NIMA TECHNOLOGY

Trough OS5 for WIN95/98/NT. Quick Installations and Operating Guide

Installation from Disk: Insert disk 1 into drive a and select Run from Windows Start Panel. Enter a:\setup.exe in the Open box (as prompted) and the software will be loaded into c:\nima or the directory of your choice. The Install Wizard will take over - just click on the 'finish' button to start (Microsoft Logic!).

If you have problems, such as 'unable to find disk 2', please copy the 3 files 'setup.exe', 'data.001' and 'data.002' into the 'c:\windows\temp' directory and run 'setup.exe' from this directory, using the 'Run' command.

Installation from the Website: Click on the 'Software' button in the margin on the left side of our home page. The instructions for downloading are given on the screen. Very briefly, these consist of:

1. Download - just click on 'download' and the 'zipped' file (2MB) is loaded onto your PC.
2. Unzip - using WinZip, unzip the file into its 3 constituents.

   A link to www.winzip.com is given, if you don't have this program.
3. Setup - as above - 'Installing from disk'.

A shortcut to the Nima.exe will be displayed in a Nima group file window, together with the Nima icon. Please use the right hand mouse button to click on the shortcut file to change its properties and select the 'trough.ico' icon.

Running: Make sure that all the files are in the same directory. These are 'Nima5xx.exe', 'sensors.cal', 'install.dat', 'standard.con' and 'serpdrv'. Double click on Nima5xx.exe to run it. After a brief introduction, a pressure-area isotherm will be displayed with a Menu Bar along the top.

Setting up the Interface: Before anything else, please click on 'Calibrations' and 'Interface Test'. This will launch the Interface program, with which you can test that the PC is communicating with the interface unit (IU).

Select the port you think you are using (if in doubt, start with port 1) and enable the interface. If the Data Reply String and the Input Values are flickering in a realistic, business-like manner and the V2 light on the interface illuminates, then communications have been established successfully. If nothing happens, increase the port no to port 2. Usually, PCs use port 1 or 2 for the external serial port connections. If you still have trouble you can try port 3 or 4, but it is unlikely they are being used. Take care, if your PC is not configured for port 3 or 4, the program may hang up and you will have to press CTRL-ALT and DELETE to start again!

If you continue to have problems, please check that there is not a clash with other serial devices, such as mouse drivers and networks.

Once communications have been established, click on Quit and confirm that you wish to save the calibration file (sensors.cal), so that the next time you launch the program, the correct serial port will be addressed immediately.

Calibrations Click on the 'Calibrations' menu (or hit the f6 key) and then click on the following:

Calibrate Pressure Sensor: This takes you through a routine where you attach the empty weighing pan to the pressure sensor, take a reading and then add the known calibration weight and take another reading. Make sure you have the correct calibration weight and plate perimeter entered before you finish. This will ensure that your sensor is correctly calibrated in mN/m. To check the calibration of the sensor, leave the weight in the pan, zero the pressure sensor reading (click on the red ZERO next to the PS display) and then remove the weight. For a 100mg weight and 21.0mm perimeter (10.5mm wide, 0.25mm thick), a change of 46.7mN/m should be observed.

Teach Barrier Positions: Use this function to teach the PC the minimum and maximum allowed positions of the barrier. Follow the instructions on the screen - you will be prompted to close the barrier, enter the minimum area and then open the barrier and enter the maximum area. Use the slide control to change the barrier speed and the Stop/Continue Motors button to enable/disable the barrier motor. Take care not to crash the barrier(s), as this routine has no fail safes!

Calibrate Barrier Speeds: Use this only when the barrier positions have been taught! Otherwise, the barrier(s) may crash. This routine is fully automatic. Just ensure that the barrier actually moves when being moved at the slowest speeed. Increase the Min Barrier Speed from the default value of 50, until you can see the position indicator change. For greatest accuracy, run this routine twice, if you have changed the Min Barrier Speed during calibration.

Calibrate Dipper Speed: Enter the dipper stroke and factor before commencing this calibration. These values are to be found on your calibration certificate. Standard dippers have a stroke of 75mm and factor of 3.84, mini dippers have a stroke of 25mm and factor of 8.28. The dipper routine is then automatic and, as for the barrier, the Minimum Speed may be changed. Remember to run the routine twice, if you do change the minimum speed.

Operation

Graph (f2) - the data can be displayed as a pressure-area isotherm, pressure & area v time or surface tension v immersion depth. Select the appropriate display form the Graph Dialog Box. The axes scaling may be changed by clicking on the minimum or maximum axes labels, or by using the attached tool-box at the top right hand corner of the graph. The auto-scaling feature is also very useful - click anywhere in the graph area with the right hand mouse button.
Barrier Menu (f3) Selects the barrier mode you require: Choose between Monolayer Menu, Open, Close, Constant Speed or Pressure Control, Isotherm cycle and Coupled Mode.
Dipper Menu (f4) - Selects the dipper mode you require - Choose between Dipper Menu, Up, Down or Programmed Dip, Creep Up and Creep Down.
Run time variables (f7 - top left of screen) - allows important values to be changed before or during a run. Choose between Target Pressure, Feedback Gain, Dipper Top End and Dipper Bottom End.
Taking an Isotherm - Enter the barrier your speed you require (left hand side of screen), choose Isotherm from Barrier Menu and keep Dipper at Stop. Zero pressure sensor by clicking on Zero button. Barrier moves at desired speed and data is displayed on graph.
Dipping: Enter target pressure, layers required, dipper top and bottom end in Dipper Menu. Then choose Barrier - Pressure Control and once at target pressure, choose Dipper - Program Dip.
Saving / Loading (File - f1) - Saves and loads data as ASCII files with *.txt extension. These can be imported into most spreadsheets.
Prining - press ALT-PrintScreen on your keyboard and graph will be copied to clipboard. Paste into other applications - graph will be pasted as it appears on the screen.

Operation

Tensiometer customers can now proceed to the 'Tensiometry' chapter - where its operation is explained in similar detail.

There are one click buttons for basic functions like 'opening' and 'closing' the barrier. These are:

O	**Open barrier**
C	**Close barrier**
D	**Dipper down**
U	**Dipper up**
STOP	**Stop all motors**
CLEAR	**Clear data from memory**
F	**Dipper forwards (alternate layer troughs only)**
B	**Dipper backwards (alternate layer troughs only)**

Other commands are embedded in the menus along the top of the screen:

File (f1) - this gives access to the **Save, Load** and **Setup Menu.** Also to **Exit** the program.

Graph (f2) - the data can be displayed as a **pressure-area isotherm, pressure & area v time** or **immersion depth-area**. Select the appropriate display form the Graph Menu. Axes are auto-scaled but may be manually re-scaled by right clicking anywhere on the graph and then typing over axes labels

Barrier Menu (f3) Selects the barrier mode you require: Choose between **Monolayer Menu, Open, Close, Isotherm** or **Pressure (Pi) Control, Isotherm cycle** and **Coupled Mode.**

Dipper Menu (f4) - Selects the dipper mode you require - Choose between **Dipper Menu, Up, Down (Forwards** or **Backwards** for alternate layer dipper) or **Programmed Dip.**

Run time variables (f7 - top left of screen) - allows important values to be changed before or during a run. Choose between **Target Pressure, Feedback Gain, Immerse To, Raise To, Cycle Max Area** and **Cycle Min Area.**

The **STOP** button can also be accessed from the **'ESC'** key.

