



**Thermo Scientific** 

# **System Start Up**

# Instructions

For Gas Chromatographs, Autosamplers, and Mass Spectrometers

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## Declaration

#### Manufacturer: Thermo Fisher Scientific

Thermo Fisher Scientific is the manufacturer of the instruments 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.

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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

Refer to your instrument's documentation set for specific EMC compliance information.

# Safety

Refer to your instrument's documentation set for specific safety compliance information.

# **FCC Compliance Statement**

Refer to your instrument's documentation set for specific FCC compliance information.



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

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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.

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# 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.

### thermo scientific

For manufacturing location, see the label on the instrument.

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# **Preface**

This guide contains detailed information for system startup (GC and GC/MS) after a period of instrument downtime.

#### Contents

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

#### **About Your System**

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

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



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

## **Related Documentation**

Visit https://gcgcms.freshdesk.com/support/solutions for current product documentation for your autosampler, GC, and GC/MS instrument.

### Safety and Special Notices

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

#### **Special Notices**

Special notices include the following:

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

**Note** Highlights information of general interest.

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

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

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



**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.



**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*.

# **Hydrogen Safety Precautions**

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

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



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

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

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

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

- Try to reduce or eliminate the higher risks by using the proper ventilation to remove hydrogen gas before an ignitable concentration can accumulate. You should also consider purging the hydrogen to further reduce hazards and ensure anyone who will be working with hydrogen has basic hydrogen safety training.
- As with laboratory safety in general, be sure to wear safety glasses, laboratory coats, gloves, etc. Typically there are no specific requirements for gaseous hydrogen, other than eye protection when working with a compressed gas. If working with liquid (cryogenic) hydrogen, insulated gloves and protective shoes should be worn in addition to eye protection.

- You should post "No Smoking" and "No Open Flames" signs to identify hydrogen sources and cylinders. Maintain, inspect and leak-test all hydrogen sources regularly.
- All hydrogen shutoff valves should be clearly marked and permanent hydrogen piping should be labeled as such at the supply or discharge point and at regular intervals along its length. Where hydrogen gas piping passes through a wall, the piping should be labeled on both sides of the wall.
- There should also be contingency plans in place should an incident occur.
- The site emergency response team, as well as the local fire department, should know the location of all hydrogen storage tanks.

#### Using Hydrogen with TRACE 1300/TRACE 1310

The use of hydrogen as a carrier gas, or as fuel gas for certain flame detectors, requires strict attention and compliance with special precautions due to the hazards involved.

**WARNING - EXPLOSION HAZARD** Hydrogen is a dangerous gas that, when mixed with air, could create an explosive mixture. The use of hydrogen as a carrier gas requires extreme caution. Special precautions must be taken because of the risk of explosion. When hydrogen is used as carrier gas the gas chromatograph must be equipped with a hydrogen sensor.



Never use hydrogen as carrier gas in your TRACE 1300/TRACE 1310 system unless your oven has a hydrogen sensor installed. Thermo Fisher Scientific FSEs are not authorized to install or repair any instrument using hydrogen as a carrier gas unless the instrument is equipped with the appropriate sensor.

If your oven does not have a hydrogen sensor already installed, contact your Thermo Fisher Scientific sales representative. To comply with instrument safety requirements, a Thermo Fisher Scientific FSE authorized service personnel should install the sensor into your TRACE 1300/TRACE 1310.

Hydrogen is a dangerous gas, particularly in an enclosed area when it reaches a concentration corresponding to its lower explosion level (4% in volume). An explosion hazard could develop in the oven when hydrogen is used as a carrier gas in the case oven elements are not perfectly connected to each other, or when the connection materials are worn out, broken, or otherwise faulty.

Use the following safety precautions when using hydrogen:

- Ensure that all hydrogen cylinders comply with the safety requirements for proper use and storage. Hydrogen cylinders and delivery systems must comply with local regulations.
- Make sure the gas supply is turned completely off when connecting hydrogen lines.
- Perform a leak test to ensure that the hydrogen lines are leak-tight before using the instrument. Repeat this test to eliminate all leaks.

• Ensure your TRACE 1300/TRACE 1310 has a Thermo Scientific hydrogen sensor installed for continuously monitoring the hydrogen level in the oven.

### Using Hydrogen with the MS System

To use hydrogen with the ISQ 7000 or TSQ 9000, you must always shut off the GC carrier gas before venting or turning off the MS instrument. There are three hydrogen safety screws on the MS that **must** be in place. These are attached to your instrument at the factory. See Figure 1 and Figure 2.



Figure 1. Hydrogen Safety Screws on the TSQ 9000



Figure 2. Hydrogen Safety Screws on the ISQ 7000 Instrument

Before powering on the MS system, ensure that:

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

#### **Hydrogen Connection Guidelines**

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

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

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

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

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

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

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

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

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

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

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

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

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

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

#### **Purchasing Hydrogen**

Use the following guidelines when purchasing hydrogen:

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

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

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

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

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

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

#### **Properly Storing Hydrogen**

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

Use the following guidelines when storing hydrogen:

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

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

#### Hydrogen Safety Codes, Standards and References

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

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

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

### **Hazardous Substances Precautions**





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

#### **Biological Hazard Warning Note**



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

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

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

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

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



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

#### **Venting Toxic Gases**

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

### **Contacting Us**

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

#### To find out more about our products

Go to www.thermofisher.com for information about our products.

To get local contact information for sales or service

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

# **Contents**

	Preface
	About Your Systemix
	Related Documentationx
	Safety and Special Noticesx
	Special Notices
	Safety Symbols and Signal Words
	Hydrogen Safety Precautionsxii
	Using Hydrogen with TRACE 1300/TRACE 1310
	Using Hydrogen with the MS System
	Hydrogen Connection Guidelinesxv
	Purchasing Hydrogen xvii
	Properly Storing Hydrogen xvii
	Hydrogen Safety Codes, Standards and References
	Hazardous Substances Precautions
	Biological Hazard Warning Note
	Venting Toxic Gasesxxi
	Contacting Usxxi
Chapter 1	Gas Equipment Requirements1
•	GC Requirements
	Cryogenic Cooling
	Using Liquid Nitrogen
	Using Carbon Dioxide
	Using Hydrogen
	MS Requirements
	1
	GC Carrier Gas
	GC Carrier Gas
	GC Carrier Gas       7         CI Gas       8         Collision Gas       9
	GC Carrier Gas7CI Gas8Collision Gas9Other Gas Specifications10

C –

Chapter 2	GC Start Up
-	Powering On the Instrument
	TRACE 1300 LEDs
	TRACE 1310 Touch Screen
	Replacing the Column
Chapter 3	Software Configuration
	Using the Touch Screen (TRACE 1310)21
	Turning on the Carrier Gas
	Leak Checking and Column Conditioning
	Turning on a Detector
	Using Chromeleon
	Turning on the Carrier Gas
	Leak Checking and Column Conditioning
	Turning on a Detector
	Using Xcalibur
	Turning on the Carrier Gas
	Leak Checking and Column Conditioning
	Turning on a Detector
	Purging the Helium Purifier (PDD Customers)
Chapter 4	
	InPlus RSH Start Up
	How to Start the IriPlus RSH
	Changing Access Level
	IriPlus 300 HS Start Up
	TriPlus 500 HS Start Up
	AI 1310/AS 1310 Start Up
Chapter 5	Mass Spectrometer Start Up51
•	Installing the Column
	Powering On the Instrument
	Tuning
	Manual Tune
	EI Tune
<b>A</b>	
Chapter 6	U Exactive GC and Exactive GC Start Up
	Installing the Column
	Powering On the Instrument

# **Gas Equipment Requirements**

Use the following guidelines to ensure you have the proper gas supplies.

#### **Contents**

- GC Requirements
- MS Requirements

### **GC Requirements**

Use the following guidelines to make sure the gas supplies for your system are ready. You will need a supply of ultra-high purity GC gases.

The gases used with the instrument are Helium, Nitrogen, Hydrogen, Air, Argon, and Argon/Methane. Other gases are rarely used.

**WARNING** Before using gases, carefully read the hazard indications and information reported in the Safety Sheet supplied by the manufacturer referring to the CAS (Chemical Abstract Service) number. It is the user's responsibility to see that all local safety regulations for the use of gases are obeyed.



All Thermo Fisher Scientific gas chromatographs normally uses an inert gas as carrier gas. If you wish to use hydrogen as a carrier gas, the hydrogen sensor must be installed. Contact a Thermo Fisher Scientific sales representative if you plan to use hydrogen as the carrier gas in your GC. If you don't have the hydrogen sensor, you **must** use an inert carrier gas. Refer to "Using Hydrogen with TRACE 1300/TRACE 1310" on page xiii for details.

**CAUTION** Secure gas cylinders to an immovable structure or wall. Handle all gases according to local safety regulations.

Do not place gas tanks in the path of the TRACE 1300/TRACE 1310 GC oven exhaust.

1. You must provide the gas supplies for your gas chromatograph. Be sure to order your gases and regulators far enough ahead of time to have them ready for the GC installation process.

The following table lists the gas recommendations:

Detector Type	Carrier Gas	Fuel Gas	Make-up Gas
FID	Helium, Nitrogen, Hydrogen <sup>1</sup>	Hydrogen + Air	Helium, Nitrogen
NPD	Helium, Nitrogen, Hydrogen <sup>1</sup>	Hydrogen + Air	Helium, Nitrogen
ECD	Helium, Nitrogen, Argon	None	Nitrogen, Argon/5% Methane
TCD	Helium, Nitrogen, Hydrogen <sup>1</sup> , Argon	None	Same as carrier
FPD	Helium, Nitrogen	Hydrogen + Air	None
PDD	Helium is the gas used	for PDD discharge and	carrier supply.
MS	Helium, Hydrogen <sup>1</sup>	None	None

**Table 1.** Gas Recommendations

<sup>1.</sup> If hydrogen is used as carrier gas, you must install a hydrogen sensor into the TRACE 1300/TRACE 1310 GC.

