

# **TSQ 8000**

# **User Guide**

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# Declaration

Manufacturer: Thermo Fisher Scientific

Thermo Fisher Scientific is the manufacturer of the instrument described in this manual and, as such, is responsible for the instrument safety, reliability and performance only if:

- installation,
- recalibration, and
- changes and repairs

have been carried out by authorized personnel and if:

- the local installation complies with local law regulations,
- the instrument is used according to the instructions provided, and
- if its operation is only entrusted to qualified trained personnel.

Thermo Fisher Scientific is not liable for any damages derived from the non-compliance with the aforementioned recommendations.

# **Regulatory Compliance**

Thermo Fisher Scientific performs complete testing and evaluation of its products to ensure full compliance with applicable domestic and international regulations. When the system is delivered to you, it meets all pertinent electromagnetic compatibility (EMC) and safety standards as described in the next section or sections by product name.

Changes that you make to your system may void compliance with one or more of these EMC and safety standards. Changes to your system include replacing a part or adding components, options, or peripherals not specifically authorized and qualified by Thermo Fisher Scientific. To ensure continued compliance with EMC and safety standards, replacement parts and additional components, options, and peripherals must be ordered from Thermo Fisher Scientific or one of its authorized representatives.

# **EMC Directive 89/336/EEC**

EMC compliance has been evaluated by Professional Testing.

- ITQ, and Ion Trap Series standards: EMC EN 61326-1:2006. Safety IEC 61010-1:2001, IEC 61010-2-081:2001
- Direct Probe Controller (DPC) standards: EMC EN 61326-1:2006. Safety IEC 61010-1:2001, IEC 61010-2-081:2001
- ISQ standards: EMC EN 61326-1:2006. Safety IEC 61010-1:2001, IEC 61010-2-081:2001
- TSQ 8000 standards: EMC EN 61326-1:2006. Safety IEC 61010-1:2001



# Low Voltage Safety Compliance

This device complies with Low Voltage Directive 2006/95/EC and harmonized standard EN 61010-1:2001.

# **FCC Compliance Statement**

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



**CAUTION** Read and understand the various precautionary notes, signs, and symbols contained inside this manual pertaining to the safe use and operation of this product before using the device.

# Notice on Lifting and Handling of Thermo Scientific Instruments

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

# Notice on the Proper Use of Thermo Scientific Instruments

In compliance with international regulations: Use of this instrument in a manner not specified by Thermo Fisher Scientific could impair any protection provided by the instrument.

# Notice on the Susceptibility to Electromagnetic Transmissions

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



For manufacturing location, see the label on the instrument.

# **WEEE Compliance**

This product is required to comply with the European Union's Waste Electrical & Electronic Equipment (WEEE) Directive 2002/96/EC. It is marked with the following symbol:



Thermo Fisher Scientific has contracted with one or more recycling or disposal companies in each European Union (EU) Member State, and these companies should dispose of or recycle this product. See www.thermoscientific.com/ rohsweee for further information on Thermo Fisher Scientific's compliance with these Directives and the recyclers in your country.

## WEEE Konformität

Dieses Produkt muss die EU Waste Electrical & Electronic Equipment (WEEE) Richtlinie 2002/96/EC erfüllen. Das Produkt ist durch folgendes Symbol gekennzeichnet:



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# **Conformité DEEE**

Ce produit doit être conforme à la directive européenne (2002/96/EC) des Déchets d'Equipements Electriques et Electroniques (DEEE). Il est marqué par le symbole suivant:



Thermo Fisher Scientific s'est associé avec une ou plusieurs compagnies de recyclage dans chaque état membre de l'union européenne et ce produit devrait être collecté ou recyclé par celles-ci. Davantage d'informations sur la conformité de Thermo Fisher Scientific à ces directives, les recycleurs dans votre pays et les informations sur les produits Thermo Fisher Scientific qui peuvent aider la détection des substances sujettes à la directive RoHS sont disponibles sur www.thermoscientific.com/rohsweee.

# **Preface**

This guide contains detailed information about how to use the Thermo Scientific TSQ 8000<sup>™</sup> triple-quadrupole GC/MS system. The TSQ 8000 system provides the selectivity and sensitivity of a triple-quadrupole GC/MS while also functioning as a high-performance single quadrupole instrument. The system is designed to stay cleaner longer to maximize your instrument's uptime and improve your lab's productivity. The need to break vacuum, cool off your system, and spend hours cleaning, reassembling, and restoring the system is greatly reduced. The extractable source incorporates a cartridge that contains the repeller, source lenses, and RF lens, all of which can be removed from the instrument without breaking vacuum. What once took hours, or even an entire day, is now accomplished in minutes.

In addition, the TSQ 8000 system includes innovative Thermo Scientific software that will help users new to triple-quadrupole GC/MS/MS systems develop selected reaction monitoring (SRM) methods.

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### **About Your System**

Thermo Scientific systems provide the highest caliber gas chromatography/mass spectrometry (GC/MS) instrumentation available on today's market.

GC/MS represents a combination of two powerful analytical techniques: GC, which acts as a separation technique, and MS, which acts as a detection technique. Complex mixtures of individual compounds can be injected into the GC, either manually or by an autosampler and then separated for presentation to the MS. The MS will generate a mass spectrum of the GC eluate and its components. The mass spectrum can then be used for qualitative identification as well as accurate and precise quantification of the individual compounds present in the sample.

A triple-quadrupole GC/MS/MS system provides the extra selectivity required for trace analysis of compounds in complex matrices.



**WARNING** Thermo Scientific systems operate safely and reliably under carefully controlled environmental conditions. If the equipment is used in a manner not specified by the manufacturer, the protections provided by the equipment might be impaired. If you maintain a system outside the specifications listed in this guide, failures of many types, including personal injury or death, might occur. The repair of instrument failures caused by operation in a manner not specified by the manufacturer is specifically excluded from the standard warranty and service contract coverage.

## **Related Documentation**

The TSQ 8000 system includes Help and these manuals as PDF files:

- TSQ 8000 Preinstallation Guide, PN 1R120505-0001
- TSQ 8000 User Guide, PN 1R120505-0002
- TSQ 8000 Hardware Manual, PN 1R120505-0003
- TSQ 8000 Spare Parts Guide, PN 1R120505-0004
- TSQ 8000 Auto SRM User Guide, PN 1R120505-0005
- ISQ and TSQ 8000 Direct Probe System User Guide, PN 1R120505-0006

#### ✤ To view product manuals

Open the desktop folder Manuals.

#### To open Help

- From the TSQ 8000 window, choose Help > TSQ 8000 Help.
- If available for a specific window or dialog box, click **Help** or press the F1 key for information about setting parameters.

For more information, visit www.thermoscientific.com.

# **System Requirements**

System	Requirements
Hardware	• 2.4 GHz processor with 4 GB RAM
	DVD/CD-ROM drive
	• Resolution display 1280 × 1024 (XGA)
	• 250 GB hard drive
	• NTFS format
Software	<ul> <li>Microsoft<sup>™</sup> Windows<sup>™</sup> 7 SP1 Professional Operating System (32-bit or 64-bit)</li> </ul>
	• Thermo Foundation 3.0 SP2 (Thermo Scientific software)
	• Xcalibur™ 3.0
	<ul> <li>TraceFinder<sup>™</sup> 3.1 or later<sup>a</sup></li> </ul>

Your TSQ 8000 data system must meet these minimum requirements.

<sup>a</sup> Your system will function without TraceFinder, but earlier versions of TraceFinder will not work with the Thermo Scientific software required for this instrument.

## **Safety and Special Notices**

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

### **Special Notices**

Special notices include the following:

**IMPORTANT** Highlights information necessary to prevent damage to software, loss of data, or invalid test results; or might contain information that is critical for optimal performance of the system.

**Note** Highlights information of general interest.

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

### **Safety Symbols and Signal Words**

All safety symbols are followed by **WARNING** or **CAUTION**, which indicates the degree of risk for personal injury, instrument damage, or both. Cautions and warnings are following by a descriptor. A **WARNING** is intended to prevent improper actions that *could* cause personal injury. A **CAUTION** is intended to prevent improper actions that *might* cause personal injury or instrument damage. You can find the following safety symbols on your instrument or in this guide.

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Symbol	Descriptor
	<b>BIOHAZARD:</b> Indicates that a biohazard <i>will</i> , <i>could</i> , or <i>might</i> occur.
	<b>BURN HAZARD:</b> Alerts you to the presence of a hot surface that <i>could</i> or <i>might</i> cause burn injuries.
	<b>ELECTRICAL SHOCK HAZARD:</b> Indicates that an electrical shock <i>could</i> or <i>might</i> occur.
	<b>FIRE HAZARD:</b> Indicates a risk of fire or flammability <i>could</i> or <i>might</i> occur.
FLAMMABLE GAS 2	<b>FLAMMABLE GAS HAZARD:</b> Alerts you to gases that are compressed, liquefied or dissolved under pressure and can ignite on contact with an ignition source. This symbol indicates this risk <i>could</i> or <i>might</i> cause physical injury.
	<b>GLOVES REQUIRED:</b> Indicates that you must wear gloves when performing a task or physical injury <i>could</i> or <i>might</i> occur.
	<b>HAND AND CHEMICAL HAZARD</b> : Indicates that chemical damage or physical injury <i>could</i> or <i>might</i> occur.
	<b>INSTRUMENT DAMAGE:</b> Indicates that damage to the instrument or component <i>might</i> occur. This damage might not be covered under the standard warranty.
<b>\$</b>	<b>LIFTING HAZARD:</b> Indicates that a physical injury <i>could</i> or <i>might</i> occur if two or more people do not lift an object.

Symbol	Descriptor
	<b>MATERIAL AND EYE HAZARD:</b> Indicates that eye damage <i>could</i> or <i>might</i> occur.
	<b>RADIOACTIVE HAZARD:</b> Indicates that exposure to radioactive material <i>could</i> or <i>might</i> occur.
	<b>READ MANUAL:</b> Alerts you to carefully read your instrument's documentation to ensure your safety and the instrument's operational ability. Failing to carefully read the documentation <i>could</i> or <i>might</i> put you at risk for a physical injury.
	<b>TOXIC SUBSTANCES HAZARD:</b> Indicates that exposure to a toxic substance could occur and that exposure <i>could</i> or <i>might</i> cause personal injury or death.
	For the prevention of personal injury, this general warning symbol precedes the <b>WARNING</b> safety alert word and meets the ISO 3864-2 standard. In the vocabulary of ANSI Z535 signs, this symbol indicates a possible personal injury hazard exists if the instrument is improperly used or if unsafe actions occur. This symbol and another appropriate safety symbol alerts you to an

imminent or potential hazard that *could cause personal injury*.

## **Hydrogen Safety Precautions**

Hydrogen is a colorless, odorless, highly flammable gas with the molecular formula  $H_2$  and an atomic weight of 1.00794, making it the lightest element. Hydrogen gas presents a hazard as it is combustible over a wide range of concentrations: at ambient temperature and pressure, this ranges from about 4% to 74.2% by volume.

Hydrogen has a flash point of - 423 °F (- 253 °C) and an auto-ignition temperature of 1,040 °F (560 °C). It has a very low ignition energy and the highest burning velocity of any gas. If hydrogen is allowed to expand rapidly from high pressure, it can self-ignite. Hydrogen burns with a flame that can be invisible in bright light.



**WARNING FIRE HAZARD:** The use of hydrogen as a carrier gas is dangerous. Hydrogen is potentially explosive and must be used with extreme care. Any use of hydrogen gas must be reviewed by appropriate health and safety staff and all installations of hydrogen systems must be performed to applicable codes and standards. Thermo Fisher Scientific assumes no liability for the improper use of hydrogen as a carrier gas.

Before you begin using hydrogen, you should conduct a risk assessment based on the quantity of hydrogen to be used and the conditions of your laboratory. You should ask yourself:

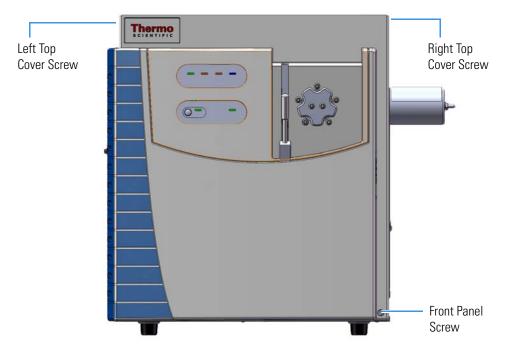
"What hydrogen hazards associated with this project are most likely to occur?"

"What hydrogen hazards associated with this project have the potential to result in the worst consequences?"

- Try to reduce or eliminate the higher risks by using the proper ventilation to remove hydrogen gas before an ignitable concentration can accumulate. You should also consider purging the hydrogen to further reduce hazards and ensure anyone who will be working with hydrogen has basic hydrogen safety training.
- As with laboratory safety in general, be sure to wear safety glasses, laboratory coats, gloves, etc. Typically there are no specific requirements for gaseous hydrogen, other than eye protection when working with a compressed gas. If working with liquid (cryogenic) hydrogen, insulated gloves and protective shoes should be worn in addition to eye protection.
- You should post "No Smoking" and "No Open Flames" signs to identify hydrogen sources and cylinders. Maintain, inspect and leak-test all hydrogen sources regularly.
- All hydrogen shutoff valves should be clearly marked and permanent hydrogen piping should be labeled as such at the supply or discharge point and at regular intervals along its length. Where hydrogen gas piping passes through a wall, the piping should be labeled on both sides of the wall.
- There should also be contingency plans in place should an incident occur.
- The site emergency response team, as well as the local fire department, should know the location of all hydrogen storage tanks.

### Using Hydrogen with TSQ 8000

To use hydrogen with the TSQ 8000, you must always shut off the GC carrier gas before venting or turning off the TSQ 8000. There are three hydrogen safety screws on the TSQ 8000 that **must** be in place. These are attached to your instrument at the factory.



#### Figure 1. Hydrogen Safety Screws on the TSQ 8000

Before powering on the TSQ 8000 system, ensure that:

- All the covers and panels of the TSQ 8000 system are firmly attached.
- The vent valve is tightly closed if you vented the system.
- All fittings, ferrules, and o-rings are sealed.

#### **Hydrogen Connection Guidelines**

Use the following guidelines to safely connect hydrogen to your system:

• **Piping**—Hydrogen must be delivered to equipment using appropriate piping and be done in such a way as to pose essentially no hazard to end-users. Piping systems for the delivery of hydrogen should be designed and installed by a person qualified by specific training and experience with hydrogen piping systems.

Stainless steel is usually recommended because it is a safe, cost-effective material. Piping of *black iron* or copper must not be used, as the pipe can become brittle with age. Elastomeric/plastic tubing of various plastics and polymers should not be used, unless the tubing is approved for use with hydrogen. If elastomeric/plastic tubing is used for hydrogen gas delivery, the tubing should be tested for hydrogen permeability to minimize leakage.

The hydrogen piping system must be flexible enough to endure routine thermal expansion and contraction. The system should also include considerations for the most severe condition of temperature and pressure expected during service. Piping and supports must be able to withstand static loading introduced by such things as ice and snow; and dynamic loading from high wind and earthquake.

Caution should be used if burying hydrogen piping. Proper controls should be used to protect against damage and corrosion, and also to prevent Hydrogen from entering a building if there is any leakage.

• **Fittings**—All fittings must be of the proper type approved or designed for use with hydrogen gas. Use as few fittings as possible to minimize the potential for leaks. After installation, ensure that leak testing is carried out prior to system use, and on a regular basis.

There must be no PTFE tape or other things like *plumber's putty* used to enhance a seal, as this actually is a detriment to a good seal. Ideally the best installation would use stainless steel tubing with appropriate gas-tight fittings.

Welding is usually preferred for joints in hydrogen piping systems since welding provides a better connection and reduces the potential for leaks compared to mechanical fittings. Soft solder joints are not permitted for hydrogen systems (due to the low melting point of soft solder and its potential for brittle failure at cryogenic temperatures). Brazed joints are permitted, but such joints should be protected against the possibility of external fire.

Tubing connections should be clamped to barbed or press-fit type connections. Hose clamps or *jubilee clamps* must not be used.

• **Valves**—All valves must be suitable for hydrogen service and for the specific operating conditions. Valves, including regulators, must not be used for hydrogen, unless they are designed and identified for such a use. Ball valves are often chosen because of their superior leak tightness through the valve seat. Pneumatic operators are usually chosen for remotely operated valves so that potential ignition sources (electricity) are remote from the valve.

Manual shutoff valves should be provided near each point of use, within immediate reach. If a hydrogen cylinder or hydrogen generation system is located within immediate reach, a separate point-of-use shutoff valve is usually not necessary.

Line regulators that have their source away from the point of use should have a manual shutoff valve near the point of use.

An emergency gas shutoff device in an accessible location outside the use area should be provided in addition to the manual point-of-use valve in each educational and instructional laboratory space that has a piped gas supply system.

If necessary, the piping system should have uninterruptible pressure relief. The pressure relief system should be designed to provide a discharge rate sufficient to avoid further pressure increase and should vent to a safe location outside or to a ventilation system exhaust.

### **Purchasing Hydrogen**

Use the following guidelines when purchasing hydrogen:

• **Hydrogen Generator**—Because it minimizes the amount of hydrogen present and reduces the degree of hazard, a hydrogen generator (also called an electrolyzer) is the safest way to purchase hydrogen in the quantity used in GC/MS.

However, to minimize the degree of hazard, the hydrogen generator must only be operated in a non-explosive environment because hydrogen buildup can be ignitable. This means that your ventilation system for the room or lab hood must maintain an air exchange rate that is at least two orders of magnitude greater than the maximum hydrogen production rate of the hydrogen generator. Be sure to follow the manufacturers' directions about proper use and maintenance of the regulator.

To prevent the possibility of releasing hydrogen, the hydrogen generator should be set to shut down if:

- There is a loss of flow to the ventilation system
- A hydrogen detector alarms at 25% of the lower flammable limit of hydrogen in air.

The oxygen exhausted by the electrolyzer should be vented to the outside as well.

• **Hydrogen Cylinder**—Hydrogen can be delivered in standard laboratory gas bottles or cylinders. These cylinders have a limited amount of hydrogen in them and are a safe way to transport and store hydrogen. However, compressed hydrogen gas cylinders, like all compressed gas cylinders, must be secured in an upright position, ideally with a non-combustible chain or cable. If the cylinder falls over, the valve can be knocked off and the pressurized cylinder can take off like a rocket, which leads to the release of hydrogen and possibly an explosion, severe injury, or death. Never crack a hydrogen cylinder valve to remove dust or dirt from fittings prior to attaching a regulator, as there is a risk of self-ignition.

### **Properly Storing Hydrogen**

Storing and handling compressed hydrogen gas and cryogenic liquid hydrogen present potential health and safety hazards. Using proper storage and handling techniques is essential to maintaining a safe work environment.

Use the following guidelines when storing hydrogen:

• Store spare hydrogen gas cylinders outside and away from doors, windows, building air intake vents, structures, and vehicle routes. This precaution applies when the hydrogen is or is not in use. Indoor storage of spare hydrogen cylinders has special requirements, which is beyond the scope of this document. Documentation for each vessel should include a description of the vessel, a list of available drawings or other documents, the most recent inspection results, and the responsible person's name.

- Prevent spare cylinders from toppling by wrapping them with chains. The chains should also be protected against corrosion and excessive heat.
- Separate spare hydrogen cylinders from oxidizing gases (such as oxygen) with a 5 ft (1.5 m) tall fire barrier with a half-hour fire rating or place the cylinders at least 20 ft (6 m) apart.
- When moving hydrogen cylinders:
  - Remove the regulator and replace the cylinder valve cap before moving.
  - Move cylinders on cylinder carts or with other appropriate transport devices.
  - Never roll or drop a cylinder and never lift a cylinder by its protective cap.
- Bulk hydrogen systems include either gaseous or liquid hydrogen in fixed installations; in some gas systems a semi-permanent trailer (tube trailer) can be used. Storage vessels for compressed hydrogen gas or liquid hydrogen should be designed, constructed, tested, and maintained in accordance with applicable codes and standards. Bulk hydrogen systems represent a level of complexity again which is beyond the scope of this document; however some general guidelines are provided.
- The bulk hydrogen storage system should not be located beneath electric power lines, close to other flammable gases/liquids, or close to public areas. It should be readily accessible to authorized personnel and delivery equipment, but protected from physical damage or tampering.
- As liquid hydrogen systems also have a cryogenic hazard, additional safety considerations for the use of cryogenic liquids might be necessary.

### Hydrogen Safety Codes, Standards and References

The following list of safety codes, standards and references is in no way an exhaustive list. In fact, there might be federal, state or local codes that apply to your specific location. Check with all appropriate agencies with jurisdiction before installing or using a hydrogen system.

- Air Products Safetygram #4 Gaseous Hydrogen
- ANSI/AIAA standard for hydrogen safety guidelines is AIAA G-095-2004, Guide to Safety of Hydrogen and Hydrogen Systems
- ASME B31.1, Power Piping Code
- ASME B31.3, Process Piping Code
- ASME B31.8, Gas Transmission and Distribution Systems
- BCGA Code Of Practice CP4 Industrial Gas Cylinder Manifolds and Gas Distribution
   Pipework
- BCGA Code Of Practice CP33 The Bulk Storage of Gaseous Hydrogen at Users' Premises
- CGA G-5, Hydrogen
- CGA G-5.4, Standard for Hydrogen Piping Systems at Consumer Locations
- CGA G-5.5, Hydrogen Vent Systems
- CGA G-5.6, Hydrogen Pipeline Systems
- CGA G-5.8, High Pressure Hydrogen Piping Systems at Consumer Locations.
- FM Global Property Loss Prevention Data Sheets 7-50: Compressed Gases in Cylinders
- FM Global Property Loss Prevention Data Sheets 7-91: Hydrogen
- IGC Doc 121/04/E, Hydrogen Transportation Pipelines System Design Features
- NASA
- NSS 1740.16 Safety Standard For Hydrogen And Hydrogen Systems Guidelines for Hydrogen System Design, Materials Selection, Operations, Storage, and Transportation
- NFPA 52, Vehicular Fuel Systems Code
- NFPA 55, Standard for the Storage, Use, and Handling of Compressed Gases and Cryogenic Fluids in Portable and Stationary Containers, Cylinders, and Tanks, 2005 Edition
- NFPA 68, Standard on Explosion Protection by Deflagration Venting
- NFPA 70, National Electrical Code

- NFPA 497, Recommended Practice for the Classification of Flammable Liquids, Gases, or Vapors and of Hazardous (Classified) Locations for Electrical Installations in Chemical Process Areas
- NFPA 13, Standard for the Installation of Sprinkler Systems
- NFPA 45, Standard on Fire Protection for Laboratories Using Chemicals
- NFPA 55, Standard for the Storage, Use, and Handling of Compressed Gases and Cryogenic Fluids in Portable and Stationary Containers, Cylinders, and Tanks
- NFPA 68, 2007 Standard on Explosion Protection by Deflagration Venting
- NFPA 69, Standard on Explosion Prevention Systems
- NFPA 91, Standard for Exhaust Systems for Air Conveying of Vapors
- NFPA 255, Standard Method of Test of Surface Burning Characteristics of Building Materials
- OSHA 29CFR1910.103 1910.103 Hydrogen

## **Hazardous Substances Precautions**



**WARNING** Before using hazardous substances (toxic, harmful, and so on), please read the hazard indications and information reported in the applicable Material Safety Data Sheet (MSDS). Use personal protective equipment according to the safety requirements.

### **Biological Hazard Warning Note**

In laboratories where samples with potential biological hazards are handled, the user must label any equipment or parts which might become contaminated with biohazardous material.



The appropriate warning labels are included with the shipment of the instrument. It is the user's responsibility to label the relevant parts of the equipment.

When working with biohazardous materials, you are responsible for fulfilling the following mandatory requirements:

- Providing instructions on how to safely handle biohazardous material.
- Training operators to be aware of potential hazards.

- Providing personal protective equipment.
- Providing instructions for what to do if operators are exposed to aerosols or vapors during normal operation (within the intended use of the equipment) or in case of single fault situations such as a broken vial. The protective measures must consider potential contact with the skin, mouth, nose (respiratory organs), and eyes.
- Providing instructions for decontamination and safe disposal of relevant parts.



**WARNING** The user or operator is responsible for the safe handling of hazardous chemicals or biological compounds including (but not limited to) bacterial or viral samples and the associated waste, according to international and local regulations.

### **Venting Toxic Gases**

When analyzing toxic compounds, be aware that during the normal operation of the GC some of the sample might be vented outside the instrument through the split and purge flow vents; therefore, be sure to vent the exhaust gases to a fume hood. Consult local environmental and safety regulations for instructions in exhausting fumes from your system.

## **Contacting Us**

There are several ways to contact Thermo Fisher Scientific for the information you need.

#### \* To find out more about our products

Go to www.thermo.com/ms for information about our products.

✤ To get local contact information for sales or service

Go to www.thermoscientific.com/wps/portal/ts/contactus.

- \* To suggest changes to documentation or to Help
  - Fill out a reader survey online at www.surveymonkey.com/s/PQM6P62.
  - Send an e-mail message to the Technical Publications Editor at techpubs-austin@thermofisher.com.

# Introduction

Use the information in this chapter to determine whether your TSQ 8000 system is working properly and to check its basic systems.

**IMPORTANT** You will likely want to change the GC column before setting up a method. See Chapter 2, "Changing the Column," for instructions on changing the column.

#### Contents

- Confirming Your Instrument is Working
- Checking Power to the System
- Verifying the Carrier Gas Flow Rate
- Checking Your Carrier Gas Tank Pressure
- Verify Collision Gas Tank Pressure
- Checking the Vacuum and Temperature
- Cleaning the Exterior of Your Instrument
- Configuring Your Instrument

### **Confirming Your Instrument is Working**

After installing a new column, confirm that your GC/MS system has power, the carrier gas flow and collision gas delivery pressure are correct, the gas tanks have enough pressure, and the system is leak-free and has reached vacuum and temperature.

### **Checking Power to the System**

To confirm that the TSQ 8000 system is powered on, check that the power light on the front panel is lit and solid green. See Figure 1. If it is not, the instrument is not powered on.

#### To power on the TSQ 8000 instrument

Lift up the power switch located on the upper left side panel of the instrument near the back.



**Figure 1.** Front Panel of the TSQ 8000 Instrument

#### ✤ To power on the TRACE 1300/1310 GC

Reach over the top right of the instrument and pull up on the large plastic ribbed power switch on the back.

#### \* To confirm that a TRACE 1300 is powered on

Confirm that the power light on the status panel is solid green.

#### To confirm that a TRACE 1310 is powered on

Confirm that the touchscreen main menu has appeared.

### Verifying the Carrier Gas Flow Rate

Once you confirm that the system is powered on, verify that the carrier gas rate is correct.

#### \* To check the carrier gas flow rate on the TRACE 1310 GC

- 1. Choose Instrument Control and then Front/Back inlet.
- 2. Display the column flow.

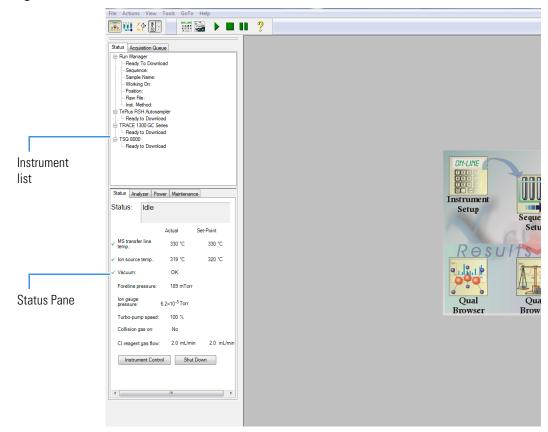
If the actual and set point amounts in **Col. Flow** are the same, then you have good carrier gas flow. If the amounts are different, refer the Troubleshooting section of the *TRACE 1300/1310 GC Series User Guide*.

#### ✤ To check the carrier gas flow rate on a TRACE 1300



- 1. Open the *Xcalibur* data system by clicking the **Xcalibur** icon on the computer desktop.
- 2. On the Xcalibur roadmap, select the **TRACE 1300** from the instrument list in the left side pane.
- 3. The Status Pane opens.

Figure 2. Status Panel in Xcalibur



4. Verify the column flow rate under the Status tab in the status pane.

### **Checking Your Carrier Gas Tank Pressure**

Make sure you have enough pressure in the carrier gas tank to accommodate the number of samples you plan to run. If the pressure is too low, you might run out of gas in the middle of a run, which could compromise the results of your data.

#### \* To check your carrier gas tank pressure

- 1. Locate your carrier gas tank. It might be in a different room, depending on how your lab is set up.
- 2. Look at the pressure gauge on the tank.
- 3. Ensure the pressure is more than 100 psi at the primary (or first) regulator stage. If it is not, you may want to replace the tank if you have to run a lot of samples.

