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ISQ 7610

Mass Spectrometers

User Guide

1R120621-0003 Revision A April 2022

ThermoFisher SCIENTIFIC

Release history: Revision A, March 2022.

Software version: ISQ Series 5.0 and later.

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

Contents

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Declaration

Manufacturer: Thermo Fisher Scientific

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

- installation,
- recalibration, and
- changes and repairs

have been carried out by authorized personnel and if:

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

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

Regulatory Compliance

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

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

EMC and Safety Standards

- 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, ISQ 7000, ISQ 7610 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, TSQ 9000, and TSQ 9610 standards: EMC EN 61326-1:2013. Safety IEC 61010-1:2010, IEC 61010-2-010:2014, IEC 61010-2-081:2015.

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FCC Compliance Statement

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

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

Notice on Lifting and Handling of Thermo Scientific Instruments

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

Notice on the Proper Use of Thermo Scientific Instruments

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

Notice on the Susceptibility to Electromagnetic Transmissions

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

EU REACH Statement

he European Commission promulgated legislation that covers the registration, evaluation, authorization and restriction of chemicals within the European Union community under (EC) No 1907/2006. This regulation is commonly known as REACH. Thermo Fisher Scientific is committed to meeting all compliance obligations under REACH. As per Article 33 of the Regulation, this product may include items which contain more than 0.1% by weight of some SVHC Candidate Substance. Some electronic parts and copper alloys can contain lead.

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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 2012/19/EU. It is marked with the following symbol:

Thermo Fisher Scientific is registered with B2B Compliance (B2Bcompliance.org.uk) in the UK and with the European Recycling Platform (ERP-recycling.org) in all other countries of the European Union and in Norway.

If this product is located in Europe and you want to participate in the Thermo Fisher Scientific Business-to-Business (B2B) Recycling Program, send an email request to weee.recycle@thermofisher.com with the following information:

- WEEE product class
- Name of the manufacturer or distributor (where you purchased the product)
- Number of product pieces, and the estimated total weight and volume
- Pick-up address and contact person (include contact information)
- Appropriate pick-up time
- Declaration of decontamination, stating that all hazardous fluids or material have been removed from the product

RoHS

For information about the Restriction on Hazardous Substances (RoHS) Directive for the European Union, search for RoHS on the Thermo Fisher Scientific European language websites.

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Declarations of Conformity

-Original-

EU Declaration of Conformity
EU-Konformitätserklärung

CE

ThermoFisher SCIENTIFIC

Thermo Fisher Scientific
2215 Grand Avenue Parkway
Austin, Texas 78728
USA

We hereby declare that the following products

Designation:	**Mass Spectrometer**
Model:	**Thermo Scientific ISQ Series** *ISQ7K Series, ISQ 7610 Series*

fulfill all the relevant requirements of the following directives:

Low Voltage Directive	**2014/35/EU**
Electromagnetic Compatibility Directive	**2014/30/EU**
RoHS Directive	**2011/65/EU and (EU) 2015/863**

The following relevant harmonized standards were used:

EN 61010-1:2020-03	**EN 61326-1:2013-07**

Person authorized to compile the technical file:

Brody Guckenberger
(Director, Applied Research)
Thermo Fisher Scientific

Signature	Austin, March 4, 2022 **Date**

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-Original-

UK Declaration of Conformity

UK CA

ThermoFisher SCIENTIFIC

Thermo Fisher Scientific
2215 Grand Avenue Parkway
Austin, Texas 78728
USA

Declares, under sole responsibility, that products

Designation:	**Mass Spectrometer**
Model:	**Thermo Scientific ISQ Series** *ISQ7K Series, ISQ 7610 Series*

as originally delivered complies with the essential requirements of the following applicable UK Regulations:

Electrical Equipment (Safety) Regulations	**2016**
Electromagnetic Compatibility Regulations	**2016**
The Restriction of the Use of Certain Hazardous Substances in Electrical and Electronic Equipment (ROHS) Regulations	**2012**

and complies with the following harmonized standards and other technical specifications:

BS EN 61010-1:2010+A1:2019	**BS EN 61326-1:2021**

Signed for and on behalf of: Thermo Fisher Scientific:

Brody Guckenberger
(Director, Applied Research)
Thermo Fisher Scientific

Signature

Austin, March 4, 2022

Date

Preface

This guide contains detailed information about how to use the Thermo Scientific™ ISQ™ 7610 single quadrupole GC-MS system. The ISQ 7610 system is designed to stay cleaner, longer, to maximize your instrument's uptime and improve your lab's productivity. The heated ion volume, lens stack, and ion optics path in the ISQ 7610 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

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 7610 includes Help and these manuals as PDF files:

- *ISQ 7610 Preinstallation Requirements Guide* PN 1R120621-0001
- *ISQ 7610 Hardware Manual* PN 1R120621-0002
- *ISQ 7610 User Guide* PN 1R120621-0003
- *ISQ and TSQ GC-MS Spare Parts Guide* PN 1R120621-0004
- *Direct Probe System User Guide* PN 1R120505-0006

❖ To view product manuals

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

❖ To open Help

- In Xcalibur software, from the ISQ 7610 window in the Method Editor, choose **Help > ISQ 7610 Help**.
- In Chromeleon software, ISQ 7610 Help is available through the Method Editor.
- 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

Your ISQ 7610 data system must meet these minimum requirements.

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

[a] Check release notes for compatibility with ISQ Series instrument control software.

[b] 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.

Symbol	Descriptor
	**BIOHAZARD:** Indicates that a biohazard *will*, *could*, or *might* occur.
	**BURN HAZARD:** Alerts you to the presence of a hot surface that *could* or *might* cause burn injuries.
	**ELECTRICAL SHOCK HAZARD:** Indicates that an electrical shock *could* or *might* occur.
	**FIRE HAZARD:** Indicates a risk of fire or flammability *could* or *might* occur.
	**FLAMMABLE GAS HAZARD:** 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 *could* or *might* cause physical injury.
	**GLOVES REQUIRED:** Indicates that you must wear gloves when performing a task or physical injury *could* or *might* occur.
	**HAND AND CHEMICAL HAZARD:** Indicates that chemical damage or physical injury *could* or *might* occur.
	**INSTRUMENT DAMAGE:** Indicates that damage to the instrument or component *might* occur. This damage might not be covered under the standard warranty.
	**LIFTING HAZARD:** Indicates that a physical injury *could* or *might* occur if two or more people do not lift an object.

Symbol	Descriptor
	**MATERIAL AND EYE HAZARD:** Indicates that eye damage *could* or *might* occur.
	**RADIOACTIVE HAZARD:** Indicates that exposure to radioactive material *could* or *might* occur.
	**READ MANUAL:** Alerts you to carefully read your instrument's documentation to ensure your safety and the instrument's operational ability. Failing to carefully read the documentation *could* or *might* put you at risk for a physical injury.
	**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
	**RISQUE D'INCENDIE** : indique qu'un risque d'incendie ou d'inflammabilité peut ou pourrait survenir.
	**RISQUE DE GAZ INFLAMMABLE** : 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.
	**GANTS REQUIS** : indique que vous devez porter des gants pour effectuer une tâche, sans quoi une blessure physique peut ou pourrait survenir.
	**RISQUE PHYSIQUE ET CHIMIQUE** : indique que des dommages chimiques ou une blessure physique peuvent ou pourraient survenir.
	**DOMMAGES À L'INSTRUMENT** : indique que l'instrument ou le composant pourrait subir des dommages. Ces dommages pourraient ne pas être couverts pas la garantie standard.
	**RISQUE SOULÈVEMENT** : indique qu'une blessure physique peut ou pourrait survenir si un objet n'est pas soulevé par deux personnes ou plus.
	**RISQUE MATÉRIEL ET YEUX** : indique que des dommages aux yeux peuvent ou pourraient survenir.
	**RISQUE RADIOACTIF** : indique qu'une exposition à des matériaux radioactifs peut ou pourrait survenir.
	**CONSULTER LE MANUEL** : vous avertit de lire attentivement 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?"

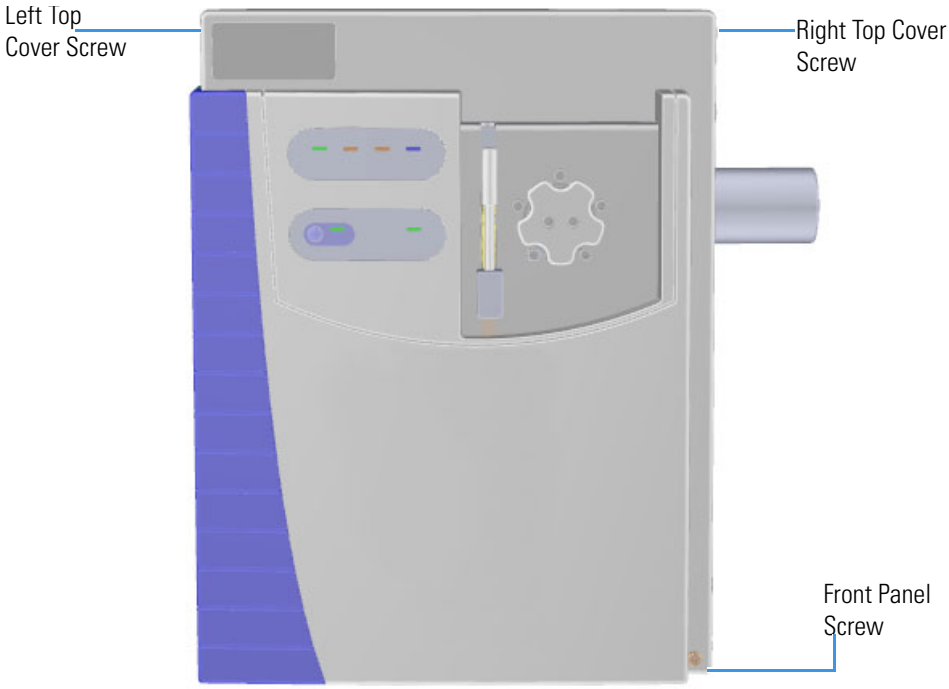
"What hydrogen hazards associated with this project have the potential to result in the worst consequences?"

- 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 7610 Mass Spectrometers

To use hydrogen with the ISQ 7610 instrument, you must always shut off the GC carrier gas before venting or turning off the ISQ 7610 instrument. There are three hydrogen safety screws on the ISQ 7610 instrument that **must** be in place. These are attached to your instrument at the factory.

Figure 1. Hydrogen Safety Screws on the ISQ 7610 Instrument



Make sure all the covers and panels of the ISQ 7610 instrument are firmly attached before powering on the ISQ 7610 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**

Send an e-mail message to the Technical Publications Editor at techpubs-austin@thermofisher.com.

1

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 supply 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

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 7610 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 7610 system is powered on, make sure the Power light on the front panel is solid green. If it is not lit, the ISQ 7610 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.

The mass spectrometer (MS) with foreline pump (system mech pump) must be on its own dedicated circuit with supply voltage matching the pump requirements.

Figure 1. Checking the Power on the ISQ LT System

To confirm that a TRACE 1600 GC is powered on, make sure that power light on the status panel is solid green. To confirm that a TRACE 1610 GC is powered on, see if the touchscreen main menu has appeared. To power-on the TRACE 1600 or 1610 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.

❖ To check the carrier gas flow rate

1. Access the carrier gas menu. On the TRACE 1610 GC, choose **Instrument Control** and then **Front/Back Inlet**. On a TRACE 1600 GC running Xcalibur open the Xcalibur software by clicking on the Xcalibur icon on the computer desktop. On the Xcalibur roadmap, select the TRACE 1600 from the instrument list in the side panel. This opens the **Status Panel**. On a TRACE 1600 running Chromeleon software, open the instrument method on Chromeleon Console to view the column flow under **GC Inlets**.
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 Supply Pressure

Make sure you have enough pressure in the carrier gas supply 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.

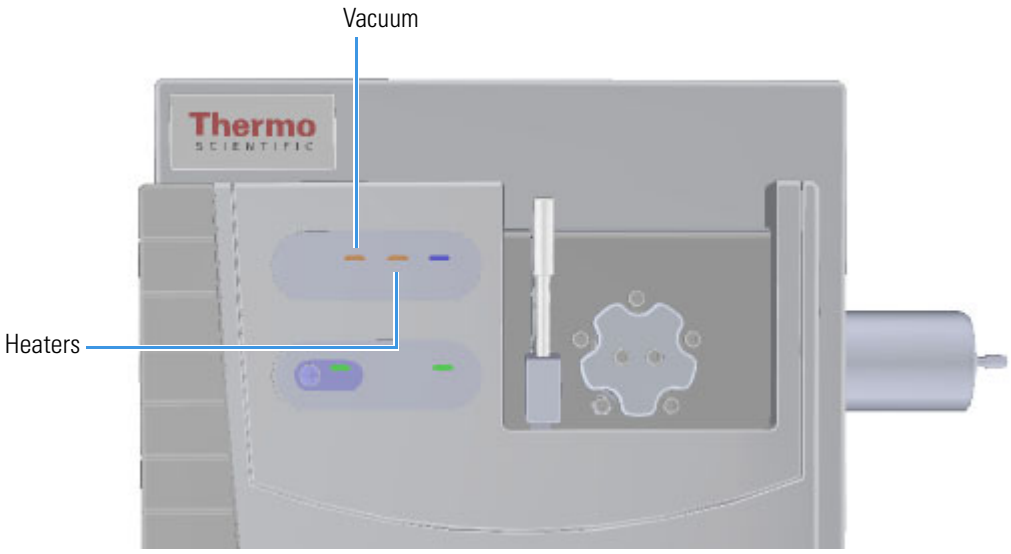
1. Locate your carrier gas supply. It might be in a different room, depending on how your lab is set up.
2. Look at the pressure gauge on the supply.
3. Ensure the pressure is more than 500 psi at the primary (or first) regulator stage. If it is not, you may want to replace the supply if you have to 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 7610 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 7610 Instrument



To check the temperature, look at the **Heaters** light. When the **Heaters** light is a solid green, the ISQ 7610 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.

Changing the Column

The ISQ 7610 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.

Tip If your system includes TRACE 1610 GC, there are videos included on the HMI touchscreen for changing the column with the various ion source types. Access the videos by clicking **Maintenance | Videos** on the HMI.

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 µm for a column with an ID of 0.25 mm. Other common film thicknesses are 0.1, 0.5, and 1.0 µm.
- **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 7610 system with no VPI or an AEI ion source" on page 7. If your ISQ 7610 MS has a vacuum probe interlock (VPI) and the ExtractaBrite ion source is installed, see "Replacing a column on a ISQ 7610 system with a VPI and an ExtractaBrite ion source" on page 17.

Replacing a column in a ISQ 7610 system with no VPI or an AEI ion source

If your ISQ 7610 system has no VPI or has an AEI ion source installed on the system, follow the instructions below to replace the column.

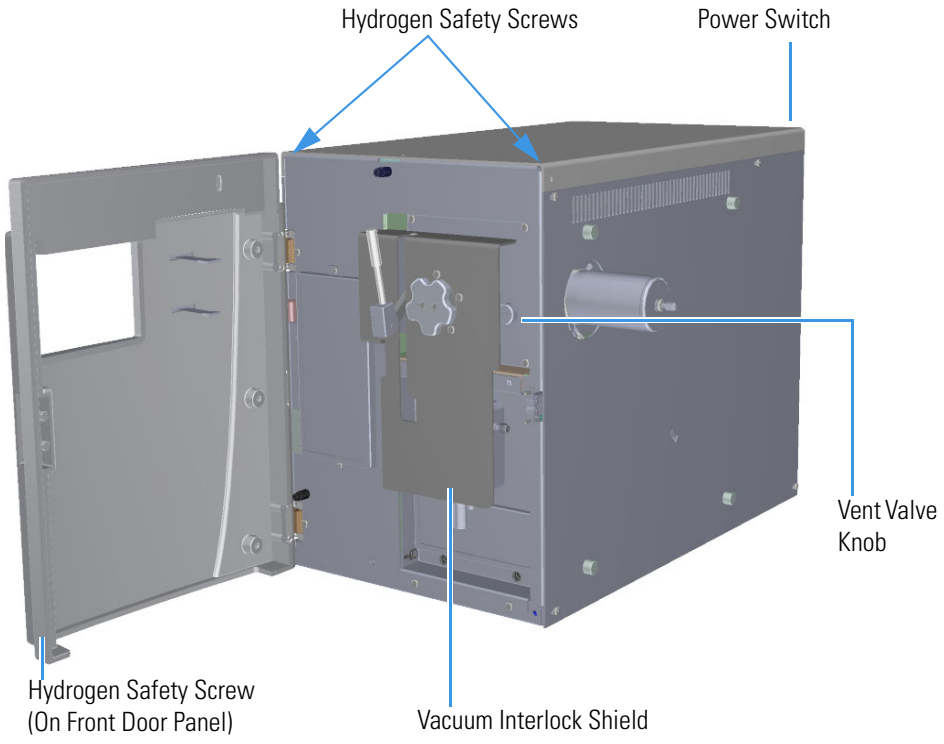
❖ To replace the factory-installed column in the ISQ 7610 system in a system with no VPI or with an AEI ion source installed

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 7610 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 7610 system.
3. On the left side of the instrument, push down on the power switch to power-off the ISQ 7610 system.

Figure 3. Powering Off the ISQ 7610 Instrument



4. Open the front door of the instrument.
5. Look behind the right side of the vacuum interlock shield and twist the vent valve knob one and a half times in a counterclockwise direction to open the vent.
6. Remove the current column 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.
7. 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™ 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 7610 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.

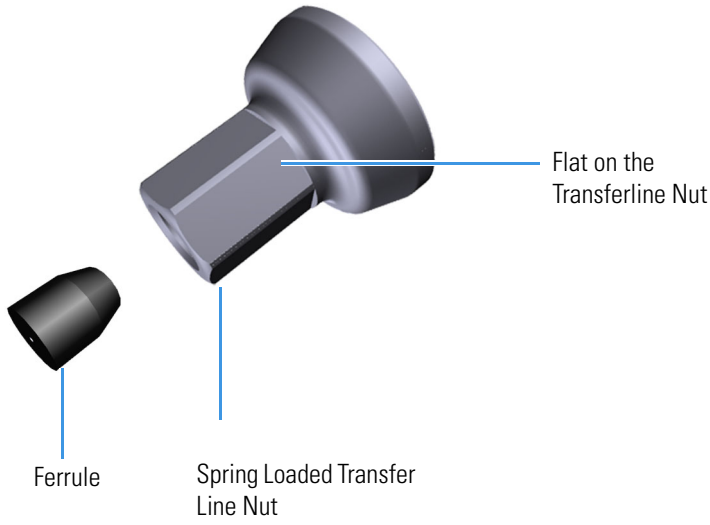
ATTENTION RISQUE DE BRÛLURE : l'injecteur, les détecteurs, le four et la ligne de transfert peuvent être chauds. Laissez-les refroidir avant de les toucher.

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

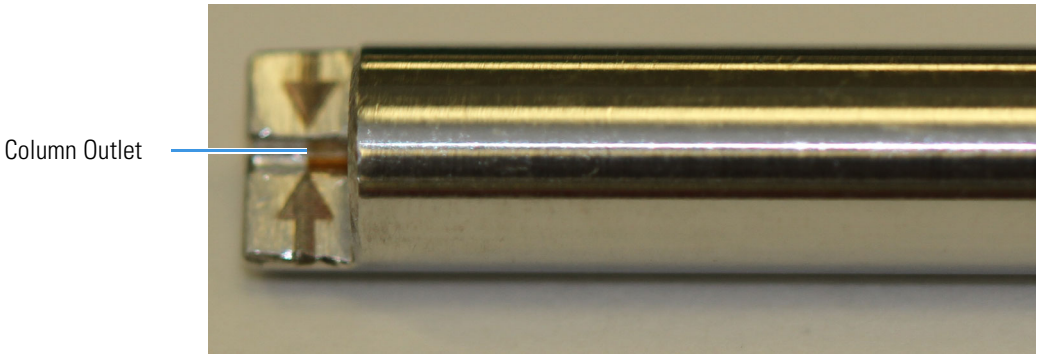


8. Insert the column into the measuring tool, which is in the MS Toolkit (See Figure 5), so that it is even with the lines at the end of the column. Figure 6 indicates proper positioning of the column in the tool for accurate measuring.
9. Use a scoring wafer to score and break the column. Use a magnifying 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

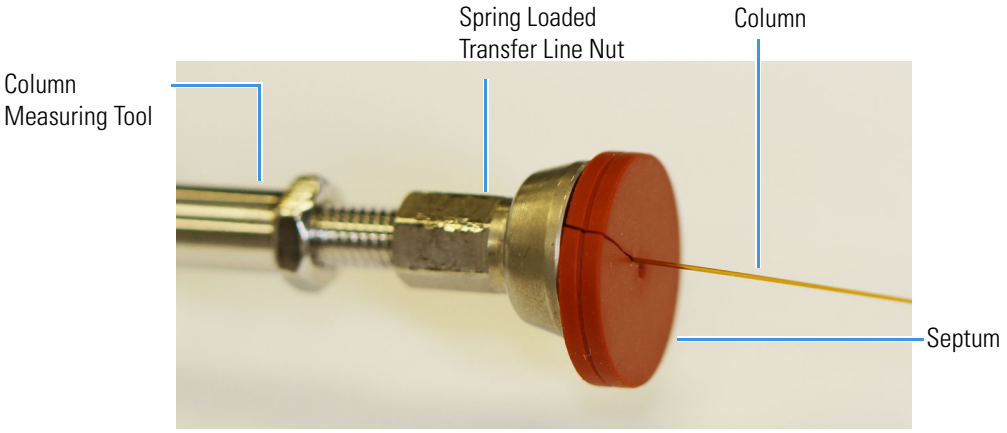
11. While holding the column measuring tool steady, tighten the spring loaded transfer line nut with a 1/4” wrench until the column just stops moving in the ferrule.
12. Turn the spring loaded transfer line nut 1 flat (60°) backward so the column is able to move in the ferrule with slight resistance.
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



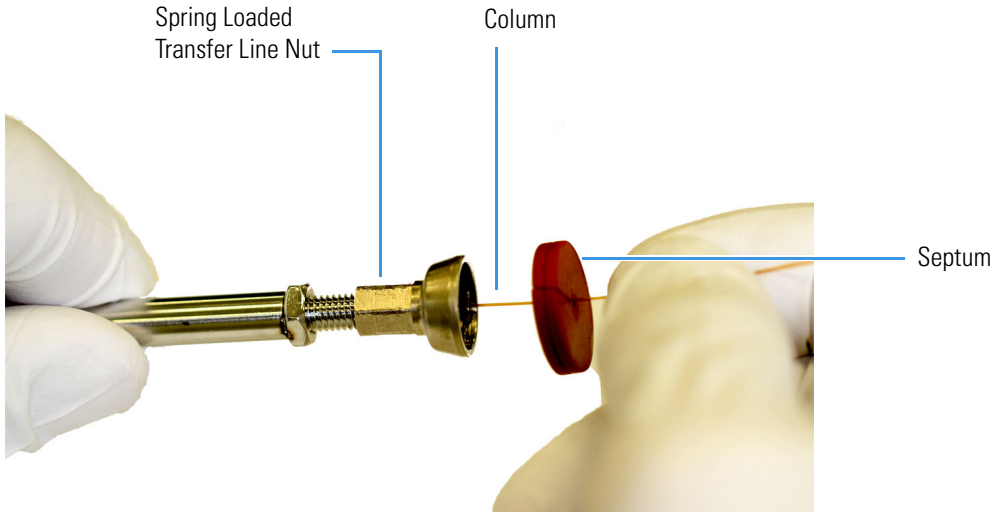
14. Place a septum with a notch cut into it behind the transfer line nut. The septum marks the place on the column where it should exit the nut.

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

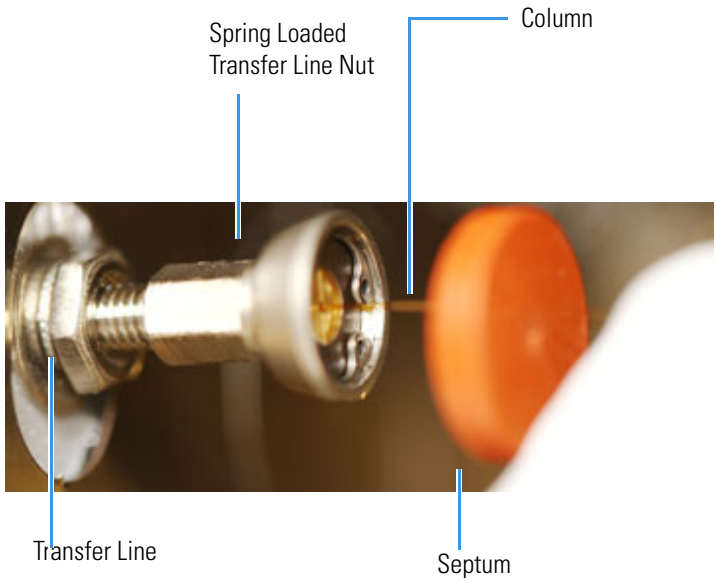


16. Loosen the transfer line nut from the column measuring tool.
17. Remove the column, transfer line nut and ferrule from the column measuring tool, making sure not to move the septum from its location on the column.

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.

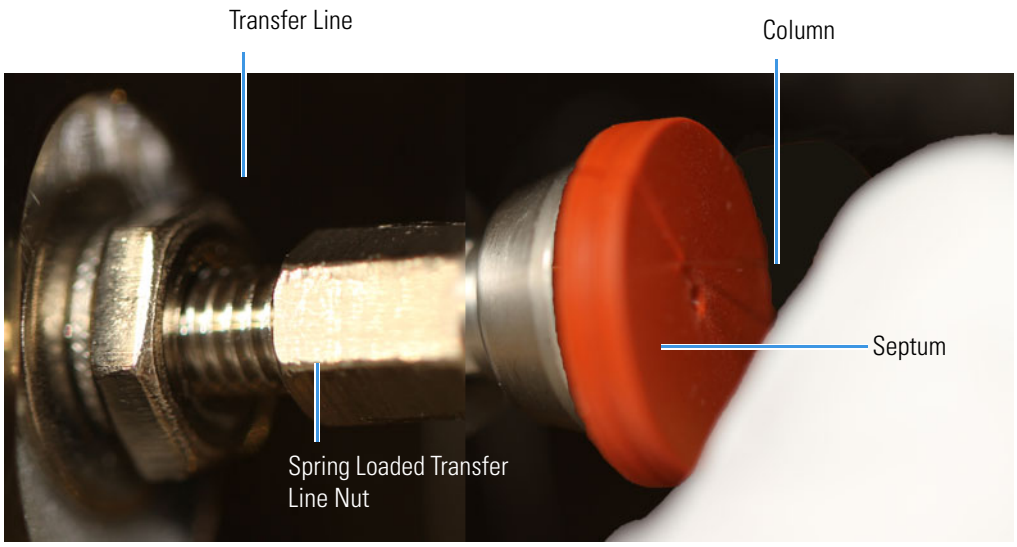
18. Insert the column into the transfer line.

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. Close the GC door.
    b. Raise the oven temperature to the maximum temperature you will operate the GC.
    c. Wait 10 minutes.
    d. 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 7610 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 7610system 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.

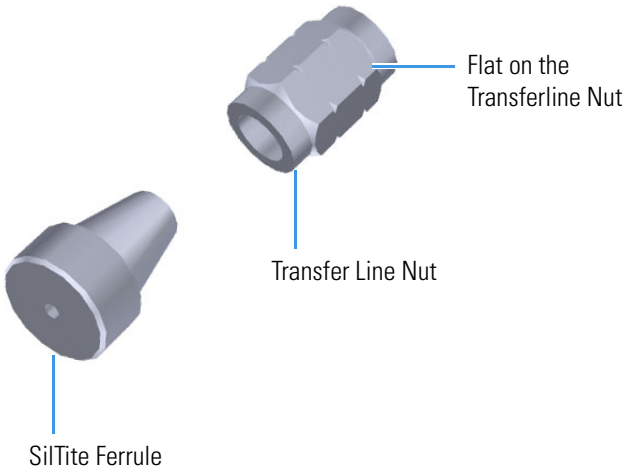
Note Wear clean, lint- and powder-free gloves when you handle the column and transfer line ferrule.

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 ≥ 290 °C (554 °F), Thermo Fisher Scientific recommends using a spring loaded transfer line nut with a graphic Vespel ferrule or a SilTite™ 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

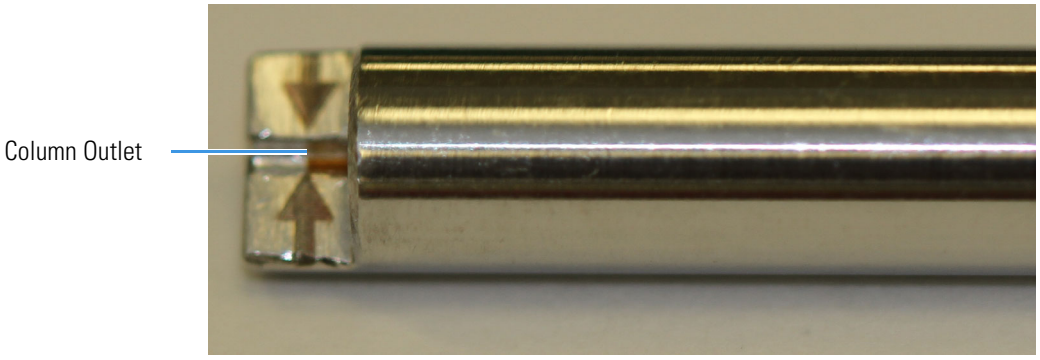


7. Insert the column into the measuring tool, which is in the MS Toolkit (See Figure 12), so that it is even with the lines at the end of the column. Figure 13 indicates proper positioning of the column in the tool for accurate measuring.
8. Use a scoring wafer to score and break the column. Use a magnifying 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

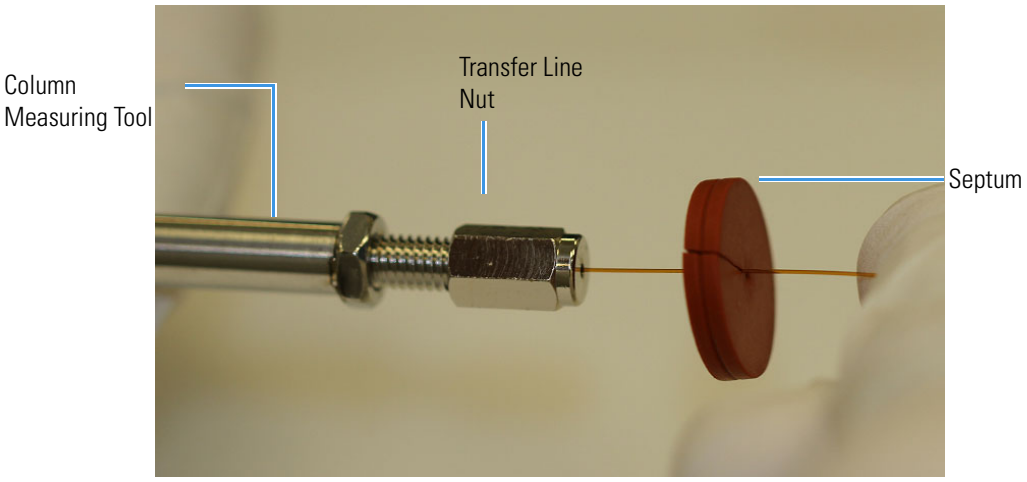
10. While holding the column measuring tool steady, tighten the transfer line nut with a 1/4" wrench until the column just stops moving in the ferrule.
11. Turn the transfer line nut 1 flat (60°) backward so the column is able to move in the ferrule with slight resistance.
12. Line up the outlet of the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

Figure 13. Lining Up the Column in the Column Measuring Tool



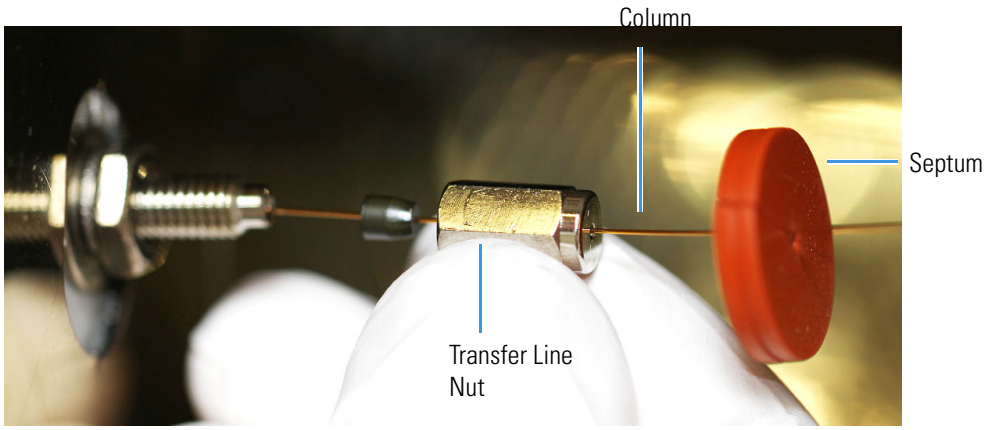
13. Place a septum with a notch cut into it behind the transfer line nut. The septum marks the place on the column where it should exit the nut.

Figure 14. Positioning the Septum



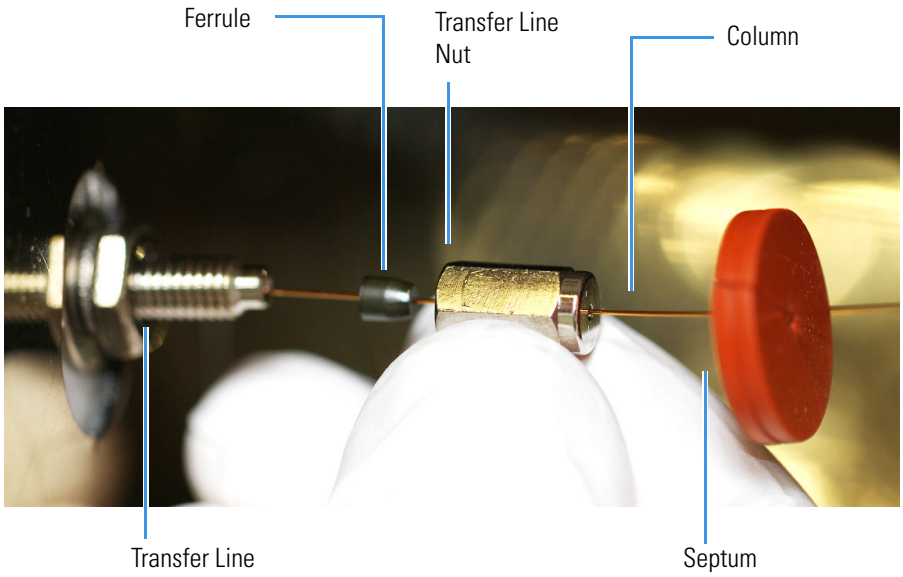
14. Pull the column back from the transfer line nut. Do not move the septum from its position on the column.

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



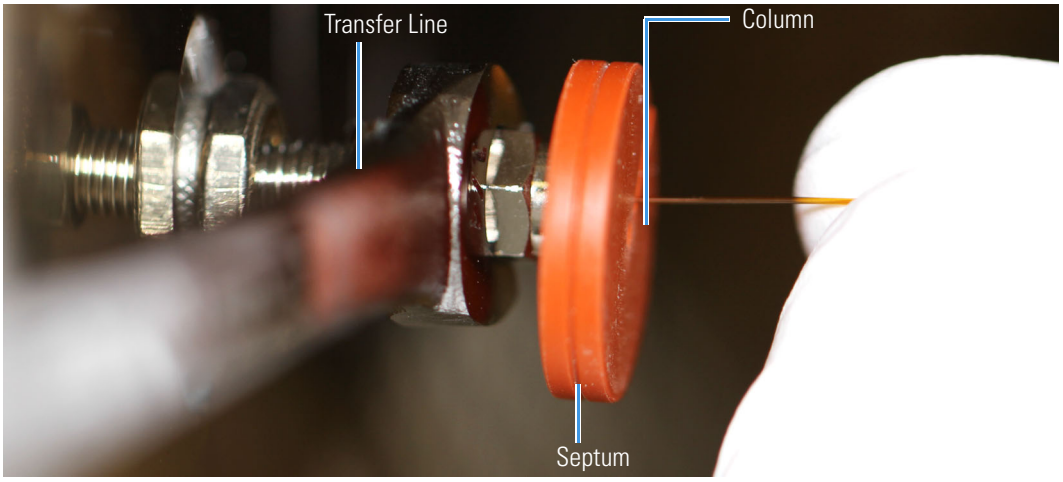
15. Loosen the transfer line nut from the column measuring tool.
16. Remove the column, transfer line nut and ferrule from the column measuring tool, making sure not to move the septum from its location on the column.
17. Insert the column into the transfer line.

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.

Figure 17. Positioning the Column in the Transfer Line



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. Close the GC door.
    b. Raise the oven temperature to the maximum temperature you will operate the GC.
    c. Wait 10 minutes.
    d. 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.

    e. 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 7610 instrument.

Once the ISQ 7610 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 7610 system with a VPI and an ExtractaBrite ion source

If your ISQ 7610 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 7610 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 7610 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 7610 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.

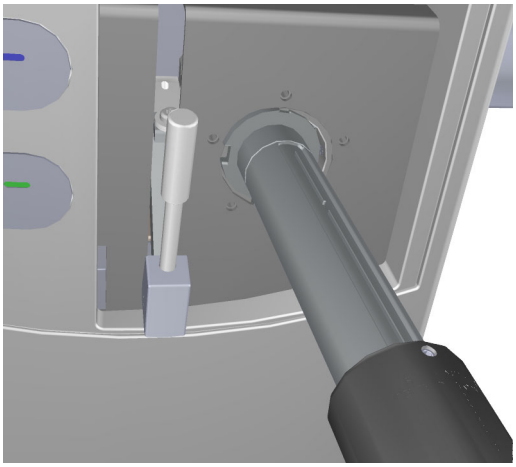
c. The **Evacuate** light will begin to flash green, and should continue to flash green for approximately 20 seconds.

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, 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.

Figure 20. V-lock Source Plug Inserted Correctly

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

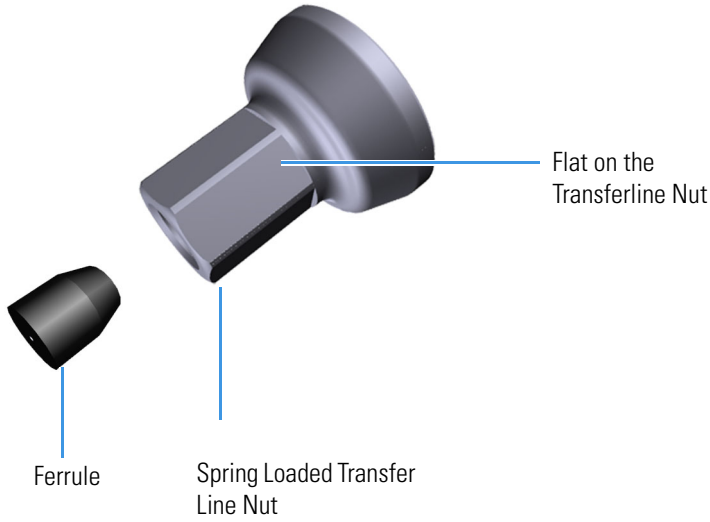
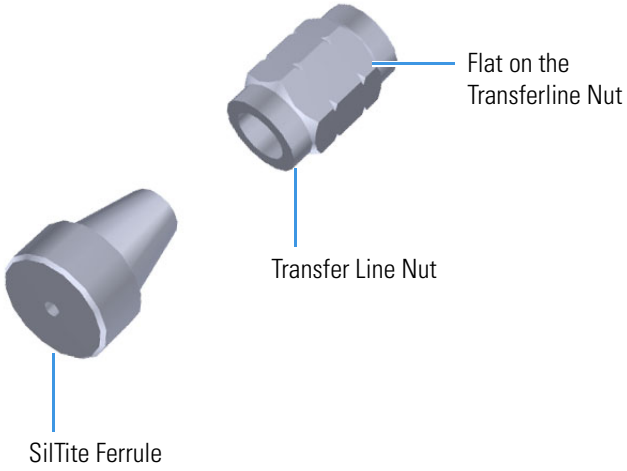


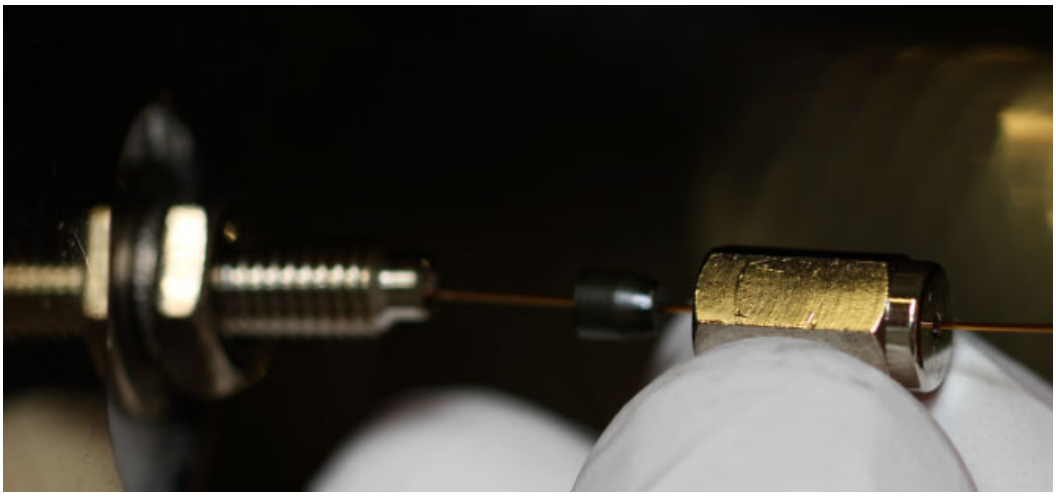
Figure 22. Transfer Line Nut and SilTite Ferrule Orientation



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.

Figure 23. Installing the Column into the MS Transfer Line



27. Loosen the nut ¼ turn and gently push the column into the MS transfer line until it just touches the V-lock source plug.
28. Pull the column ½ to 1 mm away from the V-lock source plug and tighten the nut ½ 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 7610 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 as described in "Replacing a column in a ISQ 7610 system with no VPI or an AEI ion source" on page 7 or "Replacing a column on a ISQ 7610 system with a VPI and an ExtractaBrite ion source" on page 17.

   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:

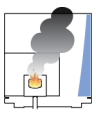
   a. On the TRACE 1610, select the **Leak Check** icon in the **Maintenance** menu. Otherwise, perform the leak check through the Chromatography Data System. Refer to the *TRACE 1600 and TRACE 1610 Series GC User Guide* for instructions.

   b. Start the leak check.

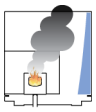
      The split and purge valves of the selected channel are automatically closed, and the channel is pressurized with carrier gas to the leak check setpoint.

      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.



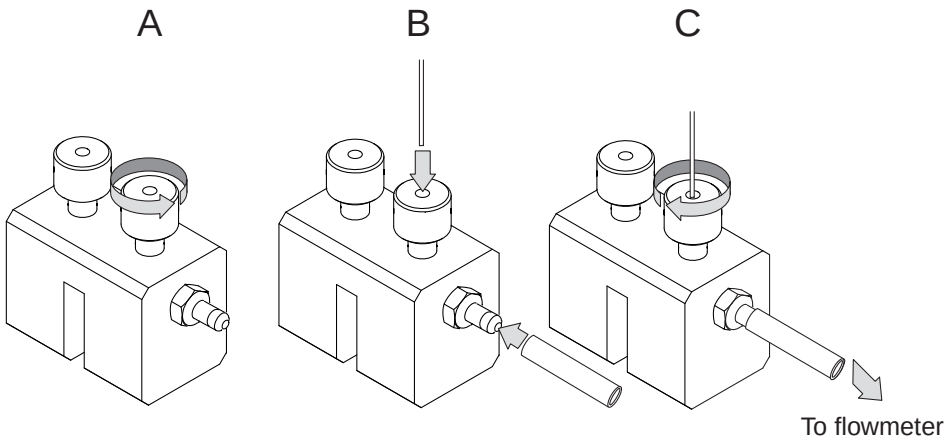
ATTENTION DOMMAGES À L'INSTRUMENT : ne laissez pas le connecteur du débitmètre de la colonne dépasser les 80 °C (176 °F). Dans le cas contraire, il va fondre et endommager l'instrument.

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.

Figure 24. Column Flowmeter Connector



b. Connect the flowmeter to the dedicated fitting on the column flowmeter connector.

c. If you have a TRACE 1610, select the **Back** or **Front Column** icon in the **Configuration** menu. Otherwise, perform the column evaluation through the Chromatography Data System. See the *TRACE 1600 and TRACE 1610 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

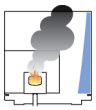
leak or broken column using the leak detector. The K-factor is a measured resistance for the column. A K-factor that is too low may indicate a leak in the system, while a K-factor that is too high may indicate a blockage.

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.

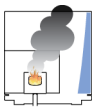
6. Disconnect the column flowmeter:
   a. Disconnect the column from the column flowmeter connector.
   b. Remove the clear plastic component, including its fittings, from the oven and set them aside.
   c. Close the GC door.
7. If necessary, condition the column before inserting it into the ISQ 7610 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.

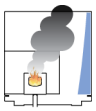


ATTENTION DOMMAGES À L'INSTRUMENT : les matières rejetées par la colonne (ressuage de la colonne) lors du conditionnement peuvent contaminer la source d'ions si la colonne est insérée dans la ligne de transfert lors de la phase à haute température du conditionnement.

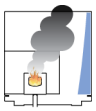
WARNING FIRE HAZARD: Do not use hydrogen as the carrier gas for conditioning your column. It could vent into the oven and present an explosion hazard.

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.

Tuning the ISQ 7610 Mass Spectrometer

Tuning will improve the performance of your ISQ 7610 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 7610 instrument (system is hot), it takes approximately 30 minutes for the components to reach vacuum and temperature.

IMPORTANT Be sure to give the ISQ 7610 instrument enough time to stabilize. Otherwise, you may see mass drift, mass spectral changes, or changes in the fragmentation of your data.

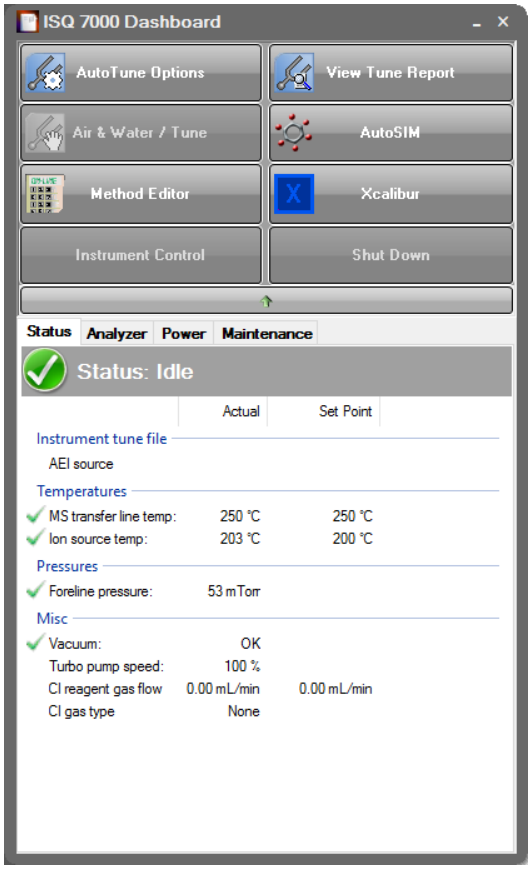
Contents

Accessing ISQ 7610 Tuning

❖ To access ISQ 7610 AutoTune in Xcalibur Software

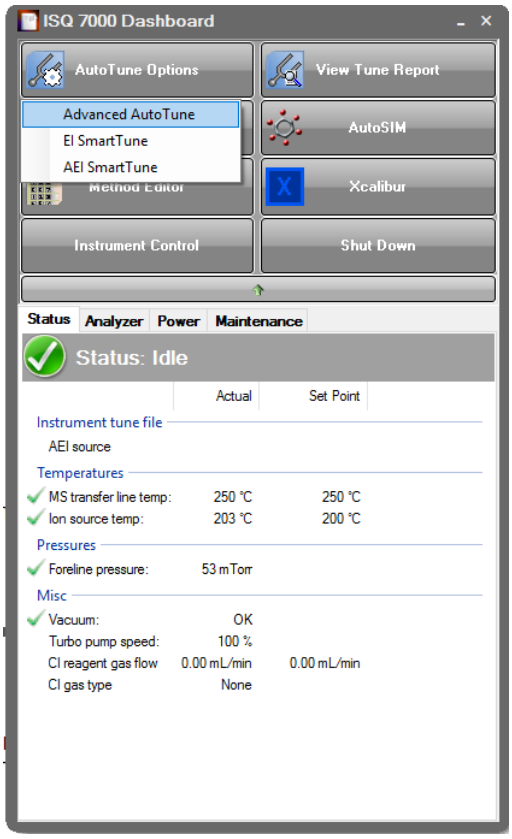
Open the ISQ 7610 Dashboard and click **AutoTune Options** to open the AutoTune window.

Figure 25. Accessing ISQ 7610 AutoTune from the ISQ 7610 Dashboard



1. 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.

Figure 26. Selecting from AutoTune Options

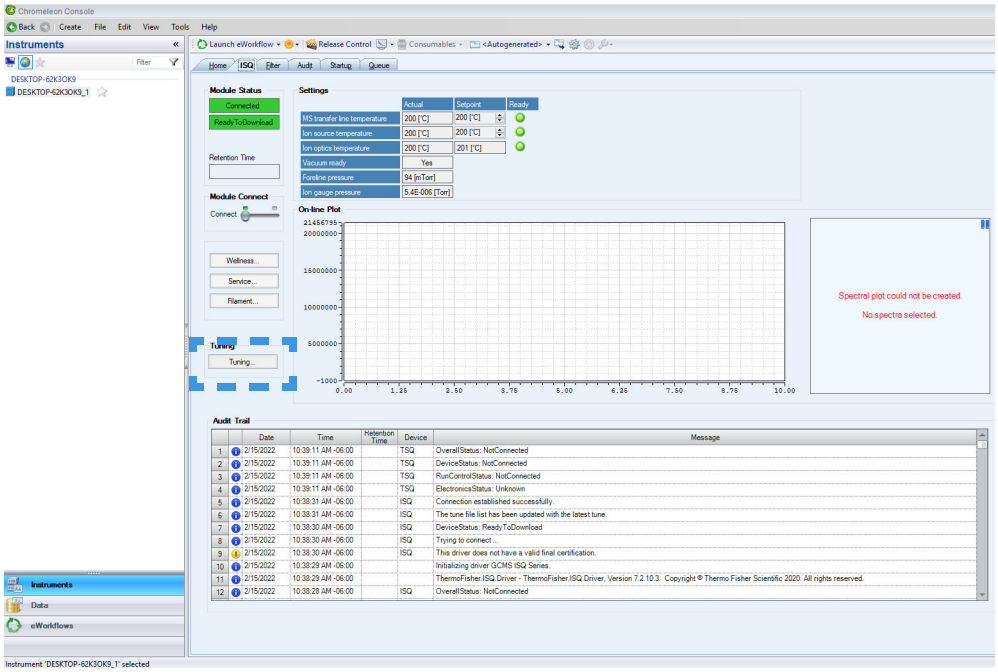


The list of available tune types opens.

❖ To access ISQ 7610 Tuning in Chromeleon Software

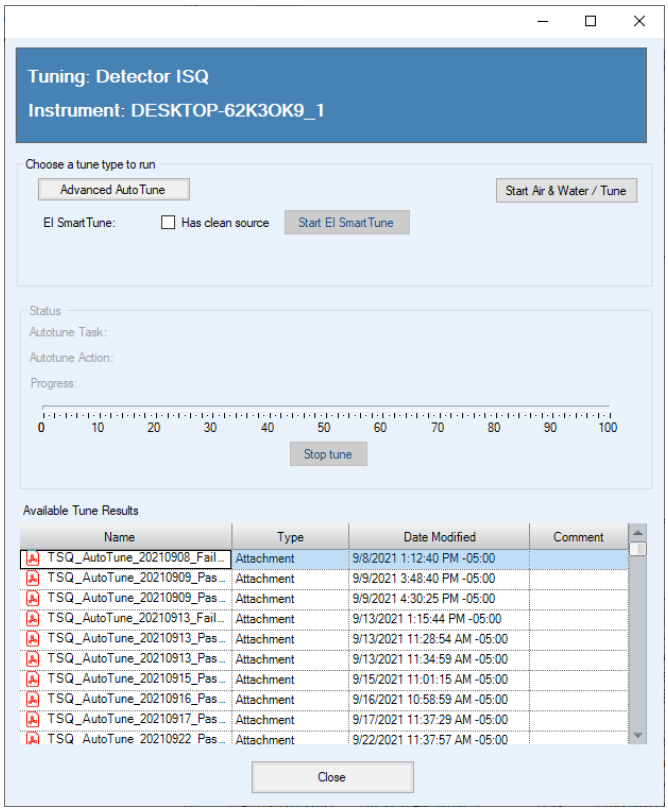
1. Go to the **ISQ** tab on the Chromeleon Console and click **Tuning** to open the tuning window. See Figure 27.

Figure 27. Accessing ISQ 7610 Tuning from the Chromeleon Console



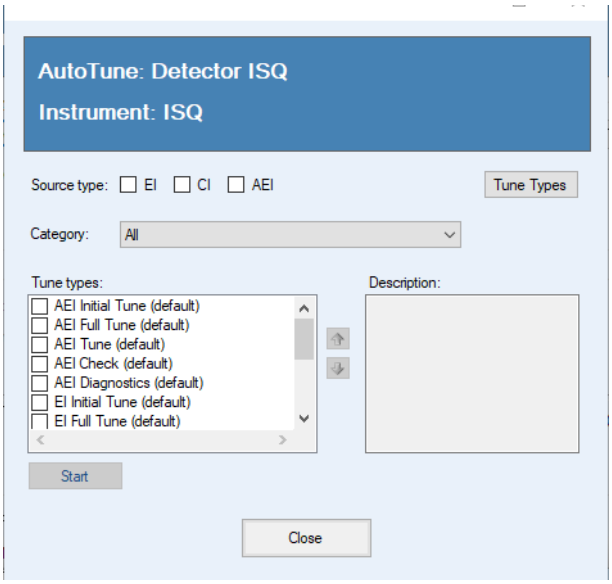
2. If you have configured Advanced AutoTune or SmartTune for your EI or AEI ion source you may access those options in the by clicking **Advanced AutoTune** in the Tuning utility. See Figure 28.

Figure 28. Opening the Tuning Panel



3. The AutoTune window with a list of available tune types opens. See

Figure 29. Opening the AutoTune Window



Tune Types

This section explains the different tune types in ISQ 7610 AutoTune.

EI 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.

EI 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.

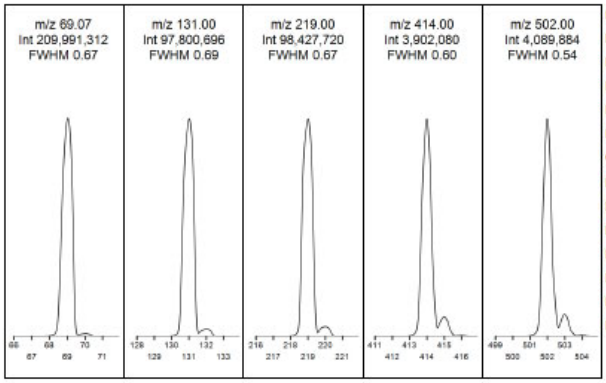
Figure 30shows a typical tune report for an EI Full Tune on a system using helium as a carrier gas.

Figure 30. Typical EI Full Tune Report

ISQ Tune Results

Lab: Austin R&D
Tune Completed - Fri Feb 16 12:49:52 2018
Tune File: AutoTune_EI_2018-02-16-12-49-52.isqtune
Tune Type: EI Full Tune (default)
Starting Tune File: (Last Saved Tune)

Instrument Name: ISQ
Instrument ID: 100992000
User:



Ion Source Type	EI
Polarity	Positive
Electron Lens Voltage	5 V
Electron Energy	70 eV
Emission Current	50 µA
Ion Guide Frequency	1653.0
Q1 Frequency	1088.2
Multiplier Voltage	1619.1 V
Detector Gain	3.0 x 10^ 5
MS Transfer Line	150.0 °C
Filament Selection	1
Ion Source Temperature	200.0 °C
Foreline Pressure	38 mTorr
Ion Gauge Pressure	1.2 x 10^ -5 Torr

Full Scan Spectrum Not Acquired

Mass	Theory	Abundance	Relative 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	215,756	9.13

Air/ Water Check:

H2O	(%18/69)	6.58
N2	(%28/69)	53.59
O2	(%32/69)	17.71
CO2	(%44/69)	1.24
N2 / H2O	(%28/18)	8.14

Leak Check: 3.19% of reference

Typical results for an EI Full Tune are listed below.

- Peak Intensities:
  - Base Peak ≥ 10,000,000
  - *m/z* 502 ≥ 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: ≥ 2200 V
- Foreline Pressure: < 100 mTorr
- Ion Gauge Pressure: < 5×10^{-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.

EI 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[1] counts.

EI 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.

[1] This number may change when using EI SmartTune.

EI 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

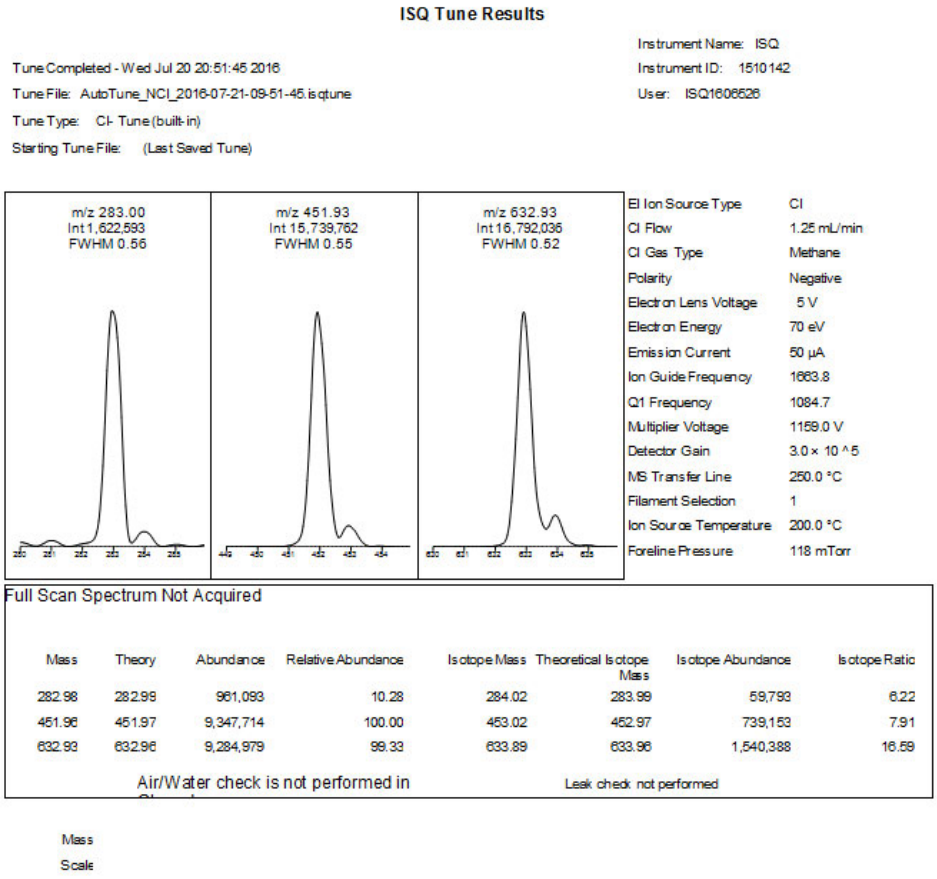
CI- Tune (default)—Used to analyze samples with negative CI. The standard NCI tune performs a mass calibration, then tunes the lenses and sets the resolution. This type of tune assumes you are using methane as the CI reagent gas and tunes the system with a 1.25 mL/min flow. This tune does not set the 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 31 shows a typical CI- Tune report where methane is the CI reagent gas.

Figure 31. Typical CI- Tune Report with Methane as a CI Reagent Gas



ISQ Tune Results

Instrument Name: ISQ
Instrument ID: 1510142
User: ISQ1606526

Tune Completed - Wed Jul 20 20:51:45 2016
Tune File: AutoTune_NCI_2016-07-21-09-51-45.isqtune
Tune Type: CI- Tune (built-in)
Starting Tune File: (Last Saved Tune)

m/z 283.00
Int 1,622,593
FWHM 0.56

m/z 451.93
Int 15,739,762
FWHM 0.55

m/z 632.93
Int 16,792,036
FWHM 0.52

EI Ion Source Type	CI
CI Flow	1.25 mL/min
CI Gas Type	Methane
Polarity	Negative
Electron Lens Voltage	5 V
Electron Energy	70 eV
Emission Current	50 µA
Ion Guide Frequency	1663.8
Q1 Frequency	1084.7
Multiplier Voltage	1159.0 V
Detector Gain	3.0 × 10 ^ 5
MS Transfer Line	250.0 °C
Filament Selection	1
Ion Source Temperature	200.0 °C
Foreline Pressure	118 mTorr

Full Scan Spectrum Not Acquired

Mass	Theory	Abundance	Relative Abundance	Isotope Mass	Theoretical Isotope Mass	Isotope Abundance	Isotope Ratio
282.98	282.99	961,093	10.28	284.02	283.99	59,793	6.22
451.96	451.97	9,347,714	100.00	453.02	452.97	739,153	7.91
632.93	632.96	9,284,979	99.33	633.89	633.96	1,540,388	16.59

Air/Water check is not performed in

Leak check not performed

Mass Scale

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
  - Replace Multiplier: ≥ 2200 V

- Foreline Pressure: < 400 mTorr
- Ion Gauge Pressure: < 1×10^{-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

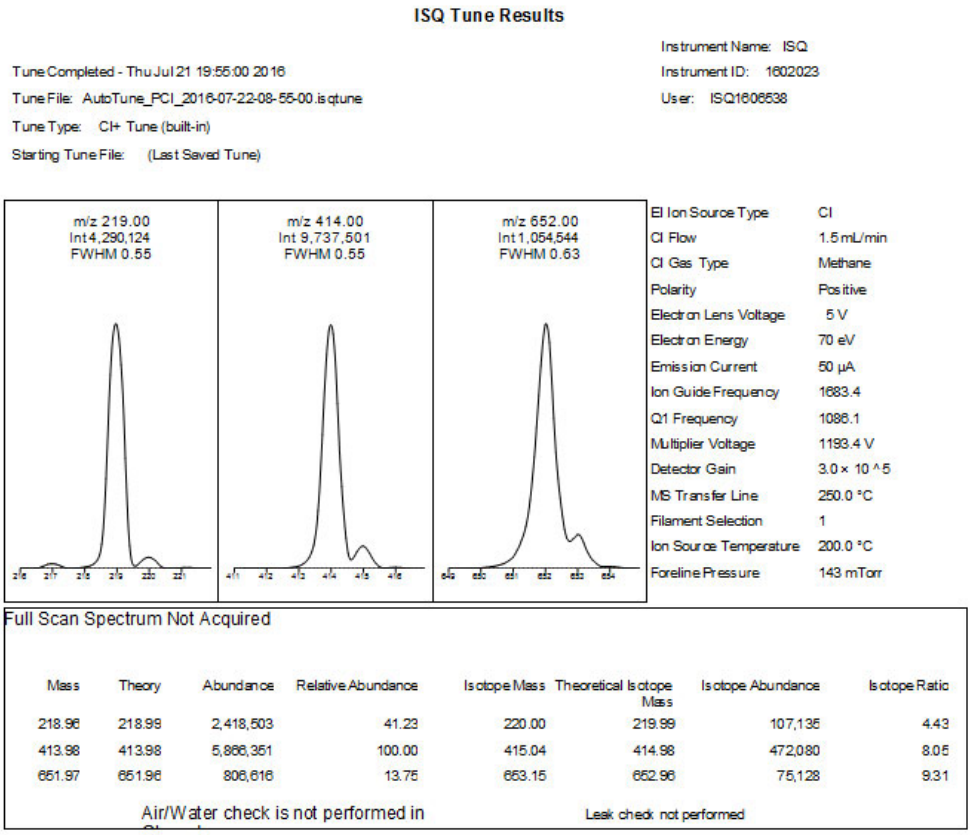
CI+ Tune—Used to analyze samples with positive CI. The standard PCI tune performs a mass calibration, then tunes the lenses and sets the resolution. This type of tune assumes you are using methane as the CI reagent gas and tunes the system with a 1.5 mL/min flow. This tune does not set the detector gain. 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 32 shows a typical CI+ Tune report where methane is the CI reagent gas.

Figure 32. Typical C+- Tune Report with Methane as a CI Reagent Gas



ISQ Tune Results

Instrument Name: ISQ
Instrument ID: 1602023
User: ISQ1606538

Tune Completed - Thu Jul 21 19:55:00 2016
Tune File: AutoTune_PCI_2016-07-22-08-55-00.isqtune
Tune Type: CI+ Tune (built-in)
Starting Tune File: (Last Saved Tune)

m/z 219.00
Int 4,290,124
FWHM 0.55

m/z 414.00
Int 9,737,501
FWHM 0.55

m/z 652.00
Int 1,054,544
FWHM 0.63

EI Ion Source Type	CI
CI Flow	1.5 mL/min
CI Gas Type	Methane
Polarity	Positive
Electron Lens Voltage	5 V
Electron Energy	70 eV
Emission Current	50 µA
Ion Guide Frequency	1683.4
Q1 Frequency	1086.1
Multiplier Voltage	1193.4 V
Detector Gain	3.0 × 10 ^ 5
MS Transfer Line	250.0 °C
Filament Selection	1
Ion Source Temperature	200.0 °C
Foreline Pressure	143 mTorr

Full Scan Spectrum Not Acquired

Mass	Theory	Abundance	Relative Abundance	Isotope Mass	Theoretical Isotope Mass	Isotope Abundance	Isotope Ratio
218.96	218.99	2,418,503	41.23	220.00	219.99	107,135	4.43
413.98	413.98	5,866,351	100.00	415.04	414.98	472,080	8.05
651.97	651.96	806,616	13.75	653.15	652.96	75,128	9.31

Air/Water check is not performed in

Leak check not performed

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 ≥ 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 3×10^5 detector gain
  - Replace Multiplier: ≥ 2500 V at 2.1×10^6 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)—(To be used only with the AEI ion source.) 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)—(To be used only with the AEI ion source.) 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[2] counts.

[2] This number may be modified when using AEI SmartTune.

AEI Check

AEI Check (default)—(To be used only 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

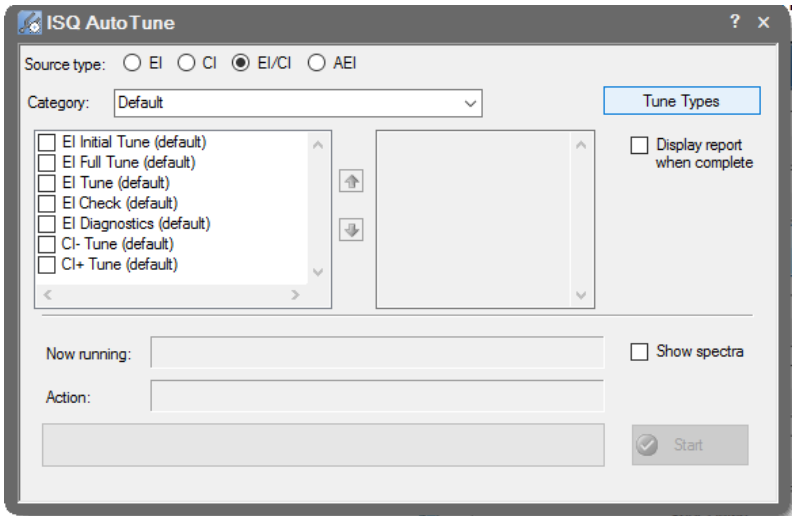
AEI Diagnostics (default)—(To be used only with the AEI ion source). Runs a complete set of diagnostics, including a leak check, and generates a report. No tuning is performed. Uses the parameters from the last saved tune.

Tuning the ISQ 7610 Mass Spectrometer

❖ To tune the ISQ 7610 Mass Spectrometer using AutoTune in Xcalibur

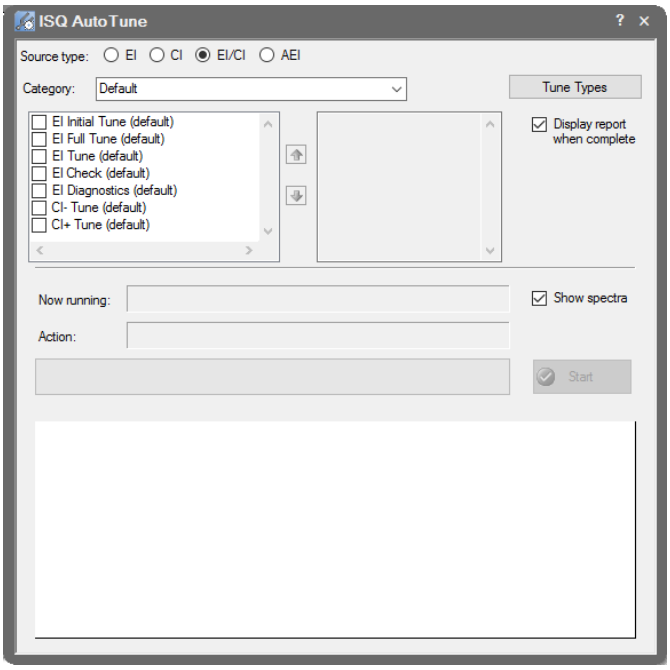
1. Select the tune type you want to use from the list of available tune types. See Figure 33.

