Microcomputer-Controlled Langmuir-Blodgett Dipping Trough

The lack of an inexpensive, commercial Langmuir-Blodgett film-deposition trough prompted us to build our own for the deposition of fatty-acid salts and tailor-made photochemically active agents upon a number of substrates. The films are characterized by their pressure-area isotherms, which are recorded by monitoring the film's surface pressure while compressing the surface area. Dipping requires slow immersion and removal of a substrate while continuously maintaining a preset surface pressure. Both of these tasks can be accomplished under microcomputer control.

The software, run on an Apple II, is written in FORTH, allowing full, interactive control of dipping. The Apple controls a dc stepping motor, which compresses the film surface, while simultaneously monitoring a Cahn microbalance which reports surface pressure on a Wilhelmy plate. Pressure-area isotherms, standardized in molecular units of square angstroms, are displayed on the Apple screen and can also be stored or plotted. The deposition - see reverse side -

16 SUPPLEMENTARY NOTATION
Prepared for publication in the Review of Scientific Instruments

19 ABSTRACT (Continue on reverse if necessary and identify by block number)

<table>
<thead>
<tr>
<th>FIELD</th>
<th>GROUP</th>
<th>SUB-GROUP</th>
<th>LANGMUIR-BLODGETT FILMS</th>
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</thead>
</table>

20 DISTRIBUTION/AVAILABILITY OF ABSTRACT
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AD-A173 772
substrate is mounted on a dipping arm driven by an ac motor. When dipping a sample, the Apple maintains the optimal surface pressure (as read from the isotherm), 0-100(±0.1) dyne cm⁻¹ throughout the deposition process, allowing multilayer structures to be built automatically. The dipping rate is variable, allowing for slow deposition of initial layers and proceeding to faster deposition for subsequent layers. Finally, the Apple monitors the change in film area during dipping, to ensure full film uptake.
Microcomputer-Controlled Langmuir-Blodgett Dipping Trough

by

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The trough used for the remaining film studies was milled from a solid piece of teflon, approximately 2 x 12 x 12 inches. The final dimensions of the trough are given in Figure 4.13, as is a schematic of the trough design. The trough was encased in aluminum to prevent distortion of the teflon. Sweeping arms and a compression arm were cut from excess teflon, and encased in aluminum metal to prevent warping. Figure 4.14 shows an expanded view of the final trough setup, including structure dimensions. The compression arm was connected to a stepping motor (Hurst Model #AS-30) to allow forward and reverse motion. A schematic of the dipping
Figure 4.13. Schematic of Langmuir trough. Inner trough dimensions are 7" x 10 1/2" x 1/2 ". Dipping well is 3/4" from one end and 2 1/4" from either side; its dimensions are 2 1/2" x 1" and it extends 1/2" below the trough floor. The entire trough is encased in 1/4" thick aluminum plate metal.
Figure 4.14. Schematic of trough setup. Floors are made of 1/2" thick aluminum plate metal. The bottom floor has dimensions of 12" x 16"; other floors are shown to scale, relative to the bottom floor, in the drawing. Support posts, shown as dashed lines in the drawing, are constructed from 3/4" diameter aluminum rods. The second floor is 3 1/2" above the bottom floor; the third floor is 15 1/2" above the bottom floor. Components of the setup, labeled in the diagram, are: (a) Bottom (motor) floor; (b) Motor block, consisting of stepping motor (not shown) and guiding mechanism for compression arm. The turn screw has 20 turns/inch; (c) Second (trough) floor; (d) Aluminum encased trough; (e) Compression arm which mates to posts on guiding mechanism; (f) Third (balance) floor; (g) Cahn Model 27 automatic electrobalance with external control unit (not shown). A hole is drilled in the third floor metal to allow suspension of the Wilhelmy plate below the balance. The entire setup is encased in a plexiglass case to prevent air drafts and contamination from dust.
arm is shown in Figure 4.15. This arm was connected to a continuous, reversible motor (Japanese Servo Co., Model # RH276P4 connected to a Japanese Servo Co. gearbox Model #6H60) to allow immersion and removal of substrates from the water surface.

