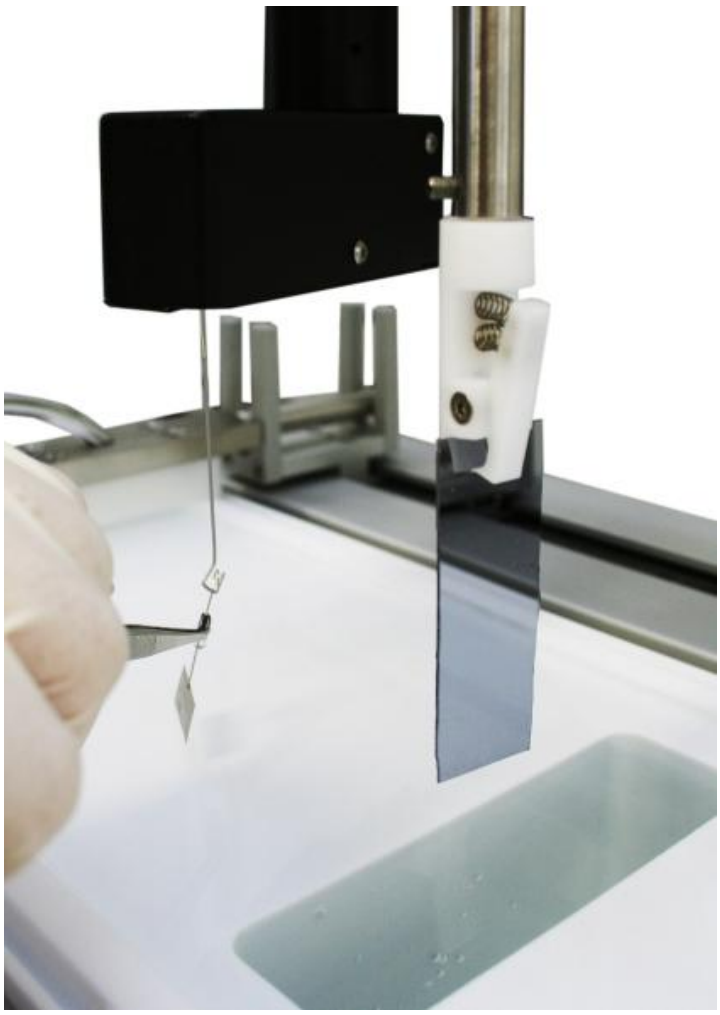
KSV Instruments

Operation Manual

LB device



KSV Minitrough

Revision 1.1

Table Of Contents

1. Introduction

The Langmuir and Langmuir-Blodgett (LB) devices available from KSV Instruments are efficient and effective in investigating the properties of floating monolayers, precise deposition of multilayers onto solid substrates or simply as platforms for use in observing surface chemistry effects such as the breakdown of an enzyme or the crystalline structure of a surfactant. The wide range of available systems and the many modules available make customisation a charm, and if the troughs available do not suit your needs then contact us about designing a particular trough to your specifications.

There are four series of LB systems available today, the KSV 5000, 2000, Minitrough and the Minimicro in descending order of size. The differences between the systems are mechanical, the same software and interface unit operates all of the LB devices. KSV 2000 is the basic multipurpose LB device. KSV 5000 adds a sturdy frame with automatic elevator arms for attaching additional measurement devices, becoming the most versatile LB device available today. The KSV Minitrough is a more compact and economical LB device. The KSV Minimicro is a small and elegant LB device brilliant for depositions on small substrates or to minimise sample volumes.

This is the device-specific manual for the KSV Minitrough LB device, featuring details such as physical descriptions and cleaning instructions. Please see the LB installation manual or the LB software manual for details on installation, calibration, measurement taking or data analysis.

2. Physical Description

2.1. Overview

The general physical layout of the KSV Minitrough Langmuir-Blodgett (LB) system consists of the trough with the subphase and the surfactant, barriers which can adjust the surface pressure by reducing the area available to the surfactant, a balance with a Wilhelmy plate which monitors the surface pressure and an interface unit to control the devices and communicate with the computer running the experiment.

The available systems are modular, based on System 1.

System	Trough	Devices	Available experiments
1	Flat	Balance	Langmuir film balance
2	Flat with well	Balance and dipper	Multilayer LB-films on solid substrate and Langmuir film balance
3	Flat with quartz window	Balance	Langmuir film balance for Microscopy experiments
4	Flat with extended range	Balance	Langmuir film balance

2.2. System 1

The appearance of the instrument depends on the system in use. System 1 is the most basic setup, consisting of a balance and a trough with a barrier drive system.

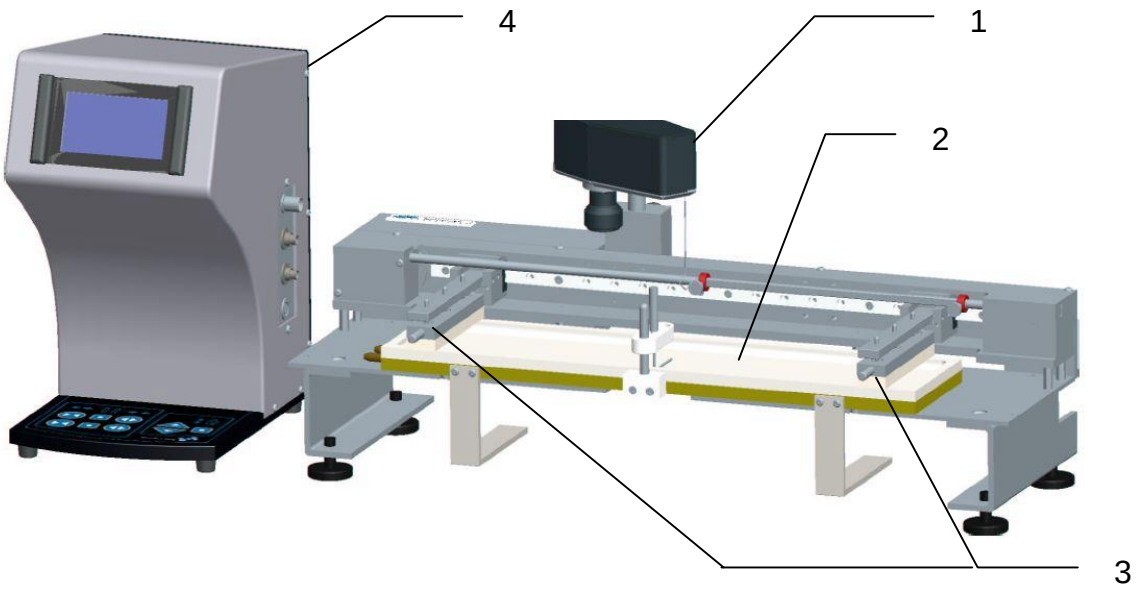


Image 2.1: *KSV Minitrough System 1.*

1) Balance
2) Trough
3) Barriers
4) LayerBuilder

System 1 is designed for the study of floating monolayers.