Figure 33. List of Available Tunes in AutoTune



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

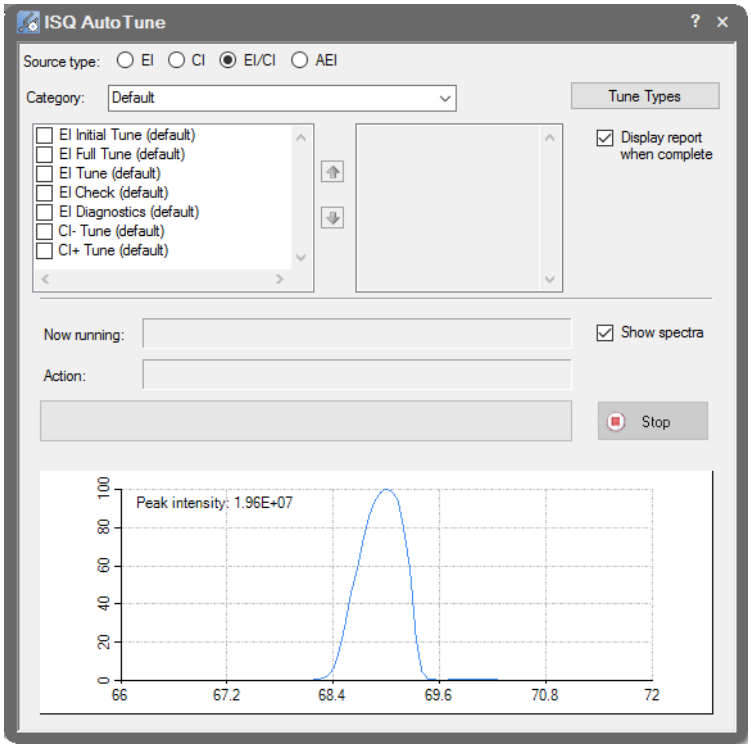
Figure 34. Displaying a Tune Report



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

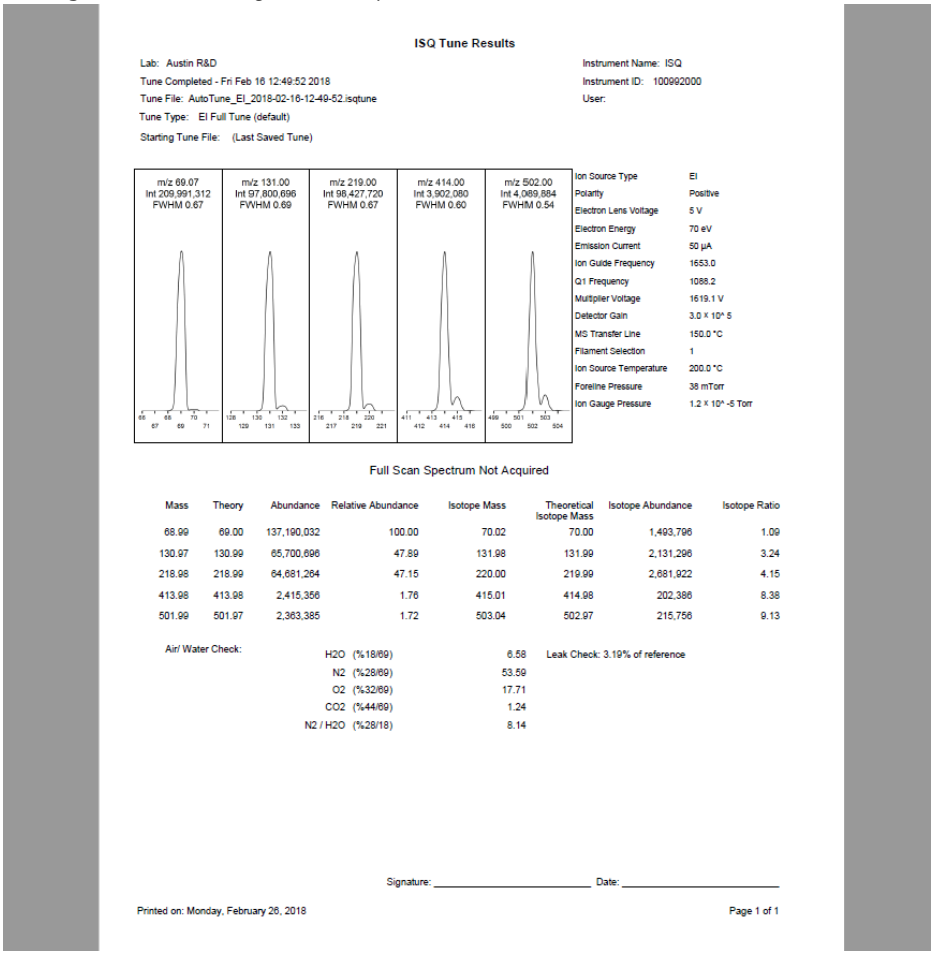
Note Make sure the power options on your computer are not set to go into Standby mode while you're acquiring data for your tune. Otherwise, it will interrupt your tune.

Figure 35. Showing the Spectra During an Automatic Tune



5. 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 7610 Dashboard and view the report.

Figure 36. Viewing a Tune Report.



ISQ Tune Results

Lab: Austin R&D
Tune Completed - Fri Feb 16 12:49:52 2018
Tune File: AutoTune_EI_2018-02-16-12-49-52.isqtune
Tune Type: EI Full Tune (default)
Starting Tune File: (Last Saved Tune)

Instrument Name: ISQ
Instrument ID: 100992000
User:

m/z 69.07	m/z 131.00	m/z 219.00	m/z 414.00	m/z 502.00
Int 209,991,312	Int 97,800,696	Int 98,427,720	Int 3,902,080	Int 4,089,884
FWHM 0.67	FWHM 0.69	FWHM 0.67	FWHM 0.60	FWHM 0.54

Ion Source Type	EI
Polarity	Positive
Electron Lens Voltage	5 V
Electron Energy	70 eV
Emission Current	50 µA
Ion Guide Frequency	1653.0
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
Ion Gauge Pressure	1.2 × 10^ -5 Torr

Full Scan Spectrum Not Acquired

Mass	Theory	Abundance	Relative 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	215,756	9.13

Air/ Water Check:

H2O	(%18/69)	6.58
N2	(%28/69)	53.59
O2	(%32/69)	17.71
CO2	(%44/69)	1.24
N2 / H2O	(%28/18)	8.14

Leak Check: 3.19% of reference

Signature: _____ Date: _____

Printed on: Monday, February 26, 2018

Page 1 of 1

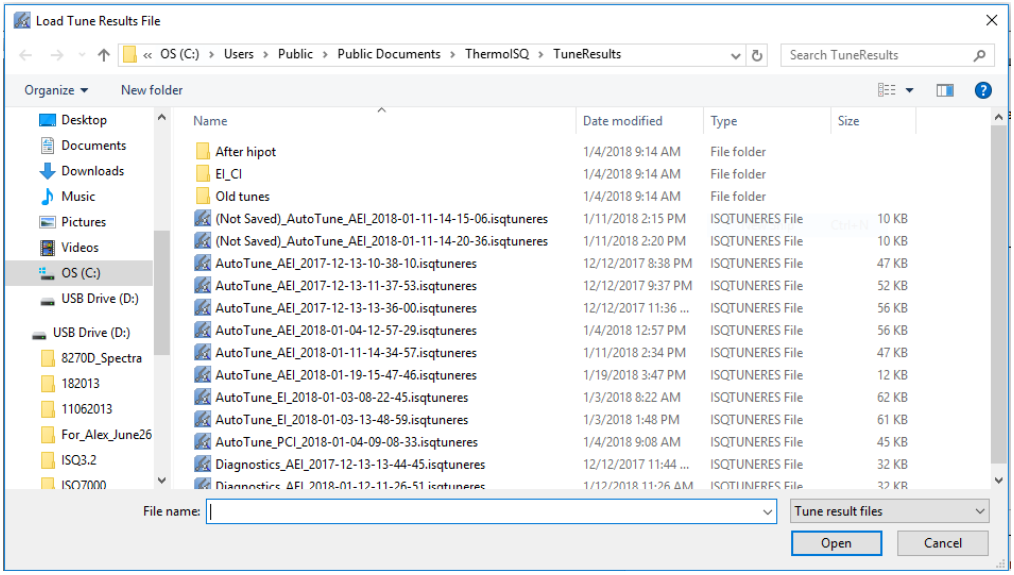
Note The **Error Action** is not diagnostic. It indicates what happens during the tune if a specific device cannot meet the tuning criteria. Any **Stop** comments on error actions do not mean the tune has failed.

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 an Adobe Acrobat PDF file.

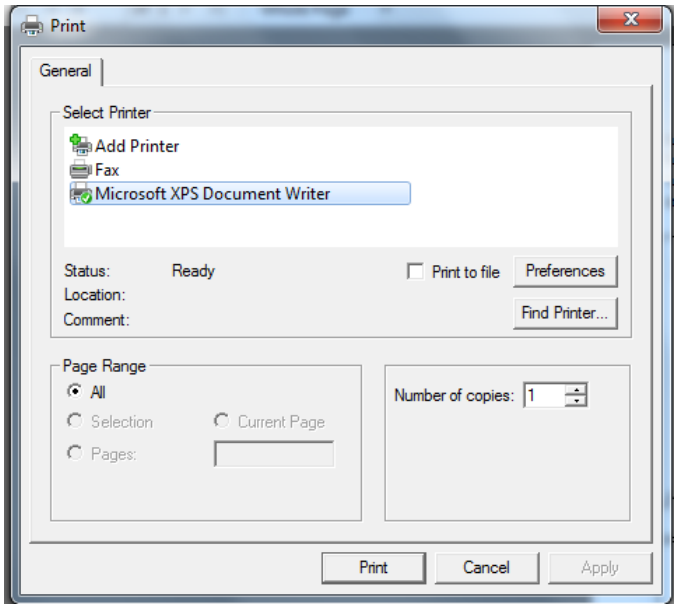
7. Click **Open Tune Results** in the top of the window and browse to another tune report on your computer. Then click **Open**.

   Figure 37. Opening a Tune Results File



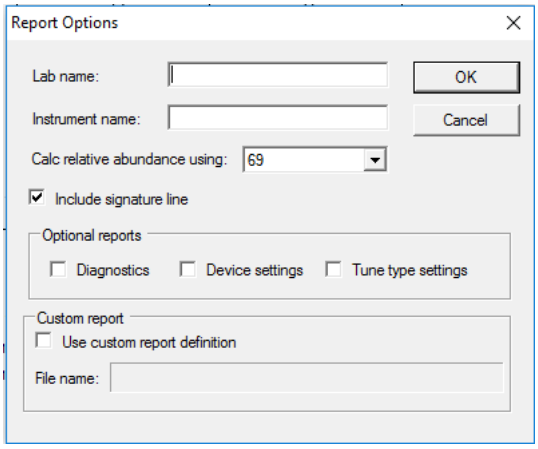
8. Click **Print Report** to open a print dialog box and print your report.

Figure 38. 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 39. Selecting the Tune Report Options



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

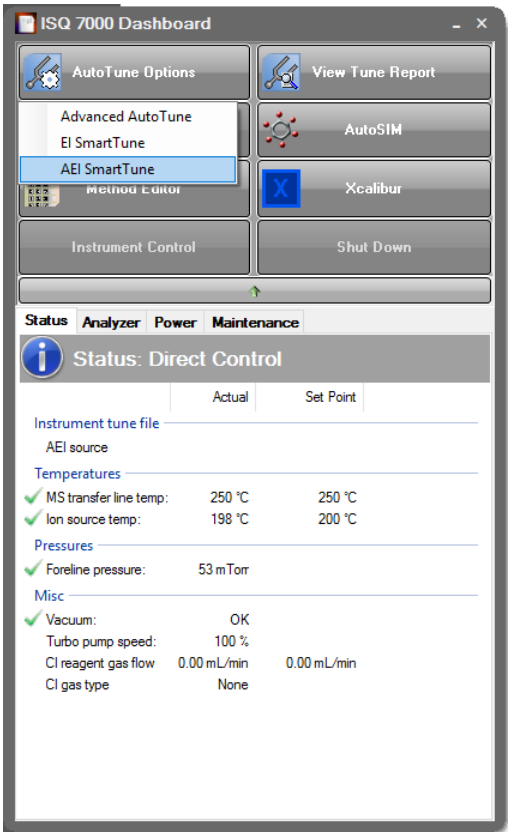
Using SmartTune

If you configured the ISQ 7610 software to use the **EI SmartTune** or **AEI SmartTune** application, follow the instructions below to use the application.

❖ To use the SmartTune application

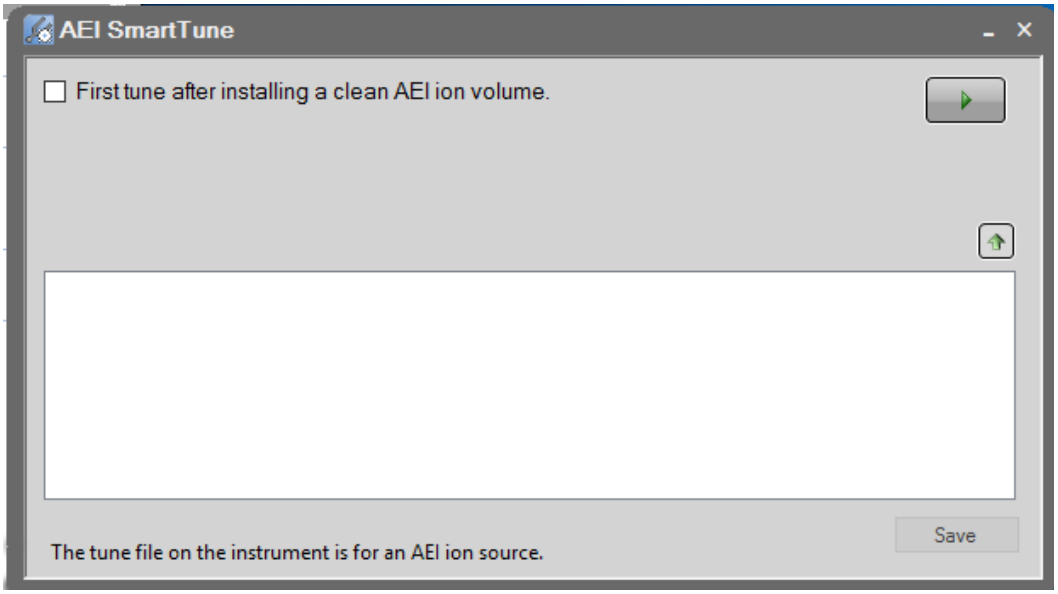
1. Right-click **AutoTune Options** on the ISQ 7610 Dashboard and choose **AEI SmartTune** or **EI SmartTune** depending on which ion source is installed on your instrument. See Figure 40.

   Figure 40. Opening the SmartTune Application



2. The SmartTune application opens. See Figure 41.

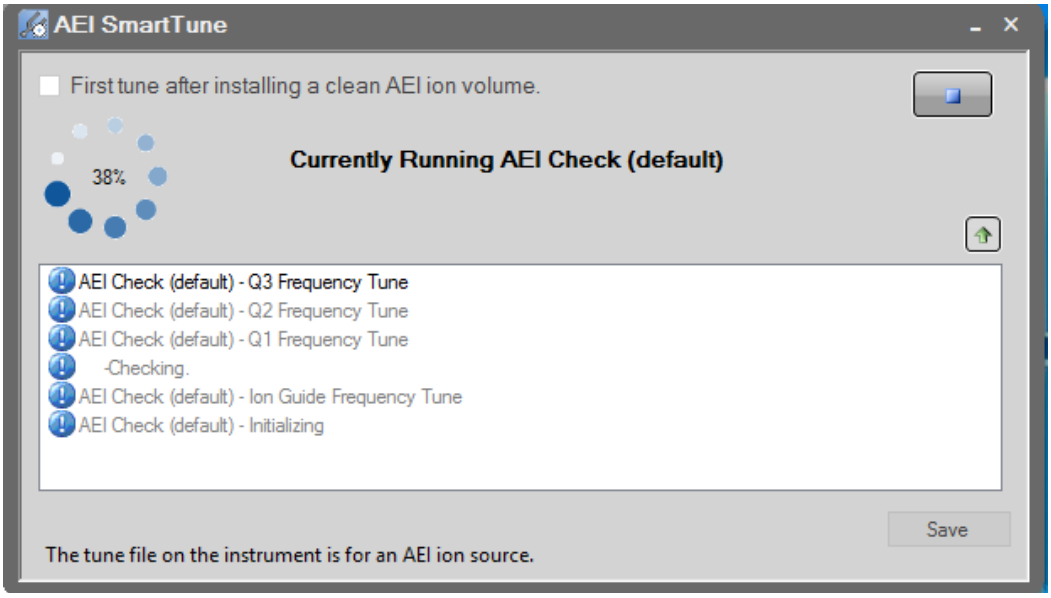
Figure 41. Viewing the SmartTune Application



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.

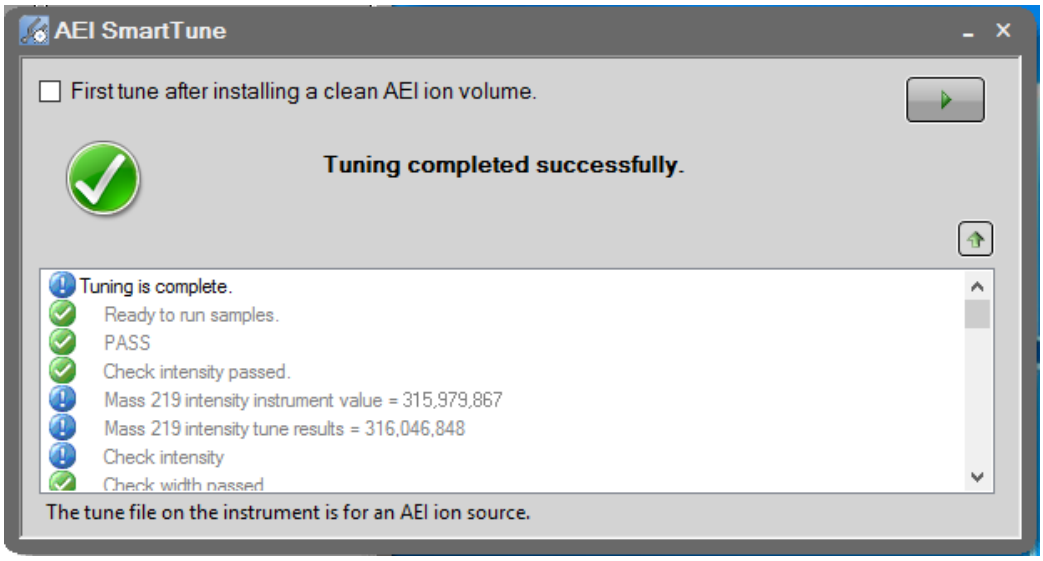
4. The SmartTune application assess the state of your instrument and chooses the proper tunes for the system. The tunes run or running and the percent complete appear in the window while SmartTune is in progress. See Figure 42.

Figure 42. SmartTune in Progress



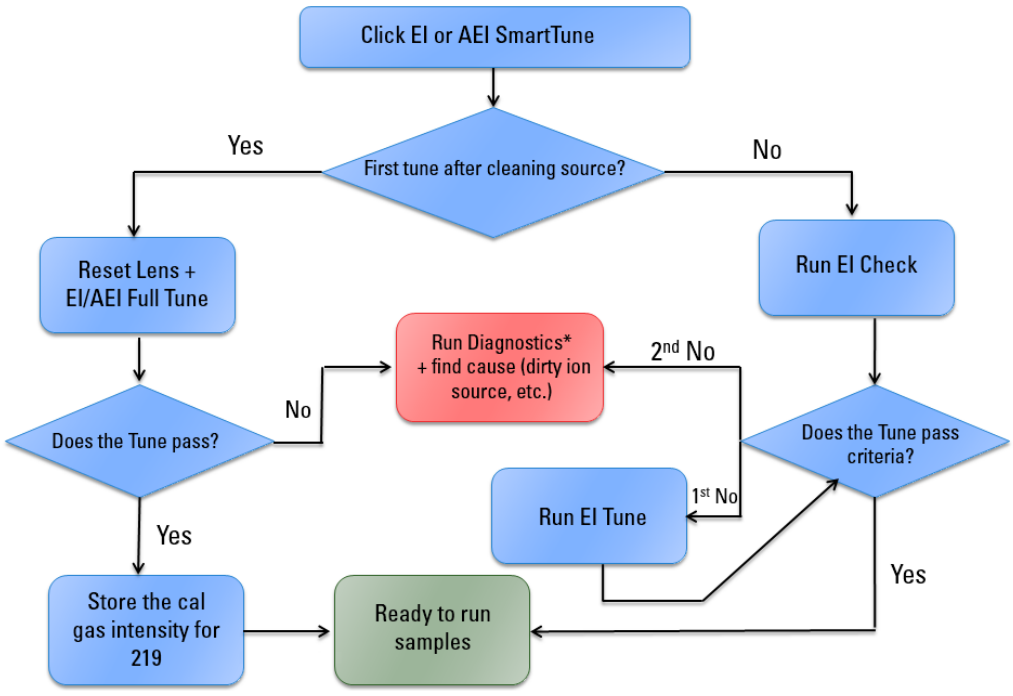
5. Once SmartTune has completed tuning your instrument, you will see a green check in the SmartTune window with a list of all the tunes it ran and a brief summary of their results. See Figure 43.

Figure 43. SmartTune Completed



6. View the tune report to see all the results of the SmartTune.
7. See Figure 44 below for the SmartTune workflow.

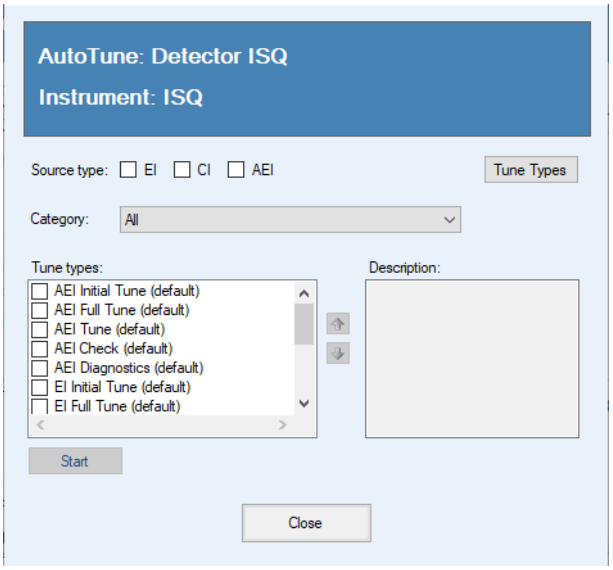
Figure 44. SmartTune Workflow



❖ To Tune the ISQ 7610 MS Using Chromeleon Software

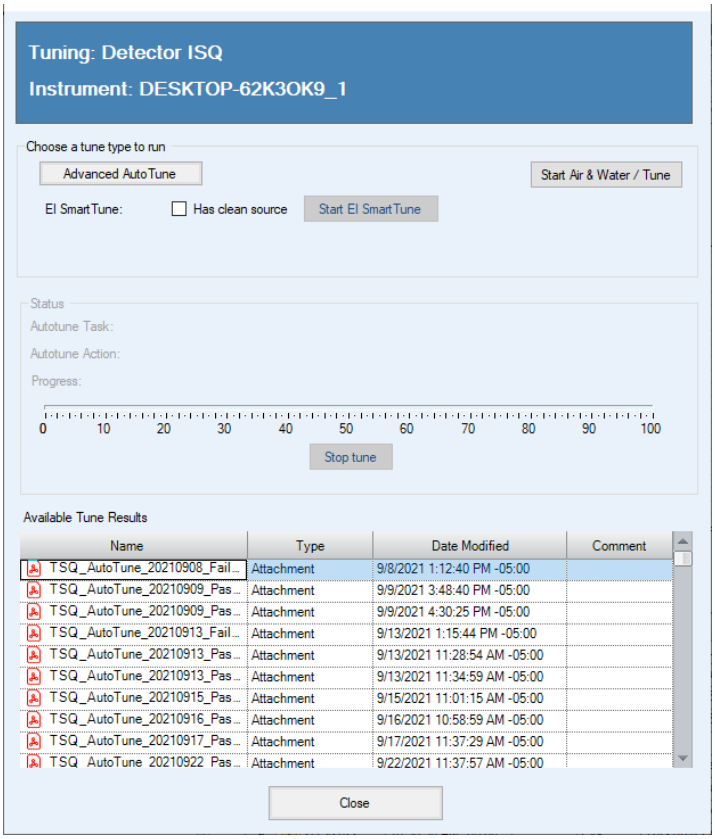
1. Choose a tune from AutoTune window. Click **Start**. See Figure 45.

Figure 45. Tuning the ISQ 7610 Using Chromeleon Software



2. View the tuning status in the audit trail under the ISQ tab on the Chromeleon Console.
3. To open a tune report, go to the Available Tune Results on the Tuning panel. See Figure 46.

Figure 46. Locating ISQ 7610 Tune Reports in Chromeleon Software

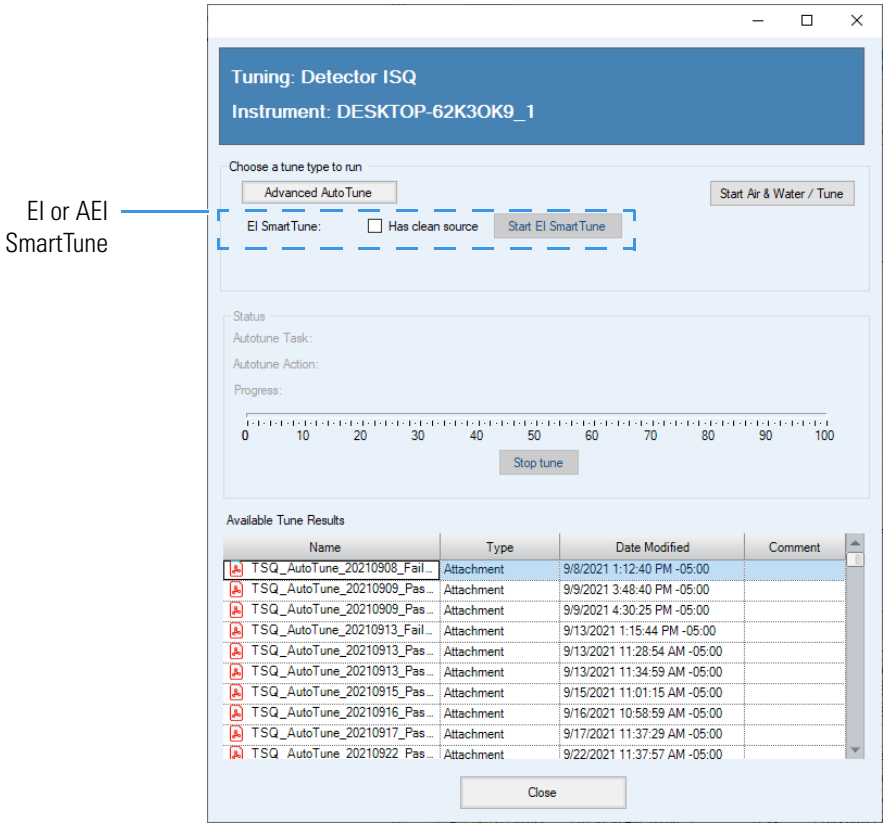


❖ To use the SmartTune application in Chromeleon Software

Note The instrument configuration settings determine whether or not Advanced AutoTune and/or the EI or AEI SmartTune options are shown on this page. If they are missing, please check the instrument configuration.

1. EI and AEI SmartTune are accessed in the **Tuning** window. Depending on which ion source is installed, the software displays **AEI SmartTune** or **EI SmartTune**. See Figure 47.

   Figure 47. Accessing EI or AEI SmartTune in Chromeleon Software



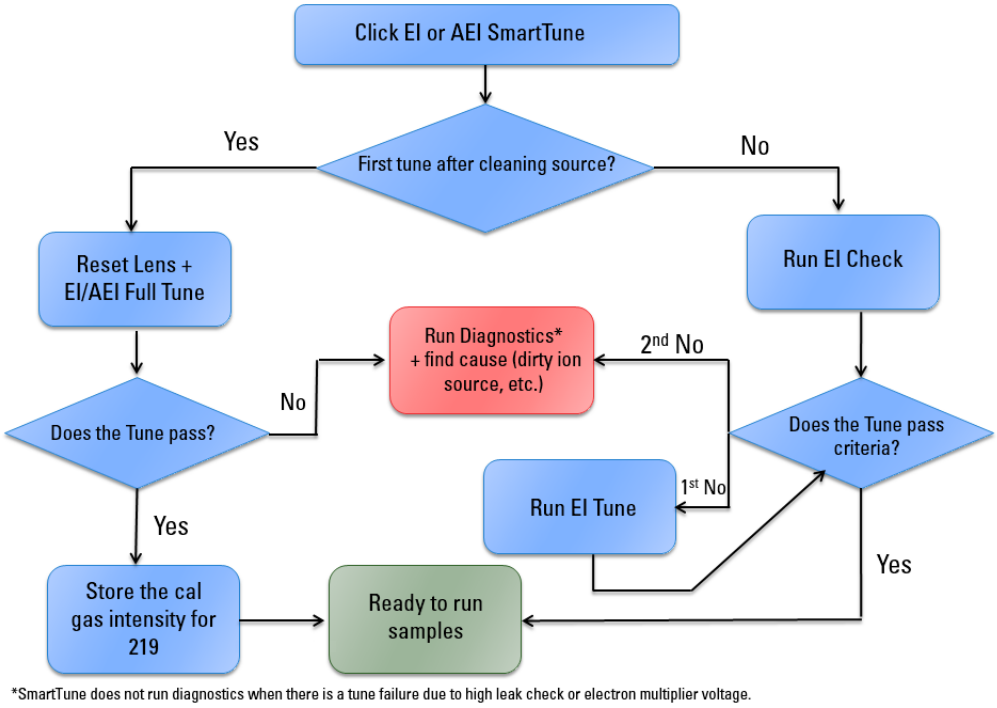
2. If this is the first smart tune to be run after installing a clean ion source, check the **Has Clean Source** box.

3. Click Start **EI SmartTune** or Start **AEI SmartTune** depending on which ion source is installed on the system.

4. View the tune report to see all the results of the SmartTune.

5. See Figure 48 below for the SmartTune workflow.

Figure 48. SmartTune Workflow



Tuning for DFTPP and BFB

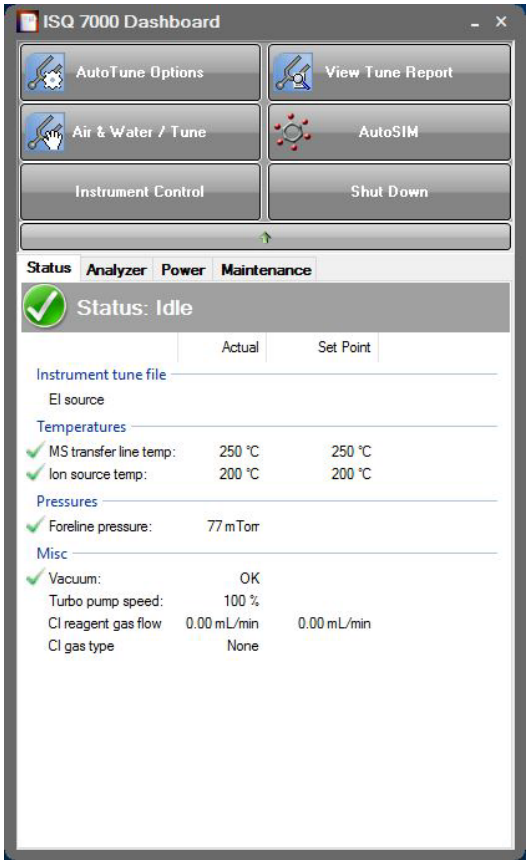
DFTPP and BFB are calibration compounds used by the EPA in the United States to determine the suitability of your mass spectrometer for specific methods for analyzing semi-volatile and volatile organic compounds. Follow the instructions below for tuning your instrument to pass the criteria for analyzing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

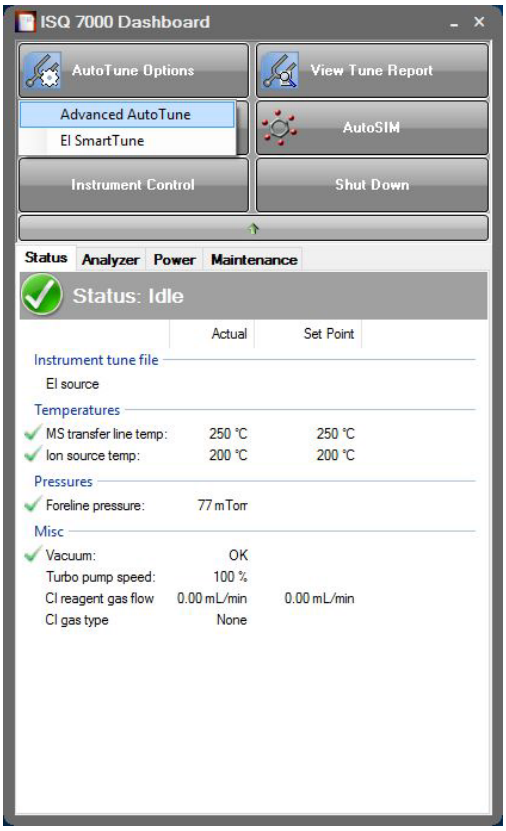
1. Open the ISQ 7610 Dashboard and click **AutoTune Options** to open the AutoTune window. See Figure 49.

Figure 49. Accessing ISQ 7610 AutoTune from the ISQ 7610 Dashboard



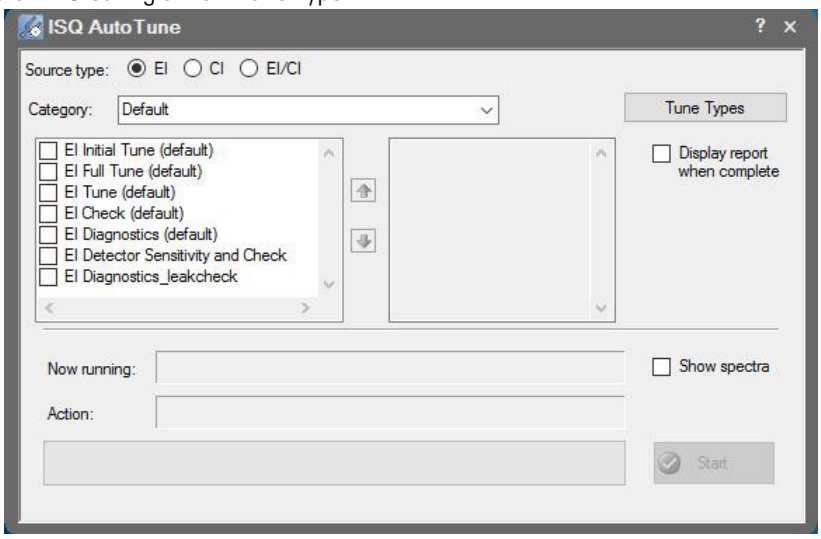
2. Select **Advanced AutoTune** from the drop-down menu. See Figure 50.

Figure 50. Selecting from AutoTune Options



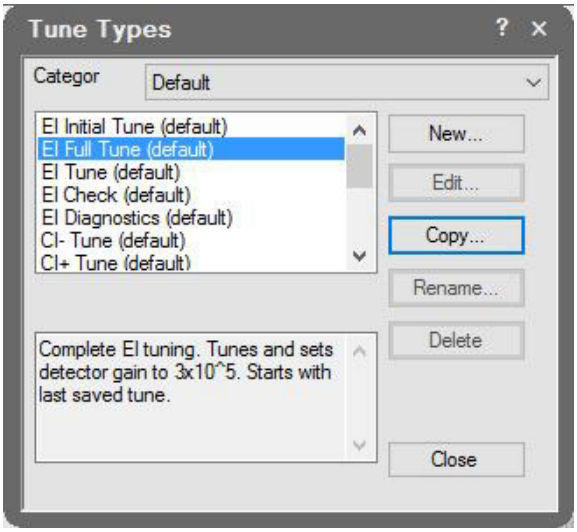
1. Click **Tune Types** to create a new tune type. See Figure 51.

Figure 51. Creating a New Tune Type



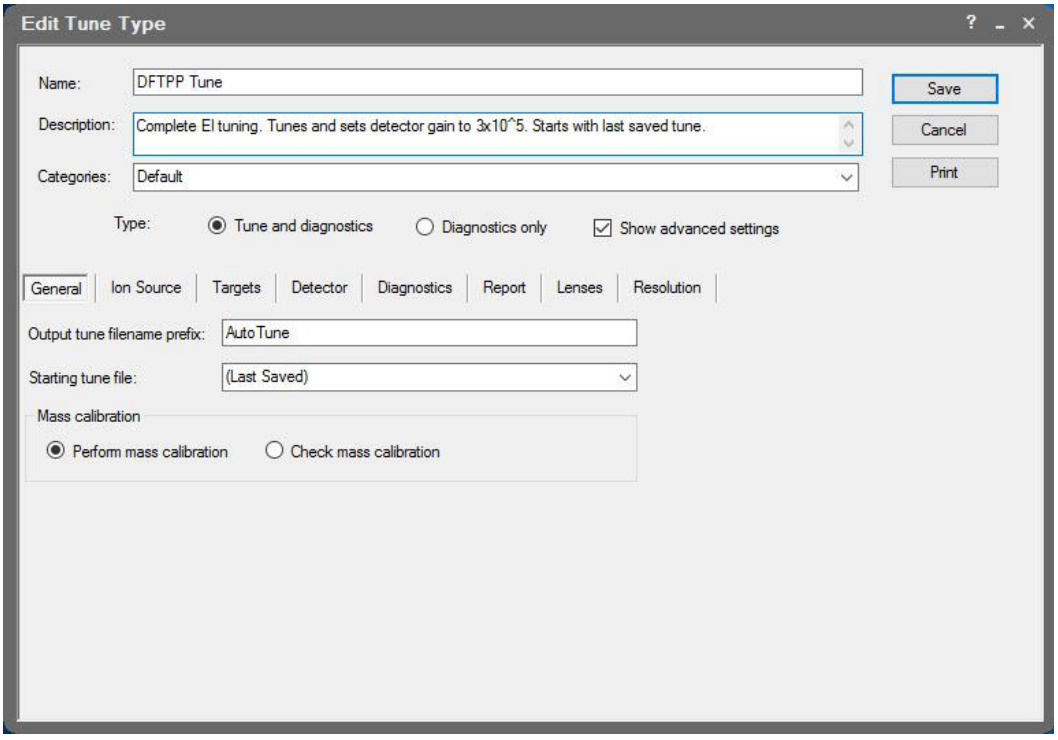
2. In the **Tune Types** dialog box, select a tune type to edit and click the **Copy** button. See Figure 52.

Figure 52. Using the Tune Types Dialog Box



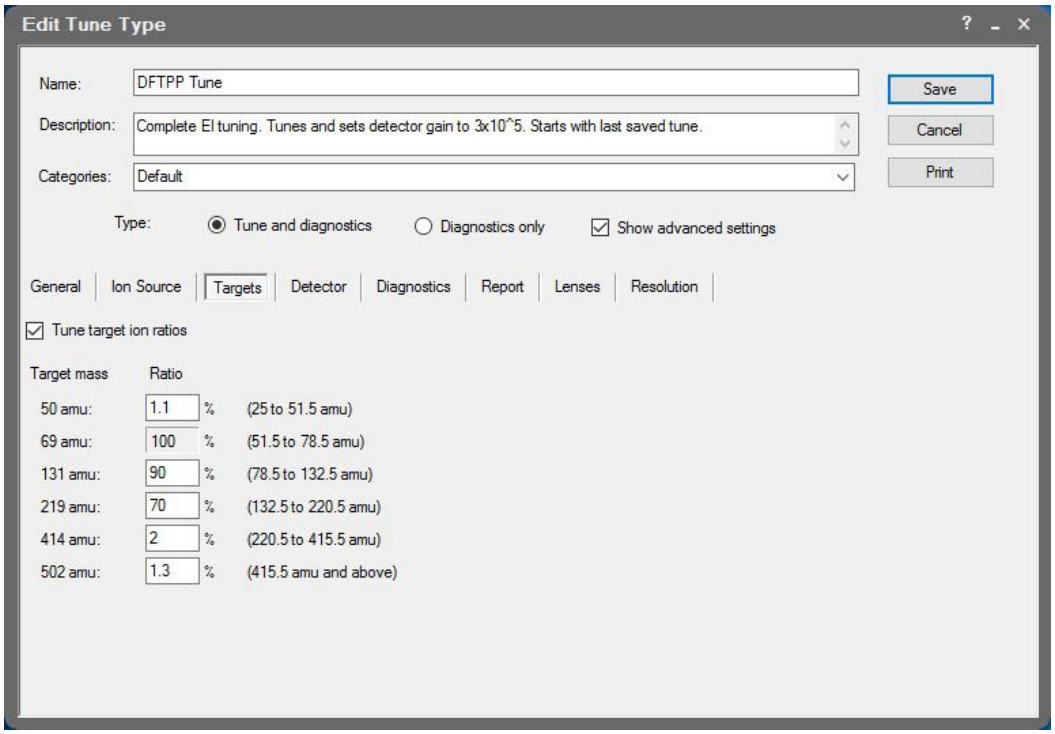
3. The **Edit Tune Type** window opens. In the **Name** field, name the new tune type **DFTPP Tune** or **BFB Tun**e depending on your requirement. Change the description if you prefer. See Figure 53.

Figure 53. Creating the DFTPP or BFB Custom Tune Type



4. Go to the **Targets** tab and check **Tune Target Ion Ratios**. See Figure 54.

Figure 54. Tuning Target Ion Ratios



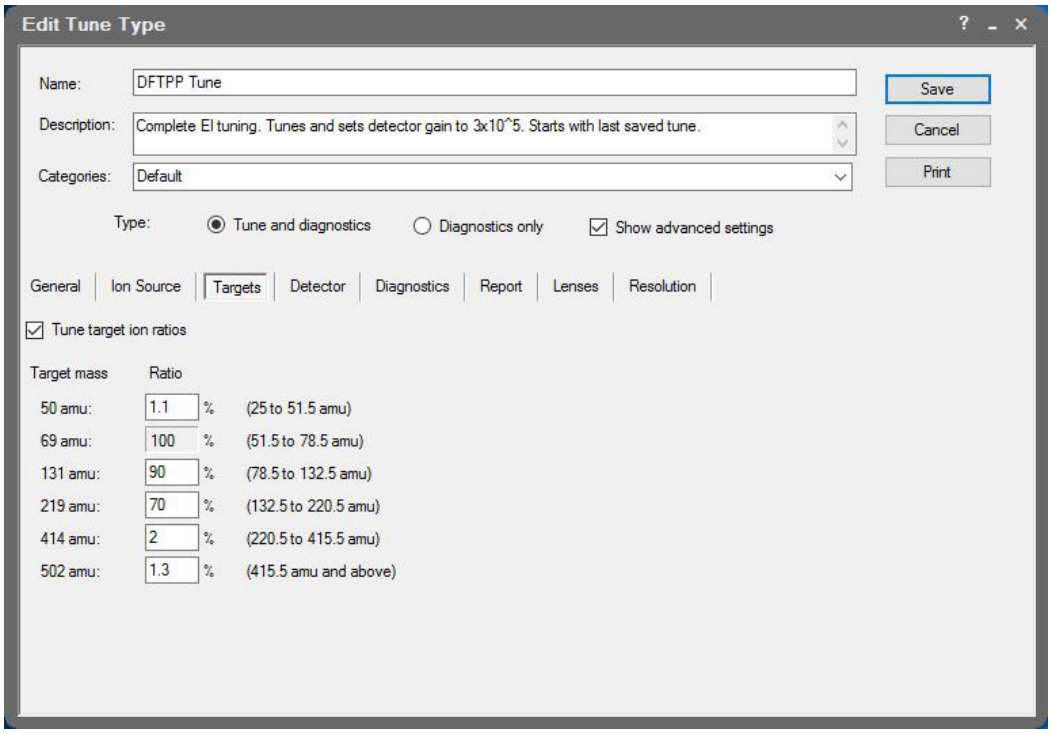
5. For DFTPP—Enter the values below for the target ion ratios. See Table 1.

Table 1. Target Ion Ratios for DFTPP

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

6. See Figure 55 for an example of the correct DFTTP Tune.

Figure 55. DFTPP Tune Targets



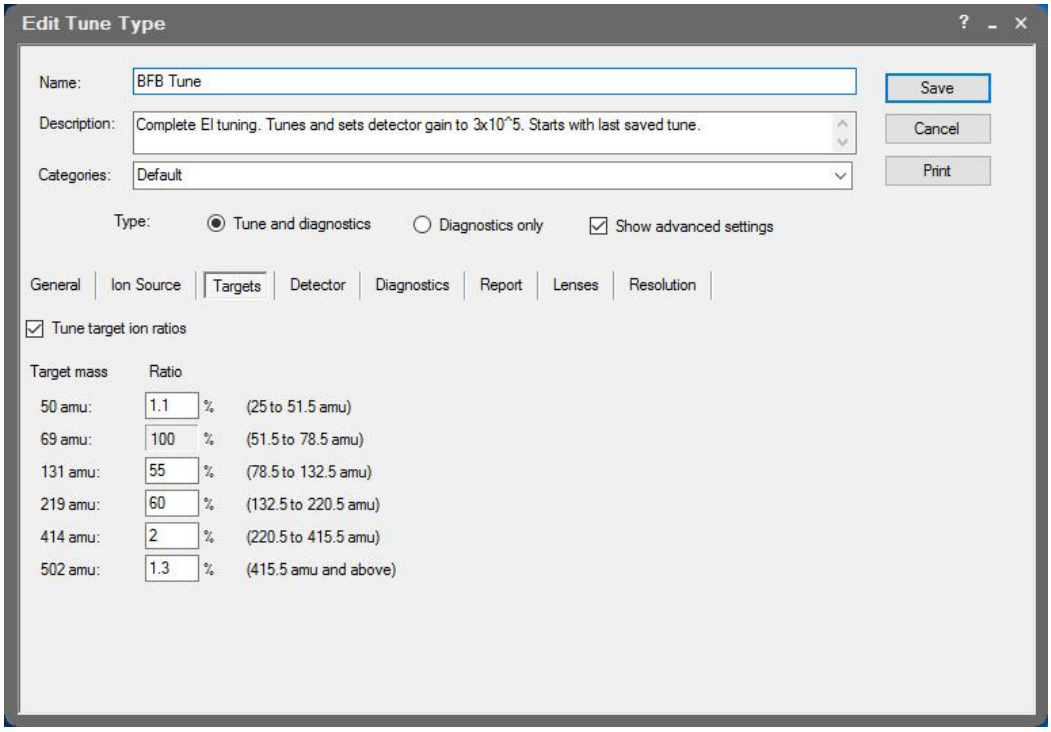
7. Save as **DFTPP Tune**.
8. For BFB—Enter the values below for the target ion ratios. See Table 2.

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 56 for an example of a correct BFB Tune.

Figure 56. BFB Tune Targets



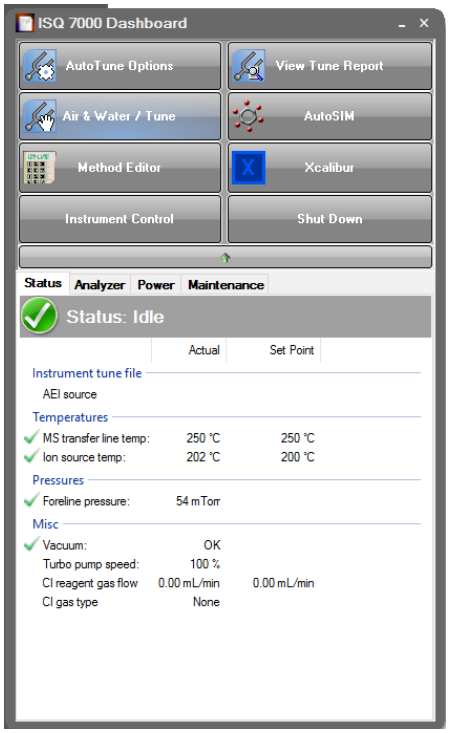
10. Save as **BFB Tune.**
11. Tune the instrument with the newly created tune types as described in Tuning the ISQ 7610 Mass Spectrometer.

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

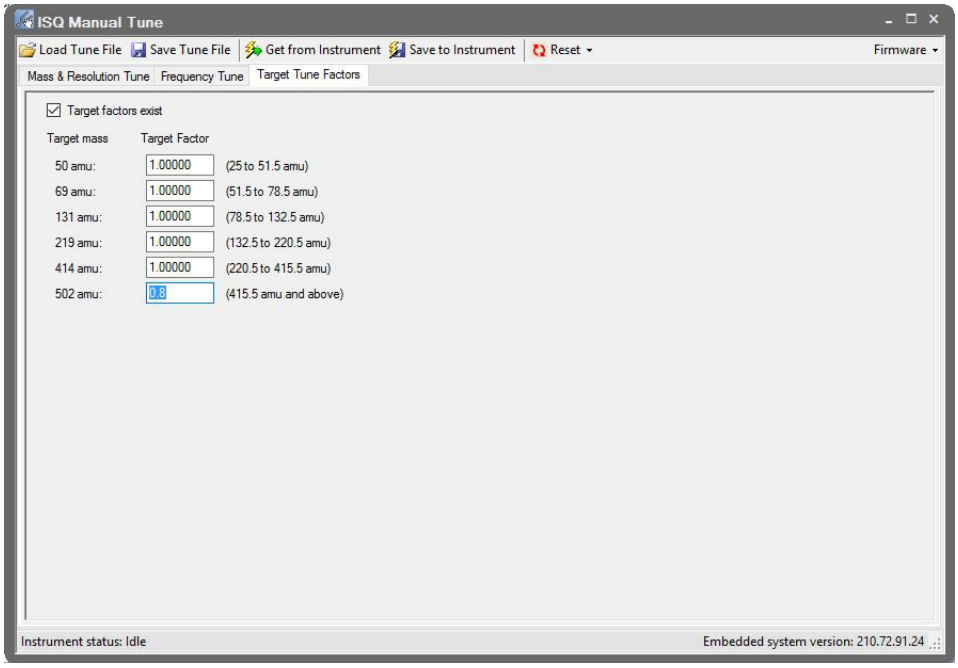
1. On the dashboard, click **Air & Water/ Tune**. See Figure 57.

Figure 57. Locating Air & Water/Tune on the ISQ 7610 Dashboard



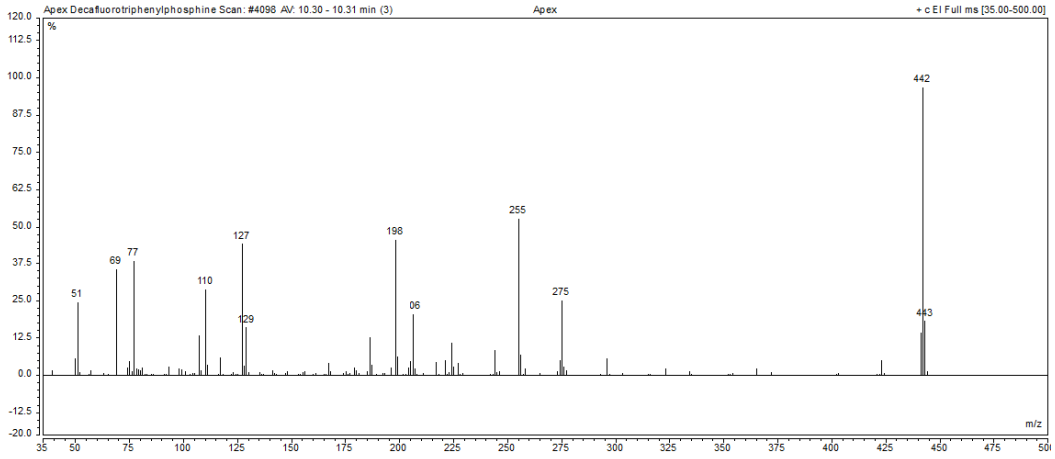
2. The **ISQ Manual Tune** utility opens. The last saved tune opens. If your DFTPP or BFB Tune was not the last saved tune, select **Load Tune File**, and open the correct tune file. Under the **Target Tune Factors** tab, adjust the tune factors as needed until the DFTPP or BFB tune criteria passes. See Figure 58.

Figure 58. Adjusting the Target Tune Factors in ISQ Manual Tune



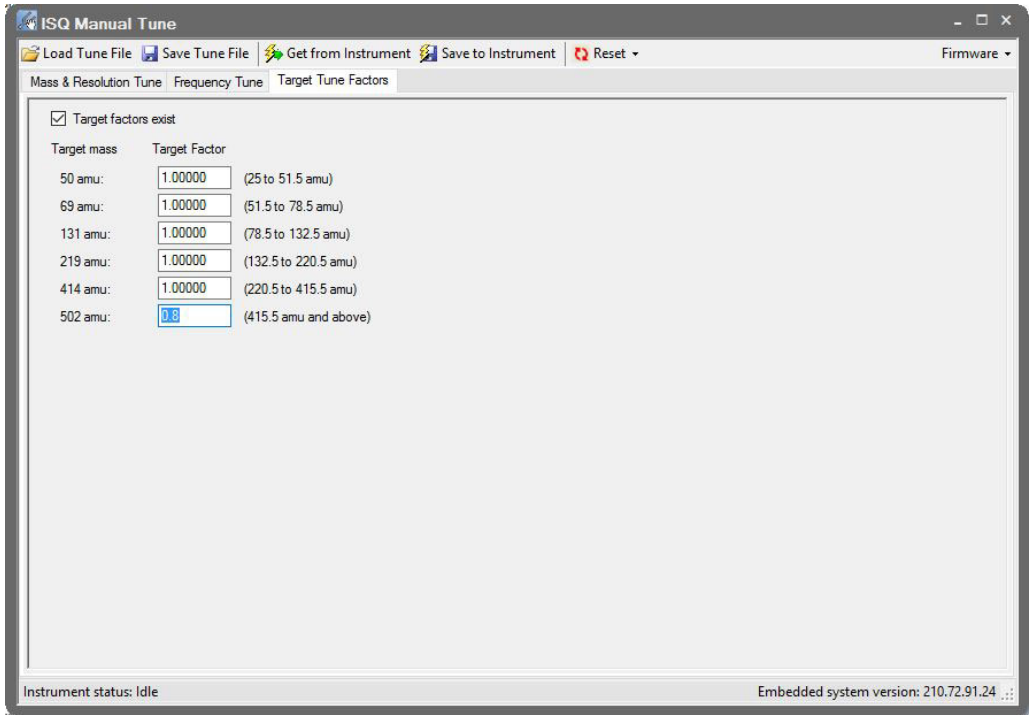
The example in Figure 59 shows a DFTPP run that had a low *m/z* 198. In this example, the *m/z* 198 intensity was low, and the tune failed. To increase the *m/z* 198 intensity, the high mass intensity was lowered in the target tune factors. Specifically, the **502 amu** target mass was lowered from **1** to **0.8**.

Figure 59. DFTPP Tune with Low *m/z* 198



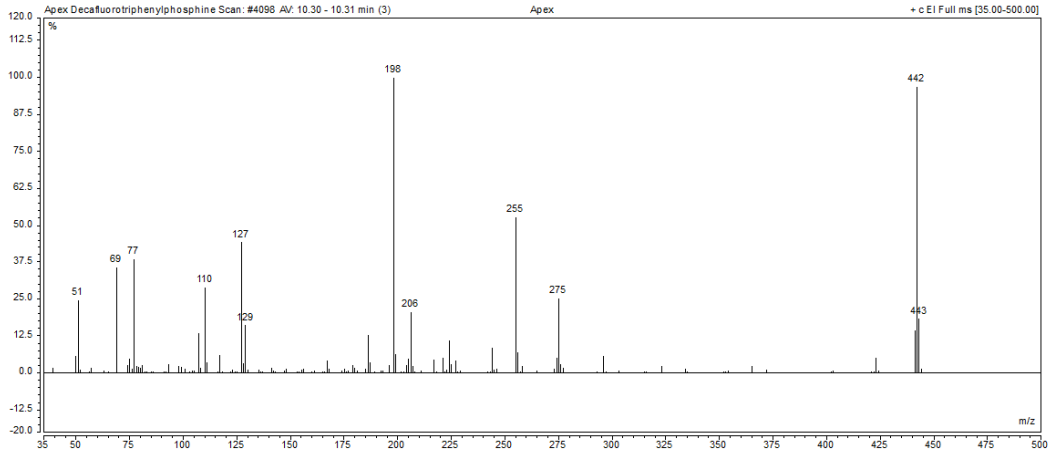
3. To increase the *m/z* 198 intensity, the high mass intensity was lowered in the target tune factors. Specifically, the 502 amu target mass was lowered from 1 to 0.8. See Figure 60.

Figure 60. 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 61.

Figure 61. 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.

4

Creating a Method

Once you have tuned the ISQ 7610 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

To create a method for the ISQ 7610 mass spectrometer, the TRACE 1600 or 1610 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.

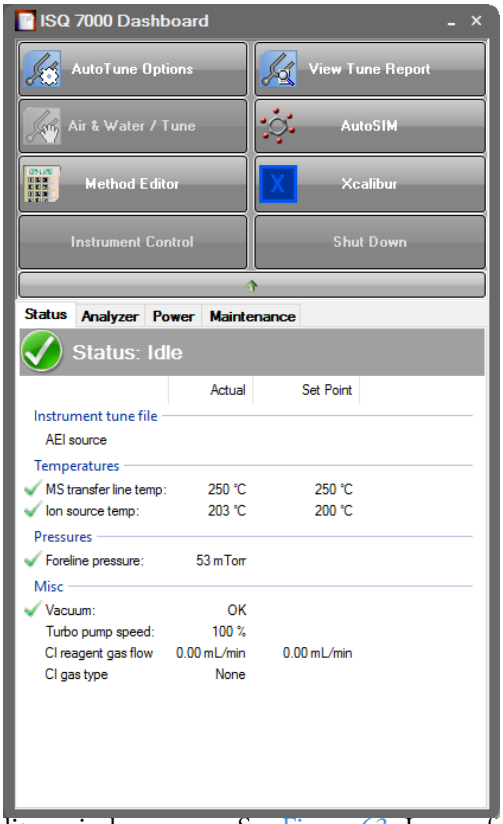
Note For information about creating a method for other instruments and software, refer to the appropriate documentation.

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 "Reconfiguring Your Instrument" for instructions.

❖ **To access the method editor using Xcalibur software**

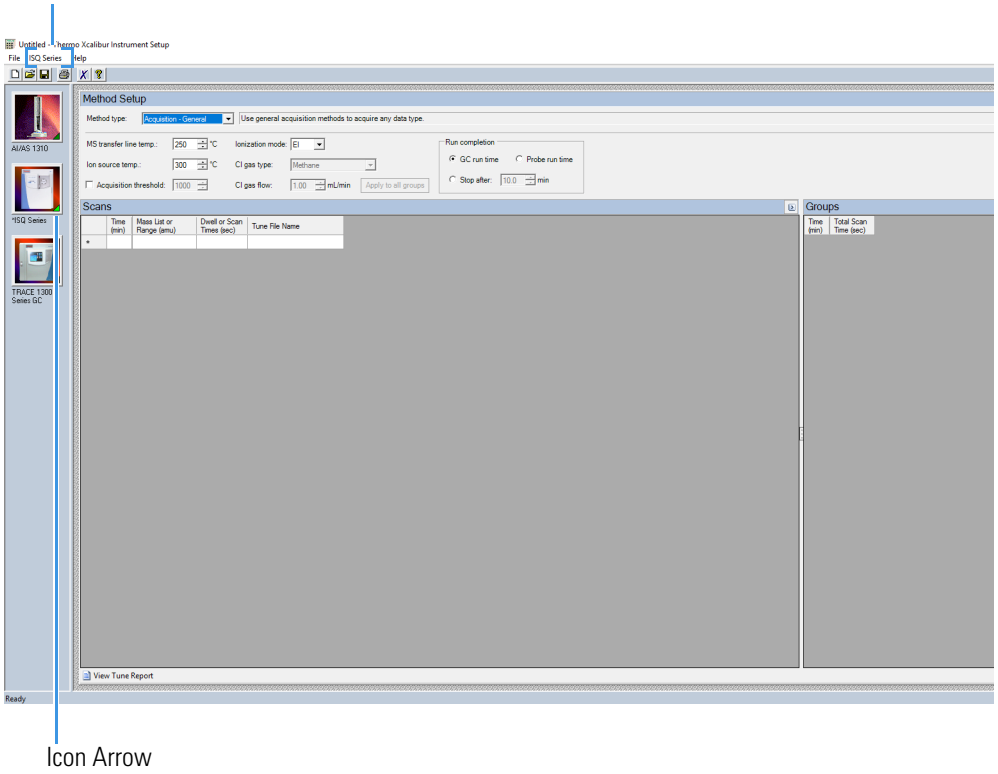
1. Click **Method Editor** on the ISQ 7610 Dashboard. See Figure 62.

Figure 62. Accessing the Method Editor from the ISQ 7610 Dashboard



2. The Method Editor window opens. See Figure 63. 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.

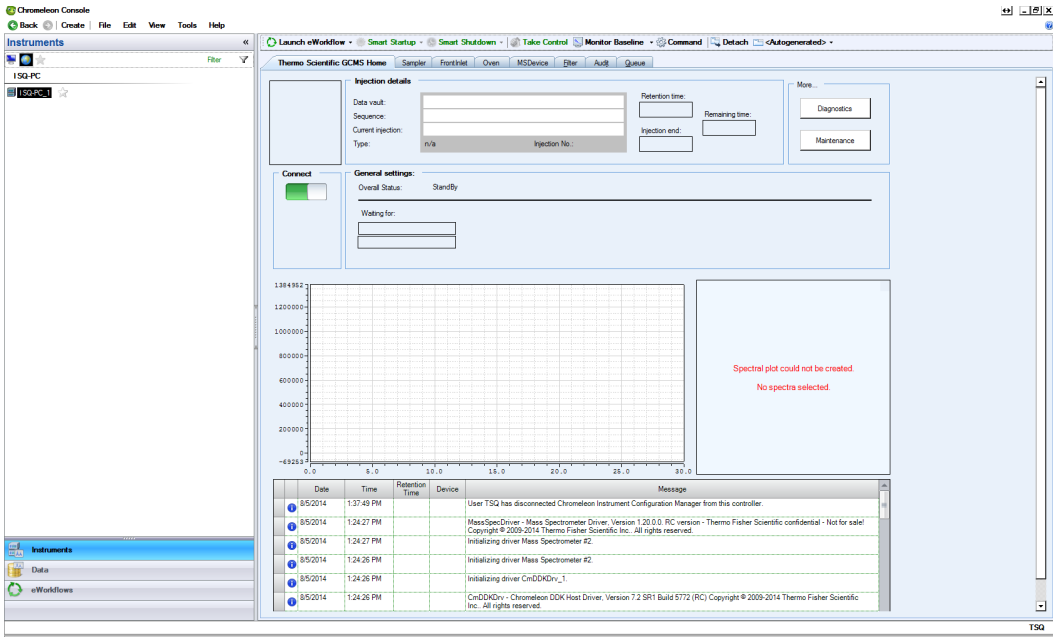
Figure 63. Method Editor Window



❖ To access the Method Editor using Chromeleon software

To access the Method Editor for the ISQ 7610 mass spectrometer, the TRACE 1600 or 1610 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 64.

Figure 64. 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 > ISQPCNAME**. If your instruments are not shown, you must configure them. See "Reconfiguring Your Instrument" for instructions.

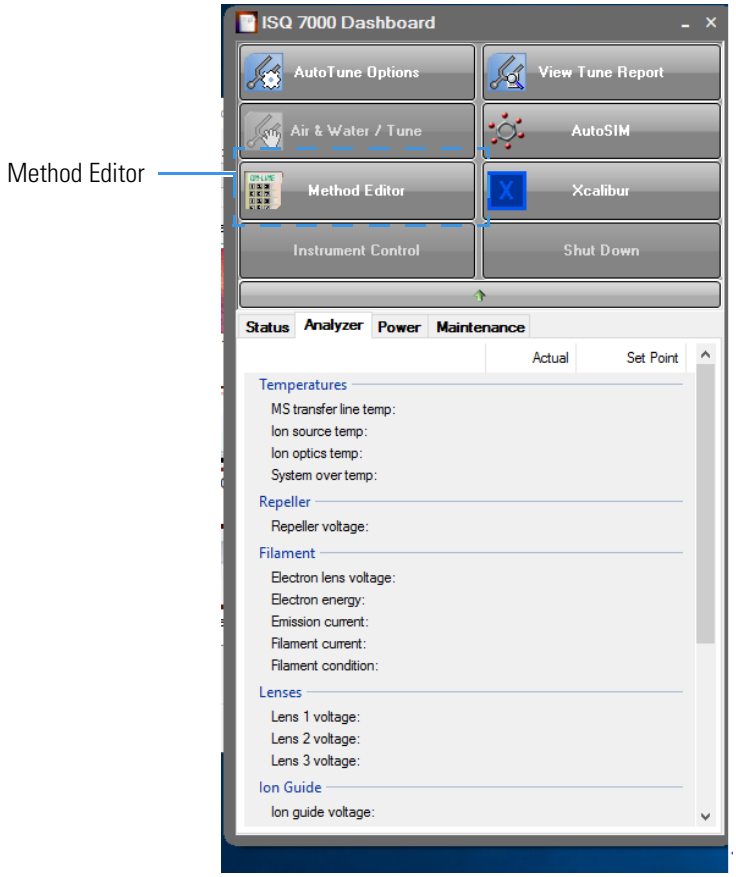
Creating a GC-MS Method in Xcalibur Software

❖ **To create a method for the ISQ 7610 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 7610 Dashboard to open the Method Editor. See Figure 65.

Figure 65. Accessing the Method Editor from the ISQ 7610 Dashboard



Creating an Autosampler Method in Xcalibur Software

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

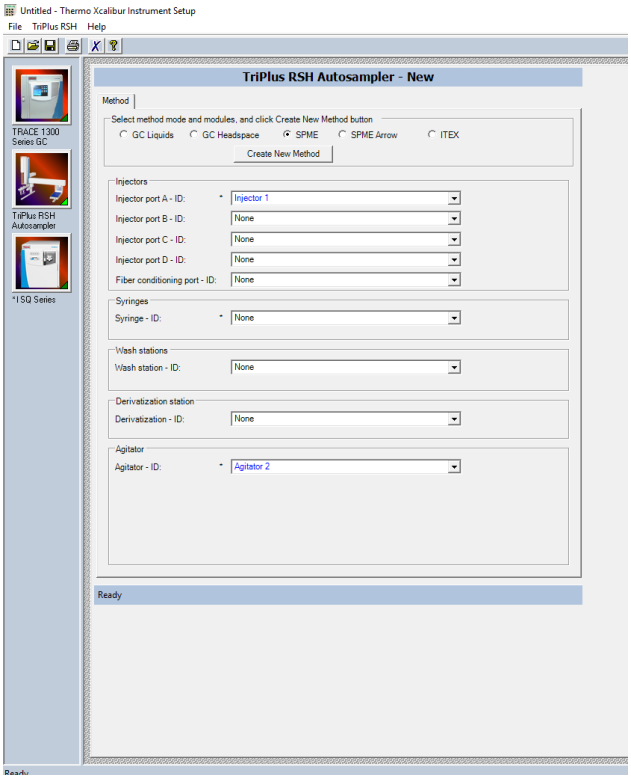
Note Instructions for setting the most common parameters for the TriPlus RSH Sampling System follow. Refer to your autosampler documentation for more detailed information about settings.

❖ To create a method for the autosampler

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

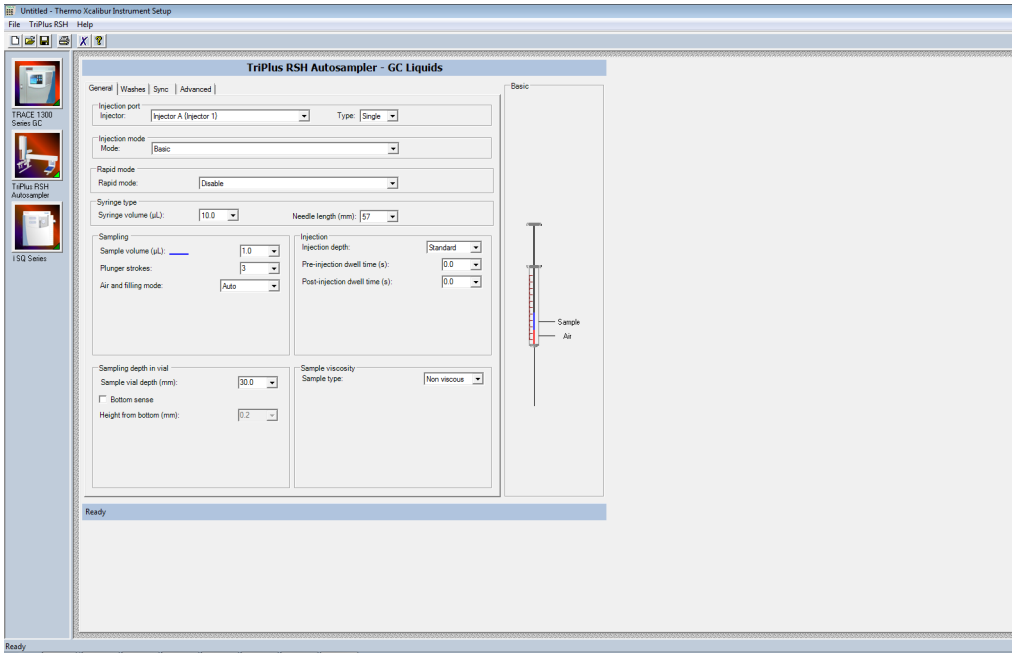
   The method view opens. See Figure 66.

   Figure 66. Autosampler Method



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 67):

Figure 67. 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 µL. 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 µL.

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.

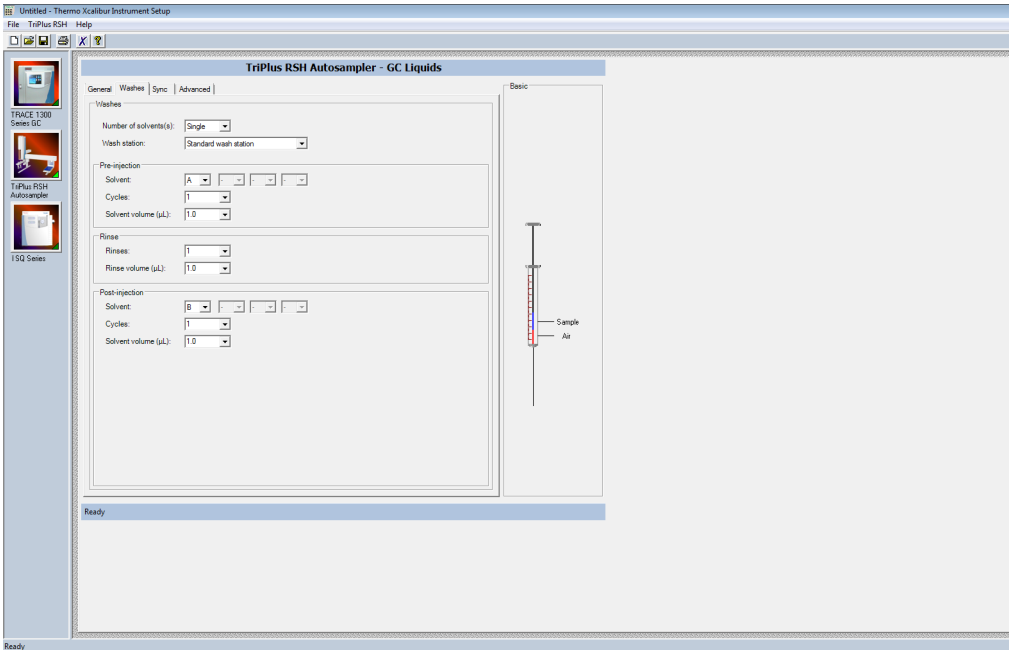
j. Injection Depth—Select Standard or Minimum to indicate how the sample is introduced into the GC. If you select Standard, the autosampler inserts the needle all the way into the injection port. If you select Minimum, the autosampler barely enters the injection port.

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.

l. 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 68):

Figure 68. 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.

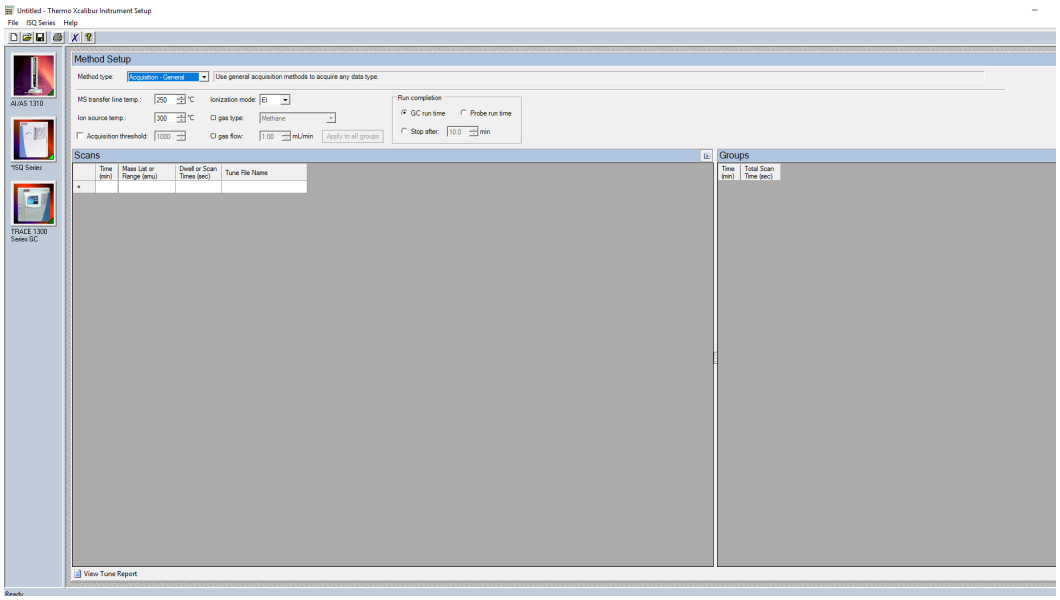
Creating a Method for the ISQ 7610 Mass Spectrometer

Click the ISQ 7610 icon in the left pane to create a method for the ISQ 7610 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 7610 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.

