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JC2000D3 Contact Angle Meter
User Manual

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Chapter 1 Foreword

As is known to all, nanomaterial science and engineering has become a hot area which attracts researchers around the world. However, contact angle is often involved in surface modification of . The contact angle is the angle created by the tangent line of gas-liquid interface and the tangent line of solid-liquid interface at the point where the solid-liquid-gas meets one another after a drop of liquid meets the horizontal surface of a solid.

JC2000D Contact Angle Meters represent a series improved on the basis of JC2000C and is intended mainly for measuring contact angles of liquids toward solids, i.e., the wetting of liquids to solids, and also for measuring contact angles of other phases to liquids. This series can measure contact angles of various liquids to various solid materials, even pellets formed by powder. It provides significant support for research and production in petroleum, printing and dyeing, medicine, painting and mineral dressing.

Chapter 2 Principles

2.1 Defection of Contact Angle

When freely placed in a space not affected by any force field, a drop of liquid is presented as a ball due to surface tension. When the drop contacts a solid surface, its final shape is determined by the relative size of its cohesion and the adhesion between the drop and the solid. When a drop of liquid is placed on a solid surface, it can automatically spread over the surface or change to be another shape that forms a certain contact angle with the surface, as shown in Figure 1.

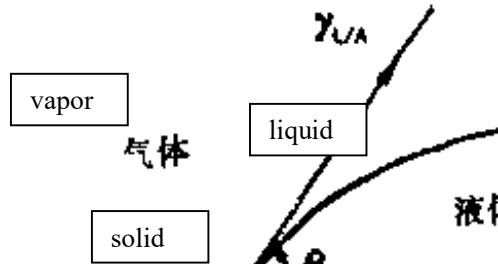


Figure 1. Contact angle

Assuming that the force between two interfaces can be presented as the interfacial tension imposing on the interaction direction, when the drop is in an equilibrium, sum of components of these interfacial tensions along the horizontal direction should be zero, namely,

$$\gamma_{S/A} = \gamma_{S/L} + \gamma_{L/A} \cos \theta \quad (1)$$

where $\gamma_{S/A}$, $\gamma_{L/A}$ and $\gamma_{S/L}$ are solid-air, liquid-air and solid-liquid interfacial tensions; θ is the angle formed by liquid-solid interface and the tangent line of the liquid surface (Including liquid), called contact angle, between 0° and 180° . The contact angle is an important measure to reflect wetting relationship between a matter and a liquid. $\theta=90^\circ$ is the line between non-wetting and wetting, $\theta<90^\circ$ means wetting exists while $\theta>90^\circ$ no wetting exists.

2.2 Wetting

In thermodynamics, if free energy (G) of a solid-liquid system can be lowered after a the solid contacts the liquid, there is wetting between the solid and the liquid. The degree to which free energy is lowered is called wettability, in $W_{S/L}$. There are three types of wetting: adhesional wetting, spreading wetting and immersional wetting. Figure 2 describes the three kinds of wetting.

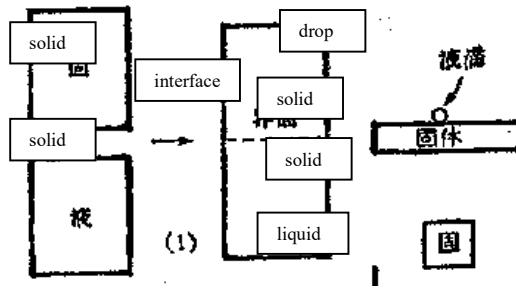


Figure 2 Three kinds of wetting

(1) Adhesional wetting

If the initial 1m^2 solid surface and 1m^2 liquid surface disappear to form a 1m^2 solid-liquid interface, the $W_{S/L}^A$ for this process is :

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$$W_{S/L}^A = \gamma_{S/A} + \gamma_{L/A} - \gamma_{S/L} \quad (2)$$

(2) Spreading wetting

When a drop of liquid spreads over a 1m^2 solid surface, the initial 1m^2 solid surface and the drop (whose area can be ignored) disappear to form a 1m^2 liquid surface and a 1m^2 solid-liquid interface, the $W_{S/L}^S$ for this process is :

$$W_{S/L}^S = \gamma_{S/A} - \gamma_{L/A} - \gamma_{S/L} \quad (3)$$

(3) Immersional wetting

When a 1m^2 solid surface is immersed into liquid, the 1m^2 solid surface disappears and a 1m^2 solid-liquid interface forms, the $W_{S/L}^I$ for this process is :

$$W_{S/L}^I = \gamma_{S/A} - \gamma_{S/L} \quad (4)$$

For the said three kinds of wetting, how to obtain $W_{S/L}$ if $\gamma_{S/A}$ and $\gamma_{S/L}$ cannot be measured?

Discussions are given as below:

① Adhesional wetting

Take (1) into (2), we have: $W_{S/L}^A = \gamma_{L/A}(1 + \cos\theta)$ (5)

For liquid surface tension $\gamma_{L/A}$ is known, only contact angle θ is needed to obtain $W_{S/L}^A$.

② Spreading wetting

Take (1) into (3), we have: $W_{S/L}^S = \gamma_{L/A}(\cos\theta - 1)$

For $\cos\theta \leq 1$, $W_{S/L}^S \leq 0$. As we know that $W_{S/L}$ represents the reduction of free energy, but the said formula means there must be a spontaneous process that causes free energy to increase or keep constant. Apparently, this does not observe the second law of thermodynamics. This is because of the wrong application of formula (1), which only applies to the equilibrium state. The drop of liquid and the solid surface do not reach the equilibrium state if the drop automatically spread to cover the entire solid surface; in this case, formula (1) cannot be taken into formula (3). It should be noted that spread wetting should not be regarded as a case when $\theta = 0^\circ$. Spread wetting means there is not contact angle, but $\theta = 0^\circ$ means there is a contact angle, which equals to 0° .

Assuming that a solid contacts a vapor whose pressure is going up to absorb the vapor. when the pressure reaches P_0 , saturated vapor pressure, there is an extreme thin liquid on the solid surface.

$$\int_{P_0}^P \Gamma d \ln P$$

According to Gibbs adsorption equation that, reduction of surface free energy = $RT \int_{P_0}^P \Gamma d \ln P$.

$$\text{Thus, } W_{S/L}^S = \gamma_{S/A} - \gamma_{L/A} - \gamma_{S/L} = RT \int_{P_0}^P \Gamma d \ln P \quad (6)$$

③ Immersional wetting

Remove the $\gamma_{L/A}$ in formula (6), we have $W_{S/L}^I$:

$$W_{S/L}^I = \gamma_{S/A} - \gamma_{S/L} = RT \int_{P_0}^P \Gamma d \ln P \quad (7)$$

It is known from formula (5) that when $\theta = 0^\circ$, $\cos\theta = 1$ and $W_{S/L}^A = 2\gamma_{L/A}$, reduction of free energy reaches the maximum, it is regarded that the solid is completely wetted by the liquid; when $\theta = 180^\circ$, $\cos\theta = -1$, $W_{S/L}^A = 0$, reduction of free energy is 0, it is regarded that the solid is totally

unwetted by the solid. They are two ideal conditions because in practice, there must be a certain force of attraction between a solid and a liquid.

2.3 Measurement of contact angle

For an ideal flat solid surface, the a drop of liquid reaches the equilibrium on the surface, there is only once contact angle that observes the Young's equation. But in fact, the surface of a solid is not ideal, which leads to a delay in the formation of contact angle and thus makes it difficult to repeat the measurement of a contact angle. It is, however, possible to have data with an acceptable repeatability, with deliberately treated surface, in particular, the surface of polymers. The aim of preparing and treating a surface is to obtain a smooth and clean surface that is ideal. The specific procedures for this aim depend on samples, which are not detailed in this manual. The following are some common methods for measurement of contact angles and they are designed for contact angles of air-liquid-solid systems. Some of them, however, are also applicable to liquid-liquid-solid systems once slightly modified.

1. Goniometry

Goniometry is one of the most common methods for measurement of contact angles, as shown in Figure 3(a, b). The contact angle is directly measured by projecting the drop of liquid on the solid surface, or the bubble formed by the solid which is immersed into the liquid, onto a screen, and then directly measuring the angle formed by the tangent and the liquid-solid phase interface.

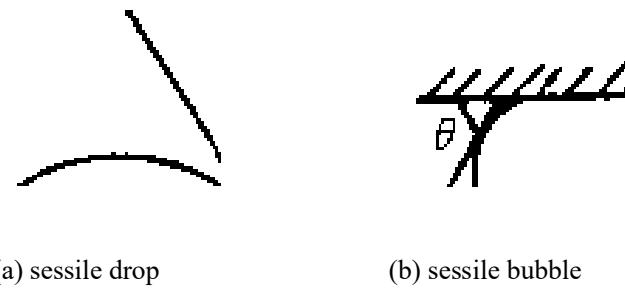


Figure 3 Goniometry schematics diagram

If vapor of the liquid is absorbed on the solid surface, which affects free energy of the solid surface, the sample should be placed in a seal container with an observation window and the measurement should not be proceeded until the system reaches an equilibrium. The advantages of this method include: less samples, simpler instrument and easier measurement. In addition, this method provides an accuracy of about $\pm 1^\circ$.

2. Height method

If the droplet is so small that its deformation caused by gravity can be ignored, the droplet can be regarded as a part of a ball, as shown in Figure 4. In this case, the contact angle can be measured through measuring the height with the following formula:

$$\tan \frac{\theta}{2} = \frac{2h}{d} \quad (8)$$

where, h is the droplet height and d is diameter of the droplet's bottom. This method applies to a droplet whose size is smaller than 10^{-4} mL. If the contact angle is less than 90° , this method also applies to a slightly larger droplet.



Figure 4 Height method schematics diagram

Goniometry can also be used to measure a contact angle formed by a droplet on a fiber by placing a straightened fiber in the sample tank and have it projected on a computer screen to directly measure the angle between the droplet and the fiber surface. If the droplet is small enough, the contact angle can also be measured through height method and calculated with formula (8).

In reality, almost all solid surfaces are not ideal, contact angle delay more or less occurs to all interfaces. For this, both advancing angle and receding angle need to be measured. In the case of sessile drop method, the contact angle can be measured by increasing the droplet volume. The contact angle measured by increasing the droplet volume is advancing angle, as shown in Figure 5(a); and that by decreasing the droplet volume is receding angle, as shown in Figure 5(b).

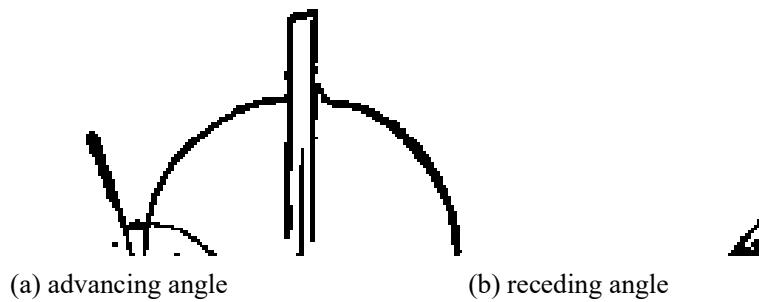


Figure 5 Measurement of advancing angle and receding angle

To avoid droplet vibration and deformation that is possibly caused by increasing and/or decreasing droplet volume, tip of the capillary tube that is able to change droplet volume is inserted into the droplet during the measurement process because tip inserting does not affect the contact angle reading.

Many factors determine and influence wetting and contact angle. For example, nature of and impurities in the solid and the liquid, additives, toughness of the solid surface, unevenness, surface contamination, etc. For a given solid surface, addition of surfactant into the liquid can often improve the wetting; and as the contact time between the liquid and solid increases, the contact angle tends to be smaller and become stable, a result caused by absorption on interfaces by the surfactant.

2.4 Measurement of surface tension

The idea of studying surface (interfacial) tension through pendant drop method was proposed in late 19th century. The first study with practical value was not performed until Andreas et al in 1937, they compared five methods and proposed to measure dimensional parameters of the pendant drop with surface selection. Andreas makes:

$$S = \frac{d_s}{d_c} \quad (9)$$

$$H = -\beta \left(\frac{d_c}{b} \right)^2 \quad (10)$$

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where d_c is the largest diameter of the pendant drop, d_s is the diameter of the circle whose appendicular distance with the pendant drop bottom equals d_c , as shown in Figure 6, and β and b are shape factor and size factor in Bashforth-Adams equation.

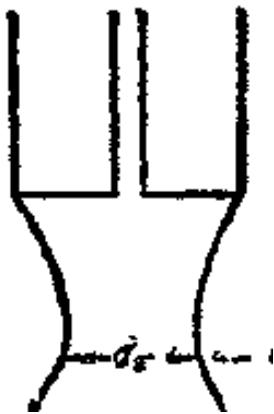


Figure 6 Pendant drop method diagram

The formula for calculating the surface tension is:

$$\gamma = \frac{(d_l - d_v) g b^2}{\beta} = \frac{(d_l - d_v) g d_c^2}{\beta} \quad (11)$$

where d_l and d_v are liquid phase and vapor phase densities. Thus, with d_c and d_s measured with the projection method and H obtained in other ways, the surface tension can be calculated. Andreas et al

found that $\frac{1}{H}$ is somehow related to S . They measured d_c and d_s of conductive water pendant drops of various sizes and shapes and calculated S . Next, given surface tension of conductive water (25°C, 72.0mN/m), they calculated $\frac{1}{H}$ value with formula (10) and made $\frac{1}{H} - S$ data list. This is the first list of parameters used for pendant method, an empirical result whose accuracy is restricted by the accuracy of surface tension of the conductive water they used. Later, Bartell, and Nicederhouser and

Fordham independently calculated the theoretical $\frac{1}{H}$ value and S value with numerical solution to

Bashforth-Adams equation and made the $\frac{1}{H} - S$ lists with a step of 0.001 for a range from 0.670 to 1.002. In relation to the two lists for the entire range, their results are identical until the fourth

decimal place, indicating the high accuracy. Then, Stauffer gave the $\frac{1}{H} - S$ list from 0.3 to 0.66 through theoretical calculation and therefore expanded the application scope of pendant drop method.

Table 5 of Appendix 3 gave the $\frac{1}{H} - S$ list from Fordham and Stauffer.

Pendant drop method is advantageous because it is a complete equilibrium and facilitates the study of liquid surface aging. The crucial point for successful use of this method lies in stability of the pendant drop and vibration prevention. According to a large number of measurements, Ambwani *et al* believe the relative error of surface tension obtained with this method is limited to 0.15%.

Chapter 3 Instrument Structure

3.1 Technical indicators

1. Measurement methods: Goniometry, height method, fitting analysis, and imaging analysis.
2. Measurement range: $0 \sim 180^\circ$;
3. Measurement precision: 0.1° or 0.5° ;
4. Temperature range: Room temperature: heating platform with electric-controlled temperature is an optional (room temperature $\sim 120^\circ$).
5. Image magnification: $55\text{pixel/mm} \sim 315\text{pixel/mm}$;
6. Solid sample size: $70 \times 100\text{mm}$;
7. Dimensions of the main unit: $500 \times 320 \times 400\text{mm}$
8. Total power: 220V 200W (including the computer);

3.2 System components

3.2.1 Hardware

Contact angle measurement meter (main unit platform), manual 3D platform for samples, precise sample feeder, CCD camera, continuous variable magnification system, manual CCD inclination angle, and mobile platform, as shown in Figure 7.

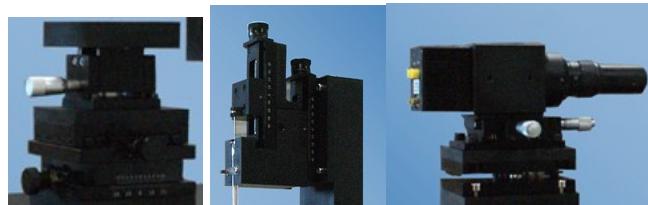


Figure 7 Manual 3D platform for samples, precise sample feeder, CCD camera.

