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Coulometer Operator's Manual

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GLOSSARY

CCS - Coulometer control system

HP - Hewlett-Packard

MC - Microcontroller

PAR - Princeton Applied Research

TW - Thumbwheel

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MASTER

COULOMETER OPERATOR'S MANUAL

by

A. L. Criscuolo

ABSTRACT

The coulometer control system automates the titration of uranium and plutonium as performed by the CMB-1 group. The system consists of a printer, microcontroller, and coulometer, all of which are controlled by an algorithm stored in the microcontroller read-only memory. This manual describes the titration procedure using the coulometer control system, its theory and maintenance.

1. INTRODUCTION

This manual contains the information necessary to operate and maintain the coulometer control system (CCS). The procedure to conduct constant potential coulometric measurements is first discussed. This is followed by a general discussion of the control algorithm accompanied by flow charts and a program listing. These should provide answers to most of the detailed questions that may arise. Lastly, the information required to maintain the hardware is presented. This information includes wire list, schematics, and references to other manufacturers' documentation.

Figure 1 is a photograph of the CCS installed in its laboratory environment. The three subsystems that comprise the CCS are the Hewlett-Packard (HP) printer, the E-5 8080 microcontroller (MC), and the Princeton Applied Research (PAR) coulometer.

The CCS should not be modified without first consulting with the E-5 Mini-Microcomputer Group, since most changes will have significant consequences.

2. CONSTANT POTENTIAL OPERATION

The constant potential coulometric measurement may be considered to consist of three major phases:

Fig. 1. Coulometer control system in a laboratory environment.

1. Cell preparation
2. Instrumentation setup
3. Automated titration and data printout

The first phase is the same with the CCS and the manual system, so it will not be discussed here. The second phase consists of two procedures. One procedure is for the initial setup of the instrumentation and need only be checked during power turn-on. The second procedure needs to be conducted at the beginning of each measurement cycle; that is, each time a new cell is introduced to the coulometer. The automated titration phase is discussed below in general terms and in more detail in the maintenance section.

2.1 Turn-On Procedure

The turn-on procedure should be followed whenever the system has been left unattended for more than a day. Before turning on the ac power, check the following:

- A. PAR Potentiostat Switch Setup
 - 1. Ext. Trig. Switch - IN
 - 2. Ext. Trig. Mode Switch - Track
 - 3. Operating Mode Switch - Contr. E
 - 4. Ext. Cell/OFF/Dummy Cell Switch - OFF
 - 5. Local/Remote Switch - Remote
 - 6. Voltage/Current Meter Switch - Current
 - 7. Reset Switch - Down
 - 8. Current Range - 100 mA
- B. HP 5150A Printer
 - 1. Print Command Switch (on rear) - LF
 - 2. Rocker Switches 1,2,3,4,5 - OFF
 - 3. Rocker Switches 6,7 - ON
- C. E-5 Microcontroller
 - 1. Continuous/Step Switch - Continuous
 - 2. Reset/Run - Run
- D. Turn on the ac power to the PAR, HP, and E-5 respectively. If manual coulometric measurements are to be done only, it is not necessary to turn on the HP or E-5 electronics. For local measurements, the local/remote switch on the PAR should be set to local.
- E. Each time the ac power is removed from the E-5 electronics, it is necessary to set the time on the clock of the controller. This is accomplished as follows:
 - 1. Set the thumbwheel (TW) to read 003.
 - 2. Push the start switch, then the load switch.
 - 3. Set the TW to correspond with the month, then push the load switch.
 - 4. Set the TW to correspond with the date of the month, push the load switch.
 - 5. Set the TW to correspond with the hour. The hour is based on a 24-hour military day not divided into a.m. and p.m. with zero hour at midnight. Push the load switch.
 - 6. Set the TW to correspond with the minute of the hour, then push the load switch.

2.2 Setup for Each Cell

Each time a new cell is introduced to the CCS, the parameters associated with that cell must be entered into the E-5 controller through the TW switches. The setting and display of these parameters is done as follows.

2.2.1 Setting of Parameters.

- a. Set TW to read 002.
- b. Push start switch, then load switch.
- c. Set TW to a 001 if cell contains plutonium, a 002 if cell contains uranium. Push the load switch.
- d. The mass of the cell sample is entered in thousands of micrograms. A four-digit number is entered, two digits at a time, starting with the most significant digits. For example, a sample weighing 2.139 mg would be entered as 21 (load switch), 39 (load switch).
- e. Next, the number of the run must be entered. This is any two-digit number. Enter OXX on the TW and push the load switch.

2.2.2 Display of Parameters. To verify that the parameters of the cell were entered correctly, they may be displayed on the printer by setting the TW to 000, then pushing the start and load switches respectively.

