Desktop

CompassCDS: main system control for data analysis

CompassCDS LINK: program for exporting data

CompassCDS Configuration: for administrator use

TeamViewer 13: allows outside control of desktop for GC support

GCVarianCP3800_ShortManual: condensed operator manual

GCVarianCP3800_Manual: full operator manual

Compass Data: short cut to Data folder, where all the files and personal folders are saved

GC_DataRunGuide.pdf: guide to running samples on the GC

GC_Guide.pdf: this comprehensive guide to running the GC

Methods

Principle of Method: Varian CP-3800 gas chromatograph (GC) equipped with a thermal conductivity detector (TCD), a flame ionization detector (FID), and an electron capture detector (ECD) to measure the concentrations of CO_2, CH_4, and N_2O, respectively. To verify accuracy, at least three standards of known concentration for each gas should be run for every 100 samples. GC measurement uncertainty: 5, 0.01, and 0.001 µmol mol^{-1} for CO_2, CH_4, and N_2O, respectively.

Create User

CompassCDS Configuration → User Identification: *admin* → click "OK" → click on "Users in "Organization box → in "Users" window, right click → New → enter your first name for "Name" and for "User identification" → click "Next" button → check box for "DESKTOP-RAGR6HF" → CLICK "Next" button → check box for "GHG" → click "OK"

Turning on the GC

Open gas flows and check gas pressures and flow rates!
- **Total amount of each gas >500 psi** (right sensor on regulator)
- **Do NOT run GC when gases are <200 psi (especially helium!)**
- **Running the GC, even in "standby" mode, without proper gas pressure will destroy the detectors!**
- **Flow rate: All gases** (Ar; H_2; He; Air) = **80 psi** (left sensor on regulator)

If gas pressures are appropriate, turn on GC switch (top, back of machine)

Upload and run "Standby.METH" (see "Running Method" section).

On the GC machine, click "Detector" button → click "2" button for Middle FID → Electronics should be "off" if the "Standby" method is running.

While the detectors are heating up, the electronics for all three detectors should be off. Turn off the electronics for the TCD and ECD detectors:

- On the GC machine, click "Detector" button → click "1" button for Front TCD → use the cursor arrow to scroll to "Electronics" → click "Enter" button to turn the electronics off until the set temperature is reached → click "3" button for Rear ECD → use the cursor arrow to scroll to "Electronics" → click "Enter" button to turn the electronics off until the set temperature is reached

Once the detectors have reached the appropriate temperatures (TCD = 120 °C; FID = 275 °C; ECD = 320 °C), upload and run "3800GHG.METH" (see "Running Method" section) for ~16 hours to allow the detectors to settle

After ~16 hours, calibrate the GC before running samples for analysis (see "Calibration" section); if unable to calibrate at this time, upload and run "Standby.METH" (see "Running Method" section) to preserve gas until calibration can occur

Running Method

   *this section details how to run a method outside of running sample analysis (the method chosen for each row in your sequence list when analyzing samples will override any method already running)

CompassCDS → Sign in with <u>YOUR</u> user ID (your first name) and click OK

After CompassCDS opens, two windows appear on the left with three tabs at the bottom; click "Systems" tab at the bottom left → check box for "3800 GHG" that appears in window to communicate with the GC and autosampler

File → Open → Open Method → open 3800GHG.METH *or* Standby.METH

Click "Control" in bottom left window; then click "Overview" box at top of large window

Upload (to run) method to GC by clicking the box that points to the GC with the **red** arrow

   *the 3800GHG.METH is for analyzing samples; the Standby.METH is a low-gas use method that should always be used when the GC is not in use, but when the GC is being used frequently enough to justify leaving it on between uses

On the GC machine, click "Detector" button → click "2" button for Middle FID → Electronics should be "off" if the "Standby" method is running or "on" if the 3800GHG.METH is running

Calibration

CompassCDS → Sign in with "lab" user ID (this will make it so that data produced from the calibration run is automatically saved in the general "lab" folder); click OK

After CompassCDS opens, two windows appear on the left with three tabs at the bottom; click "Systems" tab at the bottom left → check box for "3800 GHG" that appears in window to communicate with the GC and autosampler

Then go to File → Open → Open Method → open 3800GHG.METH

Click on "calibration" in the bottom left window (not the tab); now information about the Calibration Curve appears in the main window

