

**INSTRUCTION MANUAL  
FOR  
CONTACT ANGLE METER**

**MODEL CAM-PLUS  
MICRO/FILM**

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**CAM-PLUS MICRO OPERATING MANUAL  
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## I. Introduction and Theory

Contact angle measurements of liquid droplets on substrate surfaces are used to characterize surface wettability, surface cleanliness and the hydrophylic/phobic nature of the surface. The contact angle is defined as the angle between the substrate support surface and the tangent line at the point of contact of the liquid droplet with the substrate (Fig. 1). A liquid drop is placed on a solid substrate and the contact angle has a single value for smooth homogeneous substrate surfaces.

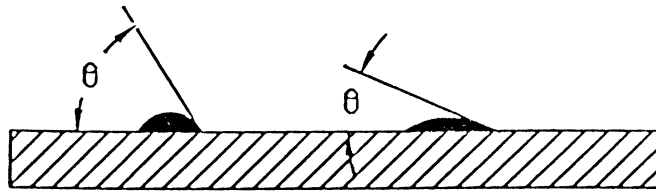


Figure 1

This value is related by Young's equation to the surface energy of the substrate. The value of the contact angle of the liquid droplet will depend upon the surface energy of the substrate and the surface tension of the liquid. If complete wetting takes place between the liquid and the substrate surface, the droplet will spread out over the substrate and the contact angle will approach zero, whereas if wetting is only partial, the resulting contact angle will lie in the range of 0 to 180 degrees. Glass and metals are examples of high energy surfaces over which most liquids spread spontaneously, with the angle tending to zero. In contrast, plastic materials are typical low energy surfaces such that liquids placed on these surfaces remain in the form of drops having finite contact angles so long as surface energy of the substrate is less than the surface tension of the liquid.

The actual time for a droplet to reach equilibrium with the surface depends upon the liquid being used. For a water droplet, the equilibrium is reached in seconds. For oil droplets, the equilibrium is reached in approximately three minutes. Contact angle readings should be taken at these time intervals. Prior to equilibrium the angle is advancing. Once equilibrium has been reached the angle starts to recede.

Solid surfaces may not be completely homogeneous so that their surface energies are not evenly distributed. Therefore, the measurement of contact angles on solid surfaces should be taken at several points on the tested surface. For these surfaces the mean value of the contact angle (i.e. ten droplets) of the particular surface should be used to characterize the surface.

### Half-Angle™ Measuring Method.

This method (U.S. Patent No. 5,268,733) for measuring contact angles allows you to take direct angle measurements, yet eliminates errors associated with arbitrary tangential alignment of a hairline to the droplet image.

Tantec's Half-Angle method is based on the formula for determining contact angles from the droplet dimensions:

$$\theta = 2 * \text{arc tan} (H/R), \quad \text{where}$$

$\theta$  is contact angle, H is the height of a droplet, and R is the radius of droplet's base (see Fig. 2).

It becomes apparent from this formula that the angle between a line connecting the contact point C with the apex A of the droplet, and the base line CO itself, is a half of the contact angle. This half-angle,  $\theta/2$  can be easily and unequivocally determined using a protractor by aligning the hairline to connect points A and C.

The dial of the protractor is then calibrated to display the contact angle value which is equal to the doubled value of the angle between lines AC and OC, or  $2 \times \theta$ .

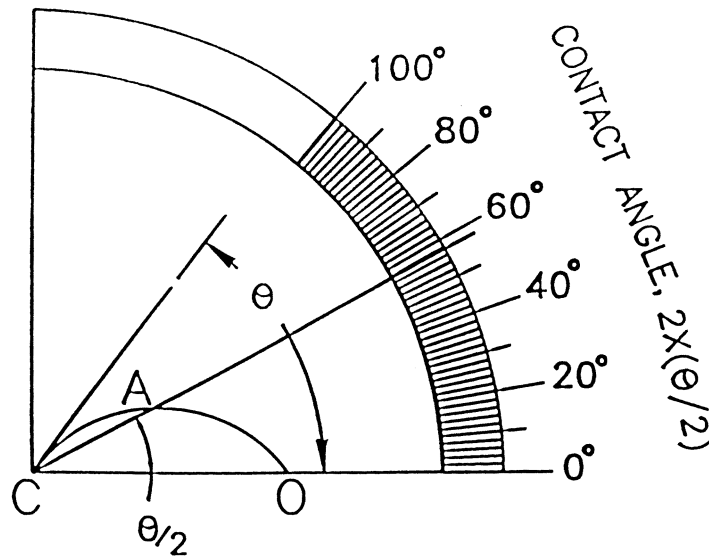


Fig. 2

Even though the above formula is derived for determining acute contact angles on flat substrates, its use can be extended to calculating slightly obtuse contact angles. It can also be used to measure contact angles on cylindrical surfaces, as long as the diameter of the cylinder is larger than the droplet size. The contact angle is formed by the tangent line, which is located in the vertical plane crossing the axis of the cylindrical surface.