Surface pressures were measured using a platinum Wilhelmy plate (obtained from Cahn Instruments); plate dimensions are 2 cm x 1 cm x ~0.005 cm. The Wilhelmy plate was flamed clean before each use and was suspended from a Cahn Model 27 automatic electrobalance, which maintained the plate at a constant immersion depth throughout the experiments. The electrobalance and both motors were interfaced to an Apple IIe computer. In this manner, computer control was available for every step in the deposition process and for obtaining $\tau$-$A$ curves. The computer program for running the trough was written in Forth, and is reproduced in Appendix I. Appendix 2 shows a schematic of the interface between the motors, the electrobalance, and the computer.
Figure 4.15. Schematic of dipping arm. The entire unit stands 14" tall. Square plates, shown in the drawing, are constructed from 1/2" thick aluminum plate metal and are 2 3/8" along each side. Posts, shown as lines in the drawing, are constructed from 1/4" diameter aluminum rods. Components of the dipping arm, labelled in the drawing, are:

(a) Continuous, reversible motor; (b) Gearbox; (c) Turn screw (20 turns/inch);
(d) Limit switches, attached to aluminum blocks mounted on aluminum rod. The limit switches can be positioned through use of set screws in the aluminum block;
(e) Microscope slide holder.
FRONT OF BOX

pilot lamp

DIP CONTROL

SLOW
FAST

A
B

A/B = 36-pin Centronics, female chassis mount
DIP CONTROL = 10kΩ variable resistor
BACK OF BOX

- Female 6-pin Jones plug
- 25-pin D-subminiature
- 36-pin D-subminiature
- 1.25 amp fuse
- 10V AC input
**CONNECTION SCHEME**

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* J= John Bell Engineering, Inc., 32-line digital I/O interface card for Apple II computer
  B= 36-pin Centronics connector, designated as B on the front of the box
  37-D= 37-D subminiature pin
**CONNECTION SCHEME (cont.)**

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16 DIP= 16 pin dual in-line package

GND
INSIDE OF BOX

16 DIP

RELAY

14 PIN

RELAY

14 PIN

HURST

#220001

STEP MOT. CONTROL

14 PIN = 14 Pin wire wrap socket
RELAY = Potter & Brumfield relay #2022
16 DIP = 16-pin dual inline package
1 MΩ variable resistor

JAMECO

# JE 210

POWER SUPPLY

FRONT
CONTROL BOX

Note: R1-2 & Tl-2 may be replaced by isolated, TTL compatible solid-state relays.
\LANGMUIR-BLODGETT DIPPING ROUTINES
\ H. VAN RYSYMK, 30 MAY 85
\ Revised 2 APR 86

\ c/o Prof. A.B. Ellis
\ Department of Chemistry
\ University of Wisconsin-Madison
\ 1101 University Avenue
\ Madison, WI 53706

\ This program coded in
\ Micromotion Masterforth 0.0
\ 12077 Wilshire Blvd, #506
\ Los Angeles, CA 90025
\ (213) 821-4340

\CONSTANTS, VARIABLES, STRINGS, BUFFERS, AND TABLES
50432 CONSTANT SWITCH 49152 CONSTANT KEYBOARD
49168 CONSTANT STROBE 40 CONSTANT BUFSIZE
45400 FCOLUMN MCONST 101.600 FCOLUMN AFLOAT
2VARIABLE PCVR 2VARIABLE CPLATE
2VARIABLE POS 2VARIABLE POSIT
2VARIABLE INC 2VARIABLE FALLOFF
VARIABLE WAIT-TIME VARIABLE TREND
CREATE BUF BUFSIZE ALLOT CREATE BUF2 BUFSIZE ALLOT
CREATE EL 2 ALLOT 1: EL 1: ENDLINE EL 1;

\TABLES
CREATE DIGITS ( I/O PORT TABLE FOR JOHN BELL CARD )
50433, 50560, 50561,

\I/O
: HALT ( HALTS T- AND DIP ARM )
255 SWITCH C!;
: UP ( MOVE DIP ARM UP )
HALT 252 SWITCH C!;
: DOWN ( MOVE DIP ARM DOWN )
HALT 253 SWITCH C!;
: PULSE ( JOES T-ARM ONE PULSE )
SWITCH DUP C8 I OR SWAP OVER OVER C! SWAP B OR SWAP
3 0 DO LOOP ( WAIT ) C!;
: FLOP SWITCH DUP C8 4 OR SWAP C! ( FLIPS DIP DIR. );

