Chemistry Lab Cookbook
Balance Software, v210.0

Instrument 1:
    Cahn Balance
        ODP   #: 02344
        Model #: 31
Website:
    http://www.thermocahn.com
Software:
    ODP Labview v.198

Instrument 2:
    Scientech Balance
        ODP   #:
        Model #: 202-004
Software:
    ODP Labview v.198

Documentation Available:

Document Name/Title/Number	Source	Version	Associated Instrument(s)	Associated Measurement(s)
Cahn 28/29 Automatic Electrobalance W/ RS232 Output (Owner's Manual)	Cahn Instruments, Inc.	n/a	Cahn balances	weights
Cahn Instruction Manual	Cahn Instruments, Inc.	Rev. 6/90	Cahn balances	Weights
Labview User Guide	In house	198		

Balance Systems User Guide
v.210

Accelerations, induced by the ship's motion, make precise weighing at sea difficult. To measure weights in this environment it is necessary to cancel out all forces acting on the balance that are not directly related to the sample weight. These include: ship's heave and vibration, electronic drift due to temperature and power fluctuations, and vertical components of roll and pitch. In addition, there is a long term (from hours to days) differential electronic drift.

To weigh at sea it is thus necessary to use a motion compensated balance. The motion compensated balances used aboard the *JOIDES Resolution* are based on the design concept described by Childress and Mickel (Deep Sea Research, Vol 27A, *A motion compensated shipboard precision balance system*).

Another aspect of weighing at sea is to collect data over a time period sufficient to define the ship's heave. The *Joides Resolution* has a natural period of about 5 to 7 seconds so it is important to weigh for at least 25 to 35 seconds. In compound swell conditions it may be necessary to double this time.

On-board the *JOIDES Resolution*, there are two types of compensated balance systems:
 Scientech Balances and Cahn Microbalance.

Both systems are controlled by a LabView software program.

A. Scientech Balances

The Scientech balance system is a traditional motion compensated balance system. This system uses a reference balance with its load matched to the unknown weight on the sample balance. The reference balance partially compensates for ship motion and vibration. Analog differences in output between the reference sensor and the weighing sensor are calculated in real time and can be removed from the true weight value by averaging.
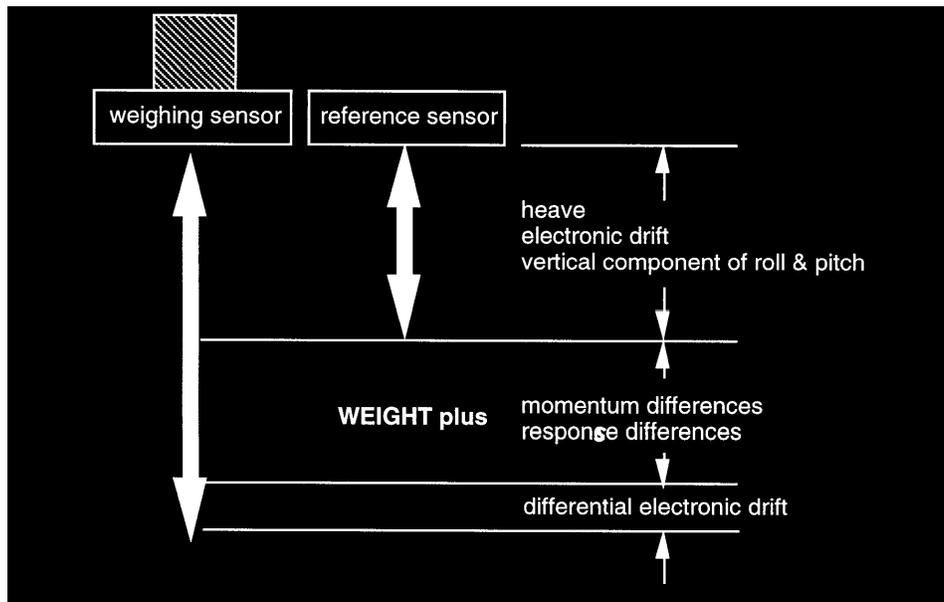
A. 1. Principle and Mode of Operation

The Scientech balance system is composed of two Scientech top loading balances (model 202-004), two controllers, a National Instrument SCB-86 Interface, and a National Instrument (NI) DAQ board.

The two top loading balances are weighing sensors. The "known" sensor acts as a reference sensor while the "unknown" sensor is the actual weighing sensor. Each sensor's basic weight signal is an unfiltered analog voltage. The no load signal is typically 1.50 volts. The signal will increase linearly at the rate of 1 volt per 10 grams of weight placed on the weighing pan to approximately 6.50 volts at full capacity (50 grams).   Each sensor has a factory rated precision of +/- 0.1 mg. On board the ship, full capacity is about 30 grams (to prevent clipping at the 6.5 volt limit during heavy seas) and the precision is of +/- 0.5 mg.

The controllers provide the power to the sensors and monitor/adjust the voltage back from/to the sensors. There is one controller per sensor. The basic controller has a power switch, an analog weight signal output (Analog Output), and a calibration adjustment for the analog output (Analog Calibrate). The Analog Output has two banana jacks (tare and gross) in the front of the unit that permit external recorders and display equipment to be used with the 202. On board the *JOIDES Resolution*, only the gross output is used. It connects to a National Instrument SCB-86 box interfaced with a NI DAQ analog-to-digital board. The exact calibration is set by the Analog Calibrate adjustment (rear panel of unit). The Analog Calibrate is factory set and should not require further adjustment.

