

# Long Term Shutdown Guide for IOMS Instruments

Welcome to the Thermo Scientific™ Long Term Shutdown Guide for the Thermo Scientific Triton Series TIMS, the Thermo Scientific Neptune Series MC-ICP-MS, the Thermo Scientific Ultra HR-IRMS, the Thermo Scientific Delta V Series IRMS, the Thermo Scientific Delta Ray IRIS, the Thermo Scientific 253 Plus and MAT 253 10 kV IRMS, the Thermo Scientific Element Series HR-ICP-MS / Element GD Series GD-MS, the Thermo Scientific DFS Magnetic Sector GC-HRMS, the Thermo Scientific ARGUS VI, HELIX MC *Plus* and HELIX SFT Noble Gas MS, and the Thermo Scientific Flash*Smart* Elemental Analyzer.

Under some circumstances, it might be necessary to shut down our supported mass spectrometers for a longer period. This includes systems and instruments related to the Thermo Fisher Scientific™ IOMS business unit.

Below, find a brief description of these processes and reference to the corresponding Operating Manuals. All safety guidelines are valid for this Shutdown Guide.

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A full description of the shutdown processes is provided in the supplied Operating Manuals. The Operating Manuals also provide safety guidelines for operation, which you must strictly follow.

- *Triton Series TIMS Operating Manual*
- *Neptune Plus Operating Manual*
- *Ultra High Resolution Isotope Ratio MS Operating Manual*
- *Delta V Series IRMS Operating Manual*
- *Delta Ray Series IRIS Operating Manual*
- *253 Plus 10 kV Isotope Ratio MS Operating Manual*
- *Element GD Operating Manual*
- *Element 2/Element XR ICP-MS Operating Manual*
- *DFS Operating Manual*
- *ARGUS VI Operating Manual*
- *HELIX MC Plus Operating Manual*
- *HELIX SFT Operating Manual*
- *FlashSmart Elemental Analyzer Operating Manual*

The Triton Series TIMS instruments can be switched to standby when you plan not to use the instrument for short periods (for example, over the weekend).

### Shutting down the System

#### ❖ To shut down the system

1. Switch off power by the red main power switch located at the rear side of the instrument.
2. Lock the analyzer gate valve manually.
3. Switch off the chiller.
4. Switch off compressed air.
5. Close the MC software and shut down the Host computer.

### Starting up the System

#### ❖ To start up the system

1. Switch on the chiller and check for leaks.
2. Switch on the compressed air supply and check for leaks.
3. Switch off the fuses F4, F7, F8, and F9 at the PD system control unit which is located behind the mid front panel.

This avoids startup peaks and protects the electronics.

4. Turn on the red main power switch (S1) located at the rear side of the instrument.
5. Switch on the fuses at the PD system control unit one by one:
  - Start with fuse F7 (LV electronics)
  - After five seconds, switch on F8 (HV electronics)
  - After five seconds, switch on F9 (Baking)
  - After five seconds, switch on F4 (frontend)
6. Switch on the host computer and start the MC software.
7. Wait until the green READY LED at the front panel is on.
8. Bake out the source head for at least four hours.



The gate valve is still mechanically closed. Open the gate valve manually when the source vacuum is below  $2 \times 10^{-7}$  mbar. The instrument is in Standby mode and can be used as usual.

To disconnect the Neptune Plus MS from the main voltage, shut down the system as described in this section.

## Shutting down the System

### ❖ To shut down the system

1. Set the instrument to standby mode as described in the *Neptune Plus Operating Manual*.
2. Switch off the connected external cooling systems as described in the respective *Operating Manuals*.
3. Secure the analyzer gate valve using an appropriate socket wrench to make sure that the vacuum system stays closed.
4. Vent the ESA module.
5. Switch off the power supply of the frontend computer.
6. Turn the switch for electronics to the Off position.  
The PD system Neptune ESA is switched off.
7. Turn the main switch to the Off position.  
The system is switched off.
8. Switch off the external controlling host computer.

The system is completely shut down and can now safely be disconnected from the main voltage.

## Starting up the System

### ❖ To start up the system

1. Turn the main switch to the On position.  
The system is switched on.
2. Switch on the connected external cooling systems as described in the respective *Operating Manuals*.
3. Turn the switch for electronics to the On position.  
The PD system Neptune ESA is switched on.  
The parts of the electronics system are automatically switched on one after another. You can hear the relays working.
4. Wait 15 seconds.  
The parts of the electronics system are automatically switched on one after another. You can hear the relays working.
5. Switch on the power supply of the frontend computer.
6. Restart the external controlling host computer.
7. Switch on the pumps.  
Wait until the vacuum pressure has dropped below about  $10^{-7}$  mbar.  
The pressure can be read off in the Instrument Status tab of the Diagnostic window.
8. Release the analyzer gate valve again using an appropriate socket wrench.
9. In the Watch Parameter window of the Tune software, click the switch **Analyzer Gate Open** to open the analyzer gate valve. The valve is open and the button shows **on** and is green.

The system is ready for operation.

## From Shutdown to Standby

This section describes the startup procedure for the instrument, that is the procedure to bring back the instrument to standby mode after it was shut down.

**Tip** The shutdown mode describes the mode when the instruments is *not fully vented*. The main circuit is switched off, but the analyzer gate valve is closed and the analyzer is not vented.