\CONVERSION
: VFT ( VFT MS -- ON PULSES )
FABS PCVR C6 F9 F11;
: DMM ( DMMS -- ON PULSES )
FLOAT MCONST C6 INT;
: DYNM ( P VOLUME )
FINV C6 FP CPLATE C6 C9 FNEGATE WTCLEAN F8 F+;
: GTC ( P VOLUME )
FINV C6 FP FNEGATE WTCLEAN F8 C6 C9 C6;
: READLN CR BUF BUFSIZE EXPECT BUF BUFSIZE CPACK ;
: GETFP ( FP )
READLN BUF2 DUP DUP C0 + OVER C! DUP
C8 + 32 SWAP C! ( ADD SPACE ) FNNUMBER FDUP DO = SWAP 0 AND
ABORT* USE SCIENTIFIC NOTATION * ;
SCRI 10 SCRI 7

\ CONVERSION & I/O MACROS
: MM S>D DMN ( INTEGER MM TO DN PULSES ) ;

: AREA-OUT ( DN PULSES --- FP MM\^2 )
  FLOAT AFNORM FP MCONST F ;

: WAIT ( TIMING LOOP ) 8000 0 DO LOOP ;

: GETDN READLN BUF2 COUNT VAL DROP ( -- DN ) ;

: POSITION ( SETS ARM POSITION VARIABLE )
  CR " ARM POSITION (IN INTEGER MM'S) " GETDN
  DMN POSIT 2! CR ;

: SHOWSCALE ( DISPLAY CURRENT CMM READING )
  READSCALE 4 9 F.R ;

SCRI 6

\ T-ARM & DIPPING ARM MACROS

\ T-ARM MOVEMENT ( DP -- DM )
: EQUALIZE ( EP'S TO FP, LEAVES DN PULSES TRAVELED )
  READSCALE F- DUP DP= SNAP 0= AND IF FDROP 0 S>D
  ELSE FDUP FO< IF VECTOR 2DUP COMpress DNEGATE
  ELSE VECTOR 2DUP EXPAND THEN
  THEN ;

: DIP
BEGIN TARGET FP= EQUALIZE DABS 20. D< WAIT UNTIL 0 S>D
DOWN BEGIN FINISHED NOT UNTIL DOWN
BEGIN TARGET FP= EQUALIZE D- WAIT FINISHED UNTIL
HALT " DELTA AREA = " AREA-OUT
3 & F.R " MM\^2 " ;

SCRI 8

\ T-ARM MOVEMENT ( DM -- -- )
1 : EXPAND ( MOVE T-ARM OUT DN PULSES )
2 : 2DUP POSIT 2E D= 2DUP Num2 2E
3 : D< IF POSIT 2!
4 : ELSE DDROP DDROP
5 : " RANGE EXCEEDED. " ABORT THEN
6 : BEGIN 2DUP DO= NOT WHILE FINISHED IF HALT THEN
7 : PULSE 1 S>D D- REPEAT DDROP ;
8 : COMPRESS ( MOVE T-ARM IN DN PULSES )
9 : FLOP ( SELECT DIRECTION )
10 : 2DUP POSIT 2E 2SNAP D- Num1 2E D= VM SWAP DROP IF 2DDROP
11 : " RANGE EXCEEDED. " ABORT ELSE 2DUP POSIT 2E 2SNAP D- POSIT ;
12 : THEN BEGIN 2DUP DO= NOT WHILE FINISHED IF HALT FLOP THEN
13 : PULSE 1 S>D D- REPEAT
14 : FLOP 2DDROP ;

SCRI 9

\ CLEANING ( SETS CLEAN WATER WEIGHT VARIABLE )
CR " WEIGHT FROM CLEAN SURFACE ( IN MB'S SCI NOT )? "
GETFP WTCLEAN F! CR ;

: DEPOSIT
CR " DESIRED SURFACE PRESSURE IN SCI. NOT. (Dynes/CN)? "
GETFP DYMETONS TARGET F! CR
" NUMBER OF COMPLETE CYCLES (INTEGER)? " GETDN DROP
1+ HOME 1 DO 1 . . " DIP DOWN, " DIP WAIT UP WAIT
BEGIN FINISHED NOT UNTIL HALT CR " UP, "
UNDIP WAIT DOWN WAIT BEGIN
FINISHED NOT UNTIL HALT CR
LOOP 3 0 DO 7 EMIT LOOP ." DONE!" CR ;

SCRI 10

: CLEANWATER ( SETS CLEAN WATER WEIGHT VARIABLE )
CR " WEIGHT FROM CLEAN SURFACE ( IN MB'S SCI NOT )? "
GETFP WTCLEAN F! CR ;

