



PAUL N GARDNER COMPANY
9104 GUILFORD ROAD, SUITE H
COLUMBIA, MD 21046 USA

EMAIL GARDCO@ALTANA.COM
PHONE +1-954-946-9454



INSTRUCTIONS

GARDCO DISTRIBUTED PRODUCTS



The *PGX+* Measuring Head



User's Manual Program version 4.0

Distributed By:

GARDCO

PAUL N. www.gardco.com

GARDNER

COMPANY, INCORPORATED 1-800-762-2478

List of Contents

1. **Unpacking your instrument**
 2. **Technical requirements**
 3. **Installing the PGX+software**
 4. **Calibration and Validation**
 - 4.1 **Calibration Procedure**
 - 4.2 **Validation Procedures**
 5. **Setting up the PGX+instrument**
 - 5.1 **Preparing a specimen**
 - 5.2 **Special specimens**
 - 5.3 **The Pump Dispenser**
 6. **Miscellaneous**
 - 6.1 **Sample preparation**
 - 6.2 **Setting up a test**
 - 6.3 **Instrument storage**
 - 6.4 **Transportation**
 7. **Test modes**
 - 7.1 **Drop application Static Mode**
 - 7.2 **Automatic testing in Static Mode**
 - 7.3 **Drop application in Dynamic Mode**
 - 7.4 **Automatic testing in Dynamic Mode**
 - 7.5 **Automatic testing in Advancing Mode**
 8. **Reports**
 9. **Software functions**
 - 9.1 **The Image Control Panel**
 - 9.2 **Main Program**
 - 9.3 **Static Mode**
 - 9.4 **Dynamic Mode**
 - 9.5 **Manual testing in Static/Dynamic Mode**
 - 9.6 **Advancing Mode**
 - 9.7 **SuperLow contact angles**
 - 9.8 **View Mode**
 - 9.9 **Surface Tension**
 - 9.10 **Surface “free” Energy (SFE)**
 - 9.11 **Options Screen**
 - 9.12 **Enhance Mode**
 - 9.13 **Baseline & Curtain settings**
 - 9.14 **Camera Gain & Threshold**
 - 9.15 **Field of View limitations**
 - 9.16 **Use of other test liquids (SFE)**
 - 9.17 **Translation of the User Interface**
 - 9.18 **System parameters (Options)**
 10. **Remote Control functions**
 11. **Spare Parts and Consumables**
 12. **Trouble Shooting**
- Appendices**
- A.1 Recommended droplet sizes
 - A.2 Detection of droplet in Dynamic Mode
 - B.1 Experienced problems
 - C.1 Surface Energy Conversion Chart
 - D.1 Replacement of Pump Tubing
 - D.2 Adjustment of Pump Volume
 - D.3 Small droplets below 0.5 µl

Technical Specifications

Image Capture Rate:	80 frames/second
Droplet size:	0,1 – 10 µL
Drop application:	Synchronized
Drop Application Modes:	Touch Down / Impact / Manual
Field of View:	6,4 x 4,8 mm
Sensor Resolution:	640 x 480 pixels
Pixel resolution:	10 µm
Communication	USB2.0



***This User's Manual supports all models of the PGX+ Measuring Head
in combination with the PG program version 4.0***

1. Unpacking your instrument

Your PGX+instrument is delivered in a carrying case including

- the PGX+software on a CD
- a one millilitre syringe
- a calibration plate
- a glass bottle
- a PGX+Accessory kit
- a copy of this User's Manual

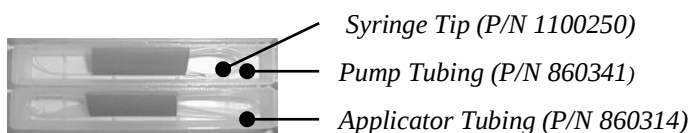


Fig. 1.2 PGX+ Accessory Kit



Fig.1.3 PGX+ Instrument Case

2. Technical requirements

This version of the PGX+software can be installed under the following operating systems:

- Windows 8
- Windows 7 (32/64-bit)
- Windows XP
- Vista 32/64



Please note the PGX+ instrument cannot be installed under Windows NT, as this operating system does not support the use of USB ports.

3. Installing the PGX+software

Your software is delivered on a CD located under the instrument padding inside the case.

 *DO NOT download new camera drivers from Internet even when Windows may suggest this as program performance might then become unreliable!*

 *DO NOT connect the PGX+ instrument to the USB port until requested by the program!
Otherwise Windows will attempt to search for other drivers and the result is unpredictable.*

- a) Insert the CD to start the installation and follow the displayed instructions.
- b) You will be prompted for a password, which is "**plus4**".
- c) The application software is then installed automatically and a message "*Software has been successfully installed*" should appear on the screen.
- d) Finally a message may appear "*This system must be restarted to complete the installation*"
Press **[OK]** to continue.
- e) Connect the PGX+ instrument to the USB port you wish to use.
- f) Start the application from the PGX+ icon on the Desktop or from C:\fibro\bin\PGPlus .EXE.
Successful installations will recognise the camera inside the PGX+ instrument and an image should appear in the Live Image Screen.

4. Calibration and Validation

Your PGX+instrument has been carefully assembled and checked during production. It should not be necessary to do any further adjustments. For a correct performance, however, the instrument must be calibrated according to the procedure below. Without a correct calibration the instrument readings will be incorrect.



Before calibration, your instrument should be connected to the USB port for 30-60 minutes in room conditions. Make sure the drop applicator is installed to avoid stray light shining into the instrument as this may have an impact on the calibration.

4.1 Calibration Procedure (Perspex plate)

- a) The steel ball must have its top 2.6-3.0 mm above the Perspex surface. With a steel ball outside these dimensions calibration might fail.
- b) Select “Calibrate” from the Image Control Panel.
- c) Locate the reference mark at the edge of the instrument sole. The calibration plate has a similar mark indicating the “full sphere”. Place the PGX+instrument on top of the calibration plate with its reference mark positioned above the dot as indicated in Figure 4.2. Push gently on top of the instrument to make sure the instrument rests firmly against the calibration plate surface.

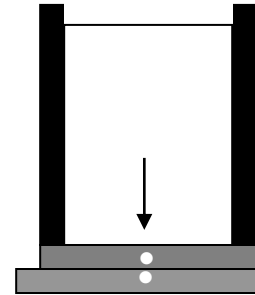


Fig 4.1 PGX Reference mark

- d) Click on the **[Start]** button to find the optimum Threshold setting.
- e) Next the automatic scale factors are determined from the image. This is a high precision operation, which requires a clean sphere. If defects are detected an error message “Unable to find sphere” will appear. If debris is found at the sphere contour, remove the instrument from the calibration plate and clean the steel ball with a cotton swab dipped in Isopropanol. Select “Camera” and “Calibrate” to restart the calibration routine.
- f) When the calibration routine has terminated, the results are displayed for the semi-sphere above the diameter with the following target numbers:
 - Height (c-top) = 1,00 ± 0.02 mm
 - Diameter = 2.00 ± 0.04 mm
 - Volume = 2.09 ± 0.4 µl
 - Contact Angle = 90.0 ± 1°

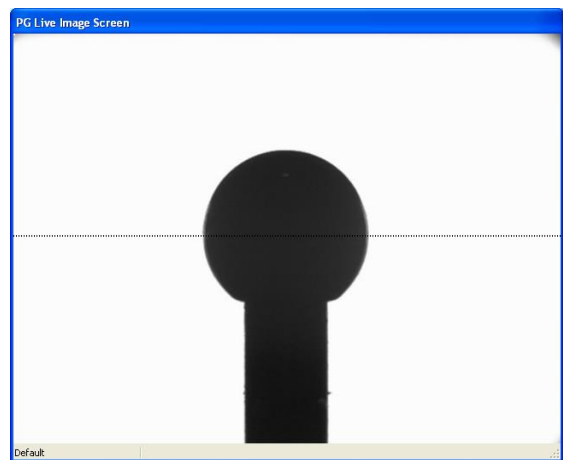


Fig 4.2 Calibration plate with a steel ball



Accepted values will appear in green.

- g) Click on [Save] to store the calibration data.

4.2 Validation Procedures

To verify a correct instrument reading it is much faster to “validate” the instrument using one of the two validation procedures described below. It is recommended to validate the instrument readings at regular intervals.

Standard Calibration plates (Perspex)

Select “Calibrate” from the Image Control Panel and place the PGX+ instrument on top of the full sphere and push the instrument gently against the calibration plate surface. This “artificial droplet” is now measured continuously. If the data are inside the tolerances, all numbers will be displayed in green. If one or more numbers are displayed in red, the instrument must be re-calibrated (Section 4.1).



An optional way to validate the instrument is to place the instrument on top of the calibration plate and perform a measurement in Static Mode. If these values are regularly stored, this information can be used for monitoring of the calibration/validations.

ISO Calibration plates (metal)

Select “Calibrate” from the Image Control Panel and place the PGX+ instrument on top of the semi-sphere and push the instrument gently against the calibration plate surface. This “artificial droplet” is now measured continuously. Enter the height of the reference found on the calibration plate. If the data are inside the tolerances, all numbers will be displayed in green. If one or more numbers are displayed in red, the instrument must be re-calibrated (Section 4.1).

5. Setting up the PGX+ instrument

Insert the Pump Dispensing Unit into the centre of the Applicator Tube.



The dispensing tip should now appear at the top of the live video image

5.1 Preparing a specimen

- Prepare a specimen as described in Section 6.1.
- Place the PGX+ instrument on top of the specimen.
- Check to make sure the test surface appears horizontally in the image.



Soft and warping materials should be placed on a proper backing to hold it flat. A good backing is flat, stiff and coated with a soft-sticky surface, which does not pull lint from the specimen surface when removed.

5.2 Special specimens

In certain applications, the test might require special arrangements like a fixture, into which the test specimen can be inserted. One example is a metal can or a round glass bottle where it will be necessary to position the droplet at the highest point of the curved surface.



Please note the groove at the centre of the instrument sole!

If the instrument is placed perpendicular across a test object with small radius (Figure 5.1) the test surface will appear higher in the image than normal. The instrument can then be placed in the direction of the object axis (Figure 5.2).



Fig. 5.1 Testing curved surfaces perpendicular to the object axis



Fig. 5.2 Testing curved surfaces in the direction of the object's axis

We can also provide special PGX+ accessories for testing on electronic circuit boards and tablets. Contact your instrument supplier for additional information.

5.3 The Pump Dispenser

The test liquid is applied from the built-in micro-pump via the pump applicator unit. It is suggested the pump system is used for a single standard test liquid (e.g. water or fountain solution).



Use different tubing for each test liquid to avoid contamination!

To pump liquids of higher viscosity, or to determine the “wetting hysteresis” (advancing/receding contact angles), it will be necessary to use the optional PG Dosing Unit (www.pocketgoniometer.com)

The integrated micro pump is designed for a standard liquid (e.g. water) where the droplet size can be set in steps of 0,5 μL . Press the PUMP button to pump out a new droplet.

- If PUMP is pressed until the LED is lit, the droplet is slowly lowered towards the test surface, which is recommended for measurements in Static Mode;
- If PUMP is pressed momentarily, the LED will blink while the droplet is pumped out. It is then lifted a short distance from which it is dropped towards the surface. This approach is recommended for Dynamic Mode measurements.
- Press PUMP a second time during the pump sequence to inhibit application of the droplet.

The liquid system is filled in the following way:

- a) Place the instrument on top of a flat surface
- b) Locate the tiny air hole on top of the white container lid. Use the attached one millilitre syringe with the RED tip to fill the container with distilled water.
 *DO NOT use tap water, as this will leave residuals inside the liquid system!*
- c) Insert the RED dispensing tip into the air hole to fill the container. Check top of liquid container to see when liquid container is full (Figure 5.3).
- d) Check the pump lid screw is tightened.
- e) Fill the pump tubing like this:
 - Set the volume selector to position FILL.
 - Press PUMP and the pump will be activated to give 50 pump strokes before it stops. This volume (25 μL) may produce a small droplet at the dispensing tip. If not, press once more on the PUMP button to restart pump. When all air is removed and a droplet appears at the dispensing tip:
press and hold the PUMP button until pump stops.
- f) Set the volume selector to “4,0 μL ”, which is the default droplet size for testing (see Appendix A).
- g) The instrument is now ready for a test (see Section “7. Test modes”).

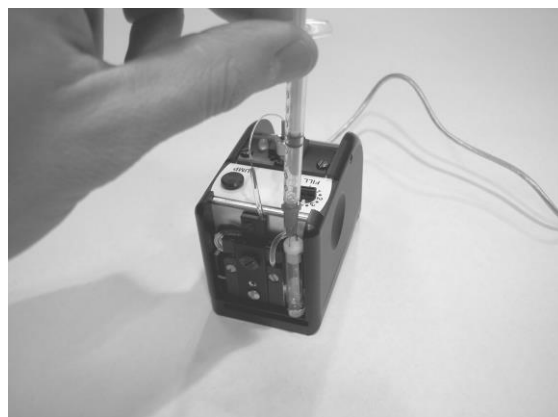


Fig. 5.3 Filling the Liquid Container

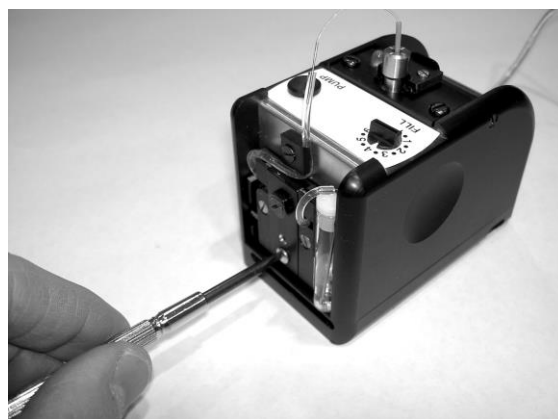


Fig. 5.4 Tighten the pump lid screw

6 Miscellaneous

6.1 Sample preparation

- a) Determine and mark the machine direction of each sample where applicable.
Be careful not to touch the areas to be tested, or contaminate them in any other way.
- b) Determine and mark the “top” and “back” sides of each sample based on the side relevant for the application. The test areas must be free of folds, wrinkles, blemishes, watermarks and other defects not normally inherent in the sample.

