



## **ISQ 7000**

## **Mass Spectrometers**

## **User Guide**

1R120617-0003 Revision D December 2018



© 2018 Thermo Fisher Scientific Inc. All rights reserved.

ISQ, TSQ, TRACE, TriPlus, TraceFinder, and Chromeleon are trademarks, and Xcalibur is a registered trademark of Thermo Fisher Scientific in the United States.

The following are registered trademarks in the United States and other countries: Microsoft, Windows, and Excel are registered trademarks of Microsoft. Adobe, and Acrobat are registered trademarks of Adobe Systems Incorporated. SilTite is a registered trademark of SGE Analytical Science in the United States. Vespel is a registered trademark of E.I. du Pont de Nemours and Company in the United States and other countries.

All other trademarks are the property of Thermo Fisher Scientific and its subsidiaries.

Thermo Fisher Scientific Inc. provides this document to its customers with a product purchase to use in the product operation. This document is copyright protected and any reproduction of the whole or any part of this document is strictly prohibited, except with the written authorization of Thermo Fisher Scientific Inc.

The contents of this document are subject to change without notice. All technical information in this document is for reference purposes only. System configurations and specifications in this document supersede all previous information received by the purchaser.

Thermo Fisher Scientific Inc. makes no representations that this document is complete, accurate or errorfree and assumes no responsibility and will not be liable for any errors, omissions, damage or loss that might result from any use of this document, even if the information in the document is followed properly.

This document is not part of any sales contract between Thermo Fisher Scientific Inc. and a purchaser. This document shall in no way govern or modify any Terms and Conditions of Sale, which Terms and Conditions of Sale shall govern all conflicting information between the two documents.

Release history: Revision A, March 2018; Revision B, April 2018; Revision C, August 2018; Revision D, December 2018.

Software version: Thermo Xcalibur 4.0 and later, ISQ Series 4.0 and later.

For Research Use Only. Not for use in diagnostic procedures.

## **Contents**

	Preface	xi
	About Your System	xi
	Related Documentationx	ii
	System Requirements	ii
	Safety and Special Notices	iii
	Special Notices	ii
	Safety Symbols and Signal Wordsx	iv
	Hydrogen Safety Precautionsxv	ii
	Using Hydrogen with ISQ 7000 Mass Spectrometers	ix
	Hydrogen Connection Guidelinesx	ix
	Purchasing Hydrogenx	xi
	Properly Storing Hydrogen	xi
	Hydrogen Safety Codes, Standards and Referencesxxi	iii
	Hazardous Substances Precautionsxx	iv
	Biological Hazard Warning Notexx	cv
	Venting Toxic Gases	cv
	Contacting Us	cv
Chapter 1	Confirming Your GC/MS System is Working	.1
•	Checking Power to the System	
	Verifying the Carrier Gas Flow Rate	
	Checking Your Carrier Gas Tank Pressure	
	Checking the Vacuum and Temperature	4
Chapter 2	Changing the Column	.5
•	Replacing the Factory-Installed Column	
	Replacing a column in a ISQ 7000 system with no VPI or an AEI	
	ion source.	6
	Replacing a column on a ISQ 7000 system with a VPI and an	
	ExtractaBrite ion source	.7
	Installing the Column in the GC Inlet2	:3
Chapter 3	Tuning the ISQ 7000 Mass Spectrometer	29
-		
	Accessing ISQ 7000 AutoTune	

C

	Tune Types	. 31
	EI Initial Tune	. 32
	EI Full Tune	. 32
	EI Tune	. 34
	EI Check	
	EI Diagnostics	. 35
	Negative CI Tune	
	Positive CI Tune	
	AEI Full Tune	. 39
	AEI Tune	. 39
	AEI Check	. 40
	AEI Diagnostics.	. 40
	Tuning the ISQ 7000 Mass Spectrometer	
	Using SmartTune	
	Tuning for DFTPP and BFB	
Chapter 4	Creating a Method	59
	Accessing the Method Editor	. 59
	Creating a GC-MS Method in Xcalibur Software	. 62
	Creating an Autosampler Method in Xcalibur Software	. 63
	Creating a Method for the ISQ 7000 Mass Spectrometer	. 67
	Creating a Method for the GC in Xcalibur Software	. 85
	Creating a GC-MS Method in Chromeleon Software	102
Chanter E		177
Chapter 5		
	Determining SIM Ions	
	Importing Transitions to the Method Editor	
	Determining SIM Ions in Chromeleon	
	Importing Transitions to the Chromeleon Instrument Method Editor	158
Chapter 6	Running a Sample	161
onaptor o	Preparing Your Sample	
	Running a Sequence	
		102
Chapter 7	Exploring Your Data	.175
Chapter 8	Optimizing Your Method	.185
•	Changing the Chromatographic Separation	
	Finding the Best Way to Make an Injection	
	Improving the Way You Prepare Samples	
	Changing the Scan Rate	
	Narrowing the Mass Range	
	Adjusting the Transfer Line Temperature	
	Modifying an Automatic Tune	

Chapter 9	Troubleshooting	203
•	Setting Instrument Conditions for Troubleshooting	. 204
	Checking Air/Water Spectra	. 205
	No Ions Present in Scans	. 211
	Checking the ISQ 7000 System Firmware	. 211
	How to Know When Your System Needs Maintenance	.214
	Investigating Baseline Issues	. 218
	Investigating Peak Issues	. 220
	Investigating Results Issues	. 223
	Reconfiguring Your Instrument	. 225
	Upgrading the Software	. 237

## thermo scientific

## **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
- re-calibration
- 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.

CE 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 and ISQ 7000 standards: EMC EN 61326-1:2013. Safety IEC 61010-1:2010, IEC 61010-2-010:2014, IEC 61010-2-081:2015.
- TSQ 8000, TSQ 8000 Evo, TSQ Duo, and TSQ 9000 standards: EMC EN 61326-1:22013. Safety IEC 61010-1:2010, IEC 61010-2-010:2014, IEC 61010-2-081:2015
- Restriction of Hazardous Substances Directive (2011/65/EU)

#### Low Voltage Safety Compliance

This device complies with Low Voltage Directive 2014/35/EU 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.



**ATTENTION** Lisez et comprenez les différentes précautions, ainsi que les signes et symboles de ce manuel relatifs à l'utilisation sûre de ce produit, avant d'utiliser le dispositif.

## 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.

## thermo scientific

# 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.

## thermo scientific

### **WEEE Konformität**

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



Thermo Fisher Scientific hat Vereinbarungen mit Verwertungs-/Entsorgungsfirmen in allen EU-Mitgliedsstaaten getroffen, damit dieses Produkt durch diese Firmen wiederverwertet oder entsorgt werden kann. Mehr Information über die Einhaltung dieser Anweisungen durch Thermo Fisher Scientific, über die Verwerter, und weitere Hinweise, die nützlich sind, um die Produkte zu identifizieren, die unter diese RoHS Anweisung fallen, finden sie unter www.thermoscientific.com/rohsweee.

### **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<sup>™</sup> ISQ<sup>™</sup> 7000 single quadrupole GC-MS system. The ISQ 7000 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 gone. The heated ion volume, lens stack, and ion optics path in the ISQ 7000 system ensure that the system stays cleaner longer, but when the system no longer meets your analytical needs, restoring performance is quick and easy. The need to break vacuum, cool off your system, and spend hours cleaning, reassembling, and restoring the system is greatly reduced if you have an ExtractaBrite ion source and a NeverVent-enabled system. 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. An AEI ion source, which provides unmatched sensitivity, is also available on the system.

#### Contents

- About Your System
- Related Documentation
- System Requirements
- Safety and Special Notices
- Hydrogen Safety Precautions
- Hazardous Substances Precautions
- Contacting Us

### **About Your System**

Gas chromatography/mass spectrometry (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 through the use of an optional autosampler, and then separated for presentation to the MS. The MS will then generate a mass spectrum of the GC eluent and its components, which can be used for qualitative identification, as well as accurate and precise quantification of the individual compounds present in the sample.



**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.



**AVERTISSEMENT** Les systèmes Thermo Fisher Scientific fonctionnent de manière sûre et fiable dans des conditions ambiantes minutieusement régulées. La protection fournie par l'équipement peut être entravée si ce dernier est utilisé d'une manière non spécifiée par le fabricant. Si vous maintenez un système en dehors des spécifications listées dans ce guide, des défaillances de types divers sont possibles, pouvant notamment entraîner des blessures, voire la mort. La réparation des défaillances d'instruments liées à une utilisation non conforme aux spécifications du fabricant est expressément exclue de la garantie standard et de la couverture prévue par un contrat de maintenance.

### **Related Documentation**

ISQ 7000 includes Help and these manuals as PDF files:

- ISQ 7000 Preinstallation Requirements Guide PN 1R120617-0001
- ISQ 7000 Hardware Manual PN 1R120617-0002
- ISQ 7000 User Guide PN 1R120617-0003
- ISQ and TSQ GC-MS Spare Parts Guide PN 1R120617-0004
- Direct Probe System User Guide PN 1R120505-0006

#### To view product manuals

Open the **Manuals** folder on your desktop.

#### ✤ To open Help

- From the ISQ 7000 window in the instrument control software, choose Help > ISQ 7000 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.thermofisher.com.

### **System Requirements**

System	Requirements
Hardware	<ul> <li>3.6 GHz dual-core processor enabled</li> <li>16 GB RAM with system managed memory enabled</li> <li>DVD drive</li> <li>Resolution display 1280×1024 (SXGA)</li> <li>20 GB available on drive C</li> <li>NTFS format</li> </ul>
Software	<ul> <li>Microsoft<sup>™</sup> Windows<sup>™</sup> 10 Operating System (64-bit) English only or Windows 7 Professional Operating System (64-bit)</li> <li>Microsoft .NET Framework 4.0 or later</li> <li>Thermo Scientific<sup>™</sup> Xcalibur<sup>™</sup> and Foundation software<sup>a</sup></li> <li>Thermo Scientific<sup>™</sup> TraceFinder<sup>™</sup> software<sup>b</sup></li> <li>Thermo Scientific<sup>™</sup> Dionex<sup>™</sup> Chromeleon software<sup>b</sup></li> </ul>

Your ISQ 7000 data system must meet these minimum requirements.

<sup>a</sup> Check release notes for compatibility with ISQ Series instrument control software.

<sup>b</sup> Check release notes for compatibility with Thermo Foundation, Xcalibur, and ISQ Series instrument control software.

### **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.

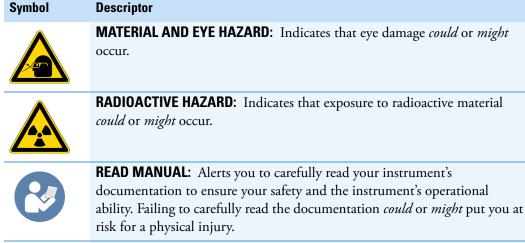
Note Highlights information of general interest.

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.

e	
Symbol	Descriptor
	<b>BIOHAZARD:</b> Indicates that a biohazard <i>will, 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>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.





**TOXIC SUBSTANCES HAZARD:** Indicates that exposure to a toxic substance could occur and that exposure *could* or *might* cause personal injury or death.



For the prevention of personal injury, this general warning symbol precedes the **WARNING** 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*.

Tous les symboles de sécurité sont suivis des mots **AVERTISSEMENT** ou **ATTENTION**, qui indiquent le degré de risque de blessures personnelles, de dommages à l'instrument, ou des deux. Les mentions « Attention » et les avertissements sont suivis d'un descripteur. Un **AVERTISSEMENT** vise à empêcher des actions inappropriées pouvant entraîner des blessures personnelles. Une mention **ATTENTION** vise à empêcher des actions inappropriées pouvant entraîner des blessures personnelles ou des dommages à l'instrument. Vous pouvez trouver les symboles de sécurité suivants sur votre instrument ou dans ce guide.

#### Symbol Descriptor



**RISQUE BIOLOGIQUE** : indique qu'un risque biologique va, peut ou pourrait survenir.



**RISQUE DE BRÛLURE** : vous avertit de la présence d'une surface chaude qui peut ou pourrait entraîner des blessures par brûlure.



**RISQUE D'ÉLECTROCUTION** : indique qu'un choc électrique peut ou pourrait survenir.

Symbol	Descriptor
	<b>RISQUE D'INCENDIE</b> : indique qu'un risque d'incendie ou d'inflammabilité peut ou pourrait survenir.
FLAMMABLE GAS 2	<b>RISQUE DE GAZ INFLAMMABLE</b> : vous avertit que des gaz sont comprimés, liquéfiés ou dissous sous pression et qu'ils peuvent s'enflammer au contact d'une source d'inflammation. Ce symbole indique que ce risque peut ou pourrait entraîner une blessure physique.
	<b>GANTS REQUIS</b> : indique que vous devez porter des gants pour effectuer une tâche, sans quoi une blessure physique peut ou pourrait survenir.
	<b>RISQUE PHYSIQUE ET CHIMIQUE</b> : indique que des dommages chimiques ou une blessure physique peuvent ou pourraient survenir.
	<b>DOMMAGES À L'INSTRUMENT</b> : indique que l'instrument ou le composant pourrait subir des dommages. Ces dommages pourraient ne pas être couverts pas la garantie standard.
	<b>RISQUE SOULÈVEMENT</b> : indique qu'une blessure physique peut ou pourrait survenir si un objet n'est pas soulevé par deux personnes ou plus.
	<b>RISQUE MATÉRIEL ET YEUX</b> : indique que des dommages aux yeux peuvent ou pourraient survenir.
	<b>RISQUE RADIOACTIF</b> : indique qu'une exposition à des matériaux radioactifs peut ou pourrait survenir.
	<b>CONSULTER LE MANUEL</b> : vous avertit de lire attentivement la documentation de votre instrument afin de garantir votre sécurité et la

documentation de votre instrument afin de garantir votre sécurité et la capacité opérationnelle de l'instrument. Ne pas lire attentivement la documentation peut ou pourrait vous exposer à un risque de blessure physique.

#### Symbol Descriptor



**RISQUE DE SUBSTANCES TOXIQUES** : indique que l'exposition à une substance toxique peut survenir et que l'exposition peut ou pourrait entraîner des blessures personnelles ou la mort.



Pour prévenir les blessures personnelles, ce symbole général d'avertissement précède le mot **AVERTISSEMENT** et est conforme à la norme ISO 3864-2. Dans le vocabulaire des signes ANSI Z535, ce symbole indique un risque de blessures personnelles si l'instrument est utilisé de manière inappropriée ou en cas d'actions dangereuses. Ce symbole et un autre symbole de sécurité approprié vous avertissent d'un risque imminent ou potentiel pouvant entraîner des blessures personnelles.

### **Hydrogen Safety Precautions**

Hydrogen is a colorless, odorless, highly flammable gas with the molecular formula  $H_2$ . 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.



**AVERTISSEMENT RISQUE D'INCENDIE** : l'utilisation d'hydrogène comme gaz vecteur est dangereuse. L'hydrogène est potentiellement explosif et doit être utilisé avec une extrême précaution. Toute utilisation d'hydrogène gazeux doit être évaluée par le personnel de santé et de sécurité approprié et toutes les installations de systèmes d'hydrogène doivent se conformer aux codes et aux normes en vigueur. Thermo Fisher Scientific décline toute responsabilité en cas d'utilisation inappropriée d'hydrogène comme gaz vecteur.

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:

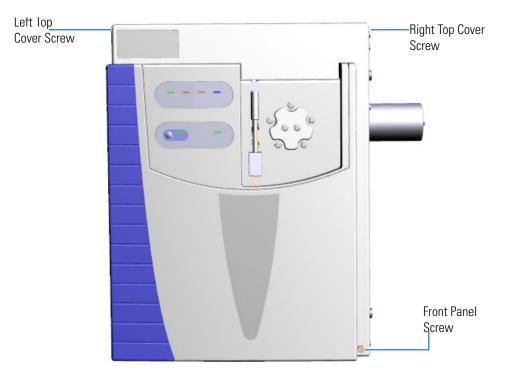
"What hydrogen hazards associated with this project are most likely to occur?"

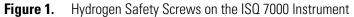
"What hydrogen hazards associated with this project have the potential to result in the worst consequences?"

- 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 ISQ 7000 Mass Spectrometers

To use hydrogen with the ISQ 7000 instrument, you must always shut off the GC carrier gas before venting or turning off the ISQ 7000 instrument. There are three hydrogen safety screws on the ISQ 7000 instrument that **must** be in place. These are attached to your instrument at the factory.





Make sure all the covers and panels of the ISQ 7000 instrument are firmly attached before powering on the ISQ 7000 instrument. If you vented the system, make sure the vent valve is tightly closed before powering on the system. Make sure all fittings, ferrules, and o-rings are sealed prior to powering on the system.

### **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 may 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 may 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.





**AVERTISSEMENT** Avant d'utiliser des substances dangereuses (toxiques, nocives, etc.), veuillez lire attentivement les indications et informations relatives au risque reprises sur la fiche de données de sécurité adéquate. Utilisez un équipement de protection individuelle conformément aux exigences de sécurité.

### **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.



**AVERTISSEMENT** L'utilisateur ou l'opérateur est responsable de la manipulation sûre des composés chimiques et biologiques dangereux, y compris, sans s'y limiter, les échantillons bactériens ou viraux et les déchets associés, conformément aux réglementations internationales et locales.

### **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.thermofisher.com for information about our products.

#### ✤ To get local contact information for sales or service

Go to www.unitylabservices.com/en/home.html.

#### ✤ 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.

## **Confirming Your GC/MS System is Working**

Use the information in this chapter to confirm that your GC/MS system has power, the carrier gas rate is correct, the gas tank has enough pressure, the system has reached vacuum and temperature, and is leak-free.

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

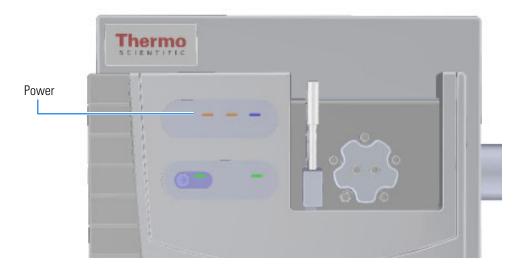
#### Contents

- Checking Power to the System
- Verifying the Carrier Gas Flow Rate
- Checking Your Carrier Gas Tank Pressure
- Checking the Vacuum and Temperature

**Note** Many nitrile and latex gloves not certified for clean room use contain silicone mold releasing agents that will contaminate the instrument. For this reason, clean room gloves are strongly recommended when you work with the ISQ 7000 mass spectrometer. We recommend Cardinal Health CP100 Nitrile Cleanroom Gloves. See the *ISQ and TSQ GC-MS Spare Parts Guide* for ordering information.

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

To confirm that the ISQ 7000 system is powered on, make sure the Power light on the front panel is solid green. If it is not lit, the ISQ 7000 system is not powered on. To power it on, reach around the right side of the instrument and pull up the power switch on the back. If the instrument still doesn't power on, check the electrical connections and wall outlet.



#### **Figure 1.** Checking the Power on the ISQ LT System

To confirm that a TRACE GC Ultra is powered on, make sure the front LCD panel displays information and that it is not blank. To power-on the TRACE GC Ultra, reach over the top center of the instrument and pull up on the large plastic ribbed power switch on the back. To confirm that a TRACE 1300 GC is powered on, make sure that power light on the status panel is solid green. To confirm that a TRACE 1310 GC is powered on, see if the touchscreen main menu has appeared. To power-on the TRACE 1300 or 1310 GC, reach over the top right of the instrument and pull up on the large plastic ribbed power switch on the back. If the instrument still doesn't power on, check the electrical connections and wall outlet.

### **Verifying the Carrier Gas Flow Rate**

Once you confirm that the system is powered on, you need to verify that carrier gas rate is what you expect.

**Note** The TRACE GC Ultra beeps to indicate that the carrier gas is turned off or set incorrectly.

#### To check the carrier gas flow rate

- Access the carrier gas menu. On the TRACE GC Ultra, press the Carrier button on the front of the GC. On the TRACE 1310 GC, choose Instrument Control and then Front/Back Inlet. On a TRACE 1300 GC, open the Xcalibur software by clicking on the Xcalibur icon on the computer desktop. On the Xcalibur roadmap, select the TRACE 1300 from the instrument list in the side panel. This opens the Status Panel.
- 2. Display the column flow.

3. 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, see the troubleshooting section of your GC user documentation.

### **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 may run out of gas in the middle of a run, which could compromise the results of your data.

- 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 500 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.
- 4. Set the second stage regulator pressure between 80 and 100 psi (560 kPa to 700 kPa).

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

Use the lights on the front of the ISQ 7000 instrument to check the vacuum and temperature of the instrument.

To check the vacuum, look at the **Vacuum** light. When the light is a solid green, the ISQ mass spectrometer is under sufficient vacuum. If it is slowly blinking orange, you have not achieved vacuum yet. If it is blinking orange quickly, you have a large leak that prevented the instrument from achieving vacuum. If this is the case, you need to find and fix the leak, then turn the power off and on. Most likely, the column nut needs to be tightened, the column was not installed correctly, or the vent valve was not completely closed.

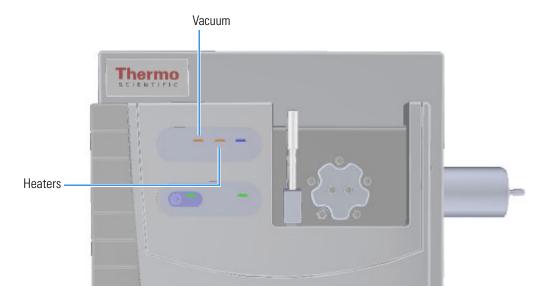


Figure 2. Using the Lights on the ISQ 7000 Instrument

To check the temperature, look at the **Heaters** light. When the **Heaters** light is a solid green, the ISQ 7000 instrument is at temperature. If it is blinking orange, the ion source or transfer line are not at temperature. If the 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.

# 2

## **Changing the Column**

The ISQ 7000 mass spectrometer ships with a factory-tested 15 m x 0.25mm i.d. TG-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 need to install a column that accommodates the type and quantity of samples you will be running. You should choose a column that gives you the best possible resolution, analysis speed, and quantitation.

**Note** Many nitrile and latex gloves not certified for clean room use contain silicone mold releasing agents that will contaminate the instrument. For this reason, clean room gloves are strongly recommended when handling the column. We recommend Cardinal Health CP100 Nitrile Cleanroom Gloves. See the *ISQ and TSQ GC-MS Spare Parts Guide* for ordering information.

**Note** Some of the cleaning procedures in this section require the use of methanol. If methanol is unavailable or prohibited, substitute LCMS-grade or GC-grade ethanol or isopropyl alcohol. Do not use denatured ethanol as it may contain impurities that contaminate the GC-MS system.

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 you are going to 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 increases the analysis time) to match the separation power of shorter columns. Typical column sizes for GC/MS have inside diameters (ID) of 0.25 mm. Smaller ID columns, such as the 0.15 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 desired phase ratio.

Thick films with small internal diameters will give very strong interactions with the analytes, which can result in longer analysis time and peak tailing. Large ID columns with thin films will 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.

• 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 meters 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 Fisher Scientific column. You can also refer to our catalog or visit our website at www.thermofisher.com.

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

The procedure for replacing a column depends on your system configuration. If your system does not have a VPI or has an AEI ion source installed, see "Replacing a column in a ISQ 7000 system with no VPI or an AEI ion source" on page 6. If your ISQ 7000 MS has a vacuum probe interlock (VPI) and the ExtractaBrite ion source is installed, see "Replacing a column on a ISQ 7000 system with a VPI and an ExtractaBrite ion source" on page 17.

### Replacing a column in a ISQ 7000 system with no VPI or an AEI ion source

If your ISQ 7000 system has no VPI or has an AEI ion source installed on the system, follow the instructions below to replace the column.

#### To replace the factory-installed column in the ISQ 7000 system in a system with no VPI or with an AEI ion source installed

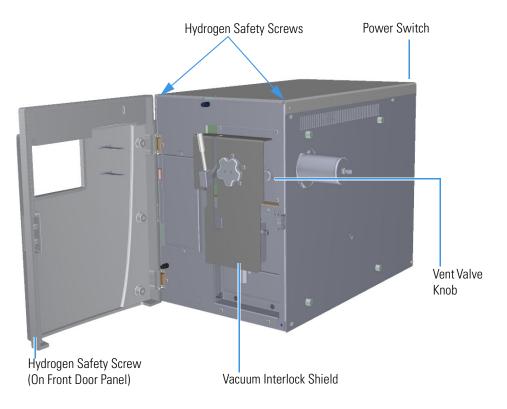
**Note** If you are running samples, stop the acquisition before powering off the system.

- 1. Cool down the GC oven and injector. See the GC documentation for information.
- 2. Open the ISQ 7000 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 ISQ 7000 system.

3. On the left side of the instrument, push down on the power switch to power-off the ISQ 7000 system.





- 4. Open the front door of the instrument.
- 5. Open the front door of the instrument.

- 6. 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.
- 7. Remove the current column from the MS transfer line. To replace the column in the GC inlet, follow the instructions in "Installing the Column in the GC Inlet" on page 23.
- Connect the column to the MS transfer line. When connecting the column to the transfer line, you may use either the spring loaded transfer line nut with the graphite Vespel<sup>™</sup> ferrule or the regular transfer line nut

**Note** For best results, we recommend you use the spring loaded transfer line nut.

\* 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 spare parts guide for ordering information.

- 1. Lower the oven temperature and allow it to cool.
- 2. If the ISQ 7000 system is running, shut down and vent it. See the instrument's hardware manual for instructions.



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

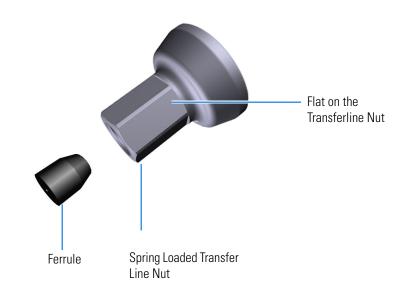


**ATTENTION RISQUE DE BRÛLURE :** l'injecteur, les détecteurs, le four et la ligne de transfert peuvent être chauds. Laissez-les refroidir avant de les toucher.

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.



**Figure 4.** Transfer Line Nut and Graphite Vespel Ferrule Orientation

- 8. Insert the column into the measuring tool, which is in the MS Toolkit (See Figure 5), so that it is even with the lines at the end of the column. Figure 6 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 5. Column Measuring Tool

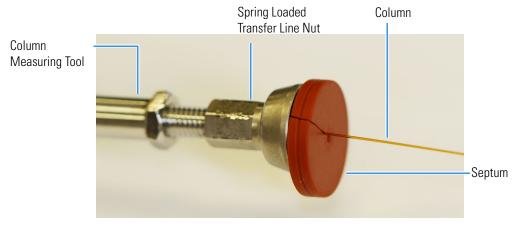


- 11. While holding the column measuring tool steady, tighten the spring loaded transfer line nut with a 1/4" wrench until the column just stops moving in the ferrule.
- 12. Turn the spring loaded transfer line nut 1 flat (60°) backward so the column is able to move in the ferrule with slight resistance.
- 13. Line up the outlet of the column with the arrows on the end of the column measuring tool.



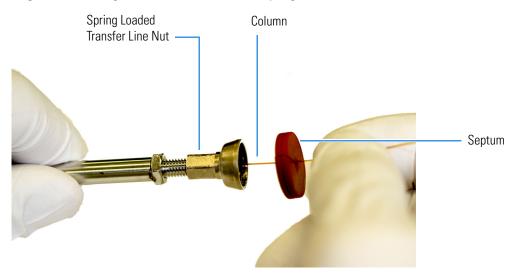
Figure 6. Lining Up the Column in the Column Measuring Tool

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.



**Figure 7.** Positioning the Septum

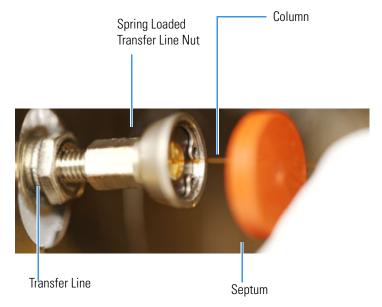
- 15. Pull the column back from the spring loaded transfer line nut. Do not move the septum from its position on the column.
  - Figure 8. Pulling the Column Back from the Spring Loaded Transfer Line Nut



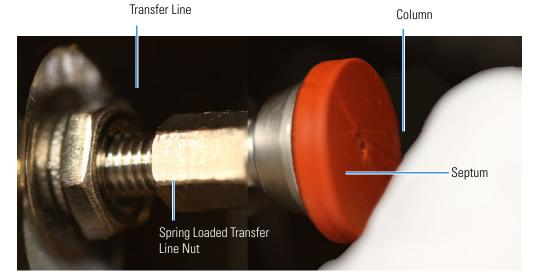
- 16. Loosen the transfer line nut from the column measuring tool.
- 17. Remove the column, transfer line nut and ferrule from the column measuring tool, making sure not to move the septum from its location on the column.

**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 9. 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 (60°) backward.
- 21. Position the column in the transfer line using the cut septum to measure the correct length you should insert the column.



#### Figure 10. Positioning the Column in the Transfer Line

- 22. Tighten the spring loaded transfer line nut 1 flat (60°) 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.



**ATTENTION RISQUE DE BRÛLURE :** l'injecteur, les détecteurs, le four et la ligne de transfert peuvent être chauds. Laissez-les refroidir avant de les toucher.

- 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 ISQ 7000 instrument.

- 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.
- \* To connect the column using the regular transfer line nut

**Note** For best results, we recommend you use the spring loaded transfer line nut. See "To connect the column using the spring loaded transfer line nut" on page 8.

- 1. Lower the oven temperature and allow it to cool.
- 2. If the ISQ 7000system is running, shut down and vent it. See the instrument's hardware manual for instructions.



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



**ATTENTION RISQUE DE BRÛLURE :** l'injecteur, les détecteurs, le four et la ligne de transfert peuvent être chauds. Laissez-les refroidir avant de les toucher.

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.

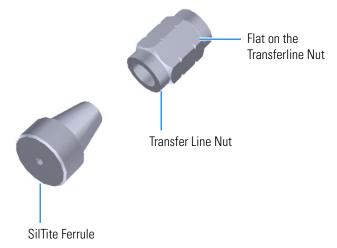


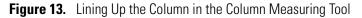
Figure 11. Transfer Line Nut and SilTite Ferrule Orientation

- 7. Insert the column into the measuring tool, which is in the MS Toolkit (See Figure 12), so that it is even with the lines at the end of the column. Figure 13 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 12. 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 (60°) 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.





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.

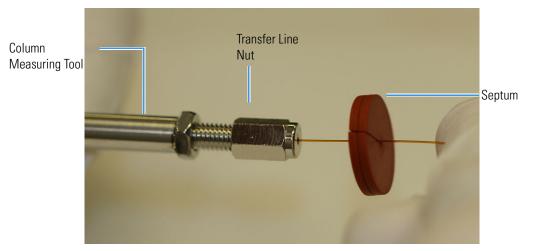
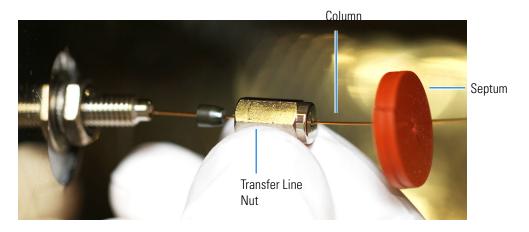


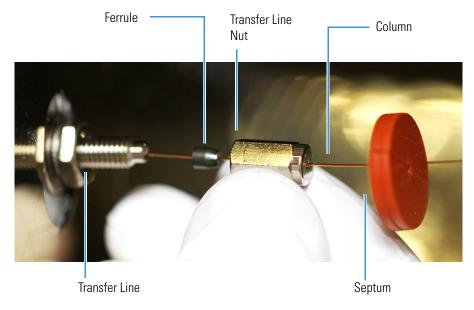
Figure 14. Positioning the Septum

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

Figure 15. Pulling the Column Back from the Transfer Line Nut



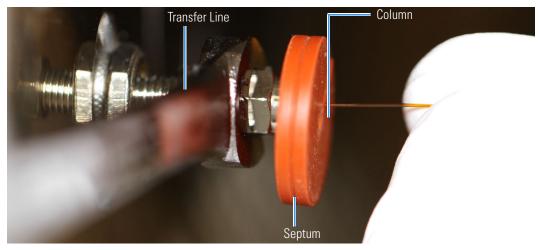
- 15. Loosen the transfer line nut from the column measuring tool.
- 16. Remove the column, transfer line nut and ferrule from the column measuring tool, making sure not to move the septum from its location on the column.
- 17. Insert the column into the transfer line.



#### Figure 16. 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 (60°) 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 (60°) 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 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.



**ATTENTION RISQUE DE BRÛLURE :** l'injecteur, les détecteurs, le four et la ligne de transfert peuvent être chauds. Laissez-les refroidir avant de les toucher.

- 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 ISQ 7000 instrument.

Once the ISQ 7000 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.

# Replacing a column on a ISQ 7000 system with a VPI and an ExtractaBrite ion source

If your ISQ 7000 system has a VPI and has an ExtractaBrite ion source installed, follow the instructions below to change the column.

- To replace the column on a ISQ 7000 system with a VPI and an ExtractaBrite ion source installed
- 1. Cool the oven, transfer line and ion source:
  - a. On the GC, set the Oven to Off.

- b. On the ISQ 7000 Dashboard set the MS transfer line temp to 40 °C and the ion source temp to 175 °C (to avoid excessive oxidation of source parts or contamination from the V-lock source plug).
- 2. Using the source removal tool and vacuum interlock, remove the ion source cartridge. See the *ISQ 7000 Hardware Manual* for instructions to remove the ion source cartridge.
- 3. Place the ion source cartridge on the source holder and set aside.
- 4. Place the V-lock source plug in the source plug holder.
- 5. Attach the source exchange tool to the V-lock source plug in the source plug holder.
- 6. Twist the plug until it aligns securely in the grooves in the source exchange tool and remove the plug from the holder. The V-lock source plug is securely attached to the source exchange tool when the alignment grooves on each piece match up.

Figure 18. Attaching the V-lock Source Plug to the Source Exchange Tool

**Note** Use compressed air to blow all the dust off the V-lock source plug before inserting it into the mass spectrometer.

7. Once the ion source temperature has dropped below 200 °C, insert the barrel end of the source exchange tool into the vacuum interlock and twist it clockwise to lock the source exchange tool into position. Be sure the black handle remains fully extended and locked.

**Note** If the ion source temperature is above 200 °C, parts of the ion source or the V-lock source plug could oxide and be damaged, causing leaks in the MS system.

- 8. Evacuate the VPI.
  - a. Confirm that the source removal tool is properly engaged in the VPI.
  - b. Press the blue **Evacuate** button on the front of the instrument.
  - c. The **Evacuate** light will begin to flash green, and should continue to flash green for approximately 20 seconds.
- 9. If the pressure has returned to an acceptable value after the 20-30 second wait, the evacuate light will turn off and the **Ready to Open** light will be solid green. At that point,

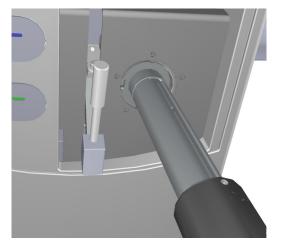
Alignment Grooves

the air has been evacuated from the instrument and it is safe to open the vacuum interlock valve.

**Note** If the vacuum light flashes amber, there is a leak in the system.

- 10. Pull the vacuum interlock handle up when the Ready to Open light is a solid green.
- 11. Twist the handle of the tool slightly to the left until it is lodged into the left-most track. See Figure 19.

Figure 19. Inserting the V-lock Source Plug



12. Push the handle toward the instrument until the end of the handle aligns with the engraved line at the end of the barrel. When you reach this line, the tool is all the way in and the V-lock source plug is securely placed in the instrument. See Figure 20.

**IMPORTANT** The V-lock source plug should remain attached to the source exchange tool. Do not rotate the source exchange tool handle to attempt to disengage the source plug.





13. Wait for oven and transfer line temperatures to drop below 50 °C to avoid burns before proceeding to touch the column and nut.

**IMPORTANT** Monitor the foreline pressure when removing the column and nut to confirm that the V-lock source plug is properly sealing the transfer line. If the pressure exceeds 1 Torr, the leak is excessive, and the source plug should be reseated or inspected for damage, especially the white o-ring on the end of the source plug. If the turbomolecular pump is forced off by the vacuum protection, the power might need to be reset to start the pumps.

14. Remove the current column from the MS transfer line. To install the column in the GC inlet, follow the instructions in "Installing the Column in the GC Inlet" on page 23.

**IMPORTANT** If a column will not be immediately added to the transfer line (such as when column conditioning or other GC maintenance is to be performed), then blank off the transfer line with a transfer line nut with a no-hole graphite Vespel ferrule. Once the transfer line is blanked off, the V-lock source plug can be removed until a column is ready to be installed into the transfer line.

15. Unwind an appropriate column length to insert into the transfer line along the front of the instrument. Leaving about an inch gap between the column and the left side of the front panel will usually give you an appropriate length of column for installation.

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

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

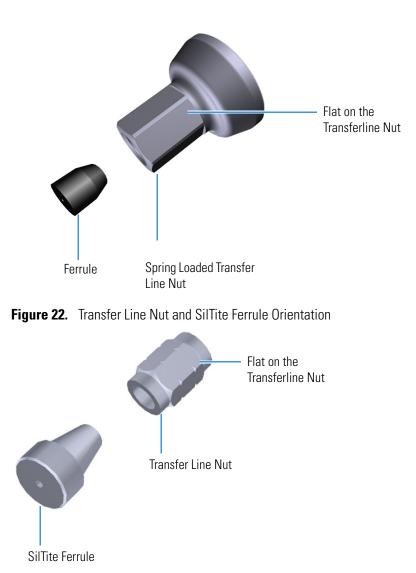
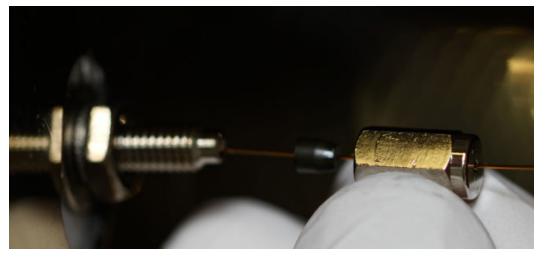


Figure 21. Transfer Line Nut and Graphite Vespel Ferrule Orientation

- 20. Insert the new column through the MS transfer line.
- 21. Carefully extend the column out the front to allow application of the nut and ferrule.
- 22. Insert the column through the nut and ferrule (flat side of the nut faces the MS).
- 23. Use a scoring wafer to score and then remove the last 10 mm of column to provide a clean, well-cut end.
- 24. Wipe the column with an alcohol soaked-wipe after inserting through the ferrule.
- 25. Carefully push the column back into the GC transfer line while keeping the nut and ferrule on the column.

26. Insert the column into the MS transfer line and tighten the nut until the column just resists sliding through the ferrule.





- 27. Loosen the nut <sup>1</sup>/<sub>4</sub> turn and gently push the column into the MS transfer line until it just touches the V-lock source plug.
- 28. Pull the column  $\frac{1}{2}$  to 1 mm away from the V-lock source plug and tighten the nut  $\frac{1}{2}$  turn.

**Note** To avoid forcing the column into the V-lock source plug when tightening the nut, the column should be pulled back approximately 1 mm before tightening the nut.

- 29. Ensure the column and nut are correctly installed in the MS transfer line.
- 30. Remove the V-lock source plug with the insertion/removal tool.
- 31. Lower the handle to close the vacuum interlock.
- 32. Remove the source exchange tool.
- 33. Allow the V-lock source plug to cool.
- 34. Carefully remove the V-lock source plug from the insertion/removal tool using the source plug holder.

**Tip** To avoid collecting dust on the V-lock source plug, store it in a closed container when not in use.

35. Using the source holder, place the ion source on the insertion/removal tool and install the ion source using the vacuum interlock.

Once the ISQ 7000 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.

# Installing the Column in the GC Inlet

- 1. Remove the current column:
  - a. 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.



**ATTENTION RISQUE DE BRÛLURE :** l'injecteur, les détecteurs, le four et la ligne de transfert peuvent être chauds. Laissez-les refroidir avant de les toucher.

- b. Unscrew the transfer line nut and remove the column from the transfer line.
- c. Unscrew the injector and detector nuts and remove the column.
- d. Remove the column from the column rack and from the GC.
- 2. 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.
- 3. 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.
- 4. 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.

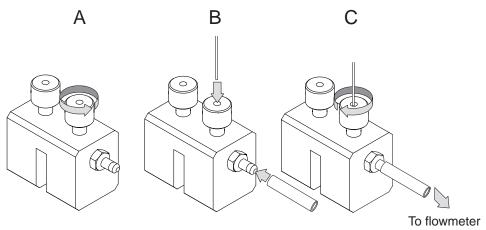


**ATTENTION DOMMAGES À L'INSTRUMENT** : ne laissez pas le connecteur du débitmètre de la colonne dépasser les 80 °C (176 °F). Dans le cas contraire, il va fondre et endommager l'instrument.

- c. Repeat the leak check until no leaks are indicated.
- 5. Calibrate the carrier gas flow (column evaluation):

a. Carefully push the capillary column end into the flowmeter section of the 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.

6. 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.
- 7. If necessary, condition the column before inserting it into the ISQ 7000 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.

**IMPORTANT** Do not leave the source plug in the instrument for an extended amount of time. Add a no-hole ferrule to the column during conditioning. Then remove the source plug from the instrument. (You do not have to reinstall the ion source cartridge.) Reinstall the source plug into the VPI before removing the no-hole ferrule.

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.



**ATTENTION DOMMAGES À L'INSTRUMENT** : les matières rejetées par la colonne (ressuage de la colonne) lors du conditionnement peuvent contaminer la source d'ions si la colonne est insérée dans la ligne de transfert lors de la phase à haute température du conditionnement.



**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.



**AVERTISSEMENT RISQUE D'INCENDIE** : n'utilisez pas d'hydrogène comme gaz vecteur pour le conditionnement de votre colonne. Il pourrait être ventilé à l'intérieur du four et présenter un risque d'explosion.

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.



**ATTENTION DOMMAGES À L'INSTRUMENT** : ne dépassez jamais la température de fonctionnement maximum de la colonne indiquée par le fabricant.

**2 Changing the Column** Replacing the Factory-Installed Column

# **Tuning the ISQ 7000 Mass Spectrometer**

Tuning will improve the performance of your ISQ 7000 mass spectrometer. For optimum stability, you should start tuning after the 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 (a cold system), it takes longer (up to 4 hours) for the instrument components to reach stable vacuum and temperature. If you did not vent the ISQ 7000 instrument (system is hot), it takes approximately 30 minutes for the components to reach vacuum and temperature.

**IMPORTANT** Be sure to give the ISQ 7000 instrument enough time to stabilize. Otherwise, you may see mass drift, mass spectral changes, or changes in the fragmentation of your data.

#### Contents

- Accessing ISQ 7000 AutoTune
- Tune Types
- Tuning the ISQ 7000 Mass Spectrometer
- Using SmartTune
- Tuning for DFTPP and BFB

# Accessing ISQ 7000 AutoTune

#### ✤ To access ISQ 7000 AutoTune

Open the ISQ 7000 Dashboard and click **AutoTune Options** to open the AutoTune window.

Figure 25. Accessing ISQ 7000 AutoTune from the ISQ 7000 Dashboard

📔 ISQ 7000 Dashboard	
AutoTune Options	View Tune Report
Air & Water / Tune	AutoSIM
Method Editor	Xcalibur
Instrument Control	Shut Down
	<b>^</b>
Status Analyzer Power Maint	enance
Status: Idle	
Actua	Set Point
Instrument tune file	
AEI source	
Temperatures	
✓ MS transfer line temp: 250 °C	
√ Ion source temp: 203 °C	200 °C
Pressures Foreline pressure: 53 mTon	
<ul> <li>Foreline pressure: 53 mTon</li> <li>Misc</li> </ul>	
Vacuum: OK	
Turbo pump speed: 100 %	
CI reagent gas flow 0.00 mL/min	0.00 mL/min
CI gas type None	

8. If you have configured Advanced AutoTune or SmartTune for your EI or AEI ion source you may access those options by right-clicking on **AutoTune Options** and choosing from a drop-down menu. See Figure 26.

AutoTune Options		View Tune Rej	port
Advanced AutoTune El SmartTune	-	AutoSIM	
AEI SmartTune Method Editor		Xcalibur	
Instrument Control		Shut Down	
<b>2</b>	\$	1	
Status Analyzer Power Status: Idle	Maintenance		
Instrument tune file AEI source	Actual	Set Point	
Temperatures			
<ul> <li>MS transfer line temp:</li> <li>Ion source temp:</li> </ul>	250 ℃ 203 ℃	250 °C 200 °C	
*	3 mTorr		
Misc Vacuum: Turbo pump speed: Cl reagent gas flow 0.00 Cl gas type	OK 100 % mL/min 0.0 None	00 mL/min	

Figure 26. Selecting from AutoTune Options

The list of available tune types opens.

# **Tune Types**

This section explains the different tune types in ISQ 7000 AutoTune.

EI Initial Tune
EI Full Tune
EI Tune
EI Check
EI Diagnostics
Negative CI Tune
Positive CI Tune
AEI Full Tune
AEI Tune
AEI Check
AEI Diagnostics

# **El Initial Tune**

EI Initial Tune (default)—This tune is used to reset parameters that could be used as a starting point for all the other tunes after cleaning the ion source. As a result, use this tune only when the ion source is clean. This tune should also be used when changing from a legacy tune file generated with earlier instrument control software. The EI Initial tune starts with the tune file stored in the instrument at the factory, which must have correct frequency set points and close mass calibration for this tune to pass. During the tune, detector gain is calibrated to generate approximately 300,000 electrons for each ion that strikes the detector. The tune then performs a resolution and mass calibration, tunes the lenses and performs a leak check. The quadrupole offset and repeller voltages are set to low values to easily tune resolution, but may make this tune less sensitive than other tunes that maximize these voltages. Additionally, this tune generates spectra that are the closest in appearance to the factory tune and can be used by your service engineer to create a new factory tune file on the instrument.

# **El Full Tune**

**EI Full Tune (default)**—This tune is used to completely retune the system. It takes the longest amount of time to run, but the advantage of using it is that it re-optimizes nearly all the parameters affecting the signal. This type of tune will perform a mass calibration, tune the lenses and resolution, and perform a leak check. The detector gain will be calibrated to generate 300,000 electrons for every ion that strikes the detector. You should run an EI full tune when the tune check is not adequate, when the electron multiplier is getting old (tuning to high electron multiplier voltages), or the first tune after you replace the electron multiplier. Unless your SOP requires it, this is not the best tune to use on a daily basis because of the length of time it takes to run it.

Figure 27shows a typical tune report for an EI Full Tune on a system using helium as a carrier gas.

#### Figure 27. Typical El Full Tune Report

			ISQ Tune Re	esults		
Lab: Austin R&D					Instrument Name: ISC	2
Tune Completed -	Fri Feb 16 12:49:52	2018			Instrument ID: 10098	2000
Tune File: AutoTu	ne_EI_2018-02-16-1	2-49-52.isqtune			User:	
Tune Type: El Fu	ll Tune (default)					
Starting Tune File:	(Last Saved Tune	•)				
m/z 69.07	m/z 131.00	m/z 219.00	m/z 414 00	m/z 502.00	Ion Source Type	EI
Int 209,991,312	Int 97,800,696	Int 98,427,720	Int 3,902,080	Int 4,089,884	Polarity	Positive
FWHM 0.67	WHM 0.67 FWHM 0.69 FWHM 0.67 FWHM 0.60 FWHM 0.54	FWHM 0.54	Electron Lens Voltage	5 V		
					Electron Energy	70 eV
					Emission Current	50 µA
Λ	1	Α	1	1	Ion Guide Frequency	1653.0
1	1		1		Q1 Frequency	1088.2
					Multiplier Voltage	1619.1 V
					Detector Gain	3.0 × 10^ 5
					MS Transfer Line	150.0 °C
					Filament Selection	1
					Ion Source Temperature	200.0 °C
					Foreline Pressure	38 mTorr
		1 h	IN	1 M	Ion Gauge Pressure	1.2 × 10^ -5 To
	128 130 132	216 218 220	411 413 415	499 501 503	A CALORINE CONTRACTOR STRUCTURES AND CONTRACTOR	

#### Full Scan Spectrum Not Acquired

Mass	Theory	Abundance R	elative Abundance	Isotope Mass	Theoretical Isotope Mass	Isotope Abundance	Isotope Ratio
68.99	69.00	137,190,032	100.00	70.02	70.00	1,493,796	1.09
130.97	130.99	65,700,696	47.89	131.98	131.99	2,131,296	3.24
218.98	218.99	64,681,264	47.15	220.00	219.99	2,681,922	4.15
413.98	413.98	2,415,356	1.76	415.01	414.98	202,386	8.38
501.99	501.97	2,363,385	1.72	503.04	502.97	215,758	9.13
Air/ V	Vater Check:	H2	0 (%18/69)	6.58	Leak Check:	: 3.19% of reference	
		N	12 (%28/69)	53.59			
		c	02 (%32/69)	17.71			
		co	02 (%44/89)	1.24			
		N2 / H2	O (%28/18)	8.14			

Typical results for an EI Full Tune are listed below.