Typical operating sequences for taking a pressure-area isotherm and depositing an LB multi-layer are given overleaf.

A typical pressure-area isotherm

The following steps are recommended to take an isotherm:

1. Clean the trough thoroughly with a Kimwipe (soaked in chloroform) and the plastic gloves provided.

2. Make up a solution of 1mg/ml arachidic acid in chloroform.

3. Fill the trough with clean water.

4. Close the barrier(s) by clicking on 'C'.
   - the barrier(s) will move to their 'close-to' position.

5. Aspirate the surface of the water enclosed by the barrier(s).
   - keep your eye on the pressure reading.

6. Open the barrier(s) by clicking on 'O'.
   -the barriers will move to their 'open-to' position.

7. Zero the pressure sensor by clicking on 'Z'
   - the current surface tension will be defined as zero surface pressure i.e. 0mN/m.

8. Take an isotherm without molecules by clicking on 'Barrier' and 'Isotherm'
   - the barriers will close and the isotherm will be displayed on the screen.
   If it the trough is clean, the curve will be horizontal on the X-axis. If there are surfactants on the surface, a small rise in pressure will be observed at smaller areas.
   Please note that the axes are set to 'auto-scaling' and adapt themselves to the data recorded.

9. Clean the surface again with the aspirator.

10. Zero the pressure sensor.
    Whenever some water has been sucked out of the trough, it will have to be re-zeroed.

11. Open the barriers and spread the solution from the micro-litre syringe.
    Wait for the pressure to return to '0' - this indicates that the solvent has evaporated.
    If the pressure does not return to zero, you have spread too much and will have to remove some monolayer with the pump!

12. Take the pressure-area isotherm
    - you should now see a good pressure area isotherm of arachidic acid on water.

13 Stop the barrier by clicking on 'STOP'
   If you do not, the barrier will continue until it passes its 'close to' area and then a message saying 'barrier too closed' will appear. The barrier will then be stopped automatically.

Laboratory Requirements

Safety

Before using any solvent, familiarise yourself with the relevant safety procedures. Solvents must never be inhaled or touch your bare skin. Work in a fume cupboard when making up the solutions. Similarly, when making up solutions of metal ions such as cadmium and potassium, always wash your hands afterwards, to prevent accidental ingestion.

Check with your laboratory supervisor on safety regulations and correct disposal after use. Remember you are responsible for your own and other's safety.

General

When studying mono-molecular layers which contain at most a few milligrams of material, even a small amount of contaminant can cause serious errors.

The trough, substrates and other apparatus must be kept as clean as possible. Extraneous greasy material concentrates at the air-water interface; the source of such contamination is usually grease from fingers or hair, this being transferred to the subphase by supposedly clean instruments which have been handled. Clean forceps or tweezers should always be used when handling substrates and for all operations it is advisable to wear disposable, powder-free polythene gloves. It is recommended that the syringes used for deposition are cleaned in between use and that they are rinsed at least once with the solution to be deposited. Clean laboratory coats should be worn, not only for protection but also to prevent the deposition of fibres from clothing onto the subphase.

When not in use the trough should be kept empty of water, covered and out of direct sunlight. If being moved, the pressure sensors and dipper mechanism should be removed from the trough to prevent accidental damage.

Lab environment

As with all aspects of Langmuir-Blodgettry, cleanliness is of the utmost importance. Airborne particulates tend to accumulate at the water surface. To combat this, the water surface should be cleaned thoroughly using the water suction pump and the trough should be situated in as clean and dust-free environment as possible. When the trough is filled with water the clear covers should be in place over the water surface and dipper mechanism to protect the trough from the vagaries of the atmosphere.

Particular care must be taken with Langmuir-Blodgett films which are to be used for optical or electronic applications, as dust particles can become incorporated into the films and give rise to light scattering, pinhole defects and ambiguous results. Many troughs are housed in clean room facilities which maintain a positive pressure and filter the air intake through micropore filters. Surfaces are usually sealed and temperature and humidity are controlled. An isolated laminar flow cabinet can also be used as an alternative. However, such facilities

are not essential if care is taken to operate the trough with regular cleaning in a relatively dust-free laboratory atmosphere. The room in which the trough is situated must be adequately ventilated to ensure that solvent fumes do not accumulate in dangerous quantities.

Excesses of temperature and humidity should be avoided, as should locating the trough in the close proximity to vibrating machinery as such vibrations can cause premature collapse of monolayer films. An anti-vibration table is useful if problems with obvious ripples from external disturbances can be discerned. However, small vibrations do not appear to cause serious problems and have been known to enhance film quality by 'annealing' the surface monolayer.

Trough

The Nima troughs are fabricated from Polytetrafluoroethylene (PTFE), a material which will not contaminate the subphase. The polymer is essentially chemically inert, will not leach plasticiser and is the most hydrophobic polymer known. PTFE can be subjected to very rigorous cleaning procedures.

In general, the best way to keep the trough clean is to use it regularly and to avoid unnecessary collapse of mono-molecular films. The tissues used to wipe the trough must be surfactant-free, such as the 'Kimwipes' supplied (type 7105, available from Kimberly-Clarke). Some other tissues, even if they are 'guaranteed 100% clean' may contain amphiphiles that prevent formation of pure monolayers.

Before and after use the trough should be thoroughly cleaned. Any leftover monolayer material should be removed from the subphase surface before the trough is emptied. To ensure that all parts of the trough are cleaned, the dipping head and barriers should be removed from the main part of the trough first. The barriers can be removed and wiped with a solvent laden tissue.

Organic solvents are the best available degreasing agents and will work over a wide range of solubilities and a thorough wipe with a chloroform laden Kimwipe cleans the PTFE well. Care must be taken to wipe off all excess chloroform, as pools of $CHCl_3$ will evaporate leaving surfactants behind. **Take great care not inhale any chloroform and always wear gloves. If your lab supervisor forbids the use of chloroform, use isopropyl alcohol instead.**

If serious contamination is suspected and solvent cleaning appears insufficient, cleaning with sodium hydroxide solution is recommended: The trough should be filled with a 0.1M solution of sodium hydroxide at approximately 50°C, soaked for about half an hour and then thoroughly rinsed with clean water after emptying.

Chemicals

The materials which are deposited should be as pure as possible. Very small quantities of impurities, especially surface active contaminants, can radically alter the molecular area calculated from the isotherm and affect the material's film forming characteristics.

To avoid contamination and hence improve results, impurities should be restricted during synthesis. Care should not only be taken with the purification of materials but also with the solvents and starting materials used, which should be of the highest available purity.

Once prepared, materials should be carefully stored and handled to prevent hydrolysis, oxidation or contamination. Ideally, samples should be kept in a clean, dry, sealable, inert vessel, preferably with a glass lid as there can be contamination from plasticisers in plastic materials. Non-contaminating methods should be devised for the weighing out and transfer of materials. Care should be taken to ensure that spatulas are kept clean.

Water

The Nima troughs have different capacities between 40 and 1500ml. This small volume reduces the demand of deionised water, reduces the risk of contamination and eases cleaning.

The water used in a Langmuir-Blodgett trough should be as pure as is possible. The quantity of particles, ions, and surface-active materials should be kept to such a low level that they will not significantly affect the surface properties of monolayer systems.

In the past, water was usually distilled a number of times, sometimes with an oxidative stage using an alkaline permanganate solution. Nowadays, reverse osmosis, ultra-filtration, and ion exchange resins are commonly used. A charcoal filter is usually located on the feed line to the still. For consistent results any of the commercially available deionisers are recommended. (Manufacturers include Barnsted, Elga, Millipore & others.)

