6](#).

Installing the GC interface

To install the GC interface:

1. In the Tune window, click .
2. Click Vacuum > Vent Source.

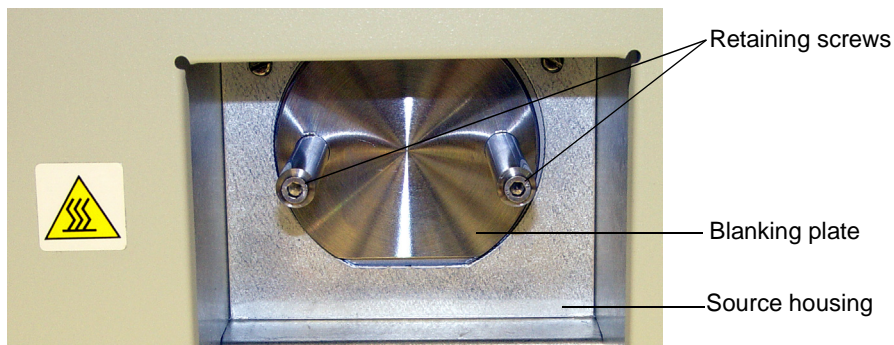
Result: A message confirms the vent command.

3. Click OK.

The turbomolecular pump is turned off. When the turbomolecular pump runs down to half normal operating speed, the vent valve opens and the instrument is automatically vented.

4. Wait for the Vacuum LED on the instrument's front panel to display yellow or red.
5. Use a 4-mm hex (Allen) key to unscrew the two retaining screws securing the blanking plate to the source housing.

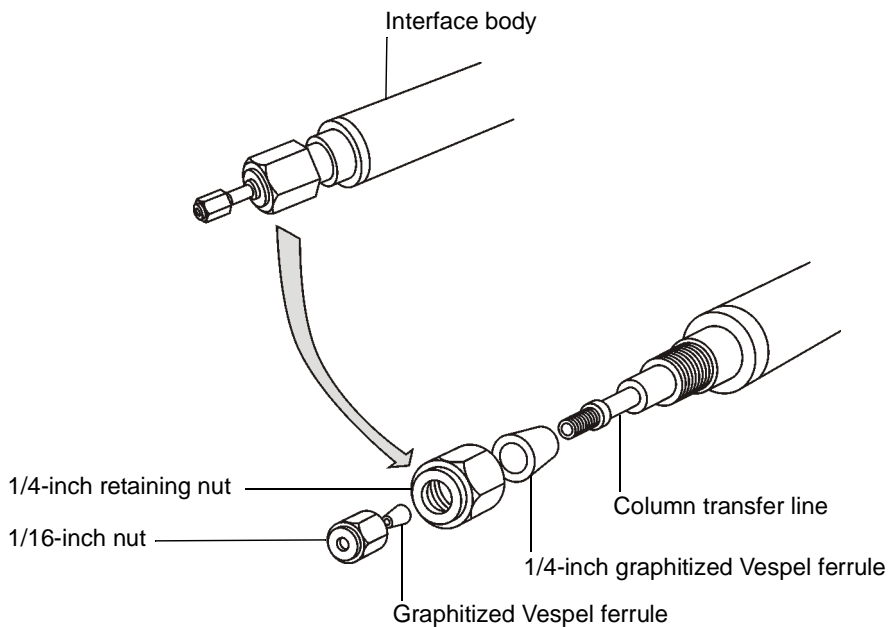
Blanking plate:




6. Remove the 1/16-inch nut and graphitized Vespel[®] ferrule from the column transfer line.

Note: The outside diameter of the column, and therefore the ferrule required, can vary depending on the column type used.

GC interface:



7. Remove the 1/4-inch retaining nut from the GC interface body.

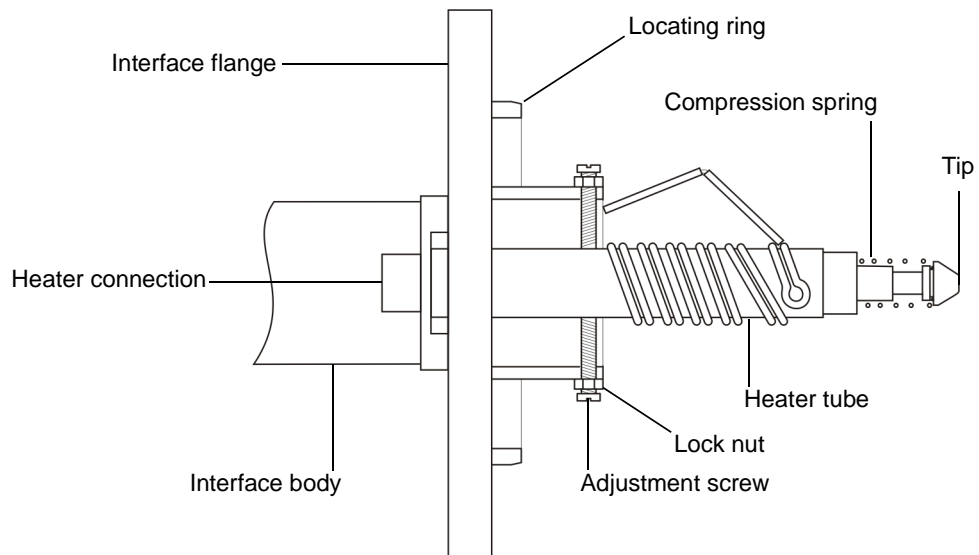
 **Caution:** Be careful not to damage the transfer line shaft when removing the graphitized Vespel ferrule with wire cutting pliers. Doing so can cause air leaks.

8. Remove the 1/4-inch graphitized Vespel ferrule by cutting part of it away with wire cutting pliers.

Requirement: If the 1/4-inch ferrule has been removed by cutting, replace it.

9. Ensure the spring-loaded tip has maximum travel when compressed. If required, the compression spring can be carefully stretched to allow maximum movement.

GC interface tip:



Note: If the GC interface has not been previously fitted to the instrument, the interface fitting tool is secured to the interface flange by two thumbscrews. In this case, follow the procedure described in [Working with the interface fitting tool on page 6-6](#).

10. Ensure that the Viton O-ring is in the groove in the GC interface flange.

11. Ensuring that the heater connection on the interface flange is at the bottom of the assembly. Then fit the GC interface to the instrument, and secure it using the two retaining screws.
12. The GC column can now be installed, or the transfer line blanked, to allow source housing pumping.

See also: [Installing the GC column on page 6-9.](#)

Reinstallation of the GC interface

If you previously installed the GC interface and determined the transfer line position, you need not renew the 1/4-inch ferrule when reinstalling the interface.

See also: [Determining the transfer line position on page 6-3.](#)

To reinstall the GC interface:

1. Loosen the 1/4-inch nut retaining the inner transfer line in the GC interface assembly and pull the transfer line back a short way.
Tip: The 1/4-inch ferrule is usually be tight on the transfer line and its position does not change.
2. Fit the GC interface to the instrument.
3. Push in the transfer line to the position dictated by the 1/4-inch ferrule, and then tighten in position.

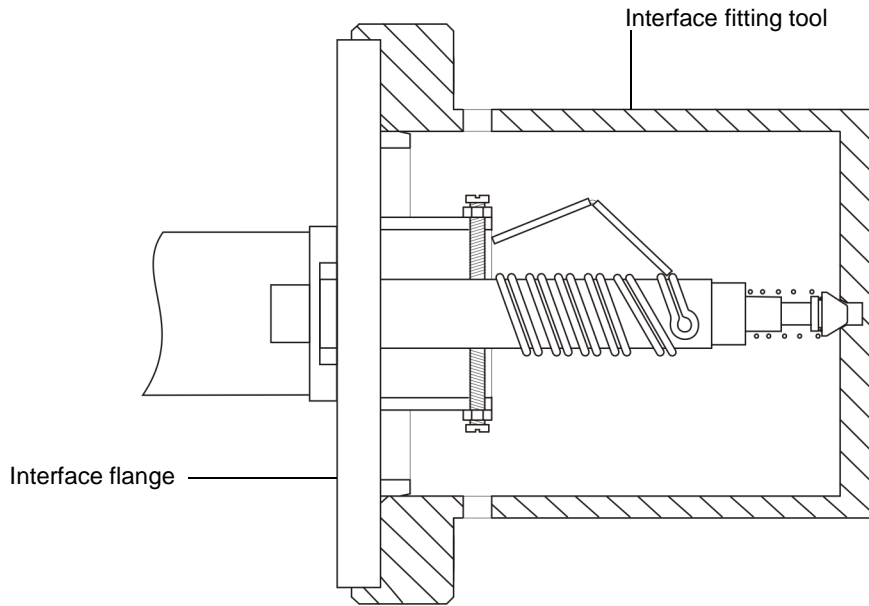
Working with the interface fitting tool

To use the interface fitting tool:

1. Remove the two thumb screws and separate the interface fitting tool and GC interface.

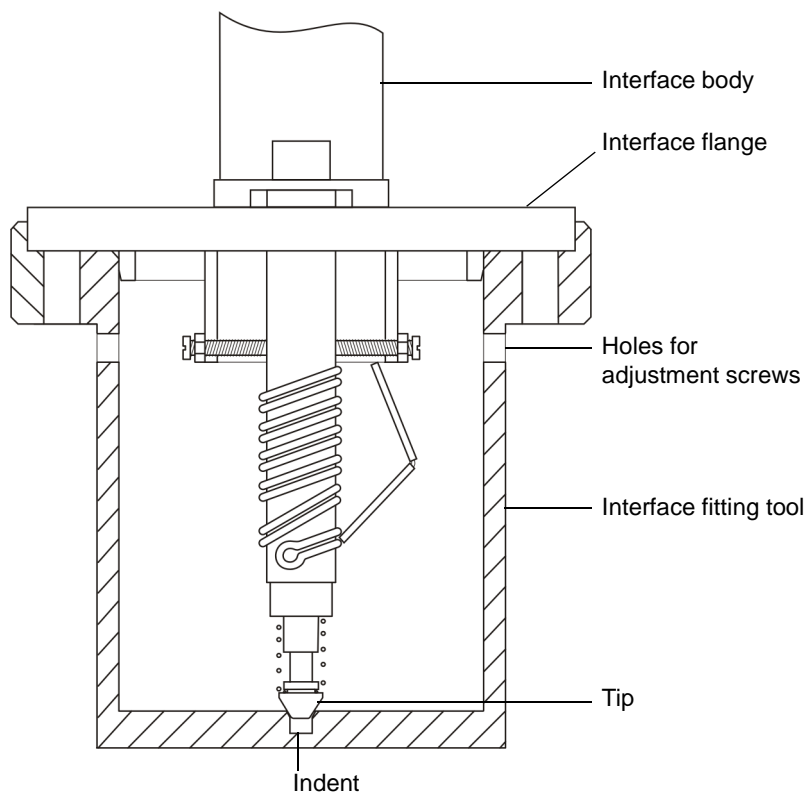
Requirement: Ensure that the Viton O-ring is not in place.

GC interface and interface fitting tool as supplied:



2. Replace the interface fitting tool, then place this and the GC interface upright on the bench. Ensure that the fitting tool fits evenly over the locating ring and interface flange.

GC interface with fitting tool attached for checking the tip position:



3. Look through the slot in the side of the interface fitting tool and assess the alignment of the tip; the tip should be central to the indentation in the interface fitting tool.

4. If required, change the position of the heater tube.

See also: [Changing the position of the heater tube on page 6-9.](#)

5. Fit a new 1/4-inch ferrule and the 1/4-inch retaining nut over the transfer line.



Caution: Fitting the tip too tightly damages the outer source as the interface expands thermally.

6. Ensure the tip is touching the indentation in the interface fitting tool without compressing the spring.

7. When the tip is in the correct position, tighten the 1/4-inch retaining nut, taking care not to change the position of the transfer line and tip.
8. Fit the 1/16-inch nut and graphitized Vespel ferrule to the transfer line.
9. Remove the GC interface from the interface fitting tool.

Changing the position of the heater tube

To change the position of the heater tube:

1. Slacken the lock nuts on the three adjustment screws.
See also: [Figure titled “GC interface tip:” on page 6-5.](#)
2. Adjust the screws to position the tip centrally.
3. Tighten the lock nuts.

Installing the GC column



Warning: To avoid contamination from samples wear, chemical-resistant, powder-free gloves at all times when handling the column.



Caution: Ensure that the helium supply is connected to the GC.

See also: The GC User Manual for more detailed information.

To install the GC column if it has not previously been fitted:

1. In the MassLynx Tune window, click Vacuum > Vent Source.
2. Wait for the Vacuum Status LED to display steady yellow.
3. Remove the 1/16-inch blanking nut from the GC interface.
4. Fit one end of the column into the GC injector.
Tip: The recommended position of the column in the GC injector is given in the GC manufacturer's instructions.
5. Remove the inner source.

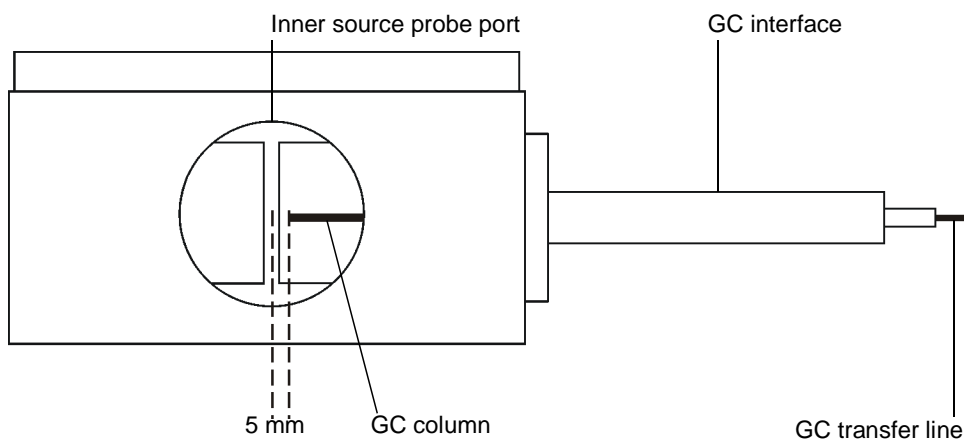
See also: [Changing the EI/CI inner source assembly on page 2-2](#) and [Changing the FI/FD inner source assembly on page 9-7.](#)

6. Insert the GC column through the GC interface transfer line until the end of the column is approximately 5 mm from the center line of the source.

Tip: To determine the correct position, look into the inner source probe port from the front with the inner source removed. The end of the GC column will be visible as it enters the source housing, as shown in the following figure.

Tip: For CI operation, the column can be positioned closer to the center line of the source.

Positioning the end of the GC column:



Caution:

- To avoid compromising sample data, ensure that no correction fluid enters the vacuum side of the instrument (see [step 7](#)).
 - To avoid damaging the GC column and inner source chamber assembly, the GC column must be retracted so that its end is within the outer source housing before fitting the inner source (see [step 8](#)).
7. Use correction fluid or a permanent marker to mark the position of the column relative to the 1/16-inch retaining nut.
 8. Retract the GC column by 50 mm into the transfer line or pull back the interface until it reaches the stopper.

9. Fit the inner source.
See also: [Changing the EI/CI inner source assembly on page 2-2](#).
10. Push the column back to the position marked in [step 7](#) so that it lies within the inner source.
11. Tighten the 1/16-inch retaining nut.
The system is now ready to pump-down (evacuate).
See also: [Starting-up the instrument on page A-2](#) and [Pumping/venting the source on page B-8](#).

Configuring the GC and autosampler

The GC and autosampler must be configured before using the GCT Premier. Configuration is carried out using the MassLynx Inlet Configuration Wizard.

See also: The MassLynx Inlet Control Guide for details.

Removing the GC interface



Warning: To avoid burn injuries, take care when working with the GC interface and GC oven. They operate at high temperatures.

To remove the GC interface:

1. On the MassLynx Tune Window, click Vacuum > Vent Source.
2. Wait for the Vacuum Status LED to display steady yellow.
3. Wait for the GC interface to cool.
4. Loosen the 1/16-inch column retaining nut on the GC interface inside the GC oven.
5. Withdraw the GC column from the GC interface.
6. Turn the black knobs on the side of the GC plinth.
Result: The two GC plinth stops are released.
7. Push the GC away from the GC interface.
8. Disconnect the GC interface heater connection.

9. Remove the two GC interface retaining thumbscrews.
10. Remove the GC interface.

7

The Direct Insertion Probe

This chapter describes the Direct Insertion (DI) probe and its operation.

Contents:

Topic	Page
Introduction	7-2
Installing the probe insertion lock	7-2
Inserting and removing the DI probe	7-4
Controlling the DI probe temperature	7-10

Introduction

The direct insertion (DI) probe is designed to admit solid samples, or liquid samples not sufficiently volatile to inject via the reference inlet, into the ion source. The DI probe is inserted into the instrument source via the probe insertion lock, which must be fitted to the instrument prior to using the probe.

The sample is loaded into a quartz sample cup, which is then located in the end of the probe. The cup is held in position by a thin strip of tungsten foil. A heating element and thermocouple are also located at the end of the probe.

A fine, stainless steel capillary, is wound close to the heater element. The capillary carries cooling water, which allows the sample temperature to be finely controlled as well as providing rapid probe cooling.

The thermocouple monitors the probe temperature. The heating current is controlled by MassLynx to maintain the required temperature ramp rate and final temperature.

Note: For more information about using the DI probe in FI mode, see [Chapter 9 FI/FD Operation on page 9-1](#).

Installing the probe insertion lock

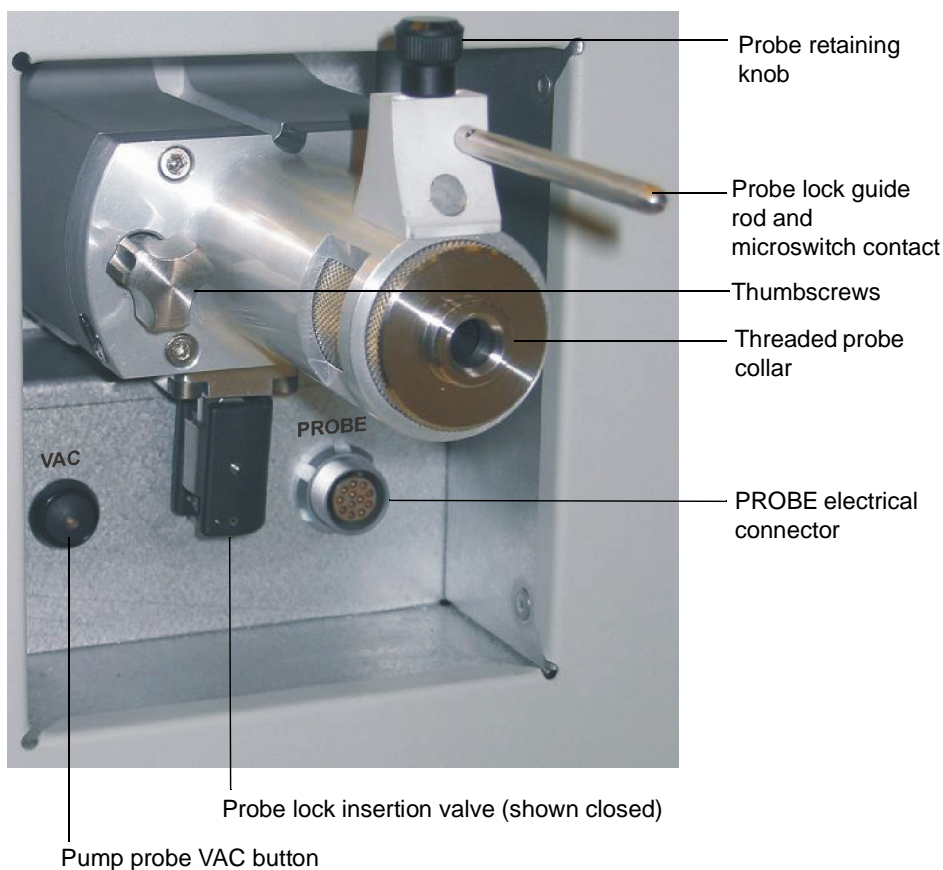
To install the probe insertion lock:



Warning: To avoid burn injuries, take care when working with the GC interface and GC oven as they operate at a high temperatures.

1. Remove the GC interface.
See also: [Installing the GC interface on page 6-3](#).
2. Ensure that the probe insertion lock valve is in the closed position.
See also: [Figure titled “Probe insertion lock:” on page 7-3](#).

Probe insertion lock:



Tip: The Pump probe button is labelled VAC on the instrument.

3. Ensure that the Viton O-ring is present in the insertion lock retaining flange groove.
4. Attach the probe insertion lock to the source housing.
5. Tighten the two thumbscrews that secure the probe insertion lock to the source housing.
6. Open the reference reservoir pump valve.
7. On the MassLynx Tune window, click Vacuum > Pump Source.

Inserting and removing the DI probe

This section explains the following tasks:

- Loading sample into the probe.
- Inserting the probe.
- Removing the probe.

Loading sample into the DI probe



Warning: To avoid contamination with samples wear chemical-resistant, powder-free gloves at all times when handling the probe.



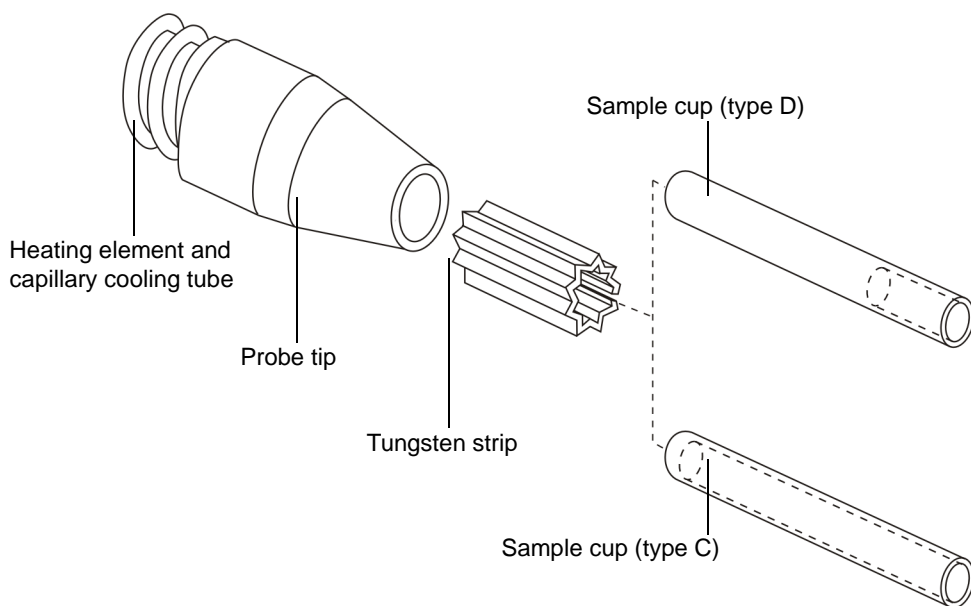
Caution: Do not use sample cups over 18 mm in length, which will damage the probe insertion lock valve.

Two types of quartz sample cup are available:

- For nonvolatile samples, the shallow cup (type D) is recommended. It is often unnecessary to heat the probe because the sample is close to the heated ion source.
- A deep cup (type C) is used for more volatile samples. The sample is placed at the bottom of the cup to reduce the heating effect of the ion source. The sample can then be evaporated into the ion source using the probe heater, if necessary.


Requirement: Used sample cups must be cleaned in a hot flame and/or washed in solvent and then allowed to cool and/or dry completely before being loaded with reasonable amounts of sample.

Direct insertion probe sample cups:



To load sample into the DI probe:

1. Push the sample cup firmly into the probe tip to ensure good thermal contact with the heater element.

 **Caution:** Ensure that the sample cup is retained firmly on the probe tip by the tungsten strip. Use two pieces of tungsten strip if necessary.

The sample cups can be loaded before or after inserting them into the probe.

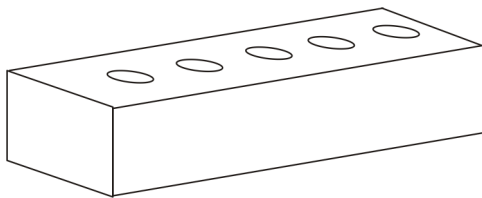
2. Load the solid samples into the cups using a thin piece of wire or a drawn-out length of glass rod.
3. Load samples in solution using a syringe.

Recommendation: Whenever possible, make the sample into a solution.

4. Ensure that the sample is deposited at the bottom of the cup and not around its neck, taking care not to leave any air pockets.

Tip: If several samples are to be run sequentially, load all the cups and stand them in the holder, provided with the DI probe.

Sample cup holder:



Any solvent used in loading can be evaporated by placing the sample cup(s) in a warm place, for example on top of the GC oven. To avoid solvent vapor blowing the sample out of the cup, be careful not to heat the cup too quickly.

To remove the sample cup after use:

Gently tap the sample cup out, or unscrew the tip of the DI probe to allow easy access to the cup.

Used sample cups must be cleaned in a hot flame or washed in solvent before using again.

Inserting the DI probe into the insertion lock



Caution: Damage to the instrument might occur if the probe insertion lock is operated with the probe incorrectly positioned.

To insert the loaded probe into the ion source:

1. Ensure that the instrument is pumped-down (evacuated) and ready for use.

See also: [Chapter A, Starting-up and Shutting-down the Instrument.](#)

2. Ensure that the probe insertion lock valve is in the closed position.

Tip: Lubricate the probe shaft with molybdenum disulphide to ensure smooth travel when inserting it into the probe lock.

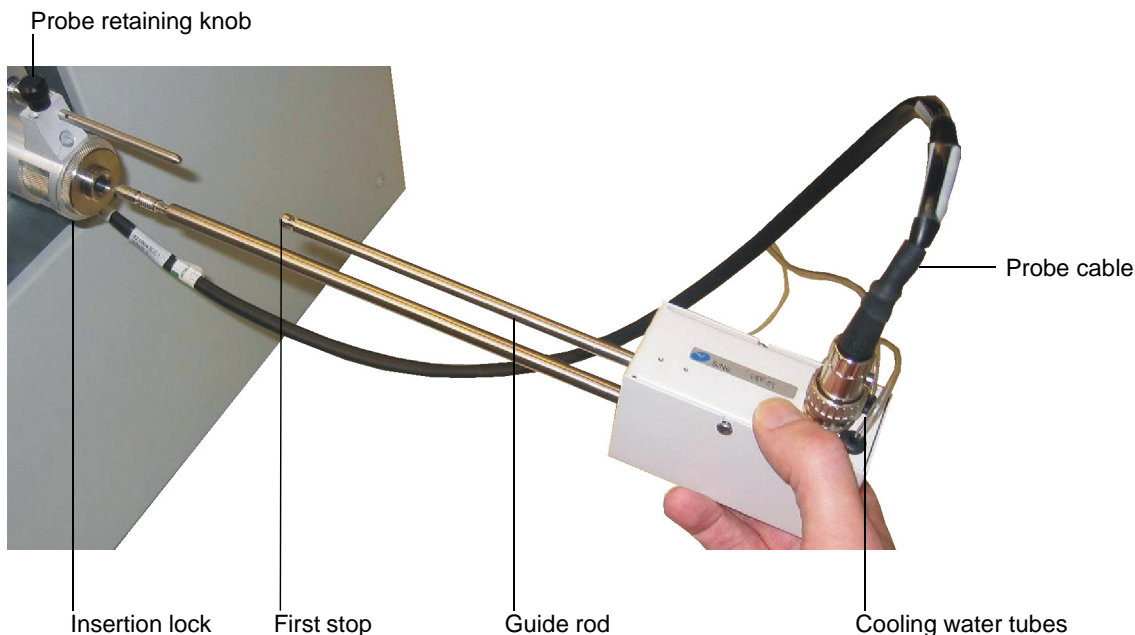


Caution: Do not use Santovac oil as a lubricant on the probe shaft; it causes a contamination peak at 446 Da in EI.

3. Insert the probe into the probe insertion lock until the probe reaches the first stop position.

Tip: The instrument has a microswitch to prevent the probe heater being activated if the probe is not fully inserted.

Inserting the DI probe:



4. Connect the cooling water tubes to the water chiller or other suitable water cooling system. Connectors are available for use with common water chillers or mains water.



Caution: To avoid damage to the instrument, do not apply more than 40 psi to the water connection.

5. Connect the probe's lead to the probe connector on the instrument's side panel.
6. Press the VAC button on the instrument's side panel.

Tip: In the MassLynx Tune window, click Vacuum > Monitor to display the vacuum levels. You should see the backing pressure rise and then fall into the green region as the probe insertion lock is pumped out (evacuated).

7. Wait until the green light in the center of the VAC button illuminates.
Rationale: The light indicates that the inlet pressure has fallen below the trip level set on the Pirani gauge. If it fails to illuminate check the insertion lock for leaks and the insertion lock O-ring for damage.
8. Open the probe lock insertion valve.
9. Pull back the spring-loaded probe retaining knob (located on the probe insertion lock).
10. Slowly push the probe fully in.
11. For CI operation, ensure that the probe is fully in by adjusting the threaded probe stop-collar.

Rationale: The probe creates a seal that retains the CI gas inside the inner source.



Caution: Do not leave the DI probe in the retracted position when not in use. Remove the probe from the instrument.

Removing the DI probe from the probe insertion lock

To remove the probe:

1. On the MassLynx Tune window Inlet tab, set Solids Probe to 20.



Warning: The probe is very hot and can cause burn injuries; take care when withdrawing the probe.



Caution: To avoid damage to its lock seals, allow the probe to cool below 100 °C before withdrawing it.

2. Wait for the probe to cool.



Caution: To avoid damage to the instrument, close the probe insertion lock valve before withdrawing the probe further than the first stop.

3. Withdraw the probe to the first stop only.
4. Close the probe insertion lock valve.

5. Pull back the spring-loaded probe retaining knob (located on the probe insertion lock).
6. Withdraw the probe from the probe insertion lock.

Controlling the DI probe temperature

Source temperature is often sufficient to evaporate the sample, particularly if the shallow sample cup is used. If the sample fails to evaporate, or if a more controlled evaporation is required, MassLynx can ramp the probe temperature during acquisition.

Ramping the probe temperature manually

To ramp the probe temperature manually:

1. In the MassLynx Tune window Inlet tab, ensure that the Use DCI mode check box is *not* selected.

Tune window Inlet tab:

The screenshot shows the 'Inlet' tab of the MassLynx Tune window. It is divided into three sections: 'Probe', 'Inlet', and 'CI'.
- The 'Probe' section contains a 'Use DCI mode' checkbox (unchecked), an 'Edit ramps' button, and a 'Direct Insertion (DI) Probe (C)' control with a numeric input field set to 0 and a slider.
- The 'Inlet' section contains three temperature controls: 'GC Re-entrant (C)' with a numeric input field set to 0 and a slider, 'Reference Reservoir (C)' with a numeric input field set to 0, and 'Reference Re-entrant (C)' with a numeric input field set to 0.
- The 'CI' section contains a 'CI Gas' checkbox (unchecked), a 'CI Purge' button, and a 'CI Gas Flow (%)' control with a numeric input field set to 0 and a slider.

2. Enter the required temperature in the Solids Probe text box.
3. Press the Enter key.

Alternative: Use the slider control to select the required temperature.

Tip: For safety reasons, the temperature is set to 0 at the start of an acquisition. Once the acquisition has started, you can manually ramp the probe temperature using this control.


The temperature readback should rise to match the set temperature.

Note: A temperature readback of 850 °C indicates that the probe cable has not been correctly connected.

Ramping the probe temperature automatically

The Probe Control dialog box automatically ramps the probe temperature.

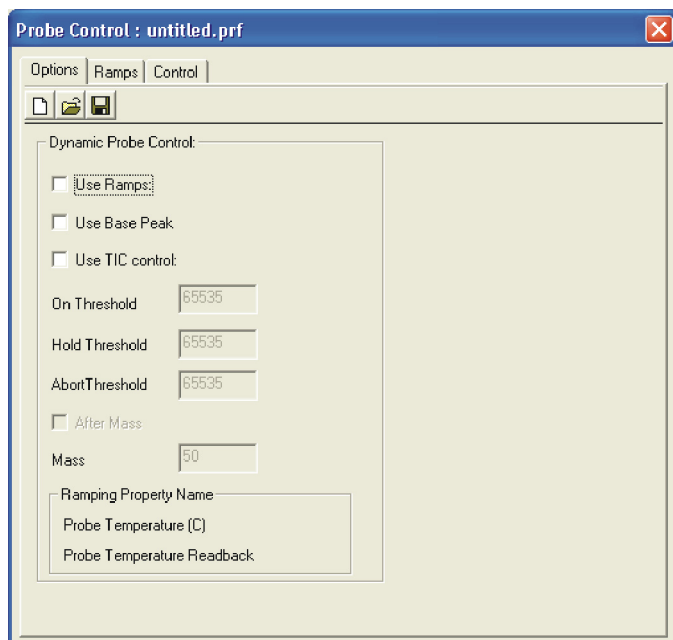
To open the Probe Control dialog box:

In the MassLynx Tune window toolbar, click .

Alternative: Click Settings > Probe Ramping.

Probe Control dialog box Options tab

Probe Control dialog box Options tab:






The Dynamic Probe Control prevents saturation of the detector.

The Options tab parameters are described in the [Table titled “Probe Control dialog box Options tab parameters:” on page 7-12.](#)

Probe Control dialog box Options tab parameters:

Parameter	Description
Use Ramps	Enables probe ramp control.
Use Base Peak	Enables probe temperature regulation based on the base peak intensity. The base peak is the most intense peak within the scanned mass range.
Use TIC control	Enables probe temperature regulation based on the Total Ion Current (TIC).
On Threshold	When temperature regulation is enabled, this parameter specifies the intensity (of the current property used for regulation: for example, Base Peak or TIC). At that, the probe ramp intensifies will increase the probe temperature after a hold or abort event. Tip: The specifies value is in counts.
Hold Threshold	Specifies the intensity at which the probe ramp control will hold the probe temperature until the intensity of the current property reaches the On Threshold or Abort Threshold.
Abort Threshold	Specifies the intensity at which the probe ramp control is turned off until the intensity reaches the On Threshold.
After Mass	If Use Base Peak is selected, masses below the value entered in the associated Mass text box are ignored when calculating the base peak value.
Ramping Property Name pane	Displays which device will be ramped, and which of its properties will be controlled. Tip: Depending on which probe is selected, the parameters displayed. See also: Figure titled “Tune window Inlet tab:” on page 7-10.

Probe Control dialog box Options tab toolbar buttons:

Button	Description
	Allows creation of a new probe ramping method file.
	Allows an existing probe ramping method file to be opened.
	Saves the probe ramping method file. See also: Use Probe Ramping Method on page 4-4 and Use Probe Ramping Method on page 4-14 .

When the Use Base Peak or Use TIC control option is selected, the base peak or TIC is monitored as the probe temperature ramp is applied. If it achieves the Hold Threshold value, the probe temperature is held at its current value until one of the following conditions applies:

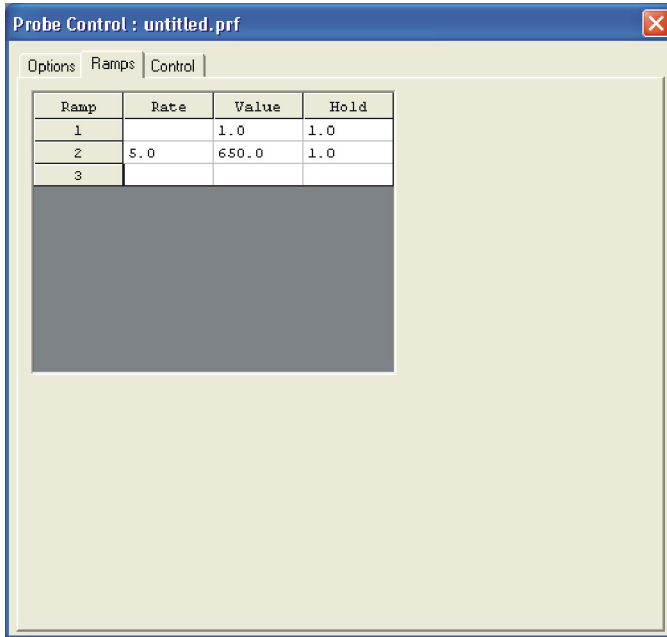
- The base peak/TIC drops to the On Threshold value, in which case the probe temperature ramp resumes.
- The base peak/TIC rises to the Abort Threshold value, in which case the probe heating is turned off until the base peak/TIC value drops below the On Threshold value.

The ramp is defined in the Ramp tab of the Probe Ramp dialog box.

Probe Control dialog box Ramps tab

You set multiple probe ramps in the Ramps tab.

Probe Control dialog box Ramps tab:



Probe Control Ramps tab parameters:

Parameter	Description
Ramp	The ramp identifying number.
Rate	The rate of the ramp, in units of °C/minute for the DI and A/minute for the DCI probe.
Value	Displays the ramp's final target temperature value, in °C for the DI probe and A for the DCI probe.
Hold	The time (in minutes) for which to hold the value, once it is reached.

Tip: The rate at which the probe is heated can be critical. It varies with the type of sample. Experiments may be required to establish the optimum conditions for evaporating of individual samples.

To create probe ramping data:

1. Type the required initial ramp value in the Value field, then press Enter.

Result: The value is entered in the field, and the focus moves to the Hold field.

2. Type the required start time for the ramp in the Hold field, then press Enter.

Result: The value is entered in the field, and a new line is created, with the focus in the Rate field.

3. Type the required value in the Rate field, then press Enter.
4. Repeat [steps 1 through 3](#) for the Value and Hold fields. When Enter is pressed for the Hold field, a new row (Ramp 2) is created.
5. Repeat [steps 3 and 4](#) as required to create additional probe ramps.

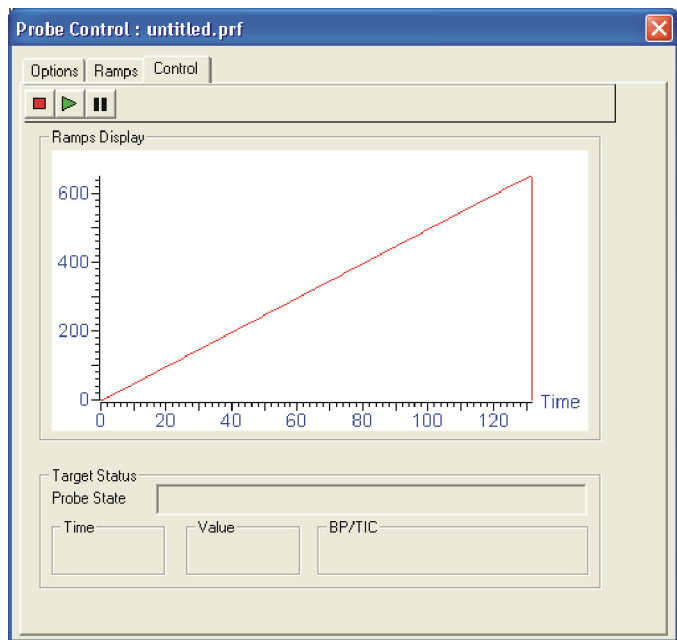
To edit probe ramping data:

1. Double-click the text box to be edited.
2. Type the new value in the box.
3. Press Enter.
4. Repeat [steps 1 through 3](#).

Probe Control dialog box

In the Control tab of the Probe Control dialog box, you manually run probe ramp methods. The tab also visually represents ramping method as it runs.