- Peak Intensities:
  - Base Peak ≥ 10,000,000
  - $m/z \ 502 \ge 300,000$
  - FWHM = 0.4–0.8
- Water Background: *m*/*z* 18:69 < 240%
- Repeller Voltage:
  - Helium carrier gas = 3–8 V
  - Hydrogen carrier gas = 7–15 V
- Multiplier Voltage

- Normal Performance: < 2200 V
- Replace Multiplier:  $\geq 2200 \text{ V}$
- Foreline Pressure: < 100 mTorr
- Ion Gauge Pressure:  $< 5 \times 10^{-5}$  Torr

**Note** Foreline and ion gauge are optional devices. Their pressures are dependent on column flow rate.

- Isotope Ratios:
  - *m*/*z* 70:69 = 0.8–3.0%
  - *m/z* 220:219 = 3.2–6.0%
  - $m/z \ 503:502 = 7.5 15\%$
- Leak Check: < 10%

Run the EI Full Tune if you suspect a system problem. The following conditions could indicate an issue:

- Increased detector gain—Detector gain is related to multiplier voltage, so if the detector gain is increased, multiplier voltage will also increase.
- Leak check change—Leak check results change over time base on instrument conditions. Recently vented systems exposed to air should be lower than 10% after one day of pumping down. Assuming the system is leak free, the instrument leak check should constantly decrease over time until stabilizing.

# **El Tune**

**EI Tune (default)**—A shortened version of the EI Full Tune recommended for use when subsequent maintenance tuning is needed after an EI Full Tune. This tunes resolution, mass, lenses, and adjusts detector sensitivity, but does not tune detector gain. It requires a starting tune file saved to the instrument to give masses within 0.5 amu and widths less than 1 FWHM. It starts with the last saved tune and sets the detector sensitivity to generate an ion at m/z 219 with an intensity of 20,000,000<sup>1</sup> counts.

#### El Check

**EI Check (default)**—Used to check how well your last tune is performing. The EI Check performs a leak check, makes sure the mass calibration is correct (that mass error is within 0.2 amu) and generates a report. If your SOP allows it, you can use this tune to rapidly verify that the previous tune is still generating good spectra.

<sup>&</sup>lt;sup>1</sup> This number may change when using EI SmartTune.

## **El Diagnostics**

**EI Diagnostics (default)**—Runs a complete set of diagnostics, including a leak check, and generates a report. No tuning is performed. Uses the parameters from the last saved tune.

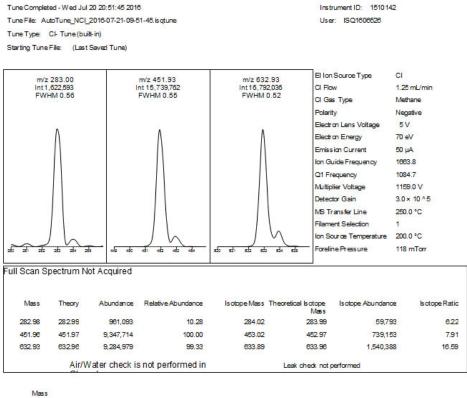
## **Negative CI Tune**

**CI-Tune (default)**—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 1.25 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. You should not use a CI tune unless your instrument has a CI ion volume and methane 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 (*i.e*—0.5 V) should be loaded in manual tune and saved to the instrument before tuning in CI mode.

Figure 28 shows a typical CI- Tune report where methane is the CI reagent gas.



ISQ Tune Results

Instrument Name: ISQ

#### Figure 28. Typical CI- Tune Report with Methane as a CI Reagent Gas

Mass

Typical results for a CI- Tune using methane as the reagent gas are listed below.

- Peak Intensities:
  - Base Peak: 452 or 633
  - Base Peak ≥ 10,000,000
- CI Gas Flow: 1.0-4.0 mL/min Methane
- Emission Current:
  - 50 μA
  - Standard Set Point

**Note** Emission current may be set in the instrument method, and the value entered in the method should match the value set in the tune.

- Multiplier Voltage:
  - Normal Performance: < 2200 V</li>
  - Replace Multiplier: ≥ 2200 V

- Foreline Pressure: < 400 mTorr
- Ion Gauge Pressure:  $< 1 \times 10^{-4}$  Torr

**Note** Foreline pressure fluctuates with CI reagent gas flow rate. As the CI reagent gas flow rate increases, the foreline pressure also increases. Ion gauge pressure also increases if an ion gauge is installed on the system.

• Isotope Ratios: m/z 453:452 = 5.8-11.8%

#### **Positive CI Tune**

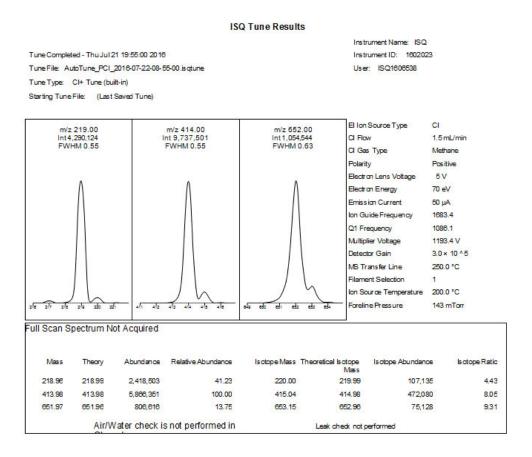
**CI+ Tune**—Used to analyze samples with positive CI. The standard PCI tune performs a mass calibration, then tunes the lenses and sets the resolution. This type of tune assumes you are using methane as the CI reagent gas and tunes the system with a 1.5 mL/min flow. This tune does not set the detector 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.

**Tip** If you intend to use ammonia reagent gas, attach methane to one CI reagent gas port and ammonia to the other port. Tune the instrument using methane, then switch to the ammonia port. Allow plenty of time for the new reagent gas to purge the CI tubing before starting your analysis.

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

Figure 29 shows a typical CI+ Tune report where methane is the CI reagent gas.

#### Figure 29. Typical C+- Tune Report with Methane as a CI Reagent Gas



**Tip** If you intend to use ammonia reagent gas, attach methane to one CI reagent gas port and ammonia to the other port. Tune the instrument using methane, then switch to the ammonia port. Allow plenty of time for the new reagent gas to purge the CI tubing before starting your analysis. Please note that the instrument is locked for approximately two minutes to evacuate the volume between gas valves when switching between gases. During this time the instrument will not respond to new commands. This is normal behavior.

Typical results for a CI+ Tune using methane as the reagent gas are listed below.

- Peak Intensities:
  - Base Peak: 414
  - Base Peak  $\geq$  1,000,000
  - CI Gas Flow: 1.5–4.0 mL/min Methane
  - Emission Current:
    - 25–50 μA
    - Standard Set Points

**Note** Emission current is an input value, and it should match the value set in the tune.

- Multiplier Voltage
  - Normal Performance: < 2200 V at  $3 \times 10^5$  detector gain
  - − Replace Multiplier: ≥ 2500 V at  $2.1 \times 10^6$  detector gain
- Foreline Pressure: < 400 mTorr

**Note** Foreline pressure fluctuates with CI reagent gas flow rate. As the CI reagent gas flow rate increases, the foreline pressure also increases.

- Ion Gauge Pressure: < 1e-4 Torr
- Isotope Ratios:

 $- m/z \, 415:414 = 5.8 - 11.8\%$ 

**IMPORTANT** Sensitivity is affected by the amount of water in the system. A recently cleaned source initially increases sensitivity in CI+, and then sensitivity drops as water is pumped out. Methane is highly reactive with water.

#### **AEI Full Tune**

**AEI Full Tune (default)**—Provides full AEI 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. It runs and sets detector gain to  $3x10^5$ . It requires a starting tune file saved to the instrument that gives masses within 0.5 amu and widths less than 1 FWHM. 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.

#### **AEI Tune**

**AEI Tune (default)**—A shortened version of the AEI Full Tune recommended for use when subsequent maintenance tuning is needed after an AEI Full Tune. This tunes resolution, mass, lenses, and adjusts detector sensitivity, but does not tune detector gain. It requires a starting tune file for the AEI ion source saved to the instrument that gives masses within 0.5 amu and widths less than 1 FWHM. It starts with the last saved tune and sets the detector sensitivity to generate an ion at m/z 219 with an intensity of 200,000,000<sup>2</sup> counts.

<sup>&</sup>lt;sup>2</sup> This number may be modified when using AEI SmartTune.

# **AEI Check**

**AEI Check (default)**—(To be used with the AEI ion source). Used to check how well your last tune is performing. The AEI check performs a leak check, makes sure the mass calibration is correct (that mass error is within 0.2 amu), and generates a report. If your SOP allows it, you can use this tune to rapidly verify that the previous tune is still generating good spectra.

## **AEI Diagnostics**

**AEI Diagnostics (default)**—(To be used with the AEI ion source). Runs a complete set of diagnostics, including a leak check, and generates a report. No tuning is performed. Uses the parameters from the last saved tune.

# **Tuning the ISQ 7000 Mass Spectrometer**

#### ✤ To tune the ISQ 7000 Mass Spectrometer using AutoTune

- 1. Select the tune type you want to use from the list of available tune types. See Figure 30.
  - Figure 30. List of Available Tunes in AutoTune

🔣 ISQ AutoTune	? ×
Source type: O EI O CI I EI/CI O AEI	
Category: Default ~	Tune Types
El Initial Tune (default)         El Full Tune (default)         El Tune (default)         El Chack (default)         El Diagnostics (default)         Cl- Tune (default)         Cl+ Tune (default)	Display report when complete
Now running:	Show spectra
Action:	🖉 Start

2. Select the **Display Report When Complete** checkbox so that you can view the tune report after running the tune.

Figure 31. Displaying a Tune Report

El Initial Tune (default) El Full Tune (default) El Tune (default) El Check (default) El Diagnostics (default)		∧ Display report
CI-Tune (default) CI+Tune (default)	~ ~	when compl
Now running:		Show spect
Action:		
		🧭 Start

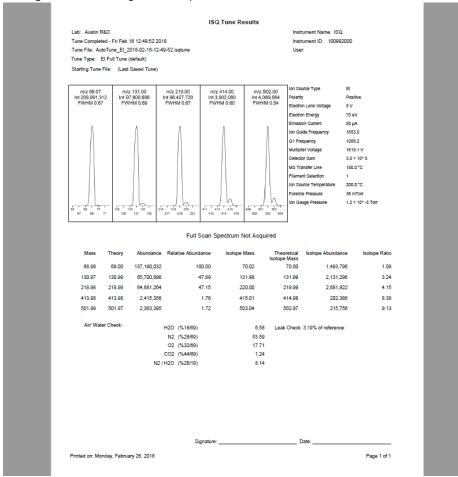
- 3. Select the **Show Spectra** checkbox to show the spectra while the system is tuning.
- 4. Click the **Start** button to begin tuning.

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

🕼 ISQ AutoTune	? ×
Source type: O EI O CI I El/CI O AEI	
Category: Default ~	Tune Types
El Initial Tune (default)         El Full Tune (default)         El Tune (default)         El Check (default)         El Diagnostics (default)         CI- Tune (default)         CI- Tune (default)         CI- Tune (default)	Display report when complete
Now running:	Show spectra
Peak intensity: 1.96E+07	72
00 07.2 08.4 03.6 70.8	12

Figure 32. Showing the Spectra During an Automatic Tune

 Once the tune completes, your tune report will open in the *ISQ Series Tune Results Viewer*. If you did not select the **Display Report When Complete** checkbox, you can click **View Tune Report** on the ISQ 7000 Dashboard and view the report.



**Note** The **Error Action** is not diagnostic. It indicates what happens during the tune if a specific device cannot meet the tuning criteria. Any **Fail** comments on error actions do not mean the tune has failed.

#### Figure 33. Viewing a Tune Report.

6. Compare this tune with a previous tune report. Some changes in peak height are normal, but if the difference is significant, see Troubleshooting. If you have recently serviced the instrument, you most likely have a leak at the column, vent valve or near the component you just serviced.

In the Tune Results window, you can open tune results, print report, or change the way you view the report. To save the report, click the 🖬 icon and save it as a Microsoft Excel file or an Adobe Acrobat PDF file.

7. Click **Open Tune Results** in the top of the window and browse to another tune report on your computer. Then click **Open**.

← → · ↑ _ « OS	(C:) > Users > Public > Public Documents > ThermolSQ > Tu	uneResults	✓ <sup>™</sup> Searce	h TuneResults
Organize 🔻 New folde	r			
Desktop ^	Name	Date modified	Туре	Size
Documents	After hipot	1/4/2018 9:14 AM	File folder	
🖶 Downloads	ELCI	1/4/2018 9:14 AM	File folder	
Music	Old tunes	1/4/2018 9:14 AM	File folder	
Pictures	🌃 (Not Saved)_AutoTune_AEI_2018-01-11-14-15-06.isqtuneres	1/11/2018 2:15 PM	ISQTUNERES File	10 KB
Videos	🌃 (Not Saved)_AutoTune_AEI_2018-01-11-14-20-36.isqtuneres	1/11/2018 2:20 PM	ISQTUNERES File	10 KB
OS (C:)	🌃 AutoTune_AEI_2017-12-13-10-38-10.isqtuneres	12/12/2017 8:38 PM	ISQTUNERES File	47 KB
	🔣 AutoTune_AEI_2017-12-13-11-37-53.isqtuneres	12/12/2017 9:37 PM	ISQTUNERES File	52 KB
🕳 USB Drive (D:)	🔣 AutoTune_AEI_2017-12-13-13-36-00.isqtuneres	12/12/2017 11:36	ISQTUNERES File	56 KB
🕳 USB Drive (D:)	🔣 AutoTune_AEI_2018-01-04-12-57-29.isqtuneres	1/4/2018 12:57 PM	ISQTUNERES File	56 KB
8270D_Spectra	🔣 AutoTune_AEI_2018-01-11-14-34-57.isqtuneres	1/11/2018 2:34 PM	ISQTUNERES File	47 KB
182013	🔣 AutoTune_AEI_2018-01-19-15-47-46.isqtuneres	1/19/2018 3:47 PM	ISQTUNERES File	12 KB
11062013	🔣 AutoTune_El_2018-01-03-08-22-45.isqtuneres	1/3/2018 8:22 AM	ISQTUNERES File	62 KB
	🔣 AutoTune_El_2018-01-03-13-48-59.isqtuneres	1/3/2018 1:48 PM	ISQTUNERES File	61 KB
For_Alex_June26	MatoTune_PCI_2018-01-04-09-08-33.isqtuneres	1/4/2018 9:08 AM	ISQTUNERES File	45 KB
ISQ3.2	🔣 Diagnostics_AEI_2017-12-13-13-44-45.isqtuneres	12/12/2017 11:44	ISQTUNERES File	32 KB
ISO7000	Diagnostics AEI 2018-01-12-11-26-51 isotuneres	1/12/2018 11·26 ΔM	ISOTHINERES File	32 KR

Figure 34. Opening a Tune Results File

8. Click **Print Report** to open a print dialog box and print your report.

× م ?

Select Printer Add Printer Fax Microsoft XPS Document Writer	ter
Status: Ready Location: Comment:	Print to file Preferences
Page Range C All C Selection C Current Page C Pages:	Number of copies: 1

**Figure 35.** Printing a Tune Report

9. Click **Report Options** to select the charts and reports you want to display and change the name of your instrument. Then click **OK**.

Figure 36. Selecting the Tune Report Options

Report Options	>
Lab name:	ОК
Instrument name:	Cancel
Calc relative abundance using: 69	
✓ Include signature line	
Optional reports	
Diagnostics Device settings Tune	type settings
Custom report	
File name:	

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

# **Using SmartTune**

If you configured the ISQ 7000 software to use the **EI SmartTune** or **AEI SmartTune** application, follow the instructions below to use the application.

#### ✤ To use the SmartTune application

1. Right-click **AutoTune Options** on the ISQ 7000 Dashboard and choose **AEI SmartTune** or **EI SmartTune** depending on which ion source is installed on your instrument. See Figure 37.



SQ 7000 Dashboard	_ ×
AutoTune Options	View Tune Report
Advanced AutoTune El SmartTune	AutoSIM
AEI SmartTune Method Editor	Xcalibur
Instrument Control	Shut Down
	•
Status Analyzer Power Main	tenance
<b>Status:</b> Direct Co	ntrol
Actua	al Set Point
Instrument tune file	
AEI source	
Temperatures MS transfer line temp: 250 °C	C 250 °C
√ Ion source temp: 198 °	
Pressures	
✓ Foreline pressure: 53 m To	π
Misc	
Vacuum: O	
Turbo pump speed: 100 ° CI reagent gas flow 0.00 mL/mi	
Cl gas type Non	

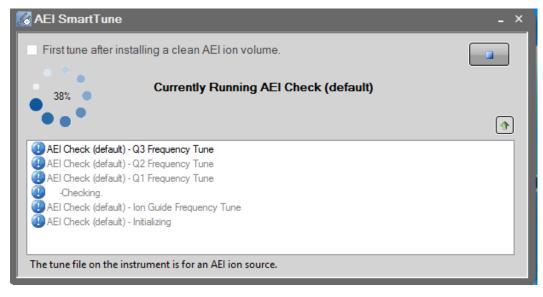
2. The SmartTune application opens. See Figure 38.

AEI SmartTune	_ ×
First tune after installing a clean AEI ion volume.	
	•
The tune file on the instrument is for an AEI ion source.	
The tune file on the instrument is for an Action source.	

Figure 38. Viewing the SmartTune Application

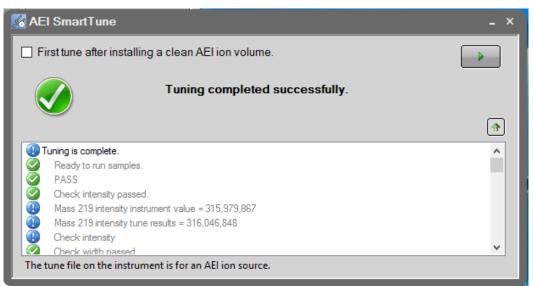
- 3. If this is the first tune after installing a clean ion source, check the box indicating that. Click the green arrow on the right side of the panel to start the SmartTune application.
- 4. The SmartTune application assess the state of your instrument and chooses the proper tunes for the system. The tunes run or running and the percent complete appear in the window while SmartTune is in progress. See Figure 39.

Figure 39. SmartTune in Progress



5. Once SmartTune has completed tuning your instrument, you will see a green check in the SmartTune window with a list of all the tunes it ran and a brief summary of their results. See Figure 40.





- 6. View the tune report to see all the results of the SmartTune.
- 7. See Figure 41 below for the SmartTune workflow.

Figure 41. SmartTune Workflow



\*SmartTune does not run diagnostics when there is a tune failure due to high leak check or electron multiplier voltage.

# **Tuning for DFTPP and BFB**

DFTPP and BFB are calibration compounds used by the EPA in the United States to determine the suitability of your mass spectrometer for specific methods for analyzing semi-volatile and volatile organic compounds. Follow the instructions below for tuning your instrument to pass the criteria for analyzing DFTPP and BFB.

The default supplied AEI or EI Full Tune should pass DFTPP or BFB criteria. If the tune does not pass for the required criteria, follow the procedures in this section.

- **\*** To tune the instrument for analyzing DFTPP or BFB using custom tune types
- 1. Open the ISQ 7000 Dashboard and click **AutoTune Options** to open the AutoTune window. See Figure 42.

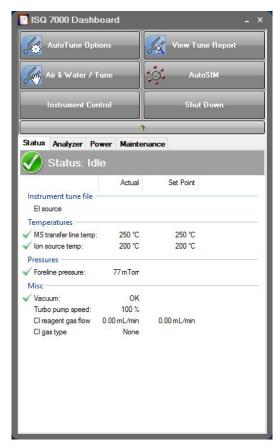


Figure 42. Accessing ISQ 7000 AutoTune from the ISQ 7000 Dashboard

2. Select Advanced AutoTune from the drop-down menu. See Figure 43.

AutoTune Options Advanced AutoTune El SmartTune Instrument Control  Status Analyzer Power Maintenance Status Analyzer Power Maintenance Status Idle Actual Instrument tune file El source Temperatures MS transfer line temp: 250 °C Ion source temp: 200 °C Pressures Foreline pressure: 77 mTorr Misc	Set Point
El SmartTune Instrument Control  Status Analyzer Power Maintenance  Status Analyzer Power Maintenance  Status: Idle  Instrument tune file El source Temperatures MS transfer line temp: 250 °C Ion source temp: 200 °C Pressures Foreline pressure: 77 mTorr	Shut Down
Instrument Control   Status Analyzer Power Maintenance  Status: Idle  Instrument tune file El source Temperatures MS transfer line temp: 250 °C Ion source temp: 200 °C Pressures Foreline pressure: 77 m Torr	Shut Down
Status Analyzer Power Maintenance Status: Idle Status: Idle Actual Instrument tune file El source Temperatures M5 transfer line temp: 250 °C Ion source temp: 200 °C Pressures Foreline pressure: 77 mTorr	e Set Point
Status Analyzer Power Maintenance Status: Idle Actual Instrument tune file El source Temperatures MS transfer line temp: 250 °C Ion source temp: 200 °C Pressures Foreline pressure: 77 mTorr	Set Point
Status: Idle Actual Instrument tune file El source MS transfer line temp: 250 °C Ion source temp: 200 °C Pressures Foreline pressure: 77 m Torr	Set Point
Actual Instrument tune file El source Temperatures MS transfer line temp: 250 °C Ion source temp: 200 °C Pressures Foreline pressure: 77 mTorr	
Actual Instrument tune file El source Temperatures MS transfer line temp: 250 °C Ion source temp: 200 °C Pressures Foreline pressure: 77 mTorr	
Instrument tune file El source Temperatures MS transfer line temp: 250 °C Ion source temp: 200 °C Pressures Foreline pressure: 77 mTorr	
El source Temperatures MS transfer line temp: 250 °C Ion source temp: 200 °C Pressures Foreline pressure: 77 mTorr	
Temperatures MS transfer line temp: 250 °C Ion source temp: 200 °C Pressures Foreline pressure: 77 mTorr	
✓ MS transfer line temp:         250 °C           ✓ Ion source temp:         200 °C           Pressures	
✓ Ion source temp: 200 °C     Pressures     ✓ Foreline pressure: 77 mTorr	
Pressures Foreline pressure: 77 mTorr	250 °C
✓ Foreline pressure: 77 mTorr	200 °C
Contraction of the second s	
Mar	
IVIISC	
Vacuum: OK	
Turbo pump speed: 100 %	
	).00 mL/min
Cl gas type None	

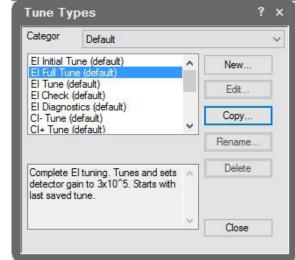
Figure 43. Selecting from AutoTune Options

- 1. Click **Tune Types** to create a new tune type. See Figure 44.
  - Figure 44. Creating a New Tune Type

Category:	Default	~	Tune Types
El Full T El Tune El Checl El Diagn El Diagn	Tune (default) une (default) (default) (default) (default) ostics (default) tor Sensitivity and Check ostics_leakcheck	*	Display report when complete
Now runnin	g:		Show spectra

2. In the **Tune Types** dialog box, select a tune type to edit and click the **Copy** button. See Figure 45.





3. The **Edit Tune Type** window opens. In the **Name** field, name the new tune type **DFTPP Tune** or **BFB Tun**e depending on your requirement. Change the description if you prefer. See Figure 46.

Figure 46. Creating the DFTPP or BFB Custom Tune Type

ame:	DFTPP Tune			Save
escription:	Complete El tur	ning. Tunes and sets detector gain to 3x10^5. Starts with last saved to	une.	Cancel
			¥.	
ategories:	Default		~	Print
put tune file ting tune fi ass calibrat	ename prefix: [A ile: [(]	rgets Detector Diagnostics Report Lenses Resolut uutoTune Last Saved) ~	vanced settings tion	

4. Go to the Targets tab and check Tune Target Ion Ratios. See Figure 47.

Name:	DFTPP Tune	Save		
Description:	Complete El tuning. Tunes and sets detector gain to 3x10^5. Starts with last saved tune.			
Categories:	Default			
Ţ	pe: <ul> <li>Tune and diagnostics</li> <li>Diagnostics only</li> <li>Show advanced settings</li> </ul>			
1	a Source Targets Detector Diagnostics Report Lenses Resolution			
Tune target	ion ratios			
Target mass	Ratio			
50 amu:	1.1 % (25 to 51.5 amu)			
69 amu:	100 % (51.5 to 78.5 amu)			
131 amu:	90 % (78.5 to 132.5 amu)			
219 amu:	70 % (132.5 to 220.5 amu)			
	2 % (220.5 to 415.5 amu)			
414 amu:				

Figure 47. Tuning Target Ion Ratios

5. For DFTPP—Enter the values below for the target ion ratios. See Table 1.

Target Mass (amu)	Ratio (%)
50	1.1
69	100
131	90
219	70
414	2
502	1.3

 Table 1.
 Target Ion Ratios for DFTPP

6. See Figure 48 for an example of the correct DFTTP Tune.

	DFTPP Tune			Save
escription:	Complete El tu	uning, Tunes and sets detector gain to 3x10^5. Starts with last saved tune.	0	Cancel
ategories:	Default		~	Print
Т	vpe: 💿	Tune and diagnostics O Diagnostics only 🗹 Show advanced settings	3	
neral lo	n Source    Ta	argets Detector Diagnostics Report Lenses Resolution		
Tune target	ion ratios			
get mass	Ratio			
) amu:	1.1 %	(25 to 51.5 amu)		
) amu:	100 %	(51.5 to 78.5 amu)		
	90 %	(78.5 to 132.5 amu)		
31 amu:	70 %	(132.5 to 220.5 amu)		
		(220.5 to 415.5 amu)		
19 amu:	2 %	(220.5 to 415.5 and)		
9 amu:	2 % 1.3 %	(415.5 amu and above)		
31 amu: 19 amu: 14 amu:				

Figure 48. DFTPP Tune Targets

7. Save as **DFTPP Tune**.

8. For BFB—Enter the values below for the target ion ratios. See Table 2.

Table 2. Target Ion Ratios for BFB

Target Mass (amu)	Ratio (%)
50	1.1
69	100
131	55
219	60
414	2
502	1.3

9. See Figure 49 for an example of a correct BFB Tune.

ame:	BFB Tune	Save	
escription:	Complete El tuning. Tunes and sets detector gain to 3x10^5. Starts with last saved tune.	Cancel	
ategories:	Default v		
Т	pe:    Tune and diagnostics    Diagnostics only   Show advanced settings		
neral lor	Source Targets Detector Diagnostics Report Lenses Resolution		
Tune target	ion ratios		
get mass	Ratio		
Damu:	1.1 % (25 to 51.5 amu)		
) amu:	100 % (51.5 to 78.5 amu)		
31 amu:	55 % (78.5 to 132.5 amu)		
19 amu:	60 % (132.5 to 220.5 amu)		
14 amu:	2 % (220.5 to 415.5 amu)		
)2 amu:	1.3 % (415.5 amu and above)		

Figure 49. BFB Tune Targets

- 10. Save as **BFB Tune.**
- 11. Tune the instrument with the newly created tune types as described in Tuning the ISQ 7000 Mass Spectrometer.

If the tune fails, you can modify the target tune factors for DFTPP and BFB in the **ISQ Manual Tune** utility.

#### \* To tune the instrument for analyzing DFTPP or BFB using the manual tune utility

1. On the dashboard, click Air & Water/ Tune. See Figure 50.

📔 ISQ 7000 Dashboard	-
AutoTune Options	View Tune Report
Air & Water / Tune	AutoSIM
Method Editor	Xcalibur
Instrument Control	Shut Down
	Ŷ
Status Analyzer Power Main	tenance
Status: Idle	
Actua	al Set Point
Instrument tune file AEI source	
Temperatures	
MS transfer line temp: 250 °	
V Ion source temp: 202 %	C 200 °C
Pressures V Foreline pressure: 54 mTo	~
Misc	
Vacuum: O	ĸ
Turbo pump speed: 100	
CI reagent gas flow 0.00 mL/mi	n 0.00 mL/min
CI gas type Non	e

Figure 50. Locating Air & Water/Tune on the ISQ 7000 Dashboard

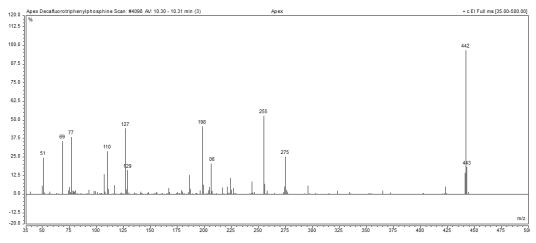
 The ISQ Manual Tune utility opens. The last saved tune opens. If your DFTPP or BFB Tune was not the last saved tune, select Load Tune File, and open the correct tune file. Under the Target Tune Factors tab, adjust the tune factors as needed until the DFTPP or BFB tune criteria passes. See Figure 51.

Figure 51. Adjusting the Target Tune Factors in ISQ Manual Tune

SQ Manual Tune - 🗆 -				
1		File 🛛 🏇 Get from Instrument 🞉 Save to Instrument	🗘 Reset 👻	Firmware -
Mass & Resolution 1	Tune Frequenc	y Tune Target Tune Factors		
Target facto	ors exist			
Target mass	Target Factor			
50 amu:	1.00000	(25 to 51.5 amu)		
69 amu:	1.00000	(51.5 to 78.5 amu)		
131 amu:	1.00000	(78.5 to 132.5 amu)		
219 amu:	1.00000	(132.5 to 220.5 amu)		
414 amu:	1.00000	(220.5 to 415.5 amu)		
502 amu:	0.8	(415.5 amu and above)		
Instrument status:	ldle		Embedded system v	version: 210.72.91.24:

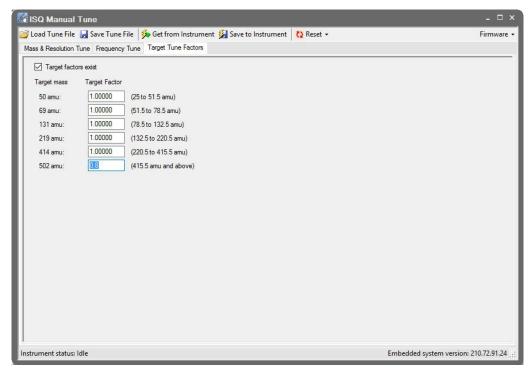
The example in Figure 52 shows a DFTPP run that had a low m/z 198. In this example, the m/z 198 intensity was low, and the tune failed. To increase the m/z 198 intensity, the high mass intensity was lowered in the target tune factors. Specifically, the **502 amu** target mass was lowered from **1** to **0.8**.



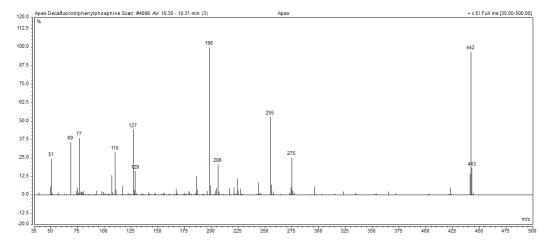


3. To increase the m/z 198 intensity, the high mass intensity was lowered in the target tune factors. Specifically, the 502 amu target mass was lowered from 1 to 0.8. See Figure 53.

Figure 53. Changing the Target Tune Factors in Manual Tune



4. When the DFTPP tune is run after the 502 amu target mass was lowered, the m/z 198 intensity increases, and the tune passes. See Figure 54.



**Figure 54.** DFTPP Tune Results with High m/z 198

5. You can make similar adjustments in case other masses fail the sensitivity criteria of your method.

Click **Save to Instrument** to save the tune settings to the instrument and close the manual tune utility.

6. Run the DFTPP sample again and check your results.

**Note** If the DFTPP or BFB tune still fails after adjusting the target tune ratios in the ISQ Manual Tune utility, clean the ion source.

# **Creating a Method**

Once you have tuned the ISQ 7000 mass spectrometer, you can create a method for each component of your system. Methods are used to indicate to the GC/MS system how to collect your data.

#### Contents

- Accessing the Method Editor
- Creating a GC-MS Method in Xcalibur Software
- Creating a GC-MS Method in Chromeleon Software

## Accessing the Method Editor

To create a method for the ISQ 7000 mass spectrometer, the TRACE 1300 or 1310 GC, and your autosampler, use the Method Editor. The Method Editor is accessed differently on systems running Xcalibur software and those running Chromeleon software. This section provides instructions for accessing the Method Editor on both systems.

**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 Instrument Setup window. If your instruments are not shown, you must configure them. See "Configuring Your Instrument" for instructions.

#### \* To access the method editor using Xcalibur software

1. Click Method Editor on the ISQ 7000 Dashboard. See Figure 55.

4

📔 ISQ 7000 Dashboard	-
AutoTune Options	View Tune Report
Air & Water / Tune	AutoSIM
Method Editor	Xcalibur
Instrument Control	Shut Down
	<b>↑</b>
Status Analyzer Power Main	tenance
Status: Idle	
Actua	al Set Point
Instrument tune file	
AEI source	
Temperatures ✓ MS transfer line temp: 250 °	C 250 ℃
<ul> <li>✓ Ion source temp; 203 °</li> </ul>	
Pressures	
✓ Foreline pressure: 53 mTo	п
Misc	
Vacuum: O	
Turbo pump speed: 100 ° CI reagent gas flow 0.00 mL/mi	
Cligas type Non	

Figure 55. Accessing the Method Editor from the ISQ 7000 Dashboard

2. The Method Editor window opens. See Figure 56. 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.

#### Figure 56. Method Editor Window

Instrument Name

х.

File ISQ Serie	mno Xcalikur Instrument Setup deg	
	Method Setup	
	Method type: Acquisition - General Use general acquisition methods to acquire any data type.	
Al/AS 1310	MS transfer line temp: 250 🔁 °C konization mode: EI 💌	
AI/AS 1310	Ion source temp: 300 🔄 IC Cligas type: Methane 🐨	
	Acquisition threshold:     I000      Cliges flow:     100      mL/min Acply to all proups     C Stop after:     100      min	
	Scans	Groups
*ISQ Series	Time Mass List or Dwell or Scan Tune Rijecci	Time Total Scan (min) Time (sec)
1.5.1		
. 💷		
TRACE 1300 Series GC		
Series GC		
	View Tune Report	
Dura du		
Ready		

Icon Arrow

#### \* To access the Method Editor using Chromeleon software

To access the Method Editor for the ISQ 7000 mass spectrometer, the TRACE 1300 or 1310 GC, and your autosampler, go to **Start > Thermo Chromeleon > Chromeleon 7** and open the Chromeleon software application The **Chromeleon Console** opens to the **Thermo Scientific GCMS Home Page**. See Figure 57.

struments	«	🗘 Launch eWorkflo	w • 🛞 Smart Startu	ip + 🛞 Smart	ihutdown +   🎲 Take Control 🔝 Monitor Baseline 🔸 🎡 Command	C Detach C <autogenerated> •</autogenerated>	
	Fiter 🏹	Thermo Scientific	GCMS Home Sa	mpler Front In	t Oven MSDevice <u>Fi</u> ter Audit Queue		
90-PC		Connect	Injection details Data vault: Sequence: Current injection: Type: General settings Overal Status:	n/a	Peterson inne. injection No :	Renaring time.	
		1384952 3	Wating for:				
		1200000					
		600000				Spectral plot could not be created. No spectra selected.	
		400000					
		-69253	5.0	10.0	15.0 20.0 25.0 30.0		
		Date	Bete	ntion	15.0 20.0 25.0 30.0		
		8/5/2014	1:37:49 PM	me Denice	User TSQ has disconnected Chromeleon Instrument Configuration Manager fr		
		<ul> <li>8/5/2014</li> <li>8/5/2014</li> </ul>	1:24:27 PM 1:24:27 PM		MassSpecDriver - Mass Spectrometer Driver, Version 1.20.0.0. RC version - Copyright © 2009-2014 Thermo Fisher Scientific Inc All rights reserved. Initializing driver Mass Spectrometer #2.	Thermo Fisher Scientific confidential - Not for sale!	
Instruments		8/5/2014	1:24:26 PM		Initializing driver Mass Spectrometer #2		
I I I I I I I I I I I I I I I I I I I							
Data		<ul> <li>8/5/2014</li> </ul>	1:24:26 PM		Initializing driver CmDDKDry_1.		

Figure 57. Chromeleon Console: Thermo Scientific GCMS Home Page

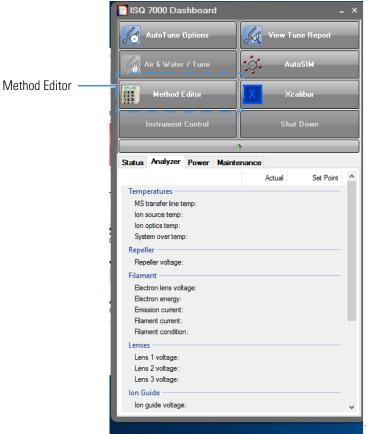
**Note** All of the configured instruments are shown in the left pane of the Chromeleon Console window under **Instruments > ISQ-PC**. If your instruments are not shown, you must configure them. See "Configuring Your Instrument" for instructions.

## **Creating a GC-MS Method in Xcalibur Software**

 To create a method for the ISQ 7000 mass spectrometer, GC, and autosampler using Xcalibur software

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

1. Click **Method Editor** on the ISQ 7000 Dashboard to open the Method Editor. See Figure 58.



#### Figure 58. Accessing the Method Editor from the ISQ 7000 Dashboard

### **Creating an Autosampler Method in Xcalibur Software**

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 59.

Figure 59. Autosampler Method

<b>2 8 8</b> ,	x 8			
		TriPlus RSH Autosam	pler - New	
	Method			
		es, and click Create New Method butto	n	
CE 1300 © GC	C GC Liquids C GC H			
.uc		Create New Method		
	Injectors			
	Injector port A - ID: *	Injector 1	•	
IS RSH	Injector port B - ID:	None	-	
ampler	Injector port C - ID:	None		
- 0	Injector port D - ID:	None	-	
	Fiber conditioning port - ID:	None	-	
Series	Syringes	1		
		None	•	
	Wash stations Wash station - ID:	None	-	
	Wdsh stauon - ID.	Trane	_	
	Derivatization station			
	Derivatization - ID:	None	-	
	Agitator			
	Agitator - ID: *	Agitator 2	-	
	Ready			
in the second				

- 2. Choose one of these three options: GC Liquids, GC Headspace, SPME, SPME Arrow, or ITEX.
- 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 60):

Intitled - Thermo Xcalibur Instrument Setup	
File TriPlus RSH Help	
TriPlus RSH Autosampler - GC Liquids	C Basic
General Washes Sync Advanced	
TRACE 1300 Injector: Injector A (hjector 1) Type: Single	
Injection mode Mode: Basic	
Papid mode	
TiFkur RSH Autosampler Syringe type	
Syringe type Syringe volume (µL): 10.0  Needle length (mm): 57	
Sampling Injection Injection Agents Sandard V	
ISQ Series Sample volume (ul.): 10 v Injection depth: Standard v Injection depth: Standard v Pre-injection duell time (s): 00 v	
Air and filling mode: Auto  Post-injection dwell time (s): 0.0	
	- Sample
Sampling depth in vial Sample vial depth (mm): 30.0  Non viscous  Non viscous	
Bottom sense	
Height from bottom (mm): 0.2 v	
Ready	
(way	

Figure 60. 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 61):

Figure 61. GC Liquids Washes Settings

- 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 Method for the ISQ 7000 Mass Spectrometer

Click the ISQ 7000 icon in the left pane to create a method for the ISQ 7000 mass spectrometer.

1. To create a acquisition method from the Method Type pull-down menu, select **Acquisition-General**. An Acquisition method is used to collect data.

**Note** The maintenance method is used to bake out or cool down the ISQ 7000 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 data acquisition.

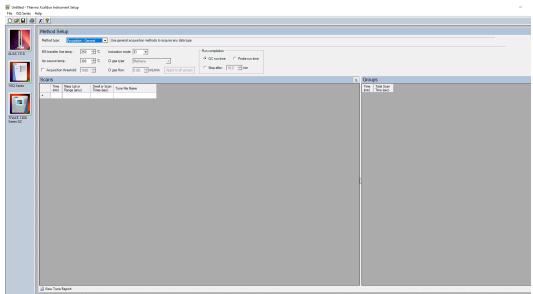
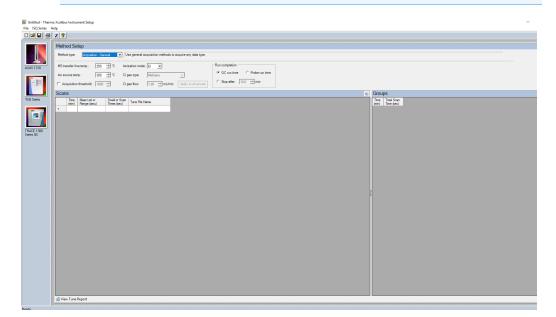


Figure 62. Creating a General Acquisition Method for the ISQ 7000 Mass Spectrometer

2. 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 ISQ 7000 mass spectrometer. The maximum allowable temperature is 400 °C.

However, you will damage the column and contaminate the ISQ 7000 instrument if you set the transfer line temperature above the maximum allowed temperature of the column.

**Note** The maximum transferline temperature of or the rate at which higher transfer line set temperatures may be reached can depend upon the ion source temperature and the GC oven temperature.



3. 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. For most compounds, a source temperature of 275 °C (default) is adequate.

**Note** For best results, tune the instrument at the same temperature you will run the analyses in your method.

- 4. Select the **Acquisition Threshold** checkbox 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.
- 5. Select **EI** from the **Ionization Mode** pull-down menu.

**Note** Only use CI if you have installed a CI ion volume in the ISQ 7000 instrument and you have 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. (There will be a two-minute delay when you change ports for the CI Gas Type.) 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.

6. In the **Run Completion** group, select the action you want to occur at the end of a run:

- a. **GC Run Time**—Select this option if you want the ISQ 7000 system run to end when the GC run is complete. This is the most common setting.
- b. **Probe Run Time**—Select this option if you have a probe controller installed and you want the ISQ system run to end when the probe run is complete.
- c. **Select Stop After**—Select this option to set the number of minutes you want the ISQ 7000 system to run. The end of the run can be between 0 and 1,000 minutes. This option allows you to 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. We recommend you select this option it because saves burn time on the filament.

**Note** In Timed Acquisition mode, the run stops when the ISQ 7000 system has completed acquisition for the final sample in your method. These settings do not apply.

7. In the **Scans** pane, click a scan row to enter scan information.

🛎 🖬 🔿 🗶 😵 250 - 10 GC run time ÷ 10 CI gas type C Stop after: 10.0 😤 min 1000 -----CL cas fir Data Type Time Total S min) Time ( Centroid 100, 150, 200 0.2, 0.2, 0.2 1.00, 1.00, 1.00 II ast Saver Centroid 35,150 01.02

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

a. The **Time (min)** column is used to set the time that the ISQ 7000 system begins to acquire data after the GC starts. It is typical to have enough of a time delay to allow the solvent to get through the column before starting an acquisition.

Figure 63. Entering the Scan Information

As an example, in this method, the mass range of 50-550 amu will be scanned in 0.2 seconds. Beginning from the same start time, three different SIM masses at 100, 150, and 200 will be looked at for 0.2 seconds each. 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 0.816 seconds.

At 4 minutes into the GC run, the scanning is completely changed. Now the full scan range is 35-150 amu, which is scanned every 0.1 seconds, and the two SIM masses are 450 and 614 amu, which are scanned for 0.1 and 0.2 seconds respectively. All these scans will repeat every 0.312 seconds until the GC run is complete.

All of these scans use the last tune file that was saved to the instrument.

b. The **Mass List or Range** column tells the ISQ 7000 system what masses it needs to scan. In full-scan mode, enter the start and end mass separated by a dash. In SIM mode, enter individual masses or multiple values in this field (as long as they are separated by a comma). You must put each full-scan range on a separate line.

**Note** Each line in a scan must only contain a Full-Scan range or individual SIM masses. They cannot be mixed in a single line.

- c. In SIM mode, the **Dwell or Scan Times** column defines the amount of time (in seconds) that the ISQ 7000 instrument will look at your SIM ion mass. If you are in Full-Scan mode, the **Dwell or Scan Times** column determines the amount of time for each individual scan. You should set this value to have 5-20 scans across your GC peak. If you have too few scans, the GC peak area is too imprecise. If you have too many scans, the ISQ 7000 instrument's signal becomes less precise. The default is 0.2 s.
- d. The **Tune File Name** column selects a tune file to be used for this scan. It should be the automatic tune file you created in Tuning the ISQ 7000 Mass Spectrometer. You can also use a specific tune file for each of the scans.
- e. The **Scan Name** column contains a description of the scan. The name may be used as a label to indicate the compound used with the scan.
- f. The **SIM Widths** column sets 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 instrument will collect all the ions from your SIM mass +/- 0.5 amu. Narrower SIM widths lead to greater specificity.
- g. The **Ion Polarity** column is only used in CI mode, which is the only mode for generating positive and negative ions. In EI mode, this column should be always be set to **Positive**.
- h. The **Data Type** column determines whether you want to collect **Profile**, **Centroided**, or **Nominal** mass spectra. Centroided mass spectra is most common because it is used by most of the libraries and provides the smallest data files. Profiled mass spectra provides detailed mass spectral peaks, which results in a large data file that contains details a centroided spectra does not contain. When you want to

perform fast scanning (up to 20,000 amu/s), select **Nominal** from the drop-down list under **Data Type**.

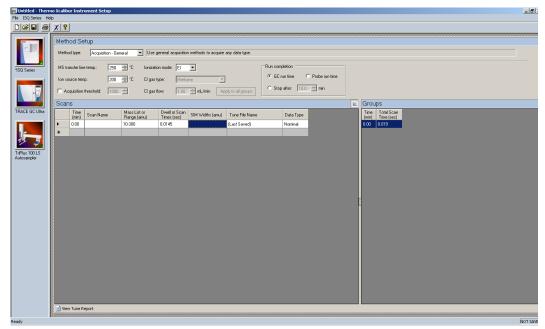
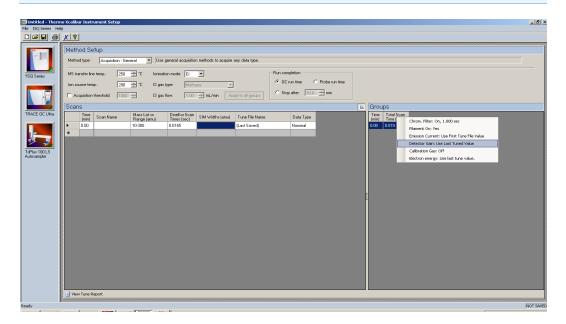


Figure 64. Selecting Nominal Mass Spectra

8. In the **Groups** pane, review the information in each row. As you create scans in the **Scans** pane, information in the groups pane is automatically displayed.