One CCD USB cable, as shown in Figure 9.



Figure 9 USB cable

One computer that matches contact angle measurement meter.

The recommended configuration of computer, if provided by the user, are at least as below:

Pentium 4 or higher CPU;

USB 2.0 port;

2G or larger memory;

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5G or larger disk space;

High performance integrated graphics card like ATI, NVIDIA, Intel

3.2.2 Software

Operating system

Win7/Win10

Included software

Contained in the CD supplied with the product are:

Instrument application software;

Drive program that goes with the equipment;

Instrument instructions and user manual.

Chapter 4 System Installation

Section 1 Software installation

The computer supplied with the contact angle measurement meter sold by the Company has system programs installed and software installation CD provided. The following steps should be followed for a computer provided by the customer or for re-installing the system:

4.2.1 CCD drive software

The CCD drive is located in the CD/chemistry/USB drive directory. Without the CCD drive installed, the CCD cannot work.

(1) Installation of CCD drive program

In relation to a CCD that is connected to the computer for the first time, a message will pop out from the computer that a new hardware is found and the hardware's drive program is required to be installed, as shown in Figure 10.



Figure 10 Find new drive wizard

The following are steps for installation of the hard drive in a win7 system

Click "cancel" button. Click "browse the computer to search hard drive software" option. Open the "instrument program/chemistry/USB drive/ mercury camera\", as shown in Figure 10, and click the MER_Setup_cn_151130.exe.

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Figure 11 Drive program installation 1

Click "next" on the screen as shown in Figure 11.

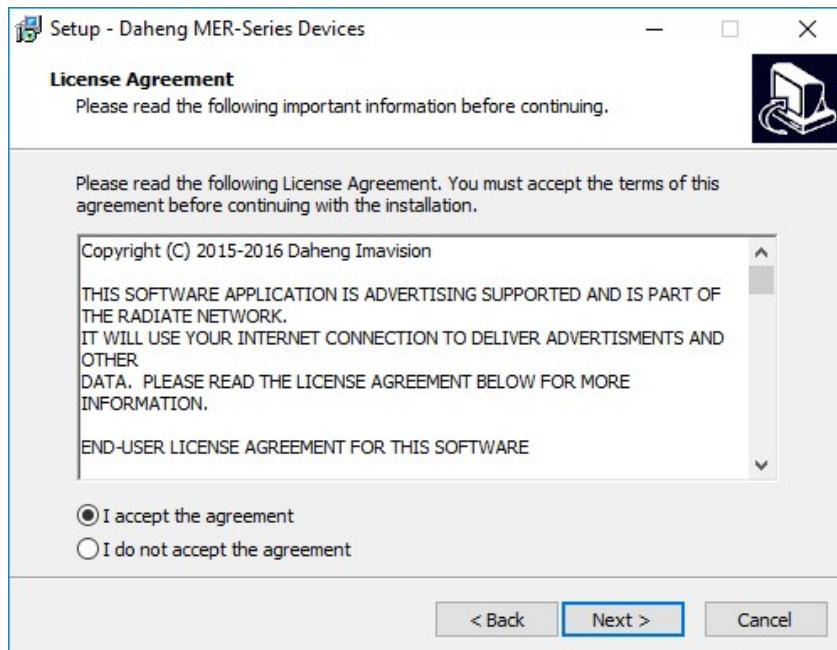


Figure 12 Drive program installation 2

Click "I accept this agreement" option and "next", as shown in Figure 12.

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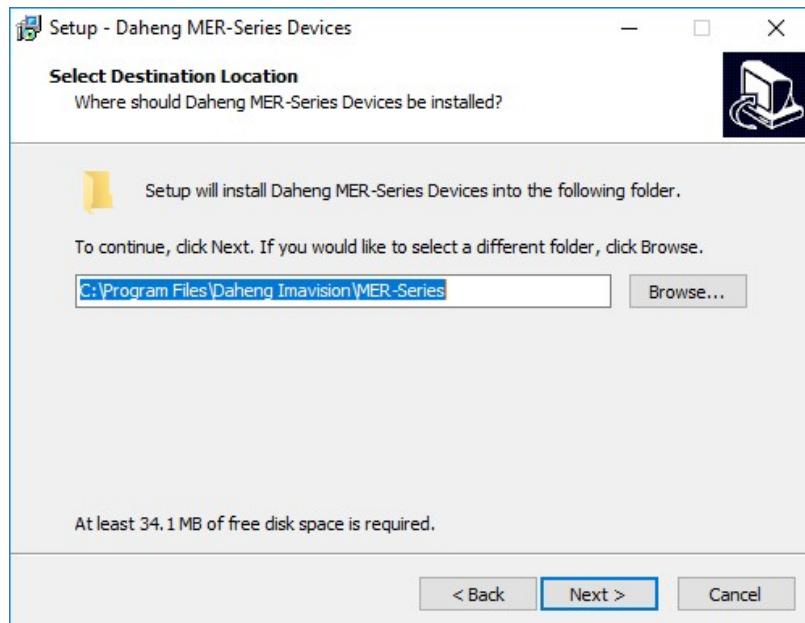


Figure 13 Drive program installation 3

The screen shown in Figure 13 will pop out. Do not change the default path. Click "next".

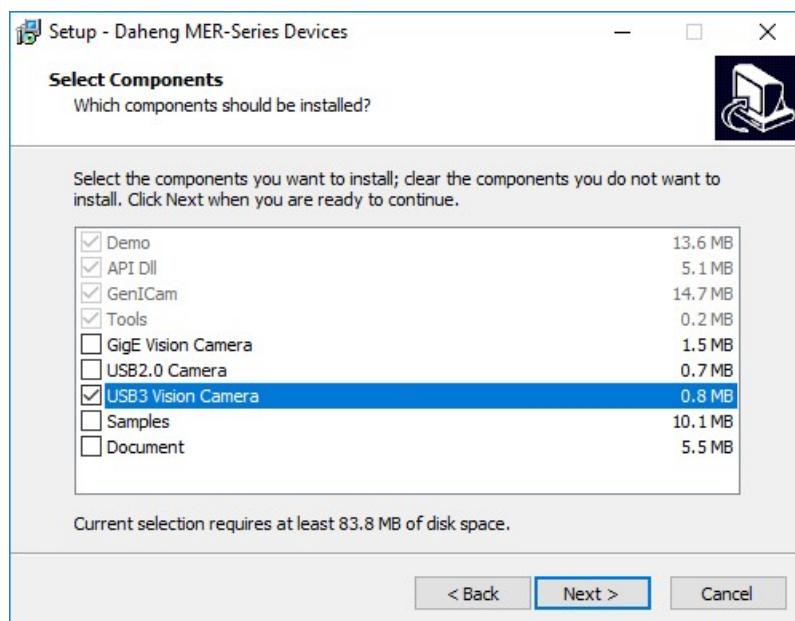


Figure 14 Drive program installation 4

The screen shown in Figure 14 will pop out. Do not change the default path. Click "next".

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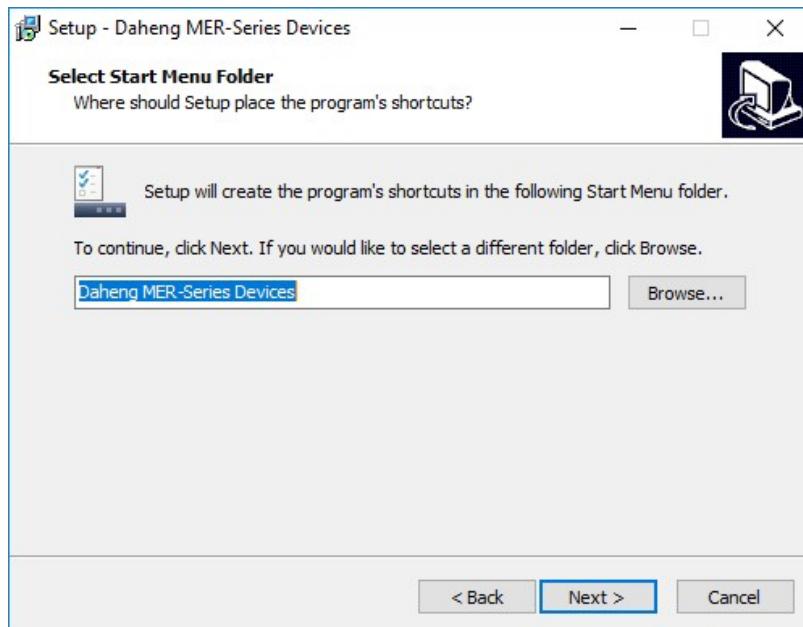


Figure 15 Drive program installation 5

The screen shown in Figure 15 will pop out. Do not change the default path. Click "next".

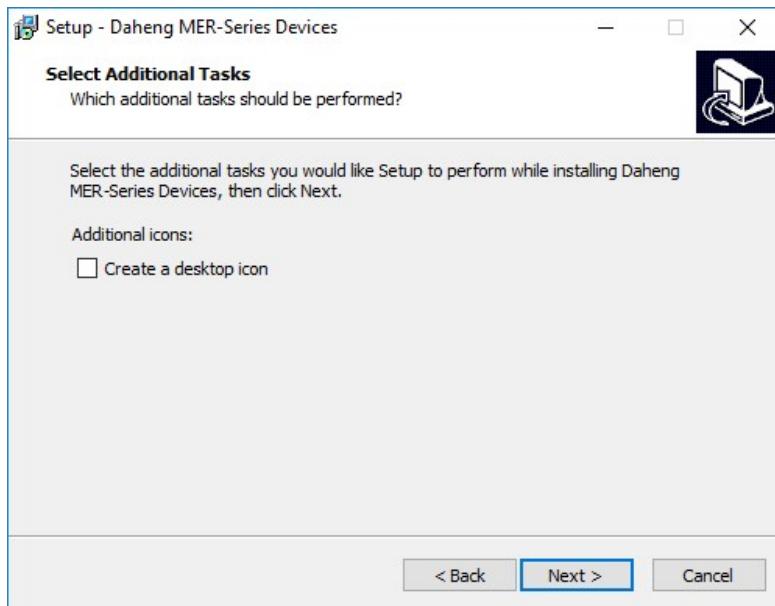


Figure 16 Drive program installation 6

Click "next" on the screen as shown in Figure 16.

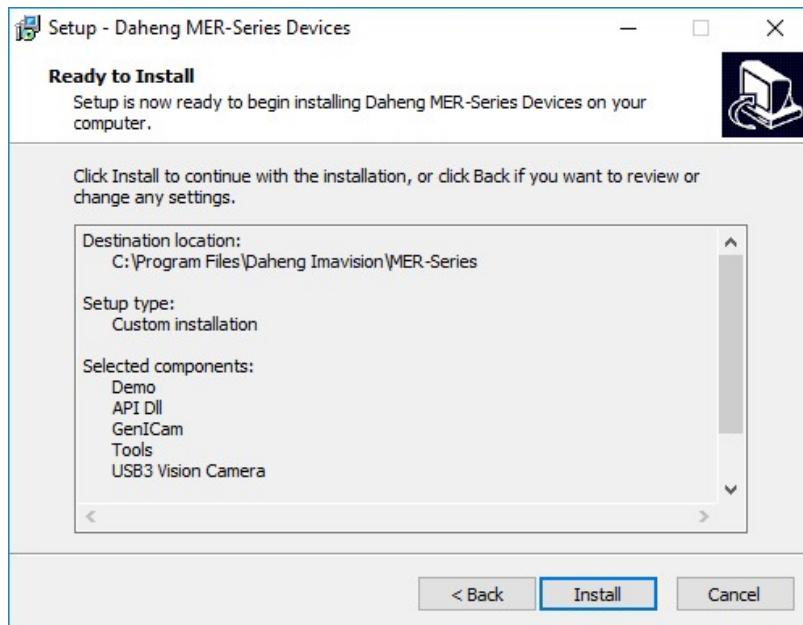


Figure 17 Drive program installation 7

Click "next" on the screen as shown in Figure 17.

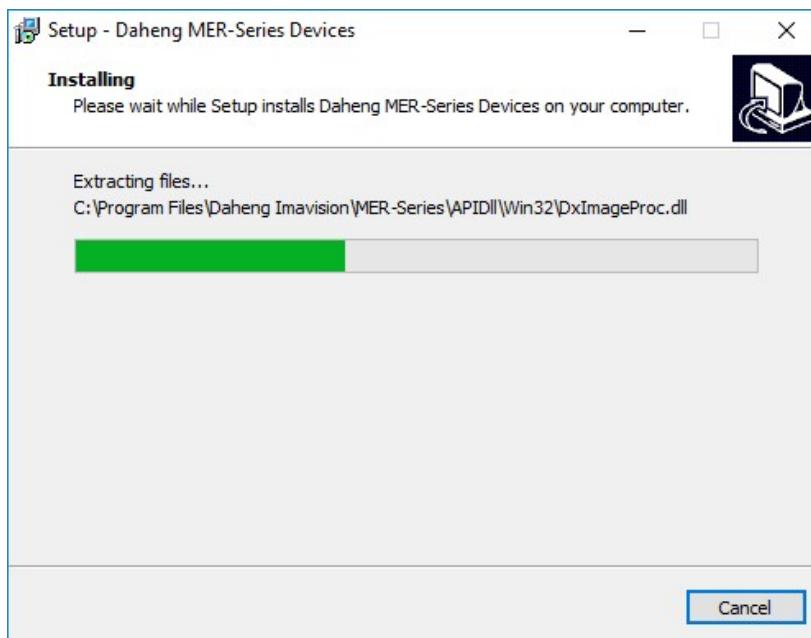


Figure 18 Drive program installation 8

A program bar will be displayed, as shown in Figure 18.

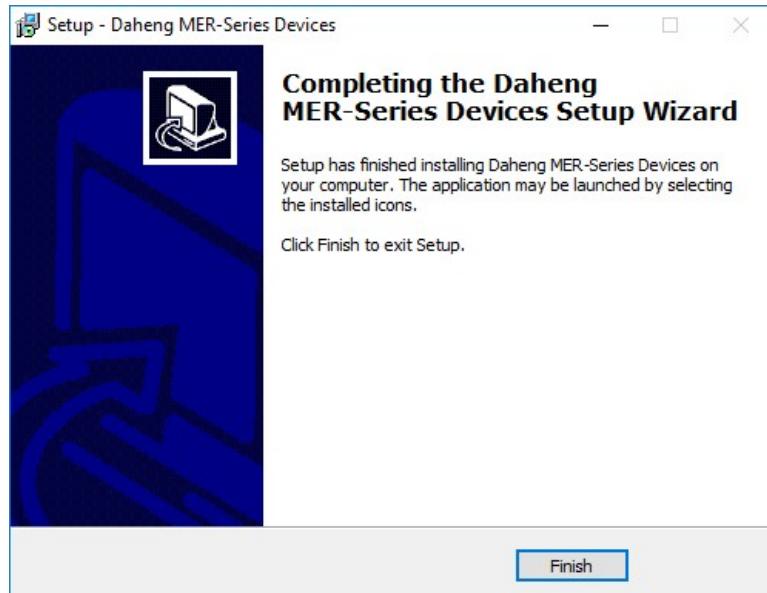


Figure 19 Drive program installation 9

Click "finish" on the screen as shown in Figure 19.

If the drive is properly installed, there will be a graphics processing unit in the device manager under the control panel.

If the drive program is either not installed or installed incorrectly, there will be a graphics processing unit-USB device together with a yellow exclamation point in the device manager under the control panel, as shown in Figure 20.



Figure 20 An unknown USB device found in device manager.

Repeat the CCD drive software installation steps in 4.2.1 to complete installation.