Probe Control dialog box Control tab:



Probe Control dialog box Control tab:

Parameter	Description
Ramps Display	Shows a graph of the active probe ramp against time. Both the target value and the actual value are shown to indicate how accurately the ramp profile is being followed.
Probe State	Displays the active state of the probe ramp.
Time	Displays the time, in minutes.
Value	Displays the active ramp value, in units appropriate to the probe type. For example, °C for DI probe and A for DCI probe.
BP/TIC	Displays the Base Peak or TIC intensity, as appropriate.

Probe Control dialog box Control tab toolbar buttons:

Button	Description
	Starts the active probe ramping method.
	Stops the active probe ramping method.
	Pauses the active probe ramping method.

Ramping the probe during a acquisition

A probe ramp can be saved as a .prf file and run from a Tune window acquisition or via the Sample list. This is done by referencing the .prf file from the acquisition tab.

See also: [Use Probe Ramping Method on page 4-4](#) and [Use Probe Ramping Method on page 4-14](#).

8 The Direct Chemical Ionization (DCI) Probe

This chapter describes the Direct Chemical Ionization (DCI) probe and its operation.

Contents:

Topic	Page
Introduction	8-2
Installing the probe insertion lock	8-2
Inserting and removing the DCI probe	8-3
Controlling the DCI probe current	8-7

Introduction

Direct chemical ionization (DCI) was originally developed as a soft ionization technique for relatively nonvolatile samples for use with EI/CI mass ionization spectrometry. The sample is placed on a filament wire, which is inserted directly into the ion source so that the filament tip is in the ion stream. A current is passed through the filament and the sample is rapidly desorbed. The desorption yields a high abundance of the molecular ion $[M+H]^+$.

On the GCT Premier mass spectrometer, the sample is loaded onto the DCI probe tip and, once inserted in the source, desorbed by ramping the probe tip current. The DCI probe is inserted into the source via the probe insertion lock, which must be fitted to the instrument prior to using the probe.



Caution: To avoid damage to the DCI probe, it must be used with the modified DCI inner source.

The DCI probe is supplied with a DCI inner source, with a larger entry hole than the standard CI inner source, to accommodate the probe tip. The DCI inner source only for DCI work.

Note: The DCI probe can also be used in EI mode with the standard EI inner source. The controls are accessed the same way as in CI. Using the DCI probe in EI mode is quicker than using the DI probe. It requires no water cooling, and it tends to produce less fragmentation.

Note: The DCI probe must not be used in FI mode.

Installing the probe insertion lock

To install the probe insertion lock:



Warning: To avoid burn injuries, take care when working with the GC interface and GC. They operate at high temperatures.

1. Remove the GC interface.

See also: [Removing the GC interface on page 6-11.](#)

2. Ensure that the probe insertion lock valve is in the closed position.

See also: Figure titled “Probe insertion lock.” on page 7-3.

3. Ensure that the Viton O-ring is present in the insertion lock retaining flange groove.
4. Attach the probe insertion lock to the source housing.
5. Tighten the two thumb screws that secure the probe insertion lock to the source housing.
6. Fit the DCI inner source.

See also: [Installing the EI/CI inner source on page 2-4.](#)

7. Open the reference reservoir pump valve.
8. In the MassLynx Tune window, click Vacuum > Pump Source.

Inserting and removing the DCI probe

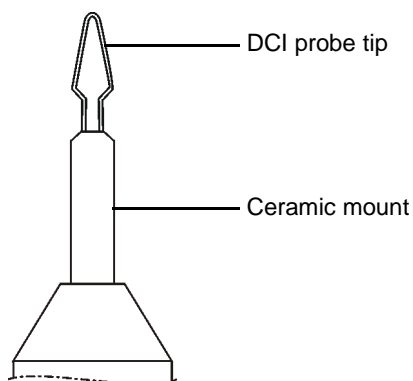
This section explains the following tasks:

- Loading sample into the probe.
- Inserting the probe.
- Removing the probe.

Loading sample into the DCI probe

The DCI probe tip consists of a small diamond of rhenium wire on a ceramic mount.

DCI probe tip:





Warning: To avoid contamination from samples, wear chemical-resistant, powder-free gloves at all times when handling the probe.

To load sample into the DCI probe:



Caution: The wire filament at the end of the DCI probe tip is fragile. Be careful not to damage the tip when handling it.

1. Push the DCI tip onto the end of the DCI probe.
2. Dissolve the sample in a volatile GC-grade solvent such as methanol to a concentration of 1 ng/ μ L.

Tip: If necessary, to increase the amount of sample introduced via the tip, use a correspondingly stronger solution. It is common to run samples of between 1 ng and 1 μ g.

3. Deposit 1 μ L (1 ng of sample) on the end of the DCI probe tip using a syringe.

Tip: The droplet stays at the tip due to surface tension.

4. Wait for the solvent to evaporate.
5. Insert the probe into the source.

See also: [Inserting the DCI probe into the insertion lock on page 8-4.](#)

Inserting the DCI probe into the insertion lock



Caution: Damage to the instrument might occur if the probe insertion lock is operated with the probe incorrectly positioned.

To insert the loaded probe into the ion source:

1. Ensure that the instrument is pumped-down (evacuated) and is ready for use.

See also: [Chapter A, Starting-up and Shutting-down the Instrument.](#)

2. Ensure that the probe insertion lock valve is closed.

Tip: Lubricate the probe shaft with molybdenum disulphide to ensure smooth travel when inserting it into the probe lock.



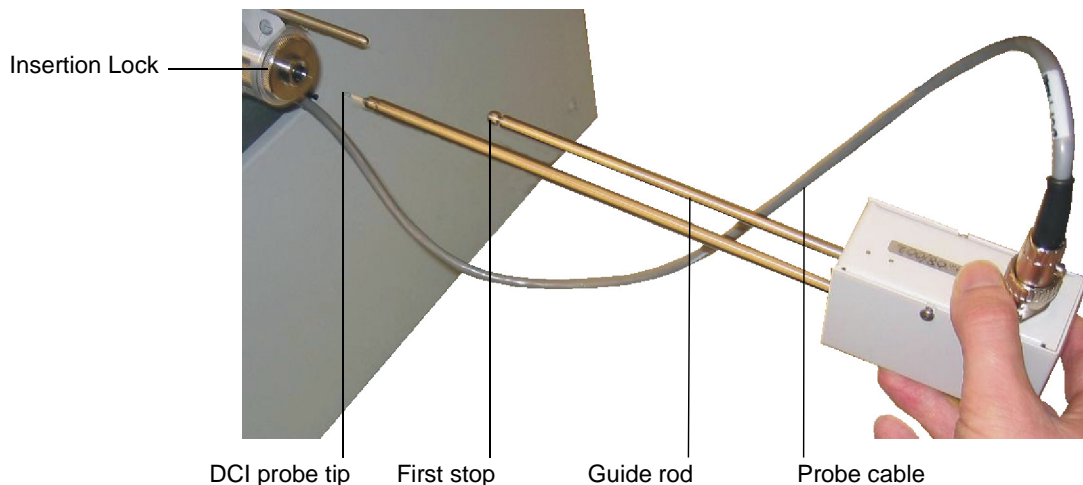
Caution:

- Do not use Santovac oil as a lubricant on the probe shaft, it causes a contamination.
- The wire filament at the end of the DCI probe tip is fragile. Be careful not to damage the tip when handling it.

3. Insert the probe into the probe insertion lock until the probe reaches the the first stop position on the guide rod.

Tip: The instrument has a microswitch to prevent the probe heater being activated if the probe is not fully inserted.

Inserting the DCI probe:



4. Connect the probe's lead to the probe connector on the instrument side panel.
5. Press the VAC button on the instrument side panel.

Tip: On the MassLynx Tune window, click Vacuum > Monitor to display the vacuum levels. You should see the backing pressure rise and then fall into the green region as the lock is pumped out (evacuated).

6. Wait until the green light in the VAC button illuminates.

Rationale: The light indicates that the inlet pressure has fallen below the trip level set on the Pirani gauge. If it fails to illuminate, check the insertion lock for leaks and the insertion lock O-ring for damage.

7. Open the probe insertion lock valve.
8. Pull back the sprung-loaded probe retaining knob (located on the probe insertion lock).



Caution: Ensure the probe is fully in by adjusting the threaded probe stop collar clockwise.

9. Slowly push the probe fully in.

Rationale: The probe creates a seal so that the CI gas is retained in the inner source.

Note: The threaded probe stop collar ensures the probe remains at required distance from the ion source.

Removing the DCI probe from the probe insertion lock

To remove the probe:

1. In the Tune window Inlet tab, set DCI Probe Current to 0.



Caution: To avoid damage to the probe, reduce the current before attempting to withdraw the probe from the probe insertion lock.

2. Withdraw the probe to the first stop only.



Caution: To avoid damage to the instrument, close the probe insertion lock valve before withdrawing the probe further than the first stop.

3. Close the probe insertion lock valve.

4. Pull back the spring-loaded probe retaining knob (located on the probe insertion lock).



Caution:

- Ensure the probe tip is not knocked during removal, as it is easily damaged.
 - To avoid damage to the probe filament, do not pass a current through the probe tip when the tip is in air.
5. Withdraw the probe from the probe insertion lock.

Controlling the DCI probe current

Ramping the probe current manually

To ramp the probe temperature manually:

1. In the MassLynx Tune window Inlet tab, select Use DCI mode.
2. Enter the required current in the DCI Probe text box. The ramp range is 0 to 1.6 A.

Tune window Inlet tab:

The image shows a software interface for the 'Inlet' tab. It is divided into three main sections: 'Probe', 'Inlet', and 'CI'.
- The 'Probe' section contains a checked checkbox for 'Use DCI mode', an 'Edit ramps' button, and a 'DCI Probe (A)' control with a numerical input of 0.00 and a slider.
- The 'Inlet' section contains three temperature controls: 'GC Re-entrant (C)' with a value of 251, 'Reference Reservoir (C)' with a value of 60, and 'Reference Re-entrant (C)' with a value of 61.
- The 'CI' section contains an unchecked checkbox for 'CI Gas', a 'CI Purge' button, and a 'CI Gas Flow (%)' control with a value of 0 and a slider.

3. Press Enter.

Alternative: Use the slider control to select the required current.

Tip: For safety reasons, the current is set to 0 at the start of an acquisition. Once the acquisition has started, you can manually ramp the probe current using this control.

Ramping the probe current automatically

The DCI probe current is controlled using the Probe Control dialog box the same way as the DI probe temperature is controlled.

See also: [Ramping the probe temperature automatically on page 7-11.](#)

9 FI/FD Operation

This chapter describes how to prepare the instrument for operation, including how to install and remove the inner and outer source options for Field Ionization (FI) and Field Desorption (FD).

Contents:

Topic	Page
Introduction	9-2
Preparing the instrument for FI/FD Operation	9-2
Setting-up for FI/FD operation	9-3
Tuning in FI mode	9-20
General considerations for tuning in FI mode	9-23
Setting up and calibrating the instrument	9-24
Running samples	9-26
Tuning in FD mode	9-27

Introduction

In field ionization (FI/FD), sample molecules pass close to the tips of a mass of carbon micro-needles on the emitter. Extraction rods maintained at high voltage produce a very high electric field around the needles. This causes ionization by quantum tunnelling of a valence electron. The process is very 'soft' often producing spectra with little or no fragmentation. The GCT Premier has a dedicated outer and inner source for FI and FD operation.

Preparing the instrument for FI/FD Operation

Choosing an emitter for the FI/FD inner source

FI emitter

Six FI emitters are supplied with the instrument (available from Carbotec [<http://www.carbotec.com>]). The emitters are made from 5- μm diameter tungsten wire. Emitters are also available from Linden (<http://linden.com>).

The advantages of 5 μm diameter emitters in FI are:

- High field strength at minimum extraction voltage.
- Minimum surface area into which analyte can condense.
- Very fast heating and cooling characteristics. This ensures fast recovery of sensitivity after flash off.

FD emitter

FD or all-round emitters give improved performance in FD mode of operation. These emitters have a 10 μm diameter wire and have longer and more highly branched carbon dendrite micro needles. Absolute sensitivity is significantly lower than when using the 5- μm diameter FI emitter.

An emitter with a higher surface area, such as a 10 μm emitter, that can partially act as a sample reservoir without completely suppressing sensitivity is required in FD mode.

See also: [Changing an emitter on page 10-33](#).

Setting-up for FI/FD operation

Recommendation: When using the FI/FD inner source probe fit the blank flange or the GC interface to the instrument. When using the FI/FD inner source probe with the DI probe, fit the FI/FD inner source direct insertion probe flange.

See also: [Installing the FI/FD inner source direct insertion probe only flange on page 9-9.](#)

Changing the FI/FD outer source assembly

Installing the FI/FD outer source

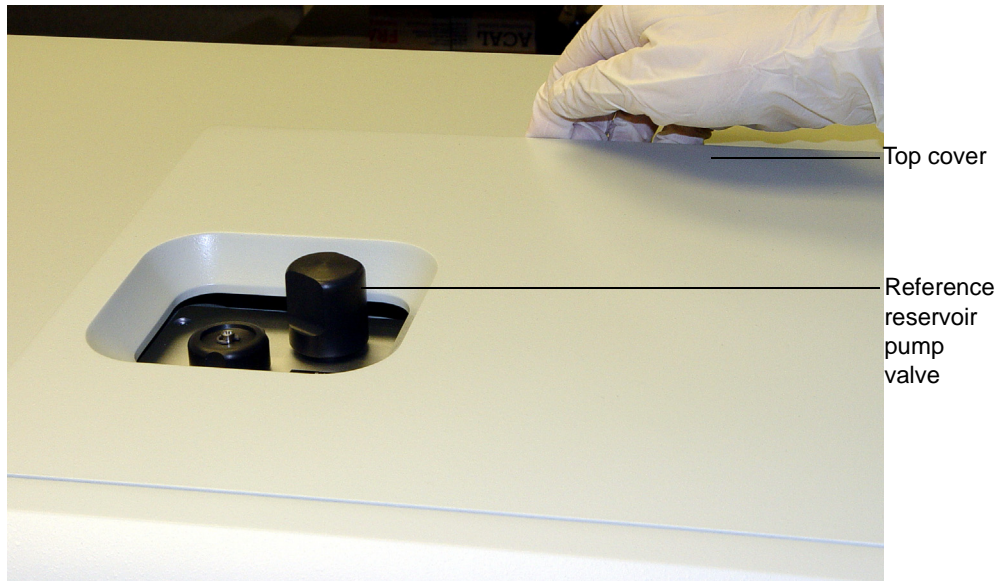


Warning: To avoid burn injuries, allow the outer source lid assembly to cool before handling.

To fit the FI/FDI outer source:

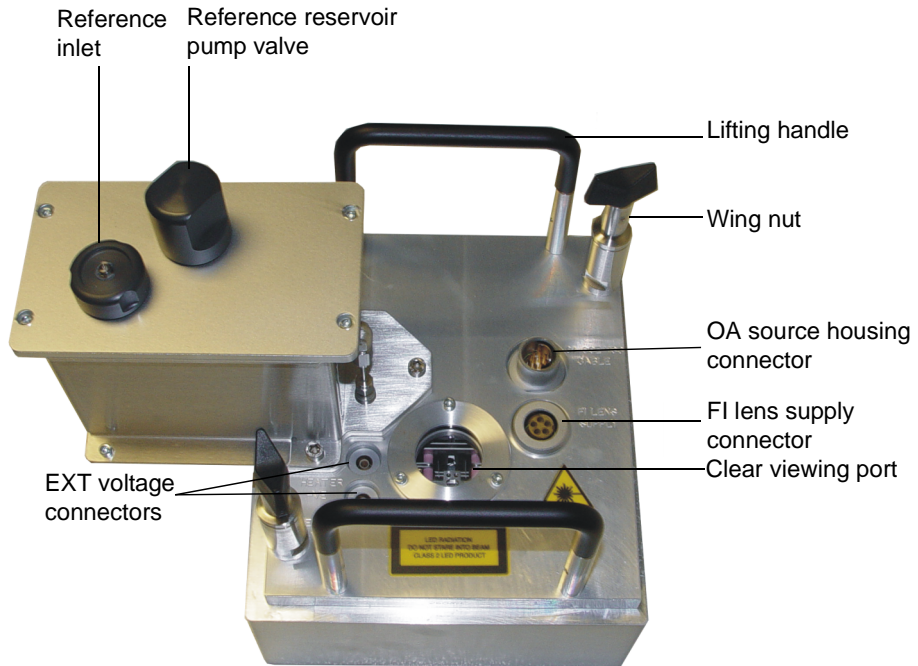
1. Remove the top cover from the instrument. Lift at the back and pull forwards to remove.

Note: FI and FD operation are run without the top cover fitted on the instrument. This allows you to see inside the source through the clear viewing port.



2. Remove the EI/CI outer source assembly from the instrument.
See also: [Removing the EI/CI outer source from the instrument on page 2-6.](#)
3. Carefully lower the FI/FD outer source lid assembly vertically into the outer source enclosure using the two studs to ensure correct alignment.

FI/FD outer source:



4. Tighten the two wing nuts on the top of the outer source lid assembly.
5. Connect the two cables to the extraction voltage (EXT voltage) connectors.
6. Connect the cable to the OA source housing connector.
7. Connect the cable to the FI lens supply connector.
8. Install the FI/FD inner source.

See also: [Installing the FI/FD inner source lock assembly on page 9-7.](#)

Removing the FI/FD outer source



Warning: The source components can be contaminated with toxic and biohazardous materials. Wear chemical-resistant, powder-free gloves at all times while handling the components.



Warning: The source is hot and can cause burn injuries to the skin. Allow the source to cool before handling it.

To remove the FI/FD outer source:

1. In the MassLynx Tune window, click Vacuum > Vent Source.
2. Wait for the Vacuum Status LED to display steady yellow.



Warning: To avoid burn injuries, allow the GC column and interface to cool before handling.

3. If fitted, pull back the carrier tube in the GC interface or pull back the whole interface to the lock point.

See also: [Installing the GC interface on page 6-3.](#)

4. Remove the inner source from the outer source.

See also: [Removing the FI/FD inner source on page 9-13.](#)

5. Disconnect the cables from the FI lens supply.
6. Disconnect the OA source housing cable.
7. Disconnect the EXT cables.
8. Unscrew the two wing nuts at the top of the outer source assembly.
9. Use the two handles to lift the outer source assembly vertically from the outer source enclosure.

Changing the FI/FD inner source assembly

Installing the FI/FD inner source lock assembly

To install the FI/FD inner source lock assembly:

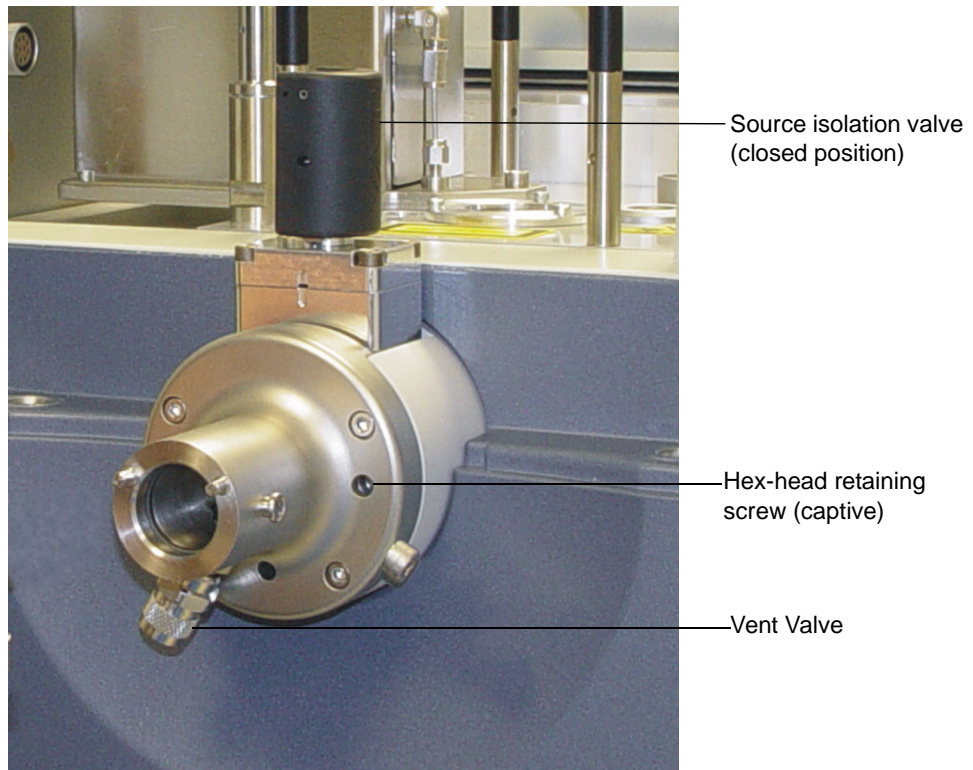


Caution: To avoid damaging the GC column, ensure the GC column is retracted before installing the FI/FD inner source assembly.

1. Ensure that the GC column is retracted.
See also: [Installing the GC column on page 6-9.](#)
2. Ensure that you are about to install the correct inner source.
See also: [Inner source assembly on page 1-4.](#)
3. On the inner source assembly, verify that the O-rings:
 - Sit correctly in the groove.
 - Are in good condition.
 - Are clean and free from dust.
4. Carefully fit the inner source lock assembly on the guide rods and present it centrally to the outer source so that the inner source assembly flange is flush with the outer source cover.
5. Use a 4-mm hex (Allen) key to tighten the two retaining screws securing the FI/FD inner source assembly to the outer source.

See also: [Figure titled “FI/FD inner source lock assembly:” on page 9-8.](#)


FI/FD inner source lock assembly:



6. Ensure that the source isolation valve is closed.
When the outer source and the inner source lock assembly has been fitted you can pump the instrument down.
7. Open the reference reservoir valve.
8. In the MassLynx Tune window, click Vacuum > Pump Source.
When the pneumatic valve opens, you should hear a small hiss.
9. Ensure that the Vacuum LED shows flashing green followed by steady green.
See also: [Status display on page 1-9.](#)
10. When the Source Turbo value in the Turbo Readbacks frame reaches 80 or more, close the reference reservoir valve.

Removing the FI/FD inner source lock assembly

To remove the FI/FD lock assembly:

1. In the Tune window, click .
2. Click Vacuum > Vent Source.
A message confirms the vent command.
3. Click OK.
The turbomolecular pump is turned off. When the turbomolecular pump has run down to half normal operating speed, the vent valve is opened and the instrument is automatically vented.
4. Wait for the Vacuum LED on the instrument front panel to display yellow or red.
5. Use a hex (Allen) key to unscrew the two hex head bolts which retain the inner source lock assembly to the outer source.
6. Carefully lift the inner source lock assembly away from the outer source.

Installing the FI/FD inner source direct insertion probe only flange

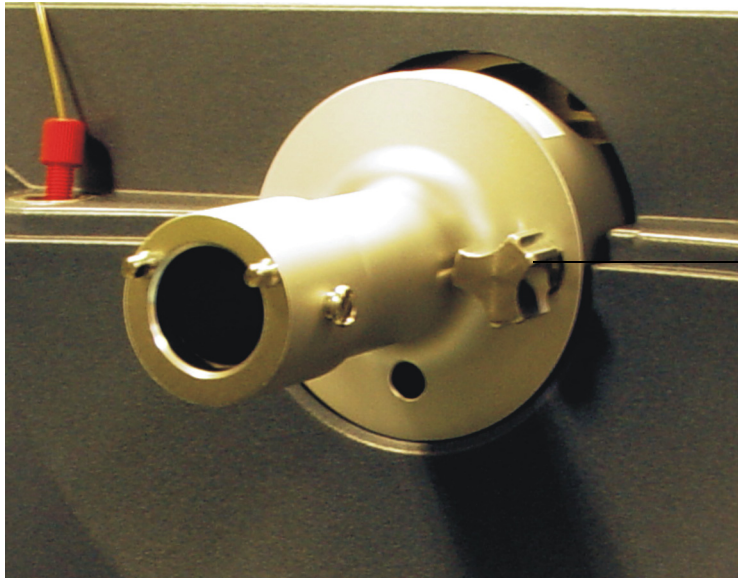
The FI/FD inner source lock assembly is not compatible with the direct insertion probe and insertion lock assembly.

To use the direct insertion probe insertion lock in FI mode, you must use the FI/FD inner source direct insertion probe only flange.

To fit install the FI/FD inner source direct insertion probe only flange:

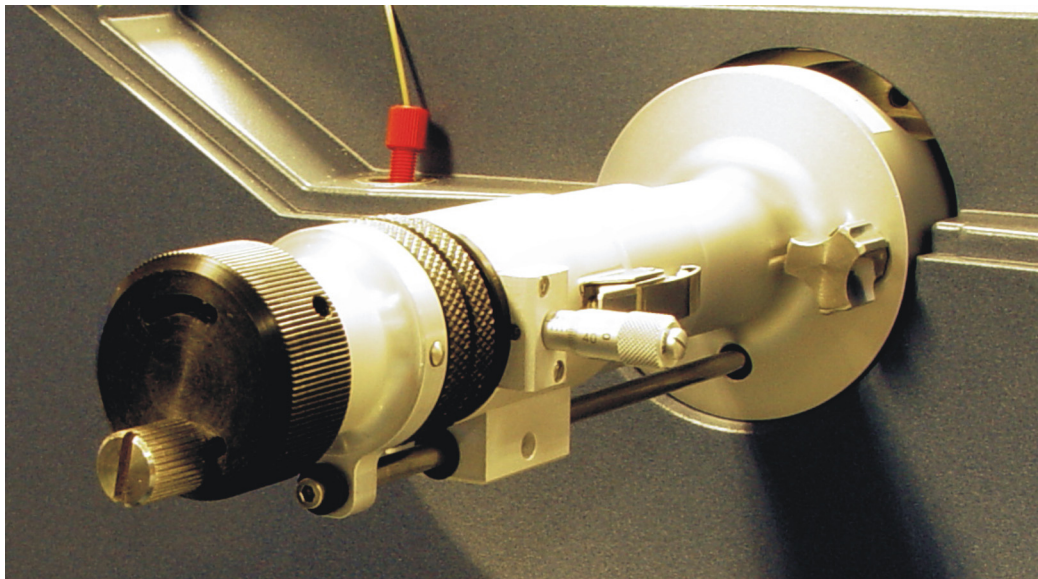
1. On the inner source assembly, verify that the O-rings:
 - Sit correctly in the groove.
 - Are in good condition.
 - Are clean and free from dust.
2. Carefully fit the FI/FD inner source direct insertion probe flange on the guide rods and present it centrally to the outer source so that the inner source assembly flange is flush with the outer source cover.
3. Tighten the two retaining thumbscrews securing the FI/FD inner source direct insertion probe only flange to the outer source.

FI/FD inner source direct insertion probe flange:



Thumbscrews

FI/FD inner source direct insertion probe flange with probe:



Inserting the FI/FD inner source probe

When the inner source lock assembly has been fitted, you can insert the FI/FD inner source probe with either an FI or FD emitter fitted. The FI/FD inner source probe can be fitted and removed without venting the source chamber by using inner source lock assembly.



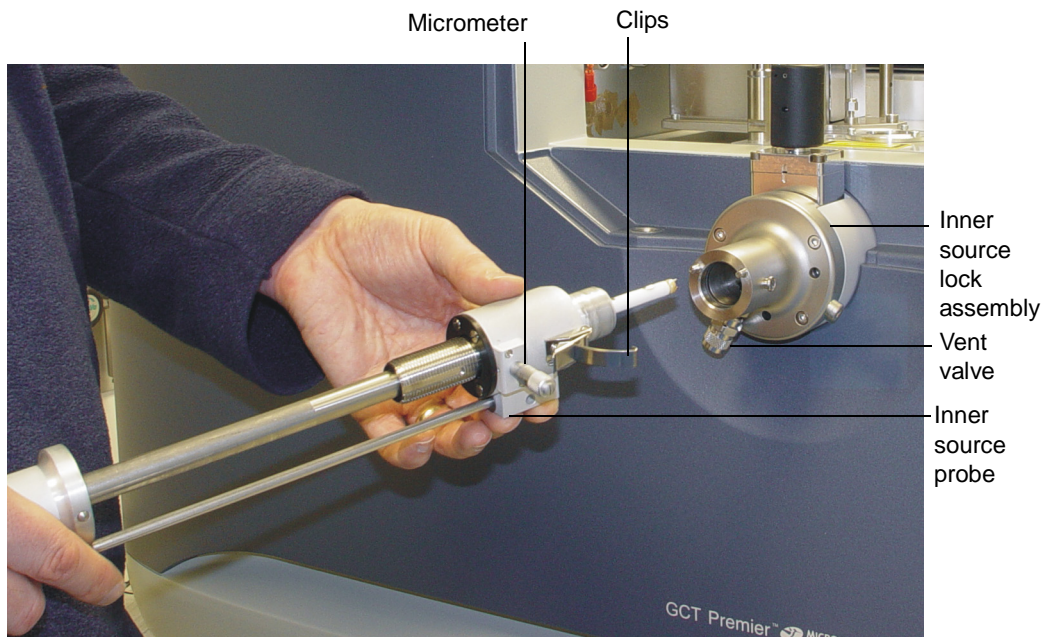
Caution: To avoid damage when installing the inner source assembly, be careful not to knock the emitter.

See also: [Figure titled “FI/FD inner source lock assembly:” on page 9-8](#)

To insert the FI/FD inner source probe:

1. Fit the correct emitter to the inner source shaft.
See also: [Changing an emitter on page 10-33.](#)
2. Ensure that the shaft is fully pulled back.
3. Carefully present the inner source probe centrally to the inner source lock assembly.

Presenting the inner source probe to the inner source lock assembly:



4. Fasten the two clips at each side of the FI/FD inner source probe.




Caution: Do not leave the FI/FD inner source probe in the retracted position when not in use. Remove the probe from the instrument.

5. Ensure that the vent valve is closed (fully clockwise).
6. Press the VAC button on the right side of the instrument.

See also: [Figure titled “Probe insertion lock:” on page 7-3.](#)

7. Wait until the green light in the center of the pump probe switch illuminates.

Tip: On the MassLynx Tune window, click Vacuum > Monitor to display the vacuum levels. You should see the Backing pressure rise and then fall into the green region as the probe insertion lock is pumped out.

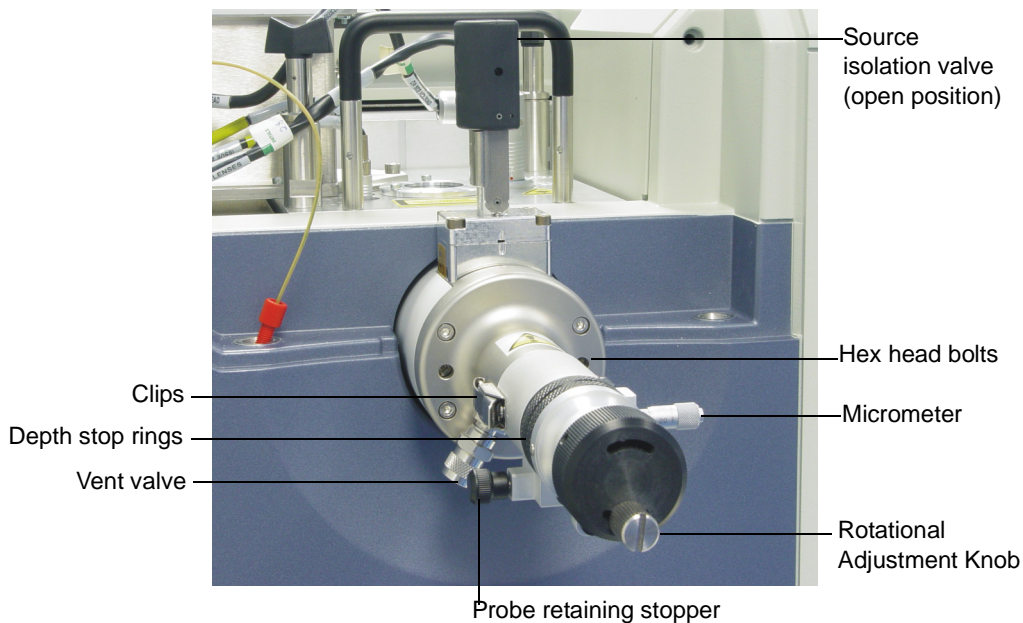
8. Click , to switch the instrument into Standby.

9. Open the source isolation valve.
10. Pull out the retaining stopper.
11. Push the FI/FD inner source probe into the outer source.
12. Adjust the micrometer until the emitter is centered between the two extraction rods.




Caution: Ensure that the emitter does not touch the extraction rods or the GC-FI column. You can view the position of the emitter through the outer source viewing port.

FI/FD inner source inserted:



Removing the FI/FD inner source

To remove the FI/FD inner source:

1. In MassLynx Tune window, click , to switch the instrument into Standby.
2. Withdraw the probe until the retaining stopper clicks in the first stop.



Caution: Do not leave the FI/FD inner source probe in the retracted position when not in use. Remove the probe from the instrument.

3. Close the source isolation valve.
4. Open the vent valve, you will hear a small hiss.
5. Close the vent valve.

6. Unfasten the two clips at each side of the FI/FD inner source probe.



Warning: To avoid burn injuries, do not touch the FI/FD tip. Allow the tip to cool before handling.

7. Pull the inner source probe away from the lock assembly.




Caution: To avoid damage when removing the inner source assembly, be careful not to knock the emitter.

Changing the FI/FD GC interface

Installing the FI/FD GC interface

To install the FI/FD GC interface:

1. In the Tune window, click  to switch the instrument into Standby.

2. Click Vacuum > Vent Source.

A message confirms the vent command.

3. Click OK.

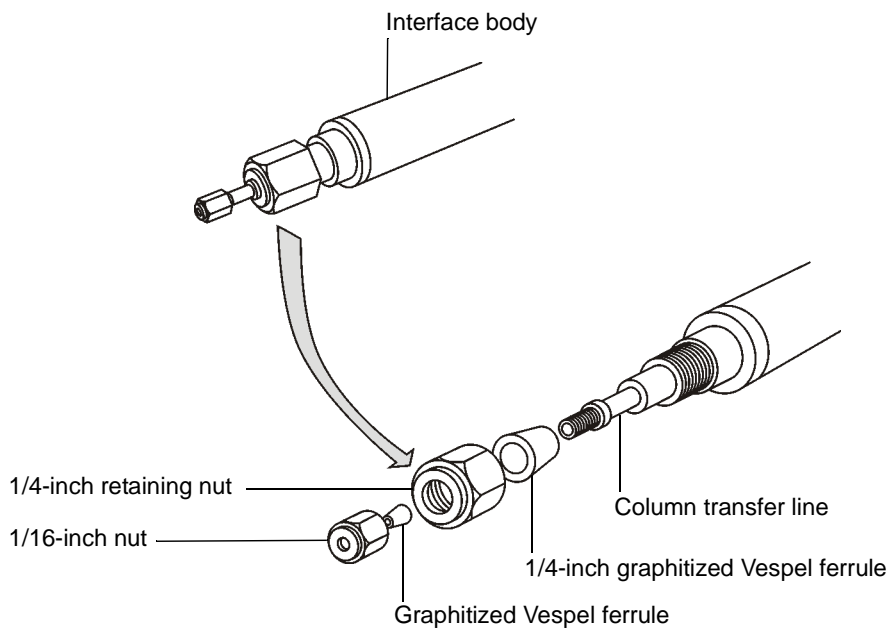
The turbomolecular pump is turned off. When the turbomolecular pump runs down to half its normal operating speed, the vent valve is opens and the instrument is automatically vents.

4. Wait for the Vacuum LED on the instrument's front panel to display yellow or red.

5. Remove the 1/16-inch nut and graphitized Vespel[®] ferrule from the column transfer line.

Note: The outside diameter of the column, and therefore the ferrule required, may vary depending on the column type used.

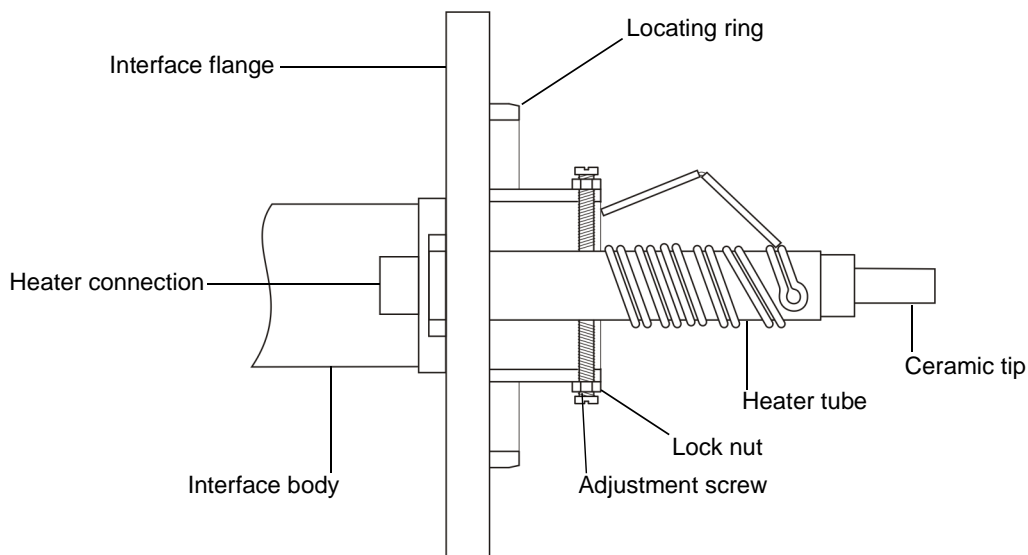
GC interface:



6. Ensure the ceramic tip is screwed securely in place, is clean and not cracked.

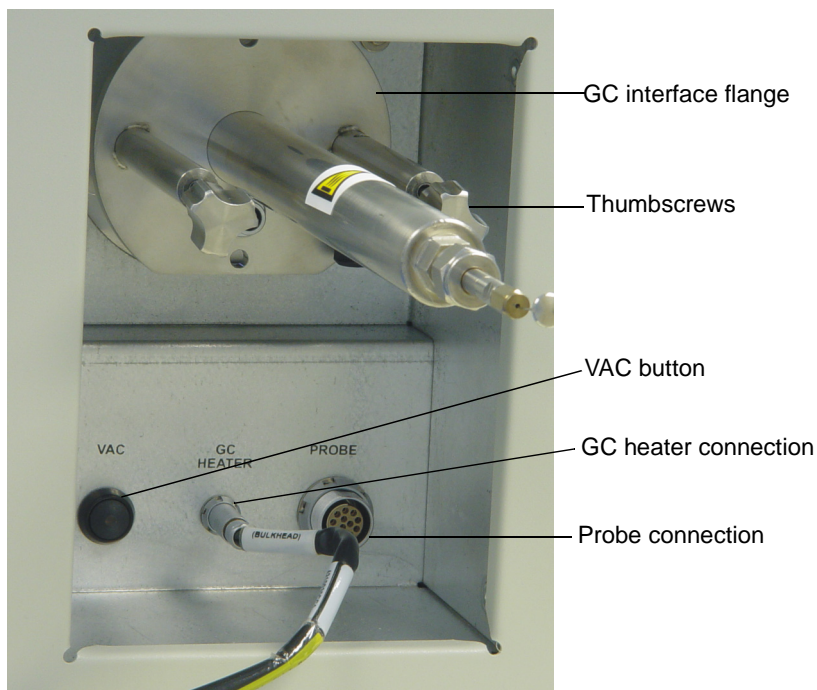
Note: The ceramic tip helps to minimize the disturbance of the electrical field close to the emitter.

