

Hogwarts

GC-FTIR

Chromeleon®7 Software

Dionex Chromeleon version 7.2.10

Ominc 9.11.745 software

Ominc Series 9.11.727

Driver version 9.11.709

Nicolet iS50

Firmware version 1.11

Thermo Scientific Hogwarts GC-FTIR Operating Procedures

Each GC-FTIR system will be identified from all other GC-FTIR systems by use of a unique identifier. This identifier could be a reference to a particular number, model number, serial number, name, etc. All GC-FTIR systems employ OMNIC software. The Hogwarts GC-FTIR additionally employs Chromeleon 7 GC software. See the instrument's maintenance logbook for the specific OMNIC and Chromeleon 7 software version.

General Operations:

1. Ensure that the power is on to all components
2. Open the Chromeleon 7 application by double clicking the icon on the computer desktop (This may take a few moments)
3. Once the Chromeleon 7 application has successfully opened, open the OMNIC application by double clicking the icon on the computer desktop

NOTE: IT IS IMPORTANT THAT YOU FIRST OPEN CHROMELEON AND THEN OMNIC. OMNIC WILL NOT COLLECT DATA IF YOU DO NOT OPEN CHROMELEON FIRST.

To Set Up for Analysis:

Make sure that the **Default iS50 GC-IR** experiment is selected on the OMNIC **Experiment** tool bar and that the blue button is lit up on the iS50 GC-IR accessory.

This should be selected automatically by the instrument. The instrument should be set to a Gain of 1, Resolution of 8 cm⁻¹, 4 Scans, 64 Scans for the background and a spectral range of 4000 to 800 cm⁻¹.

Data Acquisition:

1. All parameters for analysis on the Hogwarts GC-FTIR are electronically stored in the Chromeleon 7 software under the **Data** tab within the folder name **Chromeleon Local**. The method parameters for an analysis run will be noted in the analyst's case file.

NOTE: Remember that the column on the Hogwarts GC-FTIR has a longer column and thinner film thickness than GC/IR #1 and #2. This should be taken into consideration for certain analytes, using the necessary methods as needed.

2. A sample blank must be run each day per each case file, method, and extraction prior to running a case sample.
3. If the reservoir on the GC-FTIR is not cold, fill the reservoir with liquid nitrogen.
4. In the **OMNIC** application window select the **Series** tab and select **Heater Status...**

5. Make sure the heaters are turned on (checked box) and set the **Flow cell setpoint (deg. C):** and **Transfer line setpoint (deg. C):** to **280°C** in the white box and select the **Update Heaters** button.
6. To the right of the white box where the temperature values are set it will show the actual temperature of the flow cell and transfer line. When the values have not reached the desired set temperature, the filament background picture will become yellow. Once the values of the set temperatures have been reached the filament background will turn green.
7. Exit the **Heater Status...** window once the set temperatures have been reached.

Creating a Sequence:

8. Select the **Data** tab within the **Chromeleon Console** window.
9. Find the file titled **_Series Template_ (COPY AND PASTE)** and copy and paste it to your personal Chromeleon sequence folder.
10. Once the file has been pasted to your folder rename it with the date and your initials.
11. Fill out and select the **Name, Position, Volume, Instrument Meth, and Comment** boxes for the samples on the displayed sequence. The **Instrument Meth** boxes will have drop down arrows to select each method.
12. It is the analyst's discretion as to which method to use.
13. Make sure that in the **Name** box provides detailed sample information and the user's initials (This is how the file information will be saved in **OMNIC**)
14. **Click here to add a new injection** to add more samples to the sequence.
15. Once all of the samples have been added and the sequence is ready to run select the **OMNIC** application window.
16. Select the "Collect Ser" button on the **OMNIC** taskbar.

NOTE: IT WILL TAKE A FEW MOMENTS FOR OMNIC TO LOAD THE COLLECT SERIES IF THE SOFTWARE HAS NOT ALREADY BEEN IN USE.

17. The background will collect once the **OMNIC** software has finished loading and an **External Trigger** window will pop open stating "Ready to start data collection at external trigger."
18. Select the **Chromeleon Console** window and highlight the beginning of the sequence run (or the sample you wish to run).
19. Click the "Start" button at the top of the sequence.
20. After a few moments the sequence toolbar should load from yellow to green (New→Running) and where the "Start" button appeared it will now state "Stop".

Auto-sampler Injection:

The auto-sampler will automatically inject your sample once the run has been started in the **Chromeleon Console** and the TRACE 1310 instrument is at ready status.

OMNIC will be triggered to start collecting data once the auto-sampler has injected the sample.

Manual Injection:

The TRACE 1310 instrument has touchscreen capabilities that allow for you to monitor your run, obtain information about the instrument, and manually inject. When manually injecting, the touchscreen will provide the status of the instrument (Not Ready/Ready). When a green play/arrow button pops up on the screen, the instrument is ready for manual injection. Inject your sample and select the green play/arrow button. The **Chromeleon Console** and TRACE 1310 instrument will start collecting, triggering **OMNIC** to start collecting data.

