



LOS GATOS RESEARCH



CO₂ Carbon Isotope Analyzer

CCIA-36d

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Quick Start Guide (Standard Instrument)

Set up the CO₂ Carbon Isotope Analyzer

- Connect the instrument to main power.
- Remove the gas inlet cap.
- Remove the gas outlet cap and attach the provided vacuum pump.
- Attach the desired data connections to the rear panel of the instrument.
- Start the analyzer via the power switch. The startup process takes approximately one minute.
- Allow the instrument cell temperature to stabilize at around 45 degrees Celsius. This may take 1-2 hours and the exact final temperature will be instrument specific.

Calibrate the CO₂ Carbon Isotope Analyzer

- Enter the “Setup” panel and click the “Calibrate” button.
- Follow the on-screen directions for calibration.
- Attach a tube to the inlet, regulated at a pressure just slightly above ambient atmosphere (<10 psig), from a local gas standard.
- Enter the known total CO₂ concentration in ppm and isotopic ratios in ‰. If water is present and known, this can be entered as well.
- Disconnect the gas standard when prompted by the on-screen directions.
- *NOTE: Periodic calibration is required to maintain data quality.* Please see Appendix D – Notes concerning instrument calibration.

Starting measurement

- Attach a tube from the gas sample to the gas inlet port.
 - If the optional ‘Batch Mode’ is being used, see page 30.
 - If the optional ‘Multi-port Inlet Unit’ is being used, see page 38.
 - If the optional ‘Dynamic Dilution System’ is being used, see page 42.
 - If the optional ‘Nafion Gas Drier’ is being used, see page 46.
- If the instrument is already on, a new file can be started by entering the “Setup” panel and then exiting.
- Note the filename being written as data is taken.
- Download the data (one of the two following options) when measurement is complete.
 - Insert a USB memory device and go to the “File Transfer” menu.
 - Download through the LAN Ethernet connection if it is enabled during startup (see Appendix A – Accessing data via a LAN Ethernet Connection).

Installation

The Los Gatos Research (LGR) CO₂ Carbon Isotope Analyzer is comprised of several components. Be sure to check that each of the system components has arrived before beginning the installation procedure.

For the basic, free-flow system you should have received:

- CO₂ Carbon Isotope Analyzer
- Instrument power cord
- Users guide (this document)
- USB flash drive
- External pump
- Pump slave power cord
- Pump connection tubes
- Serial port connection cable (null modem type)

For the Batch Mode Operation (Optional):

- Syringe body
- Needle pack
- Septum pack

For the Multi-port Inlet Unit (Optional):

- Multi-port Inlet Unit
- Power Cable
- 25-pin connection cable for control signal

For the Dynamic Dilution System (Optional):

- DCS-200 dilution system
- BNC connection cable for control signal
- Sample connection tubing with flow-thru 'Tee'
- Diluter power cord
- Throttle valve for pump (if system also has Batch Mode)

Nafion Gas Drier (Optional):

- Nafion drier and manual
- Drierite canister
- Connection tubing for inlet and pump port

If you have not received all of these components, please contact LGR (650-965-7772 or sales@lgrinc.com).

Electrical Power Connection

In order to operate the CO₂ Carbon Isotope Analyzer, it must be connected to main power via the fused power entry module on the back of the unit. The unit can be switched from 115 VAC operation to 230 VAC operation via a voltage selection switch on the rear panel near the power entry module (see Figure 1). If operation from any other voltage source is desired, please contact LGR.

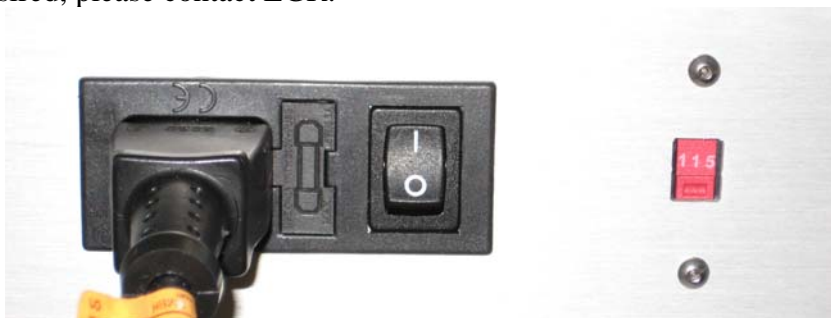


Figure 1: AC power entry module with fuse, power switch, and AC voltage selection switch

Data Interface Connections

The CO₂ Carbon Isotope Analyzer has a variety of data interface connection ports on the back panel (see Figure 2). These can vary slightly from instrument to instrument depending on the ordered configuration.

- “USB” ports are utilized for file transfer to USB memory devices and connecting peripherals.
- “Serial” port is utilized for real-time measurement output directly to a computer.
- “Ethernet” connection allows the instrument to be connected to a Local Area Network (LAN) and the data directory is made available as a Windows™ network shared directory (see Appendix A – Accessing data via a LAN Ethernet Connection).
- “Analog” port is not configured for this instrument at this time.
- “VGA” port allows for connection to an external monitor.
- “Control Out” (not labeled) is a BNC connection to the Dynamic Dilution System (Optional).
- 25-pin data port (not labeled) for connection to the Multi-Port Input Unit (optional).

The functionality of the data interface connections is described in the relevant sections later in this manual.



Figure 2: Data interface connections

Gas Inlet / Outlet Connections (Standard Instrument)

The gas inlet and outlet ports of the instrument are on the rear panel (see Figure 3 and Figure 4). The unit is shipped with all inlets and outlets plugged for protection during transit.



Figure 3: External pump connection (3/8"). The internal pump exhaust port is not used if the instrument has an external pump.



Figure 4: Gas sample inlet connection (1/4"), care must be taken to not over-pressurize.

In the normal mode of operation, the provided external pump draws sample through the instrument from the inlet (1/4" Swagelok). The inlet gas pressure range is 0 to 10 psig. The external pump is connected to the instrument with the provided 3/8" tubing and Swagelok connectors. Proper Swagelok connection requires using either a 9/16" wrench (for 1/4" tube) or an 11/16" wrench (for 3/8" tube) to tighten the connection 1/4-1/2 turn past finger-tight, leaving a gap of < 3.5mm (see Figure 5).

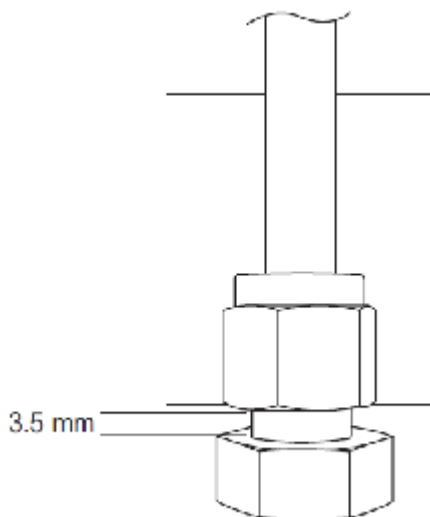


Figure 5: Swagelok connections should be tightened to 1/4-1/2 turn past finger tight, leaving a gap of < 3.5mm. Use a 9/16" wrench for 1/4" tubing, and an 11/16" wrench for 3/8" tubing.

The external pump slave power cord should be plugged into the outlet on the back panel of the instrument labeled "External Pump Power" (see Figure 6), and connected to the power connection on the pump. Make sure the pump power switch is left in the "ON" position and that the speed control knob on the pump is turned to approximately 2/3 speed (indicator at approximately a 2 o'clock position).



Figure 6: External pump power connection.

Instrument Startup

When the appropriate electrical, gas, and data connections are in place, the instrument may be started via the power switch on the rear power entry module (see Figure 1). The internal computer will boot, and automatically load and start the instrument control software. The startup process takes approximately 1 minute. Operation of the instrument is described in the next section.

Isotopic measurements require a stable environment with minimal temperature fluctuations if possible; the instrument measurement cell is temperature stabilized to help accomplish this.

NOTE: LGR recommends waiting for the cell temperature to stabilize (around 45 degrees Celsius) before starting work. The exact final cell temperature will be instrument specific.

Shutdown of the instrument is accomplished by exiting the operating software, waiting for the instrument to shut down, and turning off the power switch. Please refer to the more detailed section on page 26.

Operation

The Los Gatos Research (LGR) CO₂ Carbon Isotope Analyzer is easily operated via the user interface as described in this section.

Startup

The internal computer will boot, then automatically load and start the instrument control software once the proper power connections are made and the switch is set to the ON position. The startup process takes approximately 1 minute. Once a month, the instrument automatically performs a thorough file system integrity check during boot-up. The display shown in Figure 7 appears and the instrument will take approximately 1-2 minutes to complete the integrity check before continuing with loading the software. Do not turn off the computer during this maintenance.



Figure 7: The Routine Maintenance Screen appears during boot-up once a month. Normal operation will automatically continue after the maintenance is complete. Please do not turn off the instrument during maintenance.

Display Modes

When the instrument is turned on, it will automatically go through a one minute initialization cycle with the Los Gatos Research logo and “Please Wait” message on the screen. After initialization is complete, the instrument will begin to draw in gas and to display results in the “Spectrum” mode (see Figure 8). This display shows the laser transmission through the ICOS measurement cell (Figure 8, top frame) recorded as the laser wavelength is tuned across the selected wavelength region near 2.05 microns. The CO₂ and H₂O absorption lineshapes that result from a detailed analysis of the measured transmission signal are shown in the bottom frame of Figure 8.

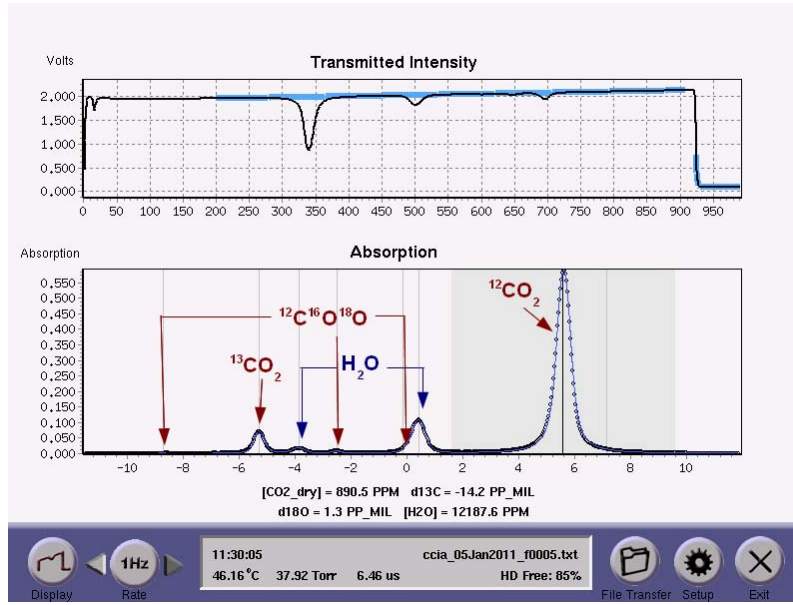


Figure 8: The spectrum display consists of two graphs: the top shows the voltage from the photodetector as the laser scans across the CO₂ and H₂O absorption features, the bottom shows the optical absorption fraction due to CO₂ and H₂O in black, and the peak fit resulting from signal analysis in blue. The respective features of isotopic CO₂ and H₂O are labeled here in the manual.

The total CO₂ concentration (¹²CO₂ + ¹³CO₂ + ¹²C¹⁶O¹⁸O) is shown in parts-per-million (ppm). For this display, this is reported as a dry mole fraction where the measured mole fraction of H₂O (also in ppm) has been removed.

$$[CO_2(ppm)]_{DRY} = \frac{[CO_2(ppm)]_{WET}}{\left(1 - \frac{[H_2O(ppm)]}{10^6}\right)}$$

Equation 1: Definition of dry mole fraction.

The isotope ratios for $\delta^{13}C$ and $\delta^{18}O$ are shown in parts-per-thousand (‰ or pp-mil). The isotope ratio is reported relative to Pee Dee Belemnite (PDB) as shown in Equation 2 and Equation 3.

$$\delta^{13}C = \left[\frac{\frac{^{13}C_{Sample}}{^{12}C_{Sample}}}{\frac{^{13}C_{PDB}}{^{12}C_{PDB}}} - 1 \right] \times 1000$$

Equation 2: Definition of isotope ratio $\delta^{13}C$ vs. Pee Dee Belemnite (PDB). PDB is defined to be $^{13}C/^{12}C = 0.0112372$.

$$\delta^{18}O = \left[\frac{\frac{{}^{18}O_{Sample}}{{}^{16}O_{Sample}}}{{}^{18}O_{PDB}/{ }^{16}O_{PDB}} - 1 \right] \times 1000$$

Equation 3: Definition of isotope ratio $\delta^{18}O$ vs. Pee Dee Belemnite (PDB). PDB is defined to be ${}^{18}O/{}^{16}O = 0.0020672$.

The user may toggle the “Display” button to see the “Time Chart” mode of instrument recordings as shown in Figure 9.

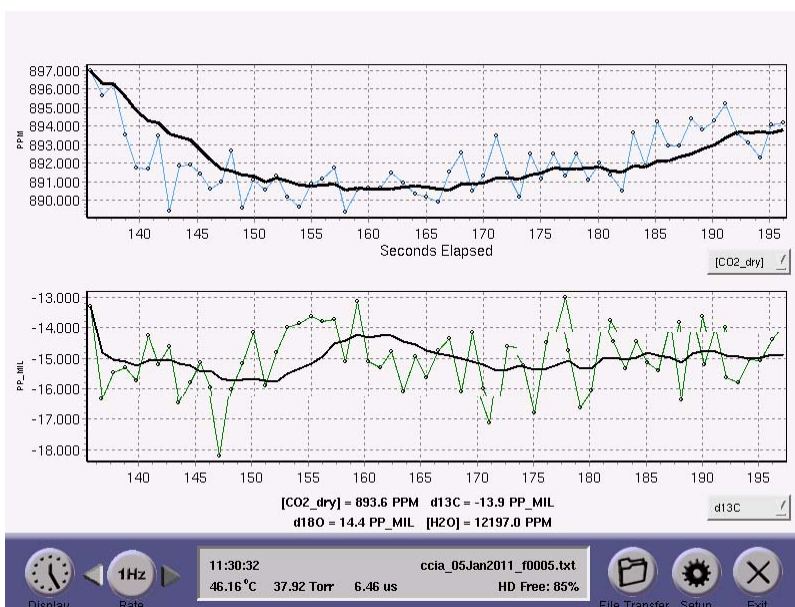


Figure 9: The time chart shows a continuous record of CO₂ concentration and isotopic ratio $\delta^{13}C$ over time. A 10-point running average (in black) is shown going through the raw data (in color). The drop down boxes associated with each graph can be toggled to show $\delta^{18}O$ and H₂O as well.

The user may use the drop down box in the lower right of either window to toggle displays of CO₂ concentration, H₂O concentration, and isotope ratios $\delta^{13}C$, $\delta^{18}O$. These data are also being saved to the file indicated in the upper right corner of the parameter window (located at the bottom middle of the screen).

Finally, the “Display” can be toggled to a “Numeric” mode which shows many of the relevant measurements simultaneously. Both wet and dry total CO₂ is reported, the isotope ratios $\delta^{13}C$ and $\delta^{18}O$ are shown, H₂O concentration is reported, and the individual isotope components are reported in ppm.