Click on one detector at a time listed under the method in the upper left window; in the "Calibration Curve" box, after "File," type in the current date for each detector (e.g., "Front TCD Apr 24 2019," "Middle FID Apr 24 2019," and "Rear ECD Apr 24 2019") and then save the method

*if the "standard gas" tanks are changed, the concentrations (ppm) must be changed under all Levels (1–5) to match the new certified concentrations; the parameters are:

Detector (gas)	Level 1	Level 2	Level 3	Level 4	Level 5
Front TCD (CO_2)	89.95	359.80	2490.00	4980.00	9960.00
Middle FID (CH_4)	0.40	1.58	2.77	5.55	11.10
Rear ECD (N_2O)	0.24	0.98	1.90	3.80	7.60
*units in ppm	Certified concentrations of "low standard" for each gas (*0.25 for Level 1)		Certified concentrations of "high standard" for each gas (*0.25 for Level 3 and *0.5 for Level 4)		

Check gas pressures and flow rates!

- **Total amount of each gas >500 psi** (right sensor on regulator)
- **Do NOT run GC when gases are <200 psi (especially helium!)**
- **Running the GC, even in "standby" mode, without proper gas pressure will destroy the detectors!**
- **Flow rate: All gases** (Ar; H_2; He; Air) **= 80 psi** (left sensor on regulator)

Use sign-in sheet:

- Record the date, your name, # of samples (20), whether you changed the septa (should be changed every 500-800 samples), and gas pressure (psi read from right sensor on regulator); also note that it is a calibration run

  > *Septa change: when GC is not in use, use the tweezers in the "Consumables" drawer to unscrew the rear injection port and to replace the used septa with a new septa (also located in "Consumables" drawer); screw injector port back in using the tweezers to make it fairly tight (note tightness when unscrewing)

Evacuate and prepare the calibration vials (located in the drawer under the desktop) using the "standard gas" tanks according to the following chart:

Gas tank	Level 1	Level 2	Level 3	Level 4	Level 5
Low standard	5	20	0	0	0
High standard	0	0	5	10	20
N_2	15	0	15	10	0
*units in mL		*each level has four replicates; be as precise as possible!			

Load the trays with the vials in the proper tray slots, facing in the correct direction (front facing toward you, Tray 1 to the left and Tray 2 to the right); **make sure the trays are set properly between the tray-holding knobs, or the needle will miss and break!**

Go to File → Open → Open Sequence → CalibrationRun.SEQU

In the "Enabled" column, check the box for all samples

Click the green "play" button to start running the calibration sequence

On the GC machine, click "Detector" button → click "2" button for Middle FID → click button below "Ignite" to ignite CH₄ analysis! Wait for the electronics to turn "on" before igniting the Middle FID or it will not work

Data will save within the "lab" folder under folders for the year, the month, and the day

When the sequence is complete, reprocess the data

Go to File → Open → Open Reprocessing List → open 3800 GHG.Repl → File → Save As → Save reprocessing list As → click on "lab" folder to save the file within this folder, and rename the reprocessing list as "Calibration_*date*"

Click on the three dots under the "Chromatogram name" column → navigate to the data you just collected (which will be in the "lab" folder under the current date) and open all the data files

In the "Sample Type" column, designate each sample as "Standard;" in the "Calibration" column, designate each sample as "Add;" in the "Calibration Level" column, designate each sample as the appropriate level (Level 1–5)

> *if the standards need to be reprocessed more than once under the same date, the first three rows of the "Calibration" column (i.e., the three detectors) should be set to "Clear old points," and the remaining rows should be set to "Add"

Click the green "play" button (click "ok" in the event of an error/warning message)

When reprocessing is complete, go to File → Open → Open Calibration Curve and open one detector from the current date at a time (the detectors will appear in the main data folder as, for example, "Front TCD Apr 24 2019.CALB"); after you have opened the Calibration Curve for one of the detectors, the tab at the bottom left will switch to the "Calibration" tab and window

In this window, choose which points to use (by unchecking and rechecking points) to try to achieve a good regression coefficient (0.9999 for CO_2; 0.999 for CH_4 and N_2O); try to keep at least 3 points from each level (Level 1–5); save the regression for each detector after editing

