MICROTESTMACHINES Co.

Langmuir–Blodgett trough

LT-111

Operating Manual

2016

Langmuir–Blodgett trough LT-111: Operating manual. – Gomel: Microtestmachines Co., 2016. – 32 p.

Attention! In view of constant improvements of the device and updating of its mechanical and electronic parts some differences may exist between real details and the illustrations, that, however, does not mean deterioration of quality and characteristics of the complex.

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CONTENTS

1 GENERAL DESCRIPTION

Device for film application LT-111 is intended for mono- and multimolecular film deposition on surface of solid substrates according to Langmuir–Blodgett (LB) technique.

Fig. 1.1 – General view of the Langmuir–Blodgett trough LT-111. Right – control electronic unit, left – LB trough.

Langmuir–Blodgett trough LT-111 (Fig. 1.1) consists of following units

1. LB trough;
2. Dipper unit;
3. Surface pressure sensor;
4. Control electronic unit with set of connection cables.

Additional accessories include:

1. a PTFE holder for substrate clamping;
2. a PTFE plate for sample placement to deposit film by liquid draining technique;

For the device operation, it is necessary to allocate additionally host PC to run the device control software. The host PC should operate under Win32 operating system and contain a free USB port to connect with the device control electronic unit. For organizing connection between host PC and control electronic unit, during first switching on of the device install specialized CDM drivers for COM-port emulation available, for example, at http://www.ftdichip.com/Drivers/D2XX.htm.

1.1 LB trough

Schematic diagram of the LB trough is shown in Fig. 1.2. LB trough itself is made of PTFE block. Free surface area of the trough is 463±1 cm^2 (290x160 mm) and depth is 14 mm. Dipping well installed at left side of the trough has width 130 mm, length 28 mm and own depth 110 mm that allows to dip substrates as wide as 125 mm. The trough holds about 1 liter of liquid.

Barrier for constraining free surface of liquid is made as a PTFE bar and slides on ledge in the trough top. Taking into account 'dead' area behind the barrier, maximum area of liquid surface is about 440±1 cm^2. Changeable (working) area of liquid free surface in working zone is about 312±2 cm^2.

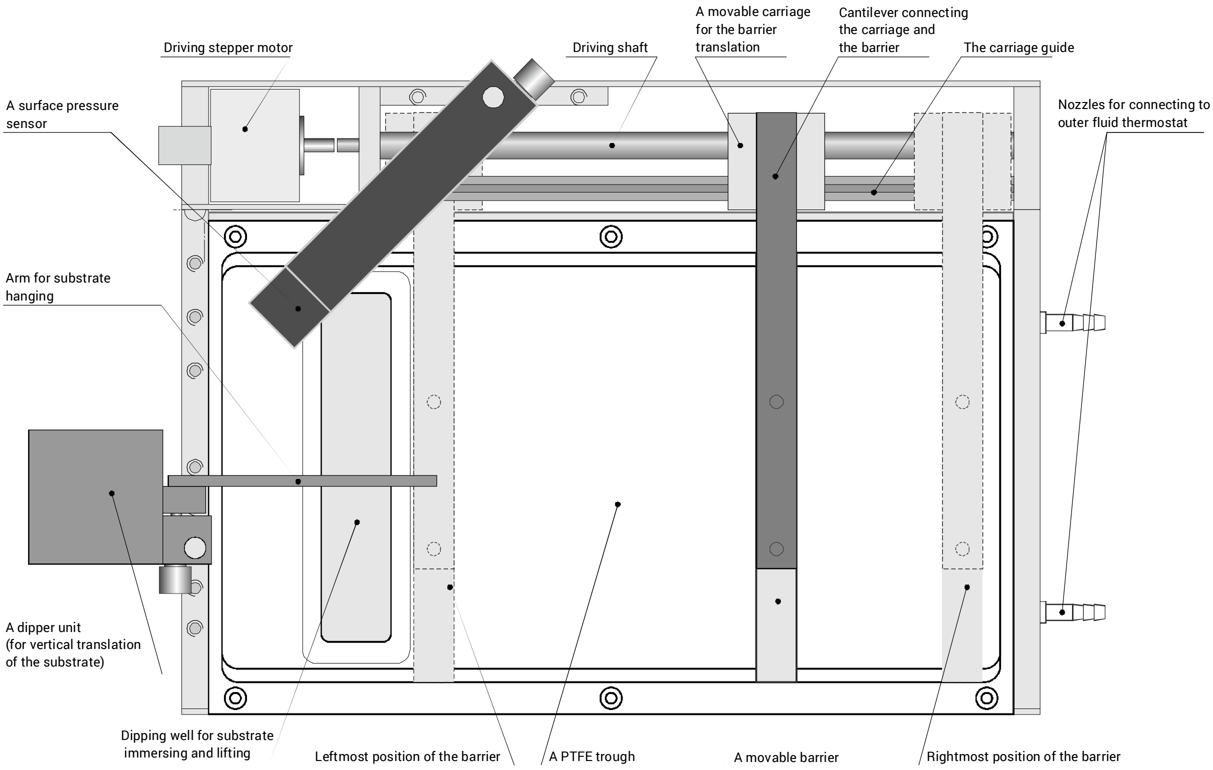


Fig. 1.2. Top view schematic diagram of Langmuir–Blodgett trough LT-111.

The PTFE trough is mounted in a frame of aluminum alloy. In extended version, the trough rests on a heat exchanger slab suitable for liquid thermostating by external device (thermostat not included). Two nozzles (outer diameter 6 mm) of the thermostating slab are located on right wall of the device to be connected to outer thermostat. To monitor temperature inside the trough, a thermal sensitive element is embedded in rear wall of the PTFE trough near the surface.

From rear side of the frame, a mechanism of the barrier horizontal movement is installed. Drive shaft is powered by a stepper motor (steps 0.9 deg.) that provides precise horizontal positioning of the barrier. Two vertical stands for installation of surface pressure sensor and dipper unit may be mounted in suitable holes (M6) on left or rear walls of the frame. Example of the unit placement is shown in Fig. 1.2. All the installed units allow their turning around the vertical stands and vertical movement along the axes. To fix the units in a desirable position on the stands, they are equipped with clamps.

Hose for draining the liquid off the trough is attached to nozzle (outer diameter 6 mm) in bottom of dipping well.

1.2 Surface pressure sensor

Surface pressure sensor serves to monitor surface pressure of liquid in the trough. This information is then used to form error signal for feedback system of the device.

The sensor unit is mounted on the stand at left or rear side of the PTFE trough frame (see example in Fig. 1.2). Schematic diagram of the sensor is shown in Fig 1.3. Sensitive element of the sensor is made as a flexible cantilever (leaf spring). To the free end of the cantilever, a Wilhelmy plate is hung to be put into liquid and to serve as initial sensor. Bend of the cantilever depends on force *S* exerted by liquid surface to submerged Wilhelmy plate. Change of surface pressure (tension) causes change of force *S* and, correspondingly, deflection of the cantilever free end off the neutral position along vertical axis. The plate usually is made of a piece of filter paper featuring good wetting with the liquid (not shown). Size of the plate should be about 14x14 mm to provide area about 200 mm^2 (this value is considered as standard for the device measuring system).

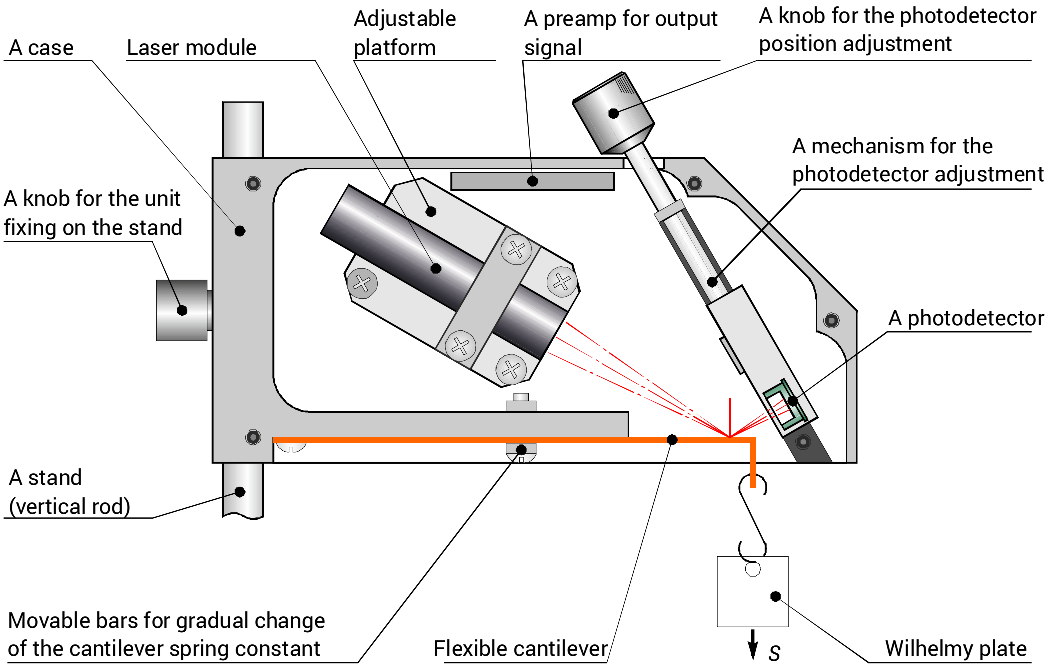


Fig. 1.3 – Schematic diagram of surface pressure sensor.

To detect sensitive cantilever deflection, a laser-beam scheme is applied (Fig. 1.3). It comprises laser source and two-section photodetector. Laser beam from the source is targeted onto mirror mounted on rear side of the cantilever free end. After reflection off the mirror, the beam gets to two-sectioned photodetector. Position of reflected spot on the photodetector sensitive area depends on cantilever vertical deflection. Electronic system compares output signals from upper and lower segments of the photodetector and determines magnitude of the cantilever deflection that in its turn describes changes of surface pressure.

Design of the surface pressure sensor provides for smooth adjustment of the cantilever spring constant by change of free end length using a sliding bars (Fig. 1.3). Installation of flexible cantilever with different thickness, width or material gives an opportunity of step-like change of the spring constant. Combination of above two ways provides possibility to select and adjust necessary range of the sensor unit sensitivity.

Laser source of the sensor is mounted on adjustable platform. Laser module includes power supply circuit, laser diode and set of lenses in a tube case. The laser module features:

aperture .... 3 mm
focus length .... 40 mm
wavelength .... 635 nm ±5%
laser diode power output .... < 5 mW
power supply .... DC 5 V

Position of the platform with laser module can be adjusted to target the beam to mirror on rear side of the cantilever. Fine adjustment of the platform position to target to the mirror and its placement in the plane perpendicular to photodetector can be performed by rotation of three adjustment screws and turning the platform around its pivot point.

Photodetector is installed in specialized mechanism enabling positioning of the photodiode sensitive area right opposite laser beam reflected off the mirror on the cantilever rear side. To adjust photodetector position, turn adjustment knob on the sensor unit top. Inside the sensor unit a preamplifier is installed near the photodetector to reduce interference and noise in the output signal. Preamplifier provides initial amplification and processing of the photodetector output signal.