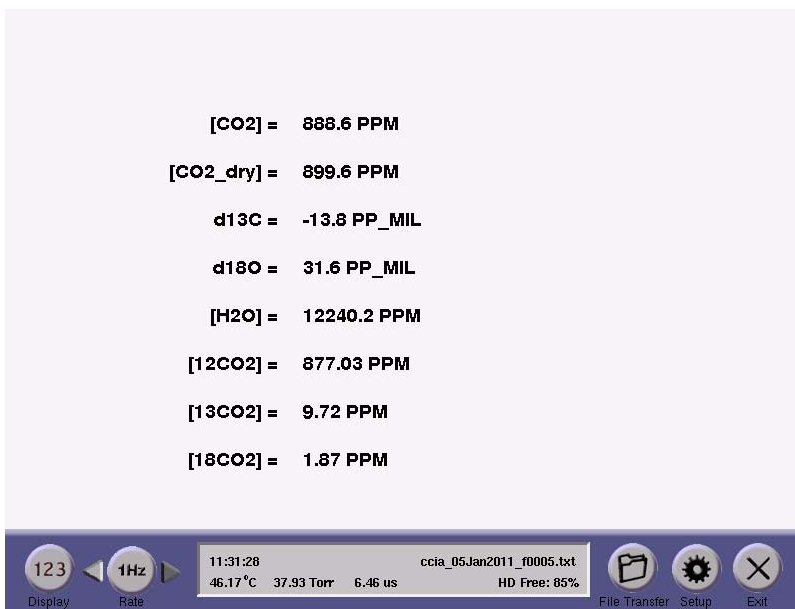


Figure 10: Numeric mode displays all results at once. In addition to total CO₂ concentration (wet and dry), the isotope ratios $\delta^{13}\text{C}$ and $\delta^{18}\text{O}$, and H₂O concentration, the individual isotope components are shown as well. Note that [18CO₂] is short-hand for ¹²C¹⁶O¹⁸O.

Rate Control

The user may change the rate at which data are written to the log file by selecting the Left or Right arrows next to the Rate indicator. In normal mode, data will be acquired at a 1 Hz rate and averaged for a selected interval (1 to 100 seconds) before being written to the data file and plotted on the time chart. Longer averaging periods (or equivalently, slower data acquisition rates) will yield better measurement precision than shorter averaging periods; so the user may trade off precision in concentration and isotope ratio for precision in time.

Parameter Window

The parameter window (bottom-center of the instrument display) shows the current time, cell temperature (Celsius), cell pressure (Torr), and cell ring-down time (micro-sec). The current data filename is also shown as well as the available hard-drive space left on the instrument.



Figure 11: Lower task bar of instrument showing parameter window between 'Rate' and 'File Transfer' controls.

If the user happens to have the Multi-port Inlet Unit (Optional) and the unit is enabled, it will display which inlet valve is currently being sampled.



Figure 12: If the Multi-port Inlet Unit (MIU) is present and enabled, the parameter window displays the current inlet valve being sampled ('16' in this figure) as well as the corresponding user-defined text tag ('Ref8'). Please see page 38 for additional details concerning the programming of the MIU.

File Transfer Menu

Each time the instrument is re-started, and each time the user enters and then exits the "File Transfer" menu or "Setup" panel, a new data file is created with a filename of the form "ccia_DDMMYYYY_fxxxx.txt". This filename has a time stamp consisting of day (DD), month (MMM), and year (YYYY) and is followed by a 4-digit serial number (xxxx); i.e., ccia_27Apr2009_f0010. The serial number counts upward to provide up to 10000 unique file names each day. If the instrument is left in continuous operation, a new data file will automatically be created in order to keep data file sizes manageable.

NOTE: In the case of continuous operation, the filename shown will be the name of the first file in the series.

The time period between creations of new files is set in the "File Settings" menu as described later (page 18). The data files are written in text (ASCII) format and contain labeled columns as shown in Figure 13.

```
2011 Jan 05 12:18:49
Time, [ht12]_ppm, [ht12]_ppm_se, [ht13]_ppm, [ht13]_ppm_se, [ht18]_ppm, [ht18]_ppm_se,
14.474, 1.33517e+03, 0.00000e+00, 1.67906e+01, 0.00000e+00, 6.14087e+00, 0.00000e+00,
15.475, 1.33371e+03, 0.00000e+00, 1.67500e+01, 0.00000e+00, 6.24950e+00, 0.00000e+00,
16.501, 1.33281e+03, 0.00000e+00, 1.67412e+01, 0.00000e+00, 6.21767e+00, 0.00000e+00,
17.493, 1.33539e+03, 0.00000e+00, 1.67632e+01, 0.00000e+00, 6.18303e+00, 0.00000e+00,
18.510, 1.33185e+03, 0.00000e+00, 1.67641e+01, 0.00000e+00, 6.21200e+00, 0.00000e+00,
19.522, 1.33390e+03, 0.00000e+00, 1.67554e+01, 0.00000e+00, 6.18797e+00, 0.00000e+00,
20.533, 1.33479e+03, 0.00000e+00, 1.67959e+01, 0.00000e+00, 6.24316e+00, 0.00000e+00,
21.540, 1.33401e+03, 0.00000e+00, 1.67536e+01, 0.00000e+00, 6.17661e+00, 0.00000e+00,
22.541, 1.33093e+03, 0.00000e+00, 1.67211e+01, 0.00000e+00, 6.15849e+00, 0.00000e+00,
23.547, 1.33402e+03, 0.00000e+00, 1.67522e+01, 0.00000e+00, 6.23914e+00, 0.00000e+00,
24.559, 1.32999e+03, 0.00000e+00, 1.67076e+01, 0.00000e+00, 6.14385e+00, 0.00000e+00,
25.571, 1.33036e+03, 0.00000e+00, 1.67296e+01, 0.00000e+00, 6.12959e+00, 0.00000e+00,
26.550, 1.33008e+03, 0.00000e+00, 1.67044e+01, 0.00000e+00, 6.15605e+00, 0.00000e+00,
27.552, 1.33332e+03, 0.00000e+00, 1.67560e+01, 0.00000e+00, 6.23718e+00, 0.00000e+00,
... ..
52.673, 1.32951e+03, 0.00000e+00, 1.67090e+01, 0.00000e+00, 6.33003e+00, 0.00000e+00,
53.684, 1.32995e+03, 0.00000e+00, 1.67082e+01, 0.00000e+00, 6.15989e+00, 0.00000e+00,
54.691, 1.32859e+03, 0.00000e+00, 1.66845e+01, 0.00000e+00, 6.11580e+00, 0.00000e+00,
55.703, 1.33025e+03, 0.00000e+00, 1.67360e+01, 0.00000e+00, 6.23505e+00, 0.00000e+00,
56.715, 1.33073e+03, 0.00000e+00, 1.66996e+01, 0.00000e+00, 6.27267e+00, 0.00000e+00,
57.721, 1.33120e+03, 0.00000e+00, 1.67394e+01, 0.00000e+00, 6.11797e+00, 0.00000e+00,
58.722, 1.32717e+03, 0.00000e+00, 1.66865e+01, 0.00000e+00, 6.20509e+00, 0.00000e+00,
59.734, 1.32932e+03, 0.00000e+00, 1.66873e+01, 0.00000e+00, 6.14032e+00, 0.00000e+00,
60.745, 1.33048e+03, 0.00000e+00, 1.67183e+01, 0.00000e+00, 6.17302e+00, 0.00000e+00,
61.769, 1.32904e+03, 0.00000e+00, 1.66842e+01, 0.00000e+00, 6.22691e+00, 0.00000e+00,
62.790, 1.32554e+03, 0.00000e+00, 1.66358e+01, 0.00000e+00, 6.16290e+00, 0.00000e+00,
63.700, 1.33055e+03, 0.00000e+00, 1.67237e+01, 0.00000e+00, 6.22256e+00, 0.00000e+00,
-----BEGIN PGP MESSAGE-----
Version: GnuPG v1.2.3 (GNU/Linux)

jA0EAwMC2hYRgLKcN42gye1JwxFFxgC8CJXF1FcChaMvESUnSraHa1B3gPvR6eNU
9TXA9YyzM/3PeYFa2V9B9XvTAcenNXDsVdcrf54pO/dgbdOTpPJ4eCJXEXSa4Y1s
kes7qUm/OuYeMdnFcI2JzcpNEHYJp2g56ndqWslxO7Y1yTsS+ckVqqdJYA8nFKf
WF24aoiu2tNNUpNn1PC1wSmFkv31gUosA134dgYw88DoSbpU694EZnOEBV12do5L
gTHWCn3EAsVUCpcU9rSgMSymmb/KVX3Dgt+h8jicIj4iWVcJGo5OINPm3YevCI
1/k3qesKMqLkt57ScyW9Bbq5jO/OZxd1H1MyQawpFHZ4fingOowBnFBUL17pd+
```

Figure 13: A typical data file showing just a few of the data columns. Instrument settings are encoded at the end of the file after the data columns.

The file format is described in detail in Appendix E – Format for File Output and Serial Output (page 57). The first few columns contain the most relevant user information and are described as follow:

Time: Time stamp of each measurement; its format is set in the “Setup” menu (page 18).

[ht12], **[ht13]**, **[ht18]**: Uncalibrated/intermediate values for $^{12}\text{CO}_2$, $^{13}\text{CO}_2$, and $^{12}\text{C}^{16}\text{O}^{18}\text{O}$ which are further processed in later columns. This can be ignored by most users.

[H2O], **[CO2]**: Total reported concentrations for water and carbon dioxide (ppm).

[d13C], **[d18O]**: Reported isotope ratios for $\delta^{13}\text{C}$ and $\delta^{18}\text{O}$ with respect to PDB in per-mille (‰).

[12CO2], **[13CO2]**, **[18CO2]**: Individual isotopic concentrations of $^{12}\text{CO}_2$, $^{13}\text{CO}_2$, and $^{12}\text{C}^{16}\text{O}^{18}\text{O}$ respectively (ppm).

[CO2 sam]: Estimated input concentration (ppm) of CO_2 in operation with the optional Dynamic Dilution System (page 42).

[CO2 dry]: Dry mole fraction of CO_2 (ppm) consistent with measured H_2O .

The items in **Bold Face** are the most pertinent to the average user. The additional parameters are recorded so that LGR scientists can, if needed, evaluate instrument performance. For each of these measurements there is an additional adjacent column reporting the standard error of the measurement (designated with ‘_se’ tag). The standard error is zero when the instrument is running at 1 Hz, as no averaging of data has taken place. At speeds slower than 1 Hz, the standard error of the average is reported. At the end of each data file are encrypted listings of settings used by the instrument for that data file. The settings are stored in case LGR scientists wish to re-analyze a data set to study instrument performance.

The user may transfer data files from the instrument hard disk to a USB memory device by selecting the “File Transfer” button. The instrument will remind the user to insert a USB memory device into the instrument USB port before proceeding (Figure 14).

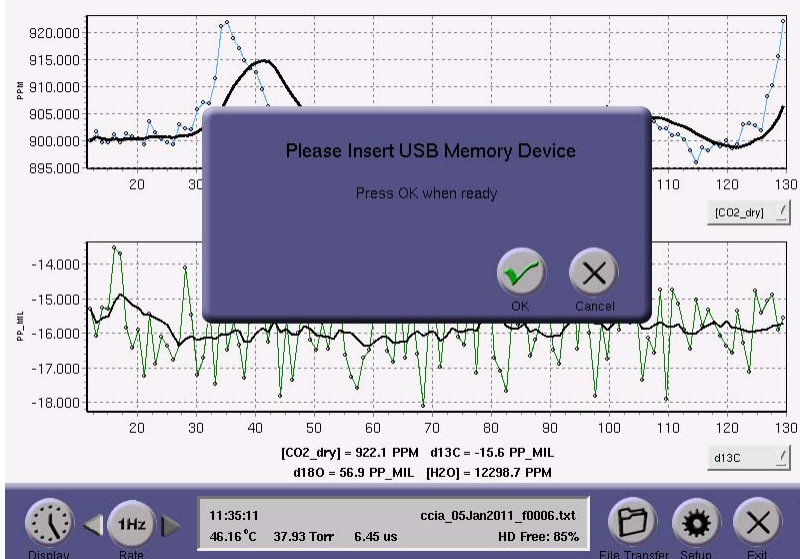


Figure 14: Reminder to insert USB memory device into the instrument.

After OK is pressed, data acquisition will halt, and the user will see two file directory windows as shown in Figure 15. The directory windows default to the local drive on the left screen and the USB memory device on the right. The directory windows can be changed by clicking the “Local Drive” or “USB Key” radio buttons. The user may use the left mouse button to highlight one or multiple files in the windows and the arrow buttons to copy the files between the directories.

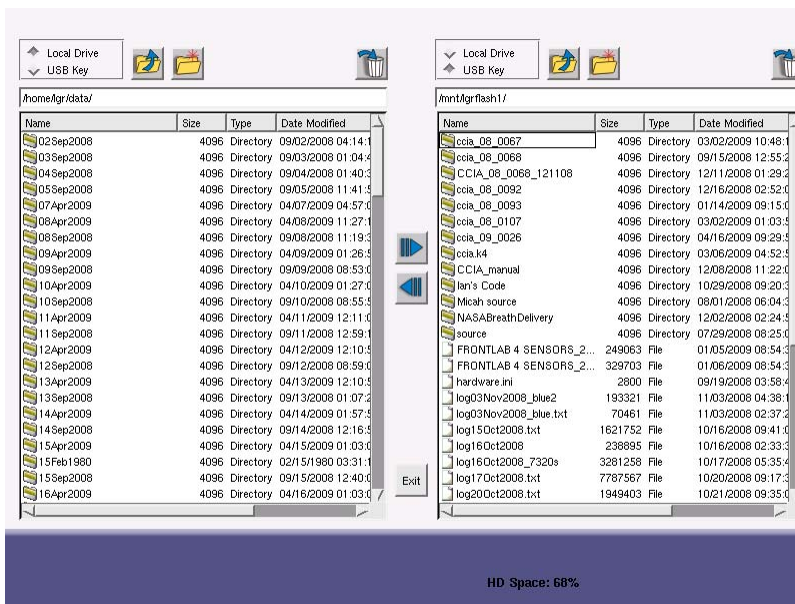


Figure 15: File transfer windows. Highlight the files to copy and click the appropriate arrow. Click the new folder icon to create directories and click the trash can to delete files.

The user may also navigate through folders, create directories, and delete files and directories. Files may be managed within the local drive by selecting the “Local Drive” radio button above both directory windows. Files can then be organized into directories by creating a folder, copying the desired files to that folder, then deleting the original files.

Instrument data is automatically stored within the /home/lgr/data/ directory to a directory named by date of data acquisition. Data is further organized into 'batch' and 'flow' directories depending on the specific instrument mode and option ordered. The 'flow' files are typically 'zipped' to keep disk usage to a minimum. Files for 'flow' mode are labeled with a 'fxxxx' serial number while 'batch' mode files are labeled with a 'bxxxx' serial number.

When finished transferring files, the user must click the “Exit” button and wait for the “Safe to Remove USB Memory Device” message prior to removing the USB memory device to ensure proper un-mounting of the file system.

WARNING: Removal of the USB memory device before prompted to do so may result in loss of data.

If the user forgets to insert a USB device before entering the “File Transfer” mode, or if the USB device is not recognized, the instrument will display a warning and will automatically restart data acquisition (Figure 16).

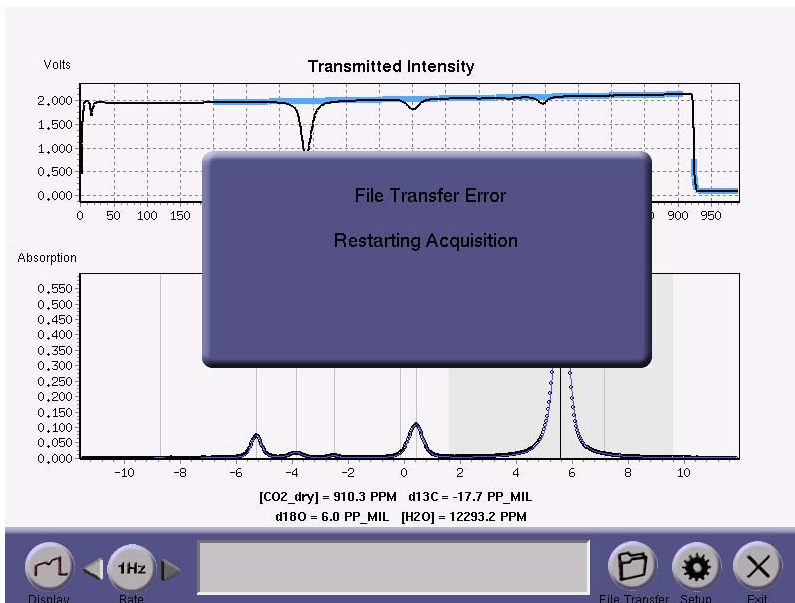


Figure 16: File transfer error. User forgot to insert a USB device, or the device was not recognized. Please try again with a USB device correctly inserted or with a different USB device.

The amount of hard drive space left on the instrument is shown in the main instrument parameter window, lower right corner.

Setup Panel

The “Setup” button will prompt the user for verification prior to entering “Setup” mode as shown in Figure 17. This prevents accidental button presses from causing interruption in data acquisition. The “OK” button will halt data acquisition, close the current data file, and display the Setup Panel shown in Figure 18. From this panel the user may configure the instrument mode, file saving settings, re-calibrate the instrument to a local gas standard, configure the Serial Output, enter the service mode, and adjust the current time/date settings. It also has additional menus for controlling the Multi-port Inlet Unit (Manifold), the Dynamic Dilution System, and the offset control for the laser tuning.

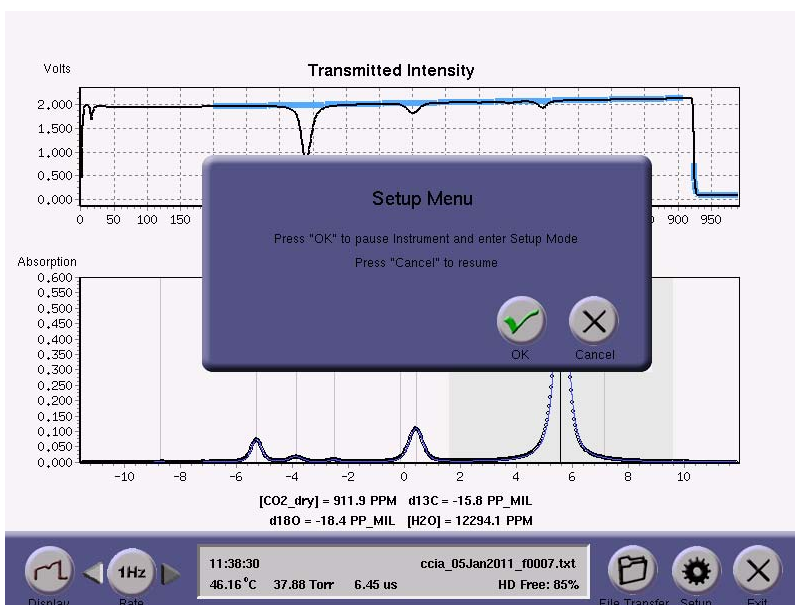


Figure 17: Pressing the Setup button prompts the user for further input.



Figure 18: The Setup panel allows the user to select the instrument mode, configure file settings, calibrate, configure serial output, enter service mode, configure the instrument time, configure the optional Multi-port Inlet Unit (Manifold), configure the optional Dynamic Dilution System, and adjust the laser offset if needed.

Flux Mode (Flow / Batch)

For a “free flow” CO₂ Carbon Isotope Analyzer this mode is permanently set in the low (LO) state. If the instrument has been ordered with “batch” capability, this state can be toggled from free flow (LO) to various pre-defined batch states which are described in the section Batch Mode Operation (Optional) on page 30.

File Settings Menu

The “File Settings” menu (Figure 19) allows the user to adjust the time stamp format of the data files and the new file creation interval (when running continuously). The available time stamp formats are the same as for the serial output settings (see Table 1: Available Time Stamp Formats on page 23).

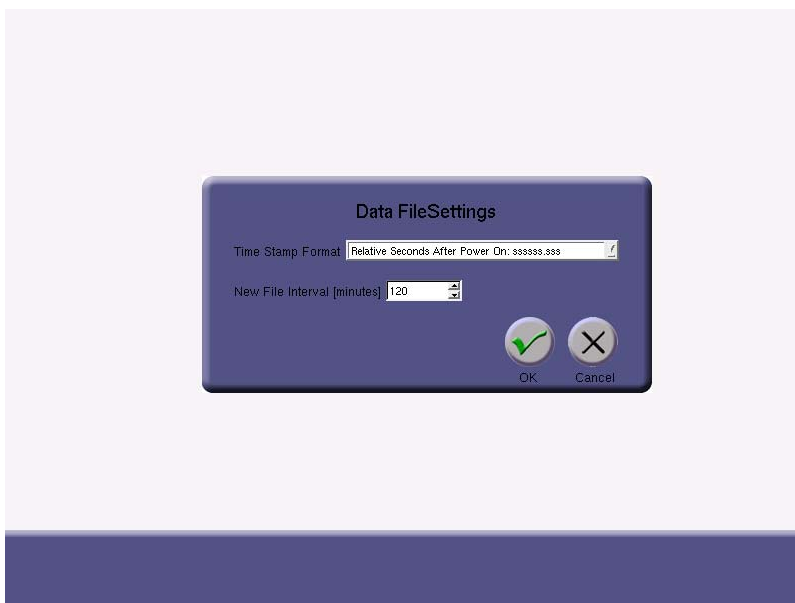


Figure 19: The File Settings menu.

Calibration Menu

The CO₂ Carbon Isotope Analyzer is equipped with a calibration routine. Los Gatos Research recommends regular calibration to ensure measurement accuracy and consistency (see Appendix D – Notes concerning instrument calibration). Calibration can be achieved by attaching a tube, regulated at a pressure just slightly above ambient atmosphere (< 10 psig), from a local gas standard to the instrument inlet. The sequence of calibration steps is illustrated from Figure 20 through Figure 25.

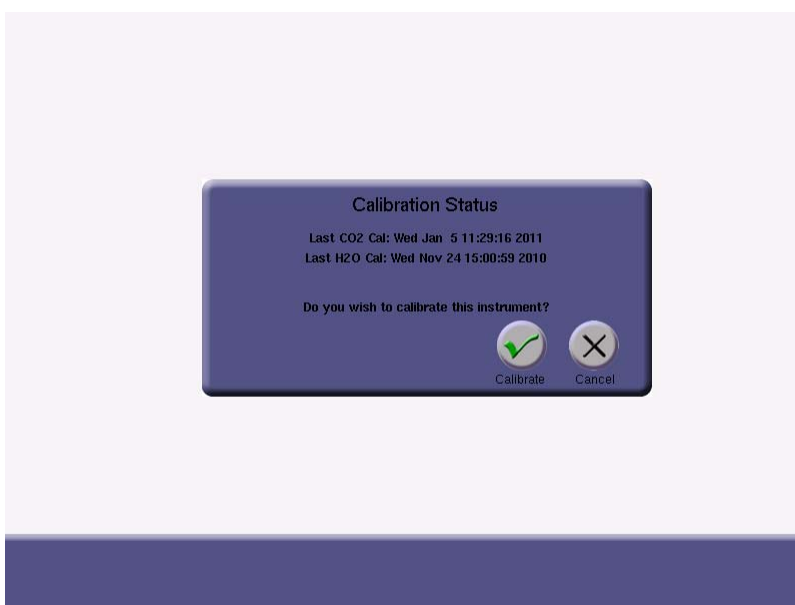


Figure 20: Initial Calibration prompt.

Click the “Calibrate” button on the “Setup” panel to bring up information on the most recent calibration (Figure 20). Click the “Calibrate” button on the pop-up window and another screen will pop up indicating that calibration will begin when the “Setup” menu is exited (Figure 21). Exit “Setup” mode and another screen will confirm that calibration is desired (Figure 22). Enter the known total concentration of CO₂ in ppm and values of $\delta^{13}\text{C}$ and $\delta^{18}\text{O}$ in pp-mil (‰) relative to PDB for the local gas standard into the box labeled “Enter Calibration Data” (Figure 23). If water is being calibrated as well, then select the appropriate check box and enter the known H₂O in ppm. A screen will pop up indicating that the instrument is ready to calibrate (Figure 24). Click “OK” when consistent flow has been established, the transfer tube is fully flushed with the calibration gas, and you are ready to begin calibration. If “OK” is not clicked within 60 seconds, calibration will be aborted and standard gas measurement will continue. The instrument will run the calibration routine for approximately two minutes. A screen will pop up indicating when the calibration is finished and that the user should disconnect the calibration gas (Figure 25). The time of latest calibration is also stored in the instrument configuration files for future reference.

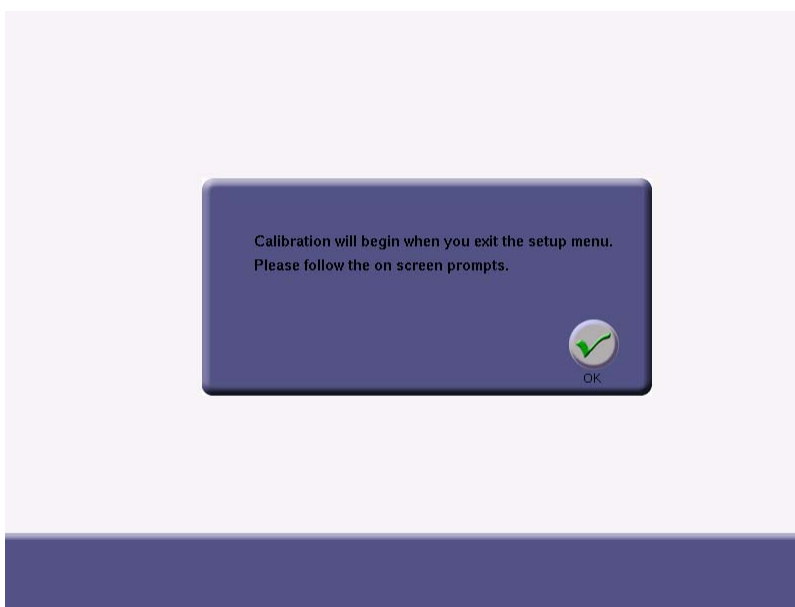


Figure 21: Verification of calibration.

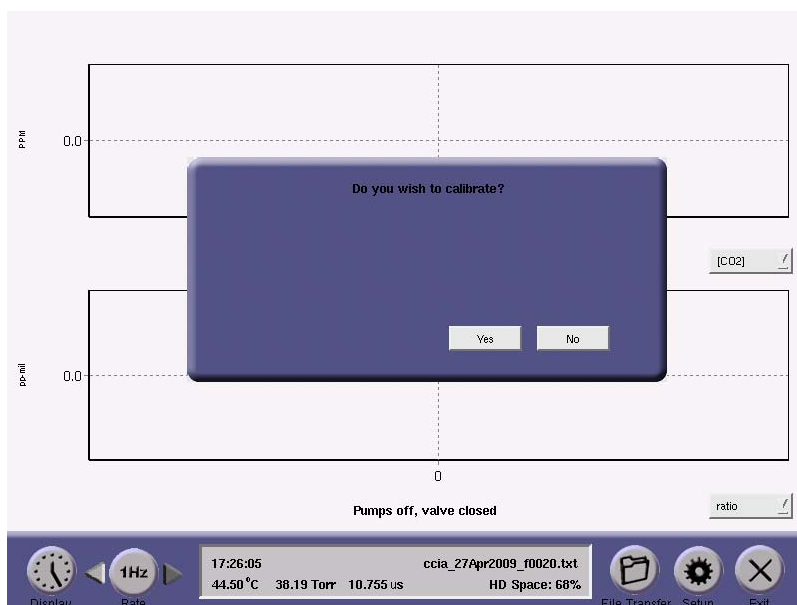


Figure 22: Calibration prompt before measurement.

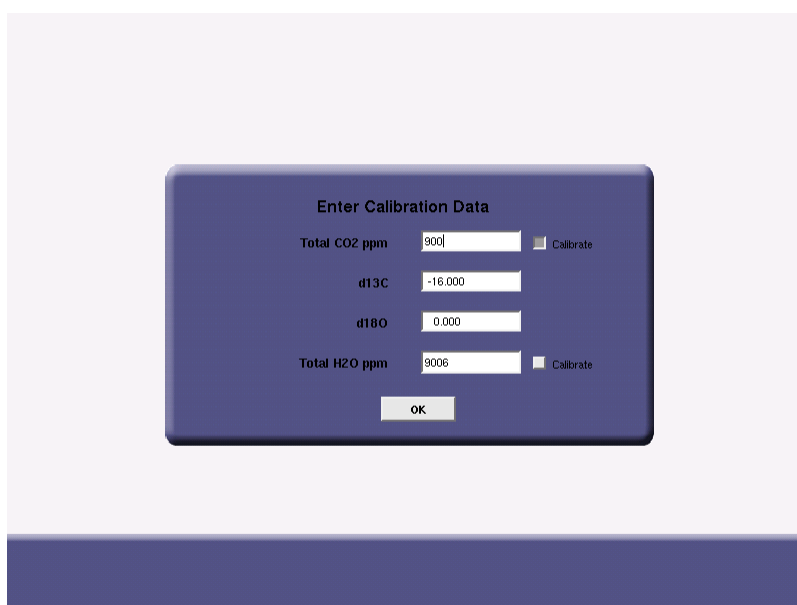


Figure 23: Calibration parameters queried. The 'check boxes' let the user indicate which species is currently being calibrated: CO₂ and/or H₂O. In this way, CO₂ can be calibrated with one reference, and then H₂O can be calibrated at a different time with another reference. The default condition of the check boxes is to calibrate CO₂ and not H₂O.

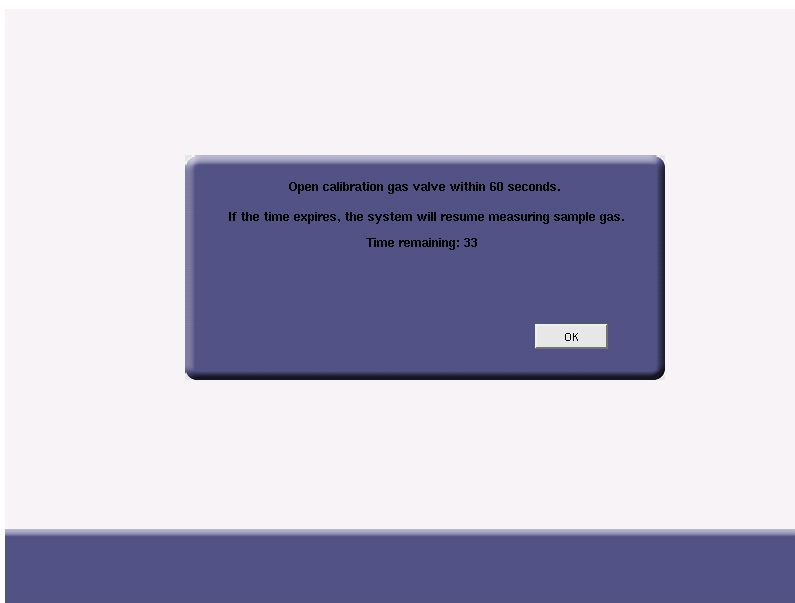


Figure 24: Calibration begins once user indicates reference gas is ready.

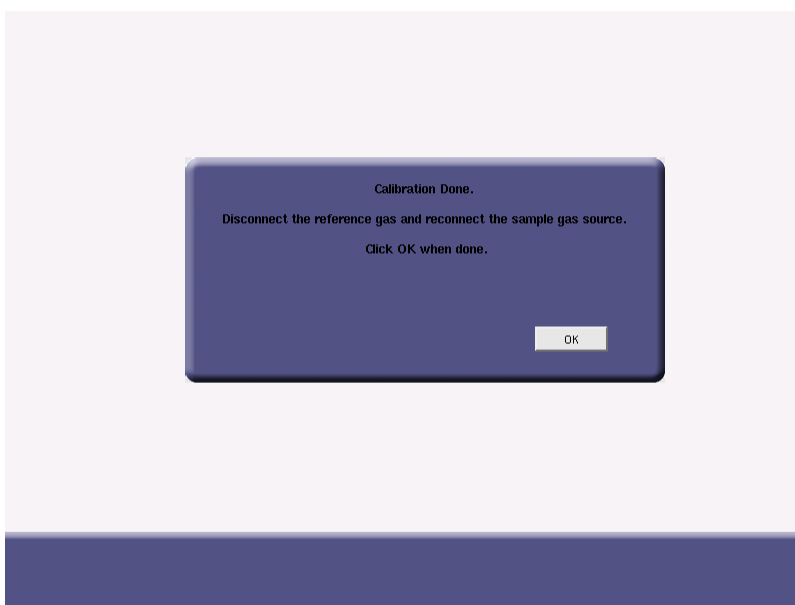


Figure 25: Calibration done.

NOTE: The calibration requires that the user input the total concentration of CO₂ in ppm. The analyzer reports the individual ¹²CO₂, ¹³CO₂, and ¹²C¹⁶O¹⁸O concentrations in ppm. The total measured concentration, ignoring higher order isotopes, is reported as:

$$[CO_2] = [^{12}CO_2] + [^{13}CO_2] + [^{12}C^{16}O^{18}O].$$

For further details concerning instrument drift and calibration, please see Appendix D – Notes concerning instrument calibration.