The water should be fresh every day and discarded if the molecule which is being deposited is changed. Storage should be in clean vessels which are inert; some plastic containers leach dialkyl phtalate esters which are used as plasticisers in their manufacture.

The prime criterion for establishing the cleanliness of the subphase is a high value of surface tension. The surface tension of pure, clean water at 293K is 72.8mN/m. A minimal change should be observed on aging and insignificant changes should be seen on compression of the surface. It should be noted that the value will decrease with an increase in temperature. The surface tension of the water in the trough can easily be measured by lifting the Wilhelmy plate out of the water after having zeroed the pressure sensor. To protect the water surface from air borne particulates the transparent lid should be kept on the trough whenever possible when the trough is full of water.

Solvents

Some Langmuir-film forming materials, such as vegetable oils, spread spontaneously on a water surface. However most materials must be deposited onto the subphase in a solution of a volatile solvent. The solvent which is chosen is critical and will affect the film forming properties of the solute. A number of factors must be considered:

Firstly a solvent must be chosen which has sufficient solvent power to dissolve a substantial quantity of the material under investigation. A solution with much less than 0.1 mg/ml will require too large a volume to be deposited to form a reasonable area of compressed monolayer.

The solvent must also be chemically inert with respect to the material under study and should also be relatively pure. Small amounts of contaminants such as grease can be removed by distilling the solvent in grease-free glass systems. The cleanliness of the solvent can be verified by spreading the solvent neat on the subphase, allowing it to evaporate and then running an isotherm of the remaining surface material. Any increase in surface pressure observed will be due to surface active contaminants in the solvent which need to be removed. This technique is known as 'blank spreading' and, as many commercially available solvents contain film forming contaminants, is highly recommended.

The volatility of the solvent must also be considered - it must be such that the evaporation time is short but not so short that the concentration of the solution cannot be determined due to evaporation. The boiling point of the solvent should lie in or near the range 40-80°C. Important factors to consider are the hazards associated with organic solvents - when allowing the solvent to evaporate the solvent vapours should not be inhaled.

Organic solvents which are very soluble in water should be avoided as they will tend to carry amphiphilic material into the subphase and precipitate it out. If a water-miscible solvent is used, a high concentration of the material to be studied must be used to counteract the effect of the material being pulled into the subphase. Care must be taken when interpreting molecular areas obtained with such solvents as they will usually be incorrect. If a material is not soluble in non-polar, volatile spreading solvents, an acetone-hydrocarbon (e.g. hexane) mixture can be used to increase the amount of solute which will dissolve without introducing serious solubility problems.

The table below shows the properties of a few solvents which have been used for depositing Langmuir film forming materials. Solubilities in water are at 25°C, in g/Kg.

Solvent	*Melting point (°C)*	*Boiling point (°C)*	*solubility in water*
n hexane	-94	69	0.01
Cyclohexane	6.5	81	0.07
Choloform	-64	61	8
Diethyl ether	-116	35	75
Acetone	-93.4	56	∞
Dichloromethane	-98	40	1.3

Table 3.3: *Properties of solvents*

Producing an isotherm

It is now time to run the software and turn the interface ON. Therefore, click on your trough icon, or, if it is not your desktop, click on the Nima5XX.exe file in c:\Nima (or whichever directory you have nstalled it into). The main screen of the software is illustrated below.

In order to produce an isotherm, the water subphase must first be clean. The molecules have to be spread on the surface and the solvent is left to evaporate. They are then slowly compressed and the resulting pressure-area isotherm displayed.

The following sections will lead you through this operation one step at a time. The software commands are printed in **bold** down the left margin with an adjacent explanatory paragraph. *Additional comments are printed in italics.*

In case of emergencies, click on the STOP button or hit the Esc key!

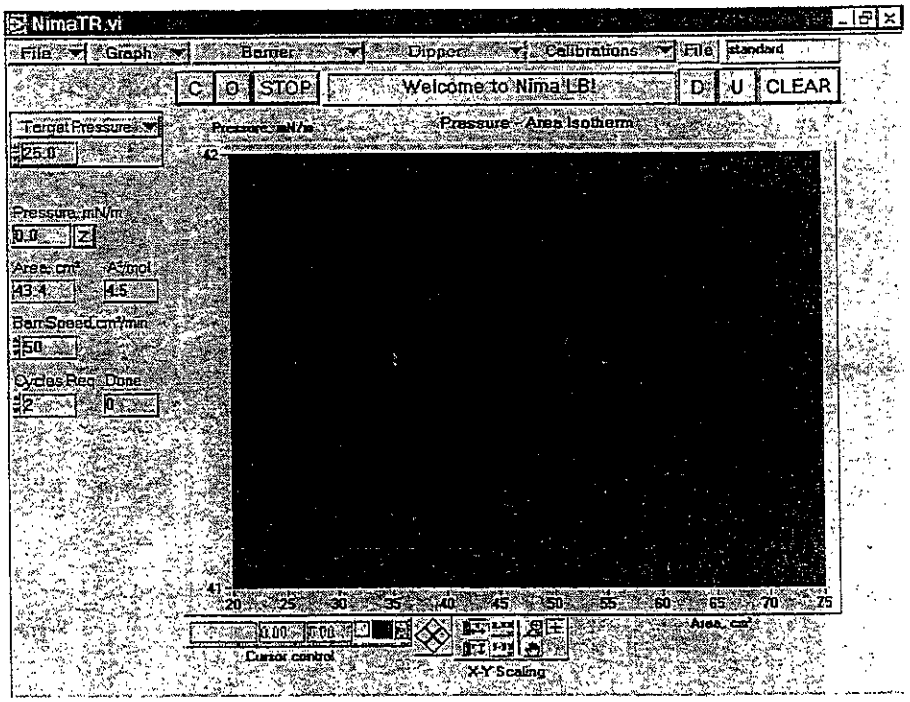

Figure 4.1: Main Screen of Nima Software

Substrate

The quality of the first layer deposited onto the substrate determines the deposition behaviour of subsequent layers - defects in the first layer can be passed through to and observed in layers subsequently deposited. Because the substrate determines the nature of the first layer deposited, it must be thoroughly cleaned.

At all stages of the cleaning procedure care should be taken not to touch the slides with fingers as the skin grease contains a mixture of surface active biological lipids.

Substrates can be divided into two groups according to their surface - they can be either hydrophilic or hydrophobic. A standard glass microscope slide or a polished silicon wafer make excellent substrates for deposition of LB films. Both are normally hydrophilic, the silicon always has an oxide layer after exposure to air.

Many substrate cleaning procedures are commonly employed. One which has been found to work well is to wash the slides with detergent, then rinse in an ultrasonic bath in deionised water and then with isopropanol. Instead of using an ultrasonic bath, Soxhlet cleaning can be used. Argon plasma cleaning (100W incident power at 13.6MHz and 0.1 Torr for 2 minutes) just prior to deposition also gives an excellent surface.

Strong oxidising agents such as acidic peroxide or chromic acid are not recommended - as they are a health hazard and require the substrate to be thoroughly rinsed post immersion.

A hydrophilic surface is prepared by soaking the clean slides overnight in a $2g/dm^3$ solution of sodium hydroxide. They are then rinsed with ultra-pure water and dried in a stream of dry nitrogen.

A hydrophobic surface is obtained by standing the clean slide overnight in a fume cupboard, in a beaker containing a few drops of hexamethyl disilazane and covered with a watchglass. On evaporation, the hexamethyl disilazane condenses onto the clean slide, rendering it hydrophobic. Evaporation of gold will also make a slide hydrophobic. Katherine Blodgett also used the technique of rubbing in molten ferric stearate (see the 'Tutorial' section) which works well and is not as hazardous to health as the other techniques.

Glass is not the only material which can be use as a substrate. Many other surfaces such as fused silica and silicon wafers can also be used. When infra-red studies of Langmuir-Blodgett multilayers are to be undertaken, calcium fluoride plates which are effectively insoluble in water and transparent to infra-red radiation can be used. All these materials can be prepared in the same manner as glass.

After preparation the slides should be stored in a clean dust-free sealable box.

Subphase cleaning

Ensure that the trough has been cleaned and filled with pure DI water.
Attach a paper Wilhelmy plate to the pressure sensor with the 'S' shaped hooks provided and lower it into the water, so that about 2mm of it is immersed.

O Open the barrier(s) by clicking on the 'O' button.

Z Zero the pressure sensor.
Check that a reading of about 70 mN/m is obtained when the Wilhelmy plate is lifted out of the water. If it does not, check the change in surface pressure for the 100mg calibration weight by adding the calibration pan and then the weight. This change should be 48.3mN/m. If it does not, recalibrate the pressure sensor as described in the 'Software' section. If the reading seems to drift initially, this is because the paper plate is still absorbing water.

Barrier Speed 100 Set the barrier speed to 100 cm^2/min by entering '100' in the 'Barrier Speed' box.
To save time, you can set it to larger values, say 250, or even 500 for larger troughs.

Barrier Isotherm Select 'Isotherm' from the 'Barrier' dialog box to start the isotherm. The barrier will move at the given speed until either the space bar is pressed or the barrier travels outside its 'allowed' position (this is set in the Monolayer Menu and prevents accidental damage to the trough.)

Examine the isotherm produced. It should be almost flat except for a slight 'tail' at small areas due to the presence of dirt on the water surface.

Turn on the aspirator pump and touch the pipette to the water surface to suck off any floating material, such as dust and amphiphilic contaminants. If the pipette end is positioned correctly, there will be a louder sucking noise as air is sucked up with water. Move the head of the pipette around to collect all the material and clean for 30-60 seconds.

O Open the barrier(s) by clicking on 'O'.

Z Re-zero the pressure sensor.
This is necessary because the water level in the trough has now changed. Make sure that the lower edge of the Wilhelmy plate is still touching the water surface.

Keep cleaning until the isotherm produced is completely flat. The change in surface pressure on compression should be less than 0.5mN/m. For a quicker check on cleanliness, simply close the barriers ('C') and watch the change in pressure sensor reading.

If you have sucked off too much water, please refill the trough until the level is again about 2mm above the rim of the PTFE. When refilling, always add the water behind the barrier i.e. the other side from where the monolayer is to be spread. This will ensure that any surfactants present in the water will rise to the surface in the unused compartment behind the barrier. Of course, the water should be clean, but you can't be too careful!

Monolayer Menu

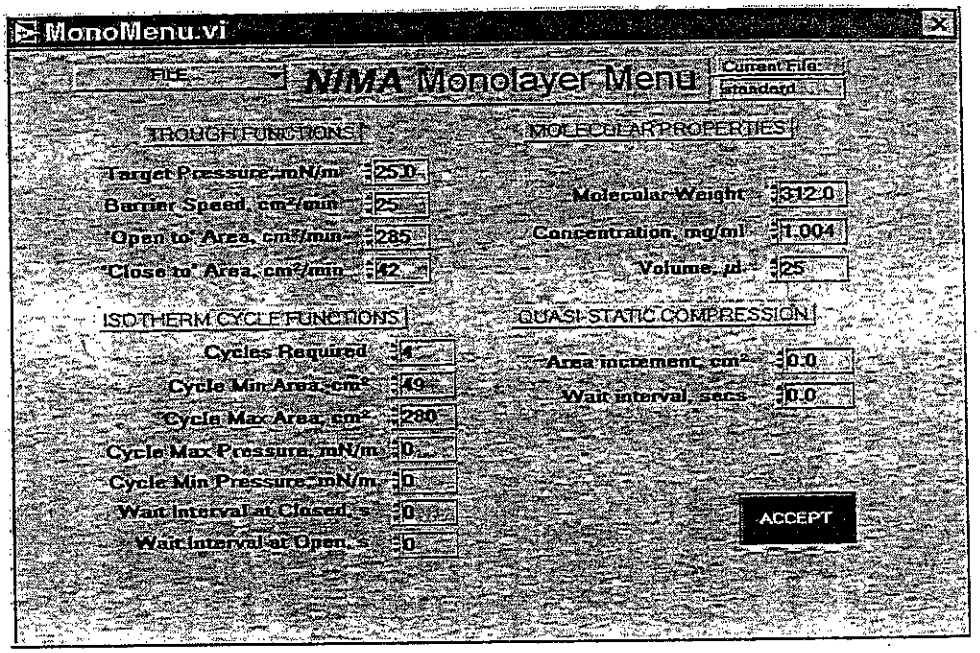

Figure 4.2: Monolayer Menu

To ensure that the area axis (in A^2 / molecule) is calibrated correctly, go to the Monolayer Menu and enter the correct molecular weight, concentration and volume of your solution.

Barrier Mono Menu Select 'Monolayer Menu' from the 'Barrier' menu.

MW Enter the molecular weight of your molecule.

Conc Enter the concentration of your solution.

Vol Enter the volume of your solution in µl.

The other variables can be left for now. They are fully explained in the 'Software' chapter.

Spreading

Now that the conditions have been set, the molecules can be spread on the subphase.

- Rinse out the syringe by drawing up and expelling a little chloroform.
- Draw up 50µl of the arachidic acid solution into the syringe and deposit it drop by drop onto the water from just above the surface.

  There is no need to deposit on different places on the trough when using arachidic acid - the molecules will spread out to cover the complete area available. With other molecules, particularly those with large dipole moments, this may not be the case and 'islands' can form.

- Leave the solvent to e vaporate.

  All the solvent must evaporate but Katherine Blodgett found that if it was given too long, the deposited films were streaky. The evaporation time does appear to have some bearing on film quality - you may wish to devote your PhD to investigate this unexplained observation! However, just wait until the pressure reading returns to zero - that will do for now.

Compression

Barrier Speed 100 — Set the barrier speed to 100 cm^2/min by entering '100' in the 'Barrier Speed' box. This is a reasonable compromise between a good isotherm (i.e. undistorted by dynamic effects) and time available. At this speed, the barrier takes about 5 minutes to close for a standard size trough.

CLEAR — Clear the memory and the graphs by clicking on 'CLEAR'. You will be asked to confirm this clear, as once cleared, all the data is irretrievably lost!
This will clear the data stored when the subphase was being cleaned. It is good practice to clear the data before taking an isotherm, to prevent the graph getting cluttered up with unnecessary data from previous runs.

Graph cm^2<>A^2 — Switch the area axis between absolute area (cm^2) and area per-molecule ($Å^2$) as calculated from the data in the Monolayer Menu.

