

Cornell Center for Materials Research *KSV Nima* *Langmuir Blodgett Trough* Standard Operating Procedure

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1 Introduction

The KSV Nima Langmuir Blodgett Trough can be used to measure the surface pressure of a monolayer formed on a sub-phase, usually water. Once the monolayer formation pressure is known, the instrument can be used to form a monolayer and hold the pressure constant while dipping (or withdrawing) a substrate to deposit a monolayer film. It is possible to make multilayers by repeating this process.

Cleanliness determines whether an experiment succeeds or fails. Monolayer formation and deposition experiments require careful attention to cleanliness of the all of the equipment that comes into contact with the sub-phase or amphile - the trough, barriers, the Whilhelmy plate (for measuring pressure) and the substrate.

2 Acknowledgments

This instrument was purchased with support of the NSF MRSEC program. All publications and patent applications which include work performed on this instrument must acknowledge this support, including the current NSF grant number: DMR-1719875. For recommended wording, please see [this link](#). Use of current grant numbers is vital

for funding agency support!

3 CCMR Shared Facility Policies

The CCMR operates its shared facilities in accordance with its shared facility policy. The full policy document can be found [here](#). All users are expected to follow these policies, and in particular:

- To use a particular instrument, a researcher is required to have been trained by facility staff unless specifically posted otherwise. An unauthorized researcher and anyone knowingly granting access to such person is in violation.
- Users are not allowed to share login IDs/passwords, or to reserve or enable an instrument on behalf of another user.
- Instrumentation must be reserved and enabled by the intended user prior to and during use.
- Disabling or circumventing instrument interlocks for the purpose of avoiding instrument permissions or machine time charges is strictly forbidden.
- Users are not to move or disturb the work of other researchers unless given permission by facility staff or the researcher. Notify facility staff about unattended samples.
- Users are expected to remove all samples and to leave the work area in as clean or cleaner condition compared to when they arrived.

4 Facility and Emergency Contacts

In the case of emergency, users should call 911 from any phone. Emergency phones are located in

Inquiries about training and use of the KSV Nima Langmuir Blodgett Trough should be made to Mark Pfeifer at map322@cornell.edu, (607)255-4161, or in person in Thurston Hall, Room 113.

Secondary contact for this instrument should be directed to Phil Carubia at pmc228@cornell.edu, (607)255-6757 or in person in Bard Hall Room B57.

5 Access

5.1 Becoming a CCMR shared facility user

Prior to accessing the CCMR shared facilities, instrument researchers will need to register to become facility users and train on the instruments of interest. Registration consists of four things:

- Register for a CCMR FOM account: Internal users can register in [FOM](#) directly with a valid net ID. External users can register with the [External FOM registration](#)
- Read the orientation for the appropriate CCMR facility - which can be downloaded [here](#) - and complete, sign and return the last page to the facility manager (hard copy or electronic).
- Take required safety training for access to the room/instrument of interest(see safety training section of this manual)
- Schedule training with appropriate facility manager.

5.2 Physical Access

Access to Bard Hall B47B is controlled through a proximity sensor which reads Cornell ID cards. Either before, or at the beginning of training, provide the lab manager with a copy of the completed last page of the [orientation form](#). After completing training, the laboratory manager will confirm that you have completed all necessary safety training courses and then grant you door access. This process may take several days to complete.

A researcher without a Cornell ID card can request a temporary access card from the laboratory manager.

5.3 Instrument access: Facility Online Manager(FOM)

The instrument control computers are interlocked with FOM and must be enabled, through FOM, by a user with an active CCMR FOM account, before an instrument can be used. FOM can be accessed through any web browser. Users can log into FOM through the data analysis computer - provided in all CCMR Lab spaces, or any other device with a web browser - at <https://fom.ccmr.cornell.edu/fom>. If FOM is accessed through a public computer, you should either: use a private browsing session, making sure to close the browser when you have finished logging on to the instrument;

or, you must log out of FOM through the browser ***and*** close all open browser windows. Failure to do so may grant another user access to your FOM account through the public computer.

It is the expectation of the Bard Materials Facility that researchers will reserve only time that they need, use the bulk of their reserved time, and be on time to their reservations. To allow flexibility for unforeseen events, reservations in FOM will stay active for the scheduling increment of the instrument. If the instrument is not enabled before the end of this grace period, the reservation will automatically be deleted and the instrument will become available to all trained users. Users with a reservation will always have precedent and it is always recommended that a user make a reservation for their session.