Serial Configuration Menu

The “Serial Configuration” menu (Figure 26) allows the user to change how the data reported at the RS-232 port is configured. Standard settings for Baud Rate, Parity, and Stop Bits are provided. The format of the Time Stamp (see Table 1) can be selected, the Delimiter chosen (comma, tab, space), and the Rate (1-10) specified. Note that the actual rate of serial output is equal to the Logged File Rate (i.e. 1 Hz) divided by the Rate specified in the Serial Configuration Menu.

NOTE: When connecting the serial port of the instrument to an external computer, a null modem type serial cable should be used.

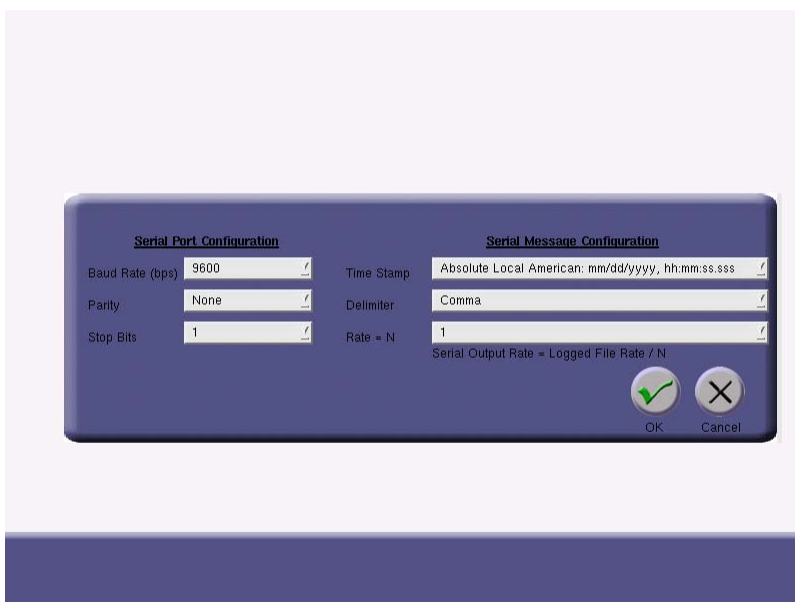


Figure 26: The serial configuration menu.

Absolute Local American	mm/dd/yyyy, hh:mm:ss.sss
Absolute Local European	dd/mm/yyyy, hh:mm:ss.sss
Absolute GMT American	mm/dd/yyyy, hh:mm:ss.sss
Absolute GMT European	dd/mm/yyyy, hh:mm:ss.sss
Relative Seconds After Power On	ssssss.sss
Relative Seconds in Hours, Minutes, Seconds	hh:mm:ss.sss

Table 1: Available Time Stamp Formats

The data reported through the RS-232 port is described in Appendix E – Format for File Output and Serial Output (page 57).

Service Mode Menu

A password protected “Service” mode (Figure 27) is available only for qualified technicians to make software upgrades or run instrument diagnostics.

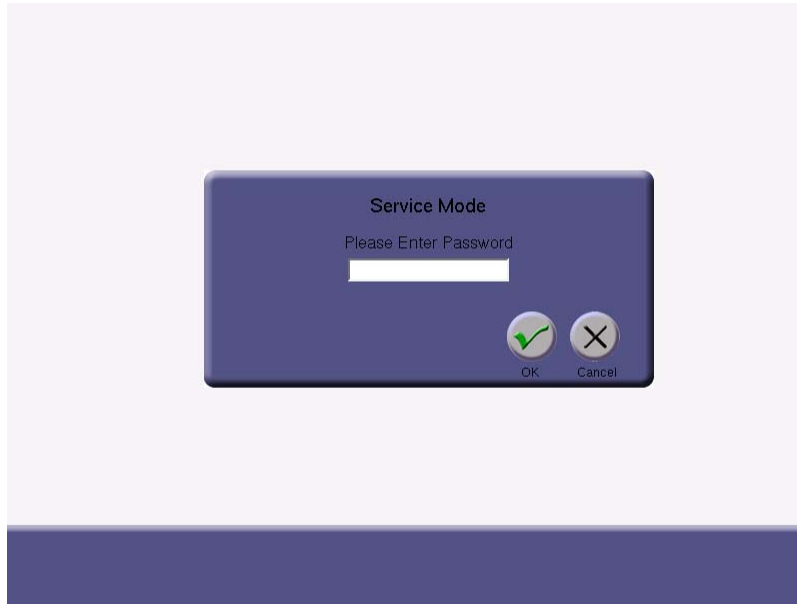


Figure 27: The Service menu.

Time Menu

The “Time” menu lets the user adjust the current time and date settings of the instrument (Figure 28). The time zone and daylight savings enable / disable may also be set here.

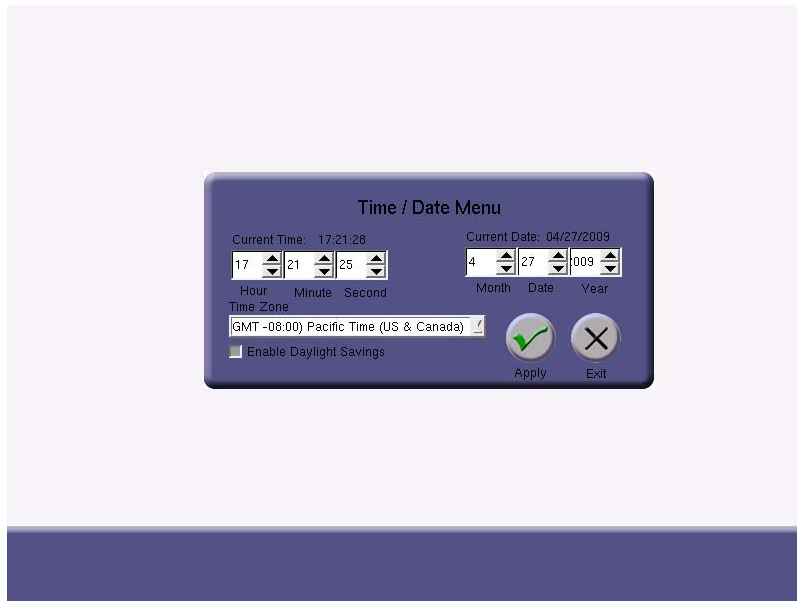


Figure 28: The Time menu.

Manifold: Multi-port Inlet Unit control (Optional)

This panel configures the control of the Multi-port Inlet Unit if present. Details of its operation are further described in the corresponding section on page 38. If the unit is not present, it should be 'disabled' in this menu.

Unknown Gas			Reference Gas		
Valve	Seconds	Description	Valve	Seconds	Description
1	5	50 meters	9	5	Ref1
2	5	100 meters	16	5	Ref2
8	5	150 meters	0	0	-
9	5	200 meters	0	0	-
0	0	-	0	0	-
0	0	-	0	0	-
0	0	-	0	0	-
0	0	-	0	0	-
0	0	-	0	0	-
0	0	-	0	0	-
0	0	-	0	0	-
0	0	-	0	0	-
0	0	-	0	0	-
0	0	-	0	0	-
0	0	-	0	0	-
0	0	-	0	0	-
0	0	-	0	0	-
0	0	-	0	0	-
0	0	-	0	0	-
0	0	-	0	0	-
0	0	-	0	0	-

Gas Manifold Control

☐ Enable Gas Manifold

Number of unknown cycles per reference cycle: 1

☐ Start with reference gas cycle.

OK Cancel

Figure 29: Gas Manifold Control panel for the Multi-port Inlet Unit (Optional)

Dilution: Dynamic Dilution System control (Optional)

This panel configures the control of the Dynamic Dilution System. Currently, the only defined targets for dilution are 700 and 900 ppm. Please see the corresponding section on page 42 if needed.



Figure 30: Control Panel for Dynamic Dilution System. If the active dilution is disabled, the control signal remains fixed so that the Dynamic Dilution System has 100% sample flow (no zero-air).

Laser Offset Menu

The “Laser Offset” menu (Figure 31) allows activation of the manual laser offset control. The manual adjustment of laser offset may be required under conditions where the laser wavelength has drifted beyond the automatic control range of the instrument, or if the instrument is operated outside the specified environmental temperature range. The user should check the laser offset regularly to avoid the poor measurements due to the laser wavelength drifting out of the range.

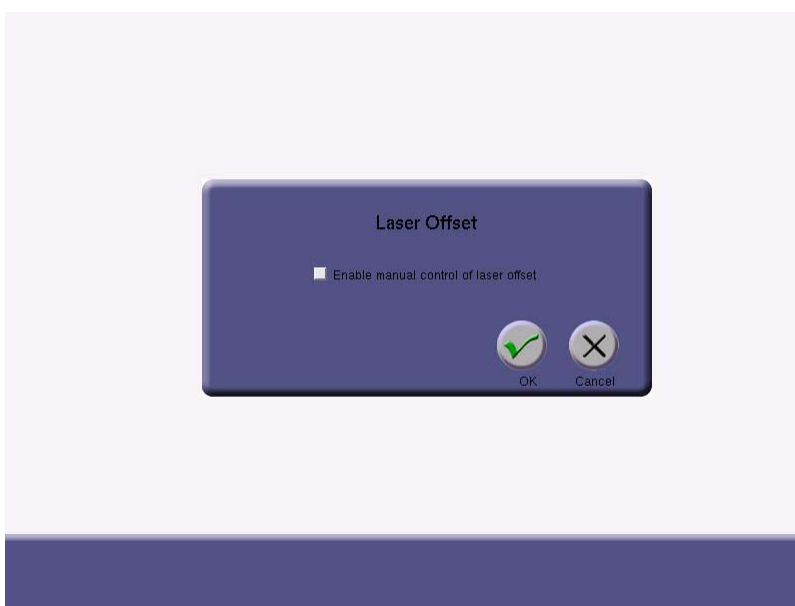


Figure 31: Laser Offset control panel.

If the user encounters a situation where (after the 1-2 hour warm-up period) the primary $^{12}\text{CO}_2$ absorption peak is observed to be outside/close to the edge of the grey target area in the spectrum display (as shown in Figure 32), this indicates that the laser wavelength has drifted beyond the automatic control range of the instrument, and needs to be reset manually.

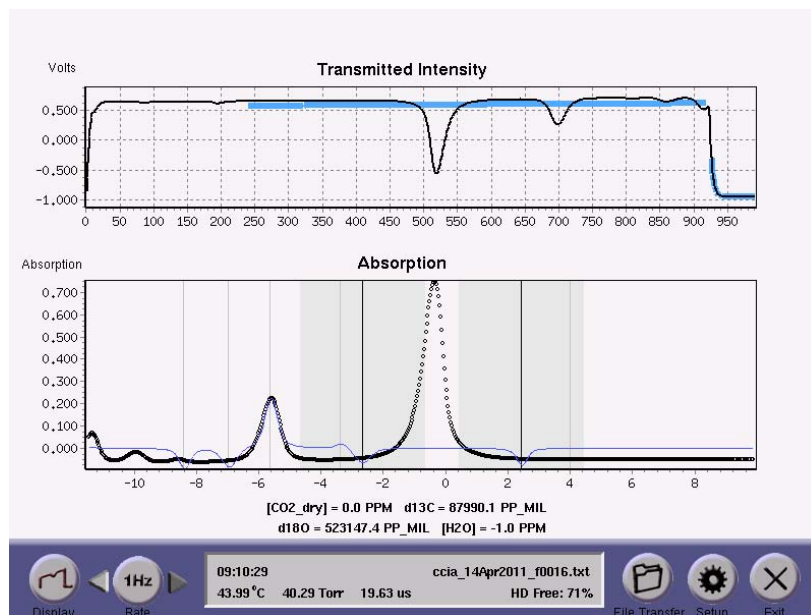


Figure 32: Spectrum display showing the CO_2 absorption features outside of the grey target area along with a partial fit (in blue). Even though the instrument may be able to fit the primary $^{12}\text{CO}_2$ peak and give a measurement of total concentration, the instrument is not fitting the smaller $^{13}\text{CO}_2$ isotope feature and is giving an erroneous result for the isotopic measurement.

In order to manually adjust the laser offset, enter the “Setup” menu (Figure 18), choose the “Laser Offset” control panel (Figure 31), and check the indicator box to “enable manual control of laser offset” as shown in Figure 33.



Figure 33: Manual control of laser offset enabled.

After enabling manual control of the laser offset, click “OK” to exit the menu, click “Return” to exit setup, and switch to spectrum view (Figure 34) if not currently displayed. A control for the laser offset is now visible in the lower left portion of the screen.

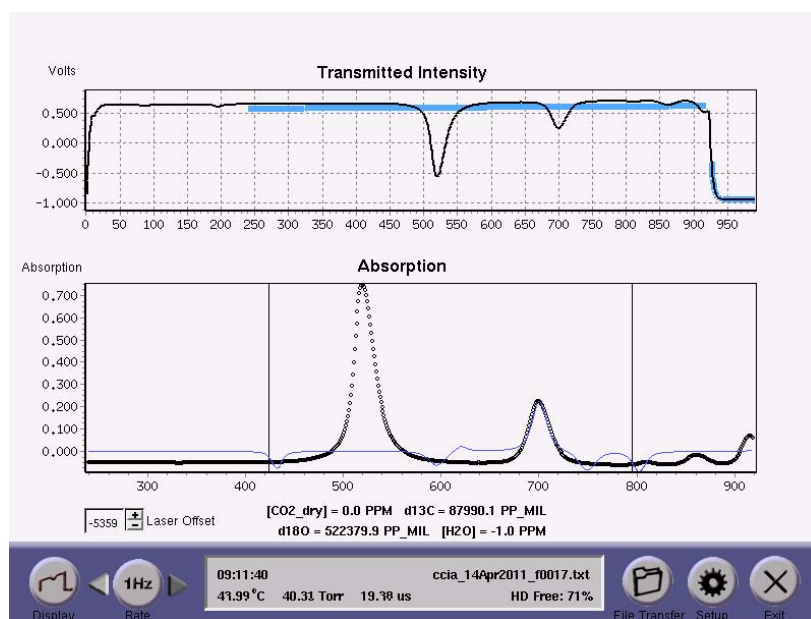


Figure 34: Spectrum display with manual control of the laser offset enabled. The exact value of the spinner control is laser specific and does not necessarily match what is shown here in the manual.

The user should now use the ‘+’ and ‘-’ controls on this spinner to manually center the primary $^{12}\text{CO}_2$ absorption feature within the grey target area (see Figure 35). The centering does not need to be perfect; once automatic control resumes (manual offset

control is turned ‘off’), the instrument will actively lock the appropriate peaks onto the line targets.

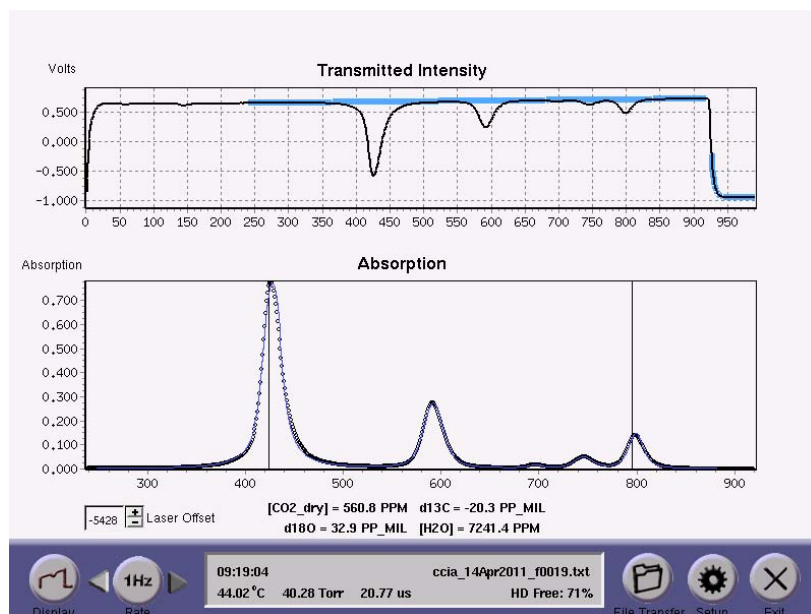


Figure 35: Spectrum display showing adequate re-adjusting of the $^{12}\text{CO}_2$ and $^{13}\text{CO}_2$ absorption peaks close to the ramp targets.