### **Verify Collision Gas Tank Pressure**

Check that the input pressure of your collision gas is at 59–61 psig for Argon or 56–58 psig for Nitrogen and that the tank is connected to the TSQ 8000 instrument.

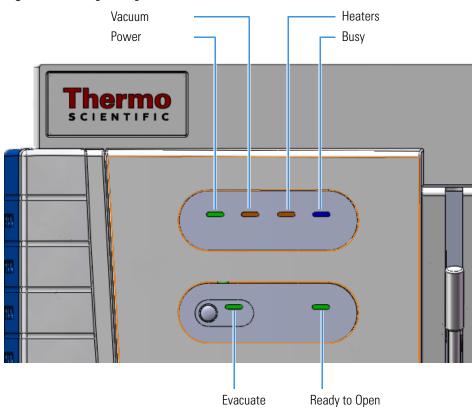


**CAUTION** Collision gas input pressure must remain constant for proper instrument performance. The regulator used to supply the collision gas must be able to deliver  $60 \pm 1$  psig. Ensure that the regulator is marked clearly at 60 psig and is stable enough to supply constant pressure at  $60 \pm 1$  psig.

### **Checking the Vacuum and Temperature**

Use the lights on the front of your TSQ 8000 system to check the vacuum and temperature of the instrument.

To check the vacuum, look at the Vacuum light. See Figure 3. When the light is a solid green, the TSQ 8000 is under sufficient vacuum. If the Vacuum light is slowly blinking orange, you have not yet achieved vacuum. If the Vacuum light is blinking orange quickly, you have a large leak that has prevented the instrument from achieving vacuum. In this case, you must turn the power off and find and fix the leak. Most likely, the column nut must be tightened, the column was not installed correctly, or the vent valve was not completely closed. See Troubleshooting for more information.



#### Figure 3. Using the Lights on the TSQ 8000 Instrument

To check the temperature, look at the Heaters light. When the Heaters light is a solid green, the TSQ 8000 system is at temperature. If the Heaters light is blinking orange, the ion source, ion optics and/or transfer line are not at temperature. If the Heaters light is not lit, the heaters are not turned on.

**Note** Until the Vacuum light is a solid green (high vacuum is achieved), the heaters will not power on and the Heaters light will not be lit.

You may also use the TSQ 8000 Dashboard to check the vacuum and temperature of your system. If your system has achieved sufficient vacuum, a green check mark appears next to **Vacuum** on the dashboard. The System Overtemp utility indicates if your TSQ 8000 system is above its maximum operating temperature. If it reads **False**, the system is within safe operating temperature. If it reads **True**, your TSQ 8000 system is above its safe operating temperature, and a red X will appear next to **System Overtemp** instead of a green check mark. You cannot operate the system while it is above safe operating temperature. Adjust your laboratory environment if necessary. You may also want to clean or replace the fans or filters on your TSQ 8000 system. See the *TSQ 8000 Hardware Manual* for instructions on cleaning and replacing the fans and filters.

TSQ 2000 Dashboard       Actual Set-Point         Actual Set-Point       Actual Set-Point         M Stransfer line temp:       310 °C       310 °C         You Start over temp:       347 °C       350 °C         System over temp:       347 °C       350 °C         Vacuum:       OK       Vacuum         Foreline pressure:       75 m Tor       Vacuum         In source temp:       12410°5 Tor       Vacuum         Collision gas on:       No       No         Cl reagent gas flow:       0.0 mL/min       0.0 mL/min         Image:       Auto Ture       Image:       Vacuum         Image:       Auto Ture       Image:       Image:         Image:       Ture Types       Image:       Auto SRM	Status:       Idle         Actual       Set-Point         MS transfer line temp:       310 °C         System Overtemp:       347 °C         System overtemp:       False         Vacuum:       OK         Foreline pressure:       75 mTor         Ion gauge pressure:       75 mTor         Ion gauge pressure:       12x10°5 Torr         Turbo-pump speed:       100 %         Collision gas on:       No         Cl reagert gas flow:       0.0 mL/min         Cl reagert gas flow:       0.0 mL/min         Catibration Gas Spectrum       Custom Spectrum         Custom Spectrum       Auto Tune         Image:       Tune Types         Image:       Method Editor	inguie 4. Checking vacuum and	
Actual Set-Point   MS transfer line temp: 310 °C   310 °C 310 °C   System Overtemp: 347 °C   System over temp: False   System over temp: State   System over temp: State	Actual Set-Point   M Strandfer line temp: 310 'C   310 'C 310 'C   System Overtemp: 347 'C   System over temp: False   Vacuum: OK   Foreline pressure: 75 m Torr   Inspauge pressure: 12×10 ° Torr   Turbopump speed: 100 'k   Collection gas on: No   Mr/Water Spectrum Vacuum   Image: Custom Spectrum Image: Custom Spectrum   Image: Custom Spectrum Image:	🞦 TSQ 8000 Dashboard 📃 🖃 🔤	
<ul> <li>Ion source temp:: 347 'C 350 'C</li> <li>System over temp:: False</li> <li>Vacuum: OK</li> <li>Foreline pressure: 75 mTorr</li> <li>Ion gauge pressure: 1.2×10<sup>-5</sup> Torr</li> <li>Turbo-pump speed: 100 %</li> <li>Collision gas on: No</li> <li>Cl reagent gas flow: 0.0 mL/min 0.0 mL/min</li> <li>Catibration Gas Spectrum</li> <li>Catibration Gas Spectrum</li> <li>Custom Spectrum</li> <li>Custom Spectrum</li> <li>Wew Tune Report</li> <li>Wew Tune Report</li> <li>Method Edtor</li> <li>Auto SRM</li> </ul>	Ion source temp:: 347 °C 350 °C System over temp:: False Vacuum: OK Foreline pressure: 75 mTor Ion gauge pressure: 1.2×10 <sup>-5</sup> Tor Turbo pump speed: 100 % Collision gas on: No Cl reagent gas flow: 0.0 mL/min Calibration Gas Spectrum Calibration Gas Spectrum Calibration Gas Spectrum Custom Spectrum Custom Spectrum Vacuum Method Editor Auto SRM Instrument Control		
		MS transfer line temp: 310 °C 310 °C Ion source temp: 347 °C 350 °C System over temp: False Vacuum: OK Foreline pressure: 75 mTor Ion gauge pressure: 1.2×10 °5 Tor Turbo pump speed: 100 °X Collision gas on: No Cl reagent gas flow: 0.0 mL/min 0.0 mL/min Cl reagent gas flow: 0.0 mL/min 0.0 mL/min Calibration Gas Spectrum Custom Spectrum Custom Spectrum View Tune Report View Tune Types Method Editor Auto SRM	

Figure 4. Checking Vacuum and Temperature on the TSQ 8000 Dashboard

## **Cleaning the Exterior of Your Instrument**

When the exterior of your instrument gets dirty, wipe it with a clean, dry, lint-free cloth.

## **Configuring Your Instrument**

Initially, the field service engineer will configure your TSQ 8000 instrument. However, if your instrument loses communication with the computer, you have reinstalled *Xcalibur*, or you have a new computer or device, follow these instructions to reconfigure it.

- ✤ To configure your TSQ 8000 instrument
- 1. Close the TSQ 8000 Dashboard and any open Xcalibur windows.
- 2. From the Start menu on your computer desktop, choose **Start | All Programs | Thermo Foundation 3.0 | Instrument Configuration**.

- 3. When the Instrument Configuration window opens, you will see icons of all connected instruments.
- 4. Select the TSQ 8000 icon in the Available Devices column and click **Add** to move it into the Configured Devices column.

🍇 Thermo Foundation I	nstrument Configuratio	on			×
Device <u>T</u> ypes : All	•				
Available Devices:			Configured Devices:		
AI/AS 1310	Al/AS 3000			Tota and	
Focus GC	TRACE GC Ultra	E			
TSQ 8000	TRACE 1300 GC Series	-			
	Add >>		<< Remove	Configure	
	Done		<u>H</u> elp		

Figure 5. Finding Available Devices

5. Select the icon for each instrument you want to configure (See Figure 6) and click Add.

🍇 Thermo Foundation I	nstrument Configuration	_		<b></b>
Device <u>T</u> ypes : All	-			
Available Devices:			Configured Devices:	
Al/AS 1310	AI/AS 3000	•	TRACE 1300 GC Series	TSQ 8000
TSQ 8000	TRACE 1300 GC Series		Al/AS 1310	
	Senes			
		Ŧ		
	Add >>		<< Remove	Configure
	Done	]	Help	]

#### Figure 6. Configuring a Device

- 6. Select the TSQ 8000 from the list of configured devices.
- 7. Click **Configure** to open the TSQ 8000 Configuration dialog box.
- 8. On the General page, set the pressure units.

Т	SQ 8000 Configuration
	General Maintenance Intervals
l	Pressure units:
	Remote start: Pa bar
	Enable Cl
	Communications
	OK Cancel

#### **Figure 7.** Setting the Pressure Units

**Note** You only need to set up the pressure units if you have an ion gauge or convectron gauge installed on your system. The readbacks from these components will display in the units set in this dialog box.

9. Set the remote start, which tells the TSQ 8000 system when the GC has started a run. When you configure the GC, you can tell it what to send out to the instrument. Make sure the value in this dialog box matches what you set on the GC. The default is Active Low. See Figure 8.

Fi	gure 8. Setting the I	Remote Start
Т	SQ 8000 Configuration	
	General Maintenance	Intervals
	Pressure units:	Torr
	Remote start:	Active Low -
	Enable CI	Active Low Active High
	Commun	ications
	0	Cancel

10. Check **Enable CI** if you will be using chemical ionization and you have installed the optional CI hardware.

T:	SQ 8000 Configuration
	General Maintenance Intervals
	Pressure units: Torr
	Remote start: Active Low -
	Enable Cl 🔽
	Communications
	OK Cancel

11. Click **Communications** to reset the network IP address and assign a TCP port. See Figure 10.

This tells Xcalibur how to find the TSQ 8000 system if you changed the IP address using Instrument Configuration. For security purposes, you may also want to modify the TCP port. Consult your local IT Department for help.

Figure 10. Setting up the Network

Communications	×
Network address:	192 168 1 234
TCP port:	30
ОК	Cancel

12. Click the **Maintenance Intervals** tab to set the number of days until you plan to perform maintenance on certain components of your GC/MS system. See Figure 11.

Figure 11. Setting the Maintenance Intervals

Т	SQ 8000 Configuration	-	×	
ſ	General Maintenance Inter	vals		
	Ena	able	Days	
	Foreline pump oil:	1	125	
	Turbo pump oil/bearing:	1	520	
	Filament 1:	1	1,000	
	Filament 2:	V	1,000	
	lon source:	1	1,000	
1	Multiplier:	1	1,000	
	Reset All			
	ОК		Cancel	

- 13. Select the Foreline Pump Oil check box to enable the maintenance intervals. Then set the number of days after which you want to be reminded to check the oil. The manufacturer recommends changing the oil every 125 days. Select the Turbo Pump Oil/Bearing check box to enable the maintenance reminder. Then set the number of days after which you want to be reminded to check the oil. Refer to the Turbo Pump manual for the manufacturer's recommended maintenance intervals.
- 14. Select the **Filament 1** check box to enable the maintenance reminder. In a leak-free system, expect the filament to last between 30-360 days, depending on usage.

- 15. Select the **Filament 2** check box to enable the maintenance reminder. Then set the number of days after which you want to be reminded to check filament 2. In a leak-free system, expect the filament to last between 30-360 days, depending on usage.
- 16. Select the **Ion source** check box to enable the maintenance reminder. Then set the number of days after which you want to be reminded to check the ion source. The time between cleaning depends very strongly on your analysis. You will have to determine the correct length of time between source cleanings.
- 17. Select the **Multiplier** check box to enable the maintenance reminder. Then set the number of days after which you want to be reminded to check the electron multiplier.
- 18. Click **OK** to return to the main Instrument Configuration window. See Figure 12.

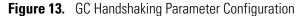
Device <u>T</u> ypes : All	•				
Available Devices:			Configured Devices:		
Al/AS 1310	AI/AS 3000		TRACE 1300 GC Series	TSQ 8000	
Focus GC	TRACE GC Ultra	E	Al/AS 1310		
TRACE GC Ultra (2)	TRACE 1300 GC Series	-			
	Add >>		<< Remove	Configure	]

Figure 12. Main Instrument Configuration Window

- 19. Select the TRACE 1300 or TRACE 1310 icon to configure the GC handshaking parameters.
- 20. Under the General tab, set the handshaking parameters as shown in Table 1.

Table 1.	GC Handshaking Parameters
----------	---------------------------

Remote Start In	High to Low
Inhibit Ready In	When High
End of Run Out	High to Low
Start of Run Out	High to Low
GC Ready Out	When Low
Prep Run Out	When Low



Device Types	3:	
All	▼	
Available De	TRACE 1300 Series GC Configuration	×
	🖉 General 🔯 Inlets 🔍 Detectors	s 🔶 Auxiliary
	Connection	Handshaking
AI/AS	Network address:	Remote start in: High -> Low ▼
	172 . 16 . 0 . 106	End of run out: High -> Low 🔻
	Advanced	
Focus	Options	GC ready out: When Low 💌
	Pressure units: kPa 💌	Start of run out: High -> Low 💌
	Lock GC input during run:	Prep run out: When Low 💌
TRACE GO	Get	OK Cancel Help
INACEG	uu	
	Add >>	Configure
	Done	Help

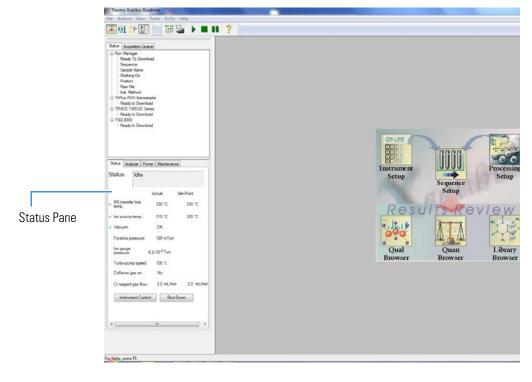
21. Click OK once you have entered the correct GC handshaking parameters.

**Note** For the remainder of the GC configuration settings, refer to the *TRACE 1300* and *TRACE 1310 Series GC User Guide*. Refer to your autosampler user documentation for the correct autosampler configuration settings.

**Note** If you are running samples with a Direct Probe System, you do not have to configure the GC.

- 22. Click **Done** to close the Instrument Configuration window.
- 23. Check the status of the configured instruments in the Status Pane of the Xcalibur Roadmap.

#### Figure 14. Xcalibur Roadmap



# 2

# **Changing the Column**

The TSQ 8000 ships with a factory-tested 15 m  $\times$  0.25 mm ID TR-SQC column, which the field service engineer uses to qualify the instrument. However, once it gets dirty, you cannot purchase a new one, so you must install a column that accommodates the type and quantity of samples that you will be running. For best results, choose a column that will give you the best possible resolution, analysis speed, and quantitation.

#### Contents

- Determining the Column Type
- Replacing the Factory Installed Column
- Connecting the Column to the Transfer Line

### **Determining the Column Type**

When determining the type of column for your particular needs, here are a few things to consider:

• **Column Material**—Columns made out of fused silica are economical and widely used. Columns made out of this material have a wide range of stationary phases and are available in many sizes that can be used with a mass spectrometer.

Large diameter columns made of steel are widely used in process gas analysis, but they are not typically used on mass spectrometers. There are also metal-clad, fused silica columns, which have the advantages of fused silica, but the metal makes them resistant to breakage. These columns are less common and more expensive.

• **Stationary Phase**—The stationary phase is the most important consideration when selecting a column. The interaction between the stationary phase and the analyte determines how well the analytes separate from each other (resolution) and also affects how quickly the separation occurs (analysis time). Choose a stationary phase that is compatible with the nature of your analytes and the maximum GC oven temperature that you will use.

- Internal Diameter—The smaller the diameter of the column, the better the separation. However, smaller diameter columns do not have as much capacity for matrix or analytes. As a result, smaller diameter columns are subject to overloading, which leads to retention time shifts and peak shape changes. Larger diameter columns can accept larger concentrations of material, but will require longer columns or slower GC oven temperature ramps (which increase the analysis time) to match the separation power of smaller-diameter columns. Typical column sizes for GC/MS have inside diameters (ID) of 0.25 mm. Smaller ID columns, such as the 0.18 and 0.10 mm, are becoming increasingly popular. Additionally, 0.32 and 0.53 mm ID columns are commonly used.
- **Film Thickness**—With larger film thicknesses, there is more capacity for the analyte. This capacity can aid in the separation of high concentration samples and in the separation of very volatile samples because thicker stationary phases allow more opportunities for the analytes to interact with the stationary phase. The optimal film thickness depends on the internal diameter of the column, and the maximum total capacity needed for sample analysis.

Thick films with small internal diameters give very strong interactions with the analytes, which can result in longer analysis time and peak tailing. Large ID columns with thin films have very little interaction with the analytes, which will result in very fast analysis times with little separation. Typical film thicknesses are 0.25  $\mu$ m for a column with an ID of 0.25 mm. Other common film thicknesses are 0.1, 0.5, and 1.0  $\mu$ m.

Be careful when selecting columns for mass spectrometers. Some columns with large inner diameters that work fine with other GC detectors may need lower head pressure when operated with vacuum at the outlet. This lower head pressure can allow air diffusion into the column through the carrier gas flow module's split and purge valves.

• Length—The length of the column affects how much time the analyte has to interact with the stationary phase. Longer columns typically have better resolutions and higher capacities, but longer analysis times. Longer columns are also more expensive. Typical column lengths are 15 or 30 m for GC/MS, but 100 m columns may be needed for very complex mixtures like gasoline. Very short columns (2.5, 5, and 10 m) are also available.

**Note** Contact your local sales representative to order a Thermo Scientific column. You can also refer to our catalog or visit our Web site at www.thermoscientific.com/columns.

### **Replacing the Factory Installed Column**

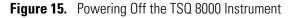
#### To replace the factory-installed column in the TSQ 8000 system

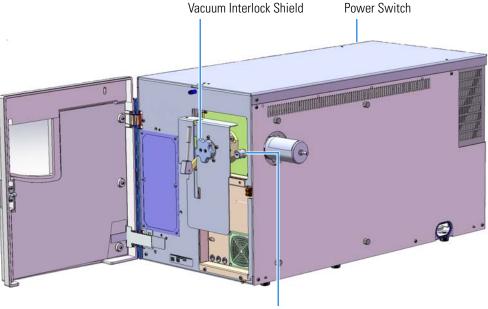
**Note** If you are running samples, stop the acquisition before powering off the system.

- 1. Cool down the GC oven and injector. See the GC documentation for information.
- 2. Open the TSQ 8000 Dashboard and click **Shut Down**.

During the shutdown procedure the vacuum and heaters lights will remain off. Once the procedure is complete and the instrument is ready to be powered off, the power light will turn amber and start blinking rapidly. At this point it is safe to power off the TSQ 8000 system.

3. On the left side of the instrument, push down on the power switch to power-off the TSQ 8000 system.





Vent Valve Knob

- 4. Open the front door of the instrument.
- 5. Look behind the right side of the vacuum interlock shield and twist the vent valve knob one and a half times in a counterclockwise direction to open the vent.
- 6. Remove the current column:
  - a. Make sure the heated zones of the GC are cooled down. Refer to the GC documentation for instructions.
  - b. Turn off the carrier gas and if used, the detector gas. See the GC documentation for information about using detector gases.
  - c. Open the front door of the GC.



**WARNING BURN HAZARD:** The injector, oven, and transfer line may be hot. Allow them to cool to room temperature before touching them.

- d. Unscrew the transfer line nut and remove the column from the transfer line.
- e. Unscrew the injector and detector nuts and remove the column.

- f. Remove the column from the column rack and from the GC.
- 7. Connect the new column to the injector inside the GC.

**Note** Wear clean, lint- and powder-free gloves when you handle the column and injector ferrule.

- a. Unwind the column enough to easily connect its ends to the injector and detector.
- b. Wipe about 100 mm (4 in.) of the column with a tissue soaked in methanol.
- c. Insert the column through the injector retaining nut and ferrule (larger end up). If the M4 retaining nut is used, slide it on the column through the side cut. Wipe the column again with a tissue soaked in methanol.

**Tip** Slide a notched septum on the column before the injector retaining nut to make it easier to measure the proper distance between the nut and end of the column.

- d. Use a scoring wafer to score and break the column about 1 cm (0.4 in.) from the end. Use a magnifying glass to check for an even, flat cut. Repeat if necessary.
- e. Insert a notched septum on the column to hold the retaining nut at this position. Thread the retaining nut into the injector but do not tighten.
- f. Ensure that the end of the column is the proper distance (splitless = 5 mm, split = 10 mm, PTV and PTVBKF = 30 mm) from the back of the injector nut.
- g. Adjust the column position so that the septum contacts the bottom of the retaining nut. Use your fingers to tighten the retaining nut until it starts to grip the column.
- h. Tighten the column nut finger-tight until it starts to grip the column plus a quarter turn.
- i. Remove the notched septum from the column.
- 8. Set up the GC parameters:
  - a. Set the oven and injector temperature to 50 °C (122 °F).
  - b. Set the carrier gas flow to 1.0 mL/min.
  - c. Turn off vacuum compensation, which is located on the Carrier menu of the GC.
  - d. Use the column flowmeter connector to verify that there is flow through the column. If you do not have a flowmeter, dip the column outlet in a small vial of methanol. Bubbles indicate there is flow through the column. If there is no flow, check that the carrier gas is on, the GC inlet is pressurized, and the column is not plugged. If there is still no flow, consult the GC documentation or contact Technical Support.
  - e. Allow the column to purge for at least 10 minutes. If you used methanol to detect column flow, remove column from methanol during purge time.
  - f. Insert the column into the fitting of the column flowmeter connector that blocks the column flow.

- 9. Perform a column leak check:
  - a. On the TRACE 1310, select the **Leak Check** icon in the **Maintenance** menu. Otherwise, perform the leak check through the Chromatography Data System. Refer to the *TRACE 1300 and TRACE 1310 Series GC User Guide* for instructions.
  - b. Start the leak check.

The split and purge valves of the selected channel are automatically closed, and the channel is pressurized with carrier gas to the leak check setpoint.

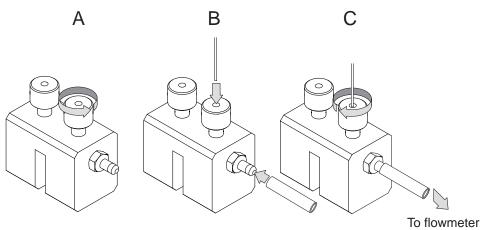
The system monitors the pressure for one minute. If the pressure does not drop more than the maximum allowed sensitivity value, then the leak check will pass. If the leak check does not pass, use the leak detector to find and fix any leaks.

**Tip** Leaks can be caused by not tightening the fitting on the column flowmeter connector. Check the fitting before looking for the leak elsewhere.



**CAUTION INSTRUMENT DAMAGE**: Do not allow the column flowmeter connector to exceed 80 °C (176 °F). Otherwise, it will melt and damage the instrument.

- c. Repeat the leak check until no leaks are indicated.
- 10. Calibrate the carrier gas flow (column evaluation):
  - a. Carefully push the capillary column end into the flowmeter section of the column flowmeter connector.
    - Figure 16. Column Flowmeter Connector



- b. Connect the flowmeter to the dedicated fitting on the column flowmeter connector.
- c. If you have a TRACE 1310, select the **Back** or **Front Column** icon in the **Configuration** menu. Otherwise, perform the column evaluation through the Chromatography Data System. See the *TRACE 1300 and TRACE 1310 User Guide* for instructions.

- d. Select **Column** and input the column's physical characteristics.
- e. If a pre-/post column is present, set the length and nominal internal diameter of the pre-/post column in the same valid ranges for the column. The following two lines are added to the menu.

**Note** For the most reproducible results, you should conduct a more detailed column evaluation. However, the following steps, while recommended, are not required.

- f. Start the column evaluation. According to the physical characteristics of the column, the system calculates and displays the relevant column K-factor. At the end of the routine, a message will indicate that the evaluation was successful.
- g. Expect a K-factor of approximately 0.7 0.9 for a 15 m, 0.25 mm i.d. column (1.3 2.0 for a 30 m, 0.25 mm i.d. column). If the column does not report a K-factor within this range or within 0.1 units of the previous stored value, check for a leak or broken column using the leak detector. The K-factor is a measured resistance for the column. A K-factor that is too low may indicate a leak in the system, while a K-factor that is too high may indicate a blockage.

Fix any issues found and rerun column evaluation until an appropriate K-factor is achieved.e a leak in the system, while a K-factor that is too high might indicate a blockage.

- 11. Disconnect the column flowmeter:
  - a. Disconnect the column from the column flowmeter connector.
  - b. Remove the clear plastic component, including its fittings, from the oven and set them aside.
  - c. Close the GC door.
- 12. Condition the column before inserting it into the TSQ 8000 system. Column conditioning consists of passing a carrier gas flow through the column heated to a programmed temperature as described in the column manufacturer's instructions.
  - a. If there are no conditioning instructions, perform the column conditioning by setting a final temperature 10 °C–20 °C below the column's recommended maximum temperature.



**CAUTION INSTRUMENT DAMAGE:** The material released from the column (column bleed) during conditioning may contaminate the ion source if the column is inserted into the transfer line during the high-temperature stage of conditioning.



**WARNING FIRE HAZARD:** Do not use hydrogen as the carrier gas for conditioning your column. It could vent into the oven and present an explosion hazard.

b. Run the slow temperature program that is recommended by the manufacturer. A typical program would hold the column at 40 °C (104 °F) for 15 minutes, and then ramp at 10 °C/min (50 °F/min) up to 10–20 °C below the maximum allowed column temperature. Hold the column at this temperature for two hours.



**CAUTION INSTRUMENT DAMAGE:** Never exceed the column manufacturer's maximum operating temperature.

### **Connecting the Column to the Transfer Line**

When connecting the column to the transfer line, you may use either the regular transfer line nut or the spring loaded transfer line nut with the graphite Vespel<sup>™</sup> ferrule.

#### To connect the column using the regular transfer line nut

- 1. Lower the oven temperature and allow it to cool.
- 2. If the TSQ 8000 system is running, shut down and vent it. See the *TSQ 8000 Hardware Manual* for instructions.



**CAUTION BURN HAZARD:** The injector, detectors, oven, and transfer line may be hot. Allow them to cool before touching them.

3. Unwind about one turn of the column from the column outlet end.

**Note** Wear clean, lint- and powder-free gloves when you handle the column and transfer line ferrule.

- 4. Wipe approximately 300 mm (12 in.) of the column with a tissue soaked in methanol.
- 5. Choose an appropriate ferrule for the outer diameter of your column.

**Note** If the maximum oven temperature in your method is  $\geq 290$  °C (554 °F), Thermo Fisher Scientific recommends using a spring loaded transfer line nut with a graphic Vespel ferrule or a SilTite<sup>TM</sup> nut and ferrule. By cycling the oven at and above this temperature, expansion and contraction of the graphite Vespel material can cause leaks in the transfer line.

6. Insert the column through the transfer line nut and ferrule, entering through the tapered end of the ferrule. Wipe the column again with a tissue soaked in methanol.

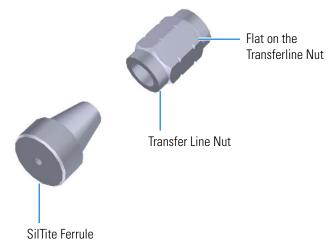


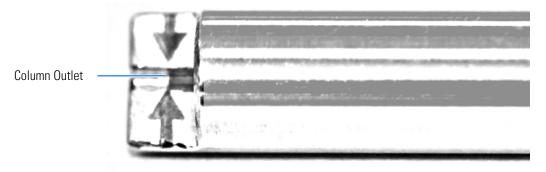
Figure 17. Transfer Line Nut and SilTite Ferrule Orientation

- 7. Insert the column into the measuring tool, which is in the TSQ 8000 Toolkit (See Figure 18), so that it is even with the lines at the end of the column. Figure 19 indicates proper positioning of the column in the tool for accurate measuring.
- 8. Use a scoring wafer to score and break the column. Use a magnifying glass to check for an even, flat cut. Repeat if necessary.
- 9. Use a 5/16 in. wrench to hold the column measuring tool steady.