2. You will need a supply of ultra-high purity GC carrier gas. Typical cylinders are about 23 cm (9 in.) wide by 140 cm (55 in.) tall and output >15,000 kPa (>2200 psig). A single full-size tank contains 8000 L of helium or 6000 L of hydrogen and each will last about three months with a typical usage rate of 50 mL/min.

Gas Type	Purity <sup>1</sup>	Outlet Pressure	Regulator	Connector <sup>2</sup>
Helium	99.999%	400-1050 kPa (58-150 psig)	Dual stage brass regulator with stainless steel	CGA-580
Nitrogen	99.999%	400-1050 kPa (58-150 psig)	<sup>–</sup> diaphragm. The regulator output	CGA-580
Hydrogen	99.999%	400-1050 kPa (58-150 psig)	pressure should be adjustable from 300 to	CGA-350
Air	99.999%	400-1050 kPa (58-150 psig)	- 1050 kPa (45–150 psig)	CGA-590
Argon/5% Methane	99.999%	400-1050 kPa (58-150 psig)	-	CGA-350

Table 2. Gas Specifications

<sup>1</sup> Ultra-high purity with less than 1.0 ppm each of water, oxygen, and total hydrocarbons and contained in one tank. Impurities below 1.0 ppm generally do not require purification. Gases with higher impurity levels may require the use of appropriate water, oxygen and hydrocarbon traps.

 $^2$  Connectors will vary with cylinder size. Confirm that your regulator will work with your gas tank. All connections to the GC are 1/8 in. Swagelok fittings.



**WARNING - FIRE HAZARD** When using hydrogen, be aware that it can flow into the oven and create a fire hazard. Turn off the supply until the GC column is in the inlet and the detector. Whenever you use hydrogen, it is critical to test all connections, lines, and valves for leaks before using the instrument. When performing maintenance, be sure to turn off the hydrogen supply.

Oxygen and moisture cannot be prevented from entering the system during cylinder changes. To minimize the impact of these contaminants on the GC system, high purity gas handling equipment should be used. To further protect the system from oxygen and moisture, point-of-use purifiers should be installed in the carrier gas lines just prior to the GC to remove any residual contaminants. See Table 3.

 Table 3.
 Trap Specifications (Sheet 1 of 2)

Traps	Use
Moisture trap	Water in the carrier or fuel gas may damage the analytical column and contaminate the system. Water content should be less than 1 ppm in all cases. If you are using multiple traps, install the moisture trap closest the gas supply, before the hydrocarbon and the oxygen trap.
Hydrocarbon trap	Hydrocarbon traps remove organic materials from gases. If you are using multiple traps, install the hydrocarbon trap after the moisture trap, but before the oxygen trap.

Traps	Use
Oxygen trap	Oxygen content in the carrier and gas lines should be less than 1 ppm. To achieve a level of oxygen of less than 1 ppm, install an oxygen-removing trap in the carrier gas line between the gas tank and the TRACE 1300/TRACE 1310 GC. If you are using multiple traps, the oxygen trap should be the last trap in the series.

3. If you have a TriPlus RSH or a TriPlus autosampler with the SPME conditioning station or a TriPlus Headspace autosampler, you need to obtain a low-pressure, single-stage regulator (0-30 psi) (P/N 1R77010-1089) for nitrogen purging.

Table 4. Other Gas Specifica	ations
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 Table 3.
 Trap Specifications (Sheet 2 of 2)

Equipment	Gas Type	Purity	Max. Pressure	Regulator	Connector
TriPlus RSH TriPlus SPME TriPlus HS	Nitrogen	99.999%	200 kPa (30 psig)	Dual stage brass regulator with stainless steel diaphragm.	CGA-580 <sup>1</sup>

<sup>1.</sup> Connectors will vary with cylinder size. Confirm that your regulator will work with your gas tank. All connections to the GC/MS are 1/8 in. Swagelok fittings.

- 4. If your TRACE 1300/TRACE 1310 GC and PTV injector will be equipped with a cryogenic cooling option, you will need a supply of coolant, such as liquid nitrogen or carbon dioxide. See the "Cryogenic Cooling" on page 4 for more information.
- 5. Gas lines should be:
  - As short as possible, run to the back or side of the GC system.
  - Made of pre-cleaned copper or stainless steel when using helium and hydrogen.
  - Free of oil and moisture.
- 6. Obtain the proper gas line filters, which help prevent impurities and contaminants from entering your system. Water, oxygen, and total hydrocarbons should be less than 1 ppm to avoid high background noise and prevent contamination. The GC is equipped with intake filters that trap moisture, oxygen, and hydrocarbons.
- 7. Store gas tanks and bottles properly so they will not damage cables or gas lines. Ensure they are secured in accordance with standard safety practices.

#### **Cryogenic Cooling**

If you have purchased a cryogenic cooling option for the oven and/or PTV injector to operate at sub-ambient temperature, you will need to provide a coolant supply. Your TRACE 1300/TRACE 1310 GC is already configured for either liquid nitrogen or carbon dioxide.

- The oven cryogenic system can reach  $-100\ ^\circ C$  with liquid nitrogen or  $-50\ ^\circ C$  with carbon dioxide.
- The PTV cryogenic system can reach -100 °C with liquid nitrogen or -50 °C with carbon dioxide.

Be sure to identify which cryogenic cooling option your instrument is configured for before you order cryogenic coolant.

#### **Using Liquid Nitrogen**

Before using liquid nitrogen, read the indications of hazard and the instructions reported in the Safety sheet supplied by the manufacturer with reference to the CAS number (Chemical Abstract Service) 7727-37-9.

**WARNING** High pressures and extremely low temperatures make liquid nitrogen a hazardous material. High concentrations of nitrogen in the air can cause asphyxiation hazard. To avoid injury, always follow the safety precautions and delivery system design recommended by your gas supplier.

Use personal protection:

- Protective gloves: Loose fitting thermal insulated or leather gloves.
- Eye protection: Full face shield and safety glasses are recommended.
- **Other protective equipment**: Safety shoes when handling containers. Long sleeve shirts and trousers without cuffs. Work clothing that sufficiently prevents skin contact should be worn.

Liquid Nitrogen must be supplied at a pressure of 1.5 bar (150 kPa; 21.75 psig). Figure 1 shows the proper configuration for a liquid nitrogen tank.





Plumbing to the GC should be 1/4 in. copper or stainless steel tubing with insulation. It is your responsibility to ensure the liquid delivery connection from the liquid nitrogen cryogenic supply is adaptable to 1/4 in. tubing.



The liquid nitrogen cryogenic valve on the TRACE 1300/TRACE 1310 GC is a 1/4 in. Swagelok fitting.

#### **Using Carbon Dioxide**

Before using carbon dioxide, read the indications of hazard and the instructions reported in the Safety sheet supplied by the manufacturer with reference to the CAS number (Chemical Abstract Service) 124-38-9.

**WARNING** High pressures and extremely low temperatures make pressurized carbon dioxide a hazardous material. High concentrations of Carbon Dioxide are dangerous. To avoid injury, always follow the safety precautions and delivery system design recommended by your gas supplier.



Use personal protection:

- Protective gloves: Loose fitting thermal insulated or leather gloves.
- Eye protection: Full face shield and safety glasses are recommended.
- **Other protective equipment**: Safety shoes when handling containers. Long sleeve shirts and trousers without cuffs. Work clothing that sufficiently prevents skin contact should be worn.

Carbon dioxide must be supplied by a high-pressure cylinder with a dip tube. Figure 2 shows the proper carbon dioxide tank configuration.

Figure 2. Carbon Dioxide Tank Configuration



It is your responsibility to ensure the liquid delivery connection from the carbon dioxide cryogenic supply is adaptable to 1/8 in. tubing.

The Carbon Dioxide cryogenic valve on the TRACE 1300/TRACE 1310 GC is a 1/8 in. Swagelok fitting.

#### **Using Hydrogen**

To safely use hydrogen, you should have a hydrogen sensor installed in your GC. Field Service Engineers can install a sensor. but they are not authorized to install or repair any instrument using hydrogen as a carrier gas unless the instrument is equipped with the appropriate sensor. The sensor must be calibrated occasionally, as described in the sensor's documentation.

Use the following safety precautions when using hydrogen:

- Ensure that all hydrogen cylinders comply with the safety requirements for proper use and storage. Hydrogen cylinders and delivery systems must comply with local regulations.
- Make sure the gas supply is turned completely off when connecting hydrogen lines.
- Perform a bubble test to ensure that the hydrogen lines are leak-tight before using the instrument. Repeat this test to eliminate all leaks.
- Ensure your GC has a Thermo Fisher Scientific hydrogen sensor installed. A hydrogen sensor continuously monitors the hydrogen level in the oven.
- Remove as many sources of ignition as possible from your laboratory. Sources can include open flames, electrostatic discharges, or devices that spark.
- Do not open a cylinder of hydrogen without a regulator attached because it may self-ignite.

### **MS Requirements**

Use the following guidelines to ensure you have the proper gas supplies.

#### **GC Carrier Gas**

• You will need a supply of ultra-high purity GC carrier gas. Typical cylinders are about 23 cm (9 in.) wide by 140 cm (55 in.) tall and output >15,000 kPa (>2200 psig). A single full-size tank will last about three months with a typical usage rate of 50 mL/min. If you have additional detectors or optional accessories, please refer to your GC or autosampler manuals for information about gas requirements.

**Note** Thermo Fisher Scientific installation specifications require helium as a carrier gas and argon as a collision gas. You must have one tank of each gas at installation, or the field service engineer will not be able to run specifications on your MS.