GC interface tip:




7. Adjust the position of the tip to approximately 5 mm from the rod.
8. See [Figure titled "GC-FI column position:"](#) on page 9-20.
9. Ensure that the two Viton O-rings:
 - Sit correctly in the groove in the GC interface flange.
 - Are in good condition.
 - Are clean and free from dust.
10. Slide the GC interface onto the two guide rods.
11. Pull out the stop and slide the GC interface towards the instrument.
12. Ensuring that the heater connection on the interface flange is at the bottom of the assembly, fit the GC interface to the instrument and secure using the two thumbscrews.
13. The GC column can now be installed, or the transfer line blanked, to allow the source housing pumping.

GC interface fitted:



Removing the FI/FD GC interface

To remove the FI/FD GC interface

1. In the Tune window, click  to switch the instrument into Standby.
2. Click Vacuum > Vent Source.
3. Wait for the Vacuum Status LED to display steady yellow.



Warning: To avoid burn injuries, do not touch the GC interface. Allow to cool before handling.

4. Wait for the GC interface to cool down.
5. Loosen the 1/16-inch column retaining nut on the GC interface inside the GC oven.
6. Withdraw the GC column from the GC interface.

7. Turn the black knobs on the side of the GC plinth.
Result: The two GC plinth stops are released.
8. Push the GC away from the GC interface.
9. Disconnect the GC interface heater connection.
10. Remove the two GC interface retaining thumbscrews.
11. Slide the interface back on the guide rods, until it clicks into the stop.
12. Pull the retaining knob out and slide the GC interface off.
Note: Use the GC interface stop to allow you to withdraw the interface to the stop, so that the GC column is out of the source but is still secure and cannot fall off.

Positioning the GC column for FI operation

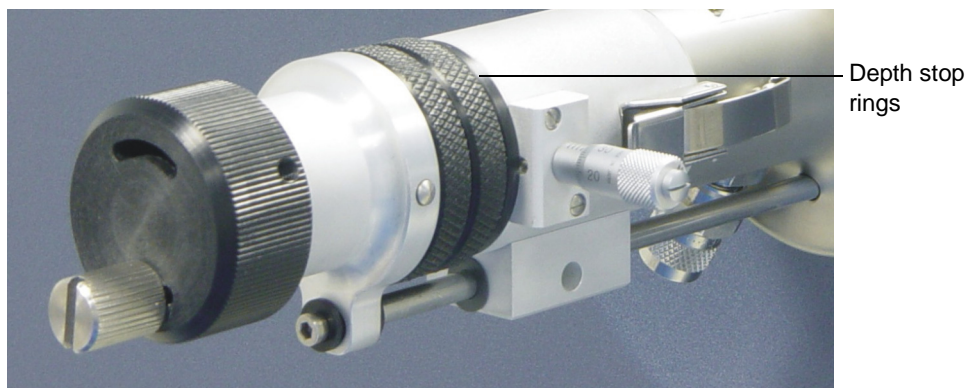
The position of the FI GC column in relation to the emitter is critical for optimum sensitivity. The end of the GC column should be as close as possible to the emitter wire. The optimum distance is 2 mm. If the end of the column is too close to the emitter wire, discharge or mechanical failure of the emitter may occur. The end of the column is too far away from the emitter, the sensitivity for analytes introduced via the GC may be reduced

Note: The extraction rods should be left cold for GC-FI operation.

To position the GC column for FI operation:

1. Insert the GC-FI transfer line and position it so that the ceramic tip is approximately 5 mm away from the extraction rods.
Tip: Use the clear viewing port on the top of the source lid.
2. Insert the GC column through the transfer line and position between 2 to 4 mm away from the center of the two extraction rods.
3. Tighten the 1/16-inch retaining nut.
4. Use the depth stop to adjust and set the position of the probe.
The depth stop rings can be locked together to keep them in place.

Probe depth stop:

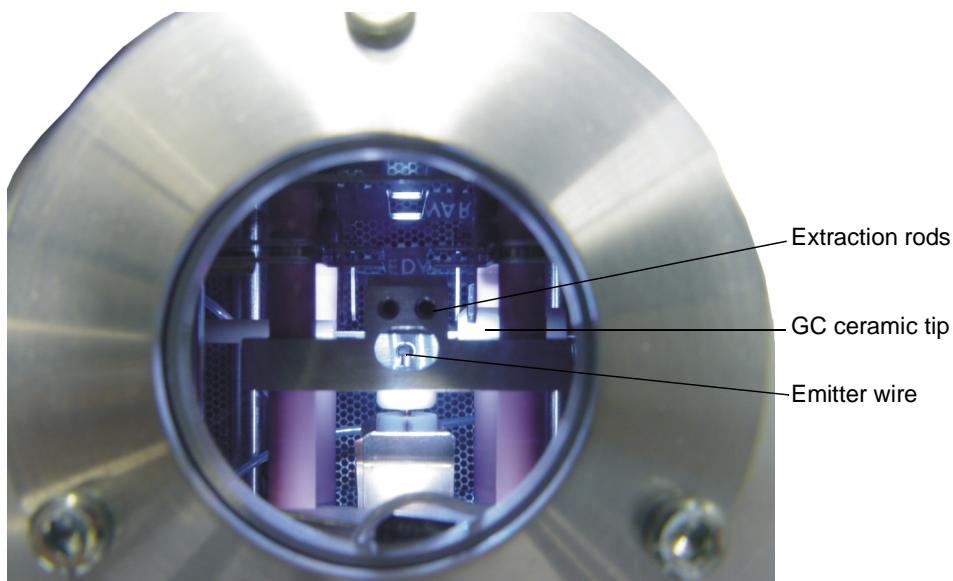


5. Position the emitter so that it is in line with or fractionally in front of the end of the GC column.

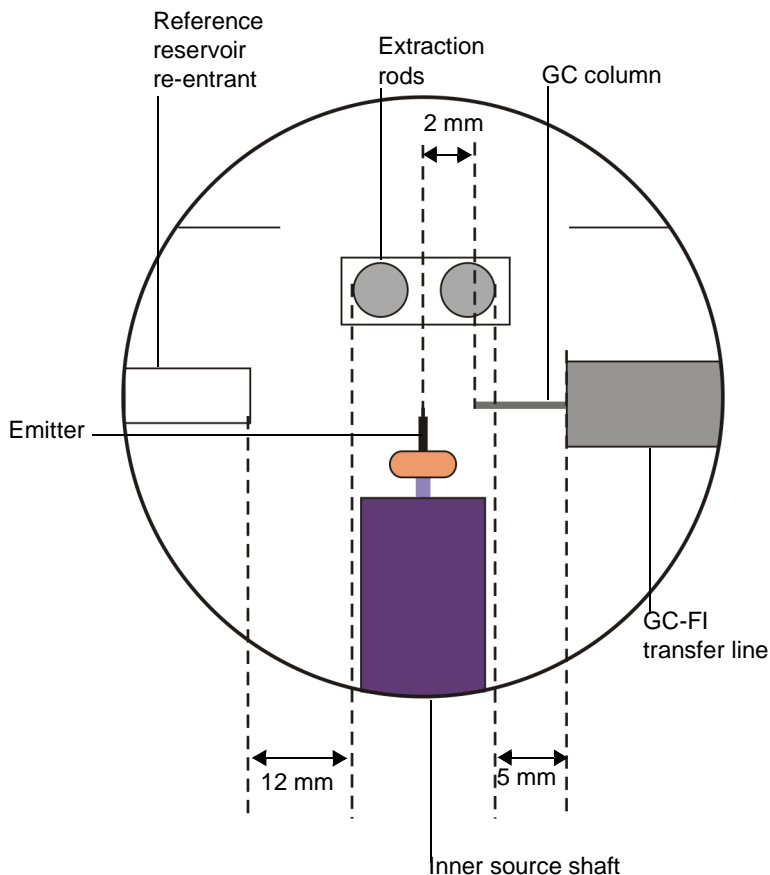
Note: Use the clear viewing port on the top of the source lid to view position of the emitter and ensure that the emitter wire does not touch the extraction rods.

6. Use the micrometer to adjust the side to side position of the inner source probe.

FI/FD outer source clear view port:



GC-FI column position:



Tuning in FI mode

When using the instrument in FI mode, sample is ionized by passing close to the very high field generated around the emitter by the voltage on the extraction rods. Sample can be introduced into the source by either the reference reservoir, the GC column or direct insertion probe (solids probe).

If you introduce the sample using the direct insertion probe, then you must use the FI/FD inner source direct insertion probe only flange.

See also: [Figure titled "FI/FD inner source direct insertion probe flange:" on page 9-10.](#)



Caution: Do not close the Tune window in FI mode with the instrument in operate. If you do, the emitter is damaged the next time you open the Tune window. To avoid damaging the emitter if you do close the Tune window while the instrument is in operate, pull the emitter back into the lock, to protect it when you open the Tune window.

Introducing the reference sample

The FI/FD outer source reference reservoir is essentially the same as the EI/CI outer source except that the reservoir has a slightly larger internal volume, and it is not heated.


The fused silica leak within the reference inlet re-entrant is 100 μm inner diameter \times 320 μm outer diameter. This allows the reference sample to bleed into the source.

To introduce the reference sample:

1. Ensure that the reference reservoir valve is closed.
2. Use a septum-piercing syringe to inject approximately 3 μL of chloropentafluorobenzene reference sample into the reference reservoir septum.

Obtaining a beam and tuning in FI operation

To obtain a beam in FI operation:

1. In the MassLynx Tune window, click Ion Mode > FI.
Note: Use the same parameters initially in the TDC dialog box and the Engineers tab as are used for the EI and CI operation.
2. In the FI Source tab set the following to 0: Flash Off Current, Emitter Current, Extraction Voltage, and Heater Current.
See also: Table titled “FI Tune tab parameters:” on page B-29.
3. Clear Enable Flash Off.
4. Click  to switch the instrument into Operate.

5. In the Tuning Setup dialog box, Time pane set the Scan Time(s) to 1.2 and the Inter Scan Delay (s) to 0.2
6. In Data pane, click Continuum from the Data Format list.
7. Increase the Extraction Voltage in increments of 2000 V to a maximum of 12,000 V.



Caution:

- Monitor the readback of the Extraction Control pane Current Leakage. If the Extraction Voltage rises above 10 μA constant readback, there is excessive current leakage to ground and indicates the source is dirty.
- Excessive leakage current can cause poor mass measurement, unstable signals, and the risk of high voltage flash-overs in the outer source, which could blow the emitter.

8. In the FI tab, specify an emitter current of 8 μA .

Ensure the readback indicates that the current is passing through the emitter. If no current or less than 8 μA passes through the emitter, the emitter could be damaged and need replacing.

See also: [Changing an emitter on page 10-33.](#)

9. Set the emitter current to the maximum value in mA for 2 to 4 seconds.

Rational: This desorbs any condensed material on the emitter and ensures maximum sensitivity.

10. Set the emitter current to zero.

11. Click Flash Off Emitter.

12. Set the Flash Off Current to 40 to 80% of the maximum emitter current.

Note: Ensure that a steady signal is maintained when the flash-off value is used. If there is a drop in sensitivity, increase the flash-off current.

13. Set the Emitter Current to 0 mA.

Tip: The emitter heats up during the interscan delay and cools down during the scan. This ensures that the sample does not condense on the emitter suppressing the sensitivity.

14. Use a septum-piercing syringe, to inject approximately 5 μL chloropentafluorobenzene into the reference reservoir.
See also: [Introducing the reference sample on page 9-21.](#)
15. Adjust the peak display to mass range 198 to 204 amu.
16. Right-click the peak display and click Intensity > Normalize Data.
17. Carefully adjust the micrometer until the peak at 201.9609 m/z is seen. If the emitter is moved to far it may hit the GC column and become damaged.
18. Use the beam steering and Focus 1, 2, and 3 values to optimize for maximum intensity.
19. Carefully adjust the rotational adjustment knob on the emitter probe to get the emitter wire as vertical as possible.
The rotational adjustment knob is very sensitive and must be adjusted carefully.
20. Tune for best resolution by adjusting the Pusher Bias and the Grid 2 values.

General considerations for tuning in FI mode

Lens tuning

The focus lenses are interactive with the beam steering and combinations of focus and steering settings can be used. The focus 1 lens often optimizes at maximum negative voltages.

The tuning of the ion source is strongly dependent on the position of the emitter with respect to the extraction rods the higher the field strength at the emitter and the higher the sensitivity.

The position of the emitter with respect to the center line of the extraction rods is also very critical. Small changes in this position can result in large changes in beam steering voltage being required. In general, the beam steering should be set close to zero and the emitter probe micrometer used to optimize the signal. Small adjustments to the beam steering can then be made using the beam steering lens.

Changes in the Focus Voltage can also result in changes in beam steering voltage. The focus lenses should be optimized in conjunction with this steering lens. If the beam steering lens optimizes at more or less than ± 4 V, the lens should be returned to zero and the micrometer used to re-optimize the beam.

See also:

- [General considerations for tuning on page G-6](#)
- [Dead time correction on page B-49](#)
- [Effects of saturation on peak shape on page B-51](#)

Setting up and calibrating the instrument

Instrument setup in FI mode

You must setup the detector voltage, veff, trigger and signal threshold in the same way as required for EI mode. These values can be set up using the Instrument Setup wizard.

See also: [Obtaining an ion beam on page 3-3](#).

Calibrating and accurate mass in FI mode

Usually the spectra produced in FI mode is simple, often exhibiting only molecular ion information, a mixture of volatile compounds must be used to initially characterize the time to square root of mass relationship. [Figure titled "Compounds and relative amount by volume:" on page 9-24](#) shows the compounds and their relative amount by volume to produce a suitable calibration mixture.

Compounds and relative amount by volume:

Compound name	% by volume	Expected masses
Heptacosane (PFTBA)	20	501.9711, 68.9952
Perfluorotrimethylcyclohexane	70	380.9760, 68.9952
Hexafluorobenzene	2	185.9998
Pentafluorobenzene	2	167.9998
Acetone	1	58.0419

Compounds and relative amount by volume: (Continued)

Compound name	% by volume	Expected masses
Chloropentafluorobenzene	2	201.9609
Xylene	2	106.0783s

Tip: These proportions can be adjusted to produce a more balanced spectra if required. The sample should be kept in a sealed vial, out of direct light and in a cool place. The volatile components (acetone) can decrease in proportion to the less volatile components over time.

Calibration in FI

Use approximately 10 to 15 μL of FI calibration mixture. Calibrate the instrument using the Calibration wizard.

See also: [Using the Calibration wizard on page 5-4.](#)

Optimization of FI calibration

The spectra produced from the FI calibration mixture contains fragment ions from some of the compounds in the mixture as well as molecular ions. The fragment ions form at a slightly different time and hence position with respect to the emitter than molecular ions.

Due to the high potential gradient between the emitter and the extraction rods the fragment ions have slightly different resultant axial energy entering the pushout region than the molecular ions. This results in some ions within calibration residuals appearing below and above the line of best fit. The differences can be minimized by adjusting the Pusher Bias voltage on the Engineer tab.

Running samples

Running MS samples in GC FI mode

You must set a use solvent delay at the beginning of an acquisition when running in GC FI mode.

See also: [Setting a solvent delay on page 4-9.](#)

The solvent delay is set so that the solvent front has completely eluted before the end of the solvent delay time. During the solvent delay period, the extraction voltage is turned off. This ensures that the extraction voltage is not at a high pressure in the ion source.



Caution: Failure to protect the system from the solvent front can damage the emitter.

Using direct insertion probe in FI mode



Caution:

- The inner source lock is incompatible with the direct insertion lock. When using the direct insertion probe in FI mode, you must fit the FI/FD inner source direct insertion probe flange.

See also: [Installing the FI/FD inner source direct insertion probe only flange on page 9-9.](#)

- You cannot use the DCI probe in FI mode.

Samples which are involatile can be run using the direct insertion probe. The rotational adjustment knob on the DI probe insertion lock allows the end of the DI probe to be set at a fixed distance away from the emitter.

The distance should be set to approximately 10 mm away from the emitter. The closer the DI probe is to the emitter, the more the field around the extraction rods will be distorted, resulting in a loss of sensitivity.

Note: Tune the instrument with the DI probe inserted into the ion source in the correct position.

See also: [Controlling the DI probe temperature on page 7-10.](#)

Tuning in FD mode

When working in FD mode, you must first obtain a beam using the reference sample, and tune the ion source. Once the instrument is tuned, you can introduce the sample onto the emitter. Using the probe ramp, the emitter current is ramped and you can run an experiment.


In FD ionization the sample is loaded directly onto the emitter, typically 10 μm tungsten wire covered with carbon microneedles. An electric current is passed through the emitter wire to raise the temperature. The sample is ionized on the surface of the emitter wire or, the sample can volatilize before ionization occurs.

Obtaining a beam in FD operation

Using this procedure, obtain a beam using a reference sample, before you inject sample onto the FD emitter. You need to use a 10 mm FD emitter for this procedure.

See also: [Changing an emitter on page 10-33](#).

To obtain a beam in FD operation:

1. In the MassLynx Tune window, click Ion Mode > FD.
2. Use a septum-piercing syringe to introduce approximately 3 μL of chloropentafluorobenzene or acetone into the reference reservoir.
3. In the FD Source tab Emitter Current, Extraction Voltage, and Heater Current, type zero.
4. Click .
5. In the Tuning Setup dialog box, set the Scan Time(s) to 1.2 and the Inter Scan Delay (s) to 0.2
6. In Data pane, click Continuum from the Data Format list.



Caution: Monitor the readback of the Current Leakage. If the current leakage rises above 10 μA constant readback, there is excessive current leakage to ground and indicates the source is dirty.

7. Increase the Extraction Voltage in increments of 2000 V to a maximum of 12,000 V, while monitoring the current leakage.



Caution: Using the system with an excessive leakage current can result in poor mass measurement, unstable signals and the risk of high voltage flash overs in the outer source, which could blow the emitter.

8. In the FD tab, click Set Maximum Emitter Current.
9. In the Emitter Current Control dialog box, click 100 mA.
10. In the FD tab, Emitter Control pane enter an Emitter Current of 20 μ A. Ensure the readback indicates that the current is passing through the emitter. If there is no current passing through the emitter, the emitter may need to be replaced.
See also: [Changing an emitter on page 10-33.](#)
11. Set the Emitter Current to the maximum value in mA for 2 to 4 seconds. This desorbs any condensed material on the emitter and ensures maximum sensitivity.
12. Carefully adjust the micrometer until the reference sample is seen, for example, chloropentafluorobenzene peak at 201.9609 m/z.
13. Use the Beam Steering and Focus 1, 2, and 3 to optimize for maximum intensity.

Introducing the sample

To introduce the sample:

1. Reduce the Emitter Current to 0 mA.
2. Ensure the Extraction Voltage is 0 V.
3. Remove the probe and load the emitter with sample.

See also: [Loading the FD inner source probe with sample on page 9-32.](#)

4. Re-insert the FI/FD inner source into the instrument.

See also: [Inserting the FI/FD inner source probe on page 9-11.](#)

5. Increase the Extraction Voltage to 12,000 V in increments of 2000 V, while monitoring the leakage current.



Caution: Do not enable the Flash Off or raise the Emitter Current above 0 at this stage.

6. Introduce acetone into the reference reservoir until the source pressure reaches 1e-5 mbar.

Note: This is approximately 20 μL .

7. In the Engineer tab Solvent Trip pane, adjust the Source Solvent Trip to allow the source pressure to rise.
8. Adjust the Beam Steering to maximize the acetone peak at 58 Da.

Rationale: The emitter position will have changed slightly when it is taken out. Adjust the beam steering to compensate for any movement. Any resulting voltage change should be less than 4 V.

Calibration and setup in FD

The calibration and set up for FD operation is the same as FI operation. If the signal is weak it is better to acquire in continuum and calibrate from the spectrum.

See also:


- [Calibrating and accurate mass in FI mode on page 9-24.](#)
- [Manual calibration on page 5-19.](#)

Alternatively, you can load a calibration solution such as polyethylene glycol (PEG) on the emitter together with your sample. Calibrate the data using the spectrum window.

Running an FD experiment manually

When the instrument has been tuned, using acetone, an acquisition can be started over the mass range of interest.

To run an FD experiment manually:

1. In the Tune window, click .

2. Set up the manual experiment as required, ensuring data is collected in continuum because signals are normally leak in FD..
3. In the MassLynx Tune window FD tab, increase the Emitter Current
This will gradually warm up the emitter and desorb the sample. As it is desorbed the sample will ionize on the emitter.
4. In the Tune window, monitor the spectrum as the acquisition progresses. It is recommended that the sample is desorbed slowly.
5. At the emitter current where ions are seen in the spectrum a large increase in signal for a small increase in emitter current may occur.
6. Increase the Emitter Current in small increments allowing the signal intensity to stabilize each time the current is increased.



Caution: Increasing the emitter current too quickly can result in thermal degrading of the analyte on the emitter surface before ionization, reduced overall ionization efficiency and in severe cases arcing between the emitter and the extraction rods due to localized increases in pressure.

7. When the signal drops to a low value you can then resume raising the Emitter Current.
Once all the sample has been desorbed from the emitter, flash off the emitter by raising the emitter current.
8. Increase the Emitter Current to 100 μA for five seconds then dropping it back to zero.

The acetone peak should be clearly visible again and the acquisition may be stopped.

Note: For some analyses it may be preferable to ramp the emitter without acetone in the source. With no reference material in the source there will be more sites available for ionization of the sample. In this case use acetone initially, to tune and then pump out the reference reservoir before commencing the ramp. However, acquiring without a lock mass increases the error of the accurate mass measurement.

Running an FD experiment using the probe ramp

If a controlled ramp is desired for the emitter current then this can be set in MassLynx Probe Control dialog box. The Probe Control is set up in the same

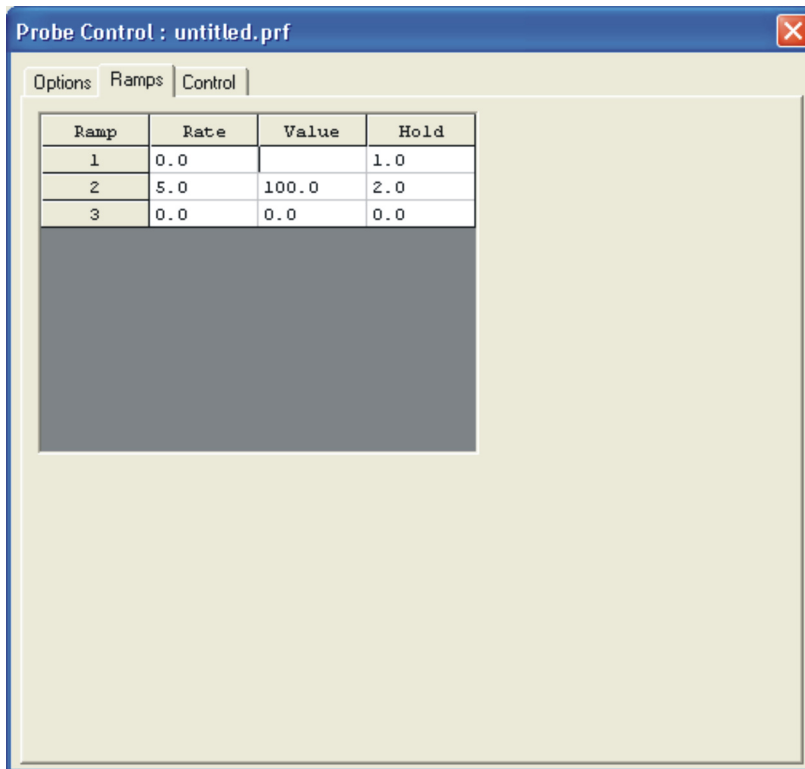
way whether you are using the DCI, DI, or FI/FD probe. The only difference is the units and values that are used in the Probe Control dialog box.

See also: [Ramping the probe temperature automatically on page 7-11.](#)

Probe Control Ramp tab

Table titled “Probe Control Ramp tab:” on page 9-31 shows the following probe ramp parameters. This ramp will hold the emitter at 0mA for the first minute. The current is then ramped at 5 mA/min in order to desorb the sample. When it reaches 100mA it will hold at that value for 2 minutes then drop to zero. This produces a total acquisition time of 23 minutes. Generally in FD it is desirable to ramp the emitter current slowly.

Probe Control Ramp tab:



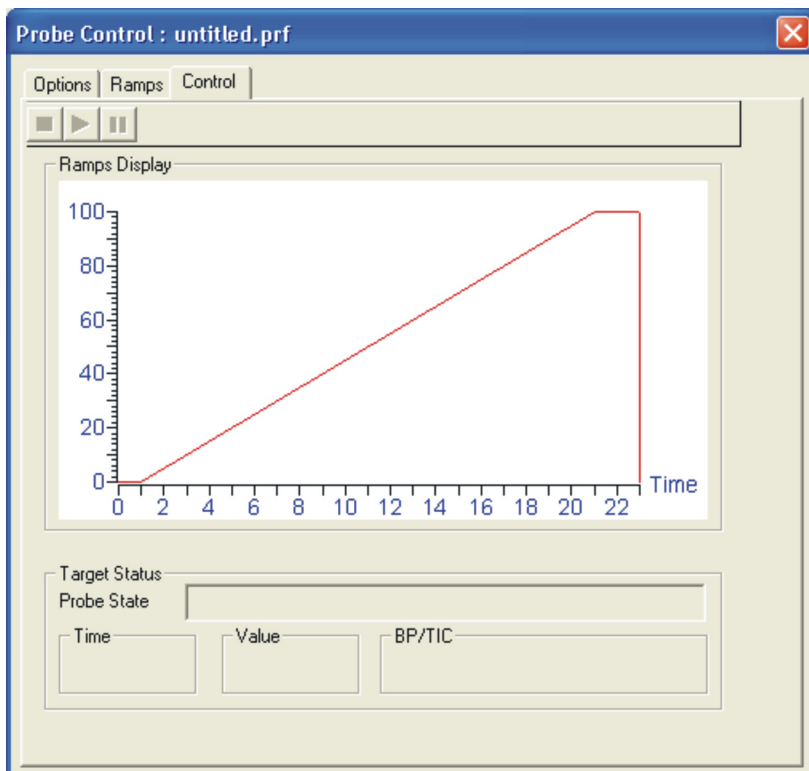
Ramp	Rate	Value	Hold
1	0.0		1.0
2	5.0	100.0	2.0
3	0.0	0.0	0.0

See also: [Probe Control dialog box Options tab on page 7-11.](#)

Probe Control Control tab

The Control tab shows a graphical representation of the probe current ramp.

Probe Control Control tab




See also: [Probe Control dialog box on page 7-16.](#)

Loading the FD inner source probe with sample


Sample concentration in the range of 500 ng/ μ L to 20 μ g/ μ L should be used. Higher concentrations of sample may sputter off the emitter, causing a discontinuous spectrum and, sometimes, arcing, which can damage the emitter. The sample should be dissolved in a volatile solvent to minimize evaporation time.

To load the FD probe with sample:

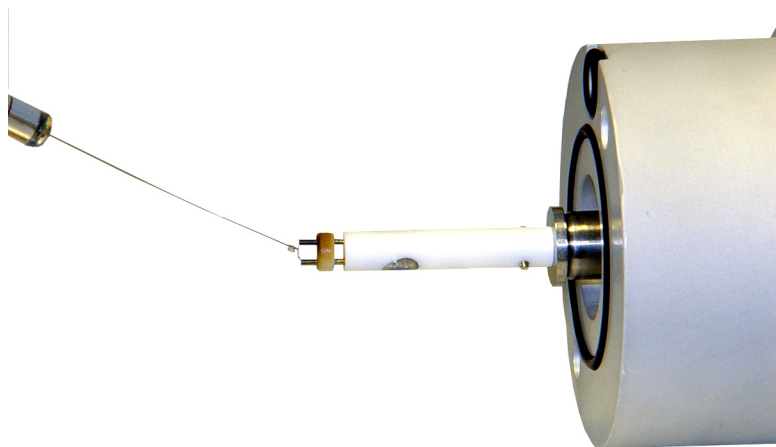
1. Remove the FI/FD inner source probe from the instrument.
See also: [Removing the FI/FD inner source on page 9-13.](#)
2. Place the FI/FD inner source probe on a flat work surface.
3. Rotate the FI/FD inner source probe shaft so that the emitter is horizontal.

 **Caution:** Do not touch the emitter wire directly with the syringe, as doing so breaks the wire.

4. Use a 10- μ L syringe, with a sharp end, to drip 1 μ L of sample directly onto the emitter wire.

 **Caution:** Do not allow the droplet to contact the legs of the emitter, or the sample may be drawn away from the emitter wire.

Loading the FD emitter with sample:



5. Ensure the sample is spread over the entire length of the emitter wire.
6. When the solvent from the sample has evaporated the FD inner source probe can be inserted into the outer source.

10 Routine Maintenance

This chapter provides maintenance guidelines and procedures required to maintain the instrument's performance.

Keep to the maintenance schedule and perform maintenance as required and described in this chapter.

Contents:

Topic	Page
Maintenance schedule	10-2
Safety and handling	10-3
Rotary pump oil	10-4
Rotary pump maintenance	10-5
Foreline trap maintenance	10-11
Reference reservoir maintenance	10-16
EI/CI inner source maintenance	10-18
FI/FD inner source maintenance	10-32
EI/CI outer source maintenance	10-35
FI/FD outer source maintenance	10-42
Checking for leaks after source maintenance	10-48
Detector maintenance	10-50

Maintenance schedule

The following table lists periodic maintenance schedules that ensure optimum instrument performance.

The maintenance frequencies shown apply to instruments that normally receive moderate use.

Maintenance schedule:

Procedure	Frequency
Gas-ballast the rotary pump.	Weekly.
Inspect and adjust the rotary pump oil level.	Weekly.
Change the rotary pump oil.	Every 3000 hours of pump operation.
Replace the rotary pump oil mist filter element.	Every six months. For applications that contaminate the rotary pump oil, this period is reduced and must be determined from experience.
Replace the rotary pump odor filter element.	Monthly, or whenever the rotary pump emits an oily odor.
Clean the foreline trap.	Every three months.
Replace scroll pump seals.	Annually. See also: Edwards document XDS 35i Instruction Manual A730-01-880.
Clean all source components.	When sensitivity decreases to unacceptable levels.
Condition the MCP detector	Each time the analyzer is vented.
Check the water hoses and connections for possible leaks and deterioration if the DI probe is fitted.	Annually, or before using the probe.

Safety and handling

Remember the following safety considerations when performing maintenance procedures.



Warning: To avoid electric shock, do not remove the instrument panels. There are no user-serviceable items inside.



Warning: To avoid electric shock, ensure that the instrument is in standby mode before commencing any maintenance.



Warning: To prevent injury, always observe good laboratory practices when handling solvents, changing tubing, or operating the instrument. Know the physical, chemical and biohazardous properties of the solvents used. See the Material Safety Data Sheets for the solvents in use.



Warning: To avoid burn injuries, take great care while working with the probe and source as they are liable to be hot.



Warning: The instrument components are liable to be contaminated with toxic and biohazardous materials. Wear chemical-resistant, powder-free gloves at all times while handling the components.

Cleanliness and care are very important whenever you remove internal assemblies from the instrument.

- Always prepare a clear, clean area in which to work.
- Make sure that any tools or spare parts that may be required are close at hand.
- Obtain some small containers in which screws, washers, spacers etc. can be stored.
- Use tweezers and pliers whenever possible.
- Use only powder-free chemical-resistant gloves.
- Avoid touching sensitive parts with fingers.
- Before reassembling and replacing dismantled components, inspect O-rings and other vacuum seals for damage. Replace with new ones if in doubt.

Should a fault occur soon after a particular part of the system has been repaired or otherwise disturbed, first ensure that the part is correctly refitted and /or adjusted. Then determine whether adjacent components have not been inadvertently disturbed.

Rotary pump oil

Oil quality

It is important that the recommended oil type is used for the associated rotary pump and vacuum conditions. For example, if the vapor pressure of the oil is higher than the ultimate vacuum of the pump, the instrument will not achieve its ultimate vacuum level. The condition of the oil is also critical to the rotary pump performance. If the oil is contaminated, the pump may operate at a reduced speed. With single phase pumps, if the pump continues to operate at reduced speed, the thermal overload opens and stops the pump. This spontaneous stopping of the pump must be avoided because it causes the instrument to perform an emergency vent.

See also: [Checking the rotary pump oil on page 10-6.](#)

Oil color

The color of the oil indicates its current quality. It can be checked through the oil level sight glass. The oil in the pump should be clear. If the oil is cloudy or discolored, it is contaminated with process vapors and must be replaced.

See also: [Changing the rotary pump oil on page 10-7.](#)

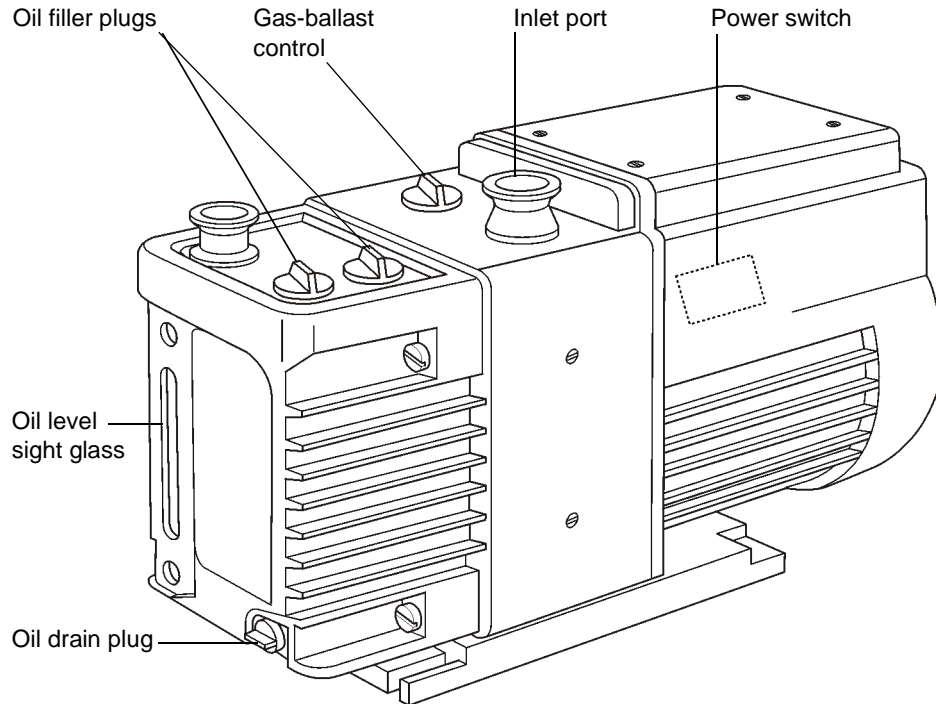
Gas ballast control

The rotary pump gas ballast control changes the flow of gas ballast into the low-vacuum stage of the pump, releasing the trapped process vapors to exhaust. The color of the oil should return to clear.

See also: [Gas-ballasting the rotary pump on page 10-5.](#)

Rotary pump maintenance

Rotary pump:



Gas-ballasting the rotary pump

When the rotary vacuum pump draws large quantities of solvent vapors, the vapors tend to condense in the pump oil and reduce pump efficiency. Gas-ballasting purges condensed contaminants from the oil.

Gas-ballast the rotary pump on a weekly basis, or when any of the following conditions apply:

- If the pump oil appears cloudy.
- If the vacuum pressure is higher than normal.
- If condensate forms in the rotary pump exhaust line.
- When changing the rotary pump oil.
- After CI or DCI operation.

To gas-ballast the rotary vacuum pump:



Warning: To avoid burn injuries, be careful while working with the rotary pump; it can be hot.



Caution:

- Failure to routinely gas-ballast a rotary pump shortens oil life and consequently shortens pump life.
 - Do not vent the instrument when a rotary pump is gas-ballasting.
 - Do not gas-ballast the rotary pumps while the instrument is in Operate.
 - Never gas-ballast a rotary pump for more than 30 minutes.
1. On the rotary pump, operate the gas-ballast control.
See also: [Figure titled “Rotary pump:” on page 10-5.](#)
 2. When the oil is clear return the gas-ballast control to its normal position.

Checking the rotary pump oil



Warning: To avoid burn injuries, be careful while working with the rotary pump; it can be hot.

The oil level can be checked while the pump is operating. However, the instrument must be vented and shut down before adding oil.

The rotary pump oil level appears in the pump's oil level sight glass. Check the oil level at weekly intervals. At all times, it should be at, or near, the MAX level indicated by the markings beside the sight glass. If oil must be added, vent and shut down the instrument before removing the oil filler plug.

See also: [Figure titled “Rotary pump:” on page 10-5.](#)

Examine the oil each time the oil level is checked. It should be colorless and free of visible contaminants. If the oil is discolored, change it.

See also: [Changing the rotary pump oil on page 10-7.](#)

Changing the rotary pump oil

Change the rotary pump oil every 3 to 4 months, or earlier, if it becomes noticeably discolored.

Required materials

- Chemical-resistant, powder-free gloves.
- Flat-blade screwdriver.
- Container to catch used oil.
- Funnel.
- Vacuum pump oil [use only Ultragrade19 or Inland Q45 (Edwards 45) vacuum pump oil].

To drain the rotary pump oil:



Warning: To avoid burn injuries, be careful while working with the rotary pump; it can be hot.



Warning: The rotary pump oil can to be contaminated with toxic and biohazardous materials. Wear chemical-resistant, powder-free gloves at all times while handling the oil, and ensure that the waste oil is disposed of according to local environmental regulations.

Tip: Operate the pump to warm the oil before draining it.

1. Gas-ballast the rotary pump.

See also: [Gas-ballasting the rotary pump on page 10-5.](#)



Caution: The ballast valve must be closed before venting the instrument.

2. Vent and shut down the instrument.

See also: [Starting-up and Shutting-down the Instrument on page A-1.](#)

3. Switch off the power supply to the pump.

4. Allow the pump to cool enough to handle it.



Warning: The pump is heavy for one person to lift. Follow the instructions in the Edwards Rotary Pump Instruction manual.

5. Raise the pump 6 to 8 inches (150 to 200 mm) above the floor, if necessary.
6. Place an object under the motor to tilt the pump toward the side on which the oil drain plug is located.
7. Remove the oil filler plug.
8. Use the flat-blade screwdriver to remove the oil drain plug.
9. Let the oil drain completely.

To fill the rotary pump with oil:



Warning: The rotary pump oil is toxic; wear chemical-resistant, powder-free gloves at all times while handling the oil.

See also: The Edwards Rotary Pump Instruction manual.

1. Fit the oil drain plug.
2. Remove the object used to tilt the pump.
3. Fill the pump until the oil in the sight glass reaches the MAX level.
4. Allow a few minutes for the oil to drain into the pump.
5. Recheck the oil level and, if necessary, add more oil.
6. Fit the oil filler plug and, if applicable, lower the pump to the floor.
7. Switch on the power supply to the pump.

Replacing the oil mist and odor filter elements

Required materials

- Chemical-resistant, powder-free gloves
- 4-mm hex (Allen) key
- Lint-free cloth

To replace the oil mist and odor filter elements:



Warning: To avoid burn injuries, be careful while working with the rotary pump; it can be hot.



Warning: The rotary pump oil can be contaminated with toxic and biohazardous materials. Wear chemical-resistant, powder-free gloves at all times while handling the oil, and ensure that the waste oil is disposed of according to local environmental regulations.

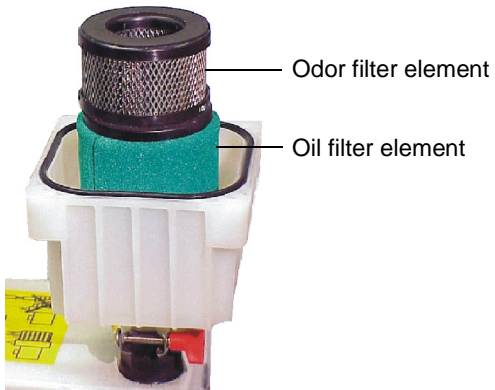
1. Turn off the power supply to the rotary pump.
2. Wipe clean the outside of the oil mist filter body.
3. Place a container underneath the drainage plug.
4. Remove the drainage plug from the filter.
5. Let the oil drain completely.