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



- a. The **Time** column displays the time that the ISQ 7000 mass spectrometer begins to acquire this particular group of scans after the GC starts.
- b. The **Total Scan Time** column indicates the sum of all the scans in each segment. The total scan time also contains the stabilization time that occurs between each scan. In this method, beginning 2.5 minutes into the GC run, you will get a complete set of scans every 0.816 seconds. At 4 minutes into the GC run, the scanning has completely changed.Now the scans will repeat every 0.412 seconds until the GC run is complete.
- c. The **Chrom Filter On** column enables the chromatographic filter. This filter smooths spectral data as it is acquired, which may increase the signal-to-noise ratio by a factor of 2 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.
- d. 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.
- e. The **Filament On** column turns 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 will be collected. Use this column if you have analytes eluting before the solvent peak. You can create a segment to turn off the filament during the solvent peak to preserve the filament.
- f. 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, deselect the Use Tune File Emission Current checkbox 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.
- g. The Use Last Tuned Detector Gain column indicates that you want to use the detector gain set in ISQ 7000 Autotune or set and scanned in manual tune. If you do not need to use the gain set in ISQ 7000 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.
- h. Use the **CI Gas Flow** column to set the flow rate of your reagent gas. Remember to set the CI gas flow in the Groups column as well as 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.
- i. Use the **CI Gas Type** column to set the type of gas attached to one or both CI gas ports (A or B). Be sure that the gas you have assigned to a port is actually attached to that port, as each CI gas has a specific calibration of flow vs. gas viscosity.
- j. The **Cal Gas** column turns on the calibration gas during a run. This setting can be useful when confirming that the system is generating ions and correctly storing the data to disk. Typically, the setting is off, but you may let a low level of calibration gas

into the source by selecting EI. If you have a dual-flow calibration gas module, you may select CI for a high level of gas.

9. (For **Acquisition-Timed** methods only) In the **Instrument Settings** pane, do the following:

	X ?			
	Method Setup			
	Method type: Acquis	ition - Timed 💌 Use ti	ned acquisition methods to acquire timed SIM dat	à
ies	MS transfer line temp.:	250 <sup>★</sup> <sup>*</sup> C Ionizatio	n mode: El 💌	
	Ion source temp.:	200 🕂 °C 🛛 gas t	pe: Methane	
19	C Acquisition threshold:	1000 🚔 Ci gas fi	ow: 1.00 🚎 mL/min	
	<ul> <li>Scans</li> </ul>			
âC Ultra	21-21-# × 🛍	🛍 🗟 🛞 M 🔺 🕨	Vink to external file	Desired scars per peak:
	Name	RT Windo		Acquisition options
	► OFN	5.00	7.20 54	Allow for asymmetric acquisition windows
4	OFN	5.00	1.80 109	
INLS	OFN	5.00	1.20 121	Allow dwell time prioritization High priority multiplier: 10 🚍
pler	OFN OFN	5.00	1.40 119 0.60 79	Fullscen
	OFN	5.00	0.40 79	Use full scan with mass range: 50-550
	OFN	5.00	0.70 105	Full scan time: 0.003 🚟 sec Positive 💌
	OFN	5.00	1.50 105	Start time: 200 min. End time: 10.00 min.
	OFN	6.00	1.00 79	
1000	OFN	6.00	0.60 84	Instrument settings
	OFN	6.00	1.20 129	Tune file
	OFN	6.00	1.20 129	Tune file (+): [Last Saved]
	OFN	6.00	0.70 167	Tune File (+): Autotune_NCI
	OFN	6.00	1.90 105	Detector gain
	OFN	6.00	6.90 84	I Use last tuned detector gain × 10.0 ÷
	*			C Use specified detector gain: 3.0E+005
				Emission current
0.000				Use tune file emission current
				C Use specified emission current:
	Total compounds: 15			
	View Tune Report			

Figure 65. Acquisition-Timed Instrument Settings

- 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 the ISQ 7000 Mass Spectrometer," 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 ISQ 7000 Mass Spectrometer," If you are using positive CI on use the most recent positive CI automatic tune file you created in Chapter 3, "Tuning the ISQ 7000 Mass Spectrometer," 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 ISQ 7000 Autotune. If you do not need to use the gain set in ISQ 7000 Autotune, then you can set the gain manually. Higher gains give larger signals, but may shorten the lifetime of the detector or saturate the electrometer with too much signal 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  $\pm 0.5 \mu$ A.

10. As appropriate, use the options in the Scan Settings area to further adjust your method.

Figure 66. Acquisition-Timed Scan Settings

File ISQ Series Help	o Xcalibur Instrument Setup	X
15Q Series	Method Setup         Use timed acquisition methods to acquire timed SIM d           Method type:         Acquisition interfect to acquire timed SIM d           MS transfer line time):         200 국가 C         Drainstoin mode         EI マ           Ion source time):         200 국가 C         Drainstoin mode         EI マ           Ion source time):         200 국가 C         Drainstoin mode         Methods           C Acquisition threshold:         1000 국가 C         Drain time         Y           S Scans         Scans         100 국가 C         Drain time	MA
TRACE GC Ultra	24-21-04 × 🖎 🛥 💀 🕐 🕅 4 🕨 🕅 Unk to external file	Scan settings
Triftur 101:S Autocompler	Name         PT         Viriados (min)         Mass           OPA         50         7.20         54           OPA         5.00         1.00         100           OPA         5.00         1.20         121           OPA         5.00         0.60         7.20           OPA         5.00         0.60         7.3           OPA         5.00         0.60         7.8           OPA         5.00         0.40         7.9           OPA         5.00         0.40         7.9           OPA         5.00         1.50         105           OPA         5.00         1.50         1.50           OPA         5.00         1.20         125           OPA         5.00         1.20         126           OPA         5.00         1.20         125           OPA         6.00         1.20         124           OPA         6.00         1.20         129           OPA         6.00         1.20         129           OPA         6.00         1.20         129           OPA         6.00         1.20         129           OPA <t< th=""><th>Time summay         Reading total can time         0.300         SM Time         0.300         Lowert dwaltime         0.00256         "Window optimizion         "Update         Desired window         0.0121         Minimum window         0.0225         Desired window         0.021         Minimum window         0.0225         Desired window         0.022         Desired window         0.022         Res         Desired window         0.022         Minimum window         0.022         Desired come pee peak         0.022</th></t<>	Time summay         Reading total can time         0.300         SM Time         0.300         Lowert dwaltime         0.00256         "Window optimizion         "Update         Desired window         0.0121         Minimum window         0.0225         Desired window         0.021         Minimum window         0.0225         Desired window         0.022         Desired window         0.022         Res         Desired window         0.022         Minimum window         0.022         Desired come pee peak         0.022
	OPN         6.00         6.30         84           *         *         *         *         *	Acquisition options Adown for asymmetric acquisition windows Adown for asymmetric acquisition windows Adown dwell these prioritication High prosty multiplen: Use foll acon with meas range: Use foll acon with meas rang

- a. The **Time Summary** section reports the resulting total scan time, the SIM time, and lowest dwell time for you method. These values are for information only and not editable.
  - i. The **Resulting Total Scan Time** is the baseline peak width divided by the number of points desired across the peak. These values should be updated if your method requirements are different from the defaults.
  - ii. The **SIM** time is the total length of all SIM scans for each compound in your list. This will match the total scan time unless the method also has a full scan event.
  - iii. The **Lowest Dwell Time** is the actual lowest dwell time achieved by the method settings. When the **Optimize** check box is selected, if the actual lowest dwell time is considerably lower than the requested dwell time, then the minimum window has been reached, and if the actual lowest dwell time is considerably higher than the requested dwell time, then the requested window has been reached.
- b. The **Window Optimization** pane allows access to the window optimizer settings. When the optimize button is checked, acquisition windows will be set automatically based on the acquisition window and dwell time targets set in this pane. For complex SIM methods, this option will help ensure a method is created that can achieve the requested scans per peak.

Algorithm Details: If the **Optimize** checkbox is checked, the SIM acquisition windows in the method are set to the **Desired Window** unless the **Desired Min Dwell Time** cannot be met with the number of **Requested Scans Per Peak**. If this occurs, then the acquisition windows are reduced until either the **Desired Min Dwell Time** is met, or the **Minimum Window** is reached. If the **Minimum Window** is reached first, the **Minimum Dwell Time** is reduced until the **Requested Scans Per Peak** is achievable. If the absolute minimum dwell time on the instrument, which is 0.5 ms, is reached before the requested points across the peak are achieved, the **Minimum Window** is lowered until the **Desired Scans Per Peak** criteria are met or until the absolute allowed minimum window on the instrument is reached, which is 0.24 min. In this very rare case you must reduce the number of **Desired Scans Per Peak**, increase the **Min Baseline Peak Width**, or reduce the number of transitions contained in your method before you will be allowed to save your method.

Settings: **Optimize** is not checked by default. When Optimize is checked, you can adjust the settings in the pane to optimize your method. When the box is not checked, you must manually input your acquisition windows. The default values for the window optimizer should give reasonable results for normal methods. The default values are:

Desired Min Dwell Time-10 ms

Desired Window-0.6 min

Minimum Window-0.3 min

- Change the minimum dwell time using the Desired Min Dwell Time combo box. If your method has many transitions, you may want to reduce the desired minimum dwell time. Note that the wider the acquisition windows in your method, the shorter the average dwell time will be.
- Change the desired acquisition window in the **Desired Window** combo box. The desired window is the amount of time to scan for a transition around a given retention time to ensure that compound will be observed. The desired window can be set from 0.24–5 min. Set the window wide enough so that a retention time shift will not cause you to miss any compounds. Include extra time in this window if there is any uncertainty in compound retention times in the method. Note that the longer the dwell time for your compounds, the narrower your acquisition windows will be.
- Change the minimum acquisition window in the Minimum Window combo box. The minimum window can be set from 0.24–5 min. This is the smallest amount of time that should be scanned for a transition around a given retention time so that you are confident the compound will be observed. Set the minimum window to the lowest safe value to prevent compound retention times from drifting outside the acquisition window.

**Note** If the dwell time limit is reached and the minimum acceptable window is forced below the 0.24 min limit, the method will fail, and a smaller list must be used.

- c. Under **Peak Width**, you can change the minimum baseline peak width and desired scans per peak. These values are used to calculate the total scan time, which includes the SIM time and the full scan time. The minimum baseline peak width should be set roughly to the shortest chromatographic peak time in your analysis.
- d. Under **Full Scan**, you indicate if a Full Scan is to be run along with SIM. 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 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.
- e. Under Acquisition Options, 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.

**Note** This option is only available when the method optimizer is not active.

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. 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 ISQ 7000 Method Editor, clear the **Link to External File** check box.

g. After clicking **Link to External File**, the **SIMBridge** dialog box opens. Choose the language of your method file from the **Source Locale** drop-down menu.

	io Xcalibur Instrument Setup		
File ISQ Series Help			
	XY		
	Method Setup		
	Method type: Acquisition - Timed	✓ Use timed acquisition methods to acquire timed SIM data.	
			al
1SQ Series	MS transfer line temp.: 250 🚔	🗃 🔔 🔄 Source locale: English (United States) 💌 Delmiter: ,	
	Ion source temp.: 200 😤	Name of Ite:	
	C Acquisition threshold	,,,,,,, _	
	Scans	Import map	
TRACE GC Ultra	§	Column in file Map to Example	
	2↓-21-AX B G G C Name RT		A 1
	* Name HI		ec
			ec
TriPlus 100 LS Autosampler			ec
		Results (8)	
			ity multiplier: 10
			50-550
		Cancel Ok	
	View Tune Report		
Ready			NOT SAVE

Figure 67. Setting the Source Language of Method Files using SIMBridge

h. Browse to your file.

Figure 68.	Linking to an	<b>External File</b>	using	SIM Bridge
------------	---------------	----------------------	-------	------------

Image: Second	File ISQ Series	rmo Xcalibur Instrument Setup Help		_ <u>_8</u> ×
152 Series     Method type:     Acquinton - Timed     Ube timed acquintion methods to acquire timed SIM data.       152 Series     No transfer line temp.     200       100 source temp.     200     Image: Source tocket:       100 source temp.     200     Image: Timed temp.				
TRACE GOUND       Income       Incom       Income       Income <th>TRACE GC Ulto</th> <th>Method Setup Method type: Acquisition Timed MS transfer fine temp: 200 2 C Acquiston Thresholt 100 2 C Acquiston Thresholt 100 2 C Scans 1 - 17 - 24 X C A C A C None RT</th> <th>It Starties       Starties       Starties       It succession         It succession       Starties       Starties       Starties         It succession       Starties       Starties       Starties</th> <th>(2)         (2)           (2)         (2)           (2)         (2)           (2)         (2)           (2)         (2)</th>	TRACE GC Ulto	Method Setup Method type: Acquisition Timed MS transfer fine temp: 200 2 C Acquiston Thresholt 100 2 C Acquiston Thresholt 100 2 C Scans 1 - 17 - 24 X C A C A C None RT	It Starties       Starties       Starties       It succession         It succession       Starties       Starties       Starties         It succession       Starties       Starties       Starties	(2)         (2)           (2)         (2)           (2)         (2)           (2)         (2)           (2)         (2)

- i. Click **Open** to open the method in SIMBridge.
- j. If necessary, change the method headings in your original file to match those in the method editor. A green check mark appears when your method is validated.

	no Xcalibur Instrument Setup								_ 5	7
File ISQ Series He										
	X ?									
								6666666		
	Method Setup									
	Method type: Acquisition - Timed	<ul> <li>Use timed acquisition</li> </ul>	methods to acquire tin	ned SIM data.						
		ISQ Series - SIMBridge						×		
1SQ Series	MS transfer line temp.: 250 🚊	🧉 🛕 🛐   Source locale	English (United State	s) 🔻 Del	imiter: 📔	]				
	Ion source temp.: 200 😤	Name of file: C:\Users\Alvi	n\Desktop\Agilent_SIM	_Method_Groups_mod2	.C\$V			=1		
	C Acquisition threshold	Import map								
	Scans	Column in file   Map to	Example							
TRACE GC Ultra	2↓- ±↑- AA X (BL NG (B (G (	Name Name	Example 1							-
	Name BT	Start Time Start time	3.2							
	*	Stop Time End time	10.4						pc .	
		Mass Mass	54						es .	
~ 🐔										
TriPlus 100 LS									ec	
Autosampler										
									(	
									(	
		Results - 15 compounds foun	d.					8		
		Name BT Window (mi	n) Ion Polarity Mass	Pre-width (min) Post-	width (min) Start time	Endtime	Vial Position Dwell Time Priority			
		31 6.8 7.2	Positive 54	0 0	3.2	10.4	Normal			
		2 11.3 1.8	Positive 109	0 0	10.4	12.2	Normal	_	(	
		3 12.8 1.2	Positive 121	0 0	12.2	13.4	Normal	- 11	(	
	2020	24 14.1 1.4 5 15.1 0.6	Positive 119 Positive 79	0 0	13.4	14.8	Normal			
	0000	6 15.6 0.4	Positive 79	0 0	15.4	15.8	Normal	- 11		
		7 16.15 0.7	Positive 105	0 0	15.8	16.5	Normal		(	
		38 17.25 1.5	Positive 105	0 0	16.5	18	Normal		ty multiplier: 10	
	200	9 18.5 1	Positive 79	0 0	18	19	Normal		,	
		210 19.3 0.6	Positive 84	0 0	19	19.6	Normal		to the	
		211 20.2 1.2	Positive 129	0 0	19.6	20.8	Normal	-	50-550	-
	📄 Yiew Tune Report						Cancel Ok			
										1070
Ready									NOT SA	AVE

Figure 69. Changing Method Headings in SIMBridge

k. Click **Open** and the external method will be opened in the method editor.

Figure 70.	Viewing a Linked File in the Method Editor
------------	--

Ne to total       Neto         Image: Series       Method Setup		io Xcalibur Instrument Set	up			X
Image: Second Sector       Method Sector         Image: Second Sector       Image: Second Sector         Image: Second Sector						
Image: Several second constructions of the sequence of the deta.         Image: Several second constructions of the sequence of the deta.         Image: Several second constructions of the sequence of the deta.         Image: Several second constructions of the sequence of the deta.         Image: Several second constructions of the sequence of the deta.         Image: Several second constructions of the sequence of the deta.         Image: Several second constructions of the deta sequence of the deta.         Image: Several second constructions of the deta sequence of the deta.         Image: Several second constructions of the deta sequence of the deta.         Image: Several second constructions of the deta sequence of		X ?				
Image: Several second constructions of the sequence of the deta.         Image: Several second constructions of the sequence of the deta.         Image: Several second constructions of the sequence of the deta.         Image: Several second constructions of the sequence of the deta.         Image: Several second constructions of the sequence of the deta.         Image: Several second constructions of the sequence of the deta.         Image: Several second constructions of the deta sequence of the deta.         Image: Several second constructions of the deta sequence of the deta.         Image: Several second constructions of the deta sequence of the deta.         Image: Several second constructions of the deta sequence of		Mothod Sotup				
View         View <th< th=""><th></th><th></th><th>ion • Timed 🔽 🖳</th><th>se timed acquisition methods to acquir</th><th>e timed SIM data.</th><th></th></th<>			ion • Timed 🔽 🖳	se timed acquisition methods to acquir	e timed SIM data.	
Image: Construction Meethod:         Cog as low:         Comment           Image: Construction Meethod:         Image: Constrult Meethod:         Image: Constrult Meet	1SQ Series	MS transfer line temp.:	250 🚔 °C Ioni	ization mode: EI		
Image: Scans         Scan settings         C           Image: Scans         Image: Scanses         Image:		Ion source temp.:	200 🕂 °C Cig	as type: Methane	<b>•</b>	
TACE GE UN         Image: Construction		C Acquisition threshold:	1000 🚍 Ci g	gas flow: 1.00 == mL/min		
Image: Section 168 mig/s       Image: Section		<ul> <li>Scans</li> </ul>				
None         B1         Window Indi         Mass           1         6.80         7.20         64           2         11.30         180         19           3         12.00         1.01         121           Advance         5         1510         0.00         78           6         1530         0.40         78         66           5         1510         0.00         78         66           6         1530         0.40         78         66           7         1515         0.70         105         9         100         100         16           8         1725         150         100         78         100         10         100         10           10         1830         0.00         14         12         12         12.40         120         12         12.40         120         12.9         12         12.40         120         12.9         12.9         12.9         12.0         12.0         12.9         12.0         12.0         12.9         12.0         12.0         12.0         12.0         12.0         12.0         12.0         12.0         12.0         12.0 <th>TRACE GC Ultra</th> <th>21-21-33 × 🗈</th> <th>🔄 🗟 🛞 🕅 4</th> <th>Link to external file</th> <th>× 1</th> <th>Scan settings 🔹 🔺</th>	TRACE GC Ultra	21-21-33 × 🗈	🔄 🗟 🛞 🕅 4	Link to external file	× 1	Scan settings 🔹 🔺
2         11.0         180 169           3         12.0         1.0         12           4         14.0         14.0         19           5         1510         0.00 78           7         1515         0.00 78           9         1920         10.0 78           9         1920         10.0 78           10         1930         0.00 78           11         20.20         1.20 123           12         24.04         13.0           13         22.25         0.00 177           14         24.06         10.2           15         20.05         6.09 04           11         20.25         0.00 177           14         24.05         19.0 165           15         20.05         6.90 04           *						
3         128         10         121           Autorardet         4         140         140         19           5         1510         0.60         73         0<		► 1	6.80	7.20 54		Resulting total scan time: 0.300 sec
IPFL INUSS       4       110       110       110       120       120       120       120       120       120       120       120       120       120       120       120       100	13 1	2	11.30	1.80 109		SIM Time: 0.300 sec
Autorarylet <ul> <li>a</li> <li>(10)</li> <li>(11)</li> <li>(10)</li> <li>(12)</li> <li>(14)</li> <li>(12)</li> <li>(14)</li> <li>(12)</li> <li>(14)</li> <li>(15)</li> <li>(15)</li> <li>(16)</li> <li>(16)</li> <li>(16)</li> <li>(16)</li> <li>(16)</li> <li>(16)</li> <li>(16)</li> <li>(16)</li> <li>(16)</li> <li>(17)</li> <li>(14)</li> <li>(16)</li> <li>(16)</li></ul>		3	12.80	1.20 121		
6       15.00       0.00       75         7       10.15       0.07       105         8       17.25       150       105         9       1120       100       13       0.02         12       21.40       120       120         14       22.55       1.91       105         15       20.05       6.50       64         *       Adjust on optimal       10-2         Adjust on optimal       10-2       10-2         *       Adjust on optimal       10-2         *       Vec have fregoet       10-2	Autosampler	§				Lowest dwell time; 0.29593 sec
7       1615       0.00 105         8       17.25       150 105         9       150 100 78         10       1330       0.80 84         11       20.20 1.20 129         12       21.40 1.20 129         13       22.25       0.70 157         14       20.65 1.90 105         15       20.05 6.90 94         *       -Acquiring rotation with constant periods         -Adjoint or acquirition with constant periods       10-3         -Adjoint or acquirition with resource       9.500         *       -Total compounds: 15         Mew hum Report		§				
0       1725       150       165         9       1163       100       73         10       153       0.00       84         11       20.20       1.20       120         12       24.40       120       120         13       22.25       0.70       167         14       23.05       6.50       0.4         -       -       -       -         -       -       -       -         -       -       -       -         -       -       -       -         -       -       -       -         -       -       -       -         -       -       -       -         -       -       -       -         -       -       -       -         -       -       -       -         -       -       -       -       -         -       -       -       -       -         -       -       -       -       -         -       -       -       -       -         -       -       -       -		8 — — · · · · · · · · · · · · · · · · ·				C Optimize
9         1150         100         79           10         1330         000.04         000		8				Desired min dwell time: 10.0 ms
9       100/75         10       1530       0.00 /15         11       20.20       1.20 /12         13       22.25       0.70 /157         14       21.05       6.50 /04         *       Adjuint option       Adjuint option         Adjuint option       Feld son         *       Feld son       Feld son         *       Use hill son with instrunge       5050						Desired window
11       20.20       1.20       129         12       21.40       1.20       120         13       22.25       0.70       167         14       22.65       1.90       105         15       23.05       6.50       94         Allow duef time production with most range       103       103         Allow duef time production       High prody multiple       103         Total computed;       15       104       103         Wew Tune Report       104       105       105		8 — — · · · · · · · · · · · · · · · · ·			r.	
12       21.40       1.20       129         13       22.25       0.70       167         14       22.05       10.10       10.10         15       20.05       6.50       64         Acquisition option       Adjust in option       Acquisition option         Allow dweld twe graduation option       Adjust in option       10.3         Full compounds: 15       15       20.50       10.3         Mew Tune Report       Use full scien with messarings       50.500       10.3		8 — B				Minimum window 0.3 🚍 min
13       22.35       0.07) 157         14       22.65       1.50 105         15       28.05       6.50 84         Allow deel fine production with most corp.       10		8				Dutuit
14         22.65         1.50         10 <sup>-</sup> 15         22.05         6.50         64           Acquisition options         Acquisition vindows         Acquisition vindows           Allow dwel time proteitization         High proteity multipler:         10 <sup>-</sup> Total compounds: 15		27				
15         20.05         6.50         64         Detected consumproperty         10-2           *         Acquisition spring room         Total compounds: 15         Total compounds: 15         Mow Turn Report         Total compounds: 15         Tota		8 8				Min. Daseline peak width: 3.0 🛨 sec
Advantise options     Advantise     Advantise options     Advantise     Advantise options     Advantise     Advantise		e 8				Desired scans per peak: 10-
Allow devel two prostization     High priority multiplier     Image: Total compounds: 15       Total compounds: 15     Image: Total accon with mess range     Image: Total accon with mess range		15	28.05	6.90 84		Acquisition options
Allow devel two prostization     High priority multiplier     Image: Total compounds: 15       Total compounds: 15     Image: Total accon with mess range     Image: Total accon with mess range		*				Allow for asymmetric acquisition windows
Full scon       Full scon       Cluschill scon with mass range       Store       Wew Tune Report						
Total compounds: 15						
Total compounds: 15  We Tune Report						
🖻 Mew Tune Report						Use ful scen with mass range:
		Total compounds: 15				
l production de la constant de		📄 View Tune Report				
	Ready					NOT SAVED

- 1. 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. This function is useful if you need to edit an analyte in a complex method.
- m. In the **RT** column, enter retention times for SIM methods. The retention time is the time it takes an analyte to pass from the column inlet to the detector.

- n. 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.
- o. In the Mass column, enter the mass of the ion you wish to monitor.
- p. 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**.
- q. You may set the number of adjacent analytes to show in your method by using the Number of Adjacent Analytes to Show selection box found in the Options dialog box accessed by the ISQ 7000 main menu. See Figure 71. View the number of analytes you set in the Show Analysis view.

Adjustments in this column are for display purposes only and will not affect your acquisition.

**Figure 71.** Options Dialog Box

III Untitled - Thermo	o Xcalibur Instrument Setup	
File ISQ Series He	elp	
	Method Setup	
10 m	Method type: Acquisition - Timed 💽 Use timed acquisition methods to acquire timed SIM data.	
1SQ Series	MS transfer line temp: 250 🚔 'C Ionization mode: 🗉 💌	
	lon source temp: 200 📩 °C Cl gas type: Methane 🗸	
	T Acquisition threshold: 1000 🛨 Cl gas flow: 100 🛨 mUmin	
TRACE GC Ultra	⊴ Scans	
INALE GE Ultra	Instrument settings         Scan settings           Tore file         Emission current         Peak width           Tore file         () (Last Stored)         () Use ture file emission current	
	Tune File (1) Autoure_NC	
*TriPlus Autosampler	Cubector gain	
Autosampier	C Use specified detector gain: 0	
	min. End time: 10.00 ∃ min.	
	□ Link to external file	Show analysis
	1-1-1-1-1-1-1-1-1-1-1-1-1-1-1-1-1-1-1-	
	Name         RT         Polarity         Window (min)         Mass           #	
	📄 View Tune Report	
Peach		NOT SAVE

r. Click **Show Analysis** (see Figure 72) to validate your method. A chart appears with all your analytes by name and in order of their start times.

File ISQ Series Hel	no Xcalibur Instrument Sel b	cup				
	Method Setup Method type: Acquisi		e timed acquisition methods to acquire	e limed SIM data		
1SQ Series	Ion source temp.:	200 💼 °C Ci ge	as type: Methane as flow: 1.00 🛫 mL/min	-		
Taha tous Adorapie	21-21-33 × 23 Name 2 3 4 5 6 7 9 10 11 12 13 14 15 * Total compands: 15		N         Unit to external file           7.20         54           7.20         54           1.80         163           1.40         113           0.60         73           0.60         74           0.70         105           1.50         175           1.60         73           0.70         105           1.50         73           0.60         84           1.20         123           1.20         123           1.20         123           1.20         124           1.20         125           1.20         124           1.20         123           1.20         124           1.20         125           1.20         124           1.20         125           1.20         125           1.20         165           6.50         64		Show ana	Scan settings     C       Time semany     000       SM Time:     0.300       SM Time:     0.300       SM Time:     0.2553       Lowest dwell time:     0.2553       Desired window     00000       Desired window     000000       Minimum window     000000000000000000000000000000000000
Bastu	View Tune Report					

Figure 72. Accessing the Show Analysis Feature

s. Use the scrolling window at the bottom of the screen to view all your analytes' expected retention times. Resize the window to view more analytes by dragging one side of the window out. As the scrolling window is decreased in size, fewer analytes are shown in the analytes chart. Increasing the size of the scrolling window allows you to view more analytes in your method. If the number of analytes retention times being viewed exceeds 50, an evenly spaced sample of the analytes shows through the window.

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 SIM windows are too congested to achieve the total scan time at the minimum dwell time, the segments are highlighted in red. This warning shows that there is not sufficient time in the segment to scan all events. In this case, the method fails to validate or save and a caution icon appears near the scans title. To correct this, reduce the number of overlapping compounds or change window times. When the peak bars are highlighted orange, this is a caution that there will be fewer scans across the peak than desired.

Also, if your list contains duplicate compounds, the middle of one of the duplicate peak bars will show an orange crossed pattern, instead of the usual white. Delete one of the duplicate compounds to avoid problems with data analysis.



	o Xcalibur Instrument Setup	_ @ ×						
File ISQ Series Help								
	<u>X</u> 3							
	Method Setup							
	Method type: Acquisition - Timed 💽 Use timed acquisition methods to acquire timed SIM data.							
*1SQ Series	MS transfer line temp: 250 🚠 "C lonization moder E 💌							
	lon source temp: 200 🚔 1C Cl gas type: Methane 💌							
	□ Acquisition threshold 1000 = Cliger flow: 100 = mL/min							
	Scans	20000						
TRACE GC Ultra	21-21-33 X 🙁 🖙 🛞 🗘 🙀 🔶 🙀 uluk to external fle 🛛 😵 🗵	≈ ∸						
	Name RT Window(min) Mass							
	9 18.50 1.00 79 Resulting total scan time: 0.300 sec							
1 J	10 19.30 0.60 84 SIM Time: 0.300 sec	00000						
TriPlus 100 LS	11 2020 1.20 129							
Autosampler		00000						
	13 22.35 0.70 167 Window spinitation -							
		2002						
	▶ 15 23.05 6.50 84	-						
	Total compounds: 15	1000						
	<b>R Q Q 3</b> *							
		1 100						
	13-12							
	12.10 17.10 22.10 27.10							
	_ Yew Tune Report							
Ready		NOT SAVED						

11. To import an ISQ 7000, TSQ 8000<sup>™</sup>, TSQ 8000 Evo, TSQ Duo, or TSQ Quantum<sup>™</sup> 7000 MS method, choose **ISQ 7000** | **Import MS Method**. See Figure 74.

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

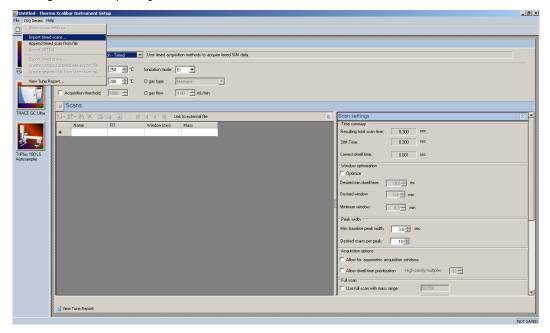
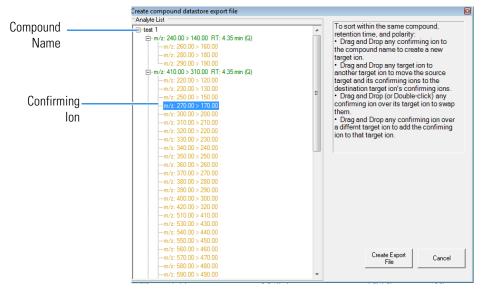


Figure 74. Importing Methods

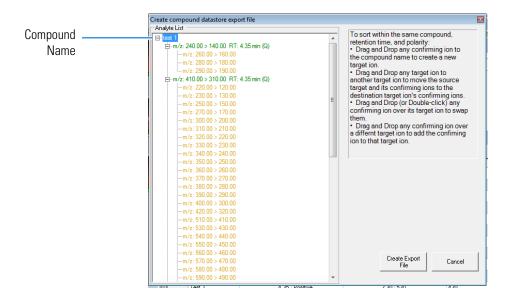
12. Choose **ISQ 7000 | Import Timed Scans** to import .csv or .xml files of previous methods. The software will only load files in valid formats. If your file is not valid you will receive an error message and will not be able to import the file into the Method Editor.

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

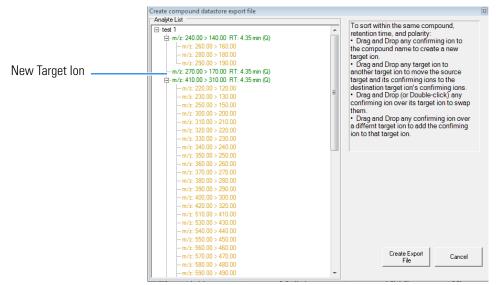
- 13. Choose **ISQ 7000** | **Append Timed Scan from File** to add scans from previous methods to the end of your open scan list. As above, you may import .csv or .xml files. The software will only load files in valid formats. If your file is not valid you will receive an error message and will not be able to import the file into the Method Editor.
- 14. Choose **ISQ 7000** | **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.
- 15. Choose **ISQ 7000** | **Create Compound Data Store Export File** to prepare your file for the compound data store in the TraceFinder application.
  - a. To create a new target ion from the list of confirming ions with identical retention times for a compound, select the confirming ion of interest.



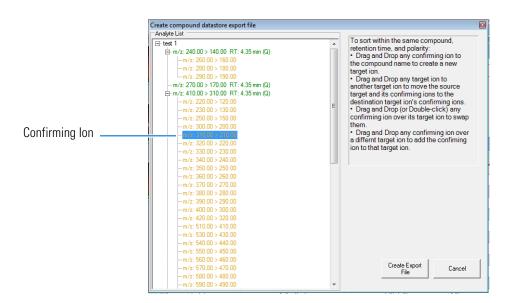
b. Drag the confirming ion to the compound name at the top of the list.



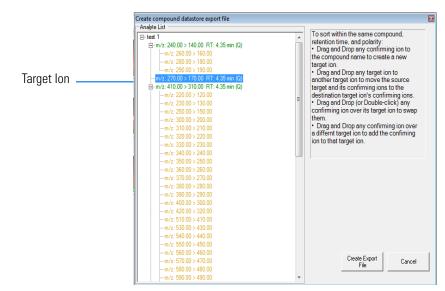
#### c. The confirming ion now appears in the list as a target ion.



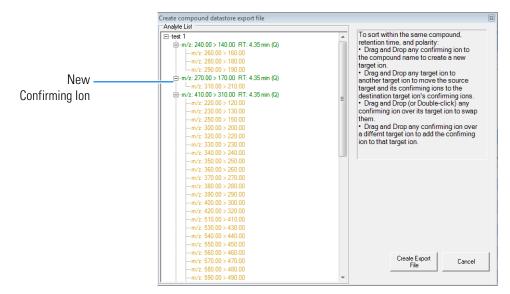
d. To add a new confirming ion to a target ion in the list, select the confirming ion of interest.



e. Drag the confirming ion under the target ion of interest.



f. The confirming ion appears in the list under the selected quantitation ion.



- g. Click **Create Export File** when you are through creating your ion list to export the list to the TraceFinder software compound database.
- 16. Choose ISQ 7000 | Create Segment List From Timed Scan List to import a general acquisition method.
- 17. Choose **ISQ 7000** | **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 75) and add identifying information to the tune report.

Figure	75.	Configu	iring the	Tune I	Report	Options

Report Options X
Lab name: OK
Instrument name: Cancel
Calc relative abundance using: 69
✓ Include signature line
Optional reports
Diagnostics Device settings Tune type settings
Custom report
Use custom report definition
File name:

# Creating a Method for the GC in Xcalibur Software

Click the GC icon in the left pane to create a GC method.

## Creating a method for the TRACE 1300 or TRACE 1310 GC using Xcalibur Software

#### ♦ To create a method for the TRACE 1300 or TRACE 1310 GC

1. Click the **Oven** tab to set the oven temperatures. 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 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 get 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 above the boiling point of your sample solvent and the initial time is usually long enough for the solvent to move through the column.

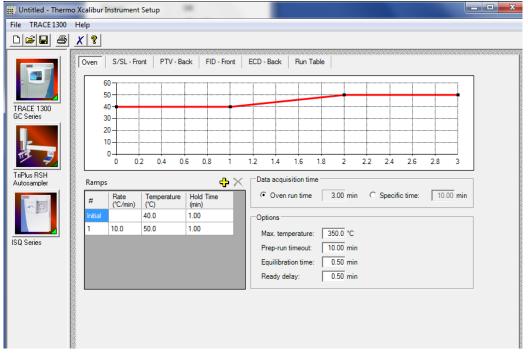


Figure 76. Setting the GC Oven Parameters on the TRACE 1300 or TRACE 1310 GC

- a. You can select a maximum of 32 temperature ramps, each with their own ramp rates, final temperatures and hold times. A typical program will have 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 will have to be optimized for your analysis needs.
- b. 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. This will prevent you from accidentally using a temperature that will damage your column. 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. The equilibration time is a delay between when the GC is at temperature and when the GC reports as being ready. This delay is typically set to 0.5 minutes.



**CAUTION INSTRUMENT DAMAGE.** Be sure not to overheat the GC column or it may contaminate the ISQ 7000 mass spectrometer.

- 2. The TRACE 1300 and 1310 GC also have the option to enable the use of cryogenics to cool the oven. If this option is selected, then the minimum allowed temperature in a temperature ramp will fall from 0 °C to -99 °C. 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 ISQ 7000 mass spectrometer, 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.
- 3. 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. The inlet should be turned on and set to a temperature that is at least 10 °C higher than the boiling point of your least volatile analyte. 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 will have to optimize the injector temperature for your analysis. The SSL temperature can be set up to 400 °C (a typical value would be 225 °C).

🗰 Untitled - Thermo Xcalibur Instr	rument Setup			x
File TRACE 1300 Help				
D 🗃 🖬 🎒 🗶 💡				
Oven S.	/SL - Front PTV - Back FID	- Front ECD - Back Run Table		
: S/SL mode:	Split 💌	Carrier mode: Constant Pressure	Backflush	
		Carrier pressure	Backflush enable:	
TRACE 1300 GC Series Temperat	ture: 200 °C	Pressure: 100.00 kPa	Backflush start time: 1.50 mir	n 🕅
Split flow		riessure.	Backflush duration:	
Split ratio			GC run time	
Splitless t			Specific duration: O 1.50 mir	n 🕅
	ime. <u>11.00</u> min		,	— <u>8</u>
TriPlus RSH Autosampler Surge —				
Surge pre	ssure: 5.00 kPa			
Surge dur	ration: 0.00 min			
	,	Carrier options		
ISO Series	urge	Vacuum compensation:		
Purge flov	w: 5.0 mL/min	Carrier gas saver:		
Constant	septum purge: 🔽	Gas saver flow: 20.0 mL/min		
Stop purg	e time: 0.00 min	Gas saver time: 2.00 min		

Figure 77. Locating the Settings for the SSL Injector on the TRACE 1300 or TRACE 1310 GC

a. Useful for diluting 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 will reduce 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 will be used, so for your analysis, lower split flows may be more acceptable. If you set the split ratio, the software will calculate the correct split flow. The reverse is true also.

**Tip** We recommend turning on 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 ISQ 7000 instrument. If you perform a splitless injection, even if the split flow is set, the split flow will be turned off for the splitless time. The septum purge will be turned off for the stop purge time. After these times, the split flow and septum purge will be reactivated.

b. You can 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 because the hotter column is more resistant to carrier gas flow. 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 ISQ 7000 instrument, which is under vacuum, the vacuum compensation *must* be on to ensure accurate flow rates.

- c. The flow can also be operated in 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 is not commonly used, but may be necessary if you have a particularly challenging separation.
- d. In an effort to reduce the amount of carrier gas used, check **Carrier Gas Saver**. When used, the split flow will be 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 ISQ 7000 instrument, which can affect the system performance. It is also possible for air to diffuse back into the column with low split flows when the column head pressure is low.
- e. 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, you can use the surge pressure to increase the column flow for the surge duration time.

File       TRACE 1300       Help         Image: Image	🔢 Untitled - Thermo Xcalibur Instrument Setup	
Tracker 1300       Oven       5/SL - Front       PTV - Back       FID - Front       ECD - Back       Run Table         Tracker 1300       Tacker 1300       FID       Fine       Carrier mode:       Constant Pressure       Image: Constant Pres	File TRACE 1300 Help	
Ven       S/SL-Front       PTV-Back       FID-Front       ECD-Back       Run Table         Image: Series       Inlet       Image: Split flow: Split fl		
Ready	Ver       S/SL - Front       PTV - Back       FID - Front       ECD - Back       Run Table         TrACE 1300       Temperature:       Split       Carrier mode:       Constant Pressure       Image: Constant Pressure         Turbus RSH       Inlet       Temperature:       Image: Constant Pressure       Carrier pressure       Image: Constant Pressu	Injection phases Press. Rate Temp. KPa "C/sec "C Injection 70:00 Transfer 210:00 14.5 200 Evaporation phase: Transfer temp. ( Cleaning phase: Post-cycle Ramped pressure: t

**Figure 78.** Locating the Carrier Gas Settings for the TRACE 1300 or TRACE 1310 GC

- 4. If you have a Programmable Temperature Vaporizer (PTV), click the **PTV-Front** or **PTV-Back** tab to configure it. The PTV is a low thermal mass injector that allows the instrument to rapidly heat or cool the inlet. You can use the PTV tab to program the temperature of the injector. See the GC documentation for details about the PTV or other types of injectors.
- 5. Click the **Run Table** tab to configure how to control external valves and devices. Consult the GC documentation for more detailed information.

In onuce - menne	Acanouri	instrument sett	νP						
File TRACE 1300	Help								
0 🗳 🖬 🎒	X ?								
		15/5/5/5/5/5/5/5/5/	1555555555555	55,55,55,55	15151515151515151				a a a a a a a a a a a a a a a a a a a
	Oven	S/SL - Front	PTV - Back	FID - Front	ECD - Back	Run Table			
	Time	Item	Ac	tion		Add			
TRACE 1300 GC Series						Modify			
						Remove			
<u>j</u>									
TriPlus RSH									
Autosampler									
ISQ Series									
			0,01,01,01,01,01,01,01,01,01	15075150751507505050	0.004504504504	0,			k
Ready	neaneaneane pàistaige.	1994 AWAR WARKEN AWAR AWAR AWAR AWAR AWAR AWAR AWAR AWA	1.442.942.442.442.442.422.524.524.524.5	wanwa.wanwa.wi.1541,541,541,541,54	1942)449949949949999999999999999	aneane an an an an 200 Mail 200 Mail 200	without without without and the line of the line of the line	na na mana na mana na mana ka ka ka ka ka ka ka	

Figure 79. Locating the Run Table Settings on the TRACE 1300 or TRACE 1310 GC .

**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.

- 6. To add an inlet or detector to the method editor user interface:
  - a. Attach the inlet or detector to the GC. See the GC documentation for instructions.
  - b. Add the inlet or detector to the current instrument configuration. Go to Start | All Programs | Thermo Foundation 3.0 | Instrument Configuration.
  - c. In the **Configured Devices** panel, click the TRACE 1300 or TRACE 1310 GC icon. Select **Configure**.

📷 Thermo Foundation Instrument Configuration	23
Device Types :       Au       TRACE 1300 GC Configuration	
🖉 General 🔽 Inlets 🔍 Detectors 🔶 Auxiliary 🖉 Configured Devices:	
Detectors and Data Channels         Detector type       Makeup gas         Front:       NPD         NPD       10         Back:       FID         None       10         Options       FCD         NPD       10         Line frequ       Tz    ISQ Series	
Get OK Cancel Help	
TRACE 1300 GC       Series       Add >>	
Done	

**Figure 80.** Configuring the Inlets or Detectors on the TRACE 1300 or TRACE 1310 GC

- d. Select the **Inlet** or **Detector** tab as appropriate.
- e. Click Get. The software automatically detects the attached modules. Select the gases as appropriate to 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.

7. When you are finished creating methods for each component in your GC/MS system, select **File | Save As...** from the main menu or click the 🔲 icon.

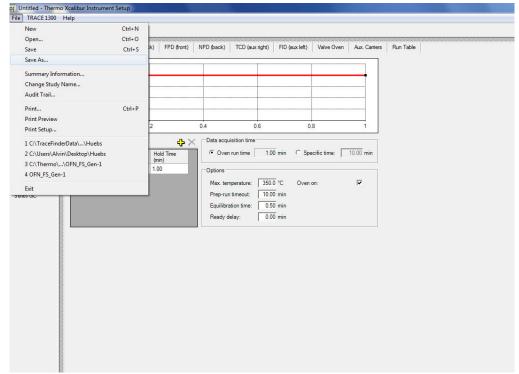


Figure 81. Saving a TRACE 1300 or TRACE 1310 GC Method

### Creating a Method for the TRACE GC Ultra using Xcalibur Software

#### To create a method for the TRACE GC Ultra

**Note** This process only covers the left SSL, but the process is the same for the right SSL. For information on how to use the other injector types, consult the TRACE GC Ultra documentation.

1. Click the **Oven** tab to set the oven temperatures. 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 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 get 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 above the boiling point of your sample solvent and the initial time is usually long enough for the solvent to move through the column.

🗱 Untitled - Thermo Xcalibur Instrument Setup
Ele IRACE Help
🛛 🖉 🖉 🖓 🖉 🖓 🖉 🖉 🖉 🖉
*AI/AS 3000 25-
<b></b> 0.00 0.20 0.40 0.60 0.80 1.00 1.20 1.40 1.60 1.80 2.00 2.20 2.40 2.60 2.80 3.00
<sup>™</sup> ISQ Ramps Post Run Conditions Oven
I Enable Cryo
Initial:         40         1.00         Time (min):         0           Max Temp (*C):         350
Ramp 1: 10.0 50 1.00 Pressure Left (kr-a):
Pressure Right (kPa): 0 Prep Run Timeout (min): 10.00
TRACE GC Ultra
© Oven Run-Time: 3.00 Equilibration
C Specific Time: 10.00 Time (min): 0.50
Ready

Figure 82. Setting the TRACE GC Ultra Oven Parameters

- a. You can select a maximum of seven temperature ramps, each with their own ramp rates, final temperatures and hold times. A typical program will have one or two ramps. However, the GC temperature profile is the primary method for separating your analytes from each other, the solvent, and the matrix. Your temperature profile will have to be optimized for your analysis needs.
- b. You can also select the maximum allowed oven temperature, which should be set to the maximum temperature allowed by your column. This will prevent you from accidentally using a temperature that will damage your column. 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. The equilibration time is a delay between when the GC is at temperature and when the GC reports as being ready. This delay is typically set to 0.1 minutes.
- 2. The TRACE GC Ultra also has the option to enable the use of cryogenics to cool the oven. If this option is selected, then the minimum allowed temperature in a temperature ramp will fall from 0 °C to -99 °C. 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 ISQ 7000 instrument, 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.



**CAUTION INSTRUMENT DAMAGE.** Be sure not to overheat the GC column or it may contaminate the ISQ 7000 instrument.

3. If you have a split / splitless inlet (SSL) injector, click the **Left SSL** or **Right SSL** tab to configure the injector port settings. The inlet should be turned on and set to a temperature that is at least 10 °C higher than the boiling point of your least volatile analyte. 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 will have to optimize the injector temperature for your analysis. The SSL temperature can be set between 50 and 400 °C (a typical value would be 225 °C).

Figure 83. Setting the TRACE GC Ultra SSL Injector Parameters

🗰 Untitled - The	rmo Xcalibur Instrument Setup
File TRACE Help	
0 🖻 🖬 🎒	
*AI/AS 3000	Oven       Left SSL       Left Carrier       Right PTV       Right Carrier       Run Table         Image: Second control of the second contrelation contrelation control of the second control of the second c
TRACE GC Ultra	
	Surge Pressure (kPa): 3
	Surge Duration (min): 0.00
	Mode: Split

a. Useful for diluting 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 will reduce 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 will be used, so for your analysis, lower split flows may be more acceptable.

**Tip** We recommend turning on the septum purge, which means an additional 5 mL/min of carrier gas will go through the injector. This reduces the buildup of contaminants in the injector, on the column, and in the ISQ 7000 instrument. If you perform a splitless injection, even if the split flow is set, the split flow will be turned off for the splitless time. The septum purge will be turned off for the stop purge time. After these times, the split flow and septum purge will be reactivated.

b. 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, you can use the surge pressure to increase the column flow for the surge duration time.

🧱 Untitled - Thermo Xcalibur Instrument Setup
Eile IRACE Help
Very         Left SSL         Left Carrier         Right PTV         Right Carrier         Run Table           ValvAS 3000         Image: Constant Pressure (kPa)         Image: Constant Pressure (kPa)

Figure 84. Setting the Carrier Gas Parameters on the TRACE GC Ultra

- 4. If you have a Programmable Temperature Vaporizer (PTV), click the **Left PTV** or **Right PTV** tab to configure it. The PTV is a low thermal mass injector that allows the instrument to rapidly heat or cool the inlet. You can use the PTV tab to program the temperature of the injector. See the GC documentation for details about the PTV or other types of injectors.
- 5. Click the **Left Carrier** or **Right Carrier** tab. The gas flow and the oven temperature work together to determine how well the analytes are separated and how long the analysis will take. You can select constant pressure or constant flow. If you use constant pressure, as the column is heated in the oven, the flow rate will fall because the hotter column is more resistant to carrier gas flow. 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 ISQ 7000 instrument, which is under vacuum, the vacuum compensation must be on.