Gas Type	Purity <sup>1</sup>	Outlet Pressure	Regulator	Connector <sup>2</sup>
Helium	99.999%	400-700 kPa (58-100 psig)	Dual-stage brass regulator with stainless steel diaphragm	CGA-580
Hydrogen	99.999%	400-700 kPa (58-100 psig)	Dual-stage brass regulator with stainless steel diaphragm	CGA-350

Table 5.	Carrier	Gas S	pecifications

<sup>1</sup> Ultra-high purity with less than 1.0 ppm each of water, oxygen, and total hydrocarbons and contained in one tank. 2 Connectors will vary with cylinder size. Confirm that your regulator will work with your gas tank. All connections to the GC/MS are 1/8 in. Swagelok fittings.

Note Systems using the Advanced EI source must use helium carrier gas only.



**CAUTION** The Helium Saver and PDD detector are not for use with oxygen - either pure oxygen or gases with a significant proportion of oxygen. The purifier's gettering alloy is pyrophoric at operating temperature. Use with significant amounts of oxygen can result in combustion of the material, potential damage to the surrounding area, and possible injury.



**WARNING FIRE HAZARD:** When using hydrogen, be aware that it can flow into the oven and create a fire hazard. Turn off the supply until the GC column is in the inlet of the MS. Whenever you use hydrogen, it is critical to test all connections, lines, and valves for leaks before using the instrument. When performing maintenance, be sure to turn off the hydrogen supply.

• Oxygen and moisture cannot be prevented from entering the system during cylinder changes. To minimize the impact of these contaminants on the GC system, high purity gas handling equipment should be used. To further protect the system from oxygen and moisture, point-of-use purifiers should be installed in the carrier gas lines just prior to the GC to remove any residual contaminants.

#### **CI Gas**

**Note** Thermo Fisher Scientific installation specifications require methane as a CI gas. You must have one tank of methane at installation, or the field service engineer will not be able to run specifications on your MS.

• If your system is equipped with the Chemical Ionization (CI) Reagent Gas Flow module, make sure you have the proper gas for it. Typical flow rates are only 1-3 mL/min, so smaller tanks like lecture bottles can be used.

Gas Type	Purity	Outlet Pressure	Regulator	Connector*
Methane	99.99% high-purity	35-240 kPa (5-35 psig)	Dual-stage brass regulator with stainless steel diaphragm	CGA-350
Isobutane	99.9% instrument grade	35-240 kPa (5-35 psig),	Dual-stage brass regulator with stainless steel diaphragm	CGA-510
Ammonia	99.99%, anhydrous grade	35-240 kPa, (5-35 psig)	Consult your gas supplier for specific regulator requirements.	CGA-240

#### Table 6. CI Gas Specifications

\* Connectors will vary with cylinder size. Confirm that your regulator will work with your gas tank. All connections to the GC/MS are 1/8 in. Swagelok fittings.



**WARNING FIRE HAZARD:** Some CI gases, such as methane and isobutane, are flammable. Make sure these gases are properly exhausted and all gas fittings on the system are leak-free. Consult your local Environmental and Safety Regulations for information about how to properly exhaust fumes from your laboratory.



**WARNING TOXIC SUBSTANCES HAZARD:** Some CI gases, such as ammonia, are toxic. Make sure these gases are properly exhausted and all gas fittings on the system are leak-free. Consult your local Environmental and Safety Regulations for information about how to properly exhaust fumes from your laboratory.



**CAUTION INSTRUMENT DAMAGE:** Do not exceed 240 kPa (35 psig) or you could damage the CI reagent gas flow module.

#### **Collision Gas**

You may use argon or nitrogen as collision gases for the MS. Typical cylinders are about 23 cm (9 in.) in diameter by 140 cm (55 in.) tall and output >15,000 kPa (>2200 psig). A cylinder should last about three years at a constant purge flow rate of 5.0 atm-mL/min. Table 7 provides the various collision gas specifications.

Gas Type	Purity	Input Pressure	Regulator	Connector*
Argon	99.999%	407-421 kPa (59-61 psig)	Dual-stage brass regulator with stainless steel diaphragm	CGA-580
Nitrogen	99.999%	386-400 kPa (56-58 psig)	Dual-stage brass regulator with stainless steel diaphragm	CGA-580

Table 7. Collision Gas Specifications

\* Connectors will vary with cylinder size. Confirm that your regulator will work with your gas tank. All connections to the GC/MS are 1/8 in. Swagelok fittings.



**CAUTION** Collision gas input pressure must remain constant for proper instrument performance. The regulator you use to supply the collision gas must be able to deliver  $60 \pm 1$  psig for argon or  $57 \pm 1$  psig for nitrogen. It should be marked clearly at 60 or 57 psig and be stable enough to supply constant pressure at  $60 \pm 1$  psig for argon or  $57 \pm 1$  psig for nitrogen.



**CAUTION** If your TRACE 1300/TRACE 1310 and PTV injector will be equipped with a cryogenic cooling option, see the *TRACE 1300 and TRACE 1310 Preinstallation Requirements Guide* for more information.

#### **Other Gas Specifications**

- If your system will be equipped with a Direct Insertion Probe, make sure you have compressed air, which is used to cool the probe.
- If you have a Thermo Scientific<sup>™</sup> TriPlus<sup>™</sup> RSH autosampler with the SPME conditioning station or a Thermo Scientific Headspace autosampler, you need to obtain a low-pressure, single-stage regulator (0-30 psi) (P/N 1R77010-1089) for nitrogen purging.

Equipment	Gas Type	Purity	Maximum Pressure	Regulator	Connector
Direct Insertion Probe	Air	90% <sup>1</sup>	700 kPa (100 psig)	Dual-stage brass regulator	CGA-346
TriPlus SPME Headspace	Nitrogen	99.999%	200 kPa (30 psi)	Dual-stage brass regulator with stainless steel diaphragm	CGA-580

#### **Table 8.** Other Gas Specifications

<sup>1</sup> Pure, particle and oil free, and contained in one tank.

<sup>2</sup> Connectors will vary with cylinder size. Confirm that your regulator will work with your gas tank. All connections to the GC/MS are 1/8 in. Swagelok fittings.

- Gas lines should be:
  - As short as possible and close to the system.
  - Made of copper or stainless steel when using helium, hydrogen, methane or isobutane.
  - Made of stainless steel when using ammonia or other corrosive gases.
  - Free of oil and moisture.
- Obtain the proper gas line filters, which help prevent impurities and contaminants from entering your system. Water, oxygen, and total hydrocarbons should be less than 1 ppm to avoid high background noise and prevent contamination. The GC is equipped with two intake filters that trap moisture, oxygen, and hydrocarbons.
- Store gas tanks and bottles properly so they will not damage cables or gas lines. Ensure they are secured in accordance with standard safety practices.

#### **Using Hydrogen**

To safely use hydrogen in your system, you should have a hydrogen sensor installed in your GC. Field Service Engineers can install a sensor. but they are not authorized to install or repair any instrument using hydrogen as a carrier gas unless the instrument is equipped with the appropriate sensor. The sensor must be calibrated occasionally, as described in the sensor's documentation.

Use the following safety precautions when using hydrogen:

- Ensure that all hydrogen cylinders comply with the safety requirements for proper use and storage. Hydrogen cylinders and delivery systems must comply with local regulations.
- Make sure the gas supply is turned completely off when connecting hydrogen lines.

- Ensure your GC has a Thermo Fisher Scientific hydrogen sensor installed. A hydrogen sensor continuously monitors the hydrogen level in the oven.
- Always turn off the GC and shut off the hydrogen at its source before venting the MS.
- If you use a hydrogen pump, ensure it has a palladium drier.
- Remove as many sources of ignition as possible from your laboratory. Sources can include open flames, electrostatic discharges, or devices that spark.
- Do not open a cylinder of hydrogen without a regulator attached because it may self-ignite.
# **GC Start Up**

This chapter describes how to start your GC after a period of instrument downtime.

#### Contents

- Powering On the Instrument
- TRACE 1300 LEDs
- TRACE 1310 Touch Screen
- Replacing the Column

## **Powering On the Instrument**

#### ✤ To power on the GC

1. If you removed the column from the inlet, re-install the column in the inlet at this time. See "Replacing the Column" on page 17 for more information. See Figure 3 to determine the column depth into the inlet.



Figure 3. Column Depth Reference

2. Turn on the gas supply at the tank for all gases configured. Typical output to the TRACE 1300 Series GC should be between 80-100 psig for each tank.

2

**Note** Customers with PDD detectors on their GC have carrier gas supplied through a getter. The connection from the getter to the GC needs to be removed from the back of the GC and purged prior to start up. See the Getter documentation for start up instructions. See "Purging the Helium Purifier (PDD Customers)" on page 37.

**Note** The maximum nominal inlet pressure for all the inputs is 1050 kPa (150 psig), as indicated on the label under the gas inlet ports on the back of the GC. The working inlet pressure range is from 400 kPa (58 psig) to 1050 kPa (150 psig).

3. Plug all external module power cables back into their respective AC inputs on the back of the GC and plug in the main power cable to the GC.



Figure 4. GC Power On



Figure 5. Aux Temperature/Cryo Module Installed into the GC

The module includes the following connections. See Figure 6.



6	A 2-pin connector marked <b>Back Inlet</b> for the connection of the solenoid valves for the back PTV/PTVBKF cryogenic system.
7	A 2-pin connector marked <b>Front Inlet</b> for the connection of the solenoid valves for the front PTV/PTVBKF cryogenic system.
8	A 2-pin connector marked <b>Oven</b> for the connection of the solenoid valves for the Oven cryogenic system.

4. With the column installed in the inlet, the gas supply turned on, and all power cables plugged in, proceed to turn the GC Power On: flip the Power Breaker Switch on the back of the GC to the On position (marked I). See Figure 4 for locations.

# **TRACE 1300 LEDs**

When the TRACE 1300 powers on, the LEDs on the status panel light up simultaneously, afterward, the POWER light becomes a solid green while all other lights turn off. The GC is now in stand-by status.

Figure 7. TRACE 1300 Status Panel at the GC Power On



# **TRACE 1310 Touch Screen**

When the TRACE 1310 GC powers on, the main menu is on the touch screen and the status reads Standby.