Barrier Isotherm — Start the compression by clicking on 'Isotherm' in the 'Barrier' dialog box.
Watch the isotherm on the screen and compare it to Figure 4.3. It should show three distinct regions - an initial flat region, corresponding to a 'gaseous' phase of the molecules on the water, a gently rising liquid-expanded or 'liquid' phase and a steeply rising, liquid-condensed or 'solid' phase.

STOP — When the isotherm begins to collapse, click on STOP to stop the compression.
At this point, the steep upward rise of pressure will begin level off. Physically, the monolayer can no longer support the force applied and the molecules move over each other to form aggregates on the surface, or are dissolved in the subphase. With highly coloured films, this is clearly visible. Remember that a surface pressure of 50mN/m is equivalent to 200 atmospheres pressure over the molecular length of 25Å !

Barrier Speed -100 Change the barrier speed to -100 cm²/min. A negative speed corresponds to the barrier moving backwards.

Barrier Isotherm Expand the floating monolayer by clicking on 'Isotherm' again.
The isotherm should have exactly the same shape as when the film was compressed but will be shifted towards smaller areas (i.e. to the left of the graph) by an amount equal to the area of collapsed film.

Barrier Pi control Clean off the surface.
Use the suction pump with its teflon tip to clean the molecules off the surface as previously described. Cleaning shouldn't take more than a minute or so.

Target Pressure 25 Select 'Target Pressure' from the 'Variables' dialog box and adjust it to 25mN/m.
This will compress the barriers, keeping the pressure at 25mN/m as you remove the molecules, ensuring that the monolayer is not collapse.

Well done - you have now taken your first pressure-area isotherm!

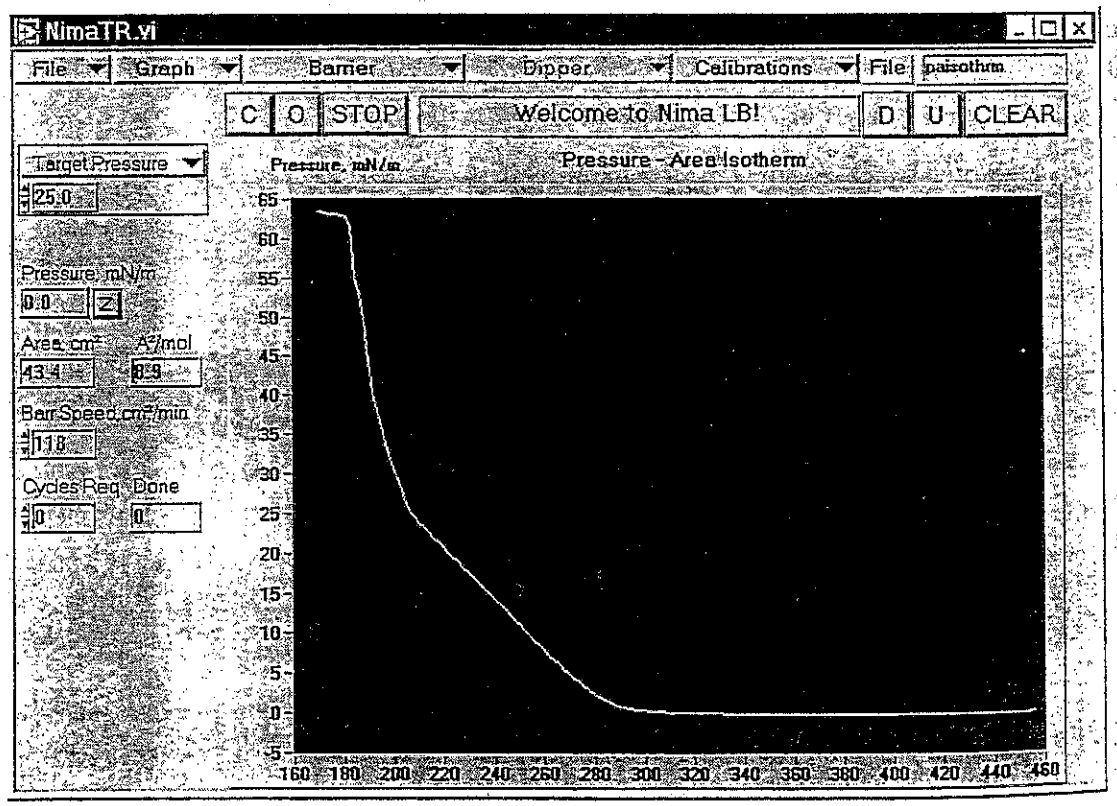

Figure 4.3: Typical isotherm of fatty acid

Loading, saving and plotting

File Save Store the isotherm, by clicking on 'Save' in the 'File' dialog box.
*Enter a name (such as 'test1') and the data will be saved as a *.txt file (i.e. test1.txt) along with its operating conditions which are saved as a *.con file. The data file can be loaded into Excel (or other spreadsheets) and the data will be displayed in columns, each headed by a label to signify what it is. It can be charted and pasted into your reports or papers.*

Graph P&A-T Click on 'P&A-Time' on the 'Graph' dialog box to view two separate plots of pressure-time and area-time.

Cursor control Click on the cursor control palette (bottom left of the graph).
There is a white square which activates the cursor controls when clicked. You can move the cursor anywhere on the screen and read off the X and Y co-ordinates. Click on the little padlock icon and select 'lock on plot' to move the cursor along the actual curve.

Right Click Right click the mouse while the mouse pointer is over the graph. Simple options will be given such as 'X-axis: autoscaling enable / disable'. The default value is always enabled. If you disable auto-scaling, then you can set the scaling of the axes by writing over the minimum and maximum abscissa values.

File Load Load an isotherm, by clicking on 'Load' in the 'File' dialog box.
Click on a previously stored file to load the data back onto the screen.

Further investigations

Repeat the isotherm procedure two or three times, varying the amount of material used, solvent evaporation time, compression speed etc. to try to improve upon your first isotherm and get a feel for the technique.

You might like also to change the concentration of Zinc sulphate in the subphase. This will alter the pressure at which the liquid-expanded changes to the liquid-condensed phase. The greater the concentration of $ZnSO_4$, the lower the pressure of the phase change, the liquid expanded phase disappearing altogether at about 10^{-3}M concentration.

Introduction

This section is written for Langmuir trough users. While some parts are applicable to tensiometer users, these are advised to proceed directly to the 'Tensiometer' chapter.

The operating software is organised as a graphics screen (the 'active screen') and a series of menus. All the 'action' takes place in the graphics screen in one of several axes such as 'pressure-area' or 'pressure & area - time', as in Figure 6.1 illustrated below.

In order to issue a command in the active screen, a single click is required such as clicking on the 'C' button for closing the barriers, or on the 'O' button for opening them. Sometimes a subsidiary choice is also given, such as 'Are you sure you want to clear all the data' when the 'CLEAR' button has been pressed.

Other functions are available from the 'menu' bar running along the top of the screen. These menus are placed into logical groups such as 'File', 'Barrier', 'Dipper' etc.