4.2.2 Installation of series port drive software

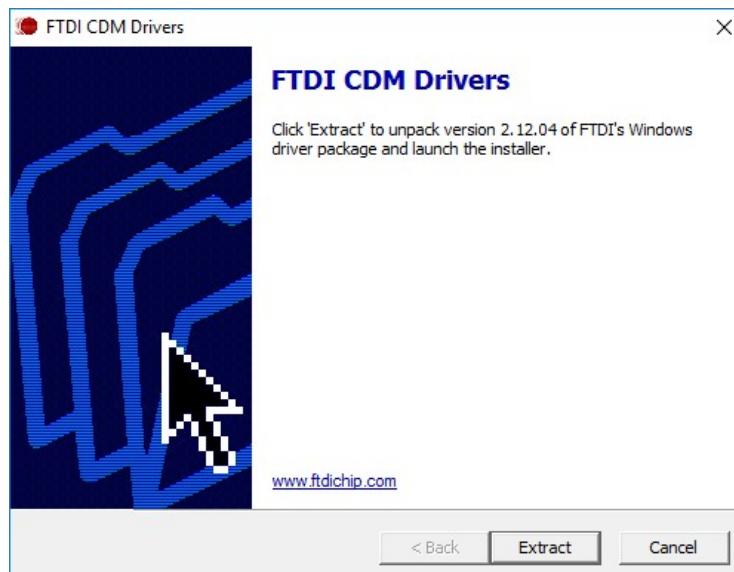


Figure 21 Installation of series port drive program 1

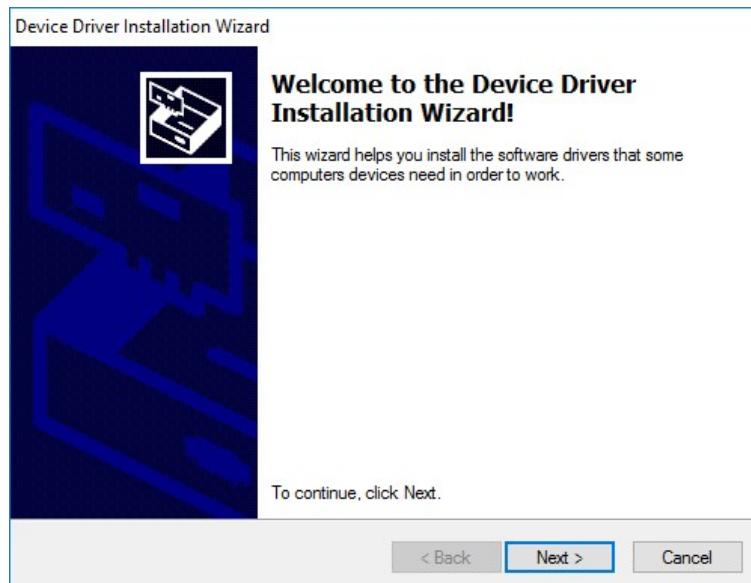


Figure 22 Installation of series port drive program 2

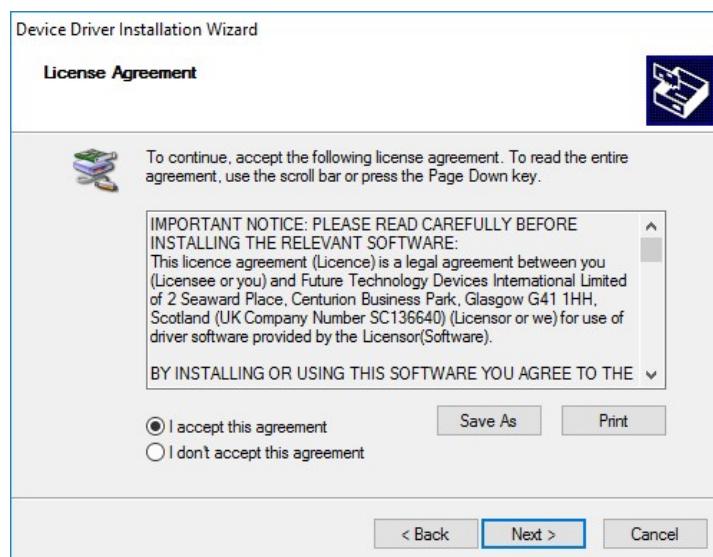


Figure 23 Installation of series port drive program 3

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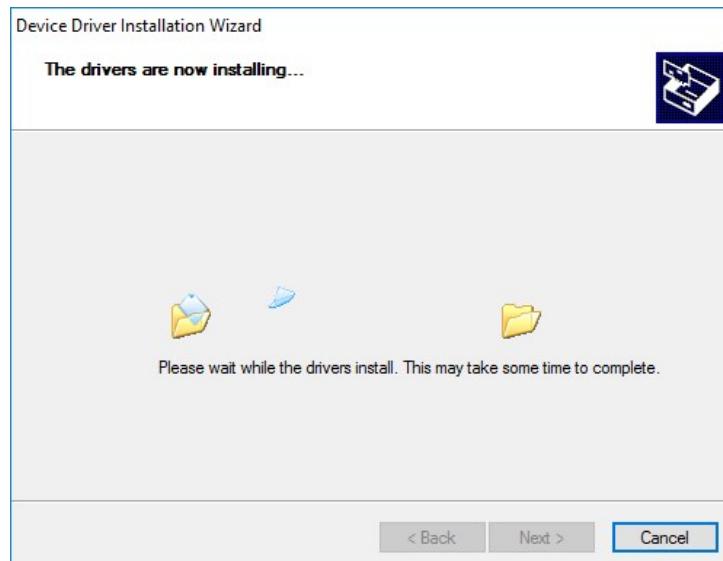


Figure 24 Installation of series port drive program 4



Figure 25 Installation of series port drive program 5

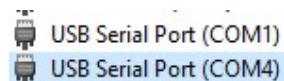


Figure 26 Installation of series port drive program 6

4.2.3 Operating software installation

Click the JC2000setup.exe in "x:\chemistry/instrument program/contact angle/contact angle program\" in the CD.

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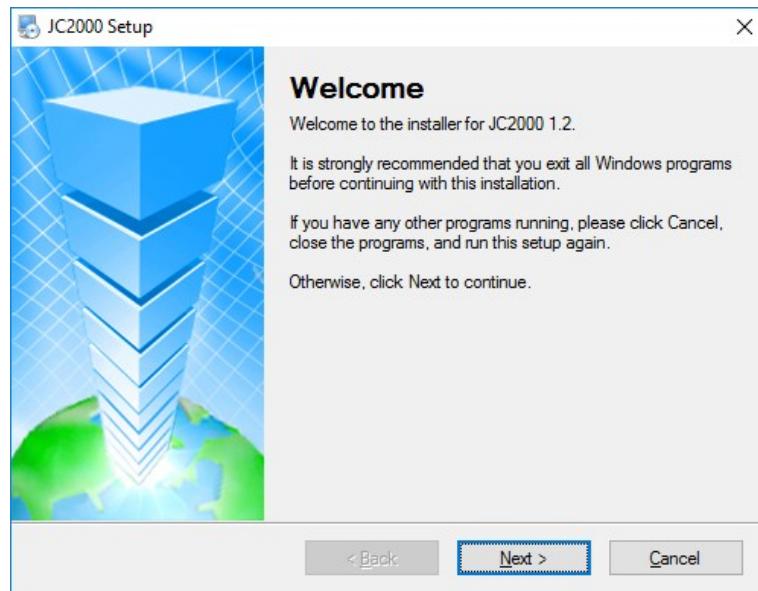


Figure 27 Installation step 1

Click "next" on the screen as shown in Figure 28.

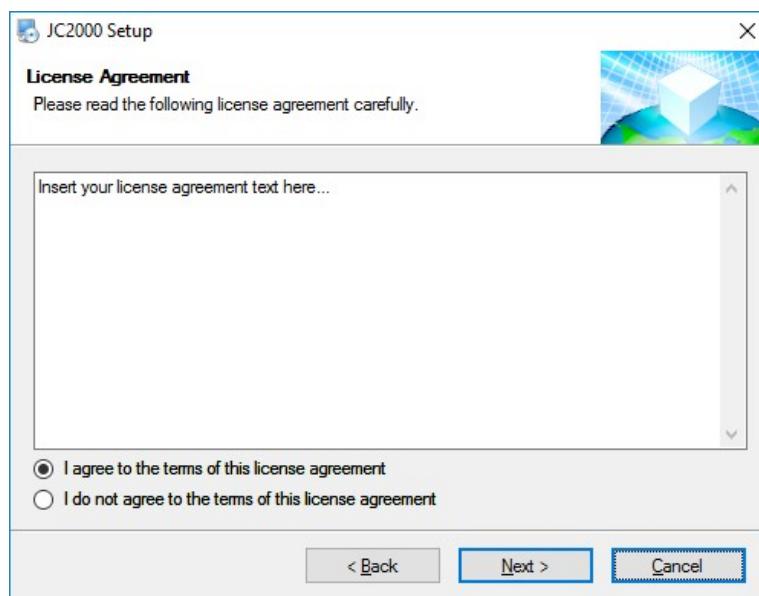


Figure 28 Installation step 2

Click "I accept this agreement" and then "next" on the screen as shown in Figure 29.

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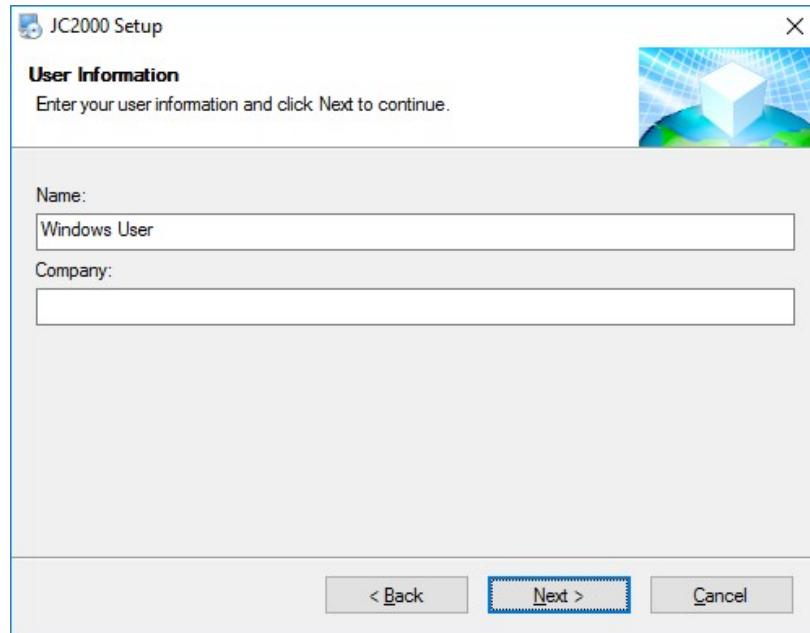


Figure 29 Installation step 3

Fill out name and company and then click "next" on the screen as shown in Figure 30.

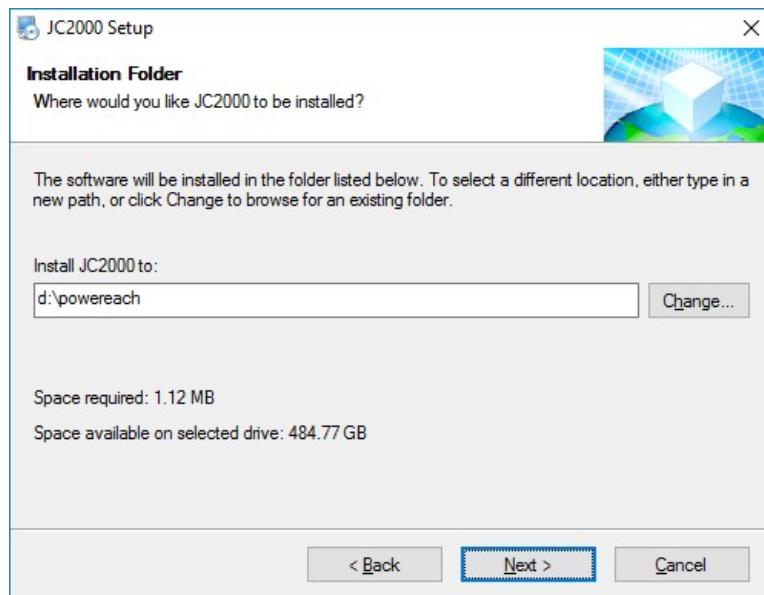


Figure 30 Installation step 4

The screen shown in Figure 31 will pop out. Do not change the default path. Click "next".

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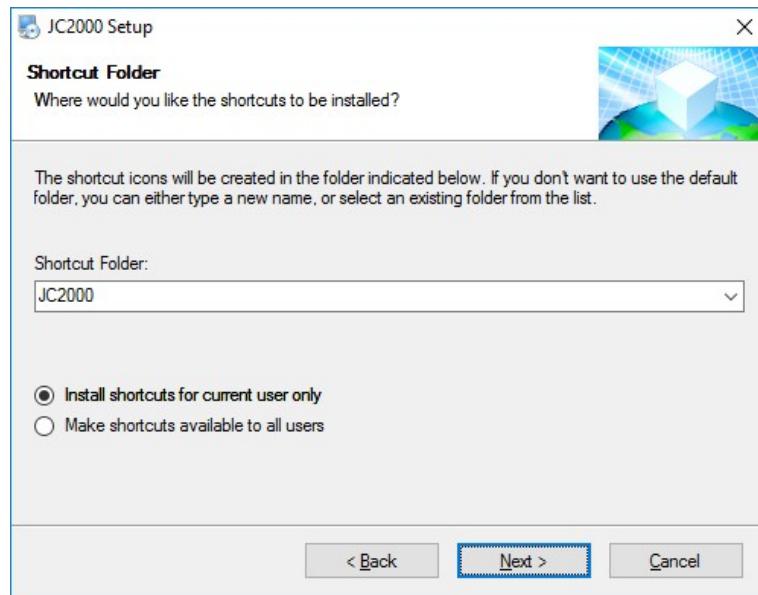


Figure 31 Installation step 5

The screen shown in Figure 32 will pop out. Do not change the default path. Click "next".

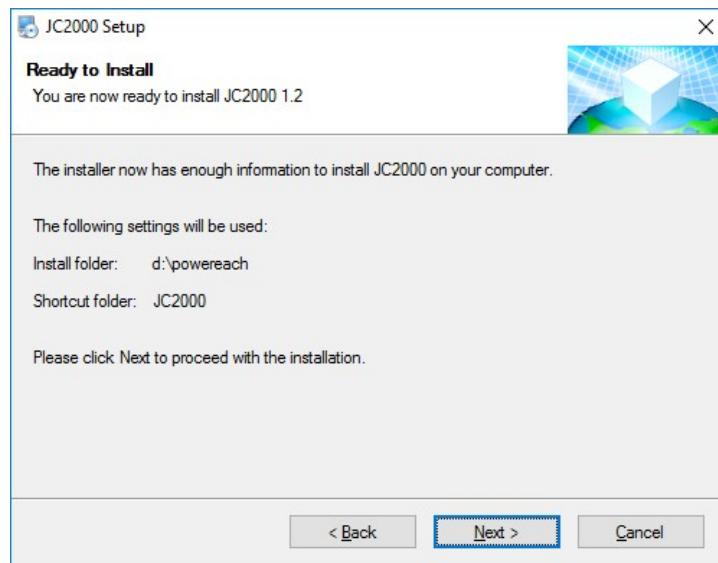


Figure 32 Installation step 6

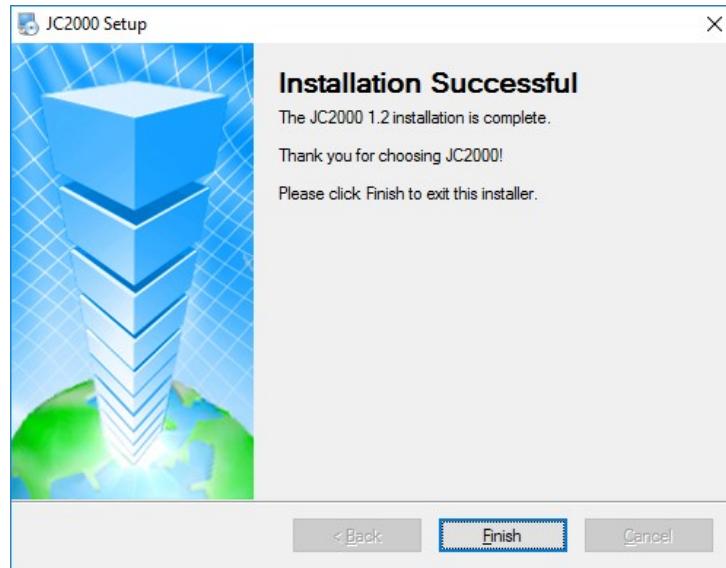


Figure 33 Installation step 7

Click "finish" on the screen as shown in Figure 33.