Figure 8. TRACE 1310 Touch Screen

# **Replacing the Column**

You may need to replace the column when your performance degrades and troubleshooting indicates that the column needs maintenance. This can mean that the end of the column needs to be trimmed, or the column needs to be replaced.

#### ✤ To replace a column

- 1. Remove the current column.
  - a. Press the **Maintenance** button to cool down the GC.
  - b. Set off the carrier and detector gases of the channel of interest, and wait for the carrier pressure to go to zero.
  - c. Open the front door of the GC.
  - d. Unscrew the injector and detector nuts, and remove the column.
  - e. Remove the column from the column rack, and from the GC.
- 2. Install the new column.
  - a. Place the new column on the two arms of the column rack inside the oven.

- 3. Connect the new column to the injector inside the GC.
  - a. Unwind the column enough to easily connect its ends to the injector and the detector.

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

- b. Wipe about 100 mm (4 in.) of the column with a tissue soaked in methanol.
- c. Insert the column through the proper injector retaining nut and ferrule (open 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.
- 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.

**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.

e. Position the column so that the end of the column extends the proper distance above the end of the ferrule as listed in Table 9.

Injector	Column Insertion Depth
SSL and HeS-S/SL	<ul> <li>5 mm (splitless)</li> <li>5 mm (He saver)</li> <li>10 mm (split)</li> </ul>
SSLBKF	<ul><li>5 mm (splitless)</li><li>10 mm (split)</li></ul>
PTV	<ul> <li>30 mm</li> <li>As far as possible into the bottom when the PTV is used as an On-Column injector.</li> </ul>
PTVBKF	• 30 mm
GSV	<ul> <li>Insert the column as far as goes and withdrawn about 2 -3 mm</li> </ul>

Table 9. Column Insertion Depth For SSL, SSLBKF, HeS-S/SL, PTV, PTVBKF, and GSV Injectors

f. Insert the notched septum on the column to hold the retaining nut at this position. Thread the retaining nut into the injector but do not tighten.

g. Adjust the column position so that the septum contact the bottom of the retaining nut.

h. Finger-tighten the retaining nut until it starts to grip the column plus a quarter turn.

i. Remove the notched septum from the column.

- 4. Setup the GC parameters.
  - a. Set the oven and injector temperature to 50 °C.
  - b. 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, contact Technical Support.
  - c. Allow the column to purge for at least 10 minutes.
- 5. Connect the column to the detector inside the GC.
  - a. Lower the oven temperature to 30 °C and allow it to cool.
  - b. Unwind the column enough to easily connect its ends to the injector and the detector.

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

- c. Wipe about 100 mm (4 in.) of the column with a tissue soaked in methanol.
- d. Use a scoring wafer to score and break the column outlet about 2.5 cm (1 in.) from the end. Use a magnifying glass to check for an even, flat cut. Repeat if necessary.
- e. Insert the column through the proper detector retaining nut and ferrule (open end up). Wipe the column again with a tissue soaked in methanol.

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

f. For FID, NPD, TCD, ECD, and FPD, position the column so that the end of the column extends the proper distance above the end of the ferrule as reported in Table 10. For PDD see the instruction described at the step g on page 20.

 Table 10.
 Column Insertion Depth For FID, NPD, TCD, ECD, FPD, and PDD Detectors

Detector	Column Insertion Depth
FID, NPD, and TCD	Insert the column as far as goes and withdrawn about 2 -3 mm
ECD	23 mm
FPD	125 mm
PDD	136 mm

i. For **FID**, **NPD**, and **TCD**, insert the column into the detector, paying attention to not force it further. Finger-tighten the retaining nut, then withdraw the column **2-3 mm**. Tighten the retaining nut an additional a quarter turn.

ii. For **ECD** and **FPD**, insert the notched septum on the column to hold the retaining nut in this position. Thread the retaining nut into the detector but do

not tighten. Adjust the column position so that the septum contact the bottom of the retaining nut. Finger-tighten the retaining nut until it starts to grip the column plus a quarter turn.

- iii. Remove the notched septum from the column.
- g. For **PDD** the column must enter **136 mm** into the pre-installed capillary column adapter.
  - i. Make a mark on the column 136 mm from the end.
  - ii. Remove the knurled nut column inlet at the bottom of the detector. Slide the nut overt the end of the column, followed by the appropriate column ferrule.
  - iii. Seat the ferrule in the detail of the column adapter and begin sliding the column through the capillary column adapter and into the column inlet.
  - iv. Get the nut started on the threads and tighten it until you feel it contact the ferrule, then back off half a turn.
  - v. Slide the column into the column inlet until the mark is flush with the surface of the knurled nut, and secure the column in the adapter by tightening the knurled nut finger tight only.

**Note** When inserting the capillary column into the PDD detector it might rarely happen to feel a slight resistance. In this case, for proper column installation, pull the column out slightly and adjust the angle before inserting it further.



**IMPORTANT** To install a packed column, the pre-installed capillary column adapter must be replaced with the **packed columns adapter** that enters into the PDD cell for the correct length.

6. Close the front door of the GC.

# **Software Configuration**

This chapter describes how to configure your GC and its components after a period of instrument downtime.

#### Contents

- Using the Touch Screen (TRACE 1310)
- Using Chromeleon
- Using Xcalibur
- Purging the Helium Purifier (PDD Customers)

## Using the Touch Screen (TRACE 1310)

If your GC has a touch screen, follow the instructions in this section to configure your GC and its components.

### **Turning on the Carrier Gas**

- To turn on the carrier gas using the touch screen
- 1. If you followed appropriate shut down procedures, it's likely the carrier gas will turn on when you power on the GC. However, this may not be the case. From the touch screen, select **Instrument Control**. See Figure 9.



Figure 9. Touch Screen Main Menu: Instrument Control

2. Select the inlet that has a column installed.

Figure 10. Instrument Control Menu

Back Inle Oven Front Inlet -Back Det FID Run Table Auxiliary Aux L Det. Aux R Det emperatures FID NPD 5) Valve Oven Aux. Carrier Valves

- 3. Depending on the mode you're in, you will either have the Column flow active or the Pressure active. If an active mode is listed as Off, turn it on and set an appropriate value for your column. See Figure 11.

Figure 11. Turning On Column Flow/Pressure



4. Once you verify **Column flow**, turn on the **Temperature** of your inlet from the same screen. Set the temperature to an appropriate value. See Figure 12.

**Figure 12.** Setting the Inlet Temperature





5. If you removed the column from your detectors/mass spectrometer, now is a good time to perform a leak check and column conditioning.

### Leak Checking and Column Conditioning

- ✤ To perform a column leak check.
- 1. Carefully push the capillary column end into the column section of the column-flowmeter connector. See Figure 13.

Figure 13. Using a Flowmeter for Leak Check



 Select the Leak Check icon in the Maintenance menu, otherwise perform the Leak Check through the Chromatography Data System by selecting the proper function. See Figure 14 and Figure 15.



Figure 14. Touch Screen Main Menu: Maintenance



Column Leak check									
Inlet for	leak check	Front -	<b>AND</b>	<b>B</b> C	<b>B</b> R				
Leak ch	eck press.	0.	Cool for	Make Log Entrv	View Logbook				
Allowed	pressure drop	0.	Ø		REAL				
		υ.	Leak Check	Quick	Video				
	Begin leak check		Select the in for leaks. P for small le ferrules or : enter an all for columns known pres characterist	hlet you wish lug the colum ak detection of septa. Alterna bwed pressur s with open e ssure drop ics.	to test in exit of atively, re drop xits and				
M	Standby	1	- °C	M 🎐	t 🏠				

- 3. Select Leak Check to begin the operation. The split and purge valves of the selected channel are automatically closed and the channel is pressurized with carrier gas to the leak check set point.
- 4. 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.
- 5. If the leak check does not pass, you should 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. We recommend that you check that fitting before looking elsewhere.

6. Repeat the leak check until no leaks are indicated.

- 7. For column conditioning, refer to the instructions provided by the manufacturer. General information can be found here:
  - Restek Column Conditioning: https://www.restek.com/Technical-Resources/Technical-Library/General-Interest/general\_GNAN1533-UNV
  - Agilent Column Conditioning: https://www.agilent.com/cs/library/eseminars/Public/Installation%20Care%20and% 20Maint%20GC%20Columns.pdf
  - Thermo Fisher Column Conditioning: https://assets.thermofisher.com/TFS-Assets/LSG/Application-Notes/TN-GC-Colum n-Installation-Maintenance-TN20684\_E.pdf
- 8. With your column installed in the inlet, the GC carrier gas on, and the column conditioned as you deem appropriate, re-install the column into the detector. See Figure 16 to determine column depth for the detector.

For column installation on a mass spectrometer see Chapter 5, "Mass Spectrometer Start Up."



Figure 16. Column Depth Quick Reference

### **Turning on a Detector**

#### ✤ To turn on a detector

1. Before turning on a detector, you will need to confirm that make-up gas (if any is present) is flowing. From the Main Menu select **Instrument Control**.

Figure 17. Touch Screen Main Menu: Instrument Control



2. Select the detector with a column installed.





3. In the **Detector** menu, if a make-up gas is present, make sure the gas is on and the flow is set. Typical make-up flows are shown in the following tables.

#### Table 11. FID Gases

Gas	Used As:	Range	Typical Condition
Nitrogen	Make-up gas (recommended)	1-50 mL/min	40 mL/min
Helium	Make-up gas	1-50 mL/min	40 mL/min

#### Table 12. NPD Gases

Gas	Used As:	Range	Typical Condition
Nitrogen	Make-up gas	1-50 mL/min	10-20 mL/min
Helium	Make-up gas	1-50 mL/min	10-20 mL/min

#### Table 13. ECD Gases

Gas	Used As:	Typical Condition
Nitrogen	Make-up gas (recommended)	15 mL/min
Argon/Methane	Make-up gas	15 mL/min

TCD–Reference Gas is coming from carrier gas. Make sure carrier gas is flowing and turn on reference gas. Typical condition is 1 mL/min.