**Figure 18.** Column Measuring Tool

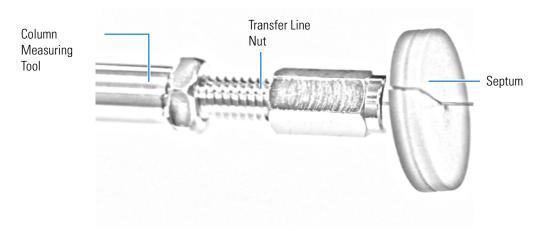


- 10. While holding the column measuring tool steady, tighten the transfer line nut with a 1/4" wrench until the column just stops moving in the ferrule.
- 11. Turn the transfer line nut 1 flat backward so the column is able to move in the ferrule with slight resistance.
- 12. Line up the outlet of the column with the arrows on the end of the column measuring tool.
  - Figure 19. Lining Up the Column in the Column Measuring Tool



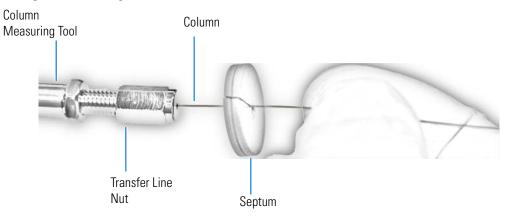
13. Place a septum with a notch cut into it behind the transfer line nut. The septum marks the place on the column where it should exit the nut.

Figure 20. Positioning the Septum

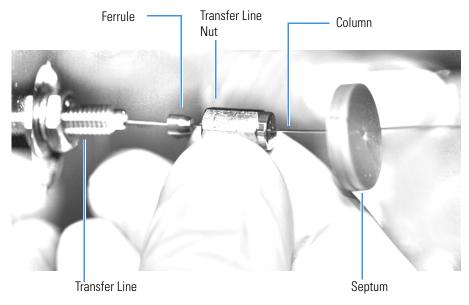


14. Pull the column back from the transfer line nut. Do not move the septum from its position on the column.



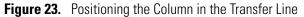


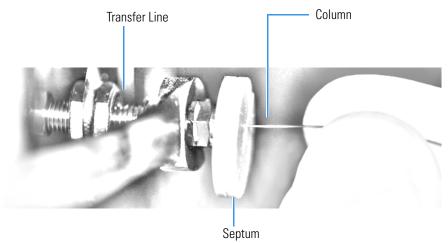
- 15. Loosen the transfer line nut from the column measuring tool.
- 16. Remove the column, transfer line nut and ferrule from the column measuring tool, making sure not to move the septum from its location on the column.
- 17. Insert the column into the transfer line.



### Figure 22. Inserting the Column into the Transfer Line

- 18. Tighten the transfer line nut until it is just secure enough so that you cannot move it.
- 19. Loosen the nut by turning it exactly 1 flat backward.
- 20. Position the column in the transfer line. Use the septum as a guide to measure the correct length you should insert the column. Be careful not to change the location of the septum on the column.





- 21. Tighten the nut 1 flat forward—back to where it is secure enough in the transfer line that you cannot move it.
- 22. Tighten the nut 1 additional quarter turn.
- 23. Remove the cut septum.

**Note** If you are using a SilTite ferrule, follow the instructions that come with SilTite ferrules. If you are using a graphite Vespel ferrule, they require conditioning to ensure a leak-tight seal. See the *TSQ 8000 Spare Parts Guide* for information about ordering these ferrules.

- 24. Condition the graphite Vespel ferrule:
  - a. Raise the oven temperature to the maximum temperature you will operate the GC.
  - b. Wait 10 minutes.
  - c. Lower the oven temperature to 40 °C (104 °F) and allow it to cool before continuing.



**WARNING BURN HAZARD**: The oven may be hot. Allow it to cool to room temperature before opening it. The injector will still be hot, so do not touch it.

- d. Retighten the transfer line nut.
- 25. Close the front door of the GC.
- 26. Restore working conditions.
  - a. Raise the oven temperature to the initial temperature that you will use.
  - b. Turn on vacuum compensation on the GC.
  - c. Power on the TSQ 8000 instrument. See Chapter 1, "Introduction," for instructions.
  - d. Once the TSQ 8000 instrument is pumped own and able to scan, view air water spectra and look for evidence of leaks with a large m/z 28 signal. If you observe a leak, stop scanning and gently tighten the nut in small increments until no leaks appear when scanning.
- \* To connect the column using the spring loaded transfer line nut

**Note** If you use a graphite Vespel ferrule with your column, Thermo Fisher Scientific recommends using the spring loaded transfer line nut with it. See the *TSQ 8000 Spare Parts Guide* for ordering information.

- 1. Lower the oven temperature and allow it to cool.
- 2. If the TSQ 8000 system is running, shut down and vent it. See the *TSQ 8000 Hardware Manual* for instructions.



**CAUTION BURN HAZARD:** The injector, detectors, oven, and transfer line may be hot. Allow them to cool before touching them.

3. Unwind about one turn of the column from the column outlet end.

**Note** Wear clean, lint- and powder-free gloves when you handle the column and transfer line ferrule.

- 4. Wipe approximately 300 mm (12 in.) of the column with a tissue soaked in methanol.
- 5. Choose an appropriate ferrule for the outer diameter of your column.
- 6. Insert the column through the spring loaded transfer line nut and ferrule, entering through the tapered end of the ferrule.
- 7. Wipe the column again with a tissue soaked in methanol.

Flat on the Transferline Nut

Figure 24. Transfer Line Nut and Graphite Vespel Ferrule Orientation

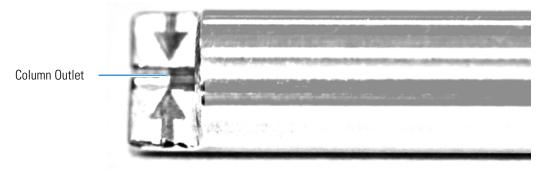
- 8. Insert the column into the measuring tool, which is in the TSQ 8000 Toolkit (See Figure 25), so that it is even with the lines at the end of the column. Figure 26 indicates proper positioning of the column in the tool for accurate measuring.
- 9. Use a scoring wafer to score and break the column. Use a magnifying glass to check for an even, flat cut. Repeat if necessary.
- 10. Use a 5/16 in. wrench to hold the column measuring tool steady.

Figure 25. Column Measuring Tool

- 11. While holding the column measuring tool steady, tighten the spring loaded transfer line nut with a 1/4" wrench until the column just stops moving in the ferrule.
- 12. Turn the spring loaded transfer line nut 1 flat backward so the column is able to move in the ferrule with slight resistance.

13. Line up the outlet of the column with the arrows on the end of the column measuring tool.

Figure 26. Lining Up the Column in the Column Measuring Tool



14. Place a septum with a notch cut into it behind the transfer line nut. The septum marks the place on the column where it should exit the nut.

Column Measuring Tool Column

**Figure 27.** Positioning the Septum

15. Pull the column back from the spring loaded transfer line nut. Do not move the septum from its position on the column.

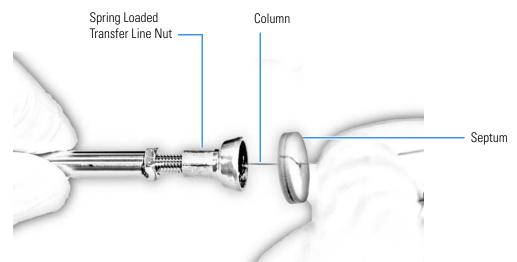


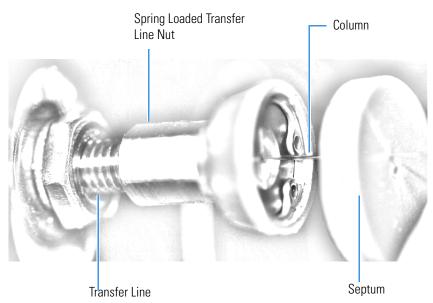
Figure 28. Pulling the Column Back from the Spring Loaded Transfer Line Nut

- 16. Loosen the transfer line nut from the column measuring tool.
- 17. Remove the column, transfer line nut and ferrule from the column measuring tool, making sure not to move the septum from its location on the column.

**Note** The ferrule should still be able to move on the column. Use the septum to mark the correct location where the column should exit the nut.

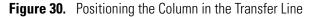
18. Insert the column into the transfer line.

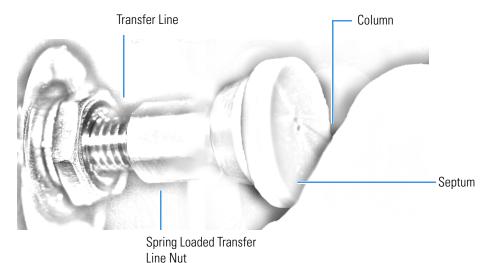
Figure 29. Inserting the Column into the Transfer Line



19. Tighten the spring loaded transfer line nut until it is just secure enough so that you cannot move it.

- 20. Loosen the nut by turning it exactly 1 flat backward.
- 21. Position the column in the transfer line using the cut septum to measure the correct length you should insert the column.





- 22. Tighten the spring loaded transfer line nut 1 flat forward—back to where it is secure enough in the transfer line that you cannot move it.
- 23. Tighten the spring loaded transfer line nut 1 additional quarter turn.
- 24. Remove the cut septum.
- 25. Condition the graphite Vespel ferrule:
  - a. Raise the oven temperature to the maximum temperature you will operate the GC.
  - b. Wait 10 minutes.
  - c. Lower the oven temperature to 40 °C (104 °F) and allow it to cool before continuing.



**WARNING BURN HAZARD:** The oven may be hot. Allow it to cool to room temperature before opening it. The injector will still be hot, so do not touch it.

- 26. Close the front door of the GC.
- 27. Restore working conditions.
  - a. Raise the oven temperature to the initial temperature that you will use.
  - b. Turn on vacuum compensation on the GC.
- 28. Power on the TSQ 8000 instrument. See Chapter 1, "Introduction," for instructions.

29. Once the instrument is pumped down and able to scan, view air water spectra and look for evidence of leaks with a large m/z 28 signal. If you observe a leak, stop scanning and gently tighten the nut in small increments until no leaks appear when scanning.

## Tuning

Tuning will improve the performance of your TSQ 8000 system. For optimum stability, you must wait until the power, vacuum, and heaters lights on the front of the instrument are a solid green. These lights indicate that the instrument has reached vacuum and that it is at the last set temperature. If the system has been powered off for a period of time (that is, a cold system), the system components take up to 4 hours to thermally stabilize after reaching the temperature setpoint. If you did not vent the instrument (that is, the system is hot), the components take approximately 10 minutes to thermally stabilize after reaching the temperature setpoint. If you exchanged the ion source without venting, the source components take approximately 30 minutes to stabilize temperature.

#### Contents

- Accessing TSQ 8000 Auto Tune
- Tuning the TSQ 8000 Instrument
- Updating Tunes for New RF Lens

You can tune the TSQ 8000 system by using Xcalibur or the TSQ 8000 Dashboard.

**IMPORTANT** Be sure to give the TSQ 8000 system enough time to stabilize. Otherwise, you may see mass drift, mass spectral changes, or changes in the fragmentation of your data.

**Note** If you are running samples with heavy matrix, running the samples before the source has had time to stabilize at high temperature will prematurely dirty the ion volume and optics.

### Accessing TSQ 8000 Auto Tune

- To access TSQ 8000 Auto Tune choose one of these two methods
- Double-click the **TSQ 8000 Dashboard** shortcut on your desktop to open the TSQ 8000 Dashboard and then click **Auto Tune**. See Figure 31.

💾 TSQ 8000 Da	shboard					
Status:	Idle					
		Actual	:	Set-Point		
<ul> <li>MS transfer I</li> </ul>	ine temp.:	310 °	С	310	°C	
<ul> <li>Ion source te</li> </ul>		347 °(		350	°C	
<ul> <li>System over</li> </ul>	temp:	False	е			
Vacuum:		OK	-			
Foreline pres		75 r				
lon gauge pr						
Turbo-pump			%			
Collision gas	on:	No				
CI reagent ga	as flow:	0.0 m	nL/min	0.0	mL/r	nin
	Air/Wa	ater Spectr	um			
	Calibratio	n Gas Spe	ctrum			
	Custo	om Spectru	m			
K	A	uto Tune				
K	View	Tune Repo	ort			
J.	Tu	ine Types				
	Me	thod Editor				
***	A	utoSRM				
	Instru	ment Contr	rol			
	Sł	nut Down				

Figure 31. TSQ 8000 Dashboard

### **Tuning the TSQ 8000 Instrument**

### ✤ To tune your instrument

- 1. In the Tune Types field, select EI, CI, or Both, depending upon which ionization mode you are using.
- 2. Select a tuning category. See Figure 32.

TSQ 8000 Autotune	? ×
Tune types:       EI       CI          Both         Category:       AI             El Standa El SRM Tune               El Standa El SRM Tune               El Standa El SRM Tune               El Standa El Standard Tune               El Target Factory                 El SRM Factory Tune (built-in)                 CI- Tune (built-in)	<ul> <li>Display report when complete</li> </ul>
Now running:	Show spectra
	Start

Figure 32. Tuning Categories

3. Select the tune type you want to use:

Figure 33. Tune Types

🔏 TSQ 8000	Autotune	? x
Tune types: Category:	© EI © CI ⊚ Both All ▼	
El Stand El Targe El Tune El SRM	ard Quick Tune (built-in) ard Tune (built-in) t Tune (built-in) Check (built-in) Factory Tune (built-in) e (built-in) (huilt-in)	Display report when complete
Now running	g:	Show spectra
Action:		Start

a. **EI Standard Tune**—Provides EI tuning and is used to completely retune the system. It takes the longest amount of time to run, but it has the advantage of reoptimizing nearly all the parameters affecting the signal. This type of tune performs a mass calibration, tunes the lenses and resolution, and performs a leak check. It adjusts the detector sensitivity to generate a m/z 219 ion with an intensity of 20,000,000 counts. Unless your SOP requires it, this is not the best tune to use on a daily basis because of the length of time required to run it.

- b. **EI Standard Quick Tune**—A shortened version of the EI Standard Tune recommended for use when subsequent maintenance tuning is needed after an EI Standard Tune. This tunes the repeller in conjunction with Q3 to overcome the effects of matrix buildup in the source. It starts with the last saved tune and sets the detector sensitivity to generate a m/z 219 ion with an intensity of 20,000,000 counts.
- c. **EI SRM Tune**—Provides EI tuning and completely retunes the system. This tune reoptimizes nearly all the parameters affecting the signal. This type of tune performs a mass calibration, tunes the lenses and resolution, and performs a leak check. Unless your SOP requires it, this is not the best tune to use on a daily basis because of the length of time required to run it. This tune is more sensitive for high mass than the EI Standard Tune, making it more sensitive to many SRM transitions. It sets the detector sensitivity to generate a m/z 219 ion with an intensity of 20,000,000 counts and verifies the correct mass calibration.
- d. **EI SRM Quick Tune**—A shortened version of the EI SRM Tune that is recommended for use when subsequent maintenance tuning is needed after an EI SRM Tune. This tunes the repeller in conjunction with Q3 to overcome the effects of buildup in the source. It starts with the last saved tune and sets the detector sensitivity to generate a *m*/*z* 219 ion with an intensity of 20,000,000 counts.
- e. **EI Tune Check**—Used to check how well your last tune is performing. As it is the fastest tune type, it allows you to quickly update the detector sensitivity as the system gets dirty. This allows the sample intensity to remain constant for longer periods without running a full retune of the instrument. The daily tune check performs a leak check, makes sure the mass calibration is correct, and sets the detector sensitivity to generate a m/z 219 ion with an intensity of 20,000,000 counts. If your SOP allows it, you can use this tune to rapidly verify that the previous lens tune is still generating good spectra.
- f. **EI Diagnostics Only**—Runs a complete set of diagnostics, including a leak check, and generate a report. No tuning is performed. Uses the parameters from the last saved tune.
- g. **EI Target Tune**—Starts with the last saved tune and adjusts the ion ratios of the calibration gas to those expected of classic single quadrupole MS analysis. This tune is intended to be run after full calibration using an EI Standard Tune. If your SOP was developed in response to regulatory requirements for classic single-quadrupole MS analysis, or you require classic single quadrupole spectra for spectral library matching, use this tune. It also sets the detector sensitivity to generate a m/z 219 ion with an intensity of approximately 20,000,000 counts and verifies correct mass calibration.
- h. **EI Initial Tune**—Creates a default tune file. It requires a clean instrument, and it sets the repeller to 0V and the quadrupole voltages to low values. This tune is used to reset parameters for all the other tunes after cleaning the ion source. As a result, use this tune only when the ion source is clean. The EI initial tune should also be used when changing from an SRM tune to a standard tune. The EI initial tune has higher resolution and lower sensitivity than the EI standard tune. This tune starts with the tune file stored in the instrument at the factory. It then performs a mass calibration and leak check, sets the repeller to 0 V, and tunes the lenses. The quadrupole offset

voltage is set to a low value to improve resolution, which is also tuned. The detector gain is calibrated to generate 300,000 electrons for every ion that strikes the detector. Additionally, this tune generates spectra that are the closest in appearance to the factory tune.

**Note** If you are starting with a clean source, first run the EI Initial Tune as a reset tune. After this tune is complete, follow it with your preferred tune.

i. **CI- Tune**—Used to analyze samples with negative CI. The standard NCI tune performs a mass calibration, then tunes the lenses and sets the resolution. This type of tune assumes you are using methane as the CI reagent gas and tunes the system with a 1.0 mL/min flow. This tune does not set the detector gain. The built-in CI- tune will start with the most recent AutoTune\_NCI tune file, so have an appropriate tune file saved on the instrument's PC.

**Note** Chemical ionization tunes are very different from the electron ionization tunes. Do not use a CI tune unless your instrument has a CI ion volume and CI reagent gas installed.

**Note** If the instrument was last used in EI mode and tuned with a high repeller voltage before switching to a clean CI ion Source, a tune file with a low repeller voltage should be loaded in manual tune and saved to the instrument before tuning in CI mode.

j. **CI+ Tune**—Used to analyze samples with positive CI. The standard PCI tune performs a mass calibration, then tunes the lenses and sets the resolution. This type of tune assumes you are using methane as the CI reagent gas and tunes the system with a 1.5 mL/min flow. This tune does not set the detector gain. The built-in CI+ tune will start with the most recent AutoTune\_PCI tune file, so have an appropriate tune file saved on the instrument's PC.

**Note** To add a tune type to the list, see Modifying an Automatic Tune.

4. Select the **Display Report When Complete** check box so that you can view the tune report after running the tune.

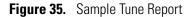
🔏 TSQ 8000 Autotune	? ×
Tune types: 🔘 EI 🔘 CI 💿 Both	
Category: All	
El Diagnostics Only (built-in) El Initial Tune (built-in) El SRM Quick Tune (built-in) El SRM Tune (built-in) El Standard Quick Tune (built-in) El Standard Tune (built-in) Fl Tarnet Tune (built-in)	✓ Display report when complete
Now running:	Show spectra
Action:	
	Start

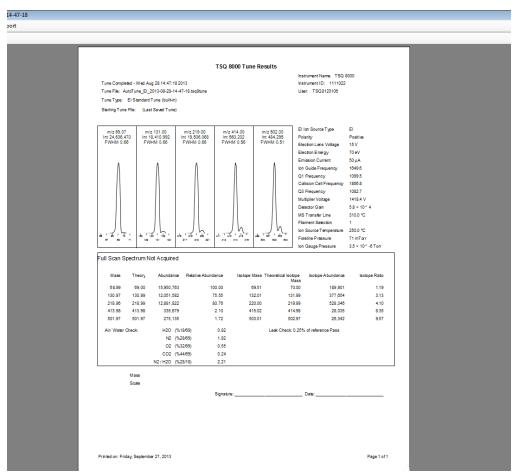
Figure 34. Displaying a Report

- 5. Select the Show Spectra check box to show the spectra while the system is tuning.
- 6. Click the **Start** button to begin tuning.

**Note** Make sure the power options on your computer are not set to go into Standby mode while you are acquiring data for your tune. Otherwise, it will interrupt your tune.

Once the tune completes, your tune report opens in the *TSQ 8000 Tune Results Viewer*. If you did not select the **Display Report When Complete** check box, you can click **View Tune Report** on the *TSQ 8000 Dashboard* and view the report.





- 7. Click **Report Options**. In the dialog box, select the charts and reports you want to display and change the name of your instrument.
- 8. Select a mass for **Calculate Relative Abundance Using**. The default is mass 69, which will reference all masses to mass 69. Some masses may have over 100% abundance. If you do not wish to have a relative abundance above 100%, select Base Peak. This option will select the reference mass as the highest abundance mass in the tune.
- 9. Click OK.

-			
	Report Options	ÎY –	164
			109
		ncy	186
	Lab name: OK		108
			141
	Instrument name: Cancel		5.8
			310
	Calc relative abundance using: 69		1
		ture	250.
129	Include signature line		71 n
			3.5
	Optional reports	⊢	
mΝ	🗌 Diagnostics 🔲 Device settings 🔲 Tune type settings		
9	Custom report	ance	
00	Use custom report definition	.901	
39		.854	
39	File name:		
79 38		,346 .035	
97		,342	

Figure 36. Tune Report Options

10. To print your tune report, click **Print Report** to open a print dialog box and print your report.

en Tune Results 🛛 ⋥ Save Report to PDF 🏼 Print Report		🛃 Re
rint EX		
neral		
	TSQ 8000 Tune Results	
Select Printer		Instrument Name: TSQ 8000
Search Add Printer	Tune Not Completed - Tue Oct 11 09:44:33 2011	Instrument ID: 1011152
Fax Microsoft XPS Document Writer	Tune File: AutoTune_E1_2011-10-11-09-44-30.tsq0tune Tune Type: El Standard Tune (bulk in)	User: Alvin
eg microsoft Ar 5 bocument mitter	Starting Tune File:	
	starting Tune File: Tuning Failed - Unable to load initial tune settings .	
Status: Ready Print to file Preferences		Electron Lens Voltage 15 V
Location: Find Printer.		Electron Energy 70 eV
Comment:		Emission Current 50 µA
Page Range		Ion Guide Frequency 1610.0 Q1 Frequency 1087.0
Al Number of copies: 1		Collision Cell Frequency 2500.0
C Selection C Current Page		Q3 Frequency 1087.0
C Pages		Multiplier Voltage 1100.0 V
		Detector Gain 1.0 × 10 ^ 6 MS Transfer Line 250.0 °C
		Filament Selection 1
		Ion Source Temperature 200.0 *C
Print Cancel Apply		Foreline Pressure 88 mTorr Ion Gauge Pressure 9.6 × 10 ^ -6 Torr
	Ful Sean Spentrum Net Acquired	
	ArrWatercheck is not performed Lask check	tot performed
	Signature:	Date:

Figure 37. Printing a Tune Report

In the Tune Results window, you can open tune results, print reports, or change the way you view the report.

11. To save the report, click the **Save** icon **□** and save it as a Microsoft Excel<sup>™</sup> file or an Adobe<sup>™</sup> Acrobat<sup>™</sup> PDF file.

You may find it useful to compare this tune with a previous tune report of the same tune type. Some changes in peak height are normal, but if the difference is significant, see Troubleshooting.

- 12. To browse to another tune report on your computer, click **Open Tune Results** in the top of the window.
- 13. Click **Open** to open a previously saved tune report.

Organize 🔻 🛛 New fo	older					1
🔆 Favorites	-	Name	Date modified	Туре	Size	
📃 Desktop		AutoTune_EI_2011-10-11-09-44-33.tsq8tu	10/11/2011 9:44 AM	TSQ8TUNERES File	6 KB	
\rm Downloads		AutoTune_EI_2011-10-11-10-08-17.tsq8tu	10/11/2011 10:08	TSQ8TUNERES File	6 KB	
📃 Recent Places		AutoTune_EI_2011-10-11-10-48-00.tsq8tu	10/11/2011 10:48	TSQ8TUNERES File	107 KB	
		AutoTune_EI_2011-10-11-12-20-59.tsq8tu	10/11/2011 12:21	TSQ8TUNERES File	100 KB	
词 Libraries		AutoTune_EI_2011-10-21-17-28-23.tsq8tu	10/21/2011 5:28 PM	TSQ8TUNERES File	104 KB	
Documents		AutoTune_EI_2011-10-27-14-42-56.tsq8tu	10/27/2011 2:42 PM	TSQ8TUNERES File	103 KB	
J Music	E	AutoTune_EI_2011-10-27-15-36-24.tsq8tu	10/27/2011 3:36 PM	TSQ8TUNERES File	52 KB	
Pictures		AutoTune_EI_2011-10-27-15-59-12.tsq8tu	10/27/2011 3:59 PM	TSQ8TUNERES File	25 KB	
🔣 Videos		AutoTune_EI_2011-10-27-16-38-03.tsq8tu	10/27/2011 4:38 PM	TSQ8TUNERES File	30 KB	
		AutoTune_EI_2011-10-28-09-58-33.tsq8tu	10/28/2011 9:58 AM	TSQ8TUNERES File	108 KB	
🖳 Computer		AutoTune_EI_2011-11-03-09-26-27.tsq8tu	11/3/2011 9:26 AM	TSQ8TUNERES File	25 KB	
📇 OS (C:)		Initial_EI_2011-10-11-09-44-38.tsq8tuneres	10/11/2011 9:44 AM	TSQ8TUNERES File	5 KB	
👝 Removable Disk I		Leak_EI_2011-10-11-10-58-51.tsq8tuneres	10/11/2011 10:58	TSQ8TUNERES File	11 KB	
		Leak_EI_2011-10-18-13-35-22.tsq8tuneres	10/18/2011 1:35 PM	TSQ8TUNERES File	11 KB	
📬 Network	÷	Leak_EI_2011-10-18-13-40-31.tsq8tuneres	10/18/2011 1:40 PM	TSQ8TUNERES File	11 KB	

Figure 38. Loading Tune Results

If the sensitivity and resolution are adequate for running your samples, you are ready to develop or run a method.

### **Updating Tunes for New RF Lens**

If you have purchased a new lens 3/RF lens (PN 1R120574-0103), you must update the ion guide frequency in the TSQ 8000 Manual Tune utility and retune your instrument.

#### \* To update the ion guide frequency using the TSQ 8000 Manual Tune utility

- 1. Open the TSQ 8000 dashboard.
- 2. The TSQ 8000 Manual Tune button is not on the dashboard by default. To add it to the dashboard, right-click in the empty space on the TSQ 8000 Dashboard. See Figure 39.

Furbo-pump speed:	100 %	
Collision gas on:	No	
CI reagent gas flow:	0.0 mL/min	0.0 mL/min
Air/	Water Spectrum	
Calibra	ation Gas Spectrum	
Cu	stom Spectrum	
J.	Auto Tune	
Ka Vie	ew Tune Report	
J.	Tune Types	
	Method Editor	
**	AutoSRM	
		Add Custom Butto
Ins	trument Control	
	Shut Down	

Figure 39. Creating a Custom Button on the TSQ 8000 Dashboard

3. The Select the Program to Add a Button For dialog box opens. Select the file TSQ8.ManualTune and click Open. See Figure 40.

Figure 40. Locating the TSQ8.ManualTune File

Computer + OS (C:) + Program	Files   Thermo	TSQ8 ►	✓ Search T.	3Q8
Organize 🔻 New folder				= -
🔆 Favorites	*	Name		Date modified
📜 Downloads		퉬 backup		12/4/2013 12:05 PM
🖭 Recent Places		퉬 Manuals		1/15/2014 5:05 PM
📃 Desktop		🔣 TSQ8.AutoTune		12/5/2013 12:07 PM
		TSQ8.Dashboard		12/5/2013 12:07 PM
🚔 Libraries		TSQ8.InstControlServe		12/5/2013 12:07 PM
Documents	E	TSQ8.InstrumentCont	trolApp	12/5/2013 12:07 PM
J Music		K TSQ8.ManualTune		12/5/2013 12:07 PM
Pictures		M TSQ8.RealTimeScanni	-	12/5/2013 12:07 PM
Videos		TSQ8.TuneResultsView		12/5/2013 12:07 PM
🖳 Computer		TSQ8.TuneTypeEditor	r	12/5/2013 12:07 PM
S Computer				
Removable Disk (F:)				
Con Matural	-	٠ ا	1	
File name: TSQ8.ManualTune			- Applicatio	ns

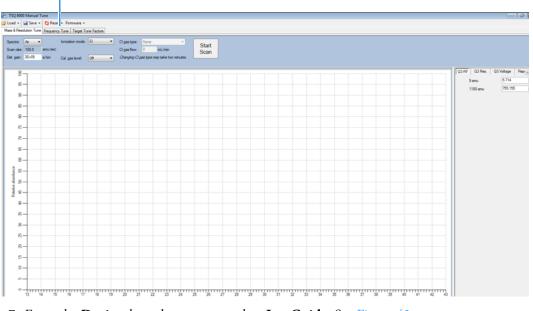
4. The TSQ8 Manual Tune button now appears on the dashboard. See Figure 41.