 *If the PGX+ instrument is used in the MD direction and then the CD direction, the ratio between $Base_{CD}$ to $Base_{MD}$ at an agreed time (e.g. one second) can be used to determine the anisotropy of a surface. For testing of anisotropic surfaces (e.g. newsprint) the PGX+ instrument should be oriented at a 45° angle to the machine direction when it is not necessary to determine the degree of anisotropic response.*

 *Soft materials (e.g. tissues, textile, film) or warping materials (e.g. newsprint, cigarette tipping paper) should be mounted on top of a flat backing material using a soft-sticky adhesive material.*

6.2 Setting up a test

- a) Check the liquid container in the pump is not empty
- b) Check your PGX+ instrument is connected to the USB port
- c) Start the PGX+ program
A live video image should now appear in the Live Image Screen
- d) Prepare a specimen as described in section 6.1
- e) Select the requested droplet size.
- f) Press PUMP to pump out a droplet, which is then automatically applied as follows:
 - in Static Mode, keep PUMP pressed until the LED is lit and the droplet will be slowly lowered towards the test surface;
 - in Dynamic Mode, press PUMP momentarily and the LED will blink while the droplet is pumped out and then lifted a short distance from which it is dropped.

 *Press PUMP a second time while pumping to inhibit drop application*
- g) Slide the instrument sideways to a new test position.
- h) Pump out a new droplet at the dispensing tip and apply the droplet for a Static or Dynamic test as described in Section “7. Test modes”.

6.3 Instrument storage

The liquid system (dispenser, tubing and liquid container) should never be left with a liquid for a long time as this may clog the system. The procedures below are recommended when your *PGX+* is to be stored for some time or shipped to another location.

When using the *PGX+* with liquids other than water (e.g. solvents, inks) it is necessary to clean the liquid system after the test has been finished. If the instrument will be stored for some time it is also recommended to empty the liquid system in the following way:

- a) Release the pump lid screw to access the pump tubing
- b) Lift out the liquid container, remove the plug and the empty container
- c) Blow liquid container dry with air
- d) Connect syringe with the RED tip to the dispensing tip and pump air through the tubing until dry
- e) Push the plug back into liquid container top and re-install the container
- f) Put the pump tubing in position
- g) Re-install the pump lid and secure the pump lid screw gently

 *DO NOT tighten the pump lid screw completely when stored!*

6.4 Transportation

Before shipping, the instrument should be prepared as described in section 6.3 above.

Please note it is important to empty even a water-based system completely if shipping might include airfreight at high altitude as this might cause freezing damages.

Always ship the instrument in a complete case with all instrument accessories.

Make sure the case is shipped in sufficient padding to avoid damaging the instrument case and its contents.

7. Test modes

The PGX+ software measures contact angles on solid surfaces in Static and Dynamic mode as described below.

7.1 Drop application in Static Mode

- a) Place the PGX+instrument on top of the test surface.
- b) Select the requested droplet size and press the PUMP button until the LED is lit. The droplet is now pumped out and then slowly lowered towards the test surface. To inhibit automatic drop application, press PUMP a second time while pumping is in progress. The droplet can then be applied manually by pressing on top of the applicator.

After droplet has been applied, three different situations may occur, which can be described as

- A “pendant” droplet hanging at the dispensing tip (Fig 7.2). To transfer droplet, apply a slightly bigger droplet or move the dispensing tip closer to the test surface.
- A “released” droplet, (Fig 7.3) which is immediately transferred to the specimen upon contact with the surface.
- A “trapped” droplet, (Fig 7.4) which is in contact with the specimen surface but has not released from the dispensing tip. Move instrument to a new test position and try using a smaller droplet size or increase distance between dispensing tip and specimen surface.



Fig 7.1 PGX instrument on a flat test surface

☞ *The principle for contact angle measurements in static mode at “equilibrium” is only valid for substrates where the test liquid does not penetrate into or reacts chemically with the specimen surface. The substrate should also be smooth and non-porous.*

☞ *It might be necessary to wait until “equilibrium” is obtained before a measurement is done.*



Fig 7.2 Pendant droplet

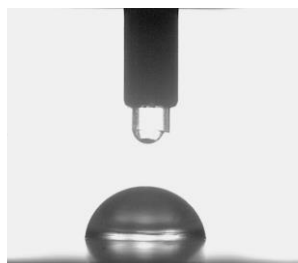


Fig 7.3 Released droplet

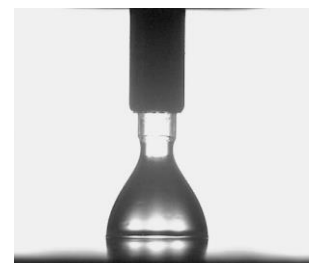


Fig 7.4 Trapped droplet

7.2 Automatic testing in Static Mode (Section 9.3)

This test gives a single measurement of contact angle, volume and droplet base width after the droplet has reached its “equilibrium” condition. Static contact angle is applicable to solid materials into which the test liquid does not penetrate. A test surface is usually characterised from an average of 6-10 test droplets and one or more additional surfaces can be tested in the same test run for comparison in the same diagram. Static contact angle is primarily used to determine the wetting characteristics of a substrate to check surface treatment, cleanliness and/or contamination effects. The purpose of this test is to determine the highest possible contact angle at “equilibrium”.

- a) Select the “Static” function in the program.
- b) Select the requested droplet size.
- c) Press the PUMP button until the LED is lit. A droplet is now pumped out and then slowly lowered towards the test surface.
- d) Wait until the droplet has reached the “equilibrium” condition, which may take several seconds.
- e) Click on “Measure” to get a reading and a new data set will appear in the Results window. Right-click on “Measure” to activate the AutoRepeat function capturing an image sequence.
- f) A single result can be erased if the result line is selected and the Delete button is pressed.
- g) Slide the instrument sideways to a new position.
- h) Select “Save” to store results when the batch is finished.

 *Always slide the PGX+ instrument sideways to prevent the liquid droplet from reaching the lens and the mirror!*

 *To test for ‘equilibrium’ condition, click on “Measure” repeatedly a few times or activate the AutoRepeat function while to capture an image sequence while the droplet rests on the surface. At ‘equilibrium’, the test result should remain stable.*

 *A single droplet applied on the surface can be studied as a function time if multiple images are captured at a fixed time interval. Click on “Measure” with the right hand mouse button to activate the “AutoRepeat” function (Section 9.3).*

7.3 Drop application in Dynamic Mode

In Dynamic Mode the droplet cannot be applied “slowly onto the surface” as in Static Mode. Such a slow application would make the initial interaction during the first second impossible to measure. Instead the droplet is gently transferred to the surface from a very short “free-fall” distance.

- a) Place the PGX+ instrument on top of the test surface.
- b) Select the requested droplet size and press the PUMP button momentarily. The LED will now blink and the droplet is slowly raised to a preset height from which it will fall towards the surface. The falling droplet will trigger the image capturing.

Three different situations may occur, which can be described as

- A “pendant” droplet hanging at the dispensing tip (Fig 7.5).
To transfer droplet, apply a slightly bigger droplet to overcome the adhesion between the droplet and the dispensing tip or reduce the distance between the droplet and the surface. As the droplet was not applied on the surface, the image capture is not triggered.
- A “released” droplet, (Fig 7.6), which will automatically trigger the image capture as the droplet contacts the surface. In this case, a measurement is performed and the test will run until the requested test time has elapsed or the droplet has disappeared from the surface.
- A “trapped” droplet, (Fig 7.7), which is in contact with the specimen surface but has not initially released from the dispensing tip. An image capture is triggered upon contact but all images will be displayed as “zero results”. Move the instrument to a new test position and try a smaller droplet size or increase the distance between the dispensing tip and the specimen surface.



Fig 7.5 Pendant droplet

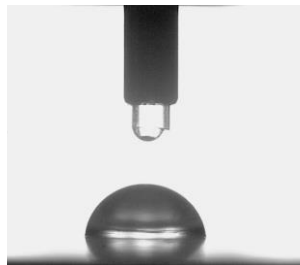


Fig 7.6 Released droplet

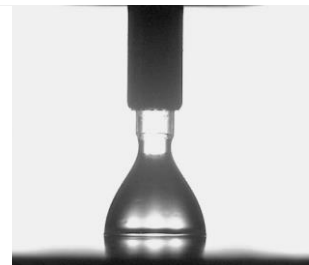


Fig 7.7 Trapped droplet

☞ *The principle for contact angle measurements in dynamic mode is to capture the interaction between a liquid and a substrate as a function of time, while the liquid droplet penetrates into or spreads across the surface.*

7.4 Automatic testing in Dynamic Mode (Section 9.4)

In this test mode a sequence of images is automatically captured as soon as the falling droplet arrives on the specimen surface. Each recorded image is analysed and the result is presented as a curve showing the dynamic change in contact angle (wetting), volume (absorption) and spreading as a function of time. This test is particularly useful for troubleshooting problems related to dynamic processes such as printing, gluing and coating applications.

- a) Select the “Dynamic” function in the program.
- b) Adjust the distance between dispensing tip and test surface to allow for a pendant droplet of the selected size.
- c) Select a suitable Drop Timeout.
- d) Press the PUMP button twice to pump out a droplet and inhibit application of the droplet. Check the droplet is not too close to the surface to risk a contact. Then push gently on top of drop applicator to remove droplet from the dispensing tip.
- e) Slide the PGX+ instrument sideways to a new test position.
- f) Press PUMP momentarily to pump out a new droplet (LED is now blinking).

- g) Immediately click on the Measure button to request a new measurement.
As the droplet falls towards the surface a test sequence is automatically triggered.
-  Please note the droplet must fall within 30 seconds after you have clicked on Measure.
- h) The camera captures up to 80 images during the first second of contact. After the first second, images will be captured at a lower rate until the droplet has vanished from the surface or the selected test time has elapsed. A dynamic curve is then displayed in the Diagram Window together with a list of all data.
- i) A single data point can be erased if a result line is selected and the Delete button is pressed. It is also possible to select a data point by clicking in the diagram.
- j) Repeat steps (e)-(i) above for 6-10 test positions.
- k) A solid line in the Diagram Window represents the average result.
Browse through the different drop results to detect possible “outliers” far away from the average. Click on the associated check box to exclude outliers from the average batch result.
- l) Select “Save” to store results.
As an option, all images in a sequence can be stored.
To store single images, double-click on the result line to bring up a particular image.
This image can then be saved or analysed manually (see Section 9.5).

 Always slide the PGX+ instrument sideways to prevent the liquid droplet from reaching the lens and the mirror!

7.5 Automatic determination of the Advancing / Receding contact angles (Section 9.6)

In some applications it is of interest to characterise the “wetting hysteresis” of a surface. The purpose is to determine the “highest” contact angle as the liquid spreads across the dry surface and the “lowest” contact angle as the liquid retracts from the wet area on the surface. These two contact angles, referred to as the advancing/receding contact angles describe the “wetting hysteresis”. See Section 9.6 for details on how to run this test.

8. Reports (Section 9.8)

To generate a report from a previously saved measurement, select the “**View**” option.

9. The PocketGoniometer program

Apart from the main program screen, there are two additional screens; the Image Control Panel and the Live Image Screen. The Live Image Screen will always reside on top of the other screens, but its size is adjustable to not interfere with other windows. Double-click on the image to switch the live image between greyscale and binary (black/white) mode.

9.1 The Image Control Panel

The control panel functions are divided into four areas to modify the camera settings, define a region of interest in the captured image, to define a target range for determined data and to calibrate the system. When a new set of test parameters has been defined the test conditions can be assigned to a test button which is named by the operator. This way a previously defined test can be restored at a later time.

The active button is indicated by a green light. When the setting of a button is modified, this indicator turns orange. To assign new test parameters to a button, click it with the right-hand mouse button and click SAVE when finished.

 PLEASE NOTE the button NORMAL is default and cannot be assigned new values.

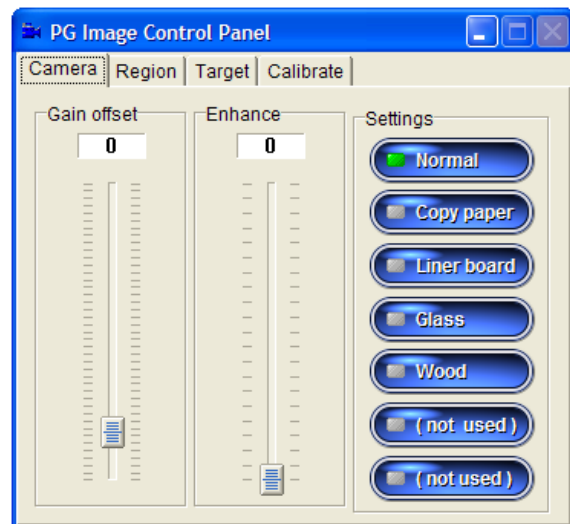


Fig. 9.1 Image Control Panel & Camera controls

Camera – Gain offset

This feature modifies the live camera image in real time. This way the full dynamic range is always available even when the test substrate appears "too dark" or "too bright" due to variations in light reflectance.

Enhance - This feature brings out the droplet image even in situations where the droplet base is partially hidden on a dark, light-absorbing surface (see Section 9.12 for more details).

Region - With these controls a selected region of interest can be defined to exclude shadows and other artefacts in the image that may disturb the image analysis. The horizontal bottom line and the top line are absolute limitations as no analysis is done outside these limits. The vertical limitations are used to create a "path" through which the top of the droplet can be detected. Once the droplet's top is found the contour will be traced down to the bottom line even if the contour is located partially outside the vertical limits for the region.

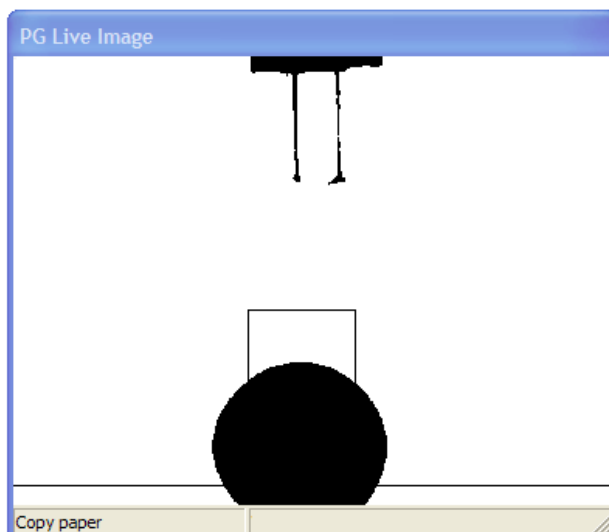


Fig. 9.2 Live Image Screen with Regions

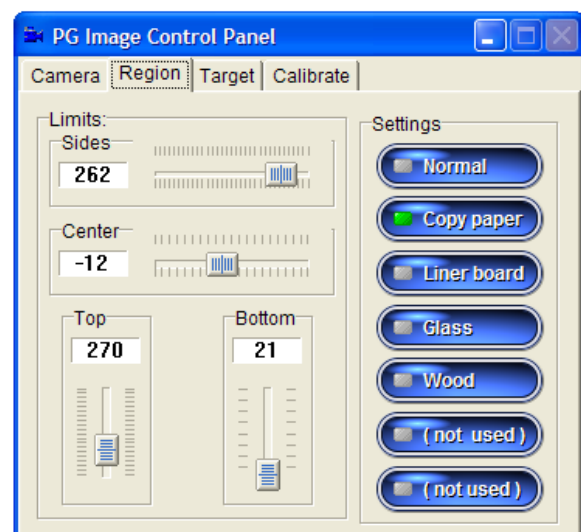


Fig. 9.3 Image Control Panel & Region controls

Target limits

When a test result is expected to fall within a specific range, the corresponding min and max values can be entered into these fields. When a test result falls outside the specified range, this value will appear in red.

Calibrate

The automatic calibration is used to determine the correct light settings and the exact size of an artificial droplet placed in front of the camera. For a detailed description of the calibration procedure, see Section 4.1.

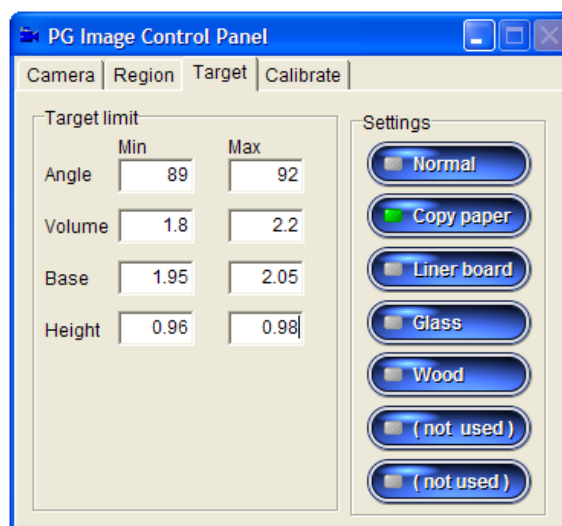


Fig. 9.4 Image Control Panel with Targets

9.2 The Main program

This section describes the functions available in the main program screen.

Static Mode is designed to capture a single reading of the static contact angle at "equilibrium". This value can be measured on smooth, non-porous surfaces not penetrated by the test liquid. Typical examples are water droplets on hydrophobic release papers and polymer barriers (e.g. liquid container board).

Dynamic Mode captures a video sequence during the interaction between the liquid and the surface. Here the dynamic wetting (contact angle), liquid penetration (volume) and spreading are measured as a function of time.

Advancing Mode is designed to characterise the "wetting hysteresis" as a liquid droplet advances across a dry surface and then retracts from a wet surface. This measurement requires a reversible pump flow, which is not possible to do with the micro pump built into the PGX+Measuring Head.

SuperLow Mode is designed for measurement of very low static contact angles below 10 degrees related to super clean surfaces.

View Mode produces a report from previously saved data.

Surface Tension enables testing of the purity of the test liquid. A sufficiently big pendant droplet is pumped out at the dispensing tip and the surface tension is determined from the droplet shape.

Surface Energy must be determined from calculations using contact angles. Different models exist for these calculations using one, two or more liquids in specific combinations.

Options opens a window with the current configuration settings and additional help text. This screen is also used for diagnostics and selection of different languages.

9.3 Static Mode

This mode captures single readings of the static contact angle at "equilibrium" each time the Measure button is pressed. Please note static contact angle measurements are only valid on smooth, non-porous surfaces not penetrated by the test liquid. Typical examples are water droplets on hydrophobic release papers and polymer barriers (e.g. liquid container board).

When a test liquid of higher viscosity is used, the reading must not be taken until the droplet has stopped spreading on the substrate surface. If repetitive readings are taken while the droplet spreads, the equilibrium condition has occurred when the contact angle does not decrease any further.

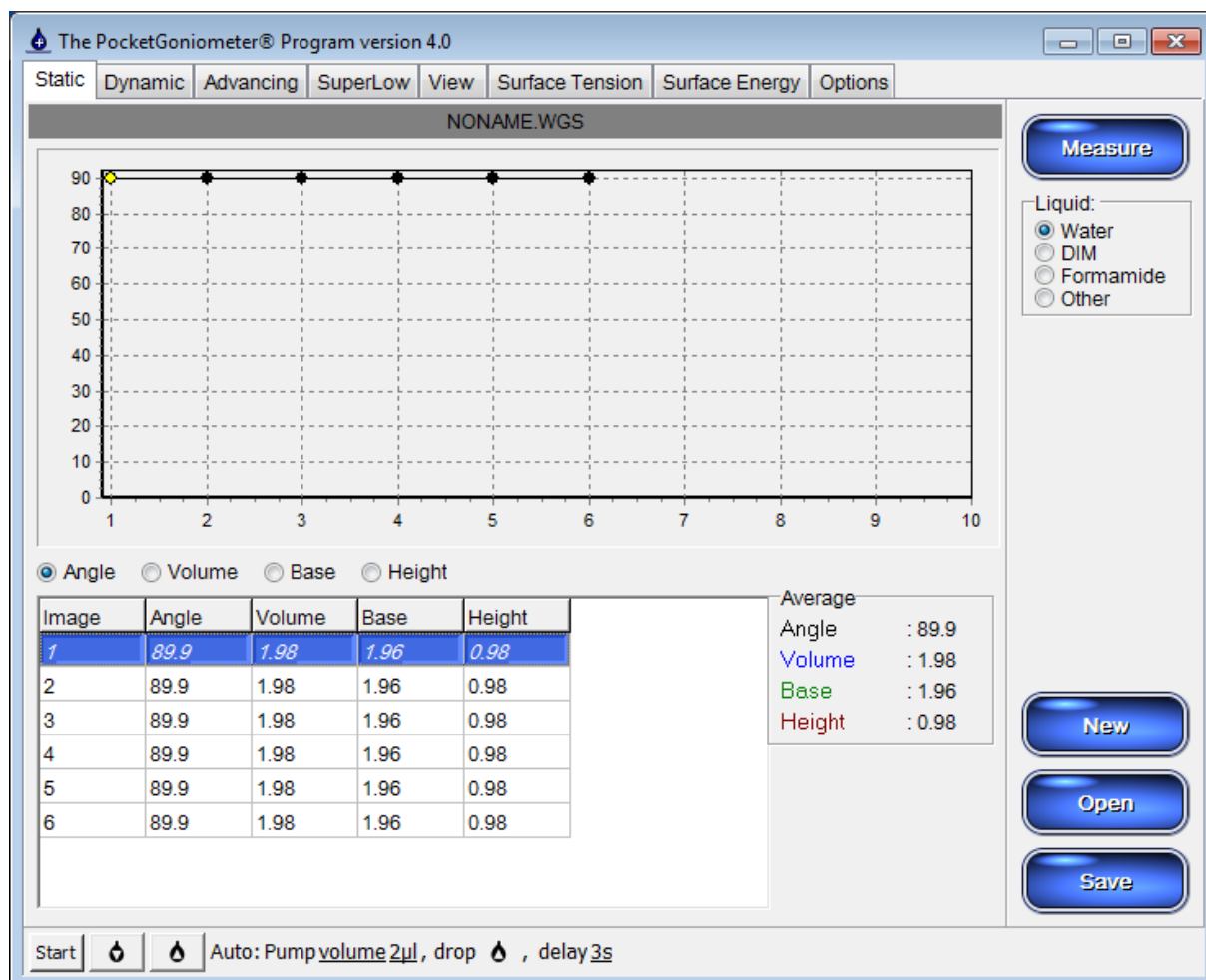


Fig. 9.5 Main Program screen in Static Mode

The Diagram window at the top of the screen is updated after each measurement with a new data point showing the contact angle, drop volume, droplet base or droplet height.

The Results window is updated with a new data set after each measurement.

How to remove a single data point

Click on a line in the Results window to highlight it and press **Delete**.

How to correct a "zero result" or make a manual reading

A "zero result" usually indicates the test is incorrectly set for a very dark or bright test surface. This is easily corrected by changing the camera settings in the Image Control Panel. In addition these new settings can be given its own test button, which makes it easy to set up different tests for very dark or shiny test surfaces. Alternatively an occasional "zero result" can be rescued using the manual approach described in Section 9.5 of this User's Manual.

AutoRepeat function

Normally a single image is captured each time **Measure** is activated. Right-click with the mouse on this button to open the **AutoRepeat** option where a number of images can be captured at user defined intervals. Right-click the button again to restore its original function.

Remote Control function

This option offers selection of droplet size and different drop application modes directly from the PC keyboard without accessing the PGX+ instrument (see section 10 for details).

Control Buttons

Measure	Captures a single image for analysis.
AutoRepeat	Captures a single image for analysis.
Display	Opens a large format instrument panel to display the results.
New	Prompts the user to save existing data and opens a new batch.
Open	A previous batch of data is re-opened and new tests can be appended to it.
Save	Stores the batch results under a filename (*.PGS) selected by the operator. The file extension PGS will be different if one of the pre-defined test liquids has been selected (e.g. Water will give the extension WGS).
Exit	Prompts the operator to store pending data before the program is terminated

For automatic calculation of SFE (Surface Free Energy) the operator can select a liquid from the following options:

- ☒ **Water**
- ☐ **DIM** (Synonyms: Diiodomethane; Methane; Methylene)
- ☐ **Formamide** (Synonyms: Methanamide; Carbamaldehyde)
- ☐ **Other**

Depending on this selection, the result file will get an extension of *wgs*, *dgs* or *fgs* used as a liquid tag. When the option 'Other' is selected, the default file extension *pgs* will be used.

 *For corona treated polymers water can be used as the single liquid to determine SFE in accordance with ASTM D5946-0. The SFE result is then presented directly in the Static window when the ASTM parameter is set to "ON" in the Options Page.*

 *To use the contact angle results for subsequent determination of SFE, all related result files must have the same filename for automatic location of associated files (e.g. 45901.wgs and 45901.dgs). See Section 9.10 for more details.*

9.4 Dynamic Mode

This mode captures a video sequence showing the dynamic interaction between the liquid and the surface. Here the dynamic wetting (contact angle), liquid penetration (volume) and spreading is measured as a function of time. After a droplet has been pumped out at the dispensing tip, a new test is started when the Measure button is pressed. The droplet must then be applied on the specimen surface within 30 seconds (see Section 7.4 for details).

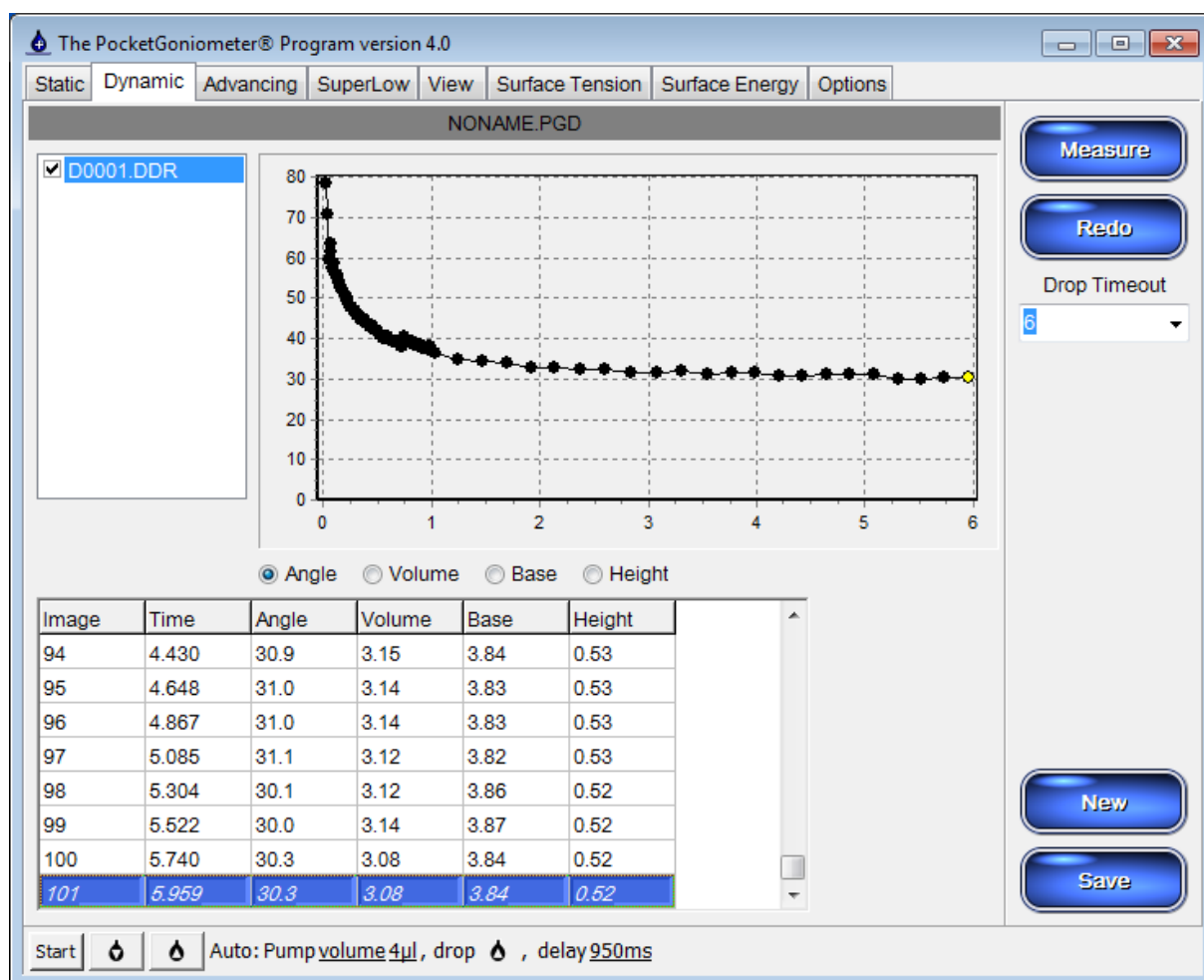


Fig. 9.6 Main Program screen in Dynamic Mode

The Diagram window at the top of the screen is updated after each measurement with a new set of data points showing the dynamic change in contact angle, drop volume, droplet base or droplet height as a function of time. When two or more droplets have been tested, the average response for the droplets in a batch is displayed as a continuous line.