1.3 Dipper unit

Dipper unit is a specialized mechanism for lifting and dipping the substrates into liquid. The unit is mounted on the stand at left side of the PTFE trough frame (scheme in Fig. 1.2). Schematic diagram of the unit is shown in Fig. 1.4. Dipper unit provides vertical stroke of the hanging arm 85±2 mm.

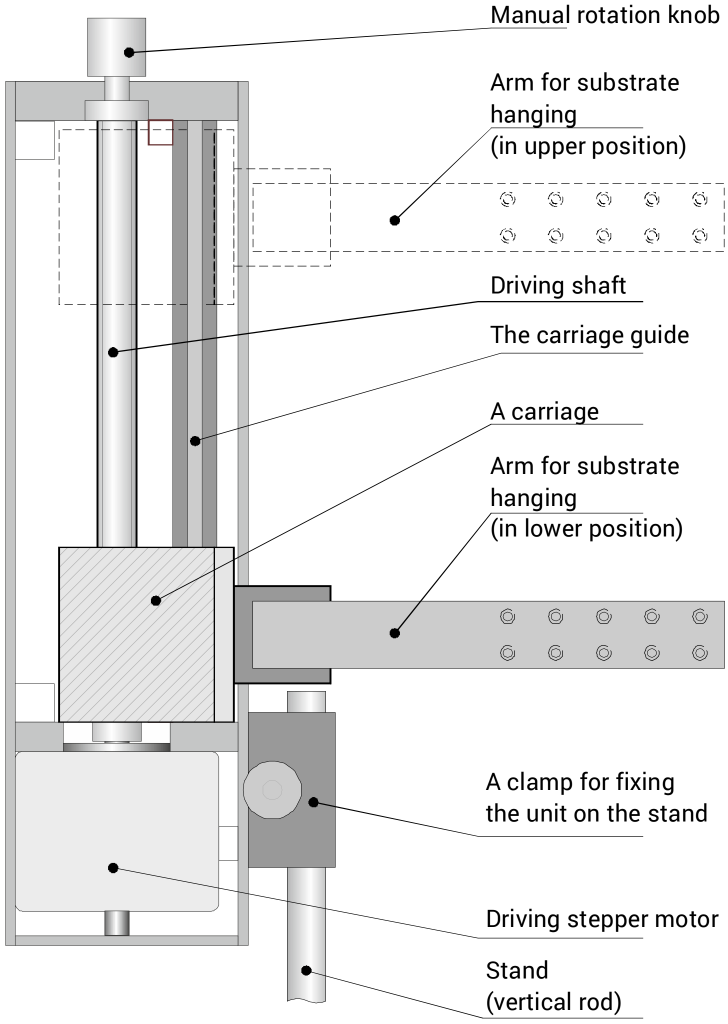


Fig. 1.4 – Schematic diagram of dipper unit.

For the sample (substarte) easy fixation on the hang arm of dipper unit use clamping PTFE holder that can be attached to threaded holes in the arm (Fig. 1.5).

Fig. 1.5 – A PTFE holder for substrate clamping.

The dipping/lifting mechanism is driven by step motor controlled by control electronic unit. Connection cable of the dipper unit is to be plugged to socket at left wall of the trough frame under the stepper motor manual rotation knob. If necessary, vertical positioning of the hang arm can be manually done with knob on top of the dipper unit.

Figure 1.6 shows the trough with all installed units necessary for the device operation.

Fig. 1.6 – LB trough filled with liquid and equipped with surface pressure sensor and dipper unit bearing substarte ready for operation. Barrier is in rightmost (initial) position.

Dipper unit is fixed on its stand (rod diam. 8 mm) by clamp on the unit front wall. If necessary, e.g. for easy access to the clamp fixing screw in alternative applications, the clamp may be remounted from left to right side of the dipper unit.

1.4 Control electronic unit

Control electronic unit is a specialized unit for control over the device for film application according to the commands from host PC. Front panel of the unit bears power switch and service buttons enabling directly perform preparatory or urgent operations with movable elements of the trough without use of control software on host PC (Fig. 1.7). Control electronic unit is connected with LB trough using special cables and with host PC using standard USB-cable (type Am-Bm). To power the unit, it should be plugged to electrical outlet (grounded 110/240 V) using power cordfrom the complete set. Switching of the unit (and the entire device) should be made by the power switch.

CAUTION! Socket of electrical outlet must have reliable grounding. If there is no grounding in the socket, you are to provide grounding for the electronic unit and LB trough to prevent electric shock at the device operation.

The service buttons on the unit front panel may be used for manual control to move the barrier and/or substrate fixed on the dipper unit arm to the desired position.

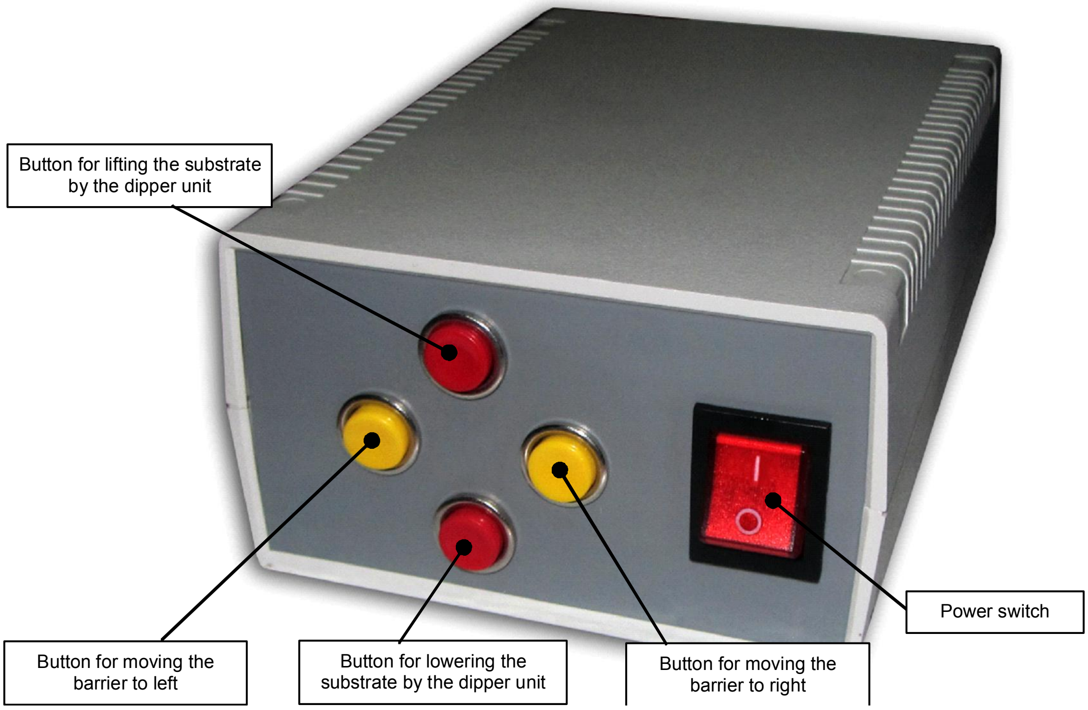


Fig. 1.7 – Front panel of the device electronic unit.

Elements located on the control electronic unit rear panel are shown in Fig. 1.8.

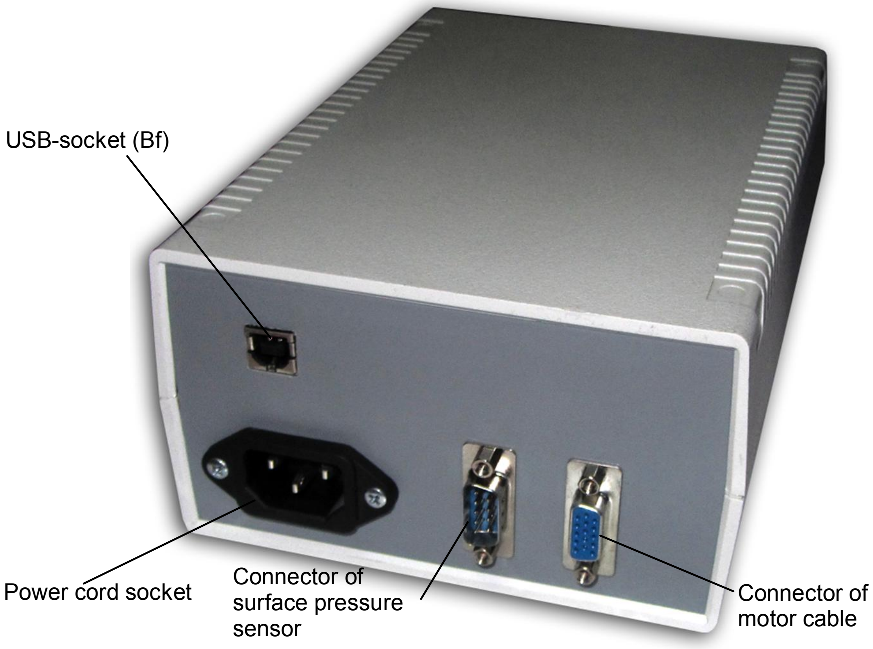


Fig. 1.8 – Elements of rear panel of control electronic unit.

2 LT-111 CONTROL SOFTWARE

Experimental device for film application LT-111 operates under the control of a specialized software **LBTrough control** delivered in the complete set and run on personal computer under Win32 operating system. The control software is installed on the host PC by executing file `lbt2inst.exe` with the setup procedure. After the installation, program **LBTrough control** can be started by running file `lbt.exe` in folder where the software locates on hard disk or by other ways used in the operating system (e.g. double click on the program shortcut in Windows desktop)

2.1 Main window

After start of **LBTrough control** software, main program window appears in the Windows desktop (Fig. 2.1). Main window contains a menu line at its top, toolbar under the menu line, working area in its middle portion and status line at the bottom. Working area is a place where control panels and other interface elements (graph windows, dialogues) are brought up for operation with the device and measured data.