Powering-up

Make sure that all the relevant files are in the same directory. These are 'Nima5xx.exe', 'sensors.cal', 'install.dat', 'standard.con' and 'serpdrv'. Double click on Nima5xx.exe to run it (or its short-cut, if it's already on the desktop). OK the introduction window and a graph will be displayed with a menu bar running along the top, as in the illustration below:

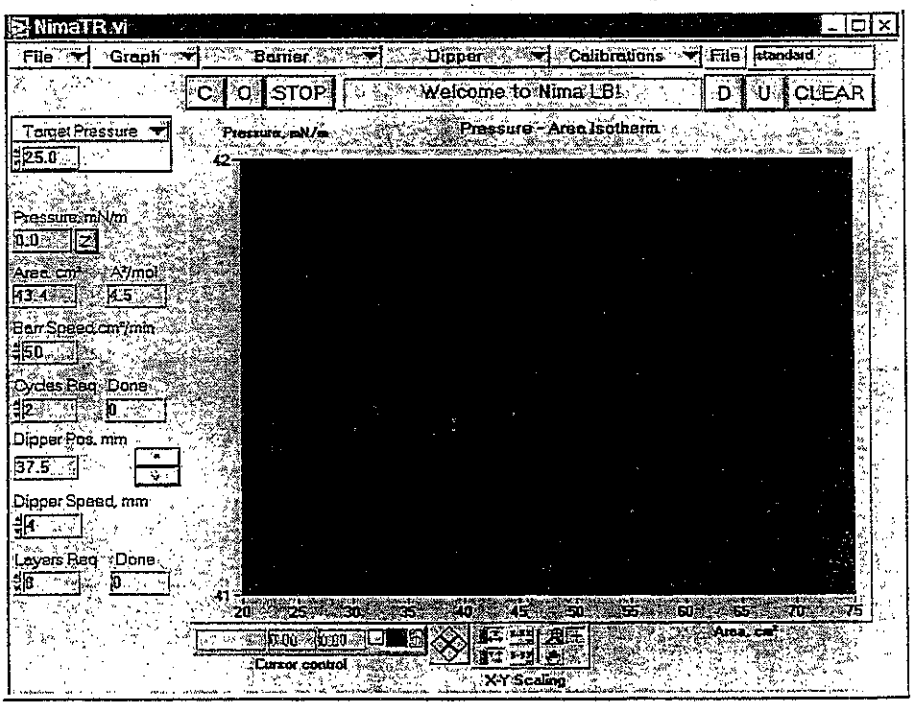

Figure 6.1: Main Screen of Nima Software

The active screen

The active graphics screen appears at power up. The main part of the screen is taken up by a graph. Then there is a menu bar running along the top with a series of 'drop down' menus. In the top right hand corner the most recently loaded file name is displayed - initially this is the 'standard' file. To the left of the graph is a margin displaying various sensor readings. Displayed data include such sensor readings as surface pressure and area.

Just below the menu bar are a series of large buttons. These are for single, often used, functions such as opening the barrier ('O') or moving the dipper up ('U') or down ('D'). The same functions can be accessed via the menu bar. For instance to close the barrier, the 'Barrier' menu is selected and then 'Close' is clicked.

The Graph Menu gives a choice of the different graphs available, such as 'Pressure-Area' or 'Pressure & Area - Time'. It also allows change of scale of the area axes between cm^2 and $Å^2$.

The Barrier Menu gives access to all the barrier functions such as 'Open' and 'Isotherm'.

The Dipper Menu gives access to all the dipper functions such as 'Dipper Up' and Program Dip'.

The Calibrations Menu gives access to all the Calibrations of the different motors and sensors.

The Variables Menu in the box at the top left of the screen allows access to less often changed variables, such as 'Target Pressure' or 'Cycle Max Area'. They can be changed by clicking on their 'up' or 'down' arrows or typing in the new value.

For a step by step guide of actual trough operation, please see the Tutorial chapter.

The following pages give the detailed contents of each menu.

Menu Bar

'File' Menu

Load	Loads previously stored data
Save	Saves current data - give file name
Print	Gives print instructions - SHIFT-PrtScreen
Setup	Setup Menu - explained in detail later in this chapter
Signature	Nima address - in case you want to get in touch
Exit	Exit the program

'Graph' Menu

Pressure-Area	Selects pressure-area isotherm axes
P&A - Time	Selects pressure and area - time axes
Dipper-Area	Selects dipper-area axes (for deposition troughs only)
Transfer Ratio	Selects Transfer Ratio axes (for deposition troughs only)
Change Axes	Changes area axes between cm^2 and $Å^2$/molecule
cm^2<>$Å^2$	(to values entered in Monolayer Menu)
Clear graphs	Clears all data in memory and from graphs

'Barrier' Menu

Monolayer Menu	Selects Monolayer Menu - explained in detail later in this chapter
Open	Moves barrier(s) to 'open to' area
Close	Moves barrier(s) to 'close to' area
Isotherm	Starts barrier moving and data logging
Pi-Control	Compresses barriers until target pressure is reached
Isocycle	Starts barrier moving under iso-cycle (see Monolayer Menu)
Coupled	Moves barriers in coupled mode (MC option only)
Stop	Stops all motors

'Dipper' Menu

Dipper Menu	Selects Dipper Menu - explained in detail later in this chapter
Dipper Up	Moves dipper to top (or clockwise for alternate dipper)
Dipper Down	Moves dipper to bottom (or anti-clockwise for alternate dipper)
Program Dip	Starts pre-programmed dipping sequence
Dipper Stop	Stops all motors

'Calibrations' Menu

Interface Test	Runs interface diagnostic routine - also selects serial port
Cali Pressure Sensor	Self
Cali Potential Sensor	evident
Teach Barr Pos	- all explained
Cali Barr Speed	later in
Cali Dip Speed	this chapter!
Show Cali Data	Displays current area and speed calibrations.

'Variables' Menu This menu changes the current variable available in the little box at the top left of the screen. It is useful to be able to change these 'secondary' variables without having to stop and enter a full menu.

Buttons and Variables

Buttons

C Closes barrier(s) to the 'close-to' area defined in the Monolayer Menu.

O Opens barrier(s) to the 'open-to' area defined in the Monolayer Menu.

STOP **Stops all motors**
The STOP function can also be accessed with the 'ESC' key.

D Down *(conventional dipper only)* moves dipper to bottom.

U Up *(conventional dipper only)* moves dipper to top.

F Forwards *(alternate dipper only)* moves dipper anti-clockwise.

B Backwards *(alternate dipper only)* moves dipper clockwise.

CLEAR Clears all data in memory and from graphs.

A-B A-B switch (alternate and multi-compartment troughs only)
Switches action between A & B (when zeroing, opening/closing or displaying data).

SYM Symmetry button (2 independent barriers only)
Switches between symmetrical and non-symmetrical compression of the barriers.

Z Zeroes pressure sensor (if AB switch present, then Z will zero either sensor A or B).

Permanently Displayed Variables

Barrier Speed
Sets the barrier speed for isotherms. The range is approximately 5cm^2/min to 1000cm^2/min depending on trough type (and gearbox ratio) and can be negative for expansions, annealing, hysteresis loops etc. The barrier speed can also be set in the Monolayer Menu.

Cycles Required **Done**
Sets the number of isotherm cycles required (only when iso-cycle function is selected in the Monolayer Menu). 'Done' displays the number already completed.

Dipper Speed
Sets the dipper speed for depositions. The range is approximately 0.5mm/min to 50mm/min depending on dipper type (and gearbox ratio). The dipper speed can also be set in the Dipper Menu.

Layers Required **Done**
Sets the number of layers required when dipping - can also be set in the Dipper Menu. 'Done' displays the number already deposited.