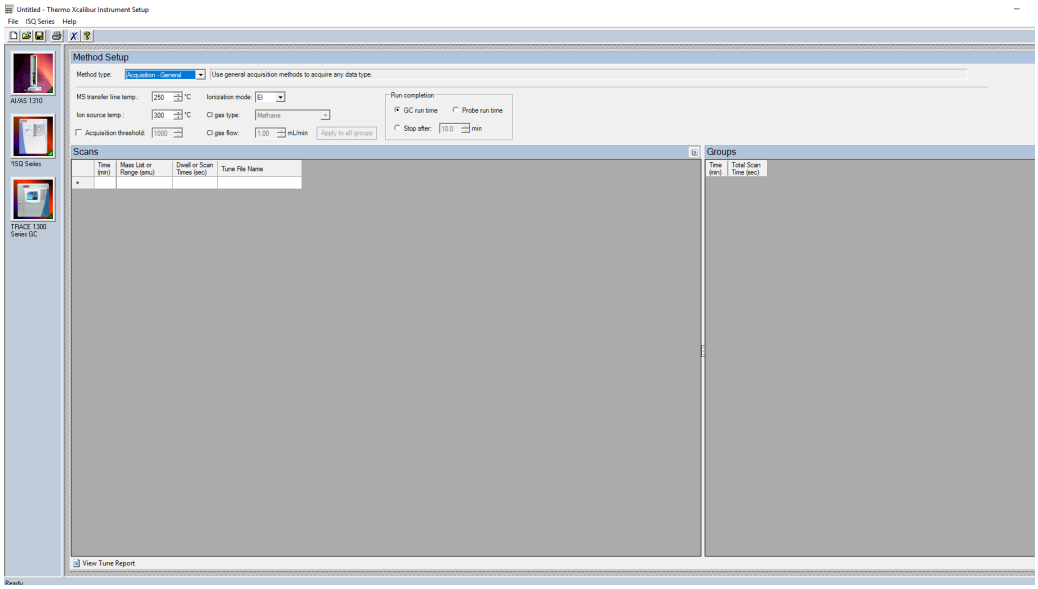
Figure 69. Creating a General Acquisition Method for the ISQ 7610 Mass Spectrometer



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 7610 mass spectrometer. The maximum allowable temperature is 400 °C.

However, you will damage the column and contaminate the ISQ 7610 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 7610 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 7610 system run to end when the GC run is complete. This is the most common setting.

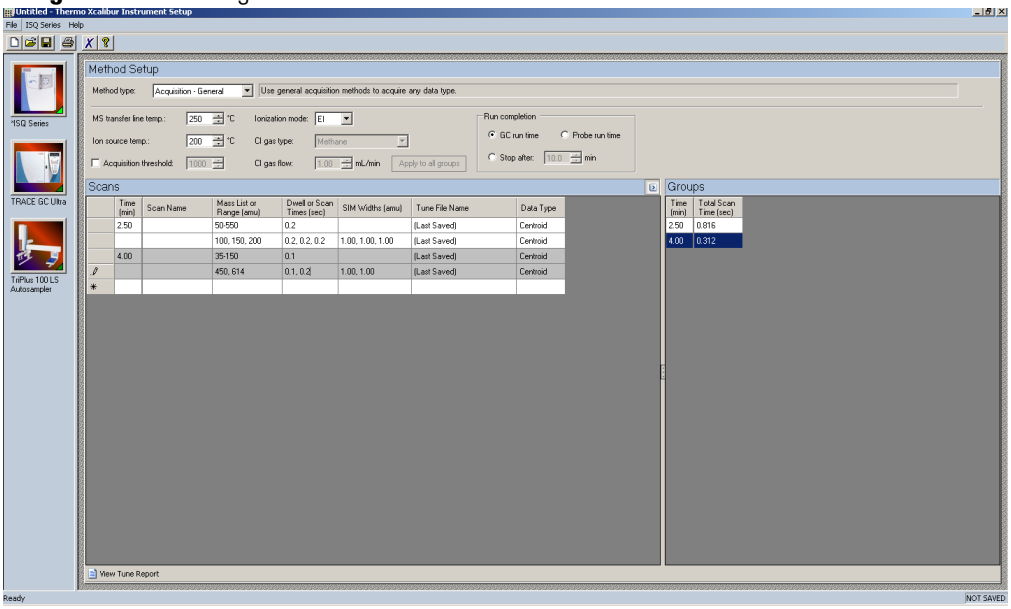
b. **Probe Run Time**—Select this option if you have a probe controller installed and you want the ISQ system run to end when the probe run is complete.

c. **Select Stop After**—Select this option to set the number of minutes you want the ISQ 7610 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 7610 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.

Figure 70. 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 7610 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 7610 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 7610 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 7610 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 7610 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.

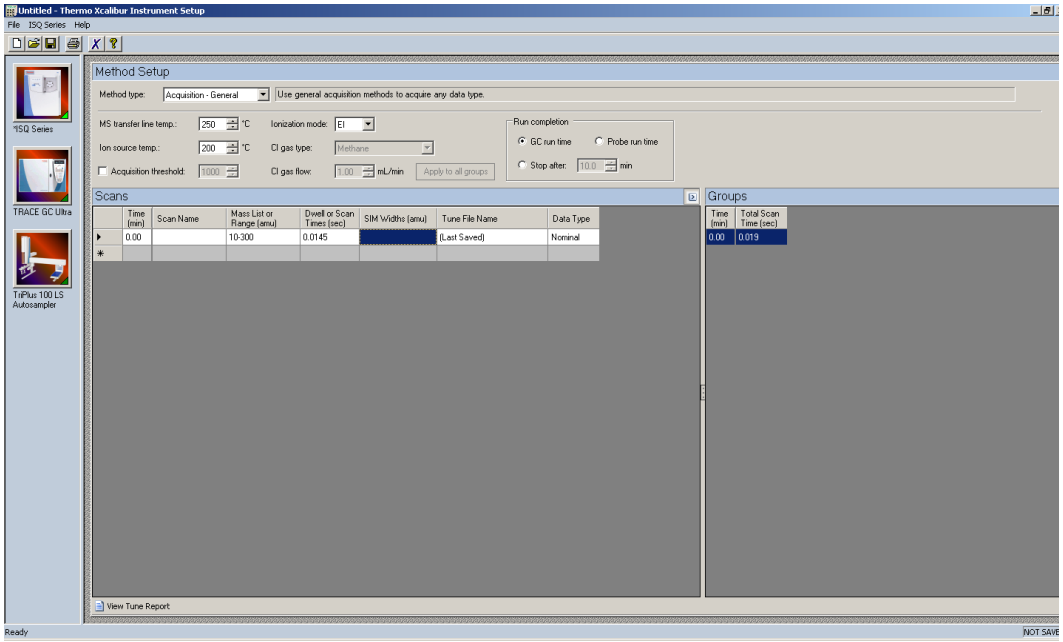
f. The **SIM Widths** column sets the width range of the SIM window. The range of values can be between 0.01 and 10. The default is 1 amu, which means the instrument will collect all the ions from your SIM mass +/- 0.5 amu. Narrower SIM widths lead to greater specificity.

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

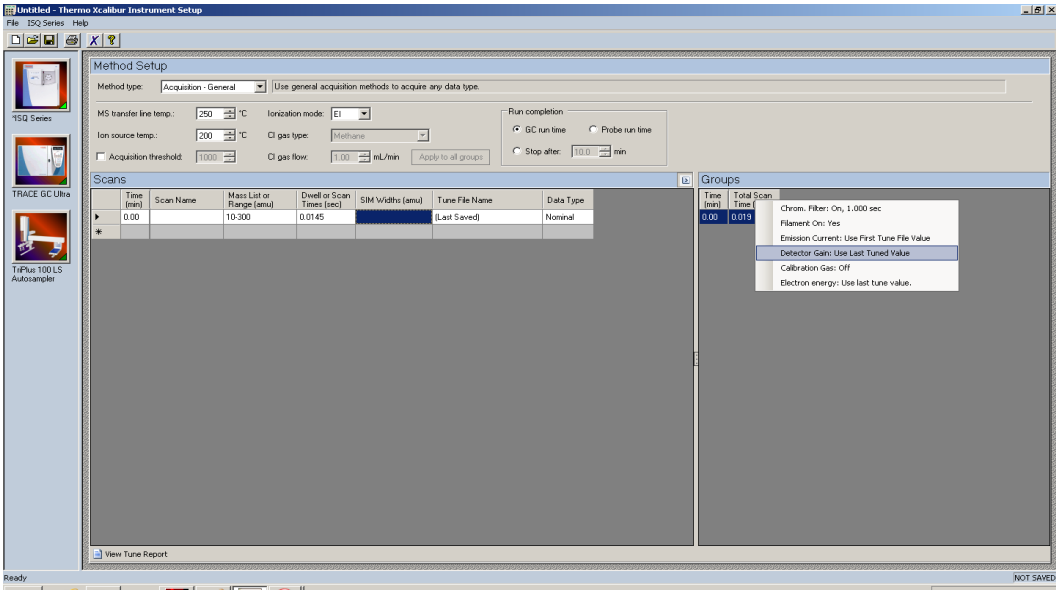
perform fast scanning (up to 20,000 amu/s), select **Nominal** from the drop-down list under **Data Type**.

Figure 71. 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 7610 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 7610 AutoTune or set and scanned in manual tune. If you do not need to use the gain set in ISQ 7610 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.

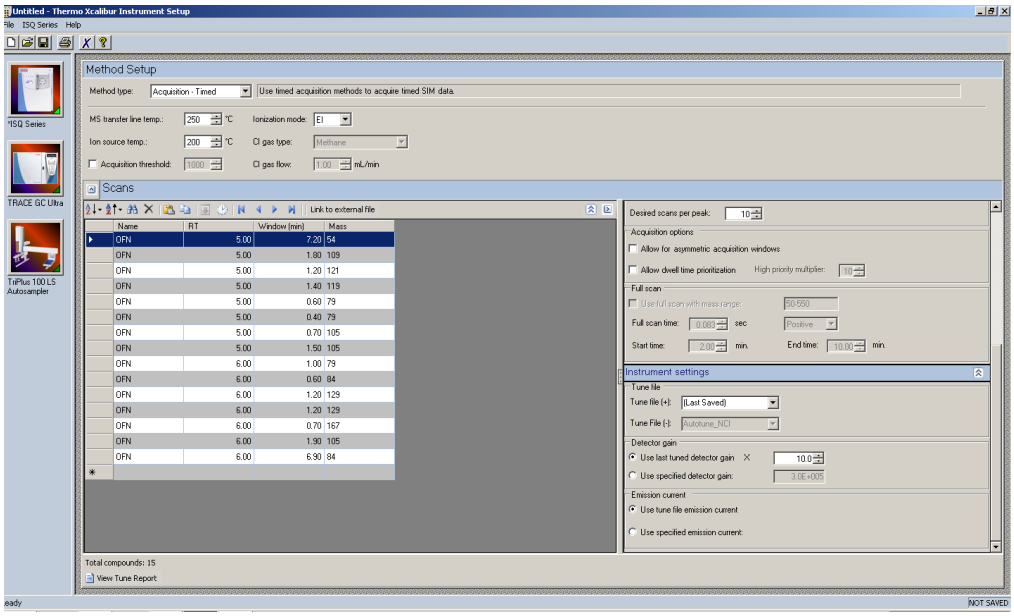
i. Use the **CI Gas Type** column to set the type of gas attached to one or both CI gas ports (A or B). Be sure that the gas you have assigned to a port is actually attached to that port, as each CI gas has a specific calibration of flow vs. gas viscosity.

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:

Figure 72. 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 7610 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 7610 Mass Spectrometer," The software defaults to the most recent **AutoTune_NCI** or **AutoTune_PCI** tune file you created.

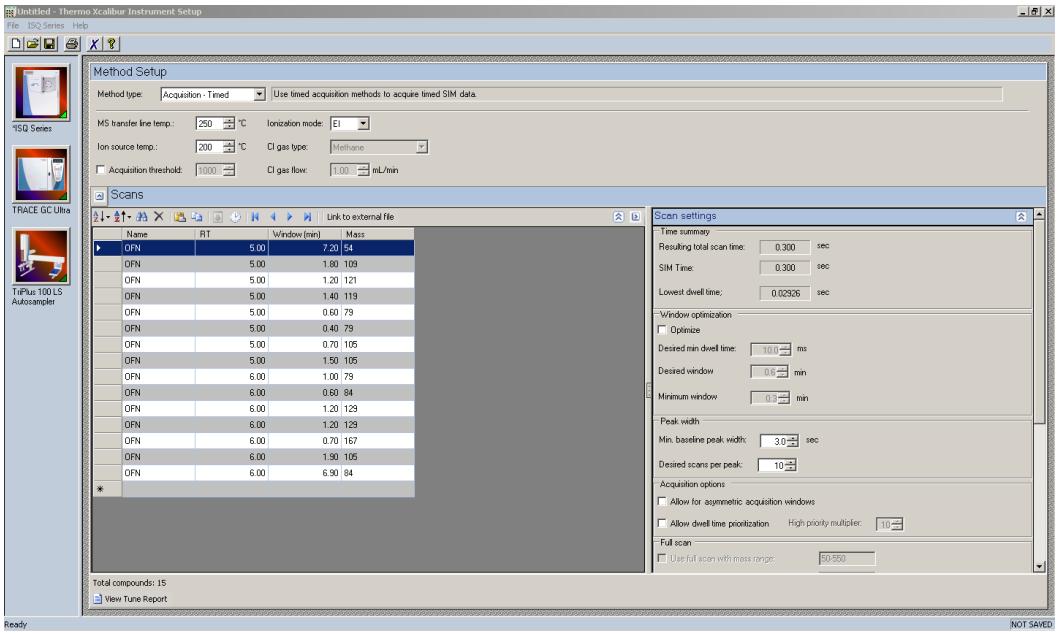
b. In the **Detector Gain** area, set the detector gain. Select the **Use Last Tuned Detector Gain** option to indicate that you want to use the detector gain set in ISQ 7610 AutoTune. If you do not need to use the gain set in ISQ 7610 AutoTune, then you can set the gain manually. Higher gains give larger 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 ± 0.5 µA.

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

Figure 73. Acquisition-Timed Scan Settings



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 Acquisition**s 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.

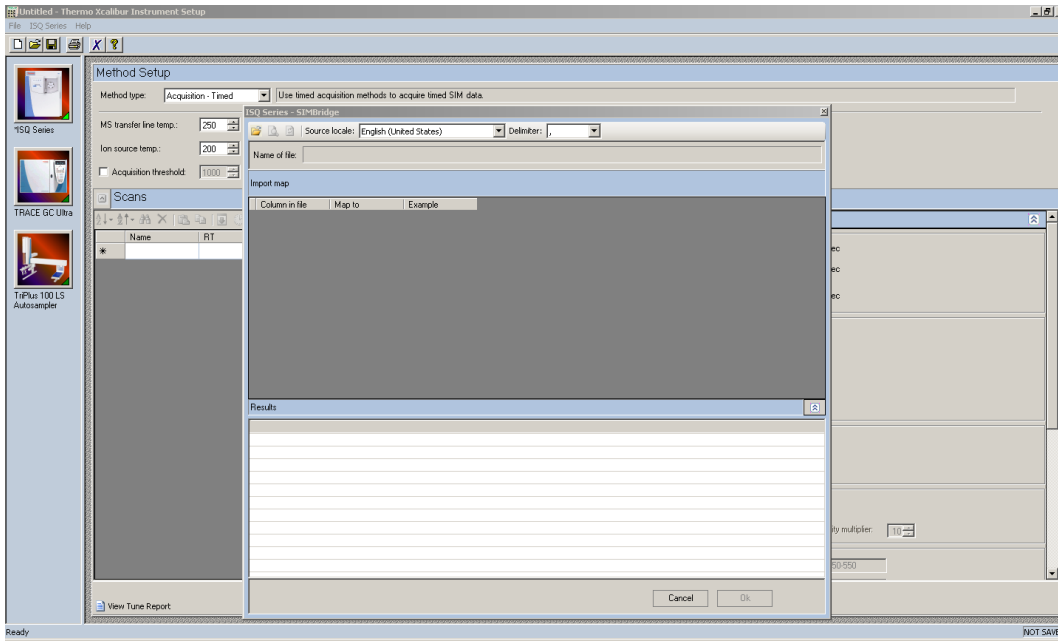
Select the **Allow Dwell Time Prioritization** check box to increase the dwell times for selected scans. The choices for each scan are Normal or High. Giving a scan high priority increases its dwell time by the value you set in the High Priority Multiplier box.

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 7610 Method Editor, clear the **Link to External File** check box.

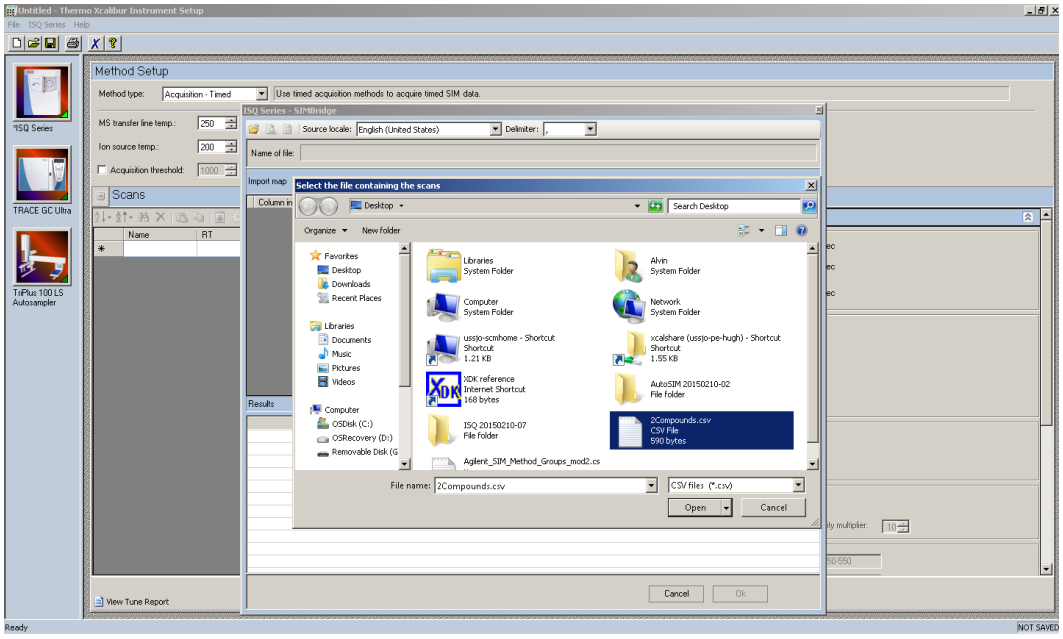
g. After clicking **Link to External File**, the **SIMBridge** dialog box opens. Choose the language of your method file from the **Source Locale** drop-down menu.

Figure 74. Setting the Source Language of Method Files using SIMBridge



h. Browse to your file.

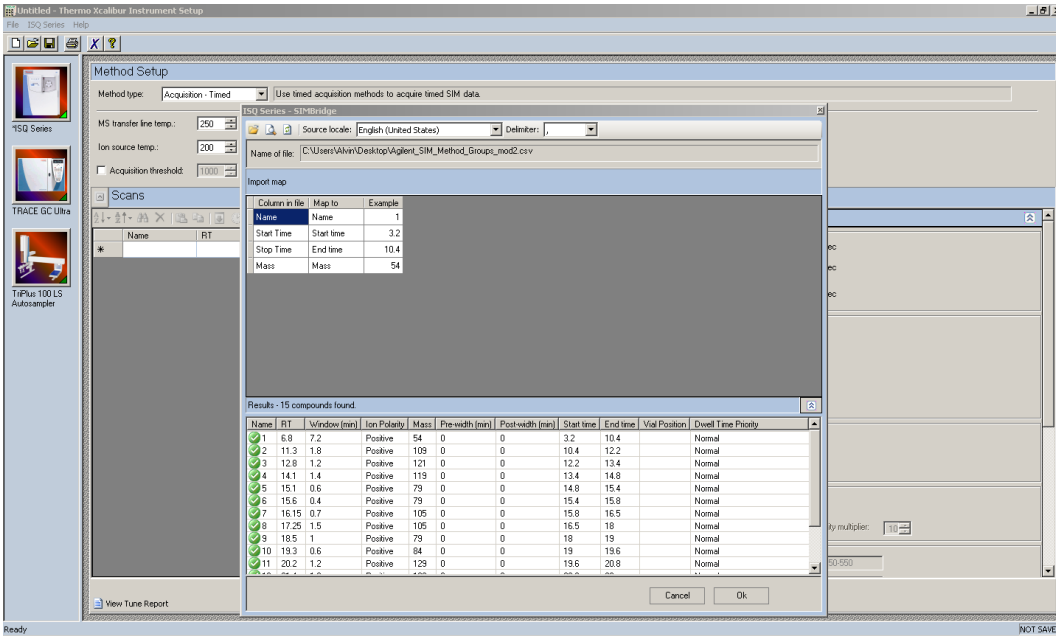
Figure 75. Linking to an External File using SIM Bridge



i. Click **Open** to open the method in SIMBridge.

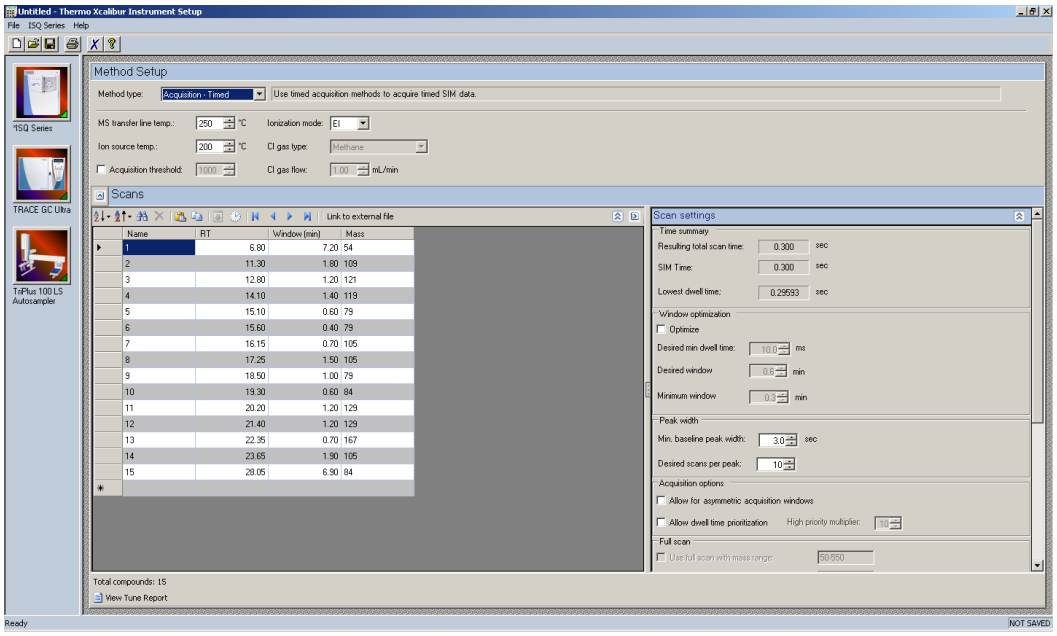
j. If necessary, change the method headings in your original file to match those in the method editor. A green check mark appears when your method is validated.

Figure 76. Changing Method Headings in SIMBridge



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

Figure 77. Viewing a Linked File in the Method Editor



l. 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.

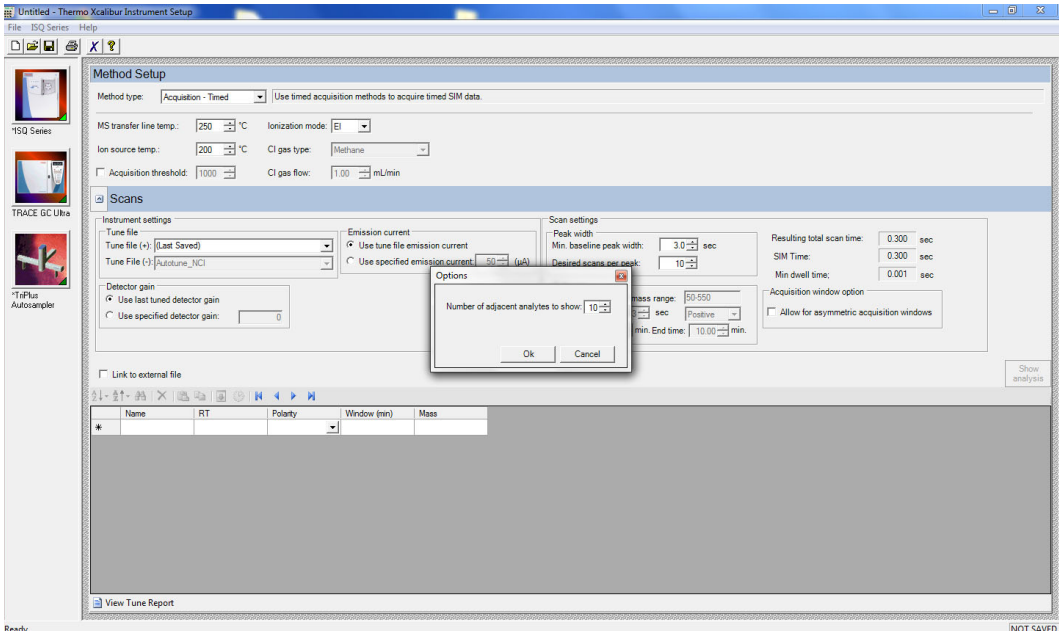
o. In the **Mass** column, enter the mass of the ion you wish to monitor.

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 7610** main menu. See Figure 78. View the number of analytes you set in the **Show Analysis** view.

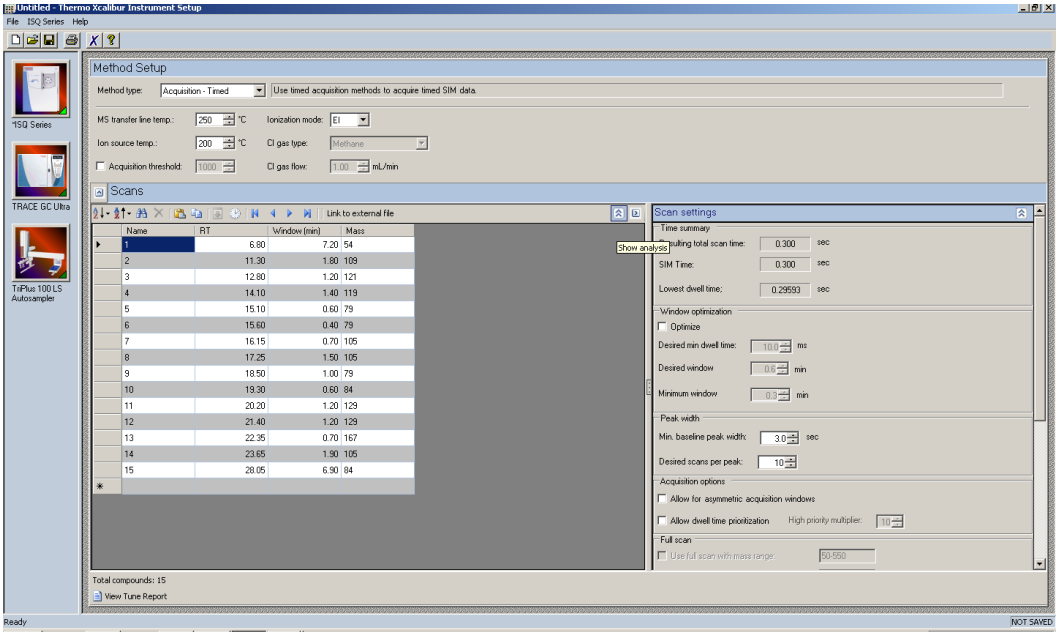
Adjustments in this column are for display purposes only and will not affect your acquisition.

Figure 78. Options Dialog Box



r. Click **Show Analysis** (see Figure 79) to validate your method. A chart appears with all your analytes by name and in order of their start times.

Figure 79. Accessing the Show Analysis Feature



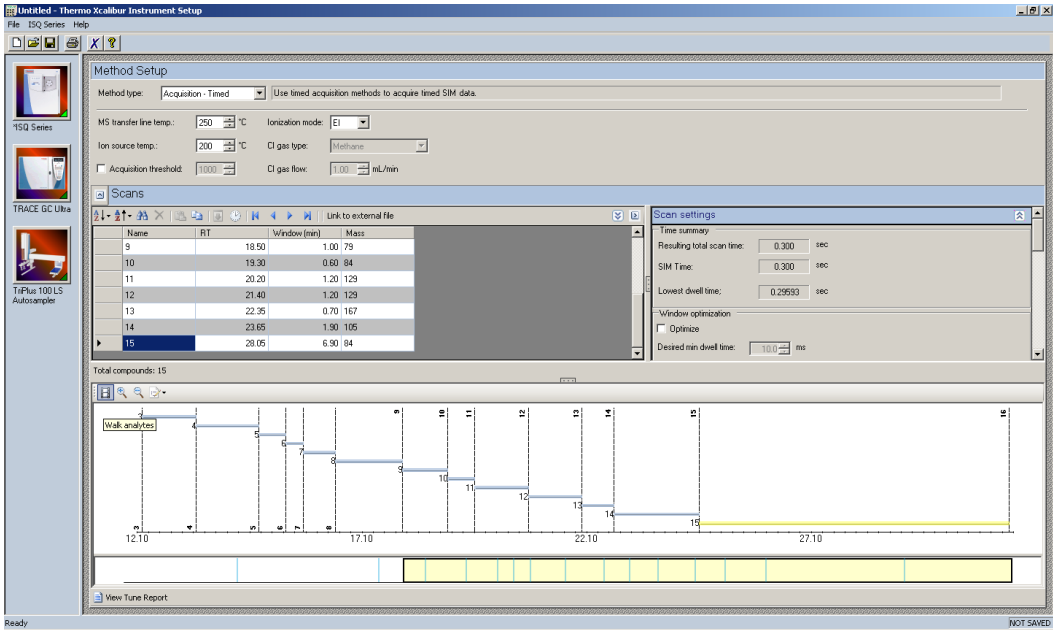
s. Use the scrolling window at the bottom of the screen to view all your analytes' expected retention times. Resize the window to view more analytes by dragging one side of the window out. As the scrolling window is decreased in size, fewer analytes are shown in the analytes chart. Increasing the size 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.

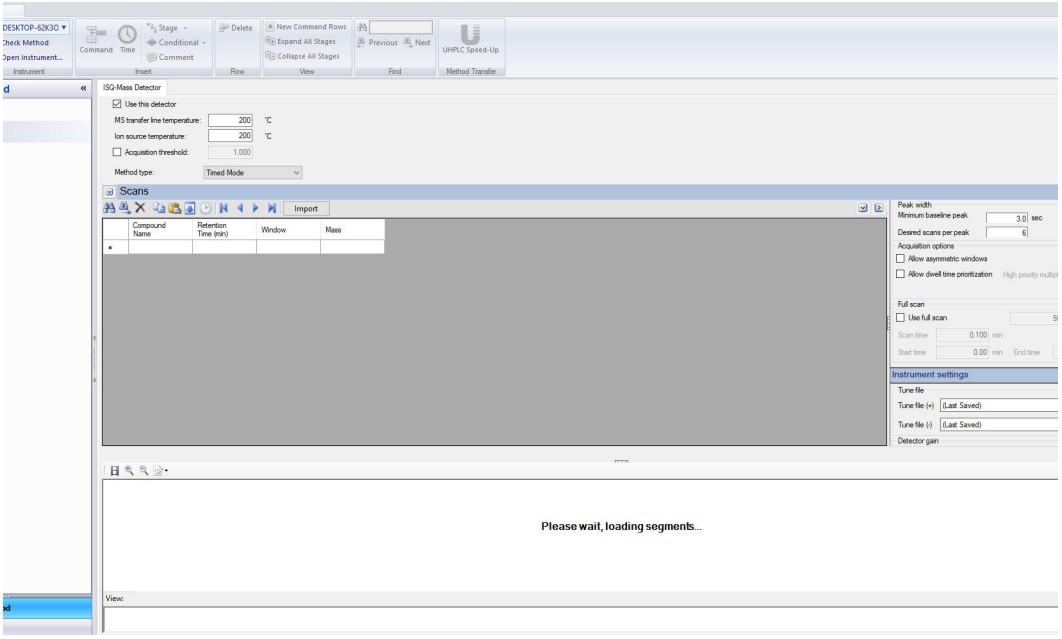
Figure 80. Validating a Method



11. To import an MS method, choose **Import.** See Figure 81.

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

Figure 81. Importing Methods



12. Choose **ISQ 7610 | 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.

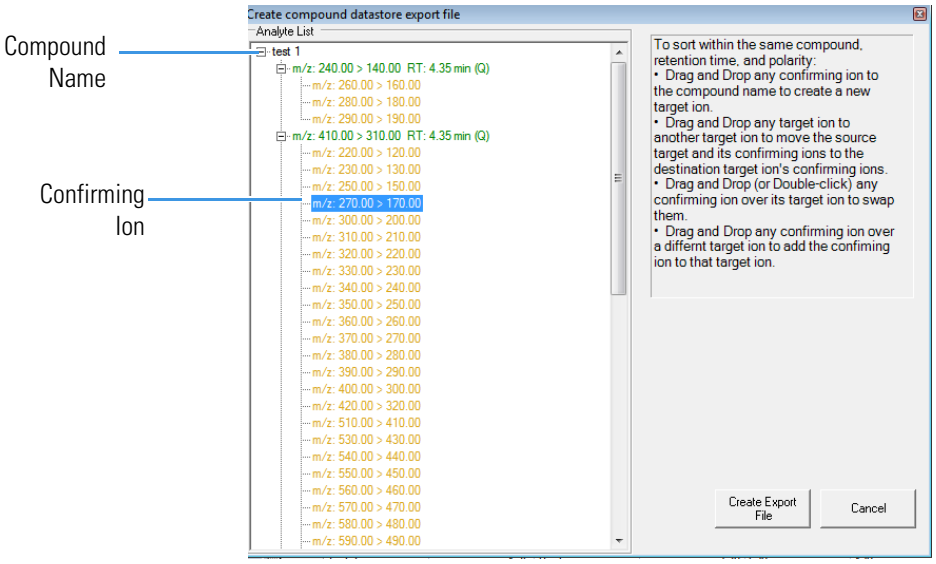
Tip Export a timed scan list to see an example of a valid format.

13. Choose **ISQ 7610 | 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.

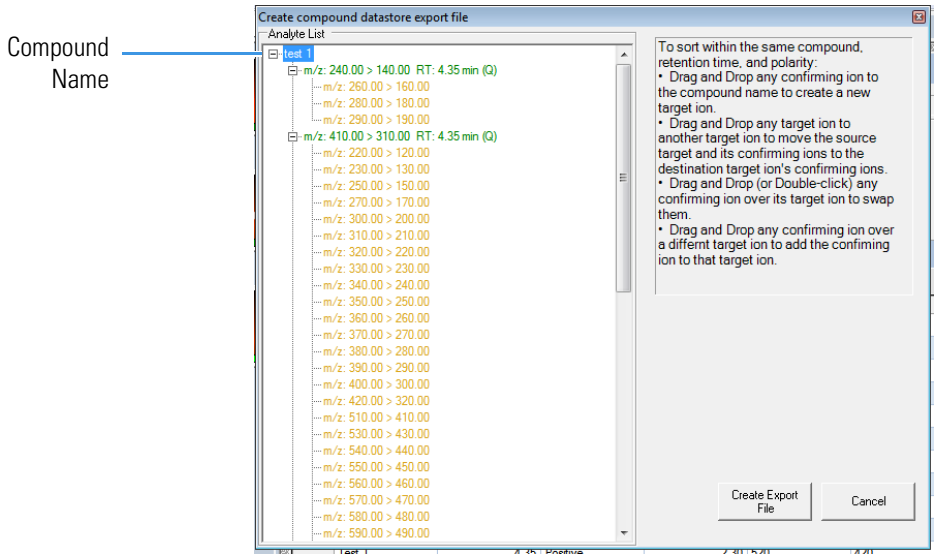
14. Choose **ISQ 7610 | Export Timed Scans** to export your method as a .csv file. If you prefer editing your methods in spreadsheet applications, you may want to use this option.

15. Choose **ISQ 7610 | Create Compound Data Store Export File** to prepare your file for the compound data store in the TraceFinder application.

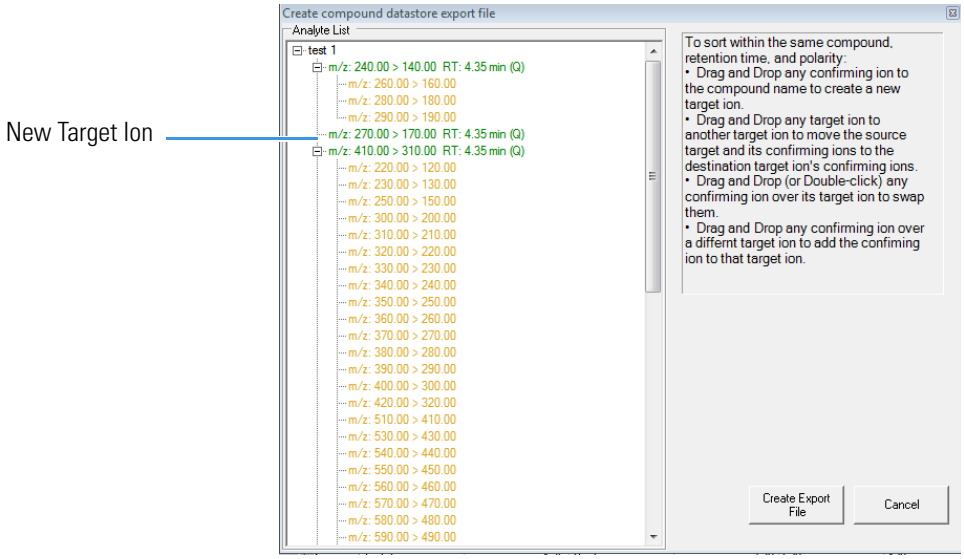
    a. To create a new target ion from the list of confirming ions with identical retention times for a compound, select the confirming ion of interest.



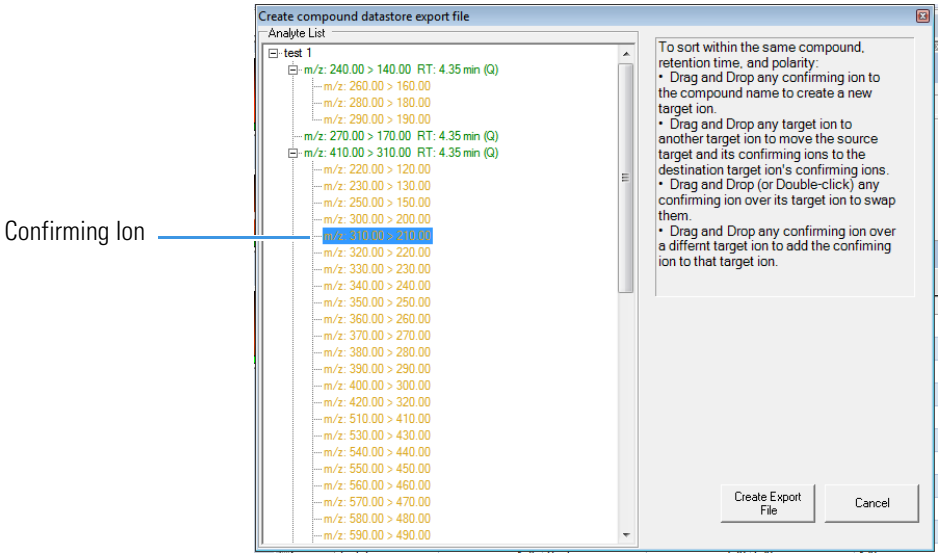
    b. Drag the confirming ion to the compound name at the top of the list.



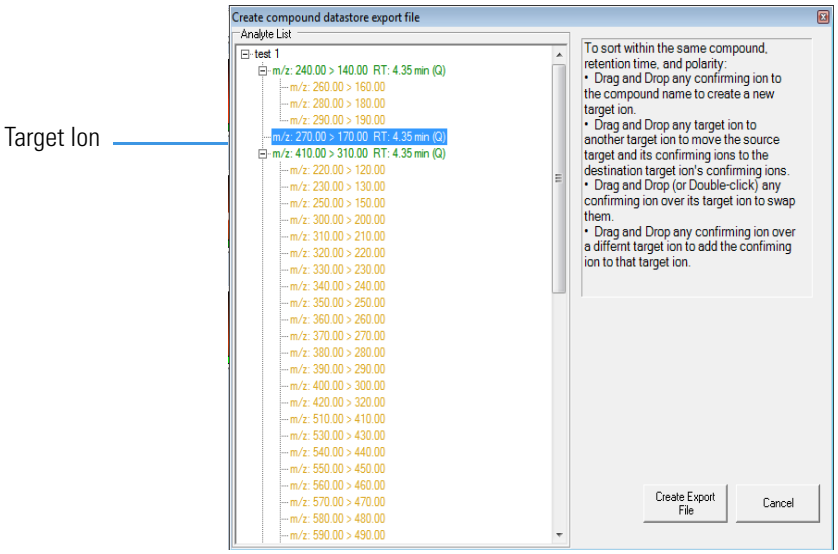
c. The confirming ion now appears in the list as a target ion.



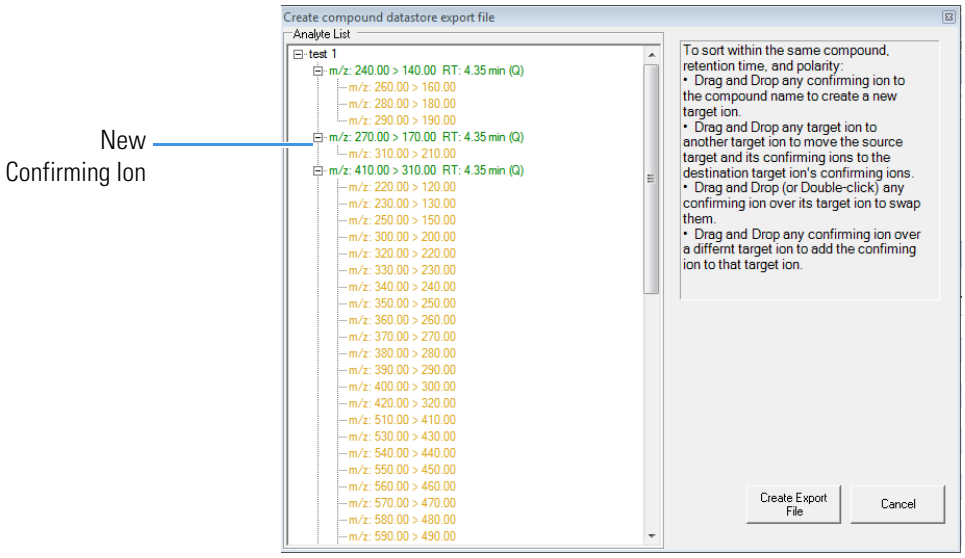
d. To add a new confirming ion to a target ion in the list, select the confirming ion of interest.



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



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

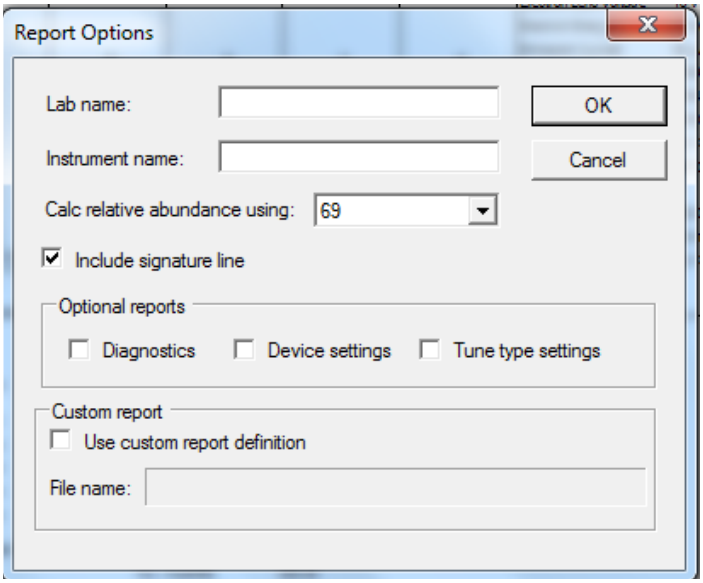


g. Click **Create Export File** when you are through creating your ion list to export the list to the TraceFinder software compound database.

16. Choose **ISQ 7610 | Create Segment List From Timed Scan List** to import a general acquisition method.

17. Choose **ISQ 7610 | 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 82) and add identifying information to the tune report.

Figure 82. Configuring the Tune Report Options



Creating a Method for the GC in Xcalibur Software

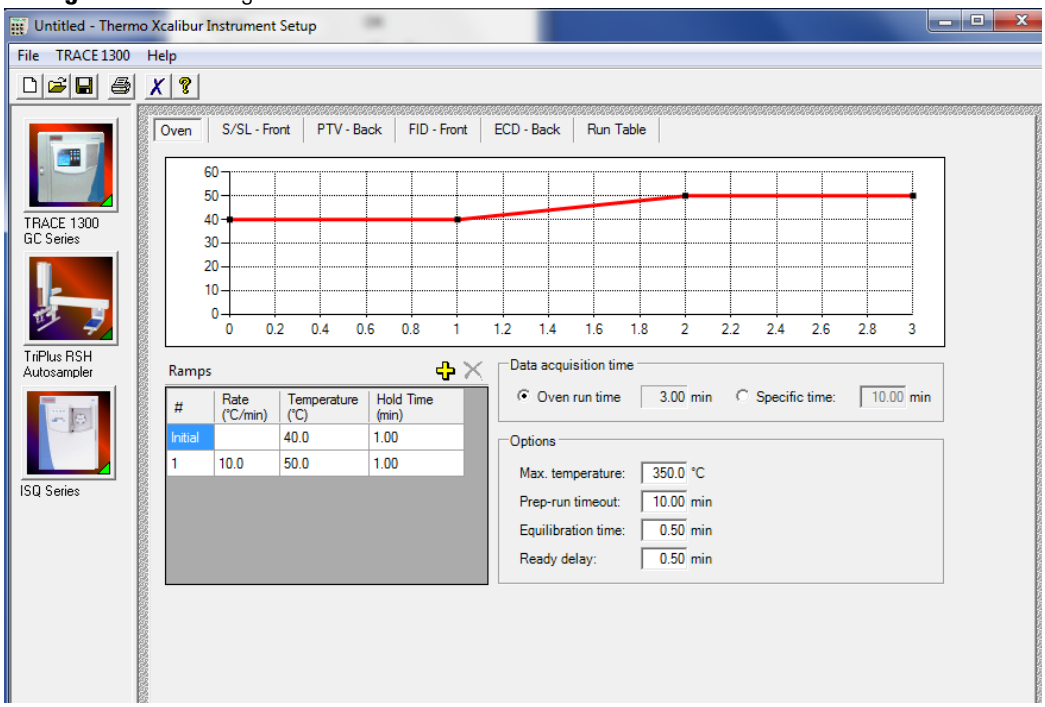
Click the GC icon in the left pane to create a GC method.

Creating a method for the TRACE 1600 or TRACE 1610 GC using Xcalibur Software

❖ To create a method for the TRACE 1600 or TRACE 1610 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 83. Setting the GC Oven Parameters on the TRACE 1600 or TRACE 1610 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.

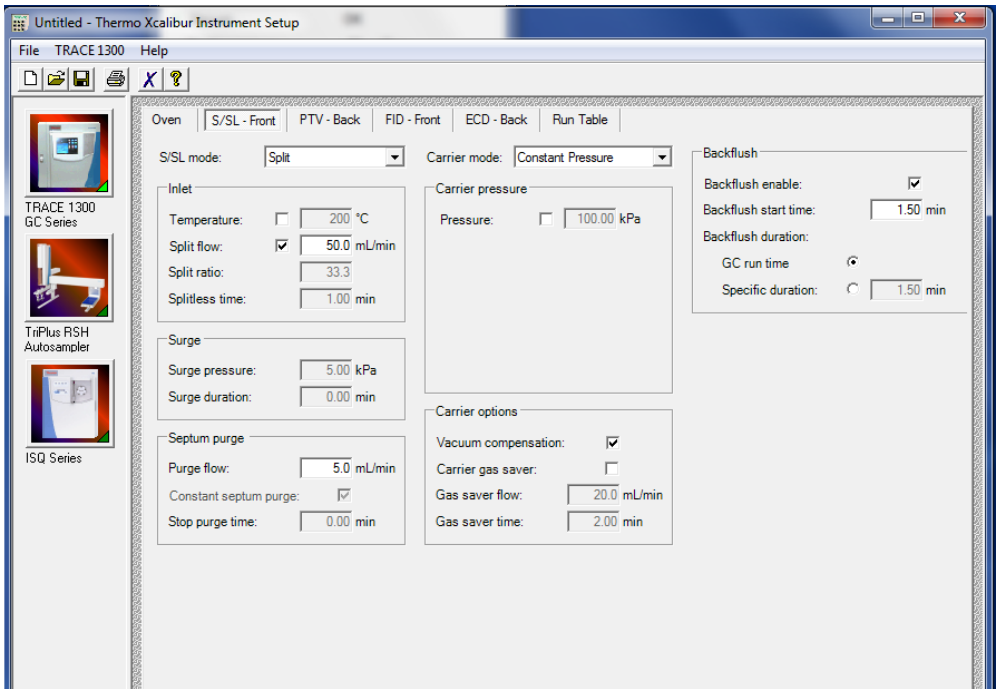
b. You can also select the maximum allowed oven temperature, which should be set to the maximum temperature allowed by your method, *not* the maximum allowed by your column. The maximum temperature allowed by the GC is 450 °C. This will prevent you from accidentally using a temperature that will damage 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 7610 mass spectrometer.

2. The TRACE 1600 and 1610 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 7610 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).

Figure 84. Locating the Settings for the SSL Injector on the TRACE 1600 or TRACE 1610 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 7610 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

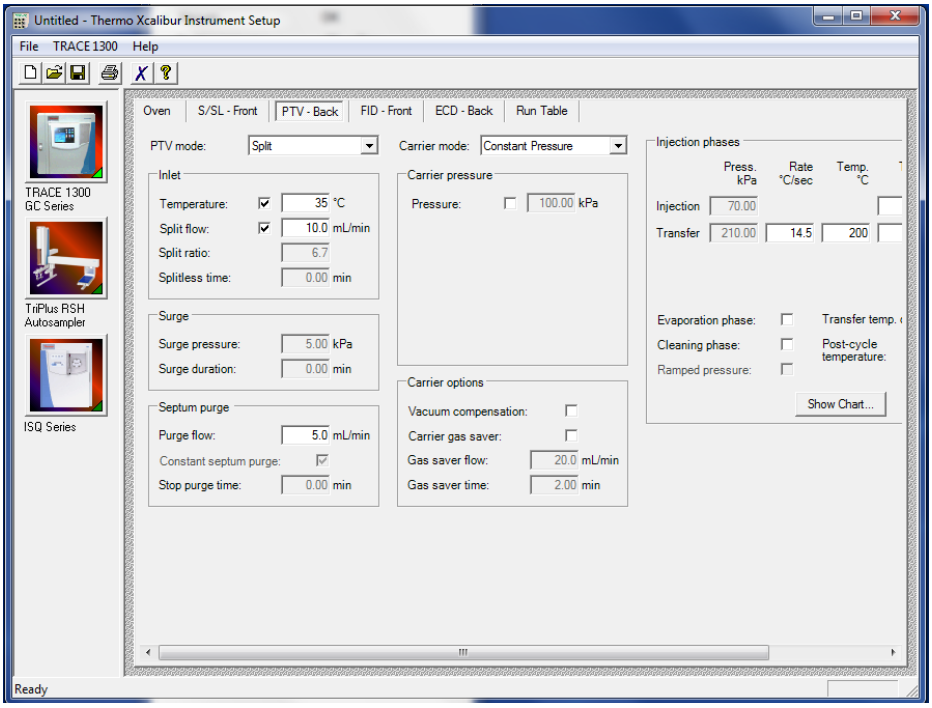
ISQ 7610 instrument, which is under vacuum, the vacuum compensation *must* be on to ensure accurate flow rates.

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 7610 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.

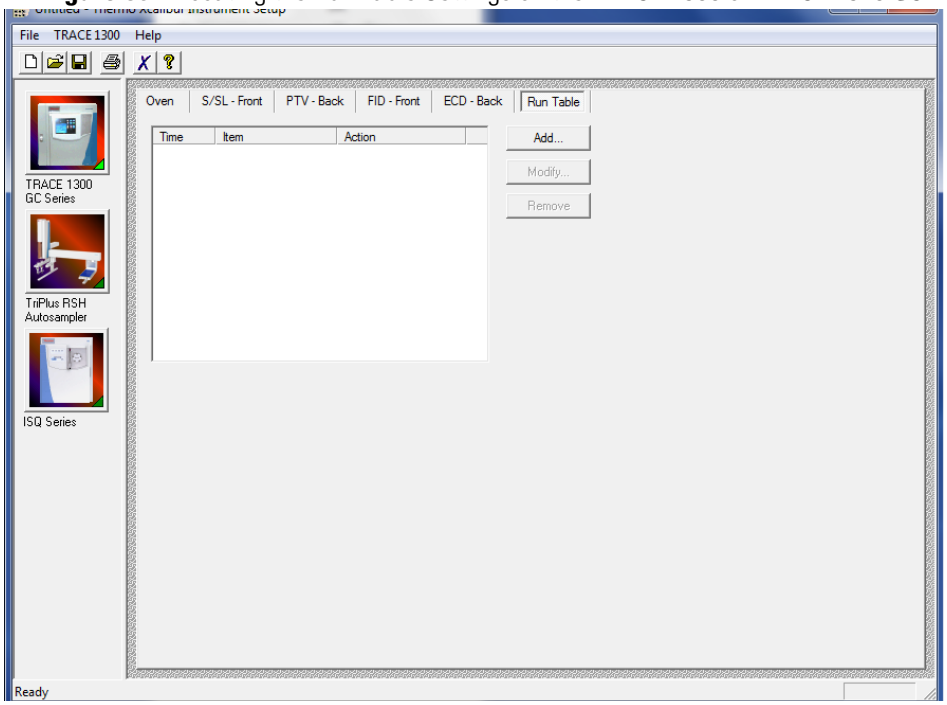
Figure 85. Locating the Carrier Gas Settings for the TRACE 1600 or TRACE 1610 GC



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.

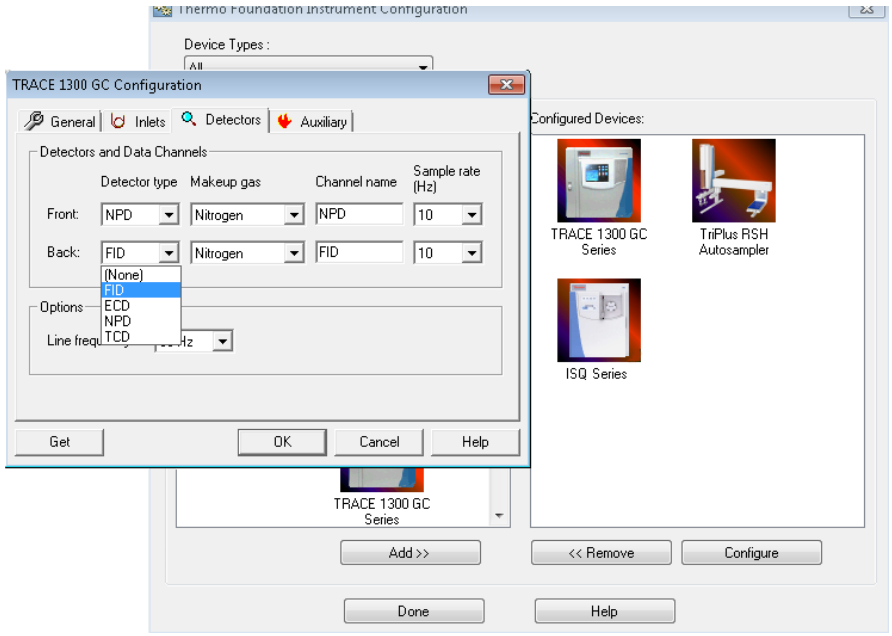
Figure 86. Locating the Run Table Settings on the TRACE 1600 or TRACE 1610 GC.



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 1600 or TRACE 1610 GC icon. Select **Configure**.

Figure 87. Configuring the Inlets or Detectors on the TRACE 1600 or TRACE 1610 GC

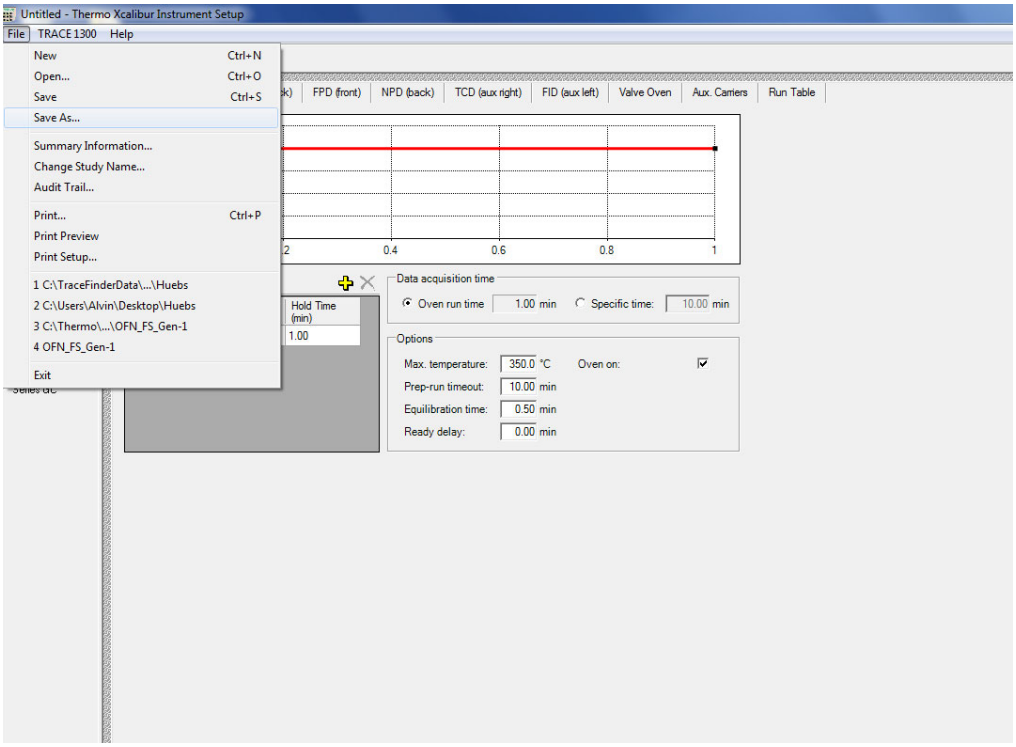


d. Select the **Inlet** or **Detector** tab as appropriate.

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 88. Saving a TRACE 1600 or TRACE 1610 GC Method



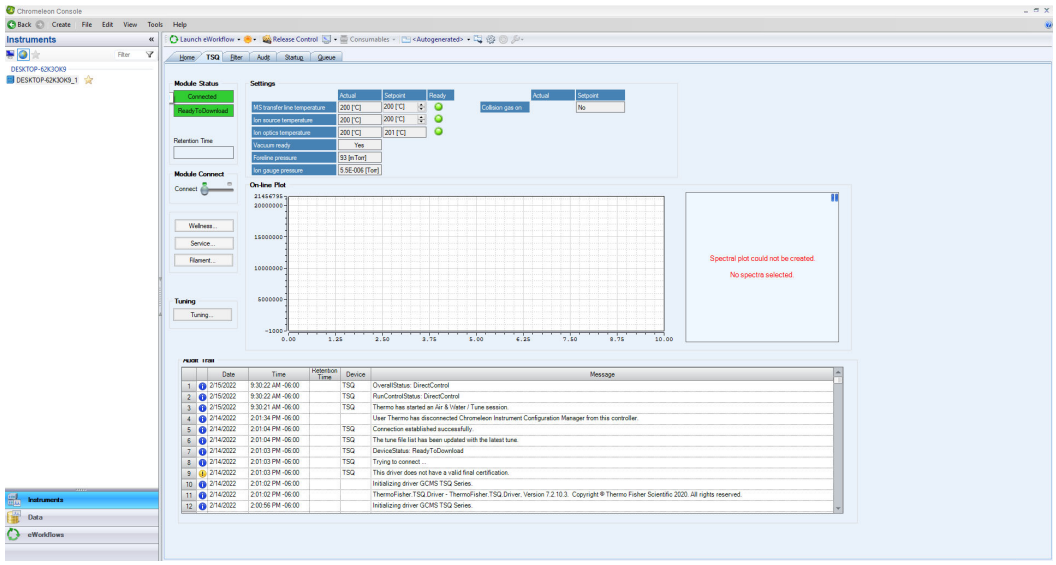
Creating a GC-MS Method in Chromeleon Software

If your system is running Chromeleon software, follow the instructions in this section to create a method for the autosampler, GC, and ISQ 7610 mass spectrometer.

❖ To create a GC-MS method in Chromeleon Software

1. Open the Chromeleon Console. Select **ISQ** from the left menu. The ISQ window opens. See Figure 89.

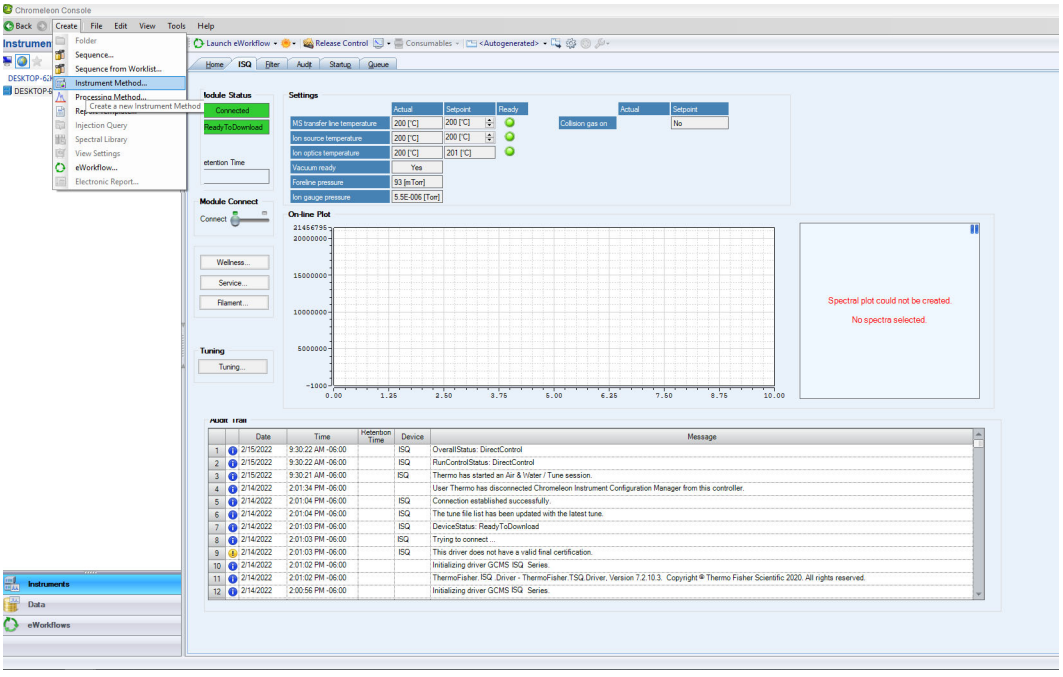
Figure 89. ISQ Window



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.

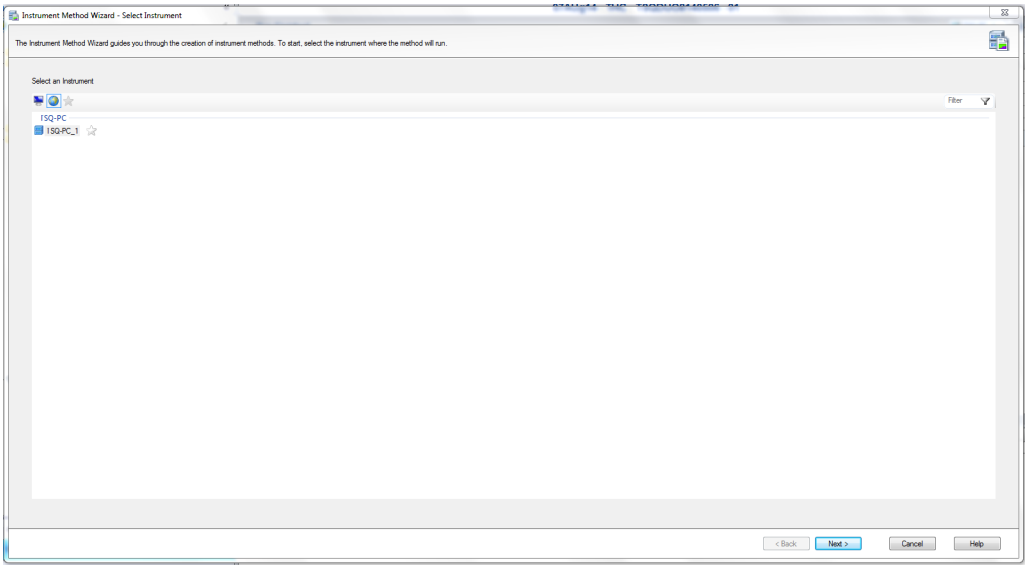
2. Go to **Create > Instrument Method**.

Figure 90. Creating an Instrument Method



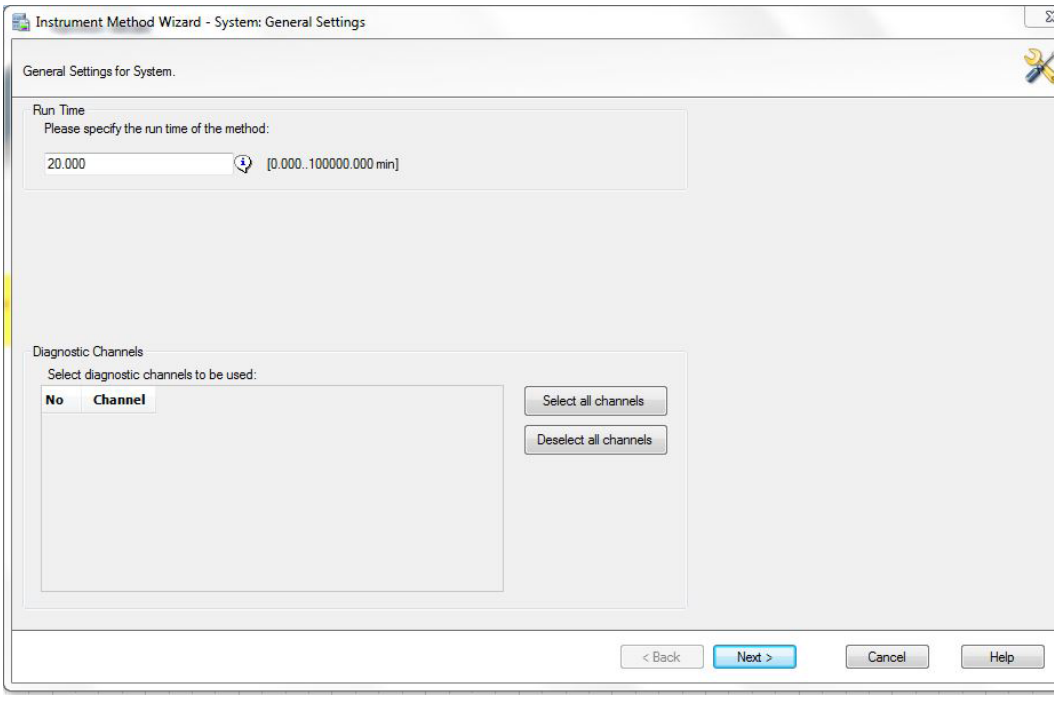
3. The **Instrument Method Wizard – Select Instrument** window opens. See Figure 91.

Figure 91. Instrument Method Wizard – Select Instrument



4. Select the instrument you want to configure and click **Next**.
5. The **Instrument Method Wizard – System: General Settings** window opens. See Figure 92.

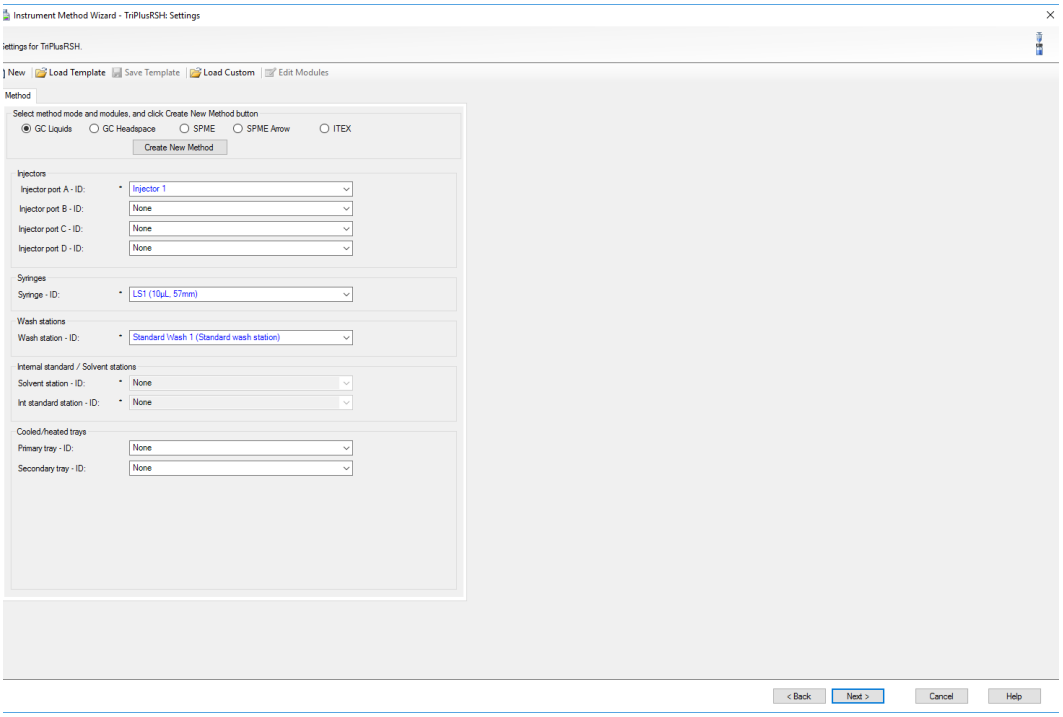
Figure 92. Instrument Method Wizard – System: General Settings



6. You can set the GC Run Time for the method here, or load it with the other GC settings later. Click **Next**.

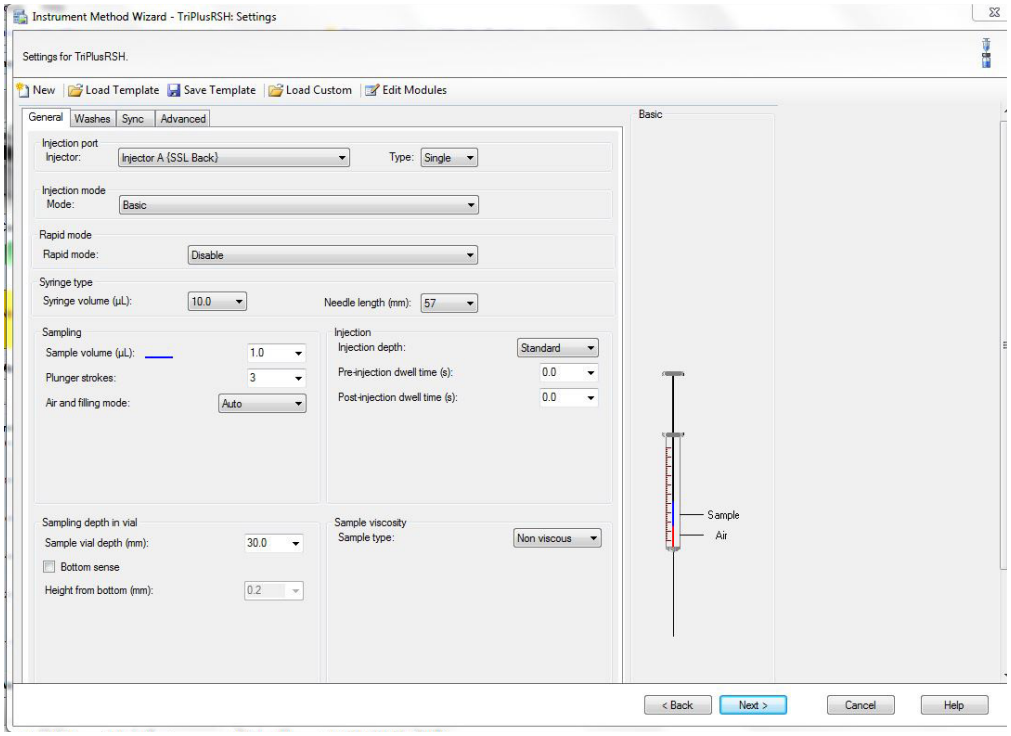
7. The **Instrument Method Wizard – TriPlusRSH: Settings** window opens. See Figure 66.