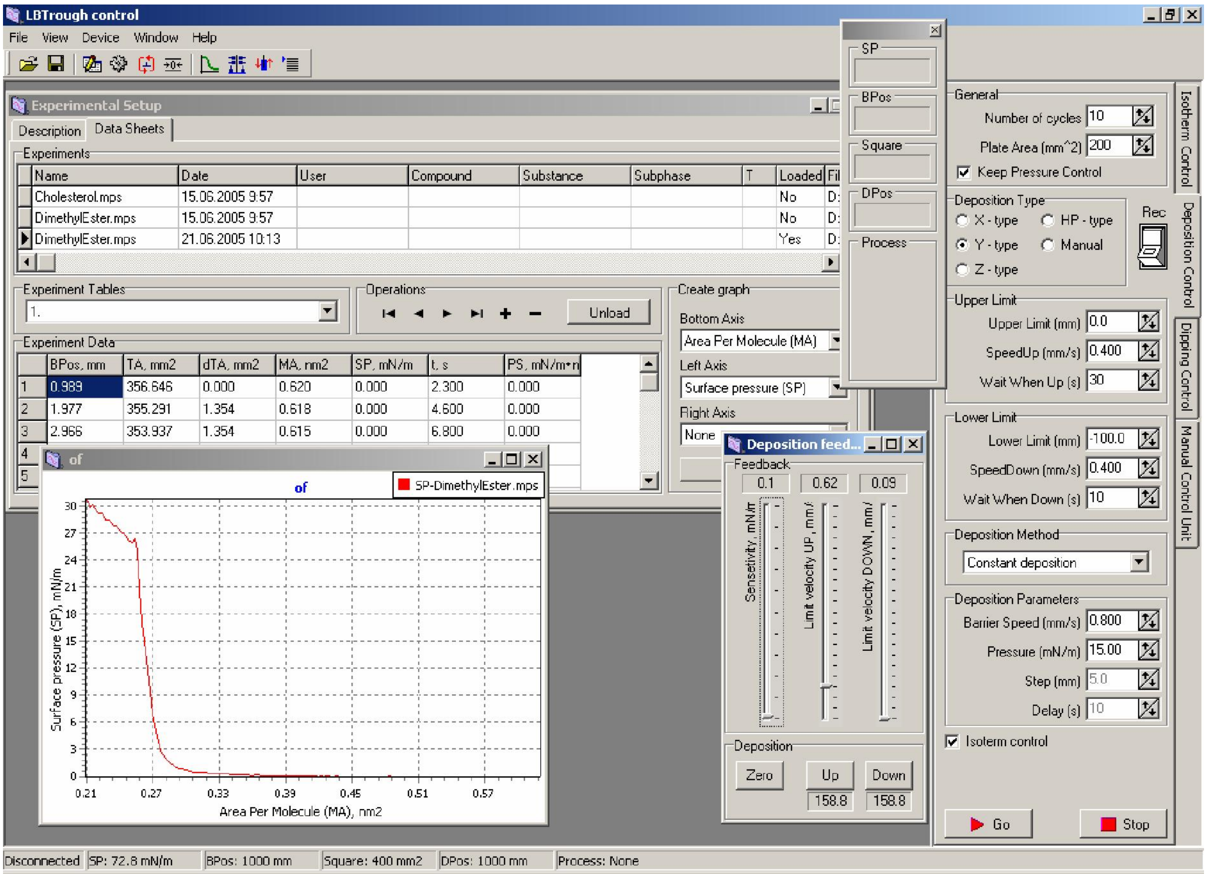
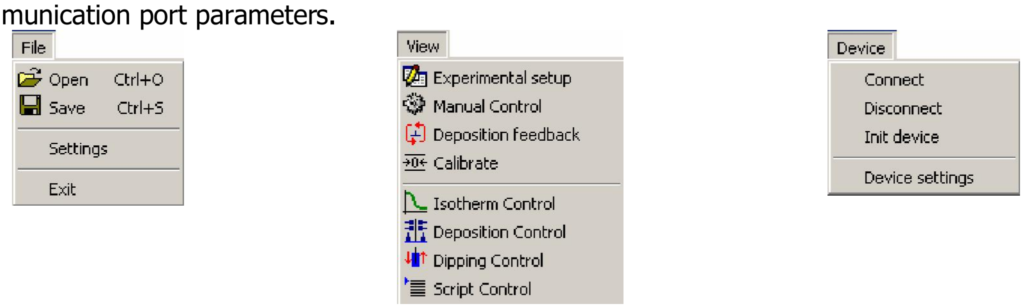


Fig. 2.1 – Main window of **LBTrough control** program.

Control panels **Deposition Control**, **Isotherm Control**, **Dipping Control** and **Manual**

Control Unit can be docked to right border of the working area (Fig. 2.1) to provide more space for other tasks. To dock a panel, drag it by its title to area at the right border. To undock panel, drag it off the docking area fixing corresponding tab at the right border. Working area can be scrolled with scroll bars appeared when panels or windows get outside existing frame.

Program menu provides access to all the functions of control software. Menu item 'File' contains commands necessary for operations with files, general settings of the program and command to exit program (Fig. 2.2 a). Menu item 'View' contains commands for bringing up specialized control panels for operation with the device. Menu item 'Device' provides commands for operations with the hardware: connection with device, disconnection of the device, device initialization, and settings for the communication port parameters.



a b c

Fig. 2.2 – Content of menu items 'File' (a), 'View' (b) and 'Device' (c).

Menu item 'Window' contains standard commands for operations with open windows in the program working area. Menu item 'Help' provides service identification information about the connected device and information about program itself.

Program toolbar contains buttons for quick access to main operations: file save and open dialogues, activation of specialized control panels necessary for work with the device (Fig. 2.3). Toolbar can be detached off the toolbar area under the menu line and placed as floated panel within the desktop.

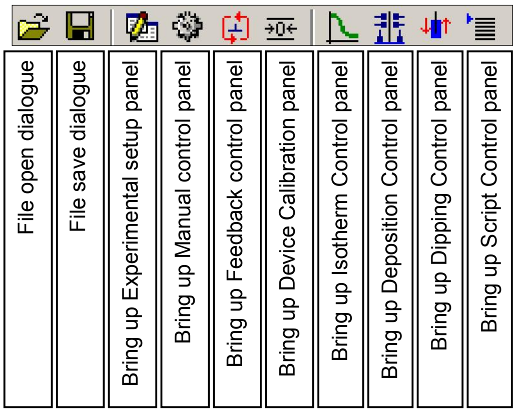


Fig. 2.3 – Functions of buttons in the program toolbar.

At bottom border of the program main window, a status line is located (Fig. 2.4). It provides service information about:

- current device communication status (Connected or Disconnected);
- current surface pressure readings (SP:###);
- current barrier position (BPos: ###);
- current value of free liquid area (Square: ###);
- current dipper mechanism position (DPos: ###);
- currently executed process or its progress status (Process: <*Description*>).

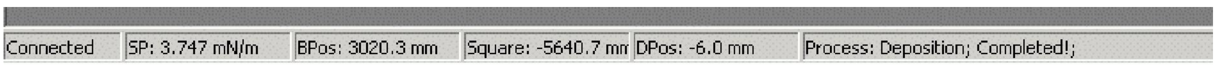


Fig. 2.4 – Example of information in the program status line.

General **Indicator** panel is always visible in the program working area (Fig. 2.5 left). It repeats information displayed in the program status line:

- current surface pressure readings (SP);
- current barrier position (BPos);
- current value of free liquid area (Square);
- current dipper mechanism position (DPos);
- currently executed process or its progress status (Process).

Indicator panel has no controls for hiding it and also a special command for bringing it up. But it is not always on top and can be overlapped by other control panels in the working area.

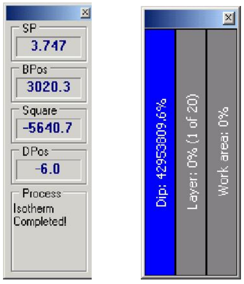


Fig. 2.5 – General indicator panel (left) and indicator of deposition progress (right).

Additionally, when deposition is executed specialized indicator graphically and in text shows progress of the procedure (Fig. 2.5 right):

- left column – relative position of dipper hang arm (100% is upper limit, 0 is lower limit);
- middle column – percentage of deposition program fulfulment (currently deposited layer of total number);
- right column – current free surface as a percentage of full working area. Full working area is 100%. As deposition is running, free surface area decreases that indicates decrease of deposited substance amount in the trough.

2.2 Panel of Experimental Setup

Experimental Setup panel (Fig. 2.6) serves as measured data organizer. It is always in the working area but it can be minimized within it. Two tabs of the panel contain means for describing measured data and used composition (tab **Description**) and controls over the data sets (tab **Data**).

Tab **Description** provides:

- general description of the experiment (group –Data List–);
- assignment of working folder on hard disk for data storing (group –Working Folder–). It is recommended to assign (type or browse to select existing) working folder right after first start of the control program;
- description of the deposited substance (group –Solution Composition–). Table in right part of group –Solution Composition– allows to enter information about the deposited composition constituents or single substance if number of constituents (Number of constituents) is one.

Tab **Data** provides:

- list of experiment sets registered in the program and their description (table –Experiment Registry–). Column Loaded of the table shows the experiment status in current session. To work with the experiment sets in program, they should be loaded (see description of controls below). If necessary, shown values can be manually edited right in the table cells;
- switch between experimental data sets (selector –Data set–). Each experimental data set (data measured at one procedure cycle) is treated as a separate set. One file can contain a series of data sets.
- a viewer pane for displaying selected set of experimental data (table –Data sheet–). If necessary, data values can be manually edited right in the cells;

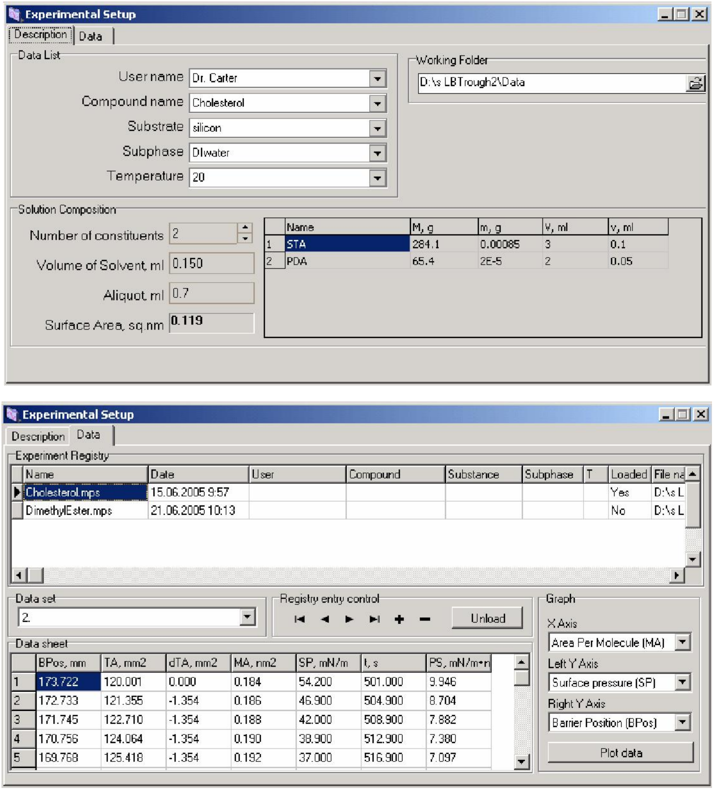
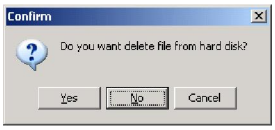


Fig. 2.6 – Experimental setup panel with active tab Description (upper) and Data Sheet (lower).