The instrument is in shutdown mode, when:

- the main circuit is switched off
- the analyzer gate valve is closed
- the analyzer region is not vented

**❖ To check before starting the system**

1. Check that all automatic fuses are on. The fuses are located at the front of the instrument.
2. Visually check that the vacuum system is closed (all flanges must be closed and tight).
3. Visually check the amplifier connection.
4. Check that the water chiller is running and the water flows.
5. Check the argon and compressed air supply.
6. Check that the host computer is switched on.

The starting conditions are checked.

**Shutting down the System****❖ To shut down the system**

1. Wait until the active data acquisition is complete.
2. Close the gate valve with Instrument Control and lock it manually.
3. Switch off both Dual Inlets with the toggle switches.
4. Stop first Emission and then switch off the HV in Instrument Control.
5. Put the main power switch at the rear instrument side into the Off position.

The instrument is switched off. Pumps will shut down. The analyzer gate valve is closed and the instrument shuts down without venting the analyzer.

6. Close all Qtegra applications (Instrument Control, Configurator and Qtegra LabBook) and switch off the host computer.
7. Switch off the chiller.
8. Switch off the compressed air supply.

**Starting up the System**

This section describes the startup procedure for the instrument, that is the procedure to set the instrument to standby mode after it was shut down.

Initial position:

- The main power switch is in the Off position.
- The analyzer gate valve is closed and manually locked.
- The analyzer region is not vented.

**❖ To start up the system**

1. Switch on the chiller and check for leaks.
2. Switch on the compressed air supply and check for leaks.
3. Put the main switch at the instrument rear side to the On position.
4. Press the **Vacuum** button at the front panel to start the turbo pumps.
5. Switch on the host computer and wait ten minutes.
6. Switch on the Dual Inlet left and wait five minutes.
7. Switch on the Dual Inlet right and wait five minutes.
8. Start Instrument Control and monitor the ESA and Source vacuum. Wait approximately four hours until the source vacuum reached the  $10^{-7}$  mbar region. Bake out the ESA and Source for four hours.

The instrument is now in standby mode, with HV and Emission off.



The analyzer gate valve is still manually closed. Open the gate valve manually when ESA and Source vacuum are below  $1 \times 10^{-8}$  mbar.

## Shutting down the System

The Delta V Series IRMS, the 253 Plus IRMS or the MAT 253 IRMS should only be shut down for maintenance as follows.

### ❖ To shut down the system

1. In Instrument Control or Acquisition of Isodat, click the **Ion source** button on the **Accessories** toolbar to switch off the ion source.
2. In the **MS State** panel of Isodat switch off all heaters.
3. Wait for approximately 30 minutes.
4. Close all needle valves.

If a Dual Inlet system is connected, close its inlets.

If a Reference Refill device is connected, close its valve.

If a liquid nitrogen (LN<sub>2</sub>) supply is connected (for a Microvolume, for example), close it.

5. Switch off the pumps at the Control panel.
6. Wait approximately 3–10 minutes for the system to vent.

The venting can be heard due to the rapidly decreasing revolution of the turbopumps.

7. Close the compressed air supply.
8. Shut down all peripherals that are powered and controlled by the IRMS.
9. Close Isodat and all other open programs.
10. Shut down the PC. It is assumed that the PC is powered by the IRMS.
11. Switch off the system by turning the main switch to the **O** position.



All devices and peripherals which are connected to the external power sockets lose their power supply.

12. Disconnect the IRMS from the power supply by unplugging it.

## Starting up the System

Start up the Delta V Series IRMS, the 253 Plus IRMS or the MAT 253 IRMS as follows.

### ❖ To start up the system

1. Make sure that compressed air is connected to the instrument.  
This is especially important if you operate a Dual Inlet system.
2. Check that the needle valve is closed.  
You can close the needle valve using the adjusting knob from the control panel.
3. Switch on the system by turning the main switch.
4. Switch on the computer.
5. Start Isodat.
6. Switch on the pumps at the Control panel.
7. In the **MS State** panel of Isodat, switch on all heaters you need.
8. Wait until the operating vacuum has been reached. Depending on the demands, this takes 24–48 hours:  
When the Vacuum LED at the front panel turns from yellow to green, the IRMS is theoretically ready for operation. However because the vacuum is still improving, wait until it becomes stable to obtain stable measurement results.
9. On the **Accessories** toolbar of Isodat (in Instrument Control or Acquisition), click the **Ion source** button to switch on the ion source.

## Starting High Voltage (10 kV) Operation on MAT 253 and 253 Plus



If the instrument has been exposed to atmospheric pressure for a longer time (the instrument has been vented for transport, or after replacing the ion source, for example), switching on the high voltage too quickly might result in arcing between the lenses in the ion source and in some cases damage the corresponding HV potential module(s).

### ❖ To start high voltage (10 kV) operation on the MAT 253 and the 253 Plus

1. Create a vacuum of about  $2 \times 10^{-7}$  mbar in the analyzer.
2. With the high voltage still switched off, let the instrument pump for additional 60 minutes.
3. Set the high voltage to only 3 kV. Turn on the high voltage.

At the same time, turn on the analyzer, the ion source, the needle valve heaters and – if present – the Dual Inlet heaters.