PTFE trough with a surface area of 273 cm^2, (L364 x W75 x D7 mm^3) and a subphase volume of 190 ml.

2.3. System 2

The System 2 additions over System 1 are the dipper, a second stand for the dipper and a trough with a well.

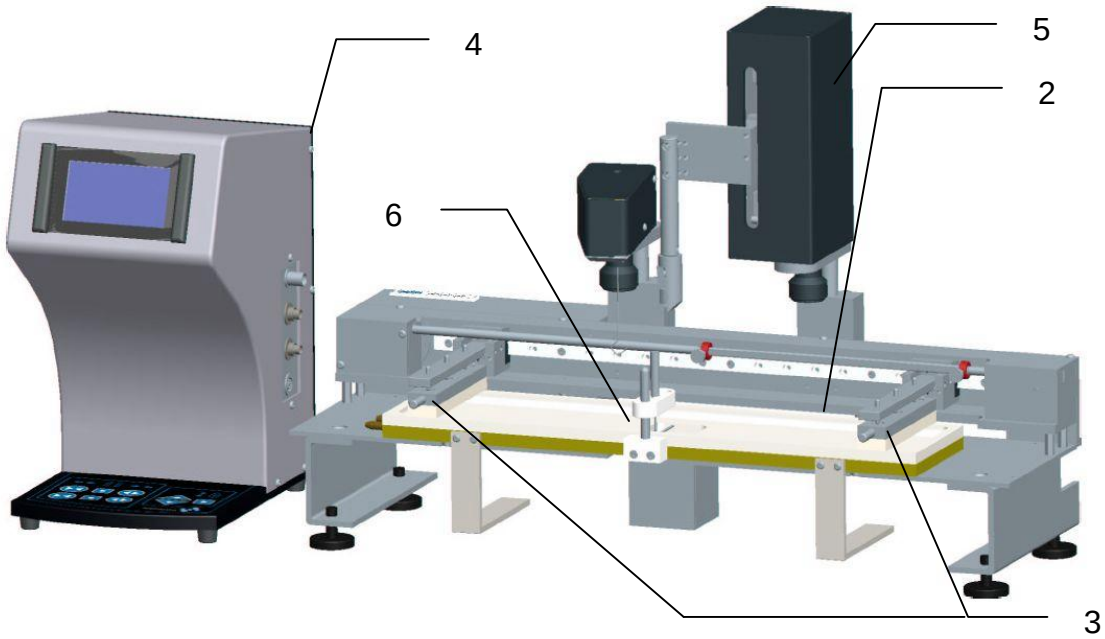


Image 2.2: *KSV Minitrough System 2.*

1) Balance
2) Trough
3) Barriers
4) LayerBuilder
5) Dipper
6) Well

System 2 is designed for the study of floating monolayers and for the deposition of multilayer films of one substance on a solid substrate.

PTFE trough with a surface area of 273 cm^2, (L364 x W75 x D7 mm^3), a dipping well (L37 x W37 x D64 mm^3) and a subphase volume of 280 ml.

2.4. System 3

In addition to system 1 the trough is fitted with a quartz window. System 3 is designed for the microscopic study of monolayers and floating monolayers. It is suitable for all conventional microscopes and inverted fluorescence microscopes.

Image 2.3: *KSV Minitrough System 2.*

2.5. System 4

In addition to system 1 the trough length is doubled. The dimensions of the trough are surface area of 587 cm^2, (L782 x W75 x D7 mm^3) and a subphase volume of 290 ml.

2.6. Mechanics

A) Computer

The computer controls all functions of the instrument. Because each device includes its own microprocessor, the computer sends only high level commands to them (for instance "Move with certain speed" or "Go to certain position" and so on). All devices are listening to every message, but they become active only when they recognise their own address.

The computer should be a PC running Windows 2000/XP/Vista. A 1GHz processor and 512MB RAM memory are recommended.

B) Barrier Driving System

The barrier position is controlled by a micro step driven stepping motor. The motor moves the barrier holder using a tooth belt. The holder itself is attached to a linear motion system, which is equipped with ball bearings.

The barrier driving system is equipped with adjustable safety switches which stop the barrier immediately when the barrier holder hits the switches. The position of the barrier is measured using an optical encoder. The position is relative and so a zero point at a known position must be set manually.

The control electronics are located inside of the barrier driving unit. There is a 15-pin connector on the rear of the unit. The barrier driving unit should be connected to the Interface unit through this connector.