Figure 93. Instrument Method Wizard – TriPlus RSH: Settings



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 94.

Figure 94. 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 µL. 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 µL.

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.

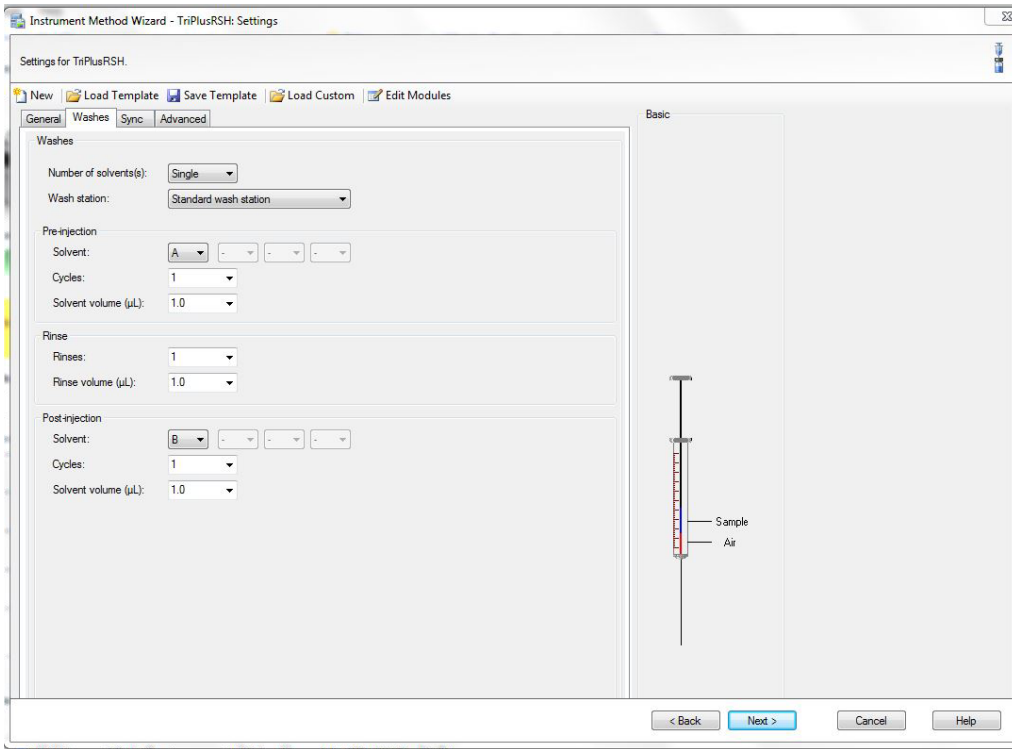
j. Injection Depth—Select Standard or Minimum to indicate how the sample is introduced into the GC. If you select Standard, the autosampler inserts the needle all the way into the injection port. If you select Minimum, the autosampler barely enters the injection port.

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.

l. 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 95):

Figure 95. 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

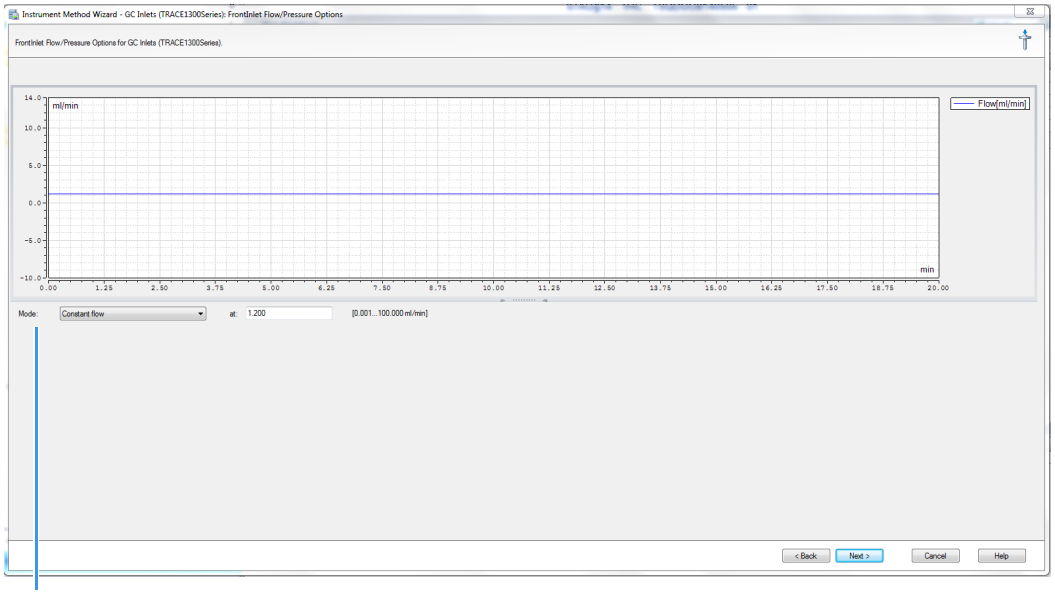
purged with the same solvent that was used in location A, B, C, or D or with solvents A and B or C and D. Typically, there is 0 or 1 cycles of pre-injection purges.

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.

13. Click **Next**. The **Instrument Method Wizard – GC Inlets(TRACE1600/1610): FrontInlet Flow/Pressure Options** window opens. See Figure 96.

Figure 96. Instrument Method Wizard – Front Inlet Flow/Pressure Options



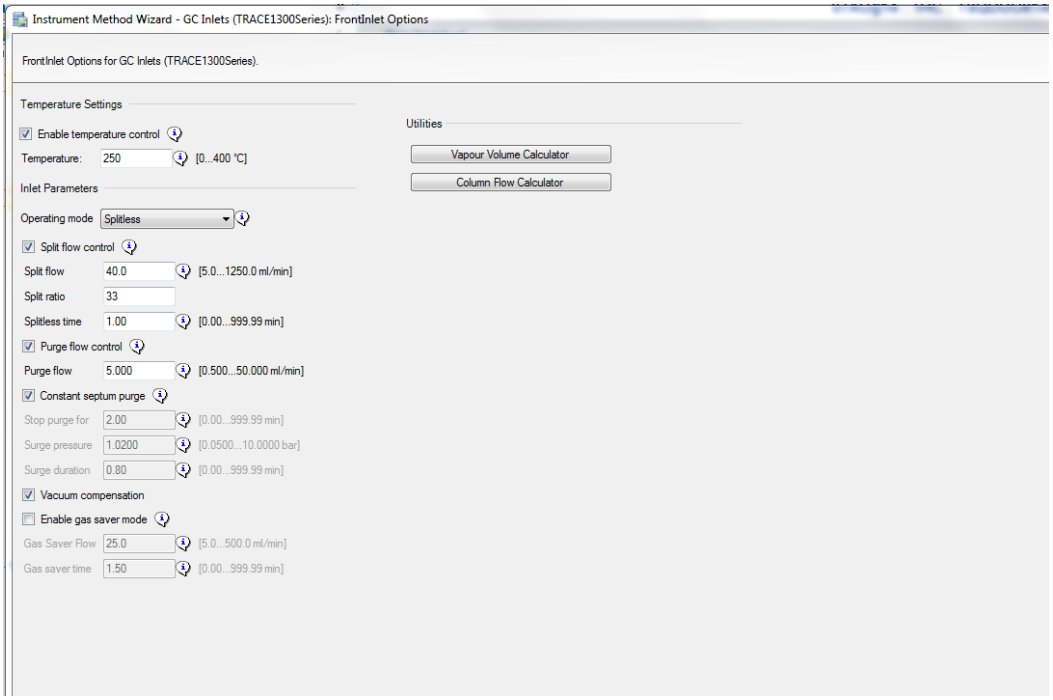
Carrier Gas
Mode

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(TRACE1600/1610): FrontInlet Options** window opens. See Figure 97.

Figure 97. Instrument Method Wizard – GC Inlets(TRACE1600/1610): Front Inlet Options



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.

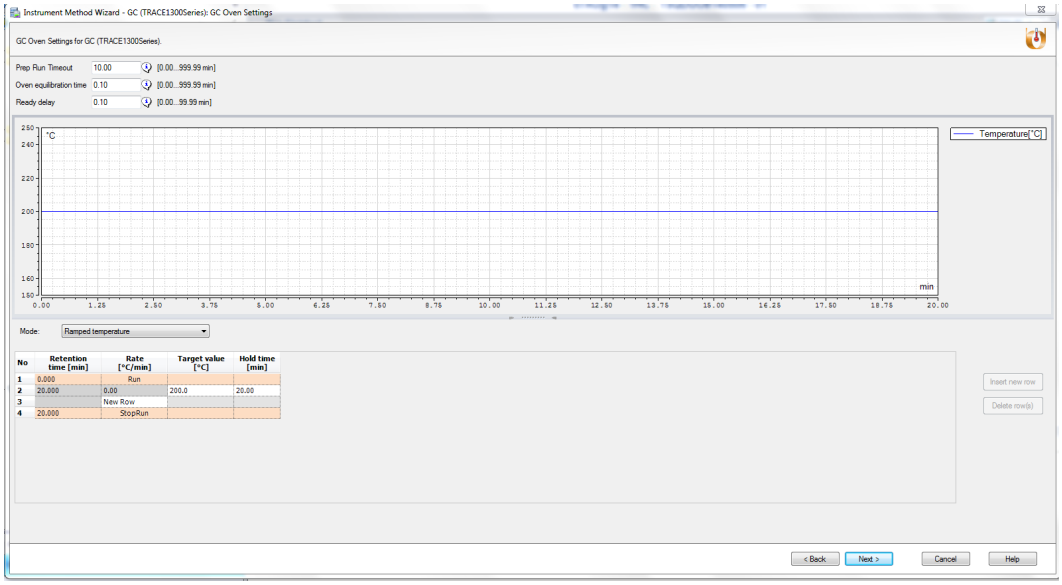
d. If your analysis requires a higher flow to quickly sweep the analytes into the column, which may be needed with high temperature injectors and thermally labile compounds, use the surge pressure to increase the column flow for the surge duration time.

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(TRACE11600/1610): GC Oven Settings** window opens. See Figure 98.

Figure 98. Instrument Method Wizard – GC Inlets: GC Oven Settings



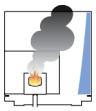
18. Set the oven temperatures in this window. There is always at least one temperature and time in any GC temperature program. In the Initial row, enter the initial temperature, which must be 4 °C (7 °F) above room temperature and less than the maximum operating temperature of your GC column. If you 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 1600/1610 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.

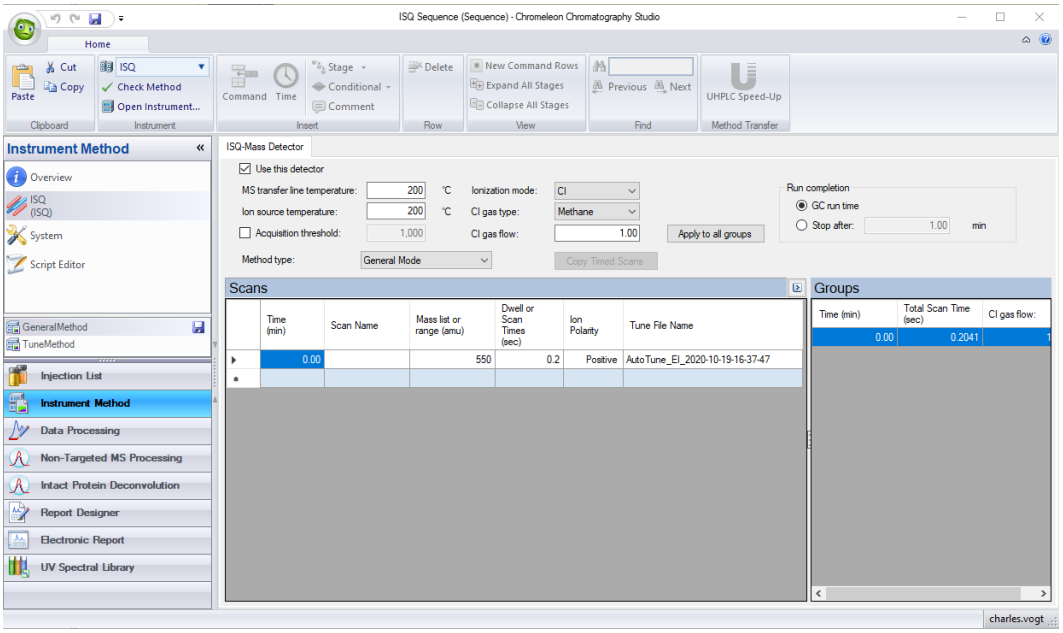
25. Click **Next**.

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

Note The maintenance method is used to bake out or cool down the ISQ 7610 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 99.

Figure 99. Creating a General Acquisition Method for the ISQ 7610 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 7610 mass spectrometer. The maximum allowable temperature is 400 °C. However, you will damage the column and contaminate the ISQ 7610 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.

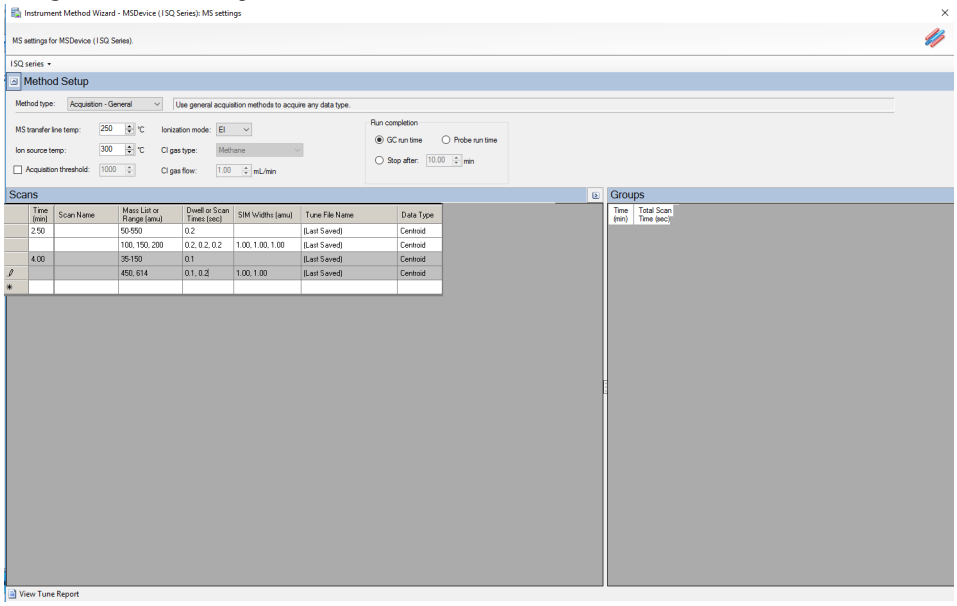
Note Only use CI if you have installed a CI ion volume in the ISQ 7610 instrument and you have connected CI reagent gas to your system. If you have CI, select a CI Gas Type from the pull-down menu and set the CI Gas Flow. (There will be a two-minute delay when you change ports for the CI Gas Type.) Typical 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 7610 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 7610 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 7610 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.

Figure 100. 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 7610 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 7610 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 7610 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 7610 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 7610 Mass Spectrometer. You can also use a specific tune file for each of the scans.

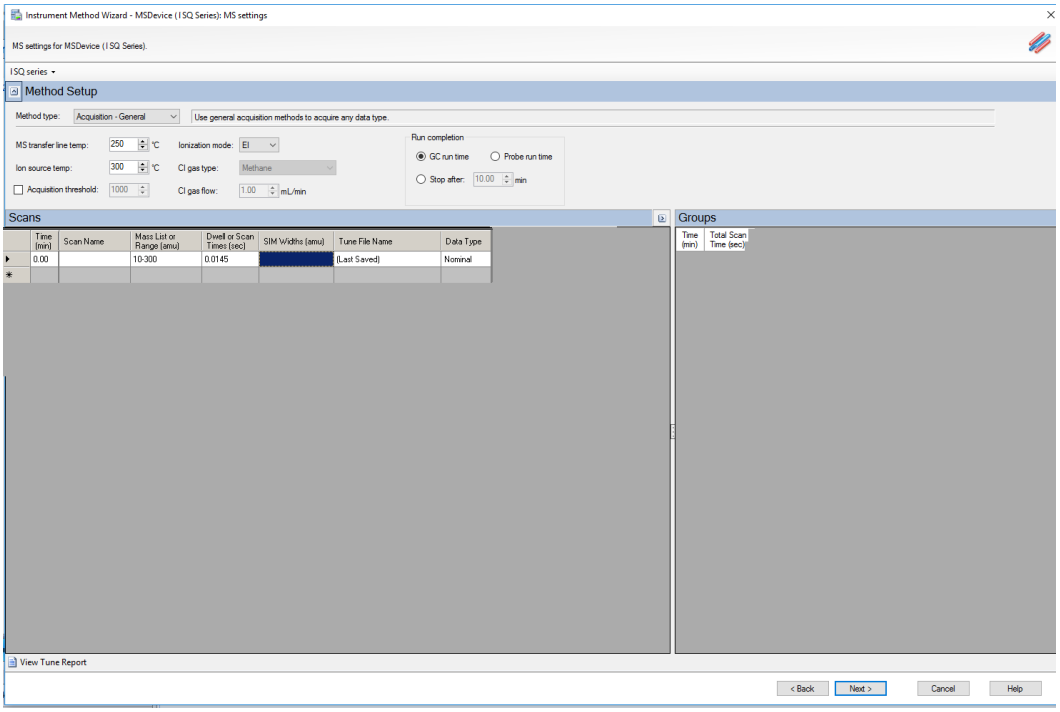
e. The **Scan Name** column contains a description of the scan. The name may be used as a label to indicate the compound used with the scan.

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**.

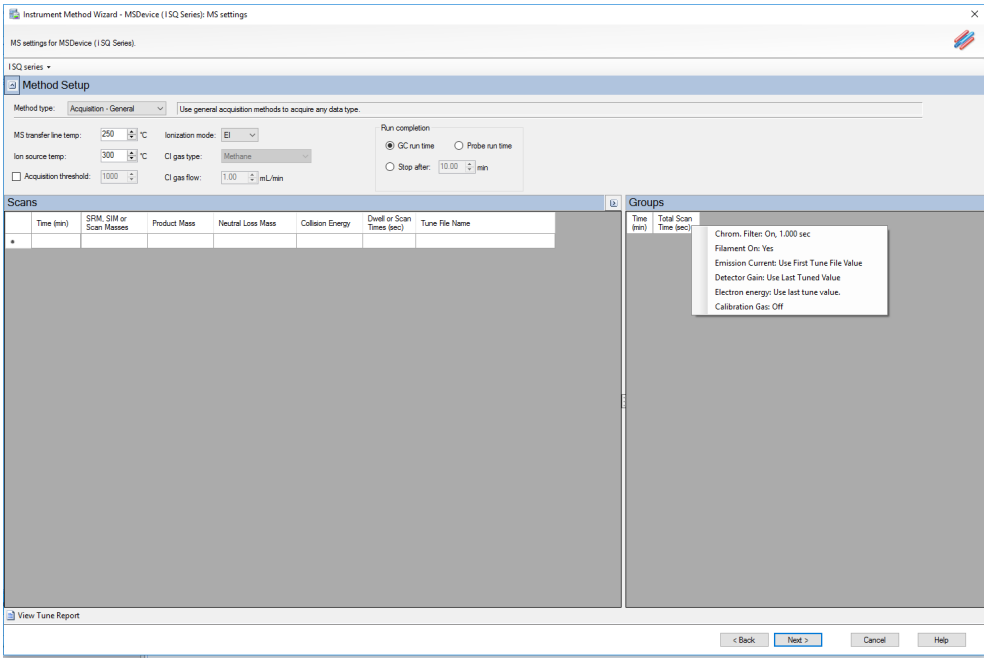
h. The **Data Type** column determines whether you want to collect **Profile**, **Centroided**, or **Nominal** mass spectra. Centroided mass spectra is most common because it is used by most of the libraries and provides the smallest data files. Profiled mass spectra provides detailed mass spectral peaks, 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 101. 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 7610 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 7610 AutoTune or set and scanned in manual tune. If you do not need to use the gain set in ISQ 7610 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.

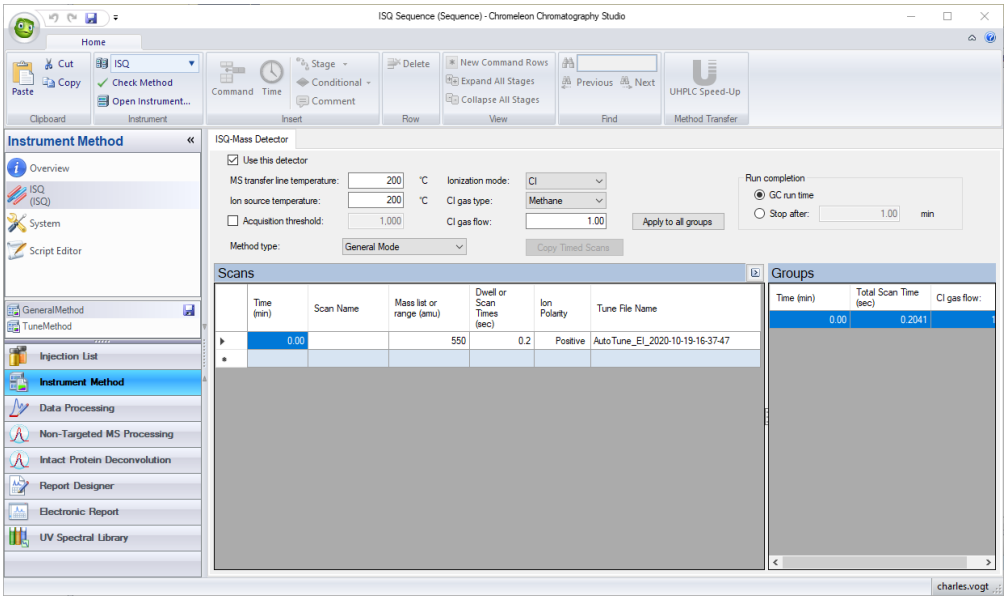
i. Use the **CI Gas Type** column to set the type of gas attached to one or both CI gas ports (A or B). Be sure that the gas you have assigned to a port is actually attached to that port, as each CI gas has a specific calibration of flow vs. gas viscosity.

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 102. 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 7610 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 7610 Mass Spectrometer," The software defaults to the most recent **AutoTune_NCI** or **AutoTune_PCI** tune file you created.

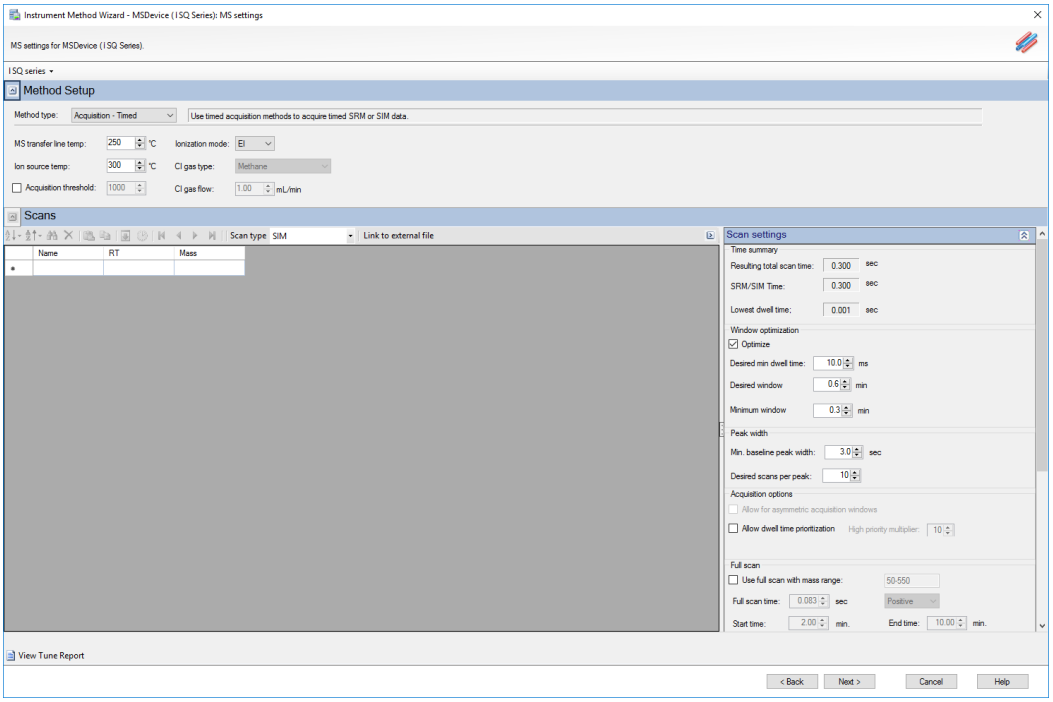
b. In the **Detector Gain** area, set the detector gain. Select the **Use Last Tuned Detector Gain** option to indicate that you want to use the detector gain set in ISQ 7610 AutoTune. If you do not need to use the gain set in ISQ 7610 AutoTune, then you can set the gain manually. Higher gains give larger 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 ± 0.5 µA.

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

Figure 103. Acquisition-Timed Scan Settings



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 Acquisition**s 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.

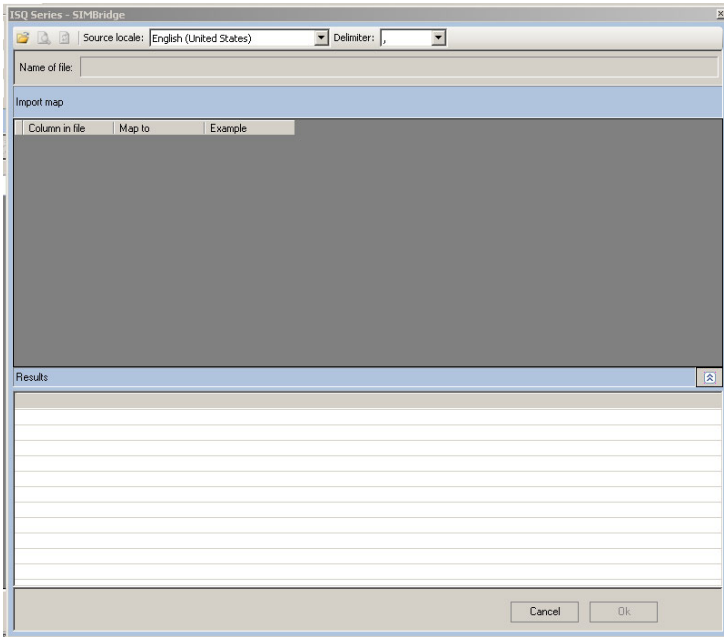
Select the **Allow Dwell Time Prioritization** check box to increase the dwell times for selected scans. The choices for each scan are Normal or High. Giving a scan high priority increases its dwell time by the value you set in the High Priority Multiplier box.

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 7610 Method Editor, clear the **Link to External File** check box.

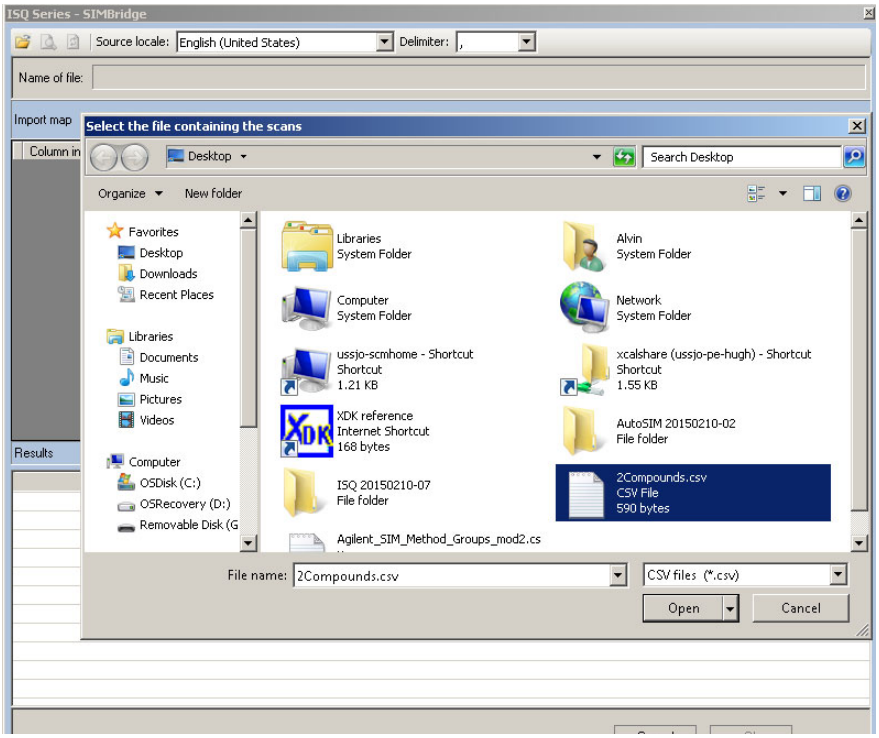
g. After clicking **Link to External File**, the **SIMBridge** dialog box opens. Choose the language of your method file from the **Source Locale** drop-down menu.

Figure 104. Setting the Source Language of Method Files using SIMBridge



h. Browse to your file.

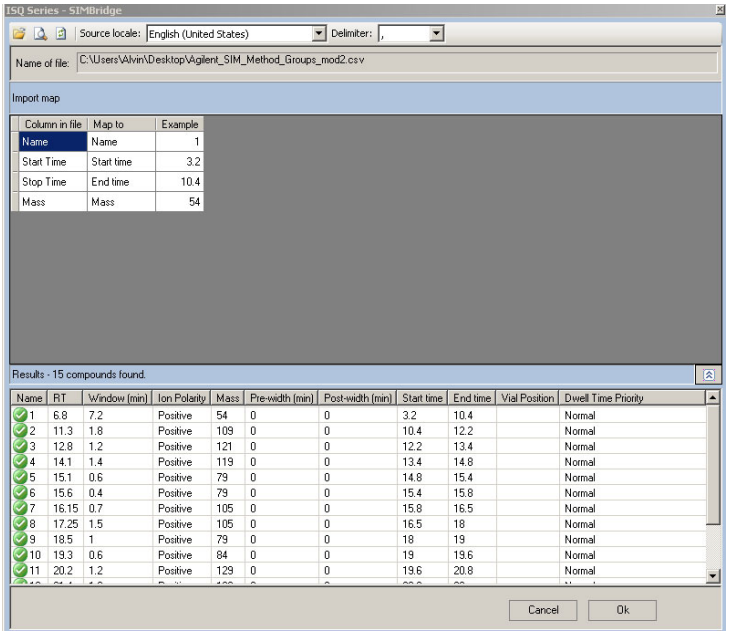
Figure 105. Linking to an External File using SIM Bridge



i. Click **Open** to open the method in SIMBridge.

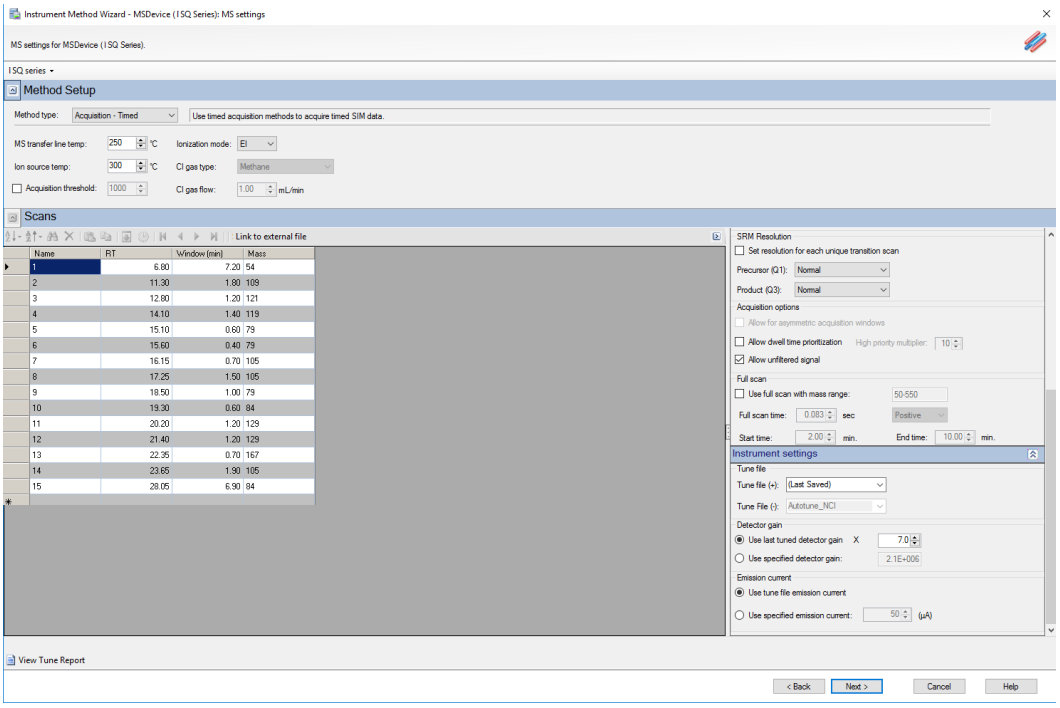
j. If necessary, change the method headings in your original file to match those in the method editor. A green check mark appears when your method is validated.

Figure 106. Changing Method Headings in SIMBridge



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

Figure 107. Viewing a Linked File in the Method Editor



l. 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.

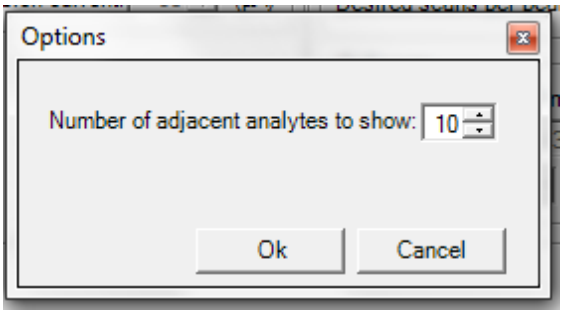
o. In the **Mass** column, enter the mass of the ion you wish to monitor.

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 7610** main menu. See Figure 78. View the number of analytes you set in the **Show Analysis** view.

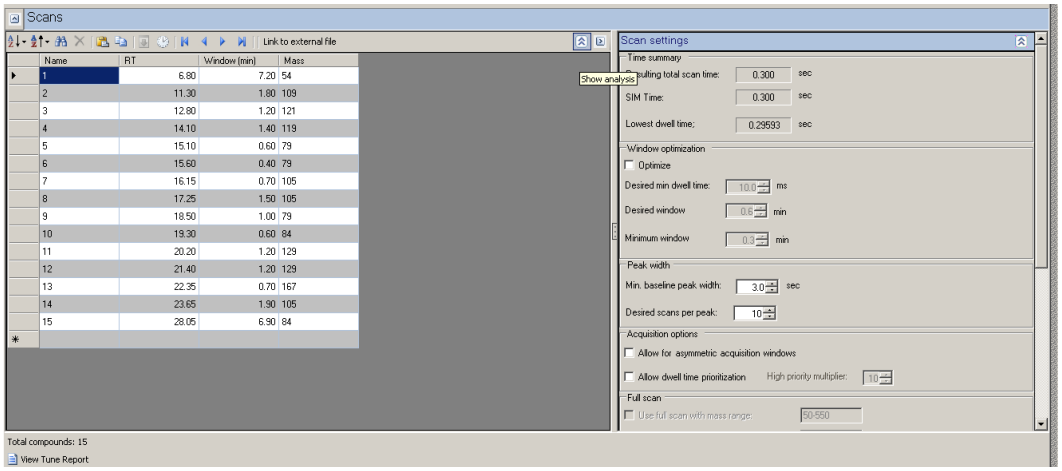
Adjustments in this column are for display purposes only and will not affect your acquisition.

Figure 108. Options Dialog Box



r. Click **Show Analysis** (see Figure 79) to validate your method. A chart appears with all your analytes by name and in order of their start times.

Figure 109. Accessing the Show Analysis Feature



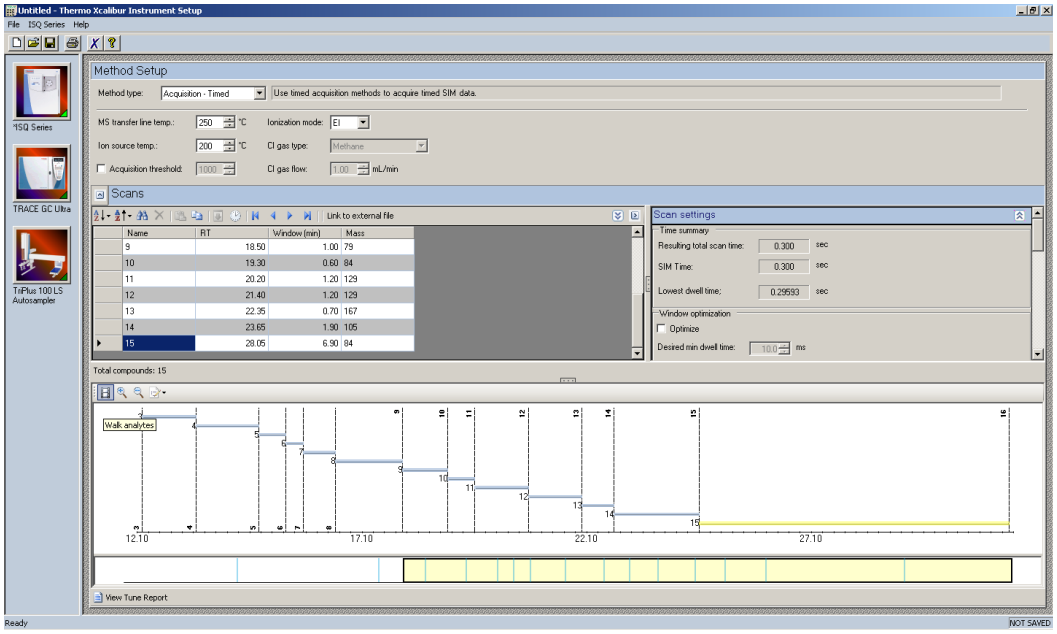
s. Use the scrolling window at the bottom of the screen to view all your analytes' expected retention times. Resize the window to view more analytes by dragging one side of the window out. As the scrolling window is decreased in size, fewer analytes are shown in the analytes chart. Increasing the size 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.

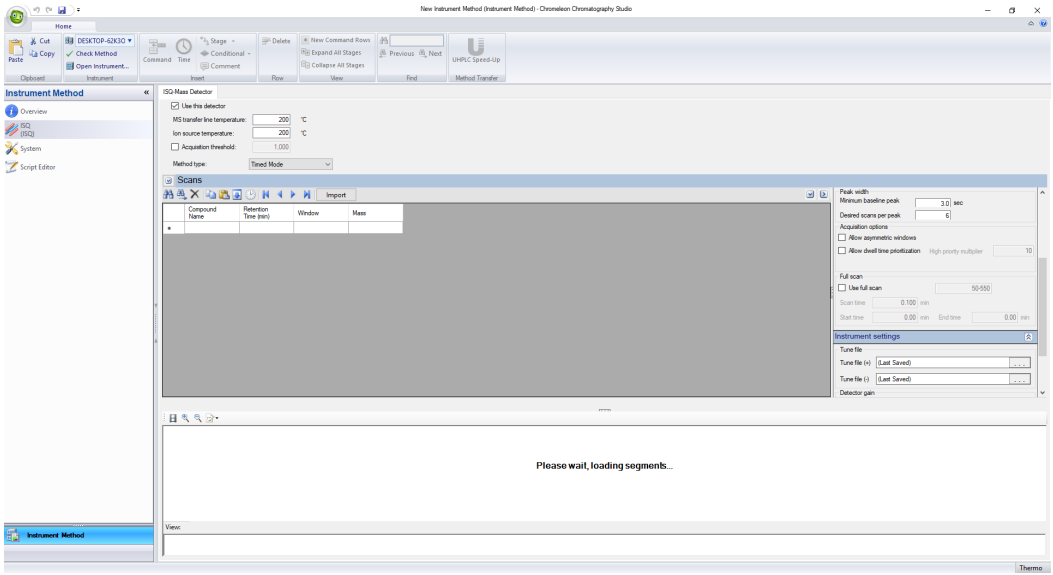
Figure 110. Validating a Method



37. To import another MS method, choose **Import**. See Figure 81.

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

Figure 111. Importing Methods



38. Choose **Import | 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.

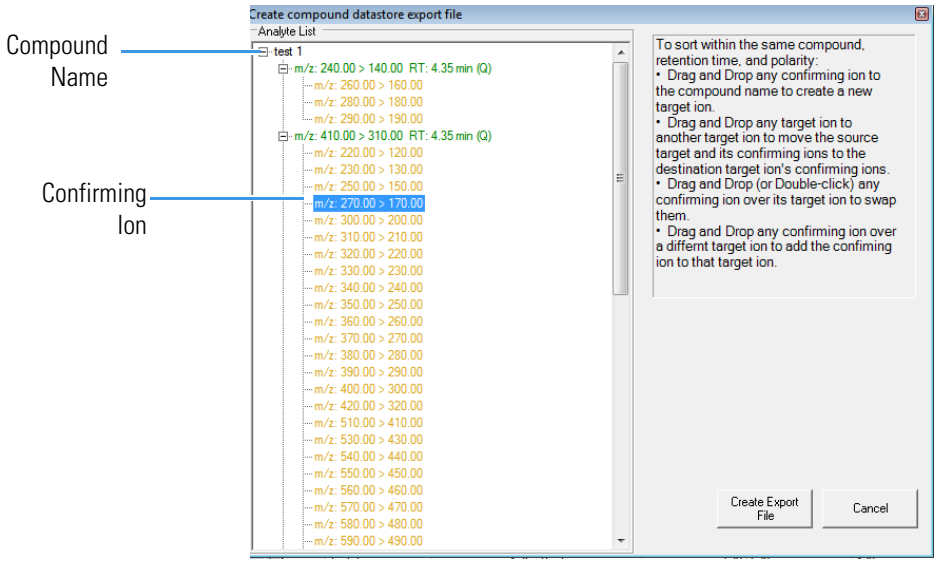
Tip Export a timed scan list to see an example of a valid format.

39. Choose **Import | 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.

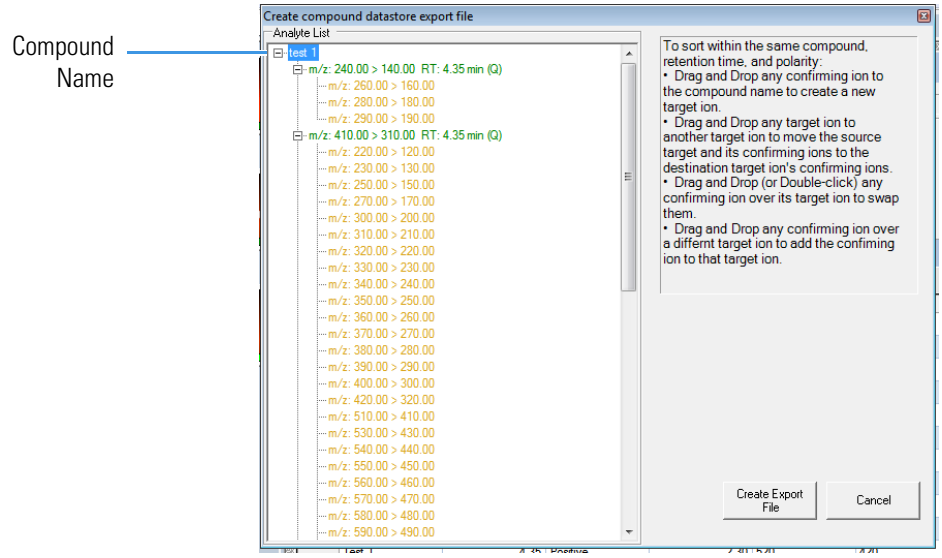
40. Choose **Import| Export Timed Scans** to export your method as a .csv file. If you prefer editing your methods in spreadsheet applications, you may want to use this option.

41. Choose **Import | Create Compound Data Store Export File** to prepare your file for the compound data store in the TraceFinder application.

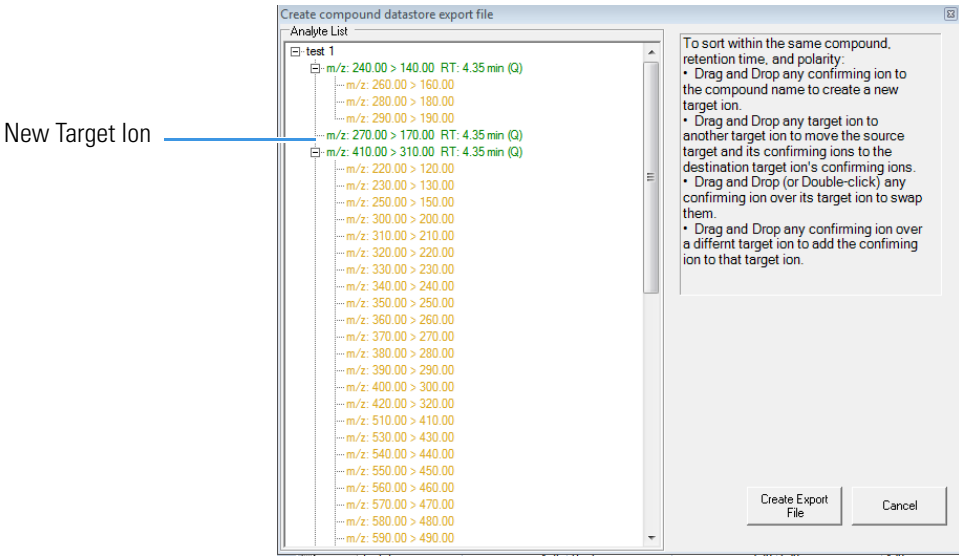
    a. To create a new target ion from the list of confirming ions with identical retention times for a compound, select the confirming ion of interest.



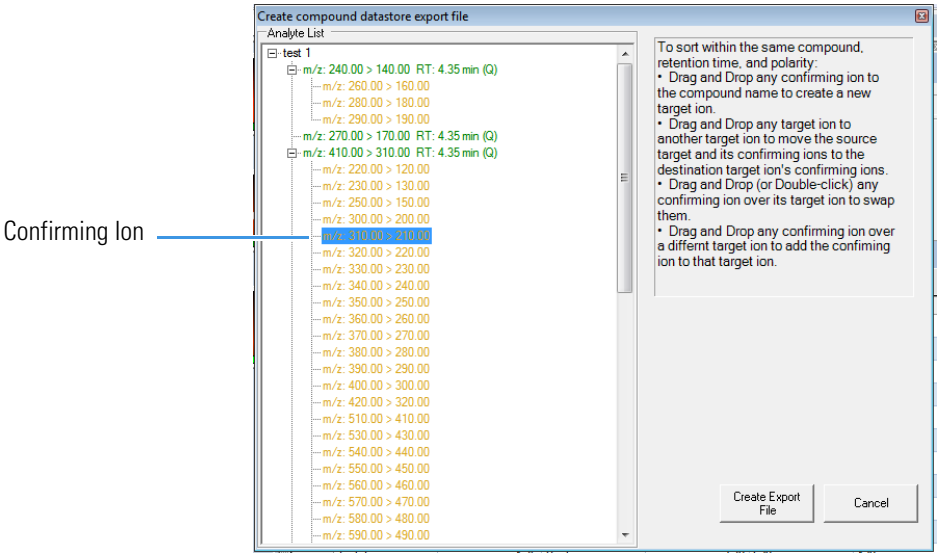
b. Drag the confirming ion to the compound name at the top of the list.



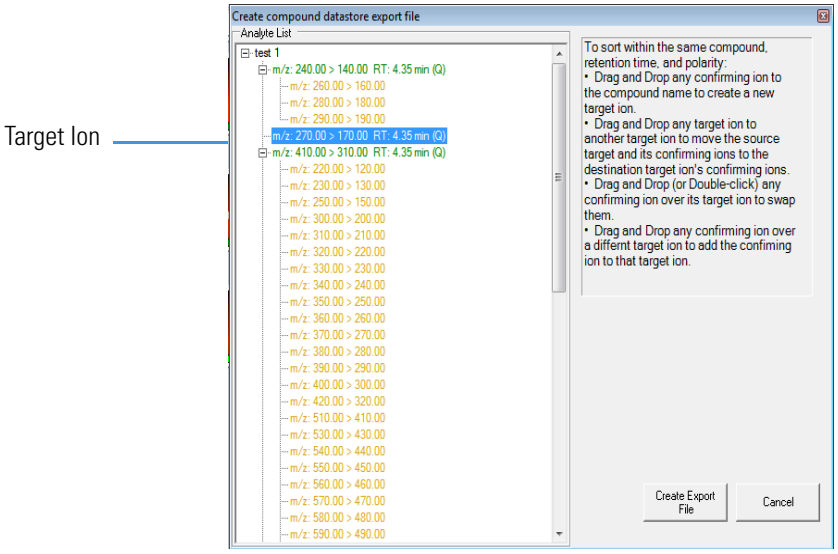
c. The confirming ion now appears in the list as a target ion.



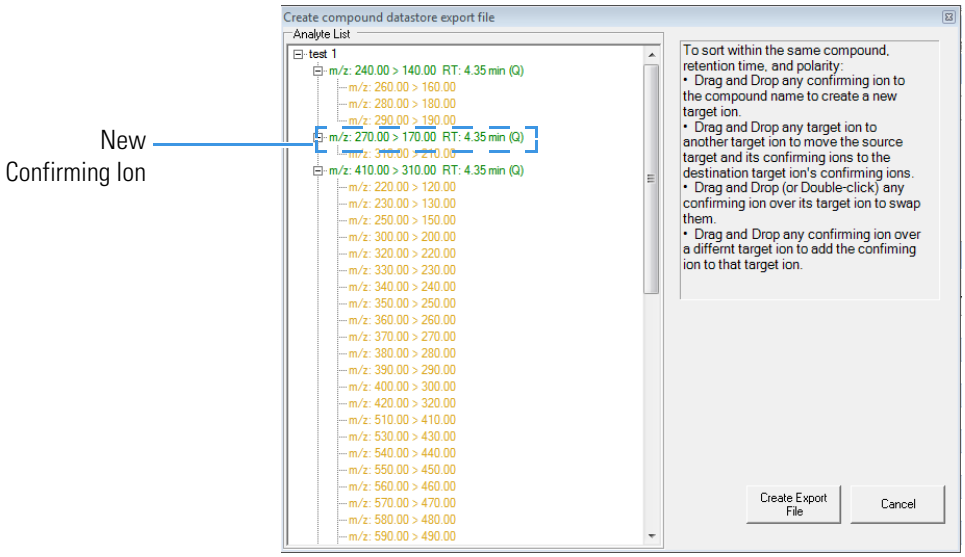
d. To add a new confirming ion to a target ion in the list, select the confirming ion of interest.



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



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

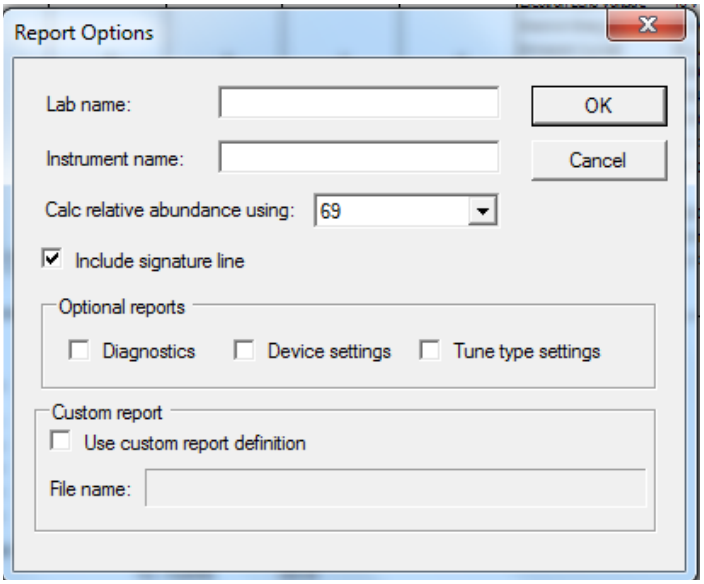


g. Click **Create Export File** when you are through creating your ion list to export the list to the TraceFinder software compound database.

42. Choose **Import | Create Segment List From Timed Scan List** to import a general acquisition method.

43. Choose **Import | 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 82) and add identifying information to the tune report.

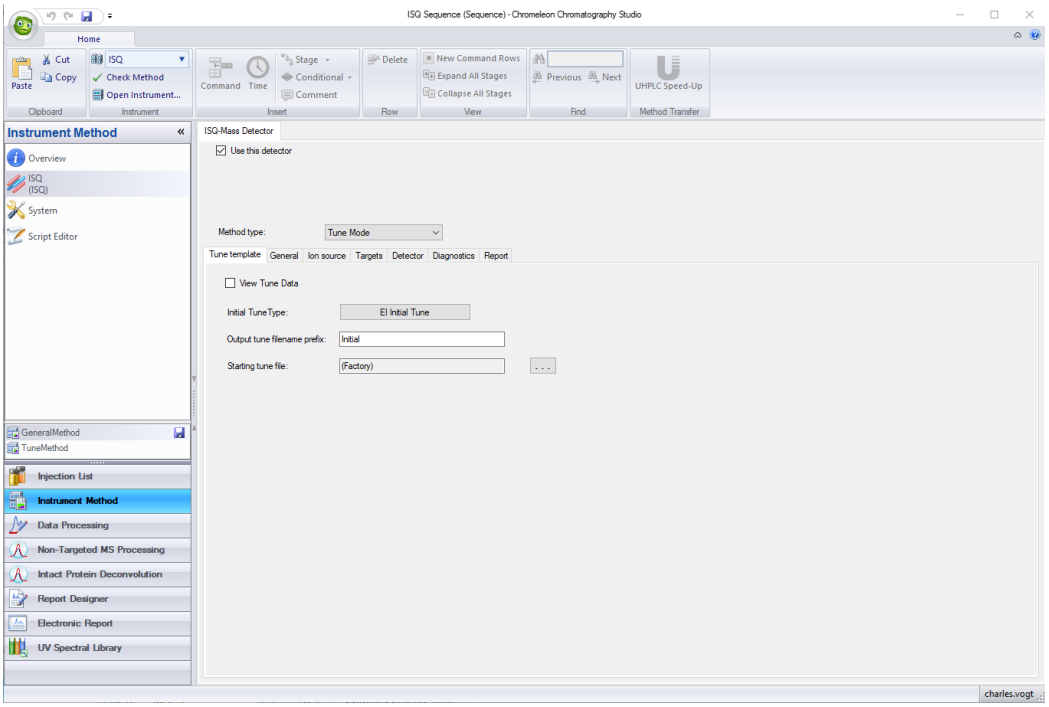
Figure 112. Configuring the Tune Report Options



44. The **Tune** method allows you to run a tune from the method editor.

    a. From the **Method Type** drop-down box, select **Tune Mode.** See Figure 113.

    Figure 113. Selecting a Tune Mode Method



    b. Under **Tune Template** set the following parameters:

        i. Check **View Tune Data** to view the tune data.

ii. **Initial Tune Type**: Select a Tune Type to use as a template. This will fill in all of the options the same as one of the built-in tune types. Options can then be manually changed as necessary to create the desired Tune Method.

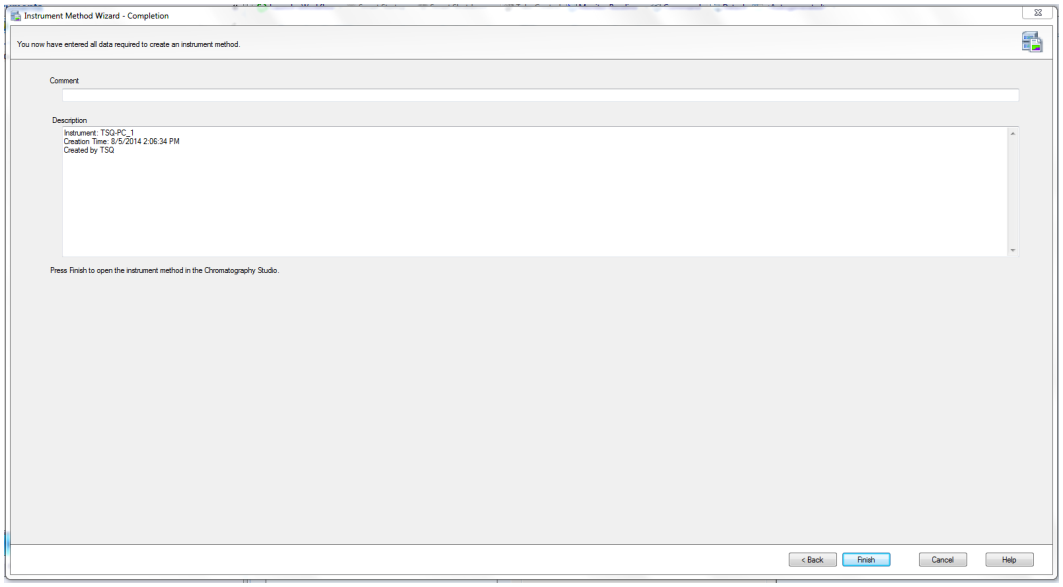
iii. **Output Tune Filename Prefix**: Autotune is the default.

iv. **Starting Tune File**: The last saved tune is the default.

c. Set up the remaining tune parameters in Tune Method as described in "To modify an automatic tune in Chromeleon software" on page 202.

45. Click **Next**. The **Instrument Method Wizard – Completion** page opens. See Figure 114.

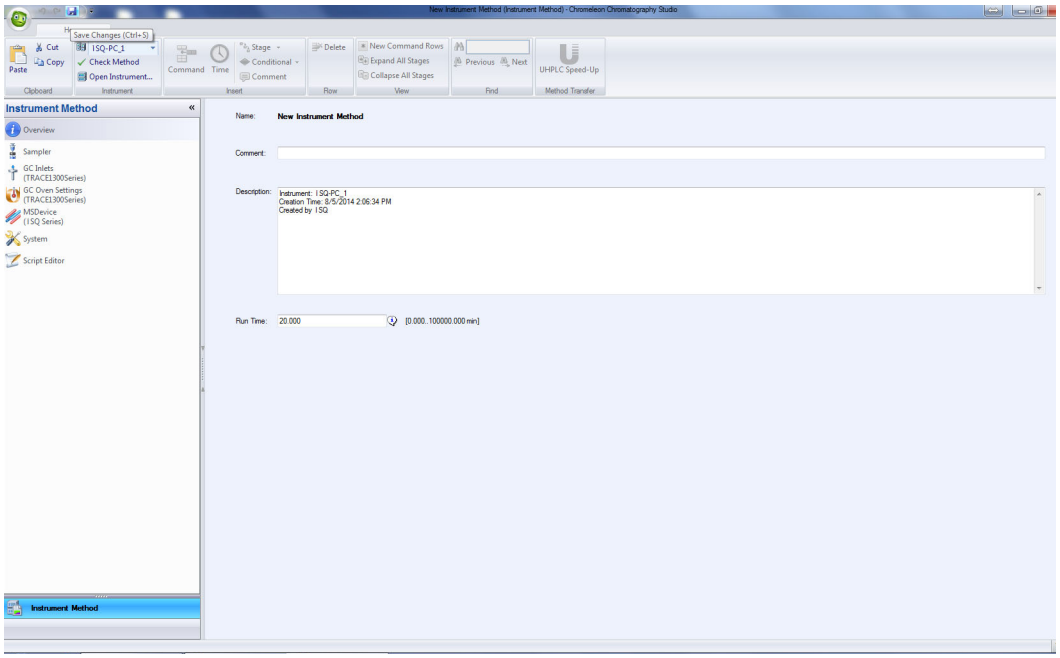
Figure 114. Instrument Method Wizard – Completion



46. Enter any comments or notes for your method and click **Finish**.

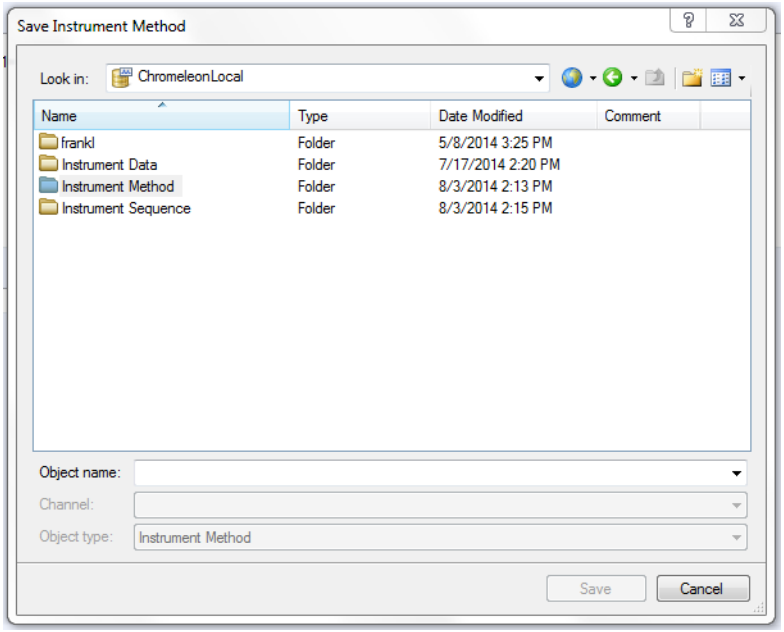
47. The New Instrument Method (Instrument Method) Chromeleon Chromatography Studio window opens. Click the **Save** icon to save your method. See Figure 81.

Figure 115. Saving Methods



48. The **Save Instrument Method** dialog box opens. Save your method in **Chromeleon Local > Instrument Method**. See Figure 116.

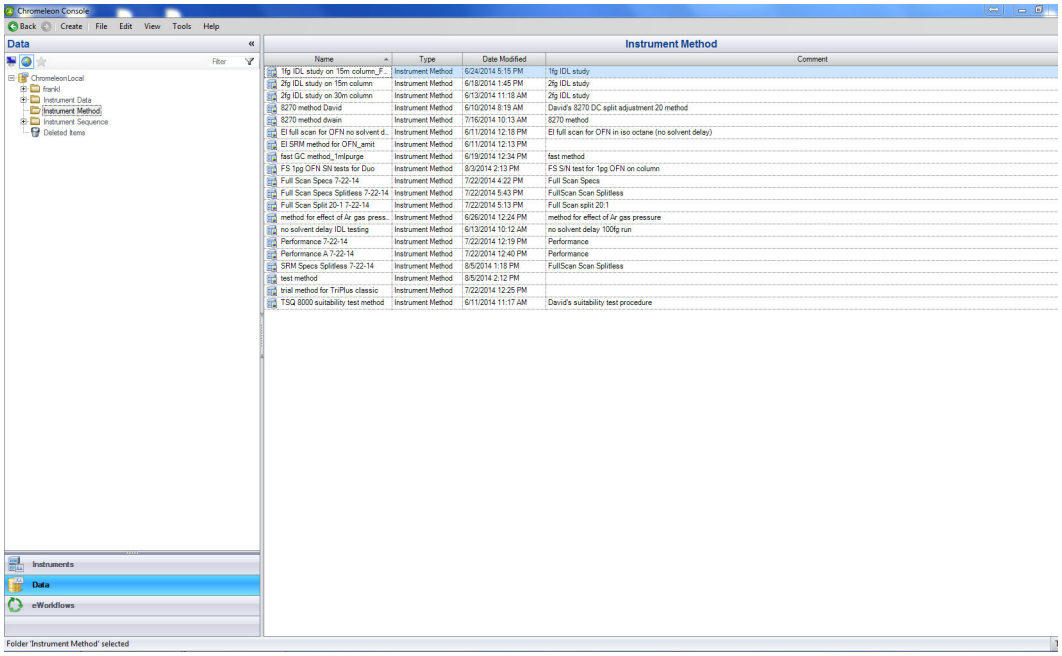
Figure 116. Finding the Instrument Method Folder



49. Enter your method in the **Object Name** box and click **Save**. Your method is now saved.

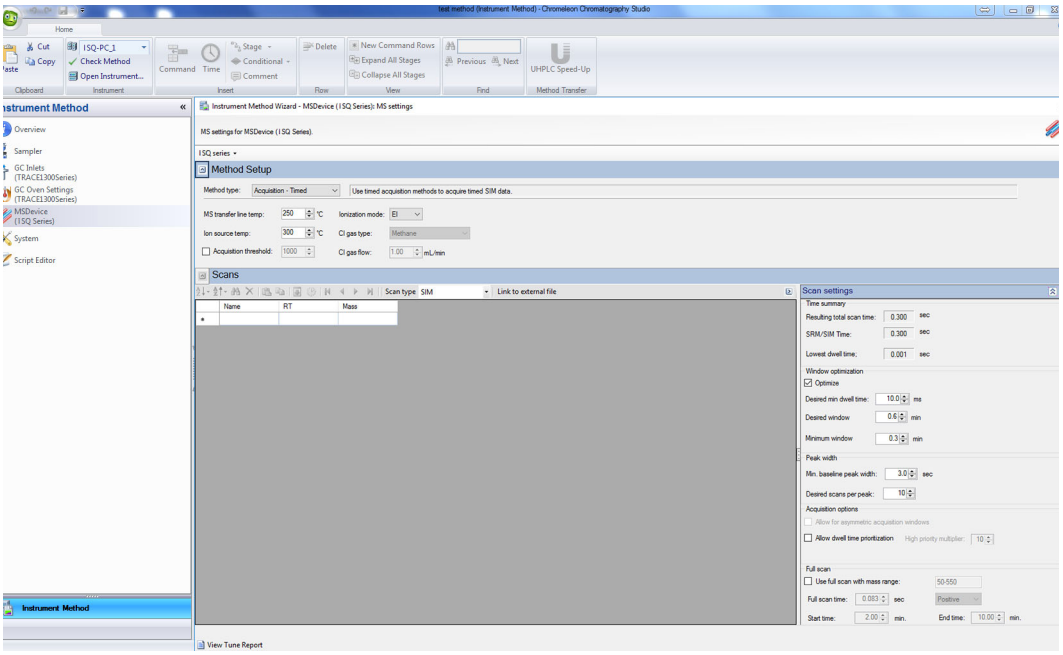
50. You can view all instrument methods on your system by opening the Chromeleon Console and selecting **Data** from the left menu. See Figure 117.

Figure 117. Viewing all Instrument Methods



51. To edit a method, select it from the list. The method opens in the Chromatography Studio. See Figure 118.

Figure 118. Editing a Method



52. 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 7610 method editor and accessing them for routine use.

Note Set up your GC and autosampler methods through the ISQ 7610 method editor before developing your AutoSIM method.

Contents

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 7610 system to run a full-scan analysis on the compounds.

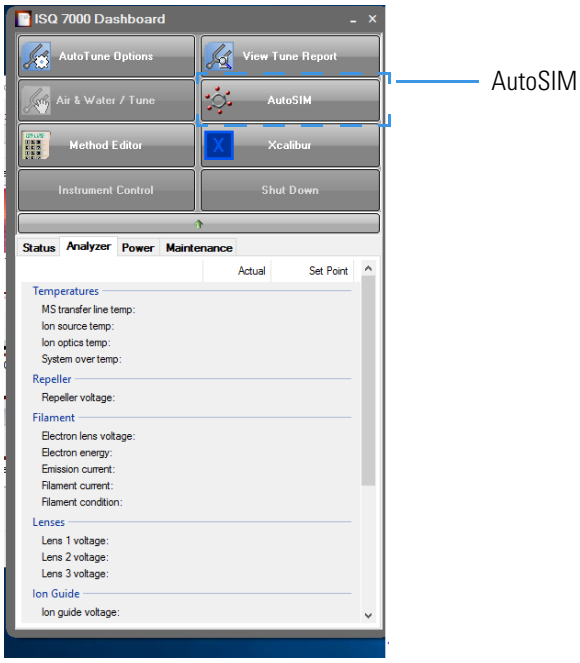
After the full-scan analysis is complete, AutoSIM presents you with the resulting chromatographic peaks and full-scan spectra, and then provides optional setting for sorting the results for your SIM ions.

Note You must have mid-range concentration standards (500 pg/µL–10 ng/µL) before setting up your AutoSIM method.

❖ To determine your SIM ions in AutoSIM

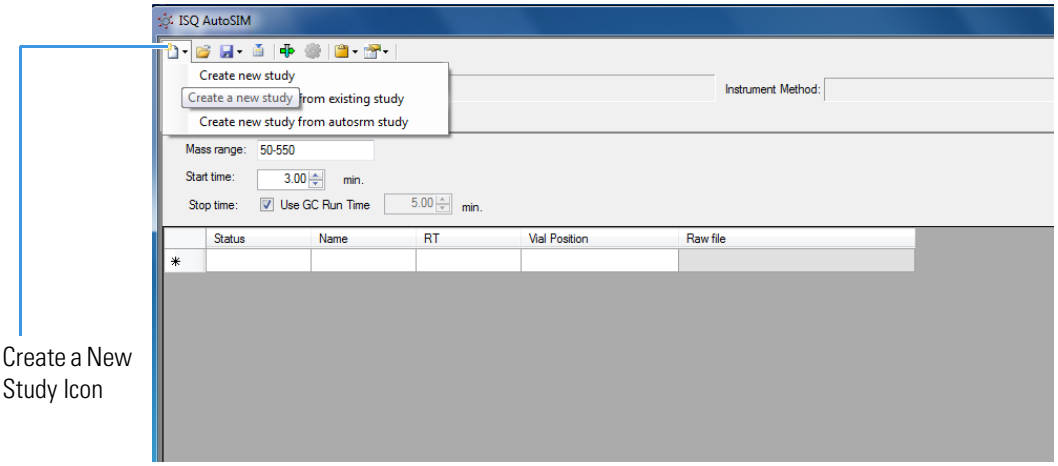
1. Click the **AutoSIM button** on the ISQ 7610 Dashboard to open the AutoSIM utility. See Figure 119.

Figure 119. Accessing AutoSIM on the ISQ Dashboard



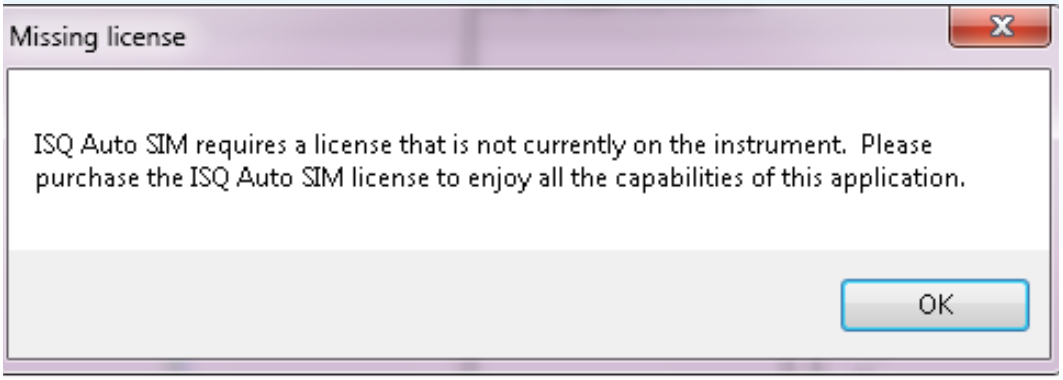
2. Click the Create a New Study icon on the left to create a new study.