6 Safety training

Before performing any laboratory work at the Cornell Center for Materials Research Bard Facility, users must have completed the following safety training courses through CULearn:

- EHS 2555 - Laboratory Safety
- EHS 2716 - Chemical Waste Disposal
- EHS 5330 - Fire Safety
- EHS 3355 - Fume Hood

7 Preparation before coming to the facility

Bring an appropriate syringe for controlled dropping of amphiphile plus solvent onto the surface of the sub-phase (other than during training). Bring a substrate if you will be depositing a monolayer (other than during training). The largest substrates that can be dipped into the trough are 50 mm x 50 mm.

8 Equipment

8.1 Hardware

8.2 Software

Open KSV NIMA software on the computer near the trough

9 Procedure

Do not calibrate the balance unless you have been specifically trained to do so and have asked permission from map322.

9.1 Chemical Waste

Chemical waste must be stored in closed containers in the fume hood. Waste containers are labeled with the types of materials in them. The labels on the waste bottles correspond to tags mounted on the outside right frame of the fume hood with magnets. There should always be one with isopropanol and stearic acid. As long as the quantities of chloroform are consistent those used in normal experiments in the trough, there should be no measurable chloroform in the waste generated and it can therefore be treated as non-halogenated solvent. If you are using solvents or materials which require more careful disposal than standard non-halogenated waste, see if one of the waste bottles is consistent with your waste class. If not, label an empty bottle and place the corresponding tag with the others on the outside. If the waste bottles are nearly full, notify map322 and pmc228.

9.2 Clean Instrument Surfaces

- Glove must be worn at all times when handling the LB trough.
- *Do not use chloroform to clean trough or barriers.*

9.2.1 Clean the barriers

- Rinse the barriers with DI water to remove any dust.
- Clean barriers by brushing them with ethanol. Use downward strokes to brush barriers. Use a large beaker to collect the ethanol.

- Make sure all sides of the barrier are brushed several times with ethanol Rinse the barriers with DI water and place them near the instrument with the metal side facing down.

9.2.2 Clean the trough

- Use the same process as used for the barriers.
- The trough clicks into the set up with two small screws on the inner side of the apparatus.

9.2.3 Clean the Whilhelmy Plate

- Handle the dipping plate with tweezers and and use extreme caution not to damage the plate.
 - Clean the plate with ethanol and finish with DI water.
 - If you know that the plate has not been used recently, clean it using a flame.
 - Take care not to put the wire into the flame, because it can fuse with the tweezers.
 - To start the torch, push down on the small tab on the back and push in the button. Once the flame is on, slide the tab to "hands-free".
 - Clean the plate by placing just the plate and not the wire in the the hottest part of the flame until the plate glows a bright orange.
 - Clean the other side of the plate in the flame.
- item Hang the plane on the balance hook of the LB trough

9.3 Prepare the Trough

- Make sure that the trough surface is clean.
- Put in the two barriers. The metal rods fit between brackets behind the trough.
- Fill the trough with clean MilliQ water until you can see a large meniscus when looking parallel to the surface of the trough.
- If the water is not level, adjust the legs to level the water. A leg rises when you turn the blue knob clockwise while holding the metal part of the leg.

- Lower the balance so that approximately 1/3 of the Wilhelmy plate is submerged. To lower the plate, hold the blue side knob and loosen the black knob. Turn the blue knob to lower the plate and then tighten the black knob.
- Open Manual Control in the software and select the Balance tab. Zero the balance.
- Close the barriers and observe the surface pressure. If it is above 0.2 mN/m, you must clean the surface of the water with the vacuum.
 - Open the barriers
 - Turn on the pump. Clean the Teflon tip with ethanol and then DI water.
 - Gently tap the tip to the water surface in different locations on the trough. Run along the barriers, taking care not to submerge the tip.
 - Do not touch the bottom of the trough with the tip.
 - Close the barriers again. If the surface pressure is still above 0.2 mN/m, repeat the vacuuming process
- Open the barriers and zero their position in the Manual Control window.

9.4 Isotherm Measurement

Click on the ISO button in the upper left hand corner. Fill out the experiment information. Molecular weight and density should be input for the surfactant and sub-phase 1.

9.4.1 Add amphile to the surface of the sub-phase

- Make sure the syringe is clean and rinse it with the solvent (*i.e.* chloroform) 3-4 times.
- Use about 20 μL of a solution of the amphile and solvent. For stearic acid (for training) use a concentration of 1 mg/mL.
- Pull 20 μL into the syringe. An appropriate syringe is available for training, but researchers must bring their own for actual experiments.
- Before dropping the solution, confirm that you have zeroed the balance, but not the barriers or dipper.