2.2.3 Set Titration Potentials. The values of potential A and potential B must be set as well as their respective polarity. Potential A switch must be depressed, as this potential is always run first by the controller.

2.2.4 Initiating Coulometric Measurement Cycle. After performing the necessary setup procedures the measurement is initiated by entering a 004 on the TWs, then pushing the start and load switches, respectively. The measurement cycle may end with any one of the following five messages. Messages are read from bottom to top.

- a. Normal Message - The printer will display the following message. The Xs take on the appropriate values. The charge is displayed in coulombs.

CHARGE:+XXX.XXXXXCLB
ELAPSED TIME XX:XX
TITRATION STOPPED
RUN: XX
MASS:XXXXMICROGRAM
ELEMENT:U

TIME XX:XX:XX
DATE XX/XX

TITRATION STARTED

- b. Elapsed-Time Error Message - If the minimum titration current is not attained within 25 minutes of the start of titration, the following message occurs. The "POT" may be either A or B.

ELAPSED TIME 25:00
TITRATION STOPPED
ATTAINED ON POT B
MINIMUM CURRENT NOT

TIME XX:XX:XX
DATE XX/XX

TITRATION STARTED

- c. Overcurrent Error Message: If a titration current in excess of five times the full-scale value occurs, the titration is stopped immediately and the following error message is printed.

ELAPSED TIME XX:XX
TITRATION STOPPED
OVERI OCCURRED
TIME XX:XX:XX
DATE XX/XX
TITRATION STARTED

- d. Overcharge Error Message: If a titration charge in excess of the coulometer range occurs, the titration is stopped immediately and the following message is printed.

ELAPSED TIME XX:XX
TITRATION STOPPED
OVER Q OCCURRED
TIME XX:XX:XX
DATE XX/XX
TITRATION STARTED

- e. Overload-of-Current Error Message. If a current in excess of 1 A occurs for more than 2 s, the titration is stopped immediately and the following message is printed.

ELAPSED TIME XX:XX
TITRATION STOPPED
OVERLOAD OCCURRED
TIME XX:XX:XX
DATE XX/XX
TITRATION STARTED

2.2.5 Time Display. The time of day and date may be printed by entering a 001 on the TW, then pressing the start and load switches respectively.

2.2.6 Termination of Coulometric Measurement. Upon receipt of one of the messages discussed in the previous paragraph, the operator may initiate another measurement cycle after entering the cell parameters and setting the potential values.

2.2.7 Reset Function. The reset switch allows a command to be terminated before it reaches its conclusion. The reset switch may be used to correct data entry errors by terminating the command and then the operator may begin again. The controller will not execute a second command until the completion of its current command. For example, a time-display command will not be executed if a titration measurement is in progress.

If the operator notices a problem with the titration process, he may terminate it by pressing the reset switch.

3. MAINTENANCE

This section will discuss the design philosophy of the CCS for the purpose of providing background information to the maintenance personnel. A system block diagram is discussed to aid in identifying system units, detailed interchassis wire lists are presented, schematics of LASL-built devices are presented or referenced, and flow diagrams of the software, as well as the program listing are discussed to aid in identifying which system unit is malfunctioning.

3.1 System Block Diagram

The CCS consists of three basic units, the PAR Model 179/173 potentiostat/coulometer, the HP5150A thermal printer, and the E-5 MC. The units and their pertinent subunits are illustrated in Fig. 2.

The E-5 MC is the central control unit of the system. Commands are entered through the TW switch to direct the controller. Experiment parameters are also entered through the TWs. The MC controls the display messages on the HP printer. When the "control coulometer" command is given, the MC takes control of the PAR coulometer until completion of the titration.

3.2 Interchassis Wiring

The three system units are interconnected through cables connected to the rear of the MC chassis. The detailed pin/function assignment of these cables is given in Appendix A, Ref. D.1.

3.3 Switch Function and Control Signal Description

A description of each of the switches on the MC is provided in Appendix B. A description of the control signals from the MC, PAR, and HP are included in Appendix B also.

Fig. 2.
System block diagram.

3.4 Schematics

The schematics, which describe the design of the MC modules, are listed in Appendix A. These drawings may be obtained from the E-2 Drafting Section.

3.5 Flow Diagrams and Program Listing

The control algorithm is described in this section through the use of flow diagrams and a program listing. The flow diagrams will act as a road map of the program listing.

The flow diagrams are presented in a sequence of increasing detail. The detailed flow diagrams are presented approximately in the order in which they occur in the program listing. The program listing is presented in Appendix A, Ref. E.

3.5.1 General Flow of Control. The supervisory program flow is illustrated in Fig. 3. The program is entered through a power-on or a reset. After initializing the output ports and program parameters, the MC stays in an idle loop until an interrupt occurs. The MC will service the interrupt and then return to the idle loop.