Figure 120. New AutoSIM Study



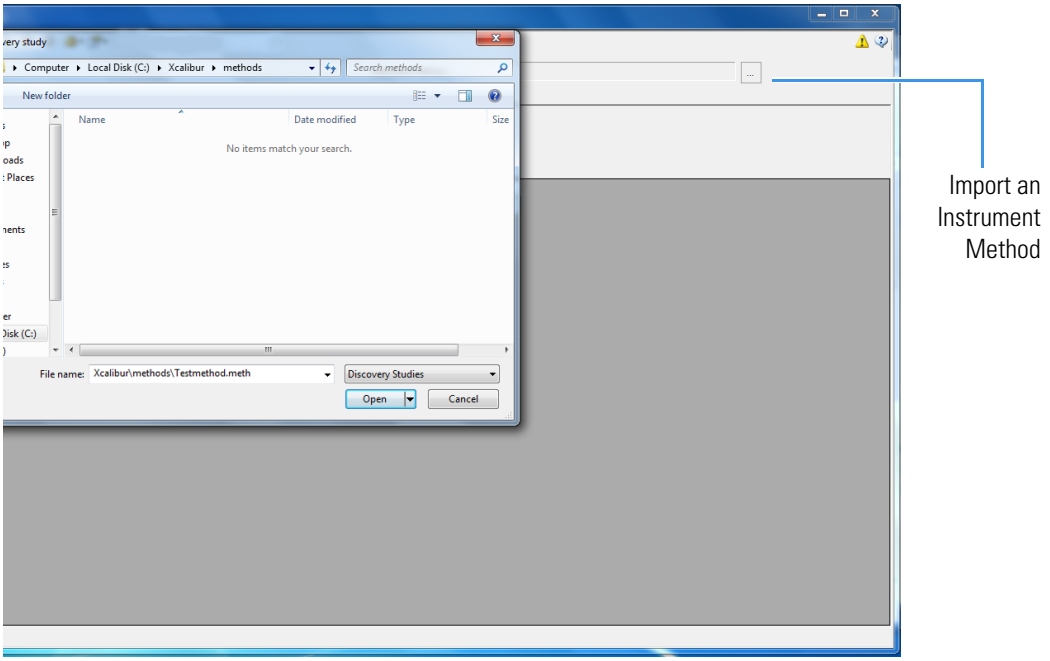
3. A new study window opens.

Note If at any time you see the error message below, go to "Upgrading the Software" on page 255 and follow the instructions for getting your software license.



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 121.

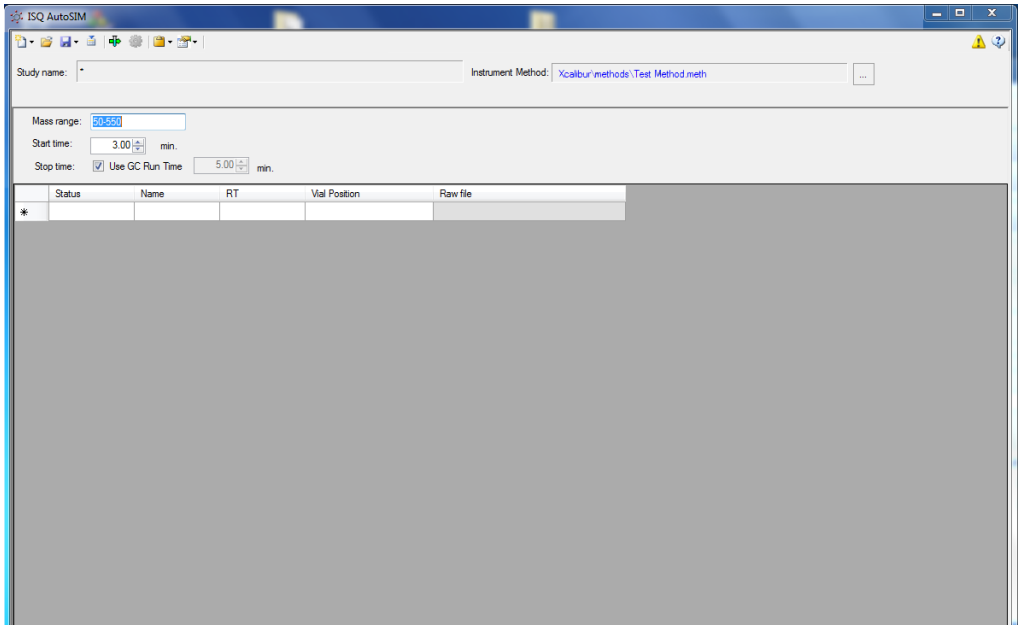
Figure 121. Retrieving an Instrument Method



5. Select an instrument method file and click Open.
6. You may set the Mass Range, Start Time, and Stop Time. See Figure 122.

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

Figure 122. Adjusting the Settings

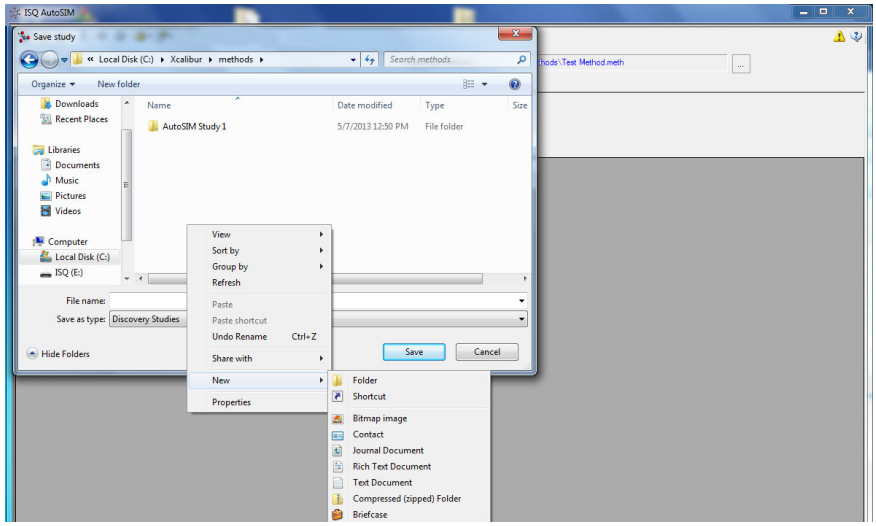


7. Enter the compound name, approximate retention time, and vial number for each compound you wish to optimize. If you already have a method for processing full scan data you can choose to import compounds from an external file. Their names and retention times will fill the compound list and their primary 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 123.

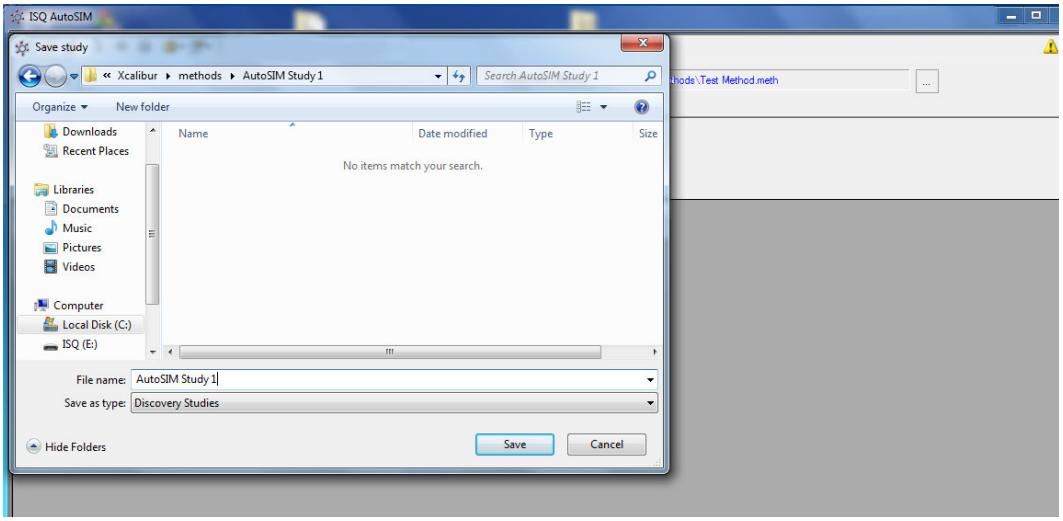
Figure 123. Creating an AutoSIM Study Folder



9. Open the folder.
10. Give your study a file name.
11. Save your study in the Study folder. See Figure 124.

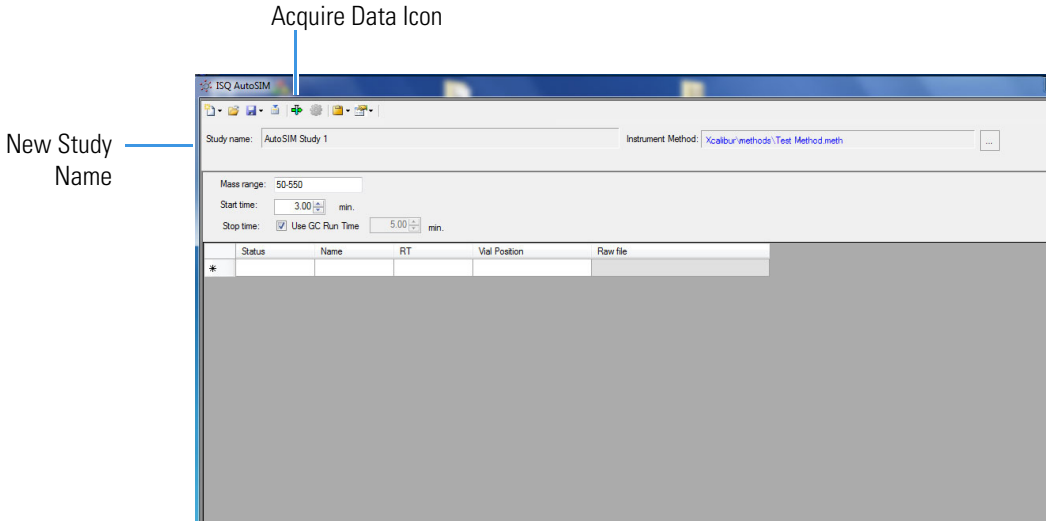
Note All files, including raw data files, that AutoSIM generates will be saved into the same folder that you save the study file. To simplify your workflow, create a folder for your study.

Figure 124. Saving an AutoSIM Study



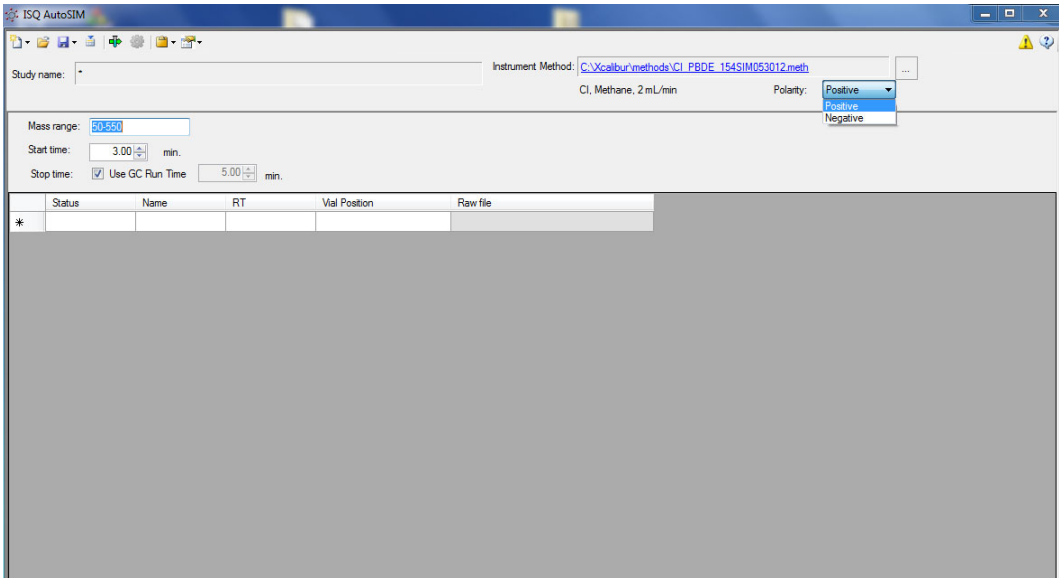
12. The Windows Explorer window closes.
13. The AutoSIM Study Name is the name you assigned. See Figure 125.

Figure 125. Acquiring Acquisition Data



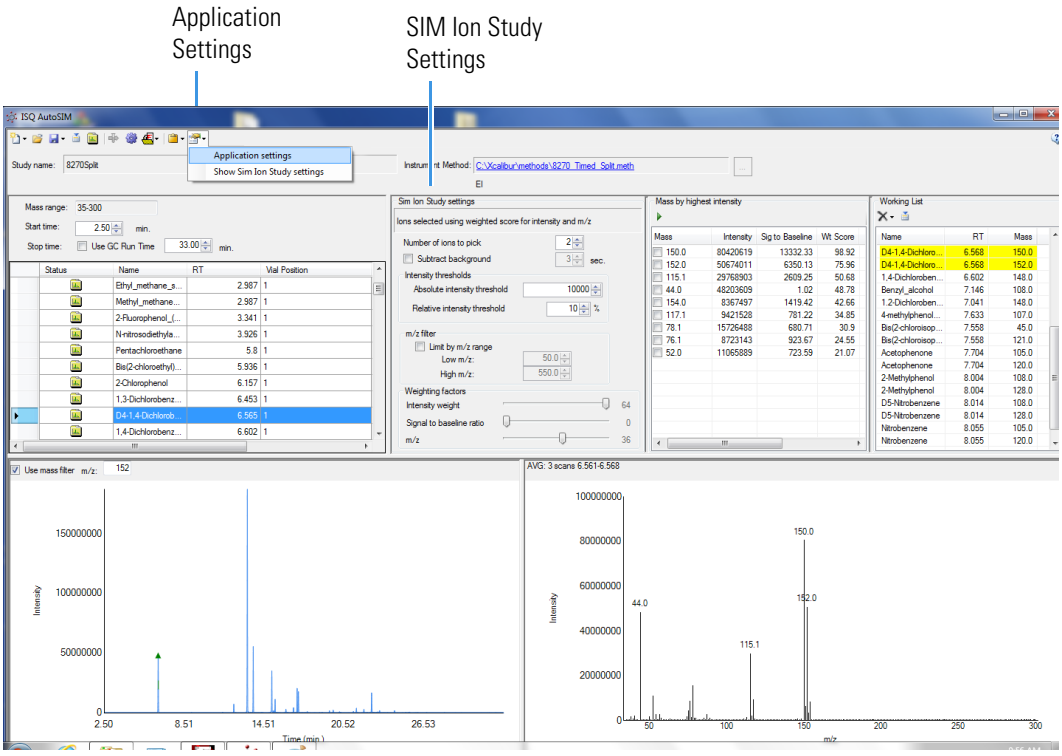
14. (CI Only) If you are running a chemical ionization (CI) study, select **Positive** or **Negative** from the **Ion Polarity** pull-down menu. See Figure 126.

Figure 126. Setting Ion Polarity in a CI Method



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 127.

Figure 127. Application Settings and SIM Ion Study Settings



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.

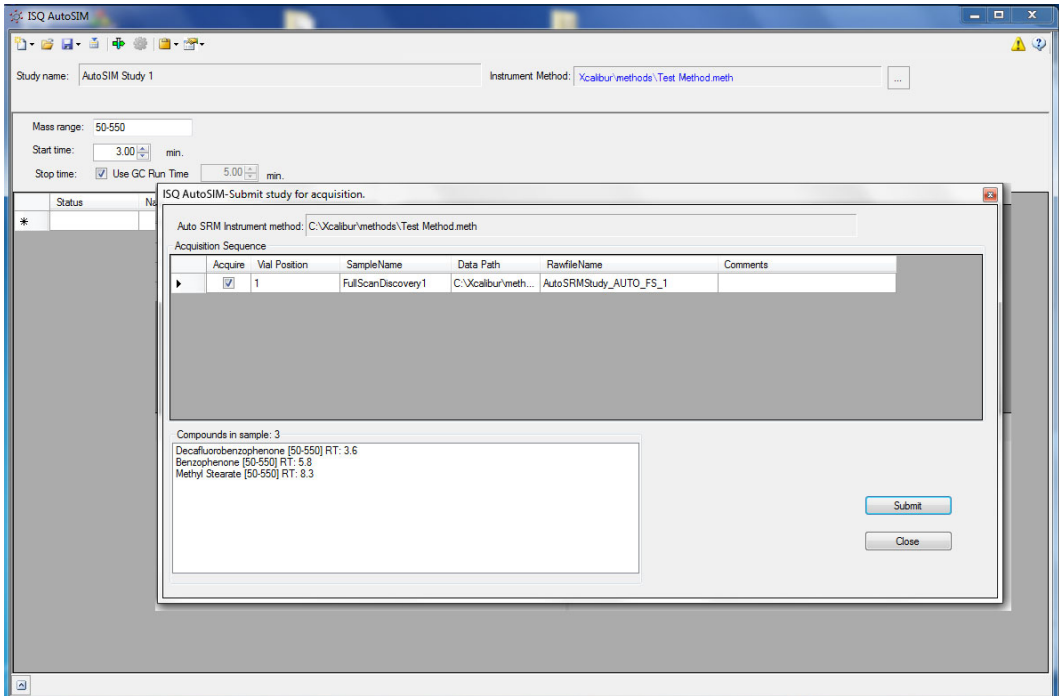
e. **Weighting Factors**: Use the sliding bars and check boxes to set the values you want to give each SIM ion study setting.

17. Click the **Acquire Data** icon to run your samples. See Figure 125.

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 128.

Figure 128. Submitting a Study for Acquisition



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

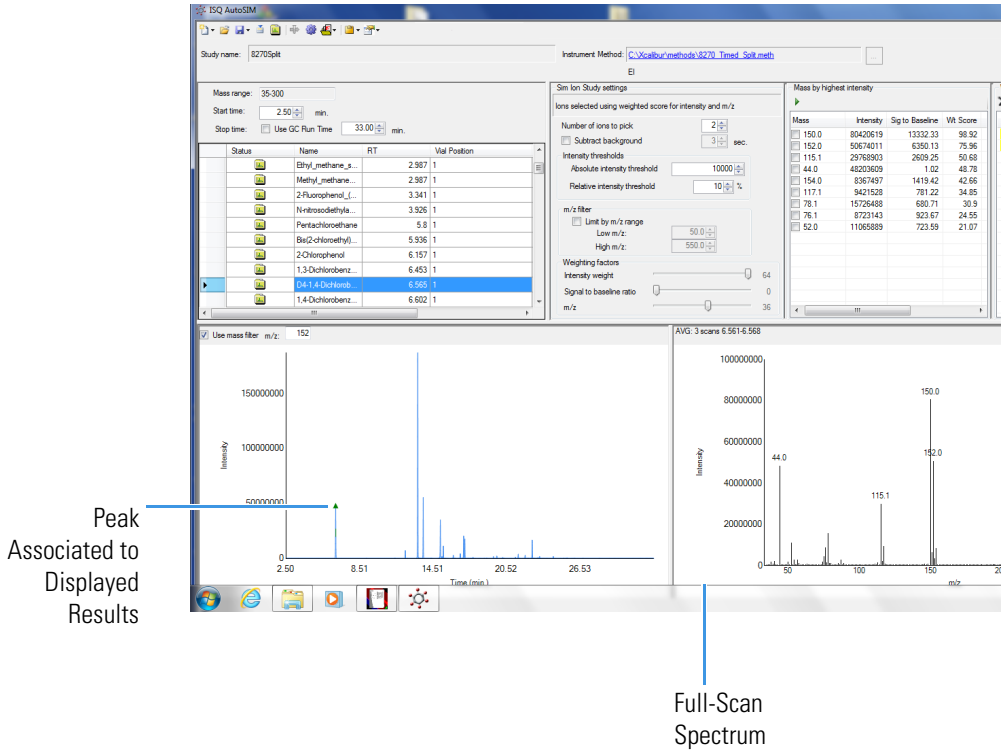
    Once the samples have finished running, the software analyzes the data.

20. The results appear in the AutoSIM window. See Figure 129.

    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.

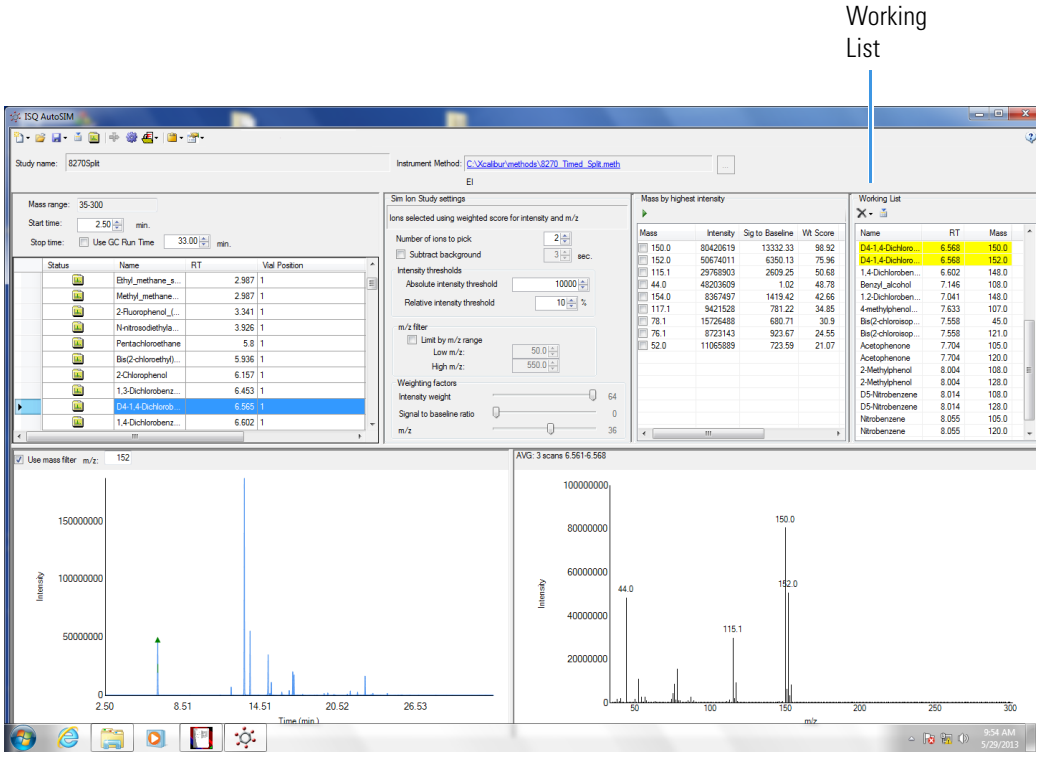
> **Note** Background subtraction updates the ions in the Mass by Highest Intensity pane.

Figure 129. 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 130.

Figure 130. Selecting SIM Ions

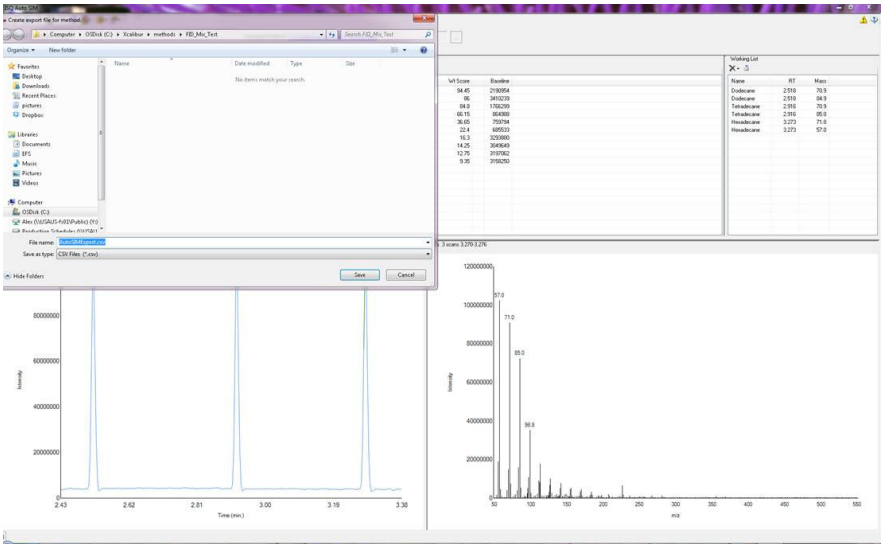


23. Repeat this process for all of your compounds.

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 131.

Figure 131. Exporting your SIM Ion Study

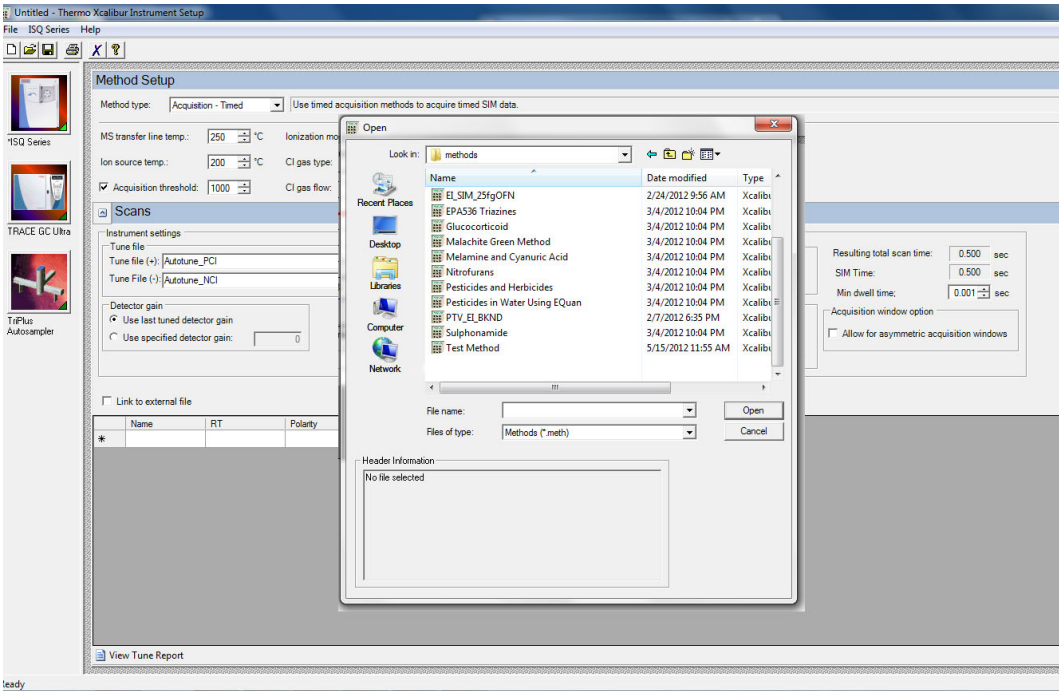


Importing Transitions to the Method Editor

❖ **To import the list of transitions you created in AutoSIM**

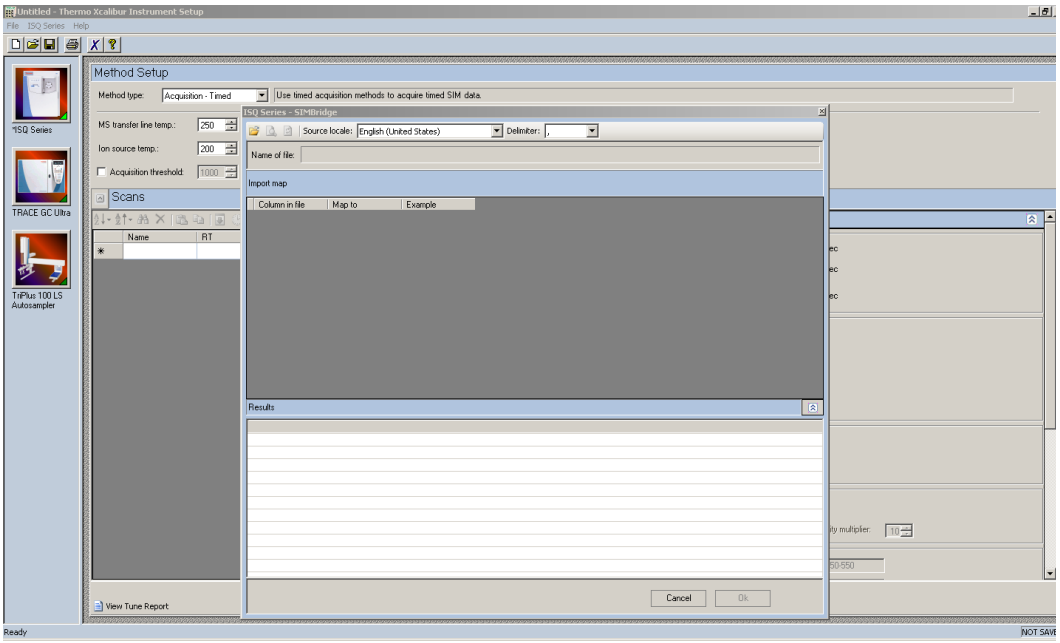
1. Open the method editor on the ISQ Dashboard.
2. Open your method in Xcalibur. See Figure 132.

Figure 132. Opening Instrument Method in Xcalibur



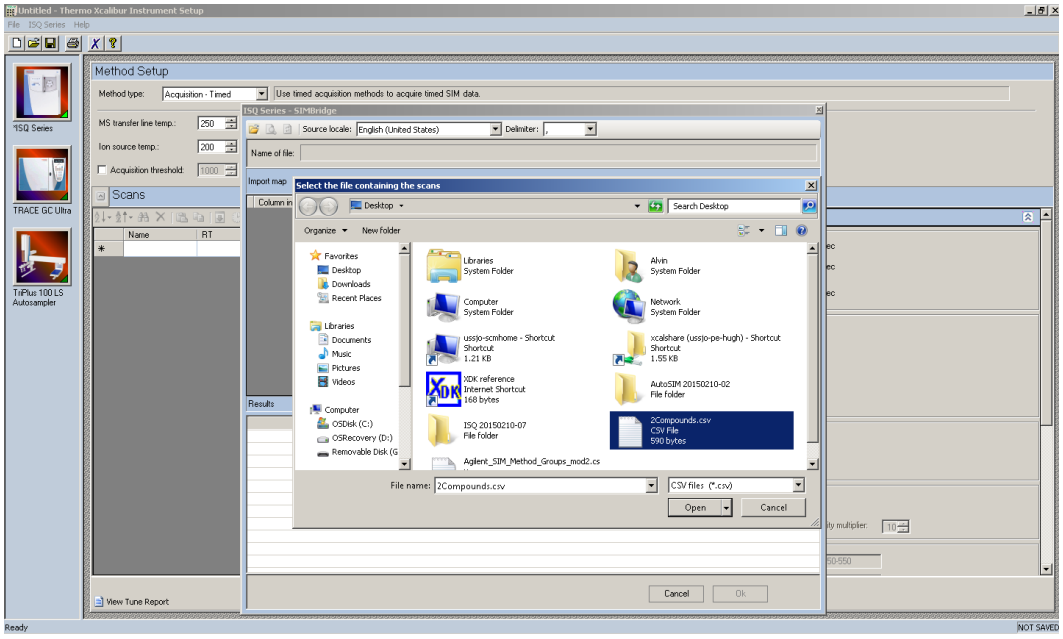
3. If you have the desired method open in AutoSIM, click the **Link to External File** in the ISQ 7610 method editor to open your method in the ISQ 7610 method editor
   a. After clicking **Link to External File**, the **SIMBridge** dialog box opens. Choose the language of your method file from the **Source Locale** drop-down menu.

Figure 133. Setting the Source Language of Method Files using SIMBridge



b. Browse to your file.

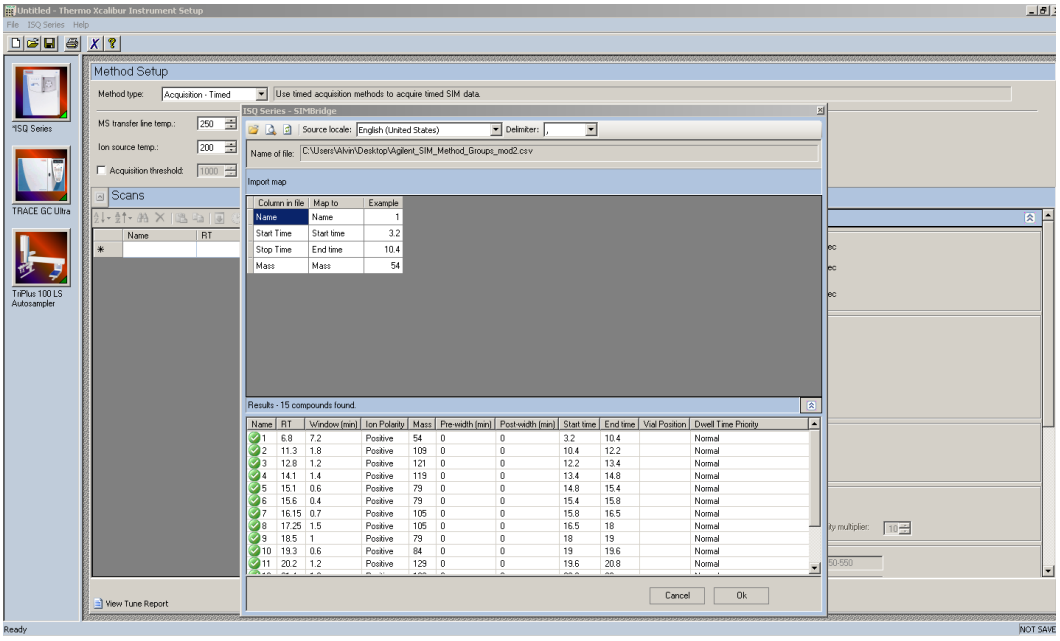
Figure 134. Linking to an External File using SIM Bridge



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.

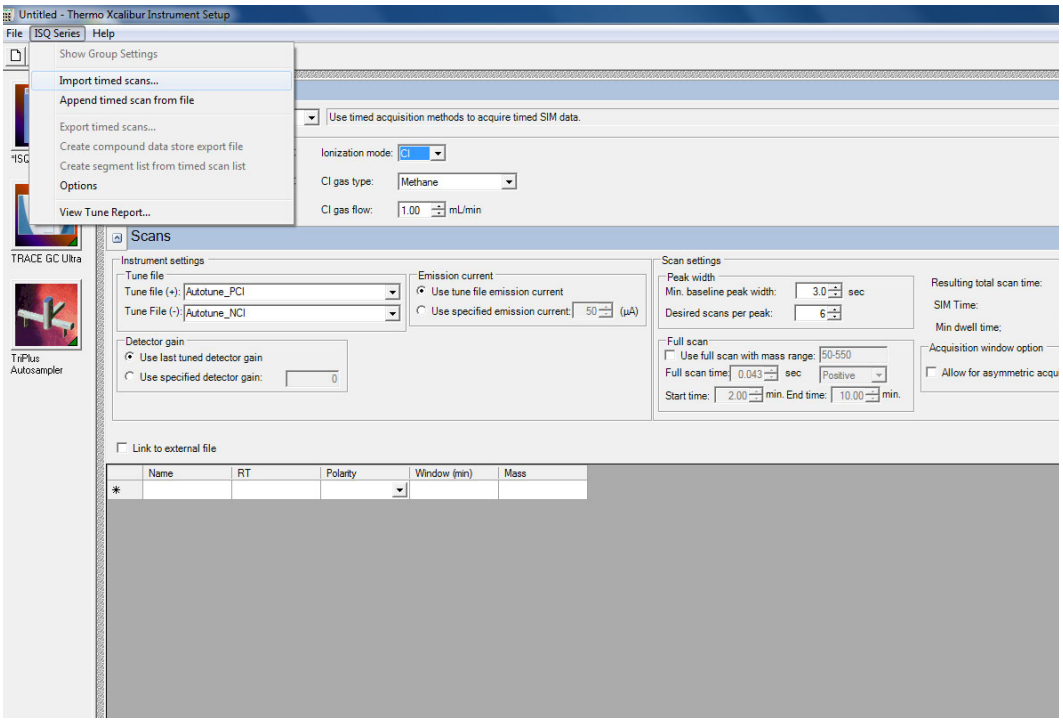
Figure 135. Changing Method Headings in SIMBridge



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

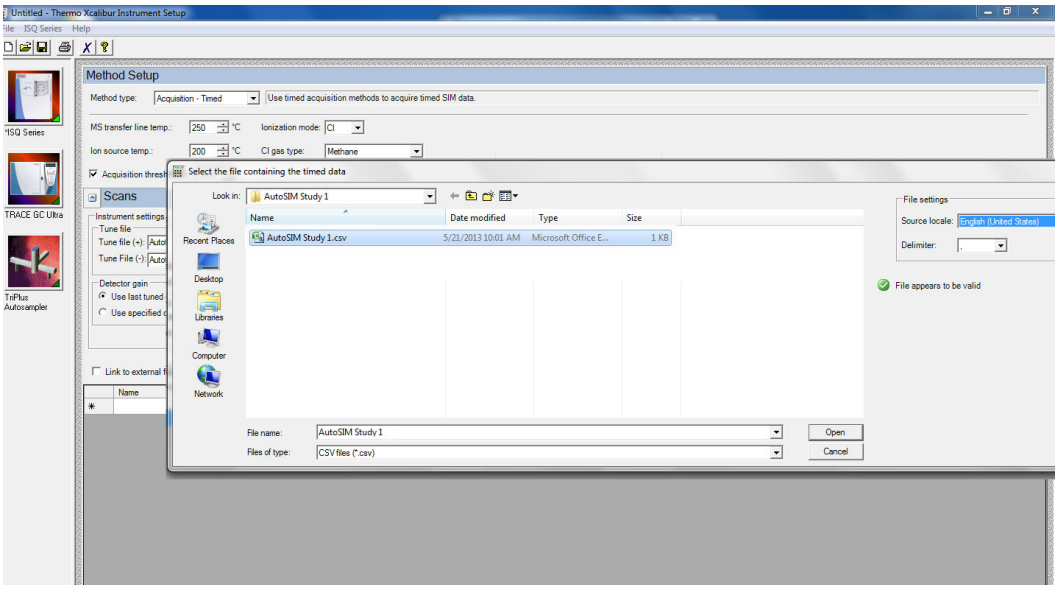
4. Click the ISQ 7610 icon in the method editor side pane.
5. Select Acquisition-Timed from the Method Type drop-down menu.
6. From the top menu, select **ISQ 7610 | Import Timed Scans**. See Figure 136.

Figure 136. Selecting Acquisition-Timed Method



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 137.

Figure 137. 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 63 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 7610 system to run a full-scan analysis on the compounds.

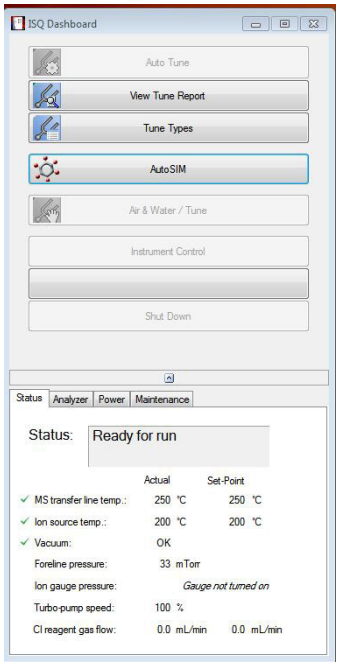
After the full-scan analysis is complete, AutoSIM presents you with the resulting chromatographic peaks and full-scan spectra, and then provides optional setting for sorting the results for your SIM ions.

Note You must have mid-range concentration standards (500 pg/µL–10 ng/µL) before setting up your AutoSIM method.

❖ To determine your SIM ions in AutoSIM

1. Click the AutoSIM button on the ISQ Dashboard to open the AutoSIM utility. See Figure 119.

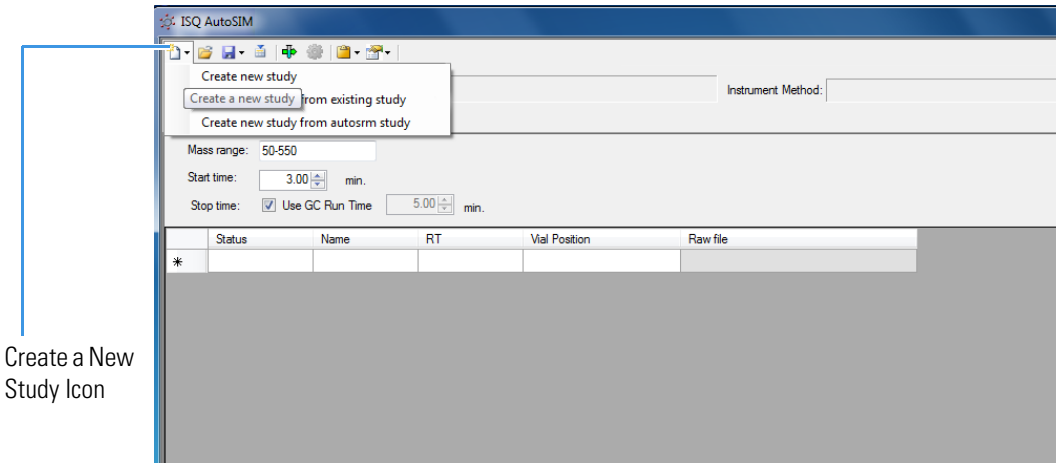
Figure 138. Accessing AutoSIM on the ISQ Dashboard



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.

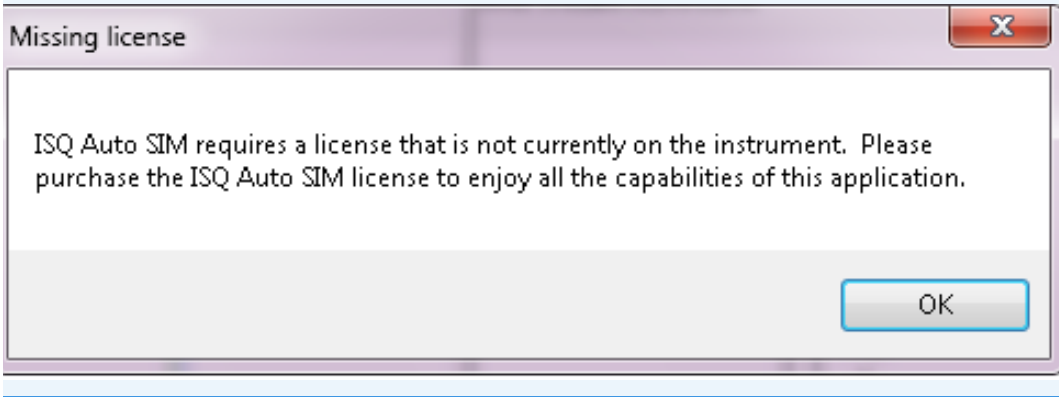
2. Click the Create a New Study icon on the left to create a new study.

Figure 139. New AutoSIM Study



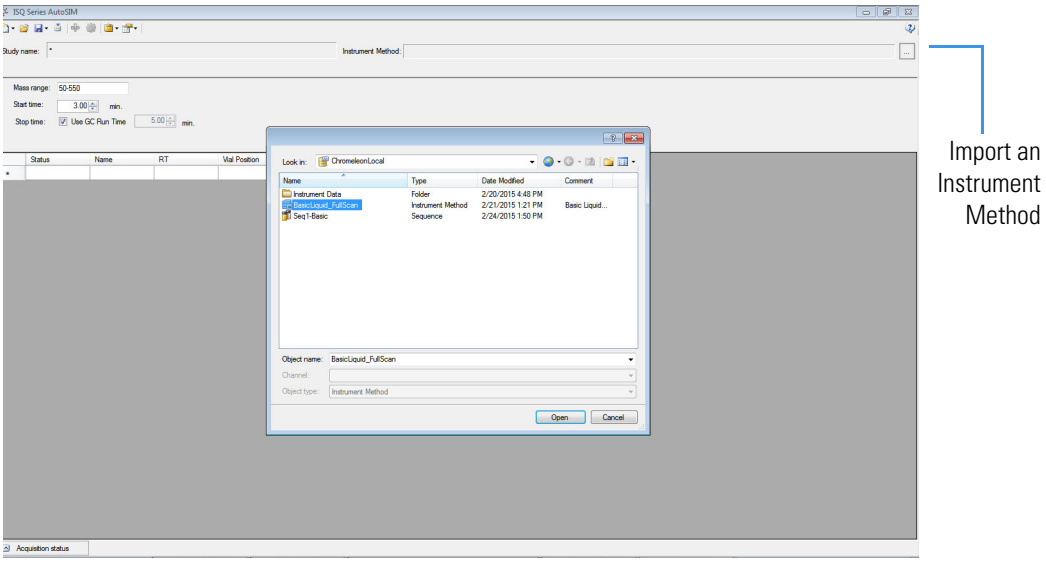
3. A new study window opens.

Note If at any time you see the error message below, go to "Upgrading the Software" on page 255 and follow the instructions for getting your software license.



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 121.

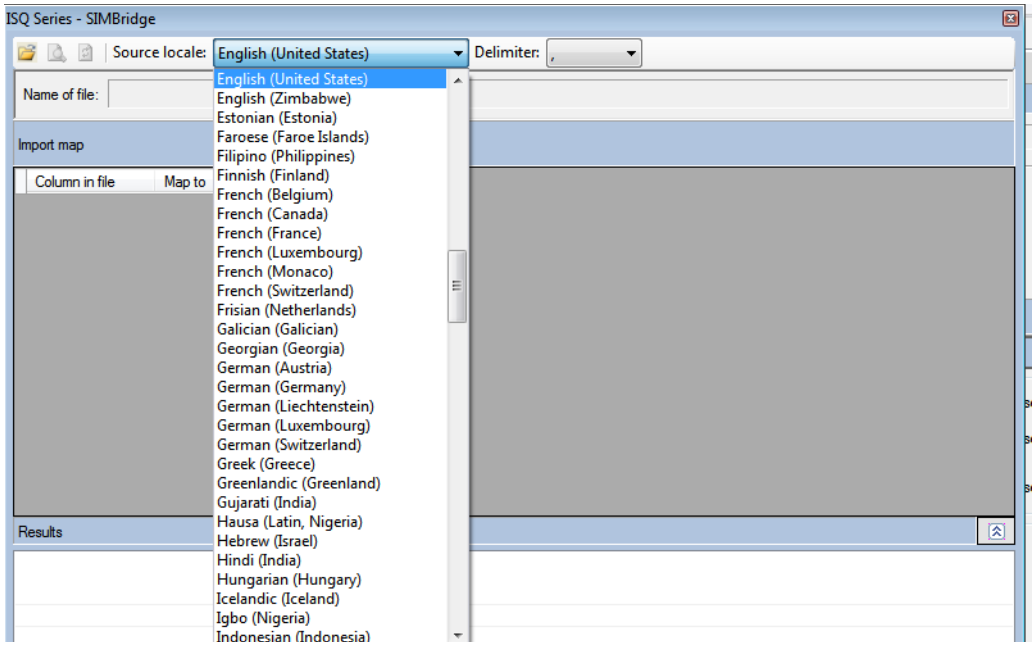
Figure 140. Retrieving an Instrument Method



5. If you are importing a file created on another system, click the import icon. Otherwise, select an instrument method file and click Open.

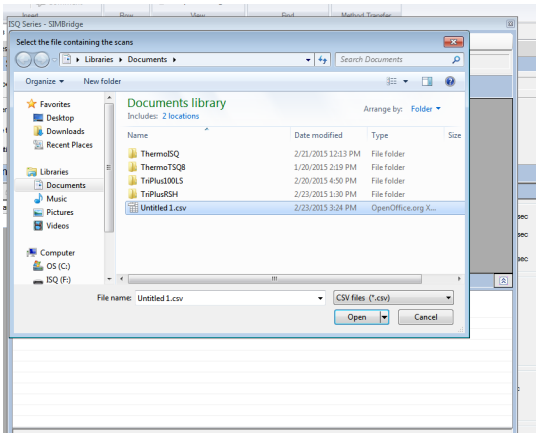
6. The **SIMBridge** dialog box opens. Choose the language of your method file from the **Source Locale** drop-down menu. See Figure 141.

Figure 141. 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 142.

Figure 142. Linking to an External File using SIM Bridge



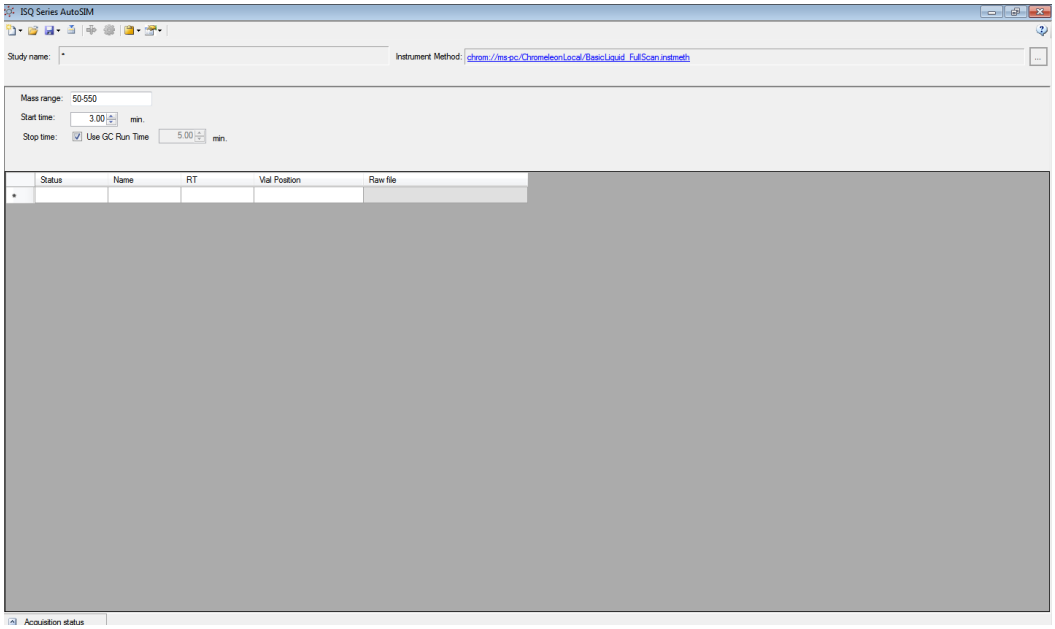
8. Click **Open** to open the method in SIMBridge.

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 122.

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

Figure 143. Adjusting the Settings

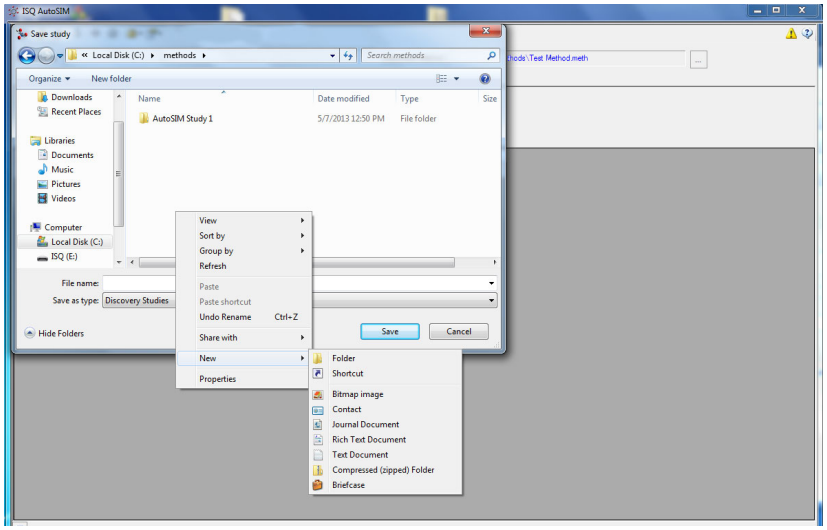


11. Enter the compound name, approximate retention time, and vial number for each compound you wish to optimize. If you already have a method for processing full scan data you can choose to import compounds from an external file. Their names and retention times will fill the compound list and their primary 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 123.

Figure 144. Creating an AutoSIM Study Folder



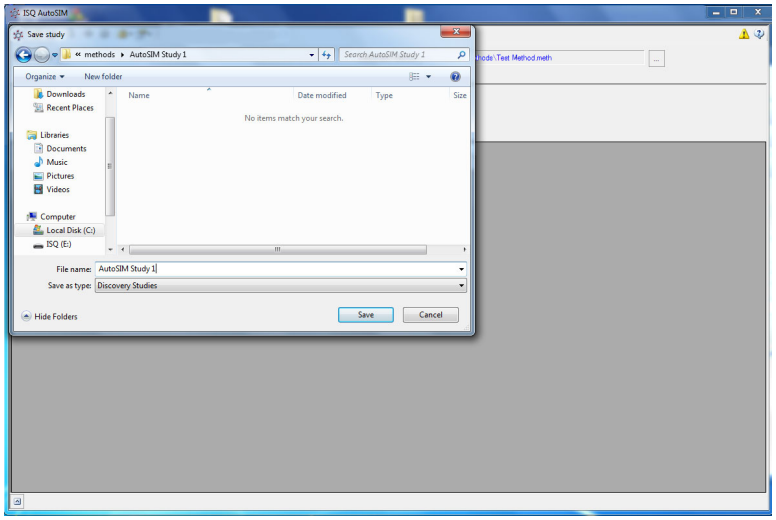
13. Open the folder.

14. Give your study a file name.

15. Save your study in the Study folder. See Figure 124.

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.

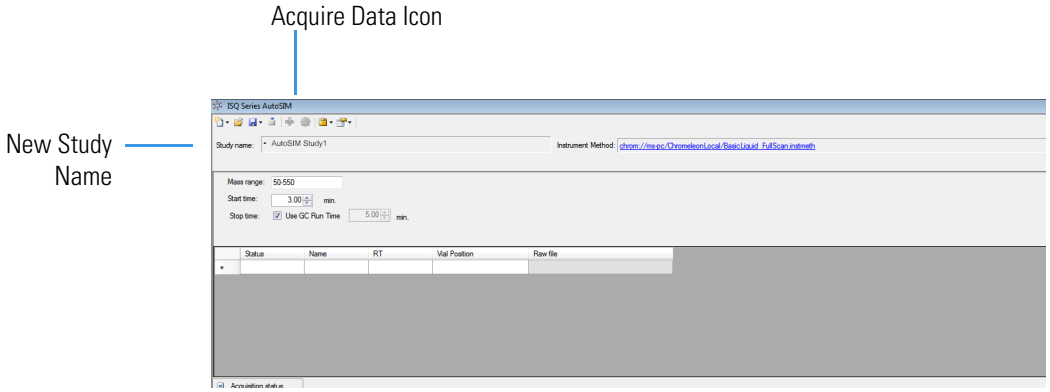
Figure 145. Saving an AutoSIM Study



16. The Windows Explorer window closes.

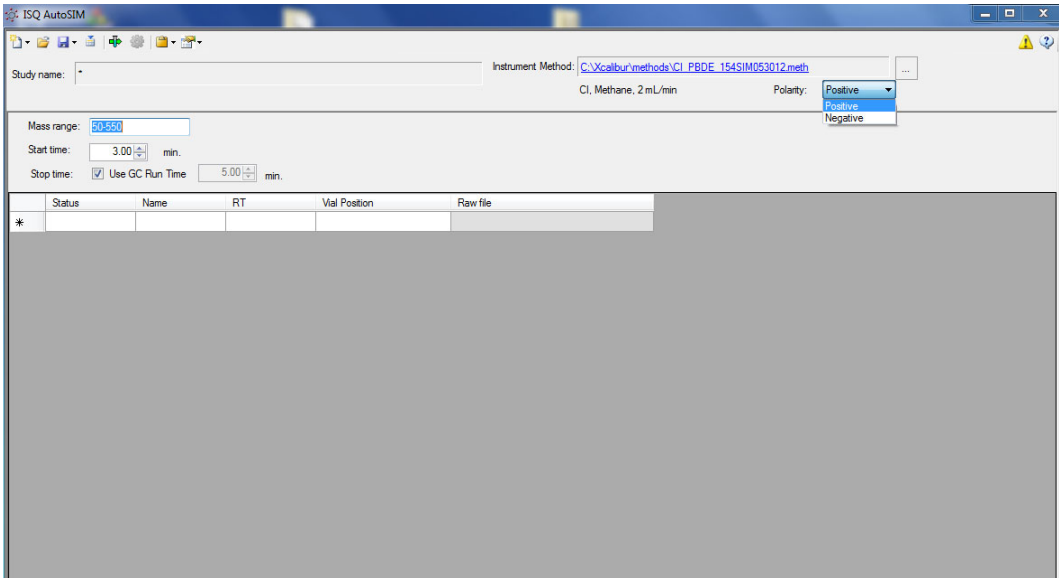
17. The AutoSIM Study Name is the name you assigned. See Figure 125.

Figure 146. Acquiring Acquisition Data



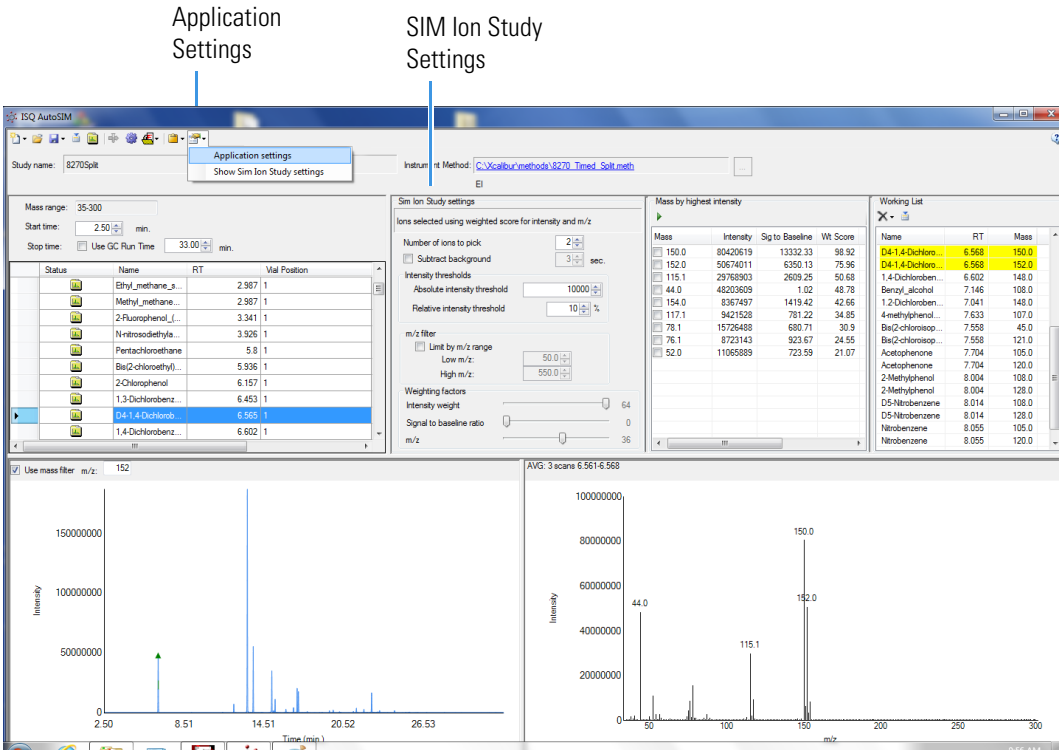
18. (CI Only) If you are running a chemical ionization (CI) study, select **Positive** or **Negative** from the **Ion Polarity** pull-down menu. See Figure 126.

Figure 147. Setting Ion Polarity in a CI 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 127.

Figure 148. Application Settings and SIM Ion Study Settings



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.

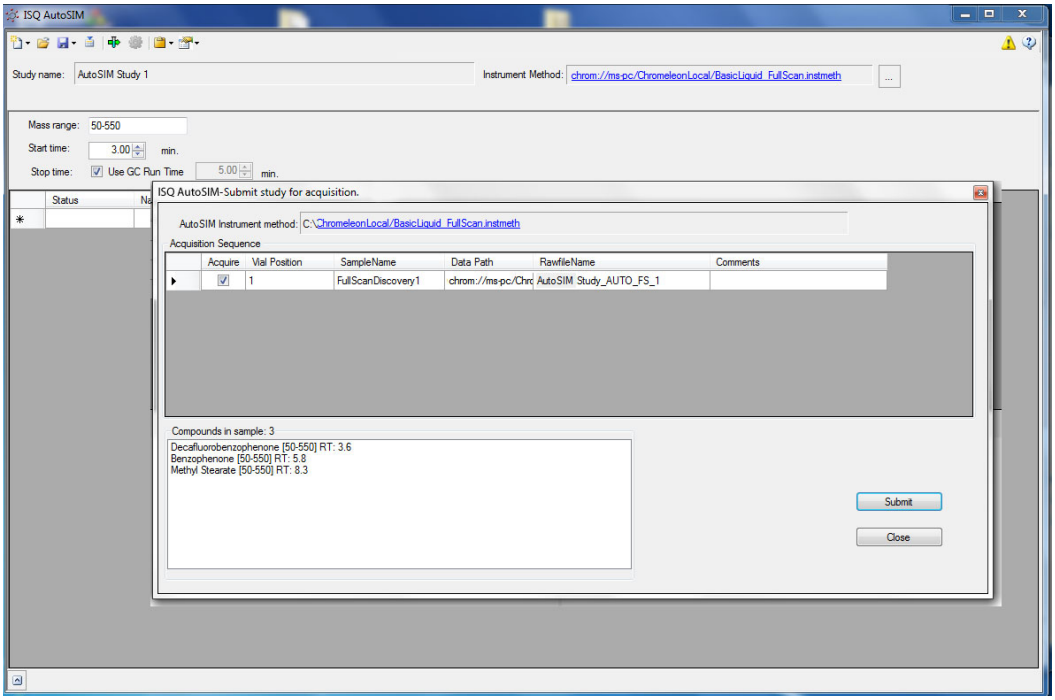
e. **Weighting Factors**: Use the sliding bars and check boxes to set the values you want to give each SIM ion study setting.

21. Click the **Acquire Data** icon to run your samples. See Figure 125.

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 128.

Figure 149. Submitting a Study for Acquisition



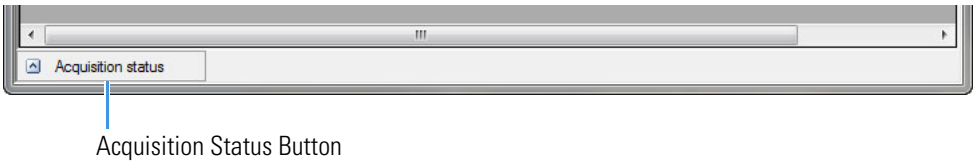
23. Click **Submit** to submit the samples to the instrument.

    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.

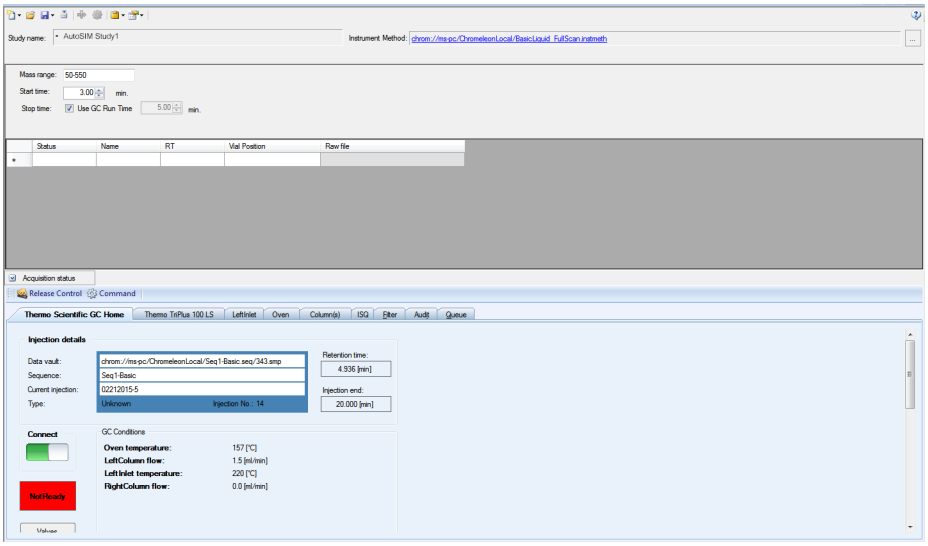
24. The Acquisition Status pane displays status information about the running instrument. If the pane is not currently visible, click the **Acquisition Status** button. See Figure 150.

Figure 150. Displaying the Acquisition Status Pane



The pane contains a Chromeleon ePanel Set that you can use to monitor the status of AutoSIM injections. See Figure 151. 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.

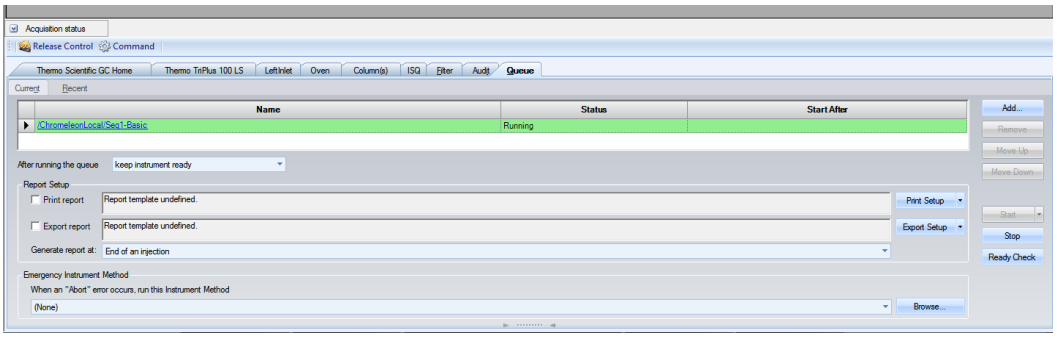
Figure 151. Viewing Instrument Status on the Home ePanel



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 152.

Figure 152. Viewing Queue Status on the Queue ePanel



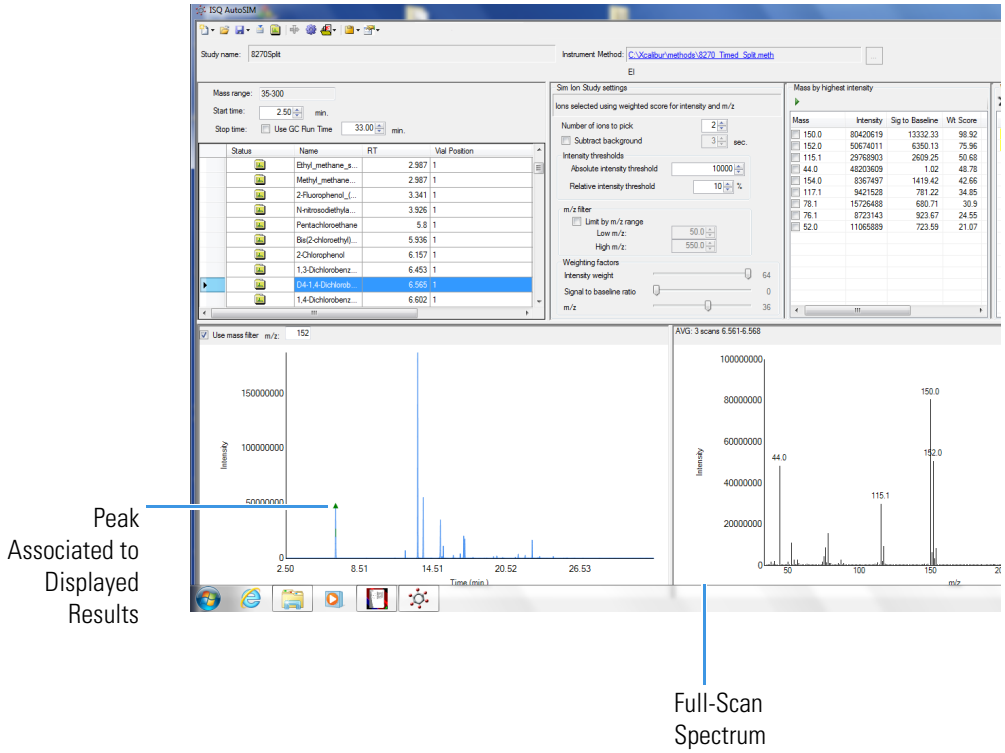
Once the samples have finished running, the software analyzes the data.

27. The results appear in the AutoSIM window. See Figure 129.

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.

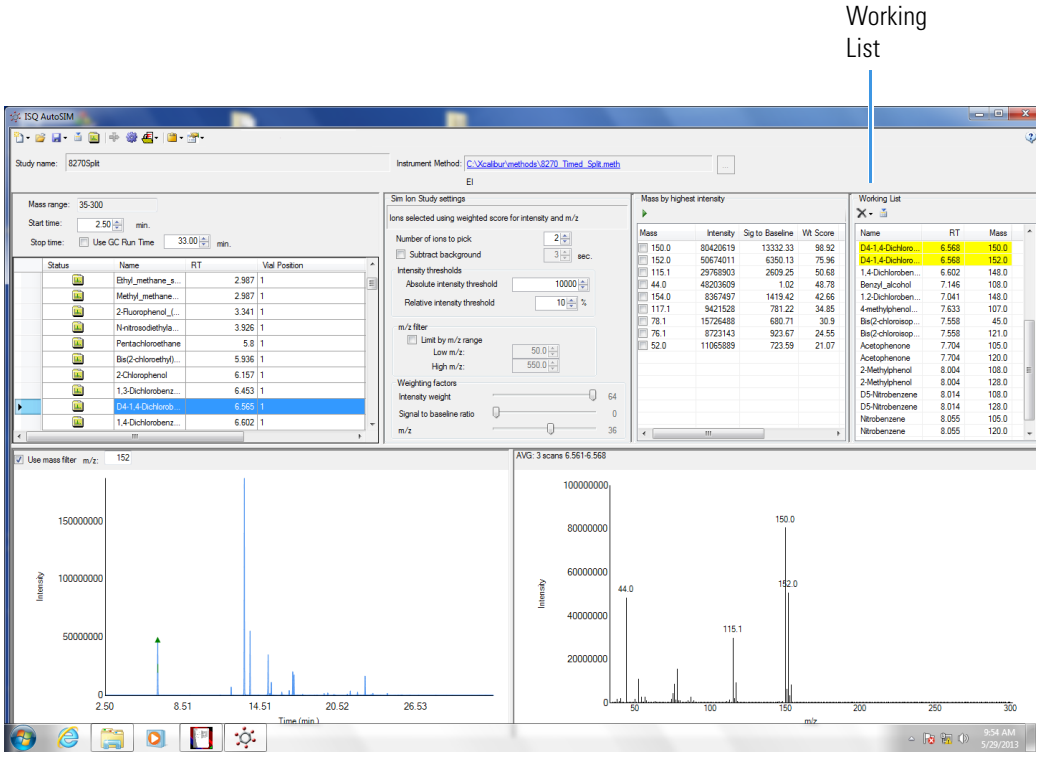
> **Note** Background subtraction updates the ions in the Mass by Highest Intensity pane.

Figure 153. 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 130.

Figure 154. Selecting SIM Ions

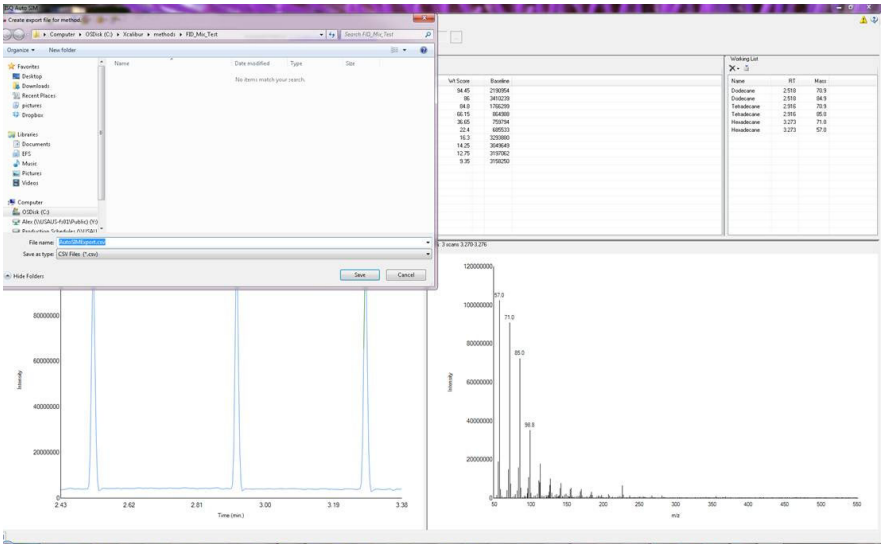


30. Repeat this process for all of your compounds.

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 131.

Figure 155. Exporting your SIM Ion Study



Importing Transitions to the Chromeleon Instrument Method Editor

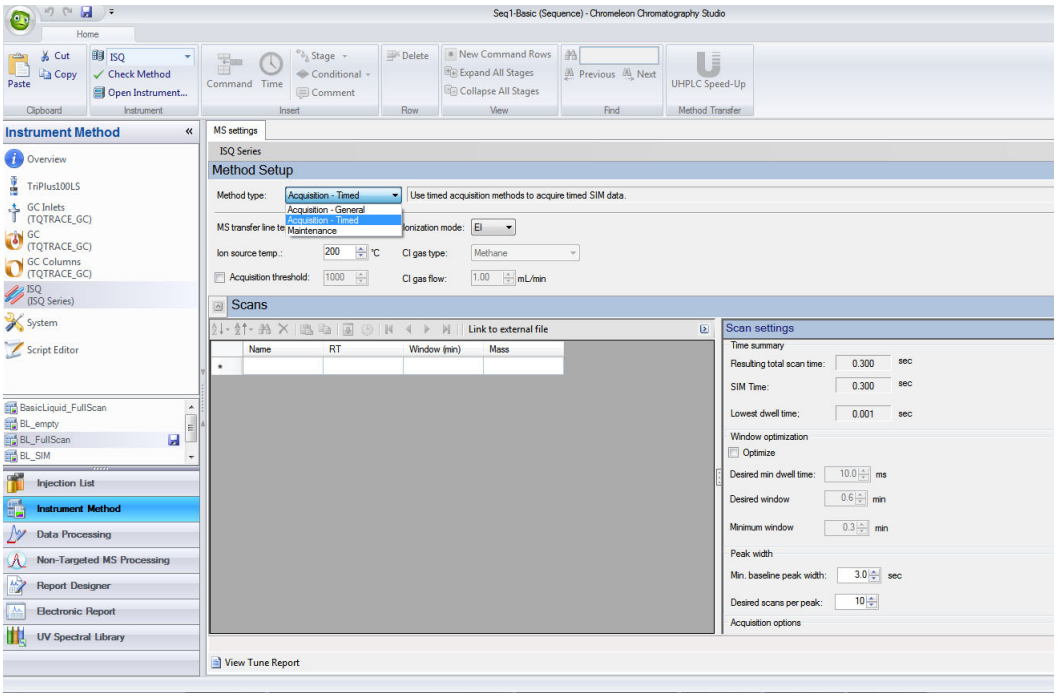
❖ To import the list of transitions you created in AutoSIM

1. Open the Chromeleon Instrument Method Editor.

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

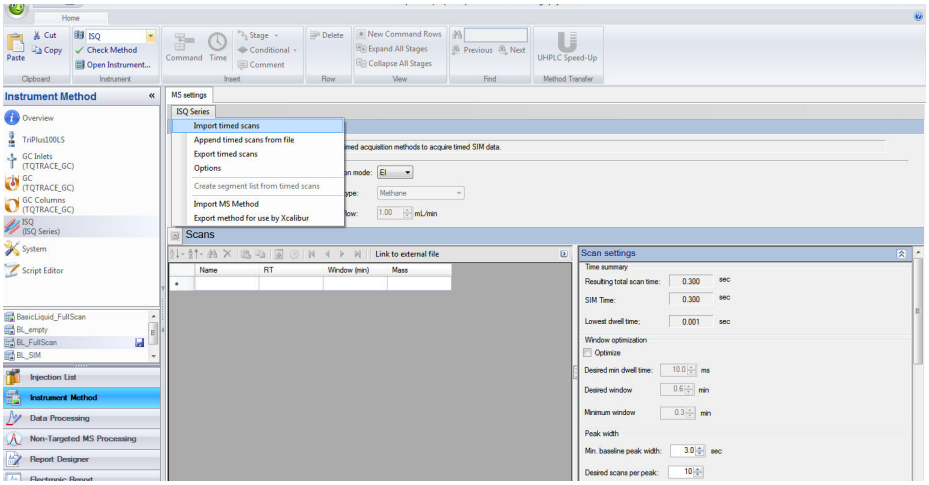
2. Select the **Acquisition – Timed** method type.

Figure 156. Opening Instrument Method in Xcalibur



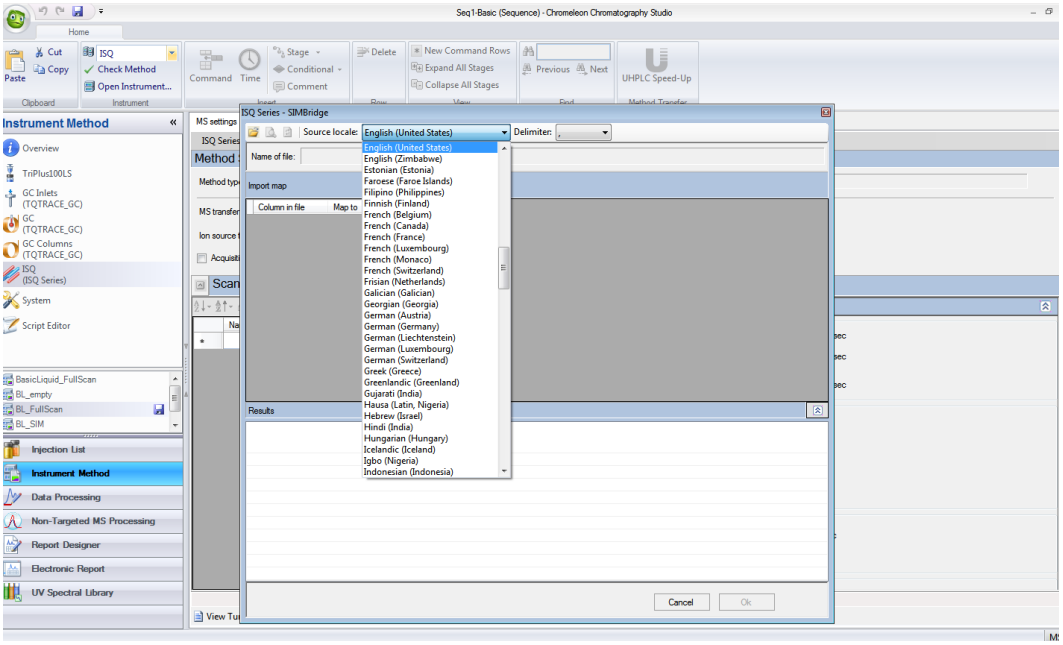
3. In the **MS Settings** pane, select **ISQ 7610 > Import Timed Scans**.

Figure 157. Importing Timed Scans



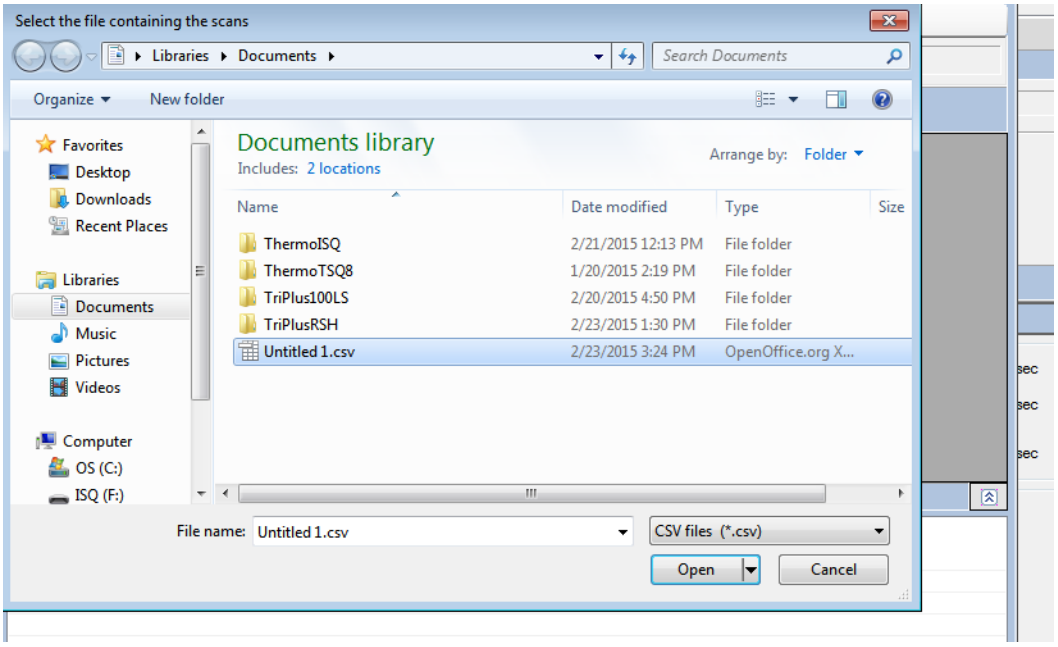
4. The **SIMBridge** dialog box opens. Choose the language of your method file from the **Source Locale** drop-down menu.

Figure 158. Setting the Source Language of Method Files using SIMBridge



5. Browse to your file.

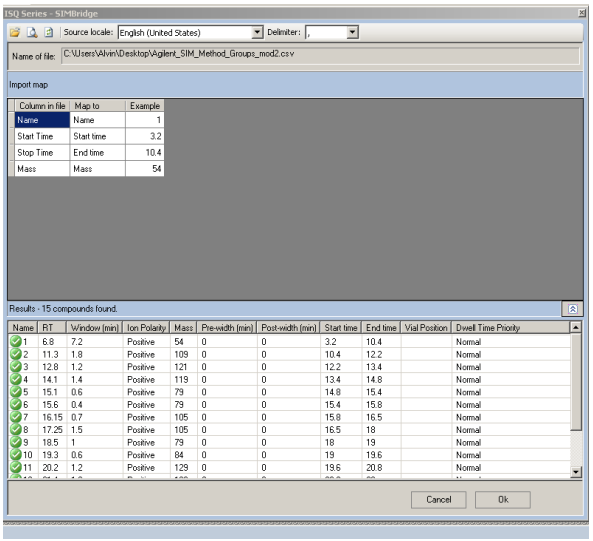
Figure 159. Linking to an External File using SIM Bridge



6. Click **Open** to open the method in SIMBridge.

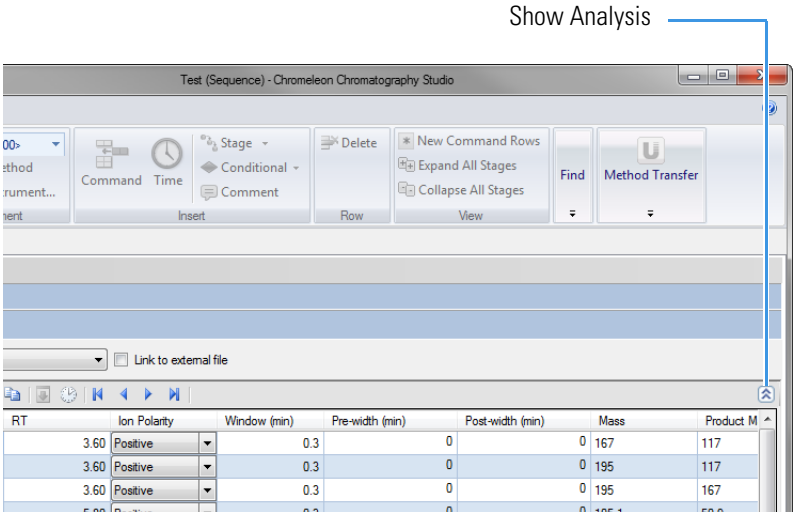
7. If necessary, change the method headings in your original file to match those in the method editor. A green check mark appears when your method is validated.

Figure 160. 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 7610 icon in the method editor side pane.
10. Click **Show Analysis** and review the imported list of compounds.

Figure 161. Selecting Show Analysis



11. Adjust your scan settings as necessary. See "Creating a Method" on page 63 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.

6

Running a Sample

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

Contents

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 in Xcalibur Software

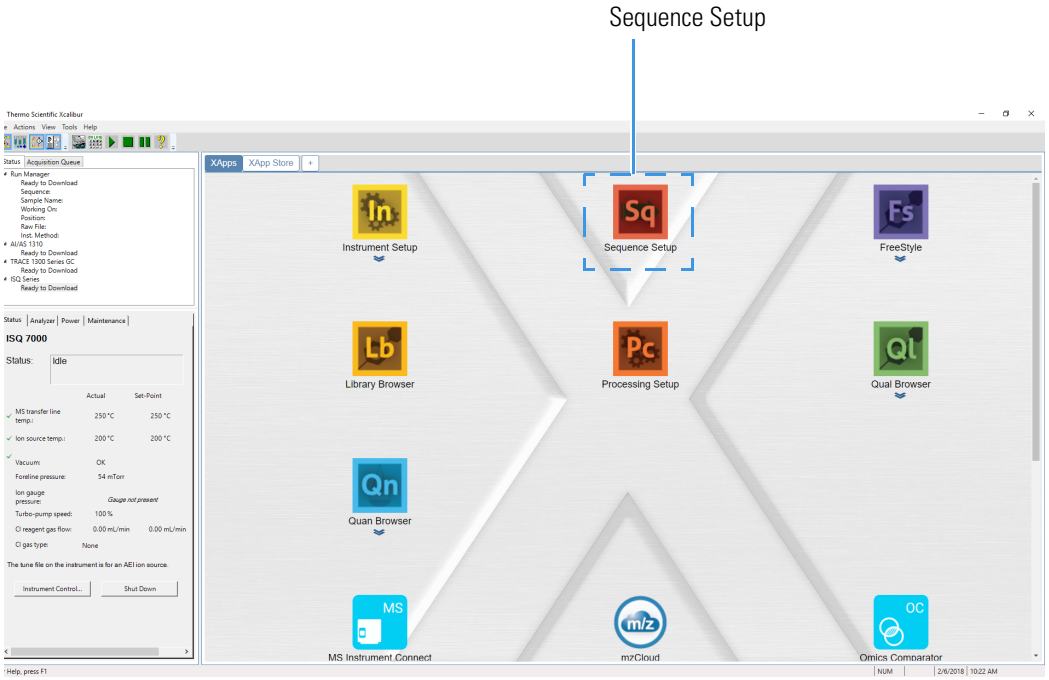
Once you've prepared your sample, you can use Xcalibur to run your sample(s). Xcalibur uses a sequence (7610 of tasks) to prepare the instruments for data acquisition, as well as monitor the injection and collection of data from the GC/MS.

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.

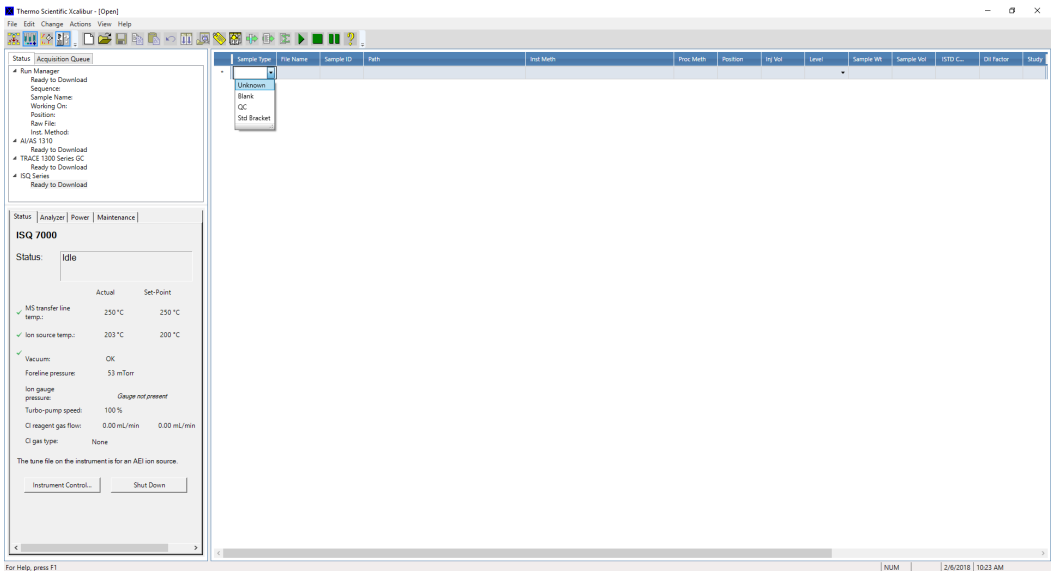
Figure 162. Sequence Setup in Xcalibur



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 icon and browse to a sequence.

Figure 163. Entering the Sample Type



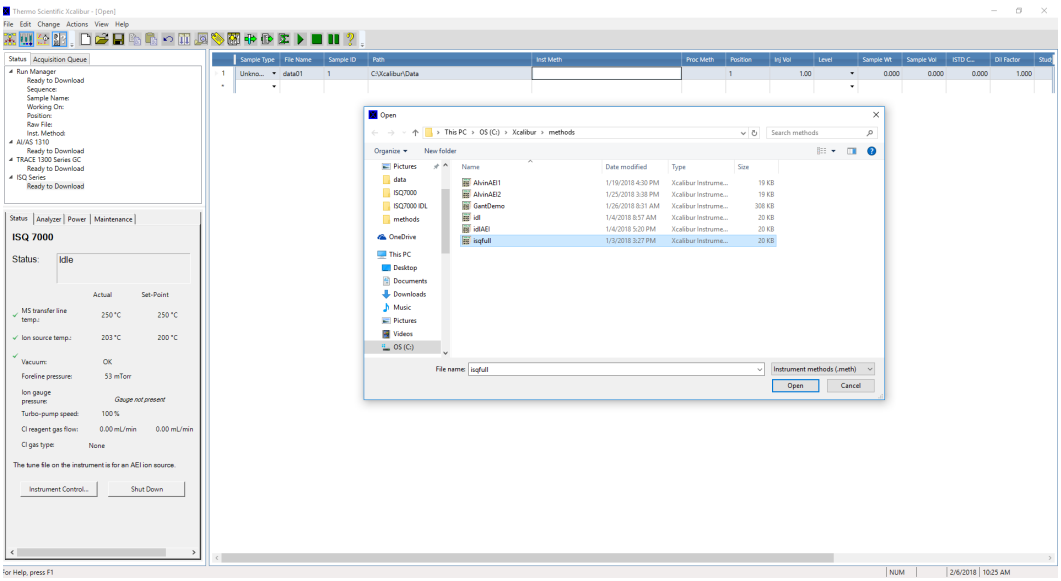
Once you select a sample type, default information automatically appears in some of the other columns. You can also right-click on the field to clear, copy, or paste into the field, as well as add and delete rows.

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 164. Naming a File

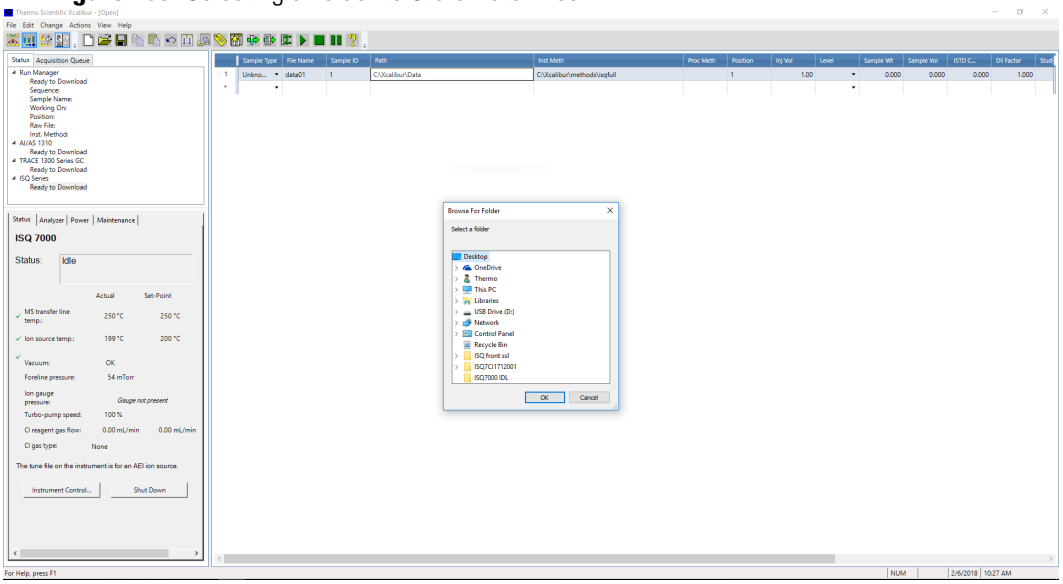


Tip You can double-click in the **File Name** field and browse to a raw file on your computer.

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.

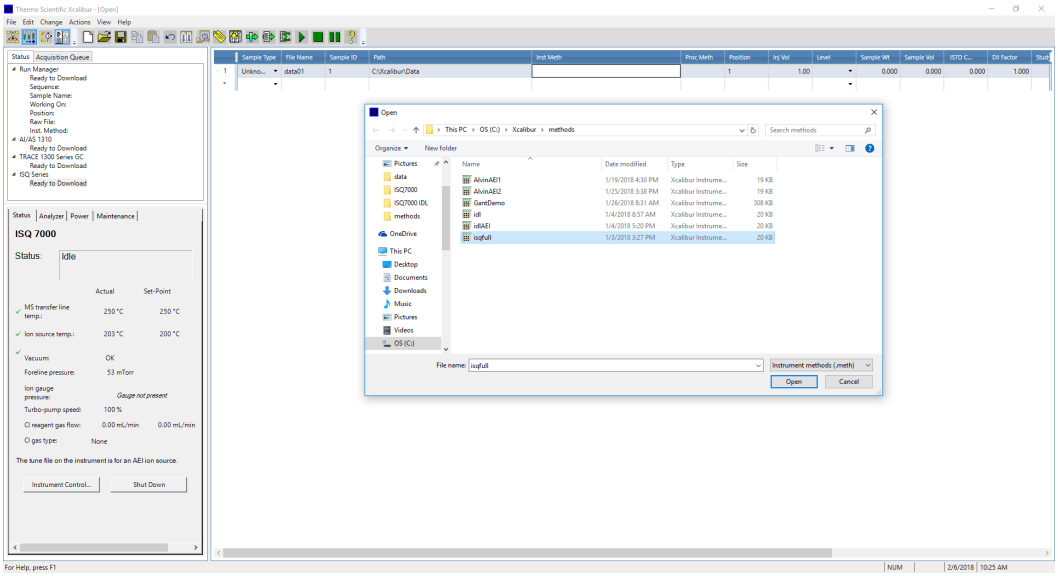
6. Double-click the first row in the **Path** column and select the folder in which to store your data files. You can also right-click on the field to clear, copy, or paste into the field, browse to a folder on your computer, or add and delete rows.

Figure 165. 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.

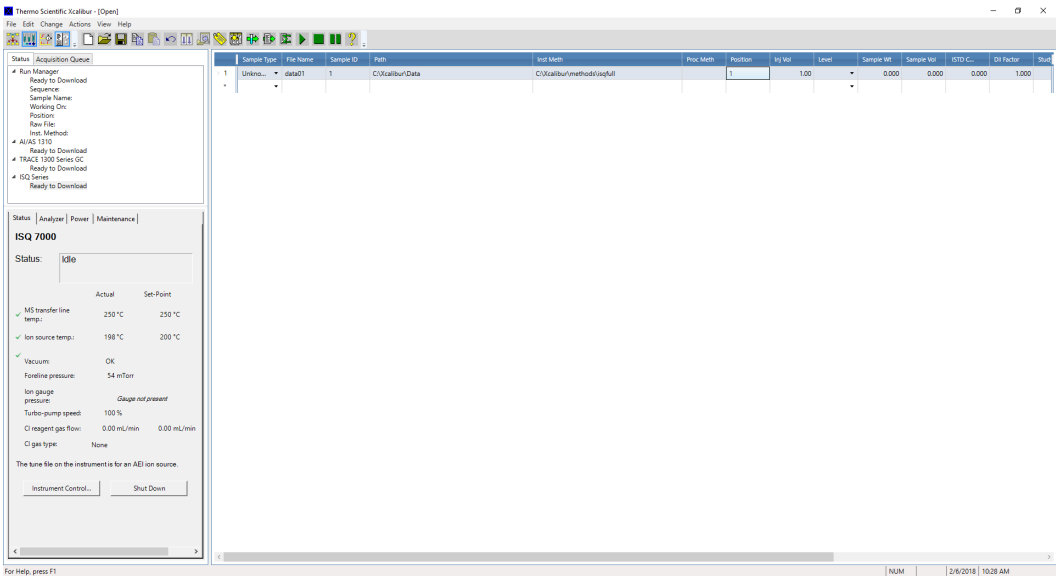
Figure 166. Opening an Instrument Method Folder



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.

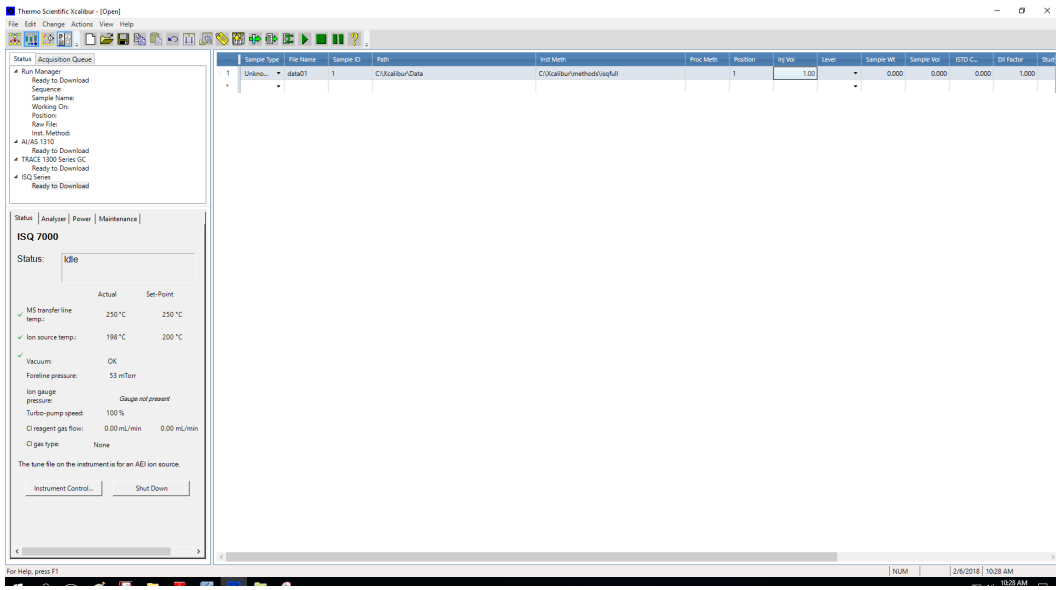
Figure 167. Entering the Sample Vial Position



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 1600, which uses an injection volume from the instrument method. The TriPlus and TriPlus RSH autosamplers must be configured to read this value.

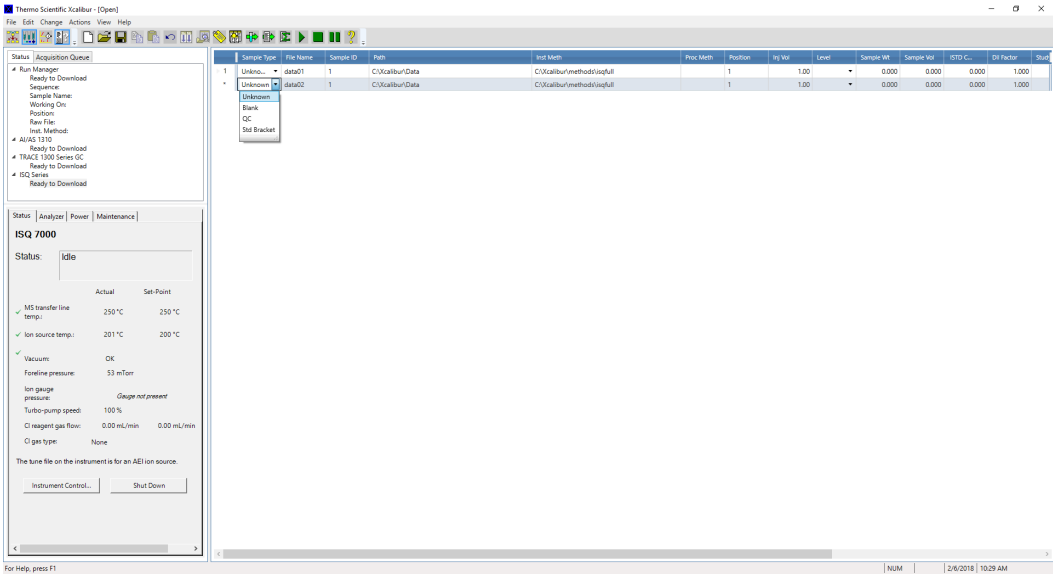
Figure 168. Setting the Injection Volume



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.

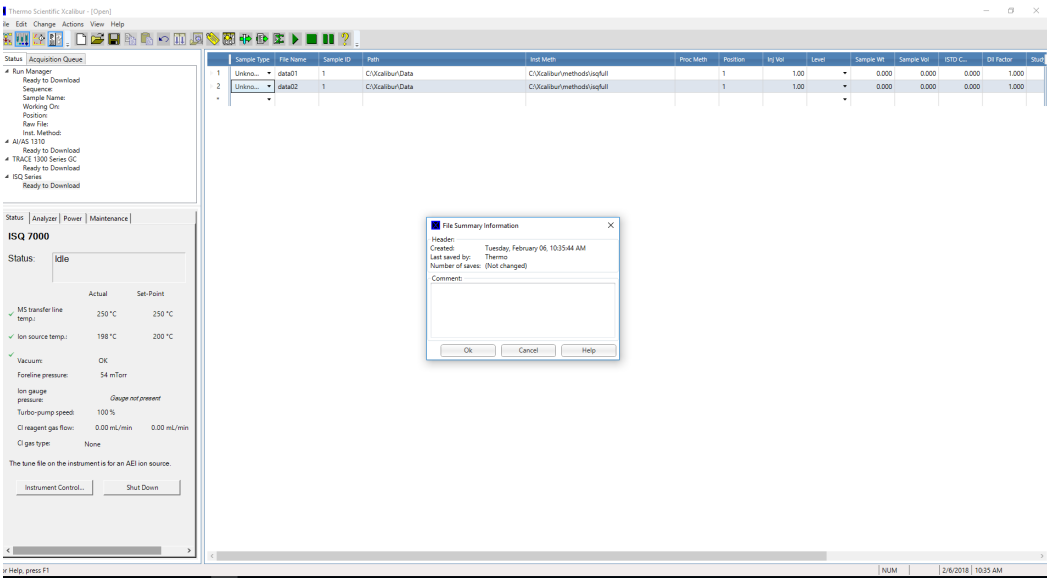
Figure 169. Adding Additional Sequence Setup Tasks



13. Once you've created a sequence of all your samples, select **File | Save** or click the icon to save the sequence.

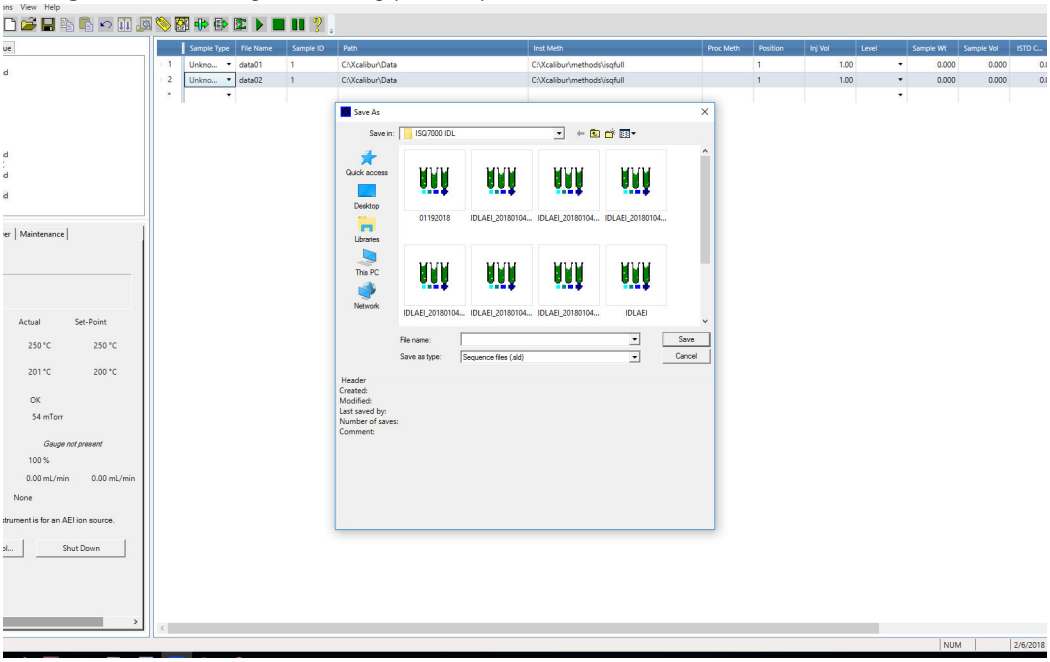
14. Enter a comment and click **OK**.

Figure 170. Entering a Comment About Your Sequence



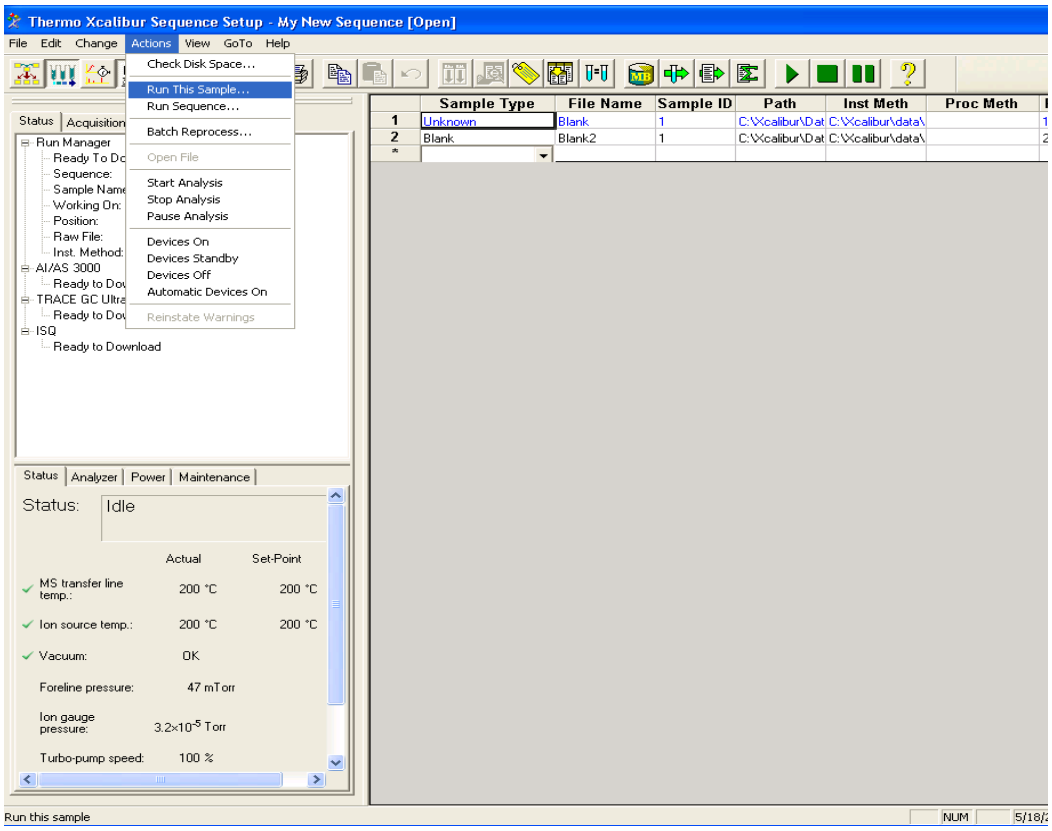
15. Enter the name of your sequence and click the **Save** button.

Figure 171. 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.

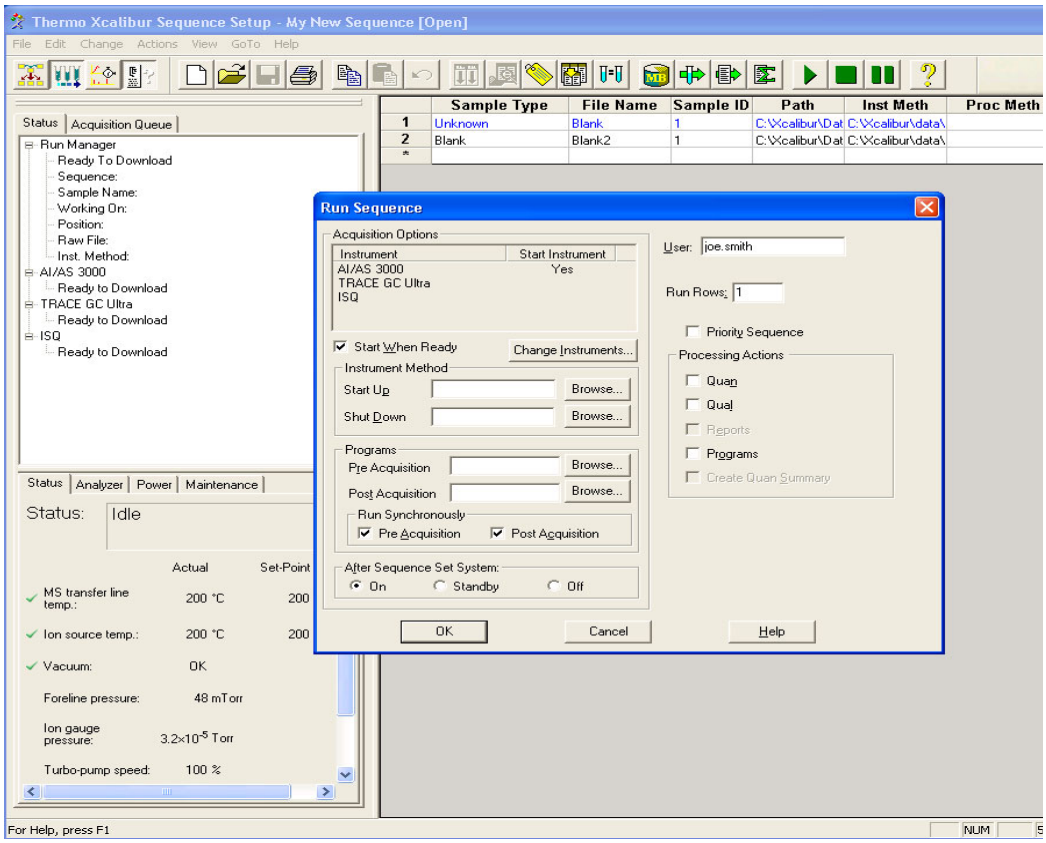
Figure 172. Selecting a Row of Samples to Run



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

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

Figure 173. 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 7610 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.

Running a Sequence in Chromeleon Software

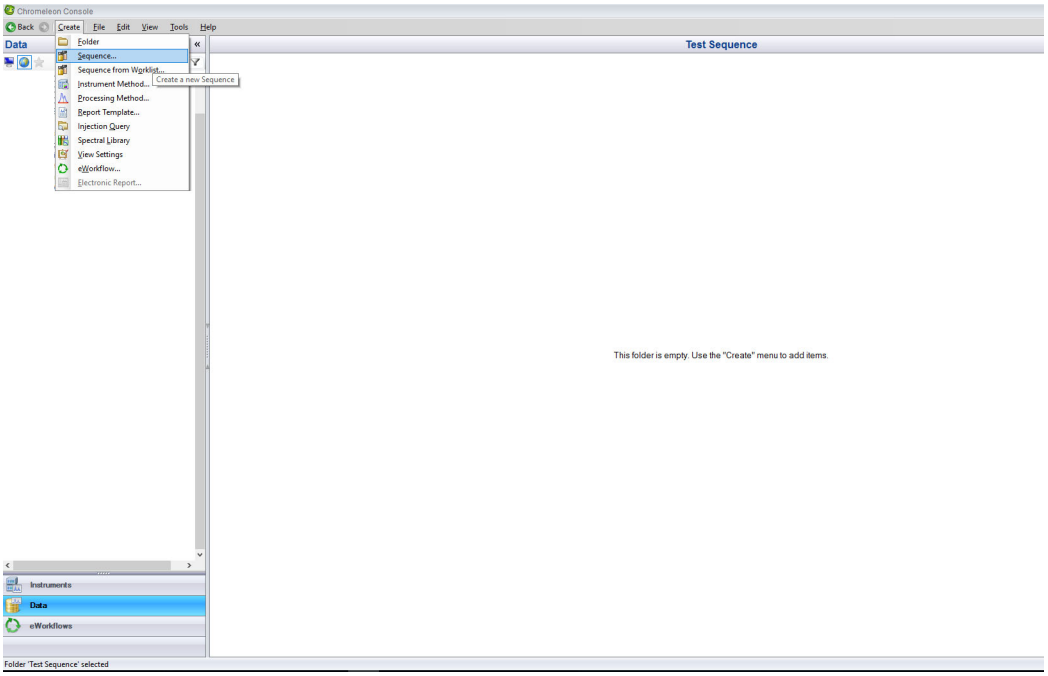
Once you've prepared your sample, you can use Chromeleon software to run your sample(s). Chromeleon uses a sequence to prepare the instruments for data acquisition, as well as monitor the injection and collection of data from the GC/MS.

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

❖ To run a new sequence of samples

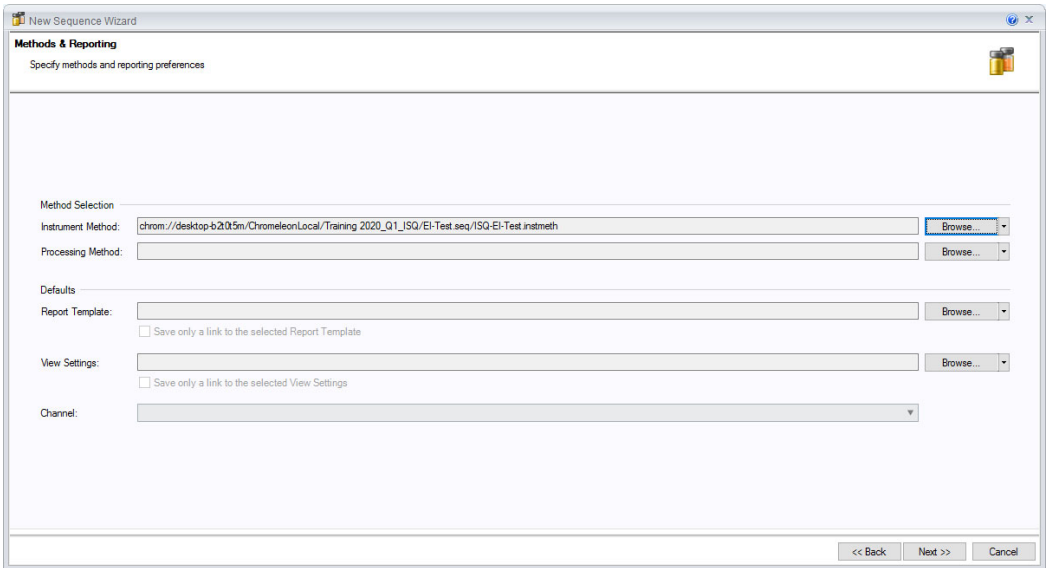
1. Open Chromeleon software on your computer.
2. In the Chromeleon Console, click the **Create > Sequence**.

Figure 174. Sequence Setup in Chromeleon



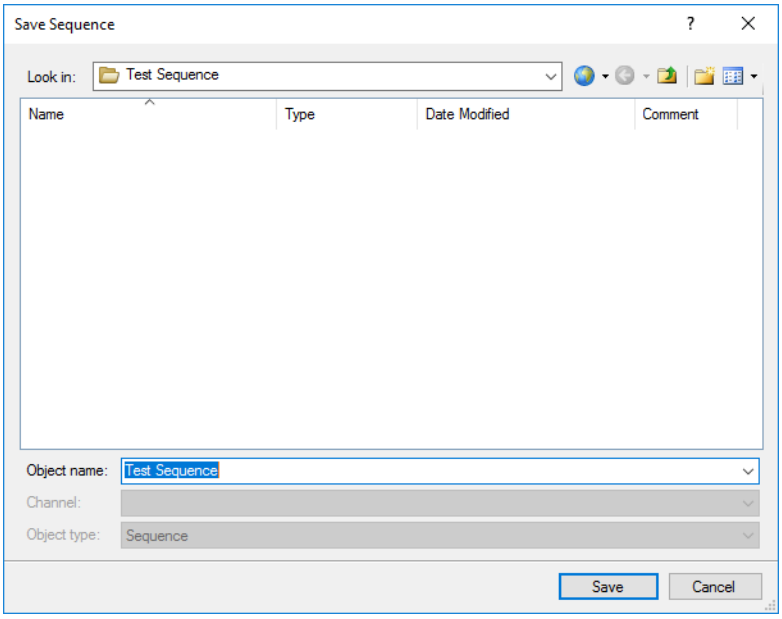
3. The **New Sequence Wizard** opens. Click **Next** to move through the wizard until the **Methods & Reporting** page opens. Browse to an instrument method and select it for your sequence.

Figure 175. Selecting an Instrument Method



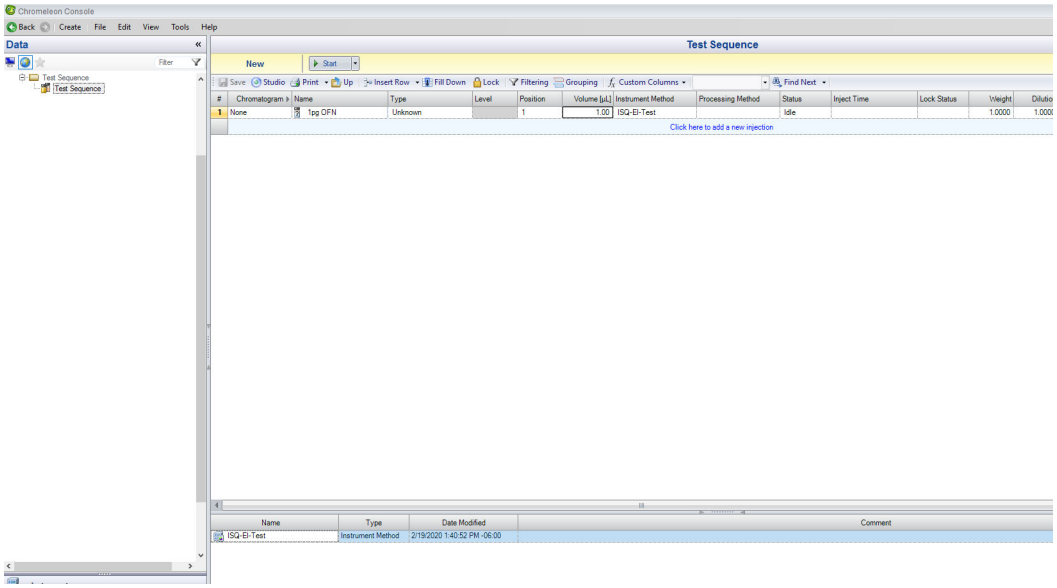
4. Click **Next** through the wizard and then click **Finish**. Name and save the sequence.

Figure 176. Naming and Saving a Sequence



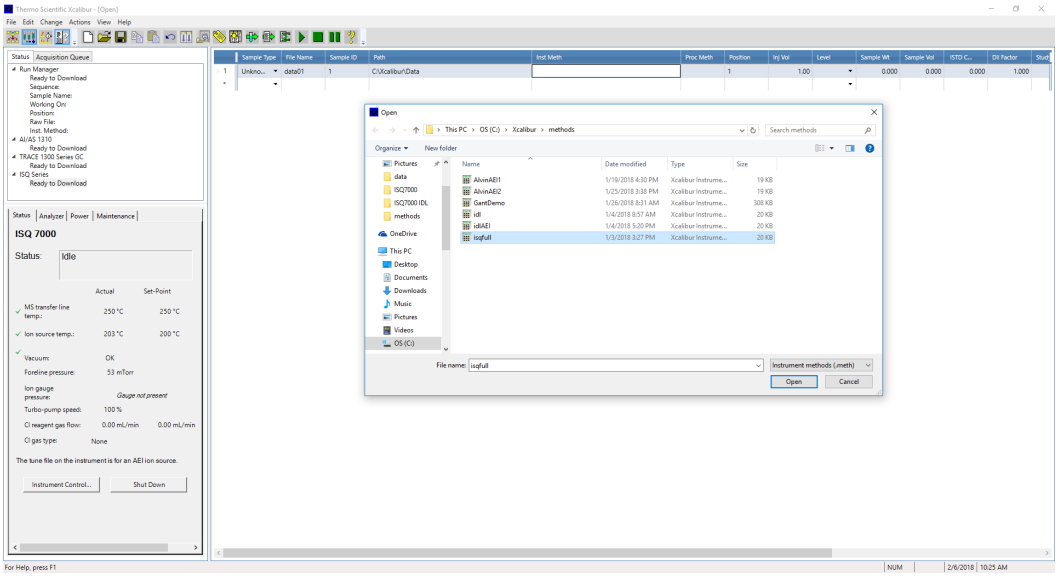
5. The sequence opens in the Chromeleon Console. Change the injection volume and vial position if needed and click **Start**.

Figure 177. Selecting a Folder to Store Data Files



6. 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.

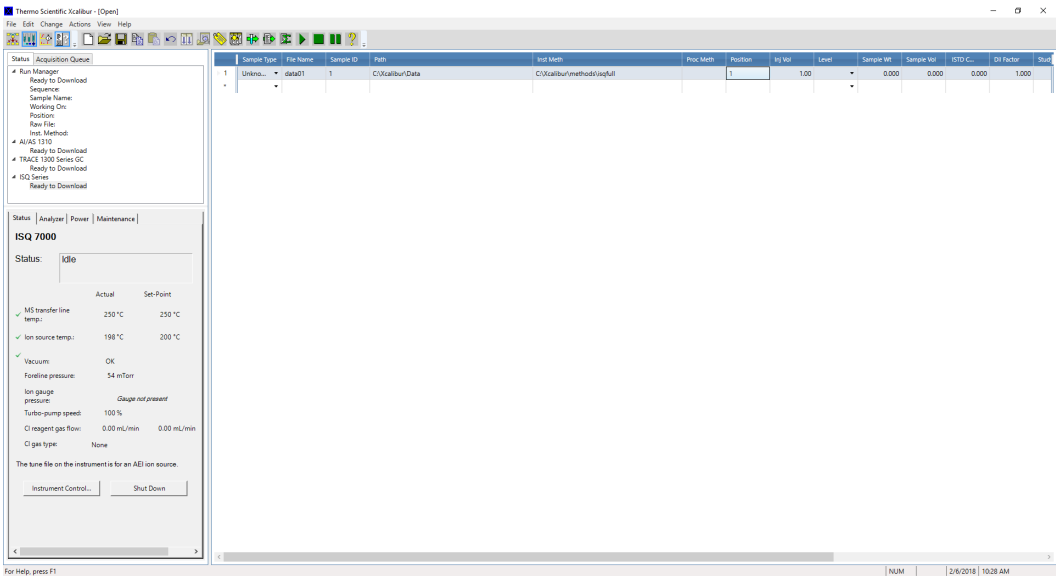
Figure 178. Opening an Instrument Method Folder



7. 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.

8. 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.

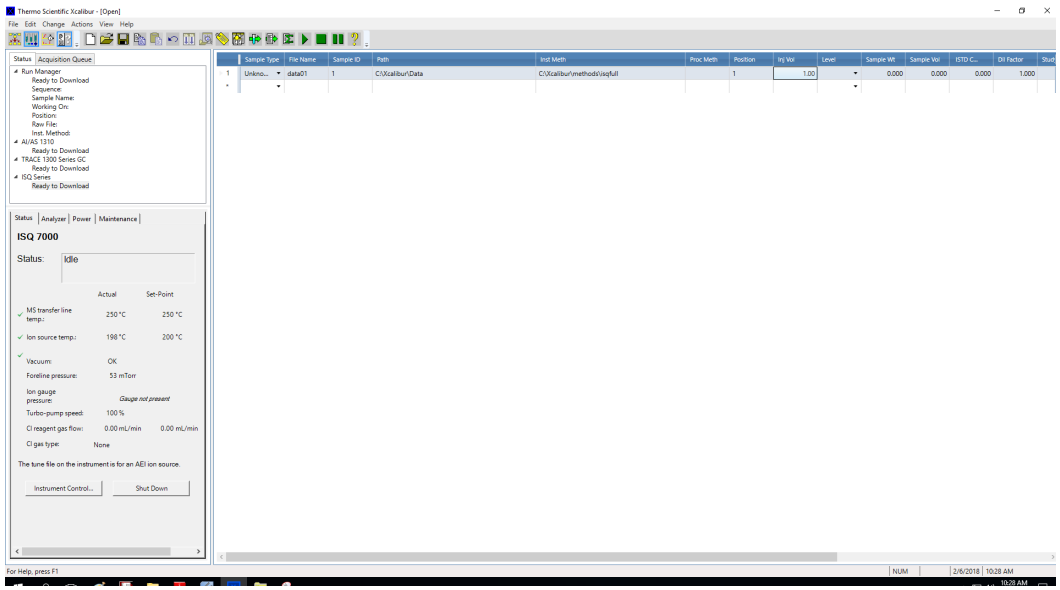
Figure 179. Entering the Sample Vial Position



9. 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 1600, which uses an injection volume from the instrument method. The TriPlus and TriPlus RSH autosamplers must be configured to read this value.

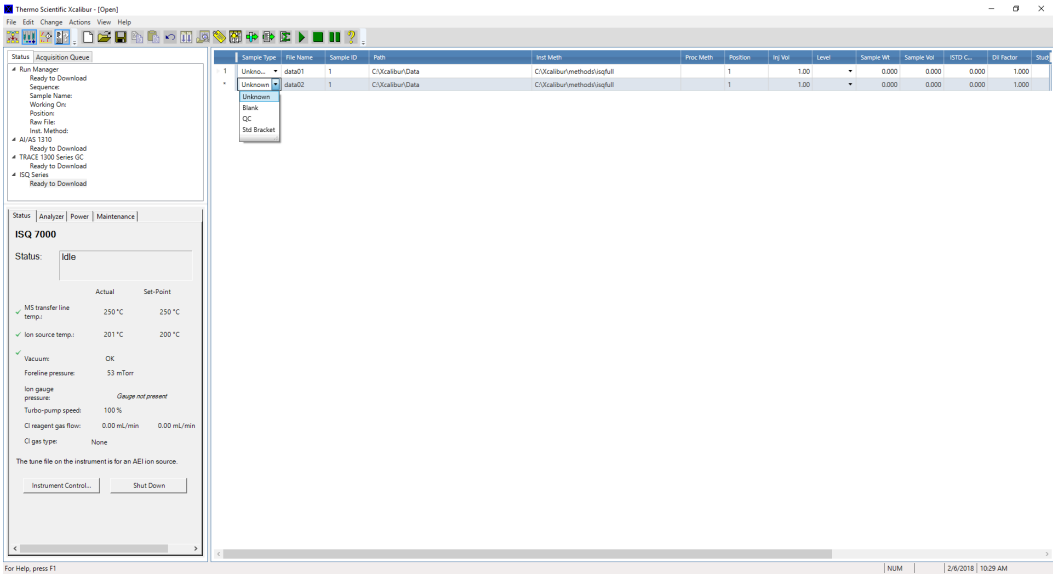
Figure 180. Setting the Injection Volume



10. 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.

11. 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.

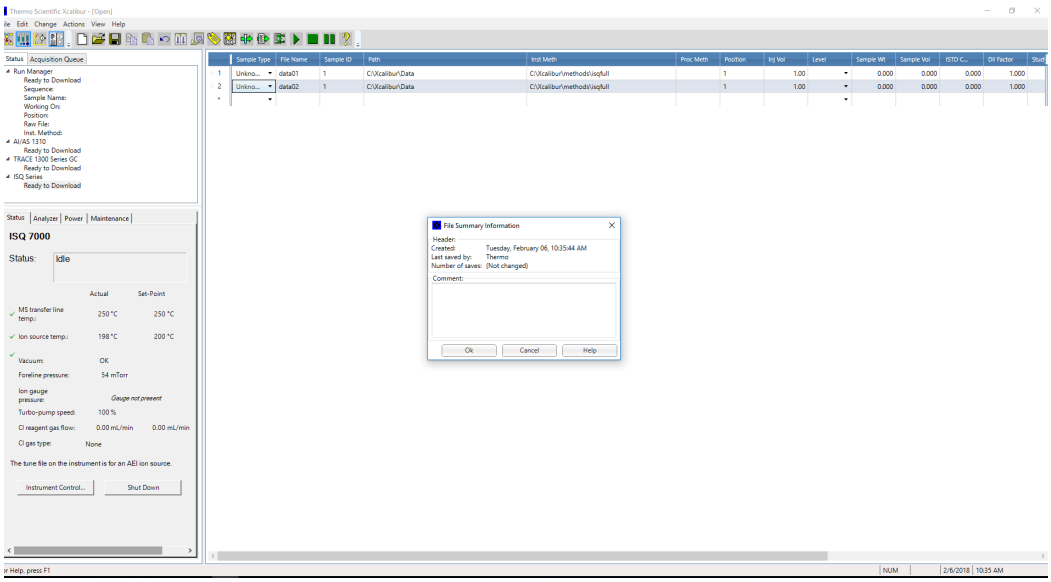
Figure 181. Adding Additional Sequence Setup Tasks



12. Once you've created a sequence of all your samples, select **File | Save** or click the icon to save the sequence.

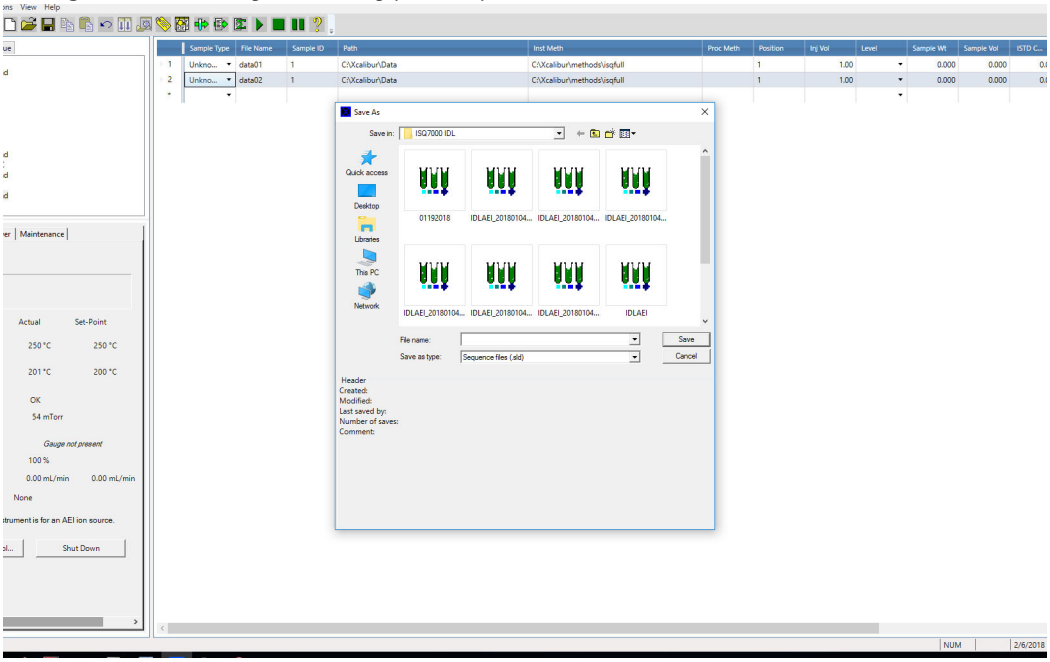
13. Enter a comment and click **OK**.

Figure 182. Entering a Comment About Your Sequence



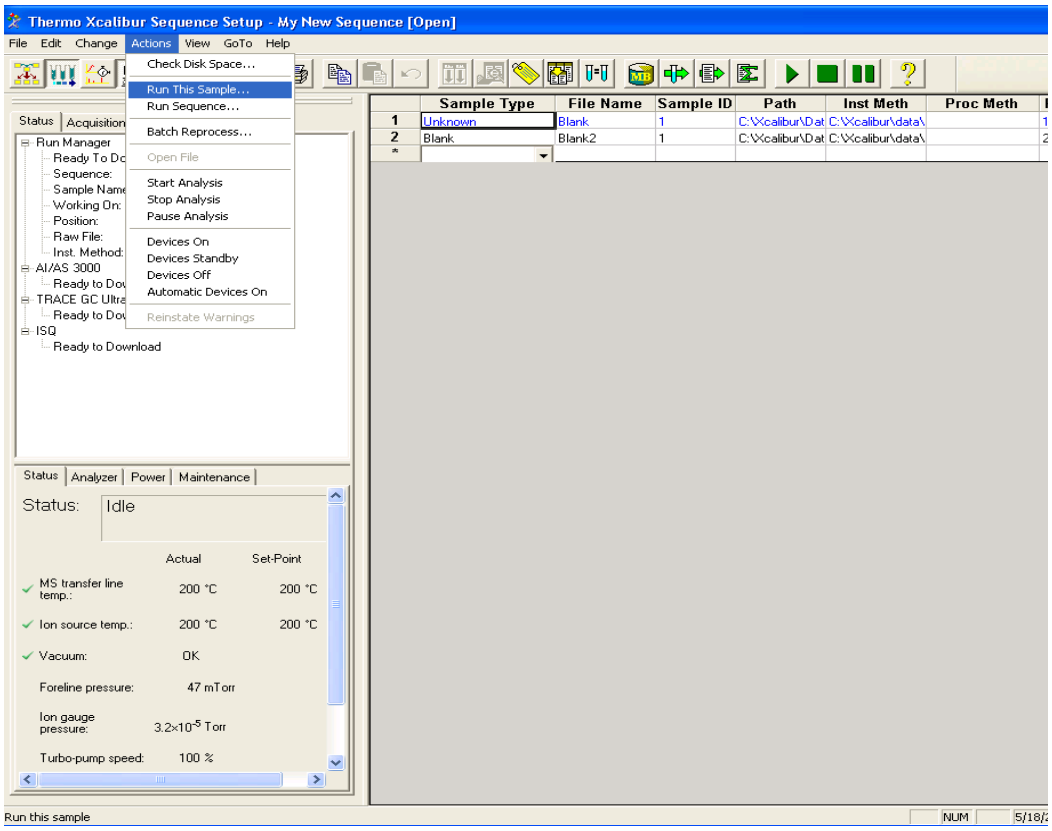
14. Enter the name of your sequence and click the **Save** button.

Figure 183. Naming and Saving your Sequence



15. 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.

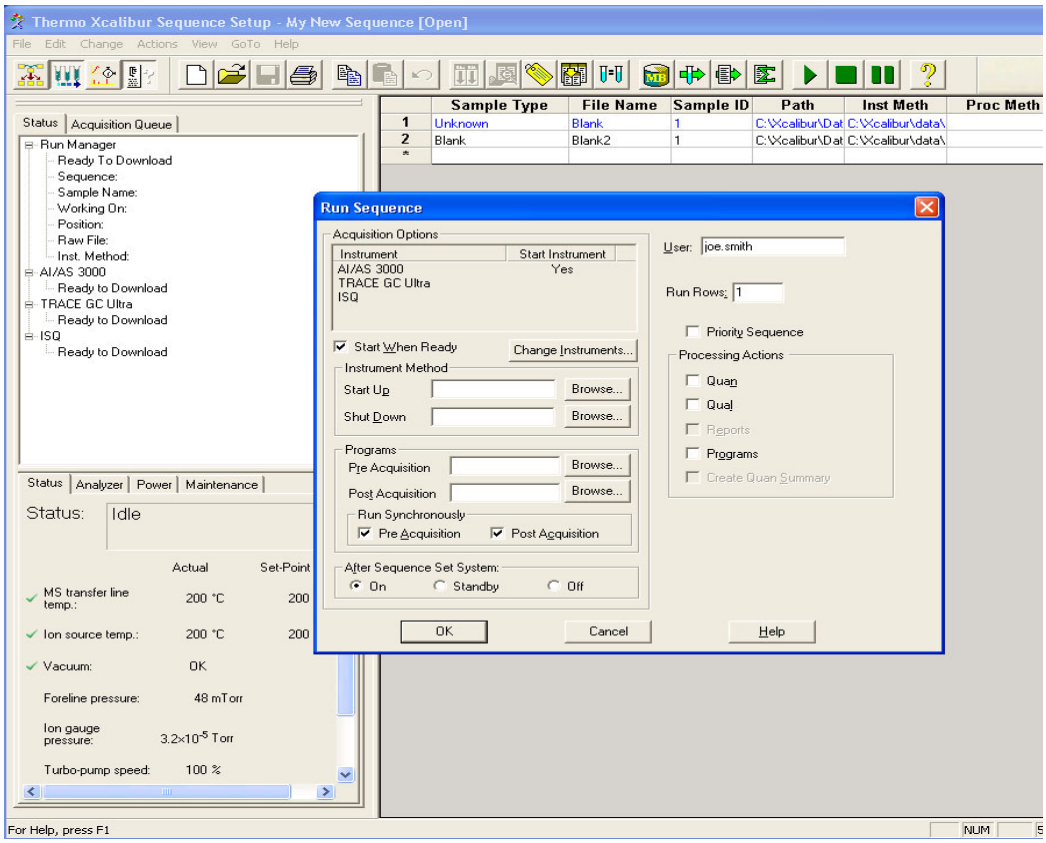
Figure 184. Selecting a Row of Samples to Run



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

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

Figure 185. 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 7610 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.

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

18. In the **Save As** window, select a location on your computer that you want to store your sequence. Then click the **Save** button.

Once your sequence completes, you are ready to explore your data.

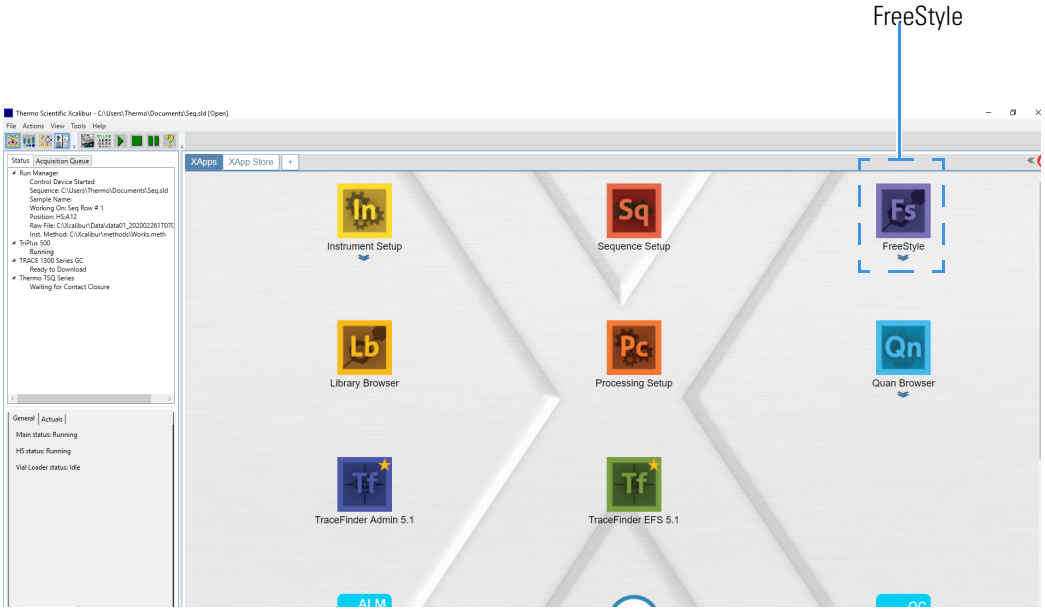
Exploring Your Data

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 FreeStyle 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 FreeStyle:**

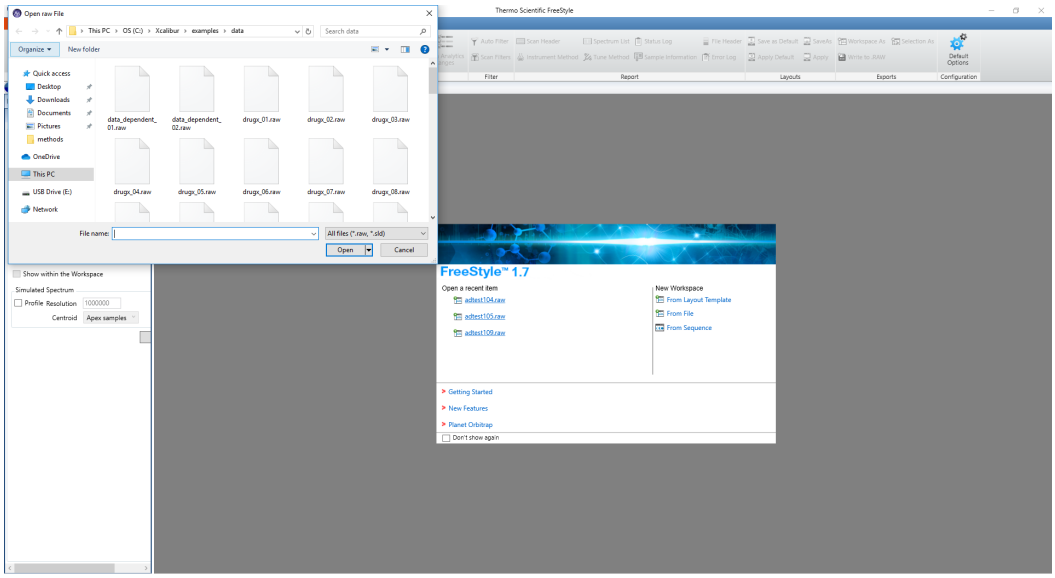
1. Double-click the **Xcalibur** software icon on your computer desktop.
2. In the Xcalibur Roadmap window, click the **FreeStyle** icon.

Figure 186. Xcalibur Roadmap



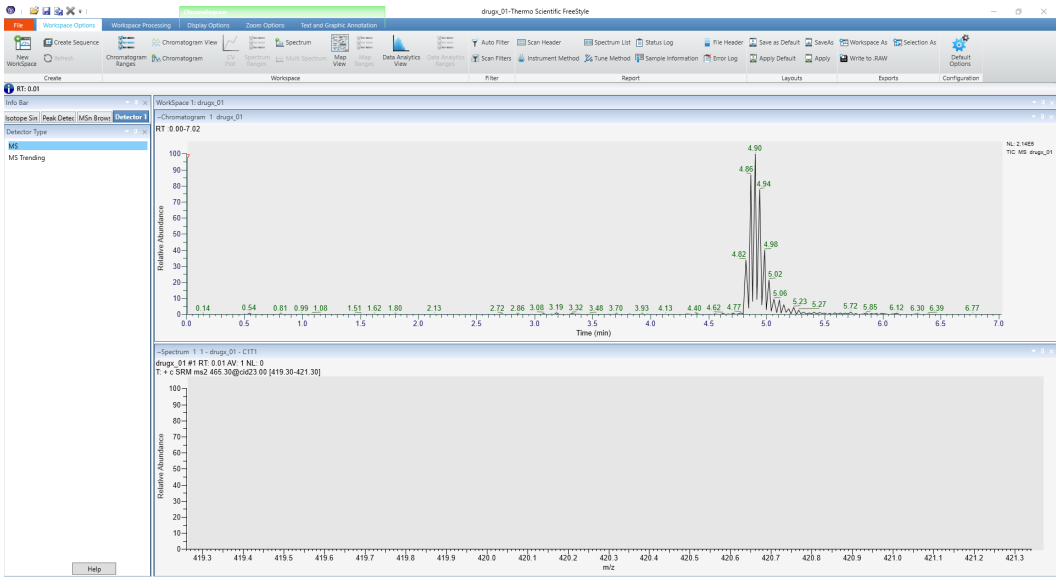
3. In FreeStyle, select the New Workspace icon.
4. Select the raw file(s) you ran in your sequence and click **Open**.