Then copy the JC2000 directory to the root directory in hard disk D. Then click on JC2000en.exe to enter the operating program.

4.2.4 Monitor setting

Click right button of the mouse on the desktop —>attributes—>selection setting; in the color quality column, select "maximum 32 bit" and in the screen definition column, select "1600 x 1200" pixels or higher (depending on graphics card and monitor).

Chapter 5 System Use

5.1 Start

Operate JC2000.exeto start the application program of contact angle measurement meter. The program's main screen is shown in Figure 35.

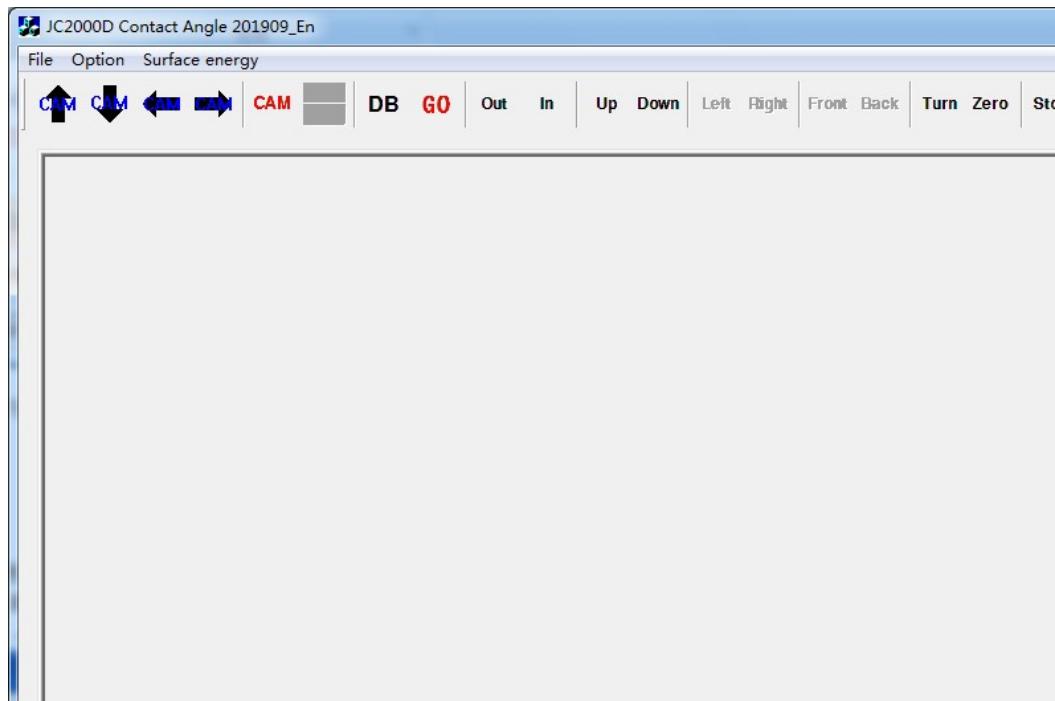


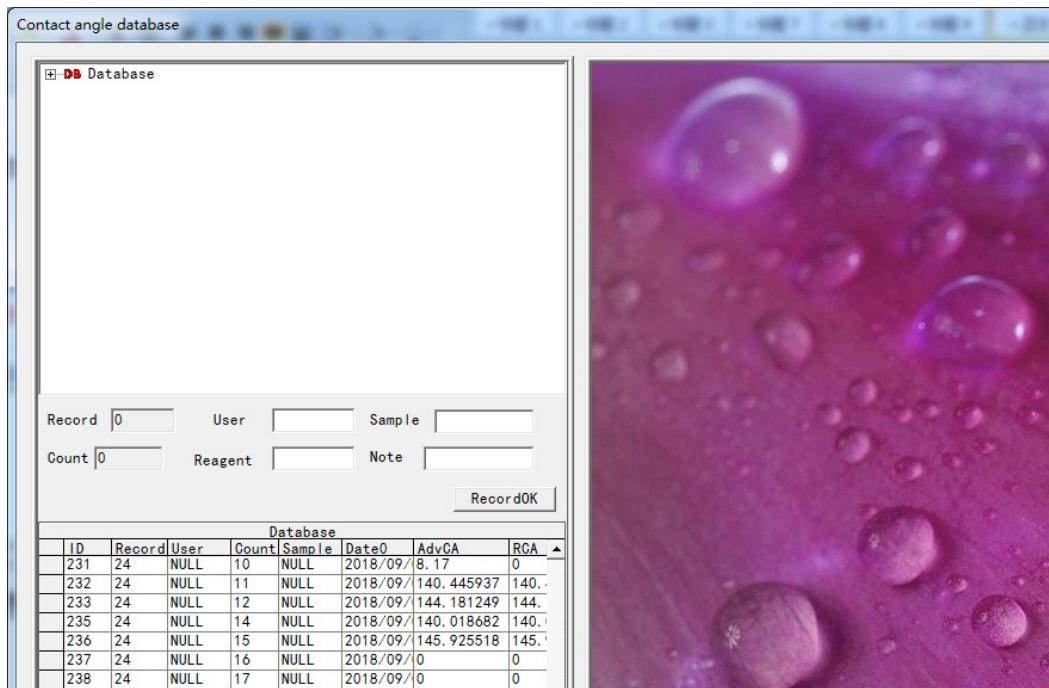
Figure 35 Main screen of the application program of contact angle measurement meter

5.2 Description of sampling module

- The large rectangular area on the left side of the screen is for image display. Click "live" button to see the current image taken by camera.
- Toolkit above the display area contains operating buttons, which are detailed as below:

- Adjust video sampling area: adjust the video display area upward, downward, leftward and rightward
- Activate image: click this button to have the image displayed activated for the next operation.
- Freeze image: click this button to free the image displayed for the next operation.
- DB** Database: Before a test, the user can set test parameters in the database.
Click data button to enter the database screen.

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With this button, the user can select to add or create a record after the original record.

In the case of creating a new record, the operator, sample name, test reagent and other necessary information must be completed.



After all information is provided, click record done button. Thus, the subsequent data will be added to the database.

Also, it is possible to add a record by selecting an existing record.

Click 12SS record and click record done button. Thus, the subsequent data will be added to the database.

G is a test button. Click this button after the sample is fed to automatically measure the contact angle.



These are buttons used when corresponding control accessories are provided.

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Out

In

Up

is used when an automatic sample feeder is available.

Left **Right**

Front

are used when an electric-controlled displacement platform is available.

Turn **Zero**

are used when an electric-controlled rotation platform is available.



are used when the transient memory is activated.

5.3 Menu description

File Option Surface energy

File menu is shown in Figure 36.

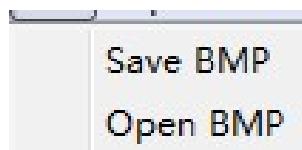


Figure 36 File menu options

The above 3 options are: "save BMP", "open BMP" and "open LIS file". Generally, only one frame is sampled in contact angle measurement test; so after the image is frozen, click on "save BMP" to save the image. Then, open the image for analysis.

"Open LIS file" is used for analysis after a transient photo-taking.

Setting menu is shown in Figure 37.

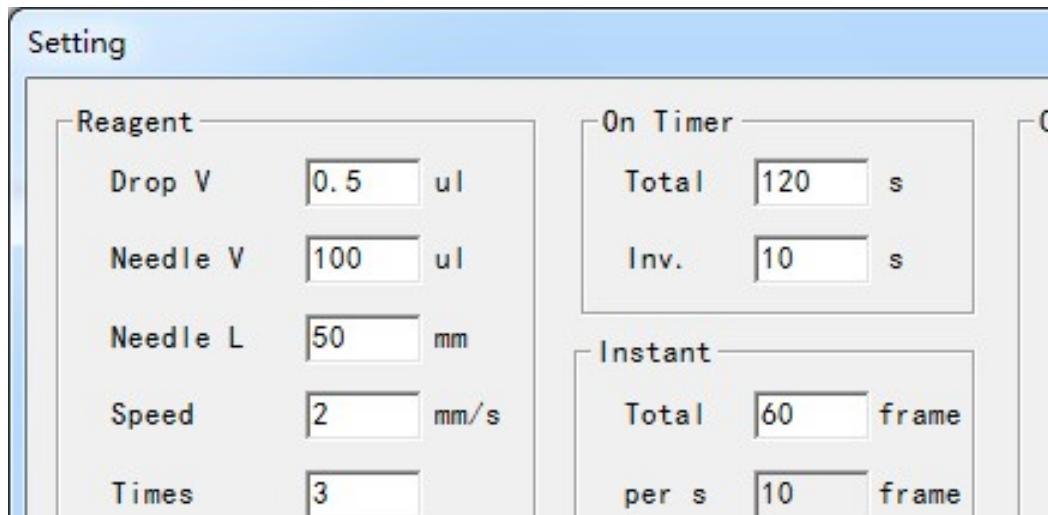


Figure 37 Setting menu options

"Setting" option in the menu is used for setting parameters. When the "setting" option is chose, a dialogue box as shown in Figure 37 will pop out.

Reagent setting is used for setting contact angle for automatic feeding.

The drop volume can be set as needed between 50 ul and 0.1 ul.

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The needle (sample feeder) is 50 ul or 100 ul.

The needle length is 50 mm.

Sampling speed

Cyclic feeding times

Timer analysis

"**Total**" is the total time set for analysis. "**Interval**" is the time between two analyses. For example, Figure 37 means the analysis is performed at an interval of 10 seconds and a total of 30 seconds is provided for information storage.

The "**Total**" in "**Instant**" (which means total frames for instant analysis) describes the total frames stored. The frame speed is measured at times per seconds. Storage time=total frames/frame speed.

Baseline If the automatic analysis is not accurate enough, this option can be ticked to provide helping lines to increase the accuracy of automatic analysis.

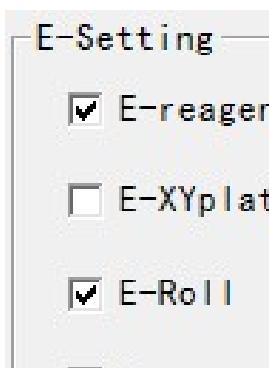


The picture below describes the modes available for contact angle analysis. There are four methods provided. The default method is single analysis (once).

Freeze is used to freeze the image after the analysis.

The other three methods are on timer, instant and continues positioning.

Click to see an advanced setting dialogue box.



Choose the module that goes with the machine configuration. **Please resort to the engineer for the machine configuration. Other parameters may not be changed without permission.**

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5.4 Use steps

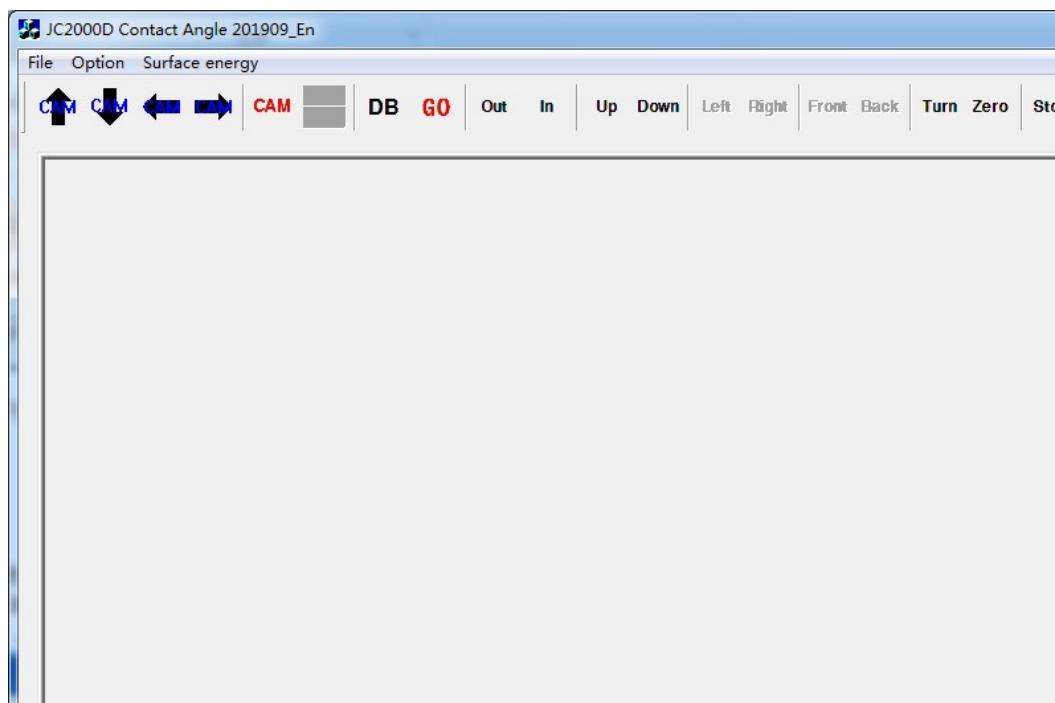
5.4.1 Testing

Connect the instrument (optional)—place the sample—connect the database (optional)—feed the sample—save image (optional)—measure the image—record

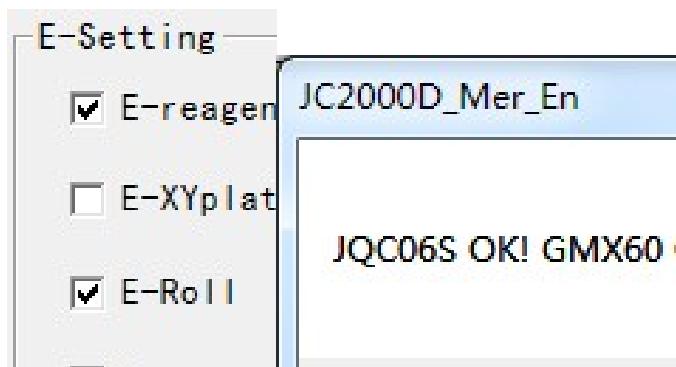
Figure 38 Test process

Steps

Open JC2000en.exe



**If an automatic module is purchased, please choose the correct module in advanced setting and then click "connect".



**This step is skipped if the module is not purchased.

#Click **CAM** button to activate the image.

#Click **DB** database button to set database connection.

#Place the sample in the sample platform.

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#Click right button of the mouse to draw an analysis box to increase the analysis accuracy.