21. Once the sample or sequence has finished, the toolbar in the sequence of the **Chromeleon Console** will state that the “Run Finished”.

Analyzing data in OMNIC:

22. Open the **OMNIC** application window and select “File” and then “Open...” in the toolbar.
23. An “Open” window will appear and in the “Files of type:” box, click the down arrow and select the “Series Files (*.SRS)” option.
24. Select the data file you wish to open and click the “Open” button.
25. Data file will display in **OMNIC**.
26. Use the bottom tools or taskbar to analyze the spectra.
27. To add a spectra region to a new window, right mouse click the bottom screen (spectra) and select “Copy To > New Window” and then click the “OK” button
28. To view a window, select the “Window” tab in the toolbar and click on the window you wish to display.

To Set Up for iS50 ATR Analysis:

Make sure that **iS50 ATR** experiment is selected on the OMNIC **Experiment** tool bar and that the blue button is lit up on the iS50 ATR accessory.

The parameters for using the iS50 ATR accessory should be set to Autogain, Resolution of 4 cm⁻¹, 16 Scans, and a spectral range of 4000 to 600.

iS50 ATR Operating Instructions:

1. At **Experiment** on the tool bar, load the **iS50 ATR** parameter file.
2. Select **Report** on the menu bar and select “Template...”.
3. Select the ATRTEMPLATE.RPT at the very top of the scrolled window and click the “Select” button below.
4. Clean ATR stage and underneath ATR accessory.
5. Collect background and add to window.
6. Add sample and turn down ATR accessory.
7. Collect sample, add to window, select stack spectra, fill out sample information in text box, and select print option on taskbar.
8. Clean ATR stage and underneath ATR accessory.
9. Select **Report** on menu bar and select “Template...”.
10. Select WILLIENEWGCFTIR.RPT template at very bottom of scrolled window and click the “Select” button below to switch template back.
11. Make sure **Default iS50 GC-IR** parameter file is switched back and loaded in **Experiment** tool bar.

MONTHLY MAINTENANCE

Alignment:

1. At **Experiment** on the tool bar, load the **Transmission E.S.P.** parameter file.
2. Select **Collect** from the menu bar and select **Experiment Setup...**
3. After the window loads go to the **Bench** tab and make sure that the Gain is set to 1.0.
4. Then select the **Diagnostic** tab and select the “Align...” button. When the alignment is complete record the number in the appropriate GC/IR maintenance log.
5. Click “OK” button to exit out.
6. Next, check and make sure the desiccant on the instrument is blue (If pink, moisture is present).

Report & Main, ATR, GC iS50 Performance Tests (KBr)

1. Make sure that the main sample compartment is empty.
2. From the menu bar select **Collect** then select **Advanced Diagnostics....**
3. After “Nicolet iS50 Diagnostics” window opens, select **Performance Test**.
4. Check all the boxes for the **Main, ATR, GC iS50 Tests (KBr)**.
5. Select the “Run Selected Test” button.
6. Fill out OMNIC with performance test values for each spectra (**Main, ATR, GC**) and print and place spectra in the appropriate GC/IR maintenance log book.

Example:

PERFORMANCE TEST (MAIN) 2200-2100 = 0.004 PASS RMH

PERFORMANCE TEST (ATR) 2800-2700 = 0.024 PASS RMH

PERFORMANCE TEST (GC) 2200-2100 = 0.012 PASS RMH

7. After tests are complete, select each spectra (**Main, ATR, GC**) and individually save by setting the filename to title under the “Monthly Performance Tests” folder under the “Spectra/Groups (*.SPA, *.SPG)” file type.
8. Select the back arrow  button and select Report, Print, and then the back arrow  button again. Place the print out in the GC/IR maintenance log book.
9. Exit out of “Nicolet iS50 Diagnostics” window.

ValPro Qualification:

1. Check to ensure the **Transmission E.S.P.** parameter file is loaded under **Experiment** from the tool bar.
2. Select **Analyze** on the menu bar and then **ValPro Qualification....**
3. Once the “ValPro Qualification” window appears use the drop down box under “Qualification test:” and select the “Nicolet iS50 system with DTGS in built-in position KBr-EP”.
4. Select the “Qualify” button once the test has been loaded.
5. When the ValPro Qualification has completed, highlight the test that just completed in the “Energy ratio” tab and click the “Report...” button.
6. A window will pop up with the report. Select the “Print” button and fill out ValPro Qualification Report and initial and place in the GC/IR maintenance log book.
7. Select the “Close” button.

For iS50 ATR Accessory

1. In the “Qualification test:” drop down box select the “Nicolet iS50 system with integrated diamond ATR and built-in DTGS-EP” option.
2. Select the “Qualify” button once the test has been loaded and click “OK” when the confirmation window pops up for the background.
3. Retrieve the SMART iTR polystyrene standard “lollipop” from the second drawer near the IR #7.
4. When a window pops up stating “Place the polystyrene in the accessory and apply pressure. Choose OK when you are ready to collect the sample data”, place the polystyrene standard “lollipop” on the sample compartment area with the “ATR STD 269-173000” words facing upwards and apply pressure.