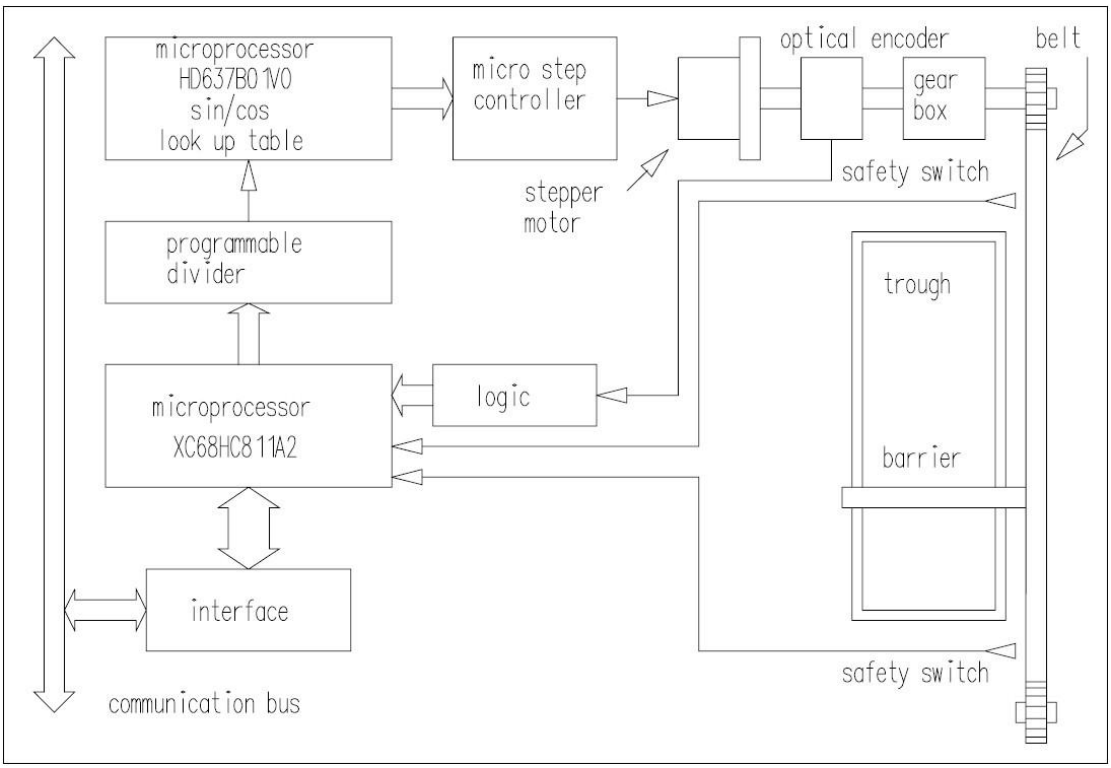


Image 2.4: *Block diagram of the barrier driving system*

C) Surface Balance

The surface pressure is measured by using the Wilhelmy plate method (round rod optional). The Wilhelmy plate is a carefully sandblasted platinum plate or a clean paper plate, which is put partly under the surface of the subphase. Normal practice is to position the plate so that one third of it is under the subphase. The force acting on the plate depends on surface pressure. This force is measured using the electro-balance. The total measurement range of the balance is +/- 2000 mg. The width of the platinum Wilhelmy plate has been chosen so that 1 mN/m corresponds to 4 mg. For paper Wilhelmy plates this value is commonly about half as great as with the platinum plates.

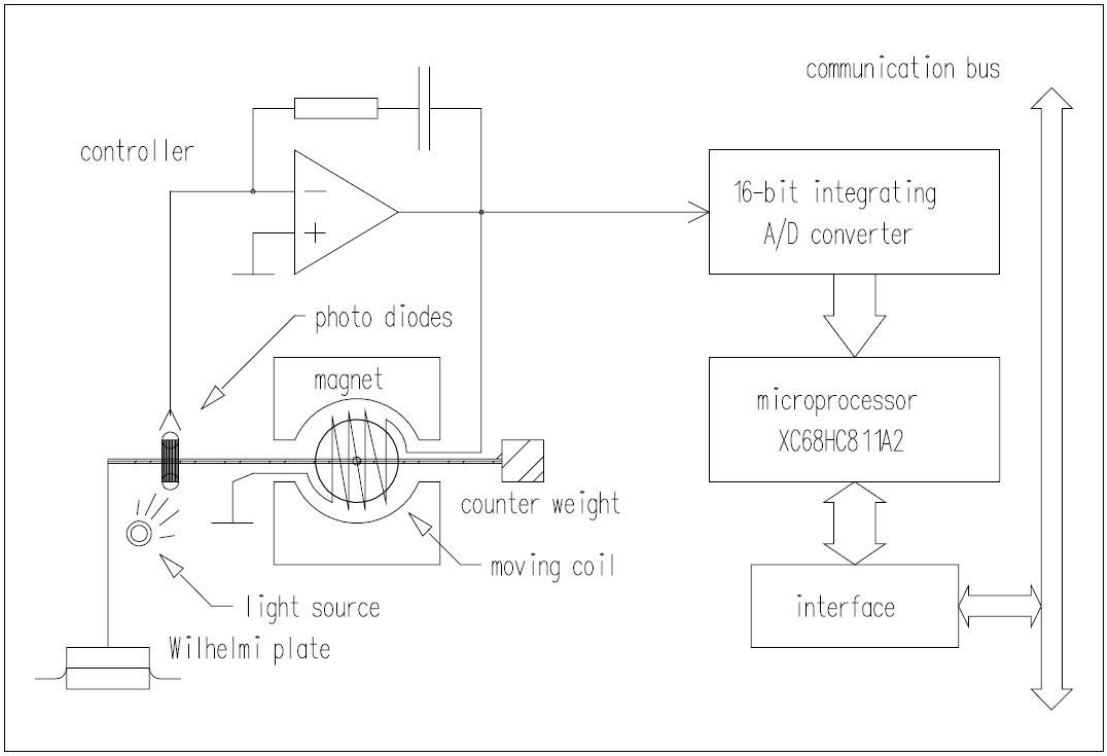


Image 2.5: *Block diagram of the surface balance.*

D) Deposition system

The deposition system is DC motor controlled to ensure vibration-free operation. The speed range of the dipping arm is 0.1...85 mm/min and maximum stroke is about 75 mm. The DC motor is servo controlled to keep the speed constant. The position is determined with an optical encoder. The deposition system is equipped with safety switches which stop the motor automatically in both ends.