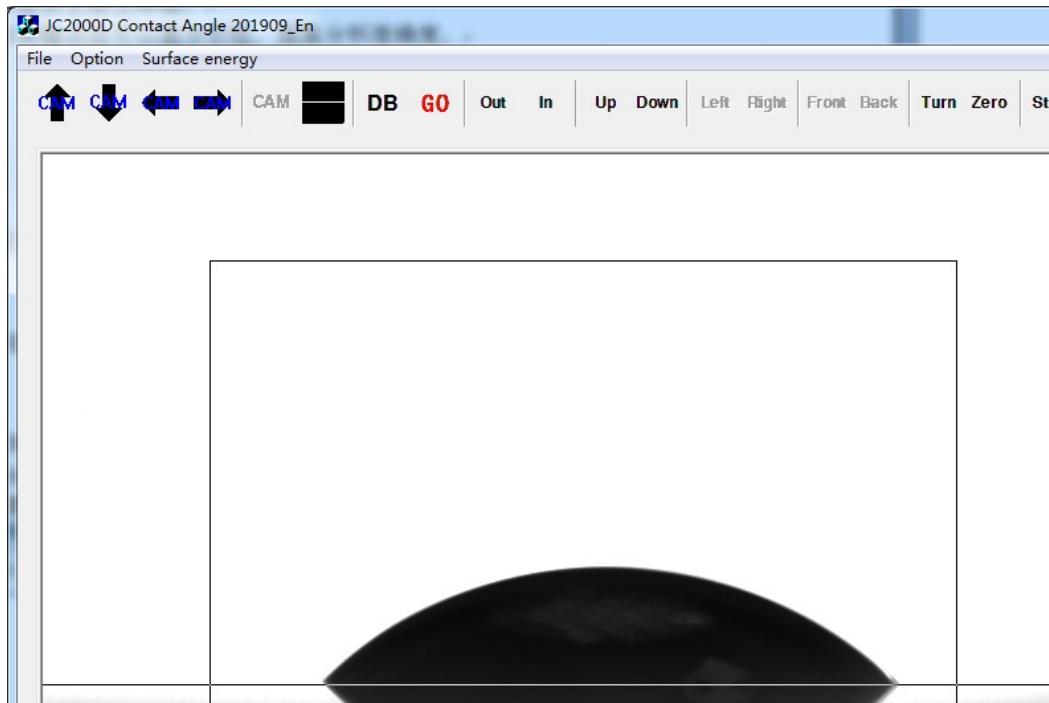
Set contact angle analysis mode. The default method is single analysis (once).

*If an automatic feeder module is available, click **GO** button; the contact angle will be automatically measured after the sample is fed.

**If the feeder module is not available, please feed the sample manually. Ensure the drop is on the

sample, then click **GO** test button to automatically measure the contact angle.

The baseline position can be adjusted to increase the analysis accuracy. This should be completed in the option setting.

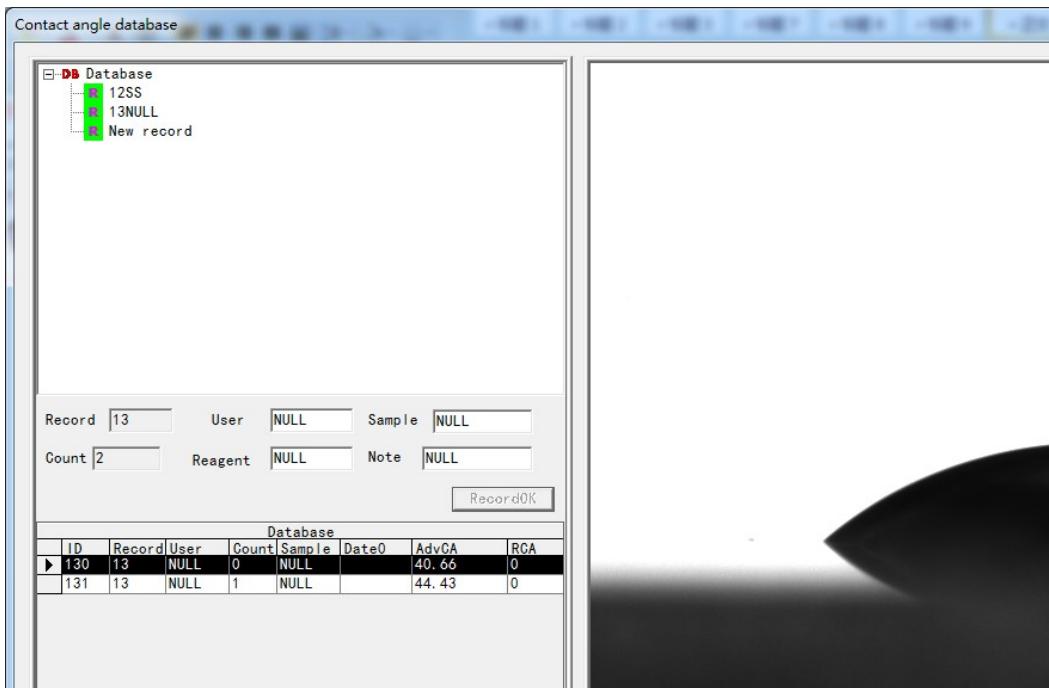


After the analysis is completed, the contact angle will be displayed on the

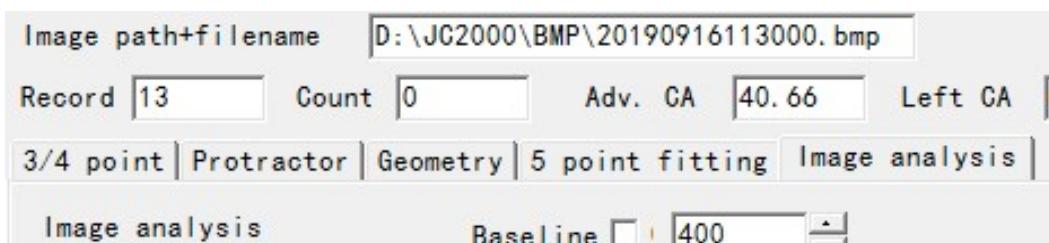
screen.

#Click **DB** button to enter the database to find relevant data.

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#If the automatic measurement is not accurate enough, alternative measurement methods are provided on data screen.



Choose a record. Choose a corresponding method and click "start" button.

5.4.2 Goniometry

Click the goniometry button to enter the main screen, as shown in Figure 36. Click "start" button.

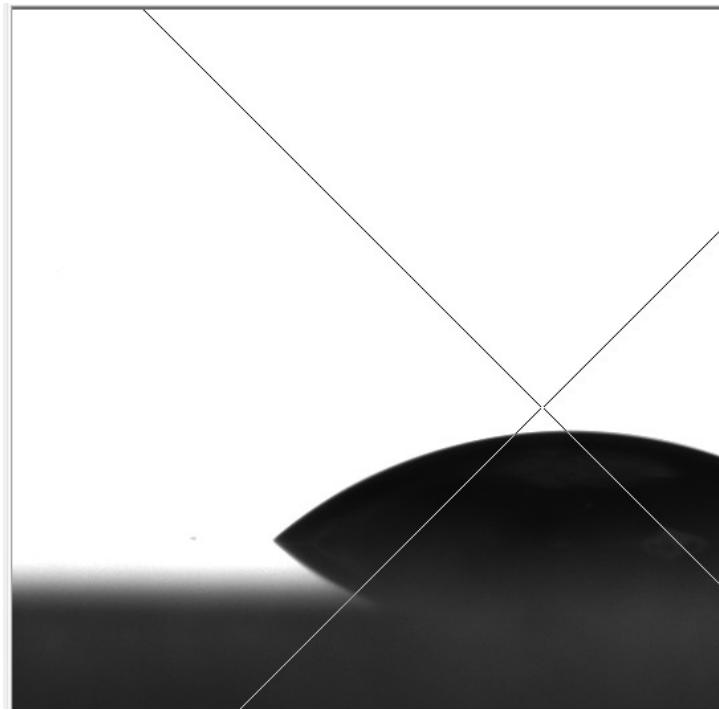


Figure 36 Main screen of goniometry method

Protractor precision: 0.1~0.5, the smaller the value is, the higher the precision is.

Measured angle: Display measuring tape

W: The measuring tape is upward

S: The measuring tape is downward

A: The measuring tape is rightward

D: The measuring tape is leftward

Clockwise rotation<: The measuring tape rotates leftward.

Anticlockwise rotation>: The measuring tape rotates rightward.

Protractor: It displays the measured angle θ and the contact angle is $2 \times (90 - \theta)$.

The measuring tape is displayed when the angle is measured. The measuring tape angle is 45° , and then have the measuring tape meets the drop edge, as shown in Figure 37.

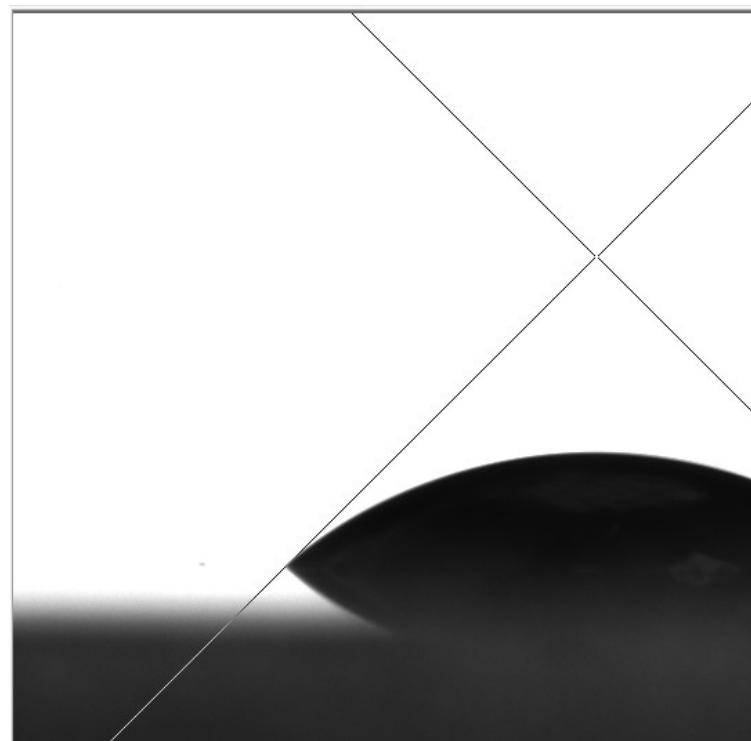


Figure 37 Goniometry method

Then move the measuring tape downward to the top of the drop, as shown in Figure 38.

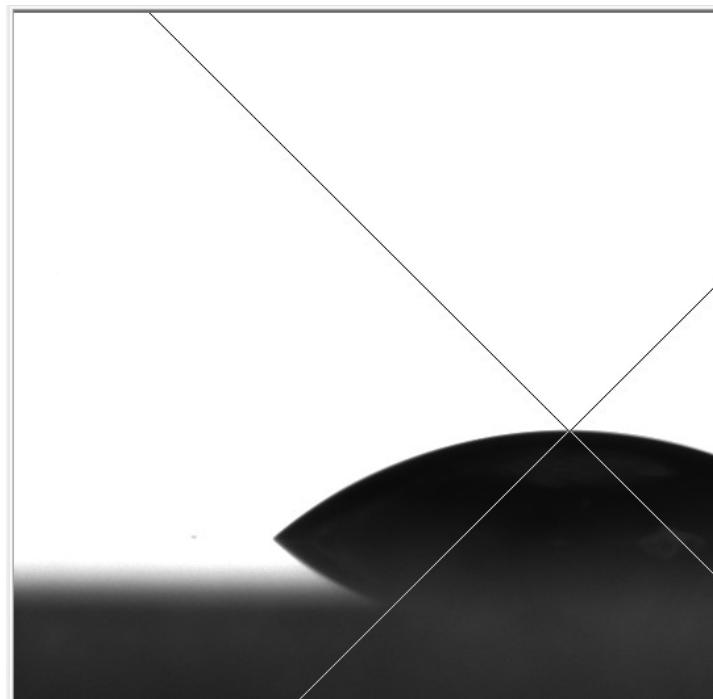


Figure 38 Goniometry method

Then rotate the measuring tape such that it meets left side of the drop to obtain the degree of left contact angle, as shown in Figure 39.

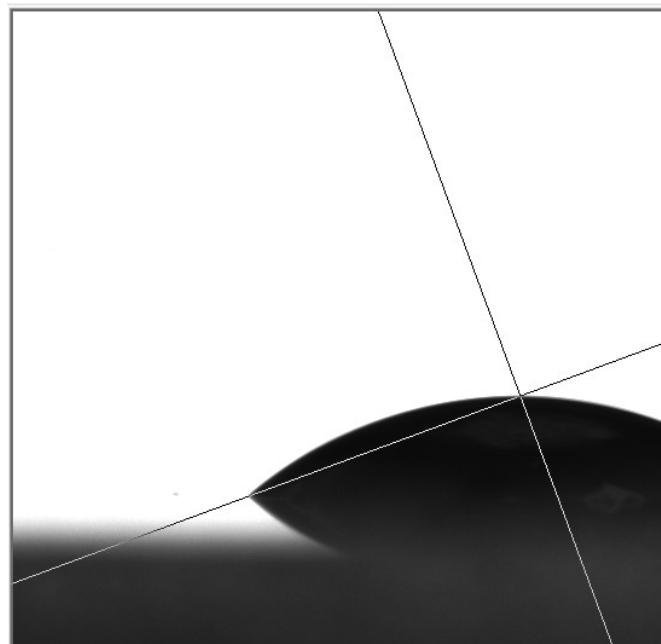


Figure 39 Goniometry method

Click "ok" button next to the left contact angle.

Alternatively, it is possible to have the measuring tape meet right side of the drop to obtain the degree of right contact angle.

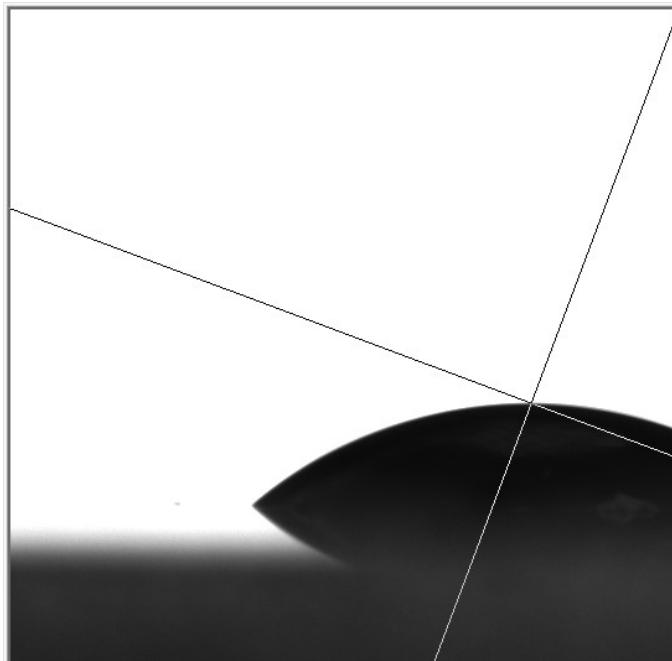


Figure 39 Goniometry method

Click "ok" button next to the right contact angle. Then, average value of the two contact angles will be calculated.

Click "save" button to save the data into database.

This method does not provide dimensioning function.

5.4.3 Height method

Click height method button to enter the main screen for height method, as shown in Figure 40. Click "start" button.

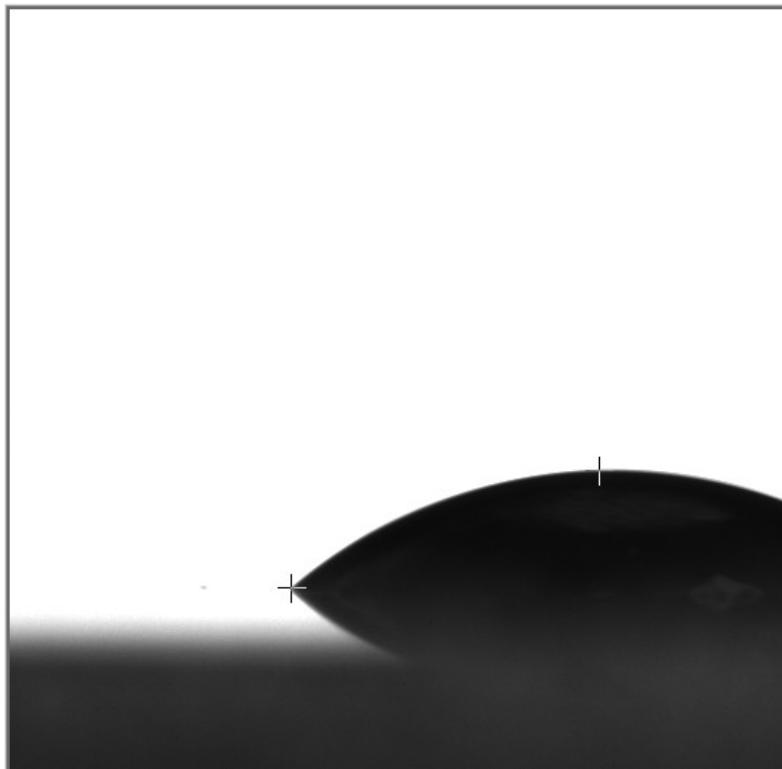


Figure 40 Main screen of height method

Click three phase contact point on left side of the drop, three phase contact point on right side of the drop and top point of the drop (the sequence must be observed) with left button of the mouse, as shown in Figure 41. If a point is wrongly clicked, click right button of the mouse to cancel the selection.