3.5.2 Thumbwheel Service Routine. The flow chart for the routine, which services the TW command switch, is illustrated in Fig. 4. When the start switch is depressed, an interrupt is generated that causes the MC to read the TW switch and execute the command that corresponds to the number set on the TW. Table I lists the commands and their TW setting.

The first reading from the TW after its interrupt is interpreted as a command code. If subsequent readings are necessary, they are considered data for either the calendar clock or the cell parameters.

Because the routine disables the TW interrupt, it will service only one command at a time. The command will be serviced to completion and then the MC will accept a new command.

3.5.3 Command Routines. The routines, which execute the commands of Table I, are similar in flow. Consequently, only the last command will be discussed. The general flow of the program is illustrated in Fig. 5. The detailed flow is shown in Fig. 6.

3.5.3.1 General Flow. As can be seen in Fig. 5, the controller is pulled out of the wait loop by an interrupt from the start switch. The MC takes the data in from the TW switch, decodes the corresponding command, and executes the command. The command will be executed to completion before the MC will service another command from the TW. The calendar clock may interrupt the MC while it

Fig. 3.
General flow of program.

Fig. 4.
Thumbwheel service routine.

TABLE I
COMMAND/THUMBWHEEL SETTING

<u>Thumbwheel Setting</u>	<u>Command Description</u>
000	Print out the cell parameters.
001	Print out the date and time.
002	Input the cell parameters from the thumbwheel.
003	Input the date and time from the thumbwheel.
004	Perform a coulometric measurement cycle.

is executing a command. However, the operation of the PAR is not affected by this. If one of the PAR "excess" conditions occurs, the appropriate message is printed and the MC reinitializes the CCS.

3.5.3.2 Detail Flow. The detail flow of the MC control, while executing a "control coulometer" command, is illustrated in Figs. 6 and 7. The MC conducts a titration at potential A until the minimum current is achieved, then it conducts the second titration at potential B until the same minimum current is attained. When this current occurs the value of the total charge accumulated is displayed on the printer along with the other parameters associated with the measurement.

If the minimum current is not attained within 25 minutes of the start of titration, the process is halted and an error message is displayed. Also, if any of the "excess interrupts" occur (overload, overcharge, overcurrent), the titration will be stopped and the appropriate error message displayed.

Some points are discussed below that may clarify certain aspects of the control procedure.

- a. The current meter on the PAR does not have the resolution required for the titration measurements, that is, less than 100 μ A. Consequently, when the titration current becomes less than 200 μ A, the MC no longer reads the current meter. Instead it reads the charge meter once per second, subtracts consecutive readings, and uses the result as the average current value for the 1-s interval. In this manner, it is possible to resolve currents as low as 10 μ A when the titration current range switch is set for 100 mA full scale.
- b. The actions taken in the flow diagram boxes D, E, F, and G are executed for potential A, but are not required. However, by including them, the procedure for A and B potentials becomes identical, thus simplifying the software.
- c. The charge meter display is limited to four digits plus exponent, although the electronic counter driving the display has an 18-digit range. Therefore, to prevent the loss of any information, the charge meter is not reset immediately after the titration current reaches 200 μ A. Instead, the MC waits for the least significant digit of the charge meter to be updated before resetting the charge meter. This is illustrated in boxes D, E, and F of the flow diagram.

3.5.4 Subroutine Flow Diagrams. The remainder of the flow diagrams are related to subroutines that are used by the supervisory routine, the TW routine, and/or the command routines. These flow diagrams are presented in Appendix A, Ref. E.1. It is believed that the commented listing and the flow diagrams are adequate to convey an understanding of the control sequence.

Fig. 5.
General flow for command to control coulometer.

Fig. 6.
Detailed flow of control coulometer command.

APPENDIX A

DRAWING LIST

- A. Princeton Applied Research Corp. Model 179/173 C
 - 1. Model 173 - Manual M186B, January 1974.
 - 2. Model 179 - Manual M198, August 1973.
 - 3. "Modification of PAR Coulometer," Group E-5 memo dated January 14, 1977, from A. Criscuolo to D. Jackson; "Modification to PAR for Remote Control," Group E-5 memo dated April 13, 1977 from A. Criscuolo to D. Temer.
- B. HP Printer Model 5150A
 - 1. Manual Number 0515C-90002, September 1974.
- C. E-5 Microcontroller
 - 1. Microcontroller CPU, LASL 4Y-168169.
 - 2. 4K PROM, LASL 4Y-168399.
 - 3. PAR Interface Module, LASL 4Y-168179.
 - 4. Microcontroller Bin, LASL 4Y-168185.
 - 5. E-5 Microcontroller User's Manual, Los Alamos Scientific Laboratory informal report, LA-6826-M.
- D. System Wiring
 - 1. "Interchassis Wire List," Group E-5 memo dated April 21, 1977, from A. Criscuolo to D. Temer.
- E. "Program Listing and Flow Charts," Group E-5 memo dated April 21, 1977, from A. Criscuolo to D. Temer.

