LOS GATOS RESEARCH

CO_2 Carbon Isotope Analyzer

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Contents

Quick Start Guide

Set up the CO_2 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 44-45 degrees Celsius. This may take 1-2 hours.

Calibrate the CO_2 Carbon Isotope Analyzer

- Enter the "Setup" panel and click the "Calibrate" button.
- Follow the on-screen directions for calibration.
- Attach a tube, regulated at a pressure just slightly above ambient atmosphere, from a local gas standard.
- Enter the known total CO_2 concentration in ppm and isotopic ratio in pp-mil.
- Disconnect the gas standard when prompted by the on-screen directions.

Starting measurement

- Attach a tube from the gas sample to the gas inlet port.
- 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 enable during startup.

Installation

The Los Gatos Research (LGR) CO_2 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. You should have received:

- CO_2 Carbon Isotope Analyzer
- Instrument power cord
- Users guide (this document)
- USB flash drive
- External pump
- Pump slave power cord
- Pump connection tube
- Serial port connection cable (null modem type)

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 CO_2 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 or frequency is desired, please contact LGR.

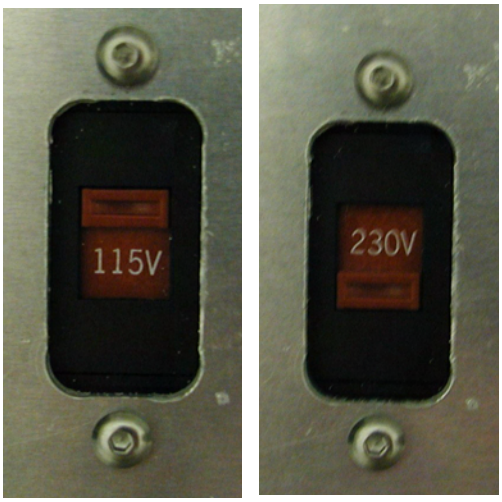

Figure 1: AC voltage selection switch.

Data Interface Connections

The CO_2 Carbon Isotope Analyzer has five data interface connection ports on the back panel (see Figure 2). The "USB" port is utilized for file transfer to USB memory devices. The "Serial" port is utilized for real-time measurement output directly to a computer. The "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. The "Analog" ports are not configured for this instrument at this time. The functionality of the data interface connections is described in the relevant sections later in the manual.

Figure 2: Data interface connections.

Gas Inlet / Outlet Connections

The gas inlet and outlet ports of the instrument are on the rear panel (see Figure 3). The unit is shipped with all inlets and outlets plugged for protection during transit. 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 20 psig.

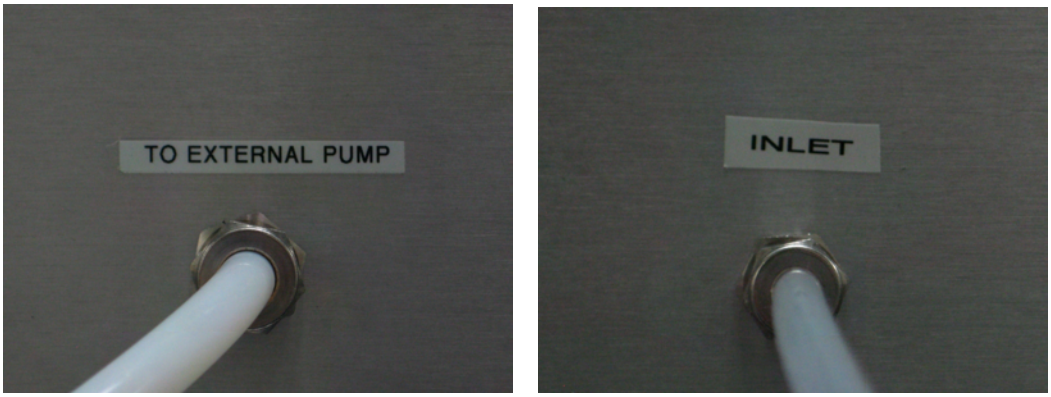

Figure 3: Pump outlet (3/8") and gas inlet (1/4") connections.

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 < 3.5 mm (see Figure 4).

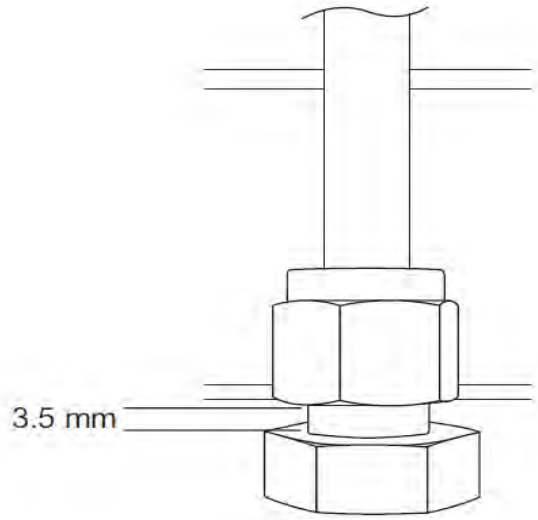

Figure 4: Swagelok connections should be tighten to 1/4 - 1/2 turn past finger tight, leaving a gap of < 3.5 mm. Use a 9/16" wrench for 1/4" tubing, and an 11/16" wrench for 3/8" tubing.

Instrument Startup / Shutdown

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. 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.

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 Startup/Shutdown section on page 16.

Operation

The Los Gatos Research (LGR) CO_2 Carbon Isotope Analyzer is easily operated via the user interface as described in this section.

Main Panel

Display Modes

When the instrument is turned on, it will automatically go through a 90-second 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 "Numeric" mode (see Figure 5). The $^{12}CO_2$ and $^{13}CO_2$ concentrations are shown in parts-per-million (ppm). The isotope ratio is shown in parts-per-thousand (pp-mil). The isotope ratio is reported relative to Pee Dee Belemnite (PDB) as shown in Equation 1.