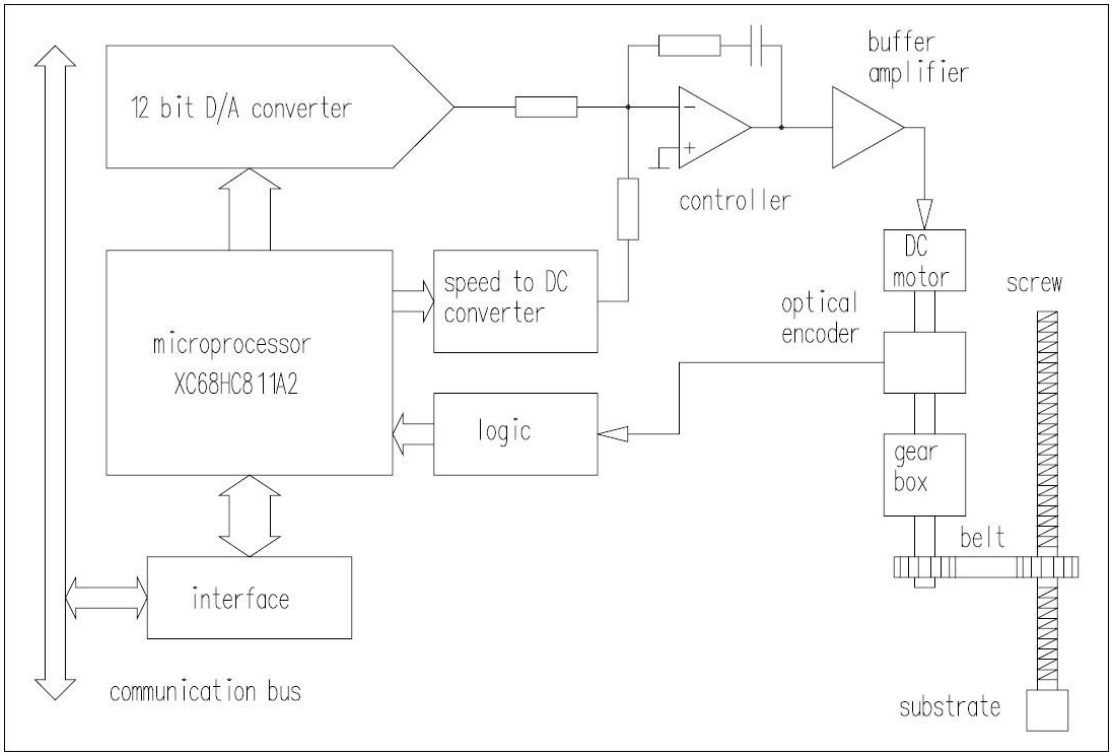


Image 2.6: *Block diagram of the deposition system.*

E) Trough and Barriers

The troughs are made of a solid piece of PTFE. The trough is glued onto an aluminium base plate, in which there are water channels for the thermostation of the subphase. The temperature is adjusted with a water bath circulator, which is connected to the trough with special connectors. These connectors include valves in both parts, so the connection can be opened without any leakage.

The maximum temperature allowed is 60C!

The barriers are made out of hydrophilic material, which ensures that the film does not escape the barrier. The barrier material is polyacetal (Delrin) and it should be noted that **Delrin is not resistant to acids or chloroform, so do not use these as cleaning agents**. Chlorinated solvents such as these can still be used as spreading solvents. The barrier is equipped with a round rod, which fits into the barrier holder.

2.7. Additional Devices

Various additional devices can be used to improve measurement conditions, enable additional measurement methods or to provide additional data.

A) Thermostation

The temperature of the subphase can be monitored with a temperature probe that connects directly to the Interface Unit. The temperature sensor is covered with a thin PTFE tube and it can be attached to the trough with a special holder. The display units are in degrees Celcius, though Fahrenheit is available on request.

This keeps a full record of the temperature conditions of the experiment, but to adjust that temperature a water bath is required. KSV troughs have built-in piping in the aluminum block which holds the teflon trough. Connect the water bath to the ports along one end of the trough. KSV Instruments recommends the Julabo water bath also available from KSV, though others can be used as well.

Connect the water bath to an available COM port on the computer. Use the software's Manual Control Unit to adjust the temperature settings for the water bath.

B) pH Probe

The acidity of the subphase can be monitored with a pH probe that connects directly to the LayerBuilder interface unit.

C) Stirrer

A magnetic stirrer consists of a rotating magnetic field source and a small magnetic bar. The rate of rotations can be adjusted with the control knob on the power supply. A magnetic stirrer is used particularly with enzyme reaction studies.

D) Minicab Cabinet

A cabinet that can be placed around the LB device to isolate it more from the environment. Includes appropriate holes for water bath circulator tubes and cables.

E) Horizontal Dipping Clamp

The standard dipper clamp can be replaced by a horizontal dipping clamp for the purpose of conducting Langmuir-Schaefer experiments. The two dipping

clamps can be interchanged quickly and easily, and no software upgrade is necessary. Set the zero position of the dipper as the level at which the plate is wetted first. With longer experiments the dipping depth might need to be increased over time to reach the surface due to evaporation.

F) Surface Potential Probe SPOT

A surface potential probe (KSV SPOT) is a tool for measuring the surface potential of an interface or a solid. KSV SPOT uses the vibrating plate capacitor method.

G) Brewster Angle Microscopy BAM

Brewster angle microscopy (BAM) provides visual information of two dimensional structures in monolayer films. It utilises a laser and a CCD camera to obtain images at 25 fps.

H) Interfacial Shear Rheometer ISR

The ISR 400 provides information on the shear stress of interfaces, particularly useful for low viscosity applications. A teflon-coated magnetic needle rests at the interface, and as a magnetic field is induced the movement of the needle is observed with a microscope.

2.8. Special Troughs

The choice of trough is one of the most important ones to make when acquiring and using a LB device. Several troughs are available designed, and many more can be manufactured according to specifications. The specialized trough types available at the moment are alternate dipping, conductivity, compartmentalised, enzyme reaction, oil/water interface and surface potential troughs. Additionally several features can be incorporated into most of the troughs, these are an injection port, a quartz window and low subphase volume.

A) Low Volume Trough

The low volume trough requires smaller subphase and sample volumes. Effective surface area is 136 cm^2 (L316 x W50 x D1) and subphase volume is 18ml.

B) Conductivity Trough

The conductivity trough is used to measure the conductivity of the monolayer. Four electrodes are placed near the center of the trough, two on each side of a central rise. The central rise divides the trough into two compartments with a

narrow band of glass connecting the subphases of the different compartments.

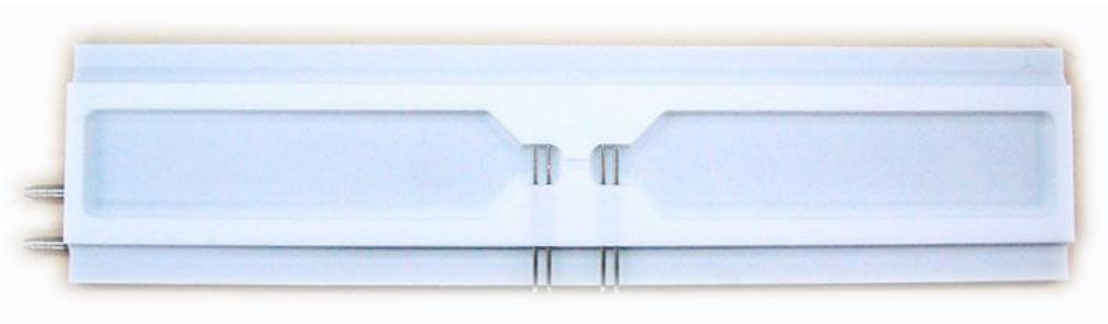


Image 2.7: *The conductivity trough*

The exterior electrodes introduce a current and the interior electrodes measure the potential between them. The subphase volume through which the current can flow is minimized with the use of the glass band.

C) Enzyme Reaction Trough

The enzyme zero order breakdown reaction can be observed with this special trough. The center compartment is connected to the outer compartments with glass bridges allowing the pressures to be adjusted without disturbing the center compartment.

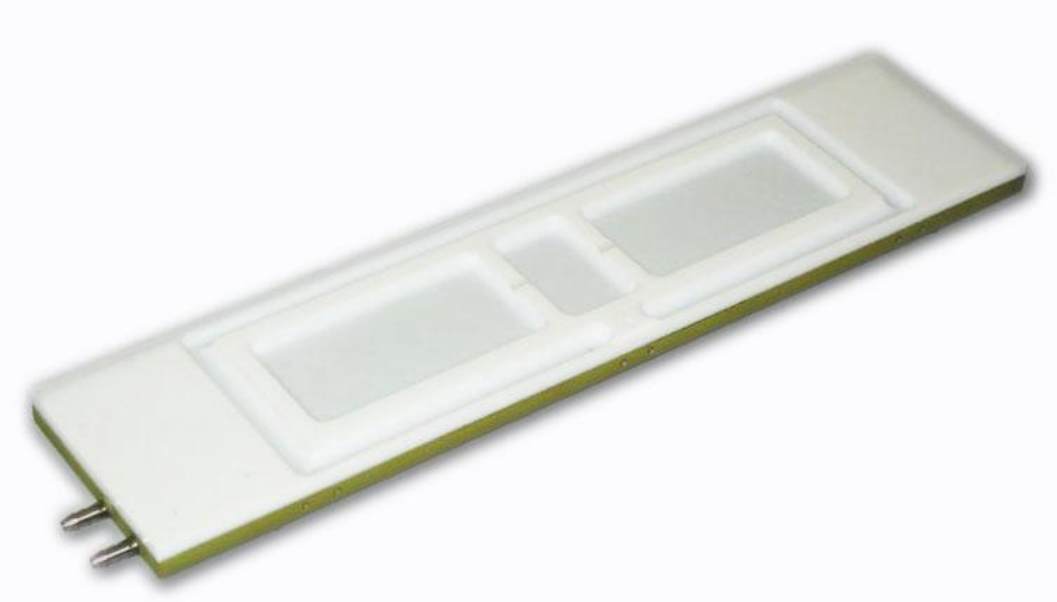


Image 2.8: *The enzyme reaction observation trough.*

The enzyme is placed in the centre trough and the initial surface pressure is measured. A protein is placed through an injection port and as the protein interacts with the monolayer the amount of monolayer material decreases and surface pressure decreases. The barriers approach the centre to keep the surface pressure (and thus mean molecular area) constant, and the area removed by them can be used to calculate the activity of the enzyme.

A stirrer might be necessary to properly observe the activity of the enzyme.

D) Oil/Water Interface

The oil/water interface trough places the monolayer material between two liquids as opposed to between a liquid and a gas. The denser liquid, usually water or another polar liquid, is poured into the trough up to the first notch. The monolayer is placed onto the surface of the polar liquid. The organic liquid is then poured gently on top of the water so that the holes in the barriers are distinctly above the interface but covered by the oil. When the barriers are brought closer together the monolayer material is compressed.

Image 2.9: *The oil/water interface trough*

Alternatively the monolayer material can be mixed into the oil and as the oil is poured the monolayer material forms a monolayer at the interface due to the strong polar attractions between the polar subphase and the hydrophilic end of the monolayer material.

E) Additional Trough Features

- A quartz window can be added to most troughs to enable live observation with a microscope or similar optical instrument.
- An injection port can be used to add a substance into the trough after the beginning of an experiment. As it is important not to disrupt the surface the injection port leads a needle tip from the side of the trough to the subphase. It is a standard feature with enzyme reaction troughs.
- A low subphase volume trough reduces the area that is available for the subphase, thus reducing the amounts of materials required.

F) Custom Troughs

Many situations arise where a custom designed trough is necessary to accomplish a particular task. KSV Instruments supplies many custom designed troughs, be they extensions of current designs or completely new concepts. Ask us for more information about a trough that suits your needs!