APPENDIX B

SWITCH FUNCTION AND CONTROL SIGNAL DESCRIPTION

Signal Description for Coulometer Control

Potentiostat Signals

1. The potentiostat current display is contained at Inport 0 and Inport 1.

2. **Inhibit Trigger:** This control line allows the micro to control the current display. When this line is HI the display will free run as normal. When this line is LO, the display will be updated only on command from the micro. This allows the micro to take stable data from the potentiostat. This line is assigned to Output 0, Bit 3.
3. **External Trigger:** When Inhibit Trigger is LO, the transition of External Trigger from a LO to HI will cause the current display to update. This line is assigned to Output 0, Bit 2.
4. **Update Complete:** This line indicates to the micro that the display has completed its update and valid data are present at Inports 0, 1. This signal is assigned to Inport 3, Bit 0. A LO indicates valid data.
5. **Polarity:** This line indicates the sign of the current display. Assigned to Inport 2, Bit 0. A HI is plus.
6. **Overrange:** A HI indicates the current has exceeded the range setting. Assigned to Inport 2, Bit 1.

7. Potential Trigger: A transition from a HI to a LO selects potential A. A transition from LO to a HI selects potential B. Assigned to Output 0, Bit 1.
8. Potential OFF: A LO removes the potential from the electrodes. This is equivalent to the off position on the dummy/cell slide switch. This line is assigned to Output 0, Bit 0.
9. Overload: When this signal is asserted, the potentiostat is being overloaded. This line is assigned to Interrupt 1, the second highest priority interrupt. This allows immediate entry into a program that will turn off the potentiostat by asserting the Zero E signal.
10. External Cell: A LO applies the selected potential (A or B) to the cell being analyzed. This line is assigned to Output 0, Bit 6.

Coulometer Signals

1. The coulometer display is contained at Inports 248, 249, 250 decimal.

2. Reset: A LO signal from the micro will reset the display. Assigned to Output 0, Bit 4.
3. Print Cmd: A LO signal from the micro will freeze the digital output of the coulometer so that the micro will read stable data. Assigned to Output 0, Bit 5.
4. V-F/Inhibit: A LO signal from the micro will freeze the coulometer display. This signal can be used to control the start of the coulomb count. This signal is not used.
5. Over I: A LO signal from the coulometer indicates that the current range has been exceeded. Assigned to Inport 2, Bit 2 and Intr 6.
6. Over Q: A LO signal from the coulometer indicates that the charge range has been exceeded. Assigned to Inport 2, Bit 3 and Intr 5.

HP Printer Signals

1. The ASCII character code is output from the micro to the printer on Output 1. The code is defined on pp. 2-17 of the HP-5150A printer manual. The port bit assignments are as follows.

The "b" refers to the ASCII chart and are LO true; that is, a 1 on the ASCII chart is a 0 at the output port.

2. DAV: A HI indicates to the printer that data are waiting to be sent. A LO indicates valid data are being sent. Assigned to Out port 0, Bit 7.
3. NRFD: A LO indicates that the printer is not ready to take new data. Assigned to Inport 2, Bit 7.
4. NDAC: A HI indicates that the printer has accepted the new data. Assigned to Inport 2, Bit 6.

Micro Signals

1. Reset: Momentary switch that causes the micro to return to the first Read-Only Memory Address. Also clears all ports and the HP printer.
2. Start: Momentary switch that asserts an interrupt on priority level 7 (lowest level).

3. Continuous: Alternate switch that allows the micro to run continuously when in the down position and halts the micro when in the up position.
4. Advance: Momentary switch that is active when the Continuous switch is up. When the Advance switch is pushed, the micro will execute one machine cycle. This may be one-third of an instruction or one complete instruction.
5. Sync: This LED lights when the micro is running.
6. Load: This momentary switch loads the contents of the TW switches into Inport 251.
7. Valid Data: This signal indicates to the micro that new data have been loaded from the TWs. A LO on Inport 2, Bit 4 indicates new data.
8. Command Switch: These three TW switches provide binary data, in BCD code, to the micro through Inport 251.

9. Real Time Clock Interval: A real time clock is assigned to Interrupt 6 to provide accurate measure of time intervals. The interrupt is automatically reset when the micro accepts it. The frequency of the interrupt is selectable by decode from 1 per 10 μ s to 1 per s. The frequency is selected by a set of switches on the printed circuit board. Only one switch may be on at any time.