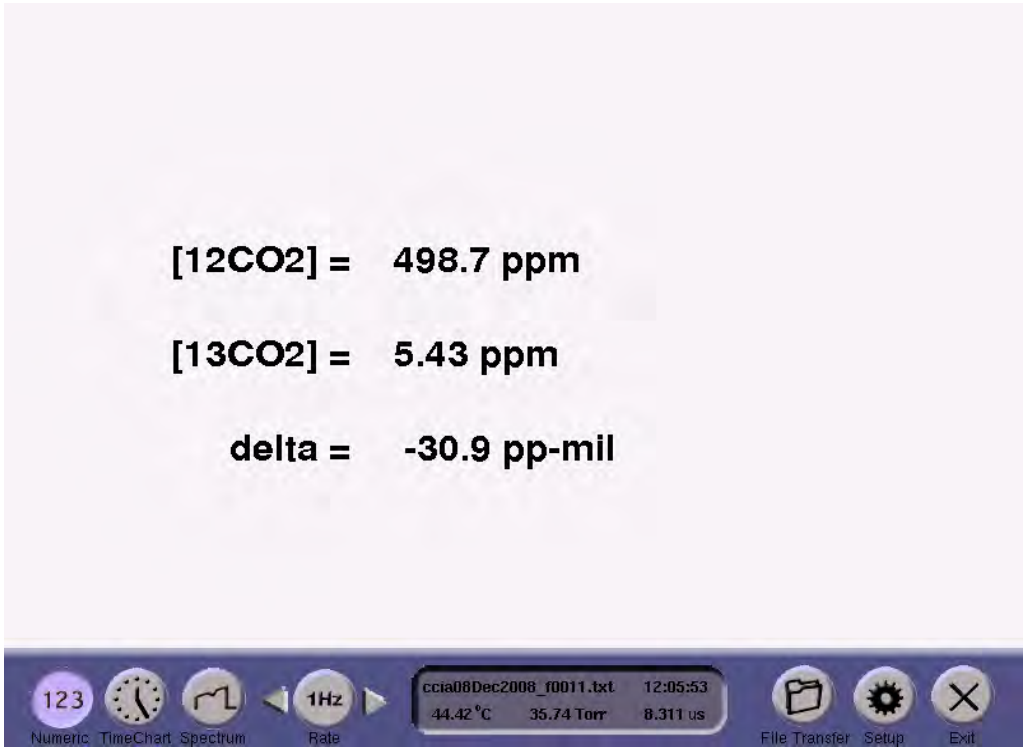

Figure 5: The CO_2 concentration and isotope measurements are reported along with the cell pressure in Torr, cell temperature in Celsius, and the current data filename. The mirror ring-down time in microseconds is measured and reported as well.

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

Equation 1. Definition of isotope ratio $\delta^{13}C$ vs Pee Dee Belemnite (PDB). PDB is defined to be $^{13}C/^{12}C = 0.0112372$.

The user may select the "TimeChart" button to see a display of $^{12}CO_2$ concentration over time, as shown in Figure 6. The user may use the drop down box in the lower right of the window to toggle between $^{12}CO_2$ concentration over time, $^{13}CO_2$ concentration over time (Figure 7) and isotope ratio $\delta^{13}C$ (delta) over time (Figure 8). These data are also being saved to the file indicated in upper left corner of the parameter window, along with a continuous record of the pressure and temperature. 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.

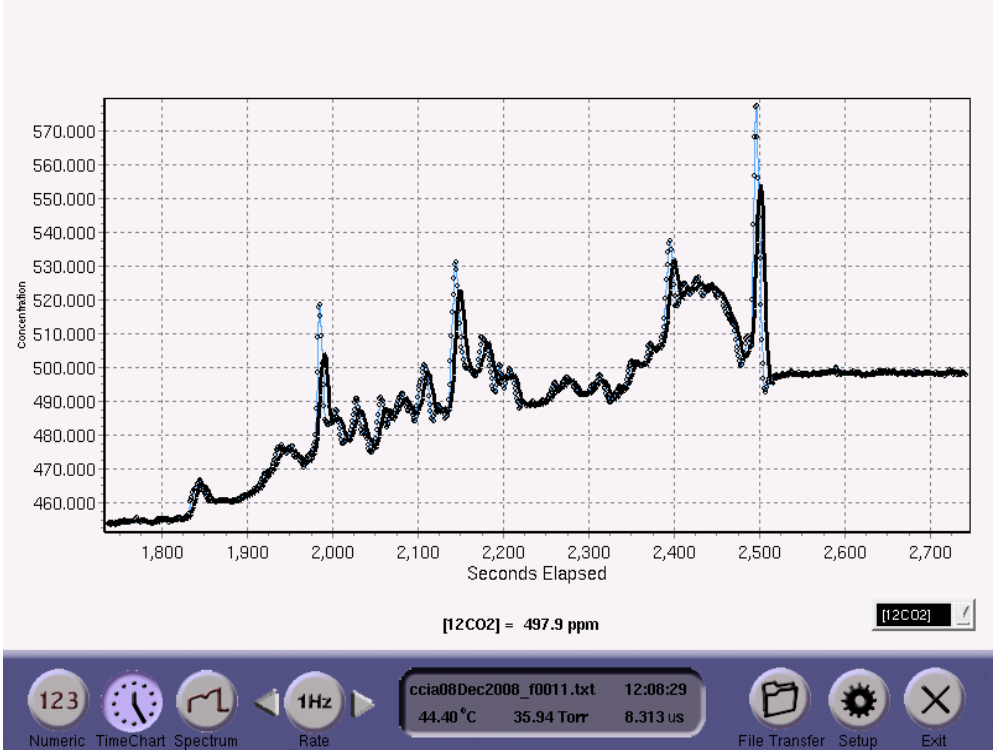

Figure 6: The time chart showing changes in the $^{12}CO_2$ concentration over time.