- controls for loading and unloading experiment sets (group –Registry entry control–). Arrows provide switching between loaded sets in table –Experiment Registry–. Button [+] adds new experiment set to the registry by opening file on hard disk. Inner file format for measured data storing is `*.mps` (MultiPurpose Set) that allows to keep unlimited number of experimental series with arbitrary data organization. Button [–] deletes current experiment set from the list. After pressing button [–] a confirmation will be asked for deleting file with experiment set from hard disk:

  If you will leave the file on hard disk (pressing button [No]), a confirmation for deleting the entry off the program registry will be asked:

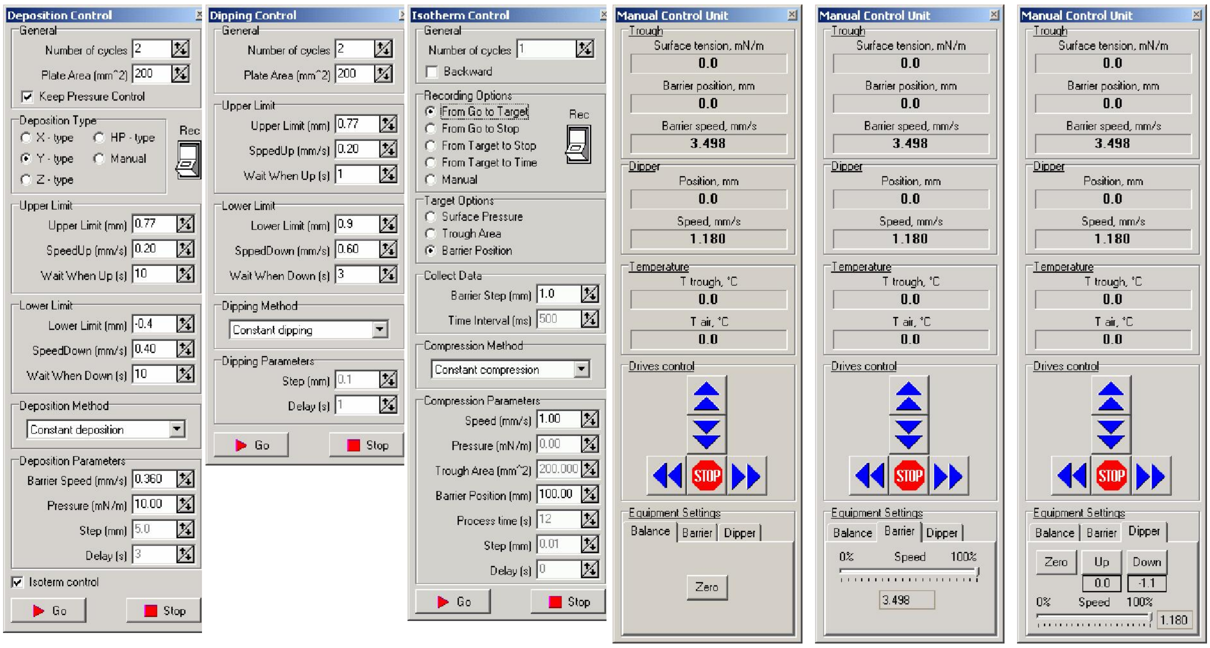
  

  After pressing button [OK], the record will be deleted from the program registry (list of experiment sets).
  Button [Load] / [Unload] serves for loading current experiment sets in the registry if it was inactive or unloading if it was previously loaded;
- plotting of measured data as a graph in specialized window (group Graph). Selectors in the group assign any data type available in the data sheet for bottom (X Axis), left (Left Y Axis) and right (Right Y Axis) axes. To bring the graph window up and create the plot, press button [Plot data] at the group bottom.

2.3 Control panels

Device for film application operates under management from control panels (Fig. 2.7). They are specialized for performing following procedures:

- panel **Deposition Control** – film deposition onto substrate with feedback control and experimental data plotting (Fig. 2.7 a);
- panel **Dipping Control** – programmable dipping of a substrate into liquid without feedback control (Fig. 2.7 b);
- panel **Isotherm Control** – isotherm curve measurement and plotting without deposition (i.e. without dipping substrate into liquid) (Fig. 2.7 c).



a b c d e f

Fig. 2.7 – Control panels of deposition (a), dipping (b), isotherm plotting (c), manual control with active tab of sensor zero point assignment (d), manual control with active tab of setting for barrier movement speed (e) and manual control with active tab of setting for dipper movement speed (f).

Switches Rec in panels **Deposition Control** and **Isotherm Control** when activated provide automated recording of the measured experimental data in the program registry appeared then in list of experiments in panel Experimental Setup and saving collected data in file placed in working directory.

After setting necessary operating parameters, procedure starts at pressing button Go in corresponding control panel. The running procedure can be manually terminated by pressing neighboring button Stop.

Panel **Manual Control Unit** displays in its upper part current readings of surface pressure sensor, positions and speed of barrier (group –Trough–), positions and speed of dipper (group –Dipper–), readings of temperature transducers (group –Temperature–) (Fig. 2.7 d–f).

Lower part of the panel contains buttons for manual control over the device stepper motors (group –Drives control–) (Fig. 2. 7 d–f). They allow to move barrier and dipper mechanism directly without feedback control or dipping program. This function is intended for fast movement of the barrier or hang arm (with or without the attached sample) to position defined by the operator, for example for the trough cleaning or sample change/installation. Control buttons have following functions:

 (up arrow) – start lifting of the dipper mechanism;

 (down arrow) - start lowering of the dipper mechanism;

 (left arrow) – start barrier movement to the left;
 (right arrow) – start barrier movement to the right;
 (a stop sign) – termination of the started motion.

Group –Equipment Settings– in panel **Manual Control Unit** contains three tabs: for sensor zero point assignment (tab Balance) (Fig. 2.7 d), barrier movement speed setting (tab Barrier) (Fig. 2.7 e), and dipper movement speed setting (tab Dipper) (Fig. 2.7 f). In tab Dipper additional buttons provide following functions under manual movement mode:

- Zero – assignment of zero point for dipper hang arm position. To set zero point, move manually the dipper hang arm to necessary position and press button Zero in the tab;
- Up – assignment of upper limit point for dipper hang arm position. To set upper limit point, move manually the dipper hang arm to necessary position and press button Up in the tab;
- Down – assignment of lower limit point for dipper hang arm position. To set lower limit point, move manually the dipper hang arm to necessary position and press button Down in the tab.

All positions manually assigned in panel **Manual Control Unit** will be automatically set as corresponding values in panel **Dipping Control** for next procedures.

Performing deposition, additional settings for feedback control should be made in specialized floating panel **Deposition feedback** (Fig. 2.8). Sliders in group –Feedback– are used for:

- Sensitivity – restriction of the sensor sensitivity. To avoid feedback system response to noise signals, it is better to decrease sensitivity. However, too low sensitivity will result in step-like deposition. Therefore it is necessary to find a balance between high and low feedback sensitivity levels;
- Limit velocity UP – restriction of the barrier motion speed when dipper moves upward. Barrier movement algorithm provides for dynamic speed variation defined by target distance and feedback signal magnitude. The slider allows to constrain maximum available speed of the barrier and avoid its too fast acceleration;
- Limit velocity DOWN – restriction of the barrier motion speed when dipper moves downward.

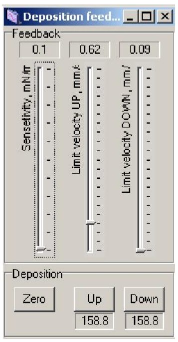


Fig. 2.8 – Control panel for adjustment of feedback parameters at deposition procedure.

Group –Deposition– of panel **Deposition feedback** contains buttons for:

- Zero – assignment of zero point for dipper hang arm position. To set zero point, move manually the dipper hang arm to necessary position and press button Zero in the panel;
- Up – assignment of upper limit point for dipper hang arm position. To set upper limit point, move manually the dipper hang arm to necessary position and press button Up in the panel;
- Down – assignment of lower limit point for dipper hang arm position. To set lower limit point, move manually the dipper hang arm to necessary position and press button Down in the panel.

All positions manually assigned in panel **Deposition feedback** will be automatically set as corresponding values in panel **Deposition Control** for next procedures.

Panel **Script Control** provides control over the device by means of scripts (Fig. 2.9). Tab Script Control serves for writing the device control script in its pane (Fig. 2.9 left). Script for the dipping procedure is automatically generated and is placed into the pane when dipping procedure is started from panel **Dipping Control**. If script already exists and it is in file on hard disk, it can be loaded using 'Open file...' dialogue brought up by button [Load]. To save the script in the pane (typed or generated by program), use 'Save file...' dialogue brought up by button [Save]. Default file name extension for scripts is `*.lds`. The scripts are saved in text format and can be manually edited.

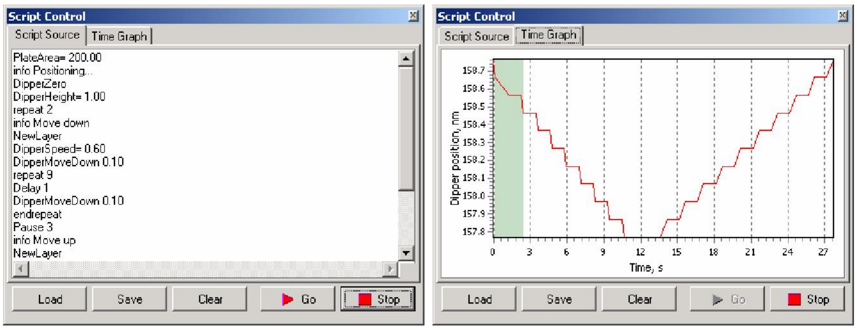


Fig. 2.9 – Panel Script Control.

Tab Time Graph presents timeline for the dipping process and it progress (Fig. 2.9 right).

2.4 Graph plotting window

When deposition, dipping or isotherm plotting procedures start, measured data are plotted in specialized graph windows (Fig. 2.10).

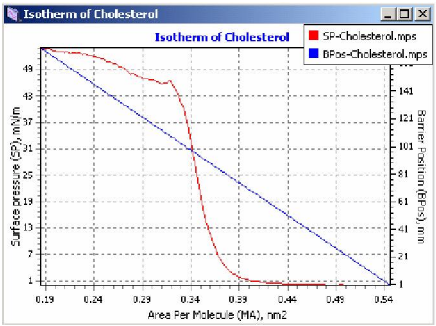


Fig. 2.10 – Example of window for plotting graph.

Isotherm graph is plotted at deposition and isotherm measurement procedures (Fig. 2.10). The curve is plotted as surface pressure (Y axis) vs. free surface area per a molecule (axis X). Surface pressure (Surface pressure, mN/m) is determined by the device as difference between absolute value of surface pressure for pure liquid medium (for water it is 72.8 mN/m) and current pressure of liquid free surface. Current pressure of liquid free surface is mesured by surface pressure sensor. Free surface area per a molecule (Area per molecule, nm^2) is calculated based on barrier position and quantity of substance applied on the liquid surface. Necessary data about the substance should be entered in group –Solution composition– in **Experimental Setup** panel (tab Description).

Deposition procedure also brings up graph window **Deposition** in addition to **Isotherm** plot. Graph **Deposition** plots dependencies of the transferred substance amount (left vertical axis Transfer) and ratio R/Z (transferred substance amount R to number of deposited layers Z, right vertical axis R/Z) on number of deposited layers (horizontal axis).

Dipping procedure brings up graph window **Dipping** where timeline of the dipping process is plotted. Timeline of the set dipping procedure and its progress can be also viewed in tab Time Graph of **Script Control** panel (Fig. 2.9 right).

Graph window is also brought up for plotting earlier collected data using the utility in group Graph of tab Data in **Experimental Setup** panel (Fig. 2.6 lower).

2.5 Configuration panels

Configuration of hardware and program itself for correct operation is performed using specialized panels.

Before the device operation, hardware settings should be configured for correct connection between host computer and control electronic unit. This task is performed in panel Setup (Fig. 2.11) of the program that can be brought up by menu command *Device > Device settings*.