Open the VISC (the so called “sulfur window”) and all internal Dual Inlet valves to pump out impurities (no exposure to outside).

4. Let the instrument stay at 3 kV for 60 minutes.
5. Raise the high voltage to 5 kV.
6. Let the instrument stay for 12 hours (overnight, for example) under the following conditions: all heaters are on, all internal Dual Inlet valves are open, the needle valve is open, helium is flushing through the peripheral attached.
7. Raise the high voltage to 7 kV.
8. Wait 30 minutes.
9. Raise the high voltage to the final desired voltage (around 9.5 kV). Turn the heaters off and let the thermal condition of the instrument stabilize until the operating temperature is reached.

**Tip** Cooling to operating temperature and temperature stabilization take about 5 hours.

10. Check the background (Argon  $m/z$  40: > 10 mbar on the middle cup).



If the high voltage shuts off under whatever condition (not only those mentioned above), do not try to turn it on again immediately. Instead, wait 10–15 minutes before you try to turn it on again.

Delta Ray IRIS  
(with URI  
Connect)

## Setting the System to Standby

The Delta Ray Series Isotope Ratio Infrared Spectrometers should not be shut down if you are not going to use it for a short time, such as overnight, over a weekend, or a few days. In this case, you can leave the system in standby mode. The standby mode is automatically activated after all scheduled LabBooks are finished.

### ❖ To set the system to standby

1. On the Home Page in the Qtegra window, click **Dashboard** to display the Dashboard page.
2. Click the **Gas Control** button in the toolbar to display the Gas Control window.
3. Select **StandBy** from the pulldown menu at Port.

The standby mode stops reference gas flows and carrier gas flows to avoid unnecessary consumption. It opens the Sample B port, and the Analyzer draws gas from this source.

## Shutting down the System

The Delta Ray Series Isotope Ratio Infrared Spectrometers should not be shut down if you are not going to use it for a short time, such as overnight, over a weekend, or a few days.

Take into account that each time the system returns from a shutdown, it takes up to 30 minutes to stabilize the internal temperature control for proper operating conditions. This section describes how to shut down the system for a relocation, maintenance, or a service procedure.

### ❖ To shut down the system

1. Shut down the Qtegra ISDS Software.
2. Press the two power buttons at the front panels of URI and Analyzer.

The systems perform a controlled shutdown of the Microsoft™ Windows™ operating systems.

Wait until the two LED bars of both instrument components are switched off. During the shutdown procedure of the operating systems that might take up to 60 seconds.

3. Place the mains switches at the rear sides of URI and Analyzer into the Off position. Place the mains switch of the external pump into the Off position.
4. Close the pressure regulators of all gas bottles.
5. Switch off the peripheral devices.

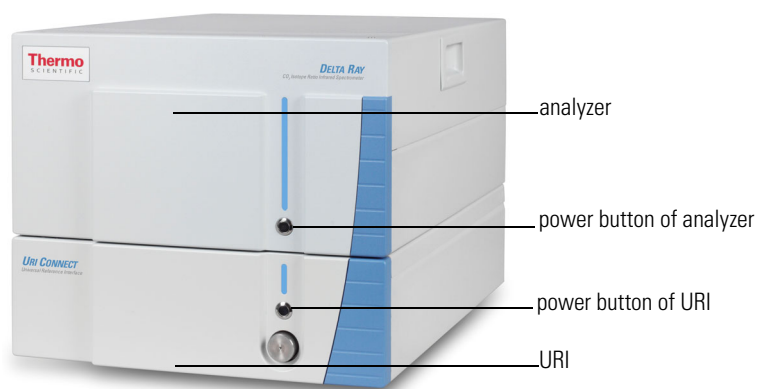
Although all valves are normally closed to avoid gas loss on power failures, this is recommended to save your gas supplies in case of small leaks.

## Starting up the System

To start up the Delta Ray Series spectrometer after it has been shut down, perform the following steps:

### ❖ To start up the system

1. Switch on analyzer, Universal Reference Interface (URI), external pump, and computer monitor with their mains switches. The mains switches of analyzer and Universal Reference Interface are located at the rear sides. The mains switch of the external pump is located at its front side.
2. Start the internal computers of analyzer and URI by pushing the power buttons at the front panels. See [Figure 1](#).



**Figure 1.** Delta Ray IRIS – front view

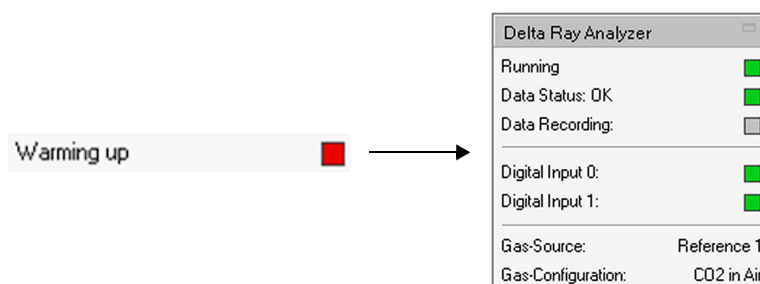
3. Wait until the internal computer of the URI is started.  
In the Log On to Windows dialog box, choose the User “**Thermo**” and enter “**optiso**” (without quotation marks) as password. Click **<Enter>** to continue.
4. The Qtegra software starts up automatically after the login<sup>1</sup>. Wait until the Qtegra window appears with the Dashboard displayed.