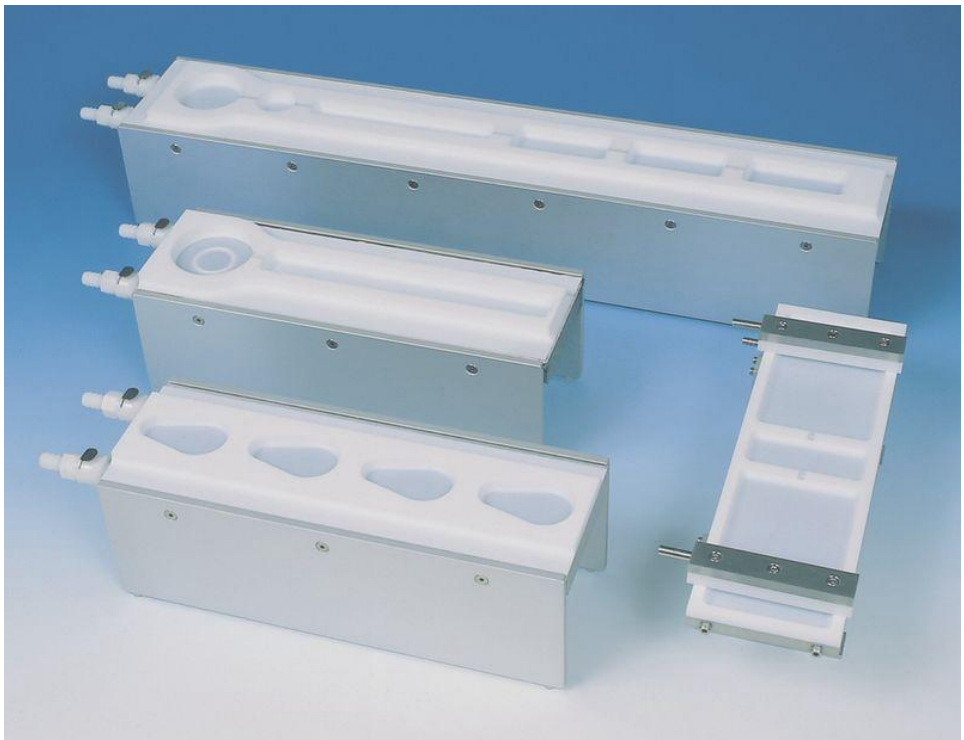


Image 2.10: *Some past custom-made troughs.*

3. Preliminaries

3.1. Connections

Check that all devices are attached to the LayerBuilder interface unit. Check that the power cable is connected to the interface unit and that it is turned on.

Turn on the computer and start the sgserver for LB systems. It can be located from the **Start** menu under **Programs > LB**. Open the **Manual Control Unit** by clicking on the menu **Control Panel**. Open the **Barrier1** tab.

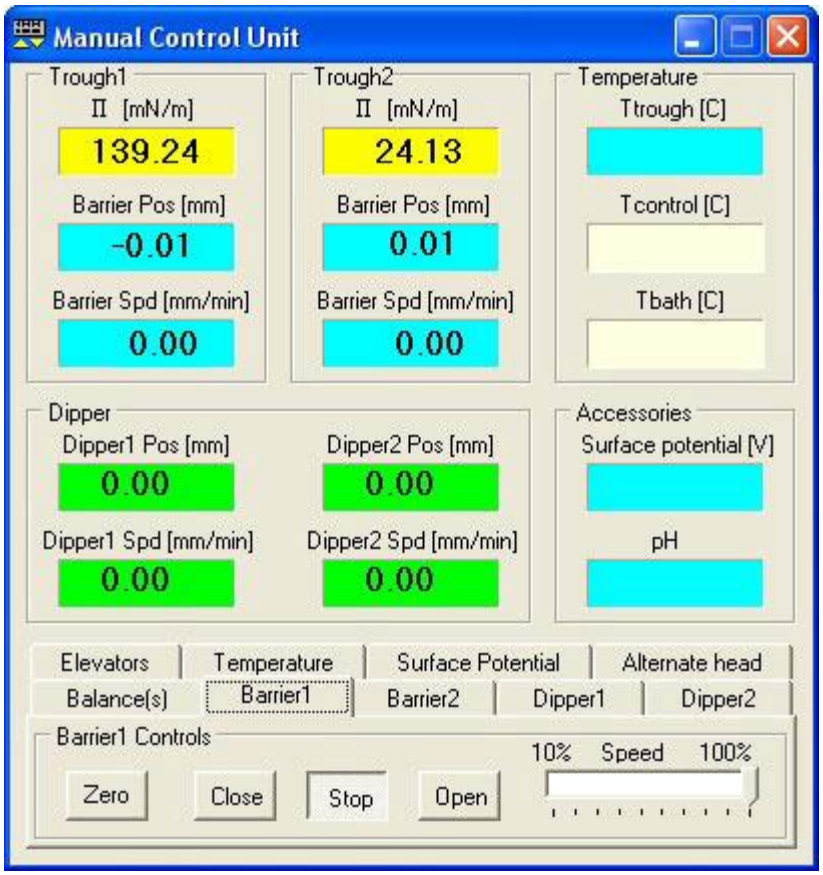


Image 3.1: *The Manual Control Unit used to control attached devices.*

Check that the safety limit switches are properly set by first closing and then opening the barriers. Adjust the positions of the switches if necessary.

3.2. Cleaning

Before an experiment the trough and barriers must be cleaned. Use rubber gloves to minimise oils from the skin contaminating the apparatus. The importance of cleanliness cannot be overstated!

The troughs used by KSV can be removed from the frame and carried to a sink for ease of cleaning. A recommended cleaning procedure is to first brush with a soft brush covered in ethanol or another organic solvent (mechanical and chemical cleaning) and then rinse with ion exchanged water. Take care not to scratch the surface!

Images 3.2 and 3.3: *Brushing the trough with ethanol (left) and rinsing the barrier with pure water.*

If the trough has not been used for a longer period of time then first washing with hot water and a commercial detergent might be appropriate before performing the standard cleaning described above. Replace the trough in the frame, checking that the screws fit into the available notches.

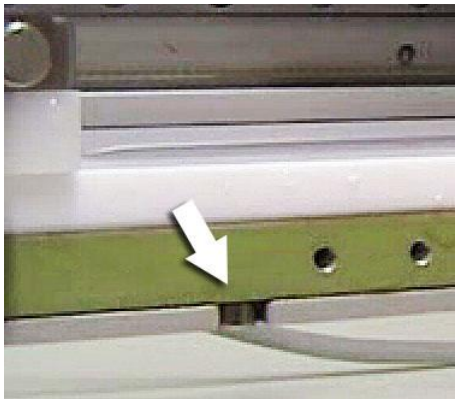


Image 3.4: *Replacing the trough so that the screws slot into the respective notches.*

Clean the barriers. The appropriate cleaning method is the same as with the trough. When one barrier is cleaned place it on top of the trough before cleaning the other barrier. Take special care in choosing the appropriate handhold on the barrier so that replacing it will be simple, avoid touching the delrin itself!