*for more precise editing of the calibration curve, go to File → New → New Summary Report → go to "ABC Variables" tab at the bottom of the new window → open current method (3800GHG.METH) in "Method File" box → choose channel → click "Add Variable" tab in "Variable List" box → add "Area" and then right click on "Chromatograms" box under "Chromatograms" to summarize → Add Chromatogram → navigate to standards data → open one level for the calibration at a time → click on "Results Mode" box in upper left of window → View Summary (note which standards should NOT be used; Rsd% closer to 0.1 is best, but should be less than 1)

*if the new calibration is not good, the method (3800GHG.METH) can be returned to the old calibration curves by opening the method, clicking on "calibration" in the bottom left window (not the tab), and changing each detector to the previous date in the "Calibration Curve" box by clicking the folder icon to navigate to the previous date (do NOT type it in); if using the old calibration curves is not appropriate, repeat calibration and try to be more precise when filling the vials with the standard gases

Once you are done calibrating the GC, run the "Standby" method (see "Running Method" section and run the "Standby.METH" method)

Data Analysis

Check gas pressures and flow rates!

- **Total amount of each gas >500 psi** (right sensor on regulator)
- **Do NOT run GC when gases are <200 psi (especially helium!)**
- **Running the GC, even in "standby" mode, without proper gas pressure will destroy the detectors!**
- **Flow rate: All gases** (Ar; H$_2$; He; Air) = **80 psi** (left sensor on regulator)

Use sign-in sheet:

- Record the date, your name, # of samples, whether you changed the septa (should be changed every 500-800 samples), and gas pressure (right sensor on regulator)
  - Septa change: when GC is not in use, use the tweezers in the "Consumables" drawer to unscrew the rear injection port and to replace the used septa with a new septa (also located in "Consumables" drawer); screw injector port back in using the tweezers to make it fairly tight (note tightness when unscrewing)

Load the trays with your vials in the proper tray slots, facing in the correct direction (front facing toward you, Tray 1 to the left and Tray 2 to the right); **make sure the trays are set properly between the tray-holding knobs, or the needle will miss and break!** If you have 3 mL vials instead of 12 mL vials, use the 3 mL converters for the trays, which fit into the middle section of the trays, allowing the trays to still function as normal with no changes to the autosampler.

CompassCDS → Sign in with <u>YOUR</u> user ID, which is your first name (this will make it so that data produced from your sample run is automatically saved in your folder, rather than in a generic, shared folder); click OK

After CompassCDS opens, two windows appear on the left with three tabs at the bottom; click "Systems" tab at the bottom left → check box for "3800 GHG" that appears in window to communicate with the GC and autosampler. Wait for communication connection to complete.

Click "Data" tab at the bottom left.

File → Open → Open Sequence → Template.SEQU → File → Save As → navigate to and click on the "Data" folder, create a folder using your name, and rename and save the file within your

folder (you can use this sequence over and over again); rename the samples, add standards if desired, and remove rows as needed.

In the "Enabled" column, check the box for all samples you want to run.

Make sure that the last row in your sequence has the "Standby.METH" selected for the method; this allows the GC to return to a low gas-use method while it is not in use after your sequence has completed.

Click the green "play" button to start running your sequence.

On the GC machine, click "Detector" button → click "2" button for Middle FID → click button below "Ignite" to ignite CH$_4$ analysis! Wait for the electronics to turn "on" before igniting the Middle FID or it will not work. Wait for the Middle FID signal on the computer screen to make sure you have ignited the detector.

Data will save in your personal folder under folders for the year, the month, and the day. Each chromatograph (contain the gas concentration data) will have the year, month, day, and given name from the sequence file.

Once your sequence is completed, verify that the "Standby" method is running: On the GC machine, click "Detector" button → click "2" button for Middle FID → Electronics should be "off" (if they are not off, run only the last row in your sequence, which should be the "Standby. METH" method, by enabling only this row.

Viewing Data

While running, double click on the desired row (sample) of your sequence to open the corresponding chromatogram once the sample has completed running. Or use File → Open → Open Chromatogram → navigate to and click on your folder (which is in the "Data" folder) and navigate to the gas chromatograms under the year, month, and day → open the desired chromatograms → click on the results tab under each detector to view the results for each gas.

Autosampler Errors

If the autosampler has an error (head connection error) during sampling run → turn off the autosampler (the on/off switch is on the side of the small box on the table behind the desktop) → stop your sequence entirely by clicking the red "stop" button → turn on the autosampler and wait for it to return to its normal position (this may take a minute) → enable only the rows (samples) you want to run in your sequence by unchecking the other rows that have already run (the final row that was running when the autosampler stopped working will be the first sample you run) → click the green "play" button to start running your sequence.