: DEPOSIT
CR " DESIRED SURFACE PRESSURE IN SCI. NOT. (Dynes/CN)? "
GETFP DYMETONS TARGET F! CR
" NUMBER OF COMPLETE CYCLES (INTEGER)? " GETDN DROP
1+ HOME 1 DO 1 . . " DIP DOWN, " DIP WAIT UP WAIT
BEGIN FINISHED NOT UNTIL HALT CR " UP, "
UNDIP WAIT DOWN WAIT BEGIN
FINISHED NOT UNTIL HALT CR
LOOP 3 0 DO 7 EMIT LOOP ." DONE!" CR ;

SCRI 11

\ DATA TRANSFERAL PRIMITIVES
1 : ARRAY ( # OF CELLS, CELL BYTES --- ) ( N "ELEMENT )
2 : CREATE DUP ; ALLOT
3 : DOES) DUP @ ROT + + 2+ ; 150 6 ARRAY PRESSURE
4 : GRAPHICS
5 : PLINT ( PLOTS PINS VS. POSIT, DF --- )
6 : POSIT 2E AREA-OUT FII 6200. D- DROP
7 : SWAP PRESSURE F! 1E2 F! FIX DROP PLOT ;
SCRI 12
\ PRIMITIVES
: KEYCHECK KEYBOARD C@ 128 > DUP IF 0 STROBE C THEN ( -- F ) ;

: (PA) 9 S)D BEGIN I. D+ 2OVER COMPRESS
WAIT-TIME @ 0 DO WAIT LOOP READSCALE MTTYDYNE
FDUP 7 PICK DUP >R PRESSURE F! R> STORE? ( QUERRY, --- F ) CR " FILE NAME? " READLN 2 DR@ BUF2 COUNT TEXT MAKE IS OUTPL
STORE? IF INC 20 STOREDATA THEN TX
DO YOU WANT TO SAVE THIS RUN? .
RECORD PRESSURE-AREA CURVES 0 PA'S, CONT.
WHERE? ( REPORTS TENSION ARM POSITION )
POSITION 20 FLOAT MCONST F/ 1 4 F.R." MM." ;

SCRI 13
\ DATA STORAGE
: STORE? ( QUERRY, --- F ) CR " DO YOU WANT TO SAVE THIS RUN? "
READLN BUF2 1+ C@ DUP ASCII Y = )R ASCII Y = R) OR ;
STOREDATA ( DN --- , WHERE N IS THE T-ARM INCREMENT )
CR " FILE NAME? " READLN 2 DR@ BUF2 COUNT TEXT MAKE IS OUTPL
" DATA TRANSFER IN PROGRESS... " CR
0 PRESSURE F# 2EO F# WRITEOUT
PO 20 0 PRESSURE F# FIX DROP
I+ I DO
2DUP AREA-OUT WRITEOUT
2OVER D+
I PRESSURE F# WRITEOUT
LOOP OUTPUT CLOSE 2DROP 2DROP ;

SCRI 14
\ RECORD PRESSURE-AREA CURVES
: PA ( --- N, WHERE N IS THE T-ARM INCREMENT )
HOME," P-A CURVES..." CR CR .
INCREMENT (INT MM'S)? " GETDN DMM 2DUP INC 2!
PLOT-INIT POSIT 20 PO 2!
10 0 DO 2DUP COMPRESS WAIT-TIME @ 0 DO WAIT LOOP
( LET SCALE SETTLE ) READSCALE MTTYDYNE
I PRESSURE F! I PLOT-IT LOOP
" PRESS ANY KEY TO STOP." CR
0 FALLOFF (PA) FLOAT G PRESSURE F! CR
STORE? IF DNEGATE STOREDATA ELSE 2DROP THEN TI;

SCRI 15
\ PA'S, CONT.
: PA-REVERSE ( REVERSES PA CURVE, --- ) POSIT 20 PO 2!
HOME 0 20 AT 6R " P-A REVERSE... "
0 PRESSURE F# FIX DROP I+ I DO
INC 20 EXPAND WAIT-TIME @ 0 DO WAIT LOOP
READSCALE MTTYDYNE I PRESSURE F! I PLOT-IT
LOOP
STORE? IF INC 20 STOREDATA THEN TI;

SCRI 16
\ MACRO MOVES
: WHERE? ( REPORTS TENSION ARM POSITION )
POSITION 20 FLOAT MCONST F/ 1 4 F.R." MM." ;

: TOP ( MOVE DIP ARM TO TOP OF RANGE, THEN OFFSET )
FINISHED NOT IF UP BEGIN FINISHED UNTIL HALT WAIT
DOWN BEGIN FINISHED NOT UNTIL HALT THEN ;