Monolayer Menu

The Monolayer Menu is accessed from the 'Barrier' Menu and is shown below:

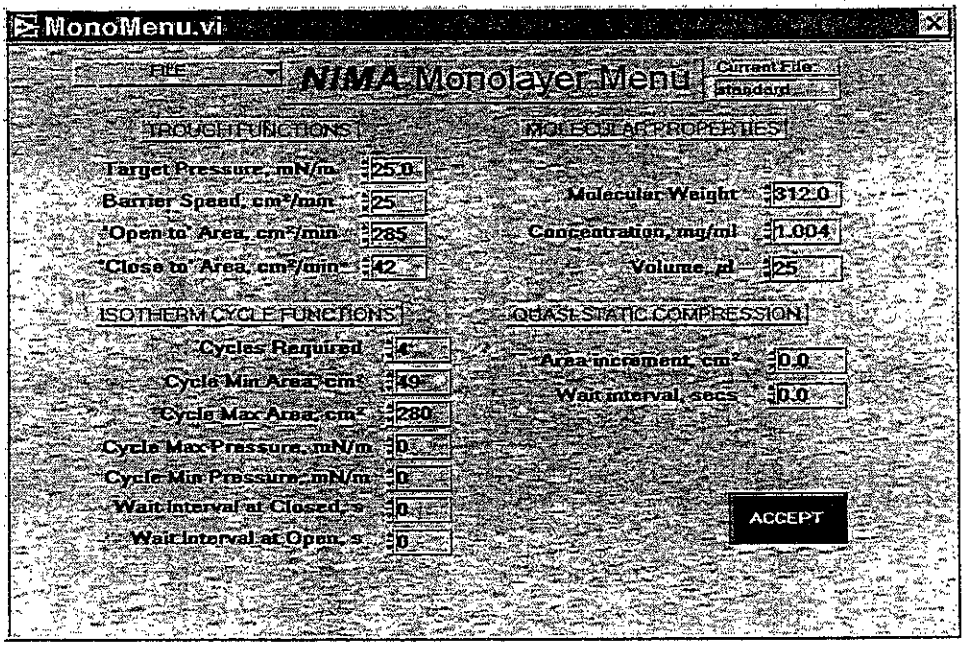

Figure 6.3: Monolayer Menu

This screen allows you to set up your isotherm run to your preferences. For instance you may wish to take a single isotherm or perform isotherm cycles. You may wish to move the barrier at a constant speed or in a 'quasi-static' way.

Entry of the molecular weight of your molecules, the concentration and volume of solution spread enables the software to calculate the absolute number of molecules spread on the subphase and hence convert the area on the subphase surface to an area per molecule.

TROUGH FUNCTIONS

Barrier speed The barrier speed of a compression run when taking an isotherm. Note that if you want to do a 'relaxation' (opening the barrier), please enter a negative barrier speed in the active screen.

Target pressure This the pressure that the film will be compressed to under pressure control ('Pi control' in the Barrier menu).

'Close to' area This is the minimum 'allowed' area. If your barrier moves to a smaller area, it will be stopped and 'barrier too closed' will be displayed. Click on 'O' to re-open it.

'Open to' area This is the maximum 'allowed' area. If your barrier moves to a larger area, it will be stopped and 'barrier too open' will be displayed. Simply click on 'C' to close it.

MOLECULAR PROPERTIES

Molecular weight Enter the MW of your molecule

Concentration, mg/ml Enter the concentration of your solution in mg/ml

Volume, µl Enter the volume of your solution in µl

Once the above data are entered, the computer can automatically label the area axes in area per molecule. MW and concentration are only printed to 3 decimal places but can be entered and are stored with greater accuracy.

A-B Switch between compartments *(alternate layer mode only)*

For an alternate layer trough (Model 622), molecule and solvent data are accessed one compartment at a time. Use this switch to select between compartment 'A' and compartment 'B'.

ISOTHERM CYCLE FUNCTIONS

It is possible to program the barrier(s) to compress and expand the monolayer repeatedly to observe hysteresis effects in isotherms. A choice of boundary conditions and the number of cycles is given. It is possible to cycle between defined areas (in cm^2) or defined pressures (mN/m) or between one of each. Please note that the barrier will reverse on the first parameter it meets, so you have to set both area and pressure parameters for the successful functioning of this routine.

Example 1

If you wish to cycle through the 'liquid-expanded' phase of a fatty acid, set the limit for minimum pressure to '0' mN/m and the limit for maximum pressure to about '30' mN/m. The area limits should then be set at points well outside the areas corresponding to these pressures, i.e. minimum area of 50 cm^2 and a maximum area of , say, 500 cm^2 (if you are using a 600 cm^2 trough).

Example 2

If you wish to cycle between the gaseous phase and the liquid condensed phase, typical values would be:

Max area: 500 cm^2 (for a 600cm^2 trough).
Minimum π: -2 mN/m - better than 0 mN/m, as the pressure reading can on occasions drop below zero.
Min area: 50 cm^2 (or any low area value)
Maximum π: 45 mN/m - or whichever pressure you choose in the liquid condensed phase.

The barrier will then cycle between the max area and the max π limits.

Note - do not set the iso-cycle mode when dipping. Pressure will not be held constant and monolayer transfer will be inhomogeneous.

Calibrations Menu

All motors and sensors are calibrated at Nima before despatch. Once calibrated, it is unlikely that the motors and potentiometers (position sensors) need to be calibrated again. However, it is good practice to check the calibration of the pressure sensor(s) before a series of runs, particularly if the data is to be published.

A pressure sensor can be quickly checked by attaching the weighing pan and checking the indicated reading in mN/m for the calibration weight in the active screen (remember that for a 10mm wide, 0.15mm thick Wilhelmy plate, 100mg is equivalent to -48.3mN/m.

Note that any software updates supplied will require calibration before use, unless you 'park' your old calibration files ('sensors.cal', 'install.dat' and 'standard.con') in another directory before installing the new version.

The following options are given in the 'Calibrations Menu':

Interface Test
Calibrate Pressure Sensor
Calibrate Potenial Sensor
Teach Barrier Positions
Calibrate Barrier Speed
Calibrate Dipper Speed
Show Calibration Data

Interface Test

This displays the 'raw' data as sent by the interface to your PC. It is also possible to move the motors 'manually' in this window. ***But take care - there are no safety stops and you can crash your barrier / dipper and you may cause damage to the gearbox.***

The most likely use you will have of this window is setting the 'serial port number'. Select the port you think you are using (if in doubt, start with port 1) and enable the interface. If the Data Reply String and the Input Values are flickering in a realistic, businesslike manner and the V2 light on the interface illuminates, then communications have been established successfully. If nothing happens, increase the port no to port 2. Usually, PCs use port 1 or 2 for the external serial port connections. If you still have trouble you can try port 3 or 4, but it is unlikely they are being used. Take care, if your PC is not configured for port 3 or 4, the program may hang up and you will have to press CTRL-ALT and DELETE to start again!

If you continue to have problems, please check that there is not a clash with other serial devices, such as mouse drivers and networks.

Once communications have been established, click on Quit. The next time you launch the program, the correct serial port will be addressed immediately.

Calibrate Pressure Sensor

The readings of the pressure sensor are recorded with the empty weighing pan and with the calibration weight in the pan. These are then automatically stored on disc.

Follow the instructions given on the screen; the 100mg calibration weight (the small metal tab) and pan are to be found in the monolayer kit. Ensure that the correct calibration weight and plate perimeter are entered in the appropriate boxes. It is good practice to check the calibration weight on a 6 figure balance and to check the dimensions of your plate with vernier calipers.

Insertion or removal of a motor or sensor lead produces a slight change in the (stabilised) voltage supply to the pressure sensors, so that their output may alter. Ensure that readings are taken with all leads connected to the interface unit.