- The flow can also be operated in programmed flow or programmed pressure modes. a. In these modes, you may have up to three flow rates or pressures to use during an analytical run. This is not commonly used, but may be necessary if you have a particularly challenging separation.
- b. In an effort to reduce the amount of carrier gas used, you can activate the Gas Saver mode. When used, the split flow will be 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 ISQ 7000 instrument, which can affect the system performance.
- 6. Click the **Run Table** tab to configure how to control external valves and devices. Consult the appropriate documentation for more detailed information.

Figure 85. Locating the Run Table Settings on the TRACE GC Ultra 🐺 Untitled - Thermo Xcalibur Instrument Setup

	X 😵	V   Right Carrier   Run Table		
*AI/AS 3000		Time Item	Setting	Add Edit Remove
TRACE GC Ultra		Initial values External Event #1 On External Event #2 On	External Event #5 On External Event #6 On	
		External Event #3 On External Event #4 On	External Event #7 On     External Event #8 On	

### Creating a Method for the FOCUS GC using Xcalibur Software

#### To create a method for the FOCUS GC \*

1. Click the **Oven** tab to set the oven temperatures. 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 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 get damaged. You can set the initial hold time to a value between 0 and 600 minutes. The typical initial temperature is at least 10 °C above the boiling point of your sample solvent and the initial time is usually long enough for the solvent to move through the column.



🗰 Untitled - The	ermo Xcalibur Instrument Setup
<u>File Focus H</u> elp	
	X 8
AI/AS 1310 Focus GC	• Oven       SSL Inlet       © Carrier       • MS Transfer Line       • Run Table         50
Ready	

- a. You can select a maximum of seven temperature ramps, each with their own ramp rates, final temperatures and hold times. A typical program will have one or two ramps. However, the GC temperature profile is the primary method for separating your analytes from each other, the solvent, and the matrix. Your temperature profile will have to be optimized for your analysis needs.
- b. You can also select the maximum allowed oven temperature, which should be set to the maximum temperature allowed by your column. This will prevent you from accidentally using a temperature that will damage your column. 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 a 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. The equilibration time is a delay

between when the GC is at temperature and when the GC reports as being ready. This delay is typically set to 0.1 minutes.

8

**CAUTION INSTRUMENT DAMAGE.** Be sure not to overheat the GC column or it may contaminate the ISQ 7000 instrument.

2. If you have a split / splitless inlet (SSL) injector, click the **SSL** tab to configure the injector port settings.

🧱 Untitled - Thermo Xcalibur Instrument Setup 👘		
Eile Fogus Help		
A/AS1310 Focus GC Focus GC ISQ	er MS Transfer Line 1 Run Table	
Mode: Split _	r	
	illi illi illi illi illi illi illi ill	
Daadu		9.9.9.9.9.9.9.9.9

Figure 87. Setting the SSL Parameters on the FOCUS GC

- a. The inlet should be turned on and set to a temperature that is at least 10 °C higher than the boiling point of your least volatile analyte. The material injected into the port will vaporize and move into the GC column quickly. Higher temperatures can lead to thermal decomposition of some analytes, so you will have to optimize the injector temperature for your analysis. Set the SSL temperature between 50 and 375 °C (a typical value would be 225 °C).
- b. Useful for diluting 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 will also reduce 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 is typically turned on and set to a flow of 50 mL/min. However, this increases the carrier gas usage, so for your analysis, lower split flows may be more acceptable.

**Tip** We recommend turning on the septum purge, which means an additional 5 mL/min of carrier gas will go through the injector. This reduces the buildup of contaminants in the injector, on the column, and in the ISQ 7000 instrument. If you perform a splitless injection, even if the split flow is set, the split flow will be turned off for the splitless time. The septum purge will be turned off for the stop purge time. After these times, the split flow and septum purge will be reactivated.

- c. 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, you can use the surge pressure to increase the column flow for the surge duration time.
- 3. Click the **Carrier** tab, which is used to set the carrier gas flows. The gas flow and the oven temperature work together to determine how well the analytes are separated and how long the analysis will take. You can select either constant pressure or constant flow. If you use constant pressure, as the column is heated in the oven, the flow rate will fall because a hotter column is more resistant to carrier gas flow. 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 ISQ 7000 instrument, which is under vacuum, the vacuum compensation must be on.

ile Fogus Help	-	Kcalibur Instrument Setup	
Al/AS 1310   Focus GC   ISQ     SSL Inlet   Corrier   MS Transfer Line   How   Mode:   Constant Pressure   Index   Corrier   Flow   Mode:   Constant Pressure   Index   In	jile Fo <u>c</u> us <u>H</u> elp		
Al/AS 1310   Focus GC   ISQ     SSL Inlet   Corrier   MS Transfer Line   How   Mode:   Constant Pressure   Index   Corrier   Flow   Mode:   Constant Pressure   Index   In	D 🚅 🖬 🎒 🗶	?	
	AI/AS 1310	Flow   Mode:   CP   CP <td></td>	

### Figure 88. Setting the Carrier Gas Parameters for the FOCUS GC

In an effort to reduce the amount of carrier gas used, you can activate the Gas Saver mode. When used, the split flow will be 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 ISQ 7000 instrument, which can affect system performance.

4. Click the **MS Transfer Line** tab to set the temperature of the transfer line for a different instrument. This tab is not used on the ISQ 7000 mass spectrometer.

- gale eer ootting the handler		
🗰 Untitled - Thermo Xcalibur Instrument Setup		
<u>File Focus H</u> elp		
Al/AS 1310 Focus GC ISQ Ready	MS Transfer Line Run Table MS Transfer Line MS Transfer Line Temperature: 30 °C	

Figure 89. Setting the Transfer Line Parameters on the FOCUS GC

5. Click the **Run Table** tab to configure how to control external valves and other external devices. Consult the appropriate documentation for more detailed information.

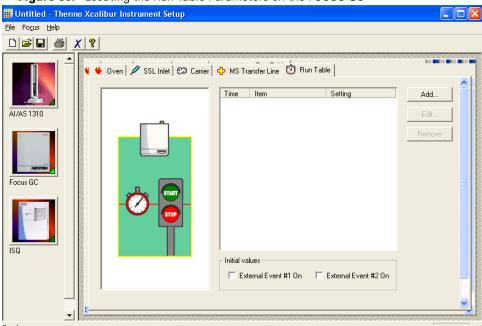


Figure 90. Locating the Run Table Parameters on the FOCUS GC

6. When you are finished creating methods for each component in your GC/MS system, select **File** | **Save As...** from the main menu or click the **■** icon.

🧱 Untitled - Thermo X	calibur I	Instrument Setup		
File TRACE Help				
Open Save	Ctrl+N Ctrl+O Ctrl+S	SL   Left Carrier Right	PTV   Right Carrier   Run Table	
Save As				Injection Phases
Summary Information Change Study Name Audit Trail			Temperature (°C): 35	Pressure Rate Temp. Time (kPa) (*C/sec) (*C) (min) (r
Print Print Preview Print Setup	Ctrl+P	rh2	Split Flow (ml/min):     10       Split Ratio:     10	Injection:         70         0.05           Evap.:         140         14.5         200         1.00           Transfer:         210         14.5         200         1.00
Recent File			Splitless Time (min):         0.00           Solv. Valve Temp. (*C):         100	Cleaning:         14.5         200         1.00
			Purge	Show Graph
TRACE GC Ultra			Stop Purge Time (min): 0.00	Sub-ambient         Backflush Enable           Evaporation Phase         Cleaning Phase
			Surge Surge Pressure (kPa): 3	Ramped Pressure
М	fode:	PTV Split 🔄	Surge Duration (min): 0.00	
Save the active document wit	h a new na	me		

Figure 91. Saving Your ISQ 7000 Instrument Method

# **Creating a GC-MS Method in Chromeleon Software**

If your system is running Chromeleon software, follow the instructions in this section to create a method for the autosampler, GC, and ISQ 7000 mass spectrometer.

#### \* To create a GC-MS method in Chromeleon Software

1. Open the Chromeleon Console. Select **ISQ** from the left menu. The MS Device window opens. See Figure 92.

nstruments	«	Launch eWorkflow 👻 🏐 Sm	nart Startup/Shutdown 👻 🧟	Release Contro	I 🔝 Monitor Baseline 🔹	Consumable	s 👻 🔚 <aut< th=""><th>togenerated</th></aut<>	togenerated
i 💽 🚖	Filter 🌱	Themo Scientific GCMS Hor	me Thermo TriPlus RSH	Frontiniet	Oven MSDevice Et	er Audt	Queue	
ESKTOP-B2T0T5M								
ISQ 😭								
		ISQ 7000	ReadyToDownload	A	nalyzer Power Maintenan			
		Connect			Analyzer	Actual	Setpoint	Ready
					MS Transfer Line temp:	249 [°C]	250 [°C]	0
			Ready		Ion Source temp:	299 ["C]	300 [°C]	õ
					Ion Optics temp:	270 [°C]	271 [°C]	0
					System over temp:	True		
		Status			Repeller voltage:	0.2 [V]	0.0 [V]	0
		MS Transfer Line temp:	Actual Setpoir 249 ['C] 250 ['		Electron lens voltage:	5.4 [V]	5.0 [V]	0
		In Source temp:	249 [*C] 250 [* 299 [*C] 300 [*		Electron lens voltage: Electron energy:	5.4 [V] 69.6 [eV]	5.0 [V] 70.0 [eV]	8
				-1	Emission current:	0.0 [µA]	0.0 [µA]	8
		Foreline pressure:	92 [mTorr]		Filament current:	0.00 [A]		-
			3 [10^-5 Torr]		Filament continuity Ok:	True		
		Turbo Pump speed:	100 %		Lens 1 voltage:	0.2 [V]	0.0 [V]	•
		Collision Gas on:	False		Lens 2 voltage:	0.2 [V] 0.6 [V]	0.0 [V]	
		CI Reagent Gas flow:	0.0 0	0	Lens 3 voltage:	0.3 [V]	0.0 [V]	ŏ –
		· 150	- 30					
		1 T	1		Ion Guide voltage:	0.5 [V]	0.0 [V]	0
			- 20		Ion Guide frequency:		1592 [kHz]	~
		1	1		Ion Guide RF amplitude:	0.015 [V]	0.000 [V]	9
		i i i i i i i i i i i i i i i i i i i	- 10		Q1 entrance lens voltage:	0.1 [V]	0.0 [V]	0
		1			Q1 voltage:	49.5 [V]	50.0 [V]	0
			- 0		Q1 frequency:		1093 [kHz]	
		Foreine Pressure	Ion Gauge Pressure		Q1 RF amplitude: Q1 exit lens voltage:	0.151 [V] 0.1 [V]	0.157 [V] 0.0 [V]	8
		Foreille Pressure	ion Gauge Pressure		u'i exit iens voitage.	0.1[0]	0.0 [4]	•
					Q2 voltage:	0.5 [V]	-150.0 [V]	•
					Q2 frequency:		1821 [kHz]	
					Q2 RF amplitude:	0.016 [V]	0.017 [V]	•
					Q3 entrance lens voltage:	0.5 [V]	0.0 [V]	0
					Q3 voltage:	-0.3 [V]	0.0 [V]	8
		Constant alust	could not be created.		Q3 frequency:	2.0 [4]	1088 [kHz]	·
					Q3 RF amplitude:	0.058 [V]	0.058 [V]	0
		No spe	ectra selected.		Q3 exit lens voltage:	0.5 [V]	0.0 [V]	ŏ
					Detector gain:		NaN	
					Multiplier voltage:	2 [V]	0 [V]	•
instruments					Dynode voltage:	0.0 [KV]	-10.0 [KV]	•
🚰 Data								
eWorkflows								

Figure 92. MS Device Window

**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.

#### 2. Go to Create > Instrument Method.

Figure 93. Creating an Instrument Method

Seguence from Works         Teams Search COM Hans         Teams Tofha RSU         Fortise         Own         MEDbrock         Event         Law           Procession Mathed Context energy Method         ISQ 7000         ReadyToDOwyHold         Addression Mathed Context energy Method         Addression Mathed Context energy Method         Addression Mathed Context energy Method         Addression Method </th <th>Folder Sequence</th> <th>C Launch eWorkflow - Smart Startup/Shutdown - 🙀 Re</th> <th></th> <th></th> <th>rogenerated &gt; • 🛶 tig 🖉 .</th> <th></th>	Folder Sequence	C Launch eWorkflow - Smart Startup/Shutdown - 🙀 Re			rogenerated > • 🛶 tig 🖉 .	
Important and with the method w		Themo Scientific GCMS Home Themo TriPlus RSH	Frontiniet Oven MSDevice Elter	Audț Queue		
Contract and instrument Muther       For Kindle       Raddy/E DODWINGad       Addyster       Raddyster	instrument Method					
Advances       Sector Larry       Connect       Sector Larry       Sector L	Procession Method	ISQ 7000 RoadyToDownload	Analyzer Dawas Maintenance	1		
Second Labory Works Hangs Works       Image: Second Labory Works Hangs Works       Image: Labory Mode       Image: Lab	Réport remplate	Ready to Download	Power Mantenance	1		
Specific Licksby/ wirked.no       Image: Control of Specific Licksby/ Wirked.no		Connect	Analyzer		Part I	
Vero Stating- Extendio - Betronic Report. Stating - Stating -			MS Transfer Line terror			
Bectronic Report.     Addate     September     Addate     September       Mit Transfer Line Inpu     2021(1)     302(1)     September     Bectronic Involge     622/0     629/0       Mit Transfer Line Inpu     2021(1)     302(1)     September     September     September     September       Total and paperature     0.0     Image pressure     82 (10-5 ford)     September     September     September       Total and paperature     0.0     Image pressure     82 (10-5 ford)     Image pressure     September     September     September       Total and paperature     0.0     Image pressure     September     September     September     September       Total and paperature     0.0     Image pressure     September     September     September     September       Total and paperature     0.0     Image pressure     September     September     September     September       Total and paperature     0.0     Image pressure     September     September     September     September     September       Total and paperature     0.0     Image pressure     September     September     September     September     September       Total and paperature     Image pressure     September     September     September     September <td></td> <td>Ready</td> <td></td> <td></td> <td></td> <td></td>		Ready				
Partin       Addati       Separati         Mith Transfer Line time       230 [C]       250 [C]         Mith Transfer Line time       230 [C]       250 [C]         Mith Transfer Line time       230 [C]       250 [C]         Torde present       82 [Mith Transfer Line time       300 [C]         Torde present       82 [Mith Transfer Line time       300 [C]         Collasso Gas en:       False         Collasso Gas en:       False         Collasso Gas en:       100 [Mith Transfer Line time         Transfer Line Thesawar       00 [Mith Transfer Line time         Foreise Pressure       00 [Mith Transfer Line time torget       00 [Mith Transfer Line time torget         Foreise Pressure       00 [Mith Transfer Line time torget       00 [Mith Transfer Line time torget       00 [Mith Transfer Line time torget         Foreise Pressure       00 [Mith Transfer Line time torget       00 [Mith Transfer Line time torget       00 [Mith Transfer Line time torget         Foreise Pressure       00 [Mith Transfer Line time torget       01 [Mith Transfer Line time torget       01 [Mith Transfer Line time torget         Spectral plot could not bia created.       No spectra selected       01 [Mith Transfer Line time torget       01 [Mith Transfer Line time torget       01 [Mith Transfer Line torget         O Spectral plot could not bia created.					õ	
Adauf     Edepart       Mid Transfer List Hum     Edepart       Bedrav intervy     66 64       Previde pressure     20 10 10       Previde pressure     8.2 (10-3 for)       Turbe Hum specific     10 9       Colsce Gas no:     Pase       Colsce Gas No:     Pase       Deferred wersy:     6.0 (10-3 for)       De	Electronic Report		System over temp:	True		
M3 Transke Late have may 200 [C] 220 [C] be 50 up can be a conserve the conserve t			Repeller votage:	0.2 [V] 0.0 [V]	•	
be Source term: 2.99 (rd) 300 (rd) Profiles present: 52 (pm)rd to deep resent: 52 (pm)rd Tota Thue speet: 100 % Colse: Ga col: Face College of 64 (rd) 700 (rd) Tota Thue speet: 100 % Colse: Ga col: Face College of 64 (rd) 700 (rd) Tota Thue speet: 100 % Colse: Ga col: Face College of 64 (rd) 700 (rd) Facehor Heaver College of 700 (rd) Facehor Col			Electron lens voltage:	5.4 IVI 5.0 IVI	0	
Prester greater     82 [mit/r]       bit Gauge greater     82 [mit/r]       bit Gauge greater     82 [mit/r]       Colsection Gas col:     Pase       Colsection Gas colsection     Pase       Freeded Paseware     No Gauge Pressure       Spectral plot could not bar created.     No spectra selected       No spectra selected     Pase       Detection gas:     Gas free mathysic       Colsection gas:     Gas free mathysic   <						
Lon Gauge pressure:       0.21 (10-3 ford)         Turbe May sevel:       100 %         Collacion Gas on:       Fade         Fade       Fade         Fade       Fade         Spectral plot could not be empeded       O.5 (M)         No spectra selected       Spectral plot could not be empeded         No spectra selected       Selector gas:         No spectra selected       Selector gas:         Spectral plot could not be empeded       O.5 (M)         Coll edato tase talonge       O.5 (M)		Foreine pressure: 92 ImTorr1			•	
Turde June seed:       100 %         Colsea Gas and the seed of the college set						
Crister Gas or: Pase Character Character Char		Turbo Pump speed: 100 %				
C Desegent Gas fors: 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0		Collision Gas on: False				
Spectral pict could not be created.         No spectra selected.		CI Reagent Gas flow: 0.0 0.0				
Image: Spectral plot could not be created.         No spectra selected		- 150 30			-	
Spectral pict could not be created.         No spectral pict could not be created.					•	
Spectral plot could not be created.         No spectra selected		- 20			0	
Spectral plot could not be meeted.         No spectral plot plot plot plot plot plot plot plo						
Image: Control of the system       0 <td< td=""><td></td><td>- 10</td><td></td><td></td><td></td><td></td></td<>		- 10				
Forekine Pressure     01 /24 areginade     01 /20 /1     01 /20 /1     01 /20 /1       01 /24 areginade     01 /20 /1     01 /20 /1     01 /20 /1     01 /20 /1       02 /24 areginade     03 /20 /1     03 /20 /1     03 /20 /1       03 /24 areginade     03 /20 /1     03 /20 /1     03 /20 /1       04 /24 areginade     03 /20 /1     03 /20 /1     03 /20 /1       05 /24 areginade     03 /20 /1     03 /20 /1     03 /20 /1       05 /24 areginade     03 /20 /1     03 /20 /1     03 /20 /1       05 /24 areginade     03 /20 /1     03 /20 /1     03 /20 /1       02 /24 areginade     03 /20 /1     03 /20 /1     04 /10       03 /24 areginade     03 /20 /1     03 /20 /1     04 /10       03 /24 areginade     03 /24 /1     03 /26 /1     04 /10       03 /24 areginade     03 /24 /1     03 /26 /1     04 /10       03 /24 areginade     03 /24 /1     03 /26 /1     04 /10       03 /24 areginade     03 /24 /1     03 /26 /1     04 /10       03 /24 areginade     03 /24 /1     03 /24 /1     04 /10       03 /24 areginade     03 /24 /1     04 /10     04 /10       03 /24 areginade     04 /10     04 /10     04 /10       04 /24 /24 /24 /24 /24 /24 /24     04 /10     04 /10 <td></td> <td></td> <td></td> <td></td> <td>U</td> <td></td>					U	
Spectral pict could not be created.       No spectra selected.						
Spectral plot could not be created     02 theyency:     0.12 (M)     0.01 (M)     0.01 (M)       03 entence ine votage:     0.4 (M)     0.01 (M)     0.01 (M)     0.01 (M)       03 entence ine votage:     0.4 (M)     0.01 (M)     0.01 (M)       03 entence ine votage:     0.4 (M)     0.01 (M)     0.01 (M)       03 entence ine votage:     0.4 (M)     0.01 (M)     0.01 (M)       03 entence ine votage:     0.01 (M)     0.01 (M)     0.01 (M)       03 entence ine votage:     0.01 (M)     0.01 (M)     0.01 (M)       03 entence ine votage:     0.01 (M)     0.01 (M)     0.01 (M)       03 entence intervotage:     0.01 (M)     0.01 (M)     0.01 (M)       03 entence intervotage:     0.01 (M)     0.01 (M)     0.01 (M)		Foreline Pressure Ion Gauge Pressure	Q1 ext lens voltage:	0.1 [V] 0.0 [V]	•	
Spectral piot could not be created.     02 theorem into a created.       No spectral selected.     03 entrance tere voltage:     0.4 (M)       Difference:     0.4 (M)     0.8 (M)       Difference:     0.8 (M)     0.8 (M)       Difference:     0.8 (M)     0.8 (M)       Difference:     0.8 (M)     0.8 (M)			02 votage:	0.5 [V] -150.0 [V]	•	
Spectral plot could not be created.       No spectral selected.			Q2 frequency:			
Spectral plot could not be created.     0.3 votage     -0.2 V/Y     -       No spectra selected.     0.0 serveroy:     1080 pH/Y       Dietector gan:     0.4 V/Y     0.8 V/Y       Dietector gan:     0.4 V/Y     0.9 V/Y       Dynode votage:     0.6 V/Y     0.9 V/Y			Q2 RF amplitude:	0.016 [V] 0.017 [V]	•	
Spectral plot could not be created.     0.3 votage     -0.2 V/Y     -       No spectra selected.     0.0 serveroy:     1080 pH/Y       Dietector gan:     0.4 V/Y     0.8 V/Y       Dietector gan:     0.4 V/Y     0.9 V/Y       Dynode votage:     0.6 V/Y     0.9 V/Y			Q3 entrance lens voltage:	0.4 [V] 0.0 [V]	9	
No spectra selected     a) of employee     a date ym ( destry )     a)       No spectra selected     a) of employee     a date ym ( destry )     a)       Detector gan:     Na selector gan:     Na selector gan:     Na selector gan:       Dynode vridge:     2 (M)     e (M)     e (M)			Q3 votage:	-0.2 [V] 0.0 [V]		
No spectra selected.		Spectral plot could not be created.				
Detector gan: Nait Multipler voltage: 2 (V) © (V) © Dymode voltage: 0.8 (V) -18.8 (V) ©		No spectra selected				
Mutpier votage: 2 [V] © Dynode votage: 0 0 [VI] •		no specific science.	do car ano ronago.	0.0101 0.0101	<b>U</b>	
Dymode votage: 0.0 [kv] -10.0 [kv]			Detector gain:	NaN		
			Multiplier voltage:		•	
	ts .		Dynode voltage:	0.0 [KV] -10.0 [KV]	•	
	13					

3. The Instrument Method Wizard – Select Instrument window opens. See Figure 94.

Figure 94. Instrument Method Wizard – Select Instrument

strument Method Wizard - Select Instrument	COC 04		
rument Method Wizard guides you through the creation of instrument methods. To start, select the instrument where the method will run.			
elect an Instrument			
₩@☆		Fiter 🖓	z
15Q-PC ISQ-PC			
			_
	< Back Next >	Cancel Help	

- 4. Select the instrument you want to configure and click Next.
- 5. The Instrument Method Wizard System: General Settings window opens. See Figure 95.

Figure 95. Instrument Method Wizard – System: General Settings

I Instrument Method Wizard - System	, General Settings				>
Run Time Please specify the run time of the method 20.000	od: [0.000100000.000 min]				
Diagnostic Channels Select diagnostic channels to be used: No Channel	1	Select all channels			
		Deselect all channels			
		< Back	Next >	Cancel	Help

- 6. You can set the GC Run Time for the method here, or load it with the other GC settings later. Click **Next**.
- 7. The Instrument Method Wizard TriPlusRSH: Settings window opens. See Figure 59.

Figure 96. Instrument Method Wizard – TriPlus RSH: Settings

Instrument Method Wiza	ard - TriPlusRSH: Settings					×
ttings for TriPlusRSH.						Ĭ
New 🛛 📸 Load Template	📓 Save Template 🛛 💕 Load Custom 🛛 🖅 Ed	it Modules				
lethod						
Select method mode and mo	odules, and click Create New Method button					
GC Liquids     G	C Headspace O SPME O SPME Arrow Create New Method	• O ITEX				
Injectors						
Injector port A - ID:	Injector 1	~				
Injector port B - ID:	None	~				
Injector port C - ID:	None	~				
Injector port D - ID:	None	~				
Syringes						
Syringe - ID:	<ul> <li>LS1 (10µL, 57mm)</li> </ul>	~				
Wash stations						
Wash station - ID:	Standard Wash 1 (Standard wash station)	~				
Internal standard / Solvent	tations					
Solvent station - ID:	• None					
Int standard station - ID:	• None					
Cooled/heated trays						
Primary tray - ID:	None	~				
Secondary tray - ID:	None	~				
Secondary hay 10.	1000	•				
				< Back Next >	Cancel	Help

- 8. Choose one of these three options: GC Liquids, GC Headspace, SPME, SPME Arrow, or ITEX.
- 9. 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.

#### 10. Click Create New Method.

11. Use the following instructions to configure the GC Liquids general method settings. See Figure 97.

ings for TriPlusRSH.			
lew 🛛 🚰 Load Template 🕞 Save Template 🛛 🚰 Load Custom 🛛 🐨 Edit M	5		
Injection pot Injector Advanced Injector A (SSL Back)		Basic	
Sample via depth in vial Sample via depth in vial Sample via depth inm): Bottom sense Height from bottom (mm): U2	Non viscous 🔹	Sample Air	

Figure 97. 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.
- 12. Configure the settings under the **Washes** tab (see Figure 98):

Figure 98. GC Liquids Washes Settings

tings for TriPlusRSH.			
	: 🛃 Save Template 🛛 📸 Load Custom 🛛 📝 Edit Modules	-	
eneral Washes Sync Washes	Advanced	Basic	
Number of solvents(s):	Single		
Wash station:	Standard wash station 🔹		
Pre-injection			
Solvent:	A • . • . • . •		
Cycles:	1 -		
Solvent volume (µL):	1.0 👻		
Rinse			
Rinses:	1 •		
Rinse volume (µL):	1.0 🔹	Т	
Post-injection			
Solvent:	B 🔻 - 👻 - 👻 - 👻		
Cycles:	1		
Solvent volume (µL):	1.0 👻		
		Sample	
		Air	
		I	

- 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.
- 13. Click Next. The Instrument Method Wizard GC Inlets(TRACE13007000): FrontInlet Flow/Pressure Options window opens. See Figure 99.

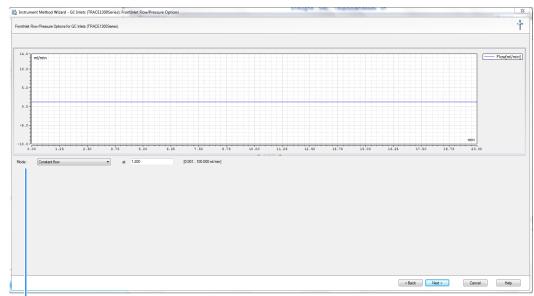


Figure 99. Instrument Method Wizard – Front Inlet Flow/Pressure Options

Carrier Gas Mode

### 14. Set the 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 MS instrument, which is under vacuum, be sure the vacuum compensation is on to ensure accurate flow rates.

15. Click Next. The Instrument Method Wizard – GC Inlets(TRACE13007000): FrontInlet Options window opens. See Figure 100.

Figure 100. Instrument Method Wizard – GC Inlets(TRACE13007000): Front Inlet Options

📩 Instrument	Method Wizard - GC Inlets (TRACE1300Series): Front	Inlet Options	and a second
FrontInlet Optio	ns for GC Inlets (TRACE1300Series).		
Temperature S	ettings		
Enable temp	erature control 🧿	Utilities	
Temperature:	250 🚯 [0400 °C]	Vapour Volume Calculator	
Inlet Parameter	S	Column Flow Calculator	
Operating mode	Splitless 🔹		
Split flow co	antrol 🚯		
Split flow	40.0 (5.01250.0 ml/min]		
Split ratio	33		
Splitless time	1.00 🔇 [0.00999.99 min]		
Purge flow	control 😲		
Purge flow	5.000 (0.50050.000 ml/min)		
Constant se	ptum purge 🚯		
Stop purge for	2.00 (0.00999.99 min]		
Surge pressure	1.0200 (0.050010.0000 bar)		
Surge duration	0.80 (0.00999.99 min]		
Vacuum co	mpensation		
Enable gas	savermode 🔇		
Gas Saver Flow	v 25.0 (5.0500.0 ml/min]		
Gas saver time	1.50 😲 [0.00999.99 min]		

- 16. 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 **Enable Temperature Control** check box and set the temperature high enough to volatize all the analytes in your sample. 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 (482 °F).
  - b. Select the **Split Flow Control** 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, check the **Constant Septum Purge** box to 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 MS 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. 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.
- e. Because the outlet of the column is in the TSQ 9000 instrument, which is under vacuum, be sure the vacuum compensation by checking the **Vacuum Compensation** checkbox is on to ensure accurate flow rates.
- f. In an effort to reduce the amount of carrier gas used, select the Enable Gas Saver Mode. 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 9000 instrument, which can affect the system performance.
- 17. Click Next. The Instrument Method Wizard GC Inlets(TRACE13007000): GC Oven Settings window opens. See Figure 101.

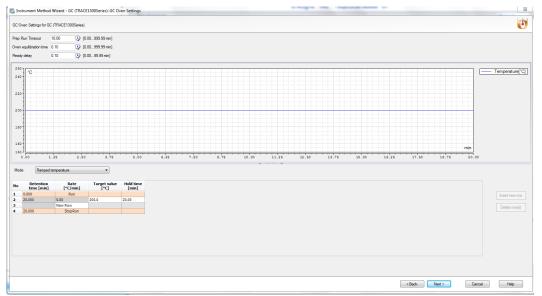


Figure 101. Instrument Method Wizard – GC Inlets: GC Oven Settings

18. Set the oven temperatures in this window. 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.

- 19. 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.
- 20. 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.
- 21. Set the oven 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 instrument.

- 22. (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 9000, 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 7000 GC User Guide* for more information.
- 23. If you have a Programmable Temperature Vaporizer (PTV), click the **PTV-Front** or **PTV-Back** tab to configure it. 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.

**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.

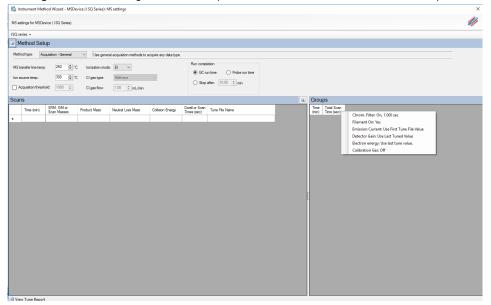
- 24. To add an inlet or detector to the Method editor user interface:
  - a. Attach the inlet or detector to the GC. Refer to the GC documentation for instructions.

- b. Add the inlet or detector to the current instrument configuration. See Chapter 1, "Introduction." for instructions to configure the GC.
- 25. Click Next.
- 26. To create a acquisition method from the Method Type pull-down menu, select **Acquisition-General** or **Acquisition-Timed**. An Acquisition method is used to collect data.

**Note** The maintenance method is used to bake out or cool down the ISQ 7000 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 data acquisition.

27. Use the **Method Setup** pane to set the temperatures and acquisition threshold in General Acquisition mode. See Figure 102.

Figure 102. Creating a General Acquisition Method for the ISQ 7000 Mass Spectrometer



28. 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 ISQ 7000 mass spectrometer. The maximum allowable temperature is 400 °C. However, you will damage the column and contaminate the ISQ 7000 instrument if you set the transfer line temperature above the maximum allowed temperature of the column.

**Note** The maximum transferline temperature of or the rate at which higher transfer line set temperatures may be reached can depend upon the ion source temperature and the GC oven temperature.

29. 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. For most compounds, a source temperature of 275 °C (default) is adequate.

**Note** For best results, tune the instrument at the same temperature you will run the analyses in your method.

- 30. Select the **Acquisition Threshold** checkbox 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.
- 31. Select **EI** from the **Ionization Mode** pull-down menu.

**Note** Only use CI if you have installed a CI ion volume in the ISQ 7000 instrument and you have 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. (There will be a two-minute delay when you change ports for the CI Gas Type.) 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.

- 32. In the **Run Completion** group, select the action you want to occur at the end of a run:
  - a. **GC Run Time**—Select this option if you want the ISQ 7000 system run to end when the GC run is complete. This is the most common setting.
  - b. **Probe Run Time**—Select this option if you have a probe controller installed and you want the ISQ system run to end when the probe run is complete.
  - c. **Select Stop After**—Select this option to set the number of minutes you want the ISQ 7000 system to run. The end of the run can be between 0 and 1,000 minutes. This option allows you to 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. We recommend you select this option it because saves burn time on the filament.

**Note** In Timed Acquisition mode, the run stops when the ISQ 7000 system has completed acquisition for the final sample in your method. These settings do not apply.

33. In the **Scans** pane, click a scan row to enter scan information.

Adampted Statuses		ent Method Wizard		Series): MS settir	0						×
Method Setup         Stander Inter Internet         Via Stander Internet <td>MS settings f</td> <td>for MSDevice (TSQ S</td> <td>Series).</td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td>44</td>	MS settings f	for MSDevice (TSQ S	Series).								44
Mindta fysie     Registration - Generality     Use general acquasion methods to acquire any did type.       Mode fysie     Registration - Generality     200 °C C gas fysie       In source temp:     900 °C C G gas fysie     Mintere       O causation Freehold     000 °C C gas fysie     Mintere       O causation Freehold     000 °C C G gas fysie     Mintere       O causation Freehold     000 °C C G gas figer     000 °C C G gas figer       Scants     U     Groups       Causation Freehold     Time Figge figer     Mintere Figer fi	ISQ series •										
Mindta fysie     Registration - Generality     Use general acquasion methods to acquire any did type.       Mode fysie     Registration - Generality     200 °C C gas fysie       In source temp:     900 °C C G gas fysie     Mintere       O causation Freehold     000 °C C gas fysie     Mintere       O causation Freehold     000 °C C G gas fysie     Mintere       O causation Freehold     000 °C C G gas figer     000 °C C G gas figer       Scants     U     Groups       Causation Freehold     Time Figge figer     Mintere Figer fi	Metho	od Setup									
NS standar lo knowne 250 3 to koration mode 2 v lo source temp: 300 3 to c past spec Mathematica A caudation threaded to c main the O Robe tan time Scans the Material And Andrew	_										
Mit Straderhorter einer     All Straderhorter     Carlot     O Straderhorter     O Straderhorter <t< td=""><td>Method type</td><td><ul> <li>Acquisition - Ge</li> </ul></td><td>eneral V</td><td>Jae general acqui</td><td>isition methods to acqu</td><td></td><td></td><td></td><td></td><td></td><td></td></t<>	Method type	<ul> <li>Acquisition - Ge</li> </ul>	eneral V	Jae general acqui	isition methods to acqu						
Non-source matrix     NO     By To     C gas type     Methods       Inclusion threaded     0000     0 gas flow     0 gas flow     0 gas flow     0 gas flow       Scanse     Mass List or project     Methods     Scanse     Description       100     0 gas flow     0 gas flow     1000 flow     0 gas flow     0 gas flow       100     0 gas flow     0 gas flow     1000 flow     0 gas flow     0 gas flow       100     0 gas flow     0 gas flow     1000 flow     0 gas flow     0 gas flow       100     0 gas flow     0 gas flow     1000 flow     0 gas flow     0 gas flow       100     0 gas flow     0 gas flow     1000 flow     0 gas flow     0 gas flow       100     0 gas flow     0 gas flow     1000 flow     0 gas flow     0 gas flow       100     0 gas flow     0 gas flow     1000 flow     0 gas flow     0 gas flow       100     0 gas flow       100     0 gas flow	MS transfer	line temp: 250	🗢 'C 🛛 loniza	ation mode: El	~						
Image Interested         Constant         Constant <td>Ion source (</td> <td>temp: 300</td> <td>÷ C Clos</td> <td>s type: Met</td> <td>hane</td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td>	Ion source (	temp: 300	÷ C Clos	s type: Met	hane						
Scars         Matrix for Record         Oracle (Sam) SM         Matrix for Record         Oracle (Sam) SM         SM         Matrix for (Sam) SM         Oracle (Sam) SM         SM         Matrix for (Sam) SM         Time (Sac) Sm         Time (Sac) S						0	Stop after: 10.00	πin			
Time Immin         Scan Name         Mail Listor Representation         Dead action Immin         Number Immin         Dead action         Mix/dth (multi Immin)         Tume Sec Immin         Time Sec Immin         Time Sec Immin           2:00         99:09         0.2         0.43 Seved)         Central           000         100,100,200         0.2,0.2.0         100,100,100         Lead Seved)         Central           4:00         35:150         0.1         Lead Seved)         Central			uga Ciga	s flow:	/ 🖃 mL/min						
Perform         Perform         Renge (ama)         Time (ama)         Performance         Land Strend         Performance         Pe									Þ		
200         5950         0.2         Lan Swed         Canad           1         100,190,200         0.2,8,2,02         100,100,100         Lant Swed         Canad           400         35/550         0.1         Lant Swed         Central	Time	Scan Name	Mass List or Bange (amu)	Dwell or Scan Times (sec)	SIM Widths (amu)	Tune File Name	Data Type			Time Total Scan	
4.00 35-150 0.1 [Last Saved] Centroid						[Last Saved]	Centroid			find the best	
					1.00, 1.00, 1.00	[Last Saved]	Centroid				
40x,614         01.0.2         100.1.00         East Saved         Denked											
	8		450, 614	0.1, 0.2	1.00, 1.00	[Last Saved]	Centroid				
E											
									-		
View Tune Report											

#### **Figure 103.** Entering the Scan Information

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

a. The **Time (min)** column is used to set the time that the ISQ 7000 system begins to acquire data after the GC starts. It is typical to have enough of a time delay to allow the solvent to get through the column before starting an acquisition.

As an example, in this method, the mass range of 50-550 amu will be scanned in 0.2 seconds. Beginning from the same start time, three different SIM masses at 100, 150, and 200 will be looked at for 0.2 seconds each. 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 0.816 seconds.

At 4 minutes into the GC run, the scanning is completely changed. Now the full scan range is 35-150 amu, which is scanned every 0.1 seconds, and the two SIM masses are 450 and 614 amu, which are scanned for 0.1 and 0.2 seconds respectively. All these scans will repeat every 0.312 seconds until the GC run is complete.

All of these scans use the last tune file that was saved to the instrument.

b. The **Mass List or Range** column tells the ISQ 7000 system what masses it needs to scan. In full-scan mode, enter the start and end mass separated by a dash. In SIM mode, enter individual masses or multiple values in this field (as long as they are separated by a comma). You must put each full-scan range on a separate line.

**Note** Each line in a scan must only contain a Full-Scan range or individual SIM masses. They cannot be mixed in a single line.

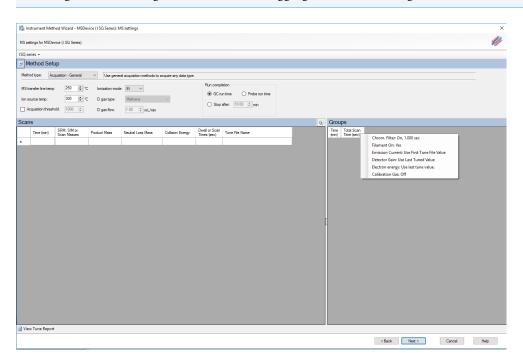
- c. In SIM mode, the **Dwell or Scan Times** column defines the amount of time (in seconds) that the ISQ 7000 instrument will look at your SIM ion mass. If you are in Full-Scan mode, the **Dwell or Scan Times** column determines the amount of time for each individual scan. You should set this value to have 5-20 scans across your GC peak. If you have too few scans, the GC peak area is too imprecise. If you have too many scans, the ISQ 7000 instrument's signal becomes less precise. The default is 0.2 s.
- d. The **Tune File Name** column selects a tune file to be used for this scan. It should be the automatic tune file you created in Tuning the ISQ 7000 Mass Spectrometer. You can also use a specific tune file for each of the scans.
- e. The **Scan Name** column contains a description of the scan. The name may be used as a label to indicate the compound used with the scan.
- f. The **SIM Widths** column sets 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 instrument will collect all the ions from your SIM mass +/- 0.5 amu. Narrower SIM widths lead to greater specificity.
- g. The **Ion Polarity** column is only used in CI mode, which is the only mode for generating positive and negative ions. In EI mode, this column should be always be set to **Positive**.
- h. The **Data Type** column determines whether you want to collect **Profile**, **Centroided**, or **Nominal** mass spectra. Centroided mass spectra is most common because it is used by most of the libraries and provides the smallest data files. Profiled mass spectra provides detailed mass spectral peaks, which results in a large data file that contains details a centroided spectra does not contain. When you want to perform fast scanning (up to 20,000 amu/s), select **Nominal** from the drop-down list under **Data Type**.

Instrument Method Wizard - MSDevice (ISQ Series): MS settings	
Instrument Method Wizard - MSUbovce (ISU Senes): MS settings Settings for MSDevice (ISQ Seites).	
2 series •	
Method Setup	
lethod type: Acquisition - General V Use general acquisition methods to acquire any data type.	
IS transfer line temp: 250 ♦ °C Ionization mode: EI ✓ ● Pun completion ● GC run time ● Probe run time	
n source temp: 300 🖆 'C Clipas type: Methane	
Acquisition threshold: 1000 0 Cl gas flow: 1.00 0 mL/min	
ans	B Groups
Time [imri] Scan Name Mass List or Dwell or Scan Range (anu) Times (sec) SIM Widths (amu) Tune File Name Data Type	Time Total Scan (nin) Time (sec)
0.00 Hange (amu) Limes (sec) On Hand (amu) Limes (sec) On Hand (amu) Limes (sec) On Hand (amu) Caller (Last Saved) Nominal	(mn) Time (sec)
View Tune Report	
	< Back Next > Cancel Help

#### Figure 104. Selecting Nominal Mass Spectra

34. In the **Groups** pane, review the information in each row. As you create scans in the **Scans** pane, information in the groups pane is automatically displayed.

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

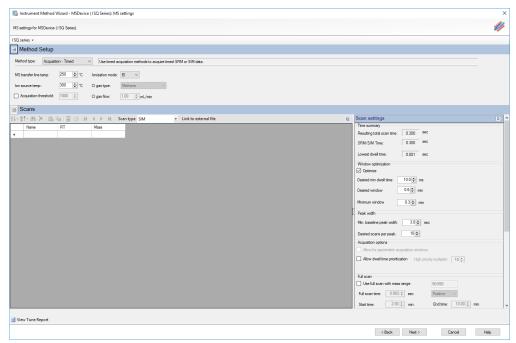


- a. The **Time** column displays the time that the ISQ 7000 mass spectrometer begins to acquire this particular group of scans after the GC starts.
- b. The **Total Scan Time** column indicates the sum of all the scans in each segment. The total scan time also contains the stabilization time that occurs between each scan. In this method, beginning 2.5 minutes into the GC run, you will get a complete set of scans every 0.816 seconds. At 4 minutes into the GC run, the scanning has completely changed.Now the scans will repeat every 0.412 seconds until the GC run is complete.
- c. The **Chrom Filter On** column enables the chromatographic filter. This filter smooths spectral data as it is acquired, which may increase the signal-to-noise ratio by a factor of 2 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.
- d. 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.
- e. The **Filament On** column turns 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 will be collected. Use this column if you have analytes eluting before the solvent peak. You can create a segment to turn off the filament during the solvent peak to preserve the filament.
- f. 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, deselect the Use Tune File Emission Current checkbox 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.
- g. The Use Last Tuned Detector Gain column indicates that you want to use the detector gain set in ISQ 7000 Autotune or set and scanned in manual tune. If you do not need to use the gain set in ISQ 7000 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.
- h. Use the **CI Gas Flow** column to set the flow rate of your reagent gas. Remember to set the CI gas flow in the Groups column as well as 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.
- i. Use the **CI Gas Type** column to set the type of gas attached to one or both CI gas ports (A or B). Be sure that the gas you have assigned to a port is actually attached to that port, as each CI gas has a specific calibration of flow vs. gas viscosity.
- j. The **Cal Gas** column turns on the calibration gas during a run. This setting can be useful when confirming that the system is generating ions and correctly storing the data to disk. Typically, the setting is off, but you may let a low level of calibration gas

into the source by selecting EI. If you have a dual-flow calibration gas module, you may select CI for a high level of gas.

35. (For **Acquisition-Timed** methods only) In the **Instrument Settings** pane, do the following:

Figure 105. Acquisition-Timed Instrument Settings



- 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 the ISQ 7000 Mass Spectrometer," 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 ISQ 7000 Mass Spectrometer," If you are using positive CI ouse the most recent positive CI automatic tune file you created in Chapter 3, "Tuning the ISQ 7000 Mass Spectrometer," 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 ISQ 7000 Autotune. If you do not need to use the gain set in ISQ 7000 Autotune, then you can set the gain manually. Higher gains give larger signals, but may shorten the lifetime of the detector or saturate the electrometer with too much signal 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** ( $\mu A$ ) 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  $\pm 0.5 \mu A$ .

36. As appropriate, use the options in the Scan Settings area to further adjust your method.

Figure 106. Acquisition-Timed Scan Settings

🔁 Instrument Method Wizard - MSDevice (ISQ Series): MS settings			×
MS settings for MSDevice (ISQ Series).			//
ISQ series +			
Method Setup			
Method type: Acquisition - Timed V Use timed acquisition methods to acquire timed SRM or SIM data.			
MS transfer line temp: 250 II (c) lonization mode: EI V			
lon source temp: 300 🖨 'C Cl gas type: Methane 🗸			
Acquisition threshold: 1000 © Ci gas flow: 1.00 © mL/min			
Scans			
21-21-21-21-21-21-21-21-21-21-21-21-21-2	Þ	Scan settings	A 1
Name RT Mass		Time summary Resulting total scan time: 0.300 sec	
		SRM/SIM Time: 0.300 sec	
		Lowest dwell time: 0.001 sec	
		Window optimization	
		Desired min dwell time: 10.0 - ms	
		Desired window 0.6 🜩 min	
		Minimum window 0.3 ÷ min	
		Peak width	
		Min. baseline peak width: 3.0 🖨 sec	
		Desired scans per peak: 10 -	
		Acquisition options	
		Allow for asymmetric acquisition windows	
		Allow dwell time prioritization High priority multiplier: 10	
		Fullscan	
		Use full scan with mass range: 50-550	
		Full scan time: 0.083 🜩 sec Positive 🗸	
		Start time: 2.00 ♀ min. End time: 10.00 ♀ min.	
View Tune Report			
			_
		< Back Next > Cancel He	(p

- a. The **Time Summary** section reports the resulting total scan time, the SIM time, and lowest dwell time for you method. These values are for information only and not editable.
  - i. The **Resulting Total Scan Time** is the baseline peak width divided by the number of points desired across the peak. These values should be updated if your method requirements are different from the defaults.
  - ii. The **SIM** time is the total length of all SIM scans for each compound in your list. This will match the total scan time unless the method also has a full scan event.
  - iii. The **Lowest Dwell Time** is the actual lowest dwell time achieved by the method settings. When the **Optimize** check box is selected, if the actual lowest dwell time is considerably lower than the requested dwell time, then the minimum window has been reached, and if the actual lowest dwell time is considerably higher than the requested dwell time, then the requested window has been reached.
- b. The **Window Optimization** pane allows access to the window optimizer settings. When the optimize button is checked, acquisition windows will be set automatically based on the acquisition window and dwell time targets set in this pane. For complex

SIM methods, this option will help ensure a method is created that can achieve the requested scans per peak.