: BOTTOM ( AS WITH TOP... )
FINISHED NOT IF DOWN BEGIN FINISHED UNTIL HALT WAIT
UP BEGIN FINISHED NOT UNTIL HALT THEN ;

SCRI 17
\ SYSTEM REPORTS
: STATUS HOME
.D. TRANSVERSE ARM IS AT " WHERE? CR
.D. WEIGHT FROM CLEAN SURFACE IS WTCLEAN F# F. " MS." CR
.D. TREND IS " TREND @ . " POINTS." CR
.D. WAIT-TIME IS " WAIT-TIME @ . " SEC." CR
SWITCH C@ DUP 3 AND 0 = IF " DIPPING ARM UP.
ELSE 2 AND 0 = IF " DIPPING ARM DOWN.
ELSE " DIPPING ARM AT REST." THEN THEN CR CR ;
\ INITIALIZATION

: STARTUP BOCOL

127 SWITCH 2+ C!  255 SWITCH C!
30418. NUM1 2!  99436. NUM2 2!  (ABSOLUTE PULSE @'S)
10E0 PCVRT F!  0.491E0 CPLATE F!
3 TREND!  6 WAIT-TIME!

*. TURN BOX ON...* CR CR
CLEANNATER

POSITION:
Dictionary of Terms

**Position** enters the correct position of the arm into the computer.

**Where?** tells where the computer thinks the compression arm is. (Position is read from leading teflon edge of the arm, towards the dipping well.)

**Cleanwater** inputs changes in the surface pressure of the clean, film free, water surface.

**Up** moves the dipping arm up (Must be stopped with the **Halt** command) Use carefully!

**Down** moves arm down (Must be stopped with the **Halt** command) Use carefully!

**Halt** stops the motion of the dipping arm in the up or down mode.

**Top** moves the dipping arm to top of the range and then offsets.

**Bottom** moves the dipping arm to the bottom of the range and then offsets.

**Status** lists the status of the system.

- Transverse arm (in motion or at rest)
- Weight from clean surface
- Trend is
- Wait-tim
- Dipping arm (in motion up/down or at rest)

**X MM Compress (or) Expand** will move the compression arm the desired mm's.

**PA** will measure the Pi-A curve. It will ask for intervals. 1mm is best to get accurate results. Program stops after recording three consecutive points where the surface pressure decreased.

**Pa-Reverse** runs a backward Pi-A curve to examine the extent of collapse and hysteresis. Unfortunately, there is often transfer of film to the plate which prevents a real measurement of the surface pressure on the reverse.

**Storedata** stores the PA curve if the program should be unwilling to let you do so automatically.
Deposit will deposit films on the slide. It will ask for pressure of transfer and for the number of dipping cycles desired. It is best to have the surface pressure close to that desired for dipping to prevent the computer from over stepping the condensed phase. There are times when the deposit cycle believes it has made several dips when in reality it has only done one. This can be seen in the area changes listed for each dip. (An area change of 0 would indicate no dip was made.) Also, changes in the dipping arm contacts are possible to vary the time for the cycles. Greater separation of the contacts gives longer times to allow the film to dry, closer distances give shorter times so each dip accomplished faster.

Dip is a sub command of Deposit and brings the slide down while maintaining the constant surface pressure given in the deposit command.

Undip is a sub command of the deposit routine. It brings the slide up while maintaining the constant surface pressure given in the deposit command. This is particularly useful if the switch fails, as it often does on the slow first dip.

XEN MGTODYNE F. converts the scale measurement of mg to the surface pressure in dynes. The number must be in scientific notation 147=1.47E2=147E0 either of the last two are acceptable.

XEN DYNETOMG F. converts the surface pressure in dynes to a force in mg readable from the scale. Again, this must be in scientific notation.

Variables that are variable!
In order to see these, type "status" which will respond with the present value for each of these (not Cplate):

Trend is the number of consecutive points which have decreasing surface pressures which the PA curve uses as the signal for collapse of the film. Not as important now that the PA can be stopped from the key-board after the first six points.

Wait-time is the time the computer waits between 1mm steps. This allows variation of the compression rate (approx. 3A2/molecule/minute).

Cplate is the inverse of the perimeter of the Wilhelmy plate, needed only with a new plate.

To Change a Variable:    # VariableName !(CR) integer
                        # VariableName F!(CR) floating pt. number