Figure 187. Selecting the Raw File



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

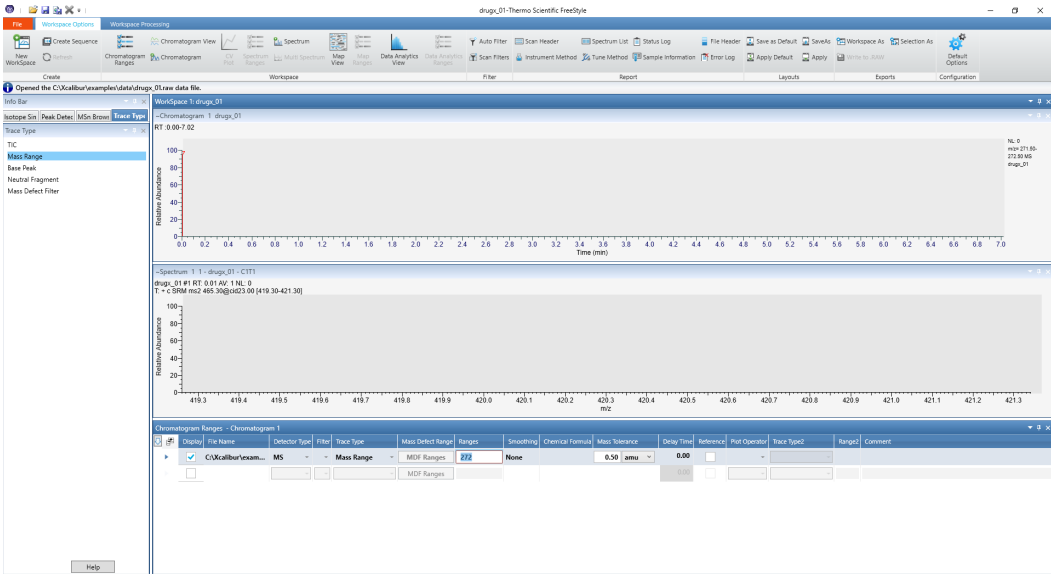
Figure 188. Viewing the Chromatogram



6. Click the Chromatogram Ranges icon in the top menu to open the pane at the bottom on the window.
7. Select Trace Type > Mass Range and enter the mass range for your compound. For OFN, which has a dominant ion at *m/z* 272, select a mass range that contains only this ion. By

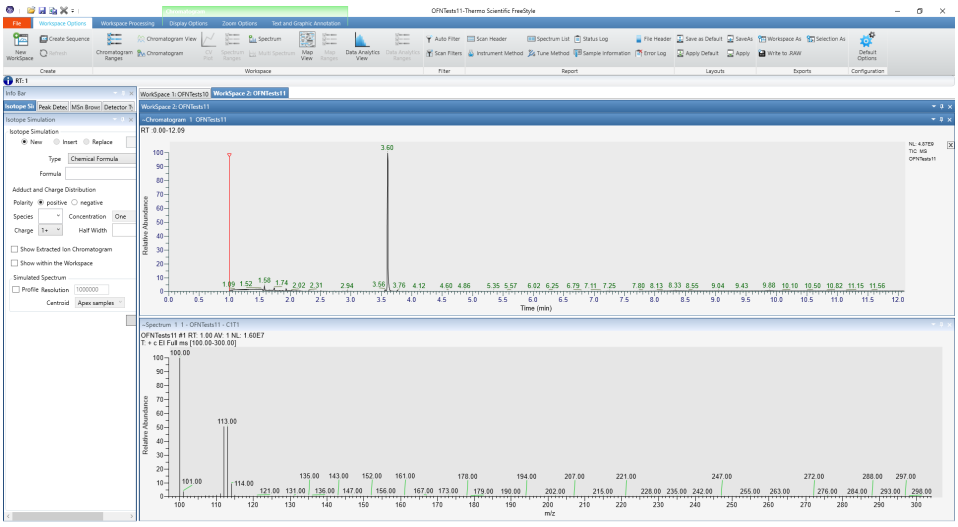
eliminating all the masses that are not generated by your compound, it is much easier to find and quantify the GC peak area.

Figure 189. Selecting the Mass Range



With only the *m/z* 272 ion shown, it is apparent that the OFN is around a retention time of 3.60 minutes.

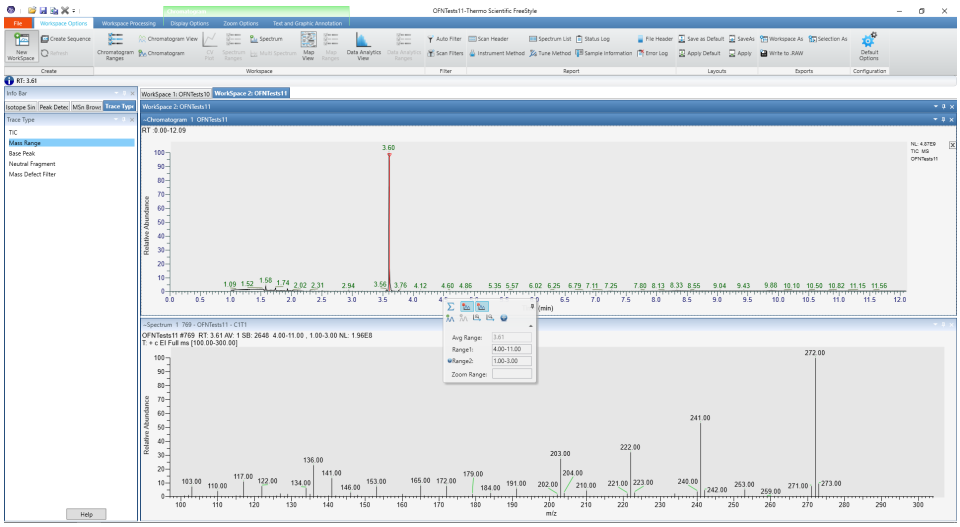
Figure 190. Determining the Retention Times



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 **Chromatogram Ranges** pane and manually enter the time range to display.

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

Figure 191. OFN in Full Scan With Background Subtraction



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

Figure 192. OFN in Full Scan Without Background Subtraction

8. There are many other ways to change the way you data displays in FreeStyle software. See the FreeStyle software online help for details.

8

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

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.

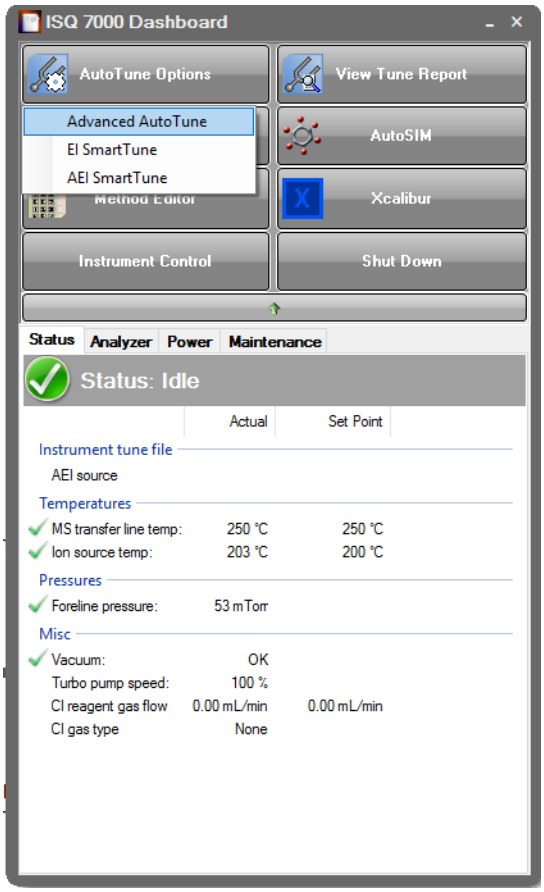
Modifying an Automatic Tune

ISQ 7610 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 in Xcalibur software

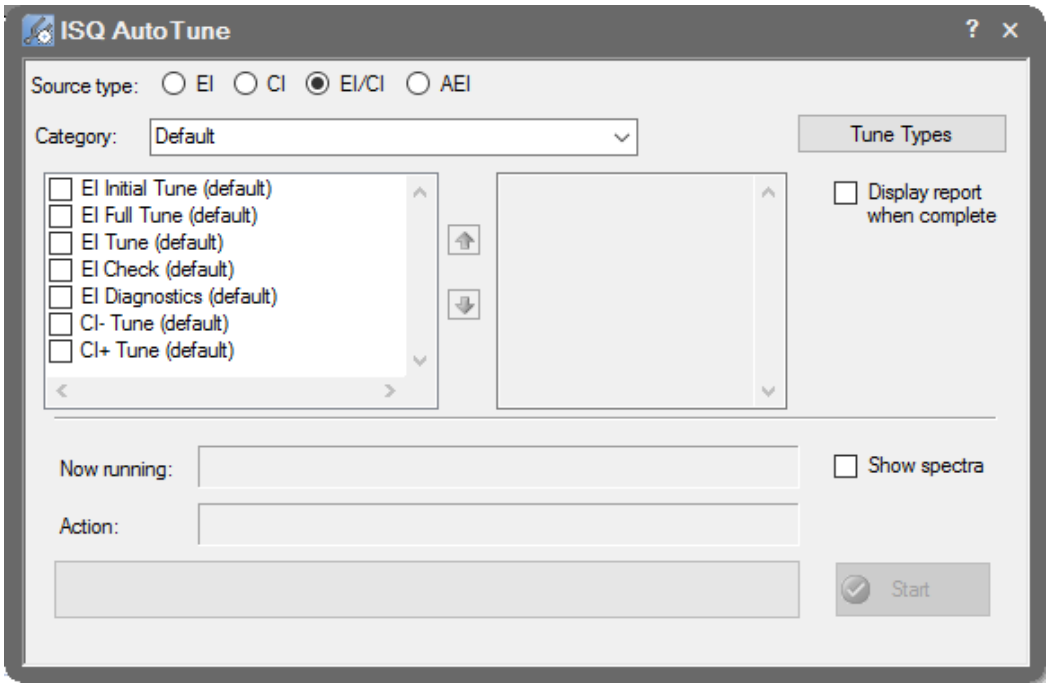
1. Select **AutoTune Options | Advanced AutoTune** on the ISQ 7610 Dashboard. See Figure 193.

   Figure 193. Accessing the Tune Type Editor from the ISQ 7610 Dashboard



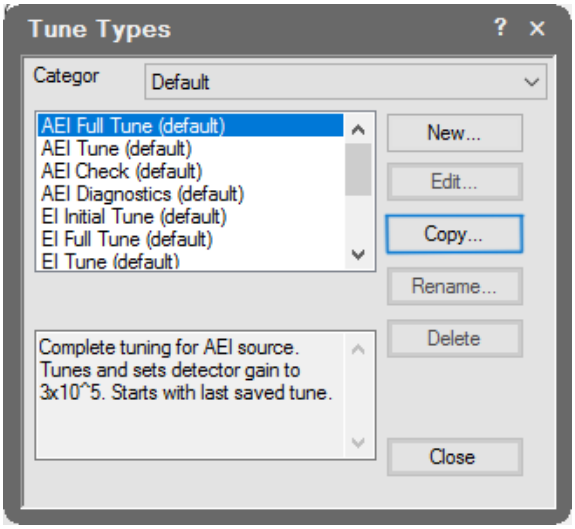
2. ISQ AutoTune opens. Click **Tune Types**. See Figure 194.

Figure 194. Accessing the Tune Type Editor from ISQ 7610 AutoTune



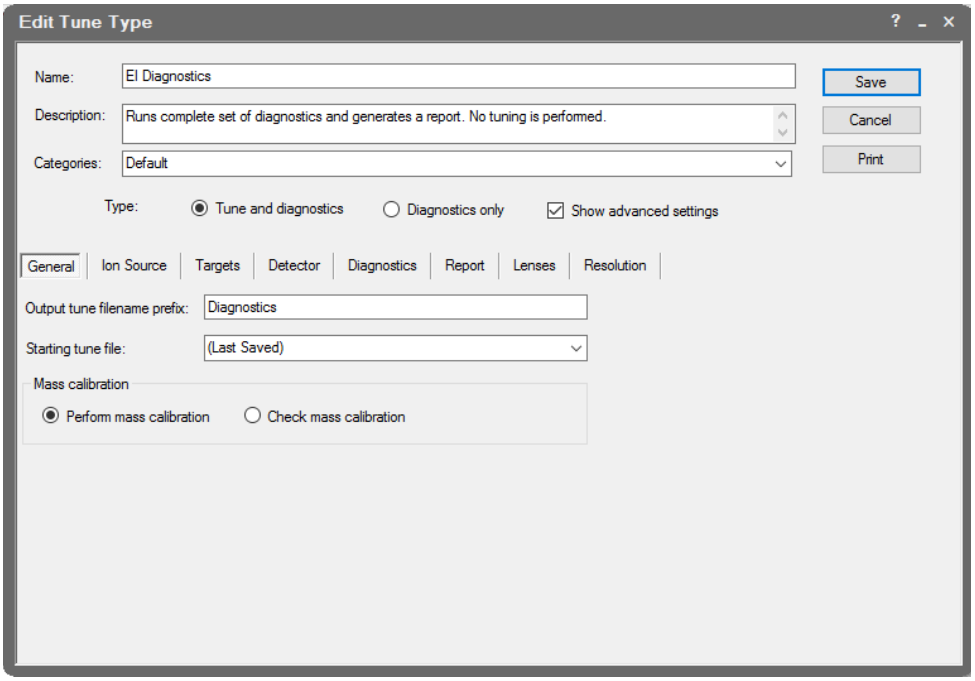
3. In the Tune Types dialog box, select a tune type to edit and click the **Copy** button. See Figure 195.

Figure 195. Using the Tune Types Dialog Box



4. Configure the options under the **General** tab. See Figure 196.

Figure 196. 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.

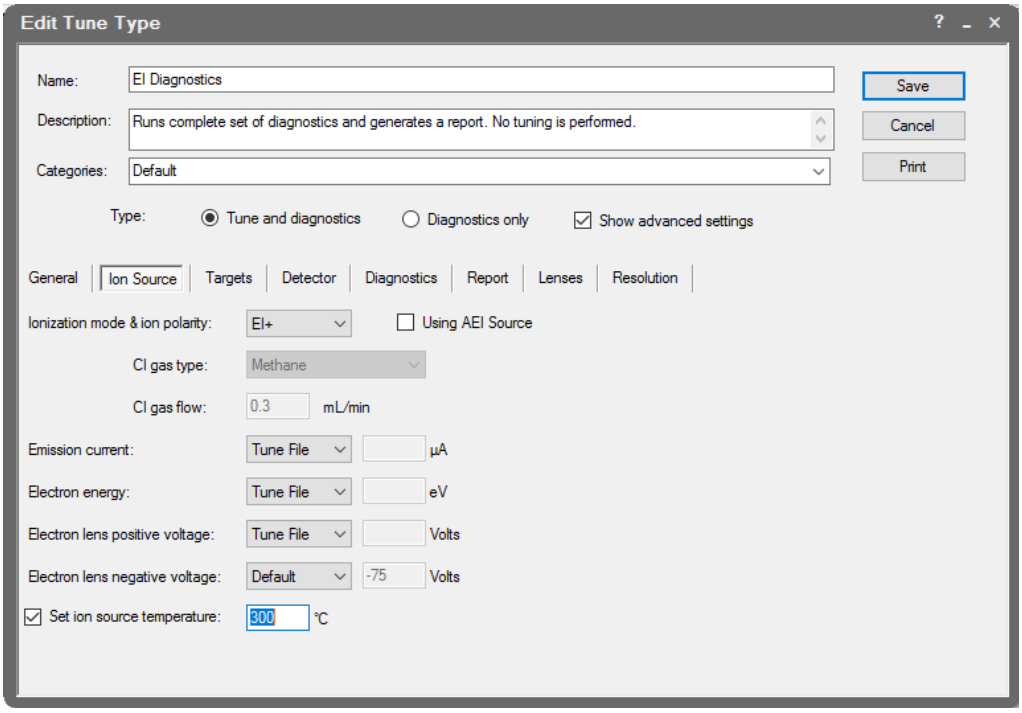
  - Select **Factory** to use a default factory-made file on the instrument that can be used to begin tuning an instrument with a clean ion source.
  - Select **Last Saved** to use a tune file saved on the instrument by the most recent successful automatic tune. A tune file may also be loaded and saved to the instrument for the Last Saved tune file using *ISQ 7610 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 197.

Figure 197. Configuring the Ion Source Options



- **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 7610 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 for EI systems and 10 µA for AEI systems.
  - **Custom**—Select this option if you want to use an emission current other than the default value to increase or decrease the sensitivity of the instrument. Tune with the same value you are planning to use for your analysis. The use of emission currents above 100 µA may lead to the generation of too many ions in the source and a degradation in resolution.

a. **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 eV 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. Changing the electron energy affects the ionization efficiency and fragmentation of the sample. This is typically set to 70 V with the EI ion source because the standard EI libraries are based on 70 eV electron beams.

> **Note** Avoid reducing the electron energy to less than 45 eV in AutoTune. The calibration compound may not be sufficiently ionized for tuning or calibrating at low electron energies.

b. **Electron Lens Positive Voltage**—Allows the electrons to enter the ion volume.

- **Default**—Select this option to use the default electron lens positive voltage, which is 5 V.
- **Tune File**—Select this option to use the value used in the tune file that you selected using the **General** tab.

- **Custom**—Select this option if you do not want to use the default value for the tune. For the electron lens positive voltage, the default is 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 in order for the instrument software to allow the filament to be turned on. The voltage applied to the filament is the same number, but the opposite sign, of the electron energy. The settable range is 0–150 V.

  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.

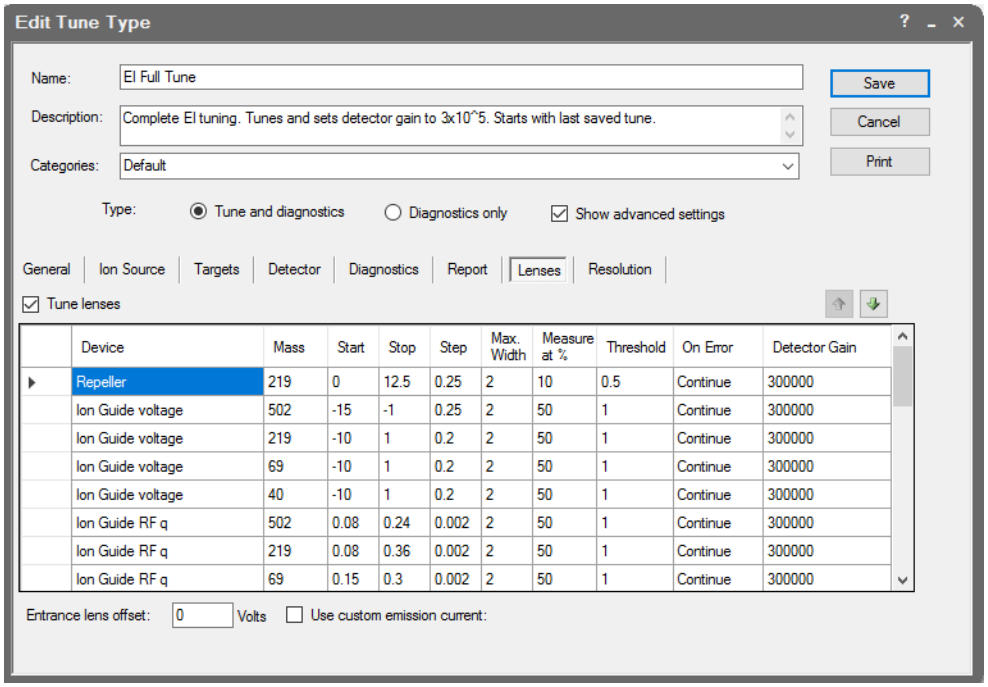
- **Electron Lens Negative Voltage**. This field is used to allow the electrons to enter the ion volume.
  - **Default**—Select this option to use the default electron lens negative voltage, which is -75 V.
  - **Tune File**—Select this option to use the value used in the tune file you selected in the 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 check box 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 198.

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

Figure 198. 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.

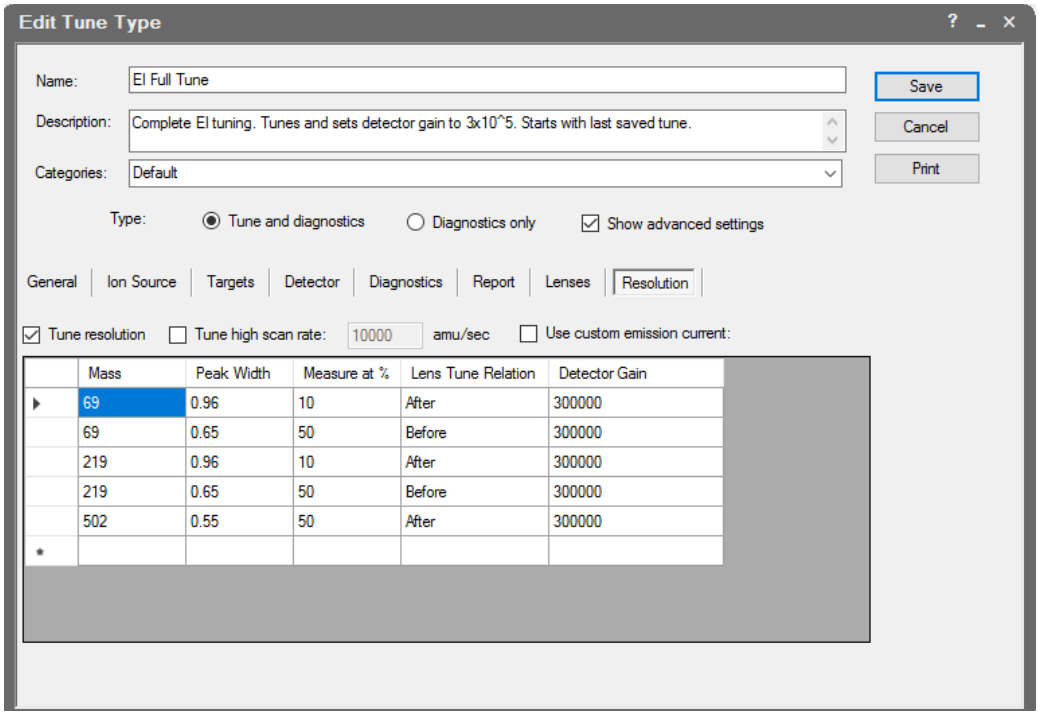
- **On Error**—Use this pull-down menu to select how to handle an error in the tune.
  - **Fail**—Select this option to stop the tune when an error occurs or if no tune points are found meeting the tune criteria.
  - **Continue**—Select this option to allow the tune to continue on to the next device 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 199.

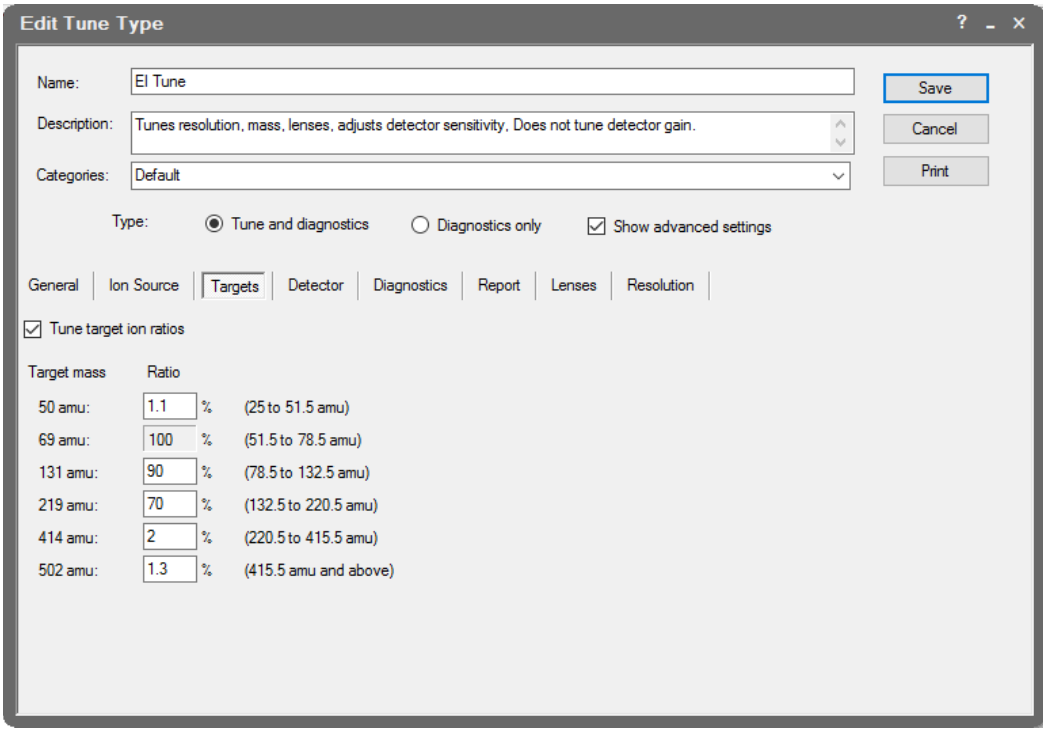
Figure 199. Configuring the Tune Resolution



- **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 7610 instrument tunes to meet regulatory requirements. See Figure 200.

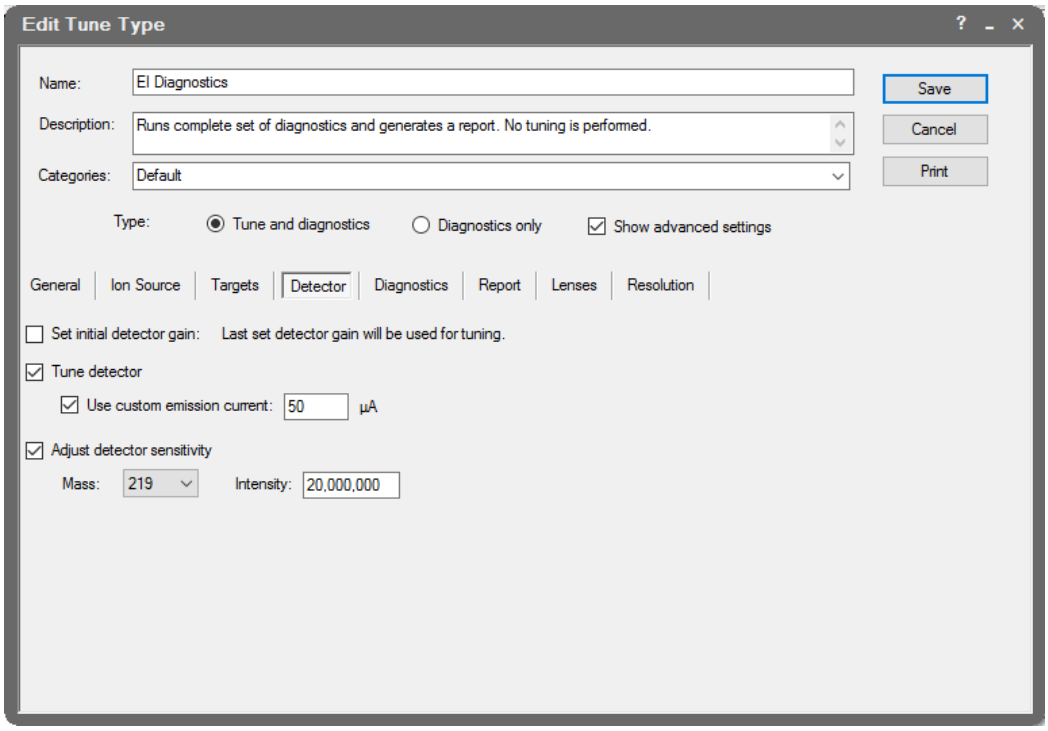
Figure 200. Configuring the Target Ion Ratios



- **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 201.

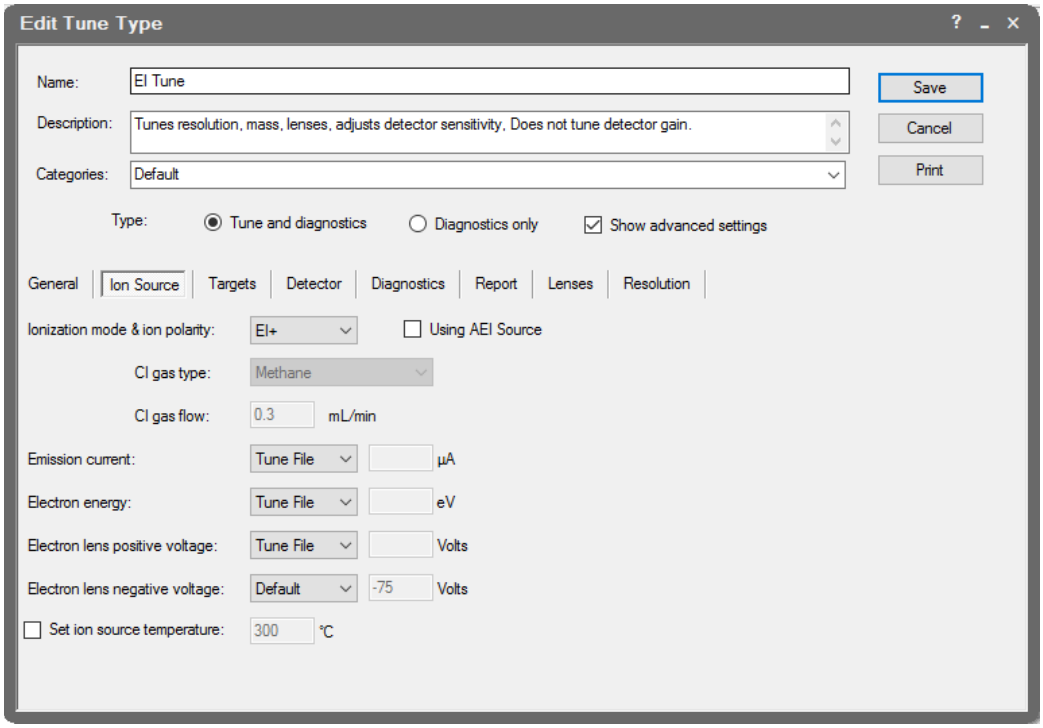
Figure 201. Configuring the Detector Options



- **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 x10^5 and 3x10^5 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.

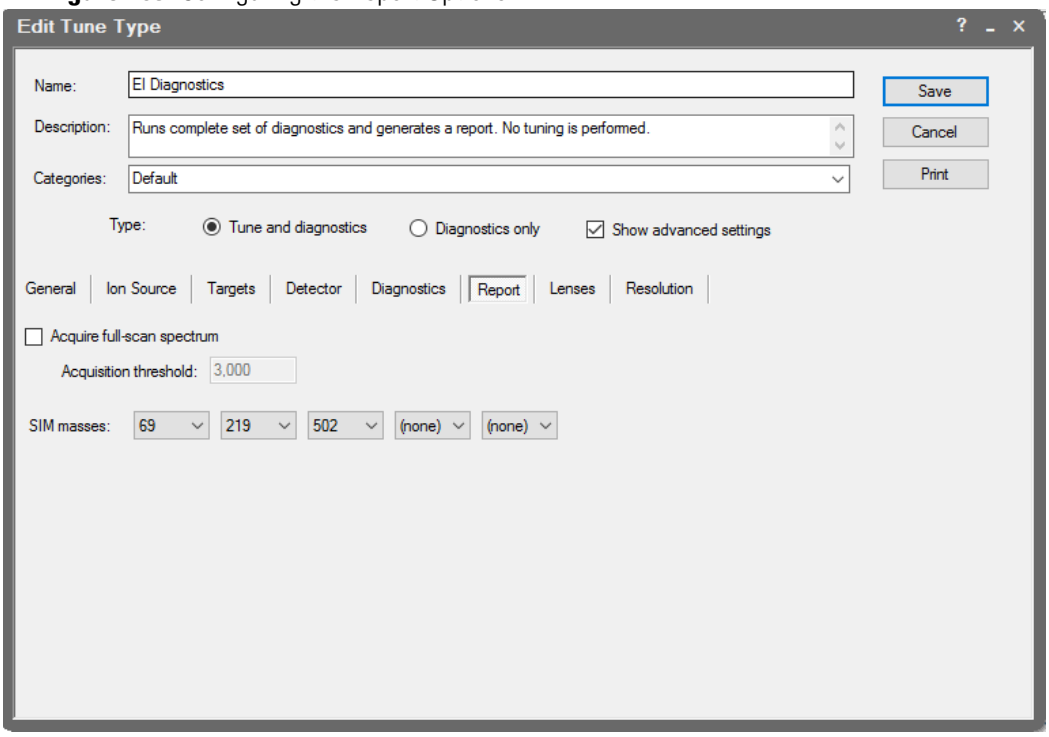
10. Click the **Diagnostics** tab and select a test to confirm the operational ability of the ISQ 7610 system. See Figure 202.

Figure 202. Accessing the ISQ 7610 System Diagnostics



11. Click the **Report** tab configure how you want to view your data. See Figure 203.

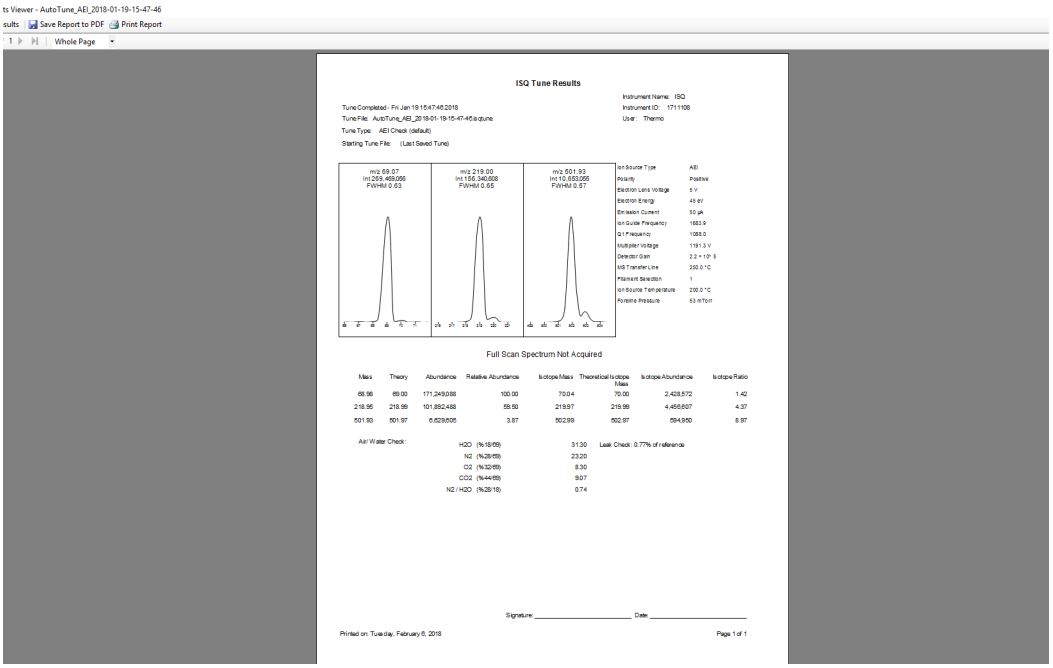
Figure 203. Configuring the Report Options



- **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 204.

Figure 204. Printing the Tune Type Information

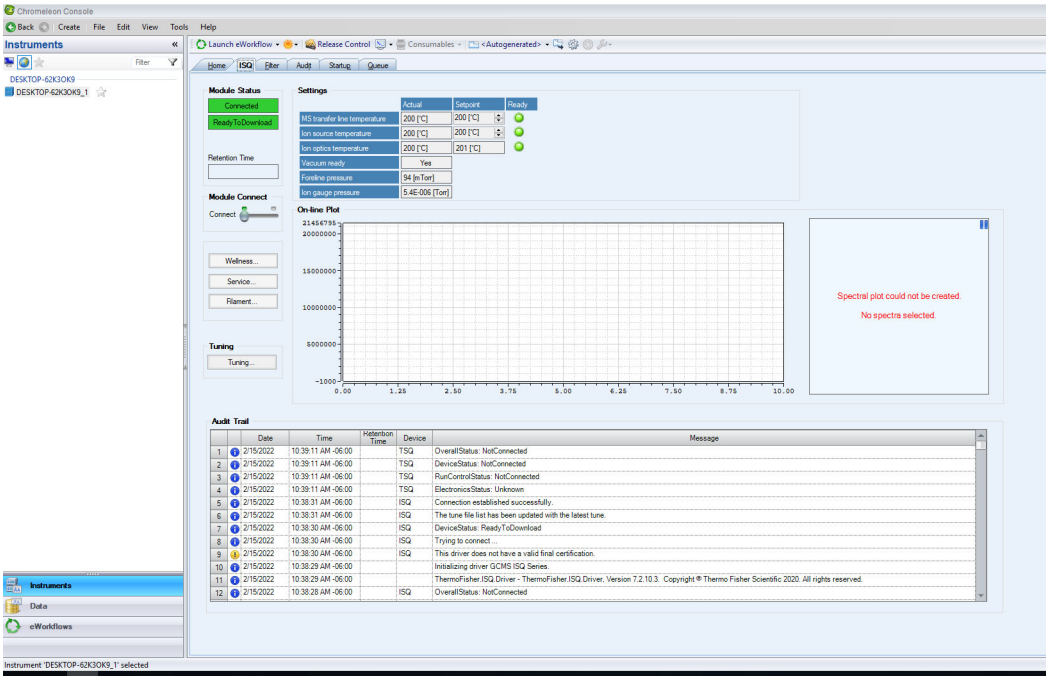


14. Click **Close** to return to the Method Setup window. Now the tune type can be selected during an automatic tune.

❖ To modify an automatic tune in Chromeleon software

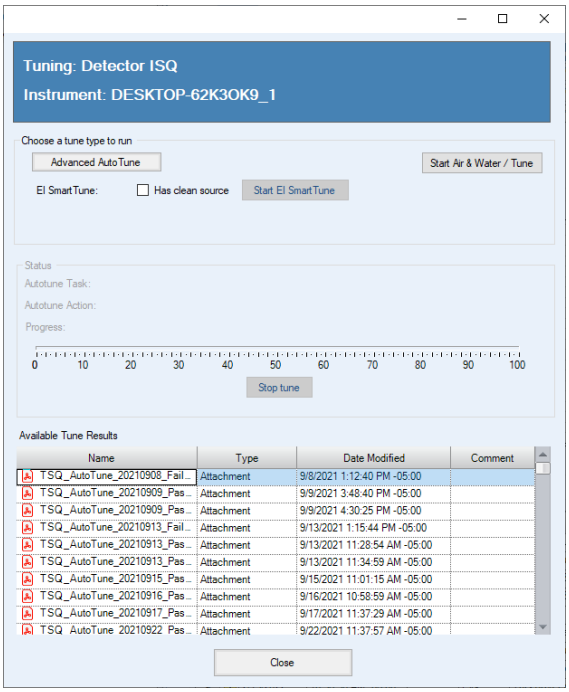
1. From the ISQ epanel, select **Tuning | Advanced AutoTune** on the ISQ 7610 Dashboard. See Figure 205.

Figure 205. Accessing the Tune Type Editor from the ISQ ePanel in Chromeleon Software



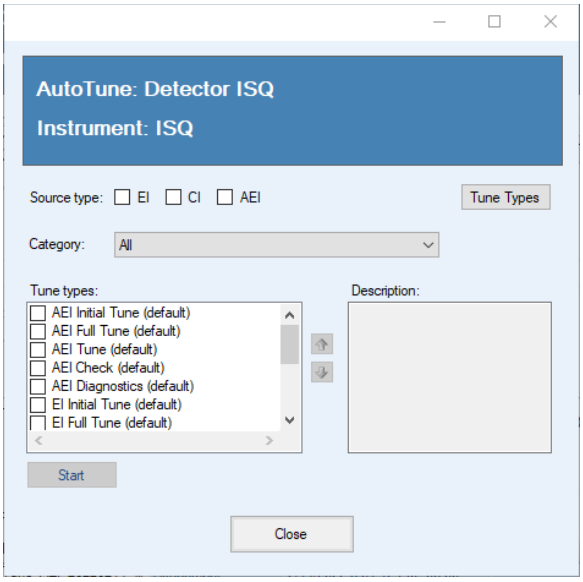
2. The **Tuning: Detector ISQ** window opens. Click **Advanced AutoTune.** See Figure 206.

Figure 206. Accessing the Tune Type Editor in Chromeleon Software



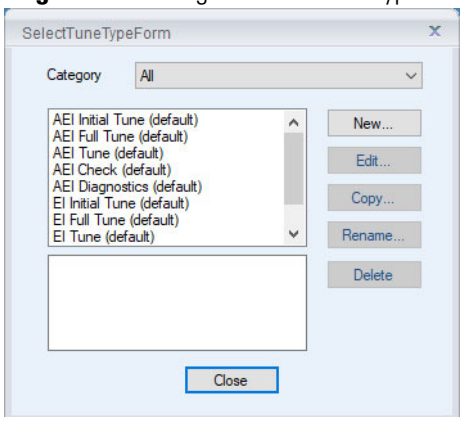
3. The **AutoTune: Detector ISQ** window opens. Click **Tune Types.** See Figure 207.

Figure 207. Accessing the Tune Type Editor from Chromeleon Software



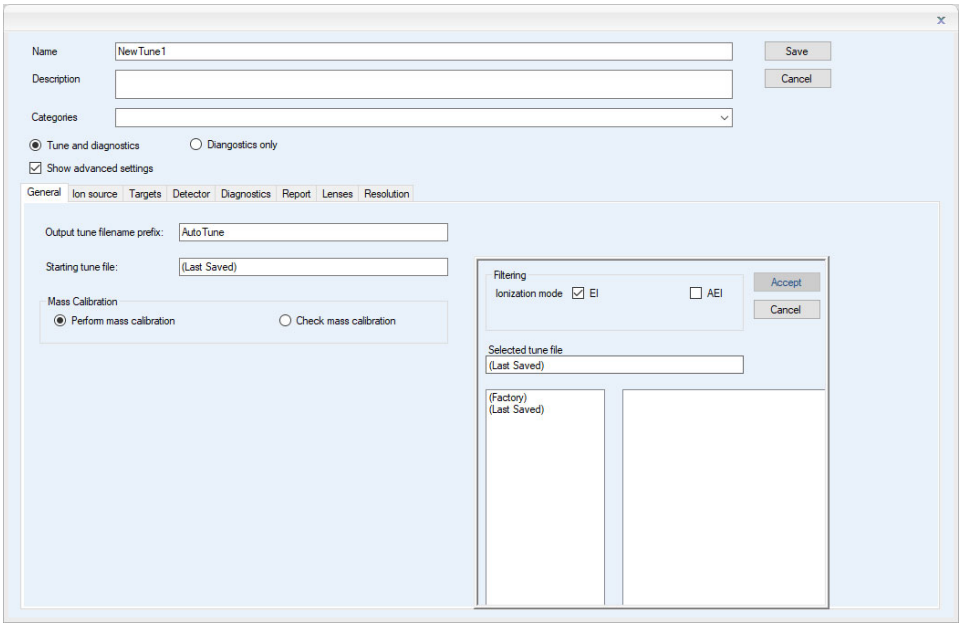
4. In the **SelectTuneTypeForm** dialog box, select a tune type to edit and click the **Copy** button. See Figure 208.

Figure 208. Using the SelectTuneTypeForm Dialog Box



5. Configure the options under the **General** tab. See Figure 209.

Figure 209. 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.

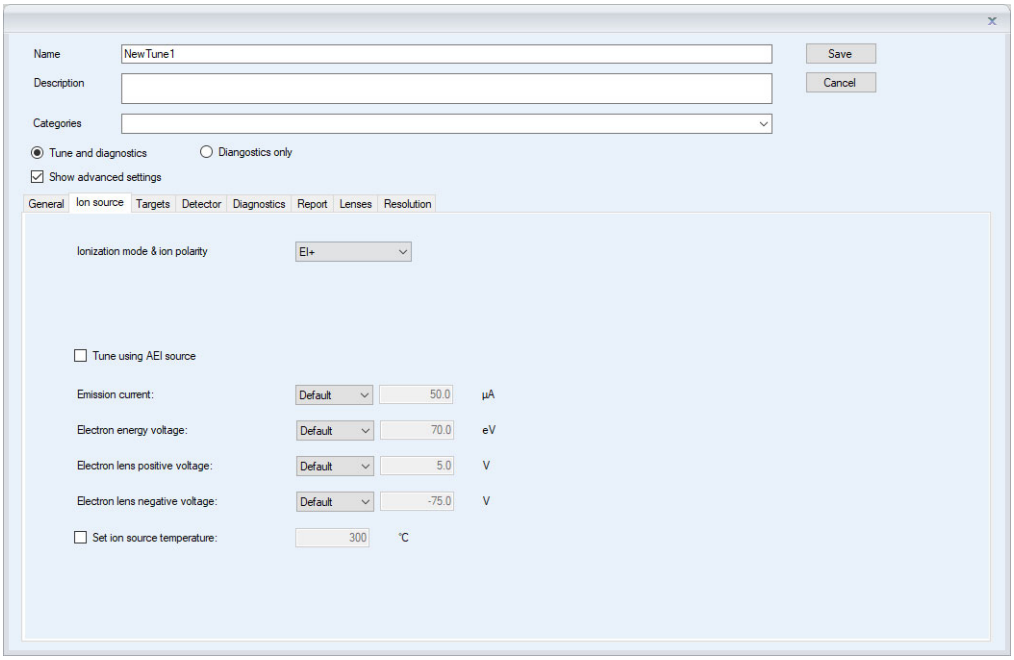
  - Select **Factory** to use a default factory-made file on the instrument that can be used to begin tuning an instrument with a clean ion source.
  - Select **Last Saved** to use a tune file saved on the instrument by the most recent successful automatic tune. A tune file may also be loaded and saved to the instrument for the Last Saved tune file using *ISQ 7610 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.

6. Configure the options under the **Ion Source** tab. See Figure 210.

Figure 210. Configuring the Ion Source Options



- **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 7610 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 for EI systems and 10 µA for AEI systems.

• **Custom**—Select this option if you want to use an emission current other than the default value to increase or decrease the sensitivity of the instrument. Tune with the same value you are planning to use for your analysis. The use of emission currents above 100 µA may lead to the generation of too many ions in the source and a degradation in resolution.

a. **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 eV 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. Changing the electron energy affects the ionization efficiency and fragmentation of the sample. This is typically set to 70 V with the EI ion source because the standard EI libraries are based on 70 eV electron beams.

Note Avoid reducing the electron energy to less than 45 eV in AutoTune. The calibration compound may not be sufficiently ionized for tuning or calibrating at low electron energies.

b. **Electron Lens Positive Voltage**—Allows the electrons to enter the ion volume.

• **Default**—Select this option to use the default electron lens positive voltage, which is 5 V.

• **Tune File**—Select this option to use the value used in the tune file that you selected using the **General** tab.

• **Custom**—Select this option if you do not want to use the default value for the tune. For the electron lens positive voltage, the default is 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 in order for the instrument software to allow the filament to be turned on. The voltage applied to the filament is the same number, but the opposite sign, of the electron energy. The settable range is 0–150 V.

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.

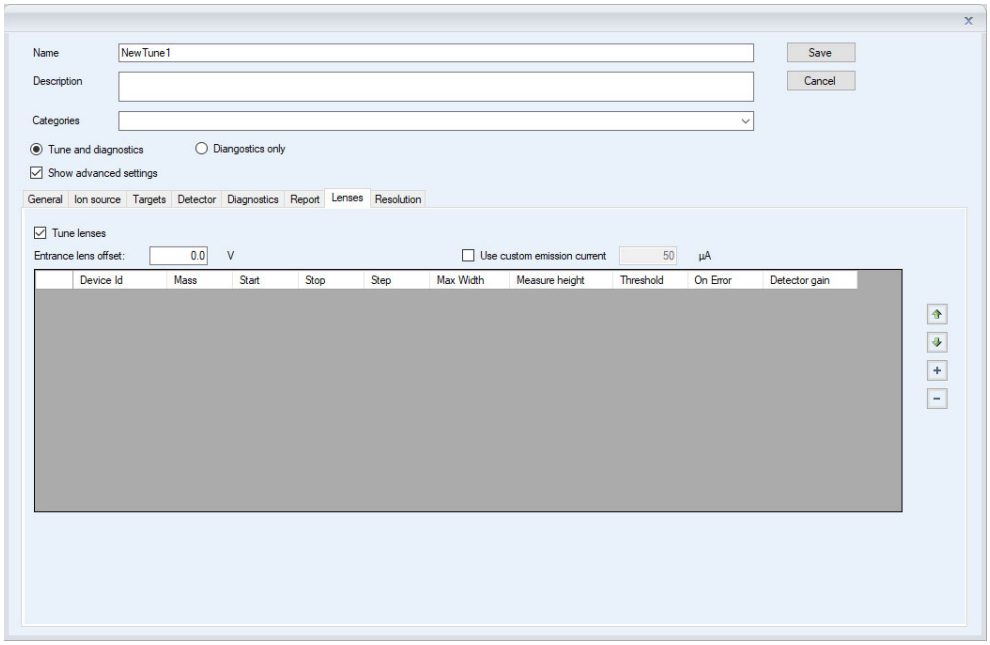
- **Electron Lens Negative Voltage**. This field is used to allow the electrons to enter the ion volume.
  - **Default**—Select this option to use the default electron lens negative voltage, which is -75 V.
  - **Tune File**—Select this option to use the value used in the tune file you selected in the 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 check box 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.

7. 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 211.

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

Figure 211. 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.

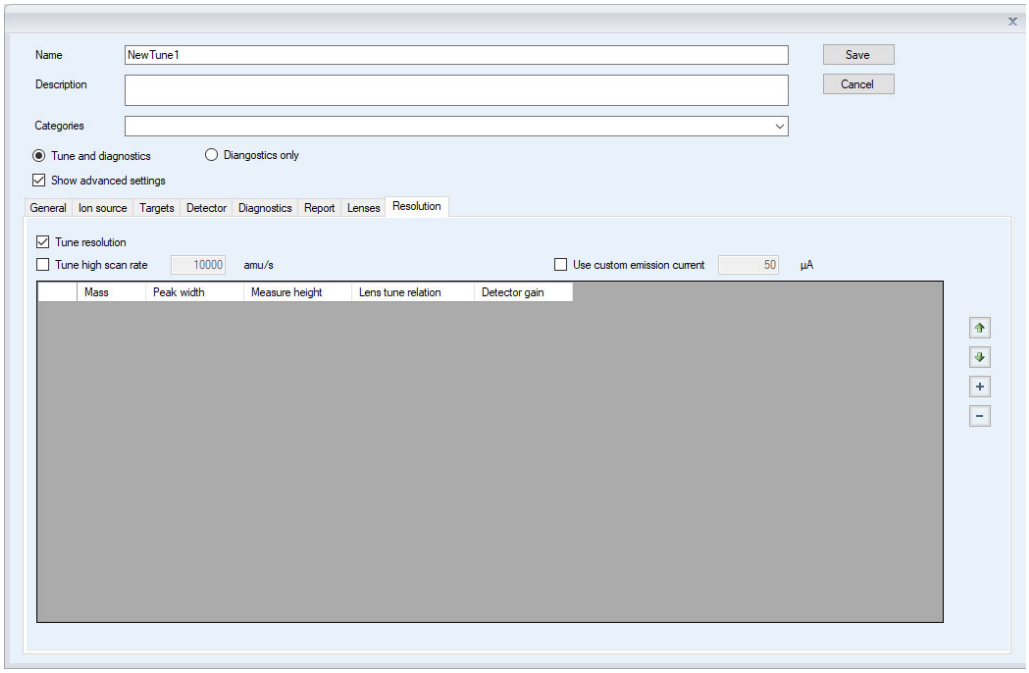
- **On Error**—Use this pull-down menu to select how to handle an error in the tune.
  - **Fail**—Select this option to stop the tune when an error occurs or if no tune points are found meeting the tune criteria.
  - **Continue**—Select this option to allow the tune to continue on to the next device when an error occurs.

8. 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 212.

Figure 212. Configuring the Tune Resolution

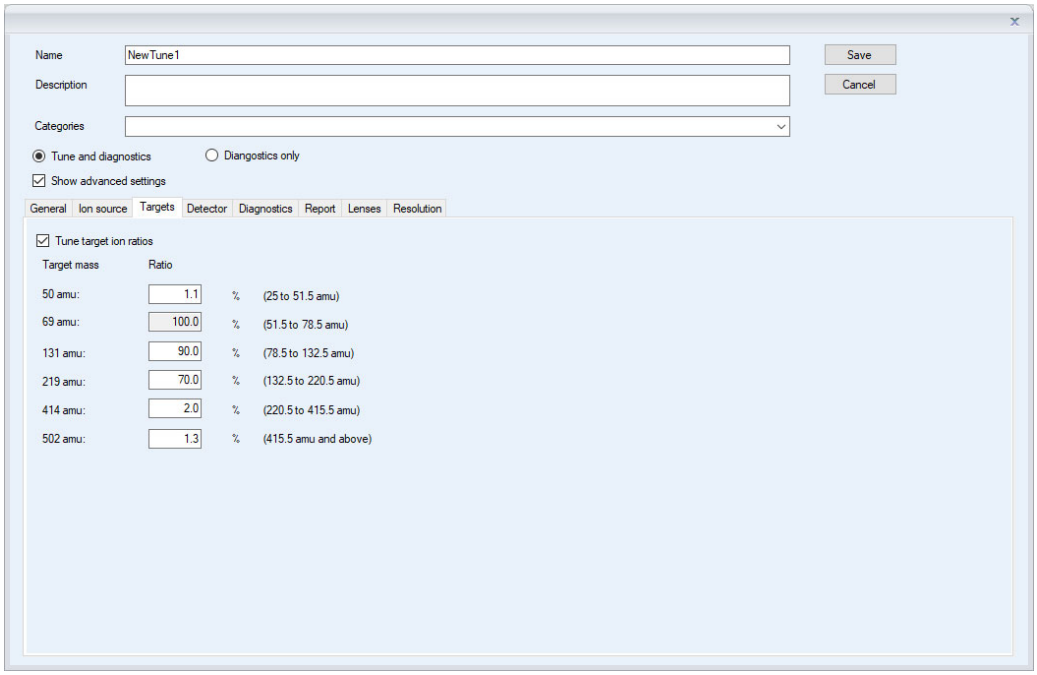


- **Mass**—Use this pull-down menu to select the ion to be used for tuning.

- **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.

9. 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 7610 instrument tunes to meet regulatory requirements. See Figure 213.

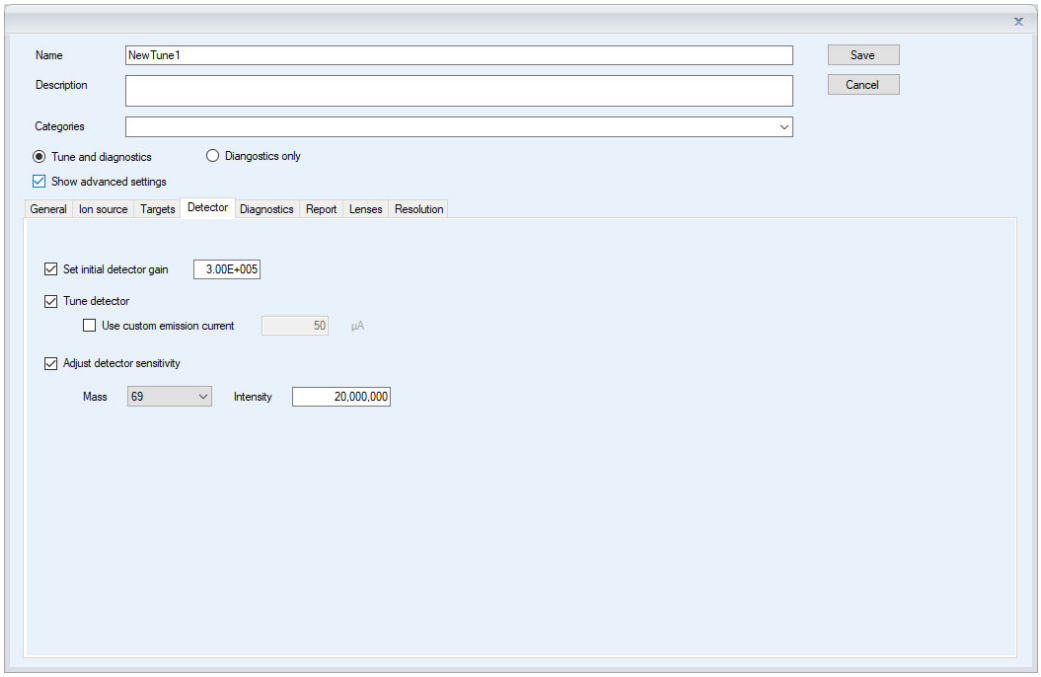
Figure 213. Configuring the Target Ion Ratios



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

10. Configure the options under the **Detector** tab. See Figure 214.

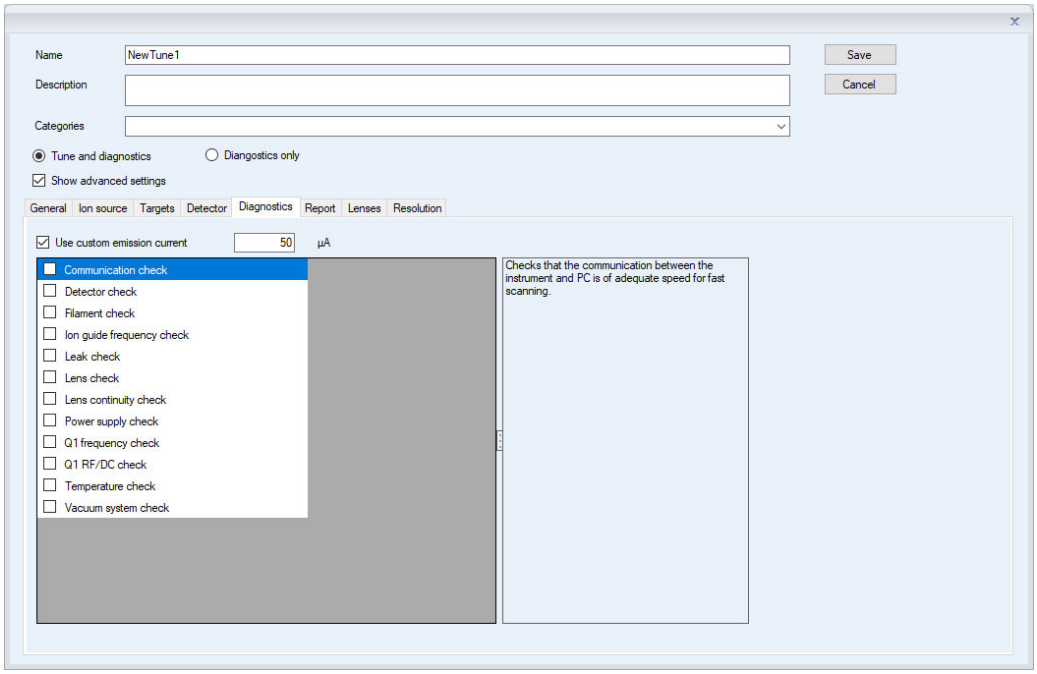
Figure 214. Configuring the Detector Options



- **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 x10^5 and 3x10^5 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.

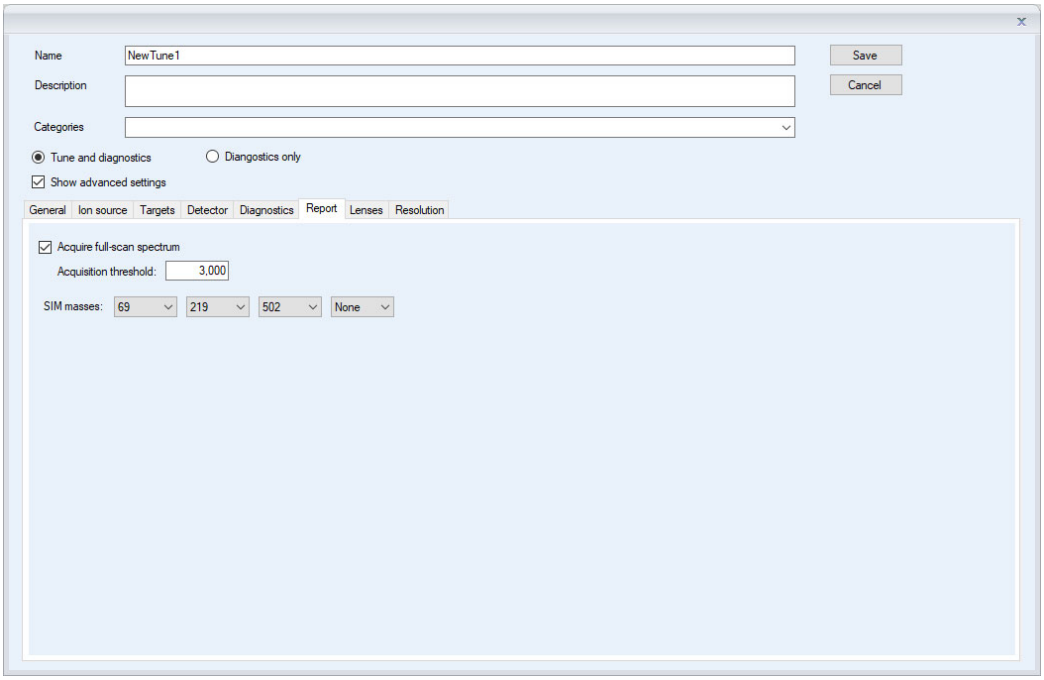
11. Click the **Diagnostics** tab and select a test to confirm the operational ability of the ISQ 7610 system. See Figure 215.

Figure 215. Accessing the ISQ 7610 System Diagnostics



12. Click the **Report** tab configure how you want to view your data. See Figure 216.

Figure 216. Configuring the Report Options

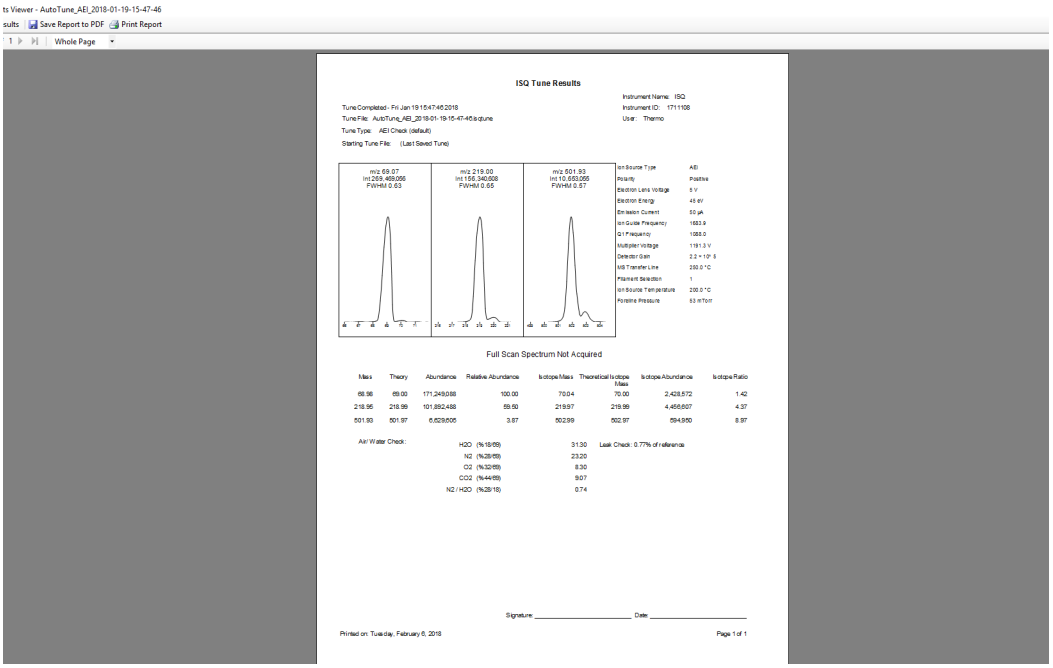


- **Acquire Full-Scan Spectrum**—Select this checkbox to display the full spectrum on your tune report.

- **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.

13. 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.
14. Click **Print** to save your tune type information as a PDF or to print a hard copy of the information. See Figure 217.

Figure 217. Printing the Tune Type Information



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

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

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 7610 Hardware Manual*.

CAUTION ELECTRICAL SHOCK HAZARD: When troubleshooting any issue that requires removing a cover on the ISQ 7610 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 7610 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 7610 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 7610 Hardware Manual* for instructions on cleaning and inserting the ion source cartridge.

Setting Instrument Conditions for Troubleshooting

Before troubleshooting the ISQ 7610 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 7610 Spare Parts Guide* for ordering information.

- Clean the ion source cartridge. See the *ISQ 7610 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 7610 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 7610 system to equilibrate, run an EI diagnostic tune even if you cannot see any ion intensities.

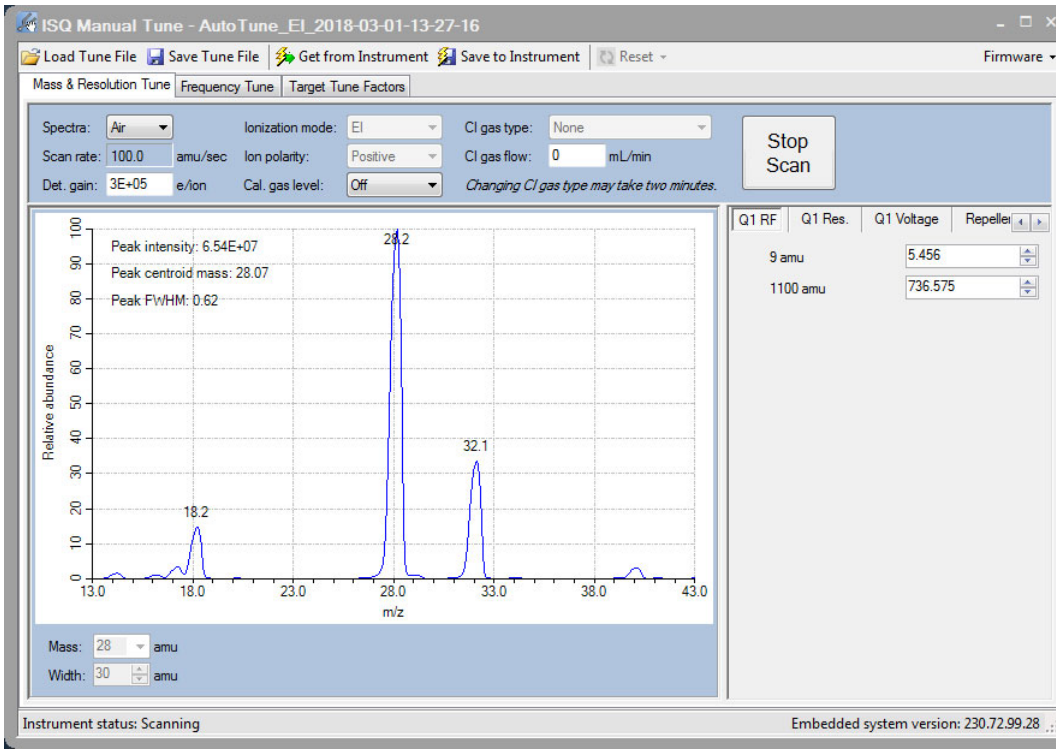
Checking Air/Water Spectra

Before running additional diagnostics, check the air/water spectra of your ISQ 7610 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 7610 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 218.

Figure 218. Typical ISQ 7610 Air/Water Spectrum with Helium Carrier Gas at 1.2mL/min



Note If using the AEI source, the air & water background ions may be too intense to observe without saturating the detector. Do not continue to scan when the detector is saturated or it will reduce detector lifetime. Make note of the detector gain and lower it (you may also find the last tuned value from the most recent tune report). If the intensity is above 1×10^9, decrease the emission current to 5 µA and reduce the detector gain to observe unsaturated ions. Be careful to set the detector gain back to the last tuned value in manual tune before running a sample method that uses last tune detector gain. Manual tune saves the detector gain to the instrument, and it can be used by subsequent methods.

The typical results (with helium carrier gas flow at 1.2mL/min) are:

- Detector Gain = 3×10^5
- Peak Intensity ≥ 3×10^6

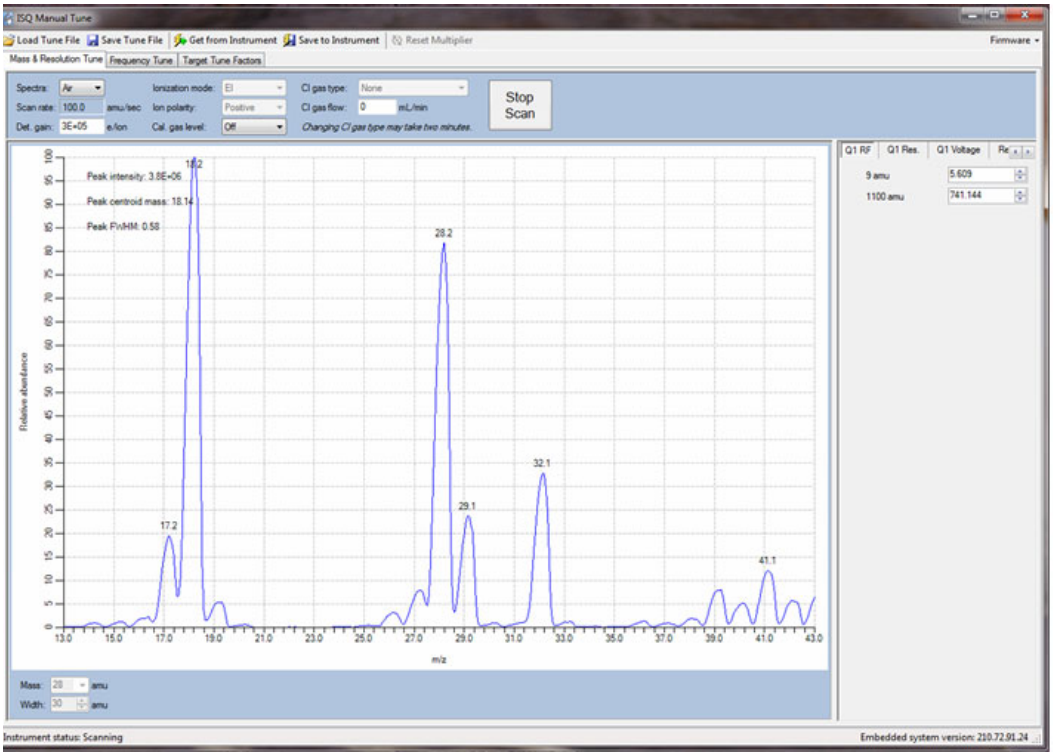
  Note Peak intensity varies depending on variables such as the amount of water and nitrogen in the helium gas supply and the column flow.

- Typical ions are present and in the following ratios:
  - 18 (Water) — Present and < 300% of N_2
  - 28 (N_2) — Reference (base) Peak or more than 33% of Water
  - 32 (O_2) — Present and < 40% of N_2
  - 40 (Argon) — <10% of N_2

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 7610 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 219 for an image of a typical ISQ 7610 air/water spectrum when hydrogen is used as a carrier gas.