The voltage from the two sensors is sampled and digitalized using a two channel NI DAQ board. The two digitalized signals are then passed directly to a LabView program. The program emulates the functions of a differential amplifier which subtracts voltages of the two sensors from each other and then adjusts the gain and zero values of the reference sensor so that the response of the reference sensor will match the response of the weighing sensor (i.e. till the difference shows 0.00 mV output).

The LabView software used to control the Scientech balance is an icon based programming language developed by National Instruments. Applications generated with LabView are called Virtual Instruments (VIs). VIs actually run under the LabView application as a collection of coded resources. The version of LabView used in all the laboratories is not a full blown version but a run-time version (4) which does not allow editing of the program or window display. To understand more about LabView 4 and VIs, refer to the LabView 4 *Getting Started Manual*.

The Scientech LabView application is located on the hard disk in the Scientech folder. The VI used to control the Scientech balances is called Scientech.vi and is launched by double clicking on the Scientech icon on the desktop.

Scientech balance systems are used on-board in three Laboratories: Chemistry, X-Ray and Physical Properties. At the beginning of each Leg, these balances need to be tuned and calibrated before being use for weighing. This should to be done in port.

A. 2. Tuning the Sensors

Once the Scientech.vi is loaded, the following Control window is displayed:
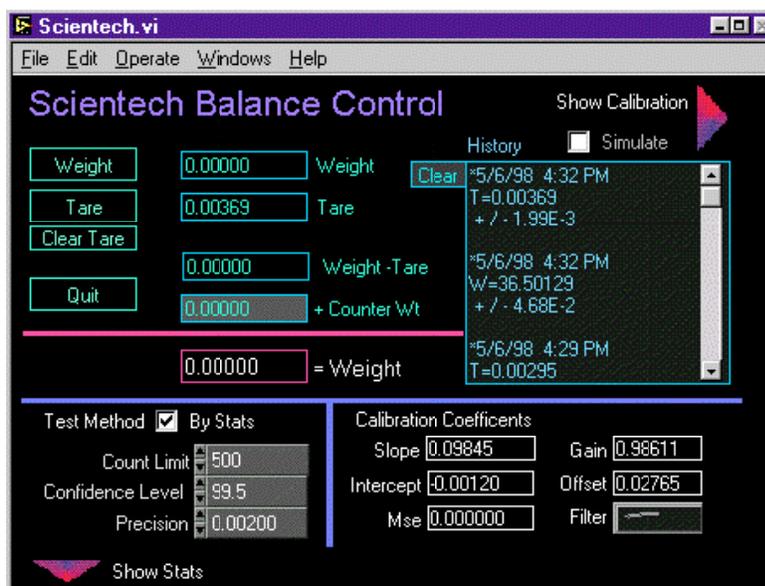
Clicking on the Show Calibration arrow in the upper right of the Control window will open an extension to the right of this window. Clicking on the Tune Sensor Utility button will load the Scientech Tuner.vi. as displayed below:
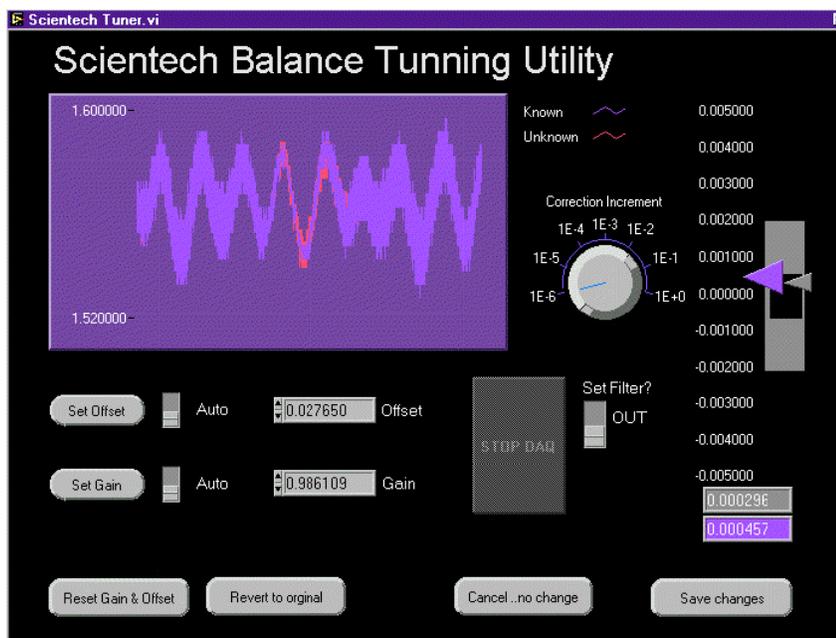
The tuner.vi allows the user to apply a gain and zero value to the reference sensor's output so as to match the zero and gain of the two balances. The matching procedure is carried out by adjusting the output to zero mV with a zero load and then placing a 20g weight on each sensor and adjusting again the output to zero. The procedure is repeated until both conditions of balanced load (zero g and 20g) shows 0.00 mV output.