### Figure 41. Locating the TSQ8 Manual Tune Button on the TSQ 8000 Dashboard

- 5. Click **TSQ8 Manual Tune**. The TSQ 8000 Manual Tune utility opens.
- 6. Select **Frequency Tune** on the top menu. See Figure 42.

**Figure 42.** Accessing Frequency Tune Frequency Tune



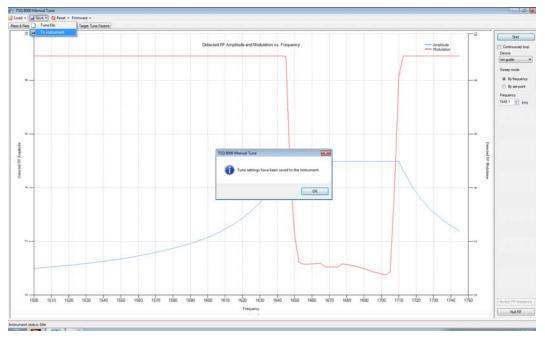
7. From the Device drop-down menu, select Ion Guide. See Figure 43.

		Device Menu
5Q 8000 Manual Tune		
ed + Ja Seve + C Reset + Firmware +		101
& Resolution Tune Frequency Tune Target Tune Factors		
	Detected RF Amplitude and Modulation vs. Frequency	Anglitude Bat Modulation Device
		Quel 3
		Start guide Guad 1
		Collision cell Quad 3
		O By set p
		Frequency
		1078.5 (0)
		RF Intelance
		Opt. Industance
		Cgt. industric

**Figure 43.** Locating the Device Menu

- 8. Click Start. The system will detect the ion guide frequency.
- 9. When the instrument has finished detecting the ion guide frequency, go to Save | To Instrument on the top menu. A dialog box confirming that the tune settings have been saved to the instrument will open. See Figure 44.

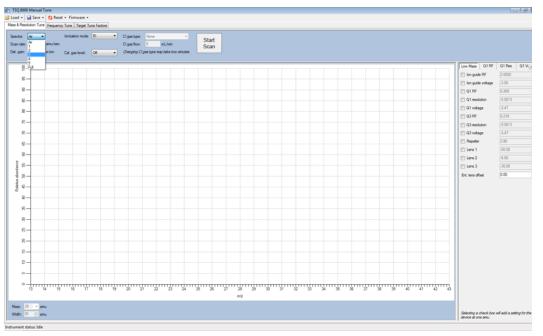
**Figure 44.** Saving the New Ion Guide Frequency to the Instrument



10. To confirm that the intensities are still correct, select **Mass & Resolution Tune** on the top menu to go back to the TSQ 8000 Manual Tune home page.

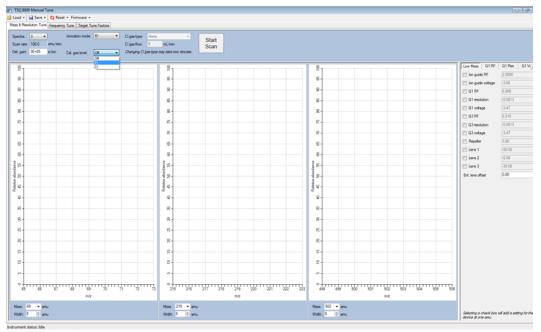
11. Choose **3** from the **Spectra** drop-down menu. See Figure 45.

Figure 45. Selecting the Correct Number of Spectra to Scan



12. Select EI from the Cal. Gas Level menu. See Figure 46.

Figure 46. Setting the Calibration Gas Level to El



13. Click **Start Scan**. When the instrument has finished scanning, check the intensities for the masses 69, 219, and 502. Set the masses using the **Mass** drop-down menu below each spectrum. See Figure 47.

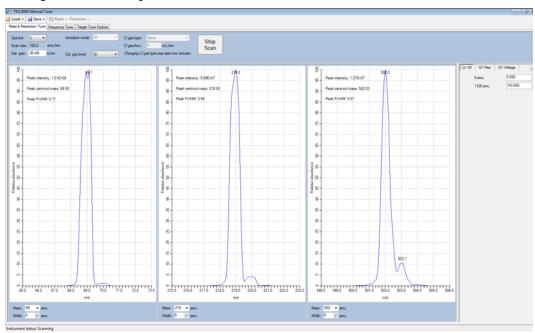


Figure 47. Checking the Mass Intensities

**Note** The intensities might be lower than they were at the previous frequency set for the ion guide until the lenses are tuned in AutoTune.

14. Once you have confirmed the intensities are sufficient for tuning, retune the TSQ 8000 system.

**3 Tuning** Updating Tunes for New RF Lens

# **Creating a Method**

Once you have tuned the TSQ 8000 system, you can create a method for each of its components. Use these methods to indicate to the GC/MS system how to collect your data.

#### Contents

- Accessing the TSQ 8000 Method Editor
- Creating an Autosampler Method
- Creating a TSQ 8000 Instrument Method
- Creating a GC Method
- Validating and Troubleshooting Methods

### Accessing the TSQ 8000 Method Editor

To create a method for the TSQ 8000 mass spectrometer, the TRACE 1300 or 1310 GC, and your autosampler, open the TSQ 8000 Method Editor.

**Note** For information about creating a method for other instruments and software, refer to the appropriate documentation.

**Note** All of the configured instruments are shown in the left pane of the Xcalibur Instrument Setup window. If your instruments are not shown, you must configure them. See "Configuring Your Instrument" for instructions.

4

#### ✤ To access the method editor

• Click Method Editor on the TSQ 8000 Dashboard.

The Method Editor window opens. See Figure 48. Icons of all connected instruments appear in the left pane. You may only configure one instrument at a time. The bright green arrow on the lower right corner of the instrument icon indicates the active instrument. The current instrument name also appears in the main menu.



Instrument Name 
 Unived - The po Xealibur

 File
 TSQ 8000

 Help

 D

 Help

 X
 Method Setur 250 土七 (E) ٠ GC run time C Probe run tim 200 🛨 °C Cigan type Met C Stop after: 10.0 = min Note 1000 - Ci gas flow 100 - mLinin Groups Time Total Scan (mn) Time (sec) SRM, SIM or Scient Manager Dwell or Scan Times Insci. Tune File Name Product Mass Neutral Loss Mass Tens Intel Collision Energy

Icon Arrow

### **Creating an Autosampler Method**

Use the method editor to create a method for the autosampler if you have one.

**Note** Instructions for setting the most common parameters for the TriPlus RSH Sampling System follow. Refer to your autosampler documentation for more detailed information about settings.

#### \* To create a method for the autosampler

1. In the left pane, select the icon for the autosampler.

The method view opens. See Figure 49.

Figure 49.	Autosample	<sup>-</sup> Method
------------	------------	---------------------

Method         Select method mode and modules, and click Create New Method button         Image: GC Liquids       C GC Headspace         Create New Method         Injectors         Injector port A - ID:         Injector port A - ID:         None         Injector port A - ID:         None         Injector port C - ID:         None         Injector port C - ID:         None         Syringes         Syringes         Syringe - ID:         Vash stations         Vash station - ID:         Internal standard / Solvent stations         Solvent station - ID:         None         Int standard station - ID:         None         Cooledheated trays         Primary tray - ID:         None         Secondary tray - ID:		TriPlus RSH Autosampler - No	ew
Select method mode and modules, and click Create New Method button         © GC Liquids       C GC Headspace         Injectors         Injector port A - ID:       Front         Injector port B - ID:       None         Injector port C - ID:       None         Syringes       Syringes         Syringe - ID:       • LS1 (10µL, 57mm)         Wash stations       •         Wash station - ID:       • None         Internal standard / Solvent stations       •         Solvent station - ID:       • None         Int standard station - ID:       • None         Int standard station - ID:       • None         Primary tray - ID:       None	Method ]		1.2019
© GC Liquids       C GC Heddspace       SPME	and the second sec	dules, and click Create New Method button	
Injectors         Injector port A - ID:         Injector port B - ID:         Injector port C - ID:         Injector port D - ID:         None         Syringes         Syringe - ID:         Vash stations         Wash station - ID:         Internal standard / Solvent stations         Solvent station - ID:         None         Int standard station - ID:         None			-
Injector port A - ID:       Front         Injector port B - ID:       None         Injector port C - ID:       None         Injector port C - ID:       None         Injector port D - ID:       None         Syringes       Syringe - ID:         Syringe - ID:       • LS1 (10µL 57mm)         Wash stations       •         Wash station - ID:       • Standard Wash 1 (Standard wash station)         Internal standard / Solvent stations       •         Solvent station - ID:       • None         Int standard station - ID:       • None         Cooled heated trays       •         Primary tray - ID:       None		Create New Method	
Injector port A - ID:       Front         Injector port B - ID:       None         Injector port C - ID:       None         Injector port C - ID:       None         Injector port D - ID:       None         Syringes       Syringe - ID:         Syringe - ID:       • LS1 (10µL 57mm)         Wash stations       •         Wash station - ID:       • Standard Wash 1 (Standard wash station)         Internal standard / Solvent stations       •         Solvent station - ID:       • None         Int standard station - ID:       • None         Cooled heated trays       •         Primary tray - ID:       None			
Injector port B - ID:       None         Injector port C - ID:       None         Injector port D - ID:       None         Syringe - ID:       • LS1 (10µL, 57mm)         Wash stations       •         Wash station - ID:       • Standard Wash 1 (Standard wash station)         Internal standard / Solvent stations       •         Solvent station - ID:       • None         Int standard station - ID:       • None         Cooled heated trays       •         Primary tray - ID:       None		• Front	-
Injector port C - ID:       None         Injector port D - ID:       None         Syringes       Syringes         Syringe - ID:       • LST (10µL, 57mm)         Wash stations       •         Wash station - ID:       • Standard Wash 1 (Standard wash station)         Internal standard / Solvent stations       •         Solvent station - ID:       • None         Int standard station - ID:       • None         Cooled heated trays       •         Primary tray - ID:       None		None	
Injector port D - ID:     None       Syringes     Syringes       Syringe - ID:     • [LS1 (10µL, 57mm))       Wash stations     •       Wash station - ID:     • [Standard Wash 1 (Standard wash station))       Internal standard / Solvent stations       Solvent station - ID:     • [None       Int standard station - ID:     • [None       Cooled heated trays     •       Primary tray - ID:     [None		None	
Syringe - ID:       •       LS1 (10µL, 57mm)         Wash stations       •         Wash station - ID:       •       Standard Wash 1 (Standard wash station)         Internal standard / Solvent stations       •         Solvent station - ID:       •       None         Int standard station - ID:       •       None         Cooled heated trays       •       •         Primary tray - ID:       None       •		None	
Syringe - ID:       •       LS1 (10µL, 57mm)         Wash stations       •         Wash station - ID:       •       Standard Wash 1 (Standard wash station)         Internal standard / Solvent stations       •         Solvent station - ID:       •       None         Int standard station - ID:       •       None         Cooled heated trays       •       •         Primary tray - ID:       None       •			
Wash stations       Wash station - ID:       Internal standard / Solvent stations       Solvent station - ID:       Int standard station - ID:       None       Cooled heated trays       Primary tray - ID:			
Wesh station - ID:       • Standard Wash 1 (Standard wash station)         Internal standard / Solvent stations         Solvent station - ID:       • None         Int standard station - ID:       • None         Cooled heated trays         Primary tray - ID:       None	Syringe - ID:	<ul> <li>LS1 (10μL, 57mm)</li> </ul>	-
Internal standard / Solvent stations Solvent station - ID: None Int standard station - ID: None Cooled heated trays Primary tray - ID: None	Wash stations		
Solvent station - ID:  None Int standard station - ID: None Cooledheated trays Primary tray - ID: None	Wash station - ID:	Standard Wash 1 (Standard wash station)	•
Int standard station - ID:     None       Cooled/heated trays       Primary tray - ID:	Internal standard / Solvent	stations	
Cooled heated trays Primary tray - ID: None	Solvent station - ID:	• None	v
Primary tray - ID:	Int standard station - ID:	* None	¥
Primary tray - ID:	Cooled/heated travs		
		None	•
	Secondary tray - ID:	None	•
		(A)	

- 2. Choose one of these three options: GC Liquids, GC Headspace, or SPME.
- 3. Enter the ID information for your samples. You must fill in any field with an asterisk (\*)
  - a. Injectors—Enter the injection ID into the corresponding injector port.
  - b. Syringes—Enter the syringe ID information.
  - c. Wash Station—Enter the wash station ID information.
  - d. (Optional) Internal Standard/Solvent Stations—Enter ID information for your solvent station or interval standard station.

- e. (Optional) Cooled/Heated Trays—Enter ID information for your primary and, if used, secondary trays.
- 4. Click Create New Method.
- 5. Use these instructions to configure the GC Liquids general method settings (see Figure 50):

Figure 50. General GC Liquids Settings on the TriPlus RSH Sampling System

- a. Injector Type—Choose Single or Double for each injector.
- b. Injection Mode—Choose Basic, Enrichment, Enrichment Needle Solvent Option, Internal Standard Double, Internal Standard Post, Needle Solvent Wash, Solvent Flush Double, or Solvent Flush Post.
- c. Rapid Mode—Choose Disable, After Sample Rinse, After Bubble Elimination, or After Sample Asp. in Home.
- d. Syringe Type—Enter your syringe volume in  $\mu$ L. Enter the syringe needle length in mm.
- e. Sample Volume—Enter the sample volume to be injected into the GC. Typical values are between 0.5 and 5  $\mu$ L.
- f. Plunger Strokes—Select the number of plunger strokes to use when drawing up the sample. Air bubbles in the syringe change the amount of sample injected, which can cause signal variation in different runs. To prevent this from occurring, increase the

number of plunger strokes to reduce the chance of an air pocket in your syringe. Typical values are between 3 and 10.

- g. Air and filling mode—Choose Auto to use the default or Custom to change the parameters.
- h. Sample Viscosity—Select Viscous if your sample is viscous or Non Viscous if your sample is non-viscous. With a viscous sample, the syringe is filled more slowly than if it was non-viscous. Since the amount of time saved is so small, setting this option to Yes might be easier.
- i. Sampling Depth in Vial—Select the Bottom Sense check box and enter the height from the bottom of the vial (in mm) at which the tip of the syringe needle will be placed in the sample vial when it is being filled.
- j. Injection Depth—Select Standard or Minimum to indicate how the sample is introduced into the GC. If you select Standard, the autosampler inserts the needle all the way into the injection port. If you select Minimum, the autosampler barely enters the injection port.
- k. Pre-inj Dwell Time(s)—Use this field to enter the time (in seconds) that the needle will be in the injection port before the plunger injects the sample.
- 1. Post-inj Dwell Time(s)—Use this field to enter the time (in seconds) that the needle will be in the injection port after the plunger injects the sample.
- 6. Configure the settings under the Washes tab (see Figure 51):

Figure 51. GC Liquids Washes Settings

	Xcalibur Instrument Setup Help	
	<u>X 8</u>	
	TriPlus RSH Autosampler - GC Liquids	
	General Washes Sync Advanced Washes	Basic
TRACE 1300 GC Series	Number of solvents(s): Single  Vash station: Standard wash station	
	Pre-injection	
TriPlus RSH Autosampler	Solvent volume (µL): 1.0 v	E
	Rinse	T
TSQ 8000	Rinses: 1	
	Post-injection Solvent: B V · V · V	
	Cycles: 1  Solvent volume (µL): 1.0	
	Image:	-
Ready		

- a. Number of Solvents—Choose Single or Multiple depending on the number of solvents you will use for your method. You may choose up to four solvents: A, B, C, or D.
- b. Wash Station—Choose Standard Wash Station or Large Wash Station. Refer to the autosampler documentation for more information about the wash stations.
- c. Pre-injection—Use the Solvent and Cycles fields to set the number of solvent purges that will occur before the autosampler touches your sample. Always include some sample rinses, either before or after injection, to make sure you do not have sample carryover from one injection to the next. Configure the settings so that the syringe is purged with the same solvent that was used in location A, B, C, or D or with solvents A and B or C and D. Typically, there is 0 or 1 cycles of pre-injection purges.
- d. Rinse—Use the Rinses list to select the number of times the syringe is rinsed with your sample before each injection. Rinses help ensure the sample being injected is not diluted by the residual rinse solvents. By purging the syringe with your sample before injection, the dilution is minimal. The standard setting is between 1 and 3 rinses. If you have a very limited amount of sample, however, you may want to set this field to 0 to conserve the sample.
- e. Post-injection—Use the Solvent and Cycles fields to set the number of solvent purges that will occur after the autosampler touches your sample. You should always have some sample rinses, either before or after injection, to make sure you do not have sample carryover from one injection to the next. You can have the syringe purged with the same solvent that was used in location A, B, C, or D or with solvents A and B or C and D. Typically, there are 1 to 5 cycles of post-injection purges.

# **Creating a TSQ 8000 Instrument Method**

Use the Method Editor to create an acquisition or maintenance method for the TSQ 8000 mass spectrometer.

- \* To create a method for the TSQ 8000 mass spectrometer
- 1. Click the **TSQ 8000** icon in the left pane to open the method view for this instrument.
- 2. From the Method Type list, select **Acquisition-General** or **Acquisition-Timed.** See Figure 52. An Acquisition method is used to collect data.

**Note** The **Maintenance** method is used to bake out or cool down the instrument during a sequence. This can be useful if you know you want to perform these tasks and you want to do them in an automated way as part of your sequence.

	lethod Set	ıp											
Metho	od type: Aca		• Use acc	quisition method to acquir	e any scan mode or ci	mbination of sca	n modes						
MStr	anafer lin Main	ation-Timed	Ionization	mode: Ci 👻		Run complet							
	Main ource temp.:	320 -		pe Ammonia	*	@ GC run	time C Probe run time						
		100				C Stop aft	er: 10.0 🚽 min						
A	quisition three	held: 1000 ±	CI gas flo	w: 0.30 _ mUmin	Apply to all groups								
Scan	s								E	Grou			
	Time (nin)	SRM, SIM or Scan Masses	Product Mass	Neutral Loss Mass	Collision Energy	Dwell or Scan Times (sec)	Tune File Name	Ion Polarity		Time (min)	Total Scan Time (sec)	Ovon. Fiter On	Chrom, Filter Peak, Width
*										1100			

#### Figure 52. TSQ 8000 Method Type Setup

a. Set the **MS Transfer Line Temperature**. This field represents the temperature of the transfer line, which is the tube that contains the column as it leaves the GC oven and enters the TSQ 8000 system. The maximum allowable temperature is 350 °C.



**CAUTION** You will damage the column and contaminate the TSQ 8000 instrument if you set the transfer line temperature above the maximum allowed temperature for the column.

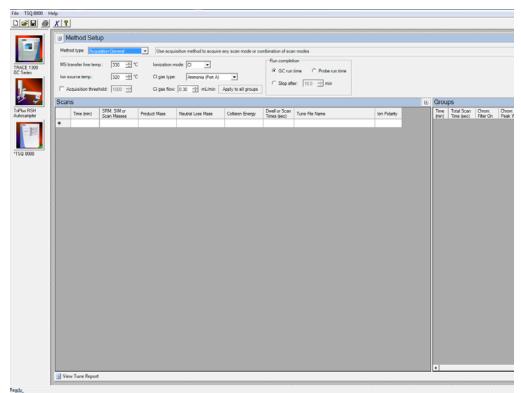


Figure 53. Setting Temperatures and Acquisition Threshold

- b. Set the **Ion Source Temperature**. You can enter a value between 0 and 350 °C. The optimal temperature depends on the analyte. Higher temperatures will keep the ion source cleaner, but will lead to increased fragmentation, which may reduce sensitivity.
- c. (Optional) Select the **Acquisition Threshold** check box and enter a value for the minimum peak height for the data file, if needed. If your peak has an intensity that is below this threshold, it will not be stored. This setting may help reduce noise, but it may also alter the reported isotope ratios because the smaller isotope signals will be preferentially reduced.
- d. Select EI or CI from the Ionization Mode pull-down menu.

**Note** Only use CI if you have installed a CI ion volume in the MS and connected CI reagent gas to your system. If you have CI, select a CI Gas Type from the pull-down menu and set the CI Gas Flow. Typical values for Methane CI for NCI are 1.0–1.5 mL/min. For Methane CI for PCI, the gas flows are typically 1.5–2.0 mL/min. Click the **Apply to All Groups Button** at the top of the method editor. The single value at the top of the method is sent when initializing the MS with the method. See Figure 55.

- e. To assign gases to the CI gas ports, open the TSQ 8000 Dashboard and click **Instrument Control**.
- f. Set CI Gas Port A or CI Gas Port B to the appropriate CI gas attached to that port. You may only use one CI Gas Port per method.

Figure 54. Setting the CI Gas Ports

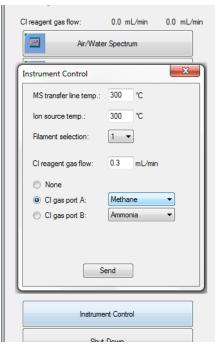
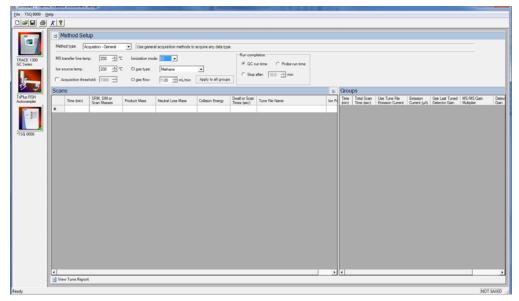


Figure 55. Acquisition-General CI Settings



**Note** The following parameters apply only to the **Acquisition-General** method type only. **Acquisition-General** is the recommend setting for full-scan only MS methods, or simple SIM or SRM methods. If you need to develop a complex SIM or SRM method with a large compound list or overlapping retention times, refer to the instructions for **Acquisition-Timed**.

3. For Acquisition-General methods only, do the following:

- a. In the **Run Completion** group, select the condition under which the run will end.
  - GC Run Time—Select this option if you want the MS run to end when the GC run is complete.
  - **Probe Run Time**—Select this option if you have a probe controller installed and you want the MS run to end with the probe run is complete.
  - Stop After—Select this option to set the number of minutes you want the MS to run. The end of the run can be between 0 and 1000 minutes. With this option you can stop the acquisition when all the compounds of interest have eluted, but the GC is still at an elevated temperature to keep the column clean. Thermo Fisher Scientific recommends that you select this option to save burn time on the filament.
- b. In the Scans pane, click a scan row to enter scan information under each column. See Figure 56.

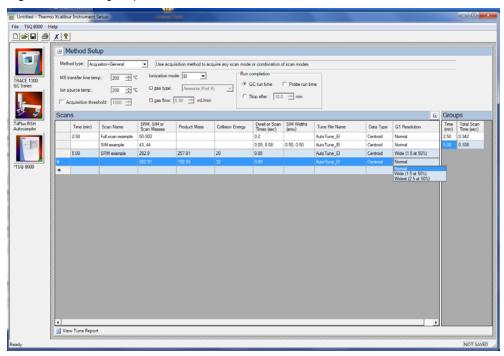


Figure 56. Setting Acquisition-General Scan Information

**Note** If some of the following columns are not shown in the Scan pane, you can right-click and display them. You can also reorganize the columns by clicking the heading of a column and dragging it to the left or right.

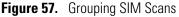
i. Use the **Time (min)** column to set the time that the MS begins to acquire the scan group. Set the time for the first group late enough to allow the solvent to move through the system, and early enough to ensure the first analyte will be captured in the segment. Set subsequent segment times so that the start time will occur between compounds of interest. You may set up different types of scans in a single group. The scans will occur in descending order.

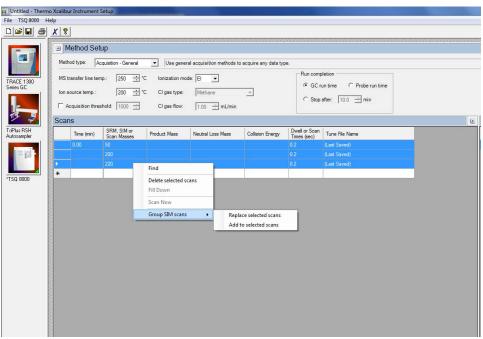
Your method may allow sequential full scans and SIM scans of a compound. As an example, in this method, the mass range of 50–300 will be scanned in 0.2 seconds. Immediately after the full scan finishes, the SIM scans begin. The SIM scans occur sequentially. Two different SIM masses, at 43 and 44, will be looked at for 0.05 and 0.08 seconds, sequentially. These simultaneous full scan and SIM scans will begin 2.5 minutes into the GC run. You will get a complete set of scans every 0.342 seconds. At 5 minutes into the GC run, the scans change to a set of SRM scans. The first SRM transition is 292.9 to 257.91 with a collision energy of 20 eV. The second SRM transition is 262.91 to 192.93 with a collision energy of 32 eV. Both SRM scans have a dwell time of 0.05 seconds. You will get a complete set of scans every 0.108 seconds.

**Note** The faster the MS scans, the more data points across a peak it will acquire. However, the precision of individual scans may be reduced.

All of these scans use the most recent AutoTune\_EI file. You can also use a specific tune file for each of the scans.

- ii. Use the **SRM, SIM, or Scan Masses** column to tell the MS which masses to scan.
  - In full-scan mode, enter the start and end mass separated by a dash. You must put each full-scan range on a separate line.
  - For SIM methods, enter individual masses of the ions you wish to monitor. You may enter multiple values in this field as long as individual values are separated by a comma. If you would like to set different collision energies for the individual SIM masses, enter the SIM masses with their associated collision energies on separate lines. Right-click on the SRM, SIM, or Scan Masses column and select Group SIM Scans | Replace Selected Scans. See Figure 57. You can also use this field to ungroup a group of SIM scans into individual masses.





- For SRM methods, enter your precursor ion mass in this column.

**Note** Each line in a scan must only contain a full-scan range, individual SIM masses, or an SRM transition. Scan types cannot be mixed in a single line. Set up simultaneous scans by giving them the same time in the **Time (min)** column.

iii. Enter the product ion mass (for SRM methods) in the **Product Mass** column. The product ion mass is the result of fragmenting the precursor ion with collision gas. If you want to perform a precursor ion scan, enter the mass of the target precursor ion into this column.

**Note** Entering a product ion mass changes a scan to SRM.

- iv. Use the **Neutral Loss Mass** column when you want to perform a neutral loss scan. Enter the mass of the target precursor ion in this column. The scan mass range must be at least 2.5 amu wide and must begin at 1 + the precursor ion mass.
- v. In the **Collision Energy** column, enter the collision energy (for SRM methods) in eV. The collision of a precursor ion with the collision gas at a particular energy fragments the precursor ions into product ions. By varying the collision energy, a single precursor ion can be fragmented into several product ions. An SRM transition is defined by a precursor ion being fragmented into a particular product ion mass using a certain collision energy. The Collision Energy column is unusable unless you define a precursor and product ion mass.

**Note** The system controls the collision gas so it will be ready when an MS/MS scan is requested.

- vi. In SIM or SRM mode, use the **Dwell or Scan Times** column to define the amount of time (in seconds) that the MS will look at your SIM ion mass or your SRM product ion mass. If you are in full-scan mode, the **Dwell or Scan Times** column determines the amount of time to scan across the designated range. Set this value to have 5–20 scans across your GC peak. If you have too few scans, the GC peak area is inaccurate. If you have too many scans, the MS signal becomes less precise. This column must be set to a value between 0.0005 and 5 s for SIM methods. For SRM methods, set to a value between 0.001 and 5 s. The default is 0.2 s.
- vii. Use the **Tune File Name** to select a tune file to be used for this scan. Select the autotune file you created in Chapter 3, "Tuning," . If you wish to use the same tune file for multiple scans, you can fill down this column by right-clicking on a tune file name.

**Note** Selecting a tune file to open without a date-time stamp opens the most recent tune of the tune type selected. To use a specific tune file, select a tune file of that prefix with the date-time stamp that you want.

**Tip** Use a tune file without the date-time stamp to create your method.

viii. (Optional) Use the **Scan Name** column to enter a description of the scan. You may use the name as a label to indicate the compound used with the scan.