Figure 219. Typical ISQ 7610 Air/Water Spectrum for a System with Hydrogen Carrier Gas



The following conditions can cause changes in the air/water spectrum on the ISQ 7610 instrument.

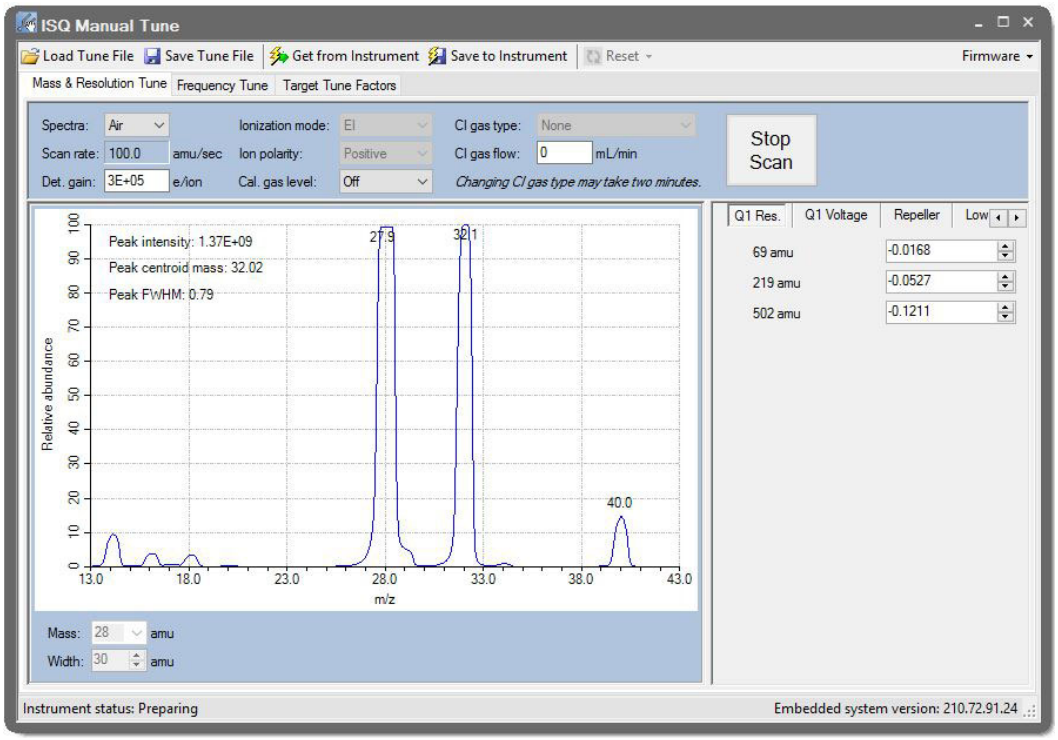
1. Standard detector gain is equal to 3e5, but this can vary depending on customer defined tunes.
2. As the instrument pumps down over time, the ratio of 18/28 will change as *m/z* 18 decreases with *m/z* 28 remaining constant. This eventually changes *m/z* 28 to the base peak.
3. Changing components 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 7610 Hardware Manual*** or "How to Know When Your System Needs Maintenance" on page 228.

The next several images show a typical air/water spectrum for several common problems.

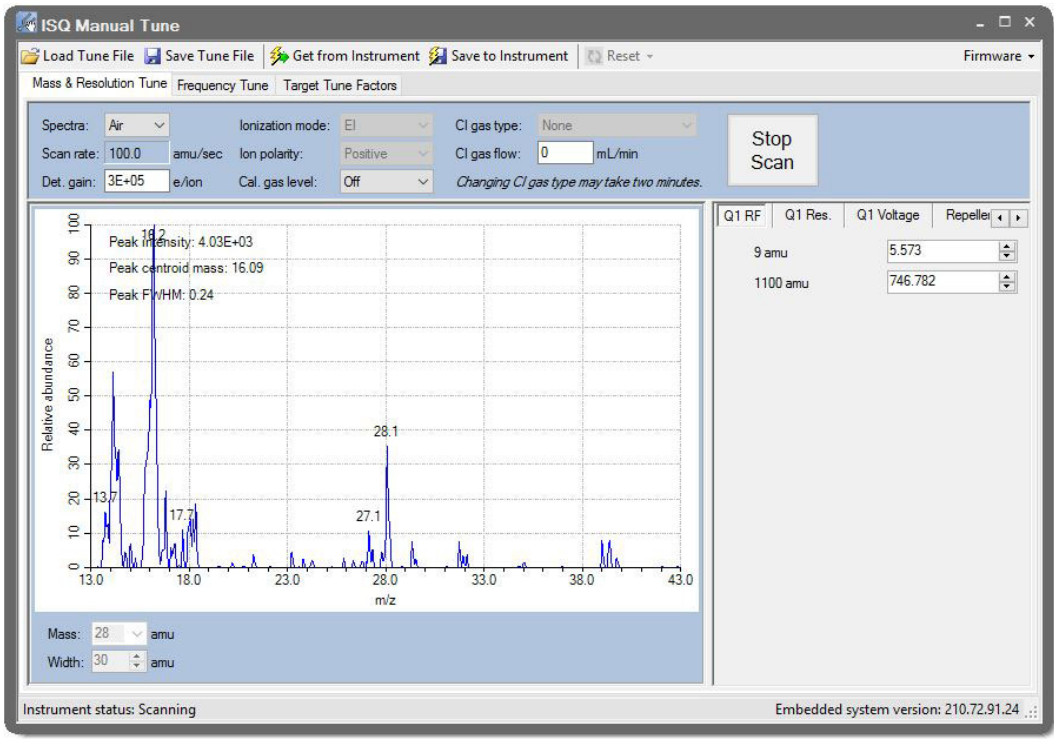
1. Figure 220 is an example of an air/water spectrum of a system with a potential air leak.

Figure 220. Typical Air/Water Spectrum of an ISQ 7610 System with an Air Leak



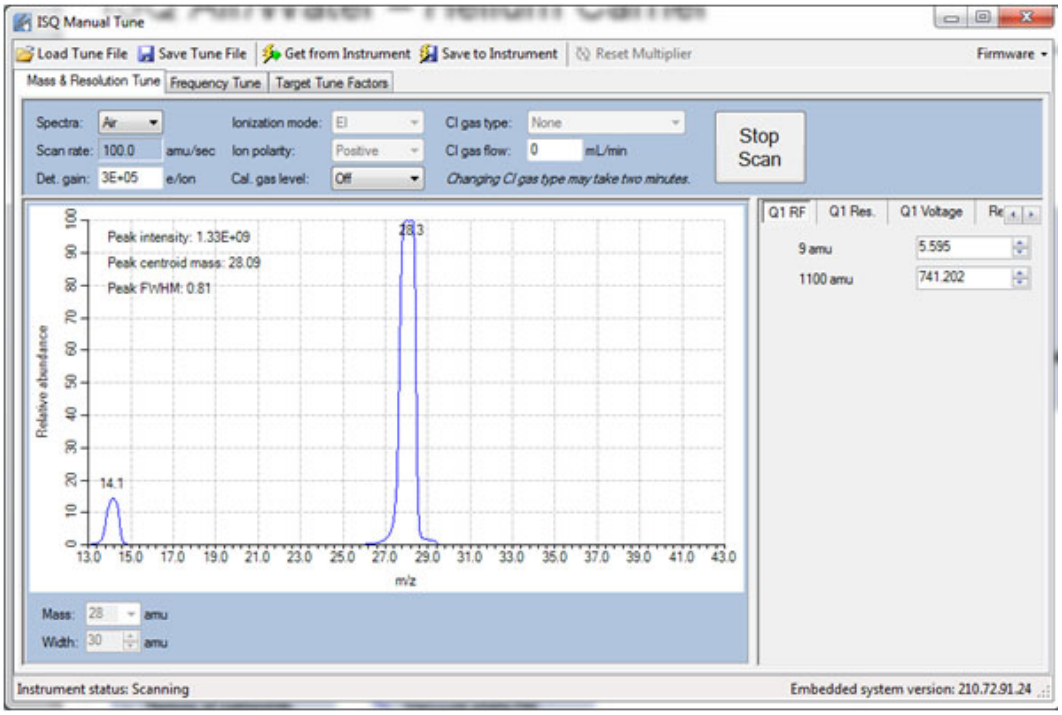
2. Figure 221 is an example of a system with an incorrectly installed ion source.

Figure 221. Typical Air/Water Spectrum of an ISQ 7610 System with an Incorrectly Installed Ion Source Cartridge



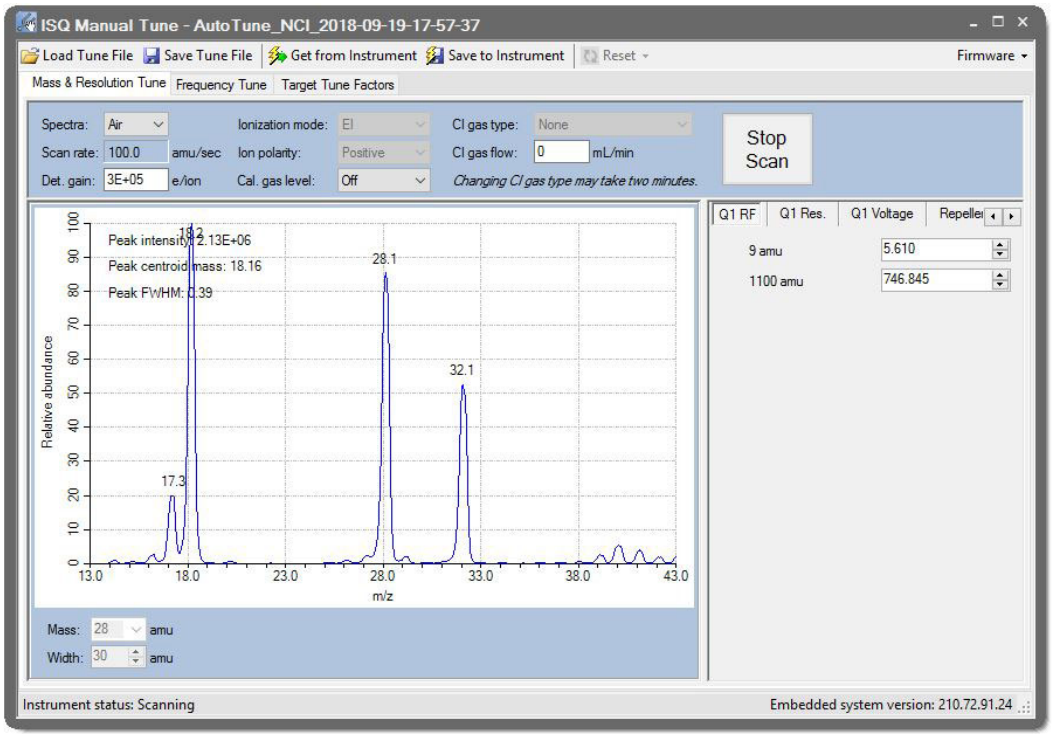
3. Figure 222 is an example of a system with no column flow.

Figure 222. Typical Air/Water Spectrum of an ISQ 7610 System with no Column Flow



4. Figure 223 is an example of a spectrum with a CI tune file run with an EI ion source.

Figure 223. Typical Air/Water Spectrum of an ISQ 7610 System with an EI Ion Source Run with a CI Tune File



No Ions 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.

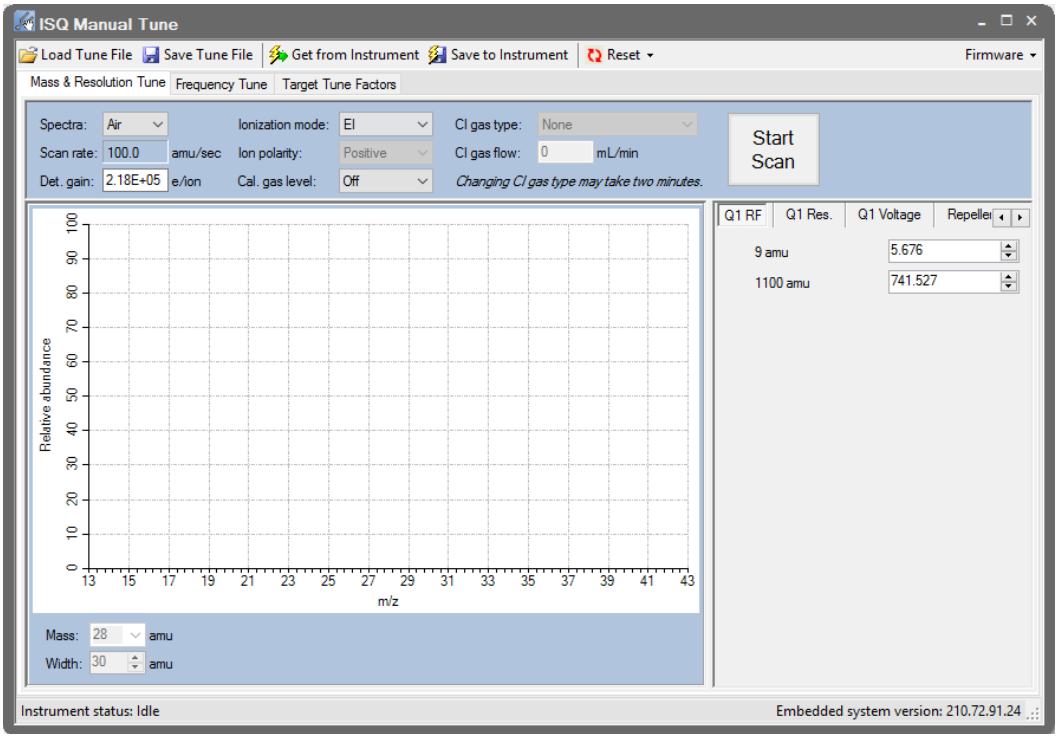
Checking the ISQ 7610 System Firmware

Confirm that you are running the correct version of the ISQ 7610 instrument firmware.

❖ **To find the version of firmware installed on your ISQ 7610 system**

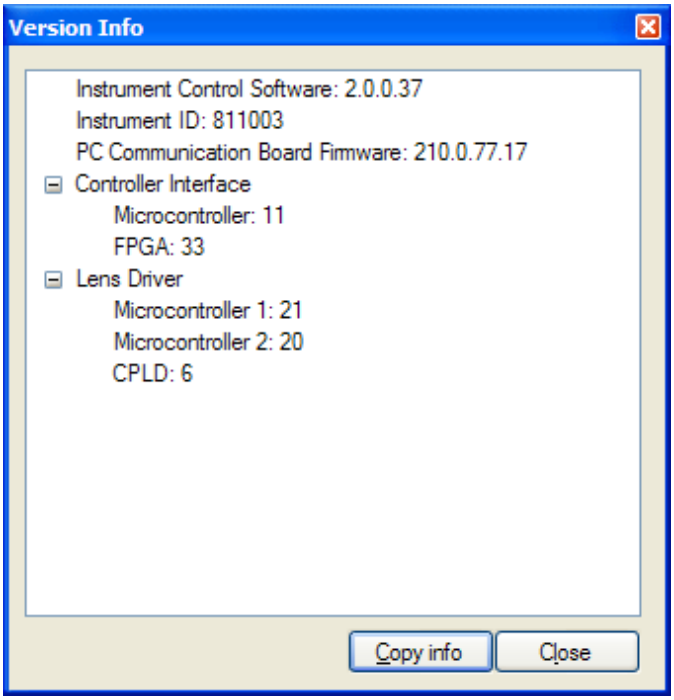
1. In Xcalibur software, on the ISQ 7610 Dashboard, click **Air & Water/Tune**. From the ISQ ePanel in Chromeleon software, click **Tuning | Start Air & Water/Tune.** This opens the Manual Tune application.

Figure 224. The ISQ Manual Tune Application



2. Click **Firmware** in the upper right-hand corner of the home screen in ISQ 7610 Manual Tune.
3. A new menu opens. Click **Version Info**.
4. A dialog box containing opens that lists the firmware versions for the ISQ 7610 instrument control drivers, the lens driver board, and the controller interface board. See Figure 225.

Figure 225. Determining the Firmware Versions



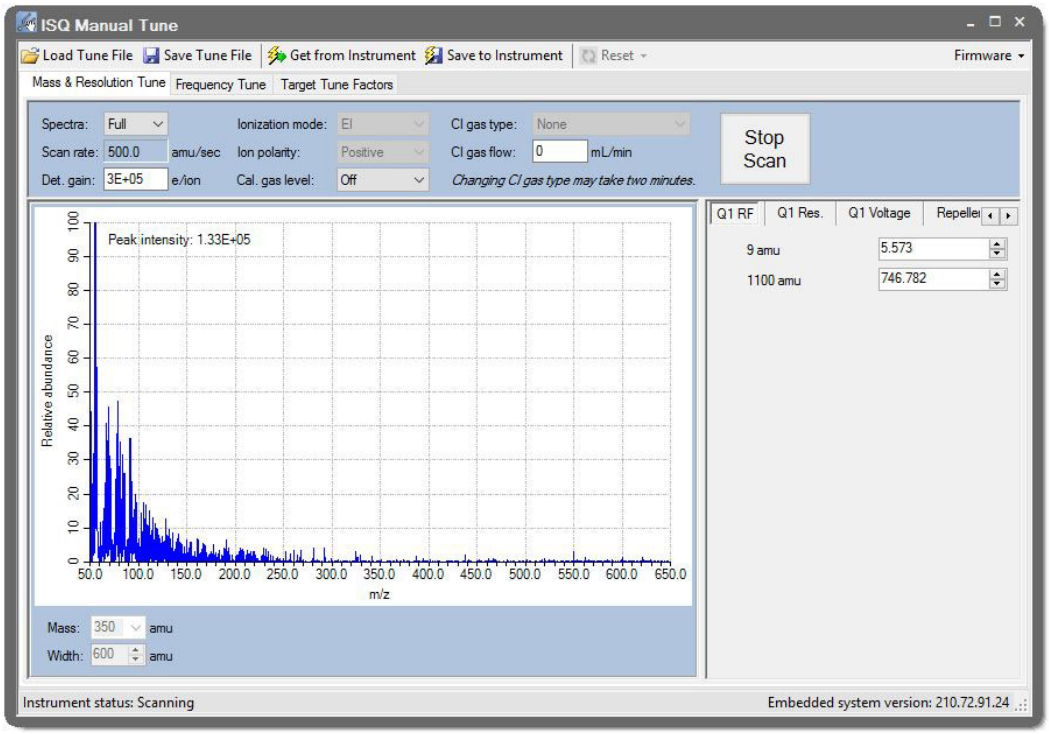
Note Depending on the version of ISQ Series instrument control software you are running, the controller interface board and lens driver board firmware may not be listed.

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 226 shows a typical full scan background spectrum on an ISQ 7610 system.

Figure 226. Typical Full Scan Background Spectrum of an ISQ 7610 System



A typical clean system meets the following:

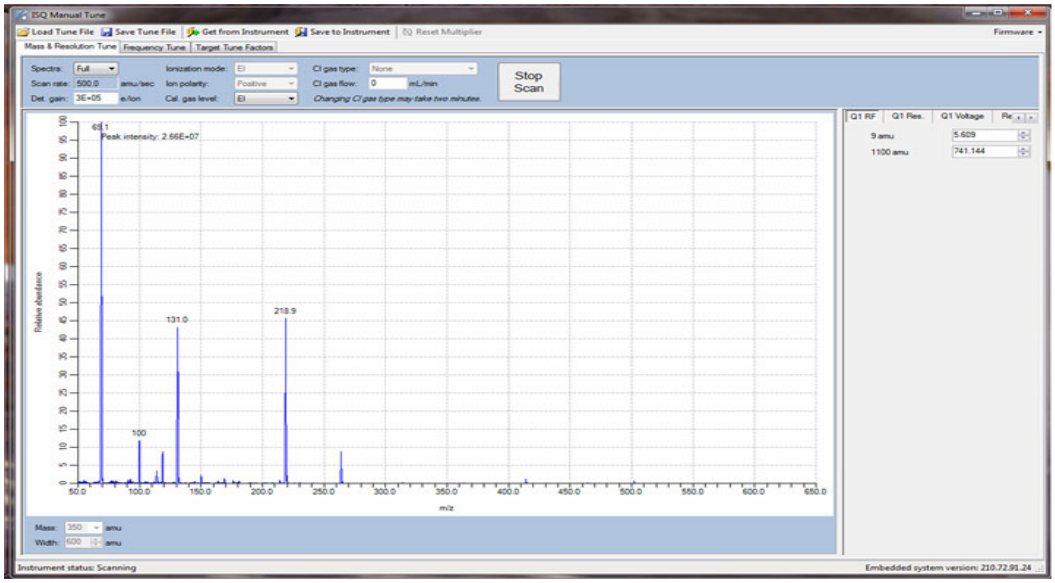
- Detector gain — 3×10^5
- Maximum intensity — $< 1 \times 10^6$
- There should be exponential decay for background noise.

> **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 227 below.

Figure 227. Typical Calibration Gas Profile Spectrum



A typical calibration gas profile meets the conditions below.

- Intensity > 8×10^6

Note The intensity will change with detector gain and SmartTune will adjust the gain to adjust intensity. To determine if the intensity is acceptable, compare the intensity to archived tune reports for the instrument when the ion source was clean.

- 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 30-100%
  - *m/z* 414 and *m/z* 502 between 1-10%

Note High mass ions decrease in relative abundance when ion source temperature increases. The spectrum above was acquired with the ion source temperature at 200 °C and with a clean ion volume.

  - *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:

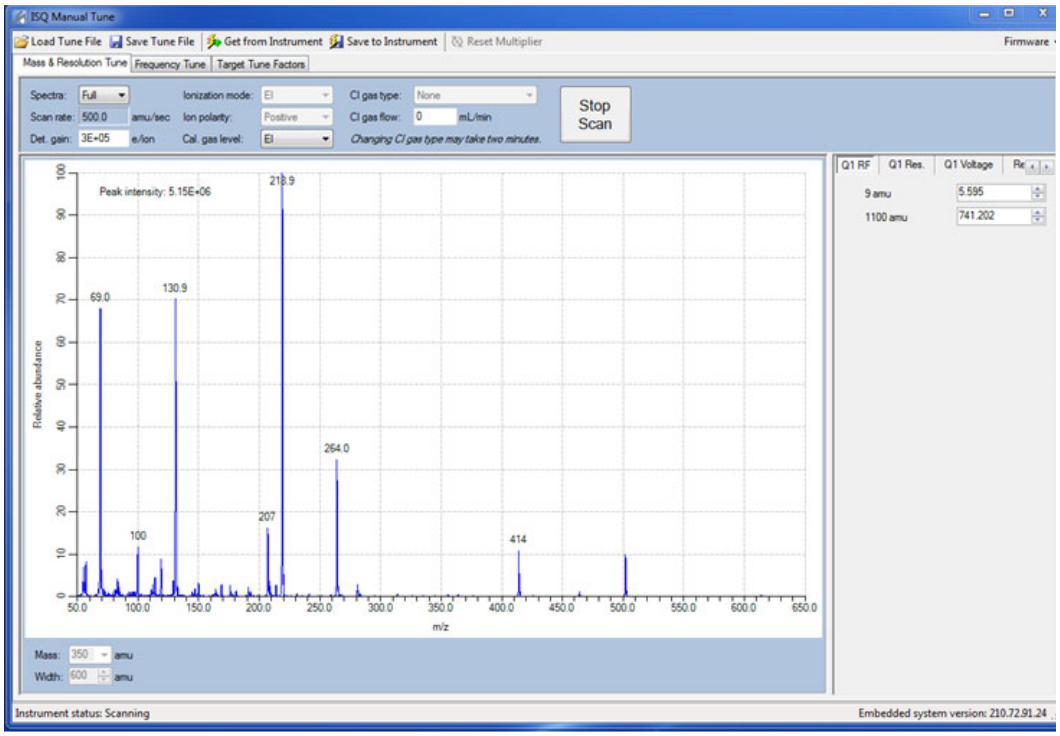
- **Contamination**—If you notice excessive background in your mass spectra, it is usually an indication that your instrument is contaminated. Use the mass spectrum in the table below to understand the origin of the contamination. If you notice cleaning solvent peaks, it is usually an indication that 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.

Table 3. Common Contaminants

Ions (*m/z*) 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 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

Figure 228 shows an atypical PFTBA profile spectrum.

Figure 228. Atypical Calibration Gas Spectrum



In Figure 228, the atypical results are as follows:

- Maximum intensity < 8×10^6 (8,000,0000)
- *m/z* 207 from column bleed is prominent relative to PFTBA ions, indicating low calibration gas levels
- 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.

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

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

Investigating Peak Issues

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.

Table 5. Troubleshooting Peak Issues in Your Data, continued (Sheet 2 of 3)

Behavior	Characteristic	Cause	Remedy
Tailing	Sample peaks	Column degradation is causing activity	Inject a test mixture and evaluate the column. Replace column if necessary.
		Liner is dirty	Clean or replace the liner.
		Ion source is dirty	Clean the ion source. 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 3 of 3)

Behavior	Characteristic	Cause	Remedy
Ghost Peaks	General	Incomplete elution of previous sample	Increase the final oven program temperature or total run time. Increase the column flow rate.
		Carrier gas is contaminated	Replace the gas cylinder or filter.
		Laboratory glassware has caused contamination	Ensure the glassware is clean and contaminant-free.
		Injected sample has decomposed	Decrease the injection port temperature. Use the on-column injection technique.
		Injection solution has matrix present	Adequately clean up your sample prior to injection.
		Inlet or pneumatics are contaminated	Remove the column and bake out the inlet. Use a high-quality septum. Replace the split vent filter. Install an in-line filter between the pneumatics and the inlet.
Missing Peaks	Baseline or background present	Column is broken	Replace the column.
		Column flow is incorrect	Make sure the septa are sealing. Make sure vacuum compensation is turned on.
		Backflush settings are incorrect	Set backflush to **off** 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 7610 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 **Reset** button on the ISQ 7610 instrument, wait ten seconds, close Instrument Configuration.

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.

Table 6. Troubleshooting Results Issues in Your Data, continued (Sheet 2 of 2)

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.

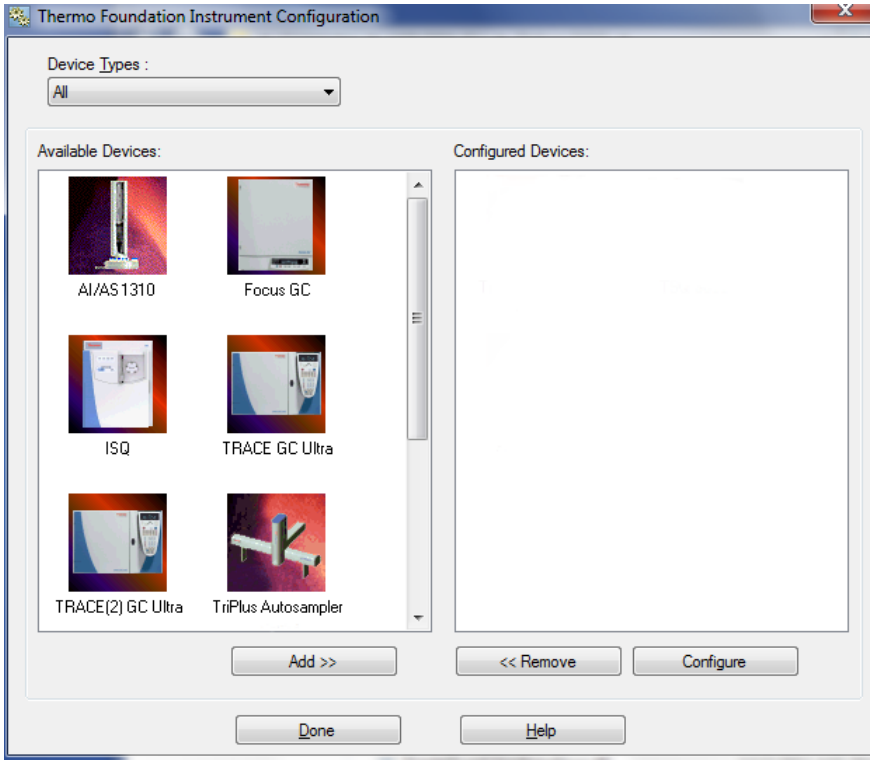
Reconfiguring Your Instrument

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 7610 instrument.

❖ To reconfigure your ISQ 7610 instrument

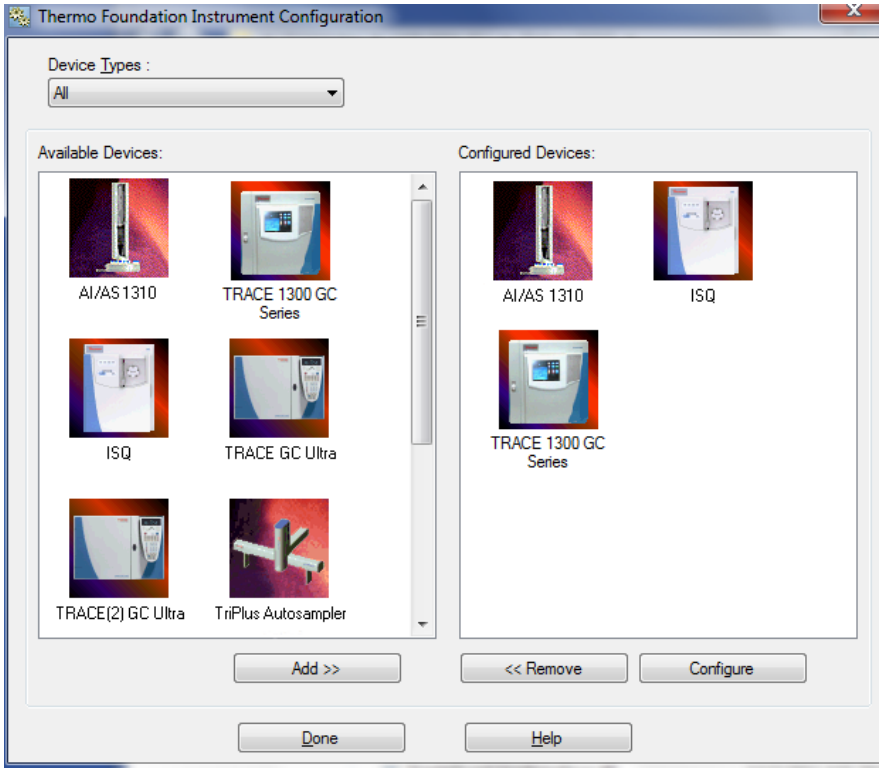
1. If you are running Xcalibur software, from the Start menu on your computer desktop, browse to **Thermo Foundation 3.1 | Instrument Configuration**. When the Instrument Configuration utility opens, you can see an icon of the instruments you have connected.
2. Click the ISQ 7610 (and other instruments) icon in the Available Devices column and click **Add** to move it into the Configured Devices column.

Figure 229. Finding Available Devices



3. Click the each instrument icon you want to configure and click **Add**.

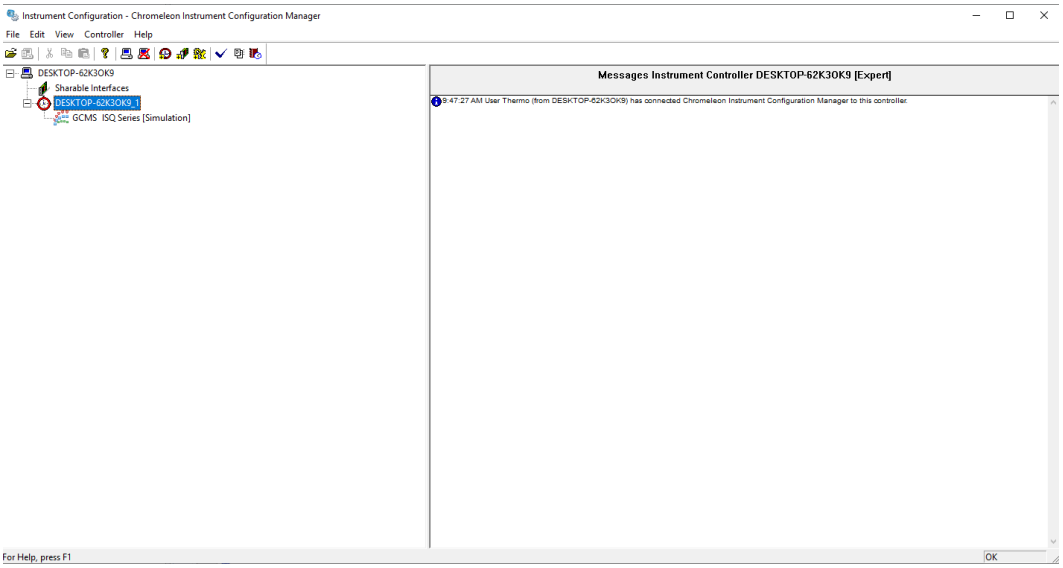
Figure 230. Adding a a Device to be Configured



4. Click **Configure**.

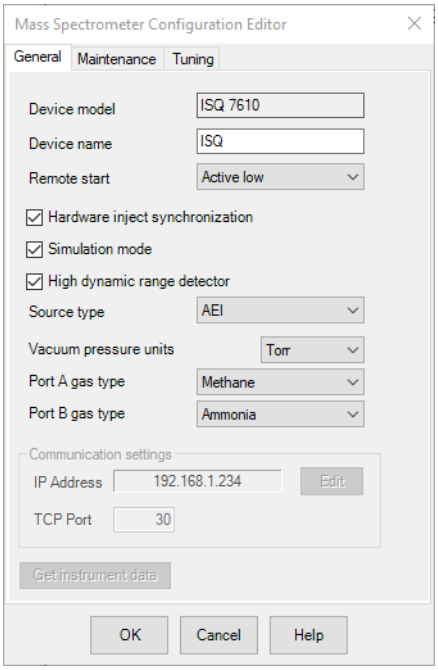
5. If you are running Chromeleon software on your system, click the Chromeleon Instrument Configuration Manager icon on your desktop to open the Chromeleon instrument configuration manager. See Figure 231.

Figure 231. Opening the Chromeleon Instrument Configuration Manager



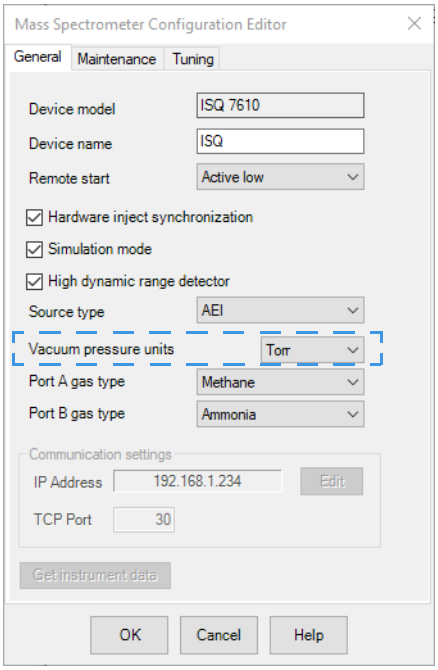
6. Click the instrument name in left pane to open the Mass Spectrometer Configuration Editor. See Figure 232.

Figure 232. Viewing the Mass Spectrometer Configuration Editor



7. In the **General** tab, set the vacuum pressure units.

Figure 233. Setting the Pressure Unites

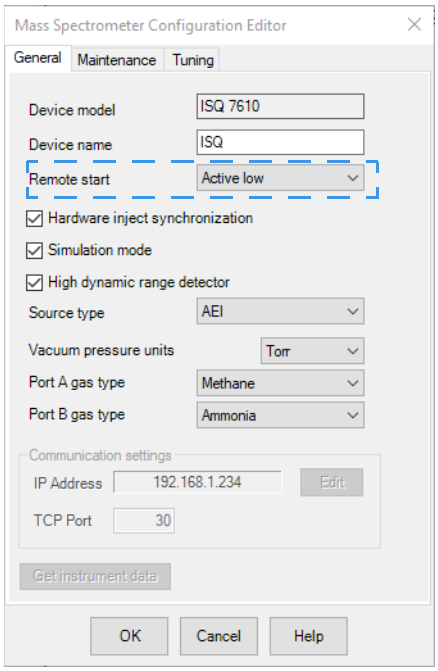


Note You only need to set up the pressure units if you have an ion gauge or convectron gauge installed on your system. The readbacks from these components will display in the units set in this window.

8. Set the remote start. It is used to let the ISQ 7610 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 7610

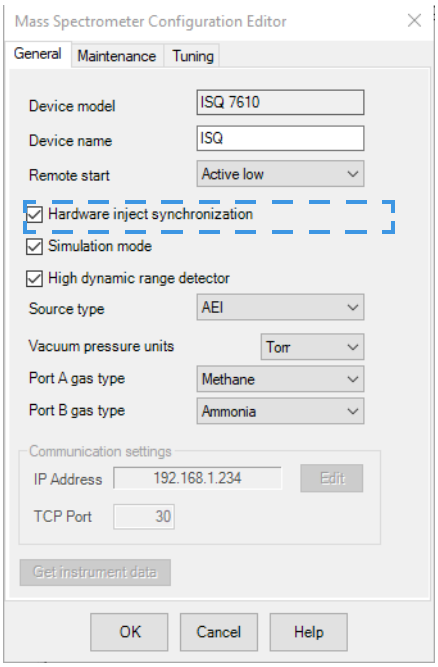
system. In this window, you need to make sure the value matches what you set on the GC. The default is **Active Low**.

Figure 234. Setting the Remote Start



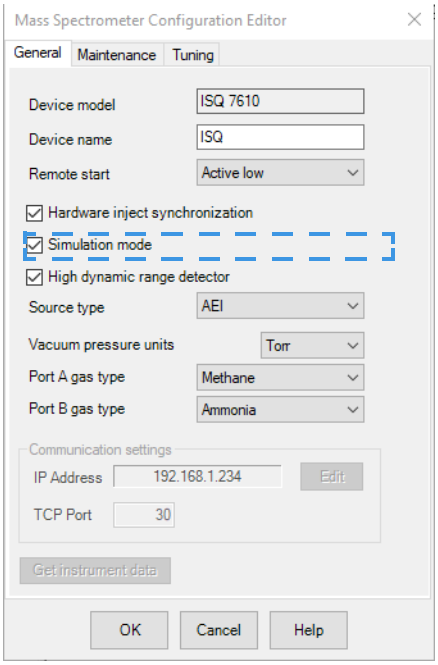
9. Check the **Hardware Inject Synchronization** box to set the instrument to inject with the GC settings. This box is checked by default. See Figure 237.

Figure 235. Configuring Hardware Inject Synchronization



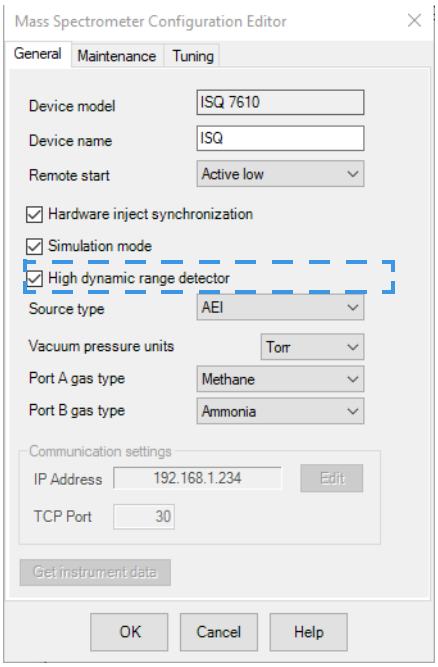
10. If you want the run the instrument in simulation mode, check the **Simulation Mode** box. See Figure 236.

Figure 236. Setting the Simulation Mode



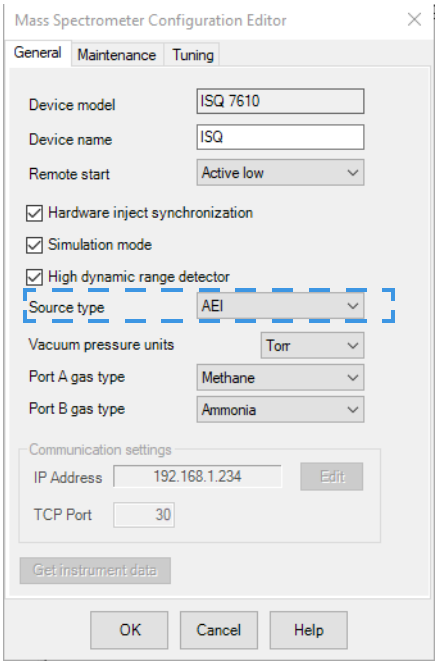
11. If the mass spectrometer is equipped with a high dynamic range electron multiplier, check the **High Dynamic Range Detection** box. See Figure 237.

Figure 237. Configuring the High Dynamic Range



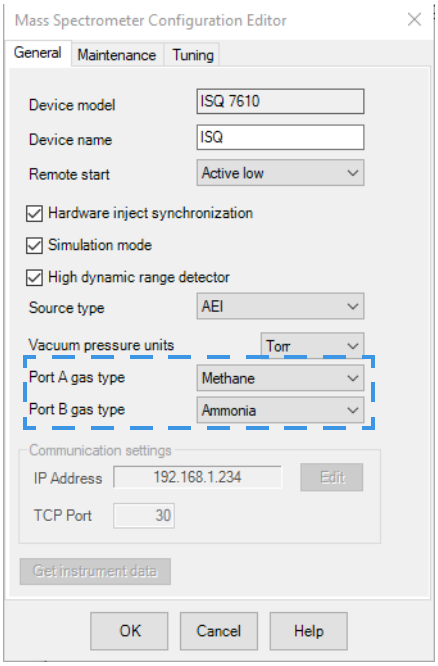
12. Configure the **Source Type**. The choices are EI, CI or AEI. See Figure 238.

Figure 238. Configuring the Ion Source Type



13. If a CI ion source is installed, set the CI gas types you are using in either Port A Gas Type or Port B Gas Type

Figure 239. Configuring the CI Gas Ports

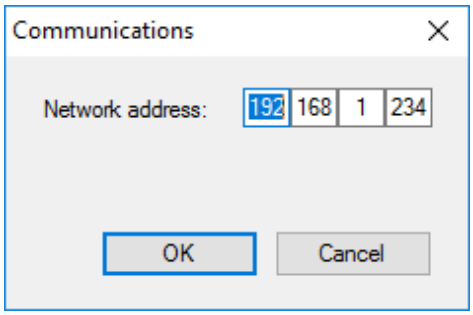


14. Click **Communications** to set the network IP address of the instrument's PC communication board and assign a TCP port.The default settings are entered by the factory and should only be changed after consulting you local IT department and

obtaining special software from customer support necessary to reprogram the IP address on your instrument. Click **Edit** to modify the Instrument IP address.

Note The instrument must be connected to a dedicated Ethernet port on the PC. The instrument cannot be connected through a LAN.

Figure 240. Setting the Network IP Address and the TCP Port



There is a reset button behind the front door of the ISQ 7610. Press and hold the reset button for more than 5 seconds to reset the instrument temporarily to the default IP address: 192.168.1.234. Make sure to send a known IP address to the instrument after reconnecting to the default IP address.

If the IP address is lost, follow the instructions below to recover the previous default IP address. Other network cards on the PC must be disabled or the network cables must be unplugged if the local network addresses begin with 192.168.1.

a. Open the Instrument Configuration on the computer and configure the Mass Spectrometer Communications to IP address 192.168.1.234. Do not close Instrument Configuration.

b. Press and hold the front panel reset button located behind the door on the mass spectrometer for at least 5 seconds. This will boot the instrument from the backup flash memory and the instrument will communicate with the original default IP address (192.168.1.234).

c. Open the ISQ computer network card and reconfigure it to its original IP address (192.168.1.XYZ, where XYZ is not 234).

d. Press **OK** and right click the ISQ network card in the lower right corner of the desktop and select **Repair**.

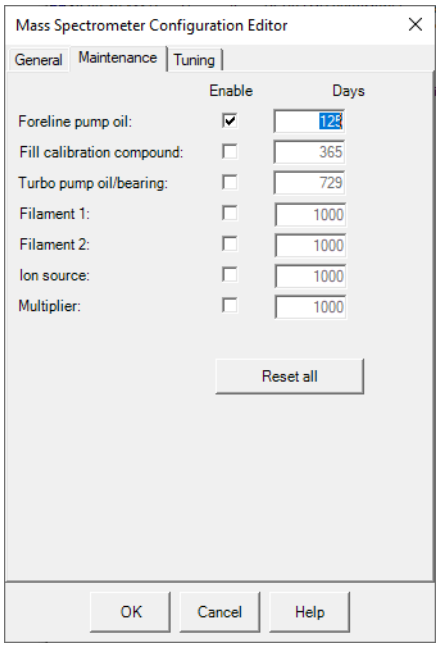
e. Close Instrument Configuration.

15. 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.

16. Check the **Foreline Pump Oil** box 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

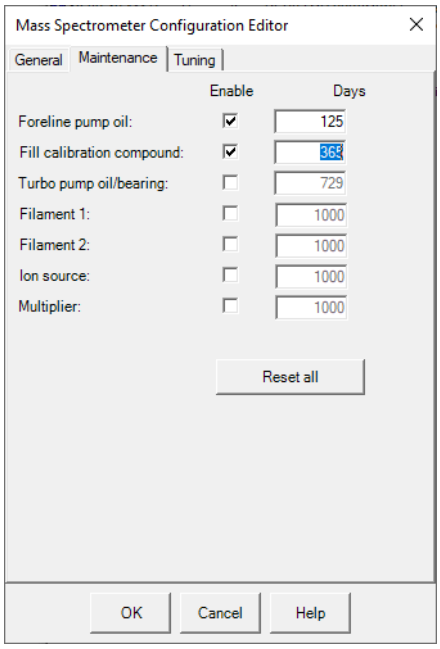
ammonia, you should change the oil every 30 days.

Figure 241. Setting the Foreline Pump Oil Maintenance Interval



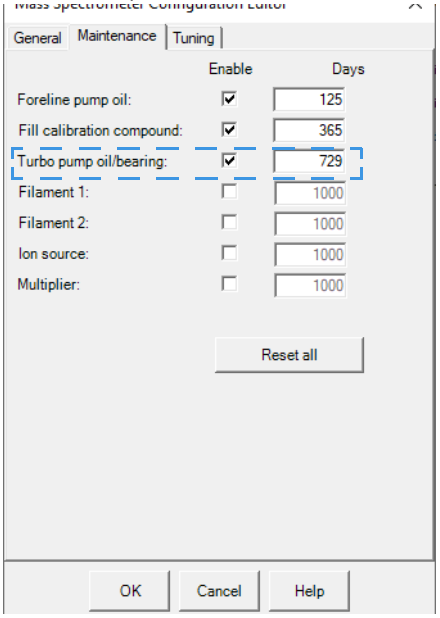
17. Check the **Fill Calibration Compound** box to enable the maintenance intervals. Then set the number of days to elapse before refilling the calibration compounds. The suggested interval is 365 days. See Figure 242.

Figure 242. Setting Calibration Compound Interval



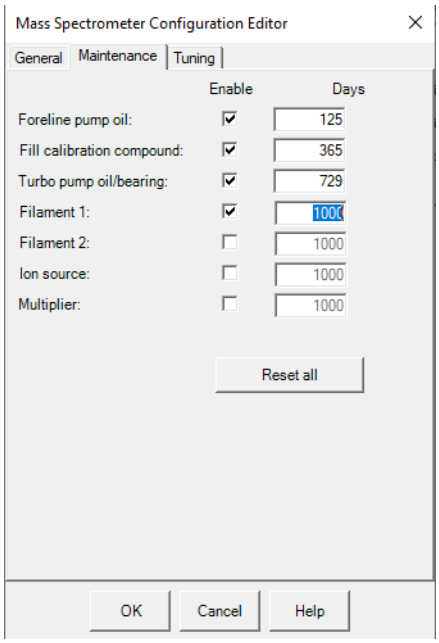
18. Check the **Turbo Pump Oil/Bearing** box to enable the maintenance reminder. Then set the number of days in which you want to be reminded to check the oil. The manufacturer recommends changing the oil cartridge every 730 days and the bearing every 1,460 days. See Figure 243.

Figure 243. Setting the Turbo Pump Oil Maintenance Reminder



19. Check the **Filament 1** box to enable the maintenance reminder. In a leak-free system, the filament should last between 30-360 days, depending on usage. See Figure 244

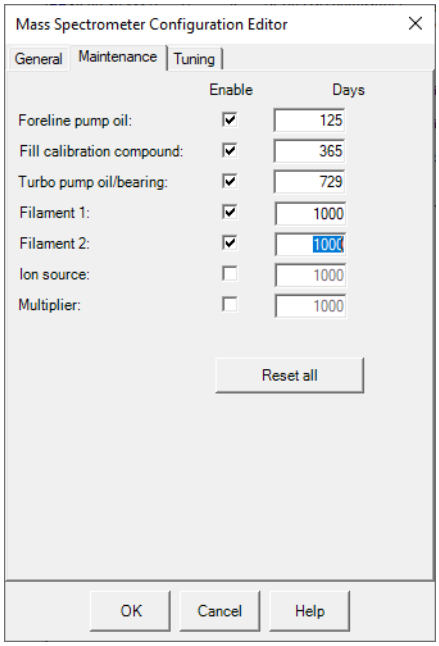
Figure 244. Setting the Filament 1 Maintenance Reminder



20. Check the **Filament 2** box 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. See Figure 245

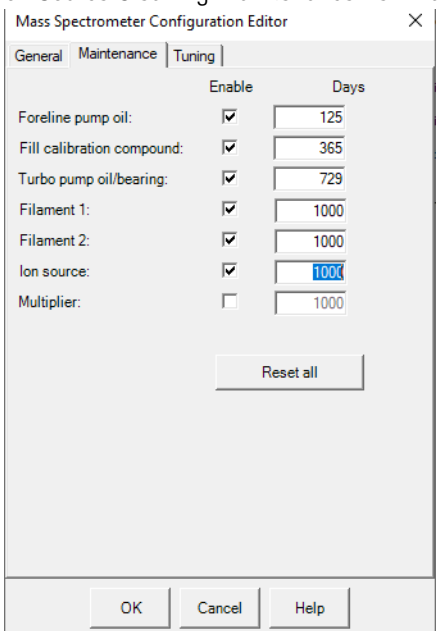
Figure 245. Setting the Filament 2 Maintenance Reminder



21. Check the **Ion Source** box 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

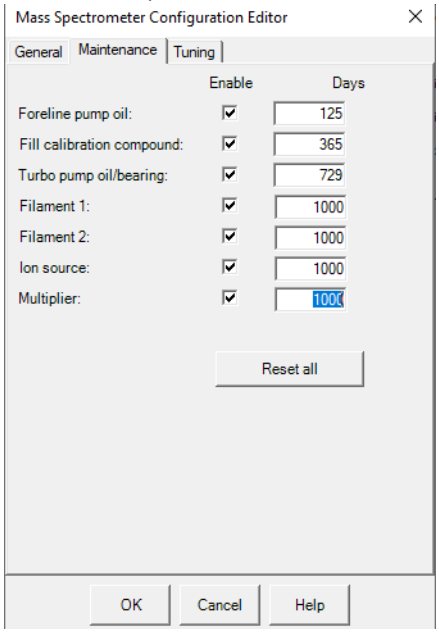
cleaning depends very strongly on your analysis. You will have to determine the correct length of time between source cleanings. See Figure 246.

Figure 246. Setting the Ion Source Cleaning Maintenance Reminder



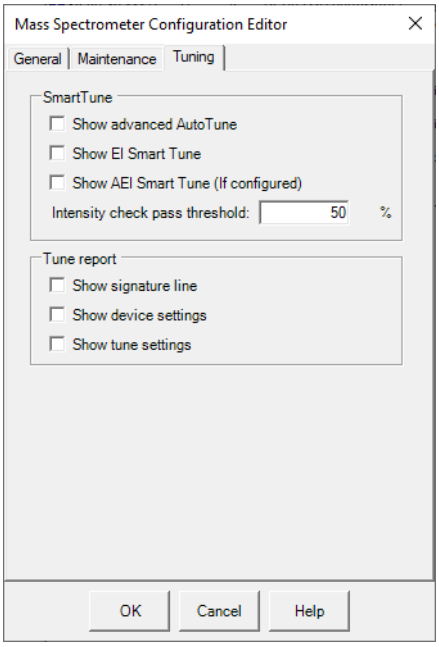
22. Check the **Multiplier** box to enable the maintenance reminder. Then set the number of days in which you want to be reminded to check the electron multiplier. See Figure 247.

Figure 247. Setting the Electron Multiplier Maintenance Reminder



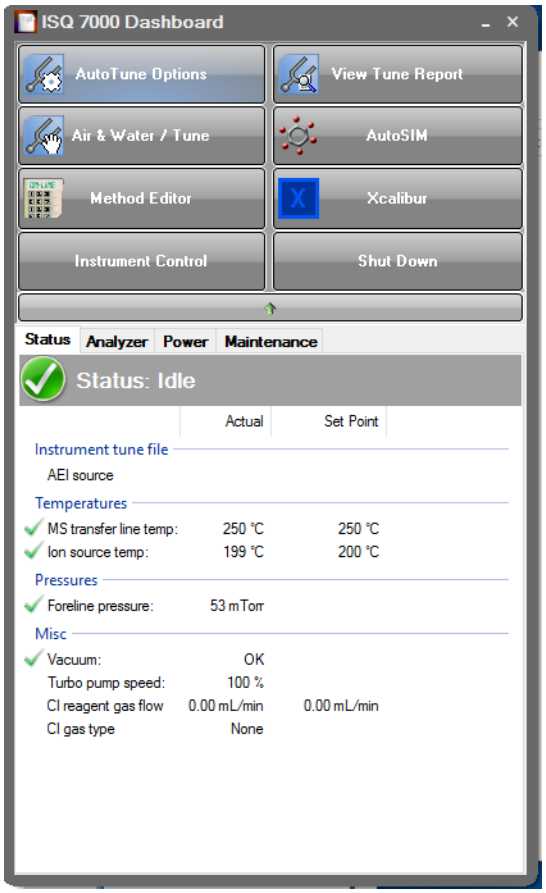
23. Click the **Tuning** tab and use the check boxes to enable **Advanced AutoTune** and the **SmartTune** application. You can also set the **Intensity Check Pass Threshold** and the **Tune Report** settings here. See Figure 248.

Figure 248. Configuring the MS Tuning



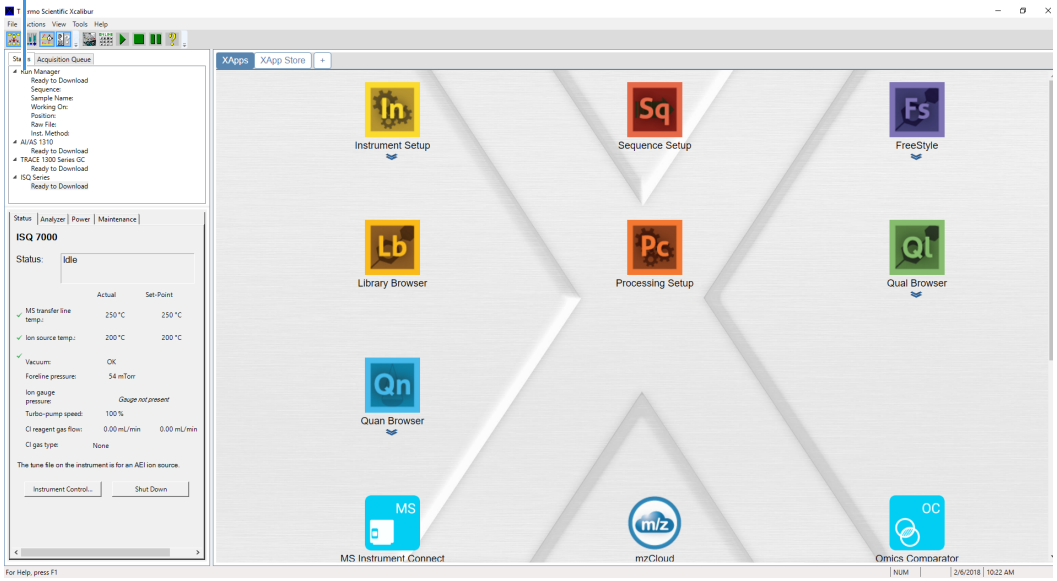
24. Click **OK** to return to the main Instrument Configuration window in both Xcalibur and Chromeleon software.
25. Click the **Done** button to close the Instrument Configuration utility in Xcalibur.
26. In Xcalibur software, you can check the status of your ISQ 7610 instrument in the Status tab of the ISQ Dashboard. See Figure 249.

Figure 249. Checking the ISQ 7610 Instrument Status on the ISQ Dashboard



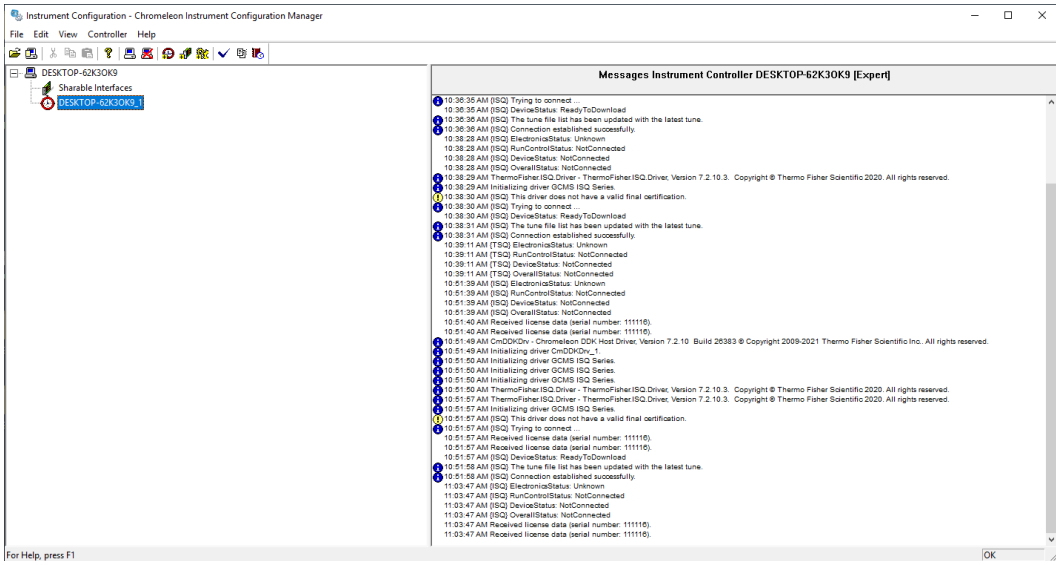
27. In the **Status Pane** of the Xcalibur Roadmap window, you can check the status of all your instruments. See Figure 251.

Figure 250. Checking All Instrument Statuses in Xcalibur Status Pane



28. You can view instrument status in Chromeleon software in the M**essages Instrument Controller** pane in the **Chromeleon Instrument Configuration Manager**. See Figure 251.

Figure 251. Checking the Instrument Status in Chromeleon Software



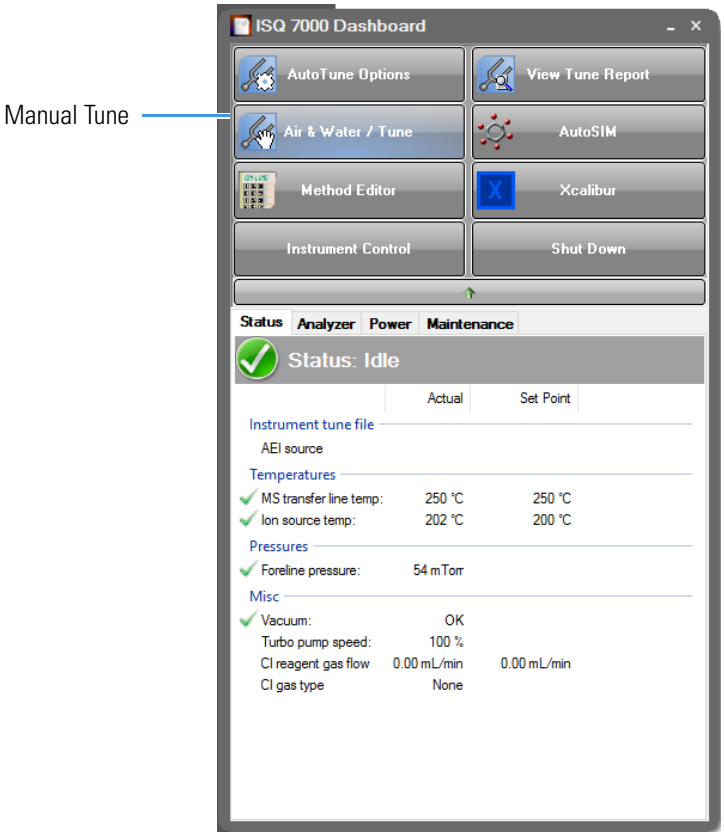
Upgrading the Software

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.

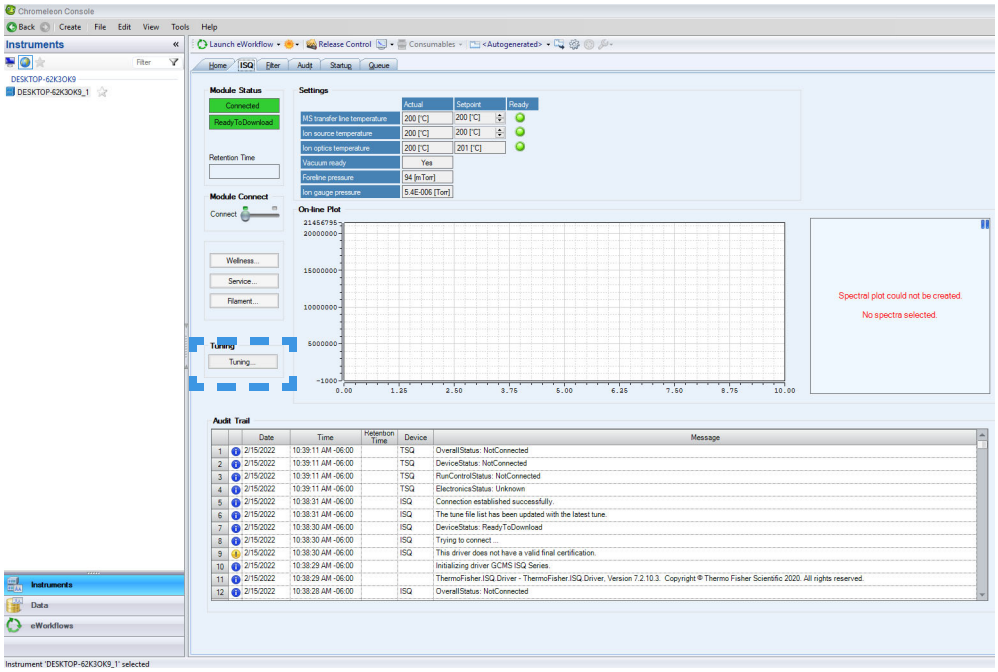
1. Ir you are running Xcalibur software, click **Air & Water/Spectrum** on the ISQ 7610 Dashboard to open the manual tune utility. See Figure 252.

Figure 252. ISQ 7610 Dashboard



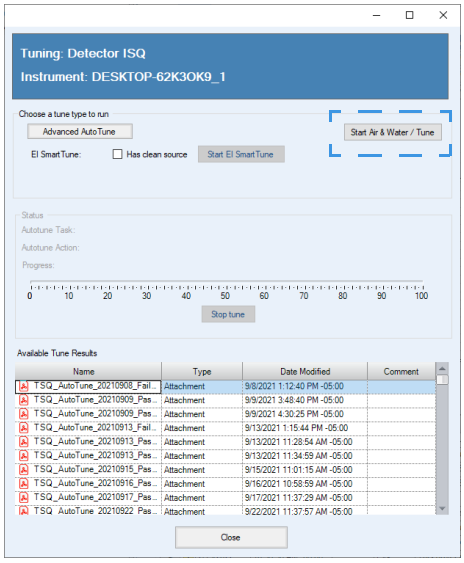
1. If you are running Chromeleon software, Go to the **ISQ** tab on the Chromeleon Console and click **Tuning** to open the tuning window. See Figure 253.

Figure 253. Accessing ISQ 7610 Tuning from the Chromeleon Console



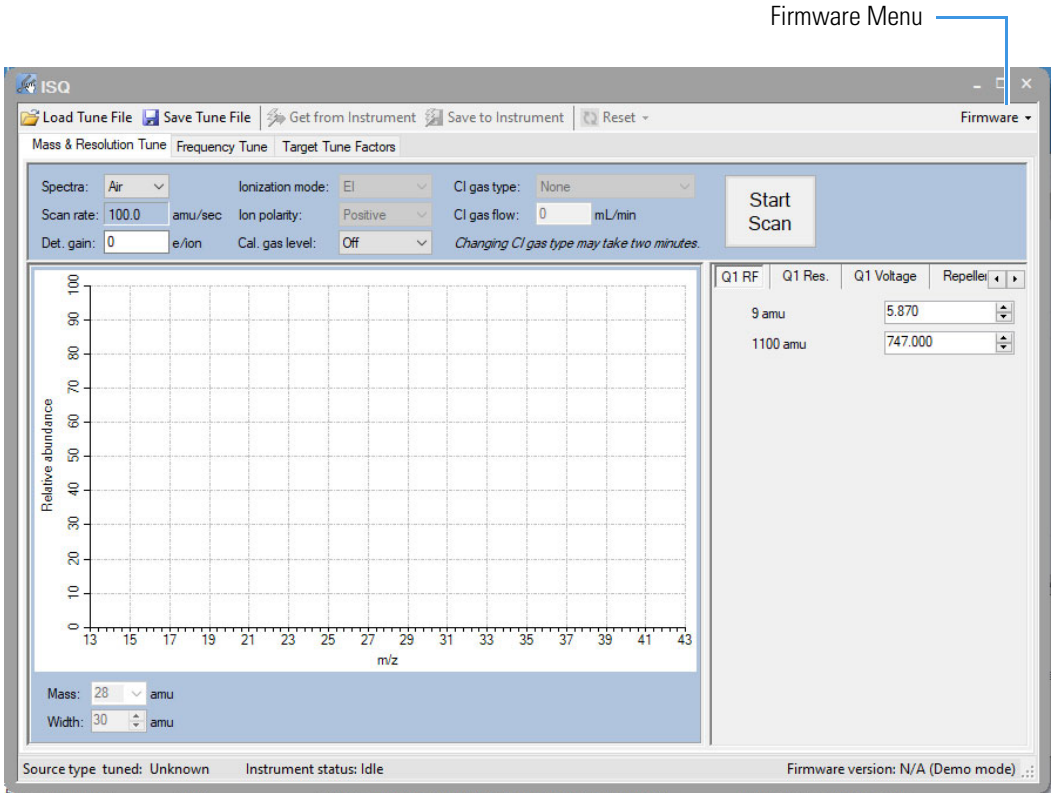
2. The **Tuning: Detector ISQ** window opens. Click **Start Air & Water/Tune**. See Figure 254.

Figure 254. Opening Manual Tune in Chromeleon Software



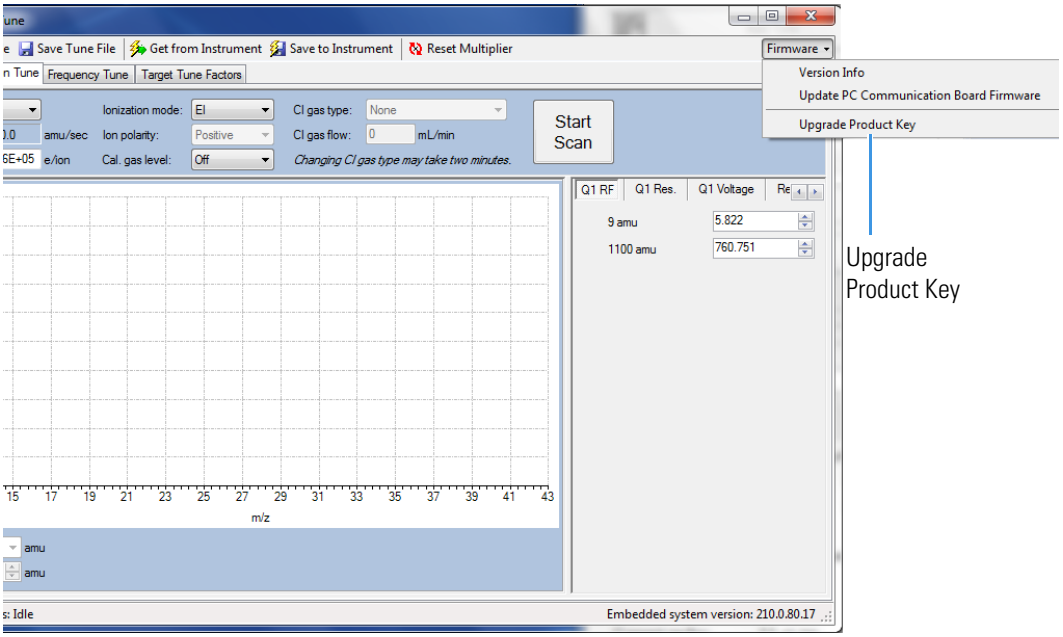
3. The **ISQ Manual Tune** application opens. Click **Firmware** on the upper right-hand side of the screen to open the firmware menu. See Figure 255.

Figure 255. ISQ Manual Tune Home



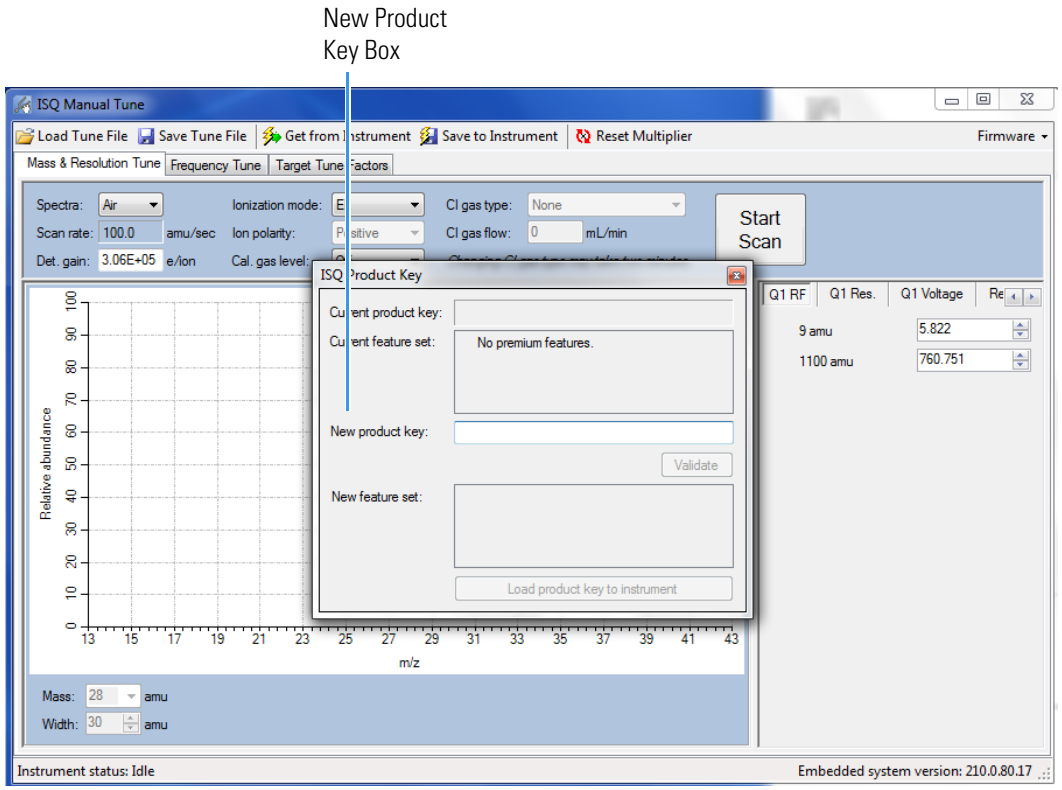
4. The firmware menu opens. Select **Update Product Key** as shown in Figure 256.

Figure 256. Selecting the Upgrade Product Key Option



5. The **ISQ Product Key** window opens. Enter your product key into the **New Product Key** box as shown in Figure 257.

Figure 257. Entering the Product Key



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