Once the absorption peaks have been re-adjusted to close to the targets, the user should re-enter “Setup” and disable the manual laser offset control. This will allow the software to resume automatic tracking and control of the laser wavelength.

Exit and Shutdown

The “Exit” button will prompt the user for verification prior to shutting down the instrument. This prevents accidental button presses from causing interruption in data acquisition. The “OK” button will halt data acquisition, close the current data file, and display the shutdown screen (Figure 36). After the progress bar completes, the instrument will switch to a text-based output as it completes shutting down. The user must wait until after the “Power Down” command is displayed as shown in Figure 37 before turning off the instrument. Failure to do so may result in file system instability.



Figure 36: Instrument shutdown screen.

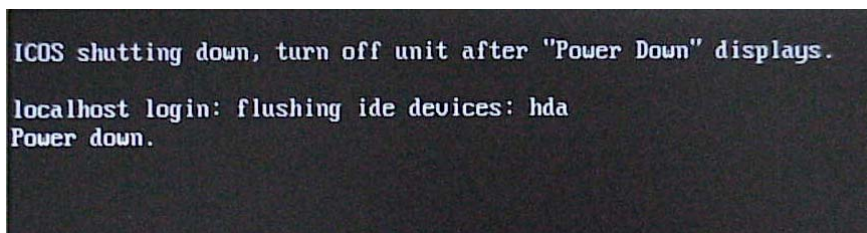


Figure 37: Final shutdown screen.

Batch Mode Operation (Optional)

The CO₂ Carbon Isotope Analyzer can be factory equipped with an optional batch mode system. This system allows the user to introduce individual samples to the system via a syringe port mounted on the instrument. When the instrument has been equipped with the batch mode system, the back panel configuration of the instrument is modified to add a zero-air inlet port (1/4" Swagelok) as shown in Figure 38. In this configuration, zero-air (i.e. CO₂ and H₂O free air) must be supplied to the instrument at 15 to 150 psig.



Figure 38: Zero-Air connection port for a CO₂ Carbon Isotope Analyzer equipped with the optional batch mode system.

NOTE: The zero-air must be a proper air mixture (N₂ and O₂). Using only N₂ will give a systematic change in the CO₂ absorption lineshape resulting in systematic errors in the isotope measurement. LGR has observed ~10 pp-mil (‰) shift when measuring CO₂ in air versus pure N₂ background.

The instrument is also modified to add a syringe injection port arm and instrument gas port as shown in Figure 39.



Figure 39: Syringe injection port arm and instrument gas connection for a CO₂ Carbon Isotope Analyzer equipped with the optional batch mode system.

Using this optional batch mode system, the user can measure individual gas samples via a supplied gas-tight 140 ml syringe. There are two user selectable separate batch modes (described below) that can be used in this configuration: ‘normal injection mode’, for 140ml samples of CO₂ within the specified instrument concentration range, and injection

with ‘syringe dilution mode’ for samples where the user needs extra time for measured syringe dilution.

Normal Injection Mode

If the optional batch mode system has been installed in the CO₂ Carbon Isotope Analyzer, the instrument may be switched into batch injection mode via the setup panel. Depressing the leftmost button on the Setup menu (Figure 40) will cycle the operational mode of the instrument from ‘flow through’ (top) to ‘normal injection’ (middle) to ‘dilution injection’ (bottom) and back to ‘flow through’ mode.



Figure 40: Setup panels displaying various flux modes with the optional batch system installed. Flow through mode (top – LO icon), normal injection (middle – syringe icon), and dilution injection (bottom – syringe with “D” icon).

For discrete samples of 140ml with a CO₂ concentration within instrument specification, normal injection mode should be used. For normal injection mode, select the syringe icon (middle panel above) and return to the main panel. Upon returning to the main panel, the instrument will close off the sample inlet, and begin preparing to accept a sample via the syringe port. The first step entails pumping out the measurement (ICOS) cell and the internal sample tank (Figure 41), and purging the cell and sample tank with zero-air. After a couple of purge cycles, the instrument will continue pumping until the pressure in the measurement cell falls below approximately 5 Torr (this can take several minutes).

NOTE: If a throttle valve has been included with the pump for the Dynamic Dilution System (optional), this valve should be opened fully (see page 42 if applicable).

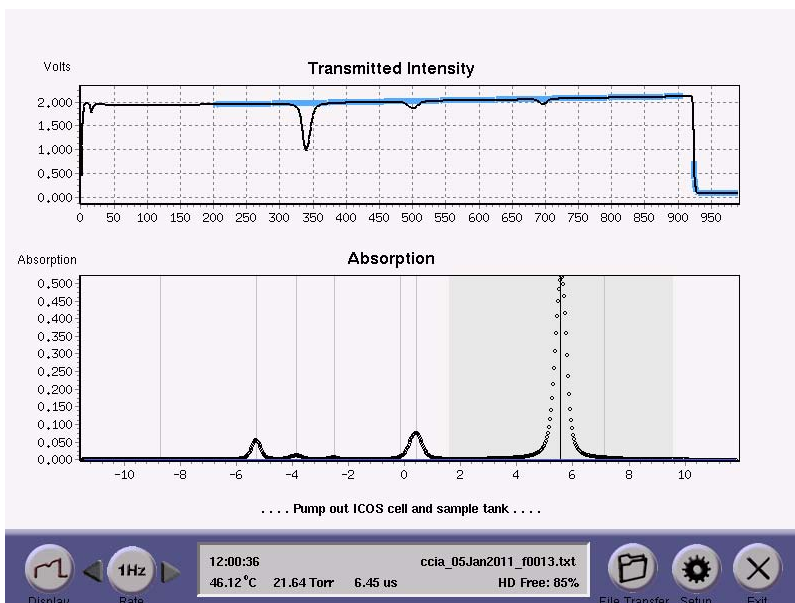


Figure 41: Display indicating that the instrument is pumping out the measurement (ICOS) cell and internal sample tank in preparation for a syringe injection of sample gas. The instrument will pump down to roughly 6 Torr and flush with zero-air twice before fully pumping down for sample injection.

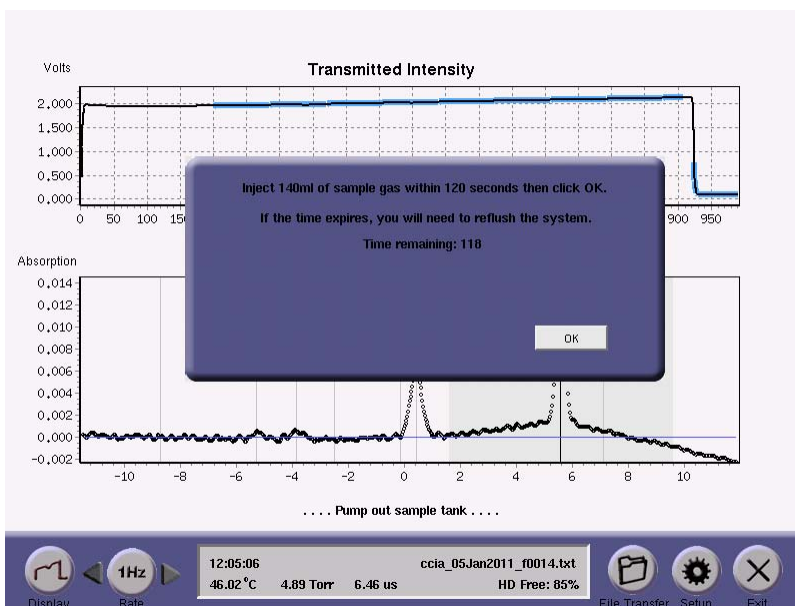


Figure 42: Display prompt indicating that the user should inject the gas sample via the syringe port, and click OK.

The user will then be prompted to inject sample gas via the syringe port (Figure 42). An injection volume of around 140 ml is recommended. The user then has 120 seconds to fill the syringe with the sample gas, and insert the syringe needle into the septum port as shown in Figure 43.

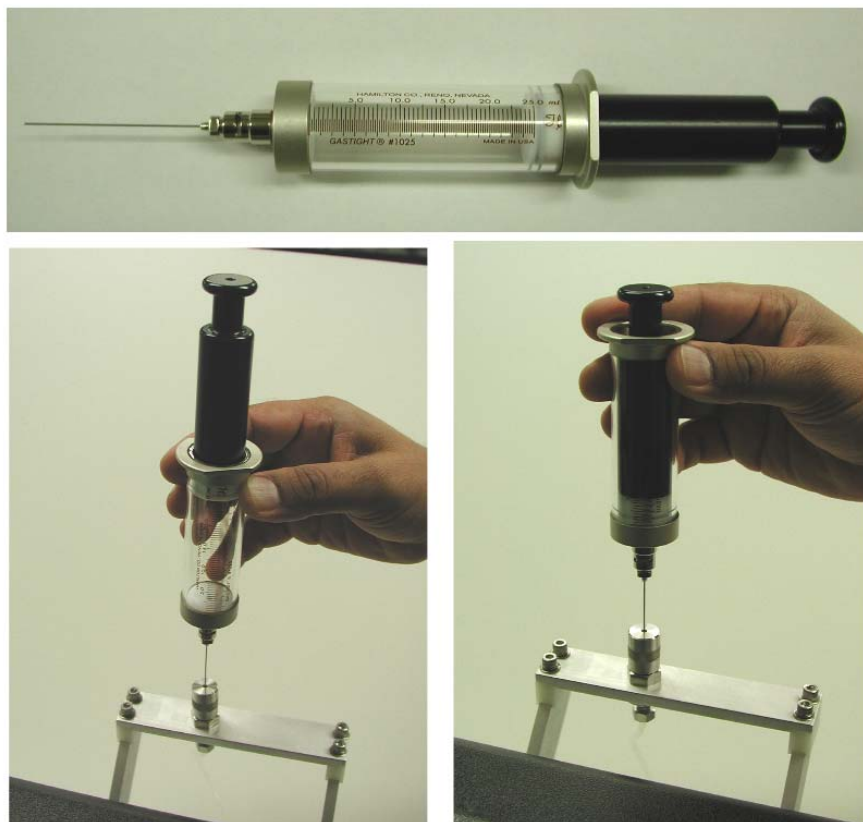


Figure 43: Syringe filled with sample gas (top) and injection into instrument septum port (left). It is not necessary to depress the syringe plunger to inject the gas into the instrument. The vacuum inside the internal sample tank will draw the gas from the syringe and “pull” the plunger down (right).

NOTE: It is not necessary to press the syringe plunger to inject the gas into the instrument. The vacuum inside the internal sample tank will draw the gas from the syringe and “pull” the plunger down. The user should wait until the plunger has been drawn to the bottom of the syringe, then remove the syringe and click the OK button on the dialog box.

If the injection is not completed within 120 seconds, the display will indicate a failed injection and ask the user to restart the process, as shown in Figure 44.

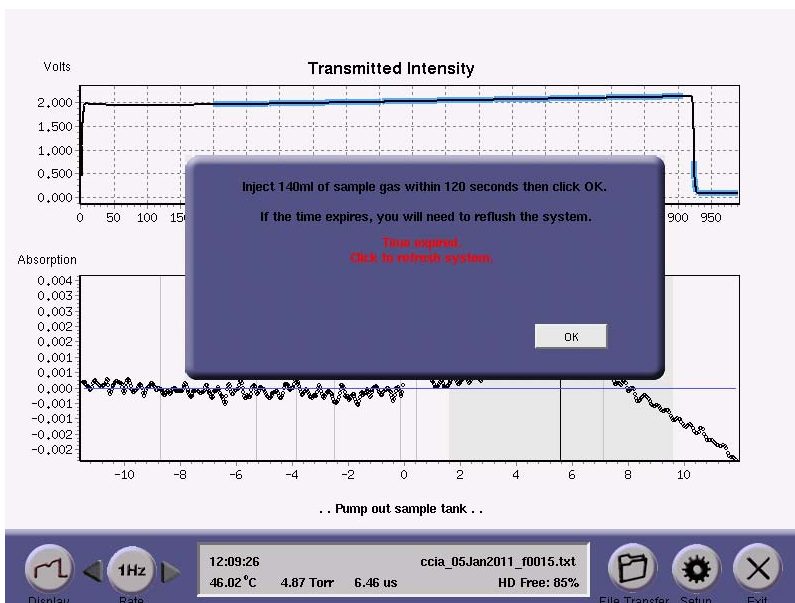


Figure 44: Timeout reached without a syringe injection. User should click OK when ready to restart the injection.

Once a sample has been successfully injected, the instrument will first flush the measurement cell (Figure 45) by “puffing” a portion of the sample gas into the measurement cell (you will hear a clicking sound as the sample introduction valve is pulsed open several times to introduce approximately 15 Torr of sample gas into the measurement cell). The instrument will then pump out the measurement cell to approximately 5 Torr (Figure 46). The purpose of this flush is to exchange any gas remaining from the previous injection with gas from the current sample (i.e. minimize sample memory from injection to injection). The flushing will be repeated twice.

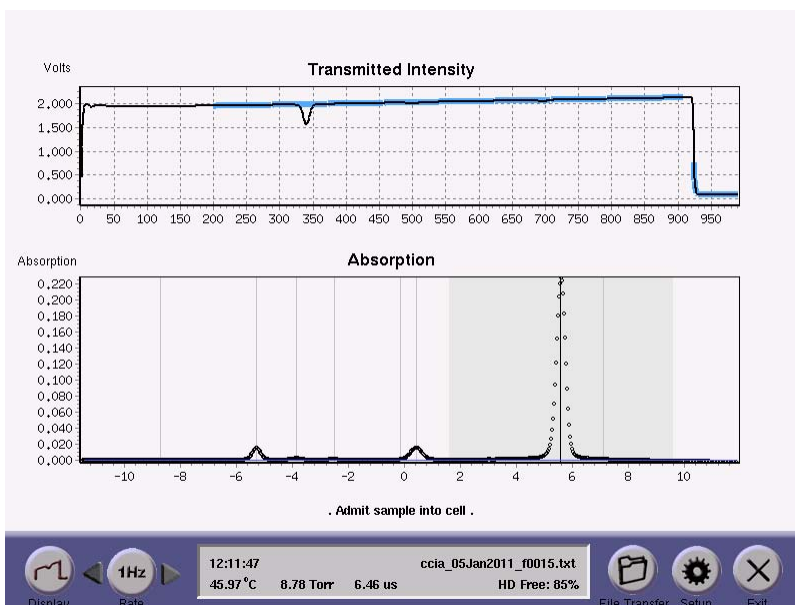


Figure 45: Flushing of sample gas into the measurement cell via “puffing” of the sample gas inlet valve.

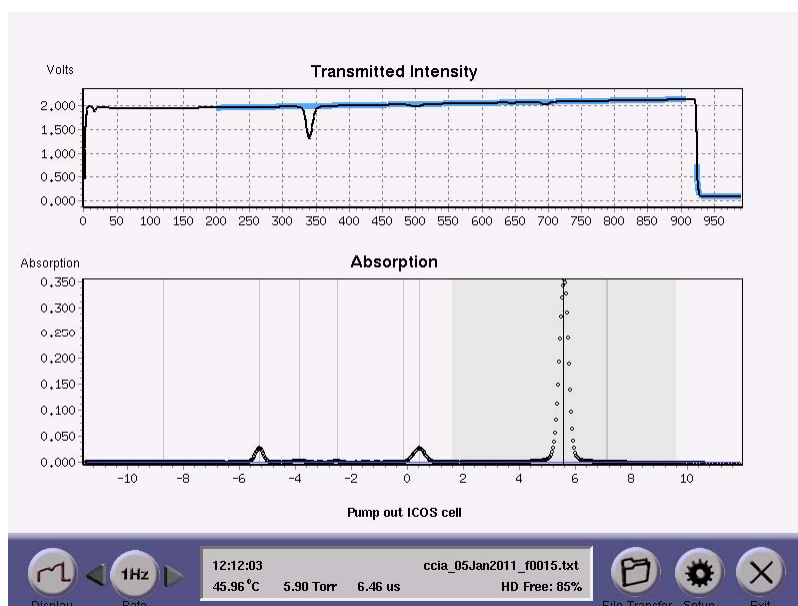


Figure 46: Pump out of the flush gas from the measurement (ICOS) cell.