Click "save" to save the data into database.



Click "image mark" button to save the image into another directory and dimension appropriate angles and tangents on the image.

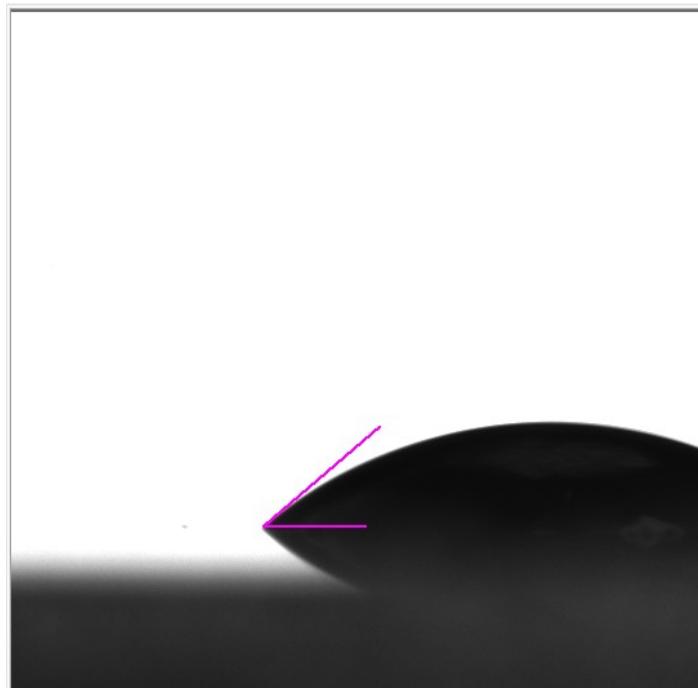


Figure 41 Height method

5.4.4 Image analysis

Click image analysis button to enter the main screen of image analysis, as shown in Figure 42. Click "start" button.

Adjust the baseline position and click "calculate" button to obtain the contact angle.

Baseline 400

Observe whether the fitting curve reasonably fits the drop contour. If so, click "save" button to save the contact angle data into data base.

Click "image mark" button to save the image into another directory and dimension appropriate angles and tangents on the image.

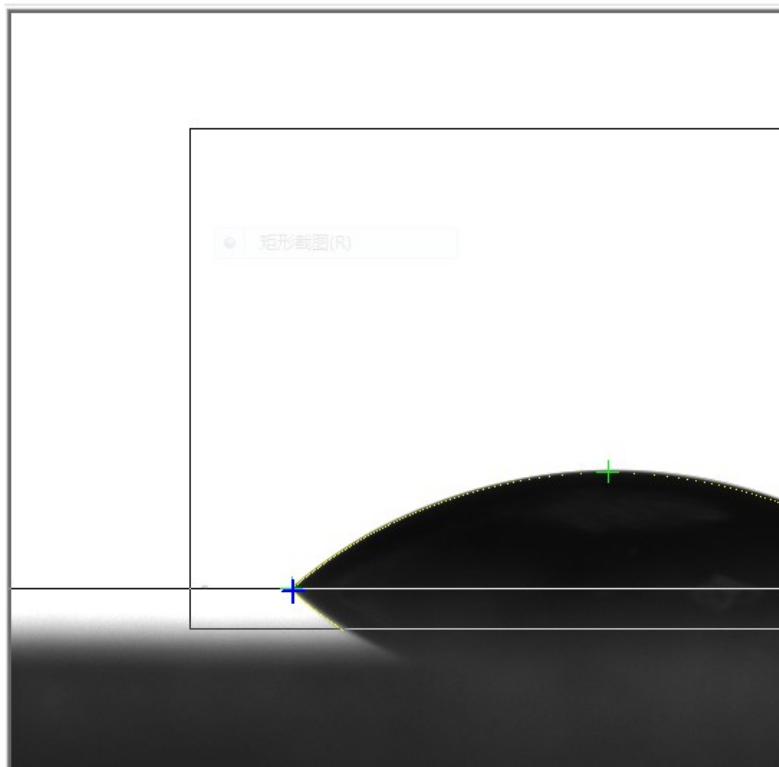


Figure 42 Image analysis

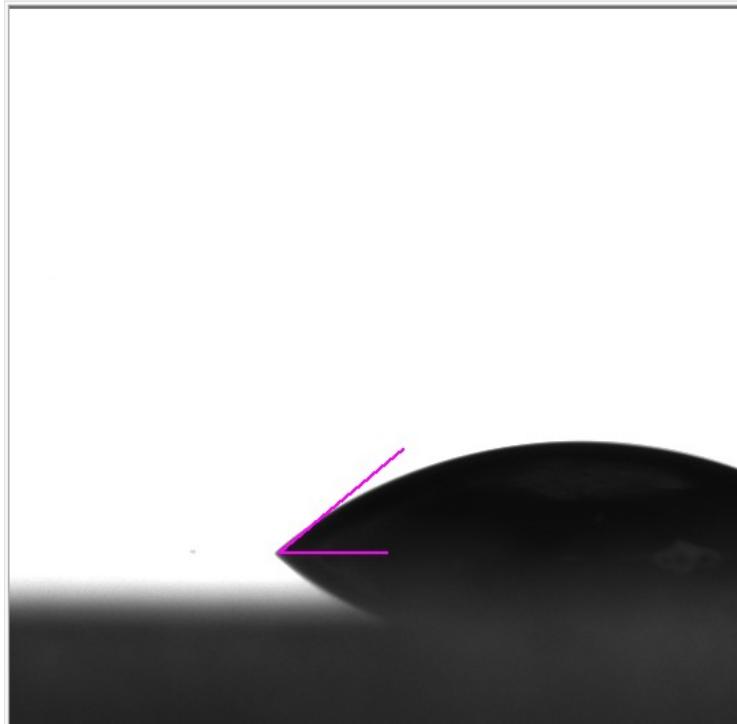


Figure 42 Image analysis

5.4.5 Fitting analysis

This method is used to measure a contact angle on a slope.

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Click fitting button to enter the main screen of fitting method, as shown in Figure 43. Click "start" button.

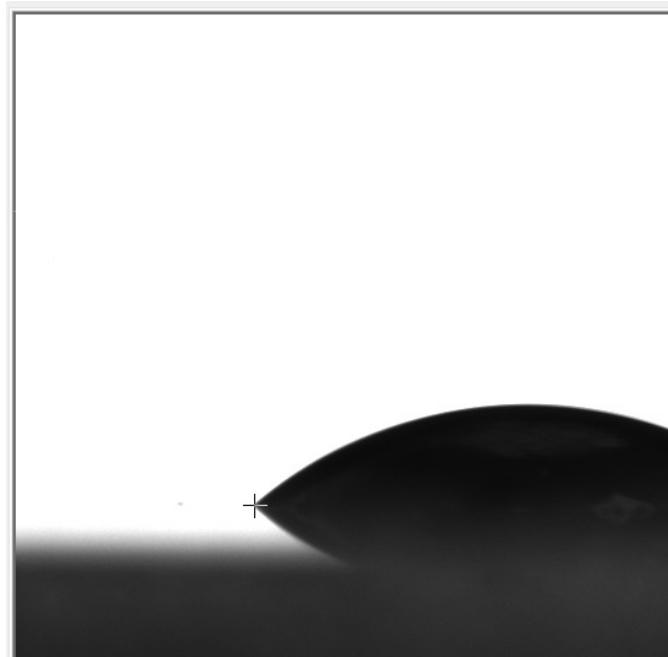
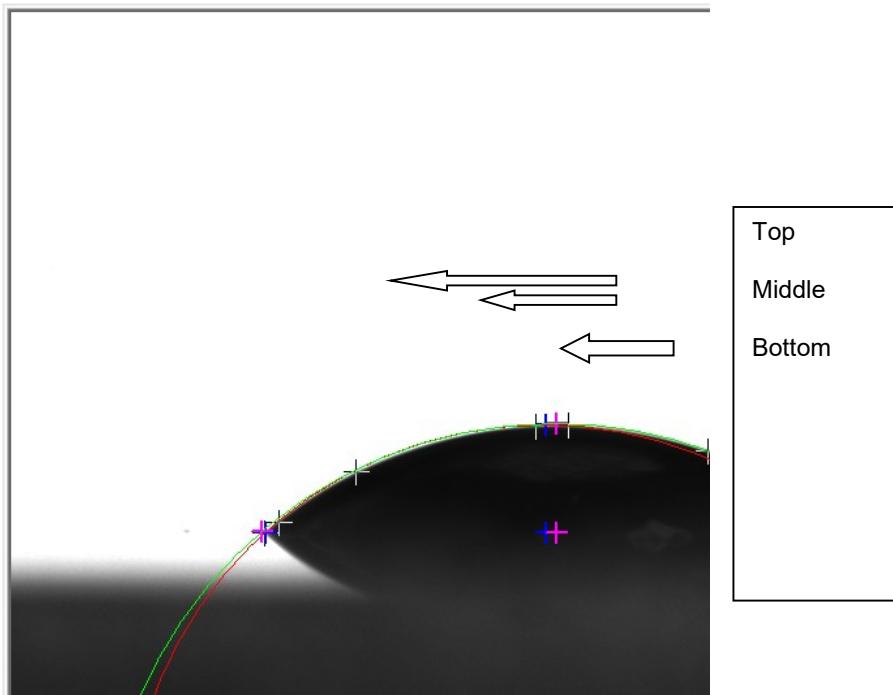


Figure 44 Receding angle fitting analysis

Click three-phase contact points at left and right sides of the drop to draw the baseline, i.e., the interface between liquid and solid.

Figure 44 Receding angle fitting analysis

Depending on nature of the measured contact angle (advancing angle or receding angle), click the bottom, middle point and top point at one side of the drop (these points are selected manually, so keep the click points as close to the drop as possible and once clicked, these points will be marked automatically).



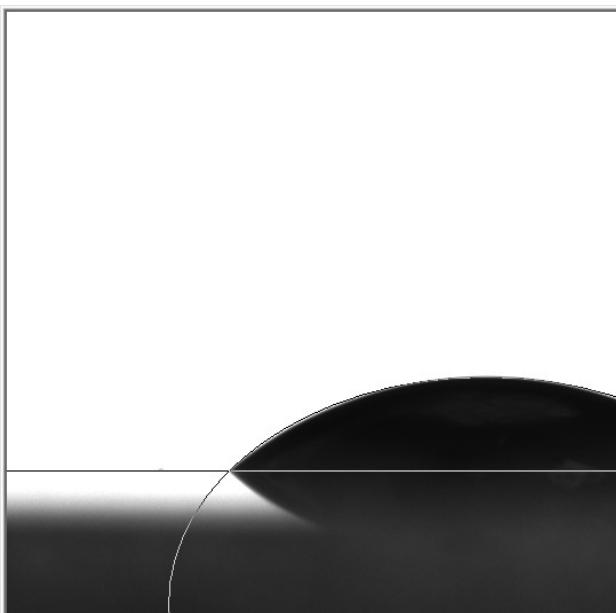
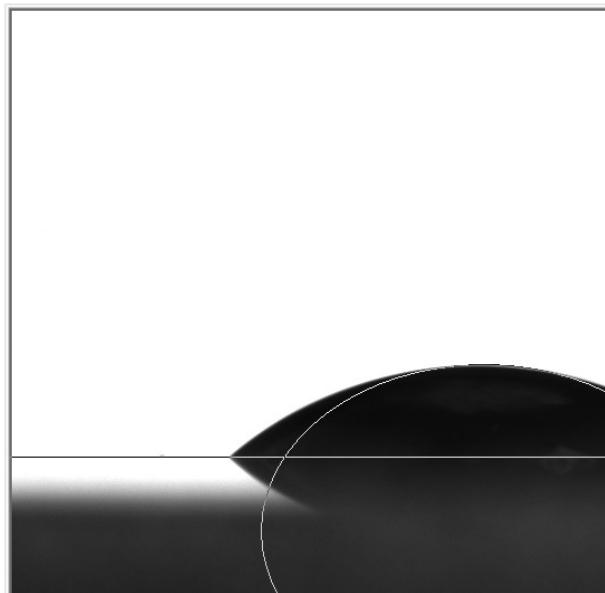
A circle will be automatically fitted with the contact angle calculated. Blue and green points indicate top and bottom points of the circle calculated by the software. If a point is wrongly clicked, click right button of the mouse to cancel the selection.

Observe whether the fitting curve reasonably fits the drop contour. If so, click "save" button to save the contact angle data into data base.

5.4.6 Geometry method

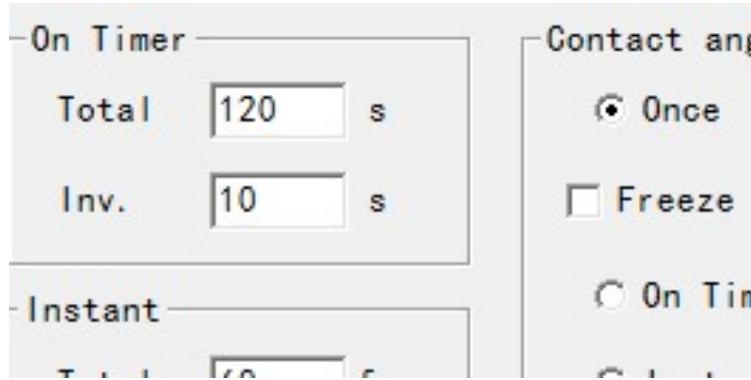
Click geometry method button to enter the main screen of geometry method, as shown in Figure 45. Click "start" button.

Click "up, down, left and right" buttons to adjust the oval position and click "x axis" and "y axis" buttons to adjust the oval size. In this way, the oval is as close to the drop contour as possible. Then, adjust the baseline position. Next, click "calculate" and "save" buttons.



5.4.7 Contact angle analysis mode

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On Timer analysis

"Total" is the total time set for analysis. "Interval" is the time between two analyses. For example, Figure 33 means the analysis is performed at an interval of 10 seconds and a total of 30 seconds is provided for information storage.

After the "on timer analysis" is chosen, keep "On Timer" chosen On Tim.

Click **G**test button and the software will analyze the image according to your setting. If database is set, the result will be stored in the database. A finish dialogue box will pop out after that.

5	1	NULL	0	NULL	2017/1
6	1	NULL	1	NULL	2017/1

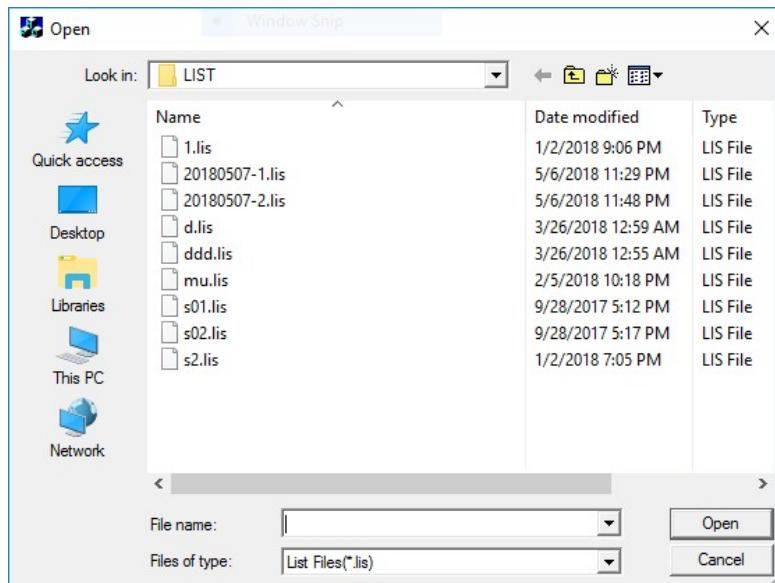
The "Total" in "Instant" (which means total frames for instant analysis) describes the total frames stored. The frame speed is measured at times per seconds. Storage time=total frames/frame speed

On Timer analysis

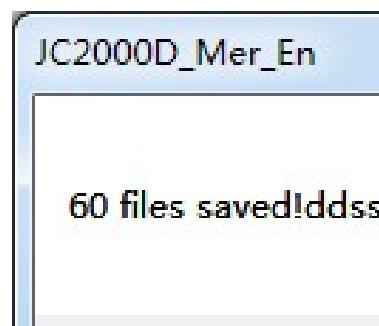
After the "Instant" is chosen, keep "Instant" chosen Instant.