4. With the column installed, carrier gas flowing, and any make-up gas turned on (if applicable), you can now turn on the detector. This will include turning on any remaining gases (H2, Air, etc) and the base/cell temperature.

See the following recommendations for each detector.

- a. FID With Make-up gas already turned on, Turn on H2, Air, and Temperature. Let temperatures / gas flows stabilize for 15 minutes then attempt to Turn the Flame On.
- b. TCD With reference gas already turned on, turn on TCD temperature, Filament temperature, and Filament Power.
- c. ECD With reference gas already turned on, turn on ECD temperature. Once at temperature, let stabilize for 4 hours or overnight.
- d. FPD Turn on Air, H2, Base Temperature, and Cell Temperature. Let stabilize for 2 hours before attempting to turn the Flame on.
- e. PDD Confirm Getter has been purged and is On, the inlet is hot, and the carrier gas and discharge gas are flowing. Turn on temperature
- f. NPD With reference gas already turned on, turn on H2, Air, and Temperature. Let stabilize for 1 hour.
  - i. Check that source is correctly switched on.
  - ii. Decrease hydrogen flow to 0.5 mL/min until the signal decreases to zero, then increase it again to its original value.

If the signal remains around zero, it means that the source is not switched on, and it is necessary to increase the current further according to the procedure just described.

If the signal rises back to original value, it means that source is correctly switched on.

iii. Increase the current value of 2% of the actual ignition current. Let the signal stabilizes until its level drops below 20 pA.



**IMPORTANT** Changes of gas flows and detector temperature affect the source current value required.

# **Using Chromeleon**

If your system is running Chromeleon software, follow the instructions in this section to configure your GC and its components.

### **Turning on the Carrier Gas**

- ✤ To turn on the carrier gas using Chromeleon
- 1. If you followed appropriate shut down procedures, it's likely the carrier gas will turn on when you power-on the GC. However, this may not be the case. Open the Chromeleon Console in the **Instrument Category** and choose the tab for your inlet.

Figure 19. Turning On Column Flow

Thermo Scientific G	CMS Home	Thermo TriPlus 500 San	npler Thermo T	nPlus RSH	Frontiniet <sup>77</sup> Back Inle	CVen Front Detector	BackDetector MSDevic
	- Status SSL						Column Functions
	Inlet type:	SSL			Actual	Setpoint	
	ll l		Carrier pressure:		0.0000 [bar]		Column Properties
Y			Column flow:	🔘 On	1.200 [ml/min]	1.200 [ml/min] 🚖	
	Gas saver:	🔘 0#	Purge flow:	Off Off	0.500 [ml/min]		Leak Check
SSI blat	Carrier mode:	FlowQtrl ~	Split flow:	🔘 Off	5.0 [ml/min]		
SSL met	Split mode:	Split $\vee$	Temperature:	i Off	0 ['C]		Column Evaluation

2. Depending on the mode you're in, you will either have the **Column flow** active or the **Pressure** active. If the active mode is listed as Off, turn it On and set an appropriate value for your column.

Use the toggle buttons. Blue signals On. You can type in the **Setpoint** box once active and press Enter to set the value. See Figure 19.

3. Once you confirm the **Column flow**, turn on the **Temperature** of your inlet from the same screen. Set the temperature to an appropriate value. See Figure 20.

Figure 20. Turning On Temperature

the same of the same of the	Status SSL									Column Functions
0	Inlet type:	SSL					Actual	Setpo	int	
	1			Carrier pressure:			0.0000 [bar]			Column Propertie
A. L				Column flow:	0	On	1.200 [ml/min]	1.200 [ml/m	n] 🖨	
	Gas saver:	🔘 0#		Purge flow:	0	Off	0.500 [ml/min]			Leak Check
	Carrier mode:	HowOtrl	~	Split flow:	0	Off	5.0 [ml/min]			
SSL INE	Split mode:	Split	~	Temperature:		On	250 ['C]	250 [°C]	÷	Column Evaluation

4. If you removed your column from your detectors/mass spectrometer, now is a good time to perform a leak check and column conditioning.

### Leak Checking and Column Conditioning

- ✤ To perform a column leak check.
- 1. Carefully push the capillary column end into the column section of the column-flowmeter connector. See Figure 21





2. In the Chromeleon Console, in the Instrument Category, choose the tab for the inlet where your column is installed. Select Leak Check. See Figure 22.

Figure 22. Leak Check

Thermo Scientific GCM	5 Home	Thermo TriPlus 500 San	mpler Thermo Ti	nPlus RSH	Front Inlet Back Inlet	t Oven Front Detector	r Back Detector MSD
	Status SSL						Column Functions
8	Inlet type:	SSL			Actual	Sctpoint	Column Properties
			Carrier pressure:		0.0000 [bar]		
			Column flow:	🔘 On	1.200 [ml/min]	1.200 [ml/min] 🗦	
	Gas saver:	🔘 O#	Purge flow:	) Off	0.500 [ml/min]		Leak Check
	Carrier mode:	FlowQtrl ~	Split flow:	🔘 Off	5.0 [ml/min]		
SSL Inlet	Split mode:	Split v	Temperature:	On (	250 ['C]	250 (°C) 🔶	Column Evaluation

- 3. Start the leak check to begin the operation. The split and purge valves of the selected channel are automatically closed and the channel is pressurized with carrier gas to the leak check set point.
- 4. 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.
- 5. If the leak check does not pass, you should use the leak detector to find and fix the leaks.

**Tip** Leaks can be caused by not tightening the fitting on the column flowmeter connector. We recommend that you check that fitting before looking elsewhere.

- 6. Repeat the leak check until no leaks are indicated.
- 7. For column conditioning, refer to the instructions provided by the manufacturer. General information can be found here:
  - Restek Column Conditioning: https://www.restek.com/Technical-Resources/Technical-Library/General-Interest/gen eral\_GNAN1533-UNV
  - Agilent Column Conditioning: https://www.agilent.com/cs/library/eseminars/Public/Installation%20Care%20and% 20Maint%20GC%20Columns.pdf
  - Thermo Fisher Column Conditioning: https://assets.thermofisher.com/TFS-Assets/LSG/Application-Notes/TN-GC-Colum n-Installation-Maintenance-TN20684\_E.pdf

If no column conditioning will be performed, go to the **Oven** tab and turn your oven on and set an appropriate temperature for Stand-by.

### **Turning on a Detector**

#### To turn on a detector

1. Before turning on a detector, you will need to confirm that make-up gas (if any is present) is flowing. From the Chromeleon Console, in the Instrument Category, select the detector tab where the column is installed. Turn on any make-up gas and enter appropriate set point. See Figure 23.

Figure 23. Make-Up Gas

Thermo Scientific GC	MS Home The	rmo TriPlus	500 Sampler Th	ermo TriPlue RSH	Frontiniet	Backiniet	Oven FrontDetector	Back
	Status FID							
	Detector type:	FID	т	emperature:	i off	Actual 0 ['C]	Selpoint	
-			FI	ane:	i off			
FID			н	ydrogen:	🔘 Off	1.0 [ml/min]		
			A	r:	🔘 Off	5.0 [ml/min]		
Signal			м	akeup gas:	On 🔘	40.0 [ml/min]	40.0 [ml/min] 🚔	
0.0000 (pA)								
Copy 📑 Report	V Filtering Expert	•	- 11,	Find Next 🏨 Fin	d Previous			
Date	Time	Retention	Device			Message		*
3/26/2020	8:38:31 AM -04:00		GC.FrontDetector	GC.FrontDetec	tor.MakeupFlow	Nominal - 40.0 n	nVmin	-
3/26/2020	8:38:28 AM -04:00		GC ErontDetector	GC FrontDeter	tor MakeunElow	Ctrl - On		-

Typical flows are shown in the following tables.

Table 14.	FID Gase	es
-----------	----------	----

Gas	Used As:	Range	Typical Condition
Nitrogen	Make-up gas (recommended)	1-50 mL/min	40 mL/min
Helium	Make-up gas	1-50 mL/min	40 mL/min

#### Table 15. NPD Gases

Gas	Used As:	Range	Typical Condition
Nitrogen	Make-up gas	1-50 mL/min	10-20 mL/min
Helium	Make-up gas	1-50 mL/min	10-20 mL/min

#### Table 16. ECD Gases

Gas	Used As:	Typical Condition
Nitrogen	Make-up gas (recommended)	15 mL/min
Argon/Methane	Make-up gas	15 mL/min

TCD–Reference gas is coming from carrier gas. Ensure carrier gas is flowing and turn on reference gas. Typical condition is 1 mL/min.

2. With the column installed, carrier gas flowing, and any make-up gas turned on (if applicable), you can now turn on the detector. This includes turning on any remaining gases (H2, Air, etc) and the base/cell temperature.

#### Figure 24. Front Detector

Thermo Scientific GCMS	6 Home Thermo TriPlus 500 Sampler	r 🔰 Thermo TriPlus RSH	Frontiniet	Back Inlet C	Yen Front Detector Ba
	Status FID				
				Actual	Setpoint
	Detector type: FID	Temperature:	🔘 On	250 [°C]	250 [°C]
		Flame:	i c <del>rr</del>		
FID		Hydrogen:	🔘 On	35.0 [ml/min]	35.0 [ml/min] 🖕
		Air:	🔘 On	350.0 [ml/min]	350.0 [ml/min] 🚔
Signal		Makeup gas:	🔘 On	40.0 [ml/min]	40.0 [ml/min] 🚔
[Aq] 0000.0					



	The	mo Scientifio GC1	4S Home The	rmo TriPlus	500 Sampler 1	Thermo TriPlus RSH	Frontiniet	Backiniet	Oven	FrontDetecto	BackDetector	MSDevice	Biter	Audt	Queue	
i.		-	Status TCD					Actual	Setpo	int	AS_GC_MS					×
		2				-	0.0	200.001	200.00	1 .	E 📰 GC	Proc	eties	Commande	ç	
			Detector type:	TCD		renperature:	Un Un	20010	20010		- 😅 BackCol	Prop.	with/		Value	-
		1		-							Backlot	ector Colu	mSource		Front	
			Polarity:	Positin	• •						- C FrontCol	mn Data	Collection	Rate	10 [Hz]	*
		TCD	Filament power:	On	$\sim$						😅 FrontDet	ector Deltz				
		100									E Frontinie	Dete	storType		TCD	
- 5	ian	4									- Inject Valve	Filen	ient		On	
				Aut	oZero						- MSDevice	Filen	entTempe	rature	250 [°C]	
1.0	000	00 lmVI				Beference cas	On	1.0 mi/mini	1.0 [m]/	min] 🜩	E-C TriPlus500	Firm	VELEVELBY	<u>n</u>	"Demo"	
							<b>•</b> • • •			1 2	H2Reul9hT 💭 -	Loca	aon		Back	
											🗄 🛤 System	NotH	eadyCaus	e		
A ID	20	Dent.	NZ Davies   Guest	r I	6	Deal New Jill Deal	Devidence				1	4 Peak	Width		Standard	
: 0	80	opy report	A Lettered Extern	•	• •	g rina nexa ye rina	Previous					Polar	nty.		Positive	
		Date	Time	Hetenbon	Device			Message			1	RefF	ow.Nomin	el	1.0 [ml/min]	
		206/2020	9.44.11 AM 04.00	lime	GC ParkDatasian	CC Real Dates	- Edward T	the second second	87.			Reff	ow.Value		1.0 [ml/min]	
1	1	3/20/20/20	0.44:11 AM -04.00		OC.beckDetector	GC. DECKDEIECK	a riiementi ei	riperature = 250				RefF	owCtrl		On	
2		3/26/2020	8:43:49 AM -04:00		GC.BackDetector	GC.BackDetects	r.Filament = (	Dn				RefG	ааТура		None	
3		3/26/2020	8:43:44 AM -04:00		GC BackDetector	GC BackDetect	r Temperatur	a Nominal - 200	°C		1	Rete	ntion		-	
4		3/26/2020	8:43:42 AM -04:00		GC BeckDetector	GC BackDetect	RefFlow No	minal = 1.0 ml/m	in			Sena	No		"Demo123"	
5		3/26/2020	8-43-39 AM -04-00		GC BackDetector	GC BackDetect	RefElenced	- On				Same				

See the following recommendations for each detector. (Some parameters are not shown on the ePanel and can only be changed or accessed by selecting F8 on your keyboard.

- a. FID With make-up gas already turned on, Turn on H2, Air, and Temperature. Let temperatures / gas flows stabilize for 15 minutes then attempt to Turn the Flame On.
- b. TCD With reference gas already turned on, turn on TCD temperature, Filament temperature, and Filament Power.
- c. ECD With reference gas already turned on, turn on ECD temperature. Once at temperature, let stabilize for 4 hours or overnight.
- d. FPD Turn on Air, H2, Base Temperature, and Cell Temperature. Let stabilize for 2 hours before attempting to turn the Flame on.
- e. PDD Confirm Getter has been purged and is On, the inlet is hot, and the carrier gas and discharge gas are flowing. Turn on temperature
- f. NPD With reference gas already turned on, turn on H2, Air, and Temperature. Let stabilize for 1 hour.
  - i. Check that source is correctly switched on.
  - ii. Decrease hydrogen flow to 0.5 mL/min until the signal decreases to zero, then increase it again to its original value.

If the signal remains around zero, it means that the source is not switched on, and it is necessary to increase the current further according to the procedure just described.

If the signal rises back to original value, it means that source is correctly switched on.

iii. Increase the current value of 2% of the actual ignition current. Let the signal stabilizes until its level drops below 20 pA.



**IMPORTANT** Changes of gas flows and detector temperature affect the source current value required.

# **Using Xcalibur**

If your system is running Xcalibur software, follow the instructions in this section to configure your GC and its components.

### **Turning on the Carrier Gas**

- ✤ To turn on the carrier gas using Xcalibur
- 1. If you followed appropriate shut down procedures, it's likely your carrier gas will turn on when you power on the GC. If this is not the case, from the Xcalibur roadmap, click **Instrument Setup**.

Figure 26. Instrument Setup



2. In the Instrument Setup, choose TRACE 1300 > Get Method from GC. See Figure 27.

Figure 27. Instrument Setup



 Check the Inlet tab to check if the gases are turned on. If not, turn on the carrier gas via Flow or Pressure and then choose Send Method to GC. You can also turn on the Temperature of the inlet at this time. See Figure 28.

Image: Untitled - Thermo Xcalibur Instru-           File         TRACE 1300           Help	nent Setup		
Send Method to GC Get Method from GC Flow Calculator Vapor Calculator Vapor Calculator Vapor Calculator Vapor Calculator Vapor Calculator Vapor Calculator Split flow: Split flow	Figure 1         Second state           Splt         ▼           200 °C         √           500 mL/min         33.3           1.00 min         1.00 min           re:         5.00 kPa           0.00 min         0.00 min           c         5.0 mL/min           c         0.00 min	FID (back)     Run Table       Carrier mode:     Constant Flow       Carrier flow     Image: Constant Flow       Flow:     Image: Constant Flow       Carrier flow:     Image: Constant Flow       Carrier options     Vacuum compensation:       Carrier gas saver:     Image: Constant Flow:       Gas saver flow:     2.00 mL/min	D (front) FID (back) Run Table Carrier mode: Constant Pressure Pressure:

Figure 28. Setting Flow and Temperature

**Note** If you did not remove the column from the MS, make sure Vacuum compensation is checked when you select Send Method to GC.

4. If you removed your column from your detectors/mass spectrometer, it is a good time to perform a leak check and column conditioning.

### Leak Checking and Column Conditioning

- To perform a column leak check.
- 1. Carefully push the capillary column end into the column section of the column-flowmeter connector. See Figure 29.

**Figure 29.** Using a Flowmeter for Leak Check



2. In the Xcalibur console select Leak Check from the General tab. See Figure 30.

Figure 30. Leak Check

eneror	
C status: Ready	
Vaiting for: Prep-run ke	Y
lun	
lapsed time:	60.00 min
Remaining time:	1.00 min
Churt .	
Start	stop
Diagnostics	Leak Check
Maintenance	Column Setup

- 3. Select Leak Check to begin operation. The split and purge valves of the selected channel are automatically closed and the channel is pressurized with carrier gas to the leak check set point.
- 4. 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.
- 5. If the leak check does not pass, you should 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. We recommend that you check that fitting before looking elsewhere.

- 6. Repeat the leak check until no leaks are indicated.
- 7. For column conditioning, refer to the instructions provided by the manufacturer. General information can be found here:
  - Restek Column Conditioning: https://www.restek.com/Technical-Resources/Technical-Library/General-Interest/gen eral\_GNAN1533-UNV
  - Agilent Column Conditioning: https://www.agilent.com/cs/library/eseminars/Public/Installation%20Care%20and% 20Maint%20GC%20Columns.pdf
  - Thermo Fisher Column Conditioning: https://assets.thermofisher.com/TFS-Assets/LSG/Application-Notes/TN-GC-Colum n-Installation-Maintenance-TN20684\_E.pdf
- With your column installed in the inlet, the GC carrier gas on, and the column conditioned as you deem appropriate, re-install the column into the detector. See Figure 31 to determine column depth in detector.

For column installation on a mass spectrometer see "Installing the Column" on page 51



#### Figure 31. Column Depth Quick Reference

### **Turning on a Detector**

#### To turn on a detector

- Before turning on a detector, you will need to confirm that make-up gas (if any is present) is flowing. In the Instrument Setup window, choose TRACE 1300 > Get Method from GC.
- 2. Look to see if the make-up gas (if any is present) is on. If not, turn it on and select Send Method to GC.

Figure 32. Make-Up Gas



See the following tables for make-up gas flows.

#### Table 17. FID Gases

Gas	Used As:	Range	Typical Condition
Nitrogen	Make-up gas (recommended)	1-50 mL/min	40 mL/min
Helium	Make-up gas	1-50 mL/min	40 mL/min

#### Table 18. NPD Gases

Gas	Used As:	Range	Typical Condition
Nitrogen	Make-up gas	1-50 mL/min	10-20 mL/min
Helium	Make-up gas	1-50 mL/min	10-20 mL/min

#### Table 19. ECD Gases

Gas	Used As:	Typical Condition
Nitrogen	Make-up gas (recommended)	15 mL/min
Argon/Methane	Make-up gas	15 mL/min

TCD–Reference gas is coming from carrier gas. Ensure carrier gas is flowing and turn on reference gas. Typical condition is 1 mL/min.

3. Before turning on the temperature of your detector, it's recommended to make sure the make-up gas is actually flowing and meeting set point.

You can check this in the Xcalibur console in the **Status** tab, and choosing **TRACE 1300 Series**, and then the **Flow** tab.

#### Figure 33. Flow Tab

3C status: Ready Waiting for: Prep-run key	,
Run	
Elapsed time:	60.00 min
Remaining time:	1.00 min
Start	Stop
Diagnostics	Leak Check
Maintenance	Column Setup

4. With the column installed, carrier gas flowing, and any make-up gas turned on (if applicable), you can now turn on the detector. This includes turning on any remaining gases (H2, Air, etc) and the base/cell temperature.

When you open Instrument Setup, it's recommended to choose TRACE 1300 > Get Method from GC before making any changes; this will ensure you do not overwrite anything already sent to the GC.

Once you choose **Get Method from GC**, make any changes necessary to turn on the detector and then select **Send Method to GC**.

See the following recommendations for each detector.