Calibrate (Surface) Potential Sensor

The surface potential probe is plugged into the 'PB' socket at the back of the interface - pin 2 is GND and pin 4 the signal.

The surface potential option is selected in the Set up Menu. The software then guides you to bias the probe at two voltages 2.0V and 1.0V and the equivalent reading of this stored.

Teach Barrier Positions

The 'minimum area' and 'maximum area' positions of the barrier have to be 'taught' to the PC. This is done by 'manually' moving the barrier to the correct position and then confirming the position and equivalent area of that position.

The barrier is moved with a slider control provided on the screen. There is also a STOP / CONTINUE button to instantly stop the barrier.

There are 4 different configurations for the various troughs made by Nima:

1. Standard - single barrier (Models 601A, 611M, 611D and 622)
2. 2 mechanically coupled barriers working symmetrically (Models 601M, 601A/2B, 611M/2B, 611D/2B)
3. 2 independent barriers working symmetrically but independently (Model 632/D1L)
4. MC - 2 barriers with the ability to cross the centre line (Models 601/MC or 611/MC)

These are explained in detail overleaf.

Teach Barrier Positions (continued)

1. Single barrier

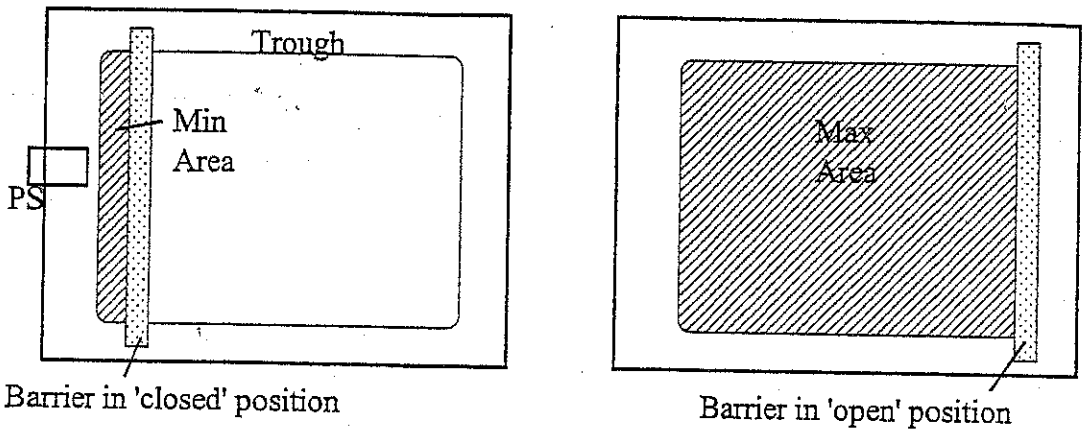

Figure 6.7: *Teaching areas for single barrier troughs*

The minimum area is the area at which the barrier is almost completely 'closed' - that is when it is nearest the pressure sensor ('PS' in the above diagram). It is good practice not to run the barrier to the end, so that when the minimum area is calculated, the curvature of the corners of the trough can be taken into consideration, thus giving a completely accurate area calibration.

To calculate the area, simply take an accurate ruler and measure the length x width. Please note that standard troughs have exact widths of whole numbers like 10 or 20cm. Most troughs have rounded corners of curvature of radius of 1cm. So ½.(4 - πr^2) (= 0.43cm^2) has to be deducted!

The maximum area is that area where the barrier is furthest away from the pressure sensor and its trailing edge is just touching the edge of the trough. Again, measure the length x width less the area of the rounded corners.

Please note in the case of 622 troughs with 2 barriers and 2 separate compartments, this procedure is done twice, first for compartment A and then B (which has to use the same areas).

Calibrating barrier speed

1. Select 'Calibrate Barrier Speed' from 'Calibrations' on the Menu Bar.

2. Click on Start and the calibration will run automatically.

Please ensure that the barrier is actually, physically moving when 'moving at the slowest speed'. So, when the software displays 'Moving Barrier at Slowest Speed', check that you can see the position indicator actually decrease (slowly!). If it's not changing, increase the 'Min Barrier Speed' from the default value of 50, until you can see it change. For greatest accuracy, run this routine twice, if you have changed the Min Speed during calibration, so that this speed is measured over the full 60 second calibration time.

Calibrating dipper speed

1. Select 'Calibrate Dipper Speed' from 'Calibrations' on the Menu Bar.

2. Check that the correct dipper stroke and dip factor have been entered. Typical values are given below and will be written in your calibration certificate:

Dipper type	Stroke	Dip factor
mini	25mm	8.28
standard	75mm	3.84

3. Click on Start and the calibration will run automatically.

Please ensure that the dipper is actually, physically moving when 'moving at the slowest speed'. So, when the software displays 'Moving Dipper at Slowest Speed', check that you can see the position indicator actually decrease (slowly!). If it's not changing, increase the 'Min Dipper Speed' from the default value of 40, until you can see it change. For greatest accuracy, run this routine twice, if you have changed the Min Speed during calibration, so that this speed is measured over the full 60 second calibration time.

Pressure sensor, PS4

This pressure sensor was developed by Dr I.R.Peterson and introduced in September 1995. It uses a taut-band meter movement which gives very low friction. The design of the electronic circuit allows infinite loop gain giving zero compliance i.e. whatever the load, the Wilhelmy plate remains at the same level. Hence it is ideal for surface tension measurements.

However, as a result of the small forces being measured, some of the component parts are delicate and can be bent out of shape. This technical note aims to help the user repair the sensor without having to return it to the manufacturer.

Mechanical set up

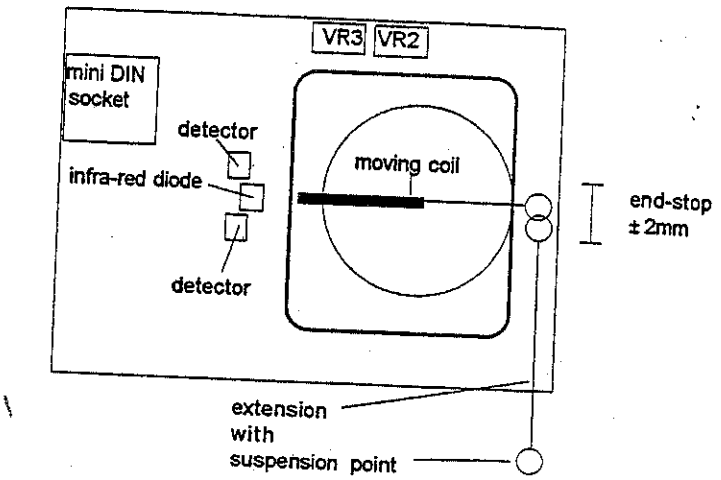

Figure A12: Schematic of PS4.

The sensor should be set up as in the diagram above.

For proper operation the following points shoud be observed:

1. The moving coil must be able to move freely.
2. The extension and suspension point must not touch the inside of the housing.
3. The moving coil should reach the horizontal position when the sensor is powered up.

Eletronic set up

There are 2 potentiometers to adjust:

VR2	offset of output signal - turn clockwise for increased output
VR3	gain of output signal - turn anti-clockwise for increased gain

When powering up, the coil should automatically reach the horizontal position. There is a wire end stop to prevent it moving by more than ±2mm from the horizontal, so that the reflective end of the coil always reflects sufficient light from the diode onto the detectors.