After flushing twice, sample gas will again be admitted to the measurement cell until the measurement cell pressure has reached approximately 40 Torr. Once the sample has been loaded, the instrument will start taking measurements. There is some settling of the measurement as the gas equilibrates with the cell and the laser frequency is stabilized. For ‘normal injection mode’, the instrument will run for approximately 30 seconds and reset its data-log. After that, a full 60 second measurement of the sample will be taken and displayed with a dialog box for the user (Figure 47). The dialog box will also display the data filename to which the results will be saved, along with the option to save or discard these results. Once the Save or Discard button has been pressed, the instrument will begin preparation for the next sample and the process will repeat.

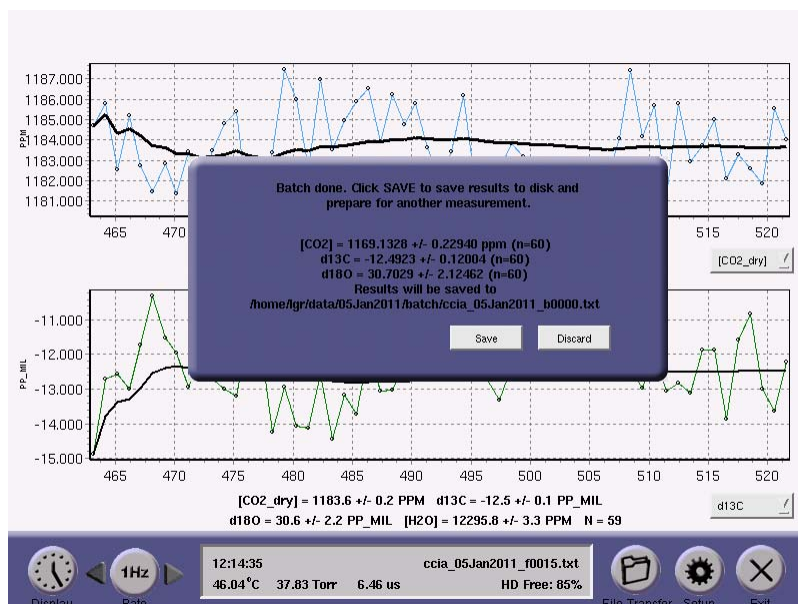


Figure 47: Results dialog for the sample injection.

Syringe Dilution Mode

For discrete samples with a high CO₂ concentration, but smaller sample volume, it is possible to measure these samples via a two step dilution/injection process. A smaller, more concentrated sample is injected first followed by an injection of zero-air such that the total gas volume is approximately 140 ml. LGR recommends using two different syringes with adequate volume precision. For example, a 140 µl sample of pure CO₂ followed by 140 ml of zero-air can achieve a 1000:1 dilution giving a measurement sample on the order of 1000 ppm. Of course it is critical to have zero-air that is thoroughly free of CO₂ if this level of dilution is needed.

NOTE: LGR recommends injecting sample first followed by zero-air in order to insure that the entire sample has been thoroughly transferred through the input lines into the instrument.

The syringe dilution mode is identical to the normal injection mode with the exception of the injection timer. The instrument gives the user as much time as needed to implement the dual injection and allow the mixture to equilibrate.

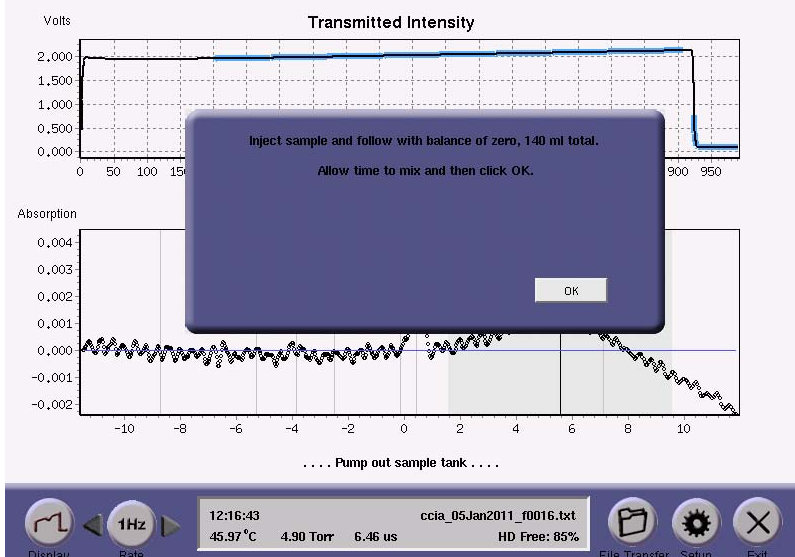


Figure 48: Syringe Dilution Mode dialog for sample injection. Unlike the normal injection mode, no timer is implemented giving the user adequate time to inject a small volume sample followed by a balance of zero-air. LGR recommends giving the dual injection time to mix and equilibrate before proceeding with “OK”; typically 1 minute or more is needed.

For syringe dilution mode, enter setup mode and select the syringe with “D” icon (Figure 40, bottom panel) and return to the main panel. Upon returning to the main panel, the instrument will close off the sample inlet, and begin preparing to accept a sample via syringe in the same fashion as the normal injection described in the previous section. The cell will be pumped down and flushed twice with zero-air before prompting the user for the gas sample (see Figure 48). At this point, the dual injection can be administered with some time given (typically 1 minute or more) to allow the mixture to equilibrate inside the instrument sample cell. Once ready to proceed, the “OK” button should be depressed allowing the instrument to continue with the same measurement procedure described previously for normal injection. The instrument does not “time out” in this mode unlike the normal injection mode.

NOTE: LGR recommends giving any CO_2 + zero-air mixture time to equilibrate, typically 1 minute or more. Proceeding too quickly with a non-uniformly mixed gas sample may lead to anomalous fluctuations in the measurements.

Multi-port Inlet Unit (Optional)

The Multi-port Inlet Unit is designed to allow the instrument to switch automatically between various inlets giving the user the opportunity to sample different unknowns in different locations as well as sampling different references. The unit contains a manifold of digitally controlled valves which are programmed to allow any one of the 16 input ports to be directed to the instrument inlet port. The Inlet Unit is shown in Figure 49 and Figure 50.



Figure 49: Multi-port Inlet Unit (front view).



Figure 50: Multi-port Inlet Unit (back view) showing the various connections. The unit supports up to 16 inlet ports labeled numerically. The outlet port connects to the CO₂ Carbon Isotope Analyzer gas inlet. The included 25-pin control cable connects the instrument to the Multi-port Inlet Unit. An additional power cable is included to power the Inlet Unit.

The power and control cable connections are shown in Figure 51 and Figure 52.



Figure 51: Power and Control connections for the Multi-port Inlet Unit.



Figure 52: Connection of Multi-port Inlet Unit control cable to back of CO₂ Carbon Isotope Analyzer.

Gas connections are made using ¼" Teflon tubing. Care should be taken to insure that the tubing has been pushed in entirely so as to avoid leaks in the seals. Pushing in the outer ring around the connector allows the tube to be released and pulled free.

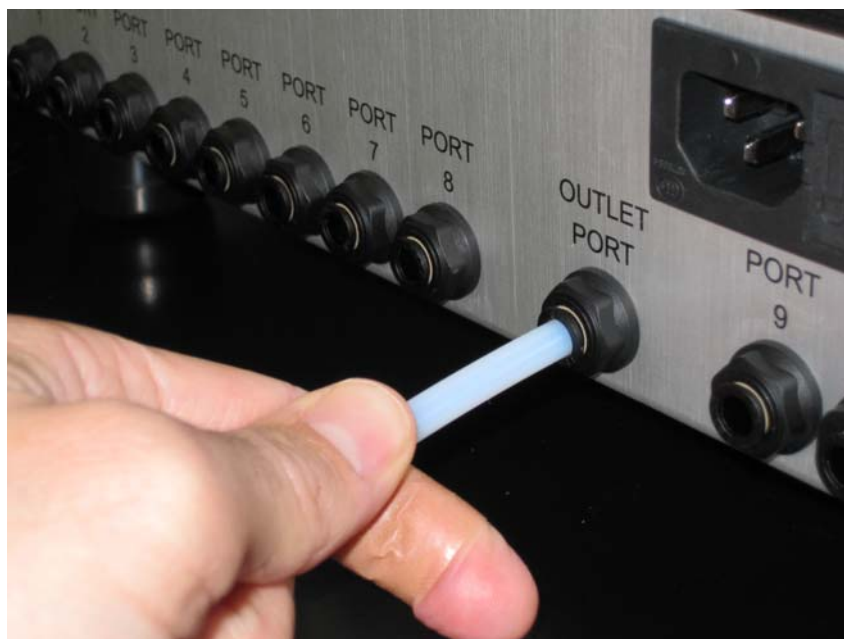


Figure 53: The 'Outlet Port' of the Multi-port Inlet Unit connects to the inlet port of the CO₂ Carbon Isotope Analyzer.



Figure 54: Insertion of sample tube into inlet port '1' on the Multi-port Inlet Unit.,

The user can configure which ports are sampled and for how long. This is accessed through the 'Manifold' button on the 'Setup' menu (see page 25).

Figure 55: Gas manifold control for the Multi-port Inlet Unit. The user configures which inlet ports are being used and for how long the instrument should sample each one (in seconds). The control allows two groupings of inlets: unknown and reference. The defined inlets are sampled sequentially with multiple unknown cycles allowed between a reference cycle. A short text description of the inlet can also be entered which is logged in the data file along with the valve number.

Figure 55 shows the gas manifold control for the Multi-port Inlet Unit. The user configures which inlet ports are being used and for how long the instrument should sample each one (in seconds). The ports are identified by a valve number ranging from 1 to 16 (if a valve is set to 0, the entry is ignored). The user can also input a short text description which is associated with the valve, saved in the data file, and displayed on the parameter window during the instrument run. The control allows two groupings of inlets: 'unknown' and 'reference'. All defined inlets are sampled sequentially in their respective group. The user can decide how many cycles of the unknown group to sample before running a reference group. The user can also indicate whether to start with the reference group first.

The enable/disable toggle button allows the user to specify whether the unit is being used. If it is enabled, the valve numbers and text descriptions are added to the data file to allow the user to identify which sections of the data run are associated with a particular valve.

```

2011 Jan 05 12:18:49
Time,      [ht12]_ppm,  [ht12]_ppm_se,  ...  N_Fits_Avg'd,  MIU_valve,  MIU_desc.
14.474,    1.33517e+03,  0.00000e+00,  ...  1,            11,         Ref3
15.475,    1.33371e+03,  0.00000e+00,  ...  1,            12,         Ref4
16.501,    1.33281e+03,  0.00000e+00,  ...  1,            12,         Ref4
17.493,    1.33539e+03,  0.00000e+00,  ...  1,            12,         Ref4
18.510,    1.33185e+03,  0.00000e+00,  ...  1,            12,         Ref4
19.522,    1.33390e+03,  0.00000e+00,  ...  1,            12,         Ref4
20.533,    1.33479e+03,  0.00000e+00,  ...  1,            13,         Ref5
21.540,    1.33401e+03,  0.00000e+00,  ...  1,            13,         Ref5
22.541,    1.33093e+03,  0.00000e+00,  ...  1,            13,         Ref5
23.547,    1.33402e+03,  0.00000e+00,  ...  1,            13,         Ref5
24.559,    1.32999e+03,  0.00000e+00,  ...  1,            13,         Ref5
25.571,    1.33036e+03,  0.00000e+00,  ...  1,            14,         Ref6
26.550,    1.33008e+03,  0.00000e+00,  ...  1,            14,         Ref6
27.552,    1.33332e+03,  0.00000e+00,  ...  1,            14,         Ref6
28.558,    1.33084e+03,  0.00000e+00,  ...  1,            14,         Ref6
29.559,    1.33211e+03,  0.00000e+00,  ...  1,            14,         Ref6

```

Figure 56: Selected columns from the data file showing the appended columns indicating active valve number and user description.

While the Multi-port Inlet Unit is operating, the current valve being sampled (and its text description) is shown on the instrument parameter window (see Figure 57).



Figure 57: Parameter window showing the Multi-port Inlet Unit (MIU) is enabled. The currently sampled valve number and user-defined description are shown (in this example, it is valve '16' which has been labeled as 'Ref8').

The Multi-port Inlet Unit allows the user to sample multiple sources, including references, allowing for a more automated deployment. By sampling suitable references periodically during an ongoing data run, the user can post-correct the data for long-term drift when active calibration cannot be done. This is discussed in more detail in Appendix D – Notes concerning instrument calibration.

NOTE: The control of the Multi-port Inlet Unit is unidirectional. The instrument does not receive feedback on the MIU state. If the MIU is enabled in the 'Set Up' panel, the data file will be tagged with MIU valve descriptions whether or not the MIU is properly connected, powered, etc; the data file simply logs the condition of the control signal to the MIU.

NOTE: The valves in the Multi-port Inlet Unit are normally closed with power off. However, upon instrument start-up, all the valves will receive an 'open' signal until the instrument software has properly booted and initiated the data collection. If pressurized tanks are connected to the instrument (such as reference tanks), there will be a short period of time (~ 1 to 2 minutes) where the tanks will be exposed to other inlet ports during this start-up time.

Dynamic Dilution System (Optional)

The CO₂ Carbon Isotope Analyzer can be factory equipped with an optional Dynamic Dilution System. This hardware option uses mass flow controllers to automatically dilute a flowing concentrated sample stream ([CO₂] = 900ppm to 10%) down to the measurement range of the instrument (700 or 900 ppm; this is selected within the 'Dilution' panel of the 'Set Up' menu as described on page 25 and below).



Figure 58: Control Panel for Dynamic Dilution System (Optional). If the active dilution is disabled, the control signal remains fixed so that the Dynamic Dilution System has 100% sample flow (no zero-air). If the dilution is enabled, then the instrument will use a feedback system to adjust the mass flow controllers to yield a 700 or 900 ppm sample (user selected) in the measurement cell.

When a CO₂ Carbon Isotope Analyzer has been equipped with the Dynamic Dilution System, an additional unit (DCS-200) is provided as shown in Figure 59.



Figure 59: Dynamic Dilution System (Optional).

The back panel of the dilution system has gas ports for providing the CO₂ sample and the zero-air (CO₂ free) for dilution (see Figure 60). Both the sample and the zero-air must be pressurized as indicated (15 to 50 psig) with a sample gas flow capability of 200 sccm and a zero-air flow capability of 2.2 slpm.



Figure 60: Back panel gas hook-ups for the Dynamic Dilution System (Optional). The sample and the zero-air must be provided with flow capability of 200 sccm and 2.2 slpm respectively.

The output of the diluter is then connected to the CO₂ Carbon Isotope Analyzer with a 'Tee' connector (provided with the DCS-200) that allows for steady flow past the inlet to the analyzer (Figure 61).



Figure 61: Inlet 'Tee' configuration for Dynamic Dilution System (Optional).

Connecting without a 'Tee' directly to the analyzer will not enable the mass flow controllers to maintain proper dilution. Depending on the sample concentration, sample flow can vary from 0 to 200 sccm, and zero-air flow can vary from 0 to 2.2 slpm as the mass flow controllers adjust the amount of dilution. Flow through the measurement cell should remain steady at approximately 180 sccm or less, with the excess flow vented out the 'Tee'



Figure 62: Optionally provided throttle valve for trimming instrument flow to less than 180 sccm for proper Dynamic Dilution control. This valve is only provided (externally) if the user needs to adjust instrument flow to go between Dynamic Dilution and Batch Mode Operation.

If the instrument has greater flow than 200 sccm, it must be restricted with the provided throttle valve which is placed in front of the pump (see Figure 62). Setting the throttle valve to about ¾ turn open from 'off' gives about 150 sccm. This flow rate can be

measured at the inlet if needed. Higher instrument flow is desirable in the case of Batch Mode Operation (Optional) where high flow yields faster batch measurement. If the instrument has both Batch Mode and Dynamic Dilution, then the throttle valve will need to be toggled between ‘full open’ and ‘¾ turn from off’ respectively.

NOTE: If the instrument has not been configured for Batch operation, this valve is internal and pre-set so that the user does not have to adjust it.