The Qtegra window contains all functions offered by Qtegra and gives access to all existing Templates and LabBooks. The Dashboard page shows on one page the instrument controls and main settings of the Delta Ray Series instrument.

5. Wait until the red Warming up LED is replaced by a green Running LED. See [Figure 2](#).

Depending on the actual ambient temperature, it requires up to 30 minutes for the analyzer to reach thermal stability.

<sup>1</sup> If the Qtegra software is not configured to start automatically after login, click the **Qtegra** button on the desktop to start Qtegra.



**Figure 2.** Warming up

If the system is not operated for a longer period, it can be switched off and vented.

To shut down the systems, for example for annual maintenance of the power system, change the status into standby mode first.

### Setting the System to Standby

In case of the **HR-ICP-MS** instrument:

1. Open the **Instrument** window.
2. Click **STOP** to stop the plasma.

An automatic sequence will be initiated.

In case of the **HR-GD-MS** instrument:

1. Click the **GD HV off** button in the Tune window.
2. Leave the argon supply open.
3. Leave the chiller on.

### Shutting down the System

#### ❖ To shut down the HR-ICP-MS or the HR-GD-MS instrument

1. Turn off the vacuum system using the key switch at the LED front panel.
2. Wait 10 minutes to let the system vent in a controlled way.
3. Turn off the electronic switch S2.

**Tip** Close the exhaust lines (especially the plasma exhaust line) when you switch off the Element2/Element XR ICP-MS or the Element GD MS.

4. To fully shut down the instrument, switch S1 to the position **Off** on the rear side of the instrument.

### Starting up the System

#### ❖ To start up the system

1. Switch on the power switch S1.

The main power for the instrument is switched on.

2. Turn the key-operated switch to the position **On**.

The backing pump and the four turbo pumps are started.

3. Switch on the power switch S2.

The instrument is in standby position again when a High Vacuum ( $5 \times 10^{-7}$  mbar) is reached. This takes several hours if argon was used for venting and up to 24 hours when humid air was used and the vacuum components were opened.



If it is possible, the mass spectrometer should not even be switched off for a weekend or for longer vacation periods. Only if you expect interruptions of the mains power and water supply, it is advisable to switch off the instrument.

## Shutting down the System

### ❖ To shut down the system

1. Cool the ion source, the GC oven, the injector and the auxiliary line.

The maximum temperature for the ion source is 100 °C when the instrument is vented with an inert gas such as argon or nitrogen (attached to the vent port at the rear side of the instrument).

If no vent gas is used and the instrument is vented by ambient air, the ion source temperature should be below 100 °C.

2. On the system control panel, press both **Pumps** buttons.

The LEDs above the Analyzer and Source button turn from green to off. The separation valve is closed by the instrument. The vacuum region is automatically vented when the rotation of the turbo pumps is slow enough. This takes approximately 15 minutes.

3. Switch off the GCs by the main power switch at the rear side of the instrument.
4. Stop the cooling water circuit.
5. Close the nitrogen gas supply and the compressed air supply of the mass spectrometer. Close the helium supply of the GCs.

## Setting the System to Standby

Because a longer period of pumping and warm-up is necessary to obtain stable vacuum conditions, the mass spectrometer should not be switched off over night or for a weekend.

The instrument can be set to standby position using the DFS Tune software.

### ❖ To set the system to standby

1. On the pull-down menu **Control** of the instrument control software, click **Standby**.

The software changes the following settings:

- The protected electronics remains switched on (power to the boards).
- The conversion dynode is switched off.
- The multiplier is switched off.
- The accelerating voltage is switched off.
- The emission current is switched off.
- The separating valve remains open.
- The temperature of the reference inlet system stays at the value it was set to (it is not affected by the standby).
- The temperature of the ion source stays at the defined value (operating temperature).

It is saved in the tune file.

2. The GC oven can be set to a low temperature and the helium flow to a minimum (0.3 mL/min, for example).
3. The auxiliary lines and the injector should be kept on temperature.

The status button will change to the Standby button when the software has completed the procedure.

## Starting up the System

### ❖ To start up the system

1. Open both gas supplies (helium for the GC and compressed air for the DFS).
2. Turn the main power switch to the **On (I)** position.
3. Turn on the main power switch for the GC located at the rear side.
4. Adjust the helium flow.
5. Set the correct temperature for the injector, the oven and the aux transfer line.
6. After two hours of heating up, check the GC for leaks.

7. To start the vacuum system, press the two buttons **Pumps Source** and **Pumps Analyzer** located at the right lower side of the Control Panel.

The roughing pumps of the DFS should start pumping and the LEDs above the buttons turn orange.

8. Stay with the instrument until the LEDs above the two Pumps buttons turn green.
9. When the vacuum LEDs turn from orange to green, let the instrument pump for at least 12 additional hours.
10. Start the computer and the DFS control software.

Starting the DFS via Xcalibur: the DFS and its peripherals, such as GC and Autosampler, need to be configured in the instrument configuration before.

11. In the instrument control software, turn the instrument from “standby” to “on”.
12. Load your last Tune file.

**Tip** The ion source temperature is also saved in the Tune file.

13. When the ion source temperature is stabilized, you are back to normal condition.
14. It is recommended to check for leaks (by looking at the argon level, for example).