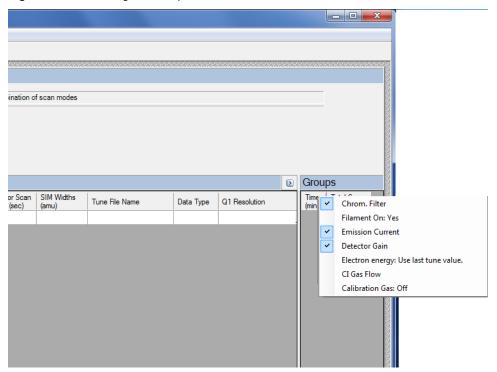
**Note** Scan Name, SIM Widths, Data Type, and Q1 Resolution columns are not shown by default. To view them, right-click one of the column headers to pull up the menu to display the hidden columns

- ix. Use the **SIM Widths** column to set the width range of the SIM window. The range of values can be between 0.01 and 10. The default is 1 amu, which means the MS will collect all the ions from your SIM mass ±0.5 amu.
- x. Use the **Ion Polarity** column if you are using CI mode to tell the instrument to generate positive or negative ions. Only use this column if you are using CI mode. In EI mode, this column should always be set to **Positive**.
- xi. Use the Data Type column to select whether you want to collect Profile or Centroided mass spectra. Centroided mass spectra are the most common because they are used by most of the libraries and provide the smallest data files. Profile mass spectra provide detailed mass spectral peaks, which result in large data files. Profile mass spectra are not available for SRM scans.
- xii. Use the Precursor (Q1) Resolution column to open the peak width of the precursor mass for SRM scans. The options are Normal, Wide (1.5 at 50%), or Widest (2.5 at 50%). The default setting is Normal, which uses the tuned resolution values from the tune file specified in the Tune File Name column.

Selecting Wide or Widest increases the precursor peak width to 1.5 and 2.5 amu (at 50%) respectively. Opening the precursor peak width will increase the ion signal into the collision cell and may improve the sensitivity of your method; however, it may also increase the background noise level.

- xiii. Use the Product (Q3) Resolution column to open the peak width of the precursor mass for SRM scans. The options are Normal, Wide (1.5 at 50%), or Widest (2.5 at 50%). The default setting is Normal, which uses the tuned resolution values from the tune file specified in the Tune File Name column. Selecting Wide or Widest increases the precursor peak width to 1.5 and 2.5 amu (at 50%) respectively.
- c. In the **Groups** pane on the right, review the information in each row. See Figure 58. As you create scans in the Scans pane, information in the groups pane is automatically displayed.

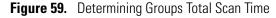
**Note** By default not all columns are displayed in the Groups pane. Right-click a heading to display them. You can also reorganize the columns by clicking the heading of a column and dragging it to the left or right.



**Figure 58.** Accessing the Groups Pane

i. The **Time (min)** column indicates the time that the MS begins to acquire data after the GC starts for that group. It is typical to have enough of a time delay for the start of the first group to allow the solvent to get through the column before starting acquisition. The Time column in the group pane cannot be modified. If necessary, modify the scan time in the Scans pane.

ii. The **Total Scan Time (sec)** column indicates the sum of all the scans in each segment. See Figure 59. The total scan time also contains the stabilization time that occurs between each scan. This method, beginning 2.5 minutes into the GC run, gives you a complete set of scans every 0.342 seconds. At 5 minutes into the GC run, the scanning completely changes. Now the scans will repeat every 0.108 seconds until the GC run is complete. As the total scan time is a calculated value, it is not editable.

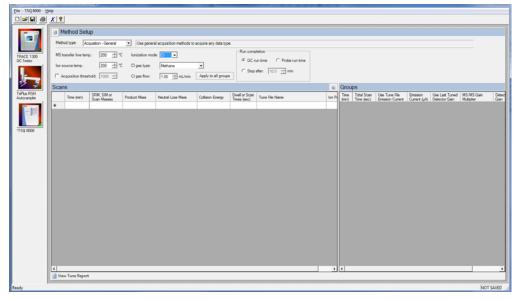


	100	Method Set	up										
MS service line temp:         200         C         Instantion mode:         B         Instantion         C Run completion           MS service lang:         200         C         C pas type:         Annual Mark         Instantion         C G c no sine         C Probe nu sine         C Step sine:         C Probe nu sine         C Step sine:		Method type: Ace	uistion General	• Use acc	suisition method to as	couire any scan mod	e or combination of	of scan modes					-
Time fireh         Scian Name         SML Station         Product Masse         Collation Firengy         Deater Scian         SML Wather (smal)         Tume File Name         Data Type         O 1 Resultion         Time (smal)         Time 2 20         SL           2 50         Full scin example         55:00         0 2         0 2         AutoTume_EI         Centroid         Nomal         22:0         0 3           5 500         SSM example         41:4         0:5;0:00         0:5;0:00         0:5;0:00         0:5;0:00         0:0;0:00 <th>k</th> <th>lon source temp.:</th> <th>200 - 1</th> <th>C Cliges type</th> <th>Annorsia (Port</th> <th>10.00</th> <th>@ GC ru</th> <th>n time C.I</th> <th></th> <th></th> <th></th> <th></th> <th></th>	k	lon source temp.:	200 - 1	C Cliges type	Annorsia (Port	10.00	@ GC ru	n time C.I					
2.50         Full scamework         50:00         0.2         Aut/Ture_EI         Central         2:00         0.5         0	S	cans										Gro	ups
2.50         Full scan seargie         50-300         0.2         Aut/Ture_II         Centrol         Normal         2.20         0.5           500         5KM seargie         41.44         0.05, 0.00         0.50, 0.50         Aut/Ture_II         Centrol         Normal         5.00         11           500         SKM seargie         232.9         257.91         20         0.05         Aut/Ture_II         Centrol         Wold (1.5 at 502.4)           6         0.21.31         152.43         32         0.55         Aut/Ture_II         Centrol         Wold (1.5 at 502.4)		Time (min)	Scan Name	SRM, SIM or	Product Mass	Collision Energy	Dwell or Scan	SIM Widths	Tune File Name	Data Type	Q1 Resolution	Time	Total
SM assight         AL4         OE5 00         050 000         AssTure_D         Cented         Nome           \$ 500         SPM assight         252.9         257.91         20         0.05         AssTure_D         Cented         Nome         \$ 500         110         \$ 500         110         \$ 500         110         \$ 500	e H							(amu)					0.342
500         SRM example         292.9         257.91         20         0.05         AutoTure_E         Centred         Wide (1.5 at 502)           P         292.41         152.53         32         0.05         AutoTure_E         Centred         Name	1	2.00				-		0.50, 0.50					0.108
▶ 282.51 152.53 32 0.05 AutoTure_E Centred Normal	1	5.00	SRM example	292.9	257.91	20	0.05			Centroid	Wide (1.5 at 50%)		and a second
				262.91	192.93	32	0.05	1		Centroid			

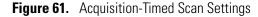
- iii. Use the **Chrom Filter On** column to enable the chromatographic filter. This filter smooths spectral data as it is acquired, which may increase the signal-to-noise ratio by a factor of two or more. The chromatographic filter is most useful when at least four full scans are acquired across a GC peak. This setting is typically left on.
- iv. Use the **Chrom Filter Peak Width** column to set the peak width to match the width of the GC peak (in seconds). If the peak width is set too large, signal intensity may be reduced. The default value is 1 s.
- v. Use the **Filament On** column to turn the filament on and off in the selected segment. Turning off the filament increases the lifetime of the filament and keeps the ion source clean longer. However, no data is collected. Use this column if you had analytes eluting before the solvent peak. You can create a segment to turn off the filament during the solvent peak to preserve the filament.
- vi. Use the **Emission Current** column to set the emission current used during the acquisition. For optimal analytical performance and stability, use the emission current at which the system was tuned. However, if you want to use a different emission current, clear the **Use Tune File Emission Current** check box and enter a value in the **Emission Current** (μA) column. A high emission current will lead

to the production of more ions, but the interaction of too many ions in the source can cause a degradation in the resolution and signal. The margin of error is  $\pm 0.5 \ \mu$ A.

- vii. Use the **Use Last Tuned Detector Gain** column to indicate that you want to use the detector gain set in *TSQ 8000 Autotune*. If you do not need to use the gain set in *TSQ 8000 Autotune* or saved to the instrument in *TSQ 8000 Manual Tune*, then you can set the gain manually. Higher gains give larger signals, but may shorten the lifetime of the detector when concentrated samples are detected.
- viii. Use the **CI Gas Flow** column to set the flow rate of your reagent gas. Click the **Apply to All Groups Button** at the top of the method editor. The single value at the top of the method is sent when initializing the MS with the method.
- Figure 60. Acquisition-General CI Settings



- ix. The **MS/MS Gain Multiplier** column only applies to groups with MS/MS events. The MS/MS gain multiplier only applies if the **Use Last Tuned Detector Gain** column is selected. The default MS/MS Gain Multiplier is 7 and is intended to give similar signal levels as a SIM scan on the precursor ion. If you enter a specific gain, the MS will use that gain for the SRM scan without the multiplier.
- 4. (For Acquisition-Timed methods only) In the Scans pane, do the following:



H Untitled - Thermo Xcalibur Instrument Setup	
File TSQ 8000 Help	
Method Setup	
Method type: Acquisition - Timed    Use timed acquisition methods to acquire timed SPIM or SIM data.	
AL/AS 1310 MS transfer line temp: 250 - C Ionization mode:	
kon source temp: 200 🕂 1C Cl gas type: Methane 💌	
Cligas flow: 1.00 🛨 mUlmin	
Scans	
TRACE 1300 Series GC Instrument settings	Scan settings
Tune file Tune file (*): [AttoTune_PCI	Peak width Min. baseline peak width: 3.0 th sec Resulting total scan time: 0.300 sec
Tune File (-) Auto Tune NCI C Use specified emission current, 50 (µA)	Desired scars per neal: 10-4 SRM/SM Time: 0.300 sec
Detector gain SRM Resolution	Lovest dvell time; 0.001 sec
TSQ 8000 G Use last funed detector gain X 70 th	Full scan     Constraints of the scan with mass range:      S0-550     Constraints of the scan with mass range:     S0-550     Constraints     S0-550     Constraints     S0-550     Constraints
C Use specified detector gain: 21E-006 Precursor (Q1) Normal	Full scan time: 0.003
Product (Q3): Nomal	Start time: 2.00 - min. End time: 10.00 - min. High priority multiplier: 10 -
Timed scan type: SRM	
21-21-21-26 × 28-20 回 ③ N 4 → N	
Name RT Ion Polarity Window (min) Mass Product Mass	Collsion Energy
•	

- a. Use the Tune File to select a tune file or files to be used for this scan. If you are using EI, only the Tune File(+) pull-down menu is available. If you are using negative CI, select a tune file from the Tune File(-) pull-down menu. Choose AutoTune\_NCI to use the most recent negative CI automatic tune file you created in Chapter 3, "Tuning." If you are using positive CI, select a tune file from the Tune File(+) pull-down menu. Choose AutoTune\_PCI to use the most recent positive CI automatic tune file you created in Chapter 3, "Tuning." If you are using positive CI, select a tune file from the Tune File(+) pull-down menu. Choose AutoTune\_PCI to use the most recent positive CI automatic tune file you created in Chapter 3, "Tuning." The software defaults to the most recent AutoTune\_NCI or AutoTune\_PCI tune file you created.
- b. In the Detector Gain area, set the detector gain. Select the Use Last Tuned Detector Gain option to indicate that you want to use the detector gain set in TSQ 8000 Autotune. If you do not need to use the gain set in TSQ 8000 Autotune, then you can set the gain manually. Higher gains give larger signals, but may shorten the lifetime of the detector when concentrated samples are detected. To manually set the detector gain, select the Use Specified Detector Gain radio button and enter the desired value in the Detector gain box.
- c. Use the **Emission Current** box to set the emission current used during the acquisition. For optimal analytical performance and stability, use the emission current at which the system was tuned. However, if you want to use a different emission current, select the **Use Specified Emission Current** radio button and enter a value in the **Emission Current (μA)** box. A high emission current will lead to the production of more ions, but the interaction of too many ions in the source can cause a degradation in the resolution and signal. The margin of error is ± 0.5 μA.
- d. In the **SRM Resolution** box, check the **Set Resolution for Each Unique Transition Scan** checkbox if you want to adjust the Precursor (Q1) Resolution or Product (Q3) Resolution for individual analytes. If this box is not checked, the settings apply to all compounds in the method.
  - i. Use the **Precursor (Q1) Resolution** column to set the full width at 50% (FWHM) of the precursor mass for SRM scans. The combo-box options are

**Normal, Wide (1.5 FWHM)**, or **Widest (2.5 FWHM)** for each scan after a precursor mass has been entered. The default setting is Normal, which uses the tuned resolution values from the tune file specified in the **Tune File Name** column. Selecting Wide or Widest increases the precursor peak width to 1.5 and 2.5 amu FWSM respectively. Opening the precursor peak width increases the ion signal into the collision cell, which might improve the sensitivity of your method; however, it may also increase the background noise level.

ii. Use the Product (Q3) Resolution column to set the full width at 50% (FWHM) of the product mass for SRM scans. The combo box options are Normal, Wide (1.5 FWHM), or Widest (2.5 FWHM) for each scan after a product mass has been entered. If the product mass is removed from a scan to run a SIM or FS scan event, the Product (Q3) Resolution column returns to the default value of Normal, which uses the tuned peak width at 10% or 50% as specified in the Tune File Name column. Selecting Wide (1.5 FWHM) or Widest (2.5 FWHM) increases the product ion peak width to 1.5 and 2.5 amu FWHM respectively, resulting in an increase of ions reaching the detector. The increase in ions at the detector may improve the sensitivity of your method; however, it may also increase the background noise level, as well as introduce isotopic interference from +/- 1 amu ions.

#### Figure 62. SRM Settings

H Untitled - Thermo	Xcalibur Instrument Setup		
File TSQ 8000 Help			
	Method Setup		
	Method type: Acquisition - Timed Use timed acquisition methods to acquire timed SRM or SIM data.		
Al/AS 1310	MS transfer line temp: 250 📩 'C Ionization mode: 🗘 💌		
	Ion source temp: 200 📩 'C Cligas type: Methane 💌		
	Cligas flow: 1.00 m mUmin		
TRACE 1300	3 Scans		
Seciel GC	Inter Set         Ensistion current           Trace Set ()         AssTrue_JCC)         Ensistion current           Trace Fel (>) (AssTrue_JCC)         C         Description           Description         C         Description         SNI Resolution           Description         Z         SNI Resolution         Point unit transition current         SNI Resolution           C         Use specified director gain         Z         Today         Point unit transition current         Point unit unit transition current         Point unit transition current         Point unit transition current         Point unit unit unit transition current         Point unit unit unit unit unit unit unit u	Scient ending         Persuiting           Michaelide peak width:         10 ±3 sec           Desired scients per peak:         10 ±3 sec           Cleared scients per peak:         10 ±3 sec           Full scient         0.000 sec           Lowerd scients per peak:         0.000 sec           Full scient with mass range         0.000 michaeling scients per peak scients peak sc	
B.,	Timed scan type (55M ) (Lick to external tipe 11-17-36 X (So (So (So (K + K H))) New (RT to Falarty (Window (Intro))) (Mass Product Mass New (RT to Falarty (Window (Intro))) (Mass Product Mass New (RT to Falarty (Window (Intro))) (Mass Product Mass New (RT to Falarty (Mass Product Mass	a Coleon Swegy (Prouver (21) Residion ν	

- e. As appropriate, use the options in the **Scan Settings** area to further adjust your method.
  - i. Under **Peak Width**, you can change the minimum baseline peak width and desired scans per peak. These values are used to calculate the SRM/SIM Time. The minimum baseline peak width should be set roughly to the shortest chromatographic peak time in your analysis.

ii. Under **Full Scan**, you indicate if a Full Scan is to be run along with SIM and SRM. The Mass Range, Scan Time, Start and End Time can be set after the Use Full Scan button is selected. The Full scan time will reduce the SRM/SIM time without increasing the total scan time. If you only want to use full scan for part of your method, you can enter full-scan start and end times.

**Tip** While the system software will calculate the minimum dwell times from your method, you may change it. If you have a method with many transitions, setting the minimum dwell time to more than 0.001 s may improve sensitivity in complex methods.

- iii. Select the Allow for Asymmetric Acquisitions check box to add extra time to the beginning or end of an acquisition window without affecting other timing in your scan. When you select this option, Pre-width and Post-width columns are added to your method. Enter the extra times in these columns.
- iv. Select the Allow Dwell Time Prioritization check box to increase the dwell times for selected scans. The choices for each scan are Normal or High. Giving a scan high priority increases its dwell time by the value you set in the High Priority Multiplier box.
- f. Select **SIM** or **SRM** in the **Timed Scan type** list. If you want to link to an external method, select the **Link to External File** check box. You may link to a .csv or .xml method file.

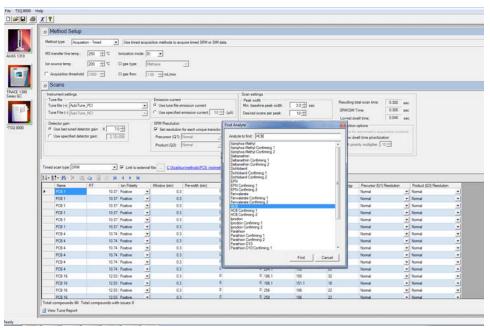
**Note** In order to edit the scans within the TSQ 8000 method editor, clear the **Link to External File** check box.

Figure 63. Linking to External Method Files

X ?									
Method Setup									
Method type: Acquistion - 1	Timed • Use timed	acquisition methods to acquire timed SF	Mar CM date						
- Performent		acquisition methods to acquire smed or	on or print uses.						
MS transfer line temp: 25	0 🕂 °C Ionization mod	fe: 🚺 💌							
Ion source temp.: 20	0 🕂 'C Cigas type:	Methane							
Acquisition threshold 10		1.00							
1	U _ Crgas now:	1100 3 40444							
<ul> <li>Scans</li> </ul>									
Instrument settings				Scan settings					
Tune file Tune file (+): AutoTune_PCI		Emission current     C Use tune file emission current		Peak width Min. baseline peak width:	3.0 1 sec	Resulting t	stal scan time: 0.300	sec	
						SRMSM	Time: 0.300	sec	
Tune File (-): AutoTune_NCI			and so they	Desired scans per peak:	10 - 2	Lowest dv	ell time; 0.046	500	
Detector gain		SRM Resolution		Full scan		Acquisition	options		
Use last tuned detector g		Set resolution for each uniq	ue transition scan	Use full scan with mass			asymmetric acquisition wind	śows	
C Use specified detector ga	ain: 2.1E+006	Precursor (Q1): Normal	~	Full scan time: 0.083 🛨			ell time prioritization		
		Product (Q3): Normal	*	Start time: 2.00 min.	End time: 10.00-+-m	in. High pric	rity multiplier: 10 🛨		
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21-21-38 × 105 Pa	<b>BON4PN</b>	ternal file <u>CSXcaliburimethoda/PS</u>	31. instructions				Processor (71) Baseletion	Product O'D Resolut	-
	<b>BON4PN</b>	derral Sie	31. instructions		Product Mass 152	Collsion Energy	Precursor (Q1) Resolution Nomal	Product (23) Resoluti	or
21-21-33 × 125 42 Name RT	In Polarty	dernal file <u>C.X.caliburimethodo/PC</u>	20. instruction, carry	h (nin) Mass	Product Mass	Collision Energy			0
21-21-23 × 125 42 Name RT	Ion Polarity 10.37 Positive	dernal file <u>G.Xcaliburimethods/PC</u> Window (min) Pre-width (m	28 instruction carrows in) Post widt 0	h (min) Mass 0 153.1	Product Mass 152	Collsion Energy	Nomal	Nomal	0
21-21-33 × 125 Ca Name RT PCB 1 PCB 1	In Polarty 10.37 Postive 10.37 Postive	terral fileC_Xcalibarimethods/PC Window (mn) Pre-width (m v 0.3 v 0.3	<ul> <li>instruction cax</li> <li>Post widt</li> <li>0</li> <li>0</li> <li>0</li> <li>0</li> <li>0</li> </ul>	h (min) Moss 0 153.1 0 188.1	Product Mass 152 126 8	Collsion Energy 12 32	Nomal Nomal	Nomal     Nomal	ior
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21-21-46 × 10, 42 Name RT PCB 1 PCB 1 PCB 1 PCB 1 PCB 1	Image:	Window (sm)         Pre-with (sm)           Window (sm)         Pre-with (sm)           Window (sm)         Pre-with (sm)           Window (sm)         Pre-with (sm)           Window (sm)         Pre-setth (sm)           Window (sm) <t< td=""><td>28 instruct, cay           in)         Post widt           0         0           0         0           0         0           0         0           0         0           0         0           0         0           0         0</td><td>h (Her) Mass 0 153.1 0 188.1 0 188.1 0 188.1 0 188.1 0 190.1</td><td>Product Mass 152 126.8 150.9 152.3 153.1 152.1</td><td>Collision Energy 12 32 34 22 12 22 22</td><td>Nomal Nomal Nomal Nomal Nomal</td><td>Nomal     Nomal     Nomal     Nomal     Nomal     Nomal     Nomal     Nomal</td><td>0</td></t<>	28 instruct, cay           in)         Post widt           0         0           0         0           0         0           0         0           0         0           0         0           0         0           0         0	h (Her) Mass 0 153.1 0 188.1 0 188.1 0 188.1 0 188.1 0 190.1	Product Mass 152 126.8 150.9 152.3 153.1 152.1	Collision Energy 12 32 34 22 12 22 22	Nomal Nomal Nomal Nomal Nomal	Nomal     Nomal     Nomal     Nomal     Nomal     Nomal     Nomal     Nomal	0
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21-21-30         None         R1           None         R1           P081         P081           P081         P081           P081         P081           P081         P081           P081         P081           P081         P081           P084         P084	Image: Construction         Image: Construction         Image: Construction           10.37         Positive         10.37         Positive	nerral file Collabornethold IS 02 03	Instructive           initial         Post widt           0         0           0         0           0         0           0         0           0         0           0         0           0         0           0         0           0         0           0         0           0         0	<ul> <li>(em)</li> <li>Mass</li> <li>153.1</li> <li>158.1</li> <li>158.1</li> <li>158.1</li> <li>158.1</li> <li>159.1</li> <li>159.1</li> <li>159.1</li> <li>159.1</li> <li>152.1</li> <li>152.1</li> <li>152.1</li> </ul>	Product Mess 152 126 8 150 9 152 3 153 1 152 1 152 1 150 150 7 152 1	Collsion Energy 12 32 34 22 12 22 12 22 14 20 12	Nomal Nomal Nomal Nomal Nomal Nomal Nomal Nomal		
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21-31-35         X         K         K         K           Name         R1         R2         R1         R2         R2	Image: Construction         Image: Construction           10.37         Posteve           10.34         Posteve           10.34         Posteve           10.34         Posteve           10.34         Posteve           10.34         Posteve	terrel file Coldensity inter-	Ite instantsh.csu           IPost widt           0	Mass           0         153.1           0         188.1           0         188.1           0         188.1           0         188.1           0         190.1           0         153.1           0         192.1           0         192.1           0         192.1           0         222.1	Product Mass 152 126 8 150 9 152.3 153.1 153.1 153.1 150.7 150.7 152.1 152.1 152.1 152.1 152.1 152.1	Collision Energy 12 32 34 22 22 12 22 14 20 14 20 12 22 8	Nomal Nomal Nomal Nomal Nomal Nomal Nomal Nomal Nomal		io
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21-21-30         X         Kare         R3           Name         R3         R4         R3           PGB 1         PGB 1         PGB 1         PGB 1           PGB 1         PGB 1         PGB 1         PGB 1           PGB 4         PGB 4         PGB 4         PGB 4           PGB 4         PGB 4         PGB 4         PGB 4           PGB 4         PGB 4         PGB 4         PGB 4	Image         Image         Image         Image           10.27         Postove         10.37         Postove           10.37         Postove         10.36         Postove           10.38         Postove         10.36         Postove           10.34         Postove         10.36         Postove           10.38         Postove         10.36         Postove           10.34         Postove         10.36         Postove	terrel fee Coldendersheel IS Voide (m) Presette (m) 03	It instructs.csc           an)         Post widt           0         0           0         0           0         0           0         0           0         0           0         0           0         0           0         0           0         0           0         0           0         0           0         0           0         0           0         0	Mass           0         155.1           0         185.1           0         186.1           0         186.1           0         186.1           0         195.1           0         195.1           0         195.1           0         195.2           0         192.1           0         222.1           0         224.1           0         195.1	Product Mass 152 126 8 150 9 152 3 153 1 152 1 153 1 53 1 54 1 55 1 55 1 55 1 55 1 55 1 55 1 55	Collision Energy 12 22 24 22 22 22 22 24 22 22 14 20 22 22 22 8 8 22 22 22 22	Nomal Nomal Nomal Nomal Nomal Nomal Nomal Nomal Nomal Nomal Nomal		ior
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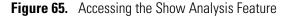
g. Either enter the analyte name in the Name column (referring to the analyte name) or, in your external file, enter the analyte name in the first column. You may also right-click this window to search for an analyte within your method. See Figure 64. This function is useful if you need to edit an analyte in a complex method.

Figure 64. Finding an Analyte in a Method



- h. In the **RT** column, enter retention times for SRM methods. The retention time is the time it takes an analyte to pass from the column inlet to the detector.
- i. In the **Window** (min) column, set the acquisition times. Smaller acquisition windows increase sensitivity but can cause you to miss your peak if set too small. Changing the window size only affects sensitivity if it reduces the number of compounds analyzed in a segment. If the windows do not overlap, you will not notice an improvement by reducing the acquisition window.
- j. In the **Mass** column, enter the precursor ion mass for SRM methods. For SIM methods, enter the mass of the ion you wish to monitor.
- k. In the **Product Mass** column, enter the product ion mass (for SRM methods). The product ion mass is the result of fragmenting the precursor ion with collision gas.
- 1. In the **Collision Energy** column, enter the collision energy (for SRM methods) in eV. The collision of a precursor ion with the collision gas at a particular energy fragments the precursor ions into product ions. By varying the collision energy, a single precursor ion can be fragmented into several product ions. An SRM transition is defined by a precursor ion being fragmented into a particular product ion mass using a certain collision energy.
- m. Use the **Ion Polarity** column if you are using CI mode to tell the instrument to generate positive or negative ions. Only use this column if you are using CI mode. In EI mode, this column should always be set to **Positive**.

n. Click the **Show Analysis** arrow (see Figure 65) to validate your method. A chart appears with all your analytes by name and in order of their retention times. See Figure 66.



ve: 0.300 sec 0.300 sec 0.046 sec : acquisition windows ritization sr: 10				
· (Q1) Resolution	Product (Q3) Resolution	Show a	analysis	<ul> <li>Show Analysis</li> <li>Button</li> </ul>
•	Normal			

#### Figure 66. Validating a Method

e: Acquistic	on - Timed 💌 Use times	d acquisition m	ethods to acquire timed S	RM or SIM data.									
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temp :	200 -1 1C Ci gas type:	Methane											
		,	_										
ion threshold:	1000 🛨 Ci gas flow:	1.00 📑	mL/min										
t settings					· Scan settings								
-			sion current		Peak width		Reading	total scan time:	0.300 sec				
(+): AutoTune,	JPCI		e tune file emission curre		Min. baseline peak width:	3.0 - Sec	SRMSIM						
(-): AutoTune,	_NCI	- CU	re specified emission cur	rent 50 🛨 (µA)	Desired scans per peak:	10 - 10							
gain		- SRM	Resolution				Lowest dr		0.046 sec				
ast tuned detect	torgain X 7.0 🛨		let resolution for each uni	que transition scan	Full scan	a ranger (50-550	Acquisition	r asymmetric acqui					
pecified detect	tor gain: 2.1E+006		ursor (Q1): Nomal	*	Full scan time: 0.003			vell time prioritizati					
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o. Use the scroll bar on the right to view all your analytes. You may also click the ladder icon walk your analytes: have the software automatically run through your list of analytes. Click the ladder icon again to stop the process at any time.

**Tip** If your SRM or SIM windows are too congested to achieve the total scan time at the minimum dwell time, the event will be highlighted in orange or red. This warning indicates that you might get fewer scans across the peak than you want. To correct this, reduce the desired scans across the peak, reduce the acquisition windows, or reduce the minimum dwell time to correct. 5. To import a Thermo Scientific<sup>™</sup> ISQ<sup>™</sup> Series or TSQ Quantum<sup>™</sup> Series MS method, choose **TSQ 8000 | Import MS Method**. See Figure 67.

**Note** This will only import the MS part of the method. You must set the GC and autosampler parameters.

Figure 67. Importing Methods

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	S	cans												
1300 C	Instr	ument settings						Scan settings						
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		e file (+): AutoTune,				tune file emission current		Min. baseline	peak width:	3.0 🛨 sec	SRM/SIM			sec
	Tun	e File (•): AutoTune	NCI		Use C Use	e specified emission current	50 : (씨)	Desired scar	is per peak:	10 🛨	Lowest de			sec
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6. Choose **TSQ 8000 | Import Timed Scans** to import .csv or .xml files of previous methods. The software will only load files in valid formats. If your file is not valid you will receive an error message and will not be able to import the file into the Method Editor.

Tip Export a timed scan list to see an example of a valid format.