Algorithm Details: If the **Optimize** checkbox is checked, the SIM acquisition windows in the method are set to the **Desired Window** unless the **Desired Min Dwell Time** cannot be met with the number of **Requested Scans Per Peak**. If this occurs, then the acquisition windows are reduced until either the **Desired Min Dwell Time** is met, or the **Minimum Window** is reached. If the **Minimum Window** is reached first, the **Minimum Dwell Time** is reduced until the **Requested Scans Per Peak** is achievable. If the absolute minimum dwell time on the instrument, which is 0.5 ms, is reached before the requested points across the peak are achieved, the **Minimum Window** is lowered until the **Desired Scans Per Peak** criteria are met or until the absolute allowed minimum window on the instrument is reached, which is 0.24 min. In this very rare case you must reduce the number of **Desired Scans Per Peak**, increase the **Min Baseline Peak Width**, or reduce the number of transitions contained in your method before you will be allowed to save your method.

Settings: **Optimize** is not checked by default. When Optimize is checked, you can adjust the settings in the pane to optimize your method. When the box is not checked, you must manually input your acquisition windows. The default values for the window optimizer should give reasonable results for normal methods. The default values are:

Desired Min Dwell Time—10 ms

Desired Window-0.6 min

Minimum Window-0.3 min

- Change the minimum dwell time using the Desired Min Dwell Time combo box. If your method has many transitions, you may want to reduce the desired minimum dwell time. Note that the wider the acquisition windows in your method, the shorter the average dwell time will be.
- Change the desired acquisition window in the **Desired Window** combo box. The desired window is the amount of time to scan for a transition around a given retention time to ensure that compound will be observed. The desired window can be set from 0.24–5 min. Set the window wide enough so that a retention time shift will not cause you to miss any compounds. Include extra time in this window if there is any uncertainty in compound retention times in the method. Note that the longer the dwell time for your compounds, the narrower your acquisition windows will be.
- Change the minimum acquisition window in the Minimum Window combo box. The minimum window can be set from 0.24–5 min. This is the smallest amount of time that should be scanned for a transition around a given retention time so that you are confident the compound will be observed. Set the minimum window to the lowest safe value to prevent compound retention times from drifting outside the acquisition window.

**Note** If the dwell time limit is reached and the minimum acceptable window is forced below the 0.24 min limit, the method will fail, and a smaller list must be used.

- c. Under **Peak Width**, you can change the minimum baseline peak width and desired scans per peak. These values are used to calculate the total scan time, which includes the SIM time and the full scan time. The minimum baseline peak width should be set roughly to the shortest chromatographic peak time in your analysis.
- d. Under **Full Scan**, you indicate if a Full Scan is to be run along with SIM. 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 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.
- e. Under Acquisition Options, 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.

Note This option is only available when the method optimizer is not active.

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. 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 ISQ 7000 Method Editor, clear the Link to External File check box.

g. After clicking **Link to External File**, the **SIMBridge** dialog box opens. Choose the language of your method file from the **Source Locale** drop-down menu.

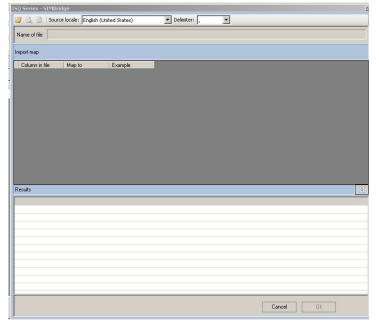


Figure 107. Setting the Source Language of Method Files using SIMBridge

h. Browse to your file.

Figure 108. Linking to an External File using SIM Bridge



- i. Click **Open** to open the method in SIMBridge.
- j. If necessary, change the method headings in your original file to match those in the method editor. A green check mark appears when your method is validated.

Neme of file: C-Wisers/Advin/Desktop/Agilent_SIM_Method_Groups_mod2.csv  mport map  Column in file Map to Example Name 1 Start Time Start time 3.2 Stop Time End time 10.4 Mass Mass 54	Import map Column in Re Map to Example Name Name 1 Start Time Start time 3.2 Stop Time End time 10.4	Import map Column in file Map to Example Name Name 1 Start Time Start time 3.2 Stop Time End time 10.4 Mass Mass 54	
Column in file     Map to     Example       Name     1       Start Time     Start time     3.2       Stop Time     End time     10.4	Column in file     Map to     Example       Name     1       Start Time     3.2       Stop Time     End time       Mass     Mass	Column in file         Map to         Example           Name         1           Start Time         Start time         3.2           Stop Time         End time         10.4           Mass         Mass         54	
Name         Name         1           Start Time         Start time         3.2           Stop Time         End time         10.4	Name Name 1 Start Time Start time 3.2 Stop Time End time 10.4 Mass Mass 54	Nane Nane 1 Start Time Start time 3.2 Stop Time End time 10.4 Mass Mass 54	
Start Time         Start time         3.2           Stop Time         End time         10.4	Start Time     3.2       Stop Time     End time       Mass     Mass	Stat Time 32 Stop Time End line 10.4 Mass Mass 54	
Stop Time End time 10.4	Stop Time     End time       Mass     Mass	Stop Time End time 10.4 Mass Mass 54	
Stop Time End time 10.4	Stop Time     End time       Mass     Mass	Stop Time End time 10.4 Mass Mass 54	
	Mass Mass 54	Mass 54	
INGS3 INGS3 34			
	esuite - 15 compounds found.	suits - 15 compounds found.	
	exuits - 15 compounds found.	suits - 15 compounds found.	
	esuits - 15 compounds found.	exuits - 15 compounds found.	
	esuits - 15 compounds found.	esuits - 15 compounds found.	
	iesuits - 15 compounds found.	esuits - 15 compounds found.	
	esuits - 15 compounds found.	exits - 15 compounds found.	
	esuits - 15 compounds found.	esuits - 15 compounds found.	
	ssuits - 15 compounds found.	suits - 15 compounds found.	
	esuits - 15 compounds found.	esuits - 15 compounds found.	
	esuits - 15 compounds found.	esuits - 15 compounds found.	
	exuits - 15 compounds found.	esults - 15 compounds found.	
	esuits - 15 compounds found.	esults - 15 compounds found.	
	esuits - 15 compounds found.	esults - 15 compounds found.	
esuits - 15 compounds found.			
		. Les las autores las les manufes manufes	
1 6.8 7.2 Positive 54 0 0 3.2 10.4 Normal			
1         6.8         7.2         Positive         54         0         0         3.2         10.4         Normal           2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal	2 11.3 1.8 Positive 109 0 0 10.4 12.2 Normal		
1         6.8         7.2         Positive         54         0         0         3.2         10.4         Normal           2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         12.2         Positive         12         0         0         12.2         Normal	2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         1.2         Positive         121         0         0         12.2         13.4         Normal	3 12.8 1.2 Positive 121 0 0 12.2	
1         6.8         7.2         Positive         54         0         0         3.2         10.4         Normal           2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         1.2         Positive         121         0         0         12.4         Normal           4         14.1         1.4         Positive         119         0         0         13.4         14.8         Normal	2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         1.2         Positive         121         0         0         12.2         13.4         Normal           4         14.1         1.4         Positive         119         0         0         13.4         14.8         Normal	3         12.8         1.2         Positive         121         0         0         12.2           4         14.1         1.4         Positive         119         0         0         13.4	
1         6.8         7.2         Positive         54         0         0         3.2         10.4         Normal           2         11.3         1.8         Positive         10.9         0         0         10.4         12.2         Normal           3         12.8         1.2         Positive         12.1         0         0         12.2         Normal           4         14.1         1.4         Positive         11.9         0         13.4         14.8         Normal           5         15.1         0.6         Positive         79         0         0         14.4         Normal	2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         1.2         Positive         121         0         0         12.2         13.4         Normal           4         14.1         1.4         Positive         119         0         0         13.4         14.8         Normal           5         15.1         0.6         Positive         19         0         0         14.8         15.4         Normal	3         12.8         1.2         Positive         121         0         0         12.2           4         14.1         1.4         Positive         119         0         0         13.4           5         15.1         0.6         Positive         79         0         0         14.8	
1         6.8         7.2         Positive         54         0         0         3.2         10.4         Normal           2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         1.2         Positive         12         0         0         12.4         12.2         Normal           4         14.1         1.4         Positive         119         0         0         13.4         14.8         Normal           5         15.1         0.6         Positive         79         0         0         14.8         15.4         Normal           5         15.1         0.6         Positive         79         0         0         15.8         Normal	2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         1.2         Positive         121         0         0         12.2         13.4         Normal           3         12.4         1.4         Positive         119         0         0         13.4         14.8         Normal           5         15.1         0.6         Positive         79         0         0         13.4         15.4         Normal           5         15.6         0.4         Positive         79         0         0         15.4         Normal	3         12.8         1.2         Positive         121         0         0         12.2           4         14.1         1.4         Positive         119         0         0         13.4           5         15.1         0.6         Positive         79         0         0         14.8           5         15.5         0.4         Positive         79         0         0         15.4	
1         6.8         7.2         Positive         54         0         0         3.2         10.4         Normal           2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         1.2         Positive         121         0         0         12.2         13.4         Normal           4         14.1         1.4         Positive         119         0         0         13.4         14.8         Normal           5         15.1         0.6         Positive         79         0         14.8         15.4         Normal           6         15.6         0.4         Positive         79         0         15.4         15.8         Normal           7         16.15         0.7         Positive         70         0         15.4         15.8         Normal	2         11.3         1.8         Positive         108         0         0         10.4         12.2         Normal           3         12.8         1.2         Positive         12.1         0         0         12.2         13.4         Normal           4         14.1         1.4         Positive         119         0         0         13.4         Normal           5         15.1         0.6         Positive         73         0         0         14.8         15.4         Normal           6         15.6         0.4         Positive         73         0         0         15.4         15.8         Normal           7         16.15         0.7         Positive         105         0         15.8         16.5         Normal	3         12.8         1.2         Positive         121         0         0         122           4         14.1         14         Positive         119         0         0         134           5         15.1         0.6         Positive         79         0         0         14.8           6         15.5         0.4         Positive         79         0         0         14.8           7         16.15         0.7         Positive         79         0         0         15.4	
1         6.8         7.2         Positive         54         0         0         3.2         10.4         Normal           2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         1.2         Positive         12         0         0         12.4         1.4         Normal           4         14.1         1.4         Positive         119         0         0         13.4         14.8         Normal           5         15.1         0.6         Positive         79         0         0         14.4         15.4         Normal           6         15.6         0.4         Positive         73         0         0         15.4         15.8         Normal           7         16.15         0.7         Positive         70         0         15.8         16.5         Normal           8         17.25         15.7         Positive         105         0         15.8         16.5         Normal	2         1.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         12.4         Positive         12.1         0         0         12.2         13.4         Normal           4         14.1         1.4         Positive         119         0         0         13.4         14.8         Normal           5         15.1         0.6         Positive         73         0         0         15.4         Normal           6         15.5         0.4         Positive         73         0         0         15.4         Normal           7         15.5         0.7         Positive         17.9         0         0         15.4         Normal           7         15.5         0.7         Positive         105         0         0         15.8         16.5         Normal           8         17.25         1.5         Positive         105         0         0         15.8         16.5         Normal	3         12.8         1.2         Positive         121         0         0         122           4         1.4.1         1.4         Positive         119         0         0         13.4           5         15.1         0.6         Positive         79         0         0         14.8           6         15.6         0.4         Positive         79         0         0         15.4           7         16.15         0.7         Positive         105         0         0         15.6           8         17.25         1.5         Positive         105         0         0         15.6	16.5 18 Normal
1         6.8         7.2         Positive         54         0         0         3.2         10.4         Normal           2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         1.2         Positive         12         0         0         12.4         12.2         Normal           4         14.1         1.4         Positive         119         0         0         13.4         14.8         Normal           5         15.1         0.6         Positive         79         0         0         14.8         15.4         Normal           5         15.5         0.4         Positive         79         0         0         15.8         15.8         Normal           7         15.5         0.7         Positive         105         0         0         15.8         16.5         Normal           8         17.25         1.5         Positive         70         0         0         16.5         18         Normal           9         18.5         1         Positive         79         0         0         18	2         11.3         1.8         Positive         109         0         0         104         12.2         Normal           3         12.8         1.2         Positive         121         0         0         12.2         13.4         Normal           4         14.1         1.4         Positive         119         0         0         13.4         14.8         Normal           5         15.1         0.6         Positive         79         0         0         14.8         15.4         Normal           5         15.5         0.4         Positive         79         0         0         15.4         Normal           6         15.6         0.7         Positive         79         0         0         15.4         Normal           7         16.15         0.7         Positive         105         0         0         15.8         Normal           8         17.25         1.5         Positive         105         0         0         18.5         Normal           9         18.5         1         Positive         79         0         0         18         19         Normal	3         1.2         Positive         1.2         0         0         1.2           4         14.1         14.4         Positive         119         0         0         1.34           5         15.1         0.6         Positive         79         0         0         1.48           6         15.6         0.4         Positive         79         0         0         1.48           7         16.15         0.7         Positive         105         0         0         1.88           8         1.25         1.7         Positive         105         0         0         18.6           9         18.5         1         Positive         105         0         0         18.6	16.5 18 Normal 18 19 Normal
1         6.8         7.2         Positive         54         0         0         3.2         10.4         Normal           2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         1.2         Positive         121         0         0         12.4         12.2         Normal           4         14.1         1.4         Positive         121         0         0         12.4         14.8         Normal           5         15.5         10.6         Positive         79         0         15.4         15.4         Normal           5         15.6         0.4         Positive         105         0         15.4         15.8         Normal           5         15.7         Positive         105         0         15.8         16.5         Normal           7         15.15         10.7         Positive         105         0         15.8         16.5         Normal           8         17.25         1.5         Positive         105         0         16.5         18         Normal           9         16.5	2         11.3         1.8         Positive         108         0         0         10.4         12.2         Normal           3         12.8         1.2         Positive         12.1         0         0         12.2         13.4         Normal           4         14.1         1.4         Positive         17.9         0         0         13.4         Normal           5         15.1         0.6         Positive         7.9         0         0         14.8         15.4         Normal           6         15.6         0.4         Positive         7.9         0         0         15.4         15.8         Normal           7         16.15         0.7         Positive         105         0         0         15.8         16.5         Normal           18         12.5         1.5         Positive         105         0         0         18.8         16.5         Normal           19         18.5         1.4         Positive         7.9         0         0         18.9         Normal           10         13.3         0.6         Positive         8         0         18         19.9         Normal	3         12.8         1.2         Positive         121         0         0         122           4         14.1         1.4         Positive         119         0         0         1.34           5         15.5         0.6         Positive         79         0         0         1.84           6         15.6         0.4         Positive         79         0         0         1.84           7         16.15         0.4         Positive         79         0         0         1.84           8         12.5         7         Positive         105         0         0         158           9         12.5         1.5         Positive         79         0         0         168           9         18.5         1         Positive         79         0         0         18           10         13.3         0.6         Positive         79         0         0         18	16.5         18         Normal           18         19         Normal           19         19.6         Normal
Results - 15 compounds found.			
Name   RT   Window (min)   Ion Polarity   Mass   Pre-width (min)   Post-width (min)   Start time   End time   Vial Position   Dwell Time Priority		2 1 6.8 7.2 Positive 54 0 0 3.2	3.2 10.4 Normal
	🔰 6.8 7.2 Positive 54 0 0 3.2 10.4 Normal		
1 6.8 7.2 Positive 54 0 0 3.2 10.4 Normal			
1         6.8         7.2         Positive         54         0         0         3.2         10.4         Normal           2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal	2 11.3 1.8 Positive 109 0 0 10.4 12.2 Normal		12.2 13.4 Normal
1         6.8         7.2         Positive         54         0         0         3.2         10.4         Normal           2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal	2 11.3 1.8 Positive 109 0 0 10.4 12.2 Normal		
1         6.8         7.2         Positive         54         0         0         3.2         10.4         Normal           2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           35         12.8         12.2         Positive         12         0         0         12.2         Normal	2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         1.2         Positive         121         0         0         12.2         13.4         Normal	3 12.8 1.2 Positive 121 0 0 12.2	13.4 14.8 Normal
1         6.8         7.2         Positive         54         0         0         3.2         10.4         Normal           2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           35         12.8         12.2         Positive         12         0         0         12.2         Normal	2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         1.2         Positive         121         0         0         12.2         13.4         Normal	3 12.8 1.2 Positive 121 0 0 12.2	
1         6.8         7.2         Poritive         54         0         0         3.2         10.4         Normal           2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         1.2         Positive         121         0         12.2         13.4         Normal           4         14.1         1.4         Positive         119         0         0         13.4         14.8         Normal	2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         1.2         Positive         121         0         0         12.2         13.4         Normal           4         14.1         1.4         Positive         119         0         0         13.4         Normal	3         12.8         1.2         Positive         121         0         0         12.2           4         14.1         1.4         Positive         119         0         0         13.4	14.8 15.4 Normal
1         6.8         7.2         Poritive         54         0         0         3.2         10.4         Normal           2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         1.2         Positive         121         0         12.2         13.4         Normal           4         14.1         1.4         Positive         119         0         0         13.4         14.8         Normal	2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         1.2         Positive         121         0         0         12.2         13.4         Normal           4         14.1         1.4         Positive         119         0         0         13.4         Normal	3         12.8         1.2         Positive         121         0         0         12.2           4         14.1         1.4         Positive         119         0         0         13.4	
1         6.8         7.2         Positive         54         0         0         3.2         10.4         Normal           2         11.3         1.8         Positive         10.9         0         0.10.4         12.2         Normal           3         12.8         1.2         Positive         12.0         0         12.2         Normal           3         14.1         1.4         Positive         119         0         0         13.4         Normal           5         15.1         0.6         Positive         79         0         0         14.4         Normal	2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         1.2         Positive         121         0         0         12.2         13.4         Normal           4         14.1         1.4         Positive         119         0         0         13.4         14.8         Normal           5         15.1         0.5         Positive         79         0         0         14.8         15.4         Normal	3         12.8         1.2         Positive         121         0         0         12.2           4         14.1         1.4         Positive         119         0         0         13.4           5         15.1         0.6         Positive         79         0         0         14.8	
1         6.8         7.2         Positive         54         0         0         3.2         10.4         Normal           2         11.3         1.8         Positive         10.9         0         0.10.4         12.2         Normal           3         12.8         1.2         Positive         12.0         0         12.2         Normal           3         14.1         1.4         Positive         119         0         0         13.4         Normal           5         15.1         0.6         Positive         79         0         0         14.4         Normal	2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         1.2         Positive         121         0         0         12.2         13.4         Normal           4         14.1         1.4         Positive         119         0         0         13.4         14.8         Normal           5         15.1         0.5         Positive         79         0         0         14.8         15.4         Normal	3         12.8         1.2         Positive         121         0         0         12.2           4         14.1         1.4         Positive         119         0         0         13.4           5         15.1         0.6         Positive         79         0         0         14.8	
1         6.8         7.2         Positive         54         0         0         3.2         10.4         Normal           2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         1.2         Positive         12         0         0         12.4         12.2         Normal           4         14.1         1.4         Positive         119         0         0         13.4         14.8         Normal           5         15.1         0.6         Positive         79         0         0         14.8         15.4         Normal           5         15.1         0.4         Positive         79         0         0         15.8         Normal	2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         12.         Positive         121         0         0         12.2         13.4         Normal           4         14.1         1.4         Positive         119         0         0         13.4         14.8         Normal           5         15.1         0.6         Positive         79         0         0         14.8         15.4         Normal           5         15.6         0.4         Positive         79         0         0         15.4         Normal	3         12.8         1.2         Positive         121         0         0         12.2           4         14.1         1.4         Positive         119         0         0         13.4           5         15.1         0.6         Positive         79         0         0         14.8           6         15.5         0.4         Positive         79         0         0         15.4	
1         6.8         7.2         Positive         54         0         0         3.2         10.4         Normal           2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         1.2         Positive         12         0         0         12.4         12.2         Normal           4         14.1         1.4         Positive         119         0         0         13.4         14.8         Normal           5         15.1         0.6         Positive         79         0         0         14.8         15.4         Normal           5         15.1         0.4         Positive         79         0         0         15.8         Normal	2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         12.         Positive         121         0         0         12.2         13.4         Normal           4         14.1         1.4         Positive         119         0         0         13.4         14.8         Normal           5         15.1         0.6         Positive         79         0         0         14.8         15.4         Normal           5         15.6         0.4         Positive         79         0         0         15.4         Normal	3         12.8         1.2         Positive         121         0         0         12.2           4         14.1         1.4         Positive         119         0         0         13.4           5         15.1         0.6         Positive         79         0         0         14.8           6         15.5         0.4         Positive         79         0         0         15.4	
1         6.8         7.2         Positive         54         0         0         3.2         10.4         Normal           2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         1.2         Positive         12         0         0         12.4         12.2         Normal           4         14.1         1.4         Positive         119         0         0         13.4         14.8         Normal           5         15.1         0.6         Positive         79         0         0         14.8         15.4         Normal           5         15.1         0.4         Positive         79         0         0         15.8         Normal	2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         12.         Positive         121         0         0         12.2         13.4         Normal           4         14.1         1.4         Positive         119         0         0         13.4         14.8         Normal           5         15.1         0.6         Positive         79         0         0         14.8         15.4         Normal           5         15.6         0.4         Positive         79         0         0         15.4         Normal	3         12.8         1.2         Positive         121         0         0         12.2           4         14.1         1.4         Positive         119         0         0         13.4           5         15.1         0.6         Positive         79         0         0         14.8           6         15.5         0.4         Positive         79         0         0         15.4	15.8 16.5 Normal
1         6.8         7.2         Positive         54         0         0         3.2         10.4         Normal           2         11.3         1.8         Positive         10.9         0         0         10.4         12.2         Normal           3         12.8         12.8         12.8         12.4         Normal           4         14.1         1.4         Positive         12.9         0         12.4         14.8         Normal           5         15.1         0.6         Positive         79         0         13.4         14.8         Normal           5         15.0         0.4         Positive         79         0         14.8         15.4         Normal           5         15.0         0.4         Positive         79         0         14.8         15.4         Normal	2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         1.2         Positive         121         0         0         12.2         13.4         Normal           4         14.1         1.4         Positive         119         0         0         13.4         14.8         Normal           5         15.1         0.6         Positive         79         0         0         14.8         15.4         Normal           5         15.6         0.4         Positive         79         0         0         15.4         Normal	3         12.8         1.2         Positive         121         0         0         12.2           4         14.1         1.4         Positive         119         0         0         13.4           5         15.1         0.6         Positive         79         0         0         14.8           6         15.5         0.4         Positive         79         0         0         15.4	15.8 16.5 Normal
1         6.8         7.2         Poritive         54         0         0         3.2         10.4         Normal           2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         1.2         Positive         109         0         0         12.4         12.4         Normal           4         14.1         1.4         Positive         119         0         13.4         Normal           5         15.1         0.6         Positive         79         0         14.8         15.4         Normal           6         15.6         0.4         Positive         79         0         15.4         15.8         Normal           7         16.15         0.7         Positive         70         0         15.4         15.8         Normal	2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         1.2         Positive         121         0         0         12.2         13.4         Normal           4         14.1         1.4         Positive         119         0         0         13.4         Normal           5         15.1         0.6         Positive         73         0         0         14.8         15.4         Normal           6         15.6         0.4         Positive         73         0         0         15.4         15.8         Normal           7         16.15         0.7         Positive         105         0         15.8         16.5         Normal	3         1.2         Positive         1.2         0         0         1.2           4         14.1         1.4         Positive         119         0         0         1.34           5         15.1         0.6         Positive         79         0         0         1.84           6         15.5         0.4         Positive         79         0         0         1.84           7         15.15         0.7         Positive         79         0         0         1.54	
1         6.8         7.2         Poritive         54         0         0         3.2         10.4         Normal           2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         1.2         Positive         109         0         0         12.4         12.4         Normal           4         14.1         1.4         Positive         119         0         13.4         Normal           5         15.1         0.6         Positive         79         0         14.8         15.4         Normal           6         15.6         0.4         Positive         79         0         15.4         15.8         Normal           7         16.15         0.7         Positive         70         0         15.4         15.8         Normal	2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         1.2         Positive         121         0         0         12.2         13.4         Normal           4         14.1         1.4         Positive         119         0         0         13.4         Normal           5         151.         0.6         Positive         73         0         0         14.8         15.4         Normal           6         156.         0.4         Positive         73         0         0         15.4         15.8         Normal           7         16.15         0.7         Positive         105         0         0         15.8         16.5         Normal	3         1.2         Positive         1.2         0         0         1.2           4         14.1         1.4         Positive         119         0         0         1.34           5         15.1         0.6         Positive         79         0         0         1.84           6         15.5         0.4         Positive         79         0         0         1.84           7         15.15         0.7         Positive         79         0         0         1.54	
1         6.8         7.2         Positive         54         0         0         3.2         10.4         Normal           2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         1.2         Positive         12         0         0         12.4         1.4         Normal           4         14.1         1.4         Positive         119         0         0         13.4         14.8         Normal           5         15.1         0.6         Positive         79         0         0         14.4         15.4         Normal           6         15.6         0.4         Positive         73         0         0         15.4         15.8         Normal           7         16.15         0.7         Positive         70         0         15.8         16.5         Normal           8         17.25         15.7         Positive         105         0         15.8         16.5         Normal	2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         12.4         Positive         12.0         0         0         12.2         13.4         Normal           4         14.1         1.4         Positive         119         0         0         13.4         14.8         Normal           5         15.1         0.6         Positive         79         0         0         14.8         15.4         Normal           6         15.5         0.4         Positive         79         0         0         15.4         Normal           7         15.5         0.7         Positive         105         0         15.8         Normal           8         17.25         1.5         Positive         105         0         0         15.8         16.5         Normal	3         12.8         1.2         Positive         121         0         0         122           4         1.4.1         1.4         Positive         119         0         0         13.4           5         15.1         0.6         Positive         79         0         0         14.8           6         15.6         0.4         Positive         79         0         0         15.4           7         16.15         0.7         Positive         105         0         0         15.6           8         17.25         1.5         Positive         105         0         0         15.6	16.5 18 Normal
1         6.8         7.2         Positive         54         0         0         3.2         10.4         Normal           2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         1.2         Positive         12         0         0         12.4         1.4         Normal           4         14.1         1.4         Positive         119         0         0         13.4         14.8         Normal           5         15.1         0.6         Positive         79         0         0         14.4         15.4         Normal           6         15.6         0.4         Positive         73         0         0         15.4         15.8         Normal           7         16.15         0.7         Positive         70         0         15.8         16.5         Normal           8         17.25         15.7         Positive         105         0         15.8         16.5         Normal	2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         12.4         Positive         12.0         0         0         12.2         13.4         Normal           4         14.1         1.4         Positive         119         0         0         13.4         14.8         Normal           5         15.1         0.6         Positive         79         0         0         14.8         15.4         Normal           6         15.5         0.4         Positive         79         0         0         15.4         Normal           7         15.5         0.7         Positive         105         0         15.8         Normal           8         17.25         1.5         Positive         105         0         0         15.8         16.5         Normal	3         12.8         1.2         Positive         121         0         0         122           4         1.4.1         1.4         Positive         119         0         0         13.4           5         15.1         0.6         Positive         79         0         0         14.8           6         15.6         0.4         Positive         79         0         0         15.4           7         16.15         0.7         Positive         105         0         0         15.6           8         17.25         1.5         Positive         105         0         0         15.6	16.5 18 Normal
1         6.8         7.2         Positive         54         0         0         3.2         10.4         Normal           2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         1.2         Positive         12         0         0         12.4         1.4         Normal           4         14.1         1.4         Positive         119         0         0         13.4         14.8         Normal           5         15.1         0.6         Positive         79         0         0         14.4         15.4         Normal           6         15.6         0.4         Positive         73         0         0         15.4         15.8         Normal           7         16.15         0.7         Positive         70         0         15.8         16.5         Normal           8         17.25         15.7         Positive         105         0         15.8         16.5         Normal	2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         12.4         Positive         12.0         0         0         12.2         13.4         Normal           4         14.1         1.4         Positive         119         0         0         13.4         14.8         Normal           5         15.1         0.6         Positive         79         0         0         14.8         15.4         Normal           6         15.5         0.4         Positive         79         0         0         15.4         Normal           7         15.5         0.7         Positive         105         0         15.8         Normal           8         17.25         1.5         Positive         105         0         0         15.8         16.5         Normal	3         12.8         1.2         Positive         121         0         0         122           4         1.4.1         1.4         Positive         119         0         0         13.4           5         15.1         0.6         Positive         79         0         0         14.8           6         15.6         0.4         Positive         79         0         0         15.4           7         16.15         0.7         Positive         105         0         0         15.6           8         17.25         1.5         Positive         105         0         0         15.6	16.5 18 Normal
1         6.8         7.2         Positive         54         0         0         3.2         10.4         Normal           2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         1.2         Positive         12         0         0         12.4         12.2         Normal           4         14.1         1.4         Positive         119         0         0         13.4         14.8         Normal           5         15.1         0.6         Positive         79         0         0         14.8         15.4         Normal           5         15.5         0.4         Positive         79         0         0         15.8         15.8         Normal           7         15.5         0.7         Positive         105         0         0         15.8         16.5         Normal           8         17.25         1.5         Positive         70         0         0         16.5         18         Normal           9         18.5         1         Positive         79         0         0         18	2         11.3         1.8         Positive         109         0         0         104         12.2         Normal           3         12.8         1.2         Positive         121         0         0         12.2         13.4         Normal           4         14.1         1.4         Positive         119         0         0         13.4         14.8         Normal           5         15.1         0.6         Positive         79         0         0         14.8         15.4         Normal           5         15.5         0.4         Positive         79         0         0         15.4         Normal           6         15.6         0.7         Positive         79         0         0         15.4         Normal           7         16.15         0.7         Positive         105         0         0         15.8         Normal           8         17.25         1.5         Positive         105         0         0         18.5         Normal           9         18.5         1         Positive         79         0         0         18         19         Normal	3         1.2         Positive         1.2         0         0         1.2           4         14.1         14.4         Positive         119         0         0         1.34           5         15.1         0.6         Positive         79         0         0         1.48           6         15.6         0.4         Positive         79         0         0         1.48           7         16.15         0.7         Positive         105         0         0         1.88           8         1.25         1.7         Positive         105         0         0         18.6           9         18.5         1         Positive         105         0         0         18.6	16.5 18 Normal 18 19 Normal
1         6.8         7.2         Positive         54         0         0         3.2         10.4         Normal           2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         1.2         Positive         12         0         0         12.4         12.2         Normal           4         14.1         1.4         Positive         119         0         0         13.4         14.8         Normal           5         15.1         0.6         Positive         79         0         0         14.8         15.4         Normal           5         15.5         0.4         Positive         79         0         0         15.8         15.8         Normal           7         15.5         0.7         Positive         105         0         0         15.8         16.5         Normal           8         17.25         1.5         Positive         70         0         0         16.5         18         Normal           9         18.5         1         Positive         79         0         0         18	2         11.3         1.8         Positive         109         0         0         104         12.2         Normal           3         12.8         1.2         Positive         121         0         0         12.2         13.4         Normal           4         14.1         1.4         Positive         119         0         0         13.4         14.8         Normal           5         15.1         0.6         Positive         79         0         0         14.8         15.4         Normal           5         15.5         0.4         Positive         79         0         0         15.4         Normal           6         15.6         0.7         Positive         79         0         0         15.4         Normal           7         16.15         0.7         Positive         105         0         0         15.8         Normal           8         17.25         1.5         Positive         105         0         0         18.5         Normal           9         18.5         1         Positive         79         0         0         18         19         Normal	3         1.2         Positive         1.2         0         0         1.2           4         14.1         14.4         Positive         119         0         0         1.34           5         15.1         0.6         Positive         79         0         0         1.48           6         15.6         0.4         Positive         79         0         0         1.48           7         16.15         0.7         Positive         105         0         0         1.88           8         1.25         1.7         Positive         105         0         0         18.6           9         18.5         1         Positive         105         0         0         18.6	16.5 18 Normal 18 19 Normal
1         6.8         7.2         Positive         54         0         0         3.2         10.4         Normal           2         11.3         1.8         Positive         109         0         0         10.4         12.2         Normal           3         12.8         1.2         Positive         12         0         0         12.4         12.2         Normal           4         14.1         1.4         Positive         119         0         0         13.4         14.8         Normal           5         15.1         0.6         Positive         79         0         0         14.8         15.4         Normal           5         15.5         0.4         Positive         79         0         0         15.8         15.8         Normal           7         15.5         0.7         Positive         105         0         0         15.8         16.5         Normal           8         17.25         1.5         Positive         70         0         0         16.5         18         Normal           9         18.5         1         Positive         79         0         0         18	2         11.3         1.8         Positive         108         0         0         10.4         12.2         Normal           3         12.8         1.2         Positive         12.1         0         0         12.2         13.4         Normal           4         14.1         1.4         Positive         17.9         0         0         13.4         Normal           5         15.1         0.6         Positive         7.9         0         0         14.8         15.4         Normal           6         15.6         0.4         Positive         7.9         0         0         15.4         15.8         Normal           7         16.15         0.7         Positive         105         0         0         15.8         16.5         Normal           18         12.5         1.5         Positive         105         0         0         18.8         16.5         Normal           19         18.5         1.4         Positive         7.9         0         0         18.9         Normal           10         13.3         0.6         Positive         8         0         18         19.9         Normal	3         12.8         1.2         Positive         121         0         0         122           4         14.1         1.4         Positive         119         0         0         1.34           5         15.5         0.6         Positive         79         0         0         1.84           6         15.6         0.4         Positive         79         0         0         1.84           7         16.15         0.4         Positive         79         0         0         1.84           8         12.5         7         Positive         105         0         0         158           9         12.5         1.5         Positive         79         0         0         168           9         18.5         1         Positive         79         0         0         18           10         13.3         0.6         Positive         79         0         0         18	16.5         18         Normal           18         19         Normal           19         19.6         Normal

#### Figure 109. Changing Method Headings in SIMBridge

k. Click **Open** and the external method will be opened in the method editor.

Figure 110.	Viewing a Linked File in the Method Editor
-------------	--

🔩 Instrument Method Wizard - MSDevice (ISQ Series): MS settings	×
MS settings for MSDevice (1SQ Series).	11
ISQ series •	
Method Setup	
Method type: Acquisition - Timed V Use timed acquisition methods to acquire timed SIM data.	
nenero you - requestori - rinno - Use ulled eculasion menodasi o eculare uned alim data.	
MS transfer line temp: 250 🚖 °C Ionization mode: EI 🗸	
Ion source temp: 300 🖶 1C Cligas type: Methane	
Acquisition threshold: 1000 \$ CI gas flow: 1.00 \$ mL/min	
Scans	
21-21-81 × [15 the line   ] ()     4 → H    :Linkto external file D	SRM Resolution
Name RT Window (min) Mass	Set resolution for each unique transition scan
2 11.30 1.80 109	Precursor (Q1): Normal ~
3 12.00 1.20 1.21	Product (Q3): Normal ~
4 14.10 1.40 119	Acquisition options
5 15.10 0.60 79	Allow for asymmetric acquisition windows
6 15.60 0.40 79	Allow dwell time prioritization High priority multiplier: 10 0
7 16.15 0.70 105	Allow unfiltered signal
8 17.25 1.50 105	Fullscan
9 18.50 1.00 79	Use full scan with mass range: 50-550
10 19.30 0.60 84	Full scan time: 0.083 - sec Positive
11 20.20 1.20 129	
12 21.40 1.20 129	: Start time: 2.00 ♀ min. End time: 10.00 ♀ min.
13 22.35 0.70 167	Instrument settings
14 23.65 1.90 105	Tune file
15 28.05 6.90 84	Tune file (+): (Last Saved) ~
	Tune File (-): Autotune_NCI
	Detector gain
	Use last tuned detector gain X 7.0
	Use specified detector gain: 2.1E+006
	Emission current
	Use tune file emission current
	O Use specified emission current: 50 ⊕ (µA)
☐ View Tune Report	
	< Back Next > Cancel Help

1. 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. This function is useful if you need to edit an analyte in a complex method.

- m. In the **RT** column, enter retention times for SIM methods. The retention time is the time it takes an analyte to pass from the column inlet to the detector.
- 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.
- o. In the Mass column, enter the mass of the ion you wish to monitor.
- p. 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**.
- q. You may set the number of adjacent analytes to show in your method by using the Number of Adjacent Analytes to Show selection box found in the Options dialog box accessed by the ISQ 7000 main menu. See Figure 71. View the number of analytes you set in the Show Analysis view.

Adjustments in this column are for display purposes only and will not affect your acquisition.

**Figure 111.** Options Dialog Box

Options		<b>x</b>
Number of adja	acent analytes to	show: 10 🛨
	Ok	Cancel

r. Click **Show Analysis** (see Figure 72) to validate your method. A chart appears with all your analytes by name and in order of their start times.

Figure 112. Accessing the Show Analysis Feature

1	• • • • • • • • •			
ŀ		3 🔁 💽 🛞 📕 -		
	Name		Window (min) Mass	Time summary
	1	6.80	7.20 54	Show analysis pulling total scan time: 0.300 sec
	2	11.30	1.80 109	SIM Time: 0.300 sec
	3	12.80	1.20 121	
	4	14.10	1.40 119	Lowest dwell time; 0,29593 sec
	5	15.10	0.60 79	- Window optimization
	6	15.60	0.40 79	C Optimize
	7	16.15	0.70 105	Desired min dwell time: 10.0 + ms
	8	17.25	1.50 105	
	9	18.50	1.00 79	Desired window
	10	19.30	0.60 84	Minimum window 0.3 🛫 min
	11	20.20	1.20 129	
	12	21.40	1.20 129	Peak width
	13	22.35	0.70 167	Min. baseline peak width: 3.0 = sec
	14	23.65	1.90 105	Desired scans per peak:
	15	28.05	6.90 84	
*				Acquisition options
				Alow for asymmetric acquisition windows
				Allow dwell time prioritization High priority multiplies:
				Fullscan
				Use full scan with mass range: 50-550

124 ISQ 7000 User Guide

s. Use the scrolling window at the bottom of the screen to view all your analytes' expected retention times. Resize the window to view more analytes by dragging one side of the window out. As the scrolling window is decreased in size, fewer analytes are shown in the analytes chart. Increasing the size of the scrolling window allows you to view more analytes in your method. If the number of analytes retention times being viewed exceeds 50, an evenly spaced sample of the analytes shows through the window.

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 SIM windows are too congested to achieve the total scan time at the minimum dwell time, the segments are highlighted in red. This warning shows that there is not sufficient time in the segment to scan all events. In this case, the method fails to validate or save and a caution icon appears near the scans title. To correct this, reduce the number of overlapping compounds or change window times. When the peak bars are highlighted orange, this is a caution that there will be fewer scans across the peak than desired.

Also, if your list contains duplicate compounds, the middle of one of the duplicate peak bars will show an orange crossed pattern, instead of the usual white. Delete one of the duplicate compounds to avoid problems with data analysis.

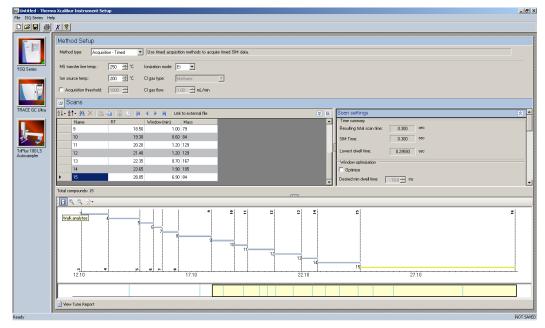


Figure 113. Validating a Method

37. To import an ISQ "Classic", ISQ LT, or ISQ QD method, 7000 MS method, choose **ISQ 7000 | Import MS Method**. See Figure 74.

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

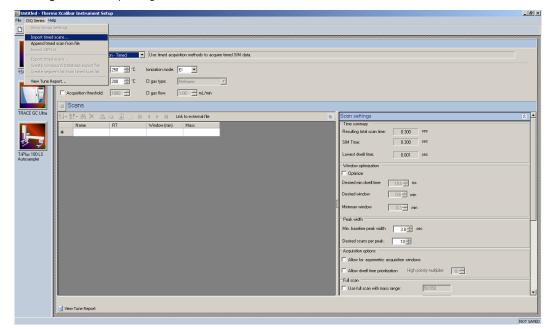
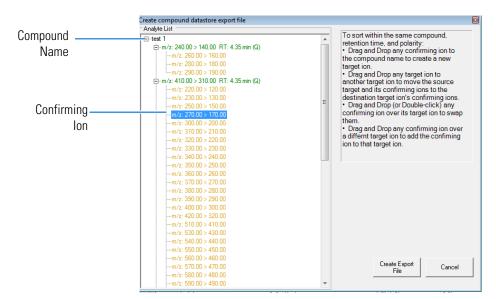


Figure 114. Importing Methods

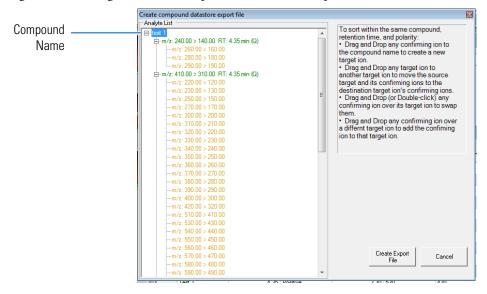
38. Choose **ISQ 7000 | Import Timed Scans** to import .csv or .xml files of previous methods. The software will only load files in valid formats. If your file is not valid you will receive an error message and will not be able to import the file into the Method Editor.

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

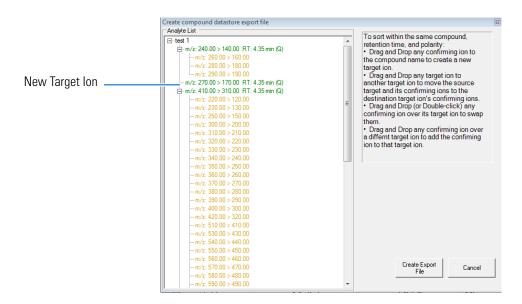
- 39. Choose **ISQ 7000** | **Append Timed Scan from File** to add scans from previous methods to the end of your open scan list. As above, you may import .csv or .xml files. The software will only load files in valid formats. If your file is not valid you will receive an error message and will not be able to import the file into the Method Editor.
- 40. Choose **ISQ 7000 | 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.
- 41. Choose **ISQ 7000 | Create Compound Data Store Export File** to prepare your file for the compound data store in the TraceFinder application.
  - a. To create a new target ion from the list of confirming ions with identical retention times for a compound, select the confirming ion of interest.



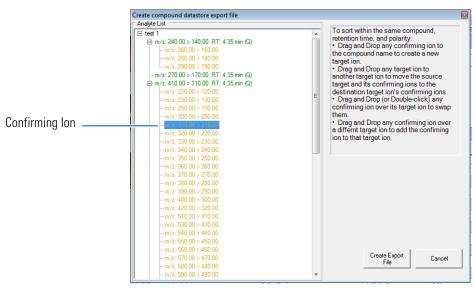
b. Drag the confirming ion to the compound name at the top of the list.



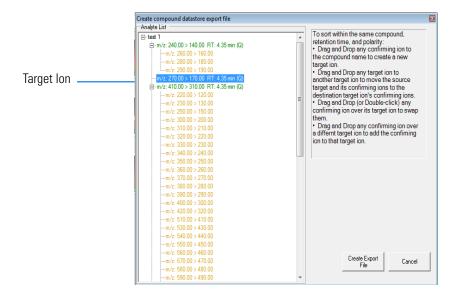
c. The confirming ion now appears in the list as a target ion.



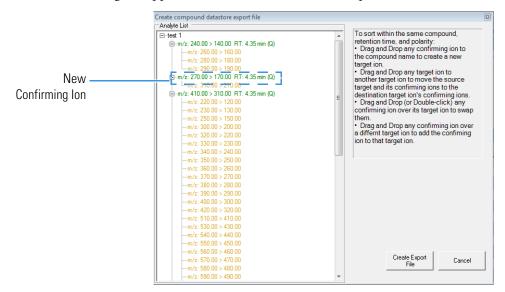
d. To add a new confirming ion to a target ion in the list, select the confirming ion of interest.



e. Drag the confirming ion under the target ion of interest.



f. The confirming ion appears in the list under the selected quantitation ion.



- g. Click **Create Export File** when you are through creating your ion list to export the list to the TraceFinder software compound database.
- 42. Choose ISQ 7000 | Create Segment List From Timed Scan List to import a general acquisition method.
- 43. Choose **ISQ 7000** | **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 75) and add identifying information to the tune report.

Report Options	×
Lab name:	ОК
Instrument name:	Cancel
Calc relative abundance using: 69	
Include signature line	
Optional reports	
Diagnostics Device settings Tune type	e settings
Custom report	

#### Figure 115. Configuring the Tune Report Options

44. Click **Next**. The I**nstrument Method Wizard – Completion** page opens. See Figure 116.

Figure 116. Instrument Method Wizard – Completion

have entered all data required to create an instrument method.		
Comment		
Description		
Network TS2-PC_1 Creation Time IS-2014 2:06:34 PM Created by TS0		
Press Findh to open the indument method in the Orionatography Studio.		

- 45. Enter any comments or notes for your method and click Finish.
- 46. The New Instrument Method (Instrument Method) Chromeleon Chromatography Studio window opens. Click the **Save** icon to save your method. See Figure 74.

#### Figure 117. Saving Methods

() () () () () () () () () () () () () (	the second s		New Ir	strument Method (Instrumer	nt Method) - Chromeleon Chromatography Studio		- 4
H Save Changes (Ctrl+S)							
A Cut Bill ISQ-PC1	d Time	ional - 🖷	New Command Rows Expand All Stages Collapse All Stages	h Previous 원, Next	UHPLC Speed-Up		
Clipboard Instrument	Insert	Row	View	Find	Method Transfer		
Instrument Method «	Name:	New Instrument Method					
🕧 Overview	ivane,	New Instrument Method					
Sampler	Comment:						
GC Inlets (TRACEL300Series)							
GC Oven Settings (TRACE1300Series)	Description:	Instrument: I SQ-PC_1	24.04				~
MSDevice (ISQ Series)		Creation Time: 8/5/2014 2:0 Created by ISQ	5.54 FM				
🔆 System							
Z Script Editor							
							-
	Run Time:	20.000	Q [0.00010000]	0.000 min]			
	4						
Instrument Method							

47. The **Save Instrument Method** dialog box opens. Save your method in **Chromeleon** Local > Instrument Method. See Figure 118.

Figure 118. Finding the Instrument Method Folder

Look in: 📴 ChromeleonLocal		- 🥥	- 🚱 - 🖄   📑 🔢
Name	Туре	Date Modified	Comment
Frankl Instrument Data Instrument Method Instrument Sequence	Folder Folder Folder Folder	5/8/2014 3:25 PM 7/17/2014 2:20 PM 8/3/2014 2:13 PM 8/3/2014 2:15 PM	
Object name:			
Object name: Channel:			

- 48. Enter your method in the **Object Name** box and click **Save**. Your method is now saved.
- 49. You can view all instrument methods on your system by opening the Chromeleon Console and selecting **Data** from the left menu. See Figure 119.