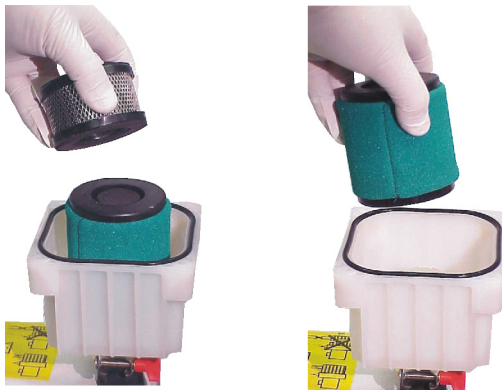
6. Remove the four screws which secure the upper body to the lower body.
7. Remove the upper body from the lower body.



This exposes the oil filter element and odor filter element.

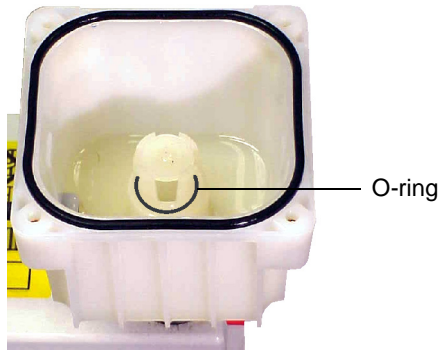


8. Lift out the element(s) you are replacing.



9. Wipe clean the insides of the upper and lower body assemblies.

10. Ensure that the oil filter element O-ring is in position on the lower body assembly.



11. Fit the new filter elements to the lower body assembly, ensuring that the foam sealing rings are correctly seated on the top and bottom of the elements.
12. Fit the upper body assembly to the lower body assembly.
13. Fit and tighten the four screws that secure the upper body to the lower body.

Foreline trap maintenance

Foreline trap:



The foreline trap prevents the back-migration of rotary pump oil vapor into the mass spectrometer. The trap consists of a metal basket located within a

cast aluminium body. The metal basket is held in place by a knurled nut, a lid, and a spring.

An absorbent material (activated alumina balls with a strong affinity towards oil vapor) is contained in the metal basket to absorb any oil that back migrates from the pump. The life of the alumina balls is typically six months. For most applications, the alumina should be replaced every three months. During normal use, the alumina nearest the port connected to the rotary pump become brown. When the discoloration reaches the alumina balls furthest from that port, the balls should be replaced.

Required materials

- Chemical-resistant, powder-free gloves
- Isopropyl or ethyl alcohol
- Lint-free cloth

To replace the alumina balls:



Warning: To avoid burn injuries, be careful while working with the rotary pump; it can be hot.



Warning: The rotary pump oil can be contaminated with toxic and biohazardous materials. Wear chemical-resistant, powder-free gloves at all times while handling the oil, and ensure that the waste oil is disposed in according to local environmental regulations.



Warning: To avoid inhaling dust from the activated alumina, wear a dust mask.

1. Vent and shut down the instrument.

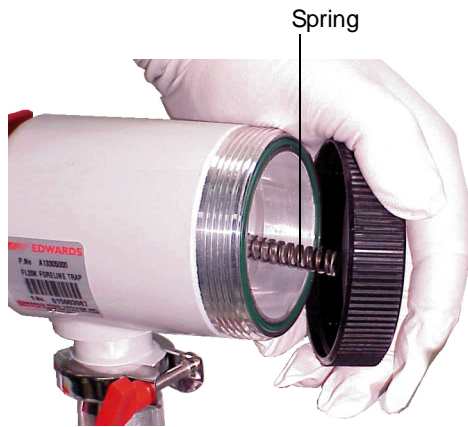
See also: [Shutting-down the instrument on page A-3.](#)



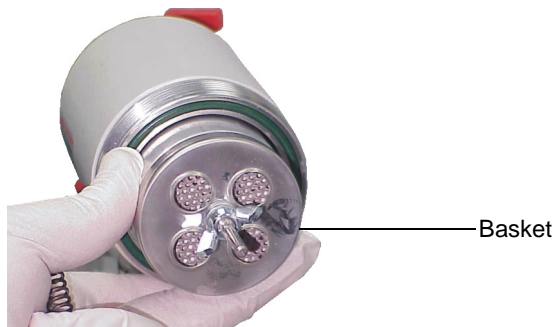
Warning: Take care when removing the foreline trap lid, It is spring-loaded.

2. Unscrew the foreline trap lid.

Tip: The lid comprises a flange and a disk, which has a strong spring pushing against it.



3. Remove the spring and the basket holding the alumina balls from the foreline trap casing.



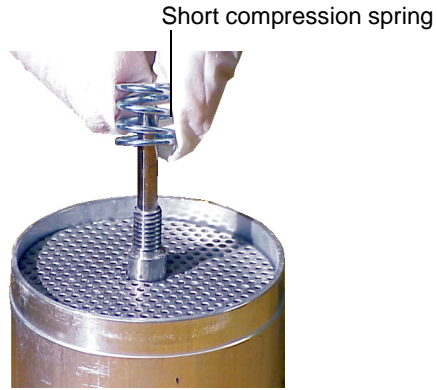
4. Clean the casing. If the inside of the foreline trap casing appears oily or dirty, wash it with isopropyl or ethyl alcohol, and then dry it thoroughly.



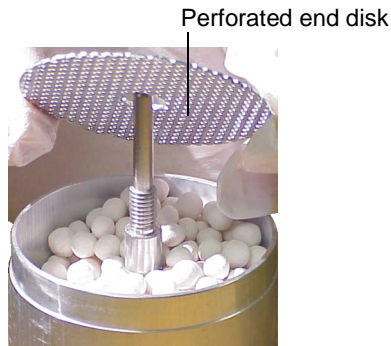
5. Remove the wing nut, washer and end casing from the basket.



6. Remove the washer and short compression spring from the inside of the basket.



7. Remove the perforated end disk from the inside of the basket.



Warning: Activated alumina, a desiccant, can absorb materials from the samples run through the mass spectrometer. Ensure that the alumina is disposed of according to local environmental regulations.

8. Remove the alumina balls from the basket, and dispose of them in accordance with local environmental regulations.
9. Clean the inside of the basket. If the inside of the basket appears oily or dirty, wash it out with isopropyl or ethyl alcohol and then dry it thoroughly.

10. Fill the basket with fresh alumina balls up to the maximum fill level marker, and refit the perforated disk, smooth side down, to avoid abrading the balls.

Tip: The perforated disk should align with the maximum fill level marker. Tap the basket gently to settle the alumina, and add or remove balls if necessary.

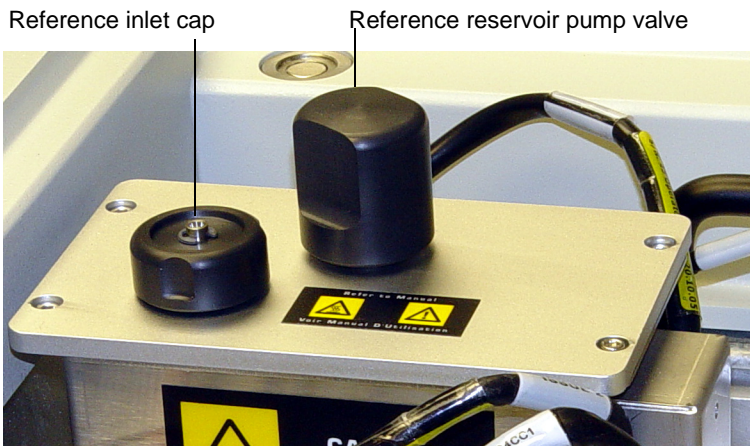


11. Slide the short compression spring over the spindle and on top of the perforated disk.
12. Refit the washer and end cap.
13. Refit the second washer and wing nut.
14. Push down the slack in the compression spring, and tighten the wing nut.
15. Fit the basket within the foreline trap casing, ensuring it is fully located on the bottom O-ring.
16. Fit and tighten the foreline trap spring lid.



Reference reservoir maintenance


Reference reservoir maintenance consists of changing the reference inlet cap septum that allows reference material to be injected into the reference reservoir. The septum is normally leak-tight. However, it can leak when repeat injections of reference material have been made.

Reference reservoir:




To renew the reference reservoir septum:

  **Warning:** The reference reservoir components can be contaminated with toxic and biohazardous materials. Wear lint-free cotton gloves at all times while handling the components.

 **Warning:** To avoid burn injuries, take care when working with the reference reservoir; it can be hot.

1. Open the reference reservoir pump valve.
2. Wait for 10 minutes.
Rationale: This removes any sample from the reference reservoir.
3. On the MassLynx Tune Window, click Vacuum > Vent Source.
4. Wait for the Vacuum Status LED to display steady yellow.
5. Unscrew and remove the reference inlet cap.
6. Remove the septum from the inlet cap.

 **Caution:** Note the color and orientation of the old septum, as an incorrectly-fitted new septum can cause air leakage.

7. Fit the new septum to the inlet cap with the grey Teflon side facing downwards, and the yellow rubber side facing upwards.
8. Refit and loosely tighten the inlet cap.
9. Push a septum-piercing syringe needle through the septum.
10. Finger tighten the inlet cap.
11. Remove the syringe needle from the septum.
12. Open the reference reservoir valve.
13. In the MassLynx Tune window, click Vacuum > Pump Source.

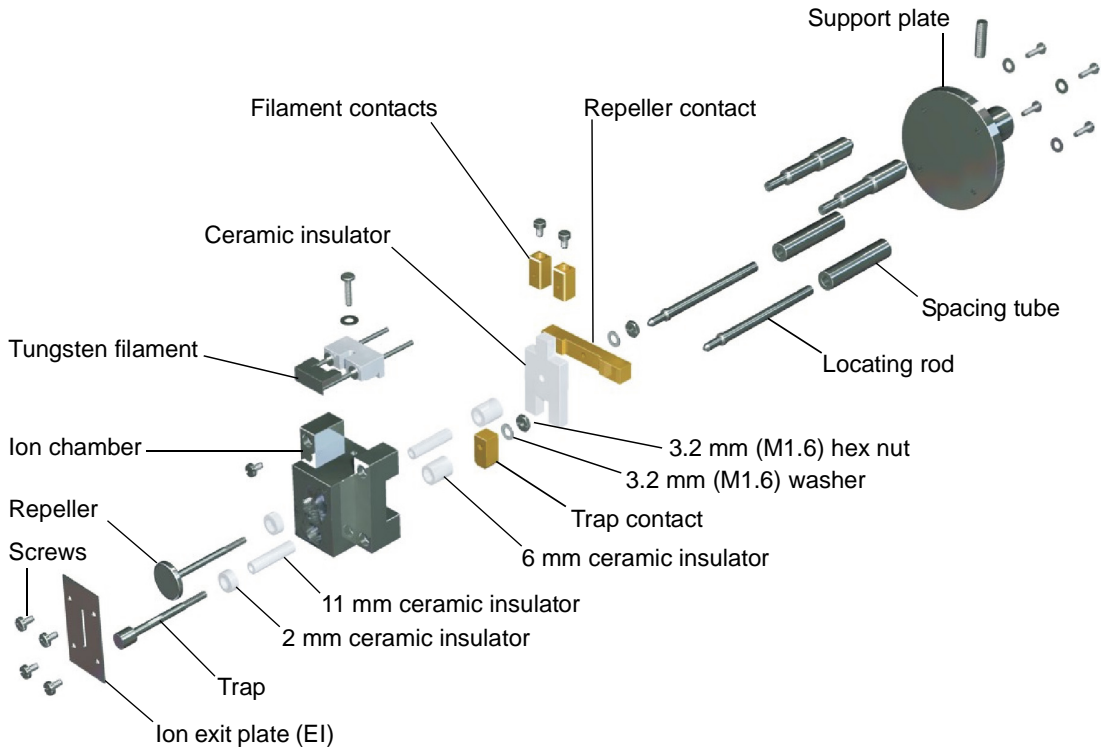
EI/CI inner source maintenance

Cleaning the EI and CI inner sources

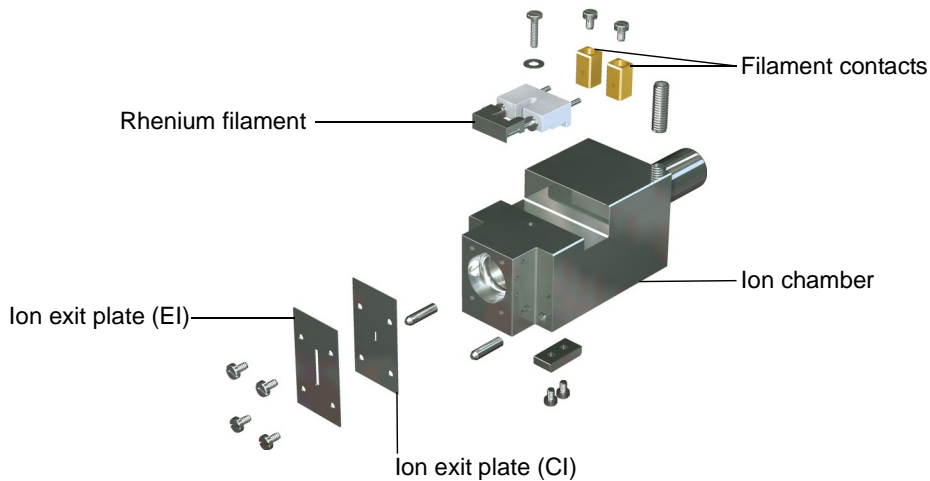
The EI and CI inner sources are easily removed for maintenance.

See also: [Inner source assembly on page 1-4](#) and [Changing the EI/CI inner source assembly on page 2-2](#).

EI inner source assembly:



CI inner source assembly (from ion chamber to exit plate only):



Cleaning the inner source components

Identify the following components for cleaning:

- Ion exit plate (see [Cleaning the ion exit plate on page 10-22](#)).
- Repeller (see [Cleaning the trap and repeller on page 10-23](#)).
- Trap (see [Cleaning the trap and repeller on page 10-23](#)).

Tip: The trap and repeller are present only on the EI source; the CI source is not fitted with a trap or a repeller.

- Filament (see [Renewing the filament on page 10-32](#)).



Warning: Personnel performing these operations should be aware of the inherent risks, and take the necessary precautions.



Warning: The source components are liable to be contaminated with toxic and biohazardous materials. Wear chemical-resistant, powder-free gloves at all times while handling the components.



Warning: Cleaning the various parts of the source requires the use of solvents and other chemicals that can be flammable and hazardous to health.



Warning: The probe and source can be hot. To avoid burn injuries take great care while working with these components.

Cleaning materials



Warning: Do not use water or solvents near the electronics units. Clean external panels with a damp cloth only.



Caution:

- Maintain the quality of the surface finish when cleaning internal components. Deep scratches or pits can cause loss of performance.
- Do not over-polish the lenses of the outer lens stack.

Where no specific cleaning procedure is given, fine abrasives should be used to remove dirt from metal components. Recommended abrasives are:

- 1200-grade wet/dry paper.
- Lapping paper (produced by 3M®).
- Polaris powder.

After cleaning with abrasives, wash all metal components in suitable solvents to remove all traces of grease and oil. Then ultra sonically clean the components in a clean beaker of solvent and subsequently to blot them dry with lint-free tissue:

- Propan-2-ol, [isopropyl alcohol (IPA)]
- Methanol
- Acetone
- Dichloromethane

Following reassembly, components should be blown with oil-free nitrogen or dried in an oven to remove dust particles.

See also: [Ultrasonically cleaning the inner source components on page 10-21.](#)

Ultrasonically cleaning the inner source components



Warning: Cleaning the inner source components requires solvents and other chemicals that can be flammable and hazardous to health.



Warning: The inner source components may be contaminated with toxic and biohazardous materials. Wear chemical-resistant, powder-free gloves at all times while handling the components.



Caution: To avoid damage, the filament and ceramic components must not be cleaned in the ultrasonic bath.

To ultrasonically clean the inner source components:

1. Immerse all the metallic inner source components (including the screws and washers, but not the filament) in a glass vessel containing a recommended solvent.

See also: [Cleaning materials on page 10-20.](#)

2. Place the vessel in the ultrasonic bath for 30 minutes.

Tip: Depending on the nature of the contamination, Waters recommends that [steps 1 and 2](#) be repeated with various solvents to thoroughly clean the components.



Caution: To avoid re-contaminating the ion source components following cleaning, use clean lint-free cotton gloves during assembly.

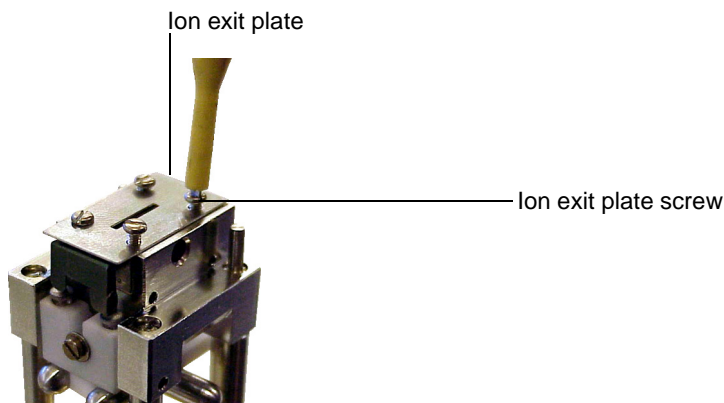
3. Carefully remove the inner source components from the vessel.
4. Blow-dry the components using inert, oil-free gas.

Alternative: Place the components on clean, lint-free towels, and allow them to air dry. Wipe off any water spots with a clean, lint-free cloth.

Cleaning the ion exit plate

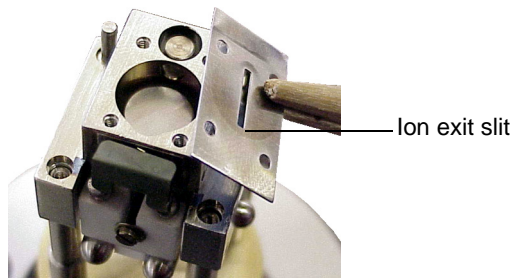
To remove the ion exit plate:

1. Use a jeweller's screwdriver to unscrew and remove the four screws on the ion exit plate.



Caution: When removing the ion exit plate, do not scratch it, especially around the slit.

2. Use tweezers to carefully remove the ion exit plate.



To clean the ion exit plate:

1. Using 1200-grade wet/dry paper, rub all the ion exit plate surfaces, including the ion exit slit edges, until they are free of visible ion burn.
2. Using 9-micron lapping paper, polish all the ion exit plate surfaces, including the ion exit slit edges.
3. Ultrasonically clean the plate in a suitable solvent to remove any remaining grease.

See also: [Ultrasonically cleaning the inner source components on page 10-21.](#)

To fit the ion exit plate:

Reassemble the ion exit plate in the reverse order to which it was disassembled.

Cleaning the trap and repeller

Tip: The trap and the repeller are present only on the EI source; the CI source is not fitted with a trap or repeller.

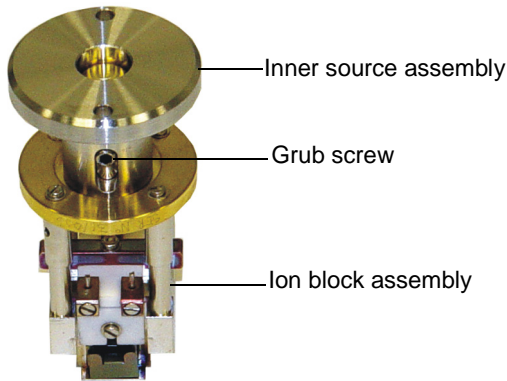
To remove the trap and repeller:

1. Remove the ion exit plate.
2. Use a hex (Allen) key to remove the grub screw at the base of the inner source assembly.

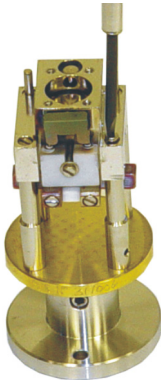


Caution: Take care when removing the inner source assembly from the source handle; it is spring-loaded.

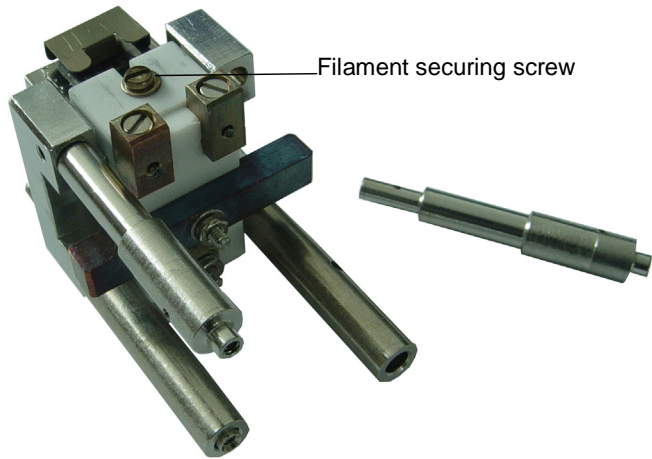
3. Remove the inner source assembly from the source handle.



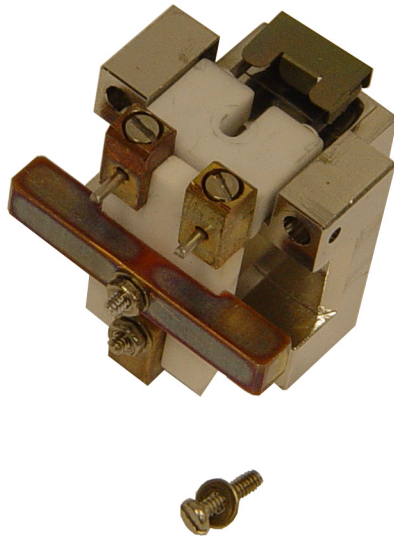
4. Remove two screws that secure the ion block assembly to the inner source assembly brass support.
5. Unscrew and remove the two screws that secure the two threaded support rods to the ion block.



6. Lift off the ion block assembly so that the two threaded support rods come out from the bottom of the ion block.
7. Remove the sleeves from the remaining two (unthreaded) support rods and remove the support rods from the top of the ion block.

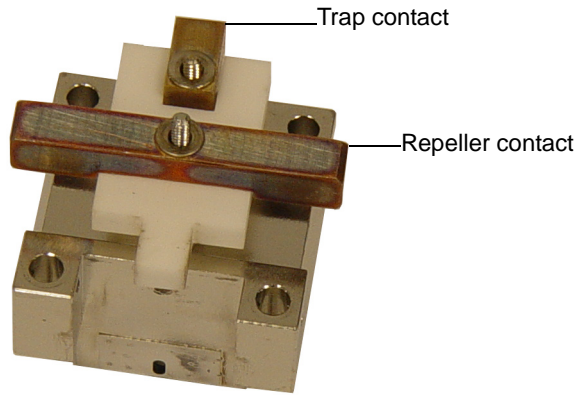
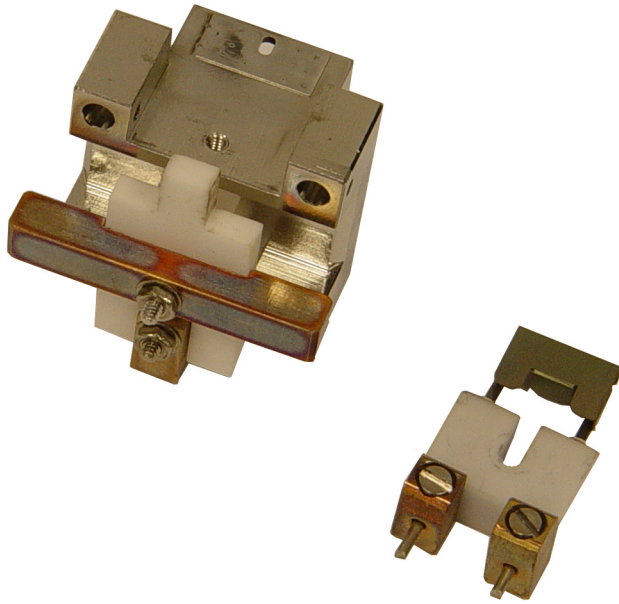


8. Unscrew and remove the filament securing screw.

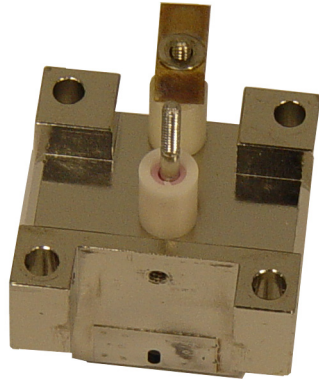



 **Caution:** The filament is fragile. To avoid damaging it, handle with care.

9. Remove the filament from the inner source.

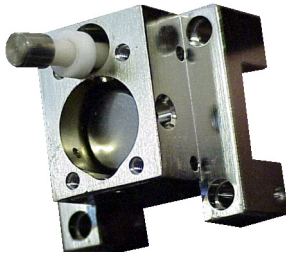


10. Remove the repeller and trap contacts, Trap/repeller ceramic isolator, and associated ceramic spacers.

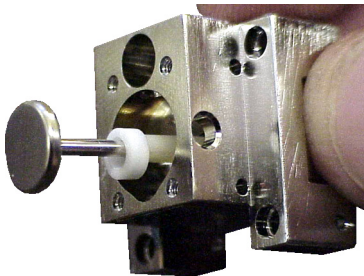


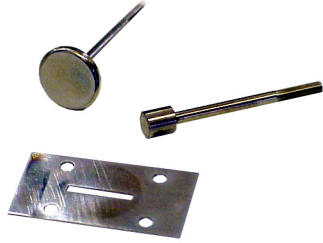
 **Caution:** When removing the trap and repeller, take care not to lose the ceramic spacers.

11. Push out the trap and ceramic spacers from underneath the ion block.



12. Push out the repeller and ceramic spacers from underneath the ion block.





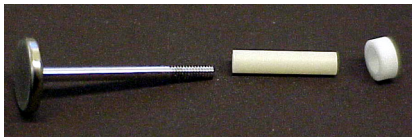
To clean the trap and repeller:

1. Using 1200-grade wet/dry paper, rub the trap edge and end surfaces until they are free of visible ion burn.
2. Using 9-micron lapping paper, polish the trap edges and end surfaces.
3. Using 1200-grade wet/dry paper laid on a flat surface, in a figure-of-eight motion, rub the repeller's flat surface until it is free of visible ion burn.
4. Using 9-micron lapping paper laid on a flat surface, in a figure-of-eight motion, polish the repeller's flat surface.
5. Ultrasonically clean the trap and repeller in a suitable solvent to remove any remaining grease.

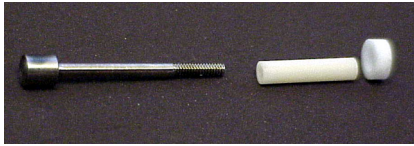
See also: [Ultrasonically cleaning the inner source components on page 10-21.](#)

To reassemble the inner source:

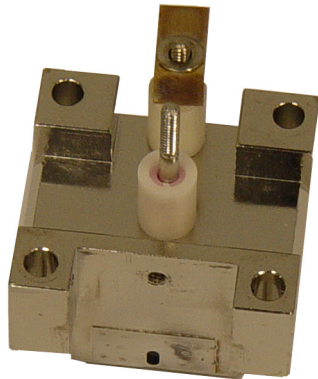
1. Fit the ceramic spacers to the repeller.



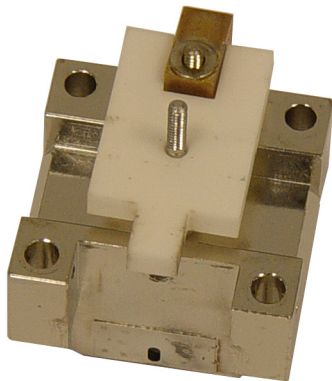
2. Fit the repeller and ceramic spacers in the ion block.
3. Fit the ceramic spacers to the trap.



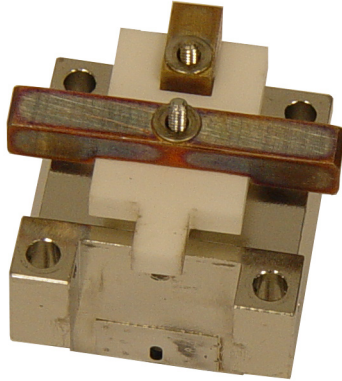
4. Fit the trap and ceramic spacers in the ion block.
5. Fit two ceramics on the rear of the ion block, one on the repeller and the other on the trap.
6. Fit the trap contact to the trap.



7. Fit the trap/repeller ceramic isolator to the repeller.



8. Fit the repeller contact to the repeller.



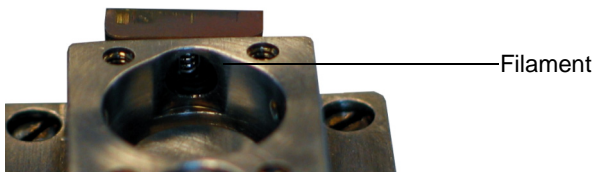
9. Fit and tighten the two contact securing nuts.



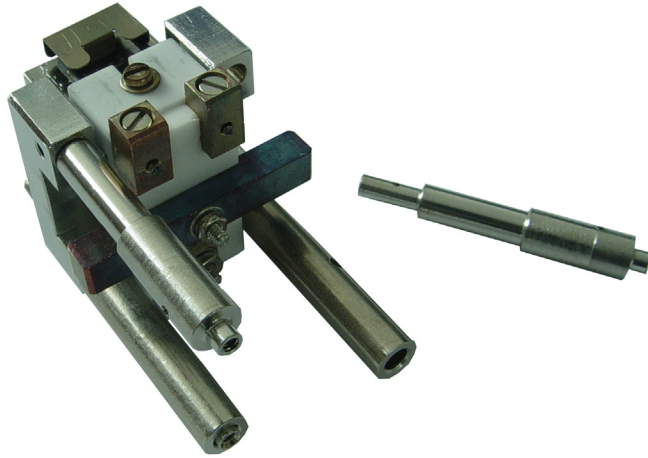
Caution: The filament is fragile. To avoid damaging it, handle with care.

10. Fit the filament to the ion block.
11. Align the center of the filament wire with the hole in the ion chamber.

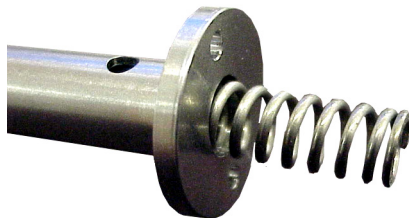
Filament wire alignment:



12. Fit and tighten the filament securing screw.
13. Fit the unthreaded support rods to the top of the ion block.
14. Fit the sleeves to the bottom of the unthreaded support rods.
15. Fit the two threaded support rods to the bottom of the ion block.



16. Fit and tighten the two screws that secure the two threaded support rods to the ion block.
17. Fit and tighten the four screws that secure the ion block assembly to the inner source assembly brass support.
18. Insert the spring into the ion block support tube.



19. Fit the inner source assembly to the source handle.
20. Use a hex (Allen) key to fit and tighten the two bolts at the base of the inner source assembly.
21. Fit the ion exit plate.

See also: [To remove the ion exit plate: on page 10-22.](#)

Renewing the filament



Caution: The filament is fragile. To avoid damaging it, handle with care.

To renew the filament:

1. Using a jeweller's screwdriver, unscrew the single screw securing the filament to the ion chamber assembly.
2. Remove the filament.
3. Use a jeweller's screwdriver to transfer the filament contact blocks to the new filament.
4. Using a pair of wire cutters, cut off the excess lengths of leg close to the contact blocks.
5. Fit the filament to the ion block.
6. Align the center of the filament wire with the hole in the ion chamber.
7. Fit and tighten the filament securing screw.
8. Reassemble the inner source.

See also: [Figure titled "Filament wire alignment:" on page 10-30.](#)

See also: [To reassemble the inner source: on page 10-28.](#)



Caution: After fitting a new filament, start applying a low emission/trap current to the filament and slowly ramp it until you reach operational values.

FI/FD inner source maintenance

Storing an emitter

Some types of emitters have a limited lifetime, between three and four months when stored at atmosphere. The sensitivity of the FI emitter decreases the longer the emitter is stored. This is normally due to oxidation of the tungsten wire used.

To extend the lifetime of the emitter:

Store the emitter under vacuum or in an atmosphere of dry nitrogen.

Changing an emitter

You must replace the emitter if it is damaged or broken. The sensitivity of the emitter is significantly compromised when a dirty, damaged or broken emitter is used. If you are swapping the emitter, remember to return the emitter that is no longer required to its transportation box.



Warning: Wear protective goggles when handling the emitter. The emitter bend is made of ceramic and can shatter if put under pressure.

To change an FI emitter:

1. Remove the FI/FD inner source probe from the instrument.

See also: [Removing the FI/FD inner source on page 9-13.](#)

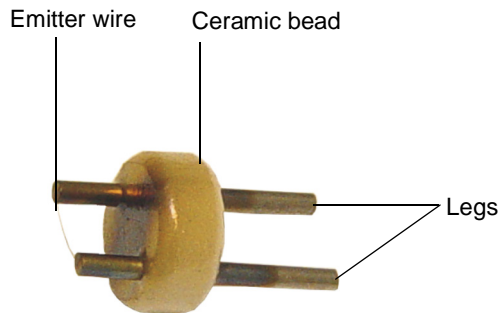
2. Use tweezers to carefully remove the emitter currently fitted onto the FI/FD inner source probe. If the emitter is not damaged, you can return it to its transportation box.



Caution: The emitter is very fragile and should not be directly touched.

3. Carefully remove the FI emitter from the transportation box using a pair of tweezers to grip one of the bead legs.

FI emitter:



4. Carefully hold the FI inner source on its side and carefully install the emitter bead into the end of the probe.
5. Use the tweezers to gently touch the ceramic bead pushing the emitter onto the probe, taking care not to touch the emitter wire.
6. Wind the threaded inner source probe stop counterclockwise until it reaches the end.

Note: You only need to set the depth stop rings the first time you insert the inner source probe. Once the depth stop rings have been set, you can lock the depth stop rings to retain the position for future use.

Rational: This prevents the inner source probe from being pushed too far into the source and hitting the extraction rods.

7. Carefully insert the FI inner source into the outer source.
See also: [Inserting the FI/FD inner source probe on page 9-11.](#)
8. Ensure that the emitter is more than 5 mm away from the extraction rods. Use the clear viewing port on the top of the source lid.
9. Adjust the depth stop rings to push the probe until the emitter is in the correct position. This is normally approximately 2 mm.
10. Lock the depth stop rings to retain the position for future use.

EI/CI outer source maintenance

A dirty outer source can cause loss of sensitivity, poor stability, or it can even affect the resolution of the GCT Premier. As the beam goes through the source it passes through collimating slits, on the EI/CI outer source, which are only 0.25 mm wide. These slits are the first to get dirty with ion burn.

This section explains the following procedures:

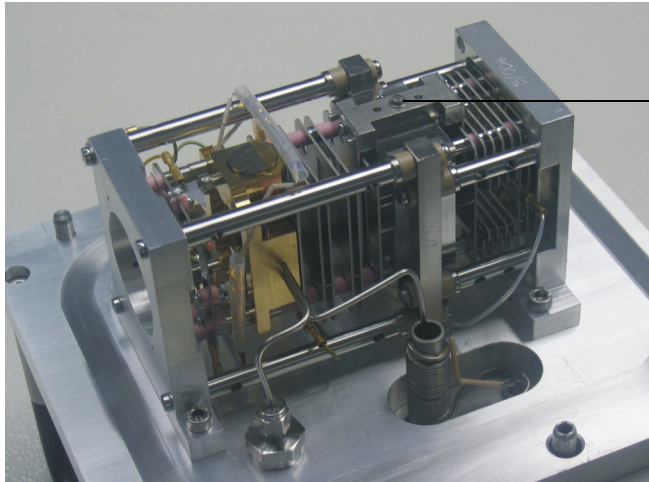
- Removing the outer source from the instrument. See [Removing the EI/CI inner source on page 2-3](#).
- Cleaning the collimating slits. See [Cleaning the collimating slits on page 10-35](#).
- Cleaning the complete outer source. See [Cleaning the complete outer source on page 10-37](#).
- Fitting the outer source to the instrument. See [Fitting the EI/CI outer source to the instrument on page 2-8](#).
- Checking for leaks after performing source maintenance. See [Checking for leaks after source maintenance on page 10-48](#).

Cleaning the collimating slits

Cleaning the collimating slits involves removing, cleaning, and refitting them.

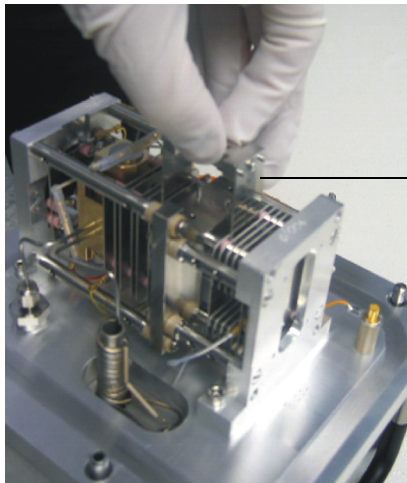
To remove the collimating slits:

1. Remove the outer source lid assembly from the instrument.
See also: [Removing the EI/CI outer source from the instrument on page 2-6](#).
2. Unscrew and remove the screw that secures the collimating slits to the outer source lid assembly.



Screw securing the collimating slits

3. Lift the collimating slits assembly out of the outer source lid assembly.



Collimating slits assembly

4. Disassemble the collimating slits assembly by unscrewing and removing the screw securing each slit to the alignment block and removing the slits.

To clean the collimating slits:

1. Rub each side of the slit with 1200-grade wet/dry paper.

2. Ultrasonically clean the slits in a suitable solvent to remove any remaining grease.

See also: [Ultrasonically cleaning the inner source components on page 10-21.](#)

To refit the collimating slits:

1. Assemble the collimating slits assembly in the reverse order to which you disassembled it.
2. Fit the collimating slits assembly into the outer source assembly.
3. Fit and tighten the screw that secures the collimating slits to the outer source lid assembly.
4. Fit the outer source lid assembly to the instrument.

See also: [Fitting the EI/CI outer source to the instrument on page 2-8.](#)

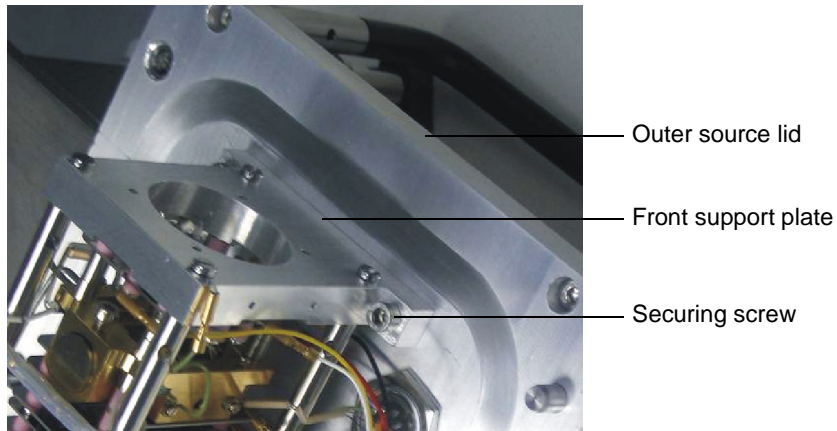
Cleaning the complete outer source

To disassemble the outer source:

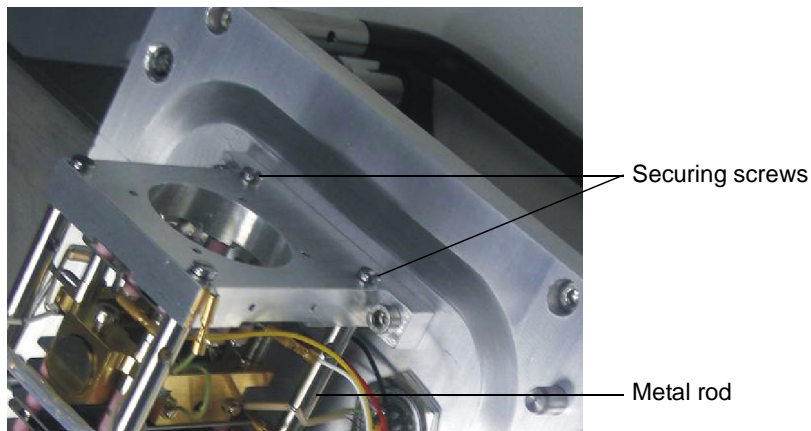
1. Remove the outer source lid assembly from the instrument.
See also: [Removing the EI/CI outer source from the instrument on page 2-6.](#)
2. Place the outer source lid assembly on a bench so that the front support plate is at the top.
3. Make a diagram of where the wires, thermocouple, and gas tubing are located.