 **PLEASE NOTE:** A drop result is excluded from the batch result if the tick box is left unchecked.

The Results window is updated with a new data set as soon as a new measurement is done. Each line corresponds to a specific image captured at the indicated time. Click on a data set to the left of the diagram to highlight it and the corresponding data will appear in the Results Window.

How to delete a droplet result

Uncheck the tick box in front of the data set to exclude it from the batch data.

You can also click on a data set to highlight it and then use the ReDo function to replace the existing data with a new measurement.

How to remove a single data point

Click on a line in the Results window to highlight it and press **[Delete]**.

How to correct a “zero result” or make a manual reading

A “zero result” usually indicates the test is incorrectly set for a very dark or bright test surface. This is easily corrected by changing the camera settings in the Image Control Panel. In addition these new settings can be given its own test button, which makes it easy to set up different tests for very dark or shiny test surfaces. Alternatively an occasional “zero result” can be rescued using the manual approach described in Section 9.5 of this User’s Manual.

Drop Timeout

The test is automatically terminated when this time has elapsed or if the droplet has vanished from the specimen surface. It is possible to change the Timeout setting during a batch.

Remote Control function

This option offers selection of droplet size and different drop application modes directly from the PC keyboard without accessing the PGX+ instrument (see section 10 for details).

Control Buttons

Measure

Starts the scanner function, which waits for a droplet during 30 seconds.

Redo

Overwrites the current set of droplet data with the next test results.

New

Prompts the user to save existing data and opens a new batch.

Save

Stores the average batch results (*.PGD) and detailed droplet results (*.DDR) under a filename selected by the operator.

Exit

Prompts the operator to store unsaved data before the program is terminated

9.5 Manual testing in Static / Dynamic Mode

In some special situations, it might be necessary to make a manual determination of the droplet. One example is when the captured droplet image is too bright and therefore does not have a solid contour resulting in a “zero result”. Another example is when the droplet base is hidden against a too dark surface and the base line is then incorrectly set. In these situations the captured image sequence can still be used for a semi-automatic or manual approach described later in this section.

Double-click on a data point in the diagram or a line in the Results Window to bring up the corresponding image as shown in Figure 9.9.

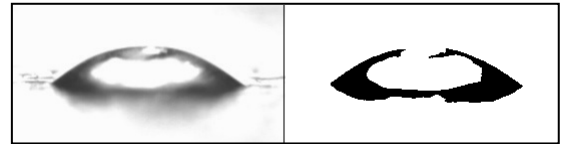


Fig. 9.7a Too bright image missing contour

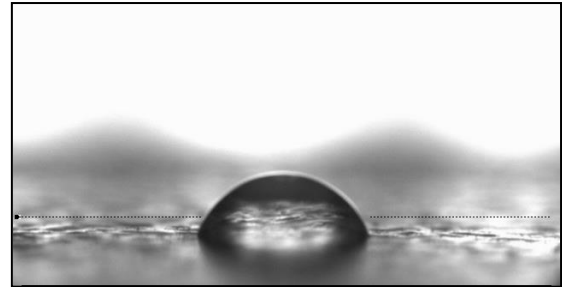


Fig. 9.7b Droplet against too dark background

9.5.1 Adjust the image contrast

Double-click the image to select binary mode and slide the handle **Gain offset** up or down to make the binary image brighter or darker. Try to find a position where the contour is continuous and the base of the droplet separated from the background. This will activate the button **Save**, with its light indicator. Right-click on this button to assign a new mode (Fig 9.8). If the button already has the appropriate selection (e.g. “All images”), simply click the button to change the contrast settings for selected images. These images will then be recalculated using the new gain setting. Use the slider below the image to step between images and check the results.

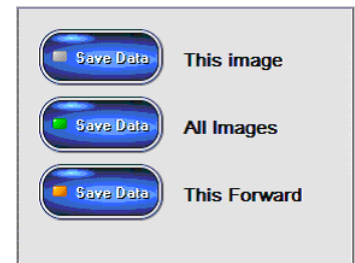


Fig. 9.8 Save status indicator

☞ If a suitable contrast setting cannot be found using the Gain offset control, it will be necessary to use the Enhance function described in Section 9.12.

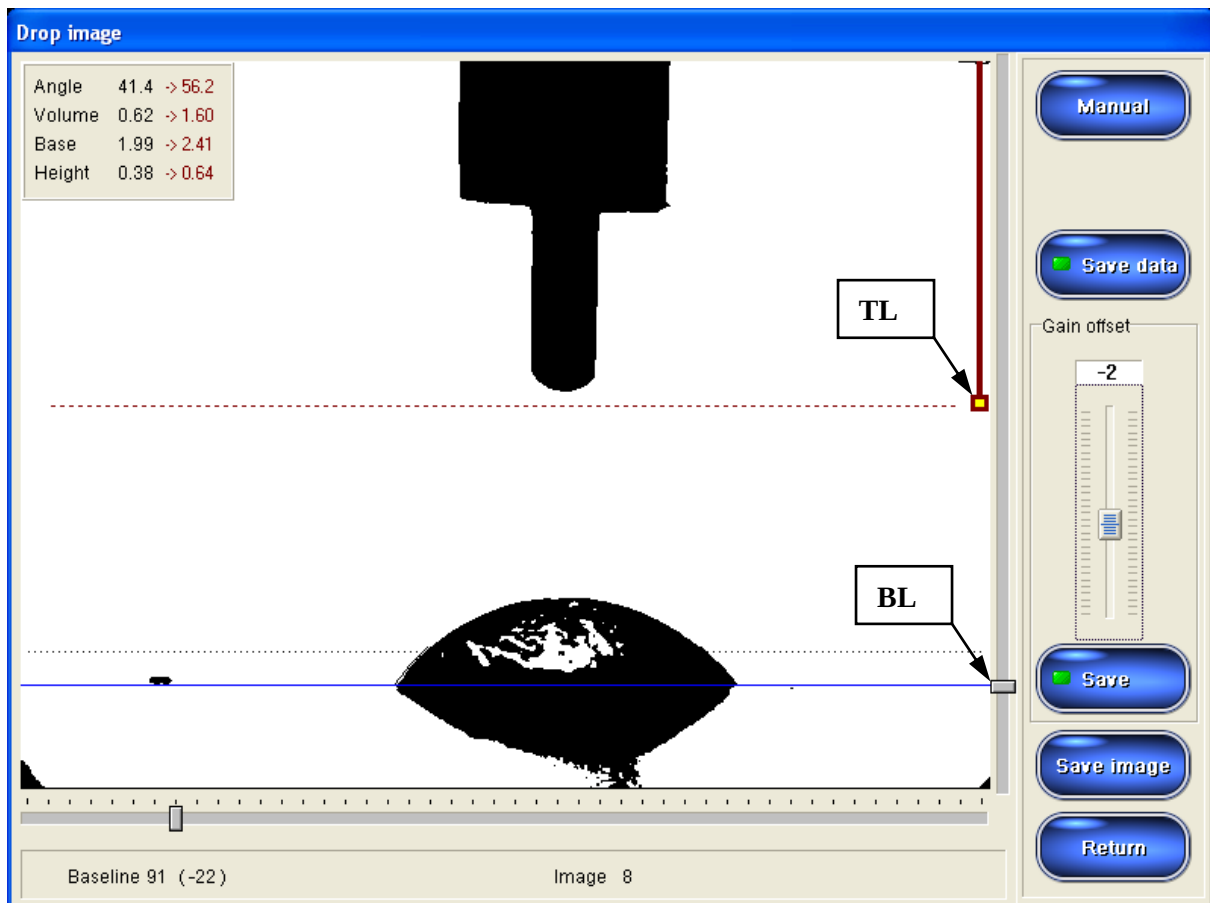


Fig. 9.9 Review of a captured image

9.5.2 Adjust the position of an incorrect Base line

When the automatic base line is set incorrectly, pick up the base line handle (BL) in Figure 9.9 with the mouse and slide it to the correct position. This will activate the button **Save data** with its light indicator. Right-click on the button to assign a new mode. If the button already has the appropriate mode (e.g. “All images”), simply click the button to move the base line for selected images. These images will then be recalculated with the new base line setting.



9.5.3 Exclude fragments above the droplet

When the automatic analysis picks up fragments of the dispensing tip, slide the top line handle (TL) below the fragments that could disturb the measurement. This will activate the button **Save data** and the changed setting will update all images in the measurement. The new top line setting will also update the region setting of the corresponding test button on the Image Control Panel.

9.5.4 Make a manual measurement

Select the greyscale view and click the button Manual to open the zoom window (Figure 9.10). Click with the cursor at the contact point between the droplet base and the test surface. Click again at the contact point on the opposite side and once at the top of the droplet. The manual results will now appear in the top left-hand corner of the image together with the original data. To save the manual results instead of the original results, click on “Save data”, which will also store the manually selected points superimposed in the image. To select three new points along the contour, click on “Reset”. To close the Zoom window, click on “Close”.

Save Image stores a copy of the image with the manual results superimposed.

 Use the slider below the image to shift to another image.

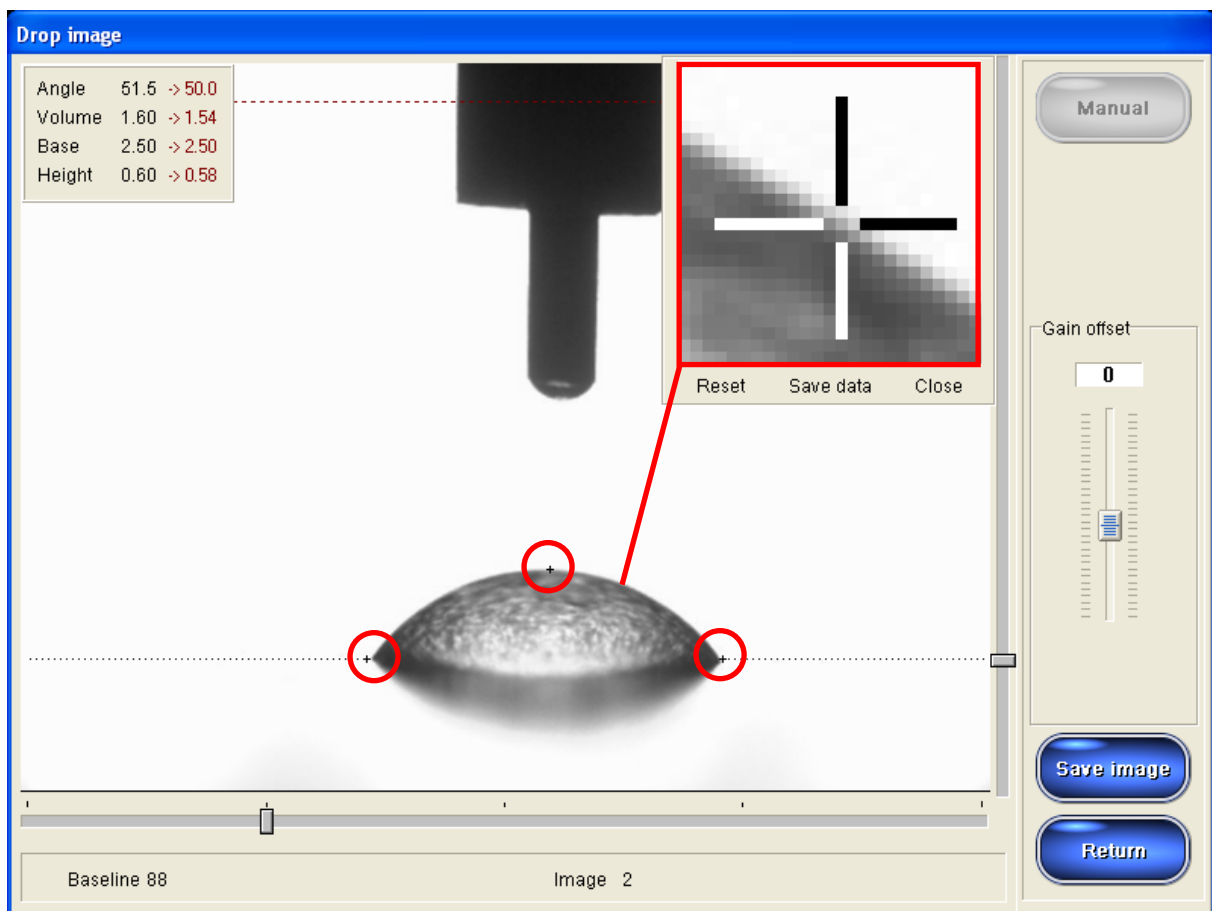


Fig 9.10 Manual measurement made with three points along the contour

9.6 Advancing Mode

This mode captures a video sequence showing the dynamic interaction between a liquid and a surface as the liquid spreads over the dry surface (“wets”) and then retracts from the wet surface (“de-wets”). This interaction, described as “wetting hysteresis”, is characterised by the highest (“advancing”) and lowest (“receding”) contact angles. This test is based on a reversible pump flow. The optional PG Dosing Unit is a programmable dispenser suitable for this task, but any stand-alone pump with reversible flow can be used for the test. With a little practice it is even possible to use a regular handheld syringe to run this test manually.

9.6.1 Automatic measurement using a reversible pump

The dispensing tip must be equipped with a fine capillary tube inserted into the PTFE tubing (P/N 860309). In addition, the distance between the dispensing tip and the test surface must be short enough to make sure the droplet remains in contact with the tip during the test sequence, as the test otherwise will be terminated.