		«				Instrument Method
7	Filter	Y	Name •	Type	Date Modified	Comment
	Pica	Y	1fg IDL study on 15m column_F_			1fa IDL study
omeleonLocal				Instrument Method	6/18/2014 1:45 PM	2fg IDL study
frankl Instrument Data			2fg IDL study on 30m column	Instrument Method	6/13/2014 11:18 AM	2fg IDL study
Instrument Data Instrument Method				Instrument Method	6/10/2014 8:19 AM	David's 8270 DC selit adjustment 20 method
Instrument Sequence				Instrument Method	7/16/2014 10:13 AM	\$270 method
Deleted items			El full scan for OFN no solvent d_	Instrument Method	6/11/2014 12:18 PM	El full scan for OFN in iso octane (no solvent delay)
				Instrument Method	6/11/2014 12:13 PM	
				Instrument Method	6/19/2014 12:34 PM	fast method
			FS 1pg OFN SN tests for Duo	Instrument Method	8/3/2014 2:13 PM	FS S/N test for 1pg OFN on column
			Full Scan Speca 7-22-14	Instrument Method	7/22/2014 4:22 PM	Full Scan Specs
			Full Scan Specs Splitless 7-22-14	Instrument Method	7/22/2014 5:43 PM	FullScan Scan Solidless
				Instrument Method	7/22/2014 5:13 PM	Full Scen split 20:1
			method for effect of Ar gas press.	Instrument Method	6/26/2014 12:24 PM	method for effect of Ar gas pressure
			no solvent delay IDL testing	Instrument Method	6/13/2014 10:12 AM	no solvent delay 100fg run
			Performance 7-22-14	Instrument Method	7/22/2014 12:19 PM	Performance
			Performance A 7-22-14	Instrument Method	7/22/2014 12:40 PM	Performance
			SRM Specs Splitless 7-22-14	Instrument Method	8/5/2014 1:18 PM	FullScan Scan Splitless
			test method	Instrument Method	8/5/2014 2:12 PM	
				Instrument Method	7/22/2014 12:25 PM	
			TSQ 8000 suitability test method	Instrument Method	6/11/2014 11:17 AM	David's suitability test procedure
truments		_				

Figure 119. Viewing all Instrument Methods

50. To edit a method, select it from the list. The method opens in the Chromatography Studio. See Figure 120.

Figure 120. Editing a Method

	test method (instrument Method) - Chromeleon Chrometography Studio	
Home		
& Cut B ISQ-PC_1 ·	■ O <sup>10</sup> a Stage - P Delete New Command Rows	
	Conditional - It Expand All Stages A Previous A Next	
Comn	and Time Comment Collapse All Stages UHPLC Speed-Up	
lpboard Instrument	Inset Row View Find Method Transfer	
ument Method	K 📓 Instrument Method Wizard - MSDevice (ISQ Series): MS settings	
verview	MS settings for MSDevice (ISQ Series).	
mpler	ISQ series +	
C Inlets RACE1300Series)	Method Setup	
C Oven Settings RACE1300Series)	Method type: Acquisition - Timed V Use timed acquisition methods to acquire timed SIM data.	
tSDevice (SQ Series)	MS transfer line temp: 250 © C lonization mode: E ~	
ystem	Ion source temp: 300 🔅 "C Cligas type: Methane	
	Acquistion threshold: 1000 C Cl gas flow: 1.00 C mL/thin	
cript Editor	- 0	
	Scans	
	2↓-2↑- 約 × 匹 哈 圖 ② N 4 P N Scan type SIM - Link to external file	Scan settings
	Name RT Mass	Time summary
	•	Resulting total scan time: 0.300 sec
		SRM/SIM Time: 0.300 PPC
		Lowest dwell time; 0.001 sec
		Window optimization
		Vindow optimization
	1	
		Desired window 0.6 🐑 min
		Minimum window 0.3 🔄 min
		Peak width
		Min. baseline peak width: 3.0 \ominus sec
		Desired scans per peak: 10 +
		Acquistion options
		Acquisition options Allow for asymmetric acquisition windows
		Allow dwell time prioritization High priority multiplier: 10 0
		Fullscan
		Use full scan with mass range: 50-550
Instrument Method		Use full scan with mass range: 50-550

51. Select a device from the left menu and edit its parameters as described in this chapter.

# 5

## **Using AutoSIM**

This chapter will help you use the AutoSIM software utility to set up and run a SIM Ion Study. As well as instructions for setting up and running each study, this chapter gives you the steps for importing the resulting list of SIM ions into the ISQ 7000 method editor and accessing them for routine use.

**Note** Set up your GC and autosampler methods through the ISQ 7000 method editor before developing your AutoSIM method.

#### Contents

- Determining SIM Ions
- Importing Transitions to the Method Editor
- Determining SIM Ions in Chromeleon
- Importing Transitions to the Chromeleon Instrument Method Editor

## **Determining SIM Ions**

The purpose of an AutoSIM study is to select your SIM ions. After you name your compounds and enter your vial numbers and retention times, AutoSIM instructs your ISQ 7000 system to run a full-scan analysis on the compounds.

After the full-scan analysis is complete, AutoSIM presents you with the resulting chromatographic peaks and full-scan spectra, and then provides optional setting for sorting the results for your SIM ions.

**Note** You must have mid-range concentration standards (500 pg/ $\mu$ L-10 ng/ $\mu$ L) before setting up your AutoSIM method.

#### \* To determine your SIM ions in AutoSIM

1. Click the **AutoSIM button** on the ISQ 7000 Dashboard to open the AutoSIM utility. See Figure 121.

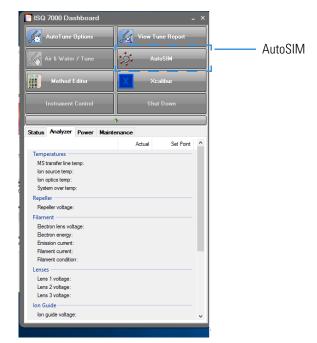


Figure 121. Accessing AutoSIM on the ISQ Dashboard

2. Click the Create a New Study icon on the left to create a new study.

Figure 122. New AutoSIM Study

	्रंः ISQ	AutoSIM						
	Cre	i i i i i i i i i i i i i i i i i i i	om existing study			Instru	ment Method:	
	Sta		min. iC Run Time	5.00 📩 min.				
		Status	Name	RT	Vial Position	Raw file		
	*							
Create a New Study Icon								

3. A new study window opens.

Note If at any time you see the error message below, go to "Upgrading the Software" on page 237 and follow the instructions for getting your software license.

Missing license

ISQ Auto SIM requires a license that is not currently on the instrument. Please purchase the ISQ Auto SIM license to enjoy all the capabilities of this application.

OK

4. Link to your saved instrument method file (that you created using the method editor) by clicking on the ellipsis icon next to the Instrument Method window. AutoSIM will use the GC and autosampler parameters from this method file. See Figure 123.

Figure 123. Retrieving an Instrument Method

rery study	▲ ♥
New folder	
Poods Poods Places File name: Xcalibur/methods/Testmethod.meth Date modified Type Size Size Control Size Size Control Size Control Size Control Size Control C	Import a Instrumen Method

- 5. Select an instrument method file and click Open.
- 6. You may set the Mass Range, Start Time, and Stop Time. See Figure 124.

Any changes you make to your MS method here will override the method editor settings.

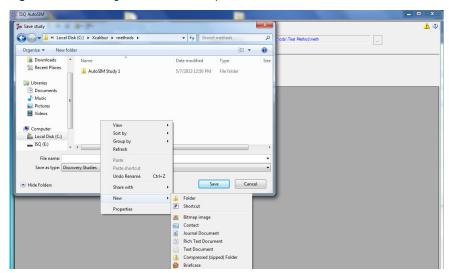
#### Figure 124. Adjusting the Settings

iột IS	Q AutoSIM						X
<del>1</del> .	😂 🖬 - 👗 👘 (	🏶   🖀 • 😭 •					ڭ 🚺
Stud	y name: •				Instrument Method: Xcalibur\method	ts\Test Method meth	
_							
	lass range: 50-550						
		🗧 min. iC Run Time 🗌	5.00 🚔 min.				
	Status	Name	RT	Vial Position	Raw file		
*							

- 7. Enter the compound name, approximate retention time, and vial number for each compound you wish to optimize. If you already have a method for processing full scan data you can choose to import compounds from an external file. Their names and retention times will fill the compound list and their primary quantitation ion will be displayed in the mass filter box once the full scan data is acquired.
- 8. Save the study.

**Tip** Create a folder for all files associated with your AutoSIM study. Otherwise, the study results files will be saved in the general instrument method folder and crowd it. See Figure 125.

Figure 125. Creating an AutoSIM Study Folder



- 9. Open the folder.
- 10. Give your study a file name.
- 11. Save your study in the Study folder. See Figure 126.

**Note** All files, including raw data files, that AutoSIM generates will be saved into the same folder that you save the study file. To simplify your workflow, create a folder for your study.

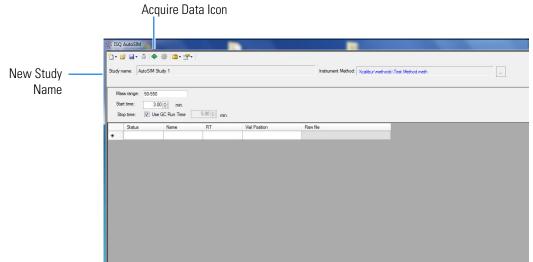
Figure 126. Saving an AutoSIM Study

袋 ISQ AutoSIM					_ 0
🔅 Save study			X		1
🚱 🔍 🗢 🕌 « Xcalibur 🕨 methods 🕨 AutoSIM Study 1	<b>√</b> 4 <sub>2</sub>	Search AutoSIM	Study 1 🔎	thods\Test Method.meth	
Organize 🔻 New folder			111 - (2)		
Downloads Recent Places Documents Music Pittures Videos Computer Local Disk (C:)	Date modif		Size		
- ISQ (E:)	m				
File name: AutoSIM Study 1 Save as type: Discovery Studies			•	1	
C Hide Folders	_	Save	Cancel	4	

12. The Windows Explorer window closes.

13. The AutoSIM Study Name is the name you assigned. See Figure 127.





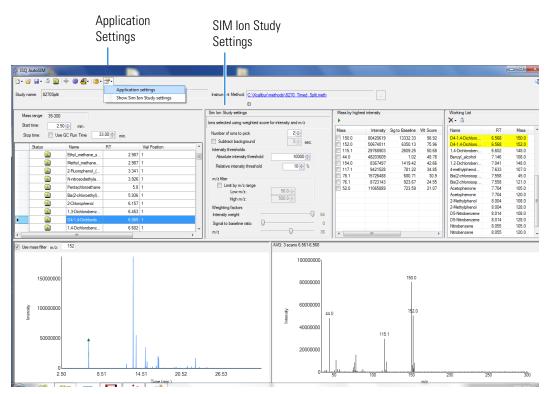
14. (CI Only) If you are running a chemical ionization (CI) study, select **Positive** or **Negative** from the **Ion Polarity** pull-down menu. See Figure 128.



ंः ISQ	AutoSIM										- 🗆 X
🔁 <b>-</b> 🖻	🖗 👗 • 🔜 🖗	🏶   🛅 <b>-</b> 🚰 -									1 🤣
Charles	ame: •					Instrument Method	: C:\Xcalibur\meth	ods\CI PBDE 154	SIM053012.meth		
Study II	ane.						CI, Methane, 2 m	L/min	Polarity:	Positive	
Mas	ss range: 50-550									Negative	
Sta	t time: 3.00	🔶 min.									
Sto		GC Run Time	5.00 🜩 min.								
	10 0	-		14.15							 
*	Status	Name	RT	Vial Position	Raw	file					
*					100						

15. To access the options for SIM ion settings, click the **Applications Settings** icon and select **SIM Ion Settings** to open the SIM Ion Settings box. See Figure 129.





16. By default, SIM ions are sorted by highest intensity. In the SIM Ion Study Settings box, you may select SIM ions according to the following criteria.

- a. Number of Ions to Pick: Selects the number of SIM ions picked for each compound.
- b. **Subtract Background**: Checking this box subtracts background from the spectrum. Subtracting the background may reduce baseline noise automatically away from the selected peak. This will help identify your target compounds, clarify intensities, and reduce column bleed. If the automatic background subtraction is not ideal (i.e., due to co-eluting peaks), you may select to manually subtract background for individual compounds by right clicking on the chromatogram and then highlighting the scan or scans to use for subtraction.
- c. Intensity Thresholds: Allows you to choose intensity levels.
  - i. **Absolute Intensity Threshold**: Sets the intensity range that all ions must fall into before being selected as SIM ion candidates. All ions must be greater than this intensity to be selected as SIM ion candidates.
  - ii. **Minimum Intensity Threshold**: Sets the minimum intensity for an ion to be a candidate for the SIM ion list. Ions must have a relative abundance greater than or equal to this percentage to be selected as SIM ion candidates.
- d. Limit by m/z Range: Check this box and set the low m/z and high m/z to limit your SIM ion selection list to certain masses within the set scan range.
- e. **Weighting Factors**: Use the sliding bars and check boxes to set the values you want to give each SIM ion study setting.
- 17. Click the Acquire Data icon to run your samples. See Figure 127.

**Note** AutoSIM calculates the number of injections needed based on the compound list and vial positions you assigned.

18. The Submit Study for Acquisition window opens. See Figure 130.

综 ISQ AutoSIM		- <b>X</b>
🖞 • 📸 🛃 • 🍈 🕸 🎯 🔛 • 🚰 •		1 2
Study name: AutoSIM Study 1	Instrument Method: Xcalbur/methods/Test Method meth	
Mass range: 50-550 Start time: 3.00 min. Stop time: V Use GC Run Time 5.00 min. Status Na Status Na Auto SRM Instrument methods C:\Xcalbur\methods\	est Method meth	
Acquisition Sequence	Data Path         RawfileName         Comments           very1         C:\Xcalibur\meth         AutoSRMStudy_AUTO_FS_1	
Compounds in sample: 3 Decafluorobenzophenone (50-550) RT: 3.6 Benzophenone (50-550) RT: 5.8 Methyl Stearate (50-550) RT: 8.3		Submt Oose
۵		

#### Figure 130. Submitting a Study for Acquisition

19. Click **Submit** to submit the samples to the instrument.

Once the samples have finished running, the software analyzes the data.

20. The results appear in the AutoSIM window. See Figure 131.

The results displayed correspond to the peak topped by the green triangle. The Mass by Highest Intensity pane contains a list the highest intensity ions at the indicated retention time.

**Note** Background subtraction updates the ions in the Mass by Highest Intensity pane.

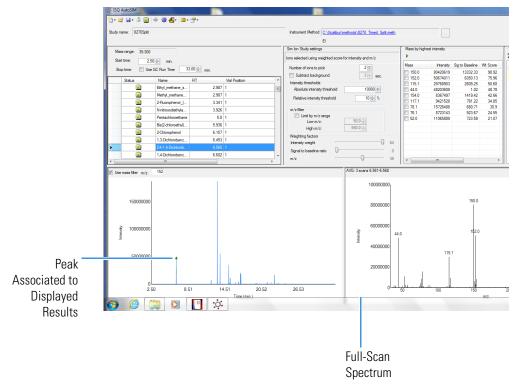
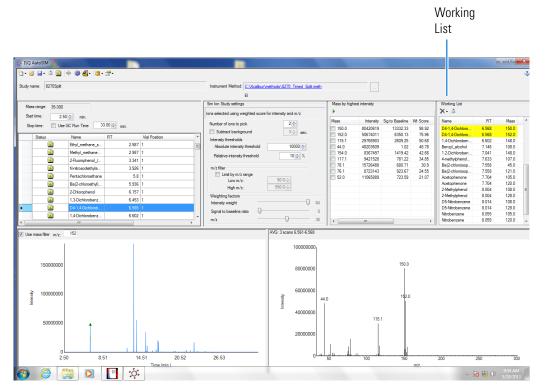


Figure 131. Study Results in AutoSIM

- 21. Select the check box next to the SIM ions you want to send to the working list.
- 22. Click the green arrow icon to push the SIM ions you selected to the working list. See Figure 132.

#### Figure 132. Selecting SIM lons

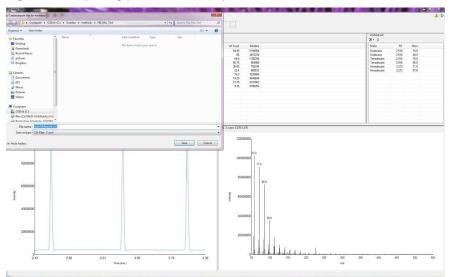


23. Repeat this process for all of your compounds.

**Note** You can select SIM ions by checking them in the mass list or send them directly to the working list by double-clicking on the ion in the spectra window.

24. Once you have selected all your SIM ions, go to **File | Save As** and export your SIM ion study as a .csv file. See Figure 133.

Figure 133. Exporting your SIM Ion Study



## **Importing Transitions to the Method Editor**

- \* To import the list of transitions you created in AutoSIM
- 1. Open the method editor on the ISQ Dashboard.
- 2. Open your method in Xcalibur. See Figure 134.

Figure 134. Opening Instrument Method in Xcalibur

ISQ Series H	
E CUba E CUba E CUba E CUba	Method Setup         Method Spec       Acquation - Timed       Use timed acculation methods to acquire timed SIM dats.         Wethod Spec       2010       C toxication methods to acquire timed SIM dats.         Wethod Spec       2010       C toxication methods to acquire timed SIM dats.         Wethod Spec       2010       C toxication methods to acquire timed SIM dats.         Wethod Spec       2010       C toxication methods to acquire timed SIM dats.         Wethod Spec       2010       C toxication methods to acquire timed SIM dats.         Wethod Spec       2010       C toxication methods to acquire timed SIM dats.         Wethod Spec       2010       C toxication time       2020         Wethod Spec       2010       C toxication time       2020         Wethod Spec       Explore the character specified context
	i View Tune Report

- 3. If you have the desired method open in AutoSIM, click the **Link to External File** in the ISQ 7000 method editor to open your method in the ISQ 7000 method editor
  - a. After clicking **Link to External File**, the **SIMBridge** dialog box opens. Choose the language of your method file from the **Source Locale** drop-down menu.

HINITIEd - Thermo Xcalibur Instrument Setup Filo: ISQ Series: Holp	
TSD Sees     Method Setup       150 Sees     Method Setup       Wethod Setup     Method Setup       Method Setup     Secarition - Timed       Use Sees     Secarition - Timed       Import map     Source temp:       2 Scarition     Source temp:       2 Scarition     Source temp:       2 Scarition     Mare of Be       Import map     Catamin Be       Import map     Catamin Be	2 RC RC RC RC
Results	8
	ly miljsler 10 -
Wew Tune Report	
Ready	NOT SAV

Figure 135. Setting the Source Language of Method Files using SIMBridge

b. Browse to your file.

Figure 136. Linking to an External File using SIM Bridge

Huntitled - Thermo Xcalibur Instrument Setup File ISQ Series Help	X
File Series         File Control         Image: Series          Image: Series         Image: Series         Image: Series         Image: Series         Image: Series         Image: Series         Image: Series         Image: Series         Image: Series         Image: Series	Image: Control of the second

- c. Click **Open** to open the method in SIMBridge.
- d. If necessary, change the method headings in your original file to match those in the method editor. A green check mark appears when your method is validated.

	N S							000000000000000000000000000000000000000				
in the l	Method Setup											
	Method type: Acquisition - Timed	🔻 Use ti	med acquisition	methods to ac	quire tim	ed SIM data.						
		ISQ Series - S	iIMBridge								×	1
Q Series	MS transfer line temp.: 250 🚊	1 📴 🖪 🖻	Source locale:	English (Unite	d State	;)	▼ Delimiter: ,		1			
	Ion source temp.: 200 🚊	_	C:\Users\Alvin				_ /		-			
		Name or me.	C. VOSEIS VAIVIN	vo esktop virgi	sri_oim,	_wearing_criticat	35_111002.03V					
•	C Acquisition threshold: 1000 🚊	Import map										
	Scans											
CE GC Ultra		Column in f	Name	Example 1								
		Start Time	Start time	3.2								l
	Name BT	Stop Time	End time	10.4								ec.
	*	Mass	Mass	54								
× 🚚		Mass	Mideo									ec.
.#: 100 LS												ec
sampler	57											
	STOCK											
		0 1 10	ompounds found								*	
		Hesuks - 15 c.			Mass	Den midth (min	Deat with fair	Start time	End time Vial Po	sition Dwell Time Priority	-	
		Name RT										
		Name RT	7.2	Positive	54	0	0	3.2	10.4	Normal		
		Name RT	7.2 1.8	Positive Positive	54 109	0	0	3.2 10.4	12.2	Normal Normal		
		Name RT 1 6.8 2 11.3 3 12.8	7.2	Positive	54	0 0 0	0	3.2		Normal		
		Name RT 1 6.8 2 11.3 3 12.8 4 14.1 5 15.1	7.2 1.8 1.2 1.4 0.6	Positive Positive Positive Positive	54 109 121 119 79	0 0 0 0 0	0 0 0 0 0	3.2 10.4 12.2 13.4 14.8	12.2 13.4 14.8 15.4	Normal Normal Normal Normal Normal		
		Name RT 1 6.8 2 11.3 3 128 4 14.1 5 15.1 6 15.6	7.2 1.8 1.2 1.4 0.6 0.4	Positive Positive Positive Positive Positive	54 109 121 119 79 79	0 0 0 0 0 0	0 0 0 0 0 0	3.2 10.4 12.2 13.4 14.8 15.4	12.2 13.4 14.8 15.4 15.8	Normal Normal Normal Normal Normal		
		Name RT 1 6.8 2 11.3 3 128 4 14.1 5 15.1 6 15.6 7 16.1	7.2 1.8 1.2 1.4 0.6 0.4 5 0.7	Positive Positive Positive Positive Positive Positive	54 109 121 119 79 79 79 105	0 0 0 0 0 0 0	0 0 0 0 0 0 0 0	3.2 10.4 12.2 13.4 14.8 15.4 15.8	12.2 13.4 14.8 15.4 15.8 16.5	Normal Normal Normal Normal Normal Normal		
		Name RT 1 6.8 2 11.3 3 128 4 14.1 5 15.1 6 15.6 7 16.1 8 17.2	7.2 1.8 1.2 1.4 0.6 0.4 5 0.7 5 1.5	Positive Positive Positive Positive Positive Positive Positive	54 109 121 119 79 79 105 105	0 0 0 0 0 0 0 0	0 0 0 0 0 0 0 0	3.2 10.4 12.2 13.4 14.8 15.4 15.8 16.5	12.2 13.4 14.8 15.4 15.8 16.5 18	Normal Normal Normal Normal Normal Normal Normal		ly multiplies: 183
		Name RT 1 6.8 2 11.3 3 128 4 14.1 5 15.1 6 156 7 16.1 8 17.2 9 18.5	7.2 1.8 1.2 1.4 0.6 0.4 5 0.7 5 1.5 1	Positive Positive Positive Positive Positive Positive Positive Positive Positive	54 109 121 119 79 79 105 105 79	0 0 0 0 0 0 0	0 0 0 0 0 0 0 0	3.2 10.4 12.2 13.4 14.8 15.4 15.8 16.5 18	12.2 13.4 14.8 15.4 15.8 16.5	Normal Normal Normal Normal Normal Normal		ly maticiae: 103
		Name RT 1 6.8 2 11.3 3 128 4 14.1 5 15.1 6 15.6 7 16.1 8 17.2	7.2 1.8 1.2 1.4 0.6 0.4 5 0.7 5 1.5 1 0.6	Positive Positive Positive Positive Positive Positive Positive	54 109 121 119 79 79 79 105 105 79	0 0 0 0 0 0 0 0 0 0 0 0	0 0 0 0 0 0 0 0 0 0 0	3.2 10.4 12.2 13.4 14.8 15.4 15.8 16.5	12.2 13.4 14.8 15.4 15.8 16.5 18 19	Normal Normal Normal Normal Normal Normal Normal Normal		
		Name RT 1 6.8 2 11.3 3 128 4 14.1 5 15.1 6 15.6 7 16.1 8 17.2 9 185 10 193	7.2 1.8 1.2 1.4 0.6 0.4 5 0.7 5 1.5 1 0.6 1.2 1.2 1.2 1.2 1.3 1.2 1.4 1.4 1.4 1.4 1.2 1.4 1.4 1.4 1.5 1.5 1.5 1.5 1.5 1.5 1.5 1.5	Positive Positive Positive Positive Positive Positive Positive Positive Positive	54 109 121 119 79 79 79 105 105 79 84	0 0 0 0 0 0 0 0 0 0 0 0	0 0 0 0 0 0 0 0 0 0 0 0	3.2 10.4 12.2 13.4 14.8 15.4 15.8 16.5 18 19	12.2 13.4 14.8 15.4 15.8 16.5 18 19 19.6	Normal Normal Normal Normal Normal Normal Normal Normal Normal		

Figure 137. Changing Method Headings in SIMBridge

- e. Click **Open** and the external method will be opened in the method editor.
- 4. Click the ISQ 7000 icon in the method editor side pane.
- 5. Select Acquisition-Timed from the Method Type drop-down menu.
- 6. From the top menu, select ISQ 7000 | Import Timed Scans. See Figure 138.

Figure 138. Selecting Acquisition-Timed Method

11 Untit	led - Thermo	Xcalibur Instrument S	setup						
File IS	Q Series He	lp							
	Show Gro	up Settings							
	Import tin	ned scans							
	Append ti	med scan from file							
	Export tim	ied scans		<ul> <li>Use timed act</li> </ul>	quisition methods to a	cquire timed SIM data.			
		mpound data store ອ	mort file						
*ISG		ment list from timed		Ionization mode	e: Cl 👻				
	Options	, nene not normanica	Jeannise	CI gas type:	Methane	•			
				CI gas flow:	1.00 ÷ mL/min				
	View Tune			Ci gas now.					
		Scans							
TRACE	GC Ultra	Instrument settings						Scan settings	
		Tune file		12	Emission curre			Peak width	Resulting total scan time:
1		Tune file (+): Auto				emission current		Min. baseline peak width: 3.0 + sec	SIM Time:
	K,	Tune File (-): Auto	tune_NCI		Use specifie	d emission current:	50 🖃 (JµA)	Desired scans per peak: 6 +	Min dwell time:
		Detector gain						Full scan	Acquisition window option
TriPlus Autosan	moler	Use last tuned	-					Use full scan with mass range: 50-550	
Autosai	inpres 1	C Use specified of	detector gain:	0				Full scan time: 0.043 ÷ sec Positive v	Allow for asymmetric acqu
	201000							Start time: 2.00 - min. End time: 10.00 - min.	
	100 March								
	100000	Link to external f	file						
	00000	Name	RT	Polarity	Window (min)	Mass			
		*			•		1		
	10100	10							
	20202								
	10100								
	20202								
	Successive Sector								
	2000								
	100020								
	20202								
	20202								
	10000								

7. Browse to the location where you saved the .csv file you created in AutoSIM. The software informs you if your .csv file is valid. See Figure 139.

Untitled - Thermo X D 🗃 🖬 🏼 🗶 😵 Method Setup Use timed acquisition methods to acquire timed SIM data Method type: Acc uisition - Timed MS transfer line temp.: 250 🛨 °C Ionization mode: CI 💌 200 🛨 °C Cligas type: Methani Ion source temp Select the file containing the timed data W 00 Scans Look in: 🔒 AutoSIM Study 1 • + 🖻 💣 🖬 • Date modified Sent Plac Tune file AutoSIM Study 1.csv 5/21/2013 10:01 AM Microsoft Office E... 1 KB ¥ Tune File (-): Desktop File appears to be valid Libraries Use spec Ċ Link to e AutoSIM Study 1 -Open Ele name CSV files (\*.csv) •

Figure 139. Linking to an External .csv File

- 8. Click Show Analysis and review the imported list of compounds.
- 9. Adjust your scan settings as necessary. See "Creating a Method" on page 59 for more information.
- 10. Once you are satisfied with your method, save it.
- 11. Run a set of samples to verify that the method meets your needs.

### **Determining SIM Ions in Chromeleon**

The purpose of an AutoSIM study is to select your SIM ions. After you name your compounds and enter your vial numbers and retention times, AutoSIM instructs your ISQ 7000 system to run a full-scan analysis on the compounds.

After the full-scan analysis is complete, AutoSIM presents you with the resulting chromatographic peaks and full-scan spectra, and then provides optional setting for sorting the results for your SIM ions.

**Note** You must have mid-range concentration standards (500 pg/ $\mu$ L-10 ng/ $\mu$ L) before setting up your AutoSIM method.

#### To determine your SIM ions in AutoSIM

 Click the AutoSIM button on the ISQ Dashboard to open the AutoSIM utility. See Figure 121.

ISQ Dashboard				0
10	Auto T	une		
V V	iew Tune	Report		
J.	Tune T	ypes		
<u>.</u> o.	Autos	SIM		
A A	r & Wate	r / Tune		
ŀ	nstrument	: Control		
	Shut D	lown		
		1		
atus Analyzer Power	Maintena			
tatus Analyzer Power Status: Ready	Maintena	ince		
	Maintena	ince	t-Point	
	Maintena for rur	ince 1 Se	t-Point 250	'n
Status: Ready	Maintena for rur Actual	nce 1 Se °C		
Status: Ready	Maintena for rur Actual 250	nce 1 Se °C	250	
Status: Ready MS transfer line temp.: In source temp.:	Maintena for rur Actual 250 200 OK	nce 1 Se °C	250	
Status: Ready MS transfer line temp.: I on source temp.: Vacuum:	Maintena for rur Actual 250 200 OK	nce 1 Se °C °C	250 200	°C
Status: Ready MS transfer line temp.: Store temp.: Yacuum: Foreline pressure:	Maintena for rur Actual 250 200 OK	nnce I "C "C "C "C "C "C "C "C "C "C "C "C	250 200	°C

Figure 140. Accessing AutoSIM on the ISQ Dashboard

**Note** If User Management is enabled in Chromeleon, the Chromeleon log on dialog box opens when you start AutoSIM. Enter your Chromeleon User Name and Password to continue.

2. Click the Create a New Study icon on the left to create a new study.

Figure 141. New AutoSIM Study

	¢ ISQ	AutoSIM						
	Cre	i i i i i i i i i i i i i i i i i i i	om existing study				Instrument Method:	
	Star	ss range: 50-550 t time: 3.00 up time: 📝 Use G		5.00 📩 min.				
Create a New Study Icon	*	Status	Name	RT	Val Postion	Raw file	B	

3. A new study window opens.

 Note
 If at any time you see the error message below, go to "Upgrading the Software" on page 237 and follow the instructions for getting your software license.

 Missing license
 X

ISQ Auto SIM requires a license that is not currently on the instrument. Please purchase the ISQ Auto SIM license to enjoy all the capabilities of this application.

4. Link to your saved instrument method file (that you created using the Chromeleon Instrument Method Editor) by clicking on the ellipsis icon next to the Instrument Method window. See Figure 123.

Figure 142. Retrieving an Instrument Method

옷 ISQ Series AutoSIM ] • 글 글 · 글   中 ②   @ • 말 • ] 3udy name:   •	hatument Method	2
Maan mage 50 550 Battime: 300 2 mm. Sop tree: 2/ Use GC Run Time 500 2 mm.	Look IN: Prometeorlacad	Import an Instrument Method
	Object name: Banclast JuliSon   Carvet  Carvet  Copied type:  Tribunet Method  Copien  Concel  Concel	

- 5. If you are importing a file created on another system, click the import icon. Otherwise, select an instrument method file and click Open.
- 6. The **SIMBridge** dialog box opens. Choose the language of your method file from the **Source Locale** drop-down menu. See Figure 143.

OK.

Source locale:	English (United States)	Delimiter: 🚬 👻
- us, us	English (United States)	
Name of file:	English (Zimbabwe)	
1	Estonian (Estonia)	
	Faroese (Faroe Islands)	
mport map	Filipino (Philippines)	
Column in file Map to	Finnish (Finland)	
Column in the Map to	French (Belgium)	
	French (Canada)	
	French (France)	
	French (Luxembourg)	
	French (Monaco)	
	French (Switzerland)	
	Frisian (Netherlands)	
	Galician (Galician)	
	Georgian (Georgia)	
	German (Austria)	
	German (Germany)	
	German (Liechtenstein)	
	German (Luxembourg)	
	German (Switzerland)	
	Greek (Greece)	
	Greenlandic (Greenland)	
	Gujarati (India)	
Results	Hausa (Latin, Nigeria) Hebrew (Israel)	
	Hindi (India)	
	Hungarian (Hungary)	
	Icelandic (Iceland)	
	Igbo (Nigeria)	
	Indonesian (Indonesia)	

Figure 143. Setting the Source Language of Method Files using SIMBridge

7. Click the File icon. The **Select the File Containing the Scans** dialog box opens. Browse to your file. See Figure 144.

Figure 144. Linking to an External File using SIM Bridge



- 8. Click **Open** to open the method in SIMBridge.
- 9. If necessary, change the method headings in your original file to match those in the method editor. A green check mark appears when your method is validated.
- 10. Your method opens in AutoSIM. AutoSIM uses the GC and autosampler parameters from this method file. You may set the Mass Range, Start Time, and Stop Time. See Figure 124.

Any changes you make to your MS method here will override the method editor settings.

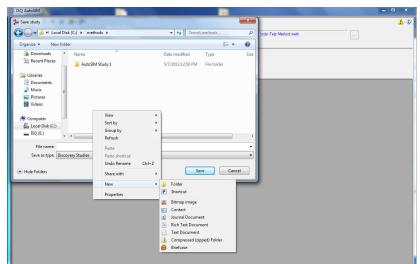
#### Figure 145. Adjusting the Settings

1. 100	Series A							
								- 8 🐱
11- 2	<b>9</b> 🚽 -	· ( 다 영	E 🗎 = 🚰 =					3
Study n	ame:					Instrument Method: chrom://ms-nc/Q	nomeleonLocal/BasicLiguid_FullScan.instmeth	
, -								
Ma	ss range:	50-550						
Sta	rt time:	3.00	min.					
Sto	p time:	Use GC	Run Time	5.00 ≑ min.				
	Status		Name	RT	Vial Position	Raw file		
•	_							
(A)	o istico e							

- 11. Enter the compound name, approximate retention time, and vial number for each compound you wish to optimize. If you already have a method for processing full scan data you can choose to import compounds from an external file. Their names and retention times will fill the compound list and their primary quantitation ion will be displayed in the mass filter box once the full scan data is acquired.
- 12. Save the study.

**Tip** Create a folder for all files associated with your AutoSIM study. Otherwise, the study results files will be saved in the general instrument method folder and crowd it. See Figure 125.

Figure 146. Creating an AutoSIM Study Folder



- 13. Open the folder.
- 14. Give your study a file name.
- 15. Save your study in the Study folder. See Figure 126.

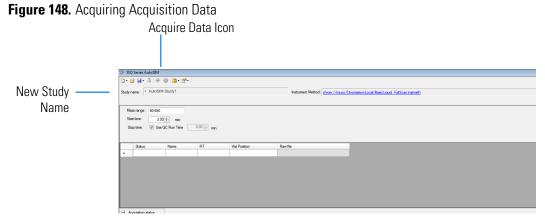
**Note** All files, including raw data files, that AutoSIM generates will be saved into the same folder that you save the study file. To simplify your workflow, create a folder for your study.

Figure 147. Saving an AutoSIM Study

1SQ AutoSIM					-	□ X
the Save study			x			1 2
😋 🕒 🗢 🕌 « methods 🕨 AutoSIM Study 1	+ + Search A	itoSIM Study 1	P	Inode\Test Method.meth		
Organize 👻 New folder		85 -	0			
Downloads ^ Name ^	Date modified	Туре	Size			
📃 Recent Places	No items match your search.					
📜 Libraries						
Documents						
Music						
Videos						
1 Computer						
= ISQ (E)						
File name: AutoSIM Study 1			•			
Save as type: Discovery Studies			-			
Hide Folders	Save	Cancel				
		_		/		

16. The Windows Explorer window closes.

17. The AutoSIM Study Name is the name you assigned. See Figure 127.

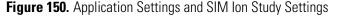


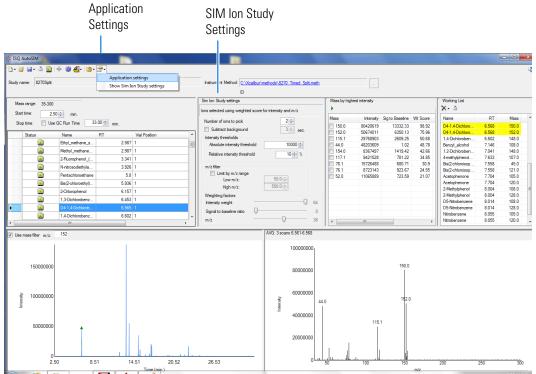
18. (CI Only) If you are running a chemical ionization (CI) study, select **Positive** or **Negative** from the **Ion Polarity** pull-down menu. See Figure 128.

ișt ISC	Q AutoSIM									_ 🗆 X
<b>℃</b> -	🚔 🖬 - 🎽 া 🏟	🏶   🖀 • 🕋 •								ې 🚹
Study	name: •				Instrument Method	C:\Xcalbur\meth	ods\CI PBDE 154SIN	1053012.meth		
,						CI, Methane, 2 m	L/min	Polarity:	Positive -	
	TATA								Positive Negative	
	ass range: 50-550									
		0≑ min.								
S	top time: 📝 Use	GC Run Time 5.	00 🔃 min.							
_	Status	Name	RT	Vial Position	Raw file					
*										

#### Figure 149. Setting Ion Polarity in a Cl Method

19. To access the options for SIM ion settings, click the **Applications Settings** icon and select **SIM Ion Settings** to open the SIM Ion Settings box. See Figure 129.





20. By default, SIM ions are sorted by highest intensity. In the SIM Ion Study Settings box, you may select SIM ions according to the following criteria.

- a. **Number of Ions to Pick**: Selects the number of SIM ions picked for each compound.
- b. **Subtract Background**: Checking this box subtracts background from the spectrum. Subtracting the background may reduce baseline noise automatically away from the selected peak. This will help identify your target compounds, clarify intensities, and reduce column bleed. If the automatic background subtraction is not ideal (i.e., due to co-eluting peaks), you may select to manually subtract background for individual compounds by right clicking on the chromatogram and then highlighting the scan or scans to use for subtraction.
- c. Intensity Thresholds: Allows you to choose intensity levels.
  - i. **Absolute Intensity Threshold**: Sets the intensity range that all ions must fall into before being selected as SIM ion candidates. All ions must be greater than this intensity to be selected as SIM ion candidates.
  - ii. **Minimum Intensity Threshold**: Sets the minimum intensity for an ion to be a candidate for the SIM ion list. Ions must have a relative abundance greater than or equal to this percentage to be selected as SIM ion candidates.
- d. Limit by m/z Range: Check this box and set the low m/z and high m/z to limit your SIM ion selection list to certain masses within the set scan range.
- e. **Weighting Factors**: Use the sliding bars and check boxes to set the values you want to give each SIM ion study setting.
- 21. Click the Acquire Data icon to run your samples. See Figure 127.

**Note** AutoSIM calculates the number of injections needed based on the compound list and vial positions you assigned.

22. The Submit Study for Acquisition window opens. See Figure 130.

🖗 ISQ AutoSIM		X
🖞 • 🗃 🖬 • 🎽   🏶 🏶   🖀 • 🖀 •		ب 🔬
Study name: AutoSIM Study 1	Instrument Method: chrom://ms-pc/ChromeleonLocal/BasicLiquid	d FullScan.instmeth
Mass range:     50-550       Sat time:     3.00 ⊕       Stop time:     IV los GC Run Time       Status     NN       Status     NN       AutoSIM Instrument method;     C. Zaromethouset	nLocal/Basciloud FullScan instructh	
Acquisition Sequence Acquire Vial Position Sa	eName Data Path RawfileName Comments	s
Compounds in sample: 3		
Decafluorobenzapierone (50-550) RT: 3.6 Benzapierone (50-550) RT: 3.8 Methyl Stearate (50-550) RT: 8.3		Submit Close
a		

Figure 151. Submitting a Study for Acquisition

23. Click **Submit** to submit the samples to the instrument.

The AutoSIM study is added to the Chromeleon sequence queue. Chromeleon performs a Ready Check to verify that the instrument is ready for operation and the instrument method is error-free. If the Ready Check passes, the run starts.

**Note** If the run does not start automatically after you click Submit, review the Chromeleon Ready Check messages listed in the Acquisition summary pane on the Submit dialog box. Close the Submit dialog box, correct the errors, and then resubmit the study.

If the following Ready Check message appears: "The instrument is currently in 'Hold' condition," follow these steps to release the Hold:

- On the Acquisition Status pane (described below), click the Command button.
- On the Command window, click the System icon. In the Properties list, select HoldMode Off and press ENTER.
- 24. The Acquisition Status pane displays status information about the running instrument. If the pane is not currently visible, click the **Acquisition Status** button. See Figure 152.

**Figure 152.** Displaying the Acquisition Status Pane

	•	
		Acquisition status
6		

Acquisition Status Button

The pane contains a Chromeleon ePanel Set that you can use to monitor the status of AutoSIM injections. See Figure 153. You can also use the ePanel Set to monitor the devices configured in the instrument and to see the status of the Chromeleon sequence queue. For details about Chromeleon ePanel Sets, refer to the Chromeleon Help.

Figure 153. Viewing Instrument Status on the Home ePanel

🖞 • 🗃 🖬 • 🎽 🕪	@   <b>@ • 😤 •</b>				٩
Study name: • AutoSIN	1 Study1		Instrument Method:	chrom://ms.pc/ChromeleonLocal/BasicLiquid_FullScan.instmeth	
Mass range: 50-550					
Start time: 3.0	min.				
Stop time: 📝 Use	GC Run Time 5.00 🖈 min	L.			
Status	Name RT	Vial Position	Raw file		
•					
	1				
Acquisition status					
Release Control					
Thermo Scientific	GC Home Themo TriPlus 10	0 LS Leftiniet Oven	Column(s) ISQ Etter	Audit Queue	
Injection details					*
			Retention time:		
Data vault:	chrom://ms-pc/ChromeleonLoca	al/Seq1-Basic.seq/343.smp	4.936 [min]		
Sequence:	Seq1-Basic				
Current injection:	02212015-5 Unknown	Injection No.: 14	Injection end:		
Type:	Chicrown	injection no 14	20.000 (min)		-
Connect	GC Conditions				
Cornect	Oven temperature:	157 [°C]			
	LeftColumn flow:	1.5 [ml/min]			
	Leftiniet temperature:	220 [°C]			
	RightColumn flow:	0.0 [ml/min]			
Not Ready					
Values					-

- 25. For an overview of the status of the run, click the **Home** tab. To monitor or control a configured device (mass spectrometer, detector, oven, etc.) click the tab for the device.
- 26. To view the queue status or to stop or start the queue, click the **Queue** tab. See Figure 154.

Figure 154. Viewing Queue Status on the Queue ePanel

Thermo Scientific GC Home Thermo	o TriPlus 100 LS Leftinitet Oven Column(s) I	ISQ Efter Audt Queue		
ent <u>R</u> ecent				
	Name	Status	Start After	Add
/ChromeleonLocal/Seg1-Basic		Running		Remov
				Move L
r running the queue keep instrument read	ly 👻			Move Do
sport Setup				
sport Setup Print report Report template undefine	ed.		Print Setup •	
Print report Report template undefine				Start
			Print Setup ·	
Print report Report template undefine				Start

Once the samples have finished running, the software analyzes the data.

27. The results appear in the AutoSIM window. See Figure 131.

The results displayed correspond to the peak topped by the green triangle. The Mass by Highest Intensity pane contains a list the highest intensity ions at the indicated retention time.

**Note** Background subtraction updates the ions in the Mass by Highest Intensity pane.

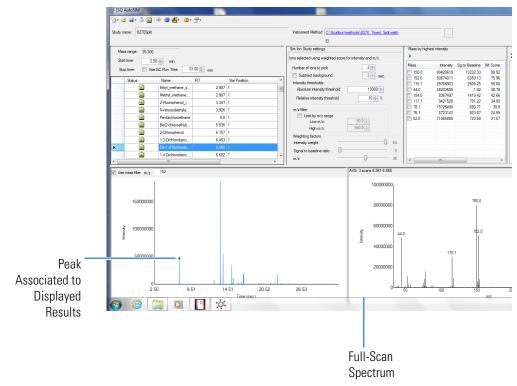
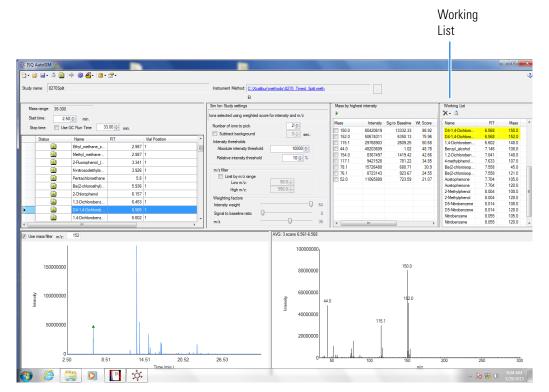


Figure 155. Study Results in AutoSIM

- 28. Select the check box next to the SIM ions you want to send to the working list.
- 29. Click the green arrow icon to push the SIM ions you selected to the working list. See Figure 132.

#### Figure 156. Selecting SIM lons

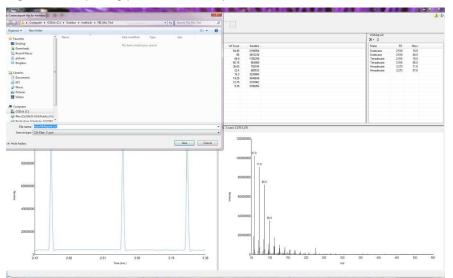


30. Repeat this process for all of your compounds.

**Note** You can select SIM ions by checking them in the mass list or send them directly to the working list by double-clicking on the ion in the spectra window.

31. Once you have selected all your SIM ions, go to **File | Save As** and export your SIM ion study as a .csv file. See Figure 133.

Figure 157. Exporting your SIM Ion Study



## **Importing Transitions to the Chromeleon Instrument Method Editor**

#### \* To import the list of transitions you created in AutoSIM

1. Open the Chromeleon Instrument Method Editor.

Tip Click the link in the Instrument Method box in AutoSIM to open Chromeleon.

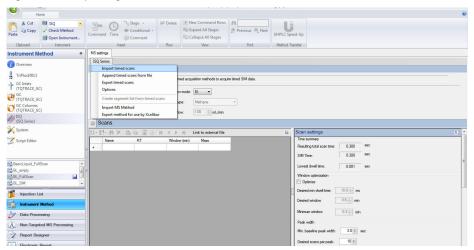
2. Select the **Acquisition – Timed** method type.

Figure 158. Opening Instrument Method in Xcalibur

n n n -	Seq1-Basic (Sequence) - Chromeleon Chromatography Studio
Home Home	
A Cut Paste Copy Cipboard Istrument	Image: State of the state o
Instrument Method «	MS settings
0 Overview           Implust00LS           GC Inites (TGTRACE_GC)           Implust00LS           <	ISQ Series         Method Setup         Method Setup         Method type:         Acquistion - Timed         Method type:         Acquistion - General         Matchourse         Matchourse         Image: Scans         Image: Scans      <
BasicLiquid_FullScan BasicLiquid_FullScan BL_entOV BL_FullScan Bectron Ltat Informant Method Mon-Tarpted MS Processing Non-Tarpted MS Processing Data P	SIM Time: 0.300 #Ac: Lowest dwell time; 0.001 sec Window optimization Desired min dwell time; 0.001 min Desired min dwell time; 0.001 min Desired window 0.61 min Minimum window 0.3 min Peak widh Min. baseline peak width; 0.30 min Peak width Min. baseline peak width; 0.30 min Peak width
	View Tune Report

3. In the MS Settings pane, select ISQ 7000 > Import Timed Scans.

Figure 159. Importing Timed Scans



4. The **SIMBridge** dialog box opens. Choose the language of your method file from the **Source Locale** drop-down menu.

🔊 🕫 🖬 📼	Seq1-Basic (Sequence) - Chromeleon Chrometography Studio	- 0
Home		
	Image: Command Time <sup>®</sup> Sage - <sup>®</sup> Conditional - <sup>®</sup> Conditional - <sup>®</sup> Conditional - <sup>®</sup> Conditional - <sup>®</sup> Collapse All Sages <sup>®</sup> Collaps	
Coveriew  TriPlud0015  Collets  Collet	Big State       Image of the i	3
	Cancel Ck	M

**Figure 160.** Setting the Source Language of Method Files using SIMBridge

5. Browse to your file.

Figure 161. Linking to an External File using SIM Bridge

Select the file containing the				
Coor Librari	ies	Documents	<ul> <li>✓ ✓</li> <li>Search Documents</li> </ul>	
Organize 🔻 New fo	olde	r	≣ - □ 0	
쑦 Favorites 📃 📃 🗮	<b>•</b>	Documents library	Arrange by: Folder 🔻	
🗼 Downloads		Name	Date modified Type Size	
Recent Places		🚡 ThermoISO	2/21/2015 12:13 PM File folder	
🥃 Libraries	E	ThermoTSQ8	1/20/2015 2:19 PM File folder	
Documents		TriPlus100LS	2/20/2015 4:50 PM File folder	
Music		🐌 TriPlusRSH	2/23/2015 1:30 PM File folder	
Pictures		🛗 Untitled 1.csv	2/23/2015 3:24 PM OpenOffice.org X	
Videos				sec
P Computer		4		sec
👝 ISQ (F:)	Ŧ			
File	le na	ime: Untitled 1.csv		
			Open 🔽 Cancel	
	-			

- 6. Click **Open** to open the method in SIMBridge.
- 7. If necessary, change the method headings in your original file to match those in the method editor. A green check mark appears when your method is validated.

nport ma	IP.										
Colum	n in file	Map to	Example								
Name		Name	1								
Start Ti	me	Start time	3.2								
Stop Ti	me	End time	10.4								
Mass		Mass	54								
and to a	15.0000	and fand									
		ounds found.									
lame	RT (		Ion Polarity	Mass	Pre-width (min)	Post-width (min)			Vial Position	Dwell Time Priority	
Name 1	RT	Window (min) 7.2	Positive	54	Pre-width (min)	Post-width (min)	3.2	10.4	Vial Position	Normal	
lame 1 2	RT ' 6.8 11.3	Window (min) 7.2 1.8	Positive Positive	54 109	0	0 0	3.2 10.4	10.4 12.2	Vial Position	Normal Normal	
lame 1 2 3	RT 1 6.8 11.3 12.8	Window (min)   7.2 1.8 1.2	Positive Positive Positive	54 109 121	0 0 0	0 0 0	3.2 10.4 12.2	10.4 12.2 13.4	Vial Position	Normal Normal Normal	
lame 1 2 3 4	RT 1 6.8 11.3 12.8 14.1	Window (min) 7.2 1.8 1.2 1.4	Positive Positive Positive Positive	54 109 121 119	0 0 0 0	0 0 0 0	3.2 10.4 12.2 13.4	10.4 12.2 13.4 14.8	Vial Position	Normal Normal	
lame 1 2 3 4 5	RT 1 6.8 11.3 12.8 14.1 15.1	Window (min) 7.2 1.8 1.2 1.4 0.6	Positive Positive Positive Positive Positive	54 109 121 119 79	0 0 0 0 0	0 0 0 0 0	3.2 10.4 12.2 13.4 14.8	10.4 12.2 13.4 14.8 15.4	Vial Position	Normal Normal Normal Normal	
lame 1 2 3 4 5 6	RT 1 6.8 11.3 12.8 14.1 15.1 15.6	Window (min) 7.2 1.8 1.2 1.4 0.6 0.4	Positive Positive Positive Positive Positive Positive	54 109 121 119 79 79	0 0 0 0 0 0	0 0 0 0	3.2 10.4 12.2 13.4 14.8 15.4	10.4 12.2 13.4 14.8 15.4 15.8	Vial Position	Normal Normal Normal Normal	
lame 1 2 3 4 5 6 7	RT 1 6.8 11.3 12.8 14.1 15.1 15.6 16.15	Window (min) 7.2 1.8 1.2 1.4 0.6	Positive Positive Positive Positive Positive	54 109 121 119 79	0 0 0 0 0	0 0 0 0 0	3.2 10.4 12.2 13.4 14.8	10.4 12.2 13.4 14.8 15.4	Vial Position	Normal Normal Normal Normal	
lame 1 2 3 4 5 6 7 8	RT 1 6.8 11.3 12.8 14.1 15.1 15.6 16.15	Window (min) 7.2 1.8 1.2 1.4 0.6 0.4	Positive Positive Positive Positive Positive Positive	54 109 121 119 79 79	0 0 0 0 0 0	0 0 0 0 0 0	3.2 10.4 12.2 13.4 14.8 15.4 15.8 16.5	10.4 12.2 13.4 14.8 15.4 15.8 16.5 18	Vial Position	Normal Normal Normal Normal Normal	
lame 1 2 3 4 5 6 7 8 9	RT 1 6.8 11.3 12.8 14.1 15.1 15.6 16.15 17.25	Window (min) [ 7.2 1.8 1.2 1.4 0.6 0.4 0.7	Positive Positive Positive Positive Positive Positive Positive	54 109 121 119 79 79 79 105	0 0 0 0 0 0 0 0	0 0 0 0 0 0 0 0	3.2 10.4 12.2 13.4 14.8 15.4 15.8	10.4 12.2 13.4 14.8 15.4 15.8 16.5 18 19	Vial Position	Normal Normal Normal Normal Normal Normal	
lame 1 2 3 4 5 6 7 8 9	RT 1 6.8 11.3 12.8 14.1 15.1 15.6 16.15 17.25 18.5	Window (min)   7.2 1.8 1.2 1.4 0.6 0.4 0.7 1.5	Positive Positive Positive Positive Positive Positive Positive	54 109 121 119 79 79 105 105	0 0 0 0 0 0 0 0 0	0 0 0 0 0 0 0 0 0	3.2 10.4 12.2 13.4 14.8 15.4 15.8 16.5	10.4 12.2 13.4 14.8 15.4 15.8 16.5 18	Vial Position	Normal Normal Normal Normal Normal Normal Normal	
lame 1 2 3 4 5 6 7 8 9 10 11	RT	Window (min) 7.2 7.2 1.8 1.2 1.4 0.6 0.4 0.7 1.5 1 0.6 0.6 1.2	Positive Positive Positive Positive Positive Positive Positive Positive Positive Positive	54 109 121 119 79 79 79 105 105 79 84 129	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	3.2 10.4 12.2 13.4 14.8 15.4 15.8 16.5 18 19 19.6	10.4 12.2 13.4 14.8 15.4 15.8 16.5 18 19 19.6 20.8	Vial Position	Normal Normal Normal Normal Normal Normal Normal Normal	
Name 1 2 3 4 5 6 7 8 9 10 11	RT	Window (min) [ 7.2 1.8 1.2 1.4 0.6 0.4 0.7 1.5 1 0.6	Positive Positive Positive Positive Positive Positive Positive Positive Positive	54 109 121 119 79 79 105 105 79 84	0 0 0 0 0 0 0 0 0 0 0	0 0 0 0 0 0 0 0 0 0 0 0	3.2 10.4 12.2 13.4 14.8 15.4 15.8 16.5 18 19	10.4 12.2 13.4 14.8 15.4 15.8 16.5 18 19 19.6	Vial Position	Normal Normal Normal Normal Normal Normal Normal Normal Normal	

#### Figure 162. Changing Method Headings in SIMBridge

- 8. Click **Open** and the external method will be opened in the Chromeleon Instrument Method editor.
- 9. Click the ISQ 7000 icon in the method editor side pane.
- 10. Click **Show Analysis** and review the imported list of compounds.

Figure 163. Selecting Show Analysis

				Sho	w Ar	nalysis —	
	Te	est (Sequence) - Chron	meleon Chromato	ography Studio			. • 🗾
00> 🔻	Command Time	<ul> <li>Stage →</li> <li>Conditional →</li> <li>Comment</li> </ul>	<b>∄</b> Delete	New Command Rows	Find	U Method Transfe	r
nent	Ins	sert	Row	View	Ŧ	÷	
	Link to exte	emal file					5
RT RT	Ion Polarity	Window (min)	Pre-width	(min) Post-width (min)		Mass	Product M
m	3.60 Positive	vvindow (min)	0.3	0		167	117
	3.60 Positive	•	0.3	0	0	195	117
	3.60 Positive	-	0.3	0	0	195	167
	E 00 D		0.0	n	0	105.1	50.0

- 11. Adjust your scan settings as necessary. See "Creating a Method" on page 59 for more information.
- 12. Once you are satisfied with your method, save it.
- 13. Run a set of samples to verify that the method meets your needs.

## **Running a Sample**

This chapter describes how to prepare a sample and then run a sequence.

#### **Contents**

- Preparing Your Sample
- Running a Sequence

### **Preparing Your Sample**

The primary goal of sample preparation is to reduce the amount of unwanted contaminant in a sample or to increase the reliability of sample detection. As a result, you should prepare your samples to increase the relative abundance of compounds you want to analyze and decrease the relative abundance of the compounds you aren't interested in. Refer to your lab's standard operating procedure (SOP) for your particular sample preparation method.

Different solvents are used to dissolve different compounds. Be sure to choose a solvent that will dissolve the compounds you want to analyze. The solvent should be compatible with the stationary phase of your GC column when making sample dilutions.

Once your sample is prepared, you can transfer it to a sealed vial so that you can inject it into the GC. It is important to use a sealed vial because you do not want the concentration to be altered, which is what happens when the solvent evaporates.

## **Running a Sequence**

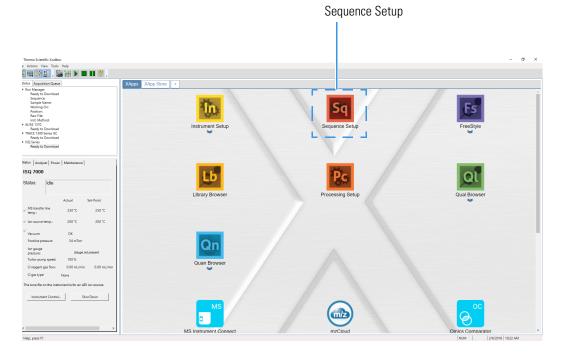
Once you've prepared your sample, you can use Xcalibur to run your sample(s). Xcalibur uses a sequence (7000 of tasks) to prepare the instruments for data acquisition, as well as monitor the injection and collection of data from the GC/MS.

A sequence can be used to acquire data from a sample or prepare the system for maintenance.

#### ✤ To run a new sequence of samples

- 1. Double-click the Xcalibur software icon on your computer desktop.
- 2. In the Xcalibur Roadmap window, click the **Sequence Setup** icon.

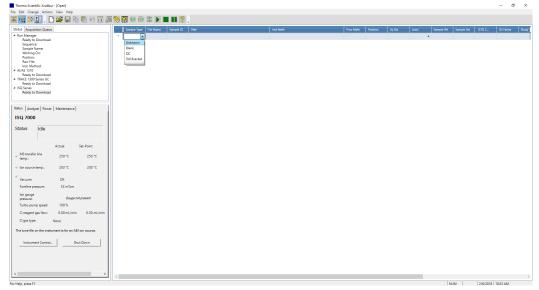
Figure 164. Sequence Setup in Xcalibur



3. In the **Sequence Setup** window, click in the first row of the **Sample Type** column and select a sample type from the drop-down menu. You should select Unknown when you are developing an analytical method. The other types of samples require a quantitation method.

**Note** To open an existing sequence, you can select **File** | **Open** from the main menu or click the  $\cong$  icon and browse to a sequence.

Figure 165. Entering the Sample Type



Once you select a sample type, default information automatically appears in some of the other columns. You can also right-click on the field to clear, copy, or paste into the field, as well as add and delete rows.

**Note** If you do not have some of the columns discussed here, you can add them by selecting **Change** | **Column Arrangement** in the main menu.

4. Click the first row in the **File Name** column and enter a file name for your first data file. If you double-click the field, you can browse to a raw data file on your computer. You can also right-click on the field to clear, copy, or paste into the field, browse to the raw data file, or add and delete rows.

**Note** Your file name cannot contain spaces.

Figure 166. Naming a File

Thermo Scientific Xcalibur - [Open]															- 0 ×
File Edit Change Actions View Help															
💥 🛄 🍄 🔛 . 🗋 🚔 🖬 🗠 🛍 🖉	i 📏 🕅 🕂 🕼 🗈 🕨 I	11 ?													
Status Acquisition Queue	Sample Type File Name														Dil Factor Stud
# Run Manager Ready to Download	► 1 Unkno ▼ data01	1	C:\Xcalibur\Data						1	1.	00	▼ 0.00	000.0	0.000	1.000
Sequence	· ·											•			
Sample Name: Working On:															
Position:			Dpen 🛛									×			
Raw File: Inst. Methodi			< → ~ ↑ 🚺	> This PC >	OS (C:) → Xcalib	ur > methods			v Ö	Search met	hods	P			
# Al/AS 1310 Ready to Download			Organize - Ne	ew folder											
# TRACE 1300 Series GC				A Nam				-			Dec				
Ready to Download # ISQ Series			data				Date modified	Туре	Size						
Ready to Download			15Q7000		AlvinAEI1 AlvinAEI2		1/19/2018 4:30 PM 1/25/2018 3:38 PM	Xcalibur Instrume. Xcalibur Instrume.							
			ISQ7000 IDL		AMINAEI2 GantDemo		1/25/2018 3:35 PM	Xcalibur Instrume. Xcalibur Instrume.							
Status Analyzer Power Maintenance			methods		dl		1/4/2018 8:57 AM	Xcalibur Instrume.							
ISQ 7000				8	IAEI		1/4/2018 5:20 PM	Xcalibur Instrume.							
ISQ 7000			🗥 OneDrive		isqfull		1/3/2018 3:27 PM	Xcalibur Instrume.	201	CB					
Status: Idle			This PC												
			Desktop												
			Documents												
Actual Set-Point			Downloads												
✓ MS transfer line 250 °C 250 °C			Music												
			Videos												
✓ Ion source temp: 203 °C 200 °C			L OS (C:)												
Vacuum: OK				~								_			
Foreline pressure: 53 mTorr				File name: is	qfull				~	Instrument	methods (.m	eth) ~			
lon gauge										Open	0	ancel			
pressure: Gauge not present															
Turbo-pump speed: 100 %															
Cl reagent gas flow: 0.00 mL/min 0.00 mL/min															
Cligas type: None															
The tune file on the instrument is for an AEI ion source.															
Instrument Control Shut Down															
< >	4										_				,
for Help, press F1												N	UM	2/6/2018 10:2	5 AM
for Heip, press FI												N	UM	2/0/2018   10/2	) AM

**Tip** You can double-click in the **File Name** field and browse to a raw file on your computer.

- 5. Click past the **Sample ID** column, which typically contains the number of the sample. Since you are in the process of developing your method, you don't have that many samples so you don't have to enter anything in this field. You can right-click on the field to clear, copy, or paste a number, as well as add and delete rows.
- 6. Double-click the first row in the **Path** column and select the folder in which to store your data files. You can also right-click on the field to clear, copy, or paste into the field, browse to a folder on your computer, or add and delete rows.

vs Acquisition Queue	Sample Type File Name	Sample ID Path		inst Meth	Proc Meth Position	Inj Vol Level	Sample Wt	Sample Vol 15	TD C DI Factor
un Managor Kandy to Download Sequence Sample Name Warking Onc Position Raw File File Onto Kang Tele Onto Kang Tele Download Read Tele Download Read Tele Download Read Tele Download		1 CAXcaliburi/Data		C/Xcailbur/methodr/uqfull	1	1.00	• 0.000	0.000	0.000 1.0
Ready to Download Q Series Ready to Download			Rectangular Snip						
Analyzer Power Maintenance			Browse For Folder	×					
Q 7000			Select a folder						
Altar: Edia Actual Set Puint Manyo Set Constraints (Set Constraints) Marken (Set Constraints)			Containing     C	OC Cover					

Figure 167. Selecting a Folder to Store Data Files

7. Double-click the first row in the **Instrument Method** column, navigate to the folder containing your instrument method. Select the instrument method and click the **Open** button. You can also right-click on the field to clear, copy, or paste into the field, browse to a folder on your computer, or add and delete rows.

atus Acquisition Queue	Sample Type File Name Sample ID	Path		Inst Meth		Proc Meth	Position	Inj Vol	Level	Sample W	t Sample Vol	ISTD C	DI Factor
Run Manager Ready to Download Seguence: Sample Name:	> 1 Unkno ▪ data01 1	C/Xcalibu/\Data					1	1.00		• 0.	000 0.000	0.000	
Working On Position Baw File:		Dpen								×			
Inst. Method: Al/AS 1310		← → ~ ↑	This PC > OS (C:) > Xcalib	r > methods			v ð	Search metho	ods	ρ			
Ready to Download RACE 1300 Series GC		Organize • New fo	older										
Ready to Download SQ Series		E Pictures 🖈			Date modified	Туре	Size						
Ready to Download		iSQ7000	AlvinAEI1			Xcalibur Instrume. Xcalibur Instrume.							
1 1 1 1		SQ7000 IDL	GantDemo			Xcalibur Instrume.							
us Analyzer Power Maintenance		imethods	idi			Xcalibur Instrume. Xcalibur Instrume.							
Q 7000		CneDrive	isqfull		1/3/2018 3:27 PM	Xcalibur Instrume.	201	08					
atus: Idle		This PC Desktop											
		Documents											
Actual Set-Point		Downloads Music											
MS transfer line 250 °C 250 °C		E Pictures											
Ion source temp: 203 °C 200 °C		📱 Videos											
Vacuum: OK		느 OS (C:)	·										
Foreline pressure: 53 mTorr		Fil	e name: isqfull				~	Instrument r					
lon gauge Gauge not present								Open	0	ancel			
pressure: Gauge not present Turbo-pump speed: 100 %													
CI reagent gas flow: 0.00 mL/min 0.00 mL/min													
Ci gas type: None													
e tune file on the instrument is for an AEI ion source.													
Instrument Control Shut Down													

Figure 168. Opening an Instrument Method Folder

- 8. Click past the **Processing Method** column, which typically contains the path to your processing method. You are in the process of developing your method and the processing method is created after your method is finalized.
- 9. Click the first row in the **Position** column and enter the sample's vial number if you are using an autosampler. You can also right-click on the field to clear, copy, or paste into the field, as well as add and delete rows.

Figure 169. Entering the Sample Vial Position

1 Unina • data01	1	C/(Kalibu/(Data	C:Xtakiburiyeshods)isqluf	1	1.00	. 0.000	0.000	0.000	1.00

10. Click the first row of the **Injection Volume** column and enter the amount of sample you are injecting if you are using an autosampler. You can also right-click on the field to clear, copy, or paste into the field, as well as add and delete rows.

**Note** This field is not used by the AI/AS 1300, which uses an injection volume from the instrument method. The TriPlus and TriPlus RSH autosamplers must be configured to read this value.

Figure 170. Setting the Injection Volume

atus Acquisition Queue				s Sample ID	-	Inst Meth	Proc Meth	Position	Inj Vol	Level	Sample Wit	Sample Wal	ISTD C	DI Extor
Nav Masger Johnson Kang to Dominal Regers to Charles Nav Poston Nav Tel Nav Te	►1 +	Unkno.	- data01	1	C/Xcalibur/Data	C (XaliburineRody)aşlul		1			- 0.000			
Marke Marker Perer Markerance   SQ2 7000 Status Marker Markerance   SQ2 Actual Sct-Point Marker Mark														
Help, press F1	¢											4		

- 11. Click past the **Level** column, which is only used when you have a processing method. Since you are in the process of developing your method, you do not have the processing method defined yet.
- 12. Now that you've set up one row of your sequence, you can stop or you can continue adding tasks. Click in the next row and repeat steps 2-10 for each additional task in your sequence. Remember, you can right-click in most fields to add more rows as you go.

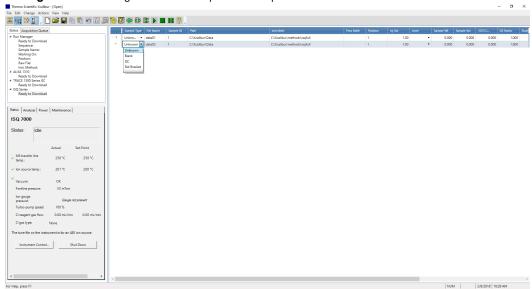


Figure 171. Adding Additional Sequence Setup Tasks

- 13. Once you've created a sequence of all your samples, select **File** | **Save** or click the **I** icon to save the sequence.
- 14. Enter a comment and click **OK**.

Figure 172. Entering a Comment About Your Sequence

Thermo Scientific Xcalibur - [Open]												0
e Edit Change Actions View Help												
s 🛄 🕼 👔 , 🗅 🚅 🖬 🗞 🗞 🗠 🛄 ,	ا 🐯 🏈 📃	• 🚯 🕼 🕨 🛯	?									
Status Acquisition Queue	Sau	nple Type File Name	Sample ID	Path	inst Meth	Proc Met	h Position	Inj Vol Leve	Sample W	Sample Vol	ISTO C D	I Factor
<ul> <li>Run Manager Ready to Download</li> </ul>	E 1 Uni	kno • data01	1	C/Xcalibur\Data	C/Xcalibur\methods\isqfull		1	1.00	+ 0.0	00 0.000	0.000	1.000
Sequence:		kno • data02	1	C:\Xcalibur\Data	C/Xcalibur\methods\isqfull		1	1.00	▼ 0.0	00 0.000	0.000	1.000
Sample Name: Working On:		•							•			
Position Raw File:												
Inst. Method: 4 AI/AS 1310												
Ready to Download												
# TRACE 1300 Series GC Ready to Download												
<ul> <li>ISQ Series Ready to Download</li> </ul>												
hady to bominate												
Status Analyzer Power Maintenance	î											
	1				File Summary Information X							
ISQ 7000					Headen Created: Tuesday, February 06, 10:35:44 AM							
Status: Idle					Last saved by: Thermo Number of saves: (Not changed)							
					Comment:							
Actual Set-Point												
MS transfer line 250 °C 250 °C												
temp.												
✓ Ion source temp.: 198 °C 200 °C												
Vacuum: OK					Ok Cancel Help							
Foreline pressure: 54 mTorr												
lon gauge												
pressure: Gauge not present												
Turbo-pump speed: 100 %												
Cl reagent gas flow: 0.00 mL/min 0.00 mL/min												
Cigas type: None												
The tune file on the instrument is for an AEI ion source.												
Instrument Control., Shut Down												
	1											
r Help, press F1										UM	2/6/2018 10:35	AM

15. Enter the name of your sequence and click the Save button.

ons View Help					0		0	,											
🗅 🚅 🖶 🗞 🖏 🗤 🛄	۵ 🏷		<b>(</b> )	•		?													
ue		1 5	ample T	lype	File Name	Sample ID	Path			Inst Meth		Proc Me	th Position	Inj Vol	Level	s	iample Wt	Sample Vol	ISTD C
d	1	U	Inkno	•	data01	1	C:\Xcalibur\Data			C:\Xcalibur\method	s\isqfull		1		1.00	•	0.000	0.000	0.0
a	2		Inkno	•	data02	1	C:\Xcalibur\Data	•		C:\Xcalibur\method	s\isqfull		1		1.00	•	0.000	0.000	0.0
				•			-					_				•			
							📓 Save As					×							
							Save in:	ISQ7000 IDL		5	) 💣 🔟 🔹								
d							*					^							
d							Quick access	UVV	AAA	UVV	<u>an</u> ñ								
d									****	<b>***</b>	***								
							Desktop	01192018	IDLAEI 20180104.	IDLAEI 20180104.	. IDLAEI_20180104								
er Maintenance	Ĩ						Lbrates												
							This PC	UVV	AAA	AAA	<u>ANN</u>								
							1		<b>***</b>	****	<b>***</b>								
							Network	IDLAEI 20180104	IDLAEI_20180104	IDLAEI 20180104.	IDLAEI								
Actual Set-Point												<u> </u>							
250 °C 250 °C								File name:	-			Save							
201 °C 200 °C								Save as type:	Sequence files (.sid)		-	Cancel							
							Header Created:												
ОК							Modified: Last saved by:												
54 mTorr							Number of saves Comment:												
Gauge not present							Comments												
100 %																			
0.00 mL/min 0.00 mL/min																			
None																			
strument is for an AEI ion source.																			
al   Shut Down																			
Shut Down																			
	<																NU		2/6/2018
			-	-	_								-				1001		2/0/2018

Figure 173. Naming and Saving your Sequence

16. Click the row you want to run and select Actions | Run This Sample from the main menu. You can also just click the 👘 icon on the main tool bar.

ile Edit Change	bur Sequence Set Actions View GoT								
🛣 🎹 🟠	Check Disk Space.				1-1	🗃 💠 🚯		<b>.</b>	
	Run This Sample Run Sequence	·	— T	Sar	nple Type File Na	me Sample I	D Path	Inst Meth	Proc Meth
Status Acquisition	n			1 Unknow		1		t C:\Xcalibur\data\	
😑 Run Manager	Batch Reprocess.		-1	2 Blank	Blank2	1	C:\Xcalibur\Da	t C:\Xcalibur\data\	
- Ready To D	c Open File			-	▼				
- Sequence: - Sample Nam	Start Analysis								
- Working On:	Stop Analysis								
- Position:	Pause Analysis								
- Raw File:	Devices On								
⊢ Inst. Method ⊨-AI/AS 3000	Devices scandby								
Ready to Do	Devices Off								
- TRACE GC Ultr		; On							
Ready to Do	Reinstate Warning	js							
i≟-ISQ ⊡ Ready to Do	wolo ad								
	Power Maintanano								
Status Analyzer	Power Maintenance	e							
		5							
Status Analyzer		8							
Status Analyzer	le								
Status Analyzer		e   Set-Point	•						
Status Analyzer Status: Id	Actual	Set-Point							
Status Analyzer Status: Id	Actual								
Status Analyzer Status: Id MS transfer line temp.:	Actual	Set-Point 200 °C							
Status Analyzer Status: Id	Actual	Set-Point							
Status Analyzer Status: Id MS transfer line temp.:	Actual	Set-Point 200 °C							
Status Analyzer Status: Id MS transfer line temp.: Ion source temp Vacuum:	le Асtual 9 200 °С р.: 200 °С ОК	Set-Point 200 °C							
Status Analyzer Status: Id	le Асtual 9 200 °С р.: 200 °С ОК	Set-Point 200 °C							
Status Analyzer Status: Id MS transfer line temp.: Vocum: Foreline pressu	le Асtual 9 200 °С р.: 200 °С ОК	Set-Point 200 °C							
Status Analyzer Status: Id MS transfer line Ion source temp Vacuum: Foreline pressu Ion gauge	le Асtual 9 200 °С р.: 200 °С ОК	Set-Point 200 °C							
Status Analyzer Status: Id MS transfer line Ion source temp Vacuum: Foreline pressu lon gauge pressure:	le Actual 200 °C p.: 200 °C OK re: 47 mTorr 3.2×10 <sup>\$</sup> Torr	Set-Point 200 °C							
Status Analyzer Status: Id MS transfer line Ion source temp Vacuum: Foreline pressu Ion gauge	le Actual 200 °C p.: 200 °C OK re: 47 mTorr 3.2×10 <sup>\$</sup> Torr	Set-Point 200 °C							

To run the whole sequence, select Actions | Run Sequence from the main menu. You can also just click the  $\blacksquare$  icon on the main tool bar.

17. In the **Run Sequence** window, customize the way you want your sequence to run.

< Run this sample

NUM

5/18/2

le Edit Change Actions View GoTo Help							
sure la companya		Sample Type	File Name			Inst Meth	Proc Met
Status Acquisition Queue	2	Unknown Blank	Blank Blank2	1		t C:\Xcalibur\data\ t C:\Xcalibur\data\	
■ Run Manager         ■ Ready To Download         > Sequence:         > Sample Name:         - Working On:         - Position:         - Raw File:         - Inst. Method:         ■ Al/AS 3000         ■ Ready to Download         ■ TRACE GC Ultra         ■ Ready to Download         ■ Status:         ■ Ready to Download         ■ Status:         ■ Ready to Download         ■ Status:         Idle         Status:         Idle         ✓ MS transfer line       200 °C         ✓ Ion source temp:       200 °C         ✓ Vacuum:       0K         Foreline pressure:       48 mTorr         Ion gauge       3.2×10.° Torr         Turbo-pump speed:       100 %	Run Sequence Acquisition Opt Instrument AI/AS 3000 TRACE GC UI ISQ ✓ Start When Instrument M Start Ug Shut Down Programs Ptc Acquisiti Post Acquisi Post Acquisi Post Acquisi	P ions Start I Ready Change ethod Change ion ion ition ronously r	Instrument Instruments Browse Browse Browse	User: [ice.smith Run Rows; ] Processing A Quan Quaj Reports Program	Sequence cetions		

Figure 175. Customizing a Sequence Run

- a. Select the **Start When Ready** checkbox to run the sequence when the instruments are ready.
- b. Click the **Change Instruments** button and use the **In Use** and **Start Instrument** columns to define the instruments that are in use and the instrument that will define the start of the sequence. Then click the **OK** button.

**Note** If you are making a manual injection, deselect the autosampler in the **Start Instrument** and **In Use** columns. Once you make the injection, press the Start button on the GC.

**Note** Even if the autosampler or GC are not in use, they can prevent the sequence from starting if they are not ready. If the autosampler or GC is not powered on or not in the ready state, remove its device driver from instrument configuration before starting a sequence that does not use the device.

- c. In the **Instrument Method** group, you can browse to the sequence to be used when the instrument starts up or shuts down.
- d. In the **Programs** group, you can browse to the executables for **Pre-Acquisition** and **Post-Acquisition**. These fields are used to automatically run processing methods after the data acquisition is complete. Because you are developing your method, you haven't defined a processing method for this field.

- e. Configure the **Run Synchronously** group:
  - **Pre-Acquisition**—Select this option to enable the executable to run before the data is acquired or deselect it to run the executable in parallel with data acquisition.
  - **Post-Acquisition**—Select this option to enable the executable to run after the data is acquired or deselect it to run the executable in parallel with data acquisition.
- f. Configure the After Sequence Set System group:
  - **On**—Select this option to leave the instrument fully ready after an acquisition.
  - **Standby**—Select this option to leave the instrument fully ready after an acquisition.
  - **Off**—Select this option to turn off the ISQ 7000 mass spectrometer by turning off the heaters, turbo pump and foreline pump.

**IMPORTANT** If you are using hydrogen as a carrier gas, manually turn off the GC after the sequence completes if you are using this setting.

- g. In the **User** field, you can change your user name. The user name defaults to the name that was used to log into the computer.
- h. In the **Run Rows** field, enter the rows to be run in the sequence. Each value must be separated by a hyphen.
- i. Check the **Priority Sequence** checkbox if this sequence has priority over any other sequence. If someone else's sequence is running in the background, this setting puts your sequence ahead of theirs.
- j. In the Processing Actions group, select **Quan**, **Qual**, **Reports**, **Programs** or **Create Quan Summary**. Since you are developing your method, you can skip this setting.

**Note** The **Reports** checkbox is only enabled when you select the **Qual** checkbox. The **Reports** and **Create Quan Summary** checkboxes are only enabled when you select the **Quan** checkbox.

18. Click **OK** to run the sample or sequence. and store your data on the computer.

- 19. In the **Save As** window, select a location on your computer that you want to store your sequence. Then click the **Save** button.
- 20. Once your sequence completes, you are ready to explore your data.

# 7

## **Exploring Your Data**

Once you have acquired your data, you need to look at your data to make sure the peaks have a good shape and that the peak area is large enough for your needs. The Qual Browser utility of Xcalibur allows you to view chromatograms and spectra from raw files or qualitative processing result files. The data in this section refers to an instrument qualification run that was performed in the factory. This same run, using 1pg octafluoronaphthalene, was also performed by a Field Service Engineer at your laboratory. Other compounds will have different retention times and ions.

#### To view your data in Qual Browser:

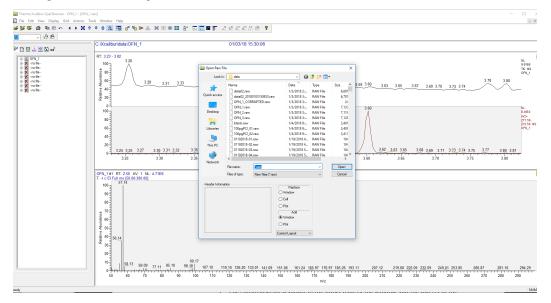
- 1. Double-click the **Xcalibur** software icon on your computer desktop.
- 2. In the Xcalibur Roadmap window, click the Qual Browser icon.

Figure 176. Xcalibur Roadmap



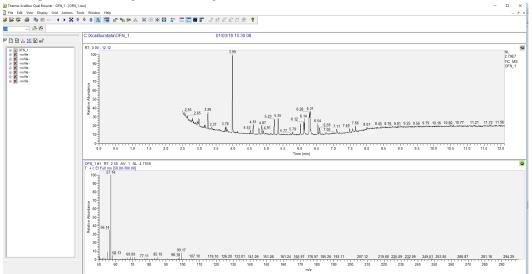
- 3. In the Qual Browser window, select **File | Open** from the main menu or click the 📂 icon.
- 4. Select the raw file(s) you ran in your sequence and click **Open**.

Figure 177. Selecting the Raw File



5. The chromatogram is shown in the upper pane of the window and the spectra in the bottom pane.

Figure 178. Viewing the Chromatogram



- 6. Right-click on the pinned chromatogram window and select **Ranges** from the pull-down menu.
- 7. Specify multiple sub-panes in the chromatogram window. For OFN, which has a dominant ion at m/z 272, select a mass range that contains only this ion. By eliminating

NL: 9.91E8 TIC MS OFN 1

8.402.4 19/2\* 271.50 272.50

3.80 3.81

all the masses that are not generated by your compound, it is much easier to find and quantify the GC peak area.

Figure 179. Selecting the Mass Range

Range Mass ra	inge: 272.00		Average	Fix so	ale: 100000.0	
Time ✓ 2.50 ✓ 2.50	Filter		Raw File C:\Xcalibur\da c:\xcalibur\dat	ta\OFN_1.raw	Subtract 1	Subt ^
	-		- - -	a wii_13aw	-	-
Plot properties Detector :	MS $\checkmark$	Time: 2.50		-	und Subtraction	>
Filter Type: Filter: Raw file:		Process	~	Time	range 2: 2.50	

With only the m/z 272 ion shown, it is apparent that the OFN is around a retention time of 3.45 minutes.



Figure 180. Determining the Retention Times

To look more closely at your data, left-click on the peak to zoom in on it. You can also hold your mouse down over the peak and draw a line over it to zoom in. Another way to zoom in is to return to the **Ranges** tab and manually enter the time range to display.

161.24 165.97 176.97 185.26 193.11

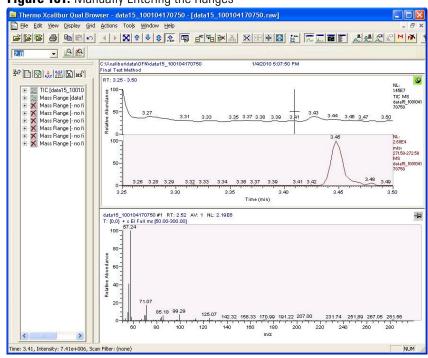
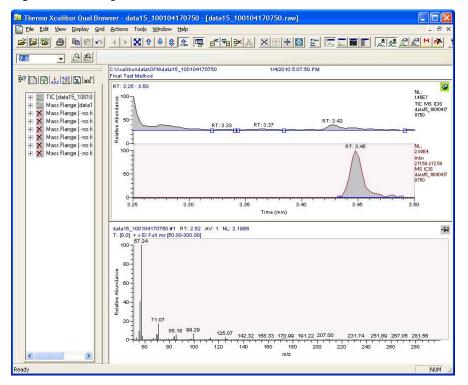


Figure 181. Manually Entering the Ranges

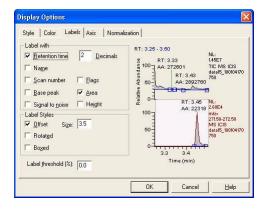
8. Right-click in the Chromatogram pane and select **Peak Detection | Toggle Detection in All Plots** to enable Qual Browser to find the peaks and define their edges.

Figure 182. Finding the Peaks in Qual Browser

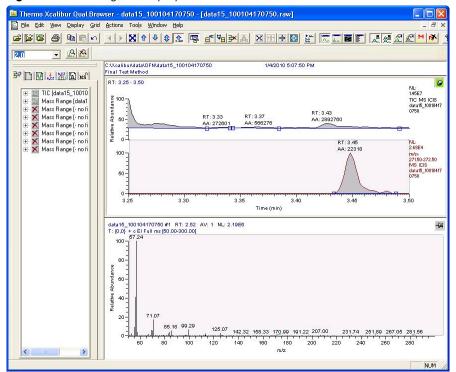


9. Right-click in the Chromatogram pane and select Display Options.

Figure 183. Selecting the Display Options



Click the Labels tab and select Retention Time and Area in the Label With group.
 Figure 184. Labeling the Display



11. With the MS window pinned, click on the peak to open the mass spectrum at the point you clicked.

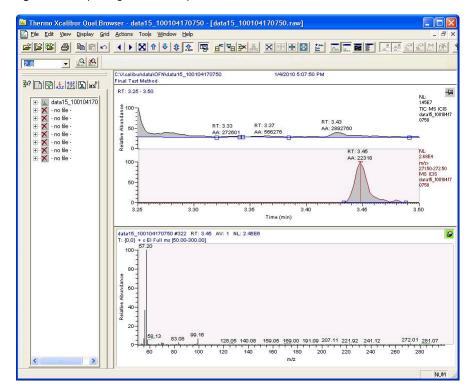


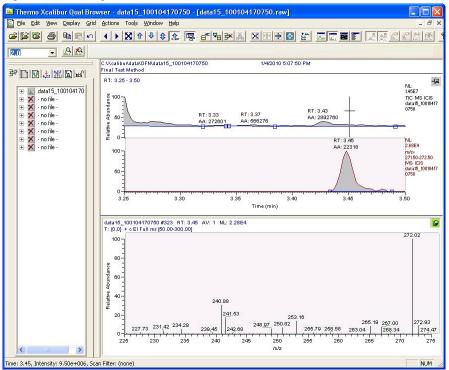
Figure 185. Opening the Mass Spectrum

12. In this spectrum, there are large peaks at m/z 57 and 99. These are very common ions from hydrocarbons. The OFN peak is near m/z 272.

#### Figure 186. Setting the Spectrum Range

Range <u>M</u> assi	ange: 225.00-275.00	Average	☐ <u>F</u> ix scale: 1000000.0	
Time	Filter	Raw File	Subtract 1	Subl
3.45		c:\xcalibur\data\	ofn\data15_100	
				~
Plot propertie <u>D</u> etector : Filter Type: Filter: <u>R</u> aw file:	MS Ii Scan C Proce	<u> </u>	Background Subitaction Time range 1: 2.52 Time range 2: 2.52 Simulation	

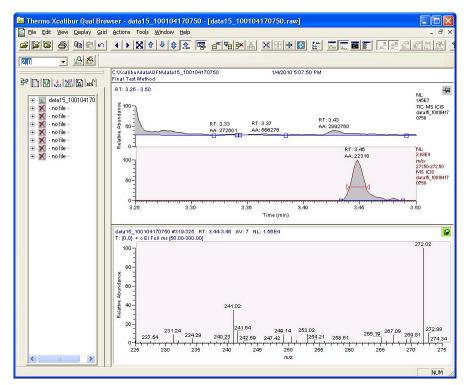
#### Figure 187. Finding the OFN Peak



13. Left-click on the GC peak and drag a line across it to average several points across the GC peak.

**Note** If the chromatogram window is pinned, this action will zoom in on the peak, but if the mass spectrum window is pinned, this action will average the spectrum.

Figure 188. Averaging the Spectrum



To perform a background subtraction and improve the spectral library searching, right-click in the MS pane, select subtract spectra and then subtract one range or two. Usually, a one range subtraction is performed before the peak eluted. The range to be subtracted out should not contain any of your chromatographic peak.

The full scan shows the ions from OFN (at m/z 222, 241, and 272) much more clearly than without the background subtraction.

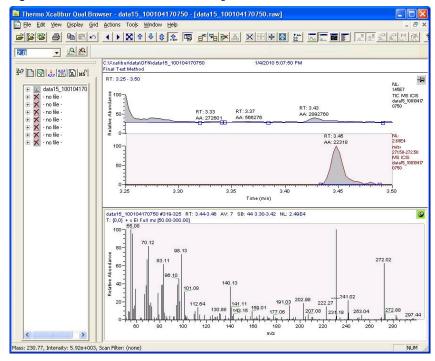
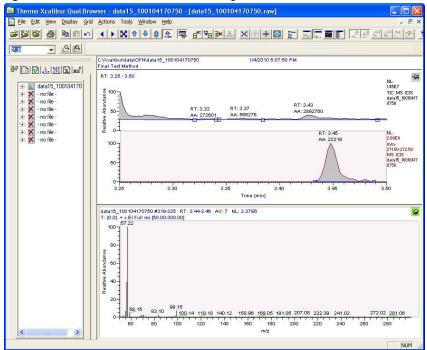


Figure 189. OFN in Full Scan With Background Subtraction

Without background subtraction, the full scan would look like the graphic below.

Figure 190. OFN in Full Scan Without Background Subtraction



14. There are many other ways to change the way you data displays in *Qual Browser*. See the *Qual Browser* online help for details.

## **Optimizing Your Method**

If you did not quite get the results you were expecting from your method, use the suggestions in this chapter to modify it to obtain better results.

#### Contents

- Changing the Chromatographic Separation
- Finding the Best Way to Make an Injection
- Improving the Way You Prepare Samples
- Changing the Scan Rate
- Narrowing the Mass Range
- Adjusting the Transfer Line Temperature
- 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. You should begin by changing the GC carrier gas flow or oven temperatures (the initial temperature, initial hold time, ramp temperature, final temperature for that ramp, and the hold time at that final temperature). These temperatures can be adjusted for each ramp.

It is important to keep in mind that 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. Changing ramp rates is usually used to separate coeluting peaks.

## 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, see 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. Try switching solvents if your method will allow it.

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 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. Increasing the scan rate increases the number of times you've sampled across the peak. However, increasing the scan rate too much results in mass spectral noise, which decreases your analytical precision. To optimize your scan rate, select a rate that will give you 8-12 points across a chromatographic peak.

## **Narrowing the Mass Range**

By narrowing your mass range, you can look directly at the compounds of interest. However, if you're 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 will also allow you to decrease the scan rate and get the same chromatographic peak sampling. Breaking your MS method into groups allows you to create compound-specific MS settings to optimize your data.

## **Adjusting the Transfer Line 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, the transfer line temperature should be 10 °C over the highest boiling point of the compounds of interest, but no more than the maximum safe operating temperature of the column. Increase the source temperature as well 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.

## **Modifying an Automatic Tune**

ISQ 7000 AutoTune is a utility that uses certain parameters in the tune types to optimize system performance when generating a tune file. To modify an automatic tune:

1. Select **AutoTune Options | Advanced AutoTune** on the ISQ 7000 Dashboard. See Figure 191.

Figure 191. Accessing the Tune Type Editor from the ISQ 7000 Dashboard

📑 ISQ 7000 Dashboard	- ×
AutoTune Options	View Tune Report
Advanced AutoTune	AutoSIM
El SmartTune	Autosim
AEI SmartTune	Xcalibur
Method Editor	
Instrument Control	Shut Down
	•
Status Analyzer Power Mainte	enance
Status: Idle	
Actual	Set Point
Instrument tune file	· · · · · · · · · · · · · · · · · · ·
AEI source	
Temperatures ✓ MS transfer line temp: 250 °C	250 °C
✓ Ion source temp: 203 °C	200 °C
Pressures	
✓ Foreline pressure: 53 mTorr	
Misc	
Vacuum: OK Turbo pump speed: 100 %	
CI reagent gas flow 0.00 mL/min	0.00 mL/min
CI gas type None	
Cl gas type None	

2. ISQ AutoTune opens. Click **Tune Types**. See Figure 192.

Kana Kata Kata Kata Kata Kata Kata Kata	? ×
Source type: O EI O CI	
Category: Default ~	Tune Types
El Initial Tune (default)   El Full Tune (default)   El Tune (default)   El Check (default)   El Diagnostics (default)   Cl- Tune (default)   Cl+ Tune (default)	Display report when complete
Now running:	Show spectra
Action:	Start

Figure 192. Accessing the Tune Type Editor from ISQ 7000 AutoTune

3. In the Tune Types dialog box, select a tune type to edit and click the **Copy** button. See Figure 193.

Figure 193. Using the Tune Types Dialog Box

Tune Types		?	×
Categor Default			~
AEI Full Tune (default) AEI Tune (default)	^	New	
AEI Check (default) AEI Diagnostics (default)		Edit	
El Initial Tune (default) El Full Tune (default) El Tune (default)	Ļ	Сору	
		Rename	
Complete tuning for AEI source.	~	Delete	
Tunes and sets detector gain to 3x10^5. Starts with last saved tune.			
	$\vee$	Close	

4. Configure the options under the **General** tab. See Figure 194.

Edit Tune T	Type ? - ×
Name:	El Diagnostics Save
Description:	Runs complete set of diagnostics and generates a report. No tuning is performed.
Categories:	Default V Print
Ту	ype: (i) Tune and diagnostics O Diagnostics only 🗹 Show advanced settings
General lor	n Source Targets Detector Diagnostics Report Lenses Resolution
Output tune file	
Starting tune file	e: (Last Saved)
Mass calibrati	
Perform	mass calibration O Check mass calibration

#### Figure 194. Configuring the General Options

- Name—Use this field to enter a name for your tune type.
- **Description**—Use this field to enter details or notes about your tune type.
- **Type**—Select **Tune and Diagnostics** to run a lens tune with diagnostics or select **Diagnostics Only** if you are creating a diagnostics test.
- **Output Tune Filename Prefix**—Use this field to enter a prefix to be added to the title of your tune report. For example, if you are always running BFB reports, you could enter BFB here to distinguish it from other reports you are generating.
- **Starting Tune File**—Use this pull-down menu to 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. A tune file may also be loaded and saved to the instrument for the Last Saved tune file using *ISQ 7000 Manual Tune*.
- Select a specific tune file if you have a reliable tune file you want to use as a test for the new tune file.
- **Perform Mass Calibration**—Select this button to enable the system to recalibrate all of the masses during a tune.

- **Check Mass Calibration**—Select this button to enable the system to confirm that your mass calibration is correct rather than performing a mass calibration.
- 5. Configure the options under the **Ion Source** tab. See Figure 195.

Figure 195. Configuring the Ion Source Options

Edit Tune Type	? _ ×
Name: El Diagnostics	Save
Description: Runs complete set of diagnostics and generates a report. No tuning is performed.	Cancel
Categories: Default ~	Print
Type: <ul> <li>Tune and diagnostics</li> <li>Diagnostics only</li> <li>Show advanced settings</li> </ul>	
General Ion Source Targets Detector Diagnostics Report Lenses Resolution	
Ionization mode & ion polarity: EI+  Using AEI Source	
Cl gas type: Methane V	
CI gas flow: 0.3 mL/min	
Emission current: Tune File V µA	
Electron energy: Tune File V	
Electron lens positive voltage: Tune File Volts	
Electron lens negative voltage: Default ~ -75 Volts	
Set ion source temperature: C	

- Ionization Mode and 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.
- Using AEI Source—Check this box if an AEI source is installed.
- **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 ISQ 7000 system's software.

• **CI Gas Flow** (enabled when you select a CI Gas Type)—Use this field to enter the flow rate of your CI gas.

- Emission Current—Defines the emission current that you use to run subsequent tunes, but not the emission current that is used for data acquisition. Lowering the emission current can extend the time between required cleanings of the ion source and replacing the filament as well as extending the detector lifetime.
  - **Tune File**—Select this option to use the value in the tune file you selected in 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. You should 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, lower filament lifetime, and cause unnecessary source degradation.
- **Electron Energy**—Use this field to indicate the energy of the electrons that come off the filament and to extend the lifetime of your filament.
  - Default—Select this option to use the default electron energy, which is 70 V for the EI ion source and 50 eV for the AEI ion source.
  - **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** Reducing the electron energy to less than 45 eV is not recommended. The calibration compound may not be sufficiently ionized for tuning or calibrating at low electron energies.

- Electron Lens Positive Voltage—Use this field to allow the electrons to enter the ion volume.
  - Default—Select this option to use the default electron lens positive voltage, which is 5 V.
  - **Tune File**—Select this option to use the value used in the tune file you selected in the General tab.
  - **Custom**—elect this option if you do not want to use the default value for the tune. For the electron lens positive voltage, the default is 5 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.

**Note** When using the AEI source, this lens acts as an ion repeller, so setting a higher value may make it more difficult to pass resolution tuning. Values above 7 to 10 V are not recommended for the AEI source.

- Electron Lens Negative Voltage. This field is used to allow the electrons to enter the ion volume.
  - 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 you selected in 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.
- Set Ion Source Temperature—Select this checkbox to enable the temperature field. Then enter a value between 0 and 350 °C (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. 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 will be tuning 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.

6. Configure the options under the **Lenses** 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) should be done about the resolution of the peak and any errors that occur. See Figure 196.