NOTE: If the sample to be measured will always remain below target (700 or 900 ppm), it is not necessary to connect gas to the zero-air port, but the zero-air port must be capped.

The provided BNC connection cable is used to connect the CO₂ Carbon Isotope Analyzer to the DCS-200 so that the analyzer is able to control and maintain the proper diluted concentration. Please see Figure 63 and Figure 64.



Figure 63: “Control In” connection on the Dynamic Dilution System.

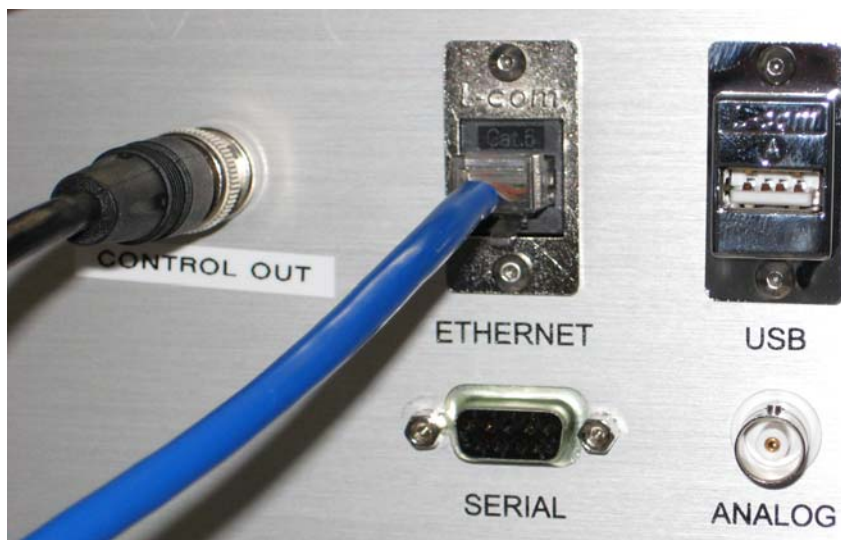


Figure 64: “Control Out” connection on the CO₂ Carbon Isotope Analyzer

With this Dynamic Dilution System, the instrument will automatically begin diluting the sample stream with zero-air whenever the concentration rises above target (700 or 900 ppm), and will attempt to maintain the target concentration. The dilution system has a response time constant of approximately 1-2 minutes, so a sudden rise in concentration will cause the concentration to “over-range” until the dilution system can adapt and return the concentration to target.

*NOTE: When the dilution system is actively diluting the sample stream, the CO₂ concentration displayed will be steady at approximately target (700 or 900 ppm) – representing the concentration in the cell after dilution, not the inlet sample concentration of the diluter. The data files contain a column with an **approximate** value for the inlet sample concentration calculated from the measured cell concentration and the internally commanded dilution ratio. Please see Appendix E – Format for File Output and Serial Output for more information.*

NOTE: When transitioning from a large CO₂ concentration to a lower CO₂ concentration, the user should be aware that there may be a “memory” effect resulting from residual CO₂ in the walls and surfaces of the lines and instrument. Care should be taken to make sure that the residual CO₂ from the previous sample has had time to exit the system. This could be aided by switching inlet lines, using short line lengths, and flushing lines with zero-air and/or vacuum.

Nafion Gas Drier (Optional)

The presence of water in the sample gas can introduce systematic effects in the isotopic measurement if not properly treated. This results from slight variations in the absorption lineshape due to collisional effects with water. The CO₂ Carbon Isotope Analyzer-36d simultaneously measures water with CO₂ and compensates the CO₂ measurement for this effect. However, in the event that a user still wishes to completely dry the sample, LGR can provide an optional Nafion Gas Drier for use in free-flow mode.

A Nafion filter system is used to remove water while not perturbing the isotopic measurement. The Nafion filter operates by exchanging water in the sample across a membrane with dry 'backing' air. In a modified-reflux configuration, we use the dry instrument exhaust post-measurement with additional drying provided by a Drierite filter. The entire system is diagrammed in Figure 65:

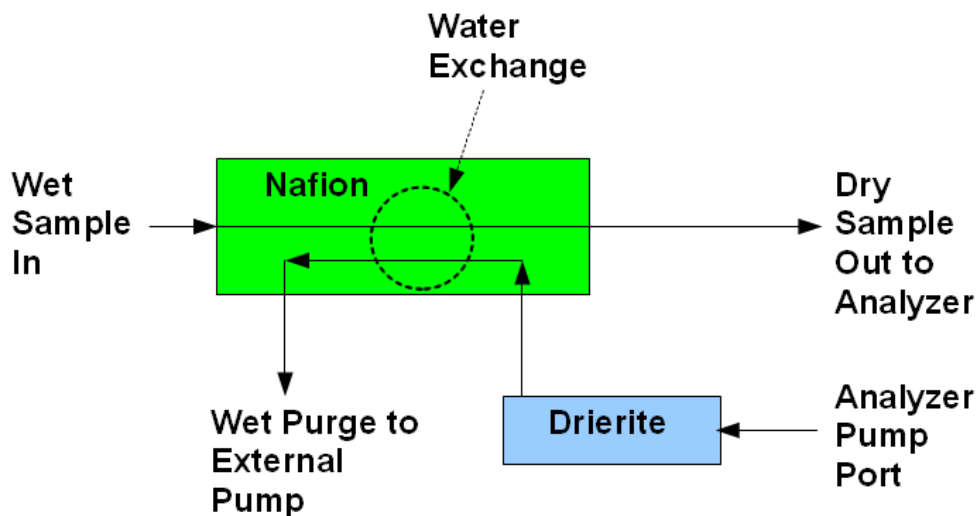


Figure 65: Schematic of Nafion filter system for drying input air sample.

The bulk of the water in the sample is removed by the Nafion and expelled in the filter wet purge. Whatever amount remains is further removed by the Drierite after the isotopic measurement before it enters the Nafion exchanger. Once the Drierite is consumed (blue to pink), it must be either replaced or recharged as detailed on the container. Maximum drying efficiency is obtained by running the sample in the opposite direction to the dry gas purge.

NOTE: Care must be taken to make sure water does not condense within the Nafion filter; the dew point of the sample must be less than the temperature of the drier. If the filter becomes saturated with water, it will cease to function and must be dried out. Please see manufacturer's instruction for further details.



Figure 66: Nafion and Drierite filters for drying gas sample.



Figure 67: Attachment of Nafion filter to the analyzer.

Tubing is provided to connect the drying filter to the inlet, the Drierite to the instrument pump port, and the external pump to the filter purge. Appropriate connections are labeled on the filter assembly. The instrument flow rate may vary depending on the pressure of the inlet with slower flow coinciding with larger inlet pressure. This is due to the Nafion tubing expanding and constricting instrument flow in the purge. LGR recommends keeping the inlet pressure under 10 psig to avoid damaging the internal pressure controller. Reducing inlet pressure further will increase instrument flow rate,

NOTE: Care must be taken to insure that the sample flow direction is opposite to the purge flow of the pump.

Appendices

The following Appendices give additional information on the instrument configuration and operation.

Appendix A – Accessing data via a LAN Ethernet Connection

This procedure describes how to access the analyzer data directory as a Windows™ Share via a Local Area Network (LAN) ethernet connection.

The data files stored on the internal hard disk drive of the analyzer may be accessed as a Windows™ Share via a Local Area Network (LAN) ethernet connection. The following prerequisites are necessary for this function to operate:

1. The analyzer must be connected to a Local Area Network (LAN) via the RJ-45 ethernet connection on the rear panel.
2. The analyzer must receive a response to a DHCP (Dynamic Host Configuration Protocol) request when the instrument is booted. If the analyzer does not receive a reply, it will disable the ethernet port and not attempt another DHCP request until the analyzer is restarted.

When these prerequisites are met, the data directory may be accessed via a Windows™ computer on the same LAN as follows:

1. Click “Start”, then “Run”, then type the following into the “Open” command field: \\LGR-XX-XXXX (where XX-XXXX is the serial number of the analyzer).
2. In a short time (usually between 10 and 60 seconds for the first access) a Windows share directory window will be displayed with a subdirectory named “lgrdata” displayed.
3. Double-click on the “lgrdata” directory, and you will see a listing of the data files stored on the internal hard disk drive of the analyzer. You may open or transfer any of the data files as you would with any Windows™ share drive.

ADDITIONAL NOTES:

1. The analyzer shared data directory may (or may not) be visible by “browsing” for it in the Windows “Network Neighborhood”. If it is, it will be in the workgroup called “LGR” and the computer name will be “LGR-XX-XXXX” where XX-XXXX is the analyzer serial number.
2. You can open the data file that is currently being written into by the analyzer without interrupting the analyzer operation (you will see a snapshot of the file as it was when you opened it). You will notice that the current data file is only updated occasionally (every 4 kB worth of data), so a new data file will appear empty until enough data is collected and written to disk.
3. If a LAN is not available, you may plug the analyzer into a simple standalone broadband router (such as a Netgear Model RP614 – approximately \$45). This will enable the analyzer to obtain a DHCP address from the router when the analyzer is started. You may then plug any Windows™ computer into the same broadband router and access the data directory.
4. A “crossover” ethernet cable will NOT allow an external computer to access the shared data directory, as the analyzer will not obtain a DHCP address at boot and will shut down its ethernet interface.
5. You may be able to access the shared analyzer data directory from computers running operating systems other than Windows™. The analyzer uses a Samba server to share the data directory, and it may be accessed by any appropriate Samba client application.

Appendix B – Mirror ring-down time and maintenance

The mirrors of the measurement cell are protected from contamination by an internal inlet filter. However, it is possible over time and with continued use that the mirrors may gradually decline in reflectivity. This will not create errors in the CO₂ concentration or the isotopic measurement, because the mirror reflectivity is continually monitored and the measurement is compensated using the mirror ring-down time. However, if a significant change occurs in the mirror ring-down time (for example, greater than 20% reduction), the precision of the instrument may be reduced. Users should occasionally take note of the ring-down time and request instrument service (mirror cleaning) from LGR if a significant reduction in ring-down time occurs.

Appendix C – Changing septa on the syringe injection port (optional)

If your CO₂ Carbon Isotope Analyzer is equipped with an optional batch mode system for syringe injection of discrete samples, the septa on the syringe injection port requires periodic replacement. Depending on use, the septa should last a minimum of 100 injections. This procedure describes how to replace the septa.

First, make sure the instrument is in flow through mode (Setup panel, Figure 40 top). Remove the septum nut from the injection port as shown in Figure 68.



Figure 68: Syringe port with septum nut removed.

Remove the septa from the septum nut, and replace with a new septa from the package included with your instrument. Make sure the septa is installed so that the Teflon side (white) is facing out (i.e. will contact the injection port surface) as shown in Figure 69. A needle can be used to center the septa by inserting the needle through the septum nut and septa; the entire nut/septa/needle assembly can then be re-installed on the syringe port before removing the needle.



Figure 69: Position of septa within septum nut. Make sure that the Teflon side (white) is facing out.

Appendix D – Notes concerning instrument calibration

The goal of any high-precision instrument is to push the limits of accuracy and precision; the CO₂ Carbon Isotope Analyzer is no exception. Los Gatos Research specifies a measured isotope ratio precision of 0.25‰ with 60 seconds of data collection and averaging. This is approaching a level of one part in 10,000 in measurement precision. At this level, a number of variables govern the fundamental instrument drift and they depend on the specific instrument and the specific environment within which it is operating. For this reason, we have included this appendix to explain various aspects of instrument specification, calibration, and data processing.

A measurement of a stable CO₂ source (a scuba tank in this case) is shown in Figure 70.

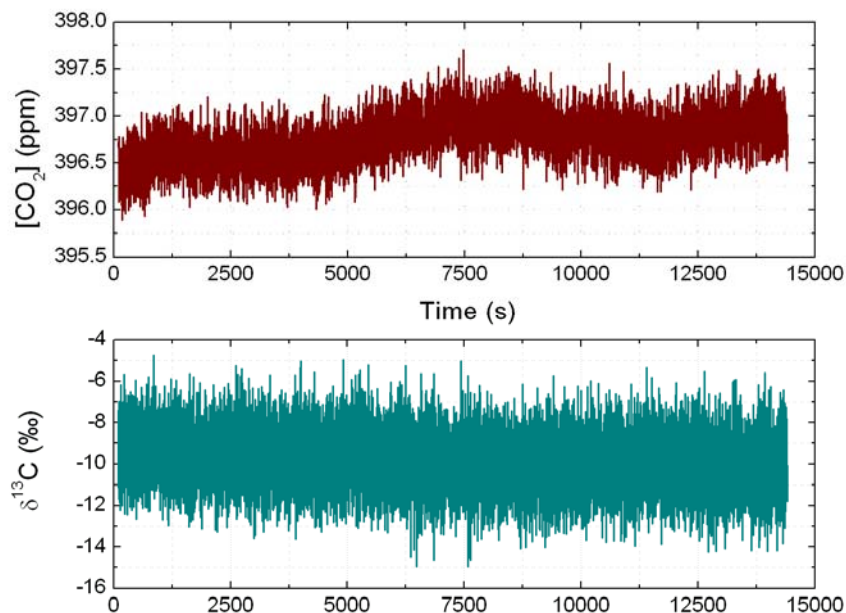


Figure 70: Data taken at 1 Hz from a scuba tank (room air) showing performance over a 4 hour period. Since the sample is assumed to be concentration and isotope stable, this test illustrates the level of residual drift in the instrument.

Over the four hours that the data has been taken, one can discern a drift in the measured concentration of almost 1 ppm out of 396 ppm and a drift in the isotope ratio of about 1‰. Typically, more drift is seen in the concentration compared to the isotope ratio; this has to do with the fact that isotope ratio is insensitive to shared drifts in the concentrations of ¹²CO₂ and ¹³CO₂.

NOTE: With respect to laser absorption spectroscopy, a higher CO₂ concentration will yield a higher precision on the measurement of the isotope ratio. Higher concentrations yield larger absorption lineshapes giving a more precise fit. One can expect precision to improve at the upper end of the instrument measurement range.

To characterize the precision specification of the CO₂ Carbon Isotope Analyzer, Los Gatos Research measures and calculates the Allan deviation. The Allan deviation is the square root of the Allan variance; a quantity also known as the two-sample variance (a detailed description can be found on many websites as well as HP Application Note 1289, *The Science of Timekeeping*). To be brief, the Allan deviation gives the expected standard deviation of the data for a given amount of averaging. Figure 71 shows the calculated Allan deviation for the isotope ratio measurement from Figure 70.

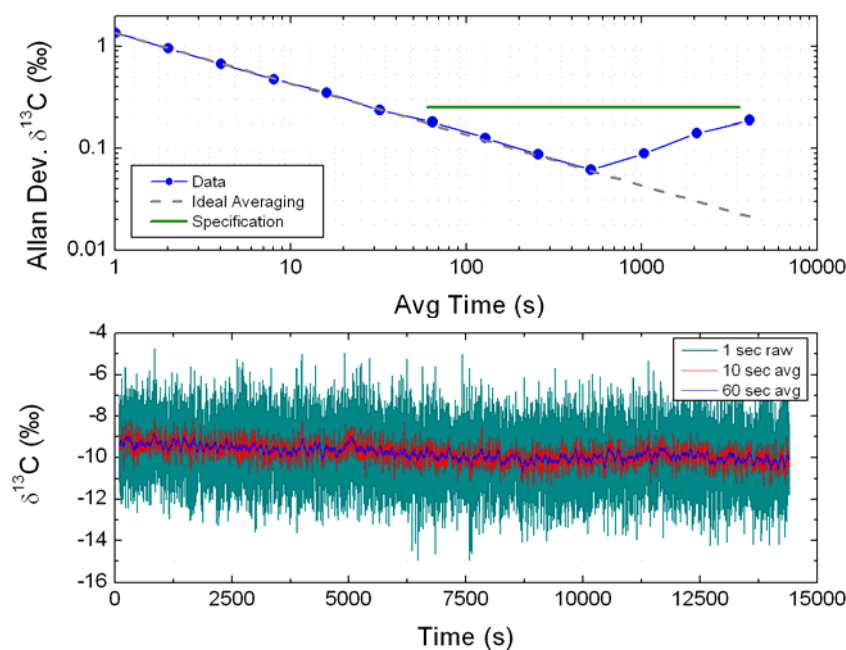


Figure 71: Analysis of noise in isotope ratio measurement from Figure 70. The measured isotope ratio at 1 Hz is shown in light blue in the bottom panel. Also shown is a 10 second and 60 second adjacent average of the same data. The upper curve is a plot of the Allan deviation of the data. It shows the standard deviation of the data versus the averaging time. Los Gatos Research specifies an instrument stability yielding better than 0.25 ‰ in 60 seconds with similar precision maintained over an hour (3600 seconds). The green line in the upper plot graphically shows this instrument specification.