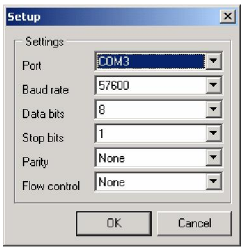


Fig. 2.11 – Hardware configuration panel.

Settings made in the panel **Setup** relate to the serial communication port of the host computer (COM port). Commonly used values for the parameters are as below:

Port	COM1/2/3/4 (depends on the host PC configuring)
Baud rate	57600
Data bits	8
Stop bits	1
Parity	None
Flow control	None

Panel **Calibrate Device** provides access to calibration procedures for the device measuring system (Fig. 2.12). It allows correctly translate relative units used by the system to physical values of distance, area and substance amount. To bring the panel up, use menu command *View > Calibrate* or press corresponding button  in the program toolbar.

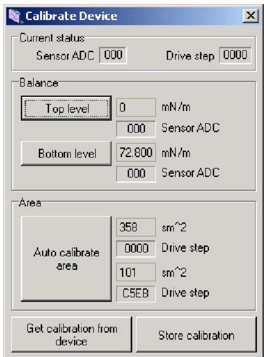


Fig. 2.12 – Panel of device calibration.

Group –Current status– shows current readings of the sensor ADC and number of steps performed by step motor. Group –Balance– contains buttons for setting *minimum* and *maximum* values of the surface pressure sensor measurement range (see also §3.2).

To set minimum value, lift the surface pressure sensor up so that wet sensor plate appeared in air and press button [Top level] ('top level' means that sensor plate is in upper position, i.e. in air). In pane from the button right manually enter number that will be considered as minimum value (usually it is '0').

To set maximum value, lower the surface pressure sensor on its stand so that sensor plate sinks into clean liquid and press button [Bottom level] ('bottom level' means that sensor plate is in lower position, i.e. is sunk in liquid). In pane from the button right manually enter number that will be considered as maximum value (if liquid is distilled water, the number is '72.8').

Group –Area– provide automated calibration of the liquid surface area. After pressing button Auto calibrate area, barrier automatically goes to leftmost and then to rightmost position till limit stops to define the stepper motor step values corresponding to the biggest (leftmost) and the smallest (rightmost) areas of liquid surface. In the panes to the button right manually enter numbers corresponding to physical values of the biggest (upper pane) and the smallest (lower pane) areas of liquid surface. These values are 440 sq.cm (the biggest area) and 128 sq.cm (the smallest area). To check these parameters, use a ruler to measure ranges that the barrier limits in its rightmost and leftmost positions on the free liquid surface correspondingly and multiply the measured values by 16 cm. (Note, exact areas and rightmost and leftmost positions of the barrier are calibrated by the device manufacturer and are stored in the device configuration file lbt.ini)

To remember the set calibration data, press button Store calibration at the panel bottom.

General program settings can be made in panel **Settings** brought up by menu command *File > Settings* (Fig. 2.13). The panel provides adjustment of program options for communication with the device and data accuracy (digits after decimal point) in tab Data and selection of the graphical interface language in tab Interface.

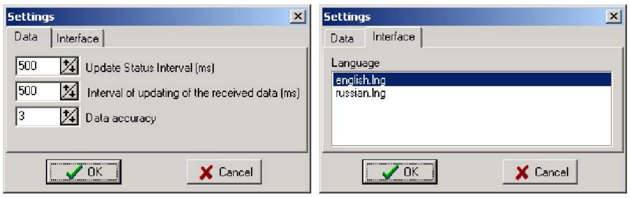


Fig. 2.13 – Panel of general program settings.

Language resources for the program interface are provided by specialized text files in subfolder `Language` located in the program folder. Used file name extension of the resource file is `*.lng`. New language resources can be added to the program by editing text of existing files (it is recommended to keep existing files without change and edit their copy with different name if necessary).

3 GETTING READY FOR OPERATION

3.1 Adjustment of surface pressure sensor

Before working with the instrument, its measuring system should be correctly adjusted. First step of the adjustment is mechanical tuning of the photodetector.

Mechanical adjustment of the photodetector position provides correct targeting of the photodetector sensitive area to laser beam reflected from the flexible cantilever rear side (Fig. 1.3). To adjust the photodetector position, rotate the adjustment knob on upper side of surface pressure sensor (Fig. 1.3). Correct adjustment results in positioning of the photodetector sensitive area center line right opposite center of spot of laser beam reflected from the flexible cantilever rear side.

During primary adjustment of the laser detection system (i.e. photodetector position), it is recommended to check visually position of laser beam spot on the photodetector sensitive area. It should be on right group of the light-sensitive segments and targeted on horizontal separating line as shown in Fig. 3.1. To maximize output signal, the spot should not overlap vertical separating line.

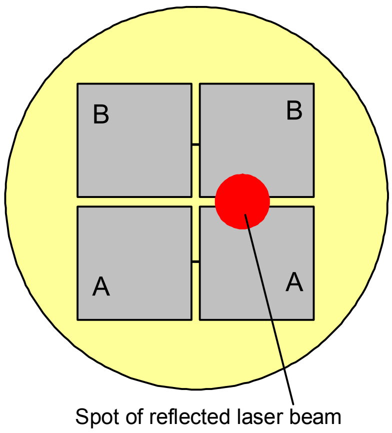
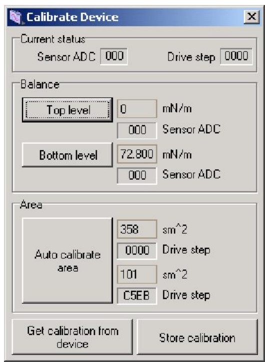


Fig. 3.1 – Correct positioning of laser beam spot on the photodetector sensitive area.



During this adjustment, refer to readings of Sensor ADC in Calibrate device panel of control software (section Current status). Since the value is a differential signal between upper and lower segments of the photodetector, accurate tuning of the photodetector with the reflected laser beam spot in the center will be reached when Sensor ADC readings make «000». Note that zero readings in Sensor ADC may be also when the reflected laser beam spot is outside the sensitive area. Therefore it is necessary to check if Sensor ADC readings change approximately in the same range at slight rotation of the adjustment knob in both directions around its optimal tuned position. As it is said, Sensor ADC readings at correct tuning should change during check rotation of the knob in both directions. After the check, please return the photodetector to the optimal adjusted position (i.e. till zero differential signal wil be reached, Sensor ADC = «000»).

3.2 Calibration of surface pressure sensor

Calibration of surface pressure sensor is second step of the device measuring system tuning. It should be done before operating the device. Aim of this procedure is calibration of the sensor output signal range to provide the measured data reliability. It is recommended to calibrate sensor before each experiment and every time after the Wilhelmy plate change and change of liquid in trough. Sensor calibration procedure includes two stages: (1) calibration of minimum value of the surface pressure sensor measurement range and (2) calibration of maximum value of measurement range of surface pressure sensor.

Preliminarily, tune the photodetector position optimal for given range of measuring cantilever deflection.

1. Attach Wilhelmy plate to the free end of the measuring cantilever. Completely moist the plate either by sinking it in separate vessel with distilled water or lowering it on the stand together with all the sensor unit till reach of liquid surface in the LB trough.
2. Lift the sensor so that the Wilhelmy plate appeared completely in air. Remember (record) the ADC reading s_p^{air1} (Sensor ADC in panel Calibrate device).
3. Lower the sensor unit so that lower edge of the Wilhelmy plate touches the surface of distilled water in LB trough. Remember the ADC reading s_p^{water1} (Sensor ADC in panel Calibrate device).
4. Calculate new readings s_p^{air2} and s_p^{water2} corresponding to new conditions:

   $$|s_p^{air2}| = |s_p^{water2}| = |s_p^{opt}|$$

   and

   $$\mathrm{sign}(s_p^{air2}) = -\mathrm{sign}(s_p^{water2}).$$

   In other words, it is necessary to calculate

   $$s_p^{opt} = (s_p^{water2} - s_p^{air2})/2,$$

   and with that new readings will be

   $$s_p^{air2} = -|s_p^{opt}|$$

   and

   $$s_p^{water2} = +|s_p^{opt}|.$$

5. Using the adjustment knob on upper side of surface pressure sensor (Fig. 1.3) shift the photodetector to new 'zero' position in which deflections of measuring cantilever at Wilhelmy plate in air will be

   $$s_p^{air2} = -|s_p^{opt}|,$$

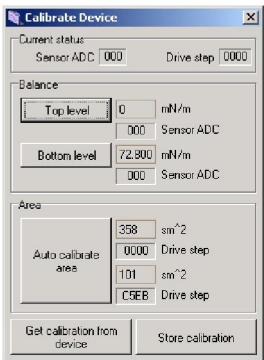
   and after touching of the aqueous surface by the Wilhelmy plate

   $$s_p^{water2} = +|s_p^{opt}|.$$

   (Main conditions here are change of sign of the ADC readings in these two utmost points and equal ADC readings for the cantilever deflections from the central (zero) point.)
   In practice, if the plate in a given case touches aqueous surface, it is necessary to shift the photodetector (using the adjustment knob) to the position in which Sensor ADC readings in panel Calibrate device will make

   $$s_p^{water2} = +|s_p^{opt}|.$$

6. Check new ADC reading for the plate in air – lift the sensor unit so that wet Wilhelmy plate appeared completely in air and check Sensor ADC reading in panel Calibrate device: it should be $s_p^{air2} = -|s_p^{opt}|$. If new readings significantly differ from the described conditions, repeat procedures 2–6 to reach maximum proximity to the conditions in point 5.



The described preliminary adjustment of optimal position of the photodetector is recommended to fulfill after change of the measuring cantilever configuration (spring constant) or after critical change of the photodetector position during previous experiments.

Directly, calibration of minimum and maximum sensor readings is performed with activated **Calibrate Device** panel (menu commands *View > Calibrate* or button  in the program) and includes following two steps.

First step – minimum value calibration. Lift the sensor unit so that wet Wilhelmy plate appeared completely in air. In panel **Calibrate Device**

press button Top level in section Balance – the measuring system will fix the value corresponding to zero surface pressure.

Second step – maximum value calibration. Lower the sensor unit so that lower edge of the Wilhelmy plate touches the surface of distilled water in LB trough (or in other vessel). In panel **Calibrate Device** press button Bottom level in section Balance – the measuring system will fix the value corresponding to surface pressure of pure liquid.

> Pay attention that number to the right from button Bottom level corresponds to typical value of surface pressure for given pure liquid in the trough. Default value for pure distilled water is 72.8 mN/m.). However, if you plan to use different medium in the experiments, enter corresponding actual value in the pane.

After finishing both calibration steps, press button Store calibration in bottom of panel **Calibrate Device** so that the measuring system remembers the changes and uses the new values in its operation.

4 PRACTICAL GUIDE TO OPERATION WITH EXPERIMENTAL DEVICE FOR FILM APPLICATION LT-111

Experimental device for film application LT-111 is intended for studies of surface active properties of amphiphilic compounds, formation of monomolecular films on aqueous surface and modification of solid substrates by mono- and multimolecular Langmuir–Blodgett films.

At the operation with LT-111 device, following stages usually should be fufilled:

1. Thoroughly (with accuracy not worse than $0.1*10^{-4}$ g) weigh the studied substances and prepare their 1–0.1 mMole solutions in appropriate solvent (e.g. chloroform, benzole, hexane).
2. Switch the control electronic unit on, run control software on host PC, and thoroughly calibrate the surface pressure sensor (it operates based on measurement of surface tension by Wilghelmy technique).
3. Apply the studied compound (its solution) on to the aqueous surface.
4. Get an isotherm in coordinates 'surface pressure – area per molecule, π-A'.
5. Choose the deposition method and construct the film of intended thickness on he substrate.

Design of LT-111 device also allows depositing compounds from solution by cyclic dipping of the substrate into the liquid at preset speed according to the assigned script. Below, you can find details for each of these steps of the device operation.

4.1 Switching the device on and calibration

Surface pressure sensor has sensitivity of order 0.02 mN/m and it is recommended to calibrate it before every isotherm measurement.

ATTENTION! Do not expose photosensitive element of the sensor to direct sunlight to avoid drifting of zero point of the surface pressure signal.

The sensor should be calibrated in two positions. Fully moist Wilghelmy plate being completely in the air (out of contact with liquid) corresponds to zero surface tension.

1. After filling the trough with liquid (usually with water), dip the surface pressure unit so that the Wilghelmy plate hung on the sensitive cantilever immersed in the liquid in got fully moist (e.g. for 3–5 min).
2. Lift the sensor so that the plate appeared completely in the air.
3. Press button UP in 'Calibrate device' window (Fig. 4.1) to store the value of the moist plate weight and assign zero level to this value.
4. Lower the sensor unit to sink the plate with its lower edge into water.
5. Press button DOWN in 'Calibrate device' window to store the surface tension value corresponding to clean aqueous surface, value 72.8 will be assigned to current ADC reading.

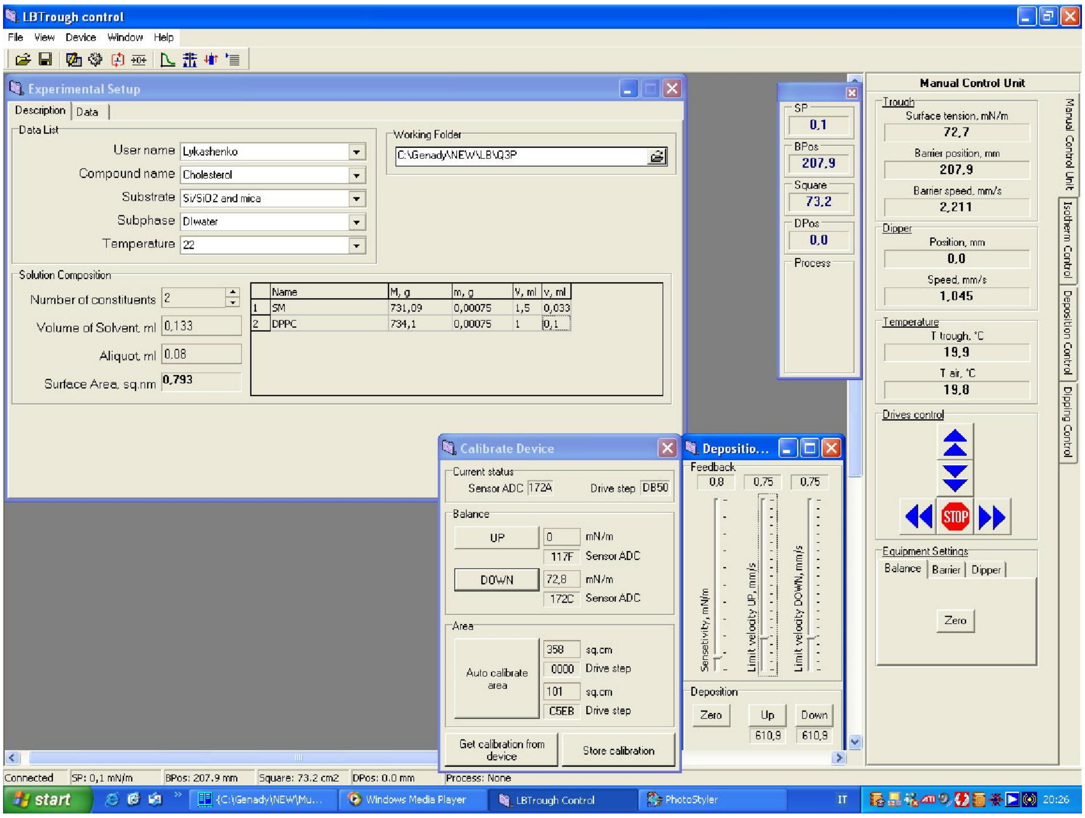


Fig. 4.1 – Main windows of the LT-111 control software.

In panel ‘Manual Control Unit’ (Fig. 4.2), parameter Surface Tension will get value 72.8 mN/m, and parameter SP (Surface Pressure, Fig. 4.1) in General Indicator panel will be 0 according to the equation

$$\pi = -(\sigma - \sigma_o) = 0,$$

where σ_o is surafce tension of clean water surface 72.8 mN/m at 25 °C; σ is current value of surface tension during the monolayer compression.

After pressing button Store Calibration in window 'Calibrate Device', Fig. 4.1, the program transfers the assigned parameters to the control electronic unit to store them there.

There is an option to calibrate the device based on the area using the standard substance with known value of minimal area per a molecule in close-packed monolayer, e.g. stearic acid. For that, record initially an isotherm and determine minimum area A =0.205 nm^2.

Note that displayed value of a parameter may differ from its actual one and it can be conditioned by two reasons: wrong weighing and application of the substance onto aqueous surface (as usual) or incorrect calibration of the device. To change minimum and maximum position of the barrier, press button Auto Calibrate Area and following prompts in the program enter minimum and maximum barrier positions so that the trough working area was as closer to its actual size as possible.

In the device sensor, there is also an option to change the sensitive cantilever spring constant to adjust its sensitivity. After change of the cantilever free end length by moving the sliding slat on sensor unit bottom side please don’t forget to readjust the laser detection system so that working stroke of the reflected spot on the photodetector during measurements appeared within photosensitive area of the latter.

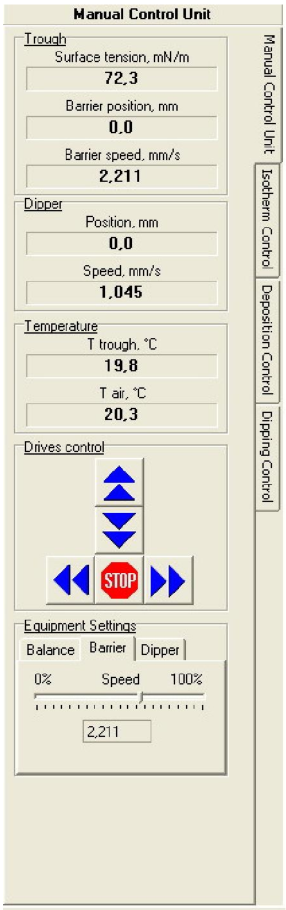


Fig. 4.2 – Panel "Manual Control unit"

4.2 Measurement of compression isotherms

Before application of the working solution onto aqueous surface, manually clean thoroughly the working area. For that, pressing buttons Left and Right in 'Manual Control Unit' panel (Fig. 4.2) move the barrier to position of minimum working area and clean the constrained aqueous surface with a water jet pump. Then move the barrier to position of maximum working area and calibrate the surface tension sensor according to §4.1.

After application of necessary amount of substance onto aqueous surface, fill information in section Data in panel 'Experimental Setup' (Fig. 4.1): operator (user) name, the substance name (abbreviated or contracted), type (nature) of the substrate, composiiton of the subphase, temperature. Enter also number of compounds in the solution, volume of solvent, molecular weight of substance, weight and volume of sample applied onto aqueous surface. If the table in pane 'Surface Area' is filled in correctly, maximum area per a molecule for given experiment will be automatically calculated and displayed.

In case of multicomponent solutions, operator instead of filling in value 'Volume of Sovent' should enter in the table the data describing solutions of individual compounds BEFORE their mixture, namely molecular weights (M, g), sample weights (m, g) and solvent volumes (V, ml) used for the sample preparation. In the last column, enter the aliquot volumes taken for the component mixture and preparation of working solution with necessary proportion of the components. The program will automatically add these values and calculate the total volume of working solution in field 'Volume of Solvent'.

Next stage of the experient is obtaining of isotherm under parameters set in panel 'Isotherm Control' (Fig. 4.3) that can be activated by selecting corresponding tab in panel 'Manual Control Unit'.

The operator can assign necessary number of cycles of the isotherm recording (usually 1), enable or disable the isotherm recording at the barrier backward motion (option ‘Backward’), enable automatic recording of the isotherm by switch ‘Rec’ (red color corresponds to enabled option). Other Recording Options allow to specify how to measure the isotherm: till reaching the preset value target parameter, till manual stop, from preset value of target parameter till manual stop, from preset value of target parameter during the assigned time interval, under completely manual control by operator (using buttons Left and Right in ‘Manual Control Unit’ panel). The target parameter can be chosen between surface pressure, the trough surface area and barrier position. The operator also assigns compression type (Constant or with Delay), compression speed and the barrier step defining points where area per molecule and corresponding surface pressure will be measured. The less value ‘Barrier Step’, the more number of measurement points recorded in data file and usd for diagram plotting. Automatic measurement of isotherm begins at pressing button Go.

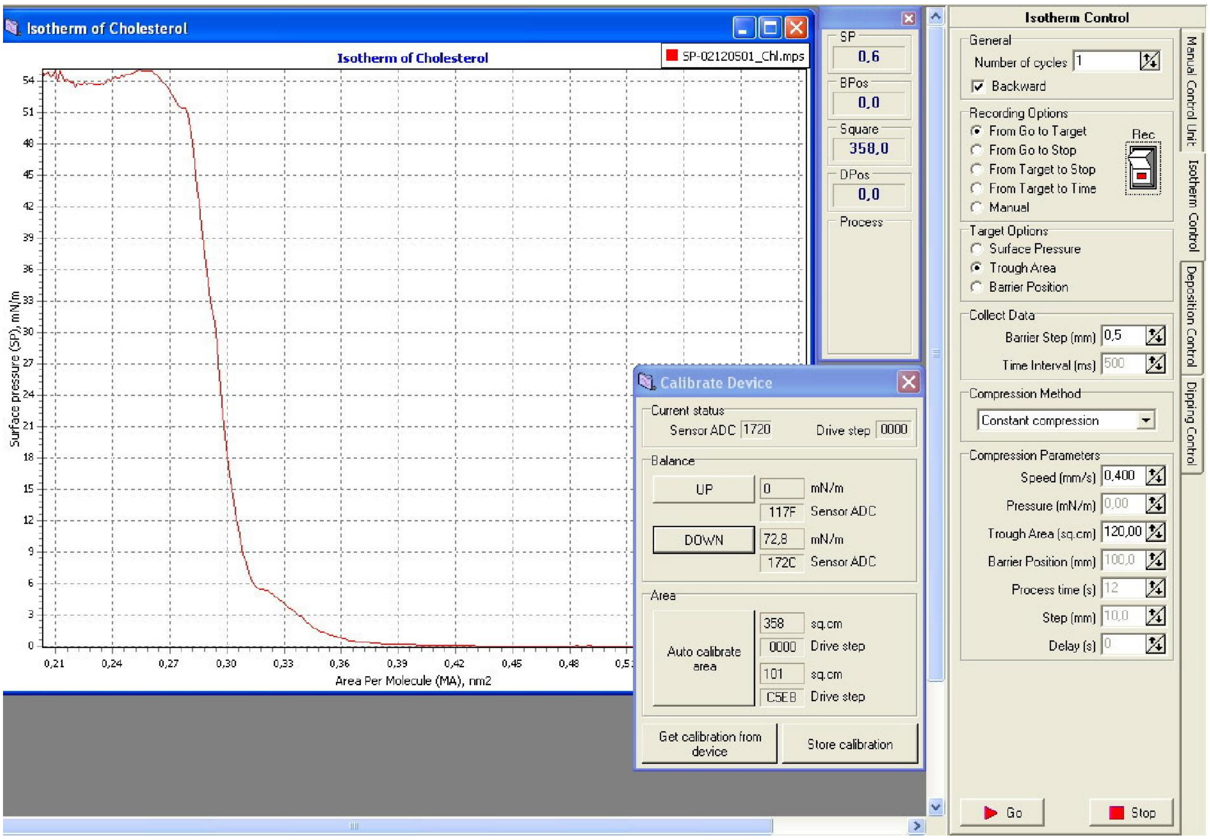


Fig. 4.3 – The program windows used during investigation of substance monolayer behavior under compression.

4.3 Deposition of mono- and multimolecular films

To deposit quality mono- and multimolecular films, the operator should use appropriate surfactants and choose optimal deposition pressure value.

Preparatory tasks before the deposition start include cleaning of the aqueous surfce from contaminations, the sensor calibration and application of a substance onto aqueous surface. Deposition process parameters should be set in panel ‘Deposition Control' activated by selecting corresponding tab in panel ‘Manual Control Unit’ (Fig. 4.4).

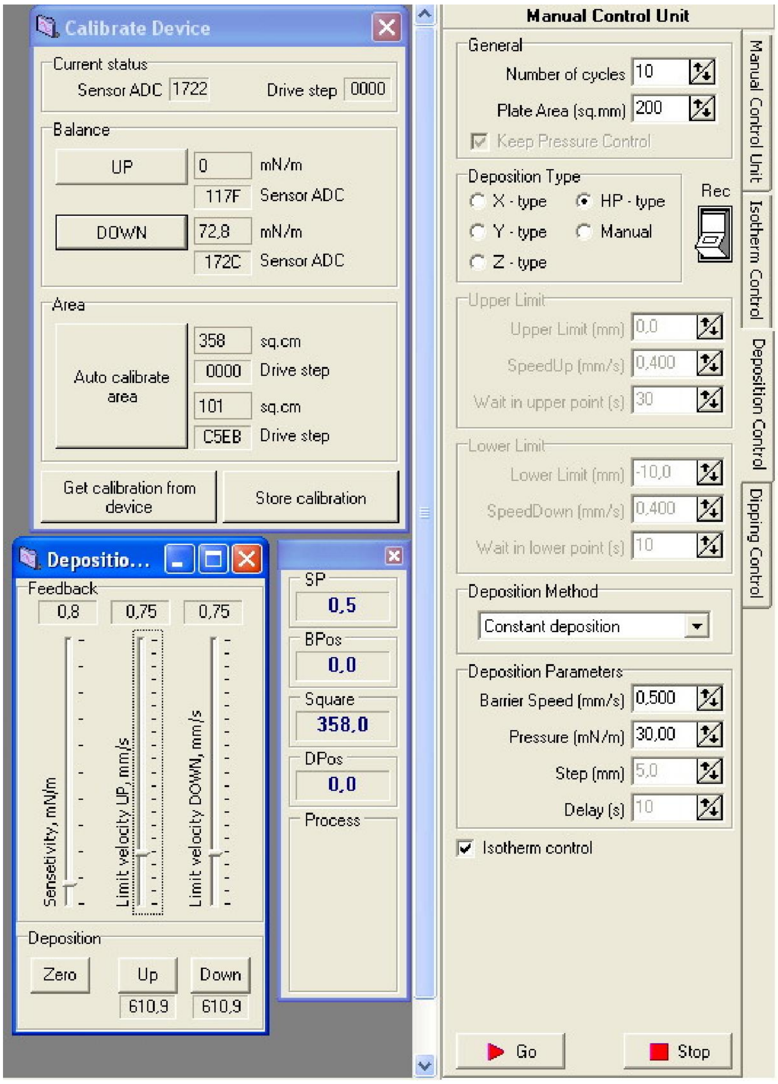


Fig. 4.4 – The program windows and panels used for control over the film deposition process.

Available are three deposition modes: automatic by traditional vertical (LB) method of formation of structures of X-, Y-, Z-types, horizontal precipitation of monolayer and film deposition under manual control. In case of vertical deposition method and formation of multimolecular films, assigned should be number of cycles, entered the substrate surface area (both sides), deposition type, way of deposition (with constant speed or by steps with delay) and other parameters like barrier speed, pressure of deposition, speeds of the substarte movement up and down, regimes of drying/moistening. additionally can be set step of the substrate motion and delay at step-like dipping/lifting of the substrate.

Depending on deposition speed, construction of films of three types is available: X- and Z-types of non-centrosymmetrical LB films and Y-type of centrosymmetrical LB films (Fig. 4.5). To deposit X-type film, substrate slowly dips through the monomolecular film at certain pressure and then quickly moves up. To deposit Z-type film, all is vice versa – substrate quickly dips through monomolecular film at certain pressure and then slowly moves up. Bilayered centrosymmetrical LB films can be constructed at various speeds of substrate motion through free surface of liquid in trough.

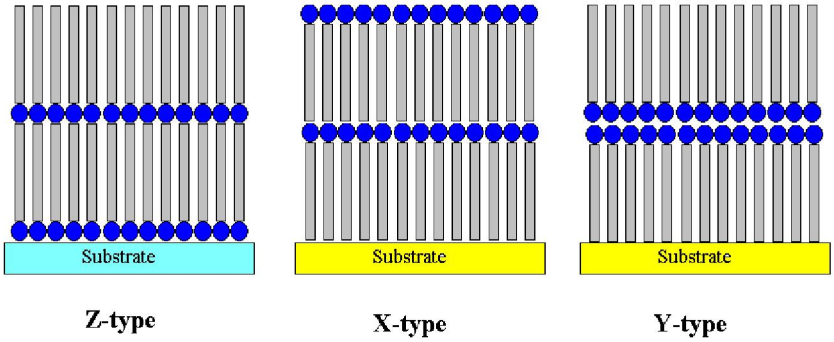


Fig. 4.5 – Schematics of Langmuir–Blodgett film of different structure.

Activated option 'Isotherm Control' means that during the deposition process not only the process progress is displayed (diagrams showing the film transfer degree) but also surface pressure changes during the deposition process will be plotted (Fig. 4.6).

From Fig. 4.6 it is seen that in case of deposition of behenic acid, quantitative transfer of the monolayer onto sustrate takes place with transfer ratio 1. Right lower image in Fig. 4.6 displays feedback control panel for system ‘substrate–barrier’.

Selection of HP mode in section ‘Deposition Type’ will result in compression of monolayer by barrier till the set value of surface pressure. Then the operator should drain the water to lower the free surface level below the substrate placed on a specialized rig (table) in the dipping well.

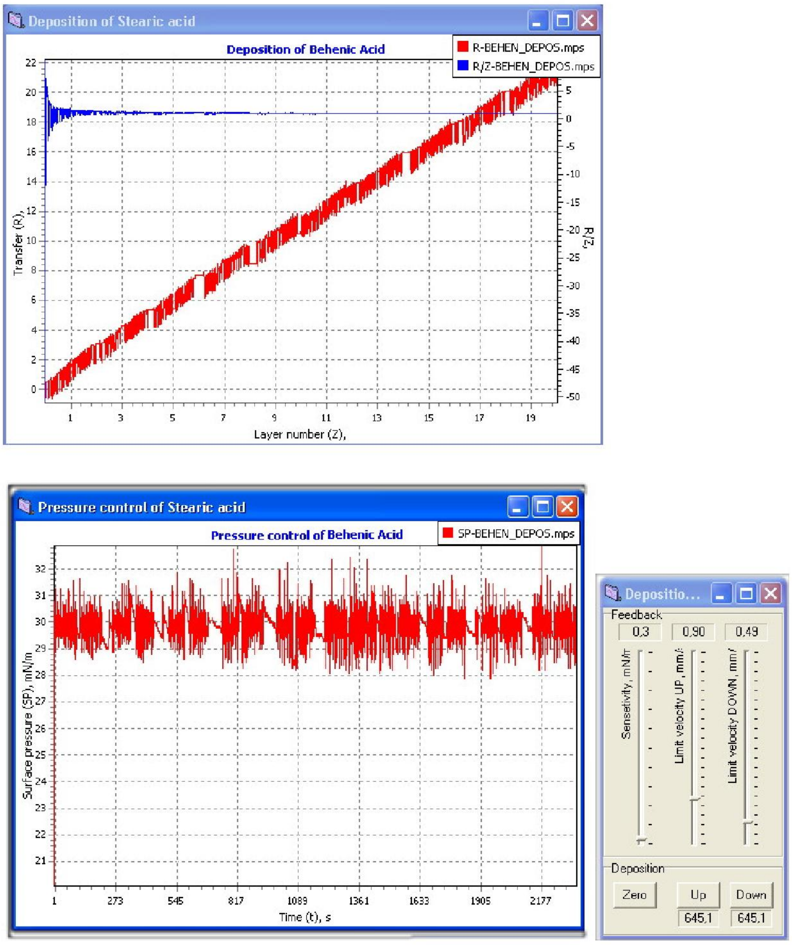


Fig. 4.6 – Example of transfer process visualization for behenic acid film.

4.4 Data processing

Operator can save and restore data using tab 'Description' in window 'Experimental Setup' (Fig. 4.7) or open recorded data files via menu 'File > Open'.