Rinse the aspirator tip with ethanol and ion exchanged water.

On the Manual Control Unit click **Open** and wait until the barriers are in the desired position, most often fully open. Press **Zero**. Fill the trough with the subphase. In most cases pure water will be a good choice. Pour the water gently on to the trough so that the level of the water rises distinctly (at least 3mm) above the level of the trough.

Turn on the aspirator and click on **Close** to start bringing the barriers together. Contaminants on the surface of the water will be picked up by the barrier. Run the tip of the aspirator along both barriers several times. The purpose of this is to remove contaminants and not to suck too much of the water so run the tip over the surface rather than plunging it into the water. When the barriers are fully closed run the aspirator tip over the surface between the barriers several times to pick up remaining contaminants. A systematic pattern such as three vertical and horizontal lines (#) is recommended.

Remove enough water to lower the water surface until it is level with the trough.

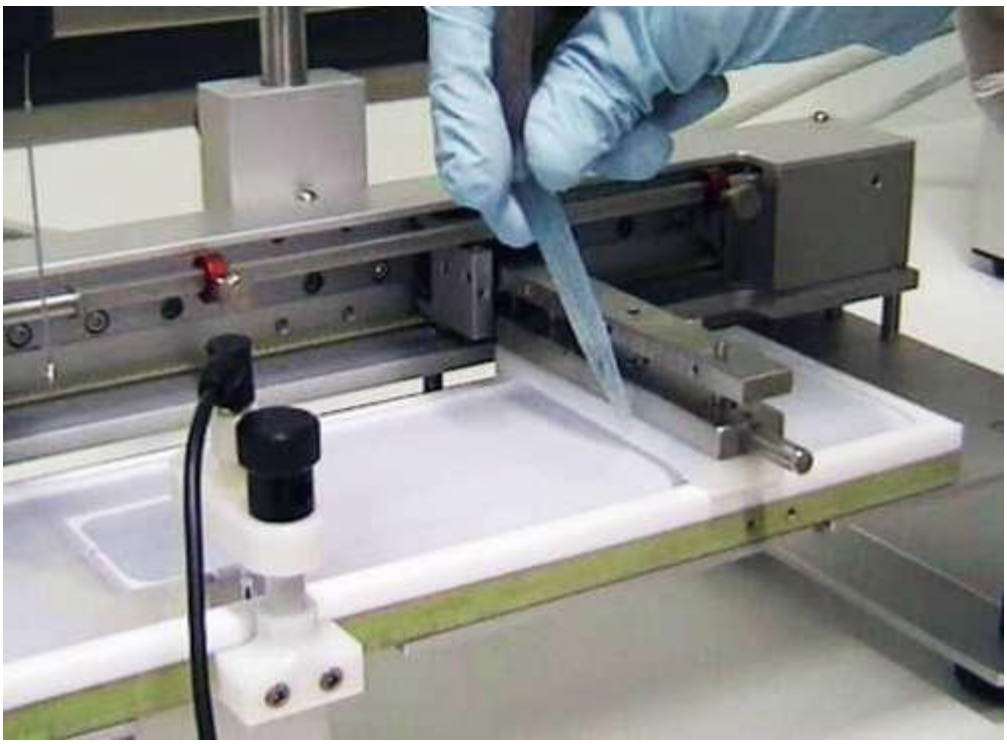


Image 3.5: *Sucking contaminants from the barriers.*

Clean the Wilhelmy plate. A thorough cleaning entails heating the platinum Wilhelmy plate over a flame (such as a Bunsen burner). Heat the plate over a blue flame hot enough to make the plate glow red in just a few seconds, a flame that is too cool will leave residues on the plate surface. Alternatively rinsing with ethanol and ion exchanged water should be enough for most cases of frequent use. Once the experiment is finished the Wilhelmy plate should be stored in a water-soluble organic solvent such as ethanol.

Attention! The platinum Wilhelmy plate is an excellent catalyst for the combustion of methanol! Beware of operating the Wilhelmy plate around methanol, especially when hot.

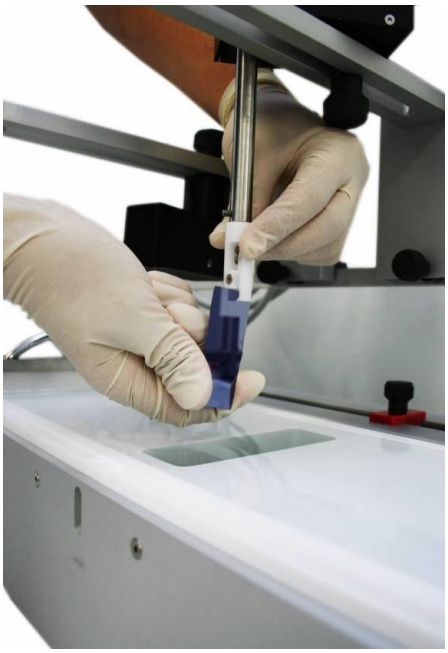
After the plate is completely glowing remove it from the flame and hold it for several seconds to allow it to cool. Dip the plate into the water in the trough and hang it from the balance hook. Set the plate so that about two thirds of it is covered and it is perpendicular to the barriers.