- Carefully drop 4-5 of the solution on the surface of the sub-phase by holding the syringe parallel to the surface and tapping the water with the drops. Do not drop the solution because the amphile may not remain on the surface in that case.

9.4.2 Set up the isotherm experiment

- Click ISO in the control software window.
- Enter parameters in the Experiment Setup section.
- If using a new amphile or sub-phase, click on the Edit Database button to store information for future use.
- Press Start (this doesn't actually start the experiment).
- Fill our parameters in the Trough Control window.
- Most common rates are 5-20 mm/min.
- Set target surface pressure.
- Wait for the solvent to evaporate, at least 10 minutes for chloroform.
- Press GO in trough controls.
- Data collection will stop once target pressure has been achieved
- Click Yes to keep data.

9.5 Deposition Experiment

If an isotherm was just completed, the trough and plate should be completely cleaned again, unless the amphile is completely elastic. Raise the plate and vacuum out the water using the Teflon vacuum piece. Then clean the trough according to the procedure described in subsection 9.2.

If a glass substrate is used, it can be cleaned with solvents. Glass can also be cleaned using Hellmanex (available in the lab) or Nochromix. Make sure the substrate is dry.

Remove the substrate clip from the dipper by loosening the set screws. Clip the substrate all the way up in the clip and reattach the clip to the dipper so that the substrate is parallel to the barriers.

Input experimental information, such as the surfactant, sub-phase, and dimension of the substrate.

Open Manual Control to zero the dipper position. Lower the dipper until it just touches the water surface, and click Zero Dipper.

If a hydrophilic substrate is used, the dipper should be lowered so that it is submerged in the water. Do not zero the dipper.

9.5.1 Add amphile to the surface of the sub-phase

Follow the procedure in 9.4.1.

9.5.2 Set up the deposition experiment

- Click Dip in the control software window.
- Enter the experimental parameters in the Experiment Set Up window.
- Press Start.
- In trough controls, input the desired surface pressure at which the monolayer should be deposited.
- Press Go/Hold (after \geq 10 minutes since the amphile was added).
- When the monolayer reaches the target pressure, the dipper controls will appear.
- Wait for the monolayer to stabilize. The pressure will oscillate and the barriers will close slightly as the layer packing becomes more uniform.
- Define the minimum height by pressing the set button if a hydrophilic substrate is being used.
- Define the upper limit as positive 2-4 mm, so the substrate will lift completely out of the sub-phase
- Press start to start the deposition.

9.6 Analyze data

On the device server, click the folder button in the top middle. Click on the run date that you would like to see. You can export data if desired. Right click on the run and choose graph surface pressure curve.

On the dipping results tab, press Graph so see the transfer ratio vs layer number graph. To find the average area per molecule click on the blue note pad icon at the top, select

Experimental Setup. Right click on test graph and select 'to calculate'. Click on two points on the graph and choose 'linear regression' and click calculate.

9.7 Transferring Data

The control computer is not connected to the internet. If you need to transfer data, please bring a USB drive.

9.8 End of experiment

Clean the trough, Wilhelmy plate, and barriers as described in section 9.2.

9.8.1 Cleaning up

This instrument is in a shared laboratory space. It is therefore important that each user take responsibility for cleanliness of the work space. Simply put, leave the lab space as clean as you found it. This includes:

- Clean any glassware and/or other lab hardware which came into contact with samples. When rinsing glassware, capture any solvents in a container which you will remove at the end of the experiment, with the exception of water used for final rinsing, which may go down the drain.
- Dispose of any pipettes (including plastic) in the trash containers labeled 'glass only'. These containers should only be used for pipettes, broken glassware and used slides.
- If you used powder or liquid samples, wipe down any work surfaces where samples were handled or transferred.
- Remove samples. Take all samples and other materials with you.

9.8.2 Disabling the control computer through FOM

Disable the instrument through FOM. Either go to the instrument schedule page and click on your present use, or go to your User Home and click on the instrument logoff link at the top of the page. A pop-up box will appear. Select the appropriate funding source (if you have more than one option), and then click on the 'Logoff' button. If you access FOM through a shared computer, you must log out of FOM *and* close all instances of the web browser to avoid unintended access of your account by another

user. Log out of FOM by clicking on the red 'X' in the top right corner of the page, next to your name, and confirm that you wish to log out.