NOTE: Be sure to place the iS50 ATR accessory on the “lollipop” polystyrene standard window in different locations when performing monthly maintenance so as to not place the iS50 ATR accessory on the same location on the “lollipop” window.

5. Select the “OK” button once the “lollipop” polystyrene standard is in place.
6. When the ValPro Qualification has completed, highlight the test that just completed in the “Wavenumber accuracy” tab and click the “Report...” button.
7. A window will pop up with the report. Select the “Print” button and place in the GC/IR maintenance log book.
8. Select the “Close” and then “Exit” button to close out of the ValPro Qualification.
9. Select and load the **Default iS50 GC-IR** parameter file under **Experiment** from the tool bar
10. Remove “lollipop” from iS50 ATR sample area and place back in Thermo box and return it to the second drawer near the IR #7.
11. Fill out ValPro Qualification Report and initial and place in the GC/IR maintenance log book.

To Run the Monthly Standard Check:

1. Check to ensure the **Default iS50 GC-IR** parameter file is loaded under **Experiment** from the tool bar.
2. Make sure the reservoir for the MCT detector is filled with liquid nitrogen.
3. Make sure that the **transfer line** and **flow cell** heaters are at 280°C.
4. Analyze the Monthly QC test standard (Amphetamine, Phentermine, Methamphetamine) that is kept refrigerated.
5. Check its expiration date. If it is past its expiration date, make up a new QC test standard following the correct procedure to ensure the proper concentrations.
6. Make sure that the **60@20AMIN** is loaded and used to run the Monthly QC standard.
7. Run this sample following the procedures for analyzing data from the GC/FTIR and place in the GC/IR maintenance log book.

This is not meant to be all-inclusive. There may be other ways to accomplish the same task.

Addendum

S/SL Injection Port with HP-5 5 meter column, 0.32 column ID, and 0.25 um film thickness:

Stored Methods for AUTOSAMPLE SPLIT INJECTION

90TO270 70AMIN 10MIN Initial 90C degrees hold for 1 minute ramped to 270C degrees at 70C/min. Constant carrier flow rate of 3.0 mL/min, Split ratio of 2.0, Split flow 5.0 mL/min, Purge flow 5 mL/min, Inlet temperature of 250C degrees. Total run time 10.00 minutes.

90TO270 70AMIN 30MIN Initial 90C degrees hold for 1 minute ramped to 270C degrees at 70C/min. Constant carrier flow rate of 3.0 mL/min, Split ratio of 2.0, Split flow 5.0 mL/min, Purge flow 5 mL/min, Inlet temperature of 250C degrees. Total run time 30.00 minutes.

200TO270 70AMIN 10MIN Initial 200C degrees hold for 1 minute ramped to 270C degrees at 70C/min. Constant carrier flow rate of 3.0 mL/min, Split ratio of 2.0, Split flow 5.0 mL/min, Purge flow 5 mL/min, Inlet temperature of 250C degrees. Total run time 10.00 minutes.

200TO270 70AMIN 20MIN Initial 200C degrees hold for 1 minute ramped to 270C degrees at 70C/min. Constant carrier flow rate of 3.0 mL/min, Split ratio of 2.0, Split flow 5.0 mL/min, Purge flow 5 mL/min, Inlet temperature of 250C degrees. Total run time 20.00 minutes.

200TO270 70AMIN 30MIN Initial 200C degrees hold for 1 minute ramped to 270C degrees at 70C/min. Constant carrier flow rate of 3.0 mL/min, Split ratio of 2.0, Split flow 5.0 mL/min, Purge flow 5 mL/min, Inlet temperature of 250C degrees. Total run time 30.00 minutes.

90TO270 35AMIN 20MIN Initial 90C degrees hold for 1 minute ramped to 270C degrees at 35C/min. Constant carrier flow rate of 3.0 mL/min, Split ratio of 2.0, Split flow 5.0 mL/min, Purge flow 5 mL/min, Inlet temperature of 250C degrees. Total run time 20.00 minutes.

Stored Methods for HAND SPLIT INJECTION

HI 90TO270 70AMIN 10MIN Initial 90C degrees hold for 1 minute ramped to 270C degrees at 70C/min. Constant carrier flow rate of 3.0 mL/min, Split ratio of 2.0, Split

flow 5.0 mL/min, Purge flow 5 mL/min, Inlet temperature of 250C degrees.
Total run time 10.00 minutes.

HI 200TO270 70AMIN 30MIN Initial 200C degrees hold for 1 minute ramped to 270C degrees at 70C/min. Constant carrier flow rate of 3.0 mL/min, Split ratio of 2.0, Split flow 5.0 mL/min, Purge flow 5 mL/min, Inlet temperature of 250C degrees.
Total run time 30.00 minutes.

Always verify that the temperature for the GC interface is at least 10 degrees higher than the maximum temperature of the GC oven.