- 7. Choose **TSQ 8000 | Append Timed Scan from File** to add scans from previous methods to the end of your open scan list. As above, you may import .csv or .xml files. The software will only load files in valid formats. If your file is not valid you will receive an error message and will not be able to import the file into the Method Editor.
- 8. Choose **TSQ 8000** | **Export Timed Scans** to export your method as a .csv file. If you prefer editing your methods in spreadsheet applications, you may want to use this option.

9. Choose **TSQ 8000** | **Create Compound Data Store Export File** to prepare your file for the compound data store in the TraceFinder application. See Figure 68.

Figure 68. Creating Compound Data Store Export Files

Create compound datastore export file	
Analyte List	Devide all the an an instance in the sector in the
	Double click on an ion to make it the quan ion for the compound.
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m/z: 170.96 > 100	
m/z: 170.96 > 136 (Q)	
🚊 Tribromoanisol Confirming 1	
m/z: 346 > 303 (Q)	
Tribromoanisol	
m/z: 346 > 331 (Q)	
🚊 Sulfotep Confirming 2	
m/z: 322.02 > 146 (Q)	
En Sulfotep Confirming 1	
m/z: 322.02 > 174 (Q)	
E Sulfotep	
m/z: 322.02 > 202 (Q)	
E- HCB Confirming 2	
m/z: 283.81 > 212 (Q)	
E- HCB Confirming 1	
m/z: 283.81 > 214 (Q)	
E HCB	
m/z: 283.81 > 249 (Q)	
E Parathion-D10 Confirming 2	
m/z: 301.14 > 83 (Q)	
E Parathion-D10 Confirming 1	
E Parathion-D10	
m/z: 301.14 > 115 (Q) ⊡ Parathion Confirming 2	
I III Paratnion Contiming 2 IIII IIII IIIIIIIIIIIIIIIIIIIIIIIIII	Create Export
⊡ Parathion Confirming 1	File <u>Cancel</u>
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1 ····· 111/2. 231.03 > 31 (k)	

- a. Double-click one ion of each analyte to set it as the quantitation ion.
- b. Click **Create Export File** to save your file in the *TraceFinder* compound data store. See the *TraceFinder* documentation for additional information.
- 10. Choose **TSQ 8000** | **Create Segment List From Timed Scan List** to import a general acquisition method.
- 11. Choose **TSQ 8000** | **View Tune Report** to view the latest tune report the method will use. Choose **Report Options** to open the Report Options dialog box (see Figure 69) and add identifying information to the tune report.

of 1 🕨 🕅 Whole Page 🔸	
	TSQ 8000 Tune Results Instrument Name: TSG 8000 Tune Results Tune Compared - Fillian 6 15 21:25 2012 Tune File: Last, DJ, 2012-01-05-53-21 21 stature Tune Type: Last Context Examp Tune Type: Last Seved Tune)
	mm 2 10 02 H 2 105 0 00 H 2 10 10 00 H 2 10 10 H 2 10
	Sgrature One Prind on Wednesder, January 11, 2012 Page 1 of 1

#### Figure 69. Configuring the Tune Report Options

### **Creating a GC Method**

You can create a method for the TRACE 1300/1310 Series GC from the TSQ 8000 Method Editor window.

#### ✤ To create a method for the TRACE 1300/1310 Series GC

- 1. Click the GC icon in the left pane.
- 2. Click the **Oven** tab to set the oven temperatures. See Figure 70. There is always at least one temperature and time in any GC temperature program. In the Initial row, enter the initial temperature, which must be 4 °C (7 °F) above room temperature and less than the maximum operating temperature of your GC column. If you set the initial temperature to a value below this limit, the GC will not reach the initial temperature. If you set the temperature above the limit, the GC column will be damaged. You can set the initial hold time to a value between 0 and 999.99 minutes. The typical initial temperature is at least 10 °C (18 °F) above the boiling point of your sample solvent, and the initial time is usually long enough for the solvent to move through the column.

ile TRACE 1300	Help X ?
TRACE 1300 GG Series	Oven         S/SL - Front         PTV - Back         FID - Front         ECD - Back         Run Table           60
TriPlus RSH Autosampler	0         0.2         0.4         0.6         0.8         1         1.2         1.4         1.6         1.8         2         2.2         2.4         2.6         2.8         3           Ramps         Data acquisition time
	#         Rate (°C/min)         Temperature (min)         Hold Time (min)         © Oven run time         3.00 min         © Specific time:         10.00 min
ISQ 8000	Instal         40.0         1.00           1         10.0         50.0         1.00           Max. temperature: 350.0 °C Cryogenics enable: Prep-run timeout: 10.00 min
	Equilibration time: 0.50 min Ready delay: 0.50 min

Figure 70. GC Oven Settings

- 3. Select a maximum of 32 temperature ramps, each with its own ramp rate, final temperature, and hold time. Each temperature ramp begins at the previous ramp's temperature after the hold time has expired. A typical program has one or two ramps. The GC temperature profile is the primary method for separating your analytes from each other, the solvent, and the matrix. Your temperature profile must be optimized for your analysis needs.
- 4. You can also select the maximum allowed oven temperature, which should be set to the maximum temperature allowed by your method, *not* the maximum allowed by your column. The maximum temperature allowed by the GC is 450 °C.
- 5. Select the prep-run timeout. The prep-run timeout is the maximum amount of time that the GC will wait before it gives up on an injection. As an example, with the default value of 10 minutes, if the GC is ready to receive an injection, but does not receive it after ten minutes, the GC will stop waiting. This usually occurs in case of an error.
- 6. Set the equilibration time. The equilibration time is a delay between when the GC is at temperature and when the GC reports as being ready. The equilibration time is typically set to 0.5 minutes.



**CAUTION INSTRUMENT DAMAGE.** Be sure not to overheat the GC column or it may contaminate the TSQ 8000 instrument.

- 7. (Optional) Enable the cryogenic option to cool the oven. If this option is selected, then the minimum allowed temperature in a temperature ramp will fall from 0 to -99 °C (32 to -146 °F). The GC also allows the use of a post-run column cleaning. This is not typically used because the material that is purged from the column in this step would go into the TSQ 8000, which can lead to contamination. If you want to use this feature, set the GC oven temperature, as well as the amount of time to remain at that temperature after the analytical run is complete. You can also set the amount of pressure used to push the carrier gas through the column. Refer to the *TRACE 1300/1310 Series GC User Guide* for more information.
- 8. If you have a split/splitless inlet (SSL) injector, click the **S/SL-Front** or **S/SL-Back** tab to configure the injector port settings. In the inlet area, do the following:
  - a. Select the **Temperature** check box and set the temperature to at least 10 °C (18 °F) lower than the boiling point of your least volatile analyte for a splitless injection. The material should be injected into the port to vaporize and move into the GC column quickly. Higher temperatures can lead to thermal decomposition of some analytes, so you must optimize the injector temperature for your analysis. The SSL temperature can be set up to 400 °C (752 °F). A typical value would be 250 °C (F).

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	<u>X</u> <u>8</u>	1474 1747 1747 1747 1747 1747 1747 1747
TRACE 1300 GC Series TriPlus RSH Autosampler TSQ 8000	Oven       \$7/5L - Front       PTV - Back       FID - Front       ECD - Back       Run Table         SVSL mode:       Splt       Image: Constant Pressure       Image: Constant Pressure       Image: Constant Pressure         Inlet       Image: Constant Pressure       Image: Constant Pressure       Image: Constant Pressure       Image: Constant Pressure         Split ratio:       33.3       Splitless time:       1.00 min         Surge       Surge pressure:       5.00 kPa         Surge duration:       0.00 min         Septum purge       Formularity         Purge flow:       5.0 mL/min         Constant septum purge:       Formularity         Stop purge time:       0.00 min	Backflush Backflush enable: Backflush start time: 1.50 min Backflush duration: GC run time Specific duration: C 1.50 min
	۲ III	
Ready		

Figure 71. Configuring the Injector Port Settings on the GC

- b. Select the **Split Flow** check box to dilute high concentrations of sample. The split flow is the amount of gas that is swept through the injector to the exhaust port. Higher values will give more dilution. The split flow reduces the amount of contamination that builds up in your system. The split flow ratio is the ratio of the split flow to the carrier gas flow. It is effectively the dilution ratio of the sample. This setting is typically turned on and set to a flow of 50 mL/min. However, more carrier gas is used, so for your analysis, lower split flows may be more acceptable. If you set the split ratio, the software calculates the correct split flow. The reverse is true also.
- c. For best results, use the septum purge, which means additional carrier gas will go through the injector. The default purge flow value is 5 mL/min. This reduces the buildup of contaminants in the injector, on the column, and in the TSQ 8000 instrument. If you perform a splitless injection, even if the split flow is set, the split flow turns off for the splitless time. The septum purge turns off for the stop purge time. After these times, the split flow and septum purge are reactivated.
- d. Set the carrier mode to **Constant Flow, Constant Pressure, Programmed Flow**, or **Programmed Pressure**. The gas flow and the oven temperature work together to determine how well the analytes are separated and how long the analysis will take. If you use constant pressure, as the column is heated in the oven, the flow rate will fall as the viscosity of the carrier gas increases. If you use constant flow, the carrier gas pressure will increase as the column temperature increases to keep the flow constant. Constant flow is more common. Typical flow rates are 1-3 mL/min. The pressure depends on the column length and internal dimensions, so there is not a typical value. Because the outlet of the column is in the TSQ 8000 instrument, which is under vacuum, be sure the vacuum compensation is on to ensure accurate flow rates.
- e. (Optional) Under Carrier Pressure, select the check box to use programmed flow or programmed pressure modes. In these modes, you may have up to three flow rates or pressures to use during an analytical run. This feature is not commonly used, but may be necessary if you have a particularly challenging separation.
- f. In an effort to reduce the amount of carrier gas used, select the **Carrier Gas Saver** option under Carrier Gas Options. When used, the split flow is reduced to the gas saver flow after the gas saver time. It is not recommended to use a flow of less than 20 mL/min because contaminants can build up in the injector, column, and TSQ 8000 instrument, which can affect the system performance.
- g. Finally, if your analysis requires a higher flow to quickly sweep the analytes into the column, which may be needed with high temperature injectors and thermally labile compounds, use the surge pressure to increase the column flow for the surge duration time.

9. If you have a Programmable Temperature Vaporizer (PTV), click the PTV-Front or PTV-Back tab to configure it. See Figure 72. The PTV is a low thermal mass injector that allows the instrument to rapidly heat or cool the inlet. You can use the PTV page to program the temperature of the injector. Refer to the GC documentation for details about the PTV or other types of injectors.

Figure 72. PTV Settings

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File TRACE 1300	Help	
	<u>X</u> 8	
TRACE 1300 GC Series	Oven     S/SL - Front     PTV - Back     FID - Front     ECD - Back     Run Table       PTV mode:     Split <ul> <li>Carrier mode:</li> <li>Constant Pressure</li> <li>Carrier pressure</li> <li>Pressure:</li> <li>Toto mu/min</li> <li>Split ratio:</li> <li>6.7</li> <li>Splitess time:</li> <li>0.00 min</li> <li>Pressure:</li> <li>Toto mu/min</li> <li>Pressure:</li> <li>Toto mu/min</li> <li>Splitess time:</li> <li>Toto mu/min</li> <li>Toto mu</li></ul>	Injection phases Press. Rate Temp. 1 kPa "C/sec "C Injection 70.00 Transfer 210.00 14.5 200
TriPlus RSH Autosampler	Surge         Surge pressure:       5.00 kPa         Surge duration:       0.00 min         Septum purge       Vacuum compensation:         Purge flow:       5.0 mL/min         Constant septum purge:       Image: Carrier gas saver:         Stop purge time:       0.00 min	Evaporation phase: Transfer temp. ( Cleaning phase: Post-cycle temperature: Ramped pressure: Show Chart
Ready	< <u> </u>	,

10. Click the **Run Table** tab to configure how to control external valves and devices. Refer to the GC documentation for more detailed information.

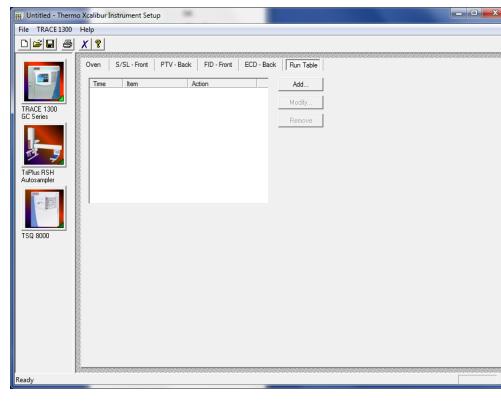


Figure 73. Configuring the GC Run Table

**Note** The user interface reflects the current configuration of your GC. If you add, remove, or change inlets or detectors, redo your instrument method according to the new GC configuration.

- 11. To add an inlet or detector to the Method editor user interface:
  - a. Attach the inlet or detector to the GC. Refer to the GC documentation for instructions.
  - b. Add the inlet or detector to the current instrument configuration. Choose **Start** | All Programs | Thermo Foundation 3.0 | Instrument Configuration.
  - c. In the **Configured Devices** pane, click the **TRACE 1300** or **TRACE 1310** icon. Click **Configure**.

1	Thermo Foundation Instrument Configuration	83
	Device Types :	
TRACE 1300 GC Configur	ration 💌	
🖉 General 🔽 Inlets	S 🔍 Detectors 🛛 🔶 Auxiliary 🔪 Configured Devices:	
Detectors and Data Ch		
Detector typ	pe Makeup gas Channel name (Hz)	
Front: NPD	Nitrogen     NPD     10     TRACE 1300 GC     TriPlus RSH	
(None)	▼ Nitrogen ▼ FID 10 ▼ Series Autosampler	
Options ECD NPD Line frequ TCD	Hz	
Get	OK Cancel Help	
	TRACE 1300 GC Series	
	Add >> Configure	
	Done	

Figure 74. Adding a GC Inlet or Detector to the Method Editor

- d. In the TRACE 1300/1310 GC Configuration dialog box, select the **Inlets** or **Detectors** tab as appropriate.
- e. Click **Get**. The software automatically detects the attached modules. Select the gases as appropriate for your setup. The default Channel names are Channel 1 and Channel 2. You may want to change them to something more descriptive. Your hardware is now configured.

12. When you are finished creating methods for each component in your GC/MS system, choose **File | Save As...** from the main menu or click the 📮 button.

File TSQ 8000 Help							
New	Ctrl+N						
Open	Ctrl+O				ちろち ひろうち ひろうちょうちょう		5057Q
Save	Ctrl+S						
Save As		isition method to acquire	timed SRM or SIM				
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1 SRM_update.meth		_ · · ·		Scan setting			_
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3 PestSRM_No_Precolumn_Hermes_Method_Adjust.meth			d emission current: 50 -	-	ns per peak:	6 ÷	
4 QuantumImport.meth		Resolution		Full scan	,	_	
Exit				Use full	scan with mass rang	e: 50-550	
C Use specified detector gain: 2	1E+006	Q1 Resolution:	lomal _		ne: 0.049 🕂 sec	Positive 👻	
				Start time:	2.00 🛨 min. End	time: 10.00 🛨 min	•
							_
Timed scan type: SRM	Link to ex	ternal file					
Name RT	IonPolarity	Window (min)	Pre-width (min)	Post-width (min)	Mass	Product Mass	Colli Ene
*		-					

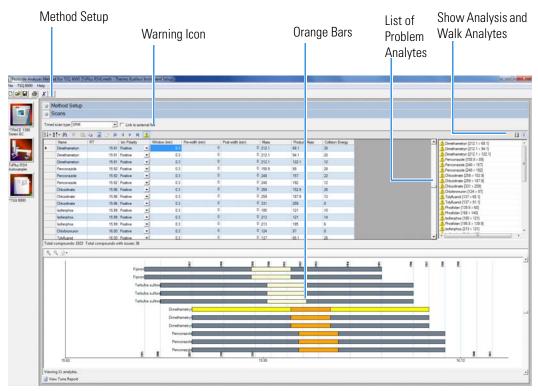
Figure 75. Saving a Method

### **Validating and Troubleshooting Methods**

If your method will not validate or save, you can fix it by reducing the desired scans across the peak or reducing the acquisition windows.

If your method will not validate, a yellow warning icon appears in the Scan pane: 🛕

Also, when you click on the yellow warning icon the **Scans** pane, you will see a list of the analytes on the right-hand side of the method editor. The **Analysis** pane will open along with the list of analytes with warnings. Selecting any of the analytes in the list will take you to its location in the Analysis pane. The Analysis pane can also be opened separately by clicking the **Show Analysis** button on the far right of the scans pane so that the arrows point down. See Figure 76.

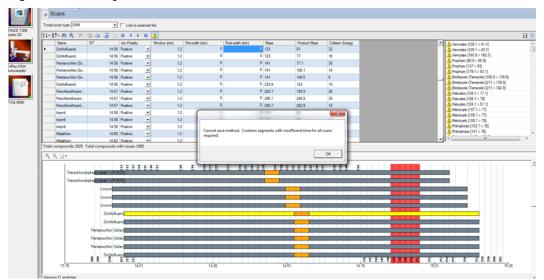


#### Figure 76. Viewing the Analytes

If there are only orange bars, the method will still validate, but you might not get the desired scans per peak.

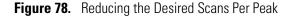
If red bars are present, however, the method will not validate and cannot be saved. An error message will appear if you try to save the method. See Figure 77. This occurs when the total scan time in a segment exceeds the time allowed for the segment. To correct this error, reduce the number of scans inside the highlighted segment.

Figure 77. Viewing Red Error Bars



#### ✤ To decrease the desired scans per peak

• In the **Scan Settings** area, reduce the number of scans in the **Desired Scans Per Peak** box. See Figure 78.



Scan settings         Peak width         Min. baseline peak width:         Joint for the set of the	
--	--

#### ✤ To reduce the acquisition windows

- 1. In the Scans pane, go to the first problem analyte and click on the **Window (min)** column. See Figure 79.
- 2. Decrease the time.
- 3. While holding down the shift key, click the last problem analyte in the list.
- 4. Go back to the first analyte you selected, right-click, and choose Fill Down.
- 5. If the warning icon still appears, and your method allows it, reduce the acquisition windows further.

**Figure 79.** Reducing the Acquisition Windows



Method Setup     Scans										
Timed scan type: SRM	• F Link to ex	durral file								
21-21-25 × 15.40 9										 
Name RT	Ion Polatty	Window (min)	Pre-setable (mint)	Fost width (min)	Mass	Product Mass	Collision Energy			 
<ul> <li>Dmethanelryn</li> </ul>	15.91 Postive		12	D	0 212.1	63.1	30			
Dimethanetryn	15.91 Postive		0.2	0	0 212.1	94.1	20			
Direthanetyn	15.91 Postive	-	12	0	0 212.1	122.1	12			
Penconazole	15.92 Postive	-	0.2	0	0 158.9	89	28			
Fenconazole	15.92 Feative	-	12	0	0 243	157	22			
Penconazole	15.92 Postive	-	1.2	0	0 248	192	12			
Orkozoknate	15.96 Positive		2	0	0 259	152.9	26			
Chiozolinate	15.96 Postive	-	0.2	D	0 259	187.9	12			
Oxiszolinate	15.96 Postive	-	2	0	0 331	259	1			
ladenphos	15.99 Postive	-	2	0	0 185	121	10			
laoferphos	15.99 Postive		1.2	D	0 213	121	14			
laoferphos	15.99 Postive	-	2	0	0 213	185	6			
Chlotromuron	16.00 Postive		12	0	0 124	37	8			
Totyluanid	16.00 Postive	•	2	0	0 137	45.1	21			
Total compounds: 1925 Total co	mpounds with issues: 0									
( C C ) -										
		Figroni				£ 5 1	2 2 2	£	夏 史	

When your method validates, the warning icon, the list of problem analytes, and the red and orange bars disappear from the method editor. See Figure 80.

1									
J Method Setup									
3 Scans									
Timed scan type: SRM	• F Link to e	sternal file							
1-11-18 4 ×	4 B 0 H 4 +	н			1000	11:			
Name RT			w (min) Pre-width (min)	Post-width (min)	Maes	Product Mass	Collision Energy		
Terbulos sulfone	15.88 Postive	-	0.3	0	0 199	143	10		
Dimethanetryn	15.91 Postve	-	0.2	0	0 212.1	68.1	30		
Dimethametryn	15.91 Positive	-	0.2	0	0 212.1	94.1	20		
Dimethametryn	15.91 Postive	-	02	0	0 212.1	122.1	12 28		
Personazsie	15.92 Positive 15.92 Positive	-	02	0	0 158.9	89	28		
Personazsie Personazsie	15.92 Postive	-	02		0 248	197	12		
Orinzolinate	15.96 Postive	-	02	0	0 259	152.9	26		
Oriozolivate	15.96 Postive	1	02	0	0 259	187.9	12		
Chiczokrate	15.96 Postve		0.2	0	0 331	259	8		
isoferphos	15.99 Postive	-	0.2	0	0 185	121	10		
laderphos	15.99 Postive		0.2	0	0 213	121	14		
Isolenphos	15.99 Postive	-	0.2	0	0 213	185	6		
Chiptromuron.	16.00 Postive	-	0.2	.0	9 124	97	8	8	
Total compounds: 1925 Total	compounds with issues: 0	2							
A A 3+									
1				3		8 7	8		8
	Terbulos sultone					-	Ale .		2
			Direthanetry						
			Dimethametry						
			Directionetry						
			Percor	4204			1	100 00	
			Percor	azole			-		
			Percor	and a			the state of the s		
				Chiczolina	_				
				Chiceolina					
				Chlozofinat					
			÷.		solenphon				
	R	C	R.	N A A	15.87	R			15.12
15.62									

Figure 80. Validated Method

You may now save the method as described earlier.

# **Optimizing Your Method**

If you are not getting the expected results from your method, you can modify it using the following procedures for better results.

#### Contents

- Changing the Chromatographic Separation
- Finding the Best Way to Make an Injection
- Improving the Way You Prepare Samples
- Changing the Dwell Time or Scan Rate
- Narrowing the Mass Range
- Adjusting the Transfer Line and Ion Source Temperature
- Optimizing an SRM Method
- Optimizing an NCI Tune
- Modifying an Automatic Tune

### **Changing the Chromatographic Separation**

Peak shapes are defined by the chromatographic conditions. If your peak is too wide, too narrow, or not symmetrical enough, then changing the chromatographic conditions are the best way to improve your method. Start by changing the GC carrier gas flow or oven temperatures (the initial temperature, initial hold time, ramp temperature, final temperature for that ramp, and hold time at that final temperature). These temperatures can be adjusted for each ramp.

Oven changes are strongly dependent on the nature of the compounds you are analyzing. At some point, the GC oven has to be above the boiling point of the compounds you are looking for. If the GC oven is not at the boiling point, the compounds will not volatilize and they will become immobilized. You can change ramp rates to separate coeluting peaks. Refer to the GC user documentation for further suggestions on optimizing chromatographic separation.

# Finding the Best Way to Make an Injection

Adjusting the way you get the sample from the needle into the column can sometimes improve the results of your data. Try modifying the autosampler method, injecting a different amount of liquid, adjusting the injector port temperature or flow, or changing the speed of your injection. You can also try using a hot or cold needle injection. In some cases, adjusting your injection port liners may give you better results. (For detailed instructions, refer to the user guide for your autosampler.)

# **Improving the Way You Prepare Samples**

Although sample preparation adds time and expense to the overall analysis, a more focused method can give you better results. Try extracting your sample in a solvent that increases the solubility of the analytes of interest, but does not increase the solubility of the other compounds. If your method allows it, try switching solvents.

You can also use or change the phase of a solid phase extraction cartridge, which gives you similar results as changing a solvent. You can affect the way you prepare samples by changing the type of cartridge you are using.

# **Changing the Dwell Time or Scan Rate**

The precision of your data depends on how well you define your chromatographic peak. Typically, you get good precision when sampling ten times across the chromatographic peak. In a full scan analysis, increasing the scan rate increases the number of times you have sampled across the peak. However, increasing the scan rate too much results in mass spectral noise, which decreases your analytical precision.

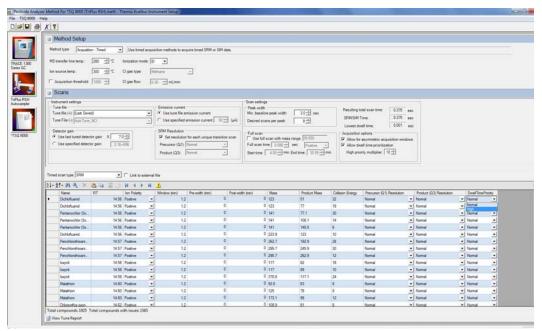
In a SIM or SRM analysis, reducing the dwell time will have a similar effect to increasing the scan rate in full scan. The dwell time is a measure of how long the instrument will average a SIM or SRM event. To optimize your scan rate or dwell time, select a value that gives you 8-12 points across a chromatographic peak. In timed acquisition mode this optimization is done automatically, providing you with the longest dwell time or slowest scan rates possible such that all compounds receive the desired scans across a chromatographic peak.

#### \* To prioritize the dwell times of the problem analytes

- 1. In the Acquisition Options area, check Allow Dwell Time Prioritization. See Figure 81.
- 2. Set the High Priority Multiplier to the desired value.
- 3. A new Dwell Time Priority option appears in your scan list. The choice are Normal or High. Choosing High multiplies the dwell times by the value set in the previous step.

**Note** The total scan time does not change. If prioritizing some dwell times reduces others to less the lowest allowed dwell time for your method, try increasing the acquisition windows.





### **Narrowing the Mass Range**

In a full scan analysis, by narrowing your mass range, you can look directly at the compounds of interest. However, if you are looking at a large number of compounds that have a broad range of mass fragments, a wide mass range makes sense. To narrow the mass range, refine your scan parameters to a smaller number. A narrower mass range also allows you to decrease the scan rate and get the same chromatographic peak sampling. By breaking your MS method into groups, you can create compound-specific MS settings to optimize your data.

### Adjusting the Transfer Line and Ion Source Temperature

If your transfer line temperature is set too low, the less volatile compounds may get stuck in the transfer line and never make it into the ion source. On the other hand, if your transfer line is too hot, you could damage the column or cause a thermal breakdown in the compounds you are analyzing. Typically, aim for the transfer line temperature to be 10 °C (18 °F) over the highest boiling point of the compounds of interest, but no higher than the maximum safe operating temperature of the column.

Use the highest ion source temperature setting that allows adequate response for targeted analytes in order to improve response for high-boiling compounds and prevent column bleed and matrix compounds from dirtying the ion source.

**Note** The transfer line temperature and ion source temperature should be similar to avoid contaminating the ion source.

# **Optimizing an SRM Method**

The following actions might improve your results when using an SRM method:

- Use timed SRM to optimize segments automatically and possibly improve dwell time and number of points across the peak.
- Reduce the Retention Time window if possible. If your sample's retention time is well known the scan window can be reduced. If this is in a high-density compound area it can dramatically improve the ion signal and number of points across the peak.
- Use AutoSRM to optimize collision energy and retention times. This can reduce errors in collision energy and improve confidence in retention times to allow shorter windows.
- Choose the optimum fragment ion for study if allowed by your method. Some fragments are higher in intensity and have less interference associated with their transition.

## **Optimizing an NCI Tune**

Use the following workflow to adjust the low mass when you create an NCI tune.

- 1. Add the Manual Tune button to the TSQ 8000 Dashboard.
  - a. Open the TSQ 8000 Dashboard and right-click anywhere on the dashboard. The message **Add Custom Button** appears.

TSQ 8000 Das	hboard				
Status:	Idle				
		Actual	Set-Point		
✓ MS transfer lin	e temp.:	310 °C	310 1	c	
<ul> <li>Ion source ten</li> </ul>	np.:	347 °C	350 °	c .	
✓ System over te	emp:	False			
Vacuum:		OK			
Foreline press	ure:	75 mTo	п		
lon gauge pre	ssure:	1.2×10 <sup>-5</sup> Torr			
Turbo-pump sp	peed:	100 %			
Collision gas o	n:	No			
CI reagent gas	flow:	0.0 mL/m	in 0.0 m	L/min	
	Air/Wa	ater Spectrum			
	Calibration	n Gas Spectrur	n	Ī	
	Custo	m Spectrum			
<b>J</b> to	A	uto Tune			
K	View	Tune Report		Ī	
J.	Tu	ne Types			
	Met	hod Editor			
***	A	utoSRM			
			Г	Add Cu	istom Bu
			L,		
	Instru	ment Control			
	9	ut Down			

Figure 82. Adding a Custom Button to the TSQ 8000 Dashboard

- b. Click the button. The Select the Program to Add a Button For box opens.
- c. Go to **Computer | OS (C:) | Program Files | Thermo | TSQ8**. Select the file named *TSQ8.ManualTune*.