Rationale: This diagram aids assembling the outer source.

4. Unscrew and remove the two hex-head screws that secure the front support plate to the outer source lid.



5. Unscrew and remove the four screws that secure the front support plate to the metal rods.



6. Remove the front support plate.
7. Remove each lens plate and its associated spacers from the outer source assembly in turn.

Tip: The outer source comprises various lens plates held in place by four ceramic rods, one at each corner. Between each lens plate there are metal or ceramic spacers of different sizes. To aid reassembly, as you disassemble the source, make a diagram of the order, position, and orientation of each lens and each spacer on its rod. Note that, on some layers, the spacers on the upper rods are different from those on the lower rods.

Alternative: You can to access the lenses from the opposite end of the outer source lid assembly, avoiding the need to remove the washers and the spacers around the inner source contacts.

To clean the lens plates:

1. Rub each side and the inner aperture of each lens plate with 1200-grade wet/dry paper to remove any visible ion burn.

Requirement: The inner aperture of each lens plate must be cleaned.

2. Ultrasonically clean the lens plates in a suitable solvent to remove any remaining grease.

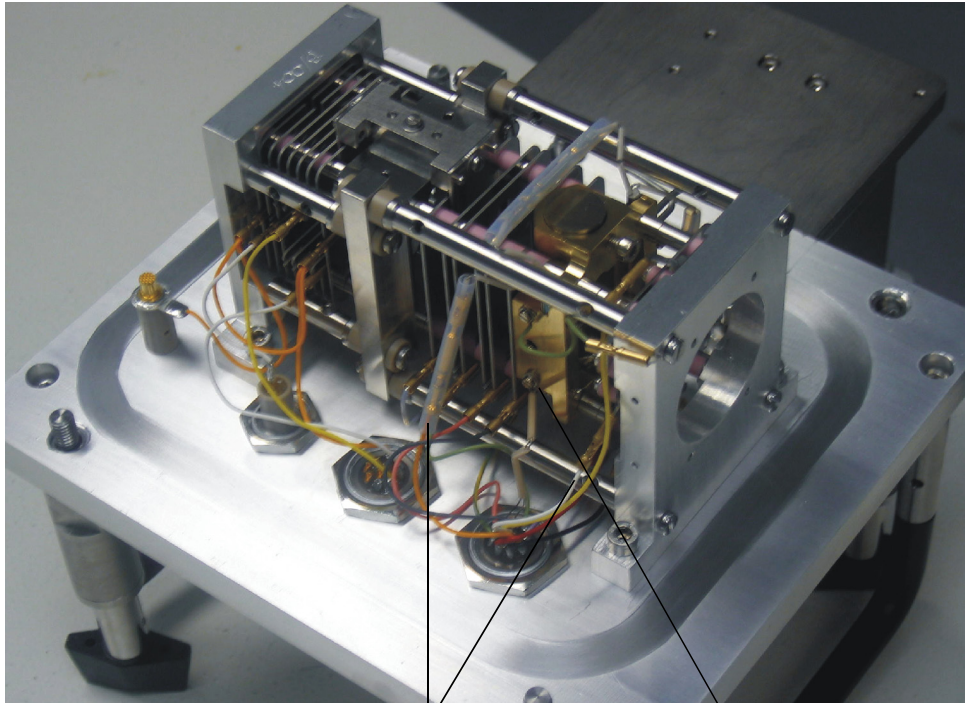
See also: [Ultrasonically cleaning the inner source components on page 10-21.](#)

To assemble the outer source:

Assemble the outer source in the reverse order to which you disassembled it, consulting the diagrams prepared during disassembly.

Note: When reassembling the outer source after completing any source maintenance, ensure that you fit the outer source magnets with the opposite poles facing each other.

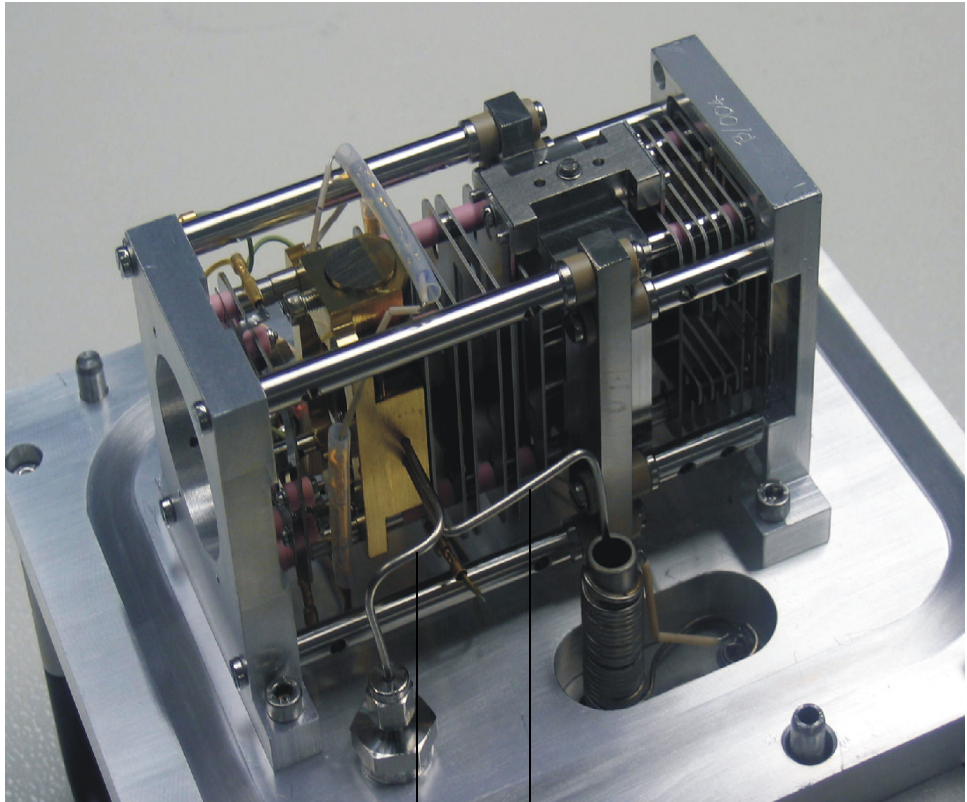
Assembled outer source (1):



Heater wires

Thermocouple

Assembled outer source (2):



Cl gas tube (top)

Reference gas tube (bottom)

Assembling the ion chamber

To fit the source magnets:

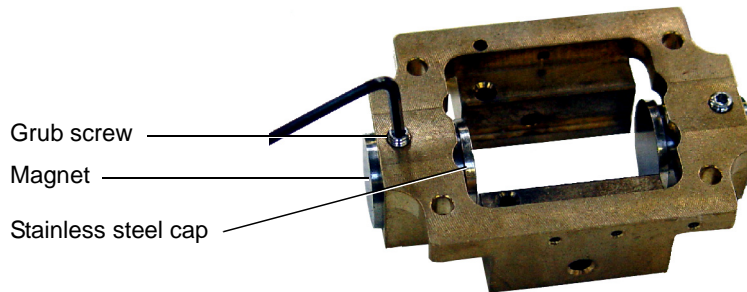
1. Fit the first magnet to the ion block.
2. Fit the stainless steel cap to the inner end of the magnet.
3. Use a 1.5-mm hex (Allen) key to fit the and tighten the grub screw securing the magnet to the ion block.

4. Test the polarity of the second magnet by ensuring it is attracted to the outer end of the first magnet in the ion block. Turn the magnet round (end-to end) if required.



Caution: When fitting the second magnet to the ion block, ensure that the polarity is the same as the first magnet. The overall orientation of the magnets in the ion block is not relevant.

5. Repeat [steps 1 through step 3](#) to fit the second magnet to the ion block, ensuring that the polarity is as determined in [step 4](#).



FI/FD outer source maintenance

A dirty outer source can cause loss of sensitivity, poor stability, or it can even affect the instrument resolution of the GCT Premier. As the beam goes through the source it passes through collimating slits, which are only 0.5 mm wide. These slits are the first to get dirty with ion burn.

If current leakage occurs, then it is recommended that you perform the following maintenance procedures:

- Change the emitter. See [Changing an emitter on page 10-33](#).
- Clean the extraction rods. See [Cleaning the extraction rods on page 10-42](#).
- Clean the focus plates in the outer source. See [Cleaning the source ceramic and focus plates on page 10-45](#).

Cleaning the extraction rods

Sometimes it is sufficient to clean the extraction rod in the ion source to improve sensitivity. The rod assembly has been designed to allow easy removal, without the need to strip away all the ion source parts.

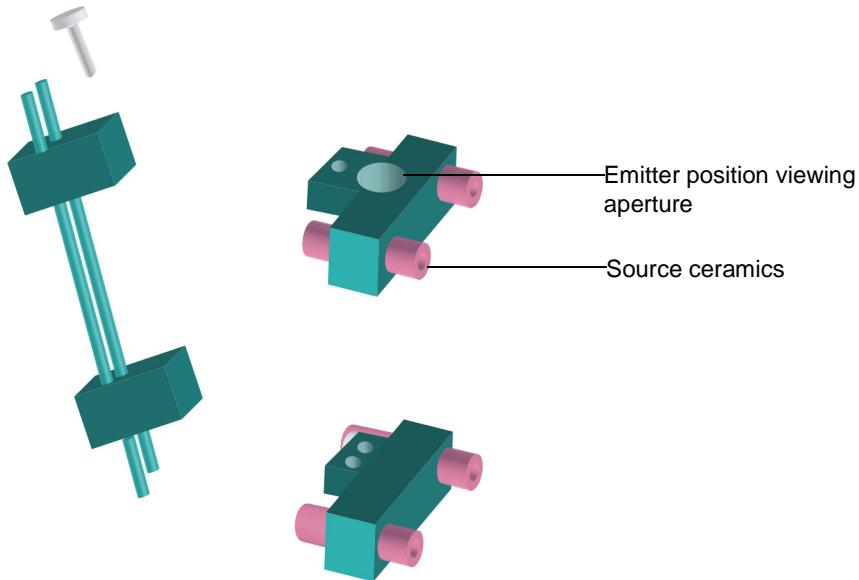
To remove the extraction rod assembly:



Warning: Cleaning the various parts of the source requires solvents and other chemicals that can be flammable and hazardous to health.

1. Close the isolation valve to isolate the source housing.
2. Remove the outer source from the instrument.
See also: [Removing the FI/FD outer source on page 9-6.](#)
3. Make a diagram of where the wires and gas tubing are located.
Rationale: This diagram aids assembling the outer source.
4. Disconnect the two extraction rod contacts.
5. Loosen the retaining screw on the extraction rod assembly.
6. Tilt the rod assembly forward and remove it from the source assembly. The extraction rods can be cleaned with disassembling.

Extraction rods:



7. Clean the rods with fine grade emery paper or fine stainless steel wool.

The surface of the rods should appear highly polished with no scratches or sharp points.

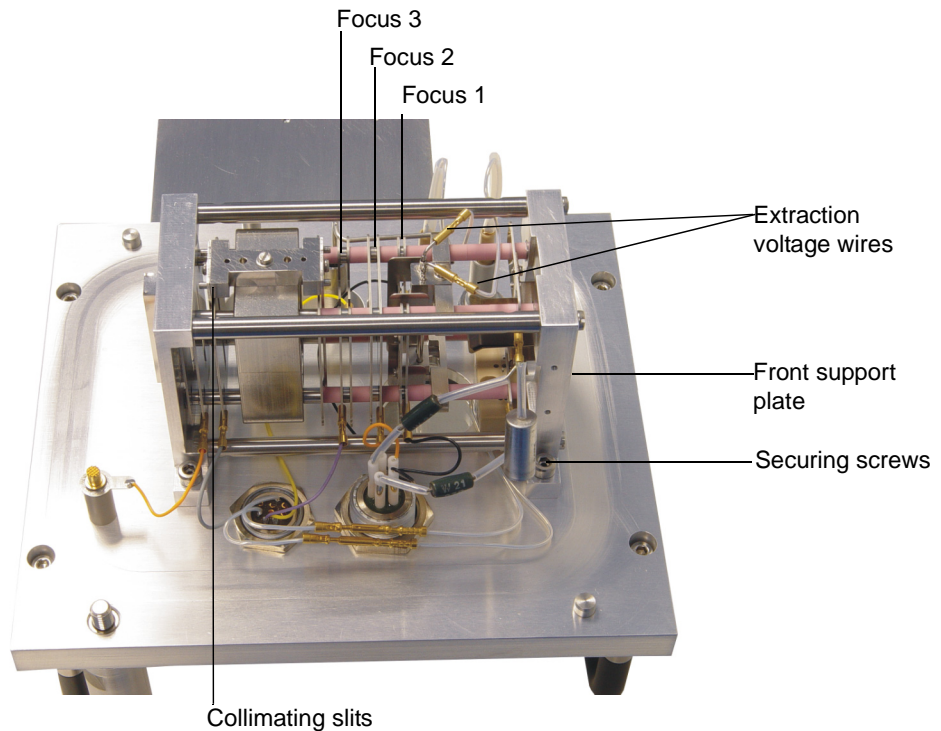
8. Ultrasonically clean the rods in a suitable solvent to remove any remaining grease or sample particles.

See also: [Ultrasonically cleaning the inner source components on page 10-21.](#)

To reassemble the rod assembly:

Refit the rods in the outer source assembly in the reverse to the above procedure, consulting the diagrams prepared during disassembly.

FI/FD outer source:




Cleaning the source ceramic and focus plates

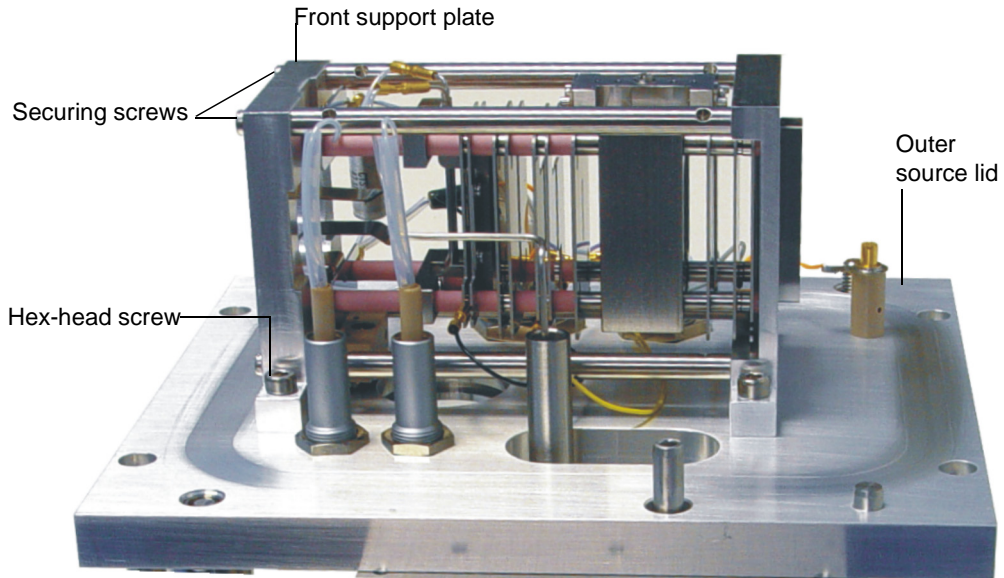
During use, sample builds up on the source ceramics and on the focus plates causing the leakage of extraction voltage and adversely affecting mass accuracy and sensitivity. If voltage leakage occurs, the source must be removed from the source lid assembly, disassembled, and the dirty components individually cleaned.

If the emitter fails due to electrical discharge in the ion source the emitter wire can be subject to a high, short current—enough to vaporize the wire.

If you notice a leakage current of more than 10 mA, this indicates a dirty source.

To clean the focus plates:

1. In the MassLynx Tune window, click  to switch the instrument into standby.
2. Vent the source and wait for the vacuum LED to go yellow.
3. Remove the outer source lid assembly from the instrument.
See also: [Removing the FI/FD outer source on page 9-6.](#)
4. Place the outer source lid assembly on a bench so that the front support plate is at the top.
5. Make a diagram of where the wires and gas tubing are located.
Rationale: This diagram aids assembling the outer source.
6. Unscrew and remove the two hex-head screws that secures the front support plate to the outer source lid.
7. Loosen the remaining two hex-head screws, to allow the outer source assembly to tilt back.



8. Unscrew and remove the four screws that secure the front support plate to the metal rods.
9. Remove the front support plate.
10. Remove each lens plate and its associated spacers from the outer source assembly in turn.

Tip: The outer source comprises the various lens plates held in place by four ceramic rods, one at each corner. Between each lens plate are metal or ceramic spacers of different sizes. To aid reassembly, record on a diagram the order, position, and orientation of each lens and each spacer on its rod. as you dismantle the source. On some layers, the spacers on the upper rods differ from those on the lower rods.

Alternative: You can also access the lenses from the opposite end of the outer source lid assembly, avoiding the need to remove the washers and the spacers around the inner source contacts.

To clean the focus lenses:

1. Rub each side, and the inner aperture, of each lens plate with 1200-grade wet/dry paper to remove any visible ion burn.

Requirement: The inner aperture of each lens plate must be cleaned.

2. Sonicate the lens plates in a suitable solvent to remove any remaining grease.

See also: [Ultrasonically cleaning the inner source components on page 10-21.](#)

Removing the collimating slits

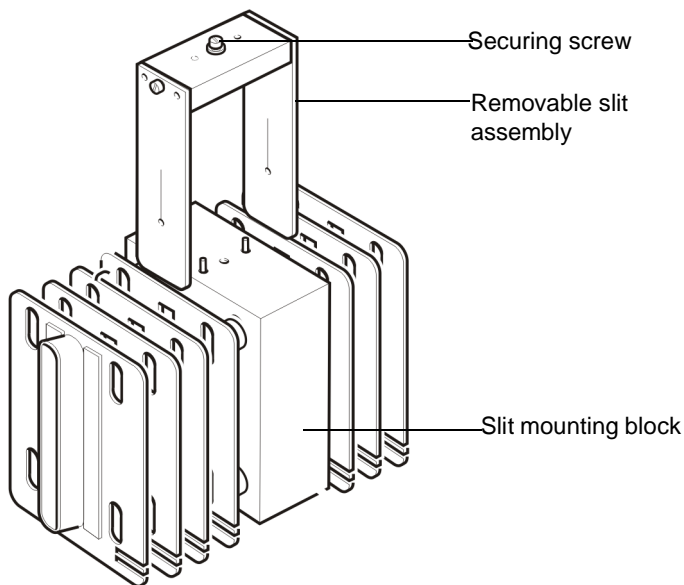
The two collimating slits are 0.25 mm wide and can be removed from the outer source when using FI or FD ion mode.

Note: When the collimating slits are removed from the outer source there is a noticeable improvement in sensitivity, but reduction in resolution.

To remove the collimating slits:

1. Unscrew and remove the securing screws that secures the slit assembly onto the slit mounting block.
2. Lift up and remove the slit assembly from mounting block.

Collimating slits:



3. Rub each slit, with 1200-grade wet/dry paper to remove any visible ion burn.

4. Sonicate the slit in a suitable solvent to remove any remaining grease.
See also: [Ultrasonically cleaning the inner source components on page 10-21.](#)

To assemble the outer source:

Assemble the outer source in the reverse order to which you disassembled it, consulting the diagrams prepared during disassembly.

Checking for leaks after source maintenance

Leaks normally occur when components have recently been removed for cleaning and/or refitted. The following are indications that air is leaking into the instrument:

- Poor source vacuum.
- Large air peaks in the spectrum (nitrogen peak at 28 Da and oxygen peak at 32 Da).
- Problems tuning the instrument.

There are a number of places where an air leak may occur. These tend to include components that are replaceable or serviceable.

- O-rings:
 - Outer source lid
 - Inner source lid
 - GC interface
 - Septum
- GC interface ferrule
- GC column
- Reference reservoir double-ended ferrule
- CI connection on top of the outer source

Inspecting the CI gas line for leaks

You must ensure the CI gas line is gas-tight because flammable or corrosive gases are used. Use the procedure to initially determine whether the CI line leaks.

When the CI purge button is clicked, the internal CI solenoid valves open and the source's pneumatic (PVPK) valve closes. If the GC is connected, the source pressure will rise because the helium cannot be backed away. If a large source pressure rise occurs when you click CI Purge in the Inlet tab, reduce the GC helium flow or remove the GC.

To check the CI gas line for leaks:



Warning: Methane is extremely flammable. When the instrument operates in chemical ionization mode (CI), which involves using of methane, you must safely vent the effluent from the rotary pump. Verify that all fittings are leak-tight and avoid naked flames.



Warning: Ammonia is a hazardous flammable gas. When the instrument operates in chemical ionization mode (CI), which involves using ammonia, you must safely vent the effluent from the rotary pump. Verify that all fittings are leak-tight.



Warning: Isobutane is extremely flammable. When the instrument operates in chemical ionization mode (CI), which involves using isobutane, you must safely vent the effluent from the rotary pump. Verify all fittings are leak-tight and avoid naked flames.

1. Remove the GC and the GC interface.

See also: [Removing the GC interface on page 6-11.](#)

2. Blank off the side flange by fitting the blanking plate to the outer source housing.

See also: [Figure titled “Blanking plate:” on page 6-4.](#)

Alternative: Remove the GC and fit a blank 1/16-inch nut to the GC interface.

3. Pump down (evacuate) the source (without GC.)

4. Connect the CI gas supply to the CI gas connector on the rear of the instrument.
See also: [Gas inlet connections on page 1-19](#).
5. Allow the backing pirani read back to reach 1.00e-2 mbar. If this cannot be achieved note the read back. (Sometimes a minor amount of condensation within the line prevents attaining the required pressure.)
6. In the Tune window Inlet tab, click CI purge.
Result: The read-back should increase to 1.00e0 mbar. The pressure should recover back to the initial backing pressure within 30 seconds.
7. Wait for approximately 10 minutes.
8. Click CI Purge.
Result: There should not be any kick in the pressure. If the read-back does not recover to the initial backing pressure in [step 5](#) or if there is a pressure kick after the second purge in [step 8](#), a leak is present and must be located and corrected.
9. Repeat [step 5 through step 8](#), until no leaks are present.

Detector maintenance

Conditioning the detector


The detector must be conditioned before use. You condition the detector by gradually increasing an applied voltage over a period of time.



Caution: The detector must be conditioned each time the analyzer is vented. Failure to follow the detector conditioning procedure will reduce lifetime of the detector and may destroy it.

To condition the MCP detector:

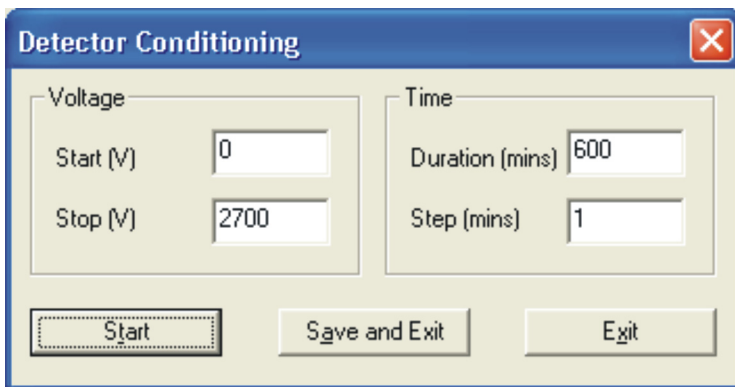
1. Ensure the analyzer and source are pumped down (evacuated). The analyzer vacuum must be 3×10^{-6} mbar for one hour before the conditioning starts.
2. In the MassLynx Tune window Source tab, ensure that the Detector Voltage is set to 0.

3. Click  and confirm that the instrument status indicator shows green.

Result: The instrument switches into operate mode.

4. Click Settings > MCP Conditioning.

Detector Conditioning dialog box:



Caution: Exposing the detector to a large ion current over an extended period reduces detector life.

5. In the Detector Conditioning dialog box, enter the required values as shown.

See also: [Detector Conditioning parameters: on page B-17.](#)

6. Click Start.

Result: The detector is conditioned over 600 minutes (10 hours). A dialog box appears when the conditioning is complete.

Note: Waters recommends that you condition the detector for 10 hours when the analyzer has been vented to air. If the analyzer has been vented to dry gas and not exposed to air, conditioning can be reduced to 60 minutes.

See also: [N₂ Vent on page 1-19.](#)

Setting the optimum gain for the detector

The optimum gain for the detector is determined automatically when the Instrument Setup wizard runs. Using the procedure described to determine the gain of the detector manually.

Note: Before setting the optimum gain for the detector, ensure you conditioned the detector.

To set the optimum gain for the detector:

1. In the MassLynx Tune window, click Settings > TDC Settings.
2. In the TDC Settings dialog box, set the TDC signal threshold to 40 or a previously determined value.
3. Click OK.
4. In the MassLynx Tune window, start acquiring data with heptacosane in continuum at a 0.9 second scan and with an inter-scan delay of 0.1 second, from 50 Da to 650 Da, for a duration of 60 minutes.
5. Set the MCP voltage to 2000 V.
6. Create a chromatogram of the peak at 263 Da.
7. Right-click and drag across the peak at 10%, and view the chromatogram in real-time as the acquisition progresses.
8. Check that the signal at 263 Da is less than 1000 counts per second in continuum mode, to ensure you are not saturating the detector. If the signal is greater than 1000 counts per second, open the reference reservoir pump-out valve to reduce the amount of heptacosane until the counts per second falls below 1000.
See also: [Effects of saturation on peak shape on page B-51.](#)
9. Increase the MCP voltage in increments of 100 V until the ion count reaches a plateau in the chromatogram.
10. Check again that the signal at 263 Da is less than 1000 counts per second in continuum mode, to ensure you are not saturating the detector. If the signal is greater than 1000 counts per second, open the reference reservoir pump out valve until the signal is less than 1000 counts per second. Then return to [step 9](#).
11. Set the MCP voltage 50 V lower than the plateau to ensure that all the ions are counted and excess electrical noise is minimized.

If the MCP voltage does not optimize, or if the increase in signal from 2800 V to 2900 V, is greater than 10%, lower the value of TDC signal threshold by 10 mV and return to [step 2](#). If you have set the TDC signal threshold to less than 10 mV the MCP, detector may need renewing. Contact Waters for advice.

See also: [Contacting Waters on page 11-14](#).

11 Troubleshooting

This chapter describes how to troubleshoot the GCT Premier mass spectrometer with the help of recommended troubleshooting procedures.

Contents:

Topic	Page
Spare parts	11-2
Safety and handling	11-2
System troubleshooting	11-2
Component hardware troubleshooting	11-3
Contacting Waters	11-14

Spare parts

Waters recommends you replace only the parts mentioned in this document.

Safety and handling

When troubleshooting the GCT Premier, keep the following safety considerations in mind.



Warning: To prevent injury, always observe good laboratory practices when handling solvents, changing tubing, or operating the GCT Premier. Know the physical and chemical properties of the solvents used (see the Material Safety Data Sheets for the solvents in use).



Warning: To avoid electric shock, do not remove the instrument's panels. There are no user-serviceable items inside the instrument.

System troubleshooting

There are a few basic steps for performing system troubleshooting:

1. Examine the system, first considering likely causes of the problem. Unresponsive instruments might mean that power or signal cables are disconnected or improperly connected. Similarly, a fluid or vacuum leak might indicate defective plumbing connections.
2. Compare current system operation with the way the system operated before the problem arose. To identify normal operating conditions, maintain a daily log noting overall system performance. Specifically, measure the performance of individual instruments using known samples, preferably the ones used for instrument acceptance.
3. Tracking system parameters and performance during normal operation is a necessary precondition of effective troubleshooting. You must know, for example, when system-tuning parameter values approximate those specified at the time a test sample was previously run or that the lens settings needed to achieve optimum sensitivity are higher than those specified before. A need to specify extreme values to attain good results implies that some part of the system requires attention.

4. Methodically investigate and eliminate possible causes of a problem to identify the atypical system parameter.
5. Consult the troubleshooting information contained in tables on the following pages. The tables identify possible causes of a symptom and suggest corrective actions. If you trace a problem to a system instrument other than the GCT Premier (for example the GC or autosampler), consult the operator's guide for that instrument.

Component hardware troubleshooting

The following tables provide suggestions for resolving most hardware problems.


Power switch fails to power-on the instrument:

Possible cause	Corrective action
Mains power fuse failure.	<p>Unplug the instrument from the main power supply, and replace both the mains power fuses with fuses of the correct type and rating. If the fuses fail continually, isolate the instrument from the power supply and contact Waters for advice.</p> <p>See also:</p> <ul style="list-style-type: none"> • Contacting Waters on page 11-14. • Power fuse on page 1-17.

No peaks in the Tune window (no ion beam):

Possible cause	Corrective action
Cables are not properly connected.	<p>Ensure all necessary cables are correctly attached to the source and probe.</p> <p>See also: Changing the EI/CI inner source assembly on page 2-2.</p>

No peaks in the Tune window (no ion beam): (Continued)

Possible cause	Corrective action
The instrument is not in Operate.	In the MassLynx Tune window, click  . Tip: When in Operate, the instrument status icon is green.
MCP is set to zero.	Set-up the MCP gain. See also: Setting the optimum gain for the detector on page 10-52.
The isolation valve is closed.	Ensure the isolation valve is open by checking the Tune window Engineer tab. See also: The Engineer tab on page B-39. Ensure the pneumatic gas is flowing and set correctly. See also: Table titled "Gas pressures:" on page 3-2.
The filament is broken.	Check the filament current readback, if it is incorrect then replace the filament. See also: Renewing the filament on page 10-32.
The wrong inner source has been fitted for the selected ion mode.	Replace the inner source with the correct one for the selected ion mode. See also: Changing the EI/CI inner source assembly on page 2-2.
The Tune window parameters are incorrectly set.	Ensure that the normal tuning parameters are set and, where appropriate, readback values are acceptable.
No sample is present.	Ensure that sample is loaded correctly in the autosampler, reference reservoir, or probe.

No peaks in the Tune window (no ion beam): (Continued)

Possible cause	Corrective action
Communication failure.	Ascertain the status of the embedded PC. If necessary, reboot the embedded PC using the embedded PC's power switch. See also: Viewing the status of, and rebooting, the instrument's embedded PC on page B-18.
The source components are dirty.	Clean the source components. See also: Cleaning the inner source components on page 10-20.
The source components have been incorrectly assembled.	Ensure that the source voltage read-backs vary with the Tune window setting. If any of these voltages are absent, disassemble and correctly reassemble the source assembly. See also: EI/CI inner source maintenance on page 10-18.
Analyzer or source trip level is set too low and has cut off the voltages.	Increase the trip levels on the engineers tab. See also: The Engineer tab on page B-39.

Unsteady or low intensity peaks (ion beam):

Possible cause	Corrective action
Problem with sample delivery (autosampler, GC system).	Troubleshoot the autosampler. Look for pressure variation on injection.
The source components are dirty.	Clean the source components. See also: Cleaning the complete outer source on page 10-37.

Unsteady or low intensity peaks (ion beam): (Continued)

Possible cause	Corrective action
The source components have been incorrectly assembled.	Ensure that the source voltage readbacks vary with the Tune window setting. If any of these voltages are absent, disassemble and correctly reassemble the source assembly.
Lens settings wrong or atypical.	Ensure all Tune window settings are correct, the readbacks are sensible, and that all lens parameters affect the beam.
Analyzer and detector parameters incorrect.	Ensure the MCP voltage is set correctly. See also: Detector maintenance on page 10-50. Ensure the parameters in the Tune window Engineer tab are set to appropriate values. See also: The Engineer tab on page B-39.

Insufficient vacuum:

Possible cause	Corrective action
Fore pump not operating.	Ensure the pump's switch is turned on.
If applicable, rotary pump not operating correctly.	Gas-ballast the rotary pumps. Inspect the rotary pump oil. If the oil is dirty, flush the pump with clean oil, then refill the pump with new oil. See also: Gas-ballasting the rotary pump on page 10-5.

Insufficient vacuum: (Continued)

Possible cause	Corrective action
Scroll pump not operating correctly.	Contact Waters for advice. See also: Contacting Waters on page 11-14.
Leak in a vacuum backing line.	Inspect the vacuum hoses for cracks or vacuum leaks.
Restriction in the vacuum pump exhaust tubing.	Inspect the exhaust lines for restrictions.
Turbomolecular pump not operating correctly.	Contact Waters for advice. See also: Contacting Waters on page 11-14.
Penning gauge is dirty.	Clean or replace the gauge. Contact Waters for advice. See also: Contacting Waters on page 11-14.
Pneumatic gas pressure too low and valves not opening.	Check the pneumatic gas connection and ensure the pressure setting is correct. See also: Table titled “Gas pressures:” on page 3-2.
Leak in the vacuum housing.	Ensure the O-rings are correctly positioned in the groove, that they are in good condition, and clean and free from dust. See also: Changing the EI/CI inner source assembly on page 2-2.
CI gas is switched on	Switch off the CI gas in the Tune window CI Tune tab CI pane, set the CI Gas Flow (%) to 0. See also: Turning off CI gas on page 3-30.

Source heater not working:

Possible cause	Corrective action
The source heater has failed.	Inspect the Tune window EI tab Source Temp (°C) readback. A readback of 503 indicates that the source heater is disconnected. Check the integrity of the heater connections. See also: Figure titled “Assembled outer source (1):” on page 10-40.

No Trap, Emission, or Filament current readback:

Possible cause	Corrective action
Damaged source filament or poor contact to the filament from the spring contacts on the outer source.	Remove the inner source and test the continuity of the filament with a digital multimeter (the resistance should be approximately 0.2 ohm). Replace the filament if it is damaged. If the filament is intact, use tweezers to adjust the spring contacts on the outer source to improve contact. See also: Renewing the filament on page 10-32.
Filament is turned off.	In the MassLynx Tune window, Source tab, click Reset Filament. See also: The EI Tune tab on page B-22.
Solvent trip level has been exceeded.	Adjust the solvent trip level or correct the vacuum housing.

No Trap, Emission, or Filament current readback: (Continued)

Possible cause	Corrective action
No filament current.	Indicates the power supply for the current has failed. See also: Contacting Waters on page 11-14.

No Filament current, Trap current maximum or Emission current zero:

Possible cause	Corrective action
Particle, or general contamination in the region of the trap, causing leakage current between the block and the trap.	Remove the inner source and exit plate. Check the cleanliness of the trap and trap ceramic. Blow out any particles using dry nitrogen. See also: Cleaning the trap and repeller on page 10-23.

High filament current, high emission current or low Trap current:

Possible cause	Corrective action
Electron entrance aperture blocked, or filament in incorrect position.	Clean the inner source. See also: Cleaning the inner source components on page 10-20.
Poor contact to the trap from the spring contacts on the outer source.	Use tweezers to adjust the spring contacts on the outer source to improve contact. See also: Cleaning the trap and repeller on page 10-23.
One source magnet is reversed in polarity.	Check the source magnet are attracting. See also: Assembling the ion chamber on page 10-41.

Ion repeller inactive in EI mode:

Possible cause	Corrective action
Poor contact to the ion repeller from the spring contacts on the outer source.	Use tweezers to adjust the spring contacts on the outer source to improve contact.

Tunes with repeller more than 5 V:

Possible cause	Corrective action
Dirty inner source.	Clean the inner source. See also: EI/CI inner source maintenance on page 10-18.

Emission: Trap ratio is greater 4:1:

Possible cause	Corrective action
Dirty inner source.	Clean the inner source. See also: EI/CI inner source maintenance on page 10-18.
One of the source magnets has the reverse polarity.	Check the source magnet are attracting. See also: Assembling the ion chamber on page 10-41.

Emission: Trap ratio is greater than 10:1:

Possible cause	Corrective action
Outer source magnets have been fitted incorrectly.	Remove the outer source magnets and reassembly with opposite poles facing each other. See also: Cleaning the complete outer source on page 10-37.

Split peaks seen on the Peak Display or Spectrum:

Possible cause	Corrective action
Incorrect TDC trigger threshold.	Change the TDC trigger threshold (mV) value by 100, within the range 200 to 700 until the peaks are no longer split. See also: Configuring the TDC settings on page B-11.

Poor sensitivity (EI or CI source):

Possible cause	Corrective action
Inefficient, sagging or misaligned filament.	Replace or reposition the filament. See also: Renewing the filament on page 10-32.
Dirty inner source.	Clean the inner source. See also: EI/CI inner source maintenance on page 10-18.
Dirty collimating slits.	Clean the collimating slits. See also: Cleaning the collimating slits on page 10-35.

No Beam or Emitter current (in FI source):

Possible cause	Corrective action
Probe is incorrectly position.	Determine whether the position of the emitter is correct. Using the emitter probe, adjust the knob. See also: Inserting the FI/FD inner source probe on page 9-11.
No extraction voltage or current leakage occurring.	Ensure the extraction voltage read-back is present and the leakage current is less than 10 mA. See also: Obtaining a beam and tuning in FI operation on page 9-21.
Emitter is damaged.	Ensure that the emitter is not damaged by visually inspecting the emitter wire. Check the emitter current readbacks match the set values. Replace the emitter if required or check continually with a multimeter. See also: Changing an emitter on page 10-33.

Excessive leakage current (in FI source):

Possible cause	Corrective action
Internal source wiring is touching grounded elements.	Inspect the source and move the wires.
Continued high leakage current.	Inspect the source. Strip and clean the source if required.

Excessive leakage current (in FI source): (Continued)

Possible cause	Corrective action
Leakage between the extraction rods and the emitter.	Withdraw the emitter by 5 to 10 mm from the extraction rods. Remove the ion source, and clean the extraction rods. See also: Removing the FI/FD outer source on page 9-6 and Cleaning the extraction rods on page 10-42 .

Poor sensitivity for reference material (in FI source):

Possible cause	Corrective action
Emitter is not intact.	Replace the emitter. Tip: There can be significant variation between performance of individual emitters.
Reference reservoir tube has been bent so that it no longer points at the emitter.	Check the reference reservoir tube. See also: GC-FI column position: on page 9-20 .

Poor GC sensitivity (in FI source):

Possible cause	Corrective action
The source end of the column is not cleanly cut.	Check the position of the emitter relative to the end of the column and adjust if required. See also: Figure titled "GC-FI column position:" on page 9-20 .
Column position not lined up with the emitter.	Check the position of the emitter relative to the end of the column and adjust if required. See also: Figure titled "GC-FI column position:" on page 9-20 .

Poor calibration/accurate mass (in FI source):

Possible cause	Corrective action
Extraction rods are dirty.	Clean the extraction rods. See also: Cleaning the extraction rods on page 10-42.

Electrical discharge resulting in damaged emitter (in FI source):

Possible cause	Corrective action
Emitter failed.	Replace the emitter. See also: Changing an emitter on page 10-33.
Discharge in ion source.	Ensure that there is no excessive source contamination by checking the leakage current. If the leakage is more than 10, the ion source needs cleaning. See also: Cleaning the complete outer source on page 10-37.

Contacting Waters

Most operational problems that arise with the GCT Premier are easily correctable. However, if you cannot correct the problem, you must contact Waters.

Customers in the USA and Canada should report maintenance problems they cannot resolve to Waters Technical Service (800 252-4752). All others should visit <http://www.waters.com> and click Offices, or phone their local Waters subsidiary or Waters corporate headquarters at 34 Maple Street, Milford, MA 01757, USA.

When contacting Waters, have the following information available:

- The nature of the symptom.

- The GCT Premier serial number.

Depending on the nature of the fault, it can also be useful to have the following information available:

- Details of the flow rate, GC conditions, and sample concentrations.
- The Tune window settings.
- The software version update reference.

A Starting-up and Shutting-down the Instrument

This appendix describes how to start-up and shut-down the GCT Premier Mass Spectrometer.

Contents:

Topic	Page
Starting-up the instrument	A-2
Shutting-down the instrument	A-3

Starting-up the instrument

To start-up the instrument:

1. Ensure the power switch is switched on at the main power supply.
2. Ensure the compressed air is set to 100 psi.

See also: [Gas inlet connections on page 1-19.](#)

3. Ensure the CI gas is set to 10 psi.
4. Switch on the instrument power on/off switch at the rear of the instrument.
5. Switch on the MassLynx PC.
6. Switch-on the embedded PC (EPC).

See also: [EPC power switch on page 1-20.](#)

7. Allow 3 minutes for the embedded PC to initialize.
8. Start the MassLynx software.

9. In the MassLynx window, click  .

See also: [Opening the MassLynx Tune window on page B-2.](#)

10. Switch on the backing pump.

See also: [Rotary pump maintenance on page 10-5.](#)

11. In the Tune window, click Vacuum > Pump Instrument.


Result: You are prompted to auto-condition the MCP detector. If you select Yes, [steps 13 to 15](#) are completed automatically.

Tip: Waters recommends that you condition the detector for 10 hours when the analyzer has been vented to air. If the analyzer has been vented to dry gas and not exposed to air, conditioning can be reduced to 60 minutes.

See also: [Conditioning the detector on page 10-50.](#)

12. Click the Engineer tab.
13. Ensure that the instrument has pumped sufficiently so that the Vacuum LED on the front panel is green.

See also: [Status display on page 1-9.](#)

14. In the Tune window, click  and confirm that the instrument status indicator shows red.
15. Condition the MCP detector.
See also: [Conditioning the detector on page 10-50](#).
16. Leave the instrument in Operate for at least one hour to warm up and stabilize before calibration or acquiring data.

Shutting-down the instrument

Emergency instrument shutdown

To shut-down the instrument in an emergency:

Turn off the power at the wall-mounted isolation switch(es), if fitted. Otherwise turn off the power at the main power on/off switch, and unplug the power lead from the rear of the instrument. Turn off all peripherals.



Caution: Data can be lost.

Temporary shutdown (switch to standby)

Switch the instrument to standby when it is to be left unattended for a long time, for example overnight or weekends.

To switch the instrument to standby:

1. Click .

Result: The trap and the emission is turned-off automatically and the isolation valve is closed when switched to Standby.


Tip: It is not necessary to switch the instrument out of operate. However, switching to standby is acceptable provided that the one-hour instrument warm-up time is considered when restarting an analysis.

2. If you have been running CI, or when running EI for prolonged periods, close the CI gas bottle valve when switching to standby.

3. In the Tune window CI tab, turn off the CI gas.

Complete shutdown

To shut down the instrument completely:

1. In the Tune window, click  and confirm that the instrument status indicator shows red.
2. Click Vacuum > Vent Instrument.
A message confirms the vent command.
3. Click OK.
The turbomolecular pumps are turned off.
Result: When the turbomolecular pumps have run down to half normal operating speed, the vent valve is opened and the instrument is automatically vented. The Vacuum and Operate status LEDs display red.
4. Exit MassLynx.
5. Shut-down the PC.
6. Turn off the backing pump and switch it off at the main power switch.
7. Turn off all peripherals.
8. Turn the power off at the main power on/off switch (mounted on the rear of the instrument).

B The MassLynx Tune window

This appendix describes the MassLynx Tune window.

Note: For the highest mass accuracy, tune and calibrate the instrument using a suitable reference compound before sample data are acquired (see [Preparing the instrument for operation on page 3-2](#) and [Calibrating the instrument on page 5-2](#)).

Contents:

Topic	Page
Opening the MassLynx Tune window	B-2
The Tune window File menu	B-5
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The Tune window Vacuum menu	B-7
The Tune window Settings menu	B-10
The Tune window View menu	B-19
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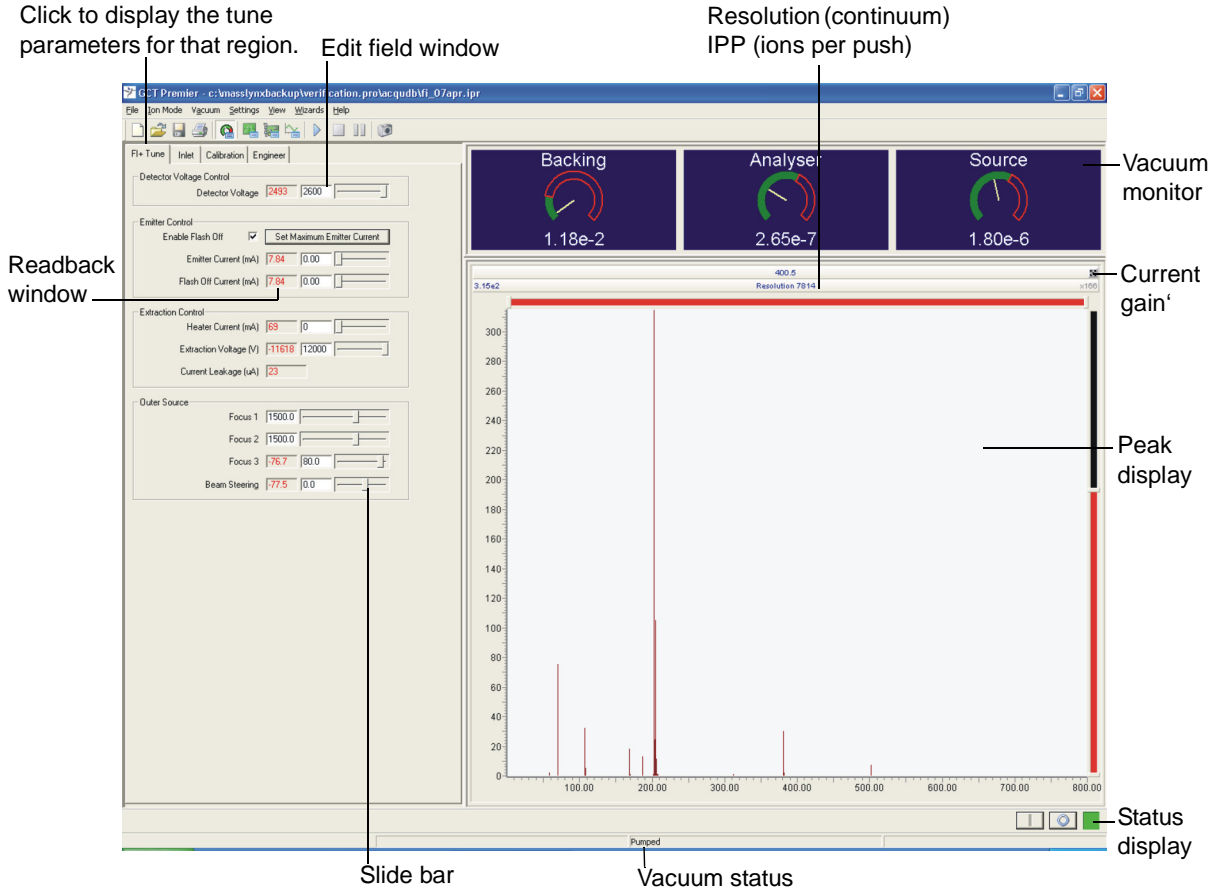
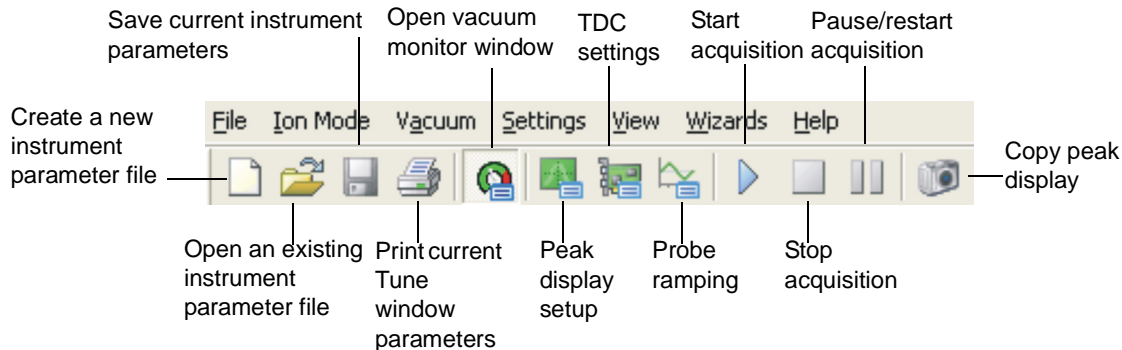
Opening the MassLynx Tune window



Click **MS Tune** in the MassLynx window Instrument shortcut bar.

Result: The Tune window opens. The [Figure titled “The MassLynx Tune window:” on page B-3](#) summarizes the Tune window layout.








The MassLynx Tune window:








The Tune window toolbar

The following table details the Tune window toolbar buttons.

Toolbar buttons:

Tool	Equivalent menu item	Function
	File > New	Creates a new set of default instrument parameters in the Tune window.
	File > Open	Opens an existing instrument parameter file (.ipr).
	File > Save	Saves all the current instrument parameters into the current instrument parameter file (which is indicated in the Tune window title bar). Tip: This option is only available if changes have been made to Tune window parameters since opening the instrument parameter file.
	File > Print	Prints a report containing the peak display and the tune parameters for the current ion mode.
	Vacuum > Monitor	Opens the Vacuum Monitor window; this displays the Pirani and ionization gauge outputs (in mbar) for the various parts of the instrument.
	Settings > Tuning Setup	Opens the Tuning Setup dialog box. This is used to define the start and end mass, scan time, and data format to be used for the peak display.
	Settings > TDC Settings	Opens the TDC Settings dialog box. This is used to define the TDC, DXC and Low Mass cut-off parameters. See also: Low Mass Cut-Off on page B-16.

Toolbar buttons: (Continued)

Tool	Equivalent menu item	Function
	Settings > Probe Ramping	Opens the Probe Control dialog box. This is used to automatically ramp the probe temperature or current. See also: Ramping the probe temperature automatically on page 7-11.
		Starts an acquisition.
		Pauses the current acquisition. Click the button again to restart the acquisition.
		Stops the current acquisition.
	Edit > Copy	Copies an image of the peak display onto the clipboard.

The Tune window File menu

The File menu is used to:

- Create a new instrument parameter file.
- Open an existing instrument parameter file.
- Save an instrument parameter file.
- Print a report of the current Tune window.

To create a new instrument parameter file:

Click File > New.

Result: Creates a new set of default instrument parameters in the Tune window.

To open an existing instrument parameter file:

1. Click File > Open.
2. In the Open dialog box, select the required instrument parameter file (.ipr).
3. Click OK.

To save an instrument parameter file:

Click File > Save.

Result: Saves all the instrument parameters in the current Tune window into the current instrument parameter file (which is indicated in the Tune window title bar).

Tip: This option is available only if changes have been made to Tune window parameters since opening the instrument parameter file.

To save an instrument parameter file to a new filename:

1. Click File > Save As.
2. In the Save As dialog box, enter a filename for the new instrument parameter file.
3. Click OK.

Tip: The Tune window title bar is updated to the new filename.

To print a report of the current Tune window:

1. Click File > Print.
2. In Print dialog box, click OK.


Result: A report containing the peak display and the tune parameters for the current ion mode is printed.

The Tune window Ion Mode menu

The Ion Mode menu is used to select the required ionization mode and update the Tune window Source tab to that mode.

Note: You cannot change the ion mode while the instrument is in operate. Before changing the ion mode, switch the instrument to standby.

To select the ionization mode:

1. In the MassLynx Tune window, click  and confirm that the adjacent instrument status indicator shows red.
2. In the Ion Mode menu, click the required ionization mode.

Tip: The current ionization mode has a check mark next to it.

The Tune window Vacuum menu

The Vacuum menu is used to:

- Monitor the instrument vacuum.
- Pump-down (evacuate) the instrument, analyzer, and source.
- Vent the instrument, analyzer, and source.

Pumping/venting the analyzer

To pump the analyzer:

In the MassLynx Tune window, click Vacuum > Pump Analyzer.

Result: The analyzer begins to pump down.

To vent the analyzer:

In the MassLynx Tune window, click Vacuum > Vent Analyzer.

Result: The analyzer begins to vent.

See also: [Status display on page 1-9.](#)

Pumping/venting the instrument

To pump the instrument:

1. open the reference reservoir pump valve.
2. In the MassLynx Tune window, click Vacuum > Pump Instrument.
The analyzer begins to pump (evacuate). When the pneumatic valve opens, you should hear a small hiss.
When the analyzer is pumped down, the source begins to pump down.
See also: [Status display on page 1-9](#).
Result: The instrument is now pumped.
3. Confirm the Vacuum LED shows green.
Note: You must condition the detector before using the instrument.
See also: [Detector maintenance on page 10-50](#).

To vent the instrument:

1. In the MassLynx Tune window, click Vacuum > Vent Instrument.
The source and analyzer begin to vent.
2. Wait for the Vacuum LED to show red.
See also: [Status display on page 1-9](#).
Result: The instrument is now vented.

Pumping/venting the source

To pump the source:

1. Open the reference reservoir pump valve.
2. In the MassLynx Tune window, click Vacuum > Pump Source.
When the pneumatic valve opens, you should hear a small hiss. Confirm the Vacuum LED shows green.
3. When the Source Turbo value in the Turbo Readbacks frame reaches 80 or more, close the reference reservoir.
Result: The source is now pumped.

To vent the source:

1. In the MassLynx Tune window, click Vacuum > Vent Source.
2. Wait for the Vacuum Status LED to display steady yellow.

See also: [Status display on page 1-9.](#)

Result: The source is now vented.

Monitoring the instrument vacuum

The instrument's vacuum status appears in the Vacuum Monitor window, in mbar. Readbacks from the instrument's pirani gauge on the backing pump and the penning gauges in the analyzer and the source are given.

If any Pirani gauge output pressure reading exceeds the trip level, the relevant fine pump is turned off. There is some hysteresis in the operation of this trip, thus the pressure at which the trip resets when the vacuum is improving is lower than that at which it trips out.

See also: [The Engineer tab on page B-39.](#)

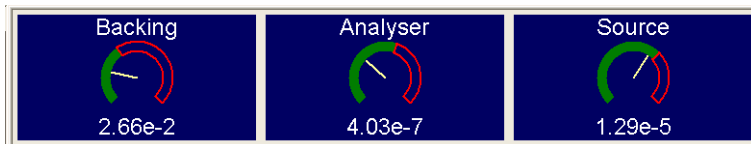
To monitor the instrument's vacuum levels:

In the MassLynx Tune window, click  to open the Vacuum Monitor window.

Alternative: Click Vacuum > Monitor.

Note: The following illustration shows typical values (in mbar) for an instrument when fully pumped-down with a GC fitted and helium flowing at 1 mL/min.

Vacuum monitor window:



The Tune window Settings menu

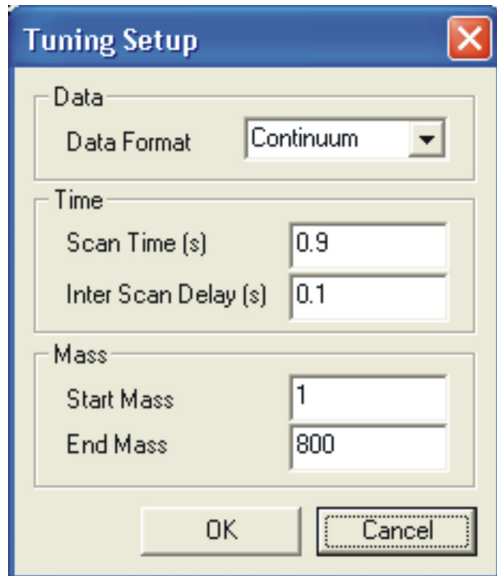
The Settings menu is used to:

- Configure the Tune setup parameters.
- Configure the TDC settings.
- Viewing Calibration parameters.
- Condition the detector.
- Ramp the probe temperature.
- View the status of the instrument's embedded PC.
- Select the instrument name.

Configuring the Tune setup parameters

Click Settings > Tuning Setup to open the Tuning Setup dialog box, which is used to configure the tuning setup parameters during the tuning procedure.

Tuning Setup dialog box:



Tuning Setup parameters

Parameter	Description
Data Format	Lists data formats on the Data Format drop-down list. See also: Types of data acquisition on page 4-14.
Scan Time (s)	Specifies the duration of each scan.
Inter Scan Delay (s)	Specifies the time between the scans. The minimum value is 0.01s (10 ms).
Start Mass	Specifies the starting mass.
End Mass	Specifies the ending mass

See also: [Obtaining an ion beam on page 3-3.](#)

Configuring the TDC settings

Before operating the instrument, you must determine that all of the ions are being counted. multi channel plate (MCP) gain must be optimized and time-to-digital converter (TDC) settings must be correct. Otherwise, the mass accuracy will be compromised.

To set up the TDC to count all the ions:

1. In the Tune window, click Settings > TDC Settings.

TDC Settings dialog box:

TDC Settings

Settings

Trigger Threshold (mV) 350

Signal Threshold (mV) 40

Threshold 1

T0 -50

Inhibit Push

Inhibit Value 0

Inhibit Help

Centroiding Parameters

Centroid Threshold 1

Min Points 5

Np Multiplier 0.7

Resolution 7000

Lock Mass 0

Mass Window +/- 0

DXC Temperature Compensation

Use DXC Temperature Compensation

Drift (PPM/D) 1

Power Port 2

Data

Veff 5285

Low Mass Cut-Off

Set Low Mass Cut-Off

Cut off below mass 50 Da

Zero transmission below -1.#IND Da

Data will be invalid above 0.0000 Da

LMC Help

OK Cancel

2. In the TDC Settings dialog box, set the TDC trigger Threshold parameter value to 350, or to a previously saved value.
3. Set the TDC Threshold parameter value to 40, or to a previously specified value.
4. Observe a beam of the reference compound.
See also: [Tuning the instrument for EI on page 3-19.](#)
5. If you observe peak splitting, change the TDC trigger threshold setting in the region of 200 to 700 until the peak is no longer split.
6. Set the detector for optimum gain.
See also: [Setting the optimum gain for the detector on page 10-52.](#)

TDC Settings dialog box parameters:

Parameter	Description
Trigger Threshold (mV)	Sets the trigger pulse level that triggers the TDC. Specify 350 mV, or the level set at instrument installation, if different. If peak splitting is seen, change the value to another within the range of 200 to 700 until peak splitting is no longer seen. See also: TOF calibration of the instrument in EI on page 5-2.
Signal Threshold (mV)	Sets the signal level that causes the TDC to record an ion arrival event. The default level is 40 mV, but it can be set at 10 to 100 mV. See also: Setting the optimum gain for the detector on page 10-52.
Threshold	Removes any point with an intensity less than the threshold value from the spectrum. For example, if the setting is 5, any peak of five counts or less will not be seen. Typically set to 0 but can be set to 1 or 2 to remove background peaks.
T0	This value is a correction factor for the base mass to time equation. The default value is -50. It affects different masses differently so adjust only if masses are correct at high mass but are poor at low mass, or vice versa.
Inhibit Push	Select the check box to enable the Inhibit Value field. This is used to blank out the “pusher-pulse” seen at approximately 6 Da. Tip: You should use the inhibit push at all times.
Inhibit Value	Enter the inhibit value, a typical value is 4 μ s.
Use DXC Temperature Compensation	Select the check box to enable the Drift (PPM/C) parameter. Adjust the mass scale to compensate for the thermal expansion of the flight tube. See also: Dynamic External Calibration Temperature Compensation on page 5-16.

TDC Settings dialog box parameters: (Continued)

Parameter	Description
Drift (PPM/°C)	Set the size of the correction to be applied to mass measurements to compensate for the effect of temperature drift. See also: Setting the DXC drift value on page 5-16.
Data pane	
Veff	Sets the instrument nominal mass scale. Default value is 5300 V. See also: Setting up nominal mass on page B-47. Note: Veff is determined using the Instrument Setup wizard. See also: Instrument Setup Wizard Nominal Mass: on page 3-11.
Centroiding Parameters pane	
Centroid Threshold	The intensity threshold for peak detection, usually set to 1.
Min Points	Sets the minimum peak width for peak detection. The range is usually within the range 2 to 6. Increase the value if peak splitting is observed in centroid acquisitions.
Np Multiplier	The default value of 0.7 usually gives optimum mass measurement on intense peaks. See also: Setting the Np multiplier on page B-50. Note: Np is customarily determined using the Instrument Setup wizard. See also: Instrument Setup Wizard DDTC Setup: on page 3-13.
Resolution	For optimum dead time correction, set to the actual resolution of the instrument. A precision ± 500 is adequate. Note: The resolution is customarily determined using the Instrument Setup wizard. See also: Instrument Setup Wizard DDTC Setup: on page 3-13.

TDC Settings dialog box parameters: (Continued)

Parameter	Description
Lock Mass	<p>When acquiring in centroid mode each spectrum is corrected by setting the mass of a known peak to an exact mass entered in this field.</p> <p>See also: Lock mass correction on page 5-3.</p> <p>Note: The lock mass is customarily determined using the Instrument Setup wizard.</p> <p>See also: Instrument Setup Wizard Lock Mass Setup: on page 3-14.</p>
Mass Window +/-	<p>The window around the lock mass that is used to look for the peak in the data. Set this value to 0.2 to 0.5.</p>
Low Mass Cut-Off	<p>When selected, enables the low mass cut-off functionality.</p> <p>See also: Low Mass Cut-Off on page B-16.</p>

Low Mass Cut-Off

The low mass cut-off prevents low masses from reaching the detector by cutting off the beam at a set time before the push.

This saves the detector from becoming saturated with data or burning out, which can occur if exposed to high signal intensities of background ions that are not ions of regularly required, such as air, water, CI gas, or helium.

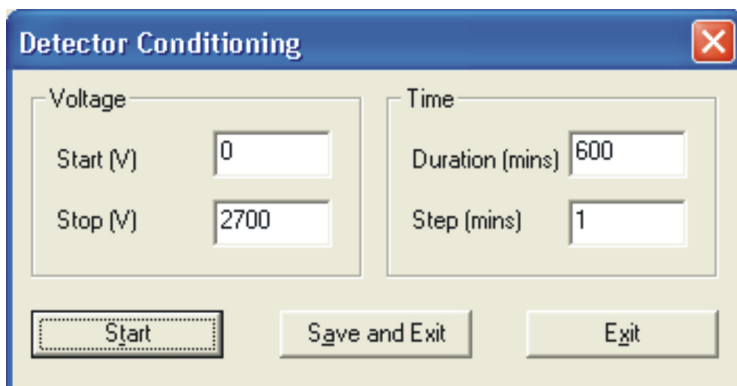
Low Mass Cut-Off parameters:

Parameter	Description
Set Low Mass Cut-Off	Select the check box to enable low mass cut-off. When selected, the cut off below mass field is enabled.
Cut off below mass	Sets a cut off mass, below which masses and attenuated.
Zero transmission below	The value where low mass cut-off starts, which prevents low mass ions being counted as they are completely attenuated below this mass.
Data will be invalid above	The mass above which data are not valid. High masses are reduced in intensity and therefore the accurate mass of the data is adversely affected. This value can be increased using the Pusher Interval on the Engineer tab in the Tune window.

Conditioning the detector

Click Settings > Detector Conditioning to open the Detector Conditioning dialog box, which is used to condition the detector.

Detector Conditioning dialog box:



Detector Conditioning parameters:

Parameter	Description
Start (V)	The voltage at which the detector conditioning starts. Set this voltage to 0.
Stop (V)	The voltage at which the detector conditioning stops. Set this voltage to 2700.
Duration (mins)	The number of minutes it will take to ramp the voltage from the start voltage to the stop voltage.
Step (mins)	The time between each voltage increment. Set this to 1 to avoid large voltage increments.

Detector Conditioning dialog box buttons:

Button	Description
Start	Starts conditioning the detector.
Save and Exit	Saves the parameters for later use, and closes the dialog box.
Exit	Closes the Detector Conditioning dialog box without saving any changes.

See also: [Detector maintenance on page 10-50.](#)

Ramping the probe temperature

Click Settings > Probe Ramping to open the Probe Control dialog box, which is used to ramp the instrument's probe temperature during tuning or data acquisition.

See also: [Ramping the probe temperature automatically on page 7-11](#).

Viewing the status of, and rebooting, the instrument's embedded PC

To view the current status of the instrument's embedded PC:

Click Settings > Options > Communications Status. The System Manager dialog box displays the state of the embedded PC.

To reboot the instrument's embedded PC:

1. Click Settings > Options > Communications Status.
2. In the System Manager dialog box, click Reboot.

Alternative: Reboot the EPC manually, using the EPC's power switch. Doing this is recommended if problems are not cleared by rebooting the EPC from the System Manager dialog box.

Selecting the instrument name

To select the instrument name:

1. Click Settings > Options > Instrument Name.
2. In the Instrument Name dialog box, type the required name in the ID text box.
3. Click OK.

Tip: The instrument name can be added to certain printouts, allowing identification if you are using a communal printer.

The Tune window View menu

The View menu is used to:

- Copy an image of the peak display.
- Hide/display the tuning parameters.
- Hide/display the engineer parameters.
- Hide/display the peak display.
- Hide/display the readback display.

Copying an image of the peak display

To copy an image of the peak display to the clipboard:

Click .

Alternative: Click Edit > Copy.

Result: An image of the peak display is copied to the clipboard, ready for pasting into another application.

Hide/display the tuning parameters

To hide/display the tuning parameters bar:

Click View > Show Tuning Parameters to toggle the tuning parameters between hidden and displayed.

Tip: A check mark is displayed beside the menu item when the tuning parameters are displayed.

Hide/display the engineer parameters

To hide/display the engineer tab:

Click View > Show Engineer tab to toggle the Engineer tab between hidden and displayed.

Tip: A check mark is displayed beside the menu item when the Engineer tab is displayed.

Hide/display the multiple peak displays

To hide/display the multiple peak displays:

Click View > Show Peak Display 2 to show a second peak display.

Click View > Show Peak Display 3 to show a third peak display.

Tip: A check mark is displayed beside the menu item when the peak display is displayed.

The Tune window Wizards menu

The Wizards menu is used to:

- Configuring the instrument for accurate mass.
- Configuring the instrument for calibration.

Note: Before running either of the wizards, ensure that your instrument is tuned for good resolution relative to the ionization mode you are using.

Tuning using the Instrument Setup wizard

To tune using the Instrument Setup wizard:

In the Tune window, click Wizards > Instrument Setup.

Result: The Instrument Setup wizard window appears.

See also: [Obtaining an ion beam on page 3-3.](#)

Calibrating the instrument using the Calibration wizard

To calibrate using the calibration wizard:

In the Tune window, click Wizards > Calibration Wizard.

Result: The Calibration Wizard window appears.

See also: [Using the Calibration wizard on page 5-4.](#)

The Tune window tuning controls

In the tuning control tabs you set the parameters used to tune the instrument. Five tabs are provided:

- Source tab
- Inlet tab
- DRE tab
- Calibration tab
- Engineer tab

Tip: The tuning control tabs can be hidden by clicking View > Tuning Parameters. Click the command again to display the tuning control tabs.

See also: [Hide/display the tuning parameters on page B-19.](#)

The Source tab

The Source tab contains the controls for the instrument's source parameters. The contents of the Source tab varies, depending on the source selected by clicking Ion Mode on the menu bar. The following sections detail the parameters available on each version of the tab.

The EI Tune tab

EI Tune tab:

The screenshot shows the 'EI+ Tune' window with several tabs: 'EI+ Tune', 'Inlet', 'DRE', 'Calibration', and 'Engineer'. The 'EI+ Tune' tab is active. It contains four main sections: 'Detector Voltage', 'Temperature', 'Inner Source', and 'Outer Source'. Each section has input fields for numerical values and graphical sliders. The values shown are: Detector Voltage (2627), Source Temperature (199), Electron Energy (71), Trap Current (1), Filament Current (0.00), Emission Current (4), Ion Repeller (0.9), Beam Steering (-83.2), Focus 1 (-81.7), Focus 2 (-11.7), and Focus 3 (-17.6).

Section	Parameter	Value	Default
Detector Voltage	Detector Voltage	2627	2750
Temperature	Source Temperature (C)	199	200
Inner Source	Electron Energy (eV)	71	70
	Trap Current (uA)	1	100
	Filament Current (A)	0.00	Filament Off
	Emission Current (uA)	4	
Ion Repeller	Ion Repeller	0.9	1.0
Outer Source	Beam Steering	-83.2	0.4
	Focus 1	-81.7	85.0
	Focus 2	-11.7	14.0
	Focus 3	-17.6	20.0

EI Tune tab parameters:

Parameter	Description
Detector Voltage	Sets the voltage across the detector. The default value is 2700 V, which provides a beam. However, for accurate mass this value must be set correctly. See also: Setting the optimum gain for the detector on page 10-52.

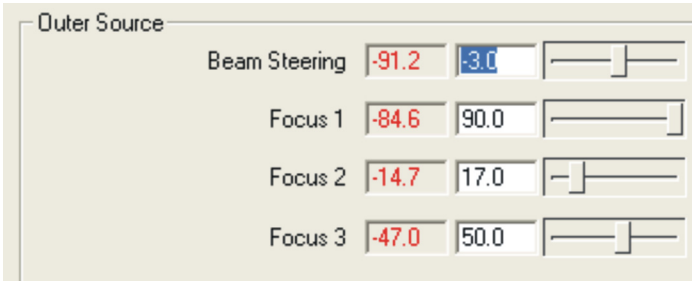
EI Tune tab parameters: (Continued)

Parameter	Description
Source Temperature (C)	<p>The temperature of the source in °C. the normal setting is between 180 and 200 °C which suits most samples. For certain applications—for example, high temperature GC—this can be increased to a maximum of 350 °C.</p> <p>Using a lower temperature can reduce fragmentation. However, this is likely to allow compounds with a high boiling point to condense in the source, which can reduce sensitivity over time.</p> <p>Tip: A readback of 503 indicates that the source heater is disconnected.</p> <p>See also: EI/CI inner source maintenance on page 10-18.</p>
Electron Energy (eV)	<p>The voltage between the filament and the source. Retain the default value of 70.</p> <p>See also: Electron Impact ionization on page G-3.</p>
Trap Current (µA)	<p>Measures the amount of electrons that hit the trap and is used to control the current through the filament in EI mode. The parameter value of between 100 to 200. Increasing the value allows more electrons to enter the source, increasing the ionization of the sample and usually giving an increase in signal intensity.</p> <p>Note: This value is not used during CI or DCI operation.</p>
Filament Current (A)	<p>The current passing through the tungsten filament. This is normally between 4 and 5 A.</p>

EI Tune tab parameters: (Continued)

Parameter	Description
Filament Off	<p>Click the button to switch the filament on and off. This enables you to leave the instrument for a long time without the filament burning out or sagging. In filament save mode (Filament Off), the button changes to Reset Filament.</p> <p>The instrument also defaults to the into filament save mode after a sample list acquisition. The filament stays off until Reset Filament is clicked, or another acquisition is started.</p>
Emission Current (μA)	<p>A measurement of the electrons emitted by the filament, including those that hit the trap. This value can be used to diagnose problems. For example, if the ratio of emission to trap is more than 4:1, the inner source is likely dirty or the filament is misaligned.</p>
Ion Repeller	<p>A plate in the inner source that repels the ions out of the inner source towards the focusing lenses. Set this value to between 0 and 1. As the source becomes dirty, the value needs to be increased. If you increase the value to more than 5 V, you may need to clean the source.</p> <p>See also: Cleaning the trap and repeller on page 10-23.</p>

El Tune tab parameters: (Continued)

Parameter	Description
Beam Steering	<p>The beam steering applies a voltage to one side of the focus lens and reduces voltage on the lens' other side. This redistribution of voltage to change the lens angle effectively steer a beam left or right.</p> <p>The readback from one side of the focus 1 lens appears in the left field of the two Beam Steering fields. The readback for the other side of the focus 1 lens appears in the left Focus 1 field. The readback values appear in red.</p>  <p>The screenshot shows a control panel titled 'Outer Source' with four rows of controls. Each row has a label, a readback value in a red box, a setpoint value in a white box, and a slider. The readback values are: Beam Steering (-91.2), Focus 1 (-84.6), Focus 2 (-14.7), and Focus 3 (-47.0). The setpoint values are: Beam Steering (-3.0), Focus 1 (90.0), Focus 2 (17.0), and Focus 3 (50.0).</p>
Focus 1	Voltage on the first focusing lens.
Focus 2	Voltage on the second focusing lens.
Focus 3	Voltage on the third focusing lens.

Note: Filament and Emission are readback fields.

The CI Tune tab

CI Tune tab:

The screenshot shows the CI Tune tab with the following parameters and controls:

- Detector Voltage:** A numerical input field set to 55 and a slider.
- Temperature:** A numerical input field set to 199 and a target value of 150.
- Inner Source:**
 - Electron Energy (eV): Numerical input field set to 71 and a target of 70.
 - Filament Current (A): Numerical input field set to 0.00 and a Filament Off button.
 - Emission Current (uA): Numerical input field set to 4 and a target of 200.
 - Limit Rhenium Filament Current: A checked checkbox.
- Outer Source:**
 - Beam Steering: Numerical input field set to -48.3 and a target of 0.0.
 - Focus 1: Numerical input field set to -46.7 and a target of 50.0.
 - Focus 2: Numerical input field set to -11.7 and a target of 14.0.
 - Focus 3: Numerical input field set to -37.2 and a target of 40.0.

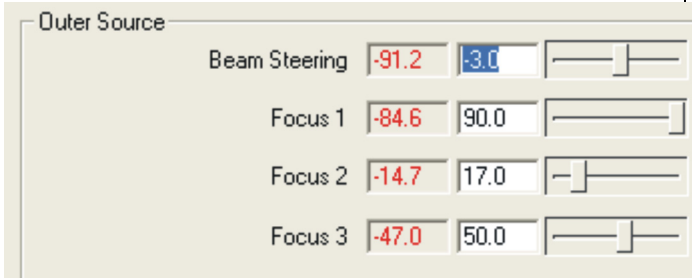
CI Tune tab parameters:

Parameter	Description
Detector Voltage	Sets the voltage across the detector. The default value is 2700 V, which provides a beam. However, for accurate mass this value must be set correctly. See also: Setting the optimum gain for the detector on page 10-52.

CI Tune tab parameters: (Continued)

Parameter	Description
Source Temperature	<p>The temperature of the source in °C is normally set at 100 to 150 °C, which suits most samples. Using a lower temperature may reduce fragmentation. However, this is likely to allow compounds with a high boiling point to condense in the source, which can reduce sensitivity over time.</p> <p>Note: A readback of 503 indicates that the source heater is disconnected.</p> <p>See also: EI/CI inner source maintenance on page 10-18.</p>
Electron Energy	<p>The voltage between the filament and the source. This parameter is set to a default value of 70.</p> <p>See also: .Figure titled “Electron impact ionization:” on page G-4.</p>
Filament Current	<p>The current passing through the filament is normally between 2 and 4 A in CI.</p>
Emission Current	<p>A measurement of the number of electrons emitted by the filament. The default value is 200 µA.</p> <p>Tip: There is no trap in the CI source.</p> <p>See also: Chemical ionization on page G-4.</p>
Limit Rhenium Filament Current	<p>When selected this parameter limits the current to a default maximum value of 3.6 A, which prevents damage to the Rhenium filament if it is accidentally inserted into the source in EI mode.</p> <p>Note: If for some reason you fit a tungsten filament on the CI source, ensure that this check box is cleared.</p> <p>Caution: To prevent damage to the rhenium filament, select the check box.</p>

CI Tune tab parameters: (Continued)

Parameter	Description
Outer Source pane	
Beam Steering	<p>The beam steering applies a voltage to one side of the focus lens and reduces voltage on the lens' other side. This redistribution of voltage to change the lens angle effectively steer a beam left or right.</p> <p>The readback from one side of the focus 1 lens appears in the left field of the two Beam Steering fields. The readback for the other side of the focus 1 lens appears in the left Focus 1 field. The readback values appear in red.</p>  <p>The screenshot shows the 'Outer Source' control panel with the following parameters and values:</p> <ul style="list-style-type: none"> Beam Steering: -91.2 (red), -3.0 (blue) Focus 1: -84.6 (red), 90.0 Focus 2: -14.7 (red), 17.0 Focus 3: -47.0 (red), 50.0
Focus 1	Voltage on the first focusing lens.
Focus 2	Voltage on the second focusing lens.
Focus 3	Voltage on the third focusing lens.

Note: Filament Current (A) is a readback only.

The FI Tune tab

FI Tune tab:

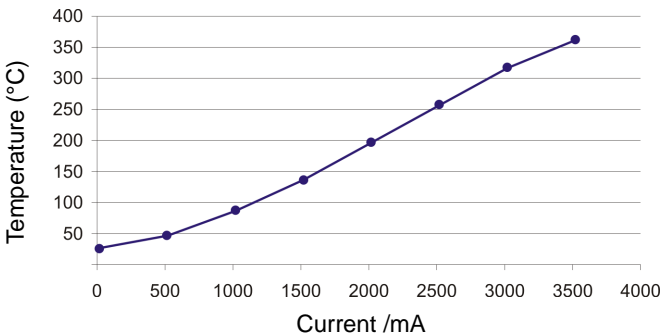
The screenshot shows the 'FI+ Tune' control panel with the following sections and parameters:

- Detector Voltage Control:** Detector Voltage is set to 0.
- Emitter Control:** 'Enable Flash Off' is unchecked. 'Set Maximum Emitter Current' is a button. Emitter Current (mA) is 0.00. Flash Off Current (mA) is 0.00.
- Extraction Control:** Heater Current (mA) is 0. Extraction Voltage (V) is 0. Current Leakage (uA) is 0.
- Outer Source:** Focus 1 is 0.0. Focus 2 is 0.0. Focus 3 is 0.0. Beam Steering is 0.0.

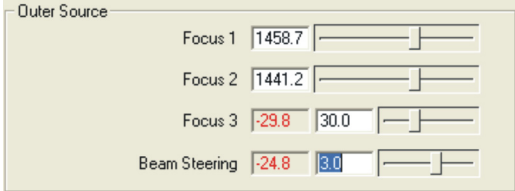
FI Tune tab parameters:

Parameter	Description
Detector Voltage	Sets the voltage across the detector. The default value is 2700 V, which provides a beam. However, for accurate mass this value must be set correctly. See also: Setting the optimum gain for the detector on page 10-52.
Enable Flash Off	When selected, enables the Flash Off Current field.

FI Tune tab parameters: (Continued)

Parameter	Description																		
Set Maximum Emitter Current button	Opens the Emitter Current Control dialog box. You can specify the maximum emitter current to be used. For FI mode, use 10 mA.																		
Emitter Current	The current that is passed through the emitter during an acquisition or tuning. Tip: For maximum sensitivity, this parameter is normally set to zero.																		
Flash Off Current	Sets the emitter current, which is used during the inter scan delay. This function desorb any condensed material on the emitter and ensures maximum sensitivity. (A value of 3 to 8 mA can be required).																		
Heater Current	The current passed through the extraction rods to heat the rods. The rods are normally heated when using waxy samples. Note: The absolute sensitivity of the instrument decreases when the rods are heated.																		
	<p>Note: The rods should normally be left cold</p> <p>The graph below shows the increase in temperature as the current applied to the rods is increased.</p> <p style="text-align: center;">Heated FI rods</p>  <table border="1"> <caption>Data points from the Heated FI rods graph</caption> <thead> <tr> <th>Current /mA</th> <th>Temperature (°C)</th> </tr> </thead> <tbody> <tr> <td>0</td> <td>30</td> </tr> <tr> <td>500</td> <td>50</td> </tr> <tr> <td>1000</td> <td>90</td> </tr> <tr> <td>1500</td> <td>140</td> </tr> <tr> <td>2000</td> <td>200</td> </tr> <tr> <td>2500</td> <td>260</td> </tr> <tr> <td>3000</td> <td>320</td> </tr> <tr> <td>3500</td> <td>360</td> </tr> </tbody> </table>	Current /mA	Temperature (°C)	0	30	500	50	1000	90	1500	140	2000	200	2500	260	3000	320	3500	360
Current /mA	Temperature (°C)																		
0	30																		
500	50																		
1000	90																		
1500	140																		
2000	200																		
2500	260																		
3000	320																		
3500	360																		

FI Tune tab parameters: (Continued)

Parameter	Description
Extraction Voltage	<p>This is the high voltage applied to the extraction rods. Normally the extraction voltage is 12000 V. The voltage and leakage current are shown as readbacks. The voltage should be increased from zero to 12000 V manually in increments of 2000 V.</p> <p>Tip: If excessive current leakage is seen, the rods should be cleaned.</p>
Current Leakage	<p>If the value rises above a steady value of 10 μA, there is an excessive leakage of the extraction voltage to the ground. This indicates that the source is dirty and requires cleaning.</p> <p>See also: Cleaning the complete outer source on page 10-37.</p>
Beam Steering	<p>The beam steering is a voltage added to one side of Focus 3 lens and taken off the other side of Focus 3 lens. This has the effect of steering the beam to the left or right.</p> <p>The readback for one side of the Focus 3 lens is shown next to Beam Steering and the other side of the Focus 3 lens is shown next to Focus 3, as shown in the following figure:</p> 
Focus 1	Voltage on the first focusing lens.
Focus 2	Voltage on the second focusing lens.
Focus 3	Voltage on the third focusing lens.

The FD Tune tab

FD Tune tab:

FD+ Tune

Detector Voltage

Detector Voltage 0 0

Emitter Control

Set Maximum Emitter Current

Emitter Current (mA) 0 0

Extraction Control

Heater Current (mA) 0 0

Extraction Voltage (V) 0 0

Current Leakage (uA) 0

Outer Source

Focus 1 0.0

Focus 2 0.0

Focus 3 0 0.0

Beam Steering 0 0.0

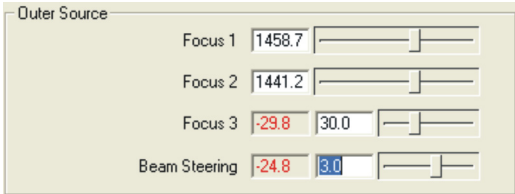
FD Tune tab parameters:

Parameter	Description
Detector Voltage	Sets the voltage across the detector. The default value is 2700 V, which provides a beam. However, for accurate mass this value must be set correctly. See also: Setting the optimum gain for the detector on page 10-52.

FD Tune tab parameters: (Continued)

Parameter	Description
Set Maximum Emitter Current button	Opens the Emitter Current Control dialog box. You can specify the maximum emitter current to be used. For FD mode, use 100 mA.
Emitter Current (mA)	The current that is passed through the emitter during an acquisition or tuning.
Heater Current (mA)	The current passed through the extraction rods to heat the rods. The rods are normally heated when using waxy samples. Note: The absolute sensitivity of the instrument decreases when the rods are heated. See also: Heater Current (mA) on page B-33.
Extraction Voltage (V)	This is the high voltage applied to the extraction rods. Normally the extraction voltage is 12000 V. The voltage and leakage current are shown as readback. The voltage should be increased from zero to 12000 V manually in increments of 2000 V. Note: If excessive current leakage is seen, the rods should be cleaned.
Current Leakage (μ A)	If the value rises above a steady value of 10 μ A, there is an excessive leakage of the extraction voltage to the ground. This indicates that the source is dirty and requires cleaning. See also: Cleaning the complete outer source on page 10-37.
Focus 1	Voltage on the first focusing lens.
Focus 2	Voltage on the second focusing lens.
Focus 3	Voltage on the third focusing lens.

FD Tune tab parameters: (Continued)

Parameter	Description
Beam Steering	<p>The beam steering is a voltage added to one side of Focus 3 lens and taken off the other side of Focus 3 lens. This has the effect of steering the beam to the left or right.</p> <p>The readback for one side of the Focus 3 lens is shown next to Beam Steering and the other side of the Focus 3 lens is shown next to Focus 3, as shown in the following figure:</p>  <p>The screenshot shows a control panel titled 'Outer Source' with four rows of controls. Each row has a label, a numerical value, and a slider. Focus 1 has a value of 1458.7. Focus 2 has a value of 1441.2. Focus 3 has a value of -29.8 and a secondary value of 30.0. Beam Steering has a value of -24.8 and a secondary value of 3.0.</p>

The Inlet tab

Tip: The Inlet tab Probe parameter configuration varies depending on the probe selected in the Tune window Inlet menu. The Probe parameter can be for a DCI Probe or DI probe (solids probe) and displayed parameter changes accordingly. [Figure titled “Inlets tab:” on page B-35](#) shows the default Inlet tab (for the solids probe), which appears when no option is selected in the Inlet menu.

Inlets tab:

Inlets tab parameters :

Parameter	Description
Use DCI mode	When selected, the parameters displayed change to reflect the DCI probe.
Solids Probe	<p>The temperature of the solids probe in °C. If the readback value of the probe shows 850, the probe cable has not been correctly connected.</p> <p>See also: Inserting the DI probe into the insertion lock on page 7-6.</p> <p>Tip: For safety reasons, the temperature is set to 0 at the start of an acquisition. Once the acquisition has started, you can manually ramp the probe temperature using this control.</p> <p>See also: Ramping the probe temperature manually on page 7-10.</p>

Inlets tab parameters (Continued):

Parameter	Description
DCI Probe	<p>The current of the DCI probe.</p> <p>See also: Ramping the probe current manually on page 8-7.</p> <p>Tip: For safety reasons, the current is set to 0 at the start of an acquisition. Once the acquisition has started, you can manually ramp the probe current using this control.</p> <p>See also: Ramping the probe current manually on page 8-7.</p>
GC Re-entrant	<p>Sets the temperature of the GC interface. A typical value is 250 °C. The parameter should normally be more than or equal to the maximum temperature of the GC oven ramp (to avoid chromatographic problems due to sample condensing in the GC-Re entrant).</p> <p>Tip: If the readback value is 503, the GC re-entrant cable is not connected.</p>
Reference Reservoir	<p>Sets the temperature of the reference reservoir. A typical value is 40 to 50 °C, which prevents condensation within the reservoir and speeds the removal of reference material when changing ionization mode.</p> <p>Note: If the readback value is 503, the reference reservoir cable is not connected.</p>
Reference Re-entrant	<p>Sets the temperature for the transfer line, which runs from the reference reservoir to the ion source. A typical value is 100 to 150 °C, which minimizes condensation of the reference material.</p> <p>Tip: If the readback value is 503, the reference reservoir cable has not been connected.</p>
CI Gas	When selected, turns on the CI gas.
CI Purge	<p>Allows the CI gas to be purged.</p> <p>Caution: Ensure that the CI gas is closed off after the regulator before purging.</p>

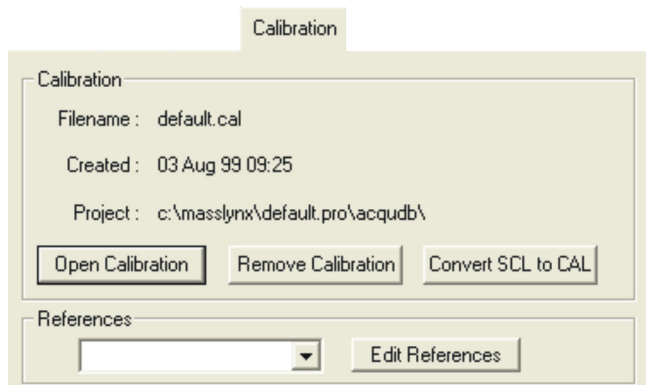
Inlets tab parameters (Continued):

Parameter	Description
CI Gas Flow%	Regulates the amount of CI gas going into the source (from 1 to 100%). Tip: 100% equals approximately 1 mL/min of methane or ammonia.

Calibration tab

The Calibration tab allows you to associate a previously created calibration file to a new calibration. You can also remove calibration files and convert SCL files to CAL files from this tab.

Calibration tab:



Calibration tab parameters:

Parameter	Description
Filename	Shows the path and file name of the calibration file currently in use.
Created	Shows the date and time the calibration file was created.
Reference list	Lists the available reference files.
Edit Reference	Opens the selected reference file in Microsoft Notepad®.

Calibration tab buttons

Button	Description
Open Calibration	Opens the Open dialog box in which you can select a new calibration file.

Calibration tab buttons

Button	Description
Remove Calibration	Replaces the current calibration file with the Uncal.cal file, an uncalibrated file.
Convert SCL to CAL	Converts a manual calibration file (.scl) to a calibration file (.cal) which can be used for subsequent acquisitions from the Tune window and Sample List.

The Engineer tab

The voltages the Engineer tab are set for optimum resolution. These voltages should not be changed during routine operation.

Tip: You should record the voltages specified on the Engineer tab for future reference.

Engineer tab:

Engineer

Time Of Flight (TOF)

Entrance

Pushout Voltage

Pusher Interval

Pusher Bias

Grid 2

Reflectron Voltage

TOF Flight Tube

Solvent Trip

Source Solvent Trip

Analyser Solvent Trip

Turbo Readbacks

Source Turbo

Analyser Turbo

Engineer tab parameters:

Parameter	Description
Entrance	The voltage of the analyzer entrance slit. Set the value to between 38 and 43 V.
Pushout Voltage	The voltage of the pulser plate that pushes ions down the flight tube. Set the value to between 960 and 970 V.

Engineer tab parameters: (Continued)

Parameter	Description
Pusher Interval	<p>Sets the frequency of the pusher pulse. Set the value to between 33 and 250 μsec.</p> <p>The default value is 40, but this can be increased to accommodate higher masses.</p> <p>Requirement: To achieve good resolution, you need to retune the Pusher Bias and the Grid 2 parameters if the pusher interval is changed.</p>
Pusher Bias	<p>The voltage on the third grid of the pusher. This is the most important parameter when tuning for the best resolution. Normally specified to 1.0 V above the entrance value, but tune for each instrument.</p>
Grid 2	<p>The voltage of the second grid in the pusher stack. This is the second most important tuning parameter for good resolution and typically is set between 0 and 60 V.</p>
Reflectron Voltage	<p>The voltage of the reflectron. The default value is between 1760 and 1790 V.</p>
TOF Flight Tube	<p>The voltage of the flight tube. The default value is between 4600 and 4700 V.</p> <p>Note: If the readback value is approximately -9900, this indicates that the voltage for the TOF Flight Tube is off.</p>
Source Solvent Trip	<p>Changing this value changes the position of the green trip-level indicator in the source vacuum display. This function protects the source filament from the rise in source pressure as the solvent front elutes in a GS/MS experiment.</p> <p>Caution: The Source Solvent Trip value must always be set to a pressure slightly higher than the current pressure reading.</p>

Engineer tab parameters: (Continued)

Parameter	Description
Analyser Solvent Trip	<p>Changing this value changes the position of the green trip level indicator in the analyser vacuum display.</p> <p>The function shuts off all analyser and source voltages if tripped and protects the instrument against pressure surges. Set a value of about 5.5, which corresponds to about $3e-6$ mbar.</p>
Source Turbo	<p>The speed at which the source turbo operates. When the source is pumped down (evacuated) the value should be between 95 and 100%. A value below 90% indicates a cooling fault.</p> <p>See also: Contacting Waters on page 11-14.</p>
Analyser Turbo	<p>The speed at which the analyser turbo operates. When the analyzer is pumped down (evacuated) the value should be between 95 and 100%. A value below 90% indicates a cooling fault.</p> <p>See also: Contacting Waters on page 11-14.</p>

Engineer tab buttons

Button	Description
Open/Close Isolation Valve	Controls the position of the isolation valve between the source and the analyser housing, regardless of whether the instrument is in standby or operate mode.
Diagnostic Message Log	Displays an updated list of diagnostic messages.

The DRE tab

DRE tab:

Sensitivity Control

High Sensitivity

z-focus (V)

z-lens steering (V)

DRE Options

Gain Drop Monitoring

Multiple Peak Stitching

Multiple Peak Stitching options

Pre Window: Da Post Window: Da

DRE Magnification Factor

Mass	Magnification factor
<input type="text" value="50.000"/>	<input type="text" value="20.000"/>
<input type="text" value="100.000"/>	<input type="text" value="20.000"/>
<input type="text" value="150.000"/>	<input type="text" value="20.000"/>
<input type="text" value="200.000"/>	<input type="text" value="20.000"/>
<input type="text" value="500.000"/>	<input type="text" value="20.000"/>

DRE tab parameters :

Parameter	Description
High Sensitivity	When the parameter is selected, the instrument operates in high-sensitivity mode for the z-focus and the z-lens steering. When the parameter is cleared, the instrument operates in low-sensitivity mode for the z-focus and the z-lens steering.

DRE tab parameters (Continued):

Parameter	Description
z-focus	The voltage applied to the z-focus. For high-sensitivity work the voltage is set to approximately 42. For low-sensitivity work the voltage is set to approximately 250.
z-lens steering	Steers the beam up and down using the DRE lenses. The specified value is normally set to 0.
Gain Drop Monitoring	When the parameter is selected, accurate mass results are produced if the gain of the detector has been reduced by a large signal. See also: Gain drop monitoring on page G-13.
Multiple Peak Stitching	When an intense ion signal strikes the detector the neighboring isotopes can be adversely affected. See also: Multiple peak stitching on page G-12.
Pre Window	A mass pre-window to the large ion signal where data is stitched in from the attenuated data.
Post Window	A mass post-window to the large ion signal where data is stitched in from the attenuated data.
Mass	Five masses across the mass range at which the DRE magnification factor has been calculated
Magnification factor	The corresponding magnification factor at each of the specified mass. See also: Setting up DRE mode on page 4-15.

The Tune window peak display

Customizing the peak display

Opening the peak display menu

Use the peak display menu to customize the peak display.

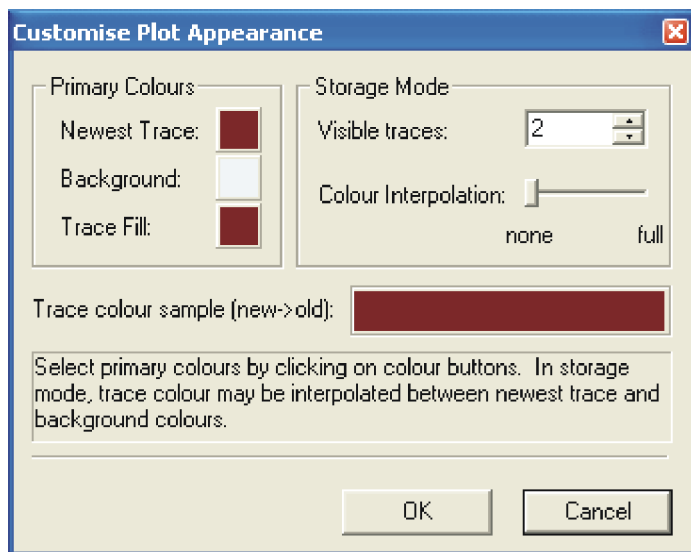
To open the peak display menu:

In the MassLynx Tune window, right-click in the peak display window.

Customizing the colors and numbers of displayed traces

To change the color of the background and traces and the number of traces displayed, on the Peak Display menu, click Customise > Plot Appearance.

Customise Plot Appearance dialog box:



To change the colors in the display:

1. In the Customise Plot Appearance dialog box, click the color box adjacent to Newest Trace, Background, or Trace Fill as required.
2. Select a new color from the Color dialog box.

To change the number of displayed traces:

1. In the Customise Plot Appearance dialog box, type the required value in the Visible traces box, within the range 2 to 20.

Customizing the peak trace line appearance

You can customize the peak trace line appearance using the Trace command in the Peak Display menu.

See also: [Customizing the peak display on page B-45](#).

Each trace may be displayed as follows:

- Outline only: click Trace > Outline.
- With the area below the line filled: click Trace > Fill.
- Maximum and minimum points only: click Trace > Min/Max.

Tip: The peaks can appear to be very narrow, so you should select the Min/Max option.

Customizing the peak intensity display

Tip: Check marks are displayed beside the selected options in the Peak Display menu.

To display the peak intensities as absolute values (counts/second):

In the Peak Display menu, click Intensity > Absolute Intensity.

To display the peak intensities as percentage values relative to the intensity of the highest peak:

In the Peak Display menu, click Intensity > Relative Intensity.

To control the way in which the peak display is scaled:

The Peak Display menu Intensity > Normalise Data option can be selected in conjunction with either of the previous options. It controls the way in which the peak display is scaled. When enabled, the display scales to the value of the intensity of the highest peak; when disabled, the display scales to the last set value.

Setting up nominal mass

The instrument's mass scale is set up to provide nominal mass measurement without calibration. This specifies a V_{eff} (effective potential) value on installation. The V_{eff} value must be routinely evaluated and changed.

See also: Table titled "TDC Settings dialog box parameters:" on page B-13.

TDC Settings dialog box:

The screenshot shows the TDC Settings dialog box with the following values:

- Settings:** Trigger Threshold (mV) 350, Signal Threshold (mV) 40, Threshold 1, T0 -50, Inhibit Push (unchecked), Inhibit Value 0.
- Centroiding Parameters:** Centroid Threshold 1, Min Points 5, Np Multiplier 0.7, Resolution 7000, Lock Mass 0, Mass Window +/- 0.
- DXC Temperature Compensation:** Use DXC Temperature Compensation (unchecked), Drift (PPM/D) 1, Power Port 2 (unchecked).
- Data:** Veff 5285.
- Low Mass Cut-Off:** Set Low Mass Cut-Off (checked), Cut off below mass 50 Da, Zero transmission below -1.##ND Da, Data will be invalid above 0.0000 Da.

Manually setting the V_{eff} correctly

The first stage of calibrating the mass scale requires that you set the V_{eff} parameter to give nominal mass. When V_{eff} is set correctly, the masses are correct to within ± 0.5 Da.

By changing V_{eff} , the effective mass of the ions changes.

V_{eff} is automatically set up during the Instrument Setup wizard.

See also: [Instrument Setup Wizard Nominal Mass: on page 3-11](#)

To set up Veff manually:

1. Introduce a known standard compound into the reference reservoir (for example, heptacosane).
2. Reduce the amount of sample using the reference reservoir value so that the intensity of the signal is less than 1000 counts per second in continuum mode to avoid saturation effects.

See also: [Effects of saturation on peak shape on page B-51](#).

3. In the Tuning Setup dialog box, Data Format list, click Centroid.
4. Observe a known peak on the Tune window.

Tip: For example, observe the peak at m/z 218.9856 when using heptacosane.

5. In the TDC Settings dialog box, set the Veff to 5300.

Note: With Veff set to 5300, the peak should be within $m/z \pm 10$ of the correct mass.

To alter the mass of the peak of interest:

1. Increase Veff to move the peak to higher mass.
2. Decrease the Veff to move the peak to lower mass.
3. In the TDC Settings window adjust the Veff so that the peak is at the correct mass (to within m/z 0.1).
4. Click OK.

Note: Veff is automatically determined by the Instrument Setup wizard.

See also: [Instrument Setup Wizard Nominal Mass: on page 3-11](#).

Setting up resolution for dead time correction manually

The resolution value on the TDC Settings window is used for digital dead time correction (DDTC). It is set up automatically during the Instrument Setup wizard.


See also: [Instrument Setup Wizard DDTC Setup: on page 3-13](#).

To set up resolution parameter manually:

1. Observe a beam of tris (trifluoromethyl) triazine.
2. Use the reference reservoir pump valve to reduce the amount of reference sample until there are less than 1000 ion counts per second in continuum mode to avoid saturation effects.
3. Acquire data in continuum mode and sum up several spectra so there are more than 2000 ion counts peak height viewed in the spectrum.
4. Measure full-width-half-maximum (FWHM) resolution of the peak at m/z 285.
5. Use the following equation to calculate the resolution:

$$Resolution = \frac{Mass}{FWHM}$$

Tip: Resolution should be greater than 7000, (when measured at the 614 peak of heptacosane) Resolution reduces as the peak mass reduces.

6. In the MassLynx Tune window, click .
7. In the TDC Settings dialog box, type the calculated resolution value in the Resolution field.

Note: Resolution is automatically determined by the Instrument Setup wizard.

See also: [Instrument Setup Wizard Introduction: on page 3-9.](#)

Dead time correction

The data acquisition system for the instrument is a time-to-digital (TDC) converter, an ion counting system that generates a mass spectrum by creating a histogram of ion arrival times of ions in memory. After the arrival and registration of an ion by the TDC, a minimum time interval occurs before a subsequent ion arrival can be registered. The interval is called the dead time of the TDC and is of the order of 5 ns. If more than one ion arrives in the same time increment, then only one count is registered.

At high ion currents, some of the ion arrivals are not registered, this leads to a shift to lower mass centroids and lower measured areas on reported peaks.


However, MassLynx incorporates digital dead time correction (DDTC™), which compensates for these effects and enables you to achieve accurate mass measurement and quantitation over a large range of ion currents.

See also:

- [Setting the Np multiplier on page B-50](#)
- [Effects of saturation on peak shape on page B-51](#)
- [Setting up resolution for dead time correction manually on page B-48](#)

Setting the Np multiplier

To set the Np Multiplier:

1. In the TDC Settings dialog box, set the Np Multiplier to 1.0.
2. Introduce reference sample tris (trifluoromethyl) triazine.
See also: [Introducing the reference sample on page 3-6](#).
3. Acquire data for calibration.
See also: [Acquiring data for calibration on tab 5-16](#).
4. Display a single spectrum.
5. In the Spectrum window, click  to view the spectrum in real-time.
6. When the peak at m/z 285 appears with the question mark over it, reduce the intensity of the peak by opening the reference reservoir valve until the moment when the symbol disappears.
Rationale: Saturation is indicated when a peak appears with a question mark above it. When setting Np the signal must be strong, but not saturated so that some dead time effect will be seen on the large isotope.
7. Recalibrate using the method above and the metri.ref reference file and examine the residuals in the calibration output.

Consider the residuals field in the calibration display:

- If the first isotope marker lies below the line of best fit compared to the second isotope marker, insufficient dead time correction is applied. Decrease Np multiplier by 0.1, and repeat [steps 3 through 7](#).

- If the first isotope marker is above the best fit line compared with the second isotope marker, then too much dead time correction is being applied. Increase Np multiplier by 0.1, and repeat [steps 3 through 7](#).

Result: In the spectrum, the first and second isotope peaks of the tris (trifluoromethyl) triazine ion should be within 1 mDa of each other on the residual output. This value of Np is typically 0.6 to 0.8 and should be used from now on.

Effects of saturation on peak shape

The time to digital converter (TDC) records the time at which an ion strikes the detector. MassLynx uses this time value to determine the mass of the ion.

As the number of ions increases, saturation of the TDC causes the peak shape to characteristically change as an increasing numbers of ions are not detected.

At the onset of saturation, the peak shifts a little to lower mass. This is followed by a failure to increase recorded intensity because the ion signal continues to increase. There will then be a sudden, sharp, high-mass cut off. Finally, the detector will appear to “ring”, causing a secondary peak to appear on the high mass side of the peak of interest.

Figure titled “Schematic diagram of peak shape saturation effects:” on [page B-52](#) represents the characteristic transition from unsaturated signal (A and B) to saturated signal (C, D, and E), which appears when tuning using the real time peak display in continuum mode. Since the recorded intensity of the saturating ions appears to reach a constant value, it is important to recognize the onset of saturation by the change in peak shape and position.

When acquiring data in centroid mode, MassLynx detects when saturation occurs and flag the saturated peaks with a question mark. These peaks do not show valid accurate mass data.

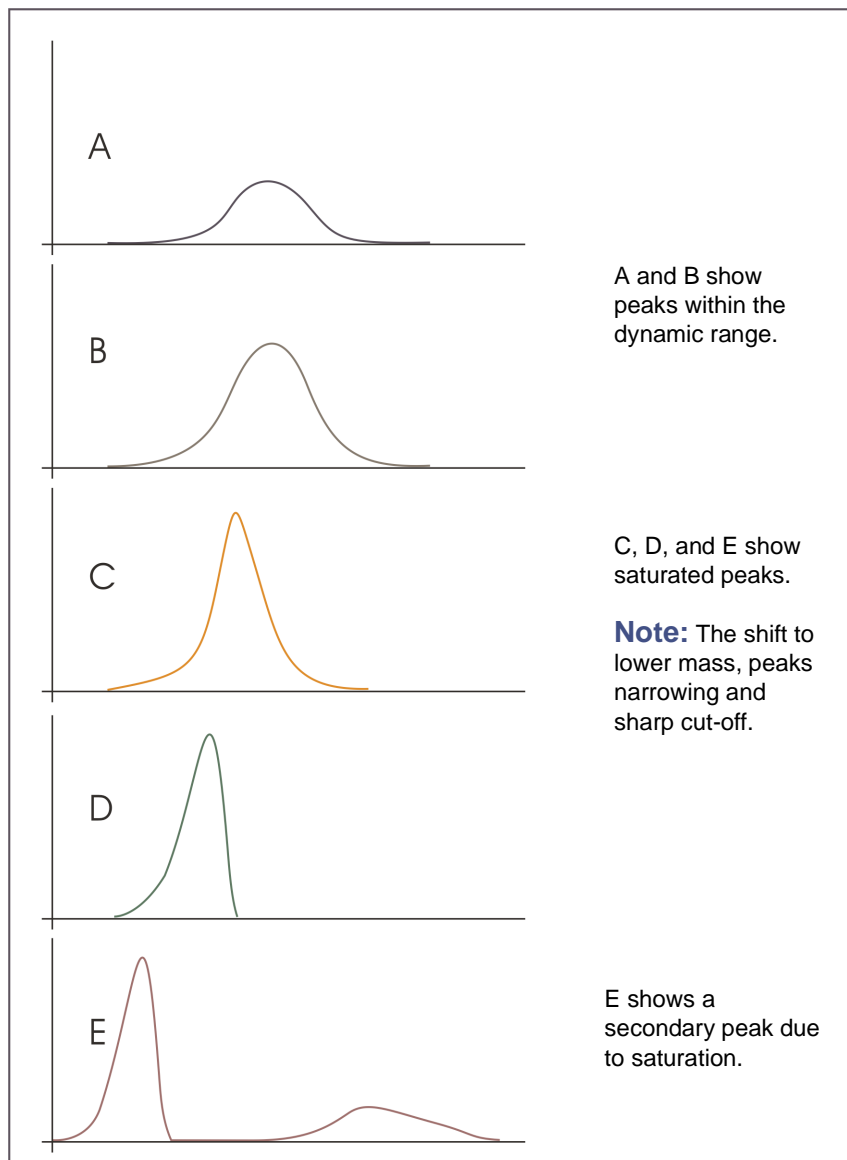
To see the (?) peak flags:

1. In the Spectrum window, click Display > Peak Annotations.
2. In the Spectrum Peak Annotation dialog box, select the Peak Flags check box.
3. Click OK.

Note: The (?) peak flag appears only on single centroid spectra. The (?) peak flag does not appear in combined or continuum spectrum.

Example E in Figure titled “Schematic diagram of peak shape saturation effects:” on page B-52 displays the effect of dead-time saturation. You can adjust the Np Multiplier and Resolution fields in the TDC Settings dialog box to correct the effect of dead time saturation.

Schematic diagram of peak shape saturation effects:



Dead-time saturation on centroid determination shifts the measured mass to a lower value than that calculated from the empirical formula. In the residuals box the calibration, insufficient dead time correction causes a systematic shift of the largest ions in the spectrum (those with the highest degree of dead time saturation) to a position below the line of best fit compared to the smaller ions in the spectrum (those with the least degree of dead time saturation).

If there is too much dead time correction applied, the converse is true. Within this particular reference file that was used to set up Np (metri.ref), the first and second carbon isotopes of the molecular ion at 284.9949 are present. Due to the difference in the intensity of these two ions this region of the calibration curve can be used to set the dead time correction to the optimum value for the system.

See also:

- [Configuring the TDC settings on page B-11.](#)
- [Setting up resolution for dead time correction manually on page B-48.](#)
- [Setting the Np multiplier on page B-50.](#)

C Calibration Reference Information

This appendix provides calibration reference file information.

Contents:

Topic	Page
Overview	C-2
Editing a calibration reference file	C-2
Calibration reference file	C-2

Overview

Calibration reference files and DRE file consist of two columns of numbers separated by any number of spaces or characters. The first column contains the reference peak masses (m/z), and the second column contains the reference peak intensities.

Most samples can be purchased from the Sigma chemical company. To order them, contact Sigma at <http://www.sigma.sial.com>. This site includes a list of worldwide Sigma offices, many with local toll-free numbers.

FC-70 can be purchased from Fluorochem at <http://www.fluorochem.net>.

Editing a calibration reference file

Calibration reference files (*.ref) are stored in the MassLynx/Ref folder.

Calibration reference files are created or edited using any Windows® text editor like Notepad.

To save the reference file after editing, click File > Save to save the file under the current name, or click File > Save As to save as a new reference file with a new name.

Textual information or comments can be stored in the reference file. Lines that are textual information or comments must start with the semicolon (;) character.

Calibration reference file

Heptacos

Use the heptacos.ref file for calibration in EI+.

Heptacos.ref file:

Calculated m/z value	Intensity	Calculated m/z value	Intensity	Calculated m/z value	Intensity
49.99680	0.73	130.99199	36.31	213.99030	0.76
68.99518	100.00	149.99039	1.29	218.98560	38.07
92.99518	0.56	163.99350	0.56	225.99030	0.56

Heptacos.ref file: (Continued)

Calculated m/z value	Intensity	Calculated m/z value	Intensity	Calculated m/z value	Intensity
99.99358	6.31	168.98877	3.24	230.98560	0.47
113.99669	2.34	175.99350	0.91	263.98705	8.38
118.99199	7.80	180.98877	1.31	313.98386	0.32
325.98386	0.16	425.97748	0.85	537.97112	0.13
375.98067	0.38	463.97429	1.02	575.96796	0.32
413.97748	1.64	501.97110	1.90	613.96471	0.64

Heptacosa negative

Use the heptaneg.ref file for calibration with heptacosa in CI-.

Heptaneg.ref file:

Calculated m/z value	Intensity	Calculated m/z value	Intensity	Calculated m/z value	Intensity
168.9888	10	311.9808	10	451.9743	20
244.9887	10	332.9823	20	475.9743	10
263.9871	10	382.9791	15	494.9727	60
280.9824	10	394.9766	10	513.9711	50
282.9855	15	413.9775	20	594.9663	50
301.9839	15	432.9759	10	632.9632	100

Tris (trifluoromethyl) triazine

Use the metri.ref file for calibration in EI+ and CI+ (when the methane reagent gas has been removed from the source).

Metri.ref:

Calculated m/z value	Intensity	Calculated m/z value	Intensity	Calculated m/z value	Intensity
49.9968	10	121.001358	100	284.9949	100
68.99521	100	170.998164	5	285.9984	5

Metri.ref: (Continued)

Calculated m/z value	Intensity	Calculated m/z value	Intensity	Calculated m/z value	Intensity
75.998881	25	189.996568	10		
102.00295	10	265.996448	100		

Perfluorotripropylamine (FC70_EI.ref)

Use the FC70_EI+.ref file for calibration in EI+.

FC70_EI+.ref:

Calculated m/z value	Intensity	Calculated m/z value	Intensity	Calculated m/z value	Intensity
68.9952	100.00	218.9856	38.07	475.9743	0.38
99.9936	6.31	230.9856	0.47	513.9711	1.90
113.9967	2.34	263.9871	8.38	525.9711	0.13
118.9920	7.80	268.9824	100	563.9679	0.32
130.9920	36.31	313.9839	0.32	601.9648	0.64
168.9888	3.24	318.9792	0.32	725.9584	0.64
180.9888	1.31	363.9807	0.16	763.9552	0.64
213.9903	0.76	413.9775	0.38		

Perfluorotripropylamine (FC70_PCI+.ref)

Use the FC70_PCI+.ref file for calibration in CI+.

FC70_PC+.ref:

Calculated m/z value	Intensity	Calculated m/z value	Intensity	Calculated m/z value	Intensity
29.0391	20	213.9903	0.76	413.9775	0.38
68.9952	100.00	218.9856	38.07	475.9743	0.38
99.9936	6.31	230.9856	0.47	513.9711	1.90
113.9967	2.34	263.9871	8.38	525.9711	0.13
118.9920	7.80	268.9824	100	563.9679	0.32

FC70_PC+.ref: (Continued)

Calculated m/z value	Intensity	Calculated m/z value	Intensity	Calculated m/z value	Intensity
130.9920	36.31	313.9839	0.32	601.9648	0.64
168.9888	3.24	318.9792	0.32	725.9584	0.64
180.9888	1.31	363.9807	0.16	763.9552	0.64

Heptacosia PCI

Use the Heptacosia PCI.ref reference file for calibration in CI+.

Heptacosia_PCI.ref:

Calculated m/z value	Intensity	Calculated m/z value	Intensity	Calculated m/z value	Intensity
68.99518	0.5	218.98560	40.07	463.97429	6.02
118.99199	7.80	263.98705	8.38	501.97110	21.90
130.99199	1.2	313.98386	1.32	537.97112	1.13
149.99039	1.29	325.98386	0.16	575.96796	7.32
168.98877	3.24	375.9806	1.38	613.96471	22.64
180.98877	1.31	413.97748	100	651.96160	3.64
213.99030	0.76	425.97748	1.85		

Perfluorotripentylamine (FC70_NCI-.ref)

Use the FC70_NCI-.ref reference file for calibration in CI-.

FC70_NCI-.ref:

Calculated m/z value	Intensity	Calculated m/z value	Intensity	Calculated m/z value	Intensity
199.9872	1	451.9743	4.38	625.9648	1.5
213.9903	1	475.9743	1.38	635.9632	2.5
218.9856	5	501.9711	2.38	644.9632	1.4
230.9856	1	513.9711	11.90	663.9616	2.5

FC70_NCI-.ref: (Continued)

Calculated m/z value	Intensity	Calculated m/z value	Intensity	Calculated m/z value	Intensity
249.9840	5	532.9695	4.50	682.9600	3.5
263.9871	8.38	551.9679	100	694.9600	2.3
268.9824	5	563.9679	2.32	706.9600	3.14
280.9805	5	575.9679	1.32	732.9568	4.24
294.9866	5	582.9663	2.32	744.9568	16.64
313.9839	1.32	594.9663	1.32	782.9536	40.64
351.9807	2.5	601.9648	2.24	820.9504	2.00
413.9775	1.38	613.9630	7.00	832.9504	2.00

DRE calculation files

Perfluorotripentylamine for DRE

Use the FC70_EI+.drf DRE reference file for DRE calculations in EI+.

FC70_EI+.drf:

Calculated m/z value	Intensity	Calculated m/z value	Intensity	Calculated m/z value	Intensity
68.9952	20	601.9648	0.64		
180.9888	1.31				
268.9824	100				
513.9711	1.90				

Heptacosia PCI DRE

Use the Heptacosia PCI DRE reference file for DRE calculation in CI+.

Heptacosia_PCI.drf:

Calculated m/z value	Intensity	Calculated m/z value	Intensity	Calculated m/z value	Intensity
29.0391	0.73	413.97748	1.64	651.96160	0.64
218.98560	38.07	501.97110	1.90		

Metri DRE

Use the metri.drf reference file for DRE calculations in EI+ and CI+.

Metri.drf

Calculated m/z value	Intensity	Calculated m/z value	Intensity	Calculated m/z value	Intensity
68.99521	100	189.996568	100	284.9949	100
121.001358	100	265.996448	100		

HeptacosiaDREPos

Use the HeptacosiaDREPos.drf file for DRE calculations in EI+.

HeptacosiaDREPos.drf

Calculated m/z value	Intensity	Calculated m/z value	Intensity	Calculated m/z value	Intensity
68.99518	100.00	218.98560	38.07	501.9711	0.64
130.99199	36.31	413.97748	1.64		

HeptacosacpfbDRENeg.drf

Use the HeptacosacpfbDRENeg.drf file for DRE calculations in CI-.

HeptacosacpfbDRENeg.drf

Calculated m/z value	Intensity	Calculated m/z value	Intensity	Calculated m/z value	Intensity
201.9609	100.00	513.9711	38.07	632.9632	0.64
451.9743	36.31	594.9663	1.64		

Typical lock mass values

Typical lock mass values

Typical lock mass	Mode	Calculated value
Heptacosac	EI+	218.9856 m/z
Metri	EI+	284.9949
FC70	EI+	268.9824
Metri	CI+	285.0027
Chloropentafluorobenzene	FI+ or FD+	201.9609
Chloropentafluorobenzene	CI-	201.9609

D External Connections

This appendix describes the GCT Premier system's external wiring, which must to be disconnected, then reconnected, if the instrument is moved.

Contents:

Topic	Page
Safety Considerations	D-2
GCT Premier system external wiring	D-3

Safety Considerations



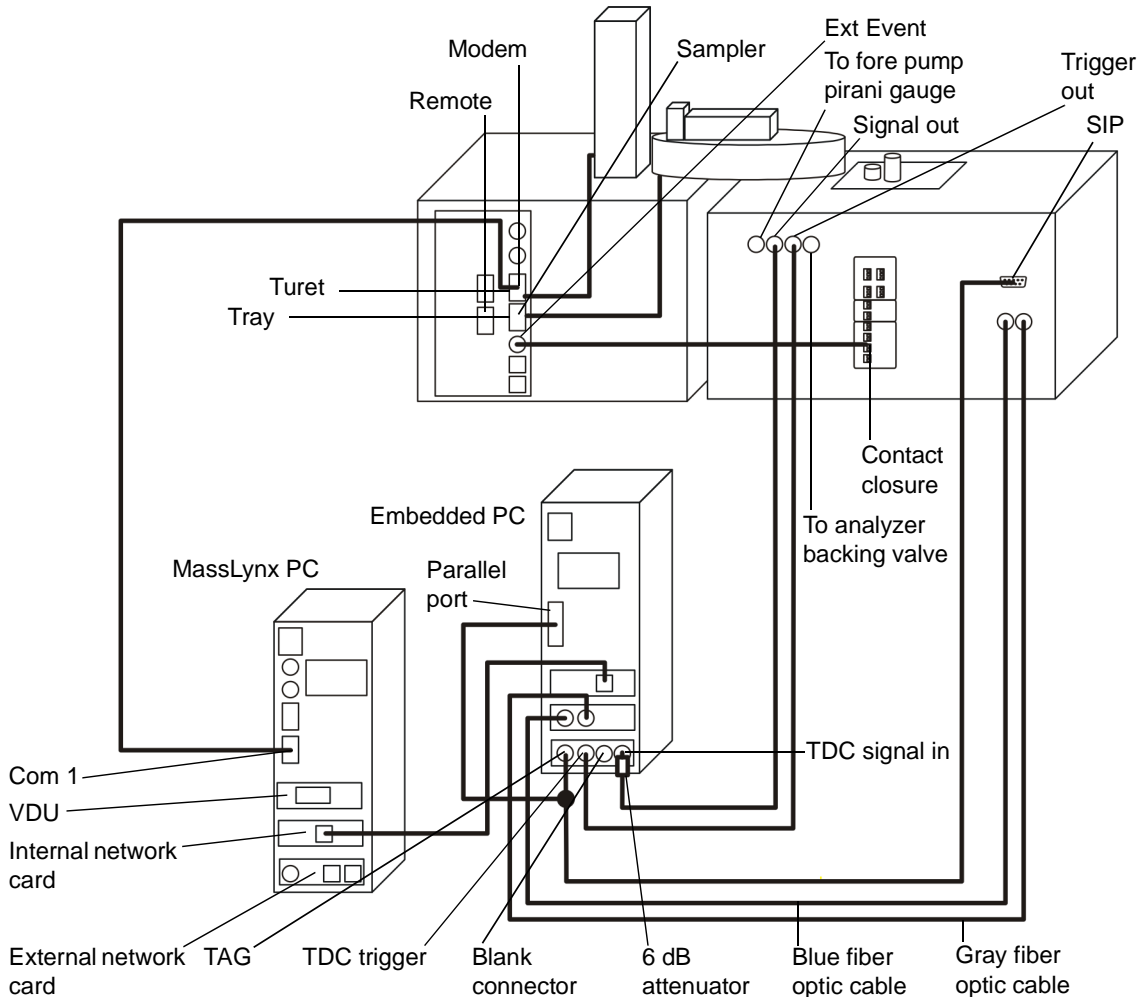
Warning: The GCT Premier is heavy; if you need to move the instrument, contact Waters.

See also: [Contacting Waters on page 11-14.](#)

GCT Premier system external wiring

The electrical connections for a GCT Premier system with a HP6890 GC and autosampler are shown in [Figure titled "GCT Premier system external wiring:"](#) on page D-3.

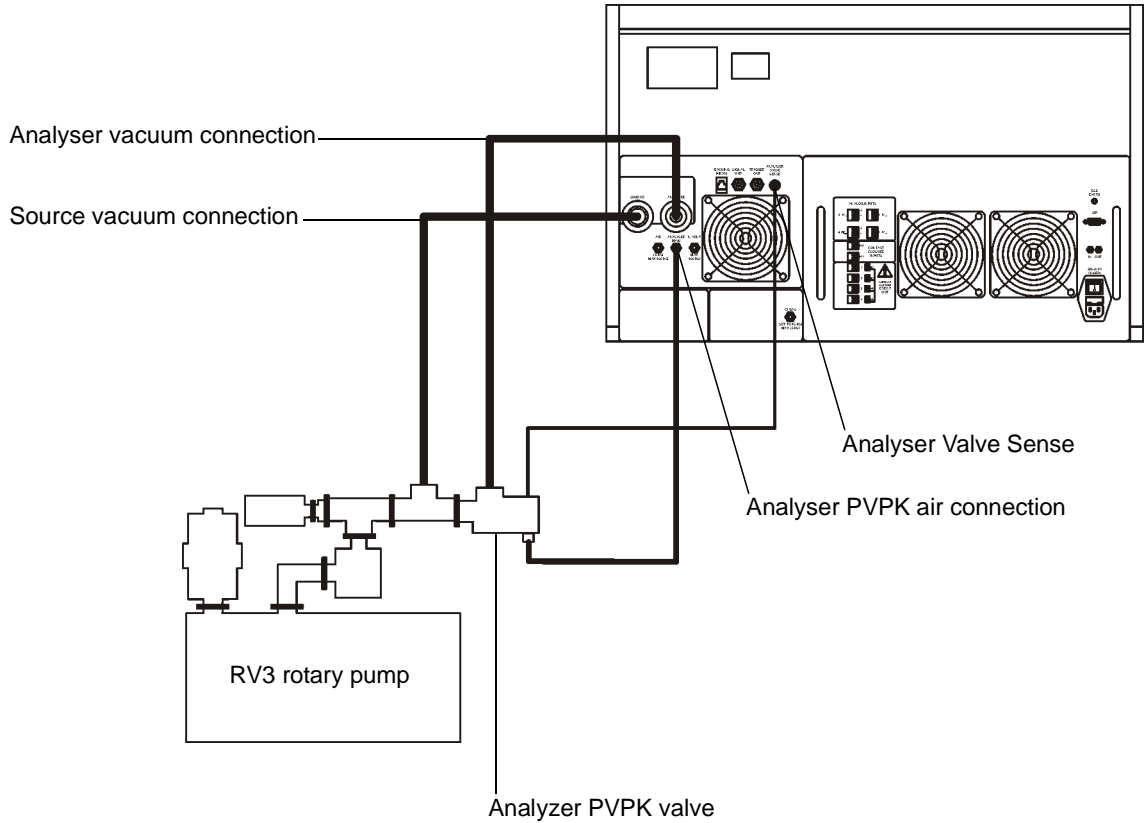
GCT Premier system external wiring:



GCT Premier system vacuum connections

The vacuum connections for a GCT Premier system are shown in [Figure titled "GCT Premier system vacuum connections:"](#) on page D-4.

GCT Premier system vacuum connections:



E

Status Display Vacuum LED

This appendix describes the status display vacuum LEDs for the various instrument states.

Contents:

Topic	Page
Vacuum LED: displays for normal instrument operation:	E-2
Vacuum LED: displays for instrument faults	E-5

Vacuum LED: displays for normal instrument operation

See also: [Table titled “Vacuum LED display:” on page 1-10.](#)

Vacuum LED: displays for normal instrument operation:

State	Status LED	Sub-state
Vented	Steady red.	Vented
Analyser pumpdown	Flashing red and yellow.	Isolation valve closing
		Source backing valve closing
		Analyser backing valve opening
		Analyser turbo run-up
Isolated	Steady yellow.	Isolated
Source pumpdown	Off to green.	Isolation valve closing
		Analyser backing valve opening
		Backing line stabilizing
		Source backing valve opening
		Source turbo run-up
		Analyser backing valve closing

Vacuum LED: displays for normal instrument operation: (Continued)

State	Status LED	Sub-state
Pumped	Steady green.	CI purge: Analyzer and source backing valves closing
		CI purge: Backing line stabilizing
		CI purge: CI gas in and pump-out valves opening
		CI Purge: CI gas in valve closing
		CI Purge: CI pump-out valve closing
		CI Purge: Analyzer and source backing valves opening
		CI Pump: Analyzer and source backing valves closing
		CI Pump: Backing line stabilizing
		CI Pump: CI pump-out valve opening
		CI Pump: CI pump-out valve closing
		CI Pump: Analyzer and source backing valves opening
		Probe Lock Pump: Analyzer and source backing valves closing
		Probe Lock Pump: Backing line stabilizing
		Probe Lock Pump: Probe lock pump-out valve opening
Probe Lock Pump: Probe lock pump-out valve closing and backing pressure stabilizing		

Vacuum LED: displays for normal instrument operation: (Continued)

State	Status LED	Sub-state
Pumped	Steady green.	Probe Lock Pump: Analyzer and source backing valves opening
		Probe Lock Pump: Probe lock pumped LED illuminated
		Pumped
Venting to isolated	Flashing green and yellow.	Isolation valve closing
		Source backing valve closing
		Analyzer backing valve closing
		Source pump run-down
Venting to vented	Flashing red and off.	Isolation valve closing
		Analyzer backing valve closing
		Source backing valve closing
		No pump run-down
		Analyzer pump run-down
		Source pump run-down

Vacuum LED: displays for instrument faults

Any combination of LED indications not described in [Table titled “Vacuum LED: displays for normal instrument operation:” on page E-2](#) can indicate an instrument normal state or fault; contact Waters for advice.

See also: [Table titled “Vacuum LED display:” on page 1-10](#) and [Contacting Waters on page 11-14](#).

Vacuum LED: displays for instrument faults:

State	Status LED	Sub State
Timeout in (analyzer pumpdown)	Flashing yellow and off.	Waiting for vent command.
Timeout in (source pumpdown)	Flashing red and green.	Isolation and source backing valves closing.
		Waiting for isolate or vent command.
Source Pump Trip	Flashing yellow, green, red, and off.	Isolation and source backing valves closing.
		Waiting for isolate or vent command.
Analyzer Pump Trip	Flashing yellow, red, green, and off.	Isolation and source backing valves closing.
		Analyzer pump run-down.
		Waiting for vent command.
Intermediate State for Aborted Pumpdown	Flashing green and yellow.	Waiting for delay.
Time-out in (pumped state)	Flashing green three seconds, red one second.	No pump run-down.
		Analyzer pump run-down.
		Source pump run-down.
		Waiting for vent command.

Vacuum LED: displays for instrument faults: (Continued)

State	Status LED	Sub State
Time-out in (source vent)	Flashing yellow one second, red three seconds.	No pump run-down.
		Analyzer pump run-down.
		Source pump run-down.
		Waiting for vent command.
Time-out in (instrument vent)	Flashing green one second, red three seconds.	No pump run-down.
		Analyzer pump run-down.
		Source pump run-down.
		Waiting for vent command.

Tips: The fault states described in the table above occur when the instrument has attempted a pump or vent which has not been successful. Usually this occurs as a result of one of the following conditions:

- The pneumatic gas supply has failed so the pneumatic valve cannot open and close in the correct sequence. Ensure the pneumatic gas pressure is correctly set to 80-100 psi.
- The Analyzer vent sense cable is disconnected. Ensure the cable is connected.
See also: [Analyzer Valve Sense on page 1-14.](#)
- The rotary pump is switched off. Ensure the switch is set to “on” on the pump and at the supply.

All fault states require a vent command from the Tune window to reset and exit the state.

F

Performance Specifications

This appendix details the performance specifications for the instrument.

Contents:

Topic	Page
Performance specifications	F-2

Performance specifications

Mass range

4000 Dalton.

Resolution

7000 resolution (FWHM) at m/z 614.

Acquisition rate

Up to 20 full spectra (to m/z 800) per second in normal mode.

Up to 10 full spectra (to m/z 800) per second in DRE extended mode.

GC sensitivity

El positive ionization mode

1 pg of hexachlorobenzene (m/z 283.8) will give a peak to peak signal-to-noise ratio (S:N) >10:1 while acquiring full spectra over a mass range up to m/z 800.

CI positive ionization mode

100 pg of benzophenone (m/z 183.1) will give a S:N >150:1 while acquiring full spectra over a mass range up to m/z 800.

CI negative ionization mode

1 pg of octafluoronaphthalene (m/z 272.0) will give a S:N >100:1 while acquiring full spectra over a mass range up to m/z 800.

Mass accuracy

When a suitable peak of known mass is used as an internal reference, the RMS error between the measured and calculated mass is less than 1 mDa for measurements below m/z 200, and is less than 5 ppm for measurements above m/z 200. Peaks are required to have sufficient intensity and be free from interference from other masses.

For example:

In EI positive ionization mode, less than 10 ng of methyl stearate (m/z 298.3) injected onto a capillary column will give, for all peaks above mass 70 and greater than 10% of the base peak intensity, 1.25 mDa RMS error or better, with the 4.0 GHz TDC.

Dynamic range

Enabled by dynamic range enhancement (DRE) and digital dead time correction (DDTC), the dynamic range of the instrument is over four orders of magnitude.

G Theory and Principles of Operation

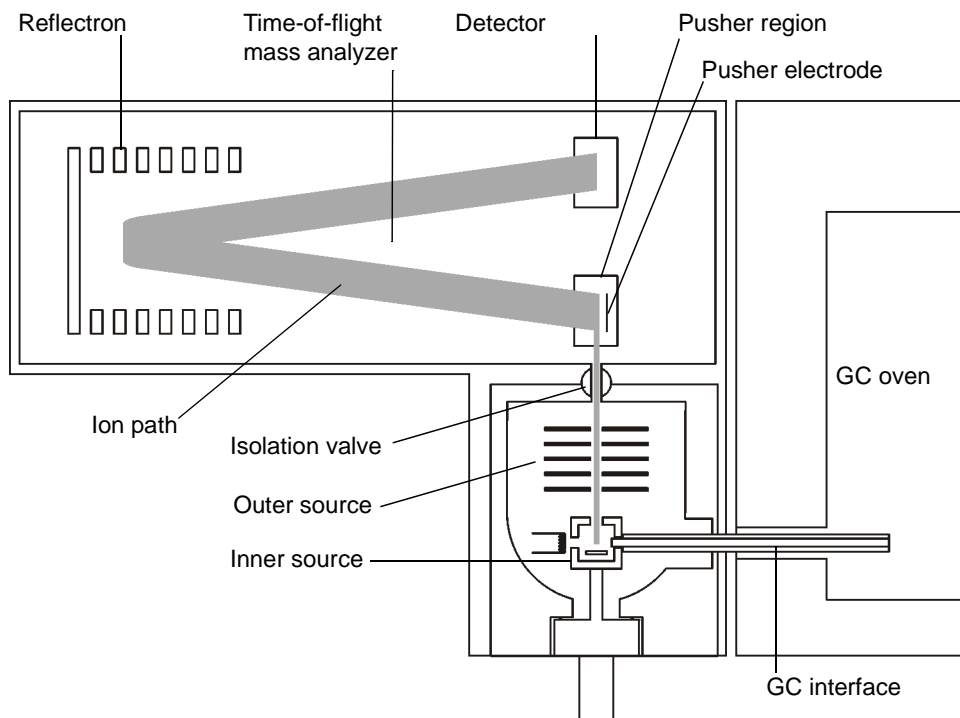
This appendix gives a brief overview of the theory and principles of operation of the instrument.

Contents:

Topic	Page
GCT Premier functionality	G-2
Ionization techniques	G-3
General considerations for tuning	G-6
Low mass cut-off	G-8
DRE mode	G-10
Multiple peak stitching	G-12
Gain drop monitoring	G-13

GCT Premier functionality

GCT Premier mass spectrometer schematic diagram (fitted with GC interface):



Sample is introduced into the inner source, where ions are generated. The ions are accelerated to 40 eV and focused into the pusher region. Voltage pulses applied to the pusher electrode, at repetition frequencies up to 30 kHz, eject sections of the ion beam orthogonally. Each discrete ion packet then passes through a two-stage acceleration region before entering the time-of-flight (TOF) mass analyzer. In the mass analyzer, the reflectron reflects the ions back to the dual microchannel plate (MCP) detector. As the ions travel through the mass analyzer, they are separated in mass according to their flight times: ions with the highest mass-to-charge ratio (m/z) take the longest to arrive at the detector. Ion arrivals are recorded using a time-to-digital converter (TDC) with a sampling rate of 4 GHz, giving high mass resolution and accuracy.

When the pusher electrode is operated at a repetition frequency of 25 kHz, a full spectrum is recorded every 40 μ s. Each spectrum is summed in the Embedded PC (EPC) memory until the completed, histogrammed, spectrum is transferred to the MassLynx PC.

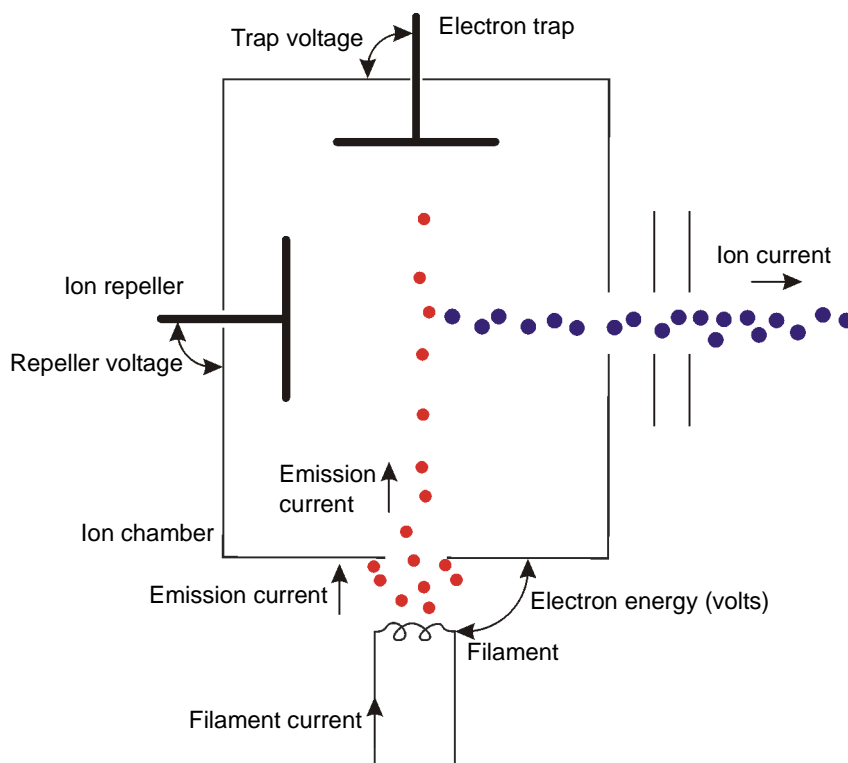
Ionization techniques

Electron Impact ionization

During EI operation, the sample enters the EI inner source via the GC interface, the solids probe, the DCI probe, or the reference inlet reservoir. The outer source is heated to vaporize the sample and the resulting gas molecules are ionized during collision with high-energy electrons, which are released from a heated filament. The electrons are drawn towards the trap in the source. The instrument extracts ions from the inner source ion chamber and introduces them into the analyzer by the ion repeller and the focusing lenses.

See also: [Tuning the instrument for EI on page 3-19.](#)

Electron impact ionization:



Chemical ionization

In contrast to EI (where the vaporized sample molecules are ionized by colliding with high energy electrons from the filament), chemical ionization (CI) uses a relatively high-pressure reagent gas (the CI gas) in the ion source to moderate the collision energy. Ammonia, iso-butane, and methane are commonly used as CI gases.

Electron bombardment ionizes the CI gas molecules within the ion source. The sample molecules are then ionized in reactions with ionized CI gas molecules.

To achieve the relatively high pressures required within the ion source ion chamber, while maintaining an adequate vacuum elsewhere, the chamber is partially gas-tight.

Unlike the EI source, the CI inner source does not include an electron trap, since insufficient electrons are likely to reach an electron trap under CI

conditions. The filament current is regulated using the total emission current and not the trap current as in EI operation.

See also: [Tuning the instrument for positive CI on page 3-25.](#)

Negative chemical ionization

Both positive and negative ions are created in EI and CI sources. Ions of the required polarity are extracted from the source by applying the appropriate polarity potential to the source and focusing lenses.

EI operation is not suited to negative ion analysis because as many organic compounds do not form molecular or quasi-molecular negatively-charged ions (anions) under conventional EI conditions, but instead provide spectra dominated by structurally insignificant low mass ions (such as CN^- , F^- , and Cl^-). Furthermore, the sensitivity for producing negative ions under EI conditions is several orders of magnitude lower than for positive ion production.

However, low energy electrons are readily captured by many organic compounds and produce negative ions without inducing excessive fragmentation. Negative CI operation facilitates this, as the CI ion source has been optimized for generating a large population of electrons with near-thermal (low) energy; the CI gas acts as a moderator for the initially energetic electrons. Negatively-charged CI gas ions can also be generated by ion-molecule reactions.

See also: [Tuning the instrument for negative CI on page 3-30.](#)

Field ionization

When using Field Ionization (FI) sample molecules pass in close proximity to the tips of a mass of carbon needles (dendrite) on an emitter. A pair of extraction rods are held at a high potential which produces a very high electrical field around the tip of the carbon needles. These high fields cause ionization by quantum tunneling of a valence electron

This process is a soft process often producing spectra with little or no fragmentation.

During field ionization, the tuning of the ion source is strongly dependant on the position of the emitter in relation to the extraction rods. The closer the emitter is to the extraction rods, the higher the field strength at the emitter and, therefore, the higher the sensitivity.

See also: [Figure titled “GC-FI column position:” on page 9-20.](#)

The position of the emitter in relation to the center line of the extraction rods is also critical. Small changes to the position can make a large changes in the beam steering voltage being required. Normally the beam steering is set close to zero, and the emitter probe micrometer is used to optimize the signal. Small adjustments to the beam steering can then be made using the beam steering lens.

When the extraction voltage is higher, the field strength is higher, which results in improved sensitivity. Adjusting the extraction voltage causes a change in the source focusing, and the source lenses will need to be reoptimized.

See also: [Tuning in FI mode on page 9-20.](#)

Field desorption

Field desorption (FD) is a technique used primarily for the analysis of non-polar or thermally labile compounds not amenable to other ionization modes. The ionization process is soft producing mainly molecular ions, or fragment ions.

During FD ionization the sample is loaded directly onto the emitter (a 10- μm tungsten wire covered with carbon microneedles). An electric current is passed through the emitter wire to raise the temperature. The sample is ionized on the surface of the emitter. Alternatively, if the sample volatilizes before ionization, field ionization occurs.

See also: [Tuning in FD mode on page 9-27.](#)

General considerations for tuning

In GC, EI and positive CI (CI+) modes of operation intense low mass background ions continuously appear. In EI mode, these ions are predominantly mass spectral peaks from the following samples:

- Helium (which is used as the GC carrier gas).
- Background nitrogen.
- Water (from residual air).

In CI+ mode, reagent gas ions dominate the spectrum. If the entire ion signal from these intense peaks was allowed to reach the detector, then the detector would rapidly degrade, causing gain loss.

In the GCT Premier, the transmission of these ions into the pushout region of the TOF is reduced by restriction slits in the ion source. The primary ion beam exiting the inner source is partially mass-dispersed by a magnetic field. The field deflects low mass ions more than high mass ions. Therefore, the beam does not travel along the centerline of the instrument.

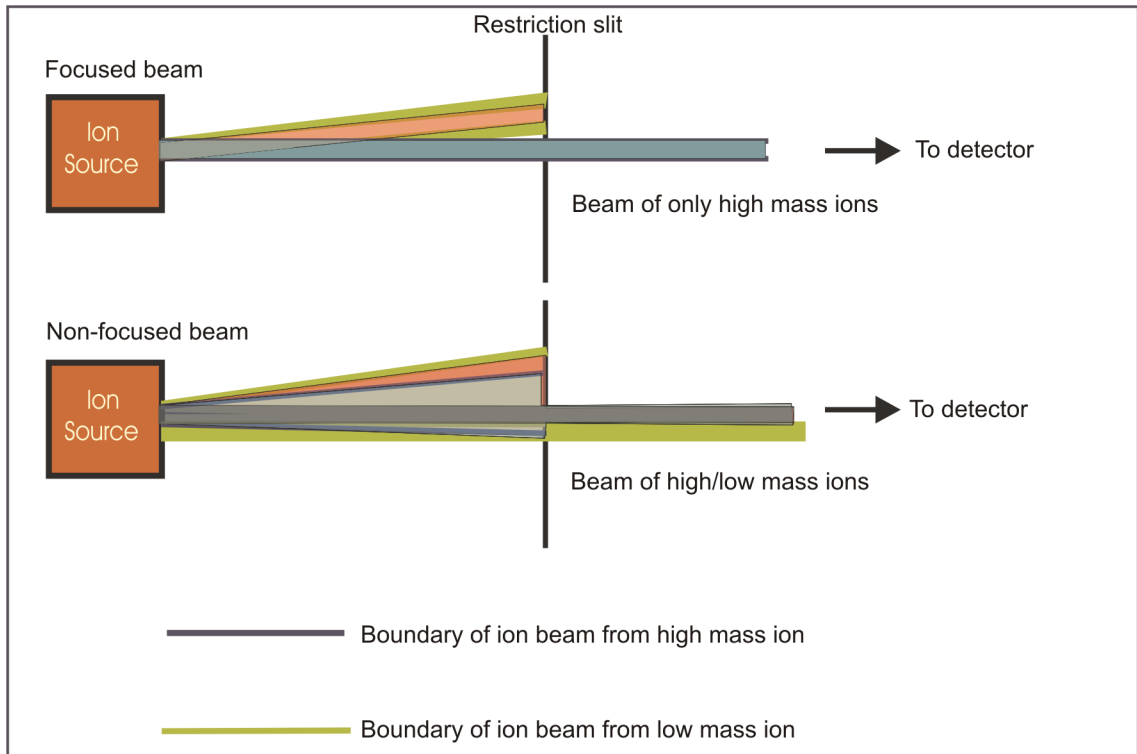
These low mass ions are incident on the restriction slits and so do not enter the analyzer. This introduces some mass discrimination into the spectra, which you can use to reduce the intensity of the low mass ions. In practice the low mass ions are so large that a proportion of the ion beam still enters the analyzer; the intensity of these ions must be monitored and minimized during tuning.

You must consider two effects when tuning in these modes.

- If the ion beam is badly focused in the source, then the beam will appear more homogenous with respect to low-mass and high-mass ions. This is equivalent to a poor mass resolution in the source region. In this case, it will be difficult to significantly reduce the intensity of the low mass ions.
- The beam steering allows the primary ion beam to move across the face of the restriction slit, which adjusts the amount of mass discrimination. When tuning in these modes, the ion signal from a reference compound in the middle of the mass range should be monitored (approximately m/z 300), as well as the ion signal from the most intense low mass background ion.

In MassLynx, you can open two peak displays to monitor these two separate mass regions simultaneously. At the point of best focusing, small changes in lens voltage should have much smaller effect on the higher mass ion and a much greater effect on the low mass ion.

Beam from high and low mass ion:



The largest constant low mass background ion should be maintained below the level of saturation of the TDC.

See also: This saturation effect is described in [Effects of saturation on peak shape on page B-51](#).

Low mass cut-off

Low mass cut-off stops low masses from reaching the detector. It does this by cutting off the beam a set time before the push so that by the time the push occurs the low masses have gone past the pusher and are not analyzed.

Air, water, CI gas and helium peaks can be of significant intensity and cutting them off from the analyzer can significantly increase the lifetime of the detector and reduces the potential for detector saturation. It is recommended therefore that LMC should be used as a matter of routine.

However viewing air peaks can provide a good diagnosis of an air leak in the system. It can therefore sometimes be helpful to turn off LMC and look at the air peaks to diagnose leaks. Typically, the air peak at 28Da should be less than 2000 counts/sec in continuum with the GC fitted and < 200 counts/sec in continuum with a closed insertion lock or the GC blanked off.

Careful tuning of the steering parameter can also reduce the air, water, CI gas peaks or helium peaks.

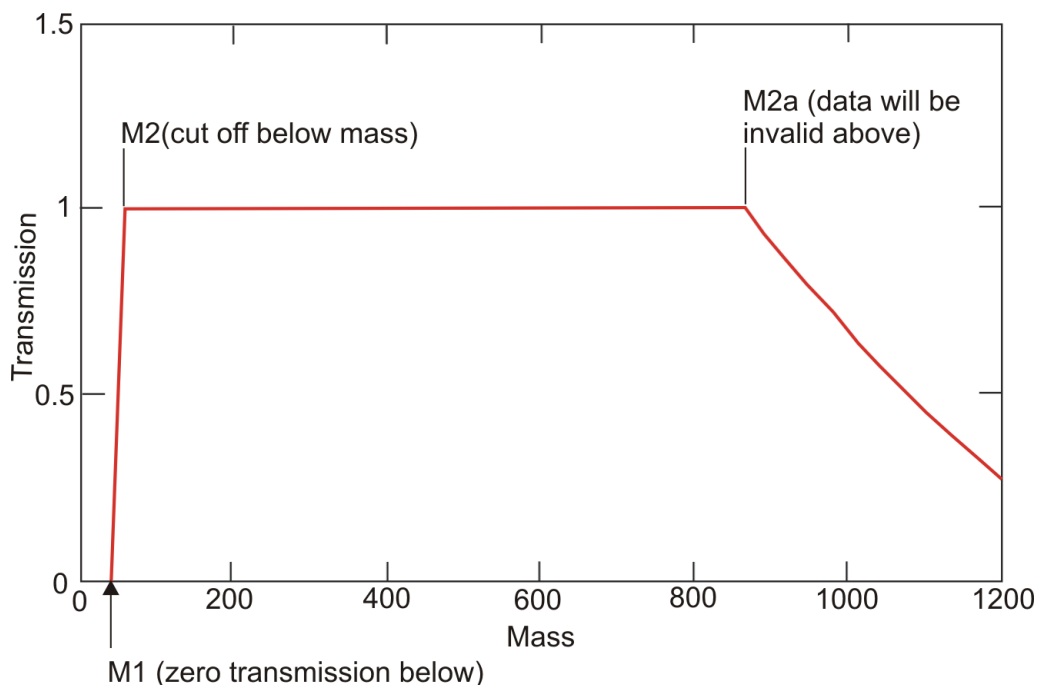
In order to set Low Mass Cut-Off you fill in the mass below which you want to cut off the beam this parameter can be called M2 "cut off below mass". Low mass cut-off gives a gradual cut-off from M1 to M2.

See also: [Figure titled "Low mass cut-off graph:" on page G-10.](#)

When you enter a value for low mass cut-off to start you will get complete cut-off at a mass (M1), which is slightly lower than M2 and is displayed in the TDC settings in the "Zero transmission below" box.

Note: Low mass cut-off also affects high masses. These effects are normally only seen above the mass range of interest for GC analysis. The high masses will be reduced in intensity and the accurate mass will be adversely affected over a certain mass (M2a) shown on the graph. This mass is displayed in the "Data will be invalid above" section of the TDC settings box. This value can be increased by increasing the pusher interval in the Engineer tab on the Tune window.

Low mass cut-off graph:



DRE mode

Dynamic range enhancement (DRE) mode increases the range of the GCT Premier. It allows the analysis of strong samples that produce intense signals. DRE is achieved by means of lenses that focus and steer the ion beam in the z-direction.

In DRE mode, the instrument acquires alternate high and reduced intensity scans and then uses the reduced intensity data to provide the mass and intensity of peaks that saturate in high intensity data stream.

The DRE z-focus and z-steering lenses are controlled via the Tune window DRE tab, by the z-focus and z-lens steering parameters.

The z-focus value is applied to both z lenses, and the z-steer value is added to one lens and subtracted from the other. For example, if z-focus is 40 and z-steer is 10, then one z lens will be at 30 V and the other at 50 V.

See also: [The DRE tab on page B-43.](#)

In a DRE acquisition the instrument acquires data using two functions, one with normal conditions (high intensity) and one with the beam de-focused (reduced intensity). Where the detector is saturated on the high-intensity scan, the reduced intensity scan will not be saturated and will enable accurate mass measurements of the peaks that would otherwise be impossible to measure.

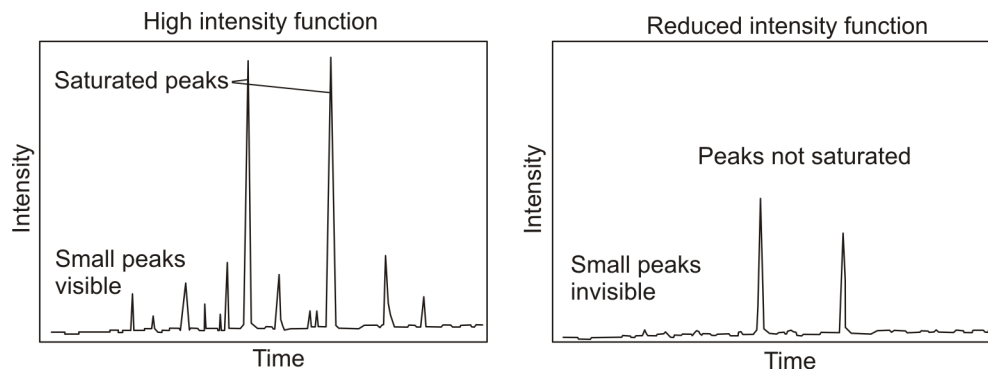
On the Tune window DRE tab, set the z-focus parameter for high intensity and reduced intensity. You can toggle between these settings by selecting the Tune window DRE tab High Sensitivity option.

To acquire data in DRE mode, click Use DRE in the GCT Premier Tune Acquire dialog box, or click Use DRE in the MS Method window, Full Scan Function Editor.

See also: [Figure titled “GCT Premier Tune Acquire dialog box:” on page 4-3](#) and the [Figure titled “Full Scan Function Editor:” on page 4-12](#).

When acquiring in DRE, the instrument acquires one high-intensity scan followed by a reduced intensity (de-focused) scan.

High intensity and reduced intensity chromatogram:



The large peaks from the high intensity chromatogram are saturated (therefore mass accuracy will be poor). However, the data are acceptable in the reduced intensity (de-focused) chromatogram. MassLynx checks each high intensity spectra for peak flags. If there are none, then it just uses the high intensity data and discards the reduced intensity data. If there are any flagged peaks, it looks in the reduced intensity data, and finds the de-focused or reduced intensity peak in the two reduced intensity scans on either side. It averages their mass and intensity, magnifies them by the magnification factor

and stitches the values into the spectrum to produce a better representation of the peak with valid accurate mass.

Rule: You must configure the DRE Magnification Factors before acquiring data in DRE mode. These factors must also be reconfigured if you change any of the tuning parameters or ion mode.

See also: [Setting up DRE mode on page 4-15.](#)

Tip: When you acquire data in the DRE mode, Waters recommends that you scan at a rate of at least 5 spectra per second. This gives better peak shapes and more accurate intensity representation.

Multiple peak stitching

When a very intense ion signal strikes the detector it can affect the neighboring ion signal from the isotope ions. This is because the baseline drops leading the to isotope ions being recorded at lower intensities than the real value.

When using Multiple Peak Stitching the DRE software will check the signal of the ion in the high intensity data. If it exceeds a level where it will affect its neighboring isotopes then the isotope peak is also stitched in from the weak data in addition to the strong main peak. In the weak data the baseline is not affected and therefore the isotope ratio will be correct.

When stitching in the weak intensity data the software will look in a window defined by the parameters “Pre Window” – for how many Da before the strong peak and “Post Window” - for how many Da after.

If you find that the isotope ratios are incorrect for very intense molecules in DRE experiments then the Multiple Peak Stitching option should be used. For most organic molecules the default values of 0 Da for Pre Window and 2 Da for Post Window are suitable. However with some molecules for example a large chlorinated sample it may be sensible to increase these numbers to catch all the ion envelope.

However using Multiple Peak Stitching will stitch in all the low intensity data around the mass of interest. This means that any small co-eluting peak in this mass range could be missed.

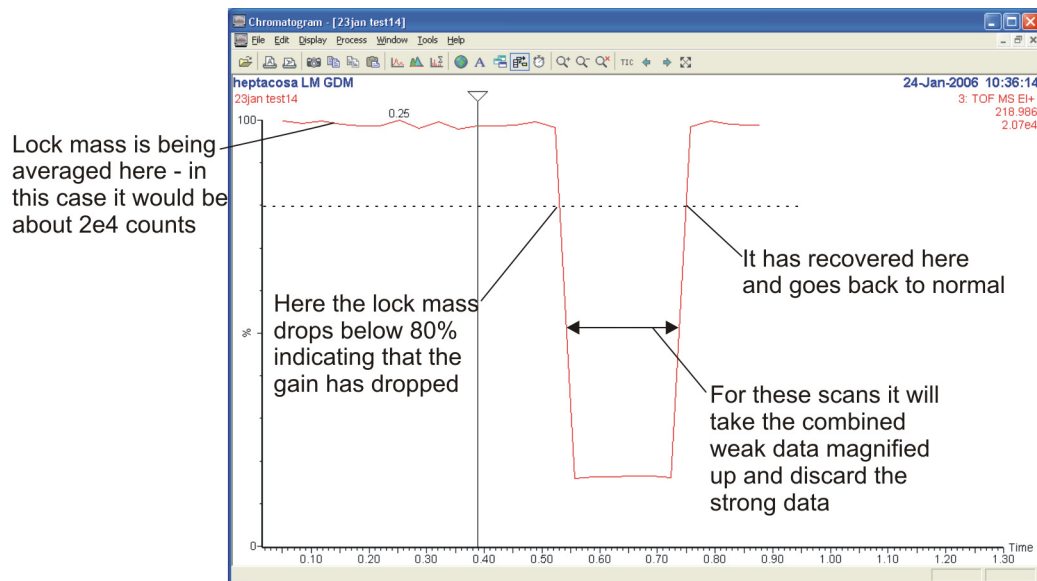
Gain drop monitoring

When the detector on the GCT Premier is hit with a very large signal it uses up the available electrons with the result that the detector gain drops until the electrons have been replenished. The effect of this gain drop is that all the peaks in a spectrum are reduced in intensity, and may not be seen at all in an extreme case. Furthermore the incorrect signal intensity means that the DDTC software does not work correctly and so accurate mass measurements will be compromised.

The Gain Drop Monitoring software tries to prevent this from happening by monitoring an ion signal that should be constant – in this case the constant signal from the reference mass used as a lock mass. If the gain of the system drops below 80% the lock mass will apparently drop in intensity then the software will stitch in the complete spectrum from the low intensity data, which the large signal strike has not affected. This retains the accurate mass measurement. This process is illustrated in the figure below.

See also: [Gain Drop Monitoring on page B-44](#).

Gain drop monitoring:



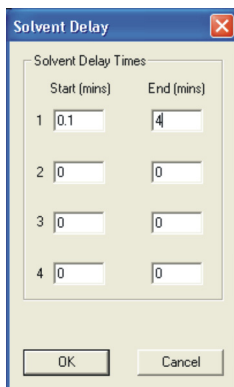
Notes:

- Gain Drop Monitoring only works in DRE mode.
- You must be using a lock mass to use GDM – this is set up in the TDC settings and can be checked because the lock mass will be flagged in the spectrum as a blue peak with a star on top of it.
- You must be using a calibration for the lock mass function to work.
- Ion statistics from the stitched weak data will be worse than from the strong. This means a very weak peak that occurs when the gain has dropped may not be measured at all.

In order to get an average of the lock mass value the system averages the first 10 scans. In a normal GC run the lock mass intensity can be affected by the solvent front so errors can occur when trying to use the data when a solvent delay is used.

Recommendation: Set the solvent delay in the MS method editor to run from 0.1 minutes to 4 minutes as shown in [Figure titled “Solvent Delay dialog box:” on page G-14.](#)

Solvent Delay dialog box:



Tip: The 4 minute duration can be adjusted to your normal solvent delay for a particular analysis. This allows the system to acquire constant lock mass data in the first 0.1 minutes before the solvent front elutes and still switches off the filament when the solvent front causes the large pressure rise in the source chamber.

Note: Gain drop monitoring successfully produces accurate mass results if the gain of the detector is reduced by a large signal. Nevertheless it is useful to verify the lock mass signal in the data. If a peak elutes from a GC peak with a

mass within approximately 15 mDa of the lock mass, it can interfere with the lock mass and thus shift all the peaks up or down the mass scale. This will produce mass errors in all the peaks in the spectrum where the lock mass peak is interfered with.

To verify this, the user should make a RIC (Reconstructed ion chromatogram) of the lock mass peak. If the lock mass peak intensity apparently increases when a GC peak elutes, then it has probably been interfered with, and you should conclude that the accurate mass measurements are invalid for the interfered with in the chromatogram. To avoid this problem you must repeat the acquisition, perhaps using a different compound for the lock mass.

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