## Starting up the Internal Computer

### ❖ To start up the internal computer

In case of communication problems between the instrument PC (Front End PC) and the computer, it is recommended to restart the instrument PC and the DFS Tune software.

1. Close all Xcalibur™ programs. Close the DFS Tune window.
2. At the power column at the rear side of the DFS instrument, switch off the fuse F1.
3. Wait for approximately 20 seconds.
4. Switch on the fuse F1 again.
5. Wait for another 60 seconds.
6. Start the Xcalibur Home Page again.

This procedure establishes a new connection between the Xcalibur data system and the Front End computer of the DFS mass spectrometer.

## ARGUS VI

Shutting down the system is only necessary to perform maintenance operations.

For most maintenance operations, it is sufficient to shut down the system without venting it (working at the turbomolecular pump, at the fore vacuum pump or at the electronics, for example). Some maintenance operations (replacing a filament or a multiplier, for example) however, require venting the system before you can shut it down.

In addition, the mass spectrometer can be placed in a Standby condition, if the system is not to be used for 12 or more hours.

In a Noble Gas mass spectrometer, the most important aspects of the system that need to be protected when shutting down are the integrity of the vacuum and the filament.

## Shutting down the System

### ❖ To shut down the system

#### **NOTICE**

If the vacuum fails and the filament is switched on, this could cause irreparable damage to the ion source, which would require disassembly, cleaning and rebuild.

1. Save the source tunings if required.
2. Switch off the source electronics by clicking the **Source/HV** button in the top left corner of the Instrument Control.
3. Switch off the ion gauge either via the Instrument Control or via the controller at the front panel of the instrument.
4. Ensure that all the automatic and manual valves fitted to the system are closed. Vacuum and ion source are now protected.

5. Power down the ion pump via Instrument Control.
6. Switch off the computer.
7. At the front panel, switch off the turbomolecular pump.
8. Isolate the main power to the instrument by using the mains isolation switch.

## Setting the System to Standby

There are two standby configurations: the first when the instrument is not to be used longer than one week, and the other for a period shorter than one week.

The main difference is whether the ion source is left powered on or not. The reason for these two options is due to the settling time required once the ion source has been turned on from cold until a stable system can be used.

### ❖ To set the system to standby if the instrument is not to be used for *more* than one week

1. Switch off the source electronics by clicking the **Source/HV** button in the top left corner of the Instrument Control.
2. Ensure that the turbomolecular pump is isolated from the mass spectrometer and that the system is pumped via the ion pump.

### ❖ To set the system to standby if the instrument is not to be used for *less* than one week

1. At the ARGUS VI Controls panel, turn high voltage to 0 V, but leave the filament/trap current on.  
You can turn down the trap current slightly to lower the filament current. This reduces the stress on the filament. Doing so increases the settling time before the instrument is ready for analysis, but it is much shorter than switching off the filament.
2. Ensure that the turbomolecular pump is isolated from the mass spectrometer and that the system is pumped via the ion pump.

## Starting up the System

The procedure of starting up the system after it was shut down depends on whether the system vacuum was affected or not.

**Tip** All valves of the system should be in standby position. Do not touch them until the integrity of the vacuum has been assessed.

### ❖ To start up the system if the vacuum *was not* affected

1. Turn on the mains isolation switch of the system.
2. Switch on the computer.
3. Start the Instrument Control of Qtegra.
4. Power up the turbomolecular pump.
5. Power up the ion pump.
6. Turn on the ion gauge and measure the vacuum level.  
If the vacuum is below  $1 \times 10^{-6}$  mbar, open the isolation valve to the ion pump or to the turbomolecular pump and allow the pressure to stabilize.
7. Once the vacuum in the mass spectrometer has been verified and is below  $1 \times 10^{-6}$  mbar, the source can be switched on.  
If the pressure exceeds  $1 \times 10^{-6}$  mbar, the instrument requires baking and/or leak checking.
8. Power up the source electronics by clicking the **Source/HV** button in the top left corner of the Instrument Control.
9. Load the appropriate source tuning file from the database.
10. Perform a background scan.

### ❖ To start up the system if the vacuum *was* affected

1. Turn on the mains support switch of the system.
2. Switch on the computer.
3. Start the Instrument Control of Qtegra.
4. Open the turbomolecular pump isolation valve to the mass spectrometer.

5. Turn on the turbomolecular pump ensuring all the valves on the mass spectrometer are open whether they are manual or automated.
6. Monitor the rotational speed of the turbomolecular pump until full speed indicated by 1500 Hz is reached. If it does not reach the necessary 1500 Hz, a significant leak exists that must be located and rectified.
7. After a period of 10 minutes at 1500 Hz, turn on the ion gauge. Monitor the pressure and check for any leaks using Analar grade acetone.
8. If the mass spectrometer has been exposed to atmosphere, water vapor will probably be in the system. Therefore, the system needs to be baked before you can turn on the ion source.
9. Once the bakeout procedure has been completed and the pressure is less than  $1 \times 10^{-6}$  mbar, the ion source can be switched on.
10. Turn the ion pump on.
11. Isolate the ion pump by closing the manual or automated ion pump valve.
12. Power up the source electronics by clicking the **Source/HV** button in the top left corner of the Instrument Control.
13. In steps of 20  $\mu$ A and intervals of 10 minutes, slowly increase the trap current until operational values. This is to prevent excessive stress on the filament.
14. Load the appropriate source tuning file from the database.
15. Perform a background scan.
16. Once the ion source has degassed into the turbomolecular pump, open the mass spectrometer ion pump valve and close the turbomolecular pump valve.

## HELIX MC Plus

Shutting down the system is only necessary to perform maintenance operations.

For most maintenance operations, it is sufficient to shut down the system **without venting** it (working at the turbomolecular pump, at the fore vacuum pump or at the electronics, for example). Some maintenance operations (replacing a filament or a multiplier, for example) however, require **venting** the system before you can shut it down.

In addition, the mass spectrometer can be set in a Standby condition, if the system is not to be used for 12 or more hours.

In a Noble Gas mass spectrometer, the most important aspects of the system that need to be protected when shutting down are the integrity of the vacuum and the filament.

### Shutting down the System under Vacuum

#### ❖ To shut down the *not vented* system

1. In Instrument Control, switch off high voltage. Thereby, the filament is automatically switched off, too.
2. Close the manual 1.5 in. UHV all metal valve towards the turbomolecular pump.
3. Close the manual 0.75 in. UHV all metal valve on the collector side and on the inlet side.
4. In Instrument Control, close the automatic pneumatic VAT valve.
5. At the front panel, switch off the turbomolecular pump.
6. In Instrument Control, switch off the ion getter pump.
7. Turn the main power switch to the position **Off**.

All valves of the mass spectrometer and the NG PREP SYSTEM (if available) are automatically closed.

8. You can work on the turbomolecular pump or on the fore vacuum pump. Afterwards, start up the system.

### Shutting down and Venting the System

#### ❖ To shut down a system before venting

1. In Instrument Control, switch off high voltage. Thereby, the filament is automatically switched off, too.
2. Wait for 30 minutes to let the filament cool.
3. Close the valves towards the turbomolecular pump, towards the ion getter pump and towards the chemical getter pumps. This minimizes the risk of air infiltration.

4. Shut down the turbomolecular pump.

Thereby, its fore vacuum pump is automatically switched off, too. The other fore vacuum pump that is used for pumping off the amplifier housing keeps running.

5. Shut down the ion getter pump in Instrument Control.
6. Close the automatic pneumatic VAT valve.
7. Turn the main power switch to the Off position to shut down the system.

Thereby, the fore vacuum pump that is used for pumping off the amplifier housing is automatically switched off, too.

You can now vent the analyzer to perform maintenance operations.

### Setting the System to Standby

After measurements are finished or when you plan not to use the instrument for some time (in the evening or before a weekend, for example), you can set the instrument to Standby mode.

#### ❖ To set the system to standby

1. Pump down all the samples.
2. Set the valves to standby position.
3. Decrease the trap current or set it even to 0  $\mu\text{A}$ .
4. Switch off high voltage.

Thereby, the filament and the power supplies of the CDDs are automatically switched off, too. The CDDs can however, also be switched off separately in Instrument Control.

### Starting up the System

The procedure of starting up the system after it was shut down depends on whether the system vacuum was affected or not.

#### ❖ To start up the system if the vacuum was not affected

1. Turn the main power switch to the position **On**.
2. Make sure that the manual 1.5 in. UHV all metal valve towards the turbomolecular pump is closed.
3. Switch on the ion getter pump.
4. Start the ion gauge and check the pressure.
5. If the pressure is in the range of  $10^{-7}$  mbar or below, open the automatic pneumatic VAT valve towards the analyzer.
6. At the ion gauge, check whether a pressure below  $10^{-8}$  mbar is reached.

#### ❖ To start up the system if the vacuum was affected

1. Turn the main power switch to the position **On**.
2. Make sure that all valves towards the vented area are open so that the turbomolecular pump can act successfully.

**Tip** If you inadvertently leave some valves closed, the enclosed air could enter the system later. However, do not open valves to areas that are still evacuated if it is not necessary. This unloads both fore vacuum pump and turbomolecular pump and keeps the degree of impurities low.

3. Start the turbomolecular pump by pressing the switch of its controller at the front panel.

The turbomolecular pump will run up. The fore vacuum pump, however, is always switched on and therefore starts automatically.

4. Wait for the turbomolecular pump to reach a frequency of 1500 Hz.
5. Set the ion gauge to the pressure range **MV**. Check whether the pressure decreases.
6. Check for leaks.
7. Bake out the system to remove moisture.

## Shutting down the System

Shutting down the system is only necessary to perform maintenance operations.

For most maintenance operations, it is sufficient to shut down the system **without venting** it (working at the turbomolecular pump, at the fore vacuum pump or at the electronics, for example).

Some maintenance operations (replacing a filament or a multiplier, for example) however, require venting the system before you can shut it down.

In addition, the mass spectrometer can be set to a Standby condition, if the system is not to be used for 12 or more hours.

In a Noble Gas mass spectrometer, the most important aspects of the system that need to be protected when shutting down the instrument are the integrity of the vacuum and the filament.

### **NOTICE**

If the vacuum fails and the filament is switched on, this could cause irreparable damage to the ion source, which would require disassembly, cleaning and rebuild.