Figure 83. Adding a Manual Tune Custom Button

Organize 🔻 New folder							•		
☆ Favorites		<b>^</b>	Name	^		D	ate modif	fied	
〕 Downloads			퉬 Manuals			9,	/27/2013 8	8:14 AM	I
🖳 Recent Places			🔣 TSQ8.AutoTu	ne		9,	/11/2013	2:41 PM	
🧮 Desktop			📑 TSQ8.Dashbo	ard		9,	/11/2013	2:41 PM	
			TSQ8.InstCon	trolServer		9,	/11/2013	2:41 PM	
🥽 Libraries			TSQ8.Instrum	entControlAp	p	9,	/11/2013	2:41 PM	
Documents		E	🛃 TSQ8.Manual	Tune		9,	/11/2013	2:41 PM	
EFS EFS			🖄 TSQ8.RealTim	eScanning		9,	/11/2013	2:41 PM	
👌 Music			🌃 TSQ8.TuneRe	sultsViewer		9,	/11/2013	2:41 PM	
Pictures			TSQ8.TuneTy	peEditor		9,	/11/2013	2:41 PM	
Videos									
🖳 Computer									
🏭 OS (C:)	L								
👝 Removable Disk (F:)									
		+ 4							

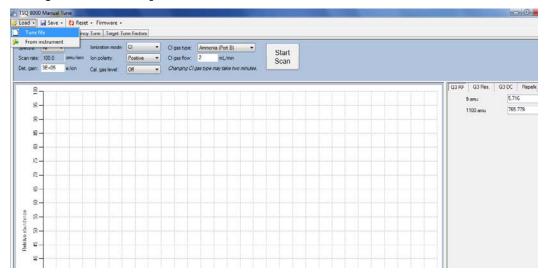
d. Click **Open**. The **TSQ8 Manual Tune** button now appears on the dashboard.**Figure 84.** Locating the TSQ8 Manual Tune Button on the TSQ 8000 Dashboard

sQ accordishooard			_	
itatus: Idle				
1	Actual Set	Point		
MS transfer line temp.:	330 °C	330 °C		
ion source temp.:	319 °C	320 °C		
	False			
Vacuum:	OK			
	182 mTorr			
Ion gauge pressure:		uge not pres	ert 🕴	
Turbo pump speed:	100 %			
Collision gas on:	Yes			
CI reagent gas flow:	2.0 mL/min	2.0 mL/m	n I	
Ar/Wate	er Spectrum	_		
Calibration	Gas Spectrum			
Custom	Spectrum			
Ka Ada	o Tune			
Kew Tu	ine Report			
F Ture	e Types			
Metho	od Editor			
🀅 ha	oSRM			
K TSGRM	lanual Tune		-	- TSQ 8000
Instrume	ent Control			Manual Tur
Shu	t Down			

- 2. Click the **TSQ8 Manual Tune** button.
- 3. Go to Load | Tune File to access the manual tune files.

......

**Figure 85.** Accessing Manual Tune Files



4. The TSQ 8000 Manual Tune Files dialog box opens. Select a PCI Tune file.



Load Tune File	s + Factory Tunes + 4	ny Tunes		
Organize • New fold	and the second se	i≕ • □1 ● art		
Favorites	Name	Date modified an		
C Desktop	AutoTune El 2012-07-26-16-56-32.tsg8tune	7/26/2012 4:56 PN		
Downloads	AutoTune_EL 2012-07-27-15-45-53.tsq8tune	7/27/2012 3:45 PN	Q3 RF Q3 Res.	Q3 DC Repele
💹 Recent Places	AutoTune_NCI_2012-08-16-14-44-55.tsq8tune	8/16/2012 2:45 PN	lon guide RF	2.0000
	AutoTune_PCI_2012-08-16-14-10-44.tsq8tune	8/16/2012 2:10 PM	Technic Content of Technic Content of Technic Content of the Conte	
Libraries	Diagnostics_EL_2012-07-26-17-54-57.tsq8tune	7/26/2012 5:55 PN	V lon guide voltage	-0.50
Documents	Initial_EI_2012-07-26-08-29-51.tsq8tune	7/26/2012 8:29 AN	C Q1 RF	0.237
Music			Q1 resolution	-0.0011
Pictures			IT Q1 DC	5.10
Videos			1 03 RF	0.143
Computer			C3 resolution	
Removable Disk			🖾 Q3 DC	6.10
Constant Oliveran			Repeler	0.00
*	( )		E Lens 1	-50.00
Filen	ame: AutoTune_PCI_2012-08-16-14-10-44. + Tune files	-	Lens 2	-7.00
	Open	Cancel	Ens 3	-26.60
	Open			
			Ent. lens offset	-10.00

5. Select the Low Mass tab on the right. Note the ion guide voltage value.

Figure 87. Finding the Ion Guide Voltage Value

uenc	y Tune   Target Tu	une Factors						
	lonization mode:			Ammonia (Port 8)	Start			
sec	lon polarity.	Positive .		State of the second	Scan			
	Cal. gas level:	Off ·	Changing C	'l gae type may take two minutee.				
-					[	Q3 DC Repeller	Low Mass Q1 + +	Ion Gui
						🔄 lon guide RF	2.0000	
						🔄 lon guide voltage	0.85	– Voltage
						🖪 Q1 RF	0.317	Value
			-			C Q1 resolution	0.0000	value
						01 DC	4.91	
						3 Q3 RF	0.185	
					1	Q3 resolution	0.0007	
t					******	03 DC	4.25	
						Repeller	0.40	
						🗄 Lens 1	17.00	
						Lens 2	4.00	
						🛄 Lens 3	-35.00	
						Ent. lens offset	-10.00 🔅	

- 6. Load an NCI tune file from Manual Tune.
- 7. Click the Ion Guide Voltage box under the Low Mass tab to activate it.

	Save - 🔇 Residution Tune Frequence		une Factors											
pectra: ican rate: let. gain:		lonization mode: Ion polarity: Cal. gas level:	CI Postive Off	• c	gas flow:	Ammonia (Po 2 ml pas type may to	/min	• (	Start Scan					
8-										 	 	 Q3 DC Repeller	Low Mass	Q1 .
												🔄 Ion guide RF	2.0000	10
8-												📝 lon guide voltage	-0.85	3
8-											 	 Q1 RF	0.223	6
¥8 —												 C Q1 resolution	-0.0013	
8-												 🔄 Q1 DC	-6.40	1
12-												🔄 Q3 RF	0.191	
												C Q3 resolution	-0.0007	ł
8-												 🖂 Q3 DC	-2.36	
8-												 Pepeler	0.40	4
8-												 E Lens 1	-50.00	
_												E Lons 2	-7.00	li i
20 22 20 22 1												E Lens 3	-27.80	
8 8-												 Ent. lens offset	-10.00	ł

Figure 88. Activating the Ion Guide Voltage in a NCI Tune File

- 8. If the ion guide voltage value is positive, change it to the PCI value you recorded earlier, which should be negative. You may also optimize the ion guide voltage value to any other negative value.
- 9. Save the changes to the tune file or rename it and save it as a new file.

Figure 89. Saving a New NCI Tune File

Save Tune File	×			
) 🔰 « ThermoTSQ8 > TuneFiles >	• 4 Search TuneFiles 👂			
Organize • New folder	jii • 🔞	art		
Desktop Name Downloads Recent Places	Date modified Type 8/29/2012 12:57 PM File folder	an	Q3 DC Repeller	Low Mass
词 Libraries			E lon guide RF	2 0000
Documents E			V Ion guide voltage	-0.85
Music			C Q1 RF	0.223
Videos			C1 resolution	0.0013
			🔲 Q1 DC	-6.40
E Computer			C Q3 RF	0.191
🚢 OS (C:)			C Q3 resolution	0.0007
Removable Disk I     Section 2014 Section 2014     Section 2014     Section 2014 Section 2014     Section 20			📰 Q3 DC	-2.36
X mater ((()))			Repeller	0.40
File name: blic\Documents\ThermoTSQ8\TuneFiles\Fac			Lens 1	-50.00
Save as type: Tune files	•		Lens 2	-7.00
Hide Folders	Save Cancel		["] Lens 3	27.80
g rade reduces		and the second	Ent. lens offset	-10.00

- 10. Click Save.
- 11. Close Manual Tune.

### **\*** To optimize the CI ion volume position using methane

Ion volume placement is the most important setting for CI. Your instrument's sensitivity is maximized when the ion volume electron hole is properly cleaned inside and outside, but this sensitivity rapidly decays until the hole gets an ion burn. This is normal behavior for your instrument. Lower emission currents cause this decay to happen less rapidly.

- 1. Scan reagent gas ions in methane PCI in manual tune, making note of the reagent gas intensities for ions at m/z 29 and 41. To do this, select **Air** for the spectra or one SIM mass 35, width 50.
- 2. Remove the source and rotate the ion volume in the sleeve and reinsert, repeating until you get the highest m/z 29 for the current flow and filament settings.
- 3. Once the orientation of the ion volume is optimized, if you are tuning in PCI, adjust the methane flow so that the m/z 41 greater than 20% of 29 at 50  $\mu$ A and 70 eV. If you are tuning in NCI, use a flow between 1 and 1.5 mL/min methane for most analytes.
- 4. Perform full scan NCI with the calibration gas on and optimize the repeller voltage. On some instruments a value of 0.5 to 1 V will generate good ion flux while almost no ions will be observed at 0 V. This is due to a possible positive offset of a few tenths of a volt on the repeller electronics.
- 5. Save the tune file with the appropriate name or save the tune file to the instrument to begin the automated tune.

**Note** You may also adjust the emission current for the ionization mode and samples. The higher emission currents often work better in NCI, and the lower emission currents often work better in PCI but the optimal emission currents are typically between 25-100 µA.

# **Modifying an Automatic Tune**

The TSQ 8000 instrument control software includes a utility that uses certain parameters in the tune types to optimize system performance when generating a tune file. Follow these procedures:

- To modify an automatic tune
- To modify advanced tune settings

### To modify an automatic tune

- 1. Open the TSQ 8000 Dashboard and click Tune Types.
- 2. In the Tune Types dialog box, select a tune type to edit and click **Copy**. See Figure 90.

Figure 90. Tune Types Dialog Box

Tune Types		S X
El Diagnostics Only (built-in) El Initial Tune (built-in) El SRM Quick Tune (built-in) El SRM Tune (built-in) El Standard Quick Tune (built-in) El Standard Tune (built-in) El Target Tune (built-in) El Tune Check (built-in) El SRM Factory Tune (built-in)		New Edit Copy Rename
Runs a complete set of diagnostics and generate a report. No tuning is performed. Starts with last saved tune.	*	Delete Close

3. The Tune Type Editor opens. Configure the parameters under the **General** tab in the Tune Type Editor.

Figure 91. Configuring General Tuning Parameters

lit Tune Type		? ×
Name:	El Diagnostics Only	Save
Description:	Runs a complete set of diagnostics and generate a report. No tuning is performed. Starts with last saved tune.	Cancel
Categories:	El SRM Tune, El Standard Tune, Factory	Print
	Tune and diagnostics     Diagnostics only     Show advanced settings      Show advanced settings	
Starting tune f Mass calibra	file: (Last Saved)	
O Perfom	n mass calibration	

a. **Name**—Enter a name for your tune type.

**Note** Create the new tune type name now. You will not be able to edit the tune type name later. Also, you will not be able to save new parameters to a built-in tune.

- b. **Description**—Enter details or notes about your tune type.
- c. **Categories**—This is an optional field that allows you to display a subset of tunes under Auto Tune on the dashboard. This is convenient for separating your most frequently used tunes.
- d. **Type**—Select the **Tune and Diagnostics** option to run a tune with diagnostics or select the **Diagnostics Only** option if you are creating a diagnostics test.
- e. **Output Tune Filename Prefix**—Enter a prefix to be added to the title of your tune report. It is a good idea to use a descriptive prefix for your tune type. For example, if you are always running BFB reports, you could enter BFB here to distinguish it from other reports you are generating.
- f. Starting Tune File—Select a tune file that your tune type will be based on:

**Note** If you select a tune file with a prefix-only name rather than a tune file with a date-time stamp, the most recent tune file of with that prefix name will be loaded at the start of each tune.

- Select **Factory** to use a default factory-made file on the instrument that can be used to begin tuning an instrument with a clean ion source.
- Select **Last Saved** to use a tune file saved on the instrument by the most recent successful automatic tune.
- Select a specific tune file if you have a reliable tune file you want to use as a starting point for a new tune file.
- g. **Perform Mass Calibration**—Select this to enable the system to recalibrate all of the masses during a tune.
- h. **Check Mass Calibration**—Select this to enable the system to confirm that your mass calibration is correct rather than performing a mass calibration.

**Note** If Tune and Diagnostics is selected, you can select only one Mass Calibration option.

4. Select the **Ion Source** tab to configure the ion source as follows:

t Tune Type		- Road to be 1	? <mark>~</mark> ×		
Name:	CI+ Tune		Save		
Description:	Positive CI tuning for with a low repeller va	se with Methane. Starts with last saved tune AutoTune_PCI. Make sure to : e as AutoTune, PCI before beginning if this is the first time tuning in PCI!	save a tune file		
Categories:	Factory				
Туре:	Tune and diagno	cs 🔘 Diagnostics only 🔄 Sho	ow advanced settings		
General	lon Source Targets	Detector Diagnostics Report			
lonization m	node & ion polarity:	Cl+ •			
	CI gas type:	Methane 👻			
	CI gas flow:	1.5 mL/min			
Emission cu	ument:	Custom ▼ 50 µA			
Electron en	ergy:	Custom   70 eV			
Electron len	ns positive voltage:	Default   Volts			
Electron len	ns negative voltage:	Default			
Set ion s	source temperature:	250 °C			

### Figure 92. Configuring the Ion Source Parameters

- a. **Ionization Mode & Ion Polarity**—Use this pull-down menu to select a mode:
  - Select **EI** + to run a tune in Electron Ionization (EI) mode.
  - Select CI+ to run a tune in Chemical Ionization (CI) mode and positive ions.
  - Select CI- to run a tune in CI mode and negative ions.
- b. CI Gas Type (only enabled if you select CI+ or CI-)—Use this pull-down menu to select a gas type, but make sure your selection is the reagent gas attached to your system and that the correct gas port is selected. Methane is commonly used for CI+. Other gases will change the efficiency and energy of the ionization process.

**Note** When changing CI gas types or gas port, there is a two minute delay while the reagent gas is purging. During this time, the Busy light will be lit and you have to wait until the light goes off before using the software.

- c. **Emission Current**—Defines the emission current that you use to run subsequent tunes, but not the emission current that is used for data acquisition.
  - **Tune File**—Select this option to use the value in the tune file that you selected on the General tab.
  - Default—Select this option to use the default emission current, which is 50 µA.
  - **Custom**—Select this option if you want to use a value other than 50  $\mu$ A when increasing or decreasing the sensitivity of the instrument. For the emission current, the default is 50  $\mu$ A. Tune with the same value you are planning to use for your analysis. The use of emission currents above 100  $\mu$ A may lead to the generation of too many ions in the source and a degradation in resolution.

- d. **Electron Energy**—Indicates the energy of the electrons that come off the filament.Lowering the electron energy will extend the lifetime of your filament.
  - Default—Select this option to use the default electron energy, which is 70 eV.
  - **Tune File**—Select this option to use the value in the tune file you selected in the General tab.
  - **Custom**—Select this option to set the energy of the electrons emitted by the filament. For example, you could change the voltage if you wanted to change the ionization efficiency and fragmentation of the sample. This is typically set to 70 V because the standard EI libraries are based on 70 eV electron beams.

**Note** Avoid reducing the electron energy to less than 70 eV. The calibration compound will not be sufficiently ionized for tuning or calibrating at low electron energies.

**Note** A custom electron energy can be selected in the instrument acquisition method even if it is not used for tuning.

- e. Electron Lens Positive Voltage—Allows the electrons to enter the ion volume.
  - **Default**—Select this option to use the default electron lens positive voltage, which is 15 V.
  - **Tune File**—Select this option to use the value used in the tune file that you selected on the General tab.
  - **Custom**—Select this option if you do not want to use the default value for the tune. For the electron lens positive voltage, the default is 15 V. You should tune with the same value you are planning to use for your analysis. This value affects the focusing of the electron beam into the source. If you change your electron energy from 70 eV, this value also changes. This voltage must always be at least 45 V above the voltage applied to the filament. The voltage applied to the filament is the same number, but the opposite sign, of the electron energy.

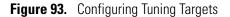
#### f. Electron Lens Negative Voltage.

- **Default**—Select this option to use the default electron lens negative voltage, which is -75 V.
- **Tune File**—Select this option to use the value used in the tune file that you selected on the General tab.
- **Custom**—Select this option if you do not want to use the default value for the tune. For the electron lens negative voltage, the default is -75 V. Tune with the same value you are planning to use for your analysis. This value affects how well the electrons are kept from entering the ion source when they are not supposed to. If you change your electron energy from 70 eV, this value also changes. Ensure this voltage is at least 5 V below the voltage applied to the filament.

g. Set Ion Source Temperature—Select this check box to enable the temperature field. Then enter a value between 0 and 350 °C. The default is 200 °C. The optimal temperature is determined by the molecular structure and weight of the compounds you are analyzing. Heavier compounds require a higher temperature. Tune at the same temperature you will use to run your samples. You should set the temperature as high as possible to keep the ion source clean and maintain the right amount of sensitivity.

**Tip** If you tune regularly between running sample sets, you can save time waiting for the temperatures to equilibrate by setting the tune temperatures to the same temperature used in your acquisition method.

5. Click the **Targets** tab to configure how you want to tune your targets. Target tuning is used to adjust the way the TSQ 8000 instrument tunes to meet regulatory requirements. Otherwise, this is an optional setting.



ame:	El Initial T	une			Save		
lescription:			n instrument. Starts with Factory tune. Sets detector gain to 3x10^5. Sets Repeller to 0 and Q3 Voltage to -3 V. Higher resolution and lower sensitivity than El Standard Tune.		Cancel		
Categories:	Factory	Factory					
Гуре:	Tune a	and dia	agnostics 💿 Diagnostics only	dvanced settings			
General	on Source	T	rgets Detector Diagnostics Report				
aeneral I	on source	Ia	rgets Detector Diagnostics Report				
		1					
Tune targe	et ion ratios						
7 Tune targe larget mass	et ion ratios Ratio						
7 7		%	(25 to 51.5 amu)				
Farget mass	Ratio	% %					
Farget mass	Ratio		(25 to 51.5 amu)				
Farget mass 50 amu: 69 amu:	Ratio 1.1 100	%	(25 to 51.5 amu) (51.5 to 78.5 amu)				
Farget mass 50 amu: 69 amu: 131 amu:	Ratio 1.1 100 90	%	(25 to 51.5 amu) (51.5 to 78.5 amu) (78.5 to 132.5 amu)				

**Tune Target Ion Ratios**—Select this check box to adjust the ratios based on the results from an injection of tuning compound.

6. Click the **Detector** tab to configure the detectors. See Figure 94.



Edit Tune Type		? ×
	El Initial Tune	
Name:		Save
Description:	Requires a clean instrument. Starts with Factory tune. Sets detector gain to 3x10^5. Sets Repeller to 0 V, Q1 Voltage to -3 V and Q3 Voltage to -3 V. Higher resolution and lower sensitivity than EI Standard Tune.	Cancel
Categories:	Factory	Print
Туре:	Tune and diagnostics O Diagnostics only	
General Id	on Source Targets Detector Diagnostics Report	
V Set initial o	detector gain: 3 × 10 <sup>5</sup>	
V Tune dete	ctor	
Adjust det	ector sensitivity	
Mass:	69 v Intensity: 10,000,000	

- a. Set Initial Detector Gain—Select this option to set the electron multiplier gain that will be used in tuning the instrument. The gain is the number of electrons generated for every ion that strikes the detector. This is typically set at  $3 \times 10^5$  electrons per ion. Gains larger than this will generate more electrons per ion, but both the analyte ion and the noise ion signals will be larger. You can also tune to lower gain values, which decreases the signal strength. Lower values also increase the chance that an ion will not be detected.
- b. **Tune Detector**—Select this check box to tune the detector. As the electron multiplier ages, the voltage required for a given gain increases. Depending on your sample load and if your system is leak tight (oxygen is bad for the detector), you should not have to perform this tune very often. Limit to 1–2 times a year.
- c. Adjust Detector Sensitivity—Select this check box to tune the detector to generate a consistent area count of a calibration gas ion for the tune report. Because the intensity of the cal gas varies depending on the atmospheric pressure and temperature of the lab, this option results in larger variation in the analytical runs, as compared to using a fixed detector gain. If you use this function, set the initial detector gain to  $3 \times 10^5$  electrons per ion.
  - Mass—Select the calibration gas mass you want to use.
  - Intensity—Enter the intensity you want to see on the tune report.
- 7. Click the **Diagnostics** tab and select a test to confirm the operational ability of the TSQ 8000 system. See Figure 95.

Edit Tune Type			? ×
Name: Description: Categories:	El Initial Tune Requires a clean instrument. Starts with Factory tune. Sets o Voltage to -3 V and Q3 Voltage to -3 V. Higher resolution an Factory		Save Cancel Print
Type: General k	Tune and diagnostics     Diagnostics only on Source   Targets   Detector   Diagnostics   Rep	Show advanced settings	
Detector ( Filament C Leak Chei Leas Chei Leas Cont Power Su Q1 Freque Q1 RF/DC Q1 Transr Q2 Freque Q3 RF/DC SRM Effic Temperatu	Theck Frequency Check ck tinuity Check pply Check ancy Check C System Check ency Check ency Check ency Check S System Check isncy Check		
			.#

Figure 95. Setting Diagnostics

8. Click the **Report** tab configure how you want to view your data

Figure 96. Selecting Data Report Options

Edit Tune Type		? ×
Name:	El Initial Tune	Save
Description:	Requires a clean instrument. Starts with Factory tune. Sets detector gain to 3x10^5. Sets Repeller to 0 V, Q1 Voltage to -3 V and Q3 Voltage to -3 V. Higher resolution and lower sensitivity than El Standard Tune.	Cancel
Categories:	Factory	Print
	Tune and diagnostics     Diagnostics only     Show advanced settings     Source   Targets   Detector   Diagnostics   Report	
	Il-scan spectrum on threshold: 3,000	
SIM masses:	69 ▼     131 ▼     219 ▼     414 ▼     502 ▼	

a. Acquire Full-Scan Spectrum—Select this check box to display the full spectrum on your tune report.

- b. **Acquisition Threshold**—Enter a minimum peak height for the full scan spectrum on the tune report. If your peak has an intensity that is below this threshold, it will not be stored.
- c. **SIM Masses**—Select masses to be displayed on your tune report. You can select a maximum of five masses, one from each list. At least one mass must be displayed on the report.

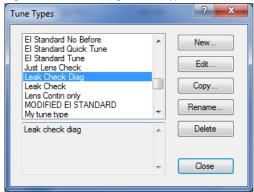
**Tip** Factory tunes cannot be altered. If a newly created tune does not perform as expected, you can always go back to a factory tune, copy it, and change the settings as described above. Save the new tune with a new name before closing the Edit Tune Type dialog box.

- 9. Once you are finished configuring all the tabs, click **Save** to save the tune type. Your new tune type appears in the Tune Types window.
- 10. Click **Close** to return to the dashboard. You can select the tune type in the automatic tune window.
- 11. If you want to rename a tune you created, open the Tune Types dialog box by clicking **Tune Types** on the TSQ 8000 Dashboard.

**Note** You cannot rename a factory tune.

a. Select **Rename** in the Tune Types dialog box.

#### Figure 97. Renaming a Tune Type



b. The **Rename Tune Type** dialog box opens. Assign a valid new name to your tune. You will not be able enter a name that is an invalid file name or already in use. See Figure 98.

### Figure 98. Changing a Tune File Name

Rename tune type - Leak	Check Diag			
New name for tune type:	Leak Check Diag			
		Ok	Cancel	
		Ok	<u>C</u> ancel	

#### c. Click OK.

**Note** The following instructions refer to advanced tuning settings that most users will never need to access. Contact your local Thermo Fisher Scientific service organization before adjusting these settings.

### To modify advanced tune settings

 To configure the Q1 Lenses, you must select the Show Advanced Settings check box. The lens tune is the main portion of the tune algorithm. In this section you can choose which components to tune, which mass to optimize for the tune, what the range of allowed values is, how to move through the range, how much the signal must change for a new value to be selected, and what (if anything) can be done about the resolution of the peak and any errors that occur. Select the **Tune Lenses** check box to access the data table. See Figure 99.



Name	e: El In	El Initial Tune									Save			
Descri		Requires a clean instrument. Starts with Factory tune. Sets detector gain to 3x10°5. Sets Repeller to 0 V, Q1 voltage to -3 V and Q3 Voltage to -3 V. Higher resolution and lower sensitivity than EI Standard Tune.									Cancel			
Categ	gories: Fact	Factory								Print				
Type:	: • т	une and diagn	ostics	Dia	aqnostics	only			V Sh	ow adva	inced setti	ngs		
_		-			-				Q1 Resolution					
Gene	eral Ion Sou	irce Targe		ctor	Diagnos		Report	Q1 Lenses	GT Resolution	Q3	Resolution	ı Q	3 Lenses	
	eral   Ion Sou une lenses	irce   large	ts   Dete	ctor	Diagnos	tics   r	Report	QT Lenses	Q I Resolution	Q3	Resolution	n   Q	3 Lenses	4
Gener	1	Ince   Targe Mass	ts Dete Start	Stop	Diagnos	Max. Width	Measure		On Error	03	Resolution	n   Q		
	une lenses		I	1		Max.	Measure			Q3	Resolution	n   Q		
	une lenses Device	Mass	Start	Stop	Step	Max. Width	Measure at %	Threshold	On Error	_ Q3	Resolution	n   Q		
	Device	Mass 219 69	Start 0	Stop 0	Step 0	Max. Width 2	Measure at %	Threshold	On Error Continue	Q3	Resolution	n   Q		
	Device Repeller Lens 1	Mass 219 69 /olt 502	Start 0 -50	Stop 0 -50	Step 0 0	Max. Width 2 2	Measure at % 50 50	Threshold 1 1	On Error Continue Continue	Q3	Resolution	n   Q		
	Device Repeller Lens 1 Ion Guide 1	Mass 219 69 /olt 502 /olt 219	Start 0 -50 -15	Stop 0 -50 -1	Step 0 0 0.25	Max. Width 2 2 2	Measure at % 50 50 50	Threshold 1 1 1	On Error Continue Continue Continue	Q3	Resolution	n   Q		
	Device Repeller Lens 1 Ion Guide 1 Ion Guide 1	Mass 219 69 /olt 502 /olt 219 /olt 69	Start 0 -50 -15 -10	Stop 0 -50 -1 1	Step           0           0.25           0.2	Max. Width 2 2 2 2 2	Measure at % 50 50 50 50 50	Threshold 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	On Error Continue Continue Continue Continue	Q3	Resolution	n   Q		

a. **Device**—Click in this box to select the component you want to tune according to the settings in the other columns.

- i. **Repeller**—Controls how the repeller pushes the ions out of the ionization region. The voltage applied to this component has a very strong effect on the energy of the ion beam, which has a strong effect on the resolution and the intensity. The lower the voltage, the better the resolution. However, higher voltages will prevent ions from striking the repeller surface, which leads to better robustness. A clean system typically tunes from 0 to 5 V, although a dirty system may have the repeller climb as high as 12.5 V. Do not set the repeller any higher.
- ii. Lens 1—Controls Lens 1, which is the first of three lenses that the ions see as they leave the ionization region. These three lenses act as a focusing element to maximize the ion beam intensity that is entering the ion guide. This field is typically set between -35 and -50 V.
- iii. **Lens 2**—Controls Lens 2, which is the second of three lenses that the ions see as they leave the ionization region. These three lenses act as a focusing element to maximize the ion beam intensity that is entering the ion guide. This field is typically set between 0 and -15 V.
- iv. Lens 3—Controls Lens 3, which is the last of three lenses that the ions see as they leave the ionization region. These three lenses act as a focusing element to maximize the ion beam intensity that is entering the ion guide. This field is typically set near the same voltage as Lens 1.
- v. **Ion Guide Voltage**—Controls the ion guide's DC offset voltage. It can potentially help focus the ions into the quadrupole while ensuring that neutral noise is eliminated. The voltage on this component is mass dependent and should be set at several different masses. This field is typically set between +1 and -15 V, depending on the mass of the ion.
- vi. **Ion Guide RF q**—Controls the ion guide's RF q (amplitude). This device is mass dependent and should be set at several different masses. This field can be tuned between 0 and 2. This device determines the ion guide RF amplitude tune value which is given as the RF q \* (m/z).
- vii. **Ion Guide RF**—(This setting is maintained for compatibility with legacy tune types. For new tune types, Ion Guide RF q is the recommended setting.) Controls the ion guide's RF voltage. It can potentially help focus the ions into the quadrupole while ensuring that neutral noise is eliminated. The voltage on this component is mass dependent and should be set at several different masses. This field is typically set between 0 and +5 V, depending on the mass of the ion.
- viii. **Q1 Voltage**—Controls the voltage that pulls the ions into the quadrupole. The voltage applied to this component has a very strong effect on the energy of the ion beam, which has a strong effect on the resolution and the intensity. The lower the voltage, the better the resolution. However, higher voltages will pull more ions into the quads, which leads to better signal. This field is typically set between 0 and -5 V.
- ix. **Q1 Resolution**—Adjusts the ratio of the quadrupole DC and RF voltages to create the resolution required for your analysis. You can set the desired peak width at a given mass and whether you measure the width at 10% or 50% of the

peak height. Because there is no static DC voltage involved, the start, stop, and step values are not used.