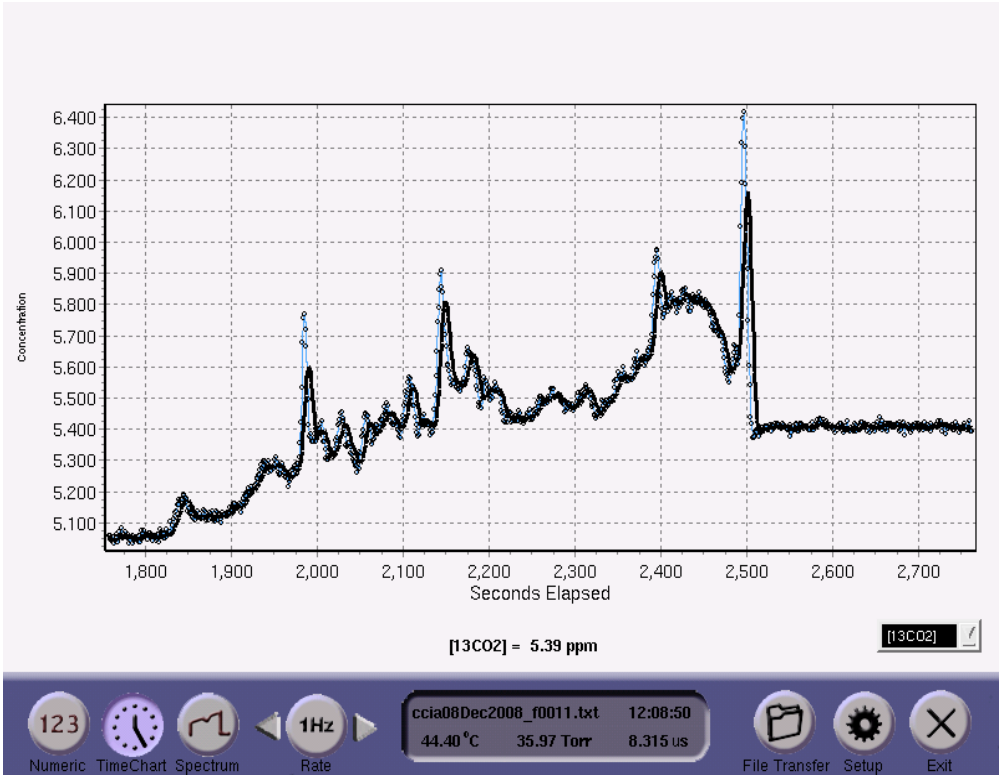

Figure 7: The time chart showing changes in the $^{13}CO_2$ concentration over time.

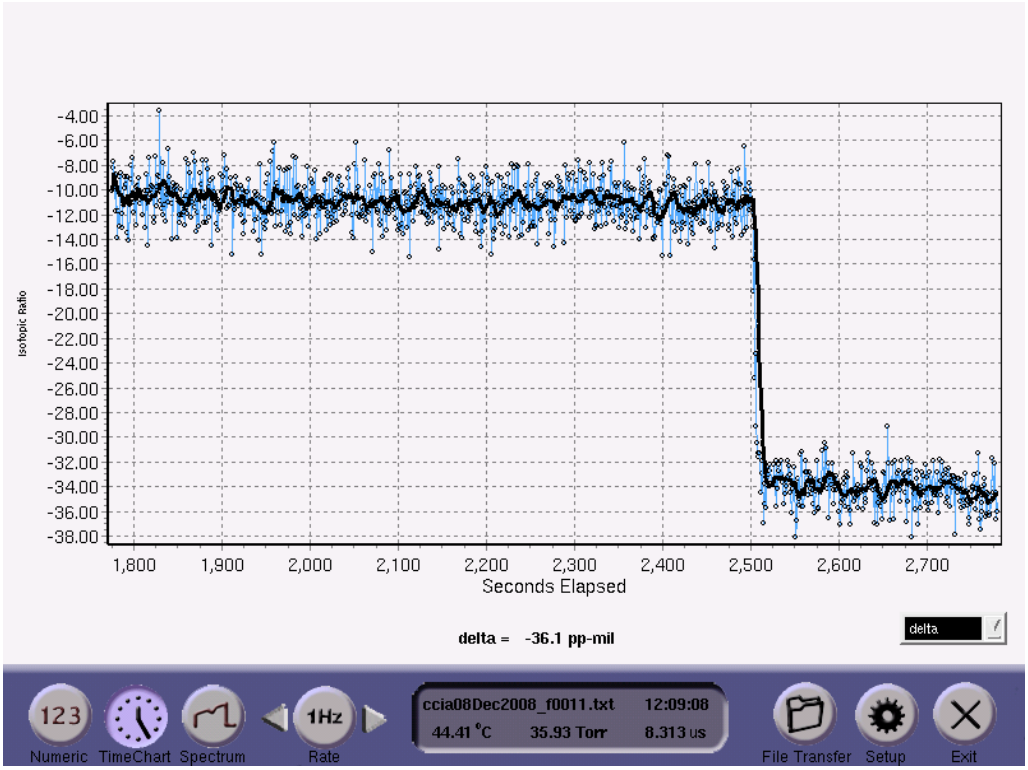

Figure 8: The time chart showing changes in the isotopic ratio $\delta^{13}C$ of carbon in CO_2 measurements over time.

The user may select the "Spectrum" button to see the laser transmission through the ICOS measurement cell (Figure 9, top frame) recorded as the laser wavelength is tuned across the selected wavelength region near 2.05 microns. The CO_2 absorption lineshapes that result from a detailed analysis of the measured transmission signal is shown in the bottom frame of Figure 9. At roughly 500 ppm of CO_2 the absorption of the larger feature should be approximately 0.55 on the lower frame.

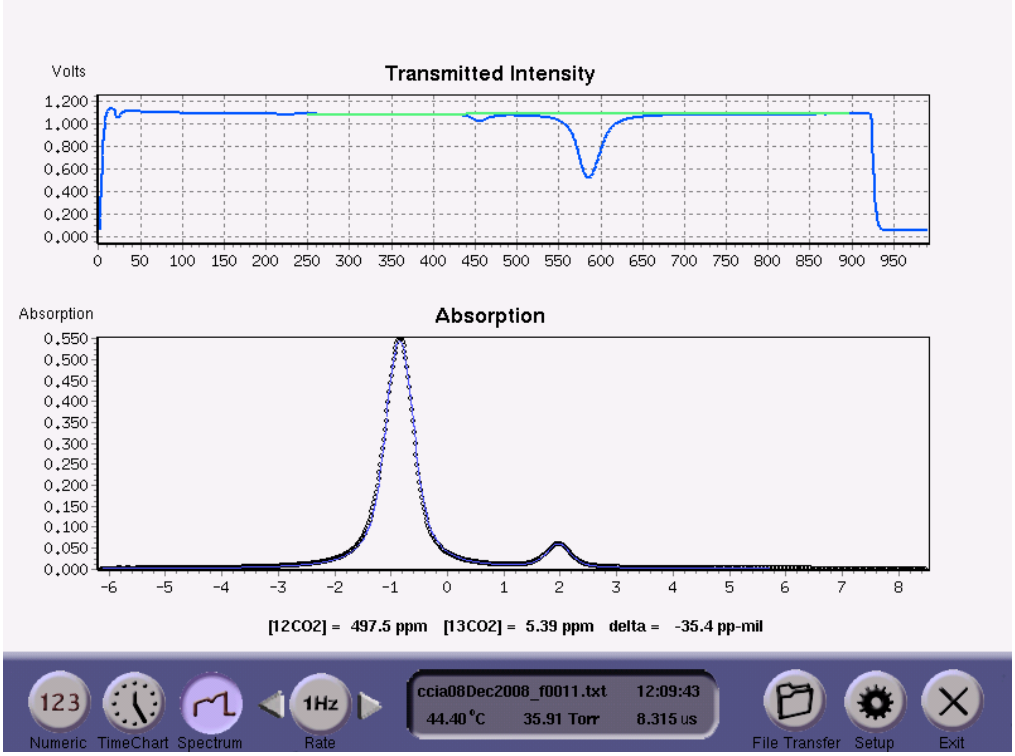

Figure 9: The spectrum display consists of two graphs: the top shows the voltage from the photodetector as the laser scans across the CO_2 absorption features, the bottom shows the optical absorption fraction due to CO_2 in black, and the peak fit resulting from signal analysis in blue.

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 "cciaDDMMMYYYY_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); ie., ccia08Dec2008_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. The time period between creation of new files is set in the "File Settings" menu as described later. The data files are written in text (ASCII) format and contain labeled columns as shown in Figure 10.

```
2008 Dec 08 14:48:31
                    Time,       [12CO2],     [12CO2]_se,       [13CO2],     [13CO2]_se,         delta,      delta_se,     Press_torr,  Press_torr_se,
                   1.418,    0.00000e+00,   -1.99410e+00,    0.00000e+00,   -1.99410e+00,    0.00000e+00,   -1.99410e+00,    0.00000e+00,   -1.99410e+00,
                   2.476,    0.00000e+00,   -1.99410e+00,    0.00000e+00,   -1.99410e+00,    0.00000e+00,   -1.99410e+00,    0.00000e+00,   -1.99410e+00,
                   3.430,    0.00000e+00,   -1.99410e+00,    0.00000e+00,   -1.99410e+00,    0.00000e+00,   -1.99410e+00,    0.00000e+00,   -1.99410e+00,
                   4.411,    0.00000e+00,   -1.99410e+00,    0.00000e+00,   -1.99410e+00,    0.00000e+00,   -1.99410e+00,    0.00000e+00,   -1.99410e+00,
                   5.391,    3.73405e+02,    0.00000e+00,    4.17584e+00,    0.00000e+00,   -4.79322e+00,    0.00000e+00,    3.58624e+01,    0.00000e+00,
                   6.376,    3.73580e+02,    0.00000e+00,    4.16100e+00,    0.00000e+00,   -8.79342e+00,    0.00000e+00,    3.58260e+01,    0.00000e+00,
                   7.358,    3.72943e+02,    0.00000e+00,    4.17470e+00,    0.00000e+00,   -3.83134e+00,    0.00000e+00,    3.59633e+01,    0.00000e+00,
                   8.350,    3.73598e+02,    0.00000e+00,    4.16631e+00,    0.00000e+00,   -7.57957e+00,    0.00000e+00,    3.58622e+01,    0.00000e+00,
                   9.365,    3.73653e+02,    0.00000e+00,    4.18415e+00,    0.00000e+00,   -3.47394e+00,    0.00000e+00,    3.58340e+01,    0.00000e+00,
                  10.363,    3.73562e+02,    0.00000e+00,    4.16876e+00,    0.00000e+00,   -6.89698e+00,    0.00000e+00,    3.58273e+01,    0.00000e+00,
                  11.300,    3.73239e+02,    0.00000e+00,    4.15980e+00,    0.00000e+00,   -8.17295e+00,    0.00000e+00,    3.59668e+01,    0.00000e+00,
                  12.292,    3.73752e+02,    0.00000e+00,    4.16103e+00,    0.00000e+00,   -9.24319e+00,    0.00000e+00,    3.58324e+01,    0.00000e+00,
                  13.282,    3.73647e+02,    0.00000e+00,    4.17151e+00,    0.00000e+00,   -6.46974e+00,    0.00000e+00,    3.58366e+01,    0.00000e+00,
                   ...
                   ...
                   ...
#=-=-=-=-=-=-=-=-=Start ../conf/ccia_ini/SerialSettings.ini =-=-=-=-=-=-=-=-=
Serial Settings -- Saved On 01/17/2006 At 01:37:23 PM
       0                //Loopback: 0=Out->Ext, 1=Out+In<->Ext, 2=Out->In2, 3=In<-Out2
       1                //Port Number: 0=Port1, 1=Port2
       0                //Baud Rate: 0=9600, 1=19200, 2=38400, 3=57600, 4=115200
       0                //Parity: 0=None, 1=Odd, 2=Even
       0                //Stop Bits: 0=1 Bit, 1=2 Bits
       0                //Message Type: 0=Normal, 1=Normal+Diagnostic
       0                //Time Stamp: 0=Abs Loc Amer, 1=Abs Loc Euro, 2=Abs GMT Amer, 3=Abs GMT Euro, 4=Rel Sec, 5=Rel hh:mm:ss
       0                //Delimiter: 0=Comma, 1=Tab, 2=Space
       1                //Rate (N): Serial Rate=Logged Rate/N
#=-=-=-=-=-=-=-=-=End ../conf/ccia_ini/SerialSettings.ini =-=-=-=-=-=-=-=-=
#=-=-=-=-=-=-=-=-=Start ../conf/ccia_ini/bootstrap.ini =-=-=-=-=-=-=-=-=
# List the hardware and algorithm specifics so that the ICOS code knows
# which ini files to read and which objects instantiate.
#
#
# Number of lasers in the instrument [1-2].
#
NUM_LASERS              1
#
# DM7520 12-bit fast analog I/O controllers installed? [Y|N]
#
```

Figure 10: The beginning of a typical data file showing just a few of the data columns. Instrument settings are saved after the end of the data columns.

The "Time" column reports seconds elapsed since the start of the data file (or since the initial start of a data file series when operating continuously). Also reported are the measured concentration of $^{12}CO_2$ in ppm, the concentration of $^{13}CO_2$ in ppm, the isotope ratio in pp-mil, cell pressure in Torr, and cell temperature in degrees Celsius. For each of these measurements there is an additional column reporting the standard error of the measurement. The standard error is zero when the instrument is running at 1 Hz or faster, as no averaging of data has taken place. At speeds slower than 1 Hz, the standard error of the average is reported. Three additional columns, LLVolt, LLVolt_se, and Fit_Flag are for diagnostic purposes only. At the end of each data file are listings of settings used by the instrument for that data file. The settings are usually not needed, but 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 11). After pressing OK, data acquisition will halt, and the user will see two file directory windows as shown in Figure 12. 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.

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.

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 13).

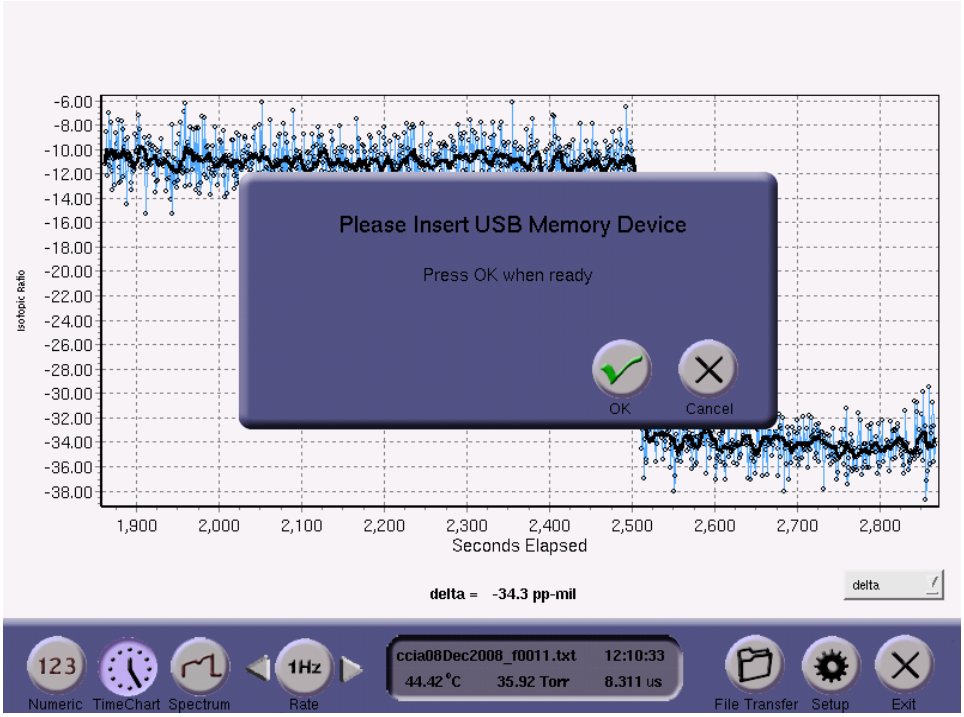

Figure 11: Reminder to insert USB memory device into instrument.

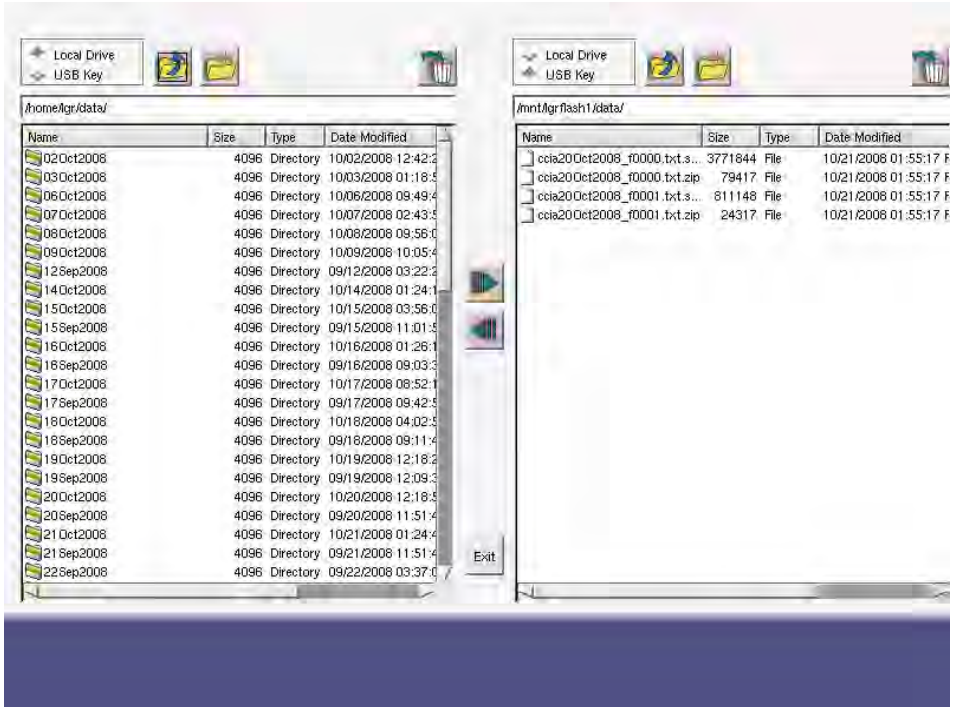

Figure 12: File transfer windows. Highlight the files to copy and click the appropriate arrow. Click the new folder icon to create directories and click the trash can to delete files.

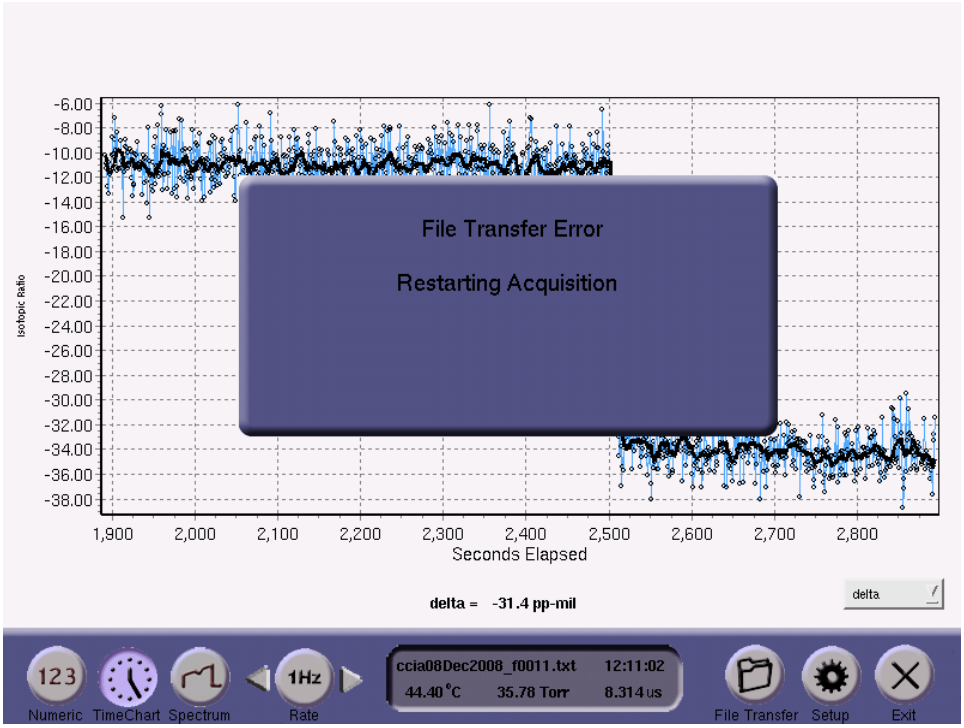

Figure 13: File transfer error. User forgot to insert a USB device, or the device was not recognized. Please try again with a USB device correctly inserted or with a different USB device.

Startup / Shutdown

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 14 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 14: 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.

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 15). After the progress bar completes, the instrument will switch to a text-based output as it completes shutting down. The user must until after the "Power Down" command is displayed as shown in Figure 16 before turning off the instrument. Failure to do so may result in file system instability.

Figure 15: Instrument shutdown screen.

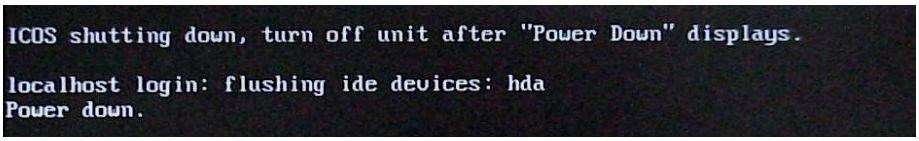

Figure 16: Final shutdown screen.

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 file saving settings, re-calibrate the instrument to a local CO_2 gas standard, configure the Serial Output, enter the service mode, and adjust the current time/date settings.

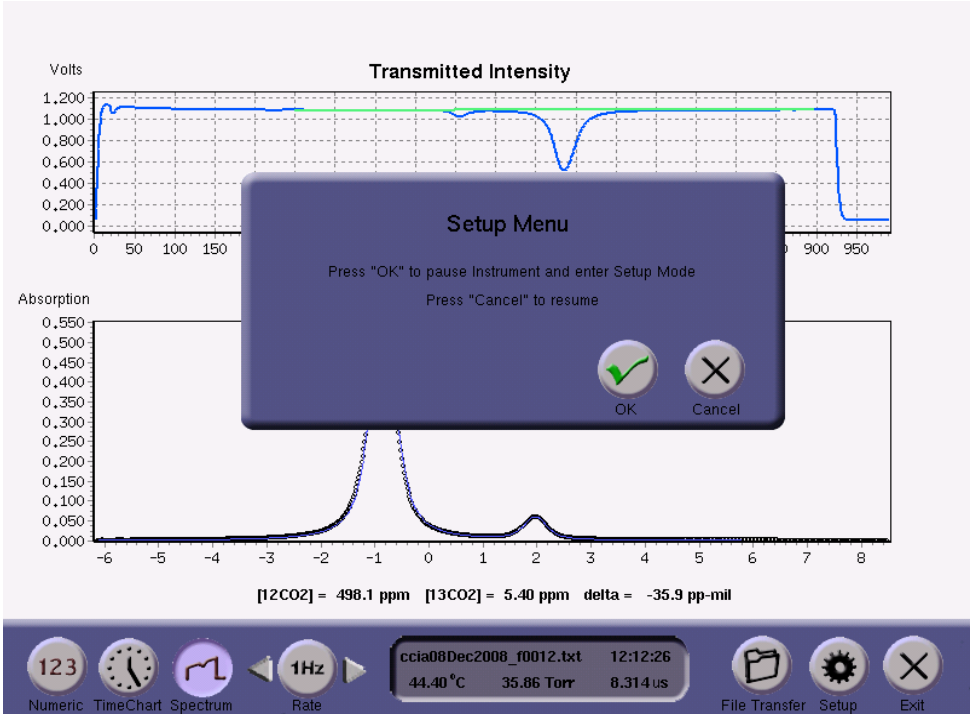

Figure 17: Pressing the Setup button prompts the user for further input.

Figure 18: The Setup panel allows the user to configure file settings, calibrate, configure serial output, enter service mode, and configure the instrument time.

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.

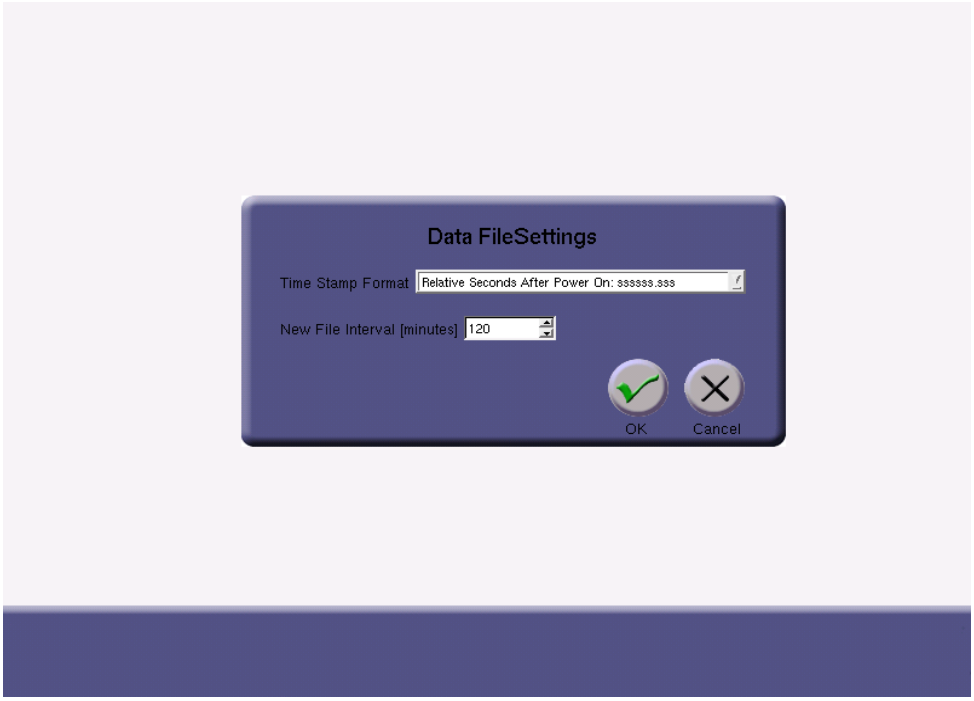

Figure 19: The File Settings menu.

Calibration Menu

The CO_2 Carbon Isotope Analyzer is equipped with a calibration routine. Los Gatos Research recommends regular calibration to ensure measurement accuracy and consistency. Calibration can be achieved by attaching a tube, regulated at a pressure just slightly above ambient atmosphere, from a local gas standard to the instrument inlet. The sequence of calibration steps is illustrated in Figure 20. Click the "Calibrate" button on the "Setup" panel to bring up information on the most recent calibration. 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. Exit "Setup" mode and another screen will confirm that calibration is desired. Enter the known total concentration of CO_2 in ppm and value of $\delta^{13}C$ in pp-mil for the local gas standard into the box labeled "Enter Calibration Data". A screen will pop up indicating that the instrument is ready to calibrate. 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 then run the calibration routine for approximately 1 minute. A screen will pop up indicating when the calibration is finished and that the user should disconnect the calibration gas. The time of latest calibration is also stored in the instrument configuration files for future reference.

Please note: The calibration requires that the user input the *total* concentration of CO_2 in ppm. The analyzer reports the individual $^{12}CO_2$ and $^{13}CO_2$ concentrations in ppm. The total measured concentration, ignoring higher order isotopes, can be calculated as:

$$[CO_2] = [^{12}CO_2] + [^{13}CO_2]$$

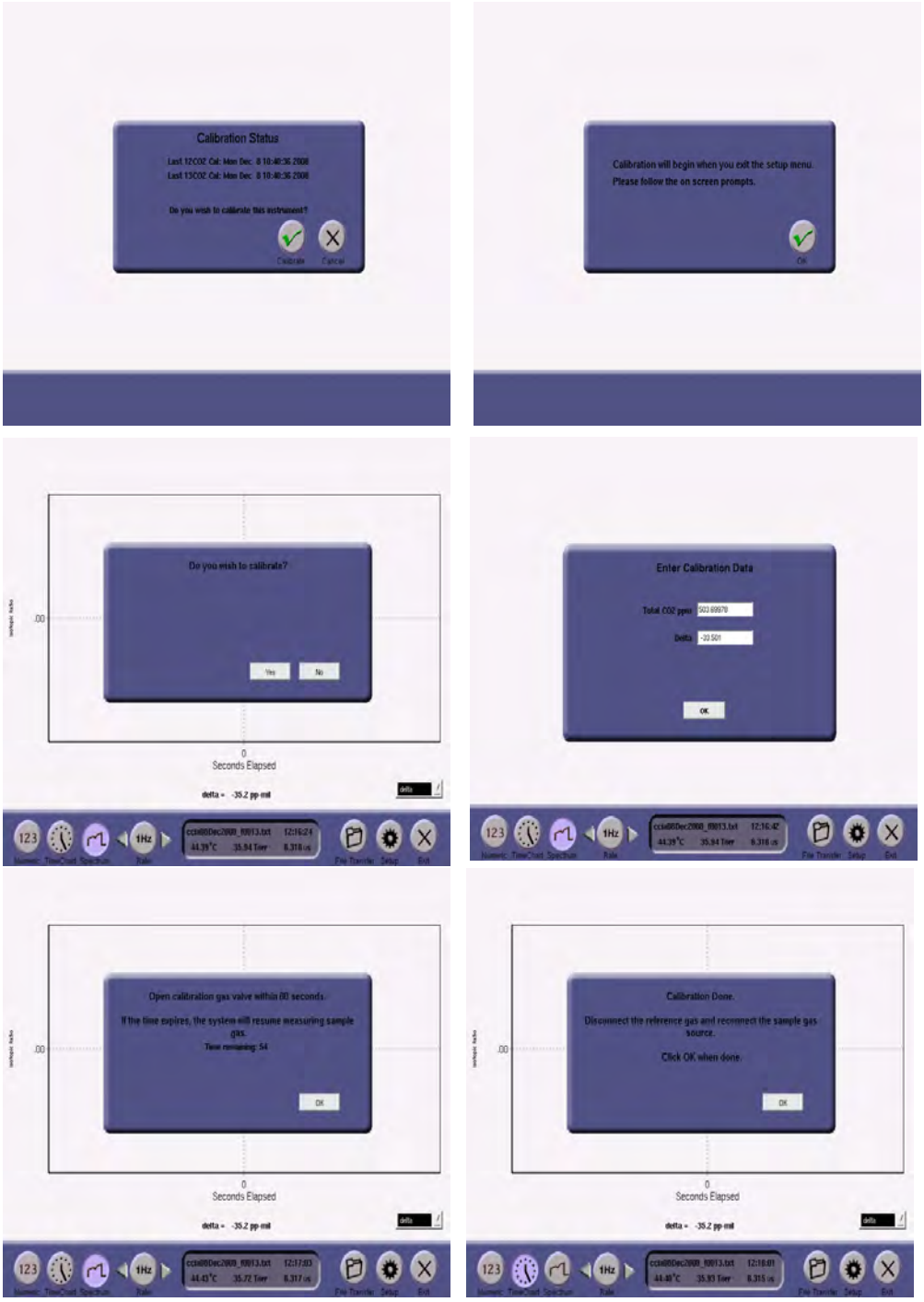

Figure 20: Screenshots of the calibration sequence.

Serial Configuration Menu

The Serial Configuration Menu (Figure 21) 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. The following data is reported through the RS-232 port: measurement time in seconds, $[^{12}CO_2]$ in ppm, $[^{13}CO_2]$ in ppm, $\delta^{13}C$ in pp-mil, pressure in Torr, and temperature in Celsius. NOTE: When connecting the serial port of the instrument to an external computer, a null modem type serial cable should be used.

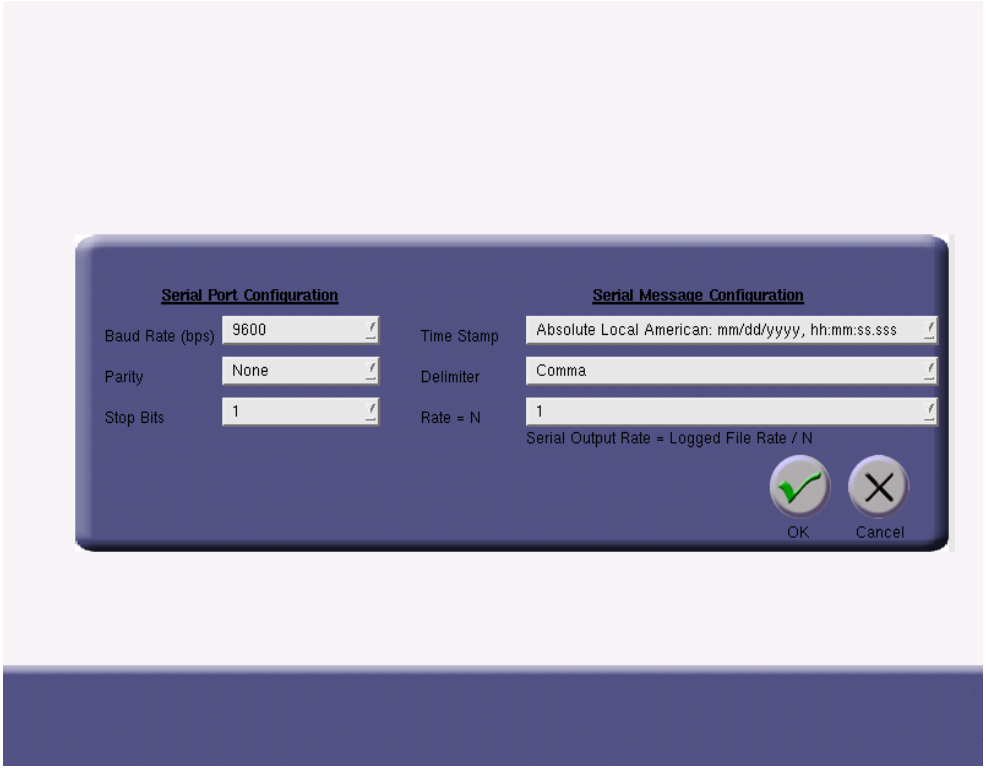

Figure 21: The serial configuration menu.

Absolute Local American	mm/dd/yyy, hh.:mm:ss.sss
Absolute Local European	dd/mm/yyyy, hh:mm:ss.sss
Absolute GMT American	mm/dd/yyy, 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.

Service Mode Menu

A password protected Service Mode (Figure 22) is available only for qualified technicians to make software upgrades or run instrument diagnostics.

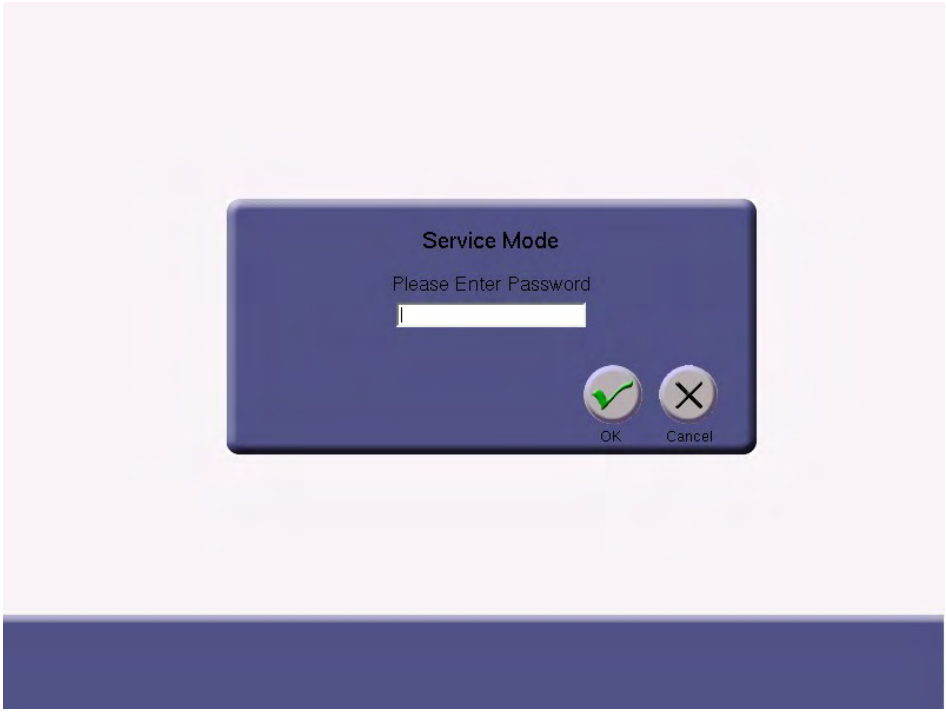

Figure 22: The Service menu.

Time Menu

The Time Menu lets the user adjust the current time and date settings of the instrument (Figure 23). The timezone and daylight savings enable / disable may also be set here.

Figure 23: The Time menu.

Appendix

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 – LGR Contact Information:

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

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