Note Click the Advanced Settings check box to access the Lenses tab.

	ne: E	I Full Tune										Save	
es	scription:	Complete El tuning. Tunes and sets detector gain to 3x10°5. Starts with last saved tune.										Cancel	
ate	egories:	Default									~	Print	
	Type eral   Ion S Tune lenses	e: ( ) Tune ar iource Targets	Detector		O Dia mostics	agnostics	ort Le		ow advanced Resolution	a acturi iga		1	
	Device		Mass	Start	Stop	Step	Max. Width	Measure at %	Threshold	On Error	Detector G		
	Repeller		219	0	12.5	0.25	2	10	0.5	Continue	300000		
		e voltage	219 502	0 -15	12.5 -1	0.25	2	10 50	0.5	Continue Continue	300000 300000		
•	Ion Guide			-			-						
•	Ion Guide	e voltage	502	-15	-1	0.25	2	50	1	Continue	300000		
,	Ion Guide Ion Guide Ion Guide	e voltage e voltage	502 219	-15 -10	-1 1	0.25 0.2	2	50 50	1	Continue Continue	300000 300000		
•	Ion Guide Ion Guide Ion Guide	e voltage e voltage e voltage e voltage	502 219 69	-15 -10 -10	-1 1 1	0.25 0.2 0.2	2 2 2	50 50 50	1 1 1	Continue Continue Continue	300000 300000 300000		
	Ion Guide Ion Guide Ion Guide Ion Guide	e voltage e voltage e voltage e voltage e RF q	502 219 69 40	-15 -10 -10 -10	-1 1 1 1	0.25 0.2 0.2 0.2 0.2	2 2 2 2 2	50 50 50 50 50	1 1 1 1	Continue Continue Continue Continue	300000 300000 300000 300000 300000		

#### Figure 196. Configuring the Lenses Options

- **Device**—Use this pull-down menu to select the component you want to tune according to the settings in the other columns.
  - Repeller—Select this option to control how the repeller pushes the ions out of the ionization region. 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 prevent ions from striking the repeller surface, which leads to better robustness. Typical values are 0 to 5 V, although a dirty system may have the repeller climb as high as 12.5 V. We do not recommend setting the repeller any higher.
  - Lens 1—Select this option to control 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.
  - Lens 2—Select this option to control 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 in EI mode. In CI, the optimal voltage may be between 0 and -30 V.
  - Lens 3—Select this option to control 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.

- Ion Guide DC—Select this option to control 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.
- Ion Guide RF—Select this option to control 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.
- Q1—Select this option to control 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.
- Resolution—Select this option to adjust 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.
- Mass—Use this pull-down menu to select the ion to be used for tuning.
- **Start**—Use this field to 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.
- **Stop**—Use this field to enter the final voltage for the tune.
- **Step**—Use this field to 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. You may notice that in some tunes devices are set to a value before tuning them in another line. This is done because ramping devices will not allow the tune to run at values higher than those chosen for higher masses for the same device.

- Max Width—Use this field to enter the maximum allowable width of the ion during the tune.
- Measure at %—Use this pull-down menu to select the location on the peak at which you want to measure the maximum width.
  - **10**—Select this option to measure the width at 10% of the peak height.
  - **50**—Select this option to measure the width at 50% of the peak height.

• **Threshold**—Use this field to enter the change in intensity that has to occur when the tune to select 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 a value less than 1 is chosen for ramped devices, the tune will step back in energy until the lower intensity is detected. For example, if 0.8 is chosen, the device value lower in energy than the device value with the maximum response which gives an intensity closest to 80% of the maximum intensity will be chosen.

- On Error—Use this pull-down menu to select how to handle an error in the tune.
  - **Fail**—Select this option to stop the tune when an error occurs or if no tune points are found meeting the tune criteria.
  - Continue—Select this option to allow the tune to continue on to the next device when an error occurs.
- 7. Click the **Resolution** tab to configure the resolution of your tune. You can tune the resolution during a lens tune or you can tune the resolution by itself.

Note Click the Advanced Settings check box to access the Resolution tab.

• **Tune Resolution**—Select this option to tune the resolution 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. See Figure 197.

Name: El Full Tune											
Description: Complete El tuning. Tunes and sets detector gain to 3x10^5. Starts with last saved tune.											
Categories: Default											
	Тур	oe: (i) Tune ar	nd diagnostics	O Diagnostics only	Show advanced s	settings					
		Course   Tomata	Detector Disc								
ne	ral lon	Source Targets	Detector Diag	gnostics Report	Lenses Resolution						
Tu	une resoluti	on 🔲 Tune high sc	an rate: 10000	Tune resolution 🔲 Tune high scan rate: 10000 amu/sec 🔲 Use custom emission current:							
Mass Peak Width Measure at % Lens Tune Relation Detector Gain											
	Mass	Peak Width	Measure at %	Lens Tune Relation	Detector Gain	-					
	Mass 69	Peak Width 0.96	Measure at %	Lens Tune Relation After	Detector Gain 300000						
	69	0.96	10	After	300000						
	69 69	0.96 0.65	10 50	After Before	300000 300000						
	69 69 219	0.96 0.65 0.96	10 50 10	After Before After	300000 300000 300000						
	69 69 219 219	0.96 0.65 0.96 0.65	10 50 10 50	After Before After Before	300000 300000 300000 300000						
	69 69 219 219	0.96 0.65 0.96 0.65	10 50 10 50	After Before After Before	300000 300000 300000 300000						
	69 69 219 219	0.96 0.65 0.96 0.65	10 50 10 50	After Before After Before	300000 300000 300000 300000						

**Figure 197.** Configuring the Tune Resolution

- Mass—Use this pull-down menu to select the ion to be used for tuning.
- **Peak Width**—Use this field to enter the target peak width.
- **Measure at %**—Use this pull-down menu to select the location on the peak at which you want to measure the target peak width.
  - **10**—Select this option to measure the width at 10% of the peak height.
  - **50**—Select this option to measure the width at 50% of the peak height.
- Lens Tune Relation—Use this pull-down menu to set the occurrence of the resolution tune parameters before or after a lens tune.
  - **Before and After**—Select this option to use the same resolution parameters before and after a lens tune.
  - **Before**—Select this option to use the resolution parameters before a lens tune.
  - After—Select this option to use the resolution parameters after a lens tune.
- **Tune High Scan Rate**—Select this checkbox to tune the resolution at a scan rate in addition to the 100 and 1000 amu/s default scan rates.
- 8. Click the **Targets** tab to configure how you want to tune your target ion ratios. Target tuning is used to adjust the way the ISQ 7000 instrument tunes to meet regulatory requirements. See Figure 198.

Edit Tune T	уре	? _ ×									
Name:	Name: El Tune										
Description:	Description: Tunes resolution, mass, lenses, adjusts detector sensitivity, Does not tune detector gain.										
Categories:											
Ту	pe: (i) Tune and diagnostics (i) Diagnostics only iii Show advanced settings										
General Ion	Source Targets Detector Diagnostics Report Lenses Resolution										
✓ Tune target	ion ratios										
Target mass	Ratio										
50 amu:	1.1 % (25 to 51.5 amu)										
69 amu:	100 % (51.5 to 78.5 amu)										
131 amu:	90 % (78.5 to 132.5 amu)										
219 amu:	70 % (132.5 to 220.5 amu)										
414 amu:	2 % (220.5 to 415.5 amu)										
502 amu:	1.3 % (415.5 amu and above)										

Figure 198. Configuring the Target Ion Ratios

- **Tune Target Ion Ratios**—Select this checkbox to adjust the ratios based on the results from an injection of tuning compound.
- 9. Configure the options under the **Detector** tab. See Figure 199.

Edit Tune T	уре	? _ ×
Name:	El Diagnostics	Save
Description:	Runs complete set of diagnostics and generates a report. No tuning is performed.	Cancel
Categories:	Default v	Print
Ту	pe:	
General Ion	Source Targets Detector Diagnostics Report Lenses Resolution	
Set initial de	tector gain: Last set detector gain will be used for tuning.	
Tune detect	tor	
Use cu	ustom emission current: 50 µA	
Adjust detec		
Mass:	219 V Intensity: 20,000,000	

Figure 199. Configuring the Detector Options

- Initial Detector Gain—Select this option to set the true gain of the electron multiplier. The gain is the number of electrons generated for every ion that strikes the detector. This is typically set between  $1 \times 10^5$  and  $3 \times 10^5$  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. As the electron multiplier ages, the voltage required for a given gain will increase. 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.
- Tune Detector—Select this checkbox to tune the detector.
- Adjust Detector Sensitivity—Select this checkbox 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 will result in larger variation in the analytical runs as compared with using a fixed detector gain.
  - Mass—Use this pull-down menu to select the calibration gas mass you want to use.
  - Intensity—Use this field to enter the intensity you want to see on the tune report.
- 10. Click the **Diagnostics** tab and select a test to confirm the operational ability of the ISQ 7000 system. See Figure 200.

Edit Tune T	уре		? _ × 1						
	[c) 7								
Name:	El Tune	Save							
Description:	Tunes resolution,	Cancel							
Categories:	Default	~	Print						
	Type:   Tune and diagnostics  Diagnostics only  Show advanced settings								
lonization mode	e & ion polarity:	EI+ V Using AEI Source							
	CI gas type:	Methane ~							
	CI gas flow:	0.3 mL/min							
Emission current	ıt:	Tune File 🗸 \mu A							
Electron energy	r:	Tune File 🗸 eV							
Electron lens po	ositive voltage:	Tune File Volts							
Electron lens ne	egative voltage:	Default Volts							
Set ion source	ce temperature:	300 °C							

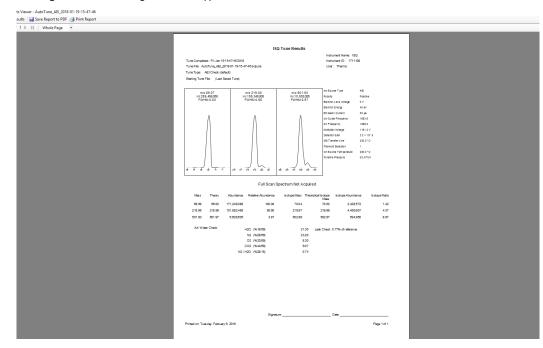
Figure 200. Accessing the ISQ 7000 System Diagnostics

11. Click the **Report** tab configure how you want to view your data. See Figure 201.

Edit Tune Type	? _ ×
Name: El Diagnostics	Save
Description: Runs complete set of diagnostics and generates a report. No tuning is performed.	Cancel
Categories: Default ~	Print
Type: <ul> <li>Tune and diagnostics</li> <li>Diagnostics only</li> <li>Show advanced settings</li> </ul>	
General Ion Source Targets Detector Diagnostics Report Lenses Resolution	
Acquire full-scan spectrum	
Acquisition threshold: 3,000	
SIM masses:         69         219         502         (none)         (none)	

### Figure 201. Configuring the Report Options

- Acquire Full-Scan Spectrum—Select this checkbox to display the full spectrum on your tune report.
  - Acquisition Threshold—Use this field to enter a minimum peak height for the data file. If your peak has an intensity that is below this threshold, it will not be stored.
  - **SIM Masses**—Use these pull-down menus to select masses to be displayed on your tune report. You can select a maximum of five masses, one from each menu.
- 12. Once you are finished configuring all the tabs, click the **Save** button to save the tune type. Your new tune type is now in the **Tune Types** window.
- 13. Click **Print** to save your tune type information as a PDF or to print a hard copy of the information. See Figure 202.



### Figure 202. Printing the Tune Type Information

14. Click **Close** to return to the Method Setup window. Now the tune type can be selected during an automatic tune.

# 9

# **Troubleshooting**

This section contains information to help you diagnose problems with your data. A lot of times, 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

- Setting Instrument Conditions for Troubleshooting
- No Ions Present in Scans
- Checking the ISQ 7000 System Firmware
- How to Know When Your System Needs Maintenance
- Investigating Baseline Issues
- Investigating Peak Issues
- Investigating Results Issues
- Reconfiguring Your Instrument
- Upgrading the Software

As you review your data, you may notice issues with the baseline, peaks, or results. Use the information in this section to troubleshoot and resolve the issue. If there is an issue with the hardware, see the *Troubleshooting* section of the *ISQ 7000 Hardware Manual*.



**CAUTION ELECTRICAL SHOCK HAZARD:** When troubleshooting any issue that requires removing a cover on the ISQ 7000 MS, you should power-off and vent the instrument to avoid any harm to yourself.



**ATTENTION RISQUE D'ÉLECTROCUTION** : lorsque vous cherchez à résoudre un problème nécessitant d'enlever le couvercle de l'instrument, mettez hors tension et ventilez l'instrument pour éviter tout risque de blessure.

A good first step for troubleshooting is to run a tune on the ISQ 7000 system. 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 an air leak, locate and address them. Pay particular attention to the transfer line ferrule, vent valve knob, front panel, and vacuum interlock on the ISQ 7000 instrument, 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 *ISQ 7000 Hardware Manual* for instructions on cleaning and inserting the ion source cartridge.

### **Setting Instrument Conditions for Troubleshooting**

Before troubleshooting the ISQ 7000 system, set the instrument to the conditions in this section in order to compare your system more accurately to the values in the section. All troubleshooting should be performed in EI mode. Once EI mode is working, check CI conditions if relevant.

**IMPORTANT** Use only Nitrile Cleanroom gloves when touching ion source components. Other types of gloves deposit contaminants on the source components that affect system performance. See the *ISQ 7000 Spare Parts Guide* for ordering information.

- Clean the ion source cartridge. See the ISQ 7000 Hardware Manual for instructions.
- Install a 15 m × 0.25 mm × 0.25 mm GC column (If using a different column, pressure readings may vary.)
- Ion Source Temperature 200 °C
- Transfer Line Temperature 250 °C
- Vacuum Compensation On
- Column Flow Rate 1.2 mL/min
- Foreline Pressure < 100 mTorr

**Note** Foreline Pressure is a function of how long the interior of the manifold has been exposed to the atmosphere, the pumping capacity of the turbo pump, length of the foreline hose, and other criteria. As an example, if the system has been recently exposed to atmosphere, the foreline pressure will be above the expected value.

• Pump down the system. Pump down time varies depending on the size of the turbomolecular pump installed.

**Note** There are no vacuum readings on any ISQ 7000 instrument that lacks a convectron gauge.

• Air/Water Check — Water (*m*/*z* 18/69) < 240%

Once you have applied the settings in this section, and have allowed the ISQ 7000 system to equilibrate, run an EI diagnostic tune even if you cannot see any ion intensities.

### **Checking Air/Water Spectra**

Before running additional diagnostics, check the air/water spectra of your ISQ 7000 system in the ISQ Manual Tune utility and use the information in this section to troubleshoot your system.

#### To check Air/Water spectra on the ISQ 7000 system

- 1. Insert the EI ion source if it is not already in place.
- 2. Open the ISQ Dashboard. Check that all the Status indicators are green and that the turbo pump is set to 100%.
- 3. Select Air & Water/Tune and check the air/water spectrum. See Figure 203.

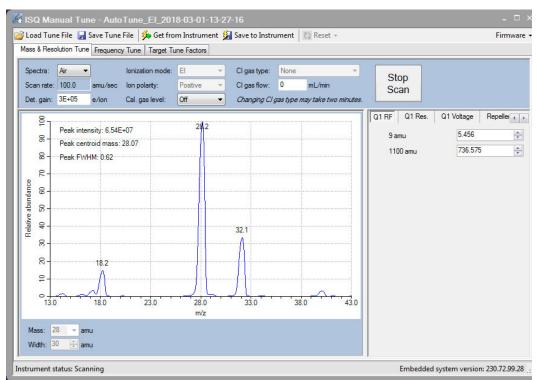


Figure 203. Typical ISQ 7000 Air/Water Spectrum with Helium Carrier Gas at 1.2mL/min

The typical results (with helium carrier gas flow at 1.2mL/min) are:

- Detector Gain =  $3 \times 10^5$
- Peak Intensity  $\ge 3 \times 10^6$

**Note** Peak intensity varies depending on variables such as the amount of water and nitrogen in the helium gas supply and the column flow.

- Normal ions are present and in acceptable ratios:
  - 18 (Water) 20–300% of N<sub>2</sub>
  - 28 (N<sub>2</sub>) Reference (base) Peak
  - 32 (O<sub>2</sub>) 10-40% of N<sub>2</sub>
  - 40 (Argon) <10% of N<sub>2</sub>

**Note** Do not expect air and water ions to be at relative abundances from atmospheric air. Nitrogen is a common contaminant within the carrier gas and is not removed with most filters, while oxygen is typically removed from helium by most gas filters.

4. Using hydrogen as a carrier gas changes the air/water spectrum on the ISQ 7000 system. It general more background peaks due to the increased reactivity of the hydrogen gas with

the components inside of the sample path. See Figure 204 for an image of a typical ISQ 7000 air/water spectrum when hydrogen is used as a carrier gas.

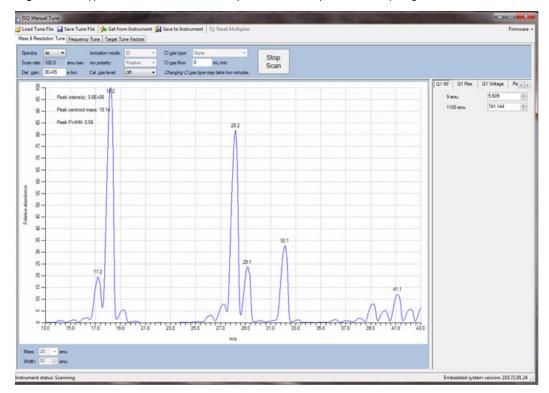


Figure 204. Typical ISQ 7000 Air/Water Spectrum for a System with Hydrogen Carrier Gas

The following conditions can cause changes in the air/water spectrum on the ISQ 7000 instrument.

- 1. Standard detector gain is equal to 3e5, but this can vary depending on customer defined tunes.
- 2. As the instrument pumps down over time, the ratio of 18/28 will change as m/z 18 decreases with m/z 28 remaining constant. This eventually changes m/z 28 to the base peak.
- 3. Changing components of the system such as the column, ion source, or gas supply affects the different masses present in the air/water spectra.
- 4. Maximum intensity may vary based on different instrument parameters, such as changing the column flow, or accessories.

If any of the previous conditions are not met and a leak is suspected as the root cause, follow "An air leak has been detected" in the *"Investigating Vacuum Issues*" section of the *ISQ 7000 Hardware Manual* or "How to Know When Your System Needs Maintenance" on page 214.

The next several images show a typical air/water spectrum for several common problems.

1. Figure 205 is an example of an air/water spectrum of a system with a potential air leak.

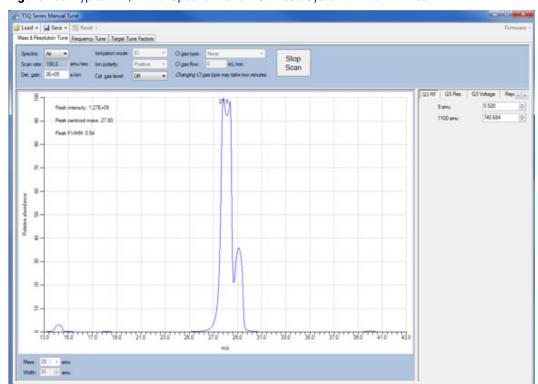
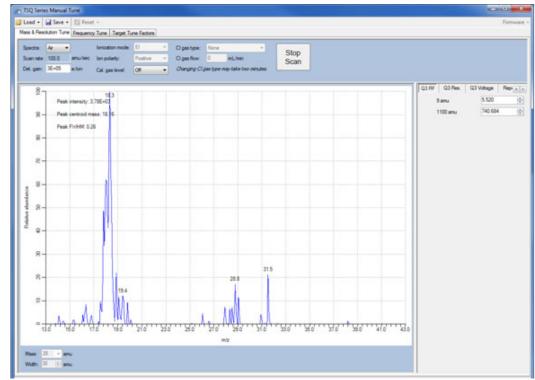


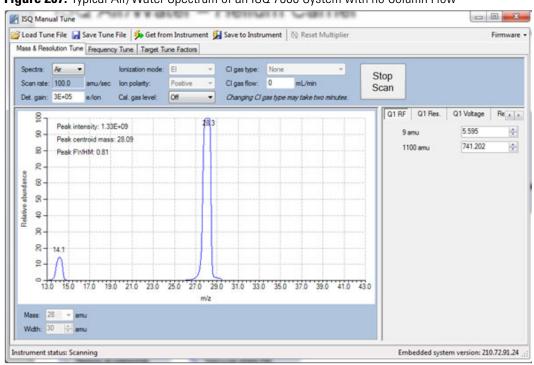
Figure 205. Typical Air/Water Spectrum of an ISO 7000 System with an Air Leak

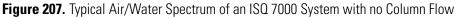
2. Figure 206 is an example of a system with an incorrectly installed ion source.





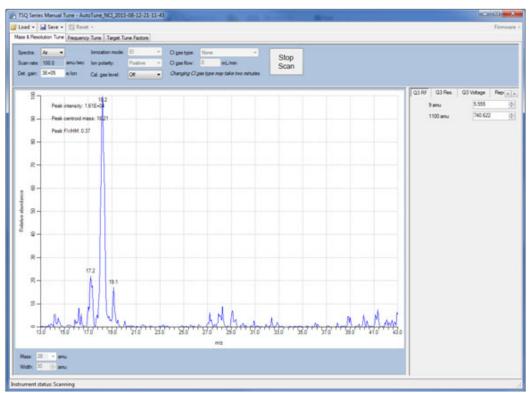
3. Figure 207 is an example of a system with no column flow.





4. Figure 208 is an example of a spectrum with a CI tune file run with an EI ion source.

Figure 208. Typical Air/Water Spectrum of an ISQ 7000 System with an El Ion Source Run with a Cl Tune File



### **No lons Present in Scans**

Lacking ions in scans or having low response can be caused by many different issues. The most common causes are instrument settings, improper tune files, or a recent change to the system. When troubleshooting, always go back to the last change made to the system and examine the delta.

#### \* Try the following solutions if you cannot see ions in any scans

- 1. Run diagnostic checks (diagnostics only tune type).
- 2. Remove and reinstall the ion source cartridge.
- 3. Switch filaments.
- 4. Confirm the starting tune file is appropriate for ionization mode or source cleanliness level.
- 5. Confirm the correct ion volume is inserted (EI or CI).
- 6. Clean the ion source.

### Try the following if you do not see ions in CI mode

- 1. Confirm that the CI ion volume is installed.
- 2. Confirm that the reagent gas is connected and turned on.
- 3. Confirm an appropriate tune file is being used.
- 4. Switch filaments.

### **Checking the ISQ 7000 System Firmware**

Confirm that you are running the correct version of the ISQ 7000 instrument firmware.

- \* To find the version of firmware installed on your ISQ 7000 system
- 1. On the ISQ 7000 Dashboard, click **Air & Water/Tune**. This opens the ISQ 7000 Manual Tune application.

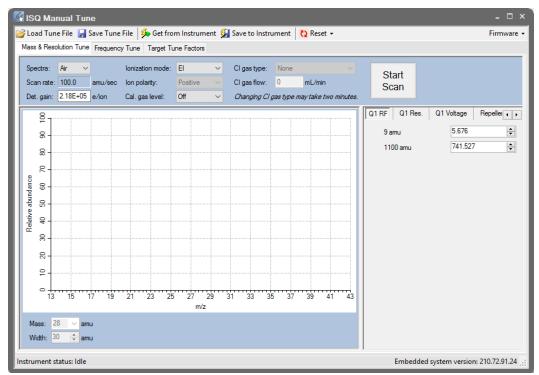


Figure 209. The ISQ Manual Tune Application

- 2. Click **Firmware** in the upper right-hand corner of the home screen in ISQ 7000 Manual Tune.
- 3. A new menu opens. Click Version Info.
- 4. A dialog box containing opens that lists the firmware versions for the ISQ 7000 instrument control drivers, the lens driver board, and the controller interface board. See Figure 210.

 Version Info

 Instrument Control Software: 2.0.0.37

 Instrument ID: 811003

 PC Communication Board Firmware: 210.0.77.17

 Controller Interface

 Microcontroller: 11

 FPGA: 33

 Lens Driver

 Microcontroller 1: 21

 Microcontroller 2: 20

 CPLD: 6

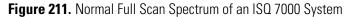
Figure 210. Determining the Firmware Versions

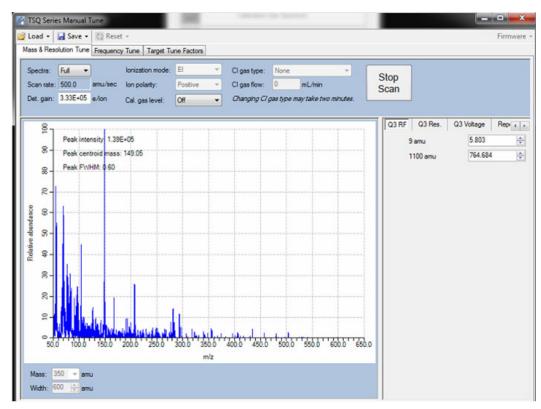
**Note** Depending on the version of ISQ 7000 instrument control software you are running, the controller interface board and lens driver board firmware may not be listed.

- 5. Click **Copy Info** to copy the information to the clipboard on your PC.
- 6. Save this information. It may be useful to your Thermo Fisher Scientific Field Service Engineer if you need a service call.

### 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. Figure 211 shows a normal full scan background on an ISQ 7000 system.





A normal system should have the following conditions met;

- Detector gain  $3 \times 10^5$
- Maximum intensity  $< 1 \times 10^6$
- There should be exponential decay for background noise.

**Note** Intensity of background ions (chemical noise) should decrease with an increase in m/z.

• There should be no extraneous peaks indicating contamination.

If you run a sample with Perfluorotributylamine (PFTBA), the tuning compound, the spectrum should look like Figure 212 below.



Spectra: Scan rate: Det. gain:	500.0	amu/sec	Ionization mode: Ion polarity: Cal. gas level:	Positive	-	Cl gas type: Cl gas flow: <i>Changing Cl</i>	0	mL/min may take to	e mitutes.	Stop Scan						
Publishe divergence Publishe divergence Publis		* 100.0 <sup>4</sup>	2 66E-07	······································	215.9	280.0****	300.0		0.0 <sup>+++</sup>		 · 5000 · ·	· '5500' ·	* kože* *	 0 1 FUF 01 Flore. 9 emu. 1100 emu	G1 Webge 5 609 741.144	Per set

A normal PFTBA profile spectrum should meet the conditions below.

- Intensity >  $2.5 \times 10^7$
- The ions m/z 69, 131, 219, 414, and 502 are all present and in the correct relative ratios.
  - m/z 69 or m/z 219 is the base peak
  - *m/z* 131 and *m/z* 219 between 60-100%
  - *m/z* 414 and *m/z* 502 between 1-10%

**Note** High mass ions decrease in relative abundance when ion source temperature increases.

- m/z 100, 119, and 264 are also present and cleanly separated from any noise.
- Mass assignments are correct. Review the tune report for true mass assignment values.
- No extraneous peaks indicating contamination are present.

Some of the most common reasons and indications your instrument needs maintenance are as follows:

• **Contamination**—If you notice excessive background in your mass spectra, it is usually an indication that your instrument is contaminated. Use the mass spectrum in the table below to understand the origin of the contamination. If you notice cleaning solvent peaks, it is usually an indication that your ion source cartridge was installed before it was completely dried. Table 3 shows a list of common contaminants.

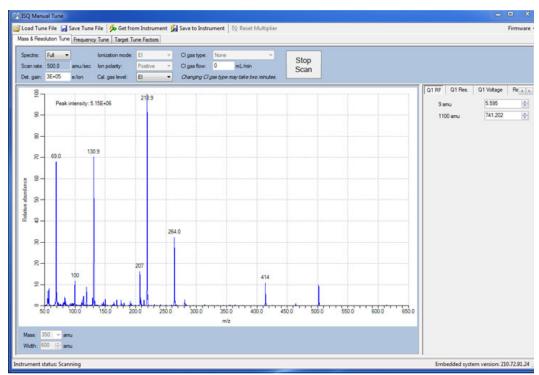
- **Fingerprints**—If you notice a series of mass peaks in your data that are 14 amu apart, it is usually an indication of fingerprint or other hydrocarbon contamination. To avoid fingerprints, you should wear clean, lint-free gloves when performing any type of maintenance on a component in the vacuum manifold of the ISQ.
- Air Leaks—If you notice a higher than normal vacuum pressure or poor sensitivity, it is usually an indication of an air leak. Check the last o-ring or ferrule 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	$H_2O, N_2, O_2, CO_2 \text{ or } N, O$	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 pump oil, or other hydrocarbons

#### Table 3. Common Contaminants

Figure 213 shows an abnormal PFTBA profile spectrum.





In Figure 213, the abnormal results are as follows:

- Maximum intensity <  $1.5 \times 10^7$  (15,000,0000)
- m/z 207 is prominent, indicating column bleed
- Many small contamination peaks present

# **Investigating Baseline Issues**

 Table 4.
 Troubleshooting Baseline Issues in Your Data (Sheet 1 of 2)

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 impurities. 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.

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.

### Table 4. Troubleshooting Baseline Issues in Your Data, continued (Sheet 2 of 2)

# **Investigating Peak Issues**

 Table 5.
 Troubleshooting Peak Issues in Your Data (Sheet 1 of 3)

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 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.

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. Run the method with higher source temperature, making certain not to start running samples in matrix until the source has had several hours to reach thermal equilibrium.
		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	Increase 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 5.
 Troubleshooting Peak Issues in Your Data, continued (Sheet 2 of 3)

Thermo Scientific

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.	
		ISQ 7000 instrument is not collecting data	Make sure the tune file is correct. Verify that the Busy light is on during acquisition. Close Xcalibur, open Instrument Configuration, press the <b>Reset</b> button on the ISQ 7000 instrument, wait ten seconds, close Instrument Configuration.	

### Table 5. Troubleshooting Peak Issues in Your Data, continued (Sheet 3 of 3)

# **Investigating Results Issues**

Table 6.	Troubleshooting Results	Issues in Your Data	(Sheet 1 of 2)
----------	-------------------------	---------------------	----------------

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 MS.
		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.

223

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.
		Handshaking is not configured correctly	Be sure the GC is inhibited by the MS and waits for contact closure by the autosampler.
		Column temperature is unstable	Check the main oven door and cooling flap. Monitor the column temperature.

### **Reconfiguring Your Instrument**

If your instrument loses communication with the computer, you have reinstalled Xcalibur, or you have a new computer, you may need to reconfigure the ISQ 7000 instrument.

#### ✤ To reconfigure your ISQ 7000 instrument

- From the Start menu on your computer desktop, browse to Thermo Foundation 3.1 | Instrument Configuration. When the Instrument Configuration utility opens, you can see an icon of the instruments you have connected.
- 2. Click the ISQ 7000 (and other instruments) icon in the Available Devices column and click **Add** to move it into the Configured Devices column.

🍇 Thermo Foundation Ir	nstrument Configuration	n		-	×
Device <u>Typ</u> es : All	•				
Available Devices:			Configured Devices:		
Al/AS1310	Focus GC	•			
ISQ	TRACE GC Ultra				
TRACE(2) GC Ultra	TriPlus Autosampler	Ŧ			
	Add >>		<< Remove	Configure	
	Done		Help		

Figure 214. Finding Available Devices

3. Click the each instrument icon you want to configure and click Add.

🐁 Thermo Foundation In		n	_	_	×
Device <u>Types</u> : All	•				
Available Devices:			Configured Devices:		
Al/AS1310	TRACE 1300 GC Series	* III	Al/AS 1310	ISQ	
ISQ	TRACE GC Ultra		TRACE 1300 GC Series		
TRACE(2) GC Ultra	TriPlus Autosampler	Ŧ			
	Add >>		<< Remove	Configure	
	Done		Help		

Figure 215. Adding a a Device to be Configured

4. Click **Configure**.

5. In the **General** tab, set the pressure units.

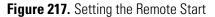
Figure 216. Setting the Pressure Unites

ISQ Series Configu	ration		×
General Mainter	ance Intervals	SmartTune	
Pressure units: Remote start:	Torr Torr Pa bar	~	
AEI Source Configured			
Con	nmunications		
	ОК	Cance	I

**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 window.

6. Set the remote start. It is used to let the ISQ 7000 MS know when the GC has started a run. When you configure the GC, you can tell it what to send out to the ISQ 7000

system. In this window, you need to make sure the value matches what you set on the GC. The default is **Active Low**.



ISQ Series Configuration	$\times$
General Maintenance Intervals SmartTune	ð
Pressure units: Torr V	
Remote start: Active Low ~	
Active Low Active High	
Communications	
OK Can	cel

7. Check **AEI Source Configured** if you are using an Advanced EI ion source on your system.

Figure 218. Configuring the Advanced El Ion Source

ISQ Series Config	uration	×	
General Mainte	nance Intervals	SmartTune	
Pressure units	: Torr	~	
Remote start:	Active	Low ~	
AEI Source Configured I			
Co	mmunications		
	ОК	Cancel	

8. Click the **Communications** button to set the network IP address of the instrument's PC communication board and assign a TCP port. The default settings are entered by the

factory and should only be changed after consulting you local IT department and obtaining special software from customer support necessary to reprogram the IP address on your instrument.

**Note** The instrument must be connected to a dedicated Ethernet port on the PC. The instrument cannot be connected through a LAN.

Figure 219. Setting the Network IP Address and the TCP Port

Communications	×
Network address:	<b>192</b> 168 1 234
ОК	Cancel

- 9. 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. As a default, the foreline pump and turbo pump oil are automatically enabled. You can monitor the progress of these settings in the Status pane of Xcalibur.
- 10. Select the Calibration Compound checkbox to enable the maintenance intervals. Then set the number of days in which you want to be reminded to refill the calibration compound. The suggested interval is 365 days.

Figure 220. Setting the Calibration Compound Maintenance Interval

15	ISQ Series Configuration			
	General Maintenance Inte	rvals	SmartTune	
	En	able	Days	
	Calibration compound:	$\checkmark$	365	
	Foreline pump oil:		125	
	Turbo pump oil/bearing:		729	
	Filament 1:		1,000	
	Filament 2:		1,000	
	lon source:		1,000	
	Multiplier:		1,000	
	Reset Al			
	ОК		Cance	1

11. Select the **Foreline Pump Oil** checkbox to enable the maintenance intervals. Then set the number of days in which you want to be reminded to check the oil. The manufacturer recommends changing the oil every 125 days. If you are using corrosive gases, such as

ammonia, you should change the oil every 30 days.

Figure 221. Setting the Foreline Pump Oil Maintenance Interval

ISQ Series Configuration				
General Maintenance Int	ervals	SmartTune		
E	nable	Days		
Calibration compound:	$\checkmark$	365		
Foreline pump oil:	$\checkmark$	125		
Turbo pump oil/bearing	:	729		
Filament 1:		1,000		
Filament 2:		1,000		
lon source:		1,000		
Multiplier: 1,000				
Reset A	NI			
OK Cancel				

12. Select the **Turbo Pump Oil/Bearing** checkbox to enable the maintenance reminder. Then set the number of days in which you want to be reminded to check the oil. The manufacturer recommends changing the oil cartridge every 730 days and the bearing every 1,460 days.

Figure 222.	Setting the	Turbo Pump	0 Oil Maintenance	Reminder
-------------	-------------	------------	-------------------	----------

ISQ Series Configuration					
General Maintenance	General Maintenance Intervals SmartTune				
Calibration compour	Enable Days Calibration compound: 🔽 365				
Foreline pump oil:		125			
Turbo pump oil/bea	ring: 🗸	729			
Filament 1:	Filament 1:				
Filament 2:		1,000			
lon source:		1,000			
Multiplier:	1,000				
Res	et All				
	OK	Cancel			

13. Select the Filament 1 checkbox to enable the maintenance reminder. In a leak-free system, the filament should last between 30-360 days, depending on usage.

FI	gure 223	. Setting the Fila	ment	1 Maintenai	nce Ke
19	SQ Series	Configuration			×
	General	Maintenance Inte	rvals	SmartTune	
		En	able	Days	
	Calibr	ation compound:	$\checkmark$	365	
	Foreli	ne pump oil:	$\checkmark$	125	
	Turbo	pump oil/bearing:	$\checkmark$	729	
	Filament 1:		$\checkmark$	1,000	
	Filament 2:			1,000	
	lon so	ource:		1,000	
	Multip	lier:		1,000	
		Reset All			
		ОК		Cancel	

ninder

14. Select the Filament 2 checkbox to enable the maintenance reminder. Then set the number of days in which you want to be reminded to check filament 2. In a leak-free system, the filament should last between 30-360 days, depending on usage.

Figure 224. Setting the Filament 2 Maintenance Reminder

ISQ Series Configuration			
General Maintenance Int	tervals	SmartTune	
E	nable	Days	
Calibration compound:	$\checkmark$	365	
Foreline pump oil:	$\checkmark$	125	
Turbo pump oil/bearing	: 🗹	729	
Filament 1:		1,000	
Filament 2:	$\checkmark$	1,000	
lon source:		1,000	
Multiplier:		1,000	
Reset /	Al		
0	к	Cancel	

15. Select the **Ion Source** checkbox to enable the maintenance reminder. Then set the number of days in 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.

Figure 225.	Setting the	Ion Source Cleaning	Maintenance Reminder

ISQ Series Configuration				
General	General Maintenance Intervals SmartTune			
	En	able	Days	
Calibr	ation compound:	$\checkmark$	365	
Foreli	ne pump oil:	$\checkmark$	125	
Turbo	pump oil/bearing:	$\checkmark$	729	
Filame	ent 1:	$\checkmark$	1,000	
Filament 2:		$\checkmark$	1,000	
lon so	ource:	$\checkmark$	1,000	
Multip	lier:		1,000	
	Reset A	I		
	ОК		Cancel	

16. Select the **Multiplier** checkbox to enable the maintenance reminder. Then set the number of days in which you want to be reminded to check the electron multiplier.

Figure 226. Setting the Electron Multiplier Maintenance Reminder

ISQ Series Configuration					
General Maintenance Intervals SmartTune					
En	Days				
Calibration compound:	$\checkmark$	365			
Foreline pump oil:	$\checkmark$	125			
Turbo pump oil/bearing:	$\checkmark$	729			
Filament 1:	$\checkmark$	1,000			
Filament 2:	$\checkmark$	1,000			
lon source:	$\checkmark$	1,000			
Multiplier:	$\checkmark$	1,000			
Reset All					
OK Cancel					



 Click the SmartTune tab and use the check boxes to enable Advanced AutoTune and the SmartTune application in TSQ AutoTune. You can also set the Intensity Check Pass Threshold here. See Figure 227.

Figure 227. Setting the Maintenance Intervals

ISQ Series Configuration X
General Maintenance Intervals Smart Tune
Show advanced AutoTune
✓ Show EI SmartTune ✓ Show AEI SmartTune (if configured)
Intensity check pass threshold: 80 🖨 %
OK Cancel

19. Click **OK** to return to the main Instrument Configuration window.

Device <u>T</u> ypes : All	•			
Available Devices:			Configured Devices:	
Al/AS1310	TRACE 1300 GC Series	* 	AI/AS 1310	ISQ
ISQ	TRACE GC Ultra		TRACE 1300 GC Series	
TRACE(2) GC Ultra	TriPlus Autosampler	Ŧ		
	Add >>	]	<< Remove	Configure
	Done	ן	Help	]

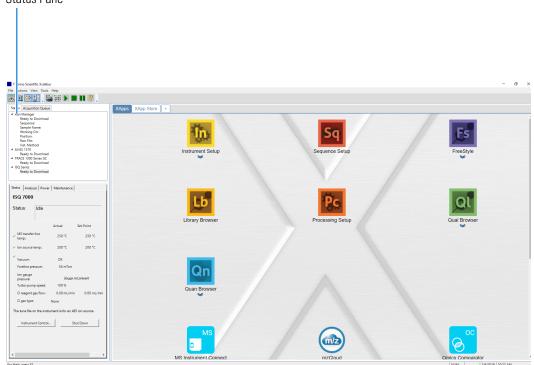
#### Figure 228. Returning to the Configuration Window

- 20. Click the **Done** button to close the Instrument Configuration utility.
- 21. You can check the status of your ISQ 7000 instrument in the Status tab of the ISQ Dashboard.

Figure 229. Checking the ISQ 7000 Instrument Status on the ISQ Dashboard

📔 ISQ 7000 Dashboard 🛛 🗛 🕹 🕹				
AutoTune Opti	ons	View T	une Report	
Air & Water / Tune		Au	itoSIM	
Method Edito	Method Editor		alibur	
Instrument Cor	Instrument Control		it Down	
	ę	}		
Status Analyzer Po	wer Mainte	enance		
Status: Idl	е			
	Actual	Set Point		
Instrument tune file – AEI source			·	
Temperatures MS transfer line temp:	250 °C	250 °C		
<ul> <li>Instansienine temp.</li> <li>Ion source temp:</li> </ul>	250°C 199 ℃	200 °C		
Pressures				
🗸 Foreline pressure:	53 mTorr			
Misc				
Vacuum: Turbo pump speed:	OK 100 %			
CI reagent gas flow				
CI gas type	None			

22. In the **Status Pane** of the Xcalibur Roadmap window, you can check the status of all your instruments. See Figure 230.



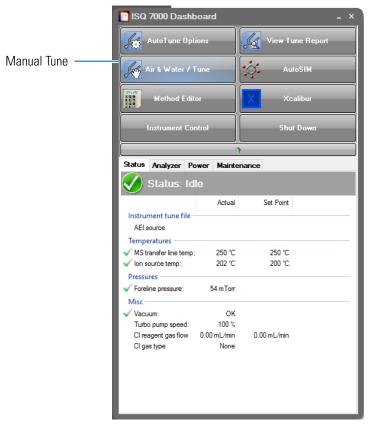
**Figure 230.** Checking All Instrument Statuses in Xcalibur Status Pane

# **Upgrading the Software**

Once you purchase optional software for your system, you will receive a product key from the factory. This product key is required to license your software. Follow the instructions below to enter the product key and activate your software.

**Note** See the *ISQ and TSQ GC-MS Spare Parts Guide* for information about ordering software upgrades.

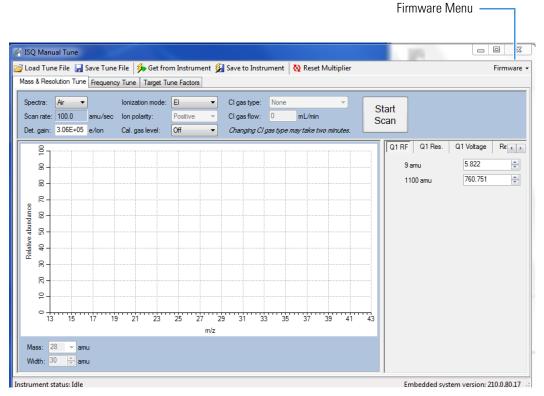
1. Click **Air & Water/Spectrum** on the ISQ 7000 Dashboard to open the manual tune utility. See Figure 231.



#### Figure 231. ISQ 7000 Dashboard

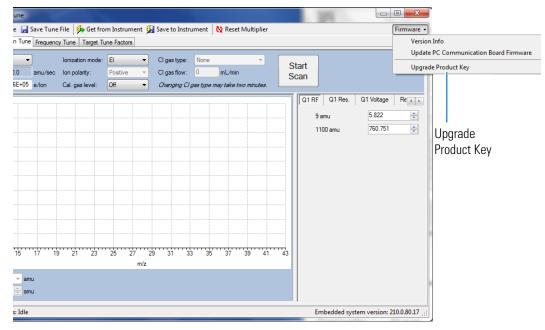
2. The ISQ 7000 Manual Tune application opens. Click **Firmware** on the upper right-hand side of the screen to open the firmware menu. See Figure 232.

#### Figure 232. ISQ Manual Tune Home



3. The firmware menu opens. Select Update Product Key as shown in Figure 233.

Figure 233. Selecting the Upgrade Product Key Option



4. The **ISQ Product Key** window opens. Enter your product key into the **New Product Key** box as shown in Figure 234.

#### Figure 234. Entering the Product Key

New Product Key Box

ISQ Manual Tune       Load Tune File       Mass & Resolution Tune       Frequency Tune       Target Tune	· · ·	E E X
R         Cu           Ne         Cu           Nu         Cu           Nu         Cu           Nu         Cu           Nu         Cu     <	stive CI gas flow: ImL/min S Product Key Iml ent product key: ent feature set: No premium features. w product key: W feature set: Load product key to instrument	tart can 9 amu 5.822 (*) 1100 amu 760.751 (*)
Instrument status: Idle		Embedded system version: 210.0.80.17

# Index

# A

acquisition methods, purpose of, 67, 112 AEI Tunes 39 AutoSIM creating a method, 134, 147 getting a license key, 135, 148 selecting SIM ions, 142, 157 SIM ion settings, 138, 152 submitting a study, 140, 154 using, 133 AutoTune, modifying, 188 AutoTune, using, 30, 49

## B

baseline (troubleshooting) drifting, 218 baseline issues, investigating, 218

# C

centroided data, about, 70, 115 chromatograms, viewing, 175 chromatographic separation, changing, 185 CI gas types, selecting, 191 CI mode, using, 68, 113 column leak checking, 24 temperature, 26 compliance FCC viii regulatory vii WEEE ix contaminants, common, 216 contamination, indications, 215 creating a method 59

# D

data result issues, investigating, 223 data, verifying, 175 diagnostics, selecting tests to run, 199 documentation survey xxvi

#### Ε

EI tune type, about, 37 electromagnetic compatibility viii EMC compliance vii

#### F

FCC compliance viii filament voltage, setting, 192 filament, turning off and on, 72, 117 fingerprint, indications, 216 flow rate, verifying, 2 full-scan mode, about, 70, 115

#### G

gas flow rate, 2 tank pressure, 3 GC carrier gas, 2 creating a method, 85, 108 powering on, 2 ghost peaks, troubleshooting, 222

#### I

instrument method, selecting, 166 ion source temperature, setting, 68, 112, 193 ISQ confirming it is working, 1 exploring data, 175 optimizing methods, 185 powering on, 1 reconfiguring, 225 running a sample, 161 troubleshooting, 203 ISQ 7000 changing the column, 5 creating a method, 59 tuning, 29 ISQ AutoTune, about, 188 ISQ Series AutoTune, about, 30

## L

leak, indications, 216 leaks, checking for, 24 lights Heater, 4 Vacuum, 4 lights on ISQ power, 1

#### Μ

maintenance, settings, 229, 234 mass range, narrowing, 186 methods acquisition, 67, 112 creating, 59 optimizing, 185 purpose of, 59

## Ν

NCI mode, using, 68, 113

#### 0

opening a sequence, 163

#### Ρ

PCI mode, using, 68, 113 peak height, minimum, 68, 113 peak issues, investigating, 220 peaks (troubleshooting) broadening, 220 fronting, 220 ghost, 222 none, 222 tailing, 221 power, checking, 1 preparing samples, 161 pressure (carrier gas tank), checking, 3 profiled data, about, 70, 115

#### R

raw file, selecting, 176 reconfiguring the ISQ, 225 regulatory compliance vii results (troubleshooting) low reproducibility of peaks area, 223 poor sensitivity, 224 retention times, 224 retention times, troubleshooting, 224 routine maintenance causes, 214

#### S

safety standards vii samples preparing, 161 running, 161 scan rate, optimizing, 186 scans, running, 69, 113 sensitivity, troubleshooting, 224 sequences about, 162 customizing, 170 opening, 163 prioritizing, 172 running, 162 saving, 168 SIM mode, about, 70, 115 SmartTune using, 45 solvents choosing, 161 purpose of, 161 source plug 18 survey link xxvi

#### Т

tailing peaks, troubleshooting, 221 temperature of the ion source, 68, 112 temperature of the transfer line, 187 temperature of transfer line, 67, 112 temperature, checking, 4 transfer line temperature, setting, 67, 112 transfer line, about, 67, 112 troubleshooting about, 203 baseline issues, 218 carrier gas tank pressure, 3 GC display is blank, 2 peak issues, 220

results issues, 223 transfer line temperature, 68, 112 tune report, 44 tune types adding, 37 tune reports example report, 44 printing, 44 settings, 44 tune types AEI Tunes 39 CI- tune, 35 CI+ tune, 37 EI full tune, 32 tuning 29 using Autotune, 30, 49 with ISQ 7000 AutoTune, 30

# U

user name, about, 172 using, 18

## V

vacuum, checking, 4 V-lock source plug 18 voltage of filament, setting, 192

#### W

WEEE compliance ix