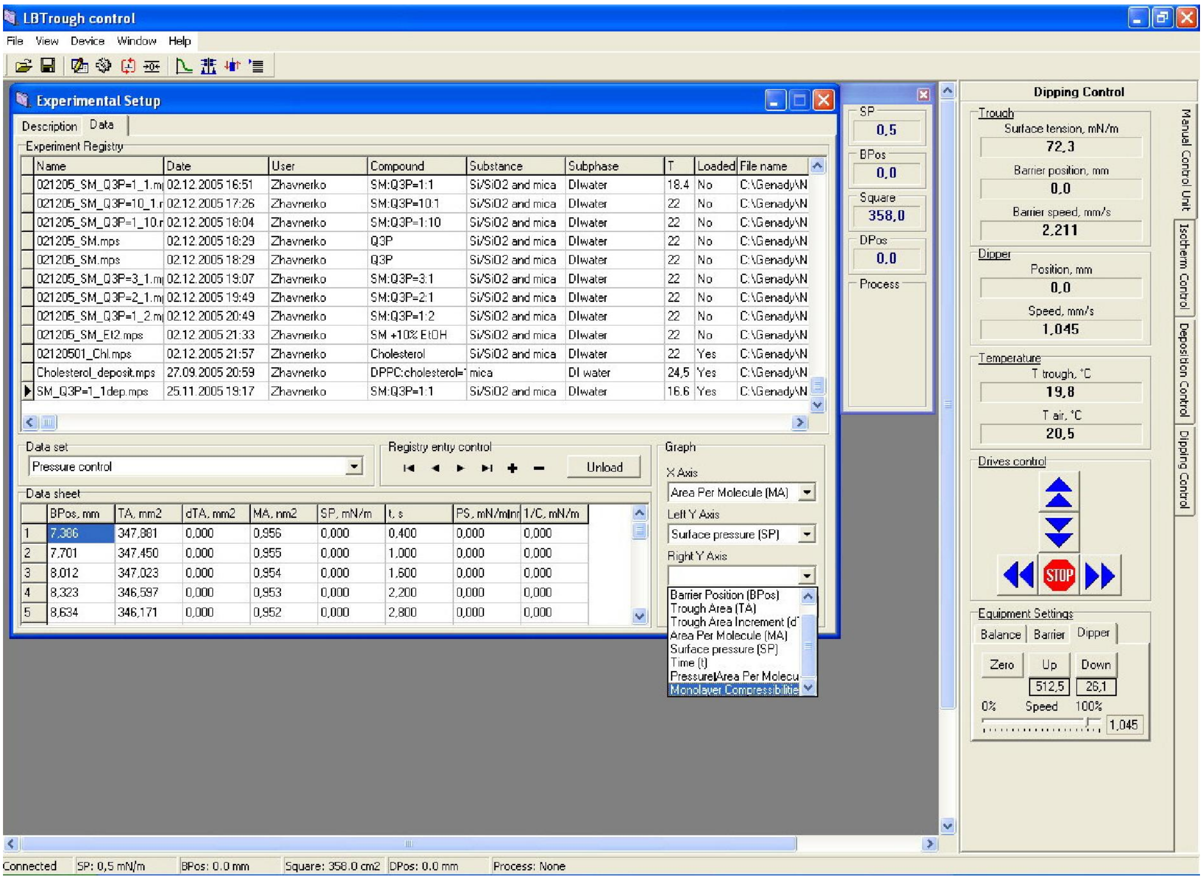


Fig. 4.7 – Main window displaying recorded isotherms and used for data file searching and opening.

In the latter case, pane Data Sheet displays the recorded data. Operator can choose mode (Isotherm or Deposition) in field 'Data Set', assign axes (X and Y) and plot the diagram by pressing button Plot Data. After that the diagram will be displayed. The diagram properties can be adjusted in additional panel 'Graph Properties' brought out at double click in any place of the diagram window (Fig. 4.8).

Tab 'Graph' allows to change the diagram title, its color and font. Additionally can be changed the diagram background color, axes color, displayed of hidden grid (option 'Grid'), adjucted the grid color, displayed or hidden the diagram legend (option 'Legend') (Fig. 4.9).

There is also an option to display multiple graph lines in the same diagram area (Fig. 4.10) that enables direct data comparison. To add additional lines into the diagram, use section 'Lines' in tab 'Axis' > 'Left' or 'Axis' > 'Right'. Operator can select name and assign data string to the selected line (correspondingly, selectors 'Experiment' and 'Data' in section 'Source'). Section 'Line Properties' allows to adjust the selected line displaying parameters.

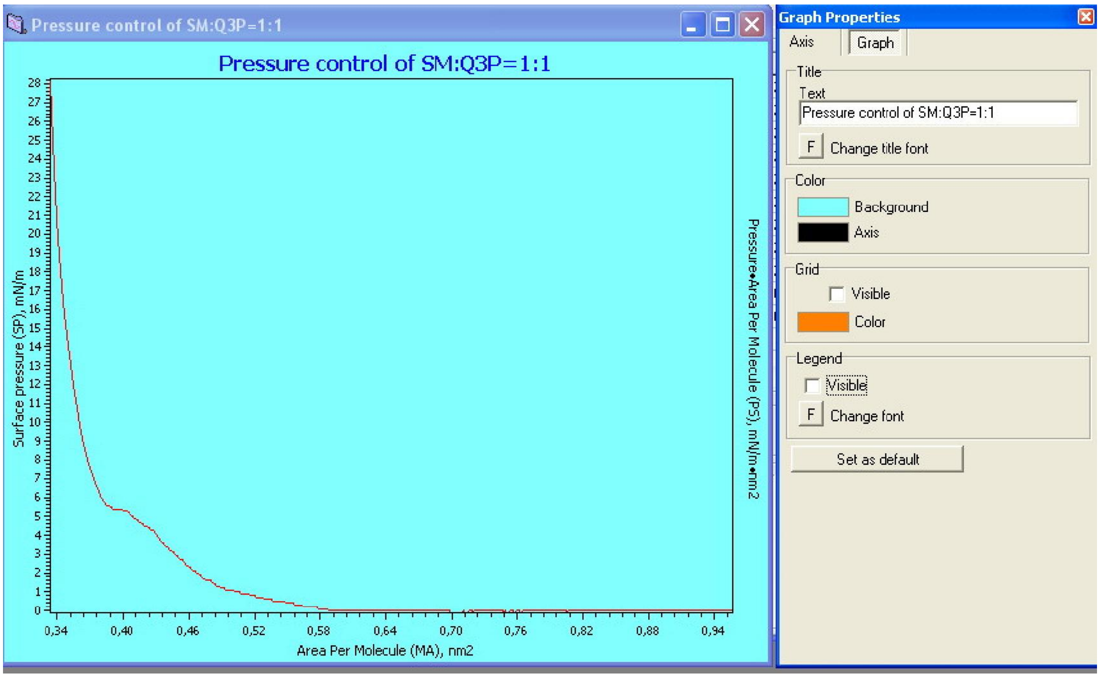


Fig. 4.8 – E[ample of editing the diagram window.

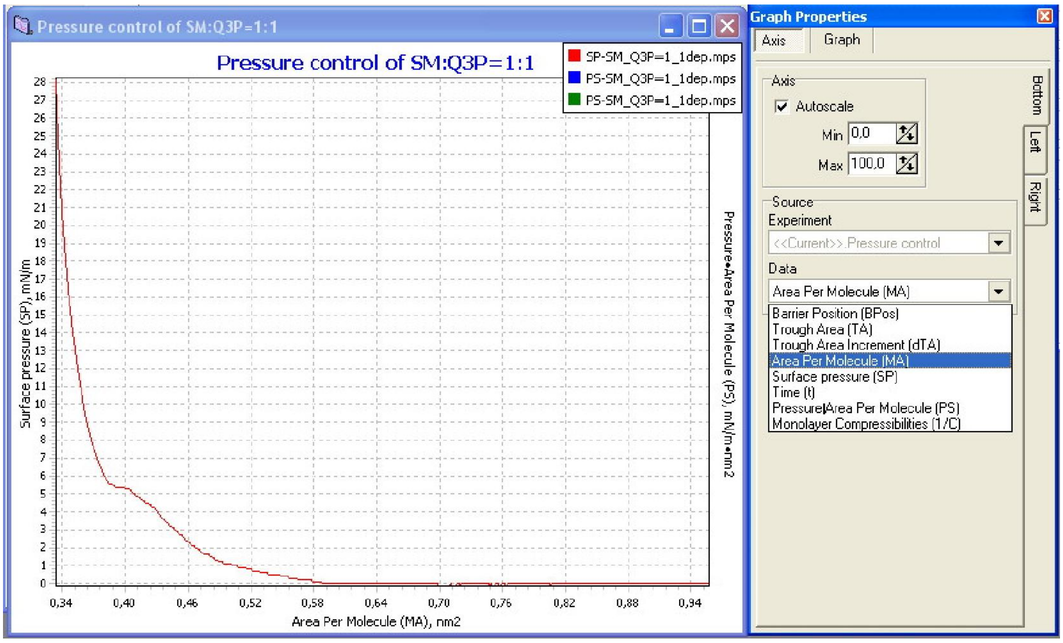


Fig. 4.9 – Example of diagram with legend (list of axes types).

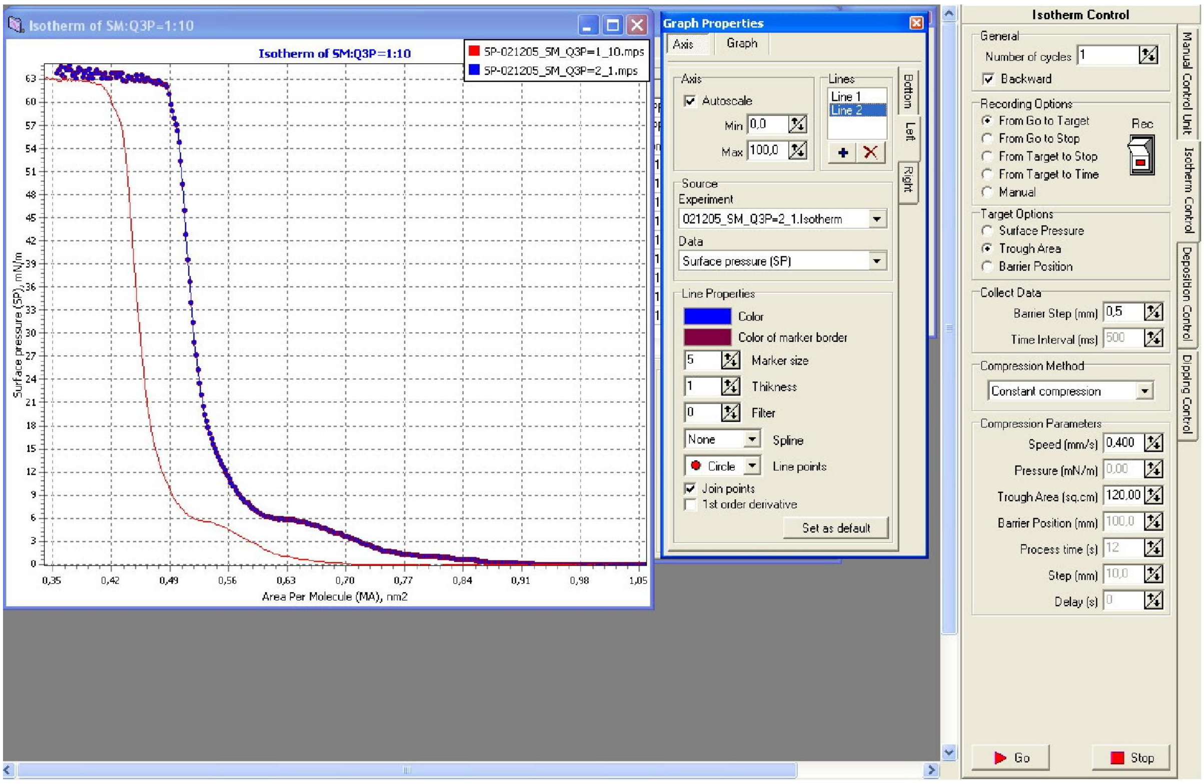


Fig. 4.10 – Example of plotting multiple diagram in one field.

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