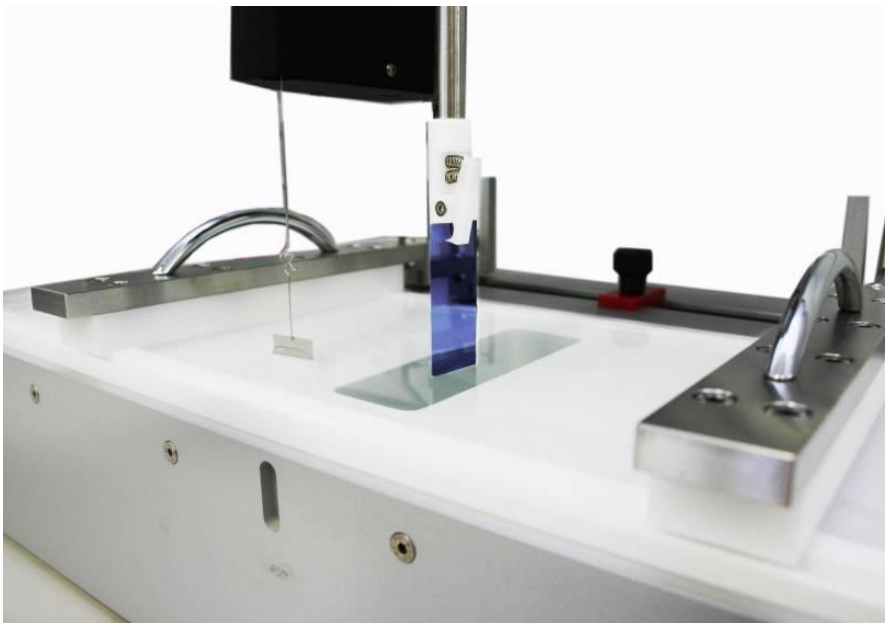
For information on using a paper Wilhelmy plate, please see the LB software manual.

Open the barriers. In the **Manual Control Unit** open the **Balance** tab and press **Zero Balance1**. The reading is now zeroed with pure water, and the measured difference in surface tension will be the surface pressure. Close the barriers. Pure water does not cause the surface pressure to change, so any change in surface pressure is caused by contaminants. If the surface pressure does not exceed 0.2mN/m the water can be considered sufficiently clean.

3.3. Dipper Substrate Attachment

If about to perform a **Dipping Experiment** with a hydrophilic substrate then clean the sample as appropriate. In the **Manual Control Unit** open the **Dipper1** tab and press **Up** and wait until the dipper arm moves well above the surface. Attach the substrate to the dipper arm and make sure that the substrate is still above the water surface. Take care not to touch the surface! Lower the substrate slowly from the Manual Control Unit and at when the water touches the substrate press **Stop** and then **Zero**. Then lower the substrate as far as it is meant to go for the experiment.





Images 3.6 and 3.7: *Attaching a substrate to the dipper (left) and zeroing the position of the substrate for a dipping experiment.*

3.4. Monolayer Material Spreading

Fill a precision syringe with a suitable volume of a prepared solution of monolayer material and a volatile organic solvent. The organic solvent should be non-polar so that it will not mix with the water and volatile so that it will evaporate from the surface, for example chloroform or hexane. A concentration of 1 mg/ml is usually a good choice to start investigations with.

Placing the solvent on the water should be done with care, gently push on the syringe to get a drop out of the needle and then touch the surface with the tip of the needle. Do not let the drop fall from the needle as some of the sample might be lost to the subphase as micelles and the surfactant does not spread evenly! Furthermore the waves caused by drops can carry monolayer materials to the edges of the trough.

Image 3.8: *Place the sample onto the water with care.*

Wait 10-15min for the solvent to evaporate. Use this time to fill in the **Experimental Setup** for this experiment. Press on the appropriate icon to start a new experiment, this will first open the Experimental Setup. Double-check that the dimensions of the trough are correct as this plays an important part in the analysis of the obtained data.

3.5. Experimental Setup

The **Experimental Setup** is used to identify the appropriate procedure and as a record for later analysis. Access this screen by beginning a new isotherm, dipping or alternative trough dipping experiment. Fill in all of the relevant sections. To add items to the drop-down menus use the **Edit Database** function. For more details please see the *LB Software Manual*.

4. Technical Specifications

4.1. Deposition System

Control and operation	Software controlled unsupervised and automatic film deposition. User defined deposition parameters.
Deposition speed	0.1 to 85 mm/min
Speed adjust	Increment 0.1 mm
Optional speed range	0.2 to 170 mm/min
Deposition cycles	1 to unlimited consecutive depositions
Dwell times	0 to unlimited seconds, individual adjustment for upper and lower end point of substrate movement.
Deposition arm	75 mm max. stroke
Max. size of substrate	60 x 35 mm
Type of motor	Servo controlled DC motor

4.2. Film Pressure Measuring System

Measuring principle	Wilhelmy plate, platinum or paper sensor element or round rod connected to micro-electronic feedback system for surface pressure control. Software controlled operation, user defined measuring parameters. A floating barrier sensor element with the same features as above also available.
Dynamic range	0 to 250 mN/m
Resolution	4 µN/m

4.3. Film Area Control System

Surface area regulation	Two symmetrically moving surface barriers. Software controlled operation and user defined process parameters.

Surface barrier	Leak proof hydrophilic (Delrin) barriers. Hydrophobic (Teflon) barriers by request or optional.
Barrier speed	0.01 mm to 400 mm/min (max. relative speed 800 mm/min)
Barrier motor	High precision micro step driven stepper motor.
Accuracy	Greater than 99%

4.4. Trough

Dimensions	Systems 1 and 3: surface area 273 cm^2, (L364 x W75 x D7 mm^3), subphase volume 190 ml. System 2: surface area 273 cm^2, (L364 x W75 x D7 mm^3), dippin well (L37 x W37 x D64 mm^3), subphase volume of 280 ml. System 4: surface area 587 cm^2, (L782 x W75 x D7 mm^3), subphase volume of 290 ml.
Trough material	Solid PTFE (Teflon), 1.5 mm bottom thickness, mounted on a thermo regulated base plate.
Barrier material	Hydrophilic Delrin (polyacetal). **Delrin is not suitable to be washed with acids or chloroform.** Teflon barriers can be provided as option.
Temperature range	0 to +60 C

4.5. General

Environmental protection	Optional vibration isolated bases and laminar flow hoods available.
Computer requirements	PC with a 1 Ghz processor and 512 MB memory running Windows™ 2000/XP/Vista.
Voltage Nominal Operational	 220 V, 50/60 Hz 110 V, 50/60 Hz 176-264 V, 40/440 Hz 90-132 V, 40/440 Hz

5. Contact Information

If any problems arise please feel free to contact a local distributor or KSV Instruments directly.

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