To perform the zero and gain tuning, proceed as follows:

 With no load on the balances, click on the Set Offset button. The user can set the zero (and gain) values by either using the Correction Increment knob (Auto) or directly by entering the values into the digital controls (Manual);
 If the Auto mode is selected, move the Correction Increment knob until the sensors' readings displayed below the right vertical scale are approximately 0.00 (i.e., the purple and grey arrows will be superimposed in the blue box), click on the Stop DAQ button to stop data acquisition;

 Put a 20g weight on each balance, click on the Set Gain button and move the Correction Increment knob until the sensors' readings are approximately 0.00. Click on Stop DAQ;
 Remove the weights and set the offset (zero) again. Repeat the previous steps until both the zero and gain are set to read 0.00;
 Saving the Changes will store the new values of the offset and gain in the program and will exit the tuner.vi back into the Control window. The offset and gain are shown in digital indicators in the Control window next to the Calibration Coefficients indicators.

Note 1: In LabView, the VI windows contain various types of controls and indicators. Controls are means to supply data to the VI. To enter or change values in a digital control, click on the control itself and enter the new value or use the increments button (up/down arrows) on the left edge of the control. Indicators display data generated by the VI and cannot be modified. Buttons perform functions as designed in the VI.

Note 2: You can tune the zero and gain by either adjusting the last Leg's readings or by starting from 0.00 after clicking on the Reset Gain and Offset button.

A graph of each sensor's signal is provided for the user's information. When the output difference is minimum, the signals will almost be superimposed.

A. 3. Calibrating the Sensors

Calibration of the sensors is performed at the beginning of each Leg, in port. Clicking on the Show Calibration arrow in the upper right of the Control window will open an extension (Calibration window) to the right of the Control window as displayed on the next page:

Every time the Calibration window is open, the Calibration Coefficients in the Control window are automatically set to the following default values: slope =1 and intercept = 0. When clicking on either the Tare or Weigh button, readings displayed in the associated digital indicators will be actual voltage readings.

Note: Calibration parameters are set differently depending on the balance usage in a specific Laboratory. In the Chemistry Lab., the Scientech balance is used most of the time only to weigh chemicals and, as a result, precision is not critical. The following paragraph illustrate a calibration as performed in the Chemistry Lab.
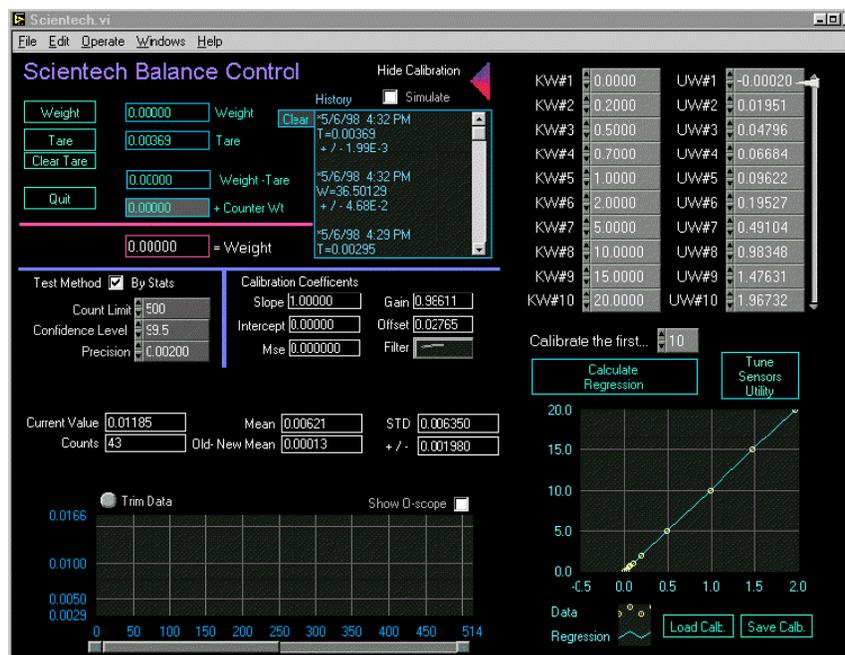
To calibrate the sensors, proceed as follows:

 Set the Test Method to "By Stats";
 Set the Confidence Interval to 99.5% and Precision to 0.05%;
 Clear the Tare;
 Open the Calibration window and type the values of all the calibration weights (0, 0.2, 0.5, 0.7, 1, 2, 5, 10, 15, and 20g) in the known weight controls (KW#1 to #10);
 With no load on either sensor (KW#1 = 0), click on the Weight button. The resulting voltage measurement is displayed in the UW#1 control;
 Place a 200mg weight on the "unknown" sensor (KW#2 = 0.2) and hit Weight. The resulting voltage measurement is displayed in the UW#2 control. Notice that the arrow on the right of the UW controls automatically goes down to the next calibration level. To remeasure a calibration level, use the arrow to slide up and down to that particular calibration level;
  Proceed as above for the remaining calibration levels in order for a regression analysis to be performed;
 When the measurement are all done, hit the Calculate Regression button and then Save Calib. to save the calibration data to a file (e.g., 179_20). The Calibration Coefficients are subsequently displayed in the main Control window;
 Place a 5g weight on the "unknown" sensor and weigh. The given weight should be within two decimal places of the theoretical value of the weight.

Note: A Calibrate the First...control allows the user to set the number of levels to be used in the linear regression calculation. You can also review how the data fits the calibration curve on the displayed graph.

A. 4. Weighing a Sample

Before Taring or Weighing, the operator has to select a Test Method: By Stats or By Counts. The values entered in the Count Limit, Confidence Level and Precision controls are used by the Tare, Weight, and Calibration functions to determine how and when to stop the weighing (other than clicking the Halt button).
The Scientech Control VI provides three methods of halting the weighing process:

Clicking on the Halt button will stop the process at any time;

When the Test Method is switched to "By Counts", weighing will stop when the number of counts entered in the Count Limit control is reached;

When the Test Method is switched to "By Stats", the operator has to select the confidence interval and precision from the Confidence and Precision controls respectively, and set the count limit from the Count Limit control. Weighing will stop when either enough counts have been taken to satisfy the precision and confidence interval selected or the count limit is reached, which ever comes first.

The "By Stats" method performs a statistical test that is based on the assumption that the mean of an infinite number of values will be true value. This also assumes that the variation of those values have a normal distribution. The question is: how many values need to be collected to have mean of +- Y mg of the true value and with a confidence level of X% .

As the Scientech VI runs, it accumulates a weighted mean. After the mean has been computed, the VI calculates the current precision value (error) using the Student's T test. This statistical test is sensitive to the value of the confidence interval selected, the size of the measured values' variation (a function of the ship's heave and electronic drift) and the degrees of freedom (i.e., number of values averaged). When the error is equal to or less than the precision selected, the weighing process will halt.

Clicking on the Show Stats arrow will open an extension window below the main Control window which displays the various statistical parameters used by the Stats method such as Mean and Standard Deviation (STD). The error is displayed in the +/ - indicator.
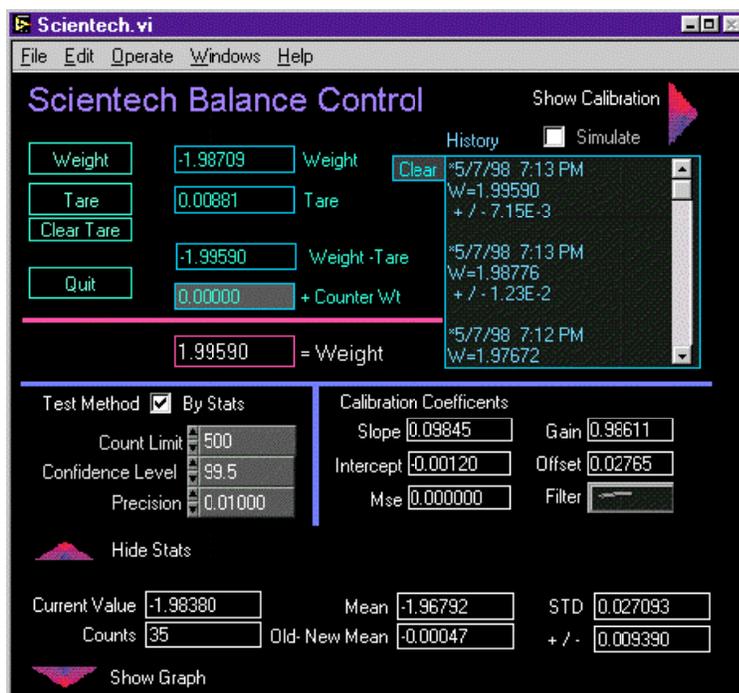
To weigh a sample, proceed as follows:

Set the Test Method to either "By Stats" or "By Counts";

Select the Confidence Interval and Precision;

Place a piece of weighing paper or a weighing boat on each balance and click on Tare. The
resulting measurement gives the difference in weight between the two papers (boats),i.e. the Tare weight;

Add the sample on the "unknown" sensor and click on Weight. The resulting measurement gives the sample
Weight as the difference between total weight and tare weight. When weighing a sample which weighs more than
one gram, a counterbalance with a known weight (close to the expected weight of the sample) should be used.
Place the counterbalance weight(s) on the "known" sensor and add the sample to the "unknown". Enter the weight
of the counterbalance in the Counter Wt control and click Weight. The weight of the sample is now equal to total
Weight -Tare + Counter Wt;

Add and remove the sample from the "unknown" sensor and weigh till satisfied with the weight.

Note: The last ten measurements can be seen on the History window. Clicking on the Clear button on the upper
left corner of this window will erase all measurements displayed in it.

The weighing process can take several tries before one obtains the desired amount of sample/chemical. To quickly
get to this weight (add and remove), weighing can be stopped at any time by clicking on the Halt button. Another
quick way to stop the weighing is to use the Trim Data function which is accessed by first opening the Stats
window and then the Graph window shown below (by clicking on the Show Graph arrow at the bottom of the
Stats window):
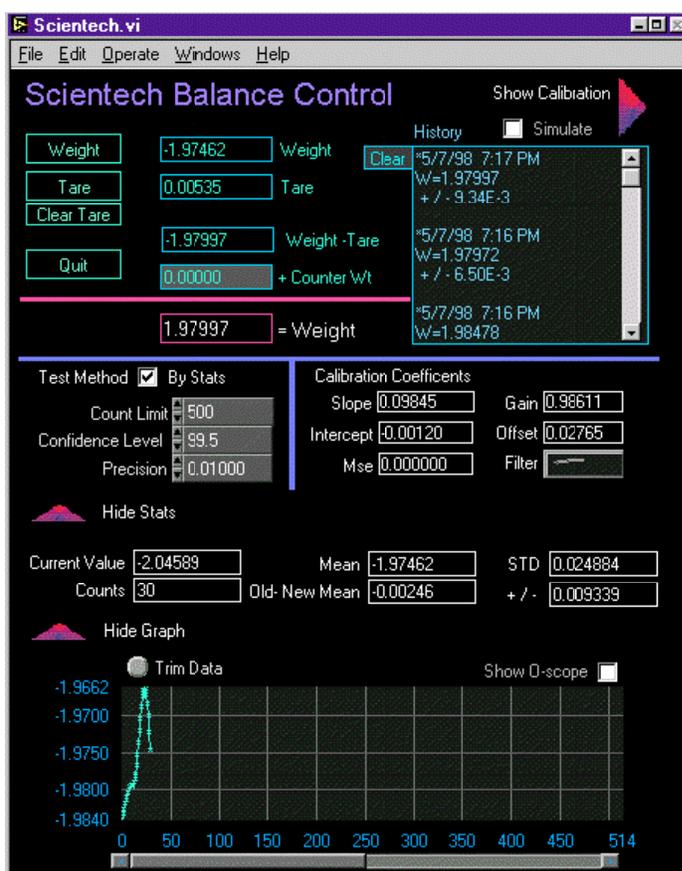
B. Cahn Microbalance

The Cahn microbalance (model C-30/31) is designed for weights up to 3.5g and is sensitive to changes as small as 0.1mg. It is best suited for weights below 1250mg. The Cahn works on the "balance beam" principle (explained below). Like the Scientech balances, the Cahn is sensitive to the ship's roll and pitch. To compensate for the ship's motion, a gimballed table is used. There is no tuning involved with the Cahn balance but a calibration (hardware and software) is performed in port.

The Cahn balance is used only in the Chemistry Lab. Samples run on the Coulometer, CHNS and Rock Eval are weighed from 0.1 to about 100mg with a precision of +/-0.001mg.

The C-30/31 has a standard RS232C port which is connected to the serial port of a Macintosh computer (MC71). A six position dip switch installed on the processor of the C-30/31 controls the configuration of the serial port (1200 baud, 8 data bits, 2 stop bits and no parity). The balance is controlled by the Coulometer/Balance LabView program which runs simultaneously on two Macs (MC67, for the Coulometer; and MC71, for the Cahn).

The following section briefly describes the principle and mode of operation of the Cahn balance when not controlled by the LabView software. Calibration and weighing using the Coulometer/ Balance program is described in section B. 2.

B. 1. Principle and Mode of Operation

The Cahn balance consists of a balance beam mounted to, supported by and pivoting about the center of a taut ribbon; a torque motor coil located in a permanent magnetic field and also mounted to the taut ribbon; sample suspension fixtures; a beam position sensor system; and controls, circuitry and indicators.
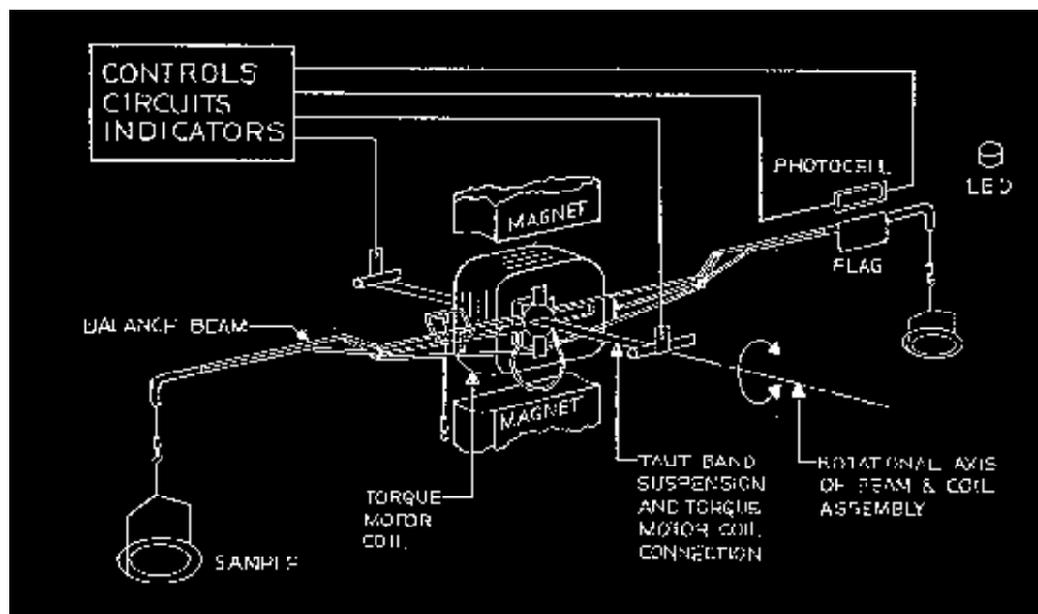
Weights to be measured are applied to the sample (left) side of the beam which produces a force about the axis of rotation. An electric current flowing in the torque motor also produces a force about the same axis which is equal and opposite to the force from the beam, if the beam is at the beam reference position. This reference position is

detected by the beam position sensing system. A greater force on the beam will require a greater opposite force from the torque motor in order to keep the beam at its reference position. Therefore, the current necessary to produce the required torque motor force is a direct measure of the force on the beam. The process of calibration allows this current to be measured in units of weight (grams).

There are three fine metal loops attached to the balance beam. Samples and counterweights are suspended from these by means of stirrups and pans. The sample suspensions are designated as A, B, and TARE loops. The A loop is on the left side of the beam and has a maximum capacity of 1.5g. The TARE loop is on the right side of the beam and has a maximum capacity of 1.5g. The B loop is 1/5 the distance from the pivot point as compared to loop A. It is for sample loads up to 3.5g. On the ship, the A loop position (range; 250mg -1µg) is used the most.
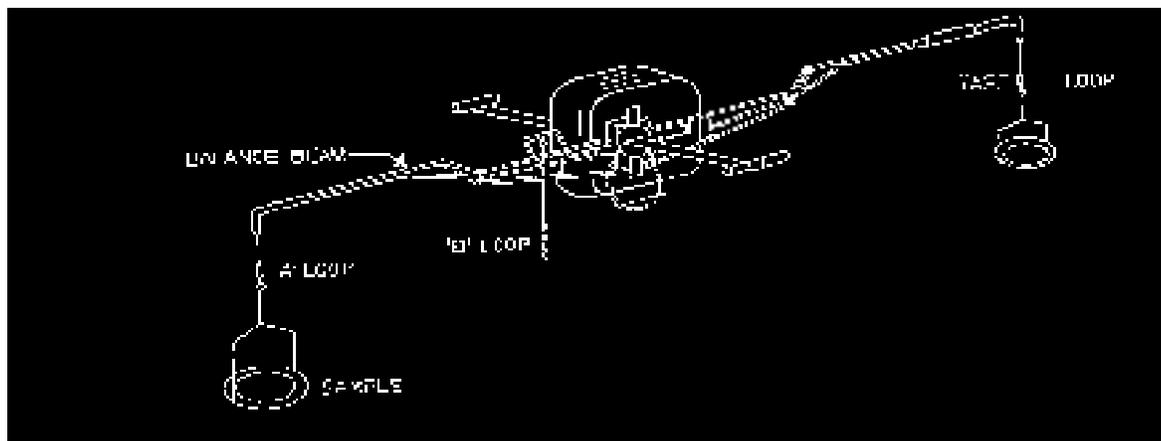
The operation of the balance is controlled from the front panel using the following controls:
The RANGE button allows to select one of the weighing ranges of the balance. Each press of the button will advance the selection to the next range;

Pushing the button labeled TARE will bring the display to zero if the sample is within the selected Weighing Range of the balance;

The CAL button will automatically calibrate the selected range when the appropriate calibrating weight is placed on the pan. A 200mg weight is used to calibrate the A loop position and a 1000mg weight is used to calibrate the B position;

The value of the weight of the sample on the sample pan is shown in milligrams on the Weight Display. Since the balance will give either positive or negative weights within the selected range, the Weight Display shows a "plus" or "minus" sign;

A PAN BRAKE button activates the Pan Brake Platform. This feature will steady the pan and allow the placing and removal of samples without creation excessive pan swing.

To calibrate the C-30/31 balance, proceed as follows:

Place a pan on the A loop and a similar one on the TARE loop and then press TARE to bring the Weight Display to zero;

Place a 200mg calibration weight on the sample pan;

When the Weight Display has stabilized, push the CAL button once;

Remove the calibration weight. You may now tare and make normal weighings.

To weigh a sample, first place a sample boat on the sample pan and a counterweight, close to the weight of the boat (usually a piece of weighing paper), on the tare pan, and press the Tare button. Place the sample in the boat, its weight will show on the Weight Display. Remove and add sample till satisfied with the weight.
Precautions:

Always use forceps to handle pans, stirrups and weights. To avoid contamination do not handle them with fingers. Dip or swab the accessories in ammonia to clean them when necessary. Do not grasp the stirrups with the forceps. Lift them from below the yoke. It is easy to break the hangdowns and beam if excessive force is exerted on the stirrups and loops. If damaged, the balance must be returned to Cahn Instruments for repair.

The weighing chamber window must be closed when zeroing, calibrating or weighing. When the balance is not in use, the chamber should be closed to reduce the possibility of contamination or damage.

B. 2. Using the Balance Program

The LabView Coulometer/Balance program automates the collection of data from the Coulometer and the associated Cahn balance. The program runs simultaneously on two Macintosh computers. MC67 runs the Coulometer (Coulometer program) and MC71, the Cahn balance (Balance program). The Macs continuously communicate with each other through the network. This gives the operator the ability, when preparing to run a sample on the Coulometer, to electronically call up the ID and weight of that sample from the Balance program.

The Cahn Balance program works very much like the Scientech Balance program. The algorithms are all the same, the differences reside mainly in the layout of the VI windows and the calibration and weighing procedures.

B. 2. 1 Calibrating the Balance

Two calibrations (0 - 10 mg for Coulometer/CHNS and 0 - 100 mg for Rock Eval) are usually performed with the Balance program during port call. One need first to calibrate the Cahn balance itself (Section B. 1) before performing a calibration using the software.

To launch the Balance program on MC 71, click on the LabView Balance icon. Check that MC67 is network connected and is shown on the desktop. If MC67 is not on the desktop, select MC67 from the Chooser on MC71. The Data Path needs to be set up in the program as "MC67:Coulometrics".

Once the Balance.vi is loaded, the control window appears as displayed on the next page.
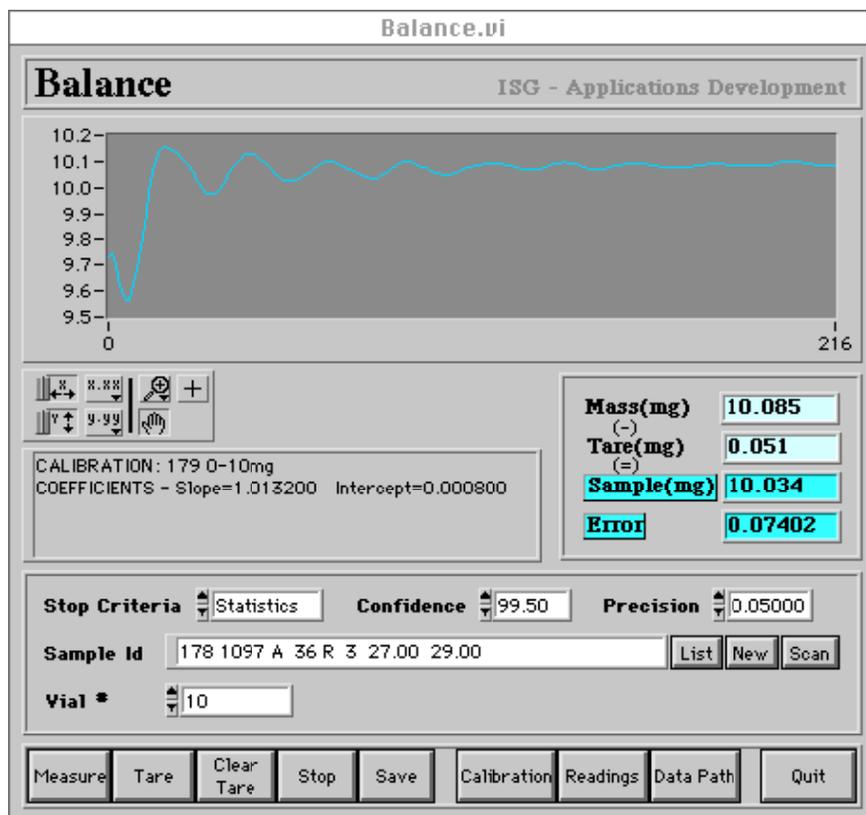
To calibrate the Cahn balance using the software, first set the Stop Criteria to "By Statistics"; the Confidence Interval to 99.5% and Precision to 0.01%; and Clear the Tare. Then click on the Calibration button to open the Select_Calibration.vi window:

Select New to open the Balance_Calibration.vi window:
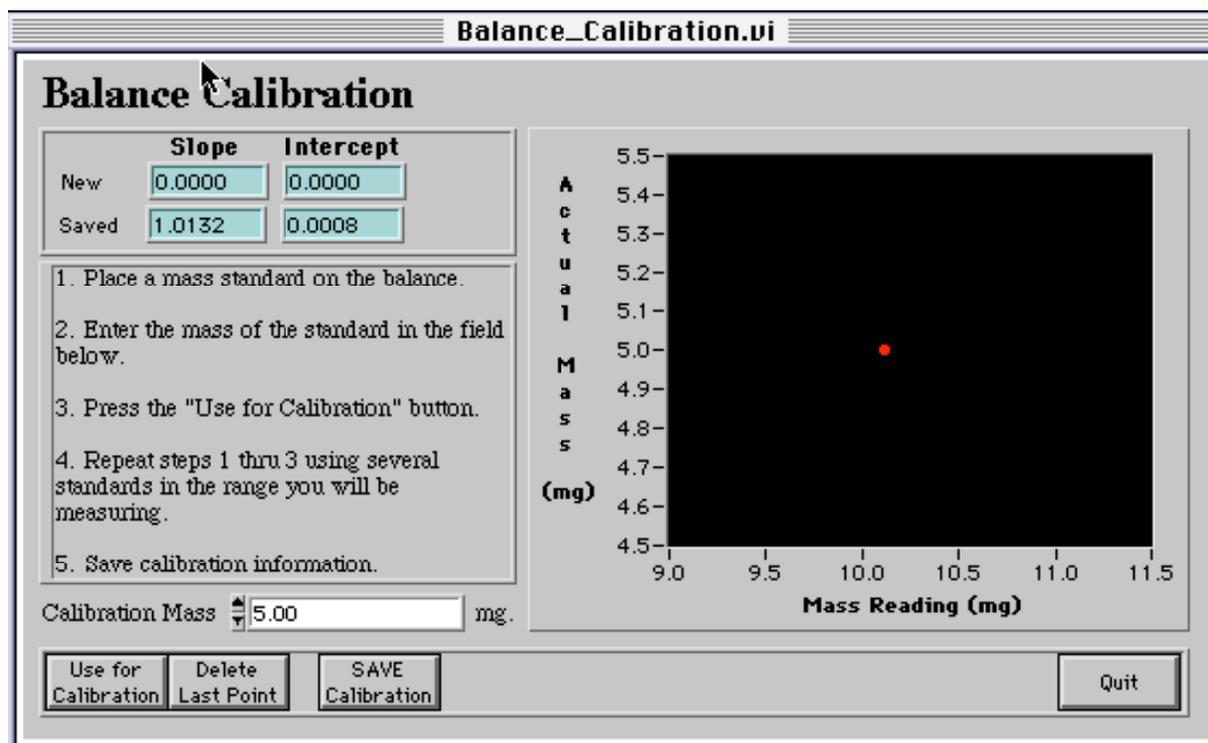
Follow the on-screen instructions:

 Start with no weight on the sample pan and enter 0 as the mass;

 Click Use for Calibration;

 When the count is finished, proceed with the remaining calibration weights of 1, 2, 5, 7, and 10mg (or 0, 5, 10, 20, 50, and 100mg for a Rock Eval calibration);

 After the last calibration level is done, Save and name the calibration.

The calibration factors in use will be displayed at all time above the Measure, Tare, etc. buttons of the control window. To change calibration during the Leg, click on the Calibration button and select the calibration you wish to use by simply clicking on its file name.

B. 2. 2 Weighing Samples

Weighing can be done by using statistical analysis or fixed counts. The Stop Criteria control is used to toggle between Statistics (Confidence Interval and Precision can be set as desired) and Counts. The Tare button is used to tare empty sample boats and the Measure button is used to weigh the boat plus sample. The difference between the two gives the actual sample weight displayed in the Sample (mg) indicator. The Readings button can be depressed to see the last ten measurements. Clicking on any of these measurements will bring the selected reading and its associated values back to the weighing screen.

If the samples IDs have been downloaded directly from the database, scanned, or manually entered in either the Coulometer or Balance program, they may be accessed from the Balance.vi window on MC71 by selecting List.

The Sample_List_View.vi window is displayed below:

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┌─────────────────────────────────────────────────────────────────────┐
│  ═══════════════════  Sample_List_View.vi  ═══════════════════        │
├─────────────────────────────────────────────────────────────────────┤
│                                                                       │
│  Leg  Site  H    Cor  T  Sct    Interval    Vial    Mass              │
│  ┌──────────────┐┌──────────────────────────────────────────────┐    │
│  │ 178  1101  A ││ 46  R  1  117.00  118.00   15    22.43     1│↑│    │
│  │ 178  1099  B ││ 45  R  1   41.00   42.00   13    12.90     1│ │    │
│  │ 178  1099  A ││ 44  R  1   37.00   38.00   16    21.13     1│ │    │
│  │ 178  1098  C ││ 42  R  2   84.00   85.00   14    15.80     1│ │    │
│  │ 178  1097  A ││ 40  R  2   40.00   41.00   12    14.70     1│ │    │
│  │ 178  1096  C ││ 39  R  2   26.00   27.00   59    17.95     1│ │    │
│  │ 178  1096  B ││ 37  R  1   79.00   80.00   11    17.82     1│ │    │
│  │ 178  1096  A ││ 36  R  3   27.00   29.00   10    10.03      │ │    │
│  │ 178  1095  D ││ 36  R  1    9.00   11.00    7    11.40      │ │    │
│  │ 178  1095  B ││ 35  R  1   35.00   36.00    5    12.28     1│ │    │
│  │ 178  1095  A ││ 34  R  1   58.00   59.00    3     8.14      │ │    │
│  │ 178  0000  0 ││ 33  R  1   24.00   25.00    6    18.25      │ │    │
│  │ 177  0000  0 ││ 28  R  1   62.00   63.00    9    20.11     1│ │    │
│  │              ││ 27  R  1   28.00   29.00    8     9.81      │ │    │
│  │              ││ 25  R  1   15.00   16.00   13    16.90     │↓│    │
│  └──────────────┘└──────────────────────────────────────────────┘    │
│  ┌────────┐  ┌────────┐                                               │
│  │ Select │  │  Quit  │                                               │
│  └────────┘  └────────┘                                               │
│                                                                       │
└─────────────────────────────────────────────────────────────────────┘
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Selecting a specific Leg - Site - Hole will load all related sample IDs. Clicking on the Select button or double-clicking on the desired sample will load that sample ID in the Sample ID field as shown on the balance.vi shown section B.2.1.

If the sample to be weighed is a CARB sample, the operator must first enter a Coulometer vial number in the Vial field of the Balance window and, then, weigh the sample. When the weighing process is finished, the operator is prompted to save the reading. Clicking on the notifier will transfer the sample ID, and associated weight and vial number, back to the List window. This List is also accessible from the Coulometer Carbon window on MC 67. Typing the vial number in the Sample ID field and hitting the tab key will load the sample ID info and associated weight.

If the sample to be weighed is a $CaCO_3$ standard, or a sample to be analyzed on the CHNS or Rock Eval, the vial number is kept at 0. For these applications, the sample ID can also remain blank. The operator simply uses the Tare and Measure buttons to weigh the sample. Clicking on the "Measurement Completed" notifier concludes the weighing process. The weight value must be written down on a piece of paper before proceeding to weigh other samples.

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