#### ❖ To shut down the system

1. Save the source tunings if required.
2. In Instrument Control, click **Source/HV** to switch off the source electronics.
3. Switch off the ion gauge either via Instrument Control or via the controller at the front panel of the instrument.
4. Ensure that all the automatic and manual valves fitted to the system are closed.

Vacuum and ion source are now protected. Shut down the rest of the system.

5. Power down the ion pump via Instrument Control.
6. Switch off the computer.
7. At the front panel, switch off the turbomolecular pump.
8. Isolate the main power to the instrument with the mains isolation switch.

## Setting the System to Standby

Depending on the duration of the standby mode, two procedures can be performed.

The main difference is whether the ion source is left powered on or not. The reason for these options is the settling time that is required after the ion source has been turned on from cold until a stable system can be utilized.

#### ❖ To set the system to standby if it is not to be used for **more than one week**

1. In Instrument Control, click **Source/HV** to switch off the source electronics.
2. Ensure that the turbomolecular pump is isolated from the mass spectrometer and that the system is pumped via the ion pump.

#### ❖ To set the system to standby if it is not to be used for **less than one week**

1. At the HELIX SFT Controls panel, set the high voltage to 0 V, but leave the filament/trap current on.

You can turn down the trap current slightly to lower the filament current. This reduces the stress on the filament. This increases the settling time before the instrument is ready for analysis, but it is much shorter than switching off the filament.

2. Ensure that the turbomolecular pump is isolated from the mass spectrometer and that the system is pumped via the ion pump.

## Starting up the System

The procedure of starting up the system after it was shut down depends on whether the system vacuum was affected or not.

#### ❖ To start up the system if the vacuum **was not** affected

1. Turn on the mains isolation switch.
2. Switch on the computer.
3. Start the Instrument Control of Qtegra.

4. Power up the turbomolecular pump.
5. Power up the ion pump.
6. Turn on the ion gauge and measure the vacuum level.  
If the vacuum is below  $1 \times 10^{-6}$  mbar, open the isolation valve to the ion pump or to the turbomolecular pump and allow the pressure to stabilize.
7. After the vacuum in the mass spectrometer has been verified and is below  $1 \times 10^{-6}$  mbar, the source can be switched on.  
However, if the pressure exceeds  $1 \times 10^{-6}$  mbar, the instrument requires baking and/or leak checking.
8. In Instrument Control, click **Source/HV** to power up the source electronics.
9. Load the appropriate source tuning file from the database.
10. Perform a background scan.

❖ **To start up the system if the vacuum *was* affected**

1. Turn on the mains support switch.
2. Switch on the computer.
3. Start the Instrument Control of Qtegra.
4. Open the turbomolecular pump isolation valve to the mass spectrometer.
5. Turn on the turbomolecular pump ensuring all valves on the mass spectrometer are open.
6. Monitor the rotational speed of the turbomolecular pump until full speed (1500 Hz) is reached.  
If it does not reach 1500 Hz, a significant leak exists that must be located and rectified.
7. After a period of 10 minutes at 1500 Hz, turn on the ion gauge. Monitor the pressure and check for any leaks with Analar grade acetone.
8. If the mass spectrometer has been exposed to atmosphere, water vapor will probably be in the system.  
Therefore, the system needs to be baked out before you can turn on the ion source.
9. After the bakeout procedure has been completed and the pressure is less than  $1 \times 10^{-6}$  mbar, the ion source can be switched on.
10. Turn on the ion pump.
11. Isolate the ion pump by closing the manual or automated ion pump valve.
12. In Instrument Control, click **Source/HV** to power up the source electronics.
13. In steps of 20  $\mu$ A and intervals of 10 minutes, slowly increase the trap current until operational values.  
This is to prevent excessive stress on the filament.
14. Load the appropriate source tuning file from the database.
15. Perform a background scan.
16. After the ion source has degassed into the turbomolecular pump, open the mass spectrometer ion pump valve and close the turbomolecular pump valve.

To obtain precise, accurate and constant time results, and at the same time to reduce operating costs, Thermo Fisher Scientific recommends following some practical suggestions. To reduce the temperatures of the furnaces, oven, TCD detector, and gas flow, refer to the *Operating Manual*.

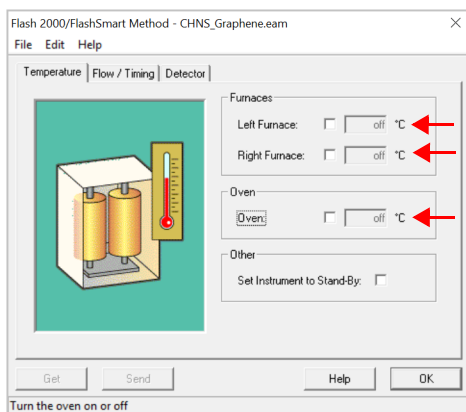
### Shutting down the System

The shutdown of furnaces and detector and the cutoff of gas flows can be performed **manually** or **programmed** as described in this section.

**Tip** The procedure described below allows protecting the functionality of the reactor(s), since the gas flow is removed only at environmental temperature. In this mode, the flow of gas is maintained up to the end, avoiding any oxidation/deterioration of the consumables inside the reactor(s).