Other Issues

The N_2O (ECD) detector tends to slowly drift during a run of a set of samples. The best way to avoid substantial drift is to run at least 5 blanks (air) at the start of your sequence or any time after the machine has been in standby or not running for about an hour. The blanks should stabilize for N_2O before you start your samples.

Exporting Data

CompassCDS LINK → click on "Settings" tab → choose your personal folder as the Output directory (do not save settings after doing this) → click on "Summarize" tab → Choose folder → navigate to the folder with the data you want to export and click on it → an Excel file is automatically created within the folder you designated as the output directory.

Reprocessing Data

File → Open → Open Reprocessing List → open 3800 GHG.Repl → File → Save As → Save reprocessing list As → navigate to and click on your folder (which is in the "Data" folder) to save the file within your folder, and rename the reprocessing list as desired

Click on the three dots under the "Chromatogram name" column → navigate to the data you want to view or reprocess and open it

Double click on the name of a sample under the "Chromatagram name" column to view the data, method, and results for that sample for all three detectors

To reprocess the sample(s), make changes to the columns as desired, and click the green "play" button (click "ok" in the event of an error/warning message)

Adjusting the Autosampler Position

*to adjust the positioning of the autosampler, it needs to be on but cannot be in communication with the GC (i.e., do not activate the System "3800 GHG" in CompassCDS)

Load both trays in the tray holders with at least one vial in each corner of each tray

On the autosampler, click "F1" for menu → scroll (rotate knob) to "Setup" → click "F3" → click enter (center button on knob) → scroll to "Objects" → enter → scroll to "Trays" → enter → scroll to "Tray 1" → enter → click "F1" for check position

The autosampler will move to position 1 of Tray 1

If the positioning is accurate, click "F1" to check the next position (position 10); if the positioning is accurate, click "F1" to check the final position (position 50)

If the positioning is inaccurate, the following axes control the positioning:

- x is left to right; y is front to back; z is up and down (should be 1-2 mm from tray)

Scroll to the axis that needs adjusted → enter (to highlight the value) → slowly scroll to adjust the positioning (note the changing value on the screen and the minor but immediate movement of the autosampler syringe) → **click enter again** to un-highlight the axis when the position is accurate → click "F1" for check position

Repeat process until all axes are appropriately positioned for positions 1, 10, and 50 of Tray 1

If the positioning is accurate, click "ESC" → scroll to "Tray 2" → enter → click "F1" for check position and repeat process for Tray 2

When the positioning of both trays is accurate, click "F4" to return to home screen

Note: use "Objects" → "Tray Holders"" → "TH1dr1" to line up hold under Tray 1 if metal plate is moved

Note: use "Objects" → "Injectors" → "Middle" to adjust injection position

Maintenance

The septa should be changed every 500-800 samples. When GC is not in use, use the tweezers in the "Consumables" drawer to unscrew the rear injection port and to replace the used septa with a new septa (also located in "Consumables" drawer); screw injector port back in using the tweezers to make it fairly tight (note tightness when unscrewing).

The syringe should be checked every ten thousand or so samples and changed if the seal is loose or the plastic stopper is deteriorating. The bungee cords within the autosampler also need to be checked every ten thousand or so samples and changed if they have lost their elasticity or are deteriorating.

Turn off the autosampler and gently move the arm so that the syringe is over the plates.

Gently open the clear protective screen and undo the latch and top screw that hold the syringe in place (take care not to move the entire tower to avoid big changes to the autosampler position). Remove and replace the syringe and/or bungee cords.

After replacing the syringe, check and adjust the autosampler position (see "Adjusting the Autosampler Position" section.

Turning off the GC

If the "Standby" method is not running, upload and run "Standby.METH" (see "[Running Method](#)" section)

>*on the GC machine, click "Detector" button → click "2" button for Middle FID → Electronics should be "off" if the "Standby" method is running or "on" if the 3800GHG.METH is running

Turn off GC switch (top, back of machine)

Close the gas flows for all the gas tanks

Update Record

Prepared By: Cole Gross   **Date:** July 2018

Update By: Cole Gross   **Date:** May 2021
Changes: Updated multiple directions and added maintenance section

Update By: **Date:**
Changes: