Woods Hole Oceanographic Institution, USA
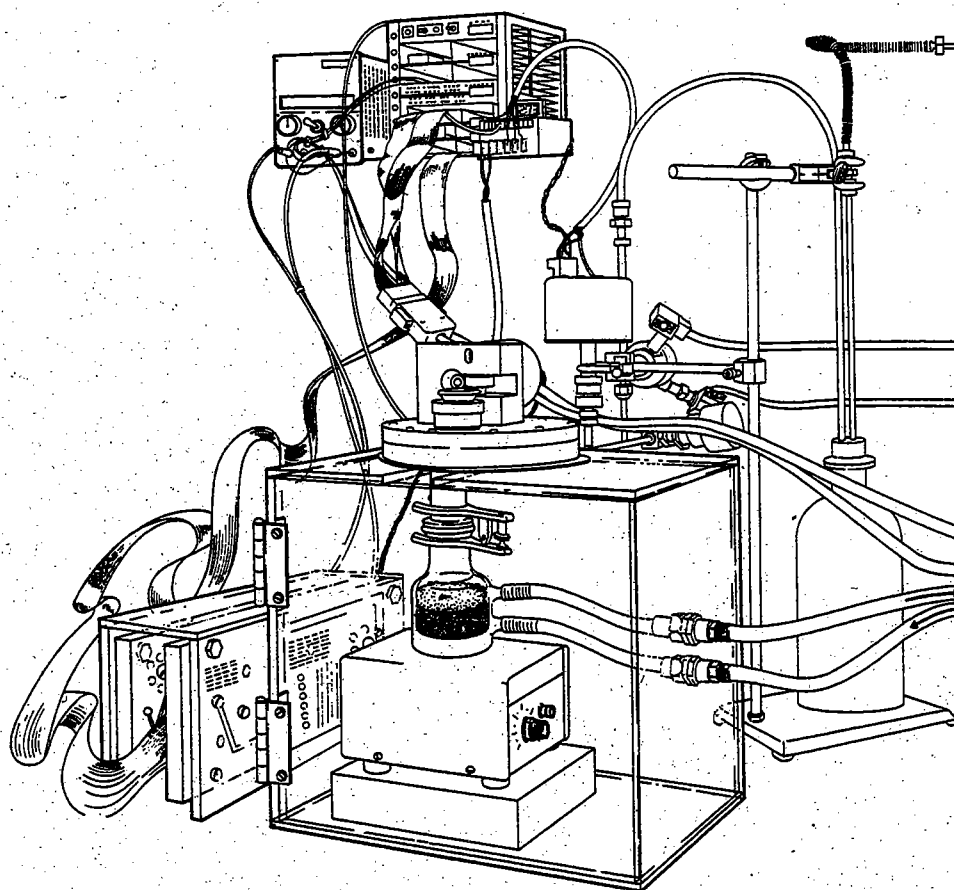
WHOI-90-03

Automated System to Measure the Carbonate Concentration of Sediments

by

Dorinda R. Ostermann, Darrell Karbott and W.B. Curry

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Technical Report

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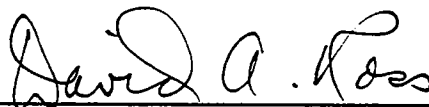
Technical Report

Approved for Distribution:

David A. Ross, Chairman
Department of Geology & Geophysics

Contents

1 INTRODUCTION

We have developed a computer controlled system to measure the calcium carbonate content of sediment samples. A menu driven program controls the analysis of each sample. The system first communicates with a Mettler digital balance to record the weights of the 40 samples which must be loaded into each run. The sample boats are next loaded into the sample carousel which is then sealed from the atmosphere. The system is first pumped down to a vacuum of 0.04 torr. The valve to the pump closes and the stepping motor turns the carousel, moving a sample boat over the delivery slot and dropping the sample into 80°C 100% phosphoric acid under vigorous spinning action. During the reaction, carbonate is evolved into H_2O and CO_2 and the resulting pressure change within the closed system is measured by a pressure transducer and recorded into memory next to the sample identification and sample weight. The system is pumped once again to 0.04 torr and the process continues until all 40 samples have been analyzed. The data can then be uploaded and converted to percent carbonate values using a regression line produced from multiple analyses of varying weights of a 100% carbonate standard. Precision of the system, based upon 120 replicate analysis ranges from 0.49% to 0.88%.

2 HARDWARE

We set out to develop an easy-to-use automated carbonate system which was inexpensive and microprocessor controlled. We based the system on the STD bus because of its modular control-oriented system design and because of the wide availability of plugin cards for specific control applications. With a minimal amount of rejumpering (see Appendix 1), the boards were snapped into the card cage, and connected to the balance, terminal, stepping motor and pressure transducer. The automated carbonate system (ACS) consists of the following major components, and is diagrammed in Figure 1.

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2.1 Microprocessor

The **VERSALOGIC©** VL-7806 Z80 microprocessor is used in this application. The STD bus compatible board features a flexible memory mapping option which allows a mixture of memory devices to be used in the system. In this system we use a 64K memory expansion board in addition to the memory contained on the Z80 board. Appendix 2 details the memory map of the Z80 processor as configured in this system. The Z80 board also features two RS232 serial I/O ports. We use port B to communicate with the **METTLER** AE100 balance through a bi-directional interface. Port A is connected to a **NEC** APC II computer which is used as a terminal for the Z80 processor and to upload the data from the carbonate analyzer. A *C4 BASIC©* 8K ROM is plugged into socket M0 on the VL-7806 processor card. The *C4 BASIC* language which runs the automation is oriented to process control tasks and includes special statememts for I/O device interface. It is designed to run programs directly from ROM storage and has an operating system, *NOVOS*, which utilizes RAM nonvolatile memory. This allows development, testing, backup, copying and transferring of *C4 BASIC* programs in a totally diskless system.

2.2 Memory Expansion

The large size of the automation program made it necessary to expand the memory of the system using a **VERSALOGIC** VL-7709 memory card. The board provides 16-bit addressing for use with the Z80 processor. The system as configured uses a total of 48K of RAM, three 8K chips on the Z80 board and three 8K chips on the memory board. This 48K of memory may be allocated for storage area and development RAM in any proportion using the system SETUP command (see Appendix 6). However, the sum of the number of Kbytes for storage and development RAM *must not* exceed 47K. This reserves 1K at the top of memory (&DC01-&DCFF) for critical machine language subroutines.

2.3 Relay Output

Solenoid actuators for the pneumatic valves are controlled by a **VERSALOGIC IPI-2** eight channel relay output card. The individual relays are powered by a 28 VDC source and the channels we use are activated through address &96. Circuitry is provided to drive eight LED indicators at the edge of the board which display the current on or off state of each channel.

2.4 Pressure Transducer

A 0-100 torr **BARATRON**© absolute pressure transducer is used as the system pressure sensor. We chose this particular transducer because it is capable of withstanding high over-pressure conditions and it is highly resistant to corrosive environments. We connected the transducer and the analog input board through a 10 K reostat (see Appendix 3A). The reostat reduced the normal 10 volt output range of the transducer to 5 volts, doubling the resolution of the transducer. Careful attention was paid to limiting the internal volume of the reaction vessel and the lengths and internal diameters of the stainless steel tubing, minimizing the overall volume of the system and maximizing the pressure transducer output (see Appendix 3B). A microvoltmeter connected to the transducer externally monitors the pressure within the system.

2.5 Analog Input

The **VERSALOGIC STD AIN-1** is an eight channel integrating analog input board compatible with the STD bus. This board uses an integrating conversion technique to perform the analog to digital conversion. The integrating method measures the actual input signal during the conversion giving a much higher accuracy in changing or moving input signals such as found in our system. The integrating conversion is also very noise tolerant. These advantages are a trade off to slower conversion times but because our system is only monitoring one signal during a 5 to 20 minute reaction time, the slower conversion time does not limit this system's efficiency. The analog input board is jumpered to a 5 volt input range. This lower voltage range provides twice the resolution for each measurment (2.5 mv versus

5 mv in the 10 mv range) without affecting stability or conversion time. The one part in 4000 (12 bit) resolution provides a digital pressure unit equivalent to .025 torr. We chose to use the differential input mode for signals that are not referenced to a ground point but are simply a voltage difference between two input wires. This mode is especially beneficial in electrically noisy environments. We communicate with this board via address &70.

2.6 Stepping Motor Driver/Controller

The **Matrix Dual Stepping Controller** (DSC) and Unipolar Stepper Driver (USD) provide microprocessor control of unipolar stepping motors. A 26-position ribbon cable connects the DSC in the card cage to the USD. The USD converts the DSC output signals to the voltage and current levels required by the stepping motor. The USD receives logic power directly from the DSC which is initialized upon power-up via a software status/command register. These commands include direction of rotation, single versus continuous step mode and the motor speed. The 4-phase stepping motor used in this application is powered by an external 24 VDC power supply connected directly to the USD.

2.7 Sample Carousel

A 'lazy susan' designed by R.G. Fairbanks served as the basis for our carousel assembly design. It was modified to our specific application and machined out of 316 stainless steel to resist acid corrosion. A four phase stepping motor is attached to the top of the carousel assembly. The motor turns a set of reducing gears connected to the carousel shaft which is sealed from the atmosphere with two 'O' rings, allowing the shaft to rotate the sample dispenser under vacuum. The carousel makes one full revolution every 2000 steps of the stepping motor. The sample carousel holds 40 samples which may be loaded through the top assembly by removing an **ULTRA-TORR**© fitting. The top and bottom carousel assemblies are secured together with allen cap screws and are sealed from the atmosphere with an 'O' ring which fits into a groove in the bottom assembly.

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2.8 Reaction Vessel

A glass reaction vessel (see Appendix 3C) is attached to the bottom of the sample carousel assembly with a pinch clamp and is sealed from the atmosphere with an 'O' ring. The 20 ml phosphoric acid (see Appendix 4) within the vessel is maintained at 80°C by a circulating water bath and a strong spinning action of a magnetic stir bar.

2.9 Vacuum Line

Any pump which can pump down to a vacuum of 10^{-4} torr is suitable for use with this system. Large internal diameter 0.25 inch copper refridgeration tubing was used between the valves and the vacuum pump to provide a more efficient initial pump down. Flexible stainless steel tubing was used between the vacuum pump and the oil trap to lessen vibration through the system. **ULTRA-TORR** fittings were used in all applications except for the attachment of the pneumatic valves to the tubing where we used swagelok fittings provided by the manufacturer. Appendix 3B details the vacuum line specifics as used in this system.

3 SOFTWARE

A complete listing of one **MICROSOFT** Fortran and four *C4 BASIC* programs can be found in Appendix 5. Each program will be individually described below.

3.1 Clear.bas

Clear.bas is a *C4 BASIC* program which clears the existing storage area so that *NOVOS* can be used to reinitialize the system. Download the program and save it in an empty slot of the directory and run it. The program will ask the user to input the starting address of the storage area. If you are unsure what the storage address is, type *NOVOS*, and the address of the storage area will be printed on the screen. In the present configuration, input '&7400'. (Remember to enter the hex symbol '&' before the address number) The system will either lock up and you will have no keyboard control or 'Enter program number' will be printed.

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In either case, power the system down for 15 seconds and reset the button on the power supply card. Now the system is ready to be reconfigured following the procedure outlined in Appendix 6.

3.2 Loader.bas

Loader.bas is a *C4 BASIC* program which loads into memory, two Z80 machine language subroutines which perform I/O operations not provided for in *C4 BASIC*. The first, RDKEY, reads a single key stroke from the NEC console, returning the ASCII code of the key pressed via the *C4 BASIC* function USR(X). The second, METIO, reads a string of up to 16 characters from the METTLER digital balance through serial port B on the Z80 board, storing their ASCII codes in a buffer from which they may be read by means of the *C4 BASIC* function PEEK. Installing these two subroutines is complicated by the fact that *C4 BASIC* has no built in commands to store machine language routines in its file system or to load them into memory. *Loader.bas* was therefore written to use the *C4 BASIC* function, POKE, to poke them into memory one byte at a time starting at &DC01. Although the subroutines are less than 100 bytes long, it was necessary to reserve a full 1K of memory for the routines because that is the minimum that *C4 BASIC* configuration procedure allows. The system has already been configured to protect the addresses &DC01-&DCFF from *C4 BASIC*. However, if the system is ever reconfigured, *it is essential that these addresses be reserved.*

3.3 Getdata.bas

Getdata.bas is a *C4 BASIC* program used to make consequetive measurements of reaction time versus digital pressure on a single sample. It is useful for leak testing of the system as well as determining the necessary reaction time needed for the complete evolution of CO_2 gas from a sample. Download the program into an empty directory location. Next load a sample boat into whichever load slot is to the right of the delivery slot, close the carousel, and run the program. The user will be asked to input the numbers of paired data to be collected (80 maximum). The user will next be asked to input the interval in seconds between measurments.

Decide the time interval over which you want to collect data and plan the number of data points collected and seconds between each collection accordingly. At the program end hit <P> to print the data to the screen or <Q> to quit. If you wish to copy the data to a disk, you will need to use a screen capture program. We use *ASYNC* as described on page 11.

3.4 Percent.for

Percent.for is a **MICROSOFT** Fortran program which determines the percent carbonate for samples run on the Automated Carbonate System. At least two runs of 100% carbonate standards are first run to produce a regression line of slope and intercept for each batch of acid (see **OPERATIONS**, p. 14). The program can then be recompiled with the new slope and intercept data or interactively on the screen each time the program is used. The user will first be asked for the input filename. (By convention we always name the raw data from the carbonate system with 'filename.crb'). The slope and intercept will then be listed. If you wish to use the default values hit <RET>, otherwise insert the values you wish to use. Finally, the user will be asked to name the output file. (By convention we always name the converted data file 'samefilename.pct'). The program will crash if 'samefilename.pct' already exists. If it does, simply delete it and rerun the program.

3.5 Animat.bas

Animat.bas is the *C4 BASIC* program which controls the Automated Carbonate System. The program is almost exculsively menu driven through two menus: (1) The Main Menu and (2) Tweak the System Menu. Each of the menus is detailed below including a few 'hidden' functions which may be called at any time.

3.5.1 The Main Menu

The Main Menu
<W> to weigh the samples
<L> to load the sample boats
<R> to run samples
<P> to print the data
<I> to reinitialize
<T> to tweak the system
<Q> to quit

<W> to weigh the samples

This option is used to weigh out the samples into the sample boats and to enter their corresponding depth values. It has several 'hidden' functions which can be accessed through the use of control characters. Typing '^ Z' at any prompt returns the user to the main menu. If the balance drifts after it has been tared, it can be retared by typing '^ T' at the 'Add the sample to the boat and hit any key when it is ready to be weighed=>' prompt. If the balance drifts after the sample has been weighed it can be reweighed by entering '^ W' at the 'Do you wish to redo this sample? (Y/N)' prompt. Occassionally the balance will refuse to tare or weigh on the first sample only. This problem seems to occur when the balance has been turned off or has been used between carbonate runs. Abort to the main menu by typing 'A'. Turn the balance off and on several times and then reboot the system by running the Loader and Automated Carbonate System programs. Finally, reinitialize the memory.

<L> to load samples

This option prompts the user to load the sample boats one at a time into their proper holes in the lazy susan's carousel. The sample carousel *must* be correctly aligned at the beginning of the load sequence for proper system operation (see Appendix 7). If the lazy susan jams while loading or is at the wrong load slot:

1. Exit to the main menu by typing '^ Z'

2. Call up the 'Tweak the system menu' by typing 'T'

3. Select the 'Home the lazy susan' option by typing 'H'

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4. Home the lazy susan so that the hole with the same number as the next sample you wish to load in in the load slot position. (see Appendix 7)

5. Exit to the 'Tweak the system menu' by typing 'E'

6. Exit to the main menu by typing 'E'

7. Reenter the Load option by typing 'L' An error message will be printed and this option will refuse to execute if (a) all 40 samples have not been weighed out before it is called or (b) the samples have already been run.

<R> *to run the samples*

This option reacts the individual samples and stores the pressure values in the systems internal file structure so that they may be uploaded using the '<P> print the data' option at the end of each run. The user is prompted to select one of two reaction times for analysis of individual samples based on the expected carbonate content. If the lazy susan jams while the 'Run the samples' option is executing, an error message will appear on the screen.

1. Hit the Escape key, '<Esc>'. This breaks the Animating Element. A system prompt, '*', should appear.

2. Type 'run 1' to reexecute the ACS program. The prompt 'Skip software initialization? (Y/N)', should appear.

3. Type 'Y' to skip the initialization. This preserves the systems internal file structure.

4. Type 'N' at the 'Is the lazy susan homed to slot #1? (Y/N)', prompt. The Homer menu will be displayed.

5. Home the lazy susan to correct the jam, preserving the jammed sample if possible.

6. Exit to the 'Tweak the system...' menu by typing 'E'.

7. At the prompt, enter the number of the hole in the carousel which is over the load slot, *not* the number of the next sample to be run. (see Appendix 7)

8. Type 'R' at the 'Tweak the system...' menu to resume running.

Do not run any other program or call *NOVOS* between the time you break the program

by typing 'Esc' and the time you reexecute the ACS program. The data in the system's file structure (i.e. the weights of all the samples and the pressure values of the samples which have already been run) will be destroyed. The 'R' option will not execute until all the samples have been weighed and loaded.

<P> *print the data*

This option is used to print out the system's data file so that it may be uploaded to a floppy disk using *ASYNC*'s 'Get a file' function.

1. After the 40 samples has been been run and the main menu is listed, type '^ VF' to call *ASYNC*'s 'Specify file name for transfer' function. The *ASYNC* prompt 'File specification:' will appear.

2. Enter the drive and filename the data is to be saved under and hit '<return>'.

3. Type '^ VG' to call *ASYNC*'s 'Get a file' function. The message 'Port-> filename/File open, rdy to rcv' will appear on the top left corner of the screen.

4. Type 'P'. The data will scroll on the terminal one line at a time.

5. When all 40 lines of data have been printed on the terminal screen, type '^ Z'. This closes the file and the message '++File received++' will appear after the main menu is printed.

<I> *to reinitialize*

This option is used to reinitialize the system so that another batch of 40 samples can be run. The execution of this option destroys all the data in memory.

<Q> *to quit*

This option is used to exit the ACS program and enter *C4 BASIC*. *Do not* execute this option unless the data has been already uploaded. It is almost always possible to reenter the ACS program program without destroying the data by typing 'Y' at the 'Skip software initialization? (Y/N) ' prompt, but it is not worth the risk. If you must, see steps #2 and #3 for unjamming under the 'Run the samples' option.

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3.5.2 Tweak the System Menu

Tweak the System Menu
<A> for atmosphere
<V> for vacuum
<W> to reweigh a sample
<R> to continue after a jam up
<C> to check the pressure in the vessel
<E> to exit to the main menu
<H> to home the lazy susan

<A> *for atmosphere*

This option opens the vessel to the atmosphere by closing the valve to the pump and opening the valve to the atmosphere.

<V> *for vacuum*

This option pumps the reaction vessel down by closing the valve to the atmosphere and opening the valve to the pump.

<W> *to reweigh a sample*

This option allows the user to reweigh a sample after all samples have been weighed. The user will be prompted for the number of the sample to be reweighed and then the normal weighing protocol is followed.

<R> *to continue after a jam up*

This option is used to continue running after the normal 'run samples' has been interrupted. Do not call this option until the lazy susan has been homed so that the carousel will drop the next desired sample into the reaction vessel. See the 'Home the lazy susan' menu above.

<C> *to check the pressure in the vessel*

This function allows the user to leak test the vessel over a selected period of time. The option closes both valves so remember to reopen the valve to the pump if you want to continue pumping on the acid after checking the pressure. An error message will be printed if this option is executed while the vessel is open to the atmosphere.

<E> *to exit to the main menu*

This option returns the user to the main menu.

<H> *to home the lazy susan*

This option calls up another menu of functions which are used to home the lazy susan.

3.5.3   Homer Menu

Homer Menu
<Right arrow> = counter-clockwise
<Left arrow> = clockwise
<Up arrow> = continuous step mode
<Down arrow> = single step mode
<S> = Smart Home Option
<E> = Exit

This menu allows the user to manipulate the lazy susan's carousel from the keyboard. The '<Right arrow>' and '<Left arrow>' keys control the direction of rotation of the lazy susan. The '<Up arrow>' and '<Down arrow>' keys switch between single step mode and continuous step mode. In single step mode, the lazy susan moves a single step in the direction of the pressed direction key. In continuous step mode, the lazy susan moves in the direction of the last pressed direction key. Pressing any key except for '<Up arrow>', '<Right arrow>' or '<Left arrow>' will cause the lazy susan to stop in continuous step mode. The carousel *must* be aligned to load slot #1 at the beginning of the load sequence (see Appendix 7).

<S> *Smart Home Option*

The smart home option asks the user for the number of the hole positioned over the load slot and automatically homes the lazy susan to load slot number 1. In order to use the smart home option, you must first use the the arrow keys to home the lazy susan to the nearest load slot. *Do not* use this option if the boats have already been loaded into

the lazy susan. This option chooses the direction of rotation based upon the shortest distance to the load slot and may dump loaded boats into the acid in the process of homing the carousel.

<E> *Exit*

This option prompts the user to enter the load slot which the lazy susan has been homed to and then returns to the 'Tweak the system...' menu. This program has no encoding mechanism and thus has no way of knowing where the carousel is other than asking the user. The program does not know which load slots have boats in them and *cannot keep you from dumping boats into the acid by accident*!!

4 OPERATIONS

Once the hardware and software have been installed and are functioning correctly, the phosphoric acid and reaction vessel can be calibrated by running at least two standard runs of 100% calcium carbonate. Data from two carbonate standard runs are shown in Figure 2. The slope and intercept of the regression line produced from such standard runs are used in *percent.for*, the fortran program which converts raw data produced on the ACS to percent carbonate data.

We have found that a constant volume of acid delivered to the reaction vessel is critical to produce consistent, accurate results. Room temperature 100% phosphoric acid is quite viscous. We found that the repipette manufactured by **LABINDUSTRIES**© consistently delivers accurate volumes. (A complete listing of components for the ACS can be found in Appendix 8). We adjust the 20 ml repipet to deliver two 10 ml aliquots of acid because in the process of drawing up 20 ml, bubbles always form at the pipette tip causing inaccurate volumes. A glass-covered spin bar is added to the acid-filled reaction vessel. The reaction vessel should now be attached to the carousel assembly, heated to 80°C, and put under vacuum to begin the outgassing process. Under atmospheric conditions, the microvoltmeter

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should read an over-pressure voltage of ±13. Once pumpdown begins, the voltage will quickly drop to below 1 volt if no leaks are present. Within 2 minutes of pumping under vigorous spinning action, the voltage should drop below 0.10 volts. The user is ready to weigh out the 100% dried reagent grade calcium carbonate standard samples into the sample boats (see Appendix 3D for sample boat specifications).

The first boat to be weighed should be listed as 'sample 1'. If the first sample listed is 'sample 0', exit the weighing procedure and reinitialize the buffer memory by hitting the <I> under 'The Main Menu'. Then proceed with the weighing process. When producing a standard carbonate run, weigh out a range of weights from 1 mg to 20 mg. The first sample should always be large, to fully saturate the acid with CO_2 (the percent carbonate value of the first sample is always 10-15% low and is deleted). Once all 40 boats have been filled, use a finnpipette to deliver $5\mu l$ of reagent grade methanol to each sample boat. We have found that on the initial pumpdown from atmosphere to vacuum, material can be lost from the boats nearest the reaction vessel delivery slot (boats 35-5). When dried, the methanol produces a thin crust which adheres the sediment particles together. Place the 40 boats into an oven to dry. The samples are dry when they are no longer shiny (5-10 minutes at 60°C). The boats can now be loaded into the sample carousel, if load slot 1 is in the correct position (see Appendix 7).

Once the **ULTRA-TORR** fitting on the carousel assembly is secure, the system is ready to run. A choice of two reaction times will appear on the screen. We have found that our initial 5 minute reaction time was not long enough to fully evolve the CO_2 from sediment samples having low carbonate contents. To determine the optimal reaction time for sediments of varying carbonate content, we conducted an experiment using *Getdata.bas*. The results of this experiment are shown in Figure 3. In general, the evolution of carbonate to CO_2 gas is complete within one minute. However, in samples with less than 25% carbonate content, it is apparent that a much longer reaction time is necessary. Data collected from a second experiment on samples with less than 40% carbonate over a 45 minute reaction time are shown in Figure 4. These results show that a 20 minute reaction time is necessary for sediment samples with less than 25% carbonate. A small but gradual increase in percent

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carbonate after 20 minutes is water vapor outgassing from the acid. The precision of replicate analyses of samples run on the ACS with high, medium and low concentrations of carbonate vary between 0.49% and 0.88% as shown in Figure 5.

As the run begins, the valve to the pump opens and the pressure in the system quickly drops to .12 volts (which corresponds to 0.04 torr) and the pump valve closes. A 'stable pressure' algorithm collects a pressure value every six seconds and waits for ten consecutive values which do not change by more than .01 volts. For the first few samples it may take the computer longer than a minute to accept a background stable pressure value. By the fifth sample, however, only one minute is necessary before a stable background pressure value is reached and the next sample boat is dropped into the acid. Based upon the reaction time chosen, the system will wait for that amount of time before beginning the 'stable pressure' algorithm to collect the final pressure value. The initial background pressure is subtracted from the final stable pressure to produce the digital pressure listed in the data table.

If a sample happens to be reacting at the end of the chosen reaction time, the system will wait for the reaction to be complete before a stable pressure value is accepted and the next sample is dropped. If there is a leak in the system the pressure will never stabalize and the run will not continue. When leak is so bad that atmospheric pressure is reached, an error message will appear on the screen and the run will be terminated.

At the end of each run, the main menu will be printed on the screen. We recommend that the data from each complete run be uploaded onto a floppy disk as soon as possible. At present, the system has no battery backup protection and when the power fails, the data is lost. You can expect a typical 'high carbonate' run to take 4.5 hours while a 'low carbonate' run will take at least 14.5 hours. For this reason we run high carbonate samples during the day and the low carbonate samples overnight for maximum data output. *Do not* increase the sample weights of low carbonate samples in order to produce a greater pressure signal. The clays in low carbonate samples will turn the acid into a thick mass making the run invalid. We keep the sediment sample weight in each boat within the range of 15 to 20 mg of material for best results. If problems are encountered, refer to the 'Trouble Shooting guide in Appendix 9. For a condensed 'System Startup' procedure, see Appendix 10.

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Coplen, T. B., Kendall, C., and Hopple, J., 1983, Comparison of stable isotopic reference samples: Nature, v. 302, p. 236–238.

Curry, W.B., and Lohmann, G.P., 1985, Carbon deposition rates and deep water residence time in the equatorial Atlantic Ocean throughout the last 160,000 years, *in* Sundquist, E. T., and W. S. Broecker, eds., The carbonate cycle and atmospheric CO_2: natural variations, Archean to present: American Geophysical Union Monograph 32, p. 285-301.

Acknowledgements

Figure 1. Automated Carbonate System Schematic.

    A. 28 volt DC power supply (for stepping motor & valve solenoids)

    B. Card cage including the following boards:

        1. power supply (5 & 12 volt DC)

        2. Z80 computer

        3. Analog input

        4. Memory expansion

        5. Dual stepper controller

        6. Relay output

    C. Microvoltmeter (to display transducer pressure)

    D. 15 volt DC power supply (for pressure transducer)

    E. Pressure transducer

    F. Pneumatic valves with attached solenoid actuator

    G. Stepping motor

    H. Lazy susan sample carousel

    I. Water jacketed reaction vessel

    J. Spin bar mixer

    K. Unipolar stepper driver

    L. Vacuum pump oil trap

Figure 1. Automated Carbonate System Schematic.

Standards @ 80 °C

slope = .150418E-04
intercept = -.305103E-04
r = 0.999547

■ 25 May 1989
○ 18 May 1989

Weight CaCO$_3$ (g)

Digital Pressure (1 unit =.025 Torr)
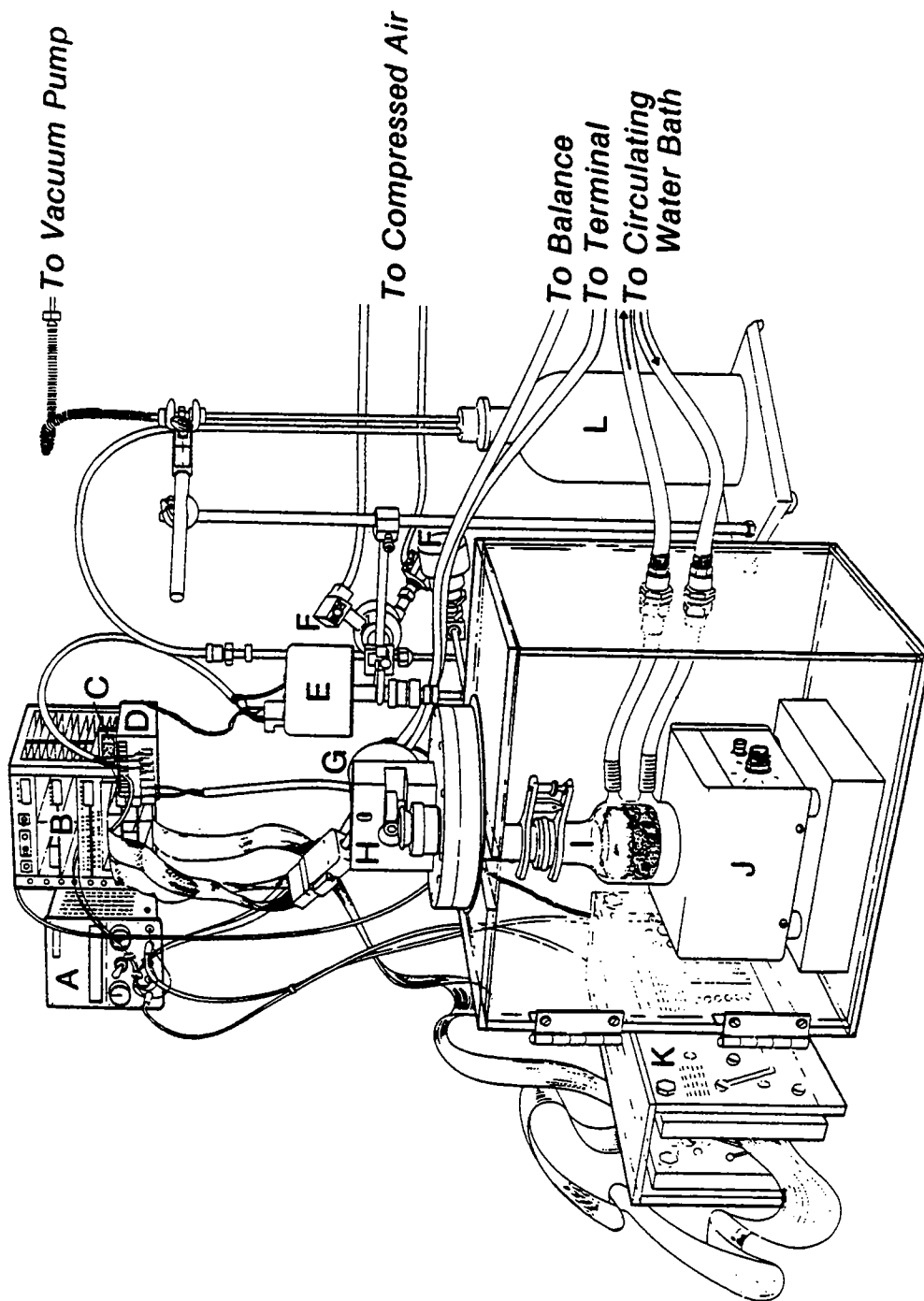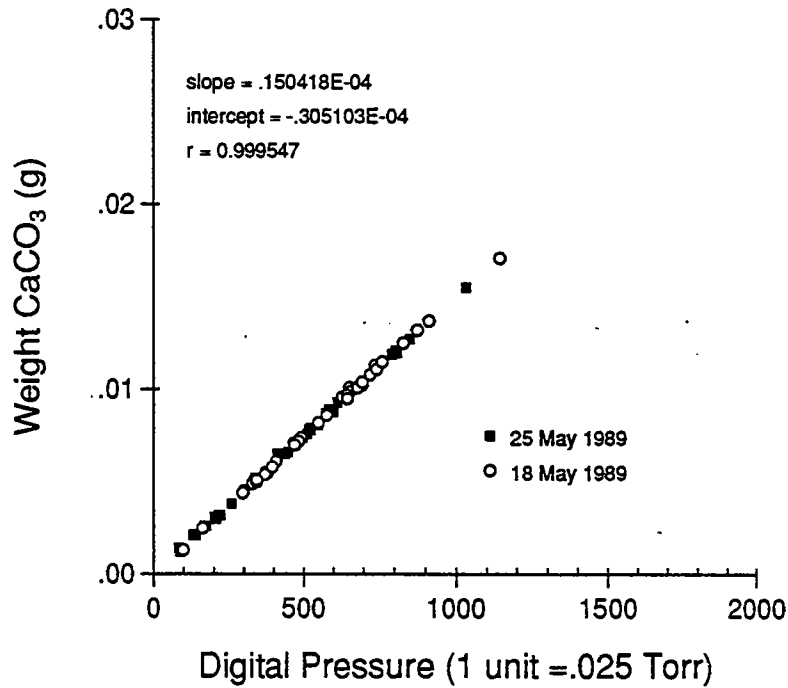
Figure 2: Two runs of 100% reagent grade calcium carbonate standards. The slope and intercept of the regression line produced from standards are used to calculate percent carbonate in unknown samples. For each new batch of phosphoric acid used, a new regression line is produced from standard runs.

c

Reaction Time vs Carbonate Content
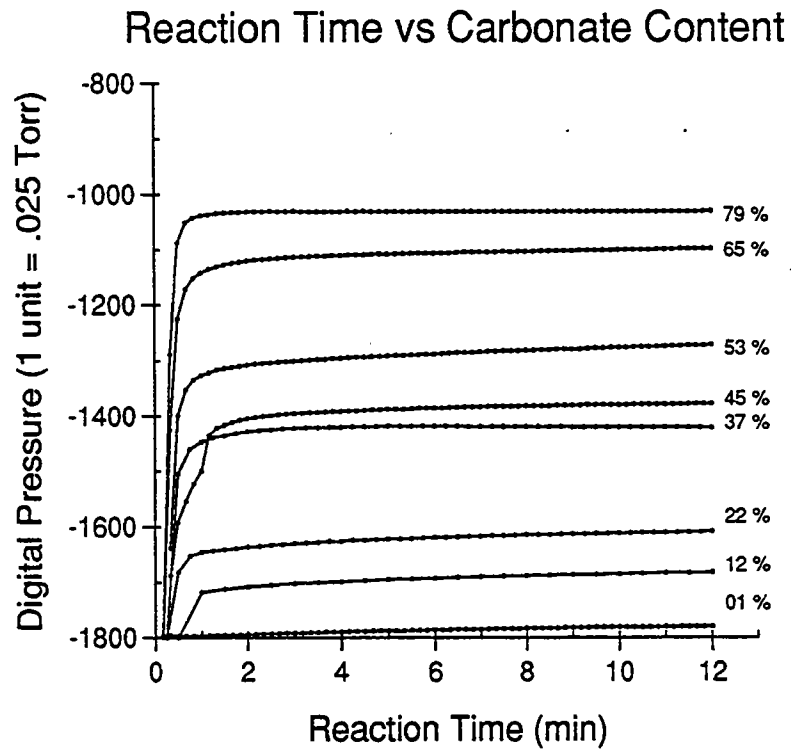
Figure 3: Reaction time for samples of varying carbonate content. For all samples, the bulk react of sample to CO_2 is complete within one minute. However, samples with less than 37% carbonte continue to react even after 12 minutes. The sediments are from cores recovered from the Sierra Leone Rise in the eastern equatorial Atlantic (Curry and Lohmann, 1985).

d

Reaction Time vs Carbonate Content
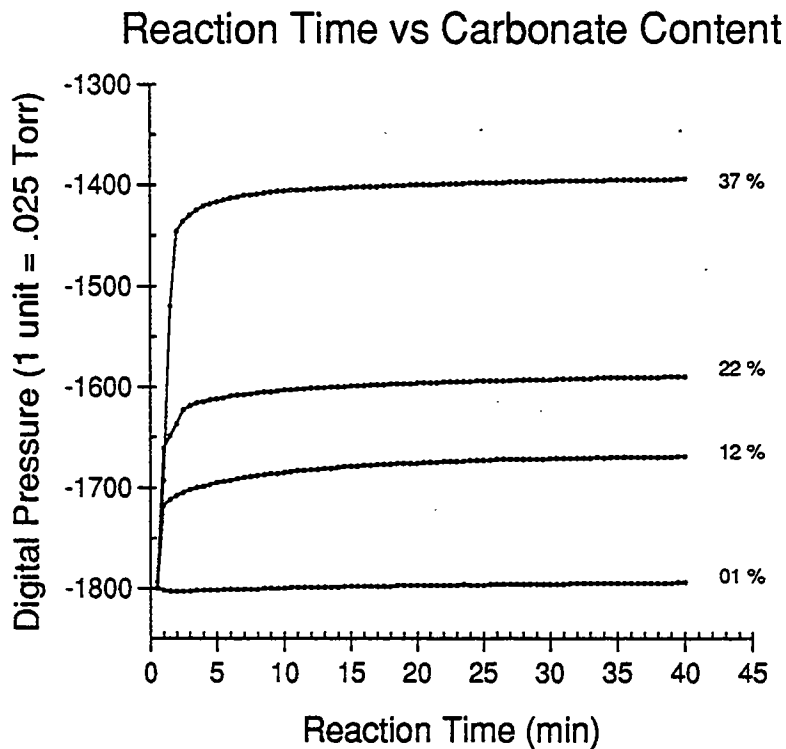
Figure 4: Reaction time for samples of low carbonate content. These samples were allowed to react for 40 minutes to find the reactime needed to evolve all the CO_2 from low carbonate samples. At 20 minutes, the reaction is complete and is 98-100% of the CO_2 value after 40 minutes. We believe that the small but gradual increase in carbonate content after 20 minutes is due to water vapor accumulation from acid outgassing. The sediments are from cores recovered from the Sierra Leone Rise in the eastern equatorial Atlantic (Curry and Lohmann, 1985).   e

Figure 5: Sample precision versus carbonate content. We show 120 replicate analysis of carbonate for samples with high, medium and low concentrations. The sediments are from cores recovered from the Sierra Leone Rise in the eastern equatorial Atlantic (Curry and Lohmann, 1985).

f

Appendix 1. Board jumpering specifics.

Board Jumpering - Various options are available on each of the following boards and are selected using removable jumper plugs. Features are selected by installing or removing the jumper plugs as noted below. See the appropriate manuals for further explanation. Only jumpers which are to be installed are detailed unless specifically noted.

VL-7806 (Z80 CPU)

J4	Memory map select/segment signal connector
M0	thru M3 use 8K Eprom chips
V1	a - connects CTC output 1 to serial channel B baud input
V1	c - connects SYSCLK/2 to CTC input 1
V2	DCE to terminal
V3	DCE to terminal
V4	DCE to terminal
V5	DTE to computer
V6	DCE to terminal
V7	DTE to computer
V8	DTE to computer
V9	DTE to computer
V10	b & c - sockets M0-M3 are 8K RAM chips
V11	a, b, c, & d - sockets M0-M3 are all enabled
V12	b - MEMEX is set low at power up
V13	MEMEX signal is controlled on the board
V14	a - IOEXP is connected to ground
V14	b - AUXGND is connected to digital ground

DSC-7911 (Dual stepper controller)

J1	no jumper - no interrupt desired
J2	center pin to NE, non-expanded I/O map
J7	E1 tied to 5, clock frequency of 6.25 kHz
SW1	1-5 on, to be address 15 matching A3-A7
SW1	6 off, enables the card

Appendix 1. Board jumpering specifics continued.

VL-7709 (64/256K memory board)

A	M0 & M1 are 8K chips and are disabled
B	M2 & M3 are 8K chips and are disabled
C	M4 & M5 are 8K chips and M4 is enabled, M5 is disabled
D	M6 & M7 are 8K chips and are disabled
V2	a-f all installed for no bank switching
V3	c, e, & g - 8K chips used for a total of 64K
V4	d & h - bank control port address EE
V5	b - IOEXP is active low
V6	no jumpers installed - 16 bit addressing without bank control

VL-STD AIN-1 (integrating analog input board)

7	= address of the board
d	= differential input mode selected

USD-7911 (Unipolar stepper driver)

E1	voltage range 8-32 volts
E2	external 5 volt supply not allowed
H3	Motor connections used
H3	pin 3 phase 4 output
H3	pin 4 phase 3 output
H3	pin 5 phase 2 output
H3	pin 6 phase 1 output
H3	pin 9 phase 1 common
H3	pin 11 motor supply voltage ground
H3	pin 12 motor supply voltage (+)

Appendix 2. Automated carbonate system memory map.

ADDRESS	K BYTES	NOTES
0000	0————————(	
	C4 BASIC ROM	
1000	4	Memory socket 0 on the Z80 board
2000	8————————(	
	System RAM	
3000	12	Memory socket 1 on the Z80 board
4000	16 (	
5000	20	Memory socket 2 on the Z80 board
6000	24 (	
7000	28	Memory socket 3 on the Z80 board
7400	29————————	
	Main Storage Area	
8000	32 (	
9000	36	Memory socket 4 on the memory board
A000	40 (	
B000	44	Memory socket 5 on the memory board
C000	48 (	
D000	52	Memory socket 6 on the memory board
DC00	55————————	
	Reserved for Machine Language Subroutines	
E000	56————————(	
	Not Used	
F000	60	Corresponds to socket 7 on the memory board (which is empty and disabled)
FFFF	64————————(	
	Top of Memory	

Appendix 3. System component diagrams.

A Connecting the transducer

* Adjust the potentiometer until the voltage at this point is
4.57 volt when referenced to the power supplie's ground

B Carousel tubing assembly

C Water jacketed reaction vessel

D Boat Specifications
.25" copper rod stock used
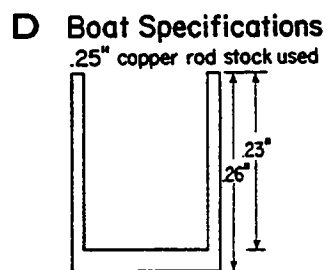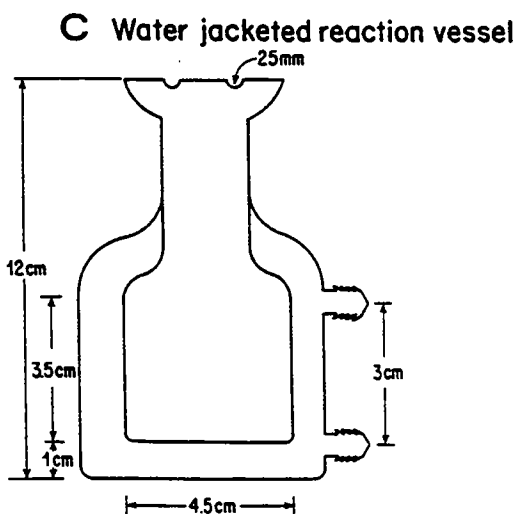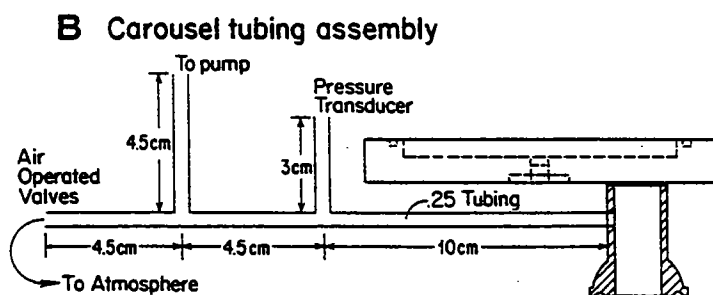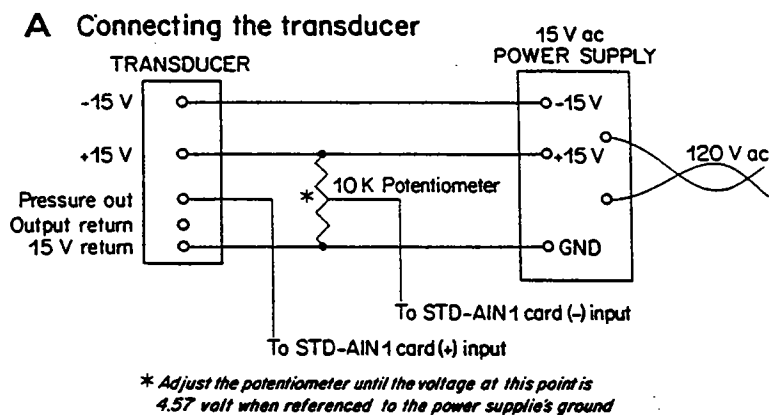
Appendix 4. Phosphoric acid mixing procedure.

This procedure is modified from Coplen et al, 1983 and is the same procedure used in the WHOI mass spectrometer facility. The 200°C hot acid will remove the ink from the thermometer so the thermometer should be inserted into a glass tube sealed at one end before immersing into the acid. Once the acid has cooled, it can be stored in a glass bottle with a cap containing a conical insert for tight sealing.

Chemicals needed:

P_2O_5 Phosphorus pentoxide (2.01 kg)
H_3PO_4 Phosphoric acid (4.30 kg or 2.5 liters 85%)
CrO_3 Chromium (ic) oxide (10 mg)
H_2O_2 Hydrogen peroxide (3 ml 50%)

Procedure:

1. Place a 4 liter beaker on a hot plate in a fume hood, wrapping the beaker in aluminum foil. The foil will help the acid maintain a constant temperature while the fume hood is operating.

2. Mix together the phosphorus pentoxide, the phosphoric acid and the chromium oxide. The solution should be yellow.

3. Heat uncovered at 200°C for 7 hours.

4. Add the hydrogen peroxide.

5. Heat uncovered at 220°C for 4.5 hours. The original 3.5 liters will evaporate to 3.25 liters.

6. The specific gravity of the final solution should be between 1.90–1.92. If it is not, more phosphorus pentoxide should be added as required and the entire process is repeated from step 3.

Appendix 5. Complete program listings for the following:
  Percent.for
  Getdata.bas
  Clear.bas
  Animat.bas
  Loader.bas

```
C
C        Percent.for
C
C        This program calculates the percent carbonate for samples
C        run on the Automated Carbonate System using a regression
C        line produced from reaction of 100% calcium carbonate
C        standards.
C
000      REAL SLOPE,INCPT
000      CHARACTER*64 FILEN,FILEM
000      CHARACTER Q
000      DATA SLOPE /.150418E-4/, INCPT /-.305103E-4/
000      WRITE(*,101) ' Input file: '
101      FORMAT(A,/)
000      READ(*,10) FILEN
010      FORMAT(A)
000      OPEN(UNIT=10,FILE=FILEN,STATUS='OLD')
031      WRITE(*,111) ' Slope: ',SLOPE
111      FORMAT(A,G15.6,/)
000      WRITE(*,101) ' Change? (y/N): '
000      READ(*,10) Q
000      IF(Q.eq.'Y'.or.q.eq.'y') THEN
000      WRITE(*,101) ' Enter new slope: '
000      READ(*,*) SLOPE
000      GO TO 31
000      ENDIF
032      WRITE(*,111) ' Intercept: ',INCPT
000      WRITE(*,101) ' Change? (y/N): '
000      READ(*,10) Q
000      IF(Q.eq.'Y'.or.q.eq.'y') THEN
000      WRITE(*,101) ' Enter new intercept: '
000      READ(*,*) INCPT
000      GO TO 32
000      ENDIF
000      WRITE(*,101) ' Output file: '
000      READ(*,10) FILEM
000      OPEN(UNIT=11,FILE=FILEM,STATUS='NEW')
001      READ(10,11,END=2)XNUM,DEPTH,WEIGHT,XPRESS
011      FORMAT(4F10.0)
C        IF(WEIGHT.LT..01)GO TO 1
000      TMP=XPRESS*SLOPE+INCPT
000      XPERC=TMP/WEIGHT*100.
000      WRITE(11,12)XNUM,DEPTH,XPERC
000      GO TO 1
002      CLOSE(10)
000      CLOSE(11)
012      FORMAT(F10.0,F10.2,F10.1)
000      STOP
000      END
```

```
C
C     Getdata.bas
C
C     This program produces paired data of pressure vs. time.
C
1         rem Getdata.bas produces pressure/time data
2         rem The boat must already be loaded in the slot to the
3         rem right of the delivery slot and the carousel must be
4         rem closed before running the program.
5         gosub 9549:rem close valves/pumpdown subroutine
6         rem (CAPS LOCK) must be depressed!
7         gosub 9525:rem close valves
10        OUT 01,50
20        OUT 00,250 : REM STARTING PULSE RARE
30        OUT 00,50 : REM HIGH SPEED PULSE RATE
40        OUT 00,&0F
50        out 00,&0F :rem larger deacceleration
100       print:print" enter # of samples ";
110       input l
120       print" enter sample interval (in secs.) ";
130       input q
140       OUT 01,77:OUT 00,254
500       gosub 1000
550       OUT 01,64
590       gosub 9500:rem open to atmosphere when done
600       print " Hit <P> to print the data or <Q> to quit =>";
610       A=USR(-9215)
620       if A=81 end
630       IF A=80 GOTO 3000
635       PRINT
640       GOTO 610
1000      REM * create pressure/time data
1010      for z=1 to l
1020      @(z)=ain(&70,0)
1030      gosub 2000
1040      next z
1050      return
1060      rem
2000      rem *delay
2010      delay Q*1000
2020      return
3000      rem * Print data
3010      for j=1 to l
3020      Z=J*q:GOSUB 6000:CPRINT 32:GOSUB 6300:GOSUB 4000:Z=@(J):GOSUB 6000
3021      GOSUB 6300
3025      CPRINT 13
3030      next j
3035      A=Usr(-9215)
3040      GOTO 600
4000      rem *do spacing
4010      for i=1 to 3
4020      CPRINT 32
4030      next i
4040      return
6000      REM DECIMAL ECT
```

Getdata.bas continued.

```
6005    REM This routine takes the var. Z and creats a
6010    REM 5 byte string of #'s ,containing the the ASCII codes for the
6015    REM characters which make up the printed representation of the # Z
6020    REM and stores them in D(0)-D(4)
6050    FOR X=0 TO 5:D(X)=32:NEXT X
6055    D(1)=48
6060    C=2:P=10000
6065    REM if Z is negitive then D(0)=45 (45=the ASCII code for "-")
6070    IF ABS(Z)<>Z D(0)=45:Z=ABS(Z)
6075    IF Z>9999 D(1)=49:Z=Z-10000
6100    D(C)=MOD(Z,P)/(P/10)+&30
6110    IF P=10 GOTO 6200
6120    P=P/10
6130    C=C+1
6140    GOTO 6100
6190    REM remove leading 0's and replace them with spaces
6200    FOR X=1 TO 4
6210    IF D(X)<>48 RETURN
6215    REM move sign 1 location to the right
6220    D(X)=D(X-1):D(X-1)=32
6230    NEXT X
6240    RETURN
6300    REM output contents of D(0)-D(4) to dart channel A
6305    D(6)=46:D(7)=48
6310    FOR X=0 TO 7
6330    CPRINT D(X)
6340    NEXT X
6350    RETURN
9549    print:print "Pumping on the acid for 27 minutes";
9500    rout 96,7,1:rem open to atmosphere at end
9501    return
9525    rout 96,6,0:delay 1000:rem close valve to pump
9526    return
9550    rout 96,7,0:rout 96,6,1:delay 9000:delay 9000:delay 9000:
9551    rem 9550 close valves/pumpdown on acid for 27 seconds
```

```
C
C       Clear.bas
C
C       This program clears the existing memory configuration so that
C       the storage area can be reinitialized.
C
05      rem Clear.bas, last revised 26.8.89 (DRO)
10      rem this program nukes the existing storage area so that NOVOS can be
20      rem re-initialized
30      rem CAUTION: this program destroys everything in the existing storage area
40      rem NOTE: after you run this the computer will lock up. Type '0' over and
50      rem over until it comes back to life (see page 2-6 of the C4-BASIC manual)
70      print " Enter the starting address of the storage area in hex ";
75      rem As presently configured, input '&7400'
80      input h
85      print " KABOOM !!!!!!!!! HA HA HA HA !!!!!"
90      for z=0 to 50:poke h+z,0:next z
```

```
C
C       Animat.bas
C
C       This program runs the Automated Carbonate System.
C
C
1       rem The Animating Element, last revised 30.8.89 (DRO)
5       print:print" Welcome stranger.":print
10      rem hardware initialization
20      gosub 6800:rem initialize serial port B to talk to the Mettler
30      gosub 7300:rem initialize motor controller card
40      print" Skip software initialization? (Y/N)";
45      gosub 700
46      rem if "Y" then skip it
47      if a=89 goto 91
48      print:print " Initializing......"
50      rem init. variables
60      for z=0 to 816:@(z)=32:next z:rem clear out array
65      rem put zeros in the pressure value slots
70      for w=1 to 40:v2=0:gosub 9310:next w
80      N=1:rem sample # of the next sample 2b weighed
81      L=40:rem # of boats
83      for z=0 to 7:f(z)=0:next z:rem zero all the flags
85      l2=5:rem minimum pressure value (used in run routine)
91      print:print" Is the Lazy Susan homed to slot #1 ? (Y/N)";
92      rem call up Homer menu if "Y"
93      gosub 700:if a=78 gosub 5100:goto 100
95      c=1
100     print:print" The Animating Element"
110     print:print
120     print" Type :"
130     print" <W> to weigh samples"
132     print" <L> to load the sample boats"
135     print" <R> to run samples"
150     print" <P> to print the data"
155     print" <I> to re-initialize"
156     print" <T> to tweak the system"
160     print" <Q> to quit"
170     print:print:print
180     print" =>";
190     gosub 700:rem call a ML single char input sub.(A=the ASCII val of char)
195     rem ASCII codes:87="A",76="L",82="R",80="P",73="I",84="T",81="Q"
199     rem weigh samples
200     if a=87 gosub 1000:goto 110
219     rem load boats
220     if a=76 gosub 2000:goto 110
224     rem run samples
225     if a=82 gosub 3000:goto 110
229     rem print data
230     if a=80 gosub 4000:goto 110
234     rem re-initialize
235     if a=73 goto 5000
240     rem call up system tweak menu
245     if a=84 gosub 500:goto 110
248     rem quit
249     if a=81 goto 300
250     goto 190
```

```
300    rem quit
310    print:print" Quitting will destroy all the data in memory. Are you sure"
320    print"you want to quit (Y/N) ?";
330    gosub 700:if a<>89 goto 100
340    print:print" goodbye....." :end
490    rem Tweak the system menu and options
500    print:print" Tweak the system........":print
510    print" Hit :"
520    print" <H> to home the Lazy Susan"
530    print" <A> for atmosphere"
540    print" <V> for vacuum"
545    print" <W> to re-weigh an entry"
550    print" <R> to continue running after a jam up"
570    print" <C> to check the pressure in the vessel"
580    print" <E> to exit to the main menu"
585    print:print:print
590    print" =>";
600    gosub 700:rem get a single keypress
605    rem ASCII codes:72="H",65="A",86="V",87="W",82="R",67="C",69="E"
609    rem call homer subroutine
610    if a=72 gosub 5100:goto 500
615    rem open to atmosphere
620    if a=65 goto 9500
629    rem pump down
630    if a=86 goto 9550
632    rem re-weigh
634    if a=87 gosub 1500:goto 500
639    rem continue running
640    if a=82 gosub 3300:goto 110
659    rem check pressure
660    if a=67 gosub 8400:goto 500
669    rem return to main menu
670    if a=69 return
680    goto 600
700    rem*******Get a single Keypress, Convert lowercase letters to Caps.*****
710    a=usr(-9215)
720    if a<97 return
730    if a>122 return
740    a=a and &DF: rem convert lowercase => uppercase
750    return
1000   rem****************** weigh samples *********************************
1002   rem This sub. prompts the user to place the boats and the samples
1005   rem on the balance,tares the boats, weighs the samples and stores
1010   rem the results in array @(x)
1020   if f4=1 print" All the samples have already been weighed." :return
1025   if f6=1 print" Sorry you can not resume weighing after loading." :return
1100   print:print
1101   print" "
1102   print:print "Sample # ";n
1105   print
1106   print" Enter the depth in cm => ";
1107   rem get depth value
1108   w=n:gosub 9010:if f1=1 f1=0:return
1109   print
1110   print" Hit <spacebar> when you are ready to tare the boat.=>";
1115   rem call ML character input sub.
1117   rem if inputted character = <CTRL> Z return
```

```
1120    gosub 700:if A=26 return
1121    if a=4 goto 1105
1122    if a<>&20 goto 1120
1123    rem 4='D',&20='<SPACEBAR>'
1125    rem
1127    print:print "taring...."
1128    rem tare, exit if escape flag set
1130    gosub 6600:if f1=1 f1=0:return
1135    rem
1140    print:print"Tare completed."
1150    print:print" Add the sample to the boat and hit any key when it is"
1160    print" ready to be weighed.=>";
1165    rem wait for a keypress. return if <CTRL><Z>
1170    gosub 700:if a=26 return
1171    rem if there was a <CTRL><T> retare.
1172    if a=20 goto 1127
1173    rem
1175    print:print"Weighing"
1179    rem ask balance for next stable weight
1180    gosub 6700:rem S command
1185    rem
1186    rem input data from balance:if no errors occured goto 1200
1190    gosub 6010:if S=0 goto 1200
1192    print" An input error has occured.Type <R> for retry or <A> for"
1193    print" abort.=>";
1194    rem get keypress:return if <CTRL><Z>
1195    gosub 700:if A=26 return
1196    rem return if "A"
1197    if a=65 return
1198    goto 1180
1200    w=n:gosub 9210: rem store weight
1210    w=n:gosub 9410: rem print data
1222    print
1223    print" Do you wish to redo this sample ? (Y/N)";
1224    rem get keypress: return if <CTRL><Z>
1225    gosub 700:if A=26 return
1226    rem if "Y" redo entire sample,if "W" re-weigh
1227    if A=89 goto 1100
1228    if A=23 goto 1175
1229    rem return to re-weigh function if re-weigh flag set
1230    if f0=1 return
1232    rem set flag and return if all samples weighed
1234    if n=l f4=1:return
1240    n=n+1
1300    goto 1100
1500    rem ****************** re-weigh a sample *****************************
1505    if n=1 print " No samples have been weighed yet.":return
1507    t=n:rem save old value
1510    print:print:print" Enter the number of the sample to re-weigh (0 aborts)";
1520    input n
1530    if n=0 goto 1570
1540    if n<1 goto 1510
1550    if n>t print" That sample hasn't been weighed yet.":goto 1510
1555    rem set "re-weigh" flag, call weigh subroutine
1560    f0=1:gosub 1100:f0=0
1565    rem restore old value
1570    n=t:return
```

```
2000    rem ***************** load boat onto lazy-susan *************************
2005    if f4=0 print:print" All the samples have not been weighed yet":goto 100
2007    d2=50:rem motor speed, used in routine at 7200
2009    f6=1:rem flag set when load routine called
2010    goto 9500
2015    if f5=1 print:print" Boats already run.....":return
2020    if f3=1 print:print" boats already loaded....":return
2040    l=n
2050    print
2055    rem keep from dumping 1st boat when loading 40th
2060    if f2=1 c=40
2070    print" Load the boat into load slot";c;" and hit the <SPACEBAR> =>";
2080    if f2=1 c=39
2085    rem get keypress: return if <CTRL><Z>
2090    gosub 700:if a=26 return
2100    if a<>32 goto 2090
2105    rem load 40th boat if f2 set and set f3 (all boats loaded flag)
2110    if f2=1 print:print" Boat ";40;" loaded.":f3=1:return
2120    if c=39 f2=1:c=40:goto 2140
2130    d=0:t=c+1:gosub 7010:c=t:rem go to hole T subroutine
2140    print:print" Boat ";c-1;" loaded."
2145    if f2=1 c=39:rem more flag gymnastics
2147    rem keep loading if there are more boats
2150    if c<l goto 2050
2170    return
3000    rem********************** run samples *******************************
3005    rem choose the reaction time in minutes
3010    gosub 9600
3020    if f3=0 print" The boats have not all been loaded yet.":return
3034    if f1=1 f1=0:goto 3060
3035    if f5=1 print" The samples have already been run.......":return
3036    f5=1
3040    if c<>39 t=39:d=0:D2=20:print " advancing to home position...":gosub 7010
3045    D2=200:rem motor speed delay used in routine at line 7200
3050    c=39
3060    l=n
3065    if l<40 l=l-1
3070    print" pumping down";
3075    gosub 8010: rem call pumpdown subroutine
3080    rem get pressure, return if escape flag set.
3085    gosub 8100: if f1=1 return
3090    v1=a: rem store average baseline pressure value
3100    print" done."
3110    r=c+2:if r>40 r=r-40
3120    t=c+1:d=0:gosub 7010
3130    print:print" running sample #";r;
3135    rem minutes changed to seconds reaction time
3140    k=K1*60
3145    rem read pressure, return if escape flag set
3150    gosub 8100:if f1=1 return
3160    v2=a-v1: rem get change in pressure
3170    w=r:gosub 9310: rem save pressure value in array
3180    print" done.":print
3190    w=r:gosub 9410:rem output pressure data to terminal
3200    c=c+1:if c>40 c=c-40
3202    if v2>l2 goto 3210
3204    for z=0 to 20:cprint(7):next z
```

```
3206    print:print" Check to make sure the Lazy-susan isn't jammed."
3207    print" Hit <A> to abort or <R> to resume =>";
3208    gosub 700:if a=65 f1=1:return
3209    if a<>82 goto 3208
3210    if r< l goto 3070
3220    if c<>1 print" advancing to load slot #1....":gosub 7100
3230    c=1
3235    rem at end of run put system under vacuum
3236    goto 9550
3240    return
3300    rem********** continue running after a jam up *************************
3310    print:print
3312    t=c: rem save old value in case user aborts
3315    print " Enter the # of the load slot the L.S is homed to (0 to abort) ";
3320    input c
3325    if c=0 c=t:return
3326    if c>40$ to 3320
3327    if c<1 goto 3320
3330    f1=1
3340    gosub 3000
3350    return
4000    rem*******print contents of weight/pressure file***********
4010    rem
4020    if n=1 print:print" Sorry, there is nothing to print.":return
4030    if n<40 n=n-1
4050    for w=1 to N
4060    gosub 9410
4070    next w
4080    if N<40 N=N+1
4090    gosub 700:rem wait for a key press
4100    return
5000    rem ******************** reset sample pointer ****************************
5010    print:print" CAUTION: re-initializing will destroy all the"
5020    print" data in memory. Are you sure you want to do it (Y/N) ?";
5030    gosub 700:if a<>&59 goto 110
5040    print:print" Forgetting.........":goto 50
5100    rem****************** home the lazy susan ****************************
5105    if f3=1 print:print:print" CAUTION: boats have already been loaded!"
5110    print:print:print" Home Menu"
5120    print:print
5140    print" < RIGHT ARROW > = counter clockwise"
5250    print" < LEFT ARROW > = clockwise"
5260    print" < DOWN ARROW > = single step mode"
5270    print" < UP ARROW > = continuous step mode"
5280    print" < S > = Smart Home option"
5290    print" < E > = exit"
5300    print:print " =>";
5310    gosub 700: rem get a single keypress
5320    rem f7= mode flag, 0 = single step mode, 1 = continuous step mode
5330    out 01,64:rem send stop command to motor controller card
5340    if a=10 f7=0: rem when booted under Async
5350    if a=24 f7=0: rem when booted under Word Star
5360    if a=11 f7=1: rem Async
5370    if a=5 f7=1: rem WS
5380    if a=29 d=1:gosub 5500:rem Async
5390    if a=19 d=1:gosub 5500:rem WS
5400    if a=12 d=0:gosub 5500:rem Async
```

```
5410    if a=4 d=0:gosub 5500:rem WS
5415    rem smart home option
5420    if a=83 goto 5600
5425    rem return to other menu
5430    if a=69 goto 5700
5440    goto 5310
5500    rem send commands to motor controller card
5510    if f7=1 goto 5550
5520    out 01,66 or (d*8): rem single step,direction d
5530    return
5550    out 01,69 or (D*8): rem continuous step mode, direction d
5555    out 00,254: rem motor speed
5560    return
5570    rem* smart home option
5600    print:print" Enter the current load slot # ";:input c
5602    if c<1 goto 5600
5604    if c>40 goto 5600
5610    gosub 7100:return
5700    rem* return to main menu
5710    print:print" Enter the current load slot # ";:input c
5712    if c<1 goto 5710
5714    if c>40 goto 5710
5720    return
6000    rem**********Mettler I/O subroutines************
6010    rem Generic input subroutine
6020    rem This routine calls a m/l routine to input data from the Mettler
6030    rem and sets the flag S on the result.
6040    rem if S=0 the data was succesfully inputed and is stored in
6050    rem @(0)-@(15)
6060    rem if S=1 a tare occurred and the data in @(0)-@(15) is invalid
6070    rem if S=2 no tare occured and no data was received
6080    S=0
6090    rem clear out input subroutines buffer        .
6100    for Z=0 to 15
6110    POKE -9201+27+Z,32
6120    next Z
6130    rem call machine language Mettler I/O subroutine
6140    A=USR(-9201)
6150    rem take data from input subroutine's buffer & put it in @(0)-@(15)
6160    for Z=0 to 15
6170    @(Z)=PEEK(-9201+27+Z)
6180    next Z
6190    rem check to see if first data byte valid (&53="S")
6200    if @(0)=&53 goto 6260
6220    rem first byte isnt the start of a data block:goto interpret error sub
6230    goto 6280
6240    rem check to see if last byte valid (&A=<lf>).if so assume
6250    rem the whole data block is valid
6260    if @(15)=&A return
6270    rem find location of <lf> in array @(X)
6280    for Z=0 to 15
6290    if @(Z)=&A goto 6340
6300    next Z
6310    rem if no <lf> found set error flag and return
6320    S=2:return
6330    rem check first char of message CASE "T"->6400
```

```
6340    if Z<3 S=2:return
6350    if @(Z-3)=&54 goto 6400
6360    rem if message doesn't start with any of above chars it is invalid
6370    rem set error flag and return
6380    S=2 :return
6390    rem if second char of message = "A" set error flag=1 (1=tare)& return
6400    if @(Z-2)=&41 S=1:return
6410    rem if first char of message ="T" & 2nd <> "A" then the message is
6420    rem invalid,set error flag and return
6430    S=2:return
6490    rem————————————————————————
6500    rem disable tare bar ie. send R1<cr><lf> to the balance
6510    gosub 6900:out &F6,&52
6520    gosub 6900:out &F6,&31
6530    gosub 6900:out &F6,&0D
6539    gosub 6900:out &F6,&0A
6550    return
6555    rem————————————————————————
6560    rem enable tare bar ie. send R0<cr><lf> to the Mettler
6570    gosub 6900:out &F6,&52
6580    gosub 6900:out &F6,&30
6590    gosub 6900:out &F6,&0D
6593    gosub 6900:out &F6,&0A
6597    return
6598    rem————————————————————————
6600    rem send tare command ie. send T<cr><lf> to the Mettler
6605    Q=0:f1=0
6610    gosub 6900:out &F6,&54
6615    gosub 6900:out &F6,&0D
6620    gosub 6900:out &F6,&0A
6625    gosub 6010:rem error check routine
6630    if S=1 return
6635    rem if the balance doesn't tare correctlly retry up to 4 times
6640    Q=Q+1:if Q<4 goto 6610
6645    print:print
6650    rem if the balance still hasn't tared give the user a chance to abort
6655    print" The balance refuses to tare.Type <R> for retry or <A> for
6660    print" abort.=>";
6665    rem wait for a keypress:if the char ="R" then retry
6670    gosub 700:if A=&52 goto 6605
6675    rem ifthe char="A" then return to the menu
6680    if A=&41 f1=1:return:rem set escape flag and return
6685    goto 6670
6690    rem————————————————————————
6700    rem ask balance for next stable weight (ie. send S<cr><lf> to balance)
6710    gosub 6900:out &F6,&53
6720    gosub 6900:out &F6,&0D
6730    gosub 6900:out &F6,&0A
6740    return
6745    rem————————————————————————
6800    rem initialize serial port B
6805    out &F1,&45
6810    out &F1,&D0
6815    out &F7,&10
6820    out &F7,&18
6825    out &F7,&4
6830    out &F7,&47
```

```
6835    out &F7,&05
6840    out &F7,&AA
6845    out &F7,&03
6850    out &F7,&41
6855    out &F7,&00
6860    return
6870    rem————————————————————————————
6900    rem do output handshaking
6910    rem see p3-14 of the Z80 ref manual
6920    if RIN(&F7,2)=0 goto 6920
6930    if RIN(&F7,5)=0 goto 6930
6940    return
7000    rem*********** motor controller card subroutine **********************
7010    rem * GO to HOLE T, IN DIRECTION D, FROM CURRENT HOLE, C
7020    if D=1 goto 7060
7030    N2=(T-C)*50
7040    if N2<0 N2=N2+2000
7050    goto 7080
7060    N2=(C-T)*50
7070    if N2<0 N2=N2+2000
7080    gosub 7200
7090    return
7095    rem————————————————————————————
7100    rem *home lazy-Susan to load slot #1
7110    if c>=20 d=0
7120    if c<20 d=1
7130    t=1:d2=50:gosub 7010
7140    c=1
7150    return
7190    rem————————————————————————————
7200    rem *GO N STEPS IN DIRECTION D
7210    for Z= 1 to N2
7220    out 01,66 OR (D*8)
7230    delay d2
7240    next Z
7250    return
7290    rem————————————————————————————
7300    rem initialize motor controller card
7310    out 01,50
7320    out 00,250
7330    out 00,50
7340    out 00,&0f
7350    out 00,&0f
7360    return
8000    rem****************** valve card and transducer routines **************
8020    goto 9550
8023    rem pump down to 4.2 Torr
8025    if ain(&70,0)>-1780 goto 8025
8040    goto 9525
8050    return
8090    rem————————————————————————————
8100    rem *wait for pressure to settle
8105    rem o=0
8110    rem for z=1 to 500
8120    rem x=ain(&70,0)
8130    rem if abs(x-o)>3 print".";:o=x:goto 9510
8140    rem next z
```

```
8200    rem * average data from pressure transducer
8201    rem by making sure 10 values in a row are equal
8202    rem with a delay of 6 seconds between each reading
8205    b1=ain(&70,0)
8207    if ain(&70,0)>16000 goto 8260
8210    b=0
8215    delay 6000
8220    b2=ain(&70,0)
8225    if abs(b2-b1)>6 goto 8205
8230    b=b+1
8235    if b<9 goto 8215
8240    a=b1
8250    return
8260    out 120,0:delay 3000: rem close all valves
8262    print" The reaction chamber is open to the atmosphere."
8263    print" <P>umpdown or <A>bort ?";
8265    gosub 700
8270    if a=65 f1=1:return
8275    if a=80 out 120,2:delay 3000:goto 8205
8280    goto 8265
8300    rem delay k seconds
8310    for m=1 to k
8320    delay 1000
8330    next m
8340    return
8400    rem*************Check pressure*****************************************
8403    gosub 9550:rem close valves/pumpdown subroutine
8404    print "Pumping on the acid for 36 seconds";
8405    delay 9000:delay 9000:delay 9000:delay 9000
8407    gosub 9525:rem close all valves
8410    print:print" Enter time interval in seconds (0 aborts) ";
8420    input k:if k<0 goto 8410
8425    print:"Getting initial pressure (1 minute)...";
8440    if k=0 return
8450    gosub 8100: v1=a: rem get initial pressure
8460    print " Waiting ";k;" seconds........";
8470    gosub 8300: rem wait k seconds
8475    print: "Getting final pressure (1 minute)....";
8480    gosub 8100: rem get pressure again
8490    print" The change in pressure was ";v1-a;" digital units."
8492    gosub 9550:rem continue pumping on acid
8495    return
8500    rem*************Decimal output routines and stuff**********************
8505    rem This routine takes the var. Z and creats a
8510    rem 5 byte string of #'s ,containing the the ASCII codes for the
8515    rem characters which make up the printed representation of the # Z
8520    rem and stores them in J(0)-J(4)
8550    for X=0 to 4:J(X)=32:next X
8560    Q=1:P=10000
8565    rem if Z is negative then J(0)=45 (45=the ASCII code for "-")
8570    if ABS(Z)<>Z J(0)=45:Z=ABS(Z)
8600    J(Q)=MOD(Z,P)/(P/10)+&30
8610    if P=10 goto 8700
8620    P=P/10
8630    Q=Q+1
8640    goto 8600
8690    rem remove leading 0's and replace wAth spaces
```

```
8700    for X=1 to 3
8710    IF j(X) <>48 RETURN
8715    rem move sig@ 1 location to the right
8720    J(X)=J(X-1):J(X-1)=32
8730    next X
8740    return
8800    rem output contents of J(0)-J(4) to dart channel A
8810    for X=0 to 4
8830    Cprint J(X)
8840    next X
8850    return
9010    rem get a 10 byte striAg and save it in array @() at address w
9015    for z=0 to 9:@((w*20)+z)=32:next z
9020    for z=0 to 10
9025    fl=0: rem escape flag
9030    rem get a single character using ML routine at -9215 (dec)
9040    gosub 700
9045    if a=26 fl=1:return
9050    rem if it was a backspace then backspace
9055    if z=0 goto 9075
9060    if a=8 z=z-1:@((w*20)+z)=32:cprint 8:cprint 32:cprint 8:goto 9040 ·
9070    rem store data in array
9075    if a=8 goto 9040
9076    rem if that character was a CR exit routine
9077    if a=13 goto 9160
9080    @((w*20)+z)=a
9110    rem wait for a CR or backspace after 10th char.
9120    if z=10 goto 9040
9130    rem print character
9140    cprint a
9150    next z
9160    if z=10 return
9162    if z=0 @((w*20)+9)=48:return
9165    for j=z to 0 step -1
9170    @((w*20)+j+10-z)=@((w*20)+J)
9180    @((w*20)+j)=32
9190    next j
9195    return
9210    rem store weight
9220    for z=0 to 8
9230    @((w*20)+z+10)=@(z+3)
9235    next z
9240    return
9310    rem store pressure
9320    @((w*20)+19)=v2
9330    return
9410    rem print a single entry
9412    cprint 32:cprint 32:cprint 32
9415    z=w:gosub 8500:gosub 8800:cprint 46:cprint 48
9420    for y=0 to 18
9430    cprint @((w*20)+y)
9440    if y=9 cprint 32
9450    next y
9460    cprint 32:cprint 32:cprint 32
9470    z=@((w*20)+19):gosub 8500:gosub 8800:cprint 46:cprint 48
9480    cprint 13
9490    return
```

```
9500    rem **************open to atmosphere subroutine***************
9505    rout 112,2,0
9510    rout 112,3,0
9515    delay 1000:rout 112,2,1
9520    return
9525    rem *******************close all valves subroutine**************
9530    rout 112,2,0
9535    rout 112,3,0
9540    delay 3000
9545    return
9550    rem ***********close valves/pumpdown subroutine******************
9555    rout 112,2,0
9560    rout 112,3,0
9565    delay 3000:rout 112,3,1
9570    return
9600    rem****************reaction time menu*********************
9610    print:print " Run the system............":print
9620    print " Choose the appropriate reaction time ":print
9630    print " Hit: "
9640    print " <L> low carbonate ( 0-25%), 20 minutes"
9660    print " <H> high carbonate (25-100%), 5 minutes"
9680    print:print:print
9690    print " => ";
9700    gosub 700: rem get a single keypress
9710    rem ASCII codes: 72='H',76='L',77='M'
9720    rem low carbonate
9730    if a=76 K1=20:goto 9800
9760    rem high carbonate or standards
9770    if a=72 K1=5:goto 9800
9800    return
9805    rem*********HARDWARE DEPENDANT SOFTWARE*********************
9810    rem
9820    rem There are a few hardware dependant lines in Animat.bas
9830    rem which will have to be changed if the hardware is
9840    rem configured differently. The lines are listed below and
9850    rem are marked with a '#' in the program listing.
9860    rem
9870    rem Relay output channels: 9505-9515, 9530-9535, 9555-9565
9880    rem Analog input address : 8025, 8120, 8205-8207, 8220
9890    rem Motor driver      : 5330, 5510-5560, 7310-7350
```

```
C
C     Loader.bas
C
C     This program loads two machine language subroutines into memory.
C
C
1     REM RDKEY & METIO loader program, last revised 29.8.89 (DRO)
2     rem This program loads 2 machine language subroutines,
3     rem RDKEY & METIO, into memory
4     rem RDKEY starts at -9215
5     rem METIO starts at -9201. It's buffer starts at -9174
6     x=&DC00
7     b1=&2A:b2=&dC
10    REM Single character input subroutine (RDKEY)
20    REM DB,F5 LOOP INA,F5H ;check RCV bit
30    REM CB,47 BIT 0,
40    REM 28,F9 JR Z,P ;i's not set wait
50    REM DB,F4 IN A,H ;g character
60    REM E6,7F AND 7F;lose the highest bit
70    REM 6F L t n L so basic can read it
80    REM 26,00 LD H,zero H so basic doesn't get garbage
90    REM C9 RTrn
100   REM poke single character input sub. into memory
110   X=X+1:POKE X,&0DB
120   X=X+1:POKE X,&0F5
130   X=X+1:POKE X,&0CB
140   X=X+1:POKE X,&047
150   X=X+1:POKE X,&028
160   X=X+1:POKE X,&0F9
170   X=X+1:POKE X,&0DB
180   X=X+1:POKE X,&0F4
190   X=X+1:POKE X,&0E6
200   X=X+1:POKE X,&07F
210   X=X+1:POKE X,&06F
220   X=X+1:POKE X,&026
230   X=X+1:POKE X,&000
240   X=X+1:POKE X,&0C9
250   REM Balance input subroutine (METIO)
260   REM b1 and b2 are variables
270   REM 06,10 START LD B,16 16=the maximum # of chars.
280   REM DD,21,b2,b1 LD IX,$b1b2 load IX with start of buffer
290   REM DB,F7 LP IN A,($F7) load A with status register
300   REM CB,47 BIT 0,A test bit 0 (bit=0 RCV flag)
310   REM 28,FA JR Z,LP if it=0 goto LP
320   REM DB,F6 IN A,($F6) load a with data register
330   REM E6,7F AND 7F
340   REM DD,77,00 LD (IX),A store A in buffer
350   REM DD,23 INC IX increment buffer pointer
360   REM FE,0A CP $A compare A to 10 (10=lf)
370   REM C8 RET Z if equal return to BASIC
380   REM 10,EC DJNZ LP decrement B and goto LPifnot0
390   REM C9 RET if b=0(ie.16 chars.have been
400   REM entered) return to BASIC
410   REM &F7= the status register of DART channel b
420   REM &F6= the data register of the same.
430   REM See pp 3-8 - 3-17 of the MULTIFUNCTION Z80CPU CARDS VL-7806 &VL-7807
440   REM for more info. on the dart chip. esp 3-14
```

```
460     rem poke balance input subroutine into memeory
470     X=X+1:POKE X,&06
480     X=X+1:POKE X,&10
490     X=X+1:POKE X,&DD
500     X=X+1:POKE X,&21
510     X=X+1:POKE X,b1: rem tell ML program where its buffer starts
520     X=X+1:POKE X,b2
530     X=X+1:POKE X,&DB
540     X=X+1:POKE X,&F7
550     X=X+1:POKE X,&CB
560     X=X+1:POKE X,&47
570     X=X+1:POKE X,&28
580     X=X+1:POKE X,&FA
590     X=X+1:POKE X,&DB
600     X=X+1:POKE X,&F6
610     X=X+1:POKE X,&E6
620     X=X+1:POKE X,&7F
630     X=X+1:POKE X,&DD
640     X=X+1:POKE X,&77
650     X=X+1:POKE X,&00
660     X=X+1:POKE X,&DD
670     X=X+1:POKE X,&23
680     X=X+1:POKE X,&FE
690     X=X+1:POKE X,&0A
700     X=X+1:POKE X,&C8
710     X=X+1:POKE X,&10
720     X=X+1:POKE X,&EC
730     X=X+1:POKE X,&C9
```

Appendix 6. System Configuration Procedure.

The system has a total of 48K RAM; three 8K RAM chips on the Z80 board and three 8K RAM chips on the memory expansion board. The present configuration is diagrammed in Figure 5 and is detailed as follows:
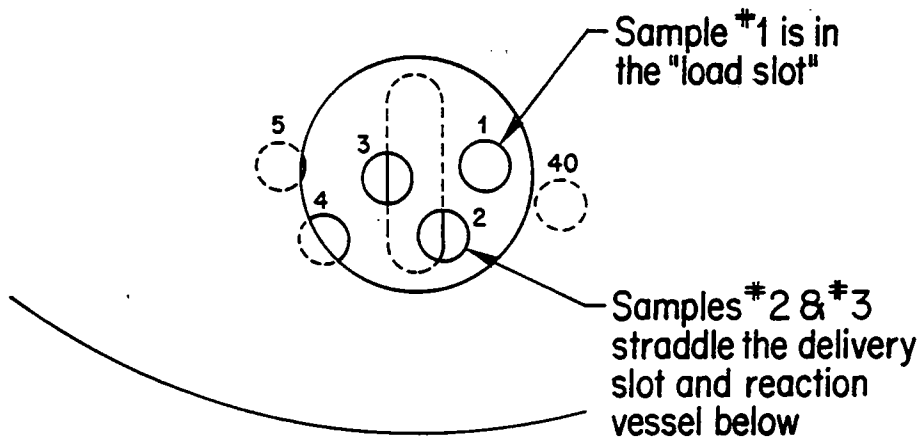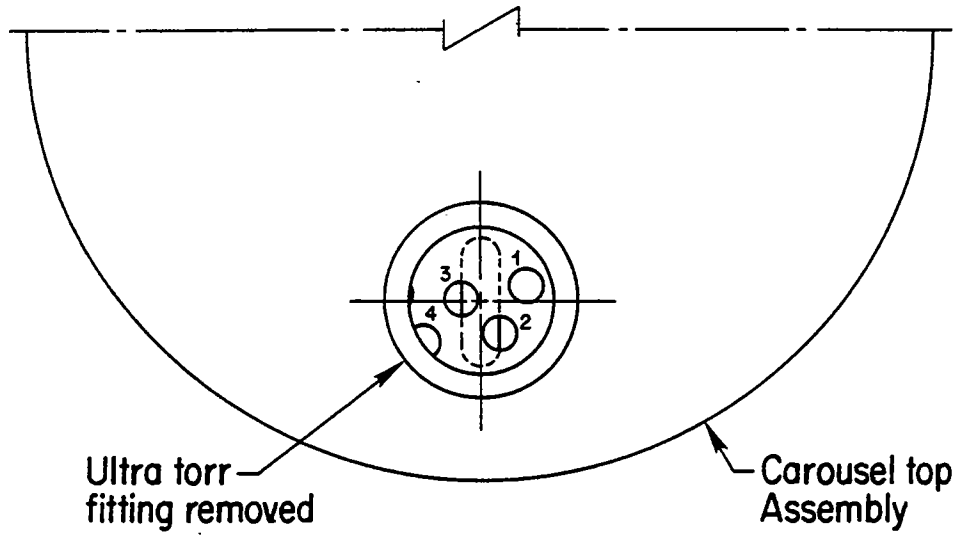
a) the storage area starts at &7400
b) the development RAM size is set to 21K
c) the storage area size is set to 26K
d) 1K at the top of memory is reserved for important machine language subroutines

If your system is not configured to reserve the necessary 1K of memory, reinitialize the system by following the procedure outlined below:

1. Enter *NOVOS* by typing '*NOVOS*'
2. Download *clear.bas* and run it (see *clear.bas* under SOFTWARE, p. 6)
3. Power down the system for 15 seconds and reset the power supply card
4. Turn the power back on and hit '0' (zero) repeatedly until the prompt appears
5. At the prompts enter;
    '21' for RAM size
    '0' for baud rate
6. Type '*NOVOS*'
7. Type '*SETUP*'
8. At the prompts enter;
    '1' for memory type (CMOS RAM)
    '7400' for the start of the storage area
    '26' for the storage area size
    '5' for the number of directory entries
9. Download *loader.bas* and run it (see *loader.bas* under SOFTWARE p 7.)
10. Download *animat.bas* and fix the downloading errors if any
11. Save *animat.bas* in slot 1 of the directory by typing 'SAVE 1'
12. Check to see that the machine language subroutines are properly loaded.
    Type 'PRINT USR(-9215)'<RET>
    Hit the space bar
    '32' should be printed on the screen (32 is the ASCII code for 'space bar')
    If not, run *loader.bas* and begin from step 9
13. Type 'DIR' to see that the following is printed:
    *DIR
    1 AUTOMATED CARBONATE SYSTEM program, last revised 16.5.89 (DRO)
    2 RDKEY & METIO LOADER program, last revised 26.8.89 (DRO)

RDKEY and METIO should now be resident in memory, starting at &DC01 (-9201) decimal. The execution addresses are -9215 for RDKEY and -9201 for METIO. METIO'S buffer, containing the character string inputed from the balance, begins at -9174 and is 16 bytes long. Whenever the system is powered up, always first run the loader program ('run 2') followed by the AUTOMATED CARBONATE SYSTEM program ('run 1') to assure that RDKEY and METIO are properly loaded.

Appendix 7. Sample carousel load slot diagram.

Ultra torr
fitting removed

Carousel top
Assembly

Sample #1 is in
the "load slot"

Samples #2 & #3
straddle the delivery
slot and reaction
vessel below

Appendix 8. Possible suppliers of equipment for the ACS

COMPANY	SPECIFIC SUPPLIES NECESSARY
Acculex 440 Myles Standish Blvd. Taunton, MA 02780	#DP-654 Voltage input meter
Ace Glass Inc. 1430 N.W. Blvd Vineland, NJ 08360-688	#9519-10 Pinch clamp for 25mm joint
All-Stainless 75 Research Rd. Hingham, MA 02043	10-32 x 3/8" allen cap screws 18-8 s.s. 10-32 x 2" allen cap screws 18-8 s.s
Analog Devices 1 Technology Way Norwood, MA 02062-9106	chassis mounted ac/dc power supply +/- 15 vdc output #952
Anderson Glass Old Turnpike Rd. R.F.D. 1 Fitzwilliam, NH 03447	glass reaction vessel; see Appendix 3.
B & D Motor Control Corp 99 Lowell Road Hudson, NH 03051	Slo-Syn DC stepping motor #MO61-FD-311
Cambridge Valve & Fitting 50 Manning Rd. Billerica, MA 01821	Cajon ultra-torr union fitting 1/2" to 1/4" #SS-8-UT-6-4 Cajon ultra-torr fitting #SS-16-UT-A-20 Cajon ultra-torr union fitting 1/4" to 1/4" #SS-4-UT-6 Cajon tube adapter NPT Male #B-2-MHC-4S for water bath Nupro air actuator valve #SS4-BK-1C Nupro DC powered soledoid valve #MS-sol-1K Cajon flexible tubing 12" #321-4-X-12 Cajon flexible tubing insert #304-4-XBA Cajon female 1/4" NPT Tee #B-4-T Cajon 1/4" NPT male x 1/4" swagelok #B-MB4-TA-1-4-24R Swagelok x 10/32" male #B-400-1-0232
Cole Parmer 7425 N. Oak Park Ave. Chicago, IL 60648	3/8" OD x 1/4" ID Norprene tubing #N-06410-05 Plastic compression quick-disconnects #YA-6360-30 & #YA-6364-05 Finnpippette 5-50ul #J-6247-01
General Supply & Metals 47 Nauset Street New Bedford, MA 02746	1/4" OD copper rod (sample boats; see appendix 4.)
Hallmark Electronics 6 Cook St. Pinehurst Park Billerica, MA 01821	8K Non-volitile ram chips #DS1225Y
Lambda Electronics 515 Broad Hollow Rd. Melville, NY 11746	Power supply 0-40 VDC #LQ-412
Matrix Corp. 1203 New Hope Rd. Raleigh, NC 27610	Stepping Motor control card for STD bus #7911/SSC-5K Unipolar motor driver card #7911/usd RCA/26 cable to connect SSC to USD
Mettler Inst. Corp. Box 71 Hightstown, NJ 08520	#012 bi-directional data interface for AE balance
Microgroup 7 Industrial Park Rd. Medway, MA 02053	304 low carbon seamless s.s. tubing 1/4" OD x .060" ID x .095" wall Military specs .021% carbon
MKS Instruments 6 Shattuck Rd. Andover, MA 01810	Absolute pressure transducer #122AA-00100-AB 0-100 Torr; 1/2" tubulation with standard accuracy plus cable and connector
Newark Electronics Route 1 South Park Walpole, MA 02081	26 pin female edge connector assembly #FCE-26-103 26 pin flat cable connector with polarity bump #S2J026 26 conductor flat laminated cable #36F658WA
Poly Sciences Corp. 7800 Merrimac Ave. Niles, IL 60648-48312	Circulating water bath #1-060-700
Versalogic 3888 Stewart Road Eugene, OR 97402	64K ram memory expansion board #VL-7709A Z-80 processor card for STD bus #VL-7806C C4 Basic/Novos ROM for VL-7806 #2066 STD-Ain-1A analog input card #2370 Power supply card #VL-PSC 8 channel relay output card #VL-IPI-2 Transformer #PSC-2532 9 slot card cage with backplate #VX-09T-MB #8560 26 pin to D8-25S cable #9553 D8-25S to D8-25S cable (for serial connection)
VWR Scientific P.O. Box 232 Boston, MA 02101	Magnetic stirrer #58940-158 1/4" x 7/8" glass stir bar #58948-422 Labindustries repipete, 20 ml #53527-121

Appendix 9. Trouble shooting guide

TROUBLE	POSSIBLE CAUSE
Carousel jams	The carousel spindle must be absolutly perpendicular to the reducing gears. Try using ball bearings of a slightly different size or number below the carousel. A copper disk can also be placed into the depression in the lower carousel assembly to change the height of the ball bearings. The two reducing gears on the carousel spindle are not perfectly aligned. Trial and error will locate . the correct allignment.
Balance won't tare	Try reinitializing the memory first. If the balance still refuses to tare, quit the Automated Carbonate System program and rerun the machine language loader program followed by the ACS program. The balance will now have no trouble taring.
Carousel doesn't stay aligned to hole one	The gears are probably slipping. Tighten.
Standards are low	There is water in the standards. Bake. Standards are being sucked away during the initial pumpdown. Standards 35-5 will be most affected. Try adding more methanol to the boats before running. The acid bath temperature is too low causing CO_2 to remain dissolved in the acid. Raise the water bath temperature.
Standards are high or erratic	Check the regression line by producing a run of standards. The pressure transducer does get dirty and needs to be sent to the factory once a year for cleaning and recalibration. Check to make sure the temperature of the water bath is at the correct setting.
Incomplete reactions	If more than one sample per run does not react fully during the 5 minute reaction time, choose a longer reaction time. Raise the temperature of the water bath to reduce the viscosity of the acid. Increase the stir bar speed.
Acid is thick at the end of the run	Low carbonate samples with lots of clay were probably run and larger weights were used to compensate for the smaller pressure expected. Lower the sample weights to the same range as used for higher carbonate samples.

Appendix 10. System startup.

The following directions allow the user to operate the carbonate system quickly and easily. For more detailed information, refer to the technical manual.

1. Make sure the compressed air source is on and adjusted to 80 psi.

2. Turn on the stepping motor power supply making sure either the (+) or the (−) terminal lead is *not* connected.

3. Once the voltage stabilizes at 28VDC, plug in the unattached lead. If there has been a power failure, the surge protection strip will probably have to be reset.

4. Power up the **NEC** terminal. *ASYNC* should be installed in drive 'A'.

5. Power up the card cage and then push the reset button.

6. The *C4 BASIC* system prompt '*' should be displayed on the screen. If not, push the reset button on the card cage.

7. Type 'DIR' at the prompt to make sure the following is printed:
   1. The Animating Elememt, last revised 30.08.89 (DRO)
   2. RDKEY & METIO loader program, last revised 29.08.89 (DRO)
   3. GETDATA program to produce pressure/time data

8. Type 'run 2 <RET>' (nothing will appear to happen) followed by 'run 1 <RET>'.
   The following should appear:
   Welcome stranger.
   Skip software initialization? (Y/N) : (in most cases type 'N')
   Is the lazy susan homed to slot #1? (Y/N) : (check the alignment)

9. 'The Main Menu' will now be displayed on the screen.

10. Attach the acid filled reaction vessel to the carousel.

11. Turn the circulating water bath on, checking that it is adjusted to 80°C.

12. Turn on the spin bar mixer to a setting of 3.

Appendix 10. System startup continued.

13. Make sure the **ULTRA-TORR** fitting on the carousel is tightened.

14. Turn on the vacuum pump with the toggle switch on the surge protection strip.

15. Go to the 'Tweak the System Menu' and hit 'V' for vacuum. The acid will now begin to outgas as it is heated to temperature under vacuum.

16. Weigh out your dried/crushed sediment samples. Add methanol to each sample and dry in the oven.

17. Load the samples into the carousel. Tighten the **ULTRA-TORR** fitting.

18. Run the samples, choosing the appropriate reaction time.

19. At the end of the run, 'The Main Menu' will be printed on the screen. Make sure there is a floppy disk inserted in drive 'B'.

20. Type '^ VF <RET>' : ('File specification:' will appear on the screen)

21. Type 'B:filename.crb <RET>' : (The file will be copied to drive 'B')

22. Type '^ VG <RET>' : ('port open' will appear on the screen)

23. Hit 'P' and the contents of the carbonate system memory will be printed to the screen one line at a time.

24. When all 40 lines of data have been printed, type '^ Z <RET>'.

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16. Abstract (Limit: 200 words)

We have developed a computer controlled system to measure the calcium carbonate content of sediment samples. A menu driven program controls the analysis of each sample. The system first communicates with a Mettler digital balance to record the weights of the 40 samples which must be loaded into each run. The sample boats are next loaded into the sample carousel which is then sealed from the atmosphere. The system is first pumped down to a vacuum of 0.04 torr. The valve to the pump closes and the stepping motor turns the carousel, moving a sample boat over the delivery slot and dropping the sample into 80°C 100% phosphoric acid under vigorous spinning action. During the reaction, carbonate is evolved into H_2O and CO_2 and the resulting pressure change within the closed system is measured by a pressure transducer and recorded into memory next to the sample identification and sample weight. The system is pumped once again to 0.04 torr and the process continues until all 40 samples have been analyzed. The data can then be uploaded and converted to percent carbonate values using a regression line produced from multiple analyses of varying weights of a 100% carbonate standard. Precision of the system, based upon 120 replicate analysis ranges from 0.49% to 0.88%.

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