- Click on Measure to start a new test.
- Pump out a droplet, which must contact the surface within 30 seconds to trigger the test.
The pump should now pump out liquid until the droplet has spread sufficiently across the dry surface (the “advancing” phase). Next the pump flow should be reversed so the liquid retracts from the wet surface (the “receding” phase).
- The test is automatically aborted when the contact is lost between the tip and the surface.

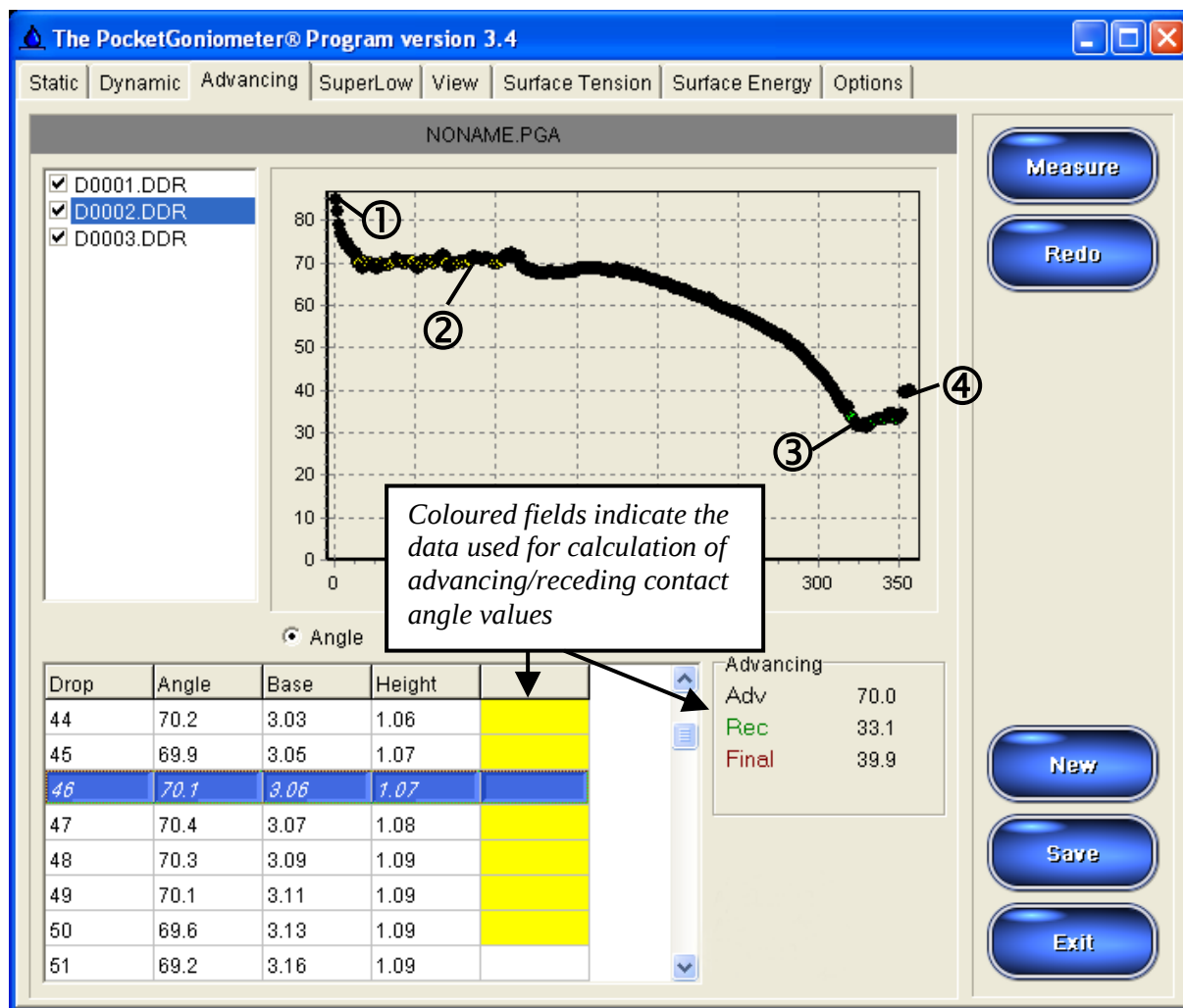


Fig 9.11 Automatic determination of advancing/receding contact angles

9.6.2 Automatic measurement without using a reversible pump

When a reversible pump is not available a liquid droplet can be applied manually from a syringe with the tubing inserted directly into the drop applicator. Alternatively, a pendant droplet of suitable size can be pumped out and the droplet is slowly pushed towards the surface after the test has been initiated. As the dispensing tip is pushed well into the sessile drop, its base will expand across the surface creating an advancing contact angle. As the dispensing tip is slowly retracted from the sessile drop, its volume will shrink and a receding contact angle will appear on the condition the droplet base is reduced.

① The initial contact phase

Immediately upon contact between the droplet and the specimen surface, the dispensing tip inside the applied droplet may “lift” the droplet upwards, which then creates a too high contact angle reading.

② The advancing phase (“wetting”)

When the droplet volume increases and spreads across the dry surface, a stable contact angle reading is obtained. This is referred to as the “advancing” contact angle.

③ The receding phase (“de-wetting”)

Once the “advancing” angle has been determined, the liquid flow is reversed until the droplet begins to retract from the wet area. The lowest contact angle obtained during this phase is referred to as the “receding” contact angle.

④ The release point

As the droplet is no longer in contact with the dispensing tip, the test is aborted and the final contact angle at equilibrium is measured. This angle should be higher than the receding

The Diagram window at the top of the screen is updated after each measurement with a new set of data points.

The Results window at the bottom of this screen displays the results.

How to remove a data point

Click on a line in the Results window to select it and press **Delete**.

Control Buttons

Measure

Starts the scanner function, which waits for a droplet during 30 seconds.

Redo

Overwrites the current set of droplet data with the next test results.

New

Prompts the user to save existing data and opens a new batch.

Save

Stores the average batch results (*.PGA) and detailed droplet results (*.DDR) under a filename selected by the operator.

Exit

Prompts the operator to store pending data before the program is terminated

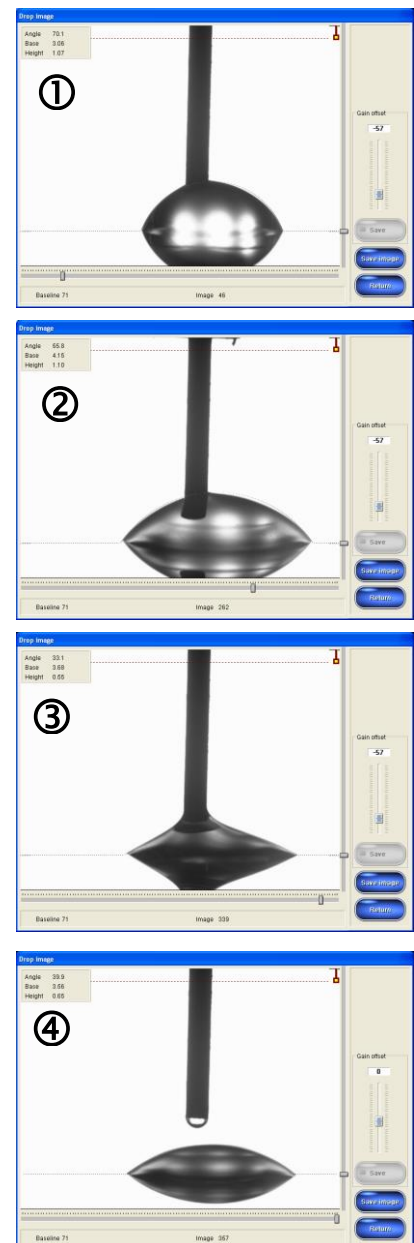


Fig. 9.12 Advancing/receding images

9.7 SuperLow contact angles

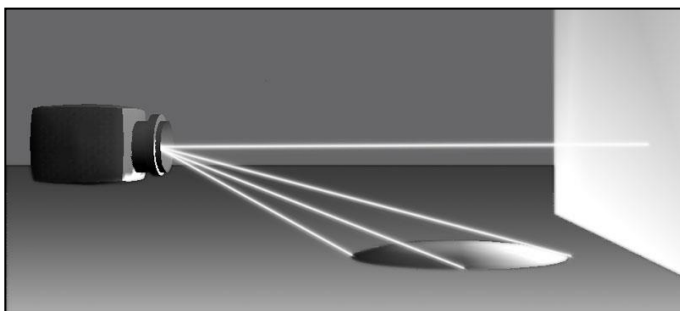


Fig 9.13a. Horizontal view of low contact angle

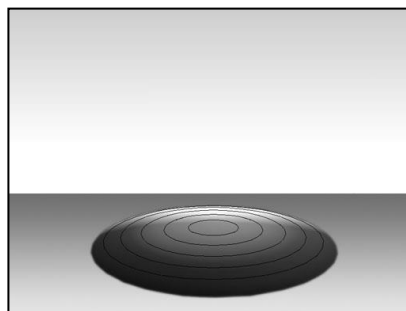


Fig 9.13b. Greyscale view

A camera view horizontal to the test surface will still view the base of the droplet at an angle of 3-6 degrees. Light reflected from the back of the droplet, then makes the top of the droplet "invisible" (Fig 9.13b). If the greyscale image in Figure 9.13b is thresholded, the top of the droplet will vanish (Fig 9.13c). As the camera cannot detect the top, it is impossible to determine its position automatically. This image can still be evaluated using the procedure described below.

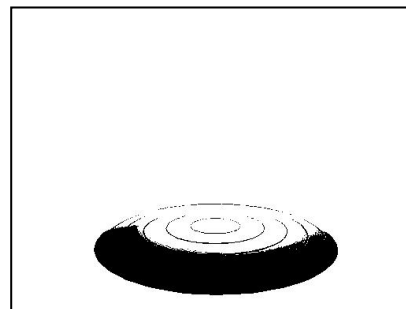


Fig 9.13c. Binary view of low contact angle

9.7.1 Volume determination

The value of a super low contact angle can only be automatically determined if the volume of the applied droplet is known. On these surfaces, even a very small water droplet of a single pump stroke (0,5 μL) may spread outside the camera view. This is why the volume often must be reduced with the volume adjustment knob located on the pump unit (see Appendix D).

 Kindly note the live camera image is enhanced using background correction.

- Fill the liquid system and make sure there are no remaining air bubbles in the liquid.
- Insert a 0,50 mm steel cannulus into the free end of the PTFE dispensing tip (P/N860309).
- Select the very first position on the volume selector (position between FILL and '1').
- Adjust the end of the cannulus in a position where the droplet barely transfers to the test surface when the drop applicator is moved downward (press and hold the PUMP button for a short while).
- Adjust the reference line on the screen to the end of the cannula.
- Press and hold the PUMP button to pump out a droplet and apply it on the test surface.
- Wipe the test area clean removing any debris.
- Click the START button to begin a new measurement once all settings are good.
- Press and hold the PUMP button to apply another droplet on the test area.
- Click the MEASURE button to take a measurement using the actual droplet size which is automatically transferred to the program.

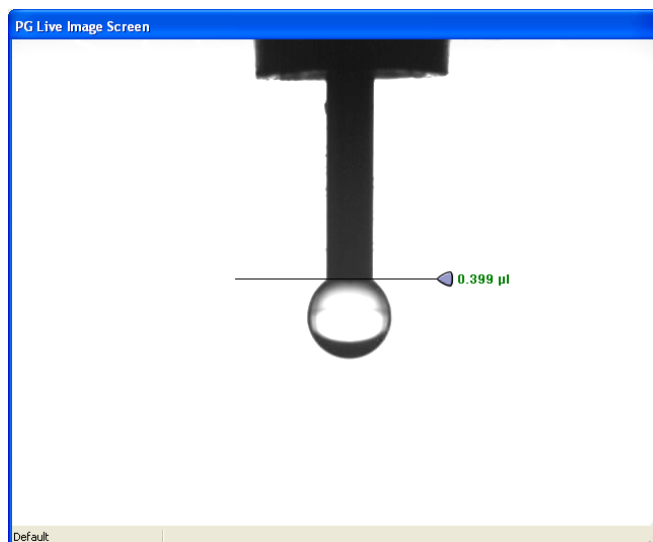


Fig. 9.14a Pendant Drop volume measured in real time

 See Section 9.15 for more details on "Field of view limitations"

9.7.2 Automatic measurement of SuperLow Contact Angles

Once the applied droplet volume has been determined an automatic test of Super Low angles can be carried out. Place the instrument on a clean test surface free from debris and water droplets and press **START** to continue. These program functions are similar to Static Mode operation (see 9.1).

 Kindly note this image is enhanced to make the droplet fully visible.

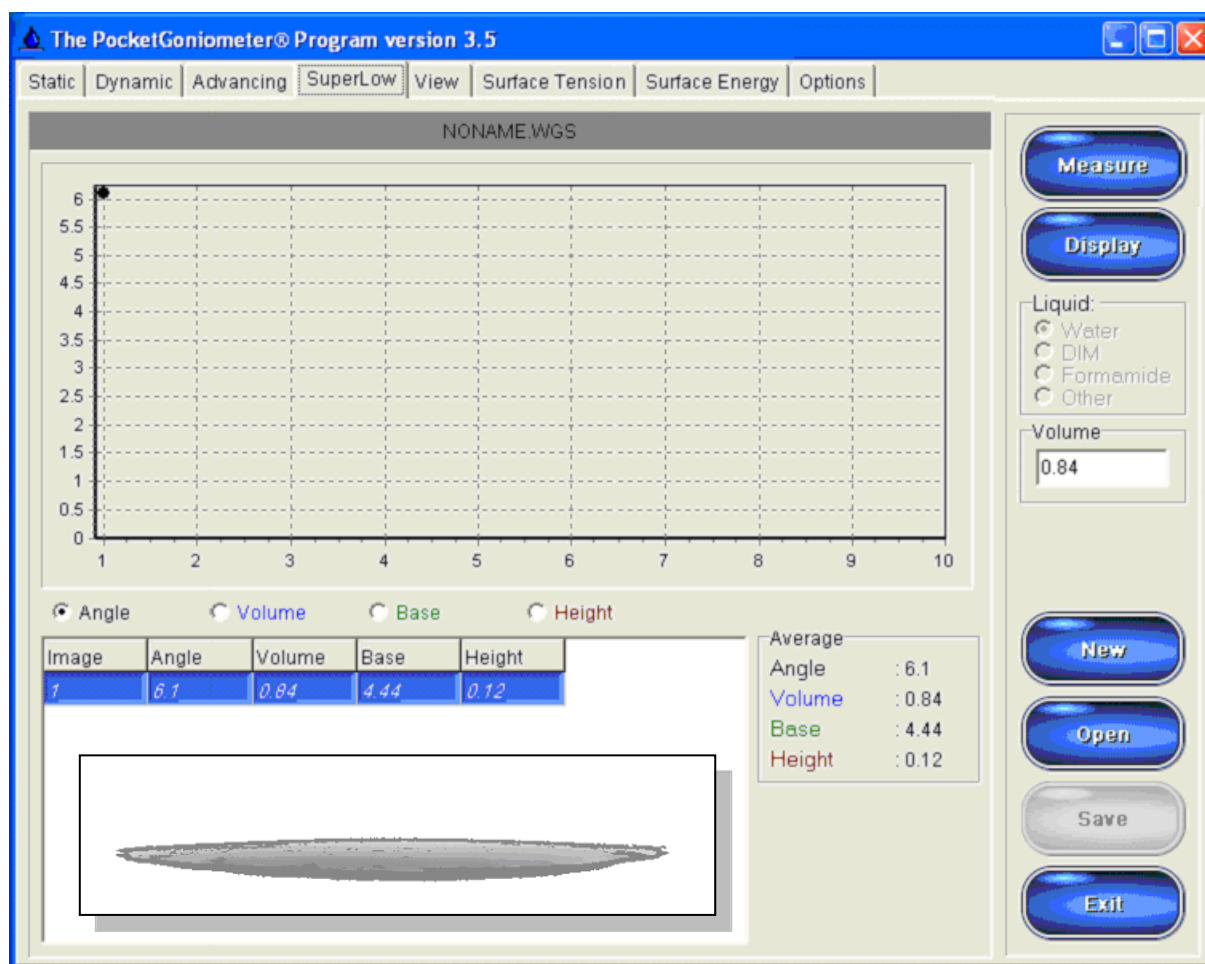


Fig. 9.14b SuperLow contact angle image

9.7.3 Manual measurement of SuperLow Contact Angles

When the contact angle falls below the viewing angle, the camera will look over the droplet and see the rear edge of the droplet. Light is then reflected from the rear droplet surface into the camera and the droplet top becomes invisible. When the automatic analysis fails or appears incorrect, the captured image can still be evaluated using the manual procedure described below.

- Double-click a result line or a data point to bring up the corresponding captured image.
- Click on "Manual" to open the Zoom window, which displays a magnified area under the cursor.
- Click on the two points (A,B) at the base and one point (D) anywhere at the bottom of the drop. These three points (left base, right base and bottom edge) can be selected in any order (Figure 9.15).

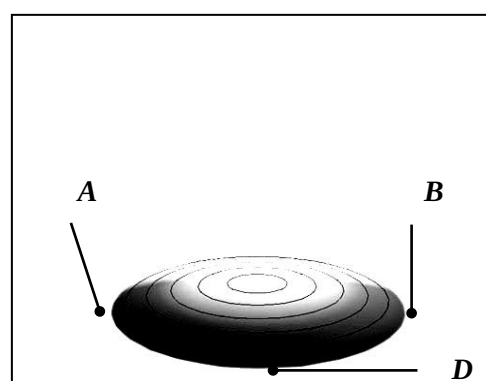


Fig 9.15 Indicate three contour points

The manual results will now appear in the top left-hand corner of the image together with the original data. To save the manual results instead of the original results, click on “Save data”, which will also store the manually selected points superimposed in the image. To select three new points along the contour, click on “Reset”. To close the Zoom window, click on “Close”.

Save Image stores a copy of the image with the manual results superimposed.

Binary / Greyscale alternates between a black/white and a greyscale image.

 Use the slider below the image to shift to another image.

9.8 View Mode

This option retrieves previously saved data showing contact angles, volumes, base diameter or height for the selected file. Double-click on a data point or on a result line to display the corresponding, captured image where the file types are:

Static results of Water, DIM or Formamide **WGS/ DGS/ FGS**

Static results of undefined liquid **PGS**

Dynamic average results for a batch of droplets ... **PGD**

Dynamic data from advancing/receding droplets ... **PGA**

Dynamic Drop Results (time sequence) **DDR**

Click on **Open** to select a file and **Report** to print the report from the requested file.

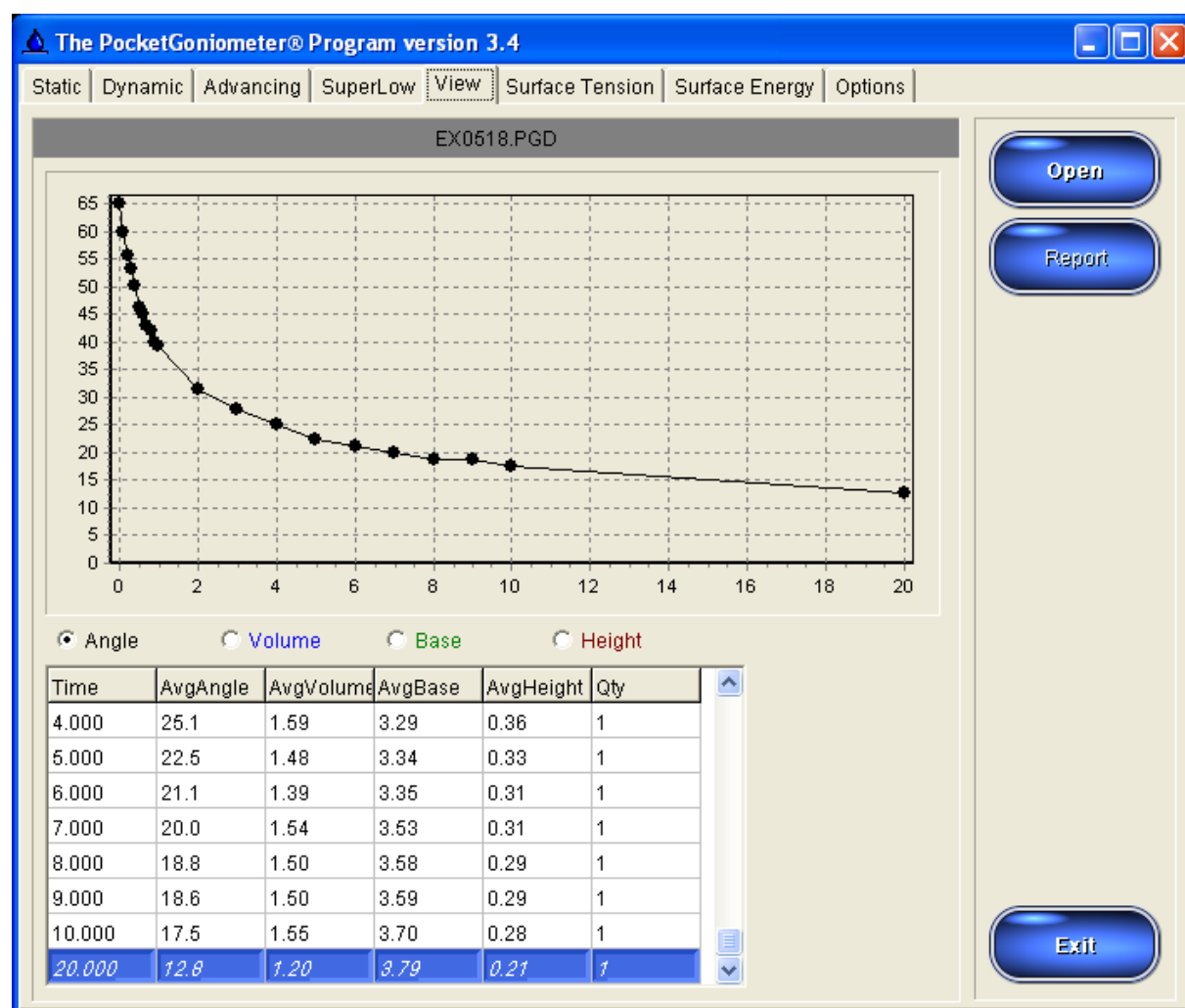


Fig 9.16 Retrieved data in View Mode

9.9 Surface Tension

The instrument must be placed on a stable, horizontal table to give correct results. A sufficiently big pendant droplet must be pumped out at the dispensing tip to determine the surface tension from the droplet shape (Bashfort-Adams). The critical dimensions are calculated from a polynomial curve fitting at sub-pixel resolution with an accuracy of 10^{-6} mm, which offers an accuracy of $\pm 0,2$ dyne/cm.

A “too small” pendant droplet at the dispensing tip will give a too high reading. When the droplet size is gradually increased, gravity will influence the droplet shape and the surface tension will gradually decrease. At a certain droplet size the droplet becomes unstable, wobbling from side to side, which may give an erroneous result. The correct droplet size is the volume used immediately before the droplet begins to wobble.

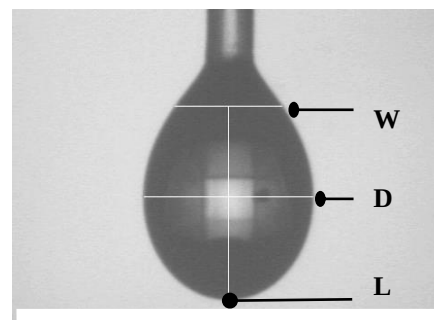


Fig. 9.17 Shape of a pendant drop

The algorithm used determines the maximum diameter “D” and the lowest point “L” of the droplet contour (Fig 9.17). The droplet width “W” is then established at a distance “D” above the point “L”. The parameters “D” and “W” are finally used to calculate the liquid’s surface tension from the droplet shape.

Please note the surface tension value is a function of the density of the liquid. This correction is done automatically if the Density field is updated before the measurement is done.

The AutoRepeat function (see 9.3) is available also for Surface Tension measurements.

The Remote Control function offers selection of droplet size and different drop application modes directly from the PC keyboard (see section 10 for details).

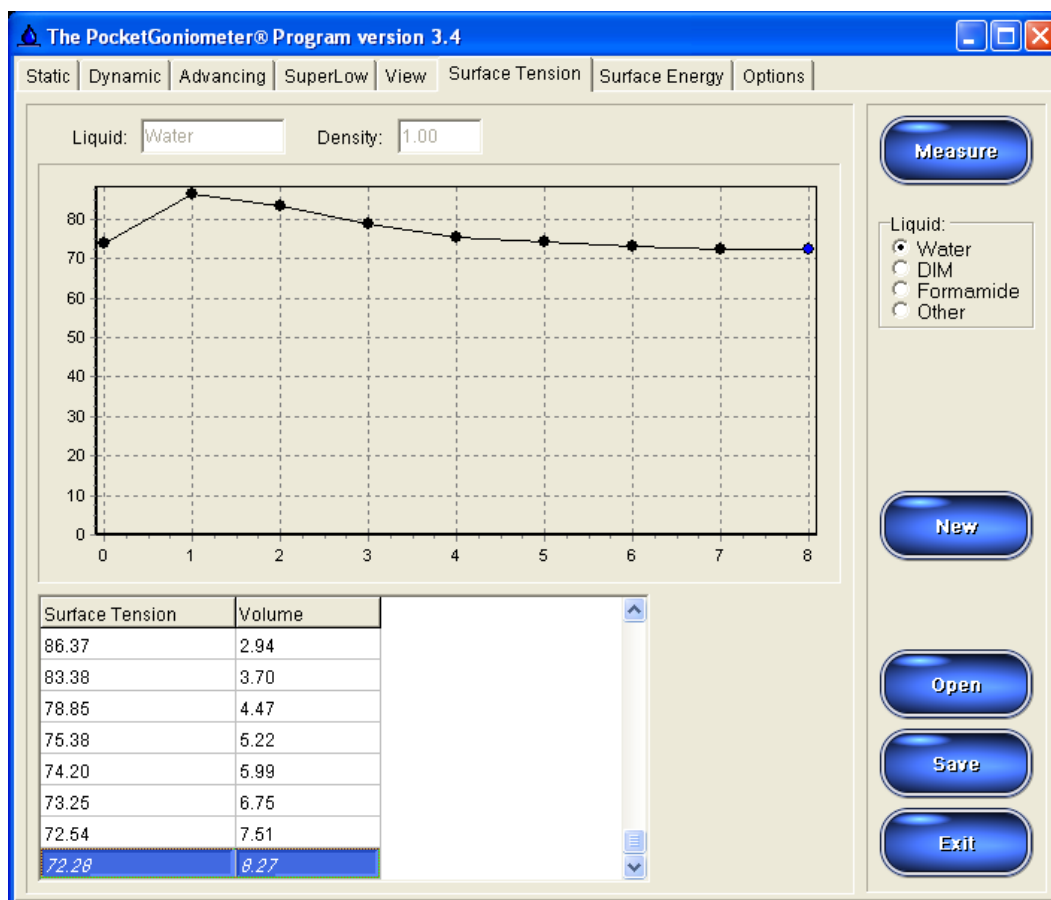


Fig 9.18 Surface tension as a function of droplet size

9.10 Surface "free" Energy (SFE)

As this surface property cannot be measured directly; it is calculated from contact angle measurements using different, properly characterised liquids. Depending on the substrate, one or more liquids are used. Please note SFE calculations are subject to the following constraints:

- No penetration of test liquid into surface (check that volume remains constant over time)
- No chemical reaction between test liquid and surface (check contact angle is constant over time)
- Surface must be sufficiently smooth

Here the operator starts by selecting a Calculation Model from the following options:

- ☒ **ASTM D5946**
- ☐ **Polar/Dispersive**
- ☐ **Acid/Base**

The first option, ASTM D5946 is the "Standard Test Method for Corona-Treated Polymer Films using Water Contact Angle Measurements" (copyright ASTM International) where the water contact angle is converted into a dyne value. For this option only the topmost liquid field (Water) is activated and used for the SFE calculations.

The screenshot shows the 'The PocketGoniometer® Program version 3.4' interface. The 'Surface Energy' tab is selected. The 'Sample ID' is 45901. Under 'Calculation Model', 'ASTM D5946' is selected. The 'Select File' section shows three fields: 'Water' with a value of 85.8 and file 45901.WGS, 'DIM' with a value of 25.8 and file 45901.DGS, and 'Formamid' with a value of 45.8 and file 45901.FGS. The 'Calc' button is visible. The results section displays: 'Sample ID: 45901', 'Contact angle with: - Water: 85.8 45901.WGS', 'Calculated results for surface energy (mJ/m²) - Total surface energy: 34.0', and 'Method used: ASTM D5946'. The 'Save', 'Print', and 'Exit' buttons are at the bottom right.

Field	Value	File
Water	85.8	45901.WGS
DIM	25.8	45901.DGS
Formamid	45.8	45901.FGS

Sample ID: 45901

Calculation Model: ☒ ASTM D5946

Contact angle with:
- Water: 85.8 45901.WGS

Calculated results for surface energy (mJ/m²)
- Total surface energy: 34.0

Method used: ASTM D5946

Fig. 9.19 SFE calculation for a corona treated polymer using a single liquid (water)

The second option 'Polar/Dispersive' uses contact angle readings from two selected test liquids to determine the SFE of a substrate. Here the two first liquid fields (Water and DIM) are activated. For these calculations, the Harmonic approach (Wu) is used as the first option as this is considered 'more accurate'. This approach can, however, end up in negative roots, so there is not always a solution to these equations. In this situation the program changes to the Geometric approach (Owens-Wendt), which always gives a solution. The SFE report indicates which method was eventually used (Harmonic or Geometric) for the calculation. It is possible to force the Geometric method if the parameter SFEMETHOD is changed from H to G (see Section 9.18 of this User's Manual).

The third option 'Acid/Base' (Lewis) uses three liquids to calculate the surface energy from an equation system. Here all three liquid fields are activated (Water, DIM and Formamide). Please note the calculated results depend on the liquid combination used.

The screenshot shows the 'The PocketGoniometer® Program version 3.4' interface. The 'Surface Energy' tab is selected. The 'Sample ID' is 45901. The 'Calculation Model' is set to 'Acid/Base'. The 'Select File' section shows three rows: 'Water' with a value of 85.8 and a file icon for '45901.WGS', 'DIM' with a value of 25.8 and a file icon for '45901.DGS', and 'Formamid' with a value of 45.8 and a file icon for '45901.FGS'. The 'Calc' button is visible. The results section displays the contact angles and calculated surface energy components.

Sample ID: 45901

Calculation Model

- ☐ ASTM D5946
- ☐ Polar/Dispersive
- ☒ Acid/Base

Select File

Water: 85.8 [Browse] 45901.WGS

DIM: 25.8 [Browse] 45901.DGS

Formamid: 45.8 [Browse] 45901.FGS

Sample ID: 45901

Contact angle with:

- Water: 85.8 45901.WGS
- DIM: 25.8 45901.DGS
- Formamid: 45.8 45901.FGS

Calculated results for surface energy (mJ/m²)

- Total surface energy: 46.8
- Dispersive component: 45.9
- Acid/Base component: 1.0
- Basic component: 0.3
- Acidic component: 0.9

Buttons: Calc, Save, Print, Exit

Fig. 9.20 SFE calculation for a solid surface using three liquids

To select an input data file, use the associated Browse button. For Water, the browse button will present files with a wgs extension only as these should contain results from water contact angles. In a similar way the other two browse buttons (if active) can be used to select other result files measured with DIM or Formamide. As soon as the result file for Water has been selected, the first part of the file name is used for the field 'Sample ID' (e.g. 45901).

The program will automatically search for associated result fields. The condition is, however, that *all files must use the same ID as a file name* (e.g. 45901.wgs and 45901.dgs). The operator can still overwrite information displayed in the contact angle fields, in which case the Sample ID window will be erased. As soon as the input fields (one, two or three) have been completed, the program will automatically calculate the SFE results. When manual data is typed into the input fields, it is necessary to click on 'Calculate' to determine the SFE values. The SFE results are displayed in the Results Window at the bottom of the screen.

The **Save** button will store a copy of the report in the default directory (e.g. \fibro\pg) with the extension *sfe* (e.g. 45901.sfe). When the file has been stored the 'Save' button becomes inactive until a new result is calculated.

☞ For corona treated polymers water can be used as the single liquid to determine SFE in accordance with ASTM D5946-0. The SFE result is then presented directly in the Static window when the ASTM parameter in the Options Menu is set to "ON" (see Section 9.11).

☞ For determination of SFE using other liquids please see Section 9.14 of this User's Manual.

☞ **Please note SFE calculations can only be done from static contact angle test results!**

9.11 Options Screen

This screen displays a window with the current configuration settings, which can be used for support and diagnostics. Some of the program functions can also be modified from this screen.

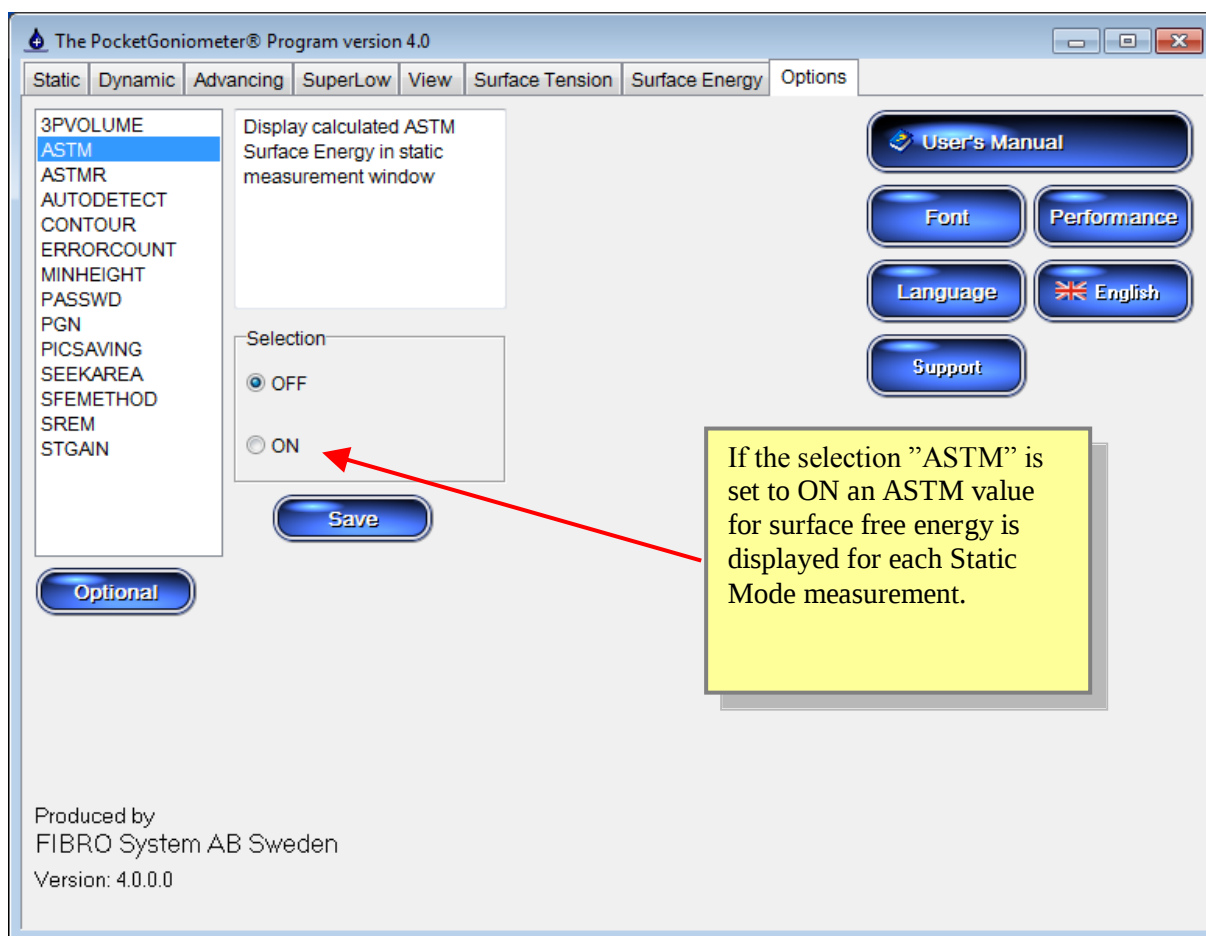


Fig. 9.21 Options Screen

Online Help is the full User's Manual supplied with the program

Font offers other fonts necessary for foreign languages

Maintenance runs diagnostic tests on the PG program

Language offers additional language versions

Support creates a diagnostic file used for support issues

Performance check

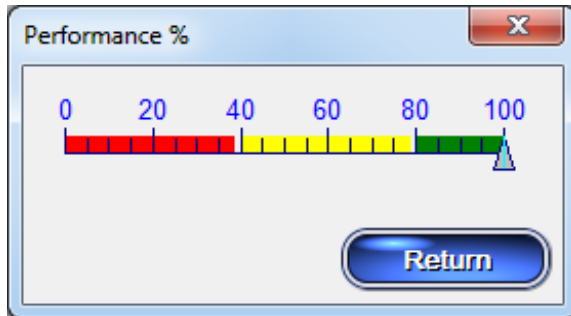


Fig. 9.22 Performance indicator

This function tests the capture rate from the USB camera. For 100% performance the PC must handle 80 images per second. If the performance is lower, too many background applications in the PC might slow down your computer. Shut down not used applications and run this check again.

9.12 Enhance Mode

When a water droplet is applied on a surface of low reflectance (e.g. dark, rough craft liner), the droplet base will appear against a dark background. If the light level is increased, or the parameter CameraGain /Threshold is adjusted to distinguish the droplet base from its background, the droplet top can become too bright against the illuminated background. This is where the image enhancing routine will be very helpful. The enhancing routine is available in Static Mode, Dynamic Mode and Advancing/Receding Mode. Please note the image enhancing function remains in operation also when a new test mode is selected (e.g. changing from Static to Dynamic Mode).

- a) Select "Normal" on the Image Control Panel (Fig 9.1) to start with the default settings.
- b) Place the instrument on the test surface and apply a liquid droplet on the surface.
- c) Double-click in the video image to switch from greyscale to a binary image if necessary. Adjust the Camera Gain control until the top of the droplet is completed. Then use the Enhance control to create a solid black contour all the way down to the base of the droplet.
- d) When the final settings have been found these test parameters can be assigned to a test button as described in Section 9.1 of this User's Manual.

 Please note the default test button "Normal" cannot be assigned new parameters!

9.13 Base Line determination

A water droplet applied on a reflecting background will appear as a dark 'saucer' when the contact angle is below 90 degrees. A high contact angle above 90 degrees will appear as a 'black snowman' with a waist against the white background. Similarly a contact angle of 90 degrees will result in a black circular shape. All these droplet silhouettes will be analysed automatically and the program will assign a base line at the interface between the droplet and the test surface. The determined base line is displayed as a dotted line in the captured image. The base line can be adjusted manually as described in Section 9.5.2.

 By definition, the base line is the lowest section of the droplet resting on top of the test surface.

9.14 Camera Gain and Threshold

The default value for Camera Gain and Threshold is established during calibration. This light level determines if a pixel is black or white in the binary image. These values can be modified by the operator to manage unusually bright or dark test surfaces as part of a test profile stored under a button on the Image Control Panel (Section 9.1).

9.15 Field of View limitations

The measurement of very low contact angles is limited by the horizontal field of view.

A standard PGX+ instrument has a horizontal view of approximately 6.5 mm. Droplets of 0.5 μL will reach a width of 3.7 mm at a contact angle of six degrees. At an angle of 2.5 degrees this droplet will have a width of five mm. In comparison a 0.25 μL droplet will reach a width of 4.6 mm at a contact angle of 1.5 degrees. As we cannot expect the droplet to be perfectly centred in the image, a useful width is some 4.5 mm at the droplet base. This means 0.5 μL droplets can be measured down to 3 degrees. Similarly, a 0.25 μL droplet can be measured down to a contact angle of 1.5 degrees. For even lower contact angles, the droplet volume has to be decreased further (see "Small droplets" in Appendix D).

9.16 Use of other test liquids for determination of 'Surface "Free" Energy'

The PG program has been configured for use with water, Diiodomethane and Formamide in order to determine the SFE values. This set of liquids is commonly used and in general there should be no reason to select another set. By using this recommended set of liquids your SFE values can be compared with other test laboratories using the same set. If you need to use another set of test liquids or translate the liquid names to another descriptor name, you can do so in the following way:

- Make a backup copy of the file \fibro\bin\SFE.INI
- Open the file SFE.INI with a regular text editor (e.g. NOTEPAD.EXE)
- You may translate the descriptor for water (e.g. 'Wasser') but DO NOT change the parameters for water, as this will ruin the ASTM D5946 standard for corona treated polymers.
- You may change the parameters for the second and third liquids as shown in the example below:

<u>Current</u>	<u>New</u>	<u>Explanation</u>
Formamide	Ethylene glycol	Descriptor name
58	48	Total surface tension γ^{TOT}
39	29	Dispersive component γ^{LW}
2.28	1.92	Basic component γ^-
39.6	47	Acidic component γ^+

Static contact angle results will still be stored as wgs, dgs and fgs files even if the liquid parameters have been changed. To keep track of files before and after a liquid change it is recommended to rename the old results directory or to move old contact angle results to a different directory each time a liquid parameter is changed.

 **PLEASE NOTE:** SFE calculations done with one set of liquids CANNOT be compared to SFE results calculated from another set of liquids;

9.17 Translation of the User Interface

The text of control buttons and the screens is controlled from a language file named **PG_NNN.LGG**, where **NNN** indicates the international short form country code (e.g. PG_CHT.LGG for traditional Chinese). The language file can be edited with a regular text editor (e.g. NOTEPAD.EXE). Try to use 'short words' as long words might exceed the space available inside a button.

This file can also be used for other modifications such as changing the default descriptor **mJ/m²** to **dyne/cm** or **mN/m**. Look up the corresponding default text in the PG_NNN.LGG file and edit the text. Please note this modification is only possible when there is a one-to-one relationship between units.

9.18 System parameters (Options Menu)

Many functions inside the PG program can be customised from the Options Menu.