Click **G**test button, a file save dialogue box will pop out. It is required to save List File in D:JC2000\LIST directory.

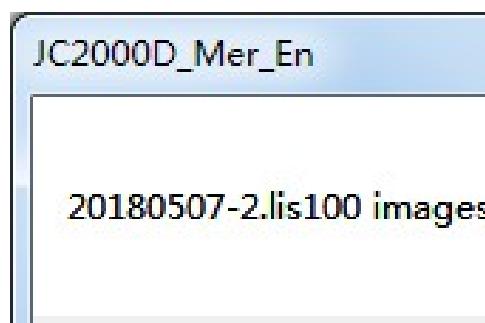
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Input the file name and the software will save images according to setting. A finish dialogue box will pop out after that.



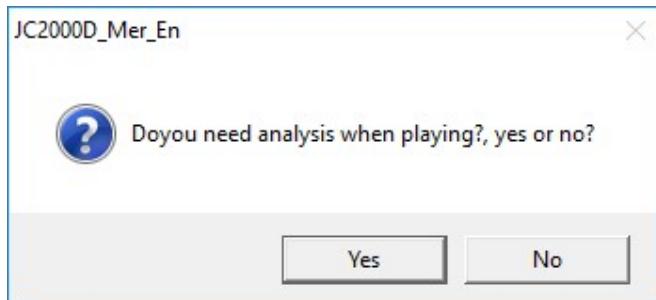
Then, go to file menu and choose "open LIS file" option. Choose the appropriate file.



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After the LIS file is read, button is activated. Click "play" button to select analysis.



Click "yes" button.

The software will automatically analyze the contact angle and display the measured value on the right side list. Click "calculate" button, the computation dialogue box will pop out.

It is possible to calculate the average value from one frame to another.

If "no" is clicked,



drag the slide bar to an image that needs to be analyzed.

Click test button to automatically analyze the contact angle. If database is set, the result will be stored in the database.

5.4.8 Surface tension determined with pendant drop method

In addition, this instrument is also able to determine surface tension with pendant drop method. Basic operations are as below: click "pendant drop method" in the program's main screen to enter the pendant drop method main screen, as shown in Figure 45.

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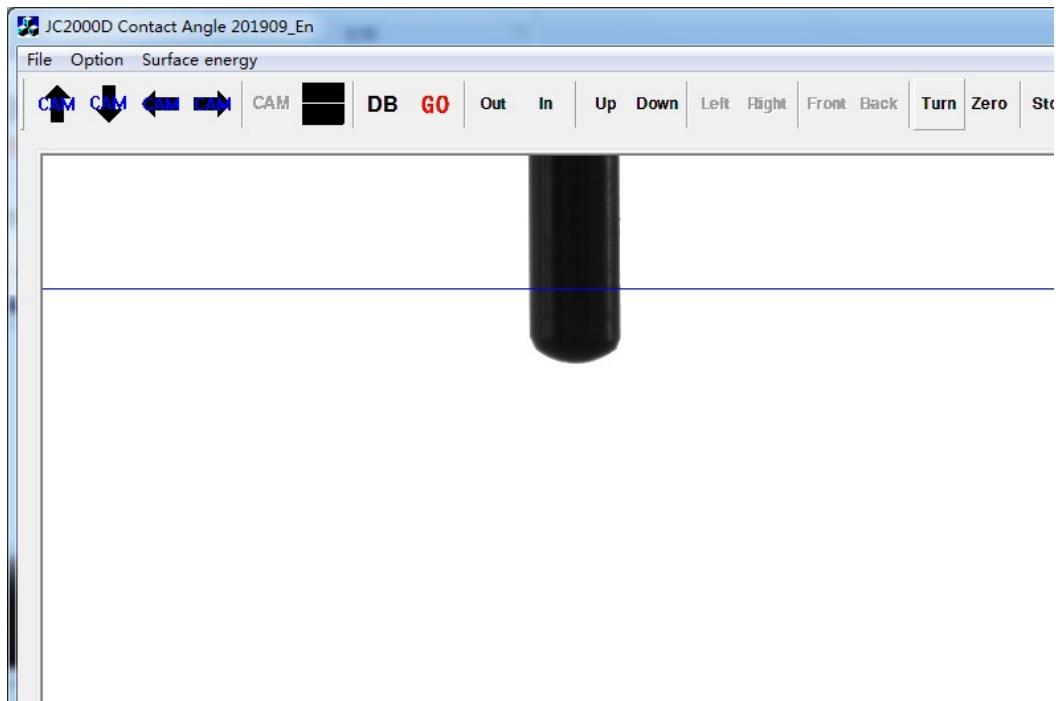


Figure 45 Main screen of determination of surface tension with pendant drop method

Fill out the needle diameter, density difference and other parameters in the main screen. Ensure the blue line is above the needle.



With this button, the user can select to add or create a record after the original record.

In the case of creating a new record, the operator, sample name, test reagent and other necessary information must be completed.

Record	16	User	NULL	S
Count	0	Note	NULL	D

After all information is provided, click record done button. Thus, the subsequent data will be added to the database.

Also, it is possible to add a record by selecting an existing record.

In the case of automatic feeding, the drop volume defined in setting screen must be large enough.

Reagent	_____
Drop V	30

Click **Go** test button such that the drop becomes large and fall off.

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*In the case of manual feeding, the drop must be large enough such that the drop can fall off itself. Surface tension of the pendant drop will be automatically calculated. If database is set, the result will be stored in the database.

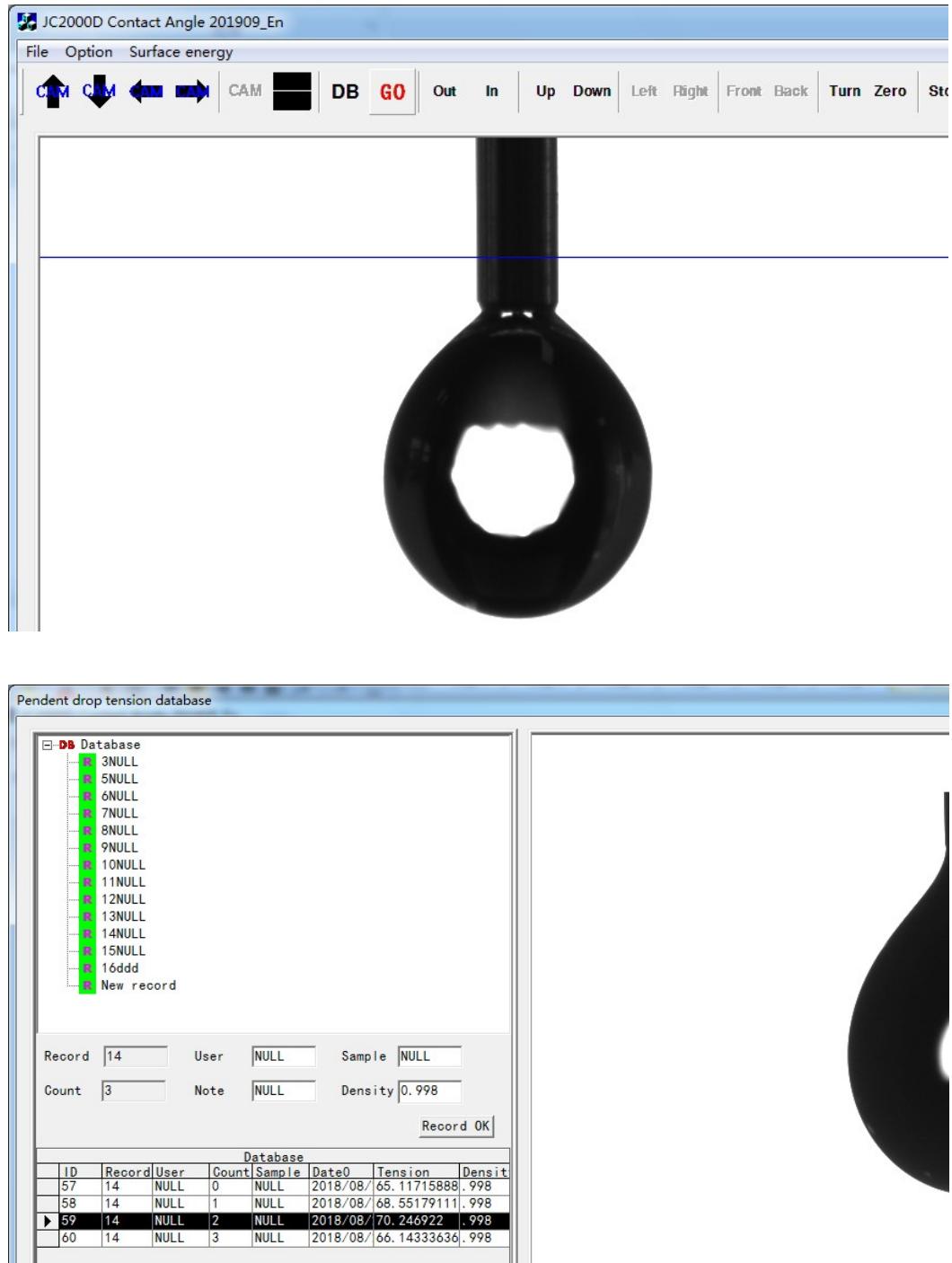


Figure 46 Determination of surface tension with pendant drop method

5.4.9 Surface tension determined with sessile drop method

Click "sessile drop method" in the program's main screen to enter the sessile drop method main screen.

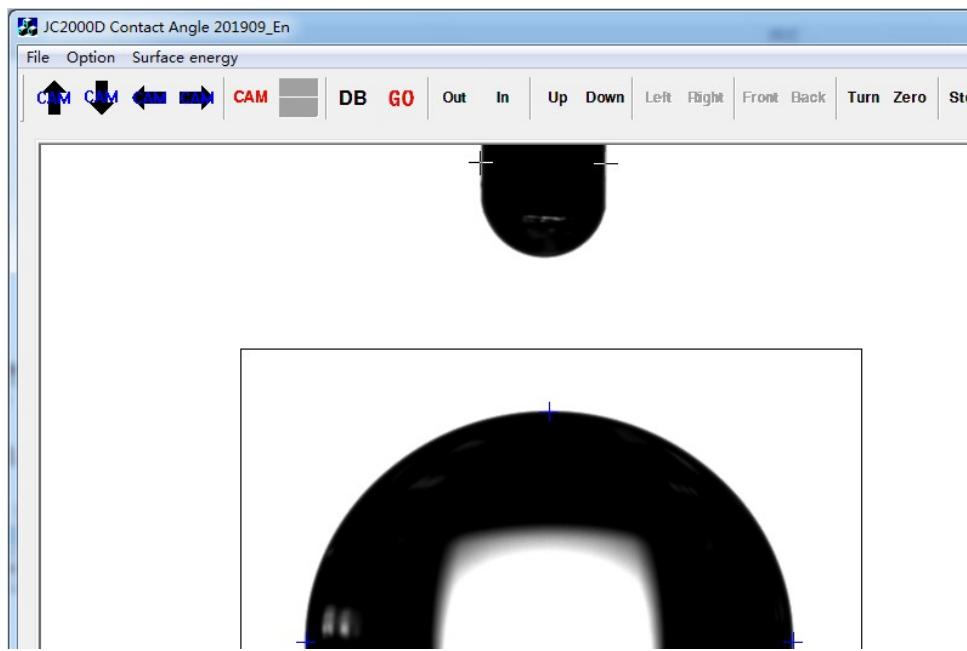
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Place a droplet on the sample and keep a part of the needle (as shown in the figure). Freeze and save the image.

Click the saved image in the menu.

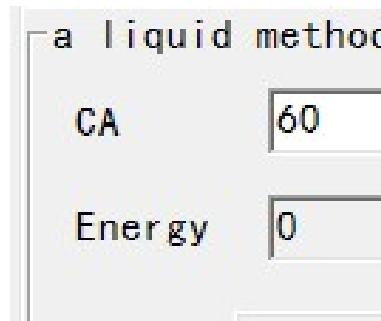
Click one point on left and right sides of the needle, click "calculate the magnification" button.

Then, select the droplet with right button of the mouse. Click "calculate surface tension" button. The surface tension will be calculated.



5.4.10 Surface energy calculation with polar liquid method

Drop a water droplet on a glass surface, measure the contact angle and fill out the angle. This method applies to contact angles which are less than 90 degrees.



5.4.11 Surface energy calculation with Owens method

This instrument also provides the function of calculating surface energy with Owens method, indicated as below:

$$\gamma_s = \gamma_s^D + \gamma_s^P$$

$$\gamma_L = \gamma_L^D + \gamma_L^P$$

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Where, γ_s is the solid surface energy, which can be divided into dispersion force term γ_s^D and polar force term γ_s^P ; γ_L the liquid surface energy, which can also be divided into dispersion force term γ_L^D and polar force term γ_L^P .

$$\text{Then: } \gamma_L(1+\cos\theta) = 2(\gamma_s^D\gamma_L^D)^{1/2} + 2(\gamma_s^P\gamma_L^P)^{1/2} \quad (12)$$

In formula (12), if the liquid surface energy γ_L and its two terms γ_L^D and γ_L^P are known, and the contact angle between the liquid and the solid, there are still two unknown terms γ_s^D and γ_s^P in the formula. To obtain these two unknown terms, two equations must be used. Thus, it is required to use two kinds of liquids, leading to the following set of equations:

$$\gamma_{L1}(1+\cos\theta_1) = 2(\gamma_s^D\gamma_{L1}^D)^{1/2} + 2(\gamma_s^P\gamma_{L1}^P)^{1/2}$$

$$\gamma_{L2}(1+\cos\theta_2) = 2(\gamma_s^D\gamma_{L2}^D)^{1/2} + 2(\gamma_s^P\gamma_{L2}^P)^{1/2}$$

γ_s^D and γ_s^P can be obtained from the set of equations, which, in turn, leads to surface energy of the solid: $\gamma_s = \gamma_s^D + \gamma_s^P$.

Surface energies of testing liquids commonly used are listed in Table 1.

Table 1 Surface energy of common testing liquid

Liquid	γ_L^P	γ_L^D	γ_L	γ_L^P/γ_L^D	Polarity
Water	51	21.8	72.8	2.36	Polar
Glycerol	26.4	37	63.4	0.71	
Formamide	18.7	39.5	58.2	0.47	
Diiodomethane	2.3	48.5	50.8	0.05	Non-polar
α -Bromonaphthalene	0	44.6	44.6	0	
Hexadecane	0	27.6	27.6	0	

For surface energy calculation with Ownes method, the two testing liquids must satisfy the following conditions:

- (1) γ_L^P/γ_L^D of each liquid must be away from 1 as much as possible.
- (2) One liquid must be polar and the other non-polar.
- (3) The testing liquid may not dissolve, expand or deform surface of the liquid.

In calculation of surface energy, dispersion forces, polar forces and contact angles of liquid A and liquid B must be filled into the screen as shown in Figure 48. Then click "calculate" to obtain surface energy of the solid.

two liquid method

Dispersion force	Polar force	
Liquid A	21.8	51
Liquid B	48.5	2.3
Static	0	0

Figure 48 Dialogue box for surface energy calculation

5.4.12 Surface energy calculation with EOS method

Fill out surface tension of the testing liquid and contact angle formed by the liquid and the solid to calculate surface energy of the solid.