- b. Mass—Click in this box to select the ion to be used for tuning.
- c. **Start**—Enter the starting voltage for the tune. The start voltage must always be less than the stop voltage. For example, -35 is smaller than 0.
- d. **Stop**—Enter the final voltage for the tune.
- e. **Step**—Enter the increment for the tuning range. For example, if you tune from 0-50 in increments of 10 V, then you would set the **Step** field to 10.

**Note** If the start and stop values are the same value, the device will be set to that value and ignore the step size. In some tunes, devices are set to a value before tuning them in another line. This presetting is done because ramping devices do not allow the tune to run at values higher than those chosen for higher masses for the same device.

- f. Max Width—Enter the maximum allowable width of the ion during the tune.
- g. **Measure at %**—Click in this box to select the location on the peak where you want to measure the maximum width.
  - 10—Measures the width at 10% of the peak height.
  - **50**—Measures the width at 50% of the peak height.
- h. **Threshold**—Enter the change in intensity that has to occur when the tune selects a new voltage. For example, if you set this field to 1.1, the tune will not select a new voltage for that component unless the intensity is 110% of the old intensity. If you set this field to 1, anytime the new voltage has an intensity larger than the old intensity, the tune will select the new intensity.

**Note** If you choose a value less than 1 for ramped devices, the tune steps back in energy until the lower intensity is detected. For example, if you choose 0.8, the device value lower in energy than the device value with the maximum response that gives an intensity closest to 80% of the maximum intensity is chosen.

- i. On Error—Click in this box to select how to handle an error in the tune.
  - **Fail**—Stops the tune when an error occurs or if no tune points are found meeting the tune criteria.
  - **Continue**—Allows the tune to continue on to the next device when an error occurs.
- j. **Entrance lens offset** and **Exit lens offset**—Changing these voltages affects ion acceleration. The default levels will give the best performance; changing the voltages will affect peak resolution.
- 2. Click the **Q1 Resolution** tab to configure the resolution of your tune. You must select the **Show Advanced Settings** check box to access this tab. Selecting this option tunes the

resolution of Quadrupole 1 by itself. This resolution tunes the system at 100 and 1000 amu/s scan rates. You can tune the resolution during a lens tune or you can tune the resolution by itself.

Figure 100. Configuring Q1 Resolution

Name:	El Initi	al Tune							Save
Descript				ctory tune. Sets detector ga her resolution and lower se				A T	Cancel
Categori	es: Facto	у							Print
		ne and diagnostic		stics only			advanced se	-	
General	lon Sour	ce Targets		gnostics   Report   Q1	Lenses C	☑ Show	advanced se Q3 Resolut	-	Lenses
Type: General	Ion Sour		Detector Diag		Lenses C			-	Lenses
General	lon Sour resolution Mass	ce Targets Peak Width	Detector Diag	gnostics Report Q1	Lenses   C			-	Lenses
General	Ion Sour resolution Mass 69	Ce Targets Peak Width 0.6	Detector Diag Measure at % 50	gnostics Report Q1 Lens Tune Relation Before and After	Lenses   [C			-	Lenses

**Note** Changing the Q1 lenses or resolution affects the Q3 lenses and resolution.

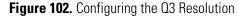
- a. Mass—Click in this box to select the ion to be used for tuning.
- b. **Peak Width**—Enter the target peak width.
- c. **Measure at %**—Click in this box to select the location on the peak where you want to measure the target peak width.
  - **10**—Measures the width at 10% of the peak height.
  - **50**—Measures the width at 50% of the peak height.
- d. **Lens Tune Relation**—Click in this box to set the occurrence of the resolution tune parameters before or after a lens tune.
  - **Before and After**—Uses the same resolution parameters before and after a lens tune.
  - Before—Uses the resolution parameters before a lens tune.
  - After—Uses the resolution parameters after a lens tune.
- 3. Select the **Q3 Lenses** tab to configure the Q3 lenses. You must select the **Show Advanced Settings** check box to access this tab. The lens tune is the main portion of the tune algorithm. In this section you can choose which components to tune, which mass to optimize for the tune, what the range of allowed values are, how to move through the range, how much the signal must change for a new value to be selected, and what (if anything) must be done about the resolution of the peak and any errors that occur.

### Figure 101. Tuning the Q3 Lenses

	El Initial Tu	100									
ame:		Ine									Save
escript									0^5. Sets Repe an El Standard		Cancel
ategori	es: Factory										Print
ype:	Tune a	nd diagnost	tics	Dia	qnostics	only			V 9	Show advanced sett	ings
ieneral	Ion Source	Targets	Dete	ctor	Diagnost	tics F	Report (	Q1 Lenses	Q1 Resolutio	on Q3 Resolution	n Q3 Lenses
Tune	lenses		1						I	I	
	Device	Mass	Start	Stop	Step	Max. Width	Measure at %	Threshold	On Error		
•	Q3 Voltage	219	-3	-3	0	2	50	1	Continue		
	Q3 Voltage	502	-3	-3	0	2	50	1	Continue		
	Q3 Voltage	69	-3	-3	0	2	50	1	Continue		
*											
Entranc	e lens offset: 📑	5 Vo	lts I	Exit lens (	offset: -	1	Volts				
MS/N	IS Voltages										
	Mass	Volts									
•	69.00	-2.50									
	219.00	-3.00									
	500.00	-7.00									
H	502.00										

- a. **Q3 Voltage**—Controls the voltage that pulls the ions into the quadrupole. The voltage applied to this component will have a very strong effect on the energy of the ion beam, which will have a strong effect on the resolution and the intensity. The lower the voltage, the better the resolution. However, higher voltages will pull more ions into the quads, which leads to better signal. This field is typically set between 0 and -5 V.
- b. **Resolution**—Adjusts the ratio of the quadrupole DC and RF voltages to create the resolution required for your analysis. You can set the desired peak width at a given mass and whether you measure the width at 10% or 50% of the peak height. Because there is no static DC voltage involved, the start, stop, and step values are not used.
- c. Mass—Click in this box to select the ion to be used for tuning.
- d. **Start**—Enter the starting voltage for the tune. The start voltage must always be less than the stop voltage. For example, -35 is smaller than 0.
- e. **Stop**—Enter the final voltage for the tune.
- f. **Step**—Enter the increment for the tuning range. For example, if you tune from 0-50 in increments of 10 V, then you would set the **Step** field to 10.
- g. Max Width—Enter the maximum allowable width of the ion during the tune.

- h. **Measure at %**—Click in this box to select the location on the peak at which you want to measure the maximum width.
  - **10**—Measures the width at 10% of the peak height.
  - **50**—Measures the width at 50% of the peak height.
- i. **Threshold**—Enter the change in intensity that has to occur when the tune selects a new voltage. For example, if you set this field to 1.1, the tune will not select a new voltage for that component, unless the intensity is 110% of the old intensity. If you set this field to 1, anytime the new voltage has an intensity larger than the old intensity, the tune will select the new intensity.
- j. **On Error**—Click in this box to select how to handle an error in the tune.
  - Fail—Stops the tune when an error occurs.
  - **Continue**—Allows the tune to continue on to the next device when an error occurs.
- k. **Entrance lens offset** and **Exit lens offset**—Changing these voltages affects ion acceleration. The default levels will give the best performance; changing the voltages will affect peak resolution.
- 1. **MS/MS Voltages**—These overwrite the Q3 voltage tune values when the instrument is in SRM mode. These values are not tuned and are added to the collision energy used in the SRM scan.
- 4. Click the Q3 Resolution tab to configure the resolution of your tune. You must select the Show Advanced Settings check box to access this tab. Select this option to tune the resolution of Quadrupole 3 by itself. This resolution will tune the system at 100 and 1000 amu/s scan rates. You may also tune at a higher scan rate. You can tune the resolution during a lens tune or you can tune the resolution by itself. See Figure 102.



Name	e: Elln	itial Tune				Save
Descr				ctory tune. Sets detector gain to gher resolution and lower sensitiv		Cancel
Categ	gories: Fact	ory				Print
Туре:	: • 1	une and diagnostic	s 🔘 Diagno	ostics only	V Show advance	ced settings
	- I I - C-		Detector Disc		and Developing Loop D	021
	eral Ion Sou une resolution	1 - 1	Detector Diag une high scan rate:	gnostics Report Q1 Lens : 10000 amu/sec	es Q1 Resolution Q3 Re	esolution Q3 Lenses
		1 - 1	une high scan rate:	: 10000 amu/sec	es Q1 Resolution Q3 Re	esolution Q3 Lenses
	une resolution	Π Τ	une high scan rate:	: 10000 amu/sec	es   Q1 Resolution   Q3 Re	esolution Q3 Lenses
	une resolution	Peak Width	une high scan rate: Measure at %	: 10000 amu/sec	es Q1 Resolution Q3 Re	esolution Q3 Lenses
	Mass 69	Peak Width	une high scan rate: Measure at %	Lens Tune Relation	es Q1 Resolution Q3 Re	esolution Q3 Lenses
	Mass 69 69	Peak Width 0.6 0.96	Measure at %	amu/sec Lens Tune Relation Before After	es Q1 Resolution Q3 Re	esolution Q3 Lenses
Gene	Mass 69 69 219	Peak Width 0.6 0.96 0.6	Measure at % 50 50 50	amu/sec Lens Tune Relation Before After Before	es Q1 Resolution Q3 Re	esolution Q3 Lenses

- a. Mass—Click in this box to select the ion to be used for tuning.
- b. **Peak Width**—Enter the target peak width.
- c. **Measure at %**—Click in this box to select the location on the peak at which you want to measure the target peak width.
  - **10**—Measures the width at 10% of the peak height.
  - **50**—Measures the width at 50% of the peak height.
- d. **Lens Tune Relation**—Click in this box to set the occurrence of the resolution tune parameters before or after a lens tune.
  - **Before and After**—Uses the same resolution parameters before and after a lens tune.
  - Before—Uses the resolution parameters before a lens tune.
  - After—Uses the resolution parameters after a lens tune.
- 5. **Tune High Scan Rate**—Select this check box to tune the resolution at a scan rate in addition to the 100 and 1000 amu/s default scan rates.

# **Computer Settings**

Your TSQ 8000 system includes a computer with all the software needed to operate the instrument.

If you replace the computer in the future, use the information in this chapter to change the computer settings so your TSQ 8000 system and the associated Thermo Scientific software will run correctly. Instructions for installing Thermo Scientific software are included on the software CD shipped with your instrument.

#### Contents

- System Requirements
- Computer Settings
- Excluding the Xcalibur Directory from Virus Scan
- Other Software

## System Requirements

The TSQ 8000 system requires a Windows 7 32-bit or 64-bit operating system. See "System Requirements" on page xi for a complete list of system requirements.

## **Computer Settings**

The following computer settings will help prevent communication interruptions in your TSQ 8000 system.

- To use the power save feature
- 1. From the Start menu choose Control Panel | System and Security | Power Options.
- 2. On the left side, click Change When the Computer Sleeps.
- 3. For **Put the Computer to Sleep**, select **Never**. See Figure 103. You may set the display to turn off at any time. It will not affect the performance of your TSQ 8000 system.

Solver Settings → Control Panel → Hardware and Sound → Power Options → Edit Plan Settings							
Change settings for the plan: Balanced Choose the sleep and display settings that you want your computer to use.							
	Turn off the display:	1 hour 30 minutes 🔹					
	Put the computer to sleep:	Never					
	Change advanced power settings	i					
	Restore default settings for this p	lan					

### Figure 103. Power Settings for the TSQ 8000 System Computer

### ✤ To use the virtual memory feature

- 1. From the Start menu, choose Control Panel | System and Security | System.
- 2. On the left side of the window, click Advanced System Settings.
- 3. In the System Properties dialog box, click Settings.
- 4. In the Performance Options dialog box, click the **Advanced** tab, and then click **Change** to open the Virtual Memory window.
- 5. Confirm that **Automatically Manage Paging File Size for All Drives** check box is selected. See Figure 104.

Figure 104. Virtual Memory Settings

System Properties	Virtual Memory
Computer Name Hardware Advanced System Protection Remote You must be logged on as an Administrator to make most of these changes. Performance Visual effects, processor scheduling, memory usage, and virtual memory	Automatically manage paging file size for all drives Paging file size for each drive Drive [Volume Label] Paging File Size (MB)  C: [OSDisk] System managed
User Profiles Desktop settings related to your logon Settings Startup and Recovery	Selected drive: C: [OSDisk] Space available: 227140 MB Custom size: Initial size (MB): Maximum size (MB):
System startup, system failure, and debugging information Settings	System managed size     No paging file     Set     Total paging file size for all drives
Environment Variables	Minimum allowed: 16 MB Recommended: 30 19 MB Currently allocated: 2669 MB
OK Cancel Apply	OK Cancel

### ✤ To confirm the network card firewall settings

- 1. From the Start menu, choose Control Panel | Network and Internet | System and Security | Windows Firewall | Advanced Settings.
- 2. In the Actions Panel on the right, (see Figure ), select Properties.
- 3. In the Windows Firewall with Advanced Security on Local Computer Properties window, select Domain Profile | State | Customize.

Figure 105. Firewall Settings for the TSQ 8000 System Computer

Windows Firewall with Advanced Security			
File Action View Help			
Windows Firewall with Advance Windows Firewall with Advance Commettion Rules Monitoring Monitoring Windows Firewall with Monitoring Windows Firewall with Monitoring Windows Firewall Windows Firewall	Windows Fiewall with Advanced Security on Local Computer Pro Domain Profile Provate Profile Public Profile (Piece Settings Specify behavior for when a computer is connected to its corporate domain. Safe Preval state: On (seconnected to its corporate Indownd connections: Block (idefault) Outbound connections: Block (idefault) Protected network connections: Customize Protected network connections: Customize Settings Specify logging settings for toubleshooting. Learn more about these settings OK Cancel Acopy toubleshooting and activity A curretly applied frewall and connection security vies and security associations for	Actions       Windows Firewall with Advanced Security on Local ▲ <ul> <li>Import Policy</li> <li>Restore Default Policy</li> <li>Diagnose / Repair</li> <li>View</li> <li>Refresh</li> <li>Properties</li> <li>Help</li> </ul>	Actions Panel

4. In the **Protected Connections for the Domain Profile** window, make sure that any instrument checkboxes are not checked.

Figure 106. Setting the Network Connections

	rewall with Advanced Security on Local Computer	Actions
Concetion and Rules Concetion Security Rules Monitoring	Sound connections that do not match a vue are allowed  Profile  Profile Profile Profile Profile Profile Profile Profile Profile Profile Profile Profile Pro	Import Policy     Export Policy     Restore Default Policy     Diagnose / Repair
Create t is as	nd cr wal ni récise récise résise out	

# **Excluding the Xcalibur Directory from Virus Scan**

If you are using anti-virus software on your computer, exclude the following folders from scanning:

- C:\Program Files\Thermo\
- C:\Thermo\
- C:\Xcalibur\

Failure to exclude these folders might cause interruptions in data acquisition. If you must, for security reasons, scan all system files, set the virus scans so that they occur when you are not using the instrument to acquire data.

# **Other Software**

Install Microsoft Office 2010 software on your system. You can use Excel spreadsheets to import large methods into the instrument software.

# **Troubleshooting**

This section contains information to help you diagnose problems within your data. Sometimes, your experience as a scientist will enable you to look at your data and detect that something is wrong either with the instrument or your sample. This chapter describes the most common indications of a problem with a baseline, peak or result.

#### Contents

- Diagnostics Checks
- How to Know When Your System Needs Maintenance
- Investigating Baseline Issues
- Investigating Peak Issues
- Investigating Results Issues

If there is an issue with the hardware, see the troubleshooting section of the *TSQ 8000 Hardware Manual*.



**CAUTION ELECTRICAL SHOCK HAZARD:** When troubleshooting any issue that requires removing a cover on the TSQ 8000 instrument, power-off and vent the instrument to avoid possible injury.

You can begin troubleshooting by running a tune with diagnostics on the TSQ 8000 instrument. If you have good ion intensities, good peak shapes, and no air leak, you might want to look first at the GC, autosampler, or carrier gas.

If you have air leaks, locate and address them. Pay particular attention to the transfer line ferrule, vent valve knob, front panel, gas quality, carrier gas vacuum compensation, and vacuum interlock on the mass spectrometer, as well as the inlet on the GC.

If your intensities are too low, make sure carrier vacuum compensation is turned on.

**IMPORTANT** When inserting a cold ion source cartridge such as after cleaning or when switching between EI and CI modes, the ion source and lens stack will expand as the source cartridge heats, often pushing the ion volume and lenses away from the rear of the instrument where they are firmly held by the RF Lens spring contacts. To avoid intermittent electrical contacts to the lenses, you should insert the ion source cartridge, wait 30 min for it to get to temperature, then remove and reinsert it. See the *TSQ 8000 Hardware Manual* for instructions on cleaning and inserting the ion source cartridge.

If you have no ions in full-scan or SIM mode, do the following:

- Switch filaments.
- Confirm the starting tune file is appropriate for ionization mode or source cleanliness level.
- Confirm the correct ion volume is inserted (EI or CI).
- Verify the ion source is correctly installed.
- Run diagnostic checks (Diagnostics Only tune type)
- Clean the ion source.

If you do not see ions in CI mode:

- Confirm that the CI ion volume is installed.
- Confirm that the reagent gas is connected and turned on.

If you do not see any peaks in SRM scans, do the following:

- Verify the collision gas supply.
- Run diagnostics checks (Diagnostics Only tune type).
- Run a full-scan method to check if your system is functional.

## **Diagnostics Checks**

The following table provides you with possible corrective actions you can take after failing a diagnostics check on your TSQ 8000 system.

**Note** If you are unsure about what to do if your system fails a diagnostic, please contact your field service engineer.

Diagnostic	Cause of Failure	Corrective Action	
Communication Check	Unknown	Contact your Field Service Engineer.	
Frequency Check	RF Dip is not correct	Run a full tune that will correct for any RF dip problems.	
		Ensure source cartridge is installed.	
		Contact your Field Service Engineer.	
SRM Efficiency Check	Collision gas is not supplied correctly	Check collision gas tank and pressure.	
	Q1 is poorly tuned	Run an EI SRM tune (not a quick tune).	
Lens Continuity Check	Ion source not inserted properly	Remove and reinsert ion source.	
	Lens plate and/or springs are damaged	Contact your Field Service Engineer.	
	A wire is disconnected or shorted inside the manifold	Contact your Field Service Engineer.	

 Table 2.
 Diagnostics and Corrective Actions

# How to Know When Your System Needs Maintenance

Typically, you will notice that your instrument needs maintenance when you are analyzing your data on the computer. Some of the most common reasons and indications are as follows:

- **Contamination**—Excessive background in your mass spectra usually indicates that your instrument is contaminated. Use the mass spectrum in the table below to understand the origin of the contamination. Cleaning solvent peaks usually indicate that the ion source cartridge was installed before it completely dried.
- **Fingerprints**—A series of mass peaks in your data that are 14 amu apart usually indicates fingerprint or other hydrocarbon contamination. To avoid fingerprints, wear clean, lint-free gloves when performing any type of maintenance on a component in the vacuum manifold of the TSQ 8000 instrument.
- Air Leaks—Higher than normal vacuum pressure or poor sensitivity usually indicates an air leak. Check the last o-ring or ferrule that you installed.

lons ( <i>m/z)</i> To Monitor	Compound	Possible Source
13, 14, 15, 16, 17, 29, 41, 57	Methane	CI gas
18, 28, 32, 44 or 14, 16, 19	H <sub>2</sub> O, N <sub>2</sub> , O <sub>2</sub> , CO <sub>2</sub> or N, O	Residual air and water, air leaks, outgassing from Vespel ferrules
69, 100, 119, 131, 169, 181, 214, 219, 264, 376, 414, 426, 464, 502, 576, 614	PFTBA and related ions	PFTBA (tuning compound)
31	Methanol	Cleaning solvent
43, 58	Acetone	Cleaning solvent
78	Benzene	Cleaning solvent
91, 92	Toluene or xylene	Cleaning solvent
105, 106	Xylene	Cleaning solvent
151, 153	Trichloroethane	Cleaning solvent
149	Plasticizer (phthalates)	Use of vinyl or plastic gloves
Peaks spaced 14 amu apart	Hydrocarbons	Fingerprints, foreline pum oil, or other hydrocarbons

 Table 3.
 Common Contaminants

Behavior	Characteristic	Cause	Remedy
Drifting Baseline	General	Stationary phase has accumulated in column	Replace the column or cut off the end of the column.
		Chromatographic baseline is high	Replace the column or cut off the end of the column.
		Carrier gas pressure is too low	Check for leaks in injector or flow path. Replace the carrier gas cylinder if it is empty or low. Increase the pressure if maximum injector pressure in method is greater than carrier line pressure set by regulator. Make sure the vacuum compensation is on.
		Carrier gas flow is drifting	Check for leaks in injector or flow path. Check the carrier gas tank.
		Impurities have accumulated in column	Run solvent blank to remove impieties. If impurities persistent after multiple solvent blanks: Inject solvent from a different source. Change syringe, liner and septum. Clean injector. Check impurity levels in your gas. Use the correct gas purity and filter.
	Falling	Carrier gas leak in the system	Perform a leak test and tighten the connections to the carrier gas line if leak is found.
		Column is baking out	Wait for the column to stabilize.
	Rising	Impurities have accumulated in column	Check impurity levels in the gas source. Use correct gas purity.
	Abnormal rise in baseline when oven temperature is high	Impurities have accumulated in column	Recondition or replace the column.

# **Investigating Baseline Issues**

Behavior	Characteristic	Cause	Remedy
Noisy Chromatographic Peaks	General	Excessive column bleed at high oven temperatures	Reduce the column temperature. Bake out the column. Install a high-temperature column.
			Install oxygen filters in carrier gas line. Check pneumatic and inlet systems for leaks. Use correct gas purity with low oxygen content.
		Column is contaminated or damaged	Condition or replace the column.
		Oven temperature is higher than column's maximum allowable temperature	Reduce oven temperature to the maximum allowable temperature of the column.
		Leak at column fittings	Find leak. Tighten fittings if loose. Replace ferrule if overtightened.
			Transfer line temperature is not set too low.

# **Investigating Peak Issues**

Behavior	Characteristic	Cause	Remedy
Broadening	General	Column higher than optimum of column	Reduce the flow. Make sure vacuum compensation is turned on.
		Column flow lower than optimum of column	Increase the flow.
		Split flow is too low for split injection	Increase the flow to 40-50 ml/min.
		Performance of the column has degraded	Test the column at the optimum flow rate.
		Injector is dirty	Clean or replace the liner.
		Ion source is dirty	Clean the ion source and tune the instrument.
		Column is not far enough into the transferline	The GC column does not extend into the TSQ 8000 MS source. Use the column measuring tool to confirm column length. If the end of the column is inside the transfer line, an excessive amount of GC effluent will contact the inside wall.
Fronting	General	Column is overloaded	Decrease the injected amount and/or analyte concentrations. Increase the split ratio. Use a column with a thicker film.

 Table 4.
 Troubleshooting Peak Issues in Your Data (Sheet 1 of 3)

Behavior	Characteristic	Cause	Remedy
Tailing	Sample peaks	Column degradation is causing activity	Inject a test mixture and evaluate the column. Replace column if necessary.
		Liner is dirty	Clean or replace the liner.
		Ion source is dirty	Clean the ion source and tune the instrument.
		Glass wool or inlet liner is causing activity	Replace wool with fresh silanized wool and install a clean inlet liner.
		Inlet temperature is too low	Increase the temperature of the inlet.
		Column connections are poor or obstructed	Reconnect the column inlet.
		Stationary phase is not appropriate for your target compounds	Replace the column and choose a more appropriate phase for your analysis.
		Final hold oven temperature is too low	crease the column/oven temperature. Do not exceed the recommended maximum temperature for the stationary phase.
		Transferline temperature is too low	If tailing occurs on late eluting compounds, it is likely the source or transferline temperature is too low.
		Source temperature is too low	If tailing occurs on late eluting compounds, it is likely the source or transferline temperature is too low.
		Poor column characterization	See Changing the Column for information about checking for leaks and column flow.

 Table 4.
 Troubleshooting Peak Issues in Your Data, continued (Sheet 2 of 3)

Behavior	Characteristic	Cause	Remedy
Ghost Peaks	General	Incomplete elution of previous sample	Increase the final oven program temperature or total run time. Increase the column flow rate.
		Carrier gas is contaminated	Replace the gas cylinder or filter.
		Laboratory glassware has caused contamination	Ensure the glassware is clean and contaminant-free.
		Injected sample has decomposed	Decrease the injection port temperature. Use the on-column injection technique.
		Injection solution has matrix present	Adequately clean up your sample prior to injection.
		Inlet or pneumatics are contaminated	Remove the column and bake out the inlet. Use a high-quality septum. Replace the split vent filter. Install an in-line filter between the pneumatics and the inlet.
Missing Peaks	Baseline or background present	Column is broken	Replace the column.
		Column flow is incorrect	Make sure the septa are sealing. Make sure vacuum compensation is turned on.
		Backflush settings are incorrect	Set backflush to <b>off</b> until after injection.
		Column position in S/SL injector is incorrect (too high)	Check the position of the column.
	No baseline or background present	Poor or missing electrical connection	Check the cable connections.
		TSQ 8000 instrument is not collecting data	Make sure the tune file is correct. Verify that the Busy light is on during acquisition. Make sure the filament is not burned out. Close <i>Xcalibur</i> , open <i>Instrument</i> <i>Configuration</i> , press the <b>Reset</b> button on the TSQ 8000 instrument, wait ten seconds, close <i>Instrument Configuration</i> .

## Table 4. Troubleshooting Peak Issues in Your Data, continued (Sheet 3 of 3)

# **Investigating Results Issues**

Behavior	Characteristic	Cause	Remedy
Low Reproducibility of Peaks Area	General	Detector gain is set too low	Retune the gain. Increase the electron multiplier voltage. Increase the target ion count.
		Concentration is not compatible with the dynamic range of the detection system	Verify that the sample concentration is suitable for the TSQ 8000 system.
		Chromatogram and spectrum are blank	Make sure the tune file is correct. Verify that the Busy light is on during acquisition. Make sure the filament is not burned out.
		Injection technique is not appropriate	Use a different injection technique.
		Injection parameters are not appropriate	Verify the injection temperature and flow rates.
		Sample injection technique is not reproducible	Evaluate the sample preparation sequences. Compare the results with a series of standard injections.
		Syringe or septum is leaking	Check and replace the syringe and/or septum at regular intervals.
		Injection port is leaking	Check the column connections. Run a leak check.
		Injection technique is not suitable	Carefully meter the injected amount. Use a clean, good-quality syringe.
		Ion source is dirty	Clean the ion source and retune the instrument.
		Split flow or ratio control is inadequate	Monitor the flow. Replace the in-line filter.

Behavior	Characteristic	Cause	Remedy
Poor Sensitivity	With increased retention time	Carrier gas flow rate is too low	Increase the carrier gas flow rate. Locate and remove possible obstructions in the carrier gas line. Check the septum for leaks. Check the injector/column ferrules for leaks.
	With normal retention time	GC carrier gas line has leaks	Run a leak test and correct leaks.
		Syringe is leaking during injection	Replace syringe or piston seals, if necessary.
		Split injection temperature is too low	Increase the temperature of the injector.
		Voltage is not reaching the lens.	Replace the lens plate and springs if damaged. Remove debris or broken pieces in the manifold. Run a lens check diagnostic. Check the connection by removing or inserting the ion source.
Retention Times	Low reproducibility	DCC is drifting or unstable	Monitor the column pressure or flow. Check and replace the controller, if necessary.
		Injection technique is inadequate	Pick injection technique suitable for the injector and liner you are using.
		Vaporization size of sample inject larger than volume of liner	Reduce the injected amount and/or volume.
		Column temperature is unstable	Check the main oven door and cooling flap. Monitor the column temperature.

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