- a. FID With Make-up gas already turned on, Turn on H2, Air, and Temperature. Let temperatures / gas flows stabilize for 15 minutes then attempt to Turn the Flame On.
- b. TCD With reference gas already turned on, turn on TCD temperature, Filament temperature, and Filament Power.
- c. ECD With reference gas already turned on, turn on ECD temperature. Once at temperature, let stabilize for 4 hours or overnight.
- d. FPD Turn on Air, H2, Base Temperature, and Cell Temperature. Let stabilize for 2 hours before attempting to turn the Flame on.
- e. PDD Confirm Getter has been purged and is On, the inlet is hot, and the carrier gas and discharge gas are flowing. Turn on temperature
- f. NPD With reference gas already turned on, turn on H2, Air, and Temperature. Let stabilize for 1 hour.
  - i. Check that source is correctly switched on.
  - ii. Decrease hydrogen flow to 0.5 mL/min until the signal decreases to zero, then increase it again to its original value.

If the signal remains around zero, it means that the source is not switched on, and it is necessary to increase the current further according to the procedure just described.

If the signal rises back to original value, it means that source is correctly switched on.

iii. Increase the current value of 2% of the actual ignition current. Let the signal stabilizes until its level drops below 20 pA.



**IMPORTANT** Changes of gas flows and detector temperature affect the source current value required.

# **Purging the Helium Purifier (PDD Customers)**

This section contains instructions to for purging the helium purifier.

#### \* To purge the helium purifier

- 1. Remove the gas line going from the getter to the GC from the back of the GC.
- 2. Turn on the gas supply at the tank and let it flow through the getter and into the room with the getter off for 15-20 minutes.
- 3. With the flow from the tank low, but not completely off, reconnect the gas line to the back of the GC.
- 4. Turn on the GC and set the carrier gas to **On**.
- 5. Turn on the Temperature of the inlet.
- 6. Turn on the getter and allow it to heat up and stabilize before turning on the PDD.



**CAUTION** This product is not for use with oxygen - either pure oxygen or gases with a significant proportion of oxygen. The purifier's gettering alloy is pyrophoric at operating temperature. Use with significant amounts of oxygen can result in combustion of the material, potential damage to the surrounding area, and possible injury.

# **Autosampler Start Up**

This chapter describes how to start up your autosampler after a period of instrument downtime.

#### Contents

- TriPlus RSH Start Up
- TriPlus 300 HS Start Up
- TriPlus 500 HS Start Up
- AI 1310/AS 1310 Start Up

4

# **TriPlus RSH Start Up**

This section describes how to start the TriPlus RSH.



**IMPORTANT** Before starting the TriPlus RSH, make sure the sampler and all the modules are properly installed. A syringe of the appropriate type, or several syringes (Tools), named using the appropriate Method, must be installed in the TR Station or ATC Station, and positioned in the slot.

### How to Start the TriPlus RSH

- 1. Reconnect the cable to the connector marked **Power** at the back of the TriPlus RSH.
- Plug in the power cable of the power module and then switch ON the power module. The following Start Screen appears either on the handheld device or through the Virtual Terminal once the TriPlus establishes a connection with the control software. See Figure 34.

Figure 34. Example of Start Screen



If an error occurs during the start up process, the Start Screen shows a blinking **Envelope** in the **Status Line** as an indicator of a Pending Message, a Service Issue, or both. See Figure 35.

TriPlus RSH 🕅 🛱 😑 12:34 12:34 TriPlus RSH TriPlus RSH 12:34 DrawerTempCtrl 1 ÷ DrawerTempCtrl 1 Act. Temp.: 20.1 ℃ Act. Temp.: 20.8 °C HS1 2500 µL; NL: 65 mm Service Issues (1) t DrawerTempCtrl 1 ht 1 G: 23; PS: 5; Sc: 60 mm; Slot1 Act. Temp.: 20.1 Pending Messages (1) LS1 10 µL; NL: 57 mm Head ij. Service Issues (1) G: 265; PS: AS; Sc: 54 mm; Head Setup by: 30.0 ℃ SPME Cond Station 1 Pending Messages (1) Maintenance Head Act. Temp.: 30.1 °C; Standby: 30.0 °C Help Setup SPME1 Fiber Length: 10 mm by: 30.0 °C G: 23; Slot3 About Maintenance ▶ ) mm Traycooler 1 Shutdown Help Slot1: -Select About Shutdown Options Select

Figure 35. Indicating Messages

The envelope symbol indicates that a message pending or a **Service Issue** is found. When powered on, the TriPlus RSH is always in the **User access level.** In this level, the message symbol is visible in the status line, but you have no access to verify or acknowledge the error. Changing the Access Level to **Extended User**, you can see details of the messages by selecting each message line and pressing **Enter**.

See the example of Figure 36:





- The **Pending Message** in the example of shows that the reconnected **Traycooler 2** has not been taught yet.
- The message retrieved from the **Service Issue** provides self-explanatory options for resolving the problem. The Service Issue in the example shows that the Agitator is missing.

- 3. To retrieve a message complete the following steps:
  - a. Change the Access Level to Extended User. See "Changing Access Level" on page 45.
  - b. Select **Options** to access the pull-up menu for further information.
  - c. Select the menu item **Pending Message** to receive detailed information on the error message.

A common reason for an error is typically when a **BUS cable** is not connected from one active module to another active module. During the boot-up procedure, the presence of all active modules is verified. If one or more modules are not found, (or it is not possible to check functions such as **Homing**), an error is recorded. See the example in Figure 37.

When an error is occurs, check all connections, try to eliminate the source of the problem, then restart the TriPlus RSH.

The example in Figure 37 shows the resulting errors when the Vortexer cable is not connected, and the homing could not be performed. All the subsequent boot-up checks failed.

Figure 37. List Items of Pending Messages



The Pending Message accompanied by the **Exclamation Mark** warning symbol indicating that a module has not been completely configured. After resolving the Service Issues, the Pending Messages, or both, restart the system. Select **Maintenance | Check Configuration**. Activating this item the entire system is checked for any open issues. If all of them are resolved, the green status light will be displayed.

See also the information provided by the error message or consult the chapter **Troubleshooting** in the *TriPlus RSH Hardware Manual*.

### **Changing Access Level**

Using the **Virtual Handheld Controller** the Access levels can be changed by pressing the two characters **A** and **B** simultaneously at the keyboard. See Figure 38.





If the TriPlus RSH is equipped with a Handheld Controller, press the two **Menu Buttons** simultaneously (see Figure 38) to access the screen shown in Figure 39. The **User Access Level** is the default level.

Figure 39. Changing Access Level



#### ✤ To change access level

1. Select the required level and press **Enter** to activate it. The asterisk indicates the current active level.

# **TriPlus 300 HS Start Up**

- 1. Turn on any auxiliary gas at the tank if not already being delivered from the GC.
- 2. Plug in the power cables for the Arm and Main Unit.
- 3. Flip the Power Switch Arm

Figure 40. Autosampler Back View





# **TriPlus 500 HS Start Up**

- 1. Turn on any auxiliary gas at the tank.
- 2. Plug in the power cables to both the Arm and Main Unit.
- 3. Flip the Power Switch on both the Arm and the Main Unit in to the **On** position.










#### Figure 44. Electrical Interface

## AI 1310/AS 1310 Start Up

- 1. Plug in the Vdc power cable of the external portable power supply into the jack marked 24 Vdc on the back of the sampling unit. See Figure 45.
- 2. Connect the power cord of the external power supply to the mains outlet. The AI 1310/AS 1310 will automatically run the self-testing routine during which the following automatic checks and settings are carried out:
  - Alignment between AI 1310/AS 1310 and the GC injector
  - Acknowledgment of the installed sample tray
  - Calculation of the syringe zero

**Note** The self-test routine is automatically carried out every time that the safety door of the turret is closed.

The back section of the sampling unit includes the following components. See Figure 45.



Figure 45. Back of the Sampling Unit

- Jack marked 24 Vdc for the instrument power supply through the external portable feeder.
- 15-pin female connector (double density) marked TRAY for the communication between the sampling unit and the motor actuating the hub positioned on the supporting plate of the 105-position or 155-position sample tray.
- 9-pin female connector marked RS232 for communication between the sampler and the GC (connector marked AUTOSAMPLER) through a proper cable.
- 6-pin female connector marked GC for connecting the sampler to the GC (connector marked SAMPLER SIGNAL) through the proper cable.
- 4-pin male connector marked TWIN SYNC for the synchronism between two AI 1310/AS 1310 installed in Gemini configuration through the proper cable.

# 5

## **Mass Spectrometer Start Up**

This chapter describes how to start your mass spectrometer after a period of instrument downtime.

#### Contents

- Installing the Column
- Powering On the Instrument
- Tuning

### **Installing the Column**

Install the conditioned column on the MS side.

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

- 1. Lower the oven temperature and allow it to cool.
- 2. 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.

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

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

5. 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.

- 6. Insert the column into the measuring tool, which is in the MS Toolkit and the VPI MS Toolkit (See Figure 46), so that it is even with the lines at the end of the column. Figure 47 indicates proper positioning of the column in the tool for accurate measuring.
- 7. Use a scoring wafer to score and break the column. Use a magnifying glass to check for an even, flat cut. Repeat if necessary.
- 8. Use a 5/16 in. wrench to hold the column measuring tool steady.

**Figure 46.** Column Measuring Tool



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

Figure 47. Lining Up the Column in the Column Measuring Tool



12. 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 48.** Positioning the Septum



- 13. Pull the column back from the transfer line nut. Do not move the septum from its position on the column.
- 14. Loosen the transfer line nut from the column measuring tool.
- 15. 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.
- 16. Insert the column into the transfer line.



Figure 49. Inserting the Column into the Transfer Line

- 17. Tighten the transfer line nut until it is just secure enough so that you cannot move it.
- 18. Loosen the nut by turning it exactly 1 flat backward.
- 19. 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 50. Positioning the Septum



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

**Note** If you are using a SilTite ferrule, follow the instructions that come with SilTite ferrules. If you are using a graphite Vespel ferrule, it requires conditioning to ensure a leak-tight seal. See your instrument's spare parts guide for information about ordering ferrules.

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



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

d. Retighten the transfer line nut.

## **Powering On the Instrument**

- ✤ To power-on the mass spectrometer
- 1. Install the GC column. If you have not done so, see "Installing the Column" on page 51.
- 2. Be sure the GC is powered on and there is carrier gas flowing through the column into the mass spectrometer.



**INSTRUMENT DAMAGE:** If you are using hydrogen as a carrier gas, you must cool down and shut off the GC to prevent the buildup of hydrogen in the vacuum manifold. See the "Hydrogen Safety Precautions" on page xii of the Preface for more information.





- 3. Open the front door of the instrument and tighten the vent valve knob. Be sure not to pinch the o-ring. Then close the front door of the instrument.
- 4. Reach around the right side of the instrument and pull up on the power switch to power-on the instrument.



**WARNING FIRE HAZARD** If you are using hydrogen as a carrier gas do NOT reach over the top of the instrument to power it on. Instead, reach around the right side or go to the back of the instrument and flip up the power switch.

5. Check the LEDs on the front of the instrument to make sure it is ready for use.

- 6. Open the Dashboard on the computer.
- 7. Check the heater status by clicking the **Status** tab in the Dashboard. If the ion source is not set to the right temperature, click the **Instrument Control** button and change the temperature.

Figure 52. Changing Ion Source Temperature

Status Analyzer Power Maintenance	Instrument Control
TSQ Duo Status: Idle Actual Set-Point MS transferline 109 °C 230 °C temp.: 138 °C 310 °C	MS transfer line temp.: 150 °C Ion source temp.: 150 °C Filament selection: 1 • CI reagent gas flow: 0.0 mL/min
Vacuum: Not Ready Foreline pressure: \$54 mTorr Ion gauge pressure: Gauge not furned on Turbo-pump speed: 0 %	None Cl gas port A: None Vone None Vone
Collision gas on: No Clireagent gas flow: 0.0 mL/min 0.0 mL/min Instrument Control Shut Down	Send

- 8. Check the status of the vacuum in the **Status** tab of the Dashboard. Within 10 minutes of powering on the mass spectrometer, the vacuum should read OK.
- 9. Once the temperature of the Ion Source reaches a minimum of 150 °C and the Foreline pressure is less than 110 mTorr, you can open Manual Tune (TSQ) or Air & Water / Tune (ISQ) to make sure you can still see ions. With the spectra mode Air selected, click Start Scan.

Figure 53. ISQ Dashboard

ISQ Dashboard			
6	Auto Tune		
Ja.	View Tune Report		
8ª	Tune Types		
k	Air & Water / Tune		
	Instrument Control		
Shut Down			

10. Allow the system to stabilize for at least 4 hours before tuning the instrument or running samples.

**Note** You can run the instrument without stabilizing it, but you may have changes in the masses and intensities as the system equilibrates at the final temperature.

## Tuning

Tuning will improve the performance of your system. For optimum stability, you must wait until the power, vacuum, and heaters LEDs on the front of the instrument are a solid green. These lights indicate that the instrument has reached vacuum and that it is at the last set temperature. If the system has been powered off for a period of time (that is, a cold system), the system components take up to 4 hours to thermally stabilize after reaching the temperature set point.

### **Manual Tune**

You're looking for clear and defined peaks. You'll most likely see Water, Nitrogen, Oxygen, and Argon. If you see sharp jagged peaks, please re-seat your source.





Following a successful pump down, tune the instrument according to recommendations provided by your Service Engineer.

#### **El Tune**

Option 1 Clean Source<sup>1</sup> El Default Tune Start No Tune Results OK? Here Yes Dirty Source Daily Tune Check El Full Tune\* Option 2 No Tune Results OK? Tune Results OK? Yes Yes Full Diagnostics Diagnostics OK? Yes No Run Samples Call Technical Support at 800-532-4752

Figure 55. ISQ QD and ISQ LT EI Tune Flow Chart

Figure 56. TSQ Duo and TSQ Evo El Tune Flow Chart







## **Q Exactive GC and Exactive GC Start Up**

This chapter describes how to start your Q Exactive GC and Exactive GC after a period of instrument downtime.

#### Contents

- Installing the Column
- Powering On the Instrument

## **Installing the Column**

To install the column in the Q Exactive GC or Exactive GC system

1. Use the GC transfer line handle to slide the GC transfer line away from the MS transfer line.

Figure 58. Separating the GC and MS Transfer Lines



- 2. Remove the current column and nut if present.
- 3. 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.

Column Gap

**Figure 59.** Measuring the Column Length

4. Insert the new column through the GC transfer line.

Figure 60. Inserting the New Column through the GC Transfer Line



Carefully extend the column out the front to allow application of the nut and ferrule.
Figure 61. Extending the Column to the Front of the Instrument



Column

6. Insert the column through the nut and ferrule (flat side of the nut faces the MS).Figure 62. Inserting the Column through the Nut and Ferrule



7. Use a scoring wafer to score and then remove the last 10 mm of column to provide a clean, well-cut end.

8. Wipe the column with an alcohol soaked-wipe after inserting through the ferrule.

Figure 63. Scoring and Cutting the Column



9. Carefully push the column back into the GC transfer line while keeping the nut and ferrule on the column.



Figure 64. Proper Placement of the Column in the GC Transfer Line

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





- 11. Loosen the nut a quarter turn and push the column into the MS transfer line until it just touches the source plug.
- 12. Pull the column 0.5 to 1 mm away from the source plug and tighten the nut a half turn.

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

#### Figure 66. Tightening the Nut



13. Ensure the column and nut are correctly installed in the MS transfer line.

Figure 67. Correctly Installed Column in MS Transfer Line



14. Pull the GC transfer line handle to the left to close it.



Figure 68. Closed MS Transfer Line

### **Powering On the Instrument**

To start up the Q Exactive GC or Exactive GC mass spectrometer after it has been shut down (and vented), you need to do the following:

#### ✤ To start up the Q Exactive GC or Exactive GC system

- 1. Power on the GC and autosampler as described in Chapter 2 and Chapter 4.
- 2. Switch on data system and monitor as described in the manuals that came with them. Wait until the operating system of the computer is completely loaded.
- 3. To start the Tune software on the data system computer, choose **Start > Programs > Thermo Exactive Series > Tune**.

**Note** The data system must be running before you start up the instrument. The instrument will not operate until software is received from the data system.

- 4. Turn on the nitrogen flow at the tank, if it is off.
- 5. Make sure that the main power circuit breaker switch is in the Off (O) position and that the electronics service switch is in the Service Mode position.
- 6. Place the main power circuit breaker switch in the On (|) position. When you place the main power circuit breaker switch in the On (|) position, the forepump and the turbomolecular pumps are started. All LEDs on the mass spectrometer front panel are off.
- 7. Set the power to the GC to On after the mass spectrometer. This allows the GC to properly identify the auxiliary temperature module controlling the GC transfer line.

When the vacuum system is switched on, the following occurs:

- a. After the main switch is switched On, the pumps of the mass spectrometer are run up. To monitor the vacuum readings in the Tune software, it is necessary to switch on the electronics switch, too. The Pirani gauge monitors the pressure at the forepump. Within a short time, a significant pressure decrease must be observed. The quality of the vacuum can be estimated by means of the rotation speed of the TMPs (for example, 80% after 15 minutes).
- b. If the working pressure is not reached after a preset time, the complete system is switched off.

**Note** The vacuum control board triggers an alert in the Tune software when a vacuum failure has occurred.

c. The Ion Gauge 1 (IKR 251) is switched on only after the source TMP has exceeded 90% of its maximum rotation speed for five minutes. To extend its life time, this ion gauge is switched off automatically after 30 minutes.

**Note** For diagnostic purposes, the ion gauge can be switched on manually in the instrument status view of the Tune software.

If the pressure exceeds 1E-4 mbar for more than 10 seconds, the ion gauge is switched off. After five minutes, the ion gauge is switched on again. After three failed attempts, it is only possible to switch on Ion Gauge 1 manually in the Tune software.

d. The Ion Gauge 2 (IKR 270) is switched on only after both TMPs have exceeded 90% of their maximum rotation speed for five minutes. It is then used to monitor the vacuum in the Orbitrap analyzer.

If the pressure exceeds 1E-4 mbar for more than 10 seconds, the ion gauge is switched off. After five minutes, the ion gauge is switched on again. After three failed attempts, it is only possible to switch on Ion Gauge 2 manually in the Tune software.

- e. The Vacuum LED on the system panel turns green when all the following conditions are met:
  - In the Tune software, all LEDs are green. (The Ion Gauge 1 is allowed to be off).
  - Analyzer temperature is below 45 °C.
  - Both TMP frequencies have exceeded 90% of their maximum rotation speed.
- f. When the vacuum measured by the Ion Gauge 2 is better than 1E-8 mbar, the power supplies of the high voltage electronics and the capillary heater are switched on.

**Note** If both ion gauges are defective, it is not possible to switch on the RF voltage and the high voltages.

8. Allow the mass spectrometer to pump down for 5 minutes.

- 9. Place the electronics service switch in the Operating Mode position. When the electronics service switch is in the Operating Mode position, the following occurs:
  - a. Power is provided to all electronic boards. (The main RF voltage and transfer multipole RF voltage remain off.)
  - b. The internal computer reboots. After several seconds, the Status LED on the front panel turns yellow to indicate that the data system has started to establish a communication link.
  - c. After several more seconds, the Status LED turns green to indicate that the data system has established a communication link. Software for the operation of the instrument is then transferred from the data system to the instrument.
  - d. After approximately 3 minutes, the System LED turns yellow to indicate that the software transfer from the data system is complete.
- 10. Start the GC or autosampler (if present) as described in the manual that came with the GC or autosampler.