The Allan deviation characterizes the limits of instrument precision and the timescale of drift. Non-statistical drift limits the ability to average away noise indefinitely; this occurs when the Allan curve ‘turns up’. LGR specifies that the CO₂ Carbon Isotope Analyzer will achieve better than 0.25‰ precision in 60 seconds and keep drift to that level for the period of an hour. This is illustrated in Figure 71 by the green line. This does assume a fairly stable, laboratory-type of environment. Depending on the specific environment, this drift could be better or worse. Measuring a stable, inexpensive source such as a scuba tank can yield a measurement of expected drift (see Figure 70). From there, the issue of calibration becomes important.

Calibration is required to correct for instrument drift and maintain accuracy. Depending on the level of accuracy needed, the interval of calibration will vary. To maintain an accuracy of 0.25‰, the LGR specification would suggest a calibration about every hour or so.

NOTE: The specific isotope ratio of the reference is not as important as the concentration. Any residual non-linear behavior is concentration dependent. LGR

recommends using a reference with a concentration similar to the samples being tested. This is true in either free-flow or batch mode operation.

In the case of continuous operation, such as in the field, the user may not be able to conveniently stop the instrument and initiate a calibration. Instead, LGR recommends periodically sampling a known reference and post-correcting the data. The Multi-port Inlet Unit (Optional) is one way of doing this in an automated manner. One could sample a reference for 60 seconds, take a run of data, and then return to sample the reference for another 60 seconds. By interpolating the measured reference throughout the sample data, a post-correction can be implemented on a point-to-point basis.

NOTE: A single reference correction involves

$$[CO_2(corr)] = [CO_2(meas)] \cdot \frac{[CO_2(ref\ value)]}{[CO_2(ref\ meas)]}$$

$$\delta^{13}C(corr) = \delta^{13}C(meas) + (\delta^{13}C(ref\ value) - \delta^{13}C(ref\ meas))$$

Where the reference values are the known concentration and isotope ratio of the reference in question, and the reference measurements are interpolated from the actual instrument readings of the reference at either end of the data run to correct.

In the event of larger drift and/or less frequent reference sampling, the data can still be corrected if the reference is not changing too quickly and can be accurately interpolated at the samples of interest. In this case, the frequency of reference sampling is limited by how many reference samplings are needed to characterize its drift to the precision required. In the case of the data shown in Figure 70, the drift (in isotope ratio) is quite linear and so a reference sample at either end (4 hours apart) would be sufficient to interpolate through the four hours and post-correct data. Figure 72 shows another extreme where the CO₂ Carbon Isotope Analyzer has been deployed in a grassland setting far from any laboratory environment.

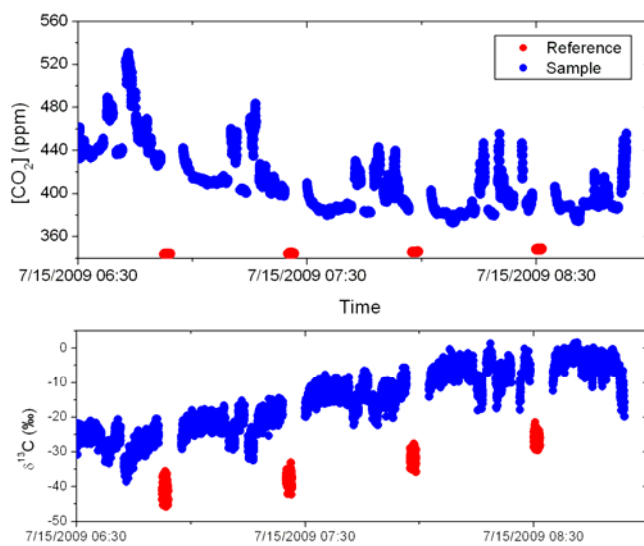


Figure 72: Section of data from a field deployment of the CO₂ Carbon Isotope Analyzer. The blue data is a measure of ambient CO₂ while the red data are periodic sampling of a reference tank. Due to the extremes of the environment, the reference measurement can clearly be seen drifting (especially its measured isotope ratio).

In this situation, the reference is drifting significantly and is being sampled roughly every 30 minutes. However, since the drift is slow on this time scale, suitable interpolation allows for correction of the data using the analysis described earlier. The corrected data is shown in Figure 73.

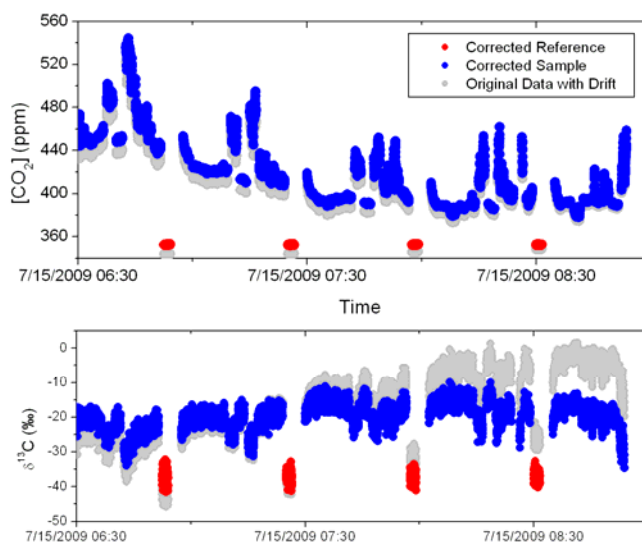


Figure 73: Suitable sampling and interpolation of the reference allows for removal of the drift. The blue and red data shown have been corrected using the analysis described in this appendix. The grey points show the uncorrected values previously shown in Figure 72.

A Keeling plot is a useful tool for visualizing isotopic measurements. It involves plotting the measured isotopic ratio versus the inverse of the measured concentration (please see various references in both literature and on the internet for more details). On a Keeling plot a reference tank is ideally a point (single concentration value, single isotope ratio). Even though the instrument drift must be corrected in the time domain, the impact of the correction is readily seen in the Keeling space. Keeling plots are typically used to characterize systems with mixing of two sources with different isotopic signatures. In the event that concentration fluctuations are correlated with one source, the Keeling plot shows a linear behavior which has a linear intercept ($1/[CO_2] = 0$) at the isotopic signature of the fluctuating source. Figure 74 illustrates the importance of proper calibration/correction in isotopic measurement and how it can impact the interpretation of Keeling analysis. Clearly the data indicates two-source mixing is present, but getting a reliable value for the Keeling intercept requires proper data correction/calibration.

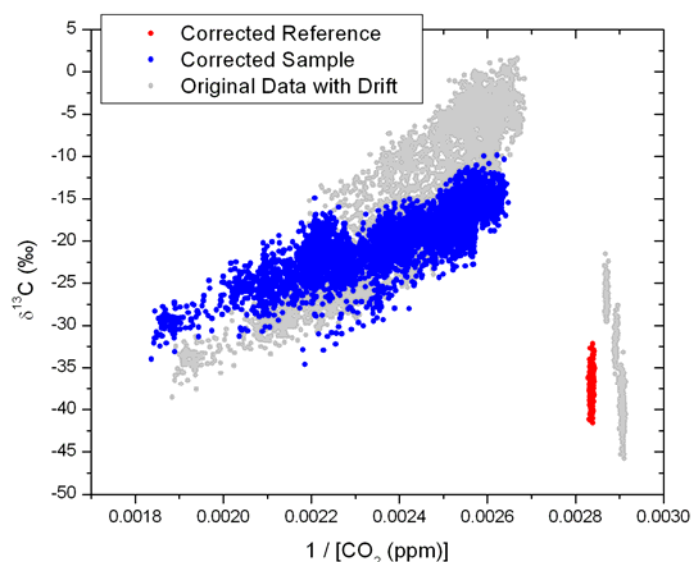


Figure 74: Keeling plot of the field data and its correction from Figure 73. One can see that the reference measurement (in red) has been properly transformed to a single value. Meanwhile, the corrected ambient samples exhibit less noise and extrapolate to a different linear intercept with post-correction.

Appendix E – Format for File Output and Serial Output

The CO₂ Carbon Isotope Analyzer has a standardized output format for file writing and a reduced version for serial output. The basic file format consists of a time stamp followed by an array of data columns.

```

2011 Jan 05 12:18:49
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14.474, 1.33517e+03, 0.00000e+00, 1.67906e+01, 0.00000e+00, 6.14087e+00, 0.00000e+00,
15.475, 1.33371e+03, 0.00000e+00, 1.67500e+01, 0.00000e+00, 6.24950e+00, 0.00000e+00,
16.501, 1.33281e+03, 0.00000e+00, 1.67412e+01, 0.00000e+00, 6.21767e+00, 0.00000e+00,
17.493, 1.33539e+03, 0.00000e+00, 1.67632e+01, 0.00000e+00, 6.18303e+00, 0.00000e+00,
18.510, 1.33185e+03, 0.00000e+00, 1.67641e+01, 0.00000e+00, 6.21200e+00, 0.00000e+00,
19.522, 1.33390e+03, 0.00000e+00, 1.67554e+01, 0.00000e+00, 6.18797e+00, 0.00000e+00,
20.533, 1.33479e+03, 0.00000e+00, 1.67959e+01, 0.00000e+00, 6.24316e+00, 0.00000e+00,
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22.541, 1.33093e+03, 0.00000e+00, 1.67211e+01, 0.00000e+00, 6.15849e+00, 0.00000e+00,
23.547, 1.33402e+03, 0.00000e+00, 1.67522e+01, 0.00000e+00, 6.23914e+00, 0.00000e+00,
24.559, 1.32999e+03, 0.00000e+00, 1.67076e+01, 0.00000e+00, 6.14385e+00, 0.00000e+00,
25.571, 1.33036e+03, 0.00000e+00, 1.67296e+01, 0.00000e+00, 6.12959e+00, 0.00000e+00,
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27.552, 1.33332e+03, 0.00000e+00, 1.67560e+01, 0.00000e+00, 6.23718e+00, 0.00000e+00,
... ..
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53.684, 1.32995e+03, 0.00000e+00, 1.67082e+01, 0.00000e+00, 6.15989e+00, 0.00000e+00,
54.691, 1.32859e+03, 0.00000e+00, 1.66845e+01, 0.00000e+00, 6.11580e+00, 0.00000e+00,
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57.721, 1.33120e+03, 0.00000e+00, 1.67394e+01, 0.00000e+00, 6.11797e+00, 0.00000e+00,
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59.734, 1.32932e+03, 0.00000e+00, 1.66873e+01, 0.00000e+00, 6.14032e+00, 0.00000e+00,
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-----BEGIN PGP MESSAGE-----
Version: GnuPG v1.2.3 (GNU/Linux)

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```

Figure 75: A typical data file showing just a few of the data columns. Instrument settings are encoded at the end of the file after the data columns.

The basic column format for FILE OUTPUT follows:

Time: Time stamp of each measurement; its format is set in the “Setup” menu (page 18).

[ht12], [ht13], [ht18]: Uncalibrated/intermediate values for $^{12}\text{CO}_2$, $^{13}\text{CO}_2$, and $^{12}\text{C}^{16}\text{O}^{18}\text{O}$ which are further processed in later columns. This can be ignored by most users.

[H2O], [CO2]: Total reported concentrations for water and carbon dioxide (ppm).

[d13C], [d18O]: Reported isotope ratios for $\delta^{13}\text{C}$ and $\delta^{18}\text{O}$ with respect to PDB in per-mille (‰).

[12CO2], [13CO2], [18CO2]: Individual isotopic concentrations of $^{12}\text{CO}_2$, $^{13}\text{CO}_2$, and $^{12}\text{C}^{16}\text{O}^{18}\text{O}$ respectively (ppm).

[CO2 sam]: Estimated input concentration (ppm) of CO_2 in operation with the optional Dynamic Dilution System (page 42).

[CO2 dry]: Dry mole fraction of CO_2 (ppm) consistent with measured H_2O .

GasP, GasT: Measurement cell pressure (Torr) and temperature (Celsius).

AmbT: Interior instrument temperature (Celsius).

RD0: Ringdown measurement of optical cell (micro-secs).

Gnd: Off-state measurement of detector.

LTC0 b, LTC0 v, Peak0: Laser locking parameters.

Xaxis0, Xaxis1: Laser scan parameters.

BL0, BL1, BL2, BL3: Baseline line fit parameters.

12CO2_0_CT, 12CO2_0_AT, 12CO2_0_DF, 12CO2_0_PF: Detailed $^{12}\text{CO}_2$ fit.

13CO2_1_CT, 13CO2_1_AT, 13CO2_1_DF, 13CO2_1_PF: Detailed $^{13}\text{CO}_2$ fit.

H2O_2_CT, H2O_2_AT, H2O_2_DF, H2O_2_PF: Detailed H_2O fit.

12C16O18O_4_CT, 12C16O18O_4_AT, 12C16O18O_4_DF,

12C16O18O_4_PF: Detailed $^{12}\text{C}^{16}\text{O}^{18}\text{O}$ fit.

Fit_Flag: Informational flag

N_Fits_Avg'd: Number of fits done per data point as dictated by ‘Rate’ setting (page 13).

MIU valve, MUI desc: If the optional Multi-port Inlet Unit (page 38) is active, then additional information regarding the active valve and user-tag is reported here.

The items in **Bold Face** are the most pertinent to the average user. The additional parameters are recorded so that LGR scientists can, if needed, evaluate instrument performance. For each of these measurements there is an additional adjacent column reporting the standard error of the measurement (designated with ‘_se’ tag). The standard error is zero when the instrument is running at 1 Hz, as no averaging of data has taken place. At speeds slower than 1 Hz, the standard error of the average is reported. At the end of each data file are encrypted listings of settings used by the instrument for that data file. The settings are stored in case LGR scientists wish to re-analyze a data set to study instrument performance.

The basic column format for SERIAL OUTPUT follows and is a subset of the previously described file output:

- Time**: Time stamp of each measurement; its format is set in the “Setup” menu (page 18).
- [ht12], [ht13], [ht18]**: Uncalibrated/intermediate values for $^{12}\text{CO}_2$, $^{13}\text{CO}_2$, and $^{12}\text{C}^{16}\text{O}^{18}\text{O}$ which are further processed in later columns. This can be ignored by most users.
- [H2O], [CO2]**: Total reported concentrations for water and carbon dioxide (ppm).
- [d13C], [d18O]**: Reported isotope ratios for $\delta^{13}\text{C}$ and $\delta^{18}\text{O}$ with respect to PDB in per-mille (‰).
- [12CO2], [13CO2], [18CO2]**: Individual isotopic concentrations of $^{12}\text{CO}_2$, $^{13}\text{CO}_2$, and $^{12}\text{C}^{16}\text{O}^{18}\text{O}$ respectively (ppm).
- [CO2 sam]**: Estimated input concentration (ppm) of CO_2 in operation with the optional Dynamic Dilution System (page 42).
- [CO2 dry]**: Dry mole fraction of CO_2 (ppm) consistent with measured H_2O .
- GasP, GasT**: Measurement cell pressure (Torr) and temperature (Celsius).
- AmbT**: Interior instrument temperature (Celsius).
- RD0**: Ringdown measurement of optical cell (micro-secs).
- Fit Flag**: Informational flag
- N Fits Avg'd**: Number of fits done per data point as dictated by ‘Rate’ setting (page 13).
- MIU valve, MUI desc**: If the optional Multi-port Inlet Unit (page 38) is active, then additional information regarding the active valve and user-tag is reported here.

As indicated previously, the items in **Bold Face** are the most pertinent to the average user. For each of these measurements there is an additional adjacent column reporting the standard error of the measurement. The standard error is zero when the instrument is running at 1 Hz, as no averaging of data has taken place. At speeds slower than 1 Hz, the standard error of the average is reported.

Appendix F – LGR Contact Information:

For questions regarding the operation of this instrument, please contact:

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