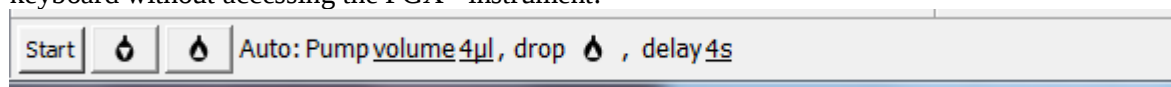
Select one of the listed parameters to see what the options are.

Please note that some of the parameters below might not exist in your list and must be added.

DYNDEF=3	Default timeout in Dynamic Mode to three seconds
REPDEF_PIC=20	Default number of images to 20 in AutoRepeat mode
REPDEF_MIN=5	Default test time to 5 minutes in AutoRepeat
CUR_ON=1	Switches Curtain= ON as default (normal default = 0)
AUTODETECT=25	Defines the distance to the trigger line in Dynamic Mode
MINHEIGHT=10	Defines the minimum height of a droplet (reduce for smooth surfaces)
DATADIR=C:\FIBRO\PG	Default directory for saved data
PICSAVING=1	Saves captured images as default (0= no saved images)
CONTOUR=0	Does not plot a contour trace (255 = plots contour as default)
3PVOLUME=0.25	Default droplet size for SuperLow contact angles
LOLIMIT= -1	Lower limit for Display Panel in Static and SuperLow Mode
HILIMIT= -1	Upper limit for Display Panel in Static and SuperLow Mode
ASTM=1	Displays the SFE value directly in the Static window
SFEMETHOD= H	Harmonic if possible (G= Geometric approach always)
PGN= -1	Will prompt for instrument model (applicable to ST672 camera only) <u>PGN=0</u> instrument is PG-2/PG-3, <u>PGN=1</u> instrument is PG-X
BASLINE	Dynamic baseline method. Adaptive (default): Baseline detected for each image. Fixed: Baseline set to a fixed value (1-200). Last valid: Uses baseline from last valid image.

10. Remote Control function

This option offers selection of droplet size and different drop application modes directly from the PC keyboard without accessing the PGX+ instrument.



Start (Stop)

Activates the pump to pump out a pendant drop until Stop is clicked.



Drop application

The pendant droplet is lowered towards the surface or lifted and dropped with a small impact.

Auto (On/Off)

Enables the remote control functions to run an automatic test profile set to On.

Droplet Size (Volume/ Time)

Select Volume or Time to pump out a desired droplet volume of liquid.

Drop Application Mode (Up / Down)

applies the pendant droplet on the test surface by lowering the droplet onto the surface (“Touch Down”) or lifts the droplet to be released with a small controlled force (“Impact”).

Delay (seconds)

The Delay parameter has different functions depending on type of measurement.

Static Mode the measurement is suspended until the set time has expired.

Dynamic Mode allows synchronisation of image capture

Advancing Mode the automatic function is disabled.

Super Low Mode the measurement is suspended until the set time has expired.

Surface Tension Mode ... measurement is suspended to avoid oscillations inside the droplet

11. Consumables

Part No.	Description	Kit Contains
860301	Liquid Container Kit (with & w/o plug)	5+5 pcs
860309	Steel cannulus tip 0.5 mm	10 pcs
860314	Applicator Tubing between Syringe and Tubing Dispenser	5 pcs
860341	Pump Tubing (PTFE) suitable for most liquids	5 pcs
860342	Pump Tubing (Silicone) suitable for Diiodomethane	2 pcs

12 Trouble shooting**Camera “not found” or missing live image or “too dark” image**

It is necessary to use the camera drivers supplied with your instrument!

If you have downloaded “later drivers” from the Internet, the results will become unpredictable and Windows might even refuse re-loading of your original drivers once “later versions” have been installed. If this situation occurs, please contact your instrument supplier for assistance.

If you have installed the PG program with the original camera drivers, please check the following:

- Check the PGX+ instrument is connected to the selected USB port.
If not connected, connect instrument cable and press OK
- If your instrument is connected, unplug the instrument from the USB port, wait a few seconds and then re-connect the instrument again. The correct driver should now be selected automatically.

The micro-pump does not run although LED indicator on top of pump is lit

Turn the volume adjustment knob (A) counter clockwise until pump starts to run.

If pump still does not run

Then tighten the hex screw (B in Figure D-2).

Finally tighten the pump lid screw (C) firmly again.

Check pump volume is correct (Appendix D2).

The micro-pump runs but does not produce a droplet

- a) Make sure liquid system is filled as described in section 5.3.
- b) If pump runs but liquid does not appear at the dispensing tip, make sure the pump lid screw (C in Figure D-2) is firmly tightened.
- c) If still no liquid is pumped, release stop screw (B in Figure D-2) ½ a turn and then tighten the pump lid screw (C) firmly again.
- d) Repeat (c) above until liquid is pumped inside tubing (use pump position FILL).

The pendant droplet continues to build up after pump has stopped

The pump tubing must be completely shut after the pumping has stopped. If not, liquid will continue to flow through the pump tubing. See Appendix D of this manual to adjust the pump function.

The droplet climbs up on the outside of the dispensing tip

The outside of the dispensing tip is wet or is not clean.

Dip a cotton swab in an evaporating solvent (e.g. alcohol) and wipe the tip clean on the outside.

Some liquids of very low surface tension (e.g. organic solvents) might still tend to wet the outside of the dispensing tip due to the tip material. To reduce this it might be necessary to use PTFE tubing with a large cross section and a small inner diameter (P/N 860315, Ø1.5x0.2 mm) without inserting a metal needle at the tip.

Droplet forms nicely at the dispensing tip but does not release from the tip in Dynamic test mode

Increase the droplet size to overcome the adhesion between the liquid droplet and the dispensing tip.

The Dynamic test gives no test result at all

The dynamic test is triggered by the drop application. Possible errors are:

- a) The image capturing might start “too early”.
If the pendant drop is too close to the surface it might trigger the image capturing before the droplet is dropped. Increase the distance to the surface slightly.
- b) The Dynamic test might have “timed out”
After the test button has been pressed the scanning process will time out after 30 seconds.
Pump out a droplet before the program button is activated and apply the droplet during the 30-second period.

Test results appear to be incorrect or “zero”

When a captured image cannot be evaluated automatically, the result is a line of zeroes.

The automatic analysis will fail if the captured image is of poor quality because of incorrect settings.

Some examples could be a droplet contour that is broken, the droplet base is hidden in the background or the image is too bright and the droplet cannot be detected.

The captured image is still available and can be displayed if the incorrect result line is double-clicked (see Section 9.5 Manual testing in Static/Dynamic Mode)

A.1 Recommended droplet sizes

The default droplet size is 4,0 μl , which has been used as the recommended size in contact angle testing for historical reasons. In general the contact angle is not affected by the droplet size as long as the droplet is not affected by the gravity. For water (having a high surface tension) the contact angle will be stable as long as the full droplet remains in view.

The droplet size may still have an input on the reading, as a bigger droplet will cover a larger test area and might easier pick up contaminants on an inhomogeneous test surface.

For absorbent surfaces it might be easier to track the penetration of liquid into the substrate if a slightly bigger droplet is used for the test. On the other hand, a bigger droplet may spread outside the field of view or cause the test surface to warp leading to erroneous test results.

If the specimen warps when the liquid droplet is applied, it might be necessary to attach the specimen strip to a flat surface with a suitable adhesive.

A.2 Detection of the droplet in Dynamic Mode

The distance between the pendant droplet and the specimen surface is necessary for the droplet to form and release from the dispensing tip before the droplet touches the surface.

The impact on the contact angle from a falling droplet of 4.0 μl is very small. This can be verified if a solid, non-absorbent surface is tested for the static contact angle with 6-8 droplets. When the same specimen is tested with the dynamic approach the contact angles should match the average result from the static test.

Smaller droplets will require a bigger falling distance to release from the dispensing tip. A droplet of 2.0 μl falling from a two-mm distance will still give the same contact angle reading.

Bigger droplets will give lower contact angles for bigger falling distances. This principle can be used to determine the “receding “ contact angle describing the de-wetting properties of a surface.

B.1 Experienced problems

Bundled operating systems

We have come across occasional problems with modified operating systems delivered with certain computers. As a result the displayed image might not appear or it will freeze up on the screen. Kindly note we can take no responsibility for the use of modified operating systems, as these are completely outside our control. Install a licensed Microsoft version on your computer, re-install the PG software and the problem should be gone.

Jumping image

With a limited number of notebook computers, the live image displayed is occasionally unstable. These errors usually occur from interfering accessories (e.g. a built in web camera) or a low refresh rate of the screen in combination with the capacity of the USB port. Use the “Performance Check” in the Help Screen, to check the image capture rate available. If performance is poor, try to switch off background tasks that are not necessary to run in parallel with the PG program. You may also try to increase the refresh rate of the display screen and reduce the resolution of the display screen.

 Please note this error is NOT related to your instrument or the PG software itself!

USB port voltage supply

Use of USB ports has become very popular and sometimes a USB port is expanded using a passive hub. Unfortunately this may affect the power available at the port when different accessories are plugged in/out (e.g. memory stick, mouse, hard disks). If you would like to connect the PGX+ instrument to a hub, please make sure the hub is of an “active” type with its own power supply to avoid possible errors.

C.1 Surface Energy Conversion Chart

Conversion chart	
<i>Water Contact Angle</i>	<i>SFE (dyne/ cm)</i>
51-53	46
54-56	45
57-59	44
60-62	43
63-65	42
66-68	41
69-71	40
72-73	39
74-76	38
77-79	37
80-81	36
82-84	35
85-87	34
88-89	33
90-92	32
93-95	31
96-97	30
98-100	29

Adapted, with permission, from D5946-01 Standard Test Method for Corona-Treated Polymer Films using Water Contact Angle Measurements, copyright ASTM International.

D.1 Replacement of Pump Tubing

- a) Remove the pump tubing from its holder
- b) Open up the pump lid screw (C) and lift up the pump lid.
- c) Disconnect the old pump tubing from the liquid container and the cannulus connected to the droplet dispenser.
- d) Install the new tubing between the liquid container and the tubing connected to the dispenser.
- e) Insert the pump tubing into its track and make sure the pump tubing goes straight through the pump unit when the liquid container is installed into the pump.
- f) Re-install the pump lid and secure the pump lid screw (C) firmly.
- g) Refill the liquid container.
- h) Fill the pump tubing like this:
 - Set the volume selector to position FILL.
 - Press PUMP and the pump will give 50 pump strokes and stop. This volume may produce a small droplet at the dispensing tip. Press once more on the PUMP button to restart the pump. When a droplet without air bubbles appears at the dispensing tip, press and hold the PUMP button until pump stops.
- i) Set the volume selector on top of the instrument to “4.0 μL ”, which is the default droplet size for testing.
- j) Slide the instrument sideways to a new test position.
- k) Use “Static” mode to measure the average volume from six droplets on a flat glass surface. The volume readings may vary around the average but must not show a trend indicating air bubbles in the liquid system. When the average becomes stable it should not vary more than 0,2 μL from the average. If the average is outside $\pm 0.2 \mu\text{L}$ compared to the target of 4,0 μL the droplet volume can be adjusted with the volume adjustment knob (A) on the pump unit.
- l) Leave instrument for 10-15 seconds and check no droplet is building up at the dispensing tip. If a droplet builds up, the pump tubing is not firmly shut. Adjust as follows:
 - Release the hex screw (B) $\frac{1}{2}$ turn.
 - Tighten screw (C) firmly.
 - Repeat if droplet is still appearing at dispensing tip.



Fig. D-1 Release the pump lid screw

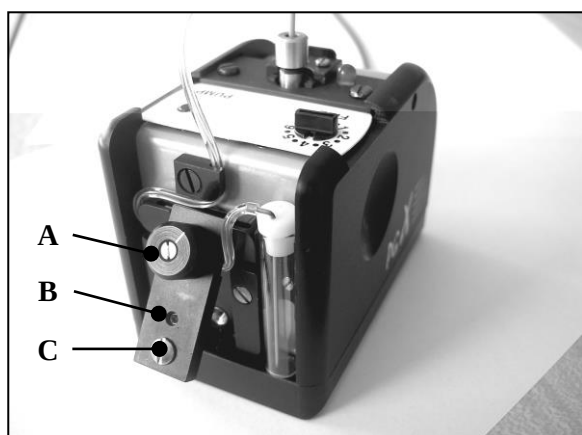


Fig. D-2 Pump tubing can now be removed

D.2 Adjustment of Pump Volume

The micro pump delivers droplets in steps of 0,5 µl and for a target volume of 4,0 µl the pump will make eight pump movements. The Static Mode can be used to measure the average volume from six droplets applied on a non-absorbent specimen surface (e.g. a metal foil or a glass plate). The volume readings will vary around an average and should not show an increasing trend indicating air bubbles in the liquid system.

Two numbers describe the pump performance where

- “repeatability” is the variation in droplet volumes around the average of six volume determinations;
- “deviation” is the difference between the target volume (e.g. 4,0 µl) and the average from the six volume determinations (e.g. 4,1 µL).

The “repeatability” is a function of the pump and cannot be adjusted. This value should not exceed 5% (corresponding to $\pm 0,2$ µl at a target of 4,0 µl).

The “deviation” is a function of the physical dimensions of the pump tubing. This value should not exceed 5% (corresponding to $\pm 0,2$ µl at a target of 4,0 µl). If the average volume from six droplets is outside 5% from the target, the droplet size can be adjusted with the procedure described below.

- a) Locate the volume adjustment knob located on the pump lid (A in Figure D-2).
- b) When the droplet volume is *too high*, turn knob in a counter clockwise direction.
When the droplet volume is *too low*, turn the knob in a clockwise direction.
- c) Take a new set of readings in Static Mode and check the average volume from six droplets.
If necessary repeat step (b) above.

D.3 Small droplets below 0.5 µl

The micro pump delivers droplets in steps of 0,5 µl but to measure SuperLow contact angles it might be necessary to use even smaller droplets. Turn the volume adjustment knob (A in Fig D-2) counter clockwise for a smaller volume. Apply a few droplets on a smooth, reflecting glass surface and determine the set volume in Static Mode.

In SuperLow Mode enter the determined volume into the Volume field (default 0.25 µl) before the measurement is carried out.

 Remember to reset the volume adjustment knob (A) to 0.5 µl when leaving SuperLow Mode.