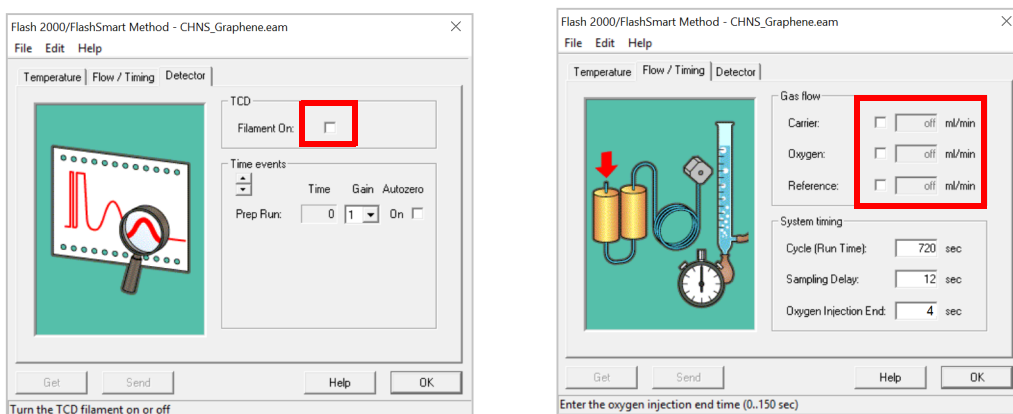
❖ **To *manually* shut down temperature, detector, and gas**

1. According to the configuration of the FlashSmart Elemental Analyzer in the EagerSmart Data Handling software switch off temperature(s) of the reactor(s) and oven.



**Figure 3.** Edit Elemental Analyzer parameters

1. After the “Actual temperature” shown in the FlashSmart Status of the EagerSmart software reaches the environmental temperature (20–25 °C), it is possible to switch off the TCD detector and the gas flow.



**Figure 4.** Switching off TCD and gas flow

2. Reduce the pressure of the carrier gas (helium or argon) and oxygen to 0 kPa.  
The reduction of the oxygen pressure is indispensable also when the analysis is not running. There is a consumption of oxygen about 10 mL/min. necessary to maintain the oxygen line cleaned.
3. Set the main switch located in the rear part of the Analyzer to the **Off** (0) position.
4. Disconnect the power cord cable.
5. Any further connected peripherals, for example, AI/AS 1310 liquid autosampler, can remain connected but have to be switched off. For the AI/AS 1310 device, it is sufficient to remove the 24 V DC power cord cable.

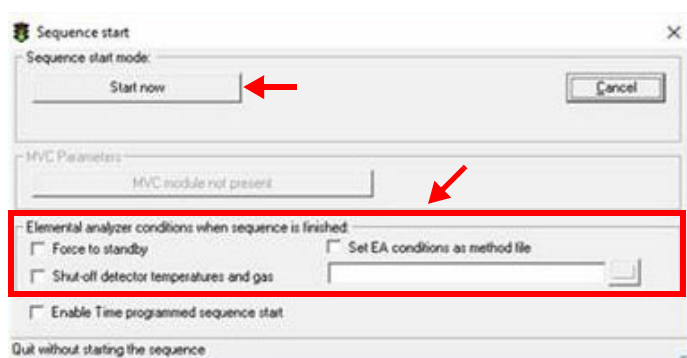
❖ **To *automatically* shut down temperature, detector, and gas**

1. From the Main Menu, select **Run > Start Sequence of Samples** or click the respective icon.

The Sequence start dialog is shown.

2. While programming the start sequence you can choose one of the following options when this sequence is finished (see Figure 5):
  - Force to standby
  - Shut off detector temperatures and gas
  - Set EA conditions as method file





**Figure 5.** Options during programming the start sequence

- When the oxidation reactor or the reduction reactor need to be replaced, the furnaces, detector and gas flow have to be shut off to operate in safe mode.

The “Force to standby” function however, is used when the crucible placed inside the reactor needs to be cleaned.

## Starting up the System

### ❖ To start up the system

- Power on the instrument by lifting the circuit breaker located at the rear of the instrument (position I). The LED **Power On** at the status panel lights up.
- Power on the computer and any complementary units with their main switches.

## Setting the System to Standby

When the work session is over, the instrument should be set in standby mode. In this condition, the temperatures of the left and right furnaces are reduced by 50% versus operating temperatures, and the helium flows on both channels are brought to 10 mL/min.

At the end of the analytical sequence, the Standby function can be activated **manually** or **automatically**.

### ❖ To *manually* set the system to standby

- On the Main menu, select **Edit > Edit Elemental Analyzer parameters** (or click the respective icon).
- On the **Temperature** tab, in the **Other** area, select **Set the instrument to standby**.
- Click **Send** to transfer the command to the analyzer.

### ❖ To *automatically* set the system to standby

- From the **Main Menu**, select **Run > Start Sequence of Samples** or click the respective icon.  
The Sequence start dialog is shown.
- While programming the start sequence you can select to set the instrument to Standby mode when this sequence is finished.

The analyzer automatically passes to the Standby condition when the last sample has been analyzed.

## Support

### Contact

If you need help, contact the Factory Product Support IOMS in Bremen:  
[support.IOMS.bremen@thermofisher.com](mailto:support.IOMS.bremen@thermofisher.com).

### QR Code



Alternatively, contact the Factory Product Support OEA in Milan:  
[support.OEA.bremen@thermofisher.com](mailto:support.OEA.bremen@thermofisher.com).



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