## II. PRODUCT DESCRIPTION

Model CAM-PLUS MICRO is designed to measure contact angles on very small areas of a variety of surfaces, flat, curved and cylindrical, such as small-diameter tubing. It is important however, that the measured surfaces are smooth and homogeneous.

This instrument is a bench-top optical device incorporating a projection screen with a protractor readout calibrated in two-degree increments. The image can be independently adjusted along both the horizontal and vertical axis and focused on the screen with an axial micrometer adjustment. A precision micrometer-based syringe accurately dispenses multiple droplets without recharging.

A droplet of testing liquid is placed on the tested surface by bringing the surface into contact with a droplet suspended from a needle of the syringe.

This instrument utilizes a projection screen design vs. microscope-based designs. A “silhouette” image of the droplet is projected on the screen and the angle is measured. The use of the projection screen makes it possible for more than one person to observe the droplet, which cannot be done with the instruments using an ocular tube. It is good for educational and discussion purposes, and helps to detect operator errors through easy supervision.

### Specifications:

- \* Measured angle range 2 to 180 degrees
- \* Substrate surface - smooth, homogeneous
- \* Droplet size - adjustable, 0.5 microliter minimum
- \* Microsyringe capacity – 75 droplets
- \* Microsyringe size std – glass syringe with 22 gauge stainless steel needles  
Optional - 15, 20, 22, 27, 30 stainless steel or Teflon-coated needles
- \* Droplet magnification - 6:1 standard  
12:1 optional (for small surface areas)
- \* Specimen holder size - 2” x 2” x 1” deep
- \* Smallest tested surface - 0.007”
- \* Accuracy - standard deviation 0.8 degrees

The contact angle meter CAM-PLUS MICRO is shown in Fig. 3 (next page).

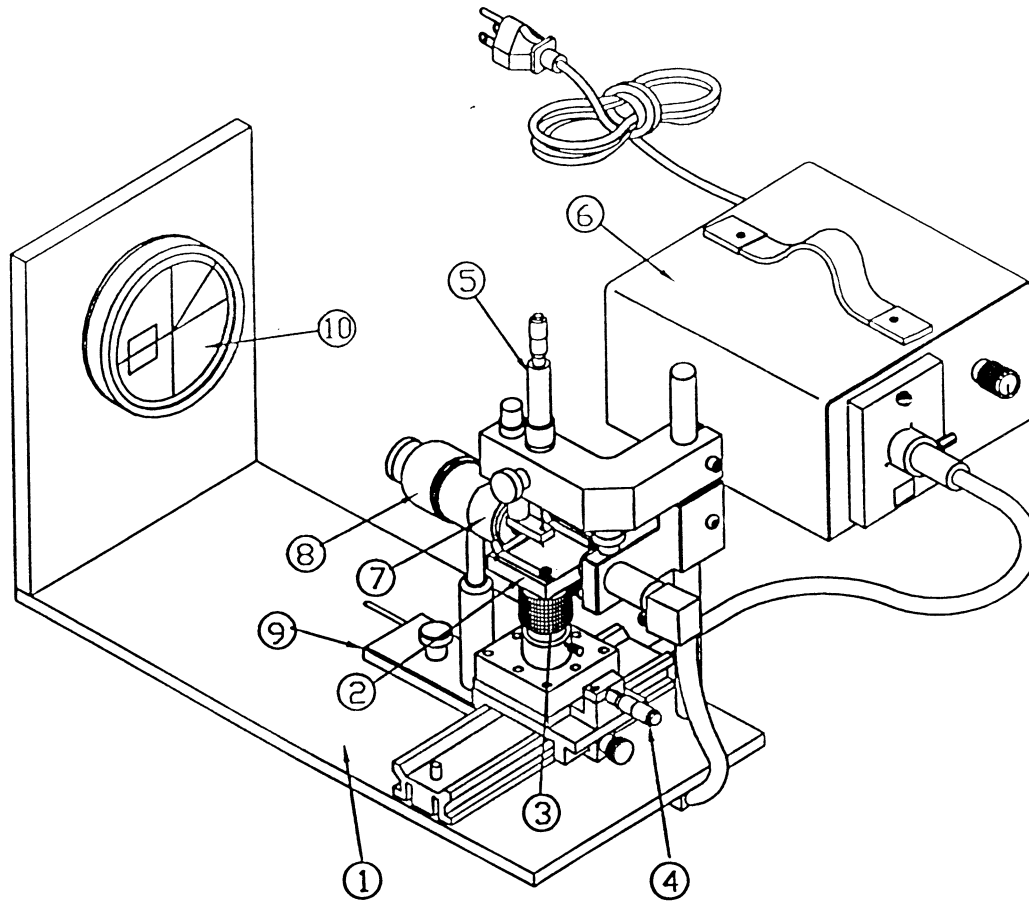


Fig. 3

It has an elongated horizontal platