

OCEAN DRILLING PROGRAM

COULOMETRICS

USER'S MANUAL

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Introduction

The *Coulometrics* program was developed to aid in the measurement and uptake of all carbonate sample data. This document contains instructions for the set-up, measurement, and saving of data. It also contains technical details on program installation for a computer technician.

Document Layout

This document is divided into several sections and two appendices. The first section details *Coulometrics* options including setting the data path. The next few sections describe how to use the balance to weigh samples. The last sections contain instructions for using the coulometer and for saving data and exporting it to Excel. The first appendix provides installation and upgrade information, intended for use by a computer technician. The second appendix provides sample file formats for the Coulometer program.

Starting Coulometrics

For the First Time

The very first time that *Coulometrics* is started, the following warning appears (see Figure 1). This message indicates that the program cannot find the folder where the data files for *Coulometrics* are located. This data path must be set before the program will run properly.

Note: the data path need only be set the very first time *Coulometrics* is run. After that, only change the data path if you change the location of the saved data.

1. Double click on the *Coulometrics* icon to start the program. The following message should appear.

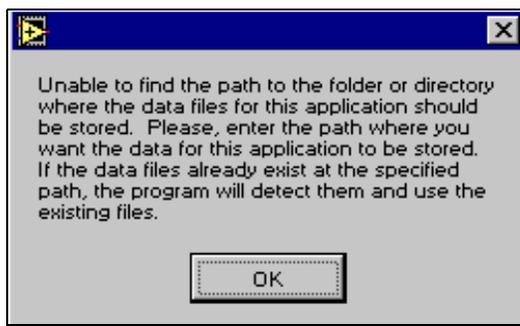


Figure 1. Data path warning.

1. Click **OK**. This opens the "Input Datapath" window (see Figure 2).

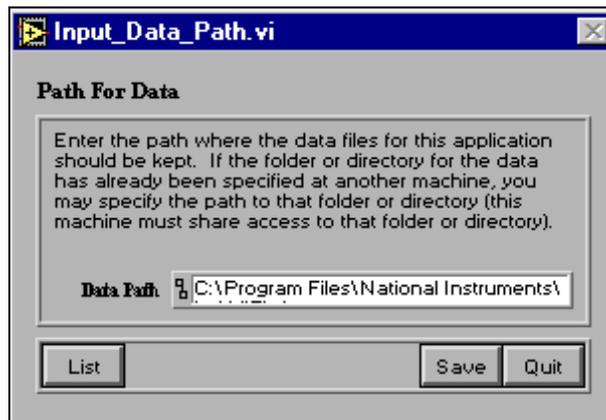


Figure 2. Input Data Path window.

2. If you already know the data path, type it into the "Data Path" field. If not, click on the **List** button. This opens a navigational dialog box (see Figure 3).

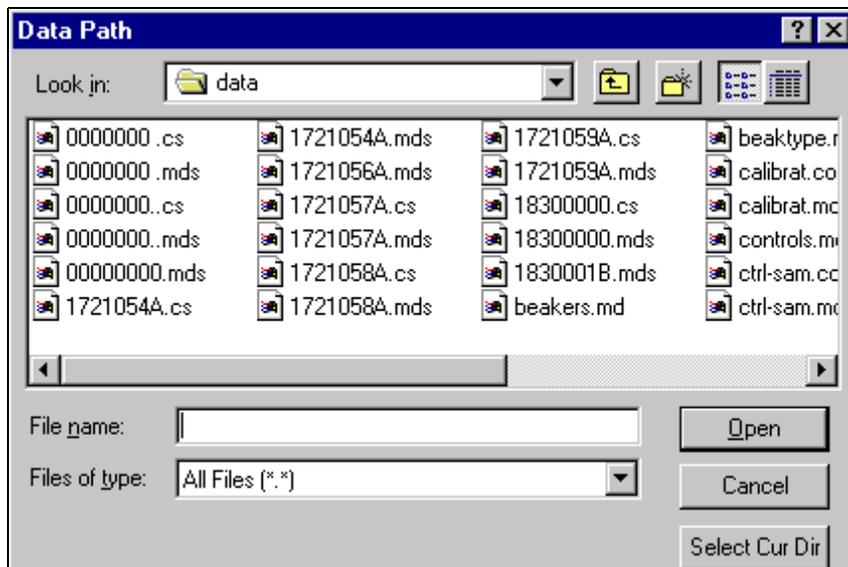


Figure 3. Set data path here.

3. Navigate through the box until you find the location where you want to save the *Coulometrics* data files. Click on **Select Cur Dir** button. The data path is now set.

The very first time *Coulometrics* is started you should also receive the following warning (see Figure 4). If you receive it at any other time, please notify the IS group. Click **Continue**.

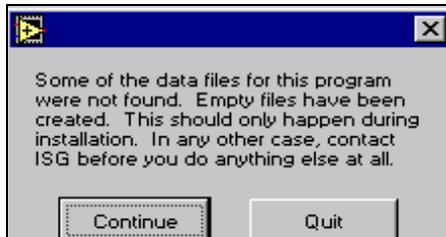


Figure 4. Startup warning.

Finally, the last message that appears during the initial startup is for User Options (see Figure 5). This message indicates that a file for user options has been created. To set the options at this time click **Yes**. You can also set these options at any other time by clicking on the **Options** button on the main menu.

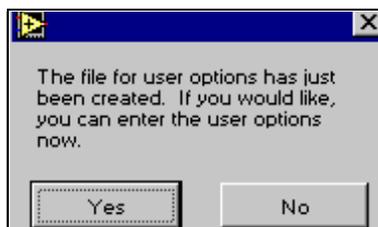


Figure 5. Message about user options.

1. If you click on **Yes**, the “Options” window will open (see Figure 6).

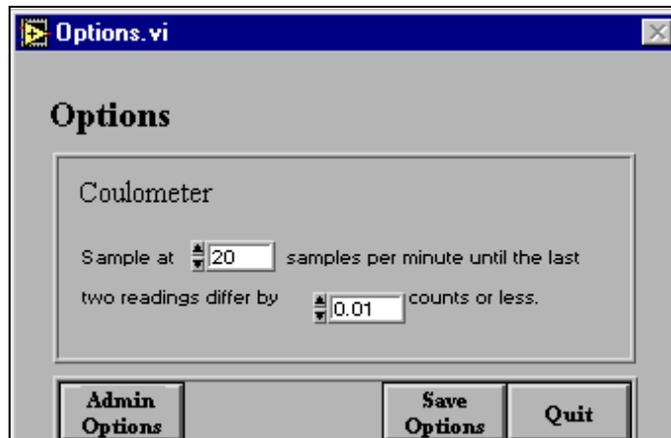


Figure 6. Set options here.

2. Use the up and down arrows to set the options, or enter them manually. Click **Save Options** when you are done, then **Quit** to return to the main menu (for more information on user options See "Coulometrics Options" p. 5).

Once the data path and options are set, the main menu of *Coulometrics* should become active (see Figure 7). The next time you start *Coulometrics*, you will not see any of the previous warnings or messages. The main menu should be the only window available.

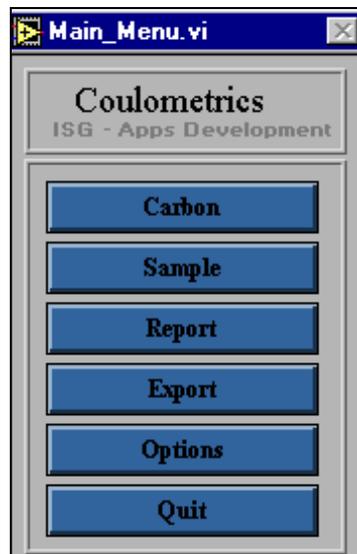


Figure 7. The Coulometrics menu menu.

Coulometrics Options

Two options, the coulometer default settings and the admin options, can be set at the “Options” window (See Figure 8).

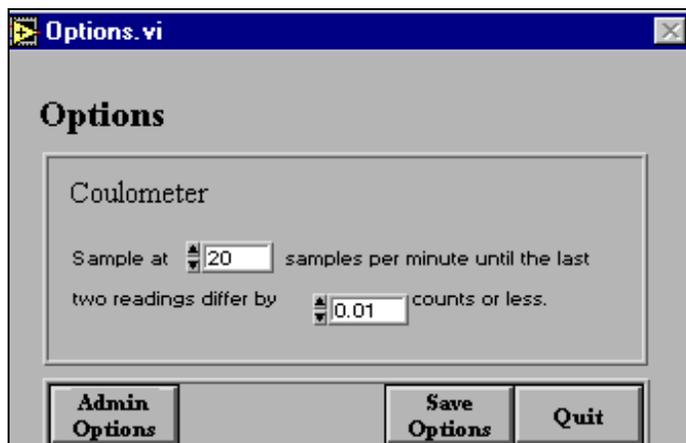


Figure 8. The “Options” window.

Coulometer Settings

The default settings are the number of samples that should be taken per minute until the last two differ by a specific count. Set the sample number and count with the up and down arrow keys or enter them manually.

Note: holding down the Shift key while clicking on an arrow speeds up the scrolling process.

Administrative Options

You must have administrative privileges and a password to access the Administrative Options window. From this window you can set the leg number, coulometer serial port, and balance serial port. You can also edit the currently selected data path by clicking on the **Edit Data Path** button (for more information, See "For the First Time" p. 2).

Note: be sure to click the Save Options button before exiting the “Options” window.

Carbonate Samples

Importing Carbonate Samples

Samples are imported into the *Coulometrics* program from the central database for analysis. These samples can then be viewed and selected on the “Sample_List” window.

1. From the main menu, click on the **Sample** button. This opens the “Sample_List” window (see Figure 9).

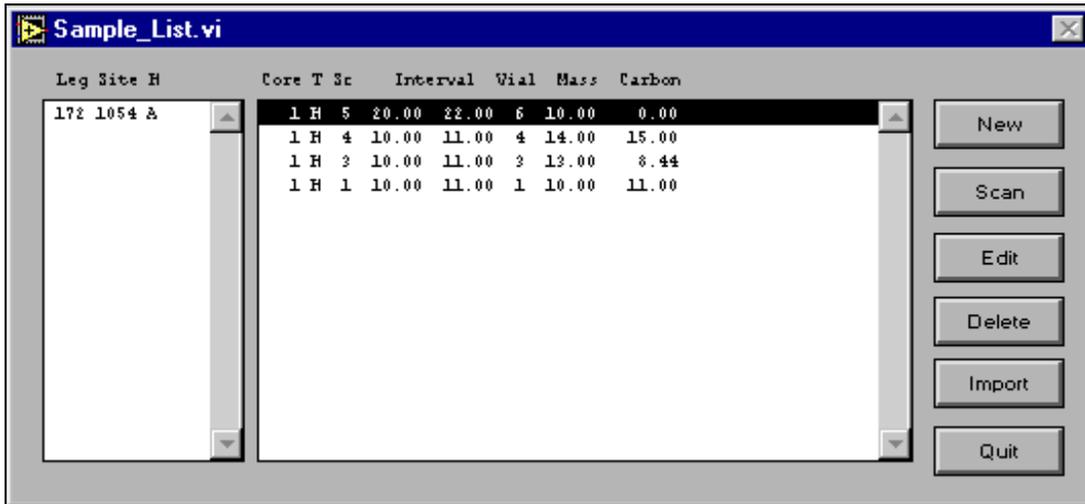


Figure 9. The “Sample_List” window.

2. Click on the **Import** button. This opens a login box, which provides access to the ODP central database (see Figure 10). Enter your username, password, and host. Click the “Remember Password” box if you want the program to retain the password during subsequent logins.

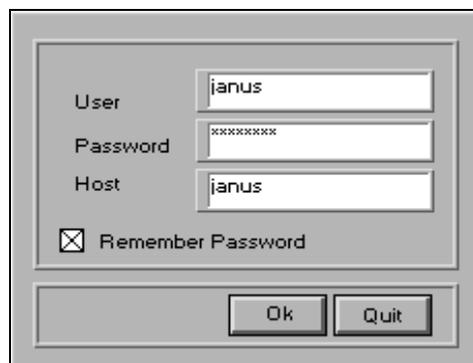


Figure 10. Login for central database.

3. Click **OK** and the “Import_Samples” window will become active (see Figure 11).

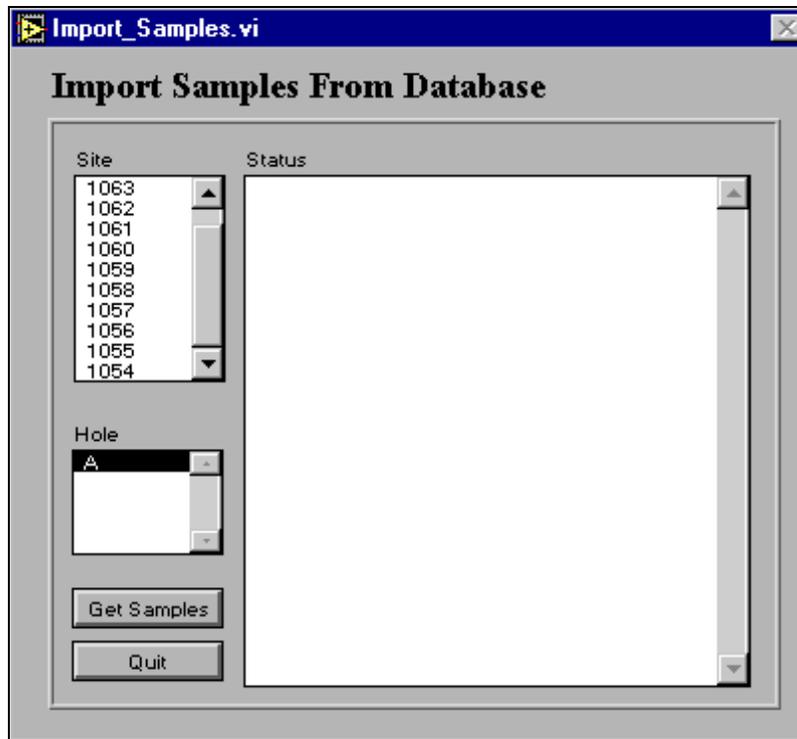


Figure 11. The "Import_Samples" window.

Note: the Sites and Holes available on this window correspond to the Leg number selected on the "Administrative Options" window (See "Administrative Options" p. 5).

4. Select a Site, then a Hole by clicking on them once.
5. Click on the **Get Samples** button. The program will notify you that the samples for your selected Site and Hole are being collected from the central database.
6. Once the samples are retrieved they will be available for selection from the "Sample_List" window (See "The "Sample_List" window." p. 6).

Adding, Editing, or Deleting Samples

From the "Sample_List" window you can scan or add a new sample, or edit or delete existing samples.

Adding a New Sample

1. Add a new sample by clicking on the **Add** button. This opens the "Sample_ID" window (See Figure 12).

The "Sample_ID" window displays the following fields and values:

| | | | | | | | |
|-----------|-----|---------|------|-------------|------|------------|------|
| Leg | 172 | Site | 0 | Hole | | Core | 0 |
| Core Type | | Section | | Top(cm) | 0.00 | Bottom(cm) | 0.00 |
| Vial# | 0 | Mass | 0.00 | Carbon (ug) | 0.00 | | |

Buttons: SAVE, QUIT

Figure 12. The "Sample_ID" window.

2. Set the sample information (e.g. site, hole, core, top, bottom, etc.,) by using the up and down arrows, then clicking **Save**. This saves the new sample to the appropriate leg, site, and hole on the "Sample_List" window.

Note: holding down the Shift key while pressing the up and down arrows speeds up the scrolling process.

Scanning a New Sample

1. Scan a new sample by clicking on the **Scan** button. This opens the "Scan_Sample" window, which has entry boxes for ODP barcode information (See Figure 13).

The "Scan_Sample" window displays the following fields and values:

| | | | | | | | |
|---------------------|---|----------|------|------------|------|------------|------|
| ODP Barcode Top: | | | | | | | |
| ODP Barcode Bottom: | | | | | | | |
| Leg | 0 | Site | 0 | Hole | Z | Core | 0 |
| Core Type | | Section | | Top(cm) | 0.00 | Bottom(cm) | 0.00 |
| Vial# | 0 | Mass(mg) | 0.00 | Carbon(ug) | 0.00 | | |

Buttons: SAVE, QUIT

Figure 13. The "Scan_Sample" window.

2. Use only the bottom two of the three barcodes on the CARB label. Click in the **ODP Barcode Top** box to activate it, then scan the middle barcode on the sample. Repeat for

the bottom barcode, first activating the **ODP Barcode Bottom** box, then scanning the bottom barcode.

Note: The leg, site, hole, etc. information is automatically set.

3. Enter the appropriate vial number in the “Vial#” entry field.
4. Click **Save** to save the new sample to the “Sample_List” window (or you can weigh the sample before saving it, which will send all information including the weight back to the “Sample_List” window).

Editing a Sample

Edit an existing sample by first clicking once on the sample in the “Sample List,” then pressing the **Edit** button. This opens the sample's ID window. Make any changes using the up and down arrow buttons and be sure to **Save** before quitting.

Deleting a Sample

Delete a sample by first clicking once on the sample in the “Sample List,” then pressing the **Delete** button. The program asks if you really want to delete the sample. Click **OK** to continue or **Cancel** to stop.

Weighing Carbonate Samples

Carbonate samples are weighed using the Cahn balance. The balance is connected to a *Coulometrics* balance program that records the sample mass, calculates a standard error for each sample, and plots each mass graphically.

Start the balance program by double clicking on its icon. This opens the “Balance” window (see Figure 14).

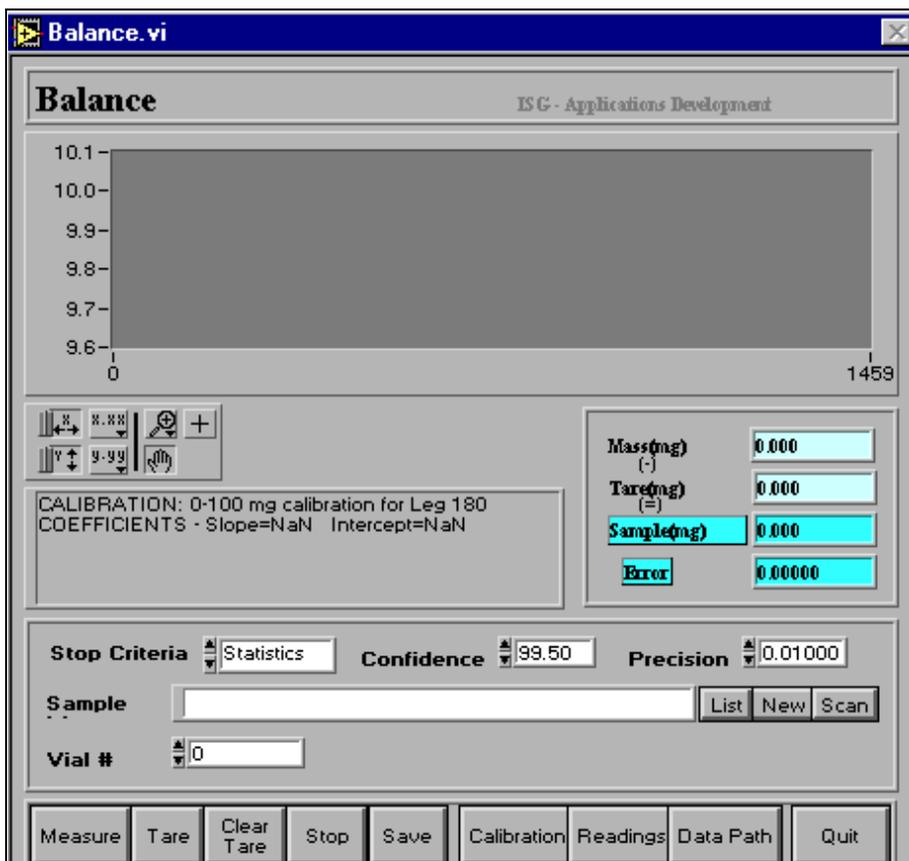


Figure 14. The “Balance” window.

Calibrating the Balance

Before samples are weighed, the Cahn balance must be calibrated. Calibrate the balance using the *Coulometrics* balance program. Two calibrations are done during port call: 0-10 mg for Coulometer/CNS; 1-100 mg for RockEval. The balance should be calibrated with the hardware first and then with the software.

Calibrating the Hardware

1. Set the Cahn balance to measure in the 250 mg to 1 microgram range.

2. After the trays are stationary, press the **Tare** button on the balance.
3. Place a 200mg weight on tray A and press the **Cal** button on the balance.

Calibrating with the Software

1. Be sure the program is set to count by statistics with a precision of 0.01 and a confidence of 99.50. Set these on the "Balance" window manually or with the up and down arrows.
2. Click on **Calibration** to open the "Select Calibration" window (see Figure 15).

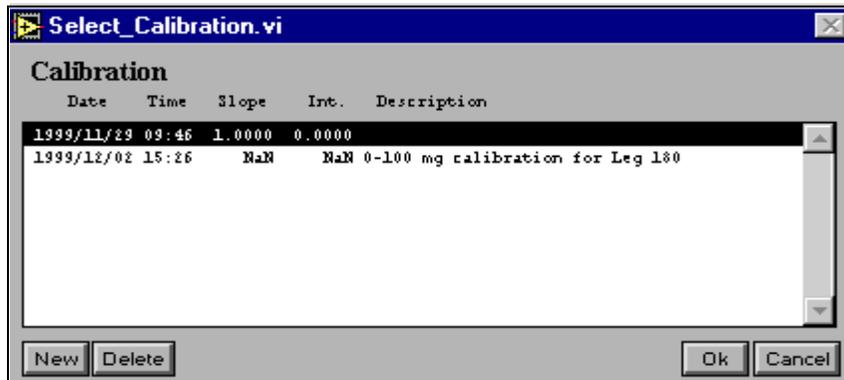


Figure 15. Select the calibration here.

3. The window opens with the default calibration automatically selected. To use this calibration click **OK**. To make a new calibration click on the **New** button.
4. Clicking on **New** opens the "Balance_Calibration" window (see Figure 16).

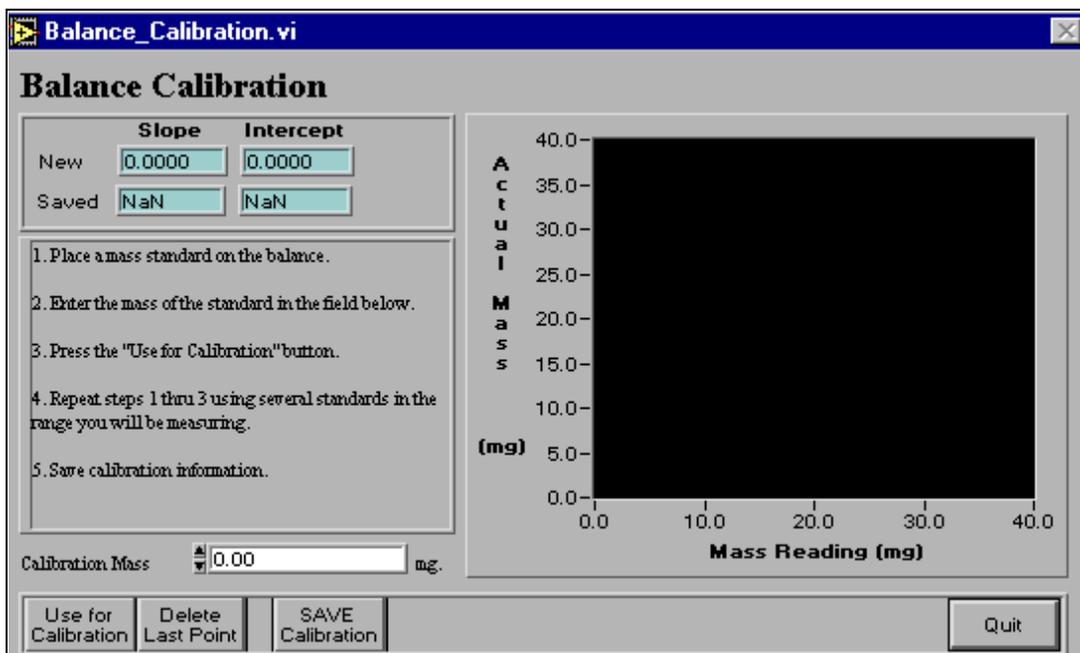


Figure 16. Make a new calibration here.

- Follow the directions on the screen. When you save the calibration, a pop-up window appears asking for a calibration description (see Figure 17). Enter a description (for example, "0-100 mg calibration for Leg 180") and click **OK**.

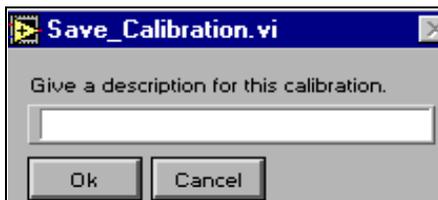


Figure 17. Enter calibration name here.

- Quit** the "Balance Calibration" window and the "Select Calibration" window will reappear with your newly-saved calibration.
- Click on the calibration that you want to use while weighing your samples, then **OK**. The calibration description will be displayed on the middle of the "Balance" window.

Weighing the Samples

Once the samples are imported from the database into *Coulometrics* and the balance calibrated, the samples are ready to be weighed. Samples are weighed using statistical analysis or fixed counts. All measurements are displayed on the "Balance" window (See Figure 18).

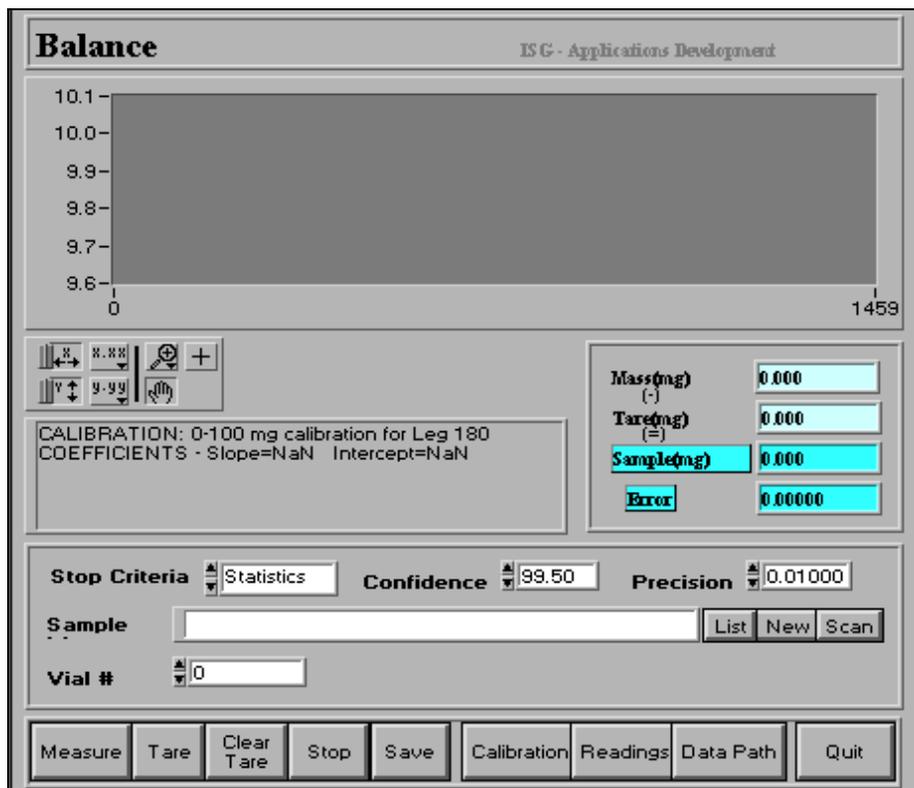


Figure 18. The "Balance" window.

Button Functions

- Use the **Tare** button to tare the empty weighing “boats.”
- Use the **Clear Tare** button to clear the “Tare” field before re-taring the balance.
- Use the **Measure** button to weigh the sample and boat. The difference between the two readings (tare and measure) is the actual sample weight, displayed in the “Sample Mass (mg)” box.
- After weighing each sample, click on the **Save** button to save the weight to the “Sample_List” window.
- Use the **Stop** button to stop the measurement after the program has started.
- Use the **Readings** button to display a list of the measurements you’ve done during the current session.
- Use **Data Path** to set a new path. Be sure to also change the data path in the *Coulometrics* program because they should both point to the same location.

Weighing Procedure

1. From the “Balance” window, click on the **List** button to open the “Sample_List” window.
2. Select the appropriate leg/site/hole in order to display the corresponding list of available samples.
3. Double click on a sample to select it for analysis. The **sample ID** will be displayed in the “Sample ID” entry field of the “Balance” window.

Note: if you already know the sample ID number, you can enter it directly into the “Sample ID” entry field on the “Balance” window.

4. Enter a vial number into the “Vial#” field.
5. Weigh the sample and be sure to **Save** before starting the next sample.

Weighing Other Types of Samples

Samples other than carbonate can also be weighed using the MAD program (CaCO₃ standard or CNS standard/sample). In these cases, keep the vial number at zero and leave the sample IDs blank. Use only the **Tare** and **Measure** buttons to weigh the samples. The program does not prompt you to save the weights. Instead, you must keep a record of the masses as the program completes each measurement.

Running the Coulometer

Once the carbonate samples are weighed, they are measured for their carbon and carbonate percentages using the Coulometer and the *Coulometrics* software.

1. From the *Coulometrics* menu, click on the **Carbon** button to open the “Coulometer” window (See Figure 19).

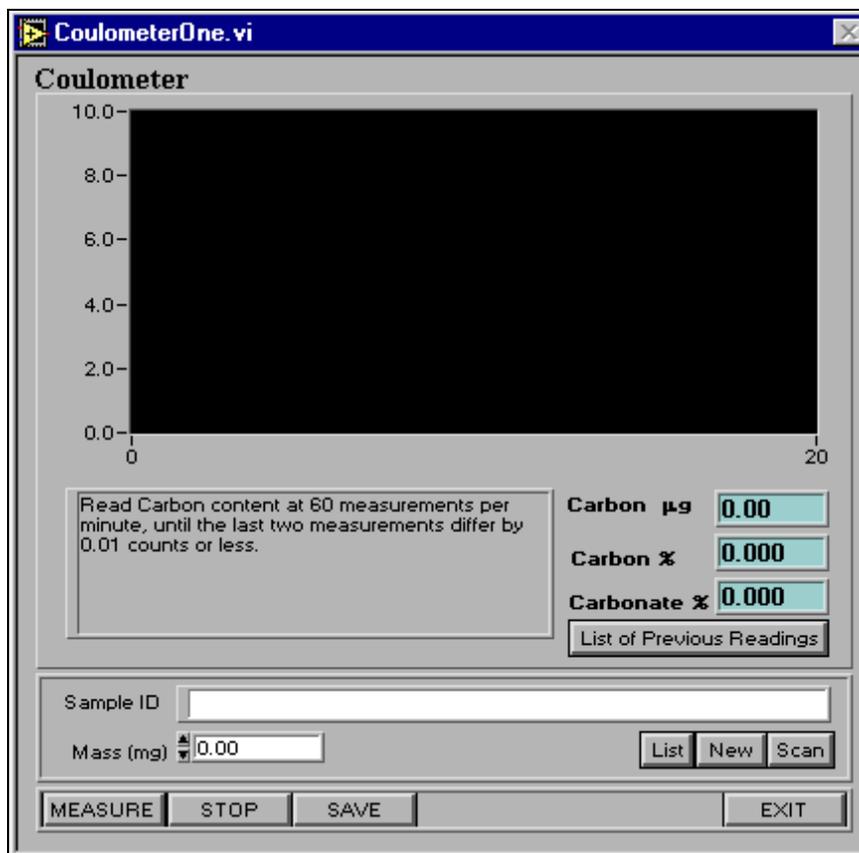
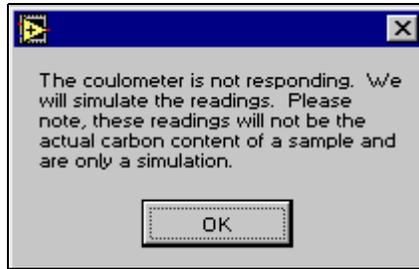


Figure 19. The “Coulometer” window.

2. Select a sample for analysis by either entering the vial number into the “Sample ID” entry field, or by selecting it from the “Sample_List” window (click on the **List** button to open the sample list, then double click on the sample you want to analyze).
3. When the sample is ready for analysis, click on the **Measure** button and inject the acid. The carbon and carbonate percentages are displayed graphically and numerically on the window.
 - If the program has a problem reading the sample, you will receive the following warning:



- The program will then simulate the readings with a graphical output (see Figure 20). **Note that these readings are not the actual carbon content of the sample.**

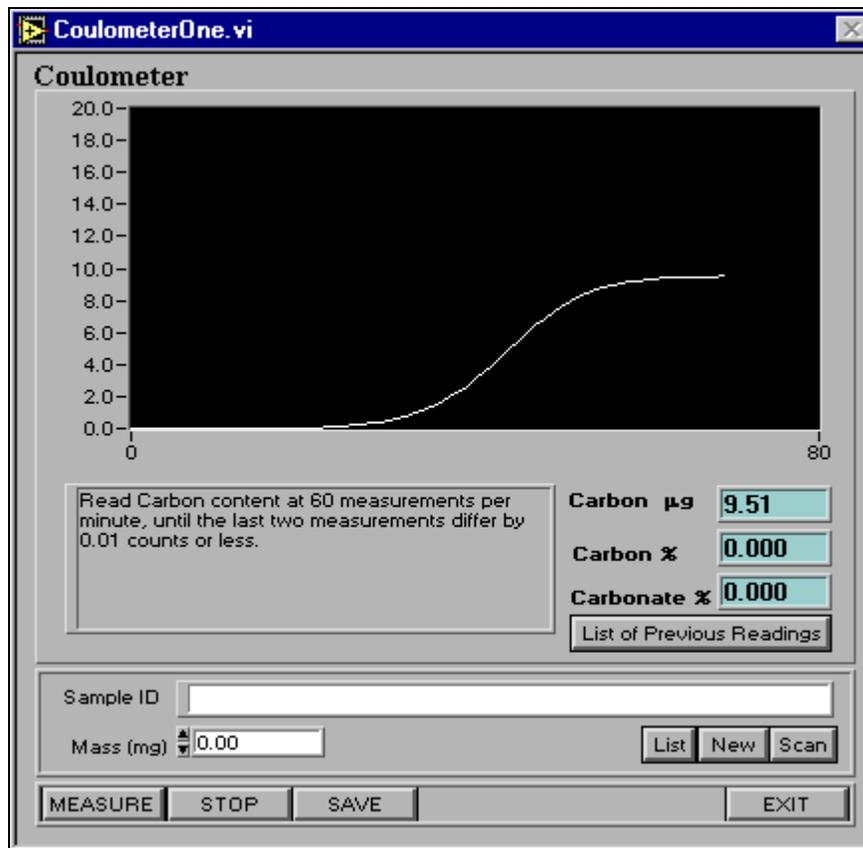


Figure 20. Simulated coulometer reading.

Note: clicking on the “List of Previous Readings” button will list the results from the last 10 readings taken.

4. After a successful analysis is run, the program will prompt you to **Save** the data.

Note: When the next sample ID and vial number are entered, the previous run results and graph default to zero.

Measuring samples not in the “Sample_List”

If a sample is not listed in the “Sample List” window (such as a standard or an ‘unofficial’ sample), use the **New** button to enter the sample.

The **New** button opens a “Sample_ID” window, with the vial number and sample ID defaulting to the last vial number and Sample ID processed. These numbers can be edited using the up and down arrows.

Enter the weight directly into the “Mass” box and click **Save**. Click on the **Tab** key to recall the vial number to the “Coulometer” window, then click **Measure**. Do not save the run once finished.

Reports

Carbonate results (%INOR_C and %CaCO₃) can be viewed at the same time that samples are being run.

1. From the *Coulometrics* menu, click on the **Reports** button to open the “Report” window (See Figure 21).

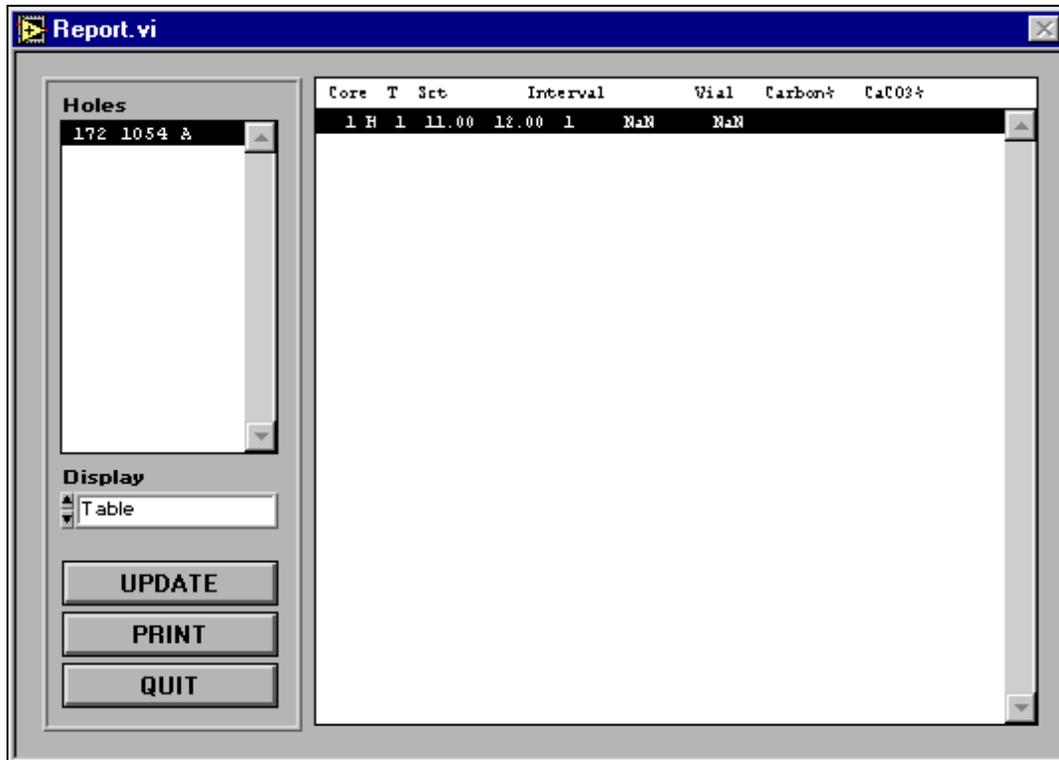


Figure 21. The “Report” window.

2. Select the appropriate leg/site/hole from the “Holes” box by clicking on it once. Use the up and down keys to scroll through the list.
3. Select the correct report format, “Table” or “Graph,” from the “Display” field. Use the up and down arrows to toggle between the two selections.
4. Click the **Update** button at any time to display the most current carbonate information.
5. Click the **Print** button to print a copy of the table or graph.

Exporting Samples

Sample data can be exported to Excel or a text file for further data analysis.

1. From the main menu, click on **Export** to open the “Export” window (See Figure 22).

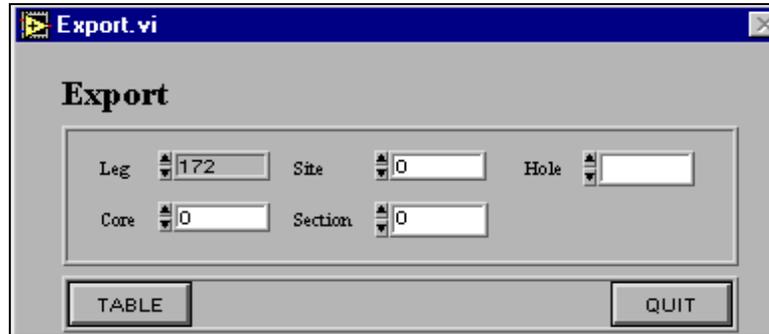


Figure 22. The “Export” window.

2. Use the “Export” window to select specific data for export by setting the appropriate fields with the arrow keys. For example, if you wanted to export all samples for leg 172, you would leave all fields blank except “Leg.” If you wanted to export samples from specific sections, you would need to set all the fields on the window.
3. Click on the **Table** button to display all selected samples (see Figure 23).

| Leg | Site | H | Cor | T | Sct | Interval | Vial | Carbon | CaCO ₃ |
|-----|------|---|-----|-------|-------|----------|------|--------|-------------------|
| 172 | 1054 | 1 | 1 | 11.00 | 12.00 | 1 | NaN | NaN | |

Figure 23. Table of carbon content data for export.

4. From the generated list, delete any samples that you don't want exported by first highlighting them (highlight more than one by holding down the **Shift** key while dragging the mouse over the samples), then clicking the **Remove** button.

Note: this does NOT delete the samples from the database, it only removes them from the list being exported.

5. From your final list you can export the samples to either a text file or Excel by clicking on the appropriate button.

Exporting to a Text File

1. When your sample list is complete, click on the **Text File** button. This opens the "Save As" dialog box (see Figure 24).

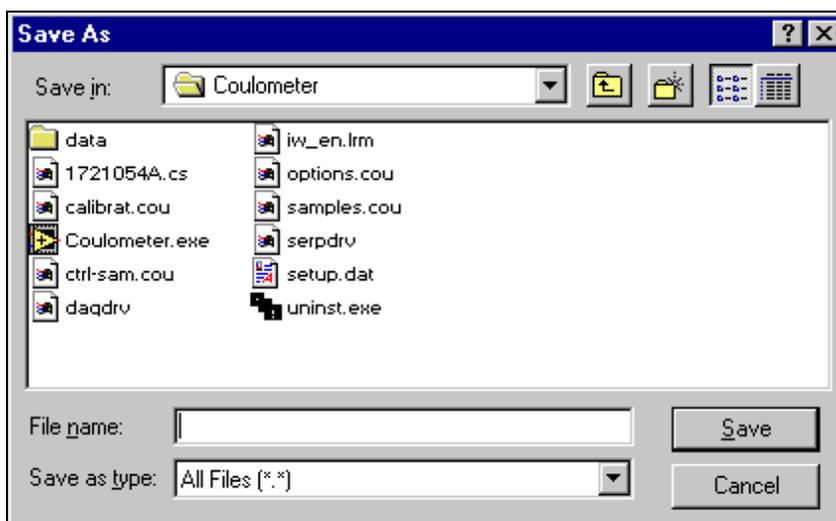


Figure 24. Saving samples as a text file.

2. Navigate through the box until you find the location where you want to save the text file. Name the file then click **Save**.

Exporting to Excel

1. When your sample list is complete, click on the **Excel** button. This opens the "Shell_to_Excel" window (see Figure 25).

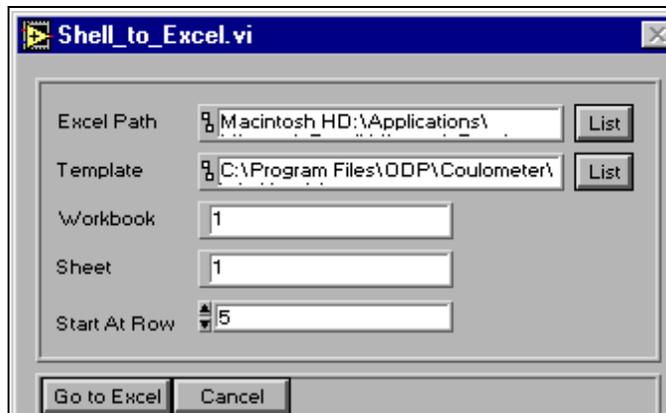


Figure 25. Exporting samples to Excel.

2. In the “Excel Path” field, enter the directory where the executable for Excel is located. If you do not know the path, click on the **List** button to open a dialog box (see Figure 26).

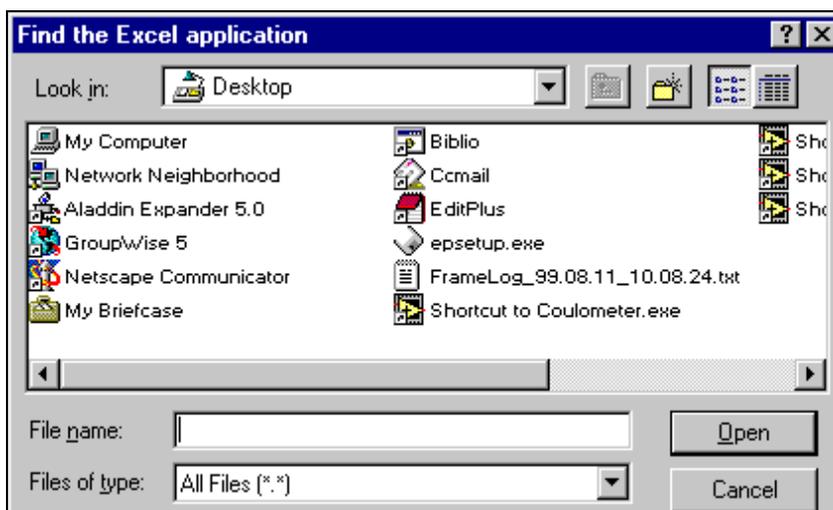


Figure 26. Find Excel application from here.

3. Navigate through the box until you find the executable Excel program, then click **Open**.
4. The path should now be in the “Shell_to_Excel” window. You can also set a path for an Excel template in the “Template” field. Do this in the same manner as the “Excel Path.”
5. Once all paths are set, click on the **Go to Excel** button. This automatically opens Excel, and any templates you specify, with your selected sample data.

Appendix A. Installation and Setup

This section explains how to install and set up the new *Coulometrics* software.

What You Need

1. A PC computer with a minimum 32 MB of RAM (64 MB recommended).

Note: the *Coulometrics* application can be run using multiple computers as long as the computers are networked together (e.g., if you want to control the balance and the coulometer with separate computers, or if you want to use more than one balance or more than one coulometer, etc.).

2. At least one coulometer (Coulometrics, Inc. model 5011).
3. At least one Cahn 28/29 automatic electrobalance with RS232 port.
4. Two serial cables. These are standard cables normally used to connect modems (with a 25pin serial connector) to the PC's serial port. For this application they will be used to connect the coulometer and the balance to the PC's serial ports.
5. A custom-made adapter cable for the balance. (The wiring of this cable is explained later.)
6. A hard disk drive with at least 10 MB of free disk space.
7. At least a 15-inch monitor (17 inch recommended).
8. A printer supported by your system (if you plant to print reports).
9. Microsoft Excel. (This is only required if you would like to do additional data manipulations in Excel.)

Note: the *Coulometrics* application can execute Excel and pass data to it directly, eliminating double entry of data.

Hardware Settings

The coulometer and the computer communicate through their serial ports, thus the Coulometrics, Inc. model 5011 coulometer must be configured properly in order for it to communicate with the computer.

The coulometer's serial port should be configured for:

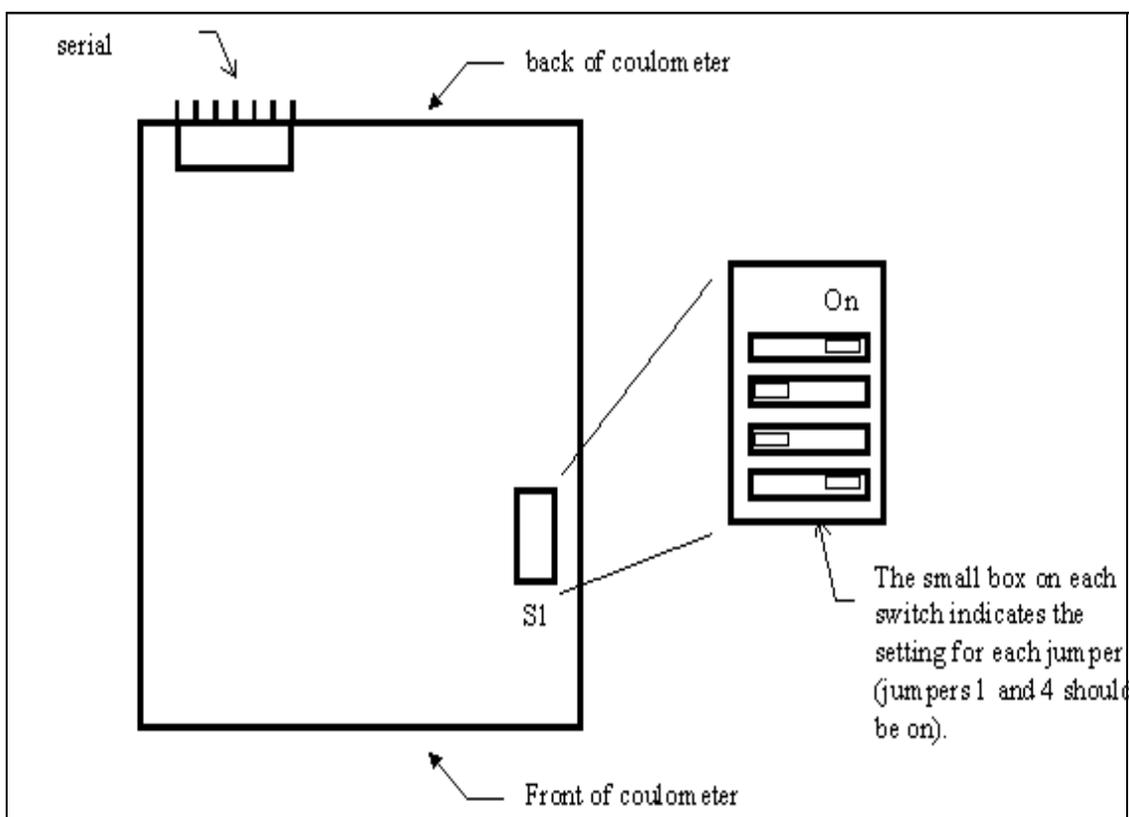
- Baud Rate: 9600
- Data Bits: 8
- Stop Bits: 1

- Parity: None

The coulometer, by default, is set to a much lower baud rate than shown above. The above baud rate is recommended because the default rate causes communication between the coulometer and the computer to be much slower, severely limiting the rate at which data can be acquired.

To configure the coulometer to the proper setting you must:

1. Turn off the power to the coulometer.
2. Remove the top cover from the coulometer to expose the circuit board.
3. Set the serial port by means of the jumper S1. Each of the 4 jumper switches must be set as shown in the figure below for the required settings.



4. The small box on each switch indicates to which side the switches should be set (switches 1 and 4 should be set to "On").
5. After all switches are properly set, replace the cover to avoid damaging the circuit.
6. Turn on the coulometer.

Hardware Settings on the Balance

The balance and the computer communicate through their serial ports, thus the Cahn 28/29 balance must also be configured properly in order for it to communicate with the computer.

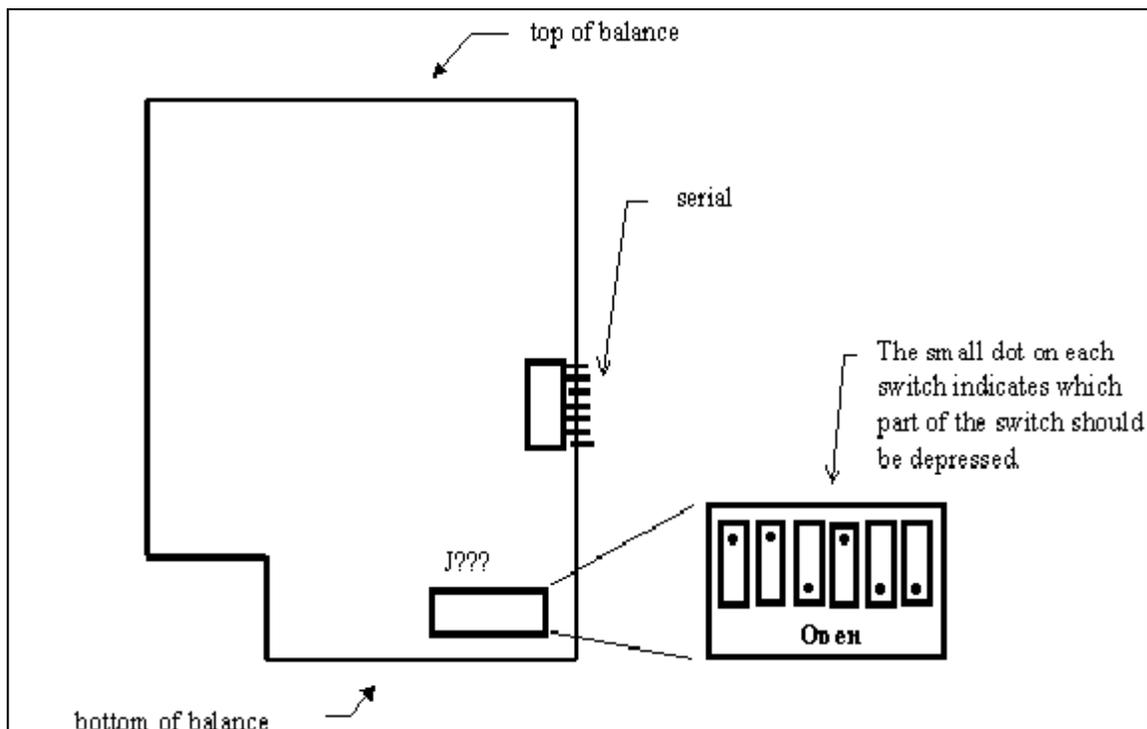
The balance serial port should be configured for:

- Baud Rate: 1200
- Data Bits: 8
- Stop Bits: 1
- Parity: None

The balance, like the coulometer, may have a different default factory setting for the baud rate. The above baud rate is recommended over the default setting.

Configure the balance to the proper settings:

1. Turn off the power to the balance.
2. Remove the top cover of the balance to expose the circuit board.
3. The settings for the serial port are accomplished by means of jumper???. Each of the 6 jumper switches must be set as shown in the figure below.

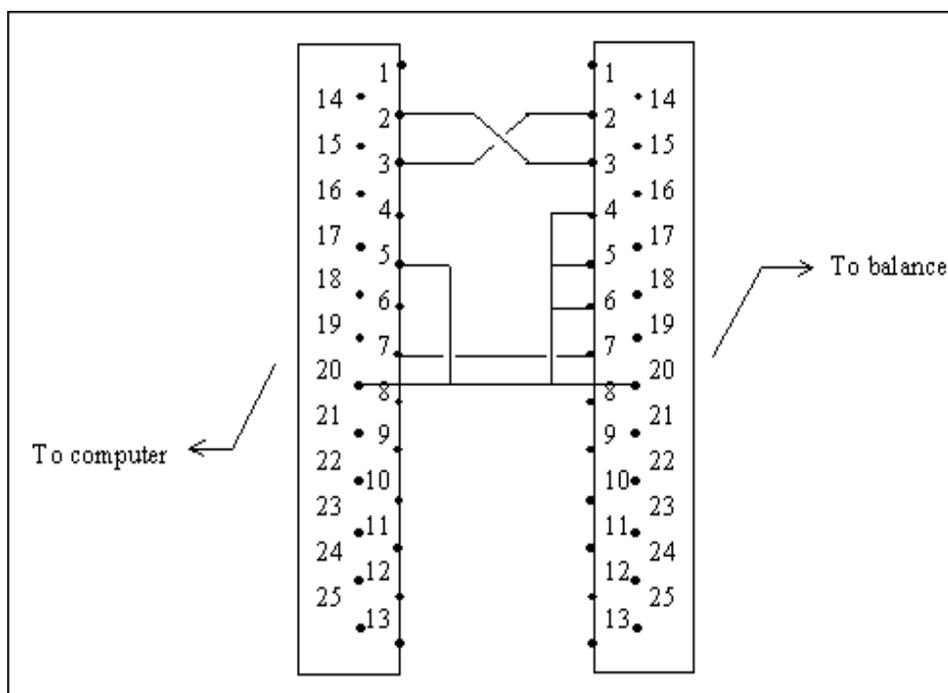


4. The small box on each switch indicates which part of the switch to depress.

5. After all switches are set correctly, replace the cover to avoid damaging the circuit.
6. Turn the balance on.
7. Press the **Tare** button and calibrate the balance (See "Calibrating the Balance" p. 10).

Connecting the Balance to Your Computer

1. You will also need a custom-made adapter cable. The wiring for this cable is described in the figure below.
2. It might be possible to replace these cables with a "null-modem" cable. This cable should go from the connector used by the PC's serial port to the 25-pin serial connector used by the balance.



Installing the Coulometrics Software

1. *Coulometrics* will either be provide as a CD or will be available on one of the ship's servers.
2. Double click on the the "setup.exe" for *Coulometrics* in order to start the installation.
3. The remainder of the installation follows a standard windows install.

Installing the Program in Other Computers

You must also install the *Coulometrics* program in all other computers that will use it. In this case, you should create a folder for the data files. The folder should be shared by all the

machines that will be processing coulometrics data.

Upgrading

To upgrade from a previous version of *Coulometrics*, install the new version in the same directory as the older version. This will ensure that the new version uses the same data files as the old version. **The installation process will NOT erase the previously saved data**; however, it is recommended that you make a backup of the data and programs before installation.

Starting Coulometrics for the First Time

The very first time the program is run, several steps must be taken which will not have to be repeated again.

1. The program will ask you for a path where it can store data files. Create a directory or folder that can be shared by all the machines that will be processing *Coulometrics* data (this includes the Coulometer and balance computers). The program will then create the data files in the path that you specified.
2. The first time you run *Coulometrics* on a second computer, you should specify the same path. The program running on the second machine will then share the files created by the first computer.
3. Finally, the program prompts you through the creation of all files and default settings for the configuration parameters.

Note: the second time you start this program these steps should NOT occur. If they do, contact a technician and do not continue until the problem is solved.

Appendix B. File Formats

The following are samples of Coulometer file formats.

Calibration

Table 1: Calibration

| Time Stamp | Bmeas | Ameas |
|---------------------|--------|--------|
| 1996/07/18 16:23 | 1.0000 | 0.0000 |

Control-Samples

Control Type Expect C Measur StdDev Time Stamp

Datapath.ini

Macintosh HD: Applications: LabVIEW 4: Data

Options

Table 2: Options

| Time Stamp | Bmea | Bsecs | Bal.SD | Pmea | PmaxSD | PPurg |
|---------------------|------|-------|--------|------|--------|-------|
| 1996/05/29 15:05 | 100 | 10 | 2.00 | 5 | 0.00 | 1 |
| 1996/05/29 15:09 | 100 | 10 | 5.80 | 5 | 0.01 | |
| 1996/05/29 15:24 | 100 | 10 | 5.80 | 5 | 3.00 | |
| 1996/05/30 15:32 | 100 | 10 | 5.80 | 20 | 0.10 | |
| 1996/05/31 09:29 | 100 | 10 | 5.80 | 20 | 0.03 | |
| 1996/05/31 10:21 | 100 | 10 | 5.80 | 20 | 0.02 | |
| 1996/05/31 10:22 | 100 | 10 | 5.80 | 30 | 0.02 | |
| 1996/07/12 14:35 | 200 | 10 | 0.10 | 20 | 0.05 | |
| 1996/07/15 10:59 | 20 | 5 | 0.20 | 20 | 0.05 | |
| 1996/07/16 15:25 | 20 | 5 | 0.20 | 20 | 0.10 | |
| 1996/07/17 14:29 | 50 | 20 | 0.10 | 20 | 0.10 | |
| 1996/07/17 14:47 | 50 | 20 | 0.20 | 20 | 0.10 | |

Samples

Leg Site H Cor Sc Intv-T Intv-B V# Depth Mass-g MassSD Carbon Comments
Time Stamps

Settings

Table 3: Settings

| Time Stamp | Leg | C. Ser | B. Ser |
|---------------------|-----|--------|--------|
| 1997/08/07 14:37 | 100 | 000000 | 000000 |
| 1997/08/07 14:37 | 166 | 000000 | 000000 |