EOS

Tension	72.6
CA	108.7
Energy	0

5.4.13 Solid-liquid interfacial tension calculation

With surface energy of the solid calculated with the above method, and fill surface tension of the liquid and contact angle formed by the liquid and the solid to calculate solid-liquid interfacial tension.

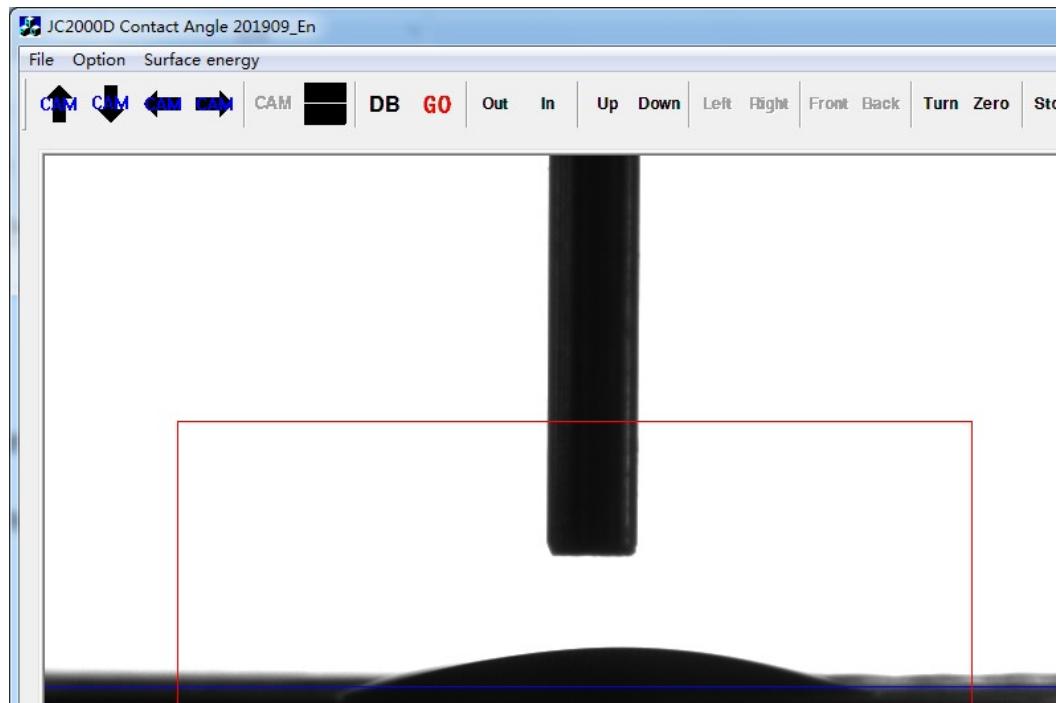
Solid-liquid interface tension

Energy (Solid-gas)	Tension (Liquid-gas)
0 mN/m	0 mN/m

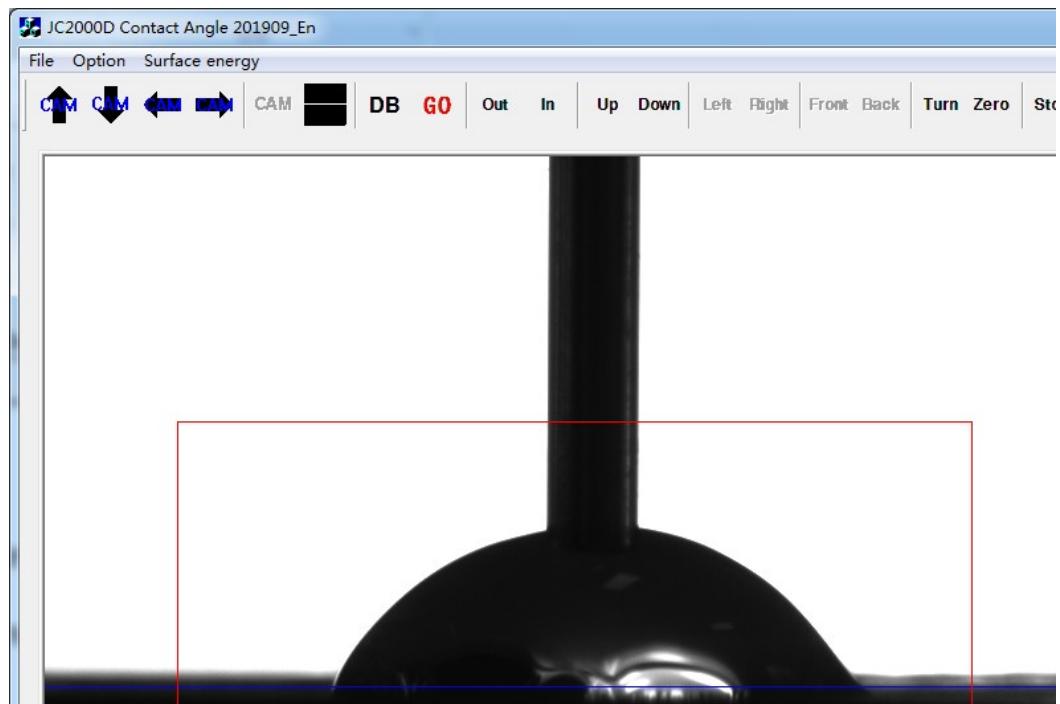
5.4.14 Advancing angle and receding angle

Select the testing option to advancing angle page, and click "go" button to feed sample.

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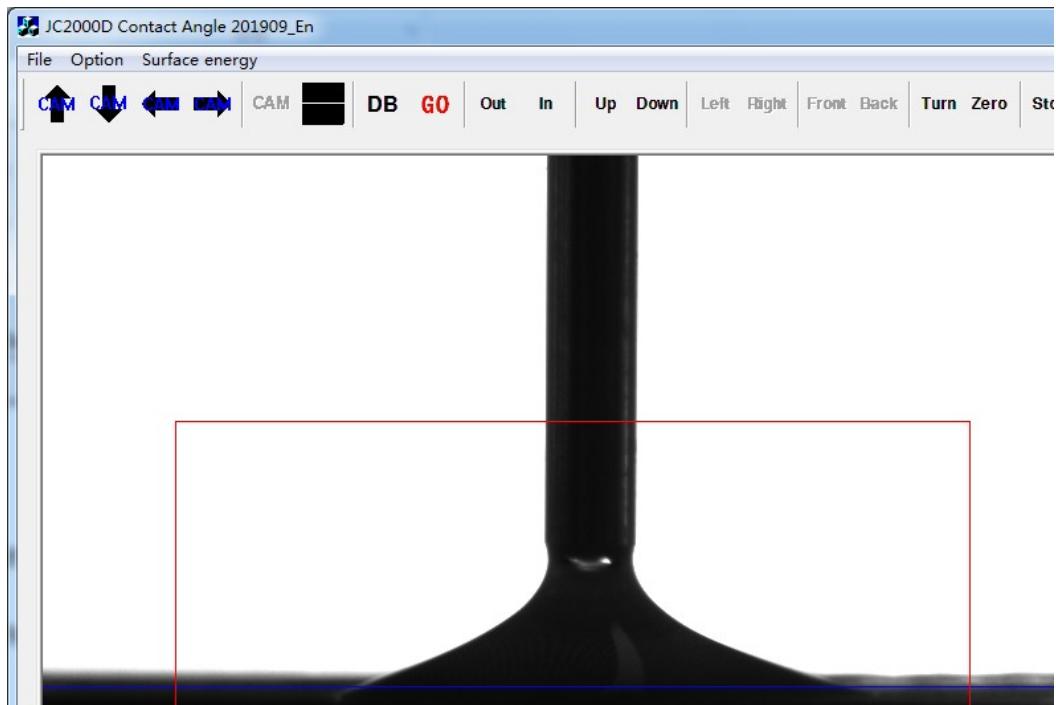


If the drop size needs to be reduced, click "receding angle" button, the motor will automatically pump excessive liquid out.

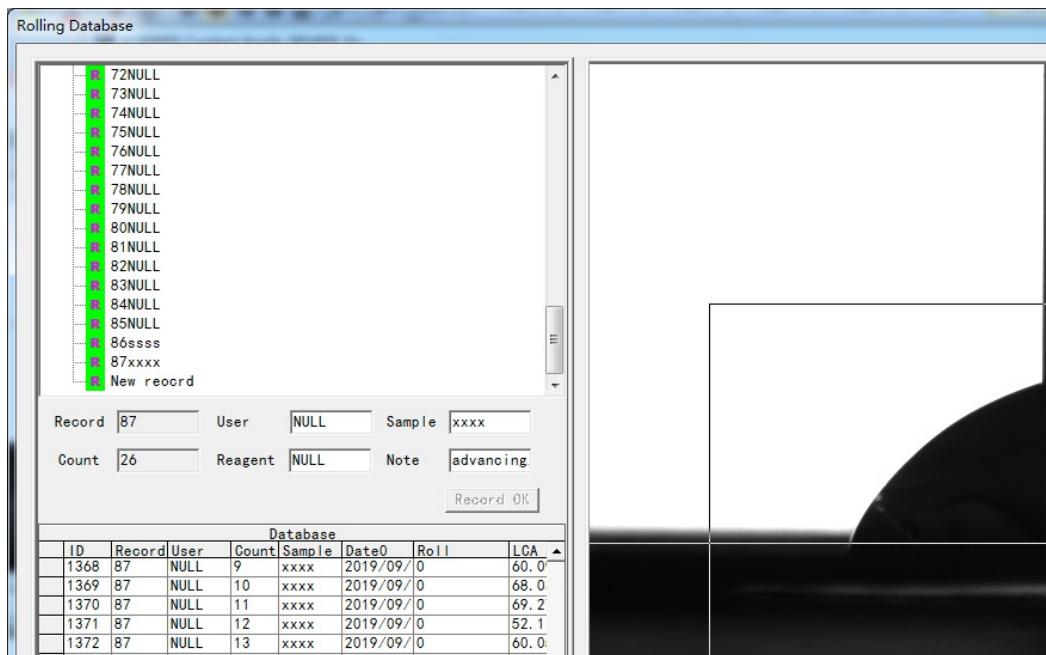


Click "advancing/receding angle stop" to end the testing.

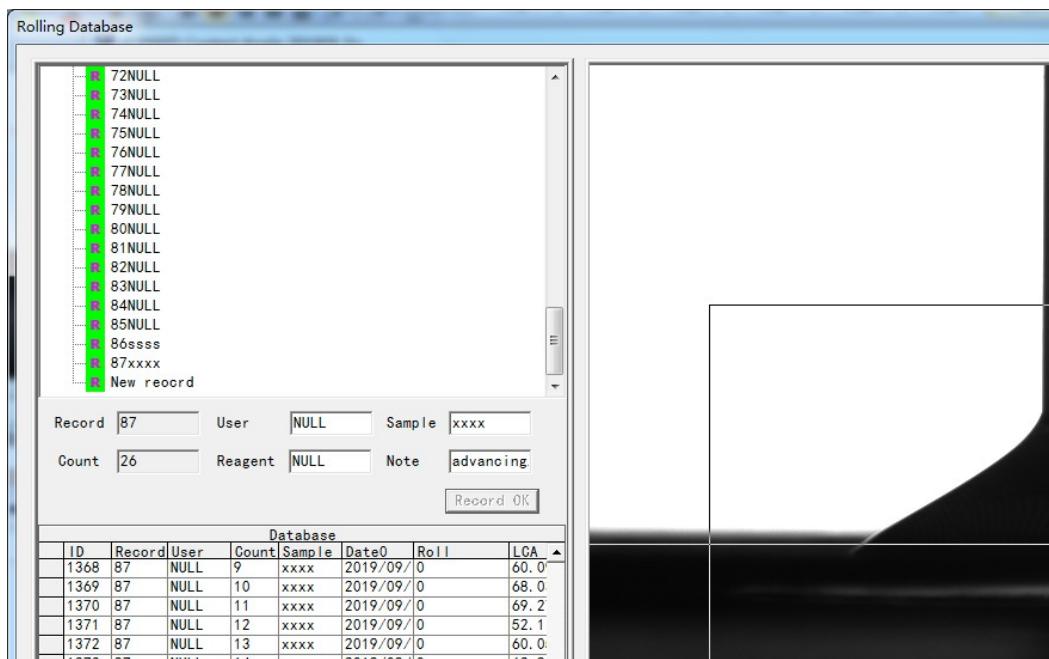
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Access to data base page for the testing result.



Advancing angle



Receding angle

Appendix 1:

Contact angles of some systems and surface tensions of some liquids

Some useful data about contact angles from the literature are listed in Table 2. Contact angles formed between solids that cannot be easily melted, solids that are polar, and polymers with low surface energy, and some liquids are included. Due to influence from different factors, values of contact angles reported from different sources vary from one another. For this, a reference range is provided for each contact angle. Also, some liquid-solid-liquid interfacial contact angles are also provided.

Table 2 Contact angles of some systems (advancing angles) (20-25°C)

Liquid (γ , $\times 10^{-3}$ J/m 2)	Solid	θ (degrees)	$d\theta/dT$ (degrees/K)	Liquid (γ , $\times 10^{-3}$ J/m 2)	Solid	θ (degrees)	$d\theta/dT$ (degrees/K)
Mercury (484)	Poly(tetra-fluoroethylene)	150		CH ₂ I ₂ (50,8)	Poly(tetra-fluoroethylene)	85	
	Glass	128-148				60-61	
	N-hexatriacontane	111			Paraffin	53	
	Paraffin	110			Steatite	40 (single crystal)~46	
	Poly(tetra-fluoroethylene)	98-112			Polyethylene		
	Tetrafluoroacetone	108	-0.05		Formamide		
Water (72)		78-103	-0.02	(58)	Tetrafluoroethylene-hexafluoropropylene	92	-0.06
						75	-0.01

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hylene-hexafluoropropylene copolymer	75-90	-0.11~ -0.01	CS ₂ (about 35)	copolymer	35	0.35
Polypropylene	88		Benzene (28)	Polyethylene	46	
Polyethylene	84	-0.13		Ice (about -10°C)	42	
Polyethylene	86				0	
	72-82			Poly(tetra-fluoroethylene)	43	
Human skin	80		n-propanol (23)		22	
Naphthalene (single crystal)	66			N-hexatriacontane		-0.12~
	40			Paraffin	32-40	-0.11
Sb ₂ S ₃	17		n-decane (23)			
Stibnite	Regarded as 0			Poly(tetra-fluoroethylene)	26-30	-0.12
Graphite	0		n-octane (21.6)			
Carbon ^a	0			Paraffin		
Stearic acid ^b	0					
Gold	0					
Platinum	40-90					
Silver iodide						
Glass						
SiO ₂						
TiO ₂						
SnO ₂						
Steel						

Note: a Carbons prepared with different methods may have different contact angles.

b Langmuir-Blodgett membrane deposited on copper.

Table 3 Contact angles of some liquid A-solid-liquid B interfaces

Solid	Liquid A	Liquid B	θ
Sb ₂ S ₃ (Stibnite)	Water	Benzene	130
Al ₂ O ₃	Water	Benzene	22
Poly(tetra-fluoroethylene)	Water	n-decane	about 180
	Benzenemethanol	Water	30
Polyethylene	Water	n-decane	about 180
	Paraffin oil	Water	30
Mercury (unknown natural oil)	Water	Benzene	about 100
	Mercury	Gallium	about 0
Glass			

Table 4 γ of some liquids (20°C)

Liquid	γ (mN•m ⁻¹)

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	Liquid-vapor	Water-liquid
Water	72.75	
Octane	21.69	51.68
Dodecane	25.44	52.90
Hexane	27.46	53.77
Benzene	28.88	35.0
Carbon tetrachloride	26.77	45.0
Octanol	27.53	8.5
Butanol	24.6	1.6
Aniline	42.9	5.9
Ether	17.0	10.7
Ethyl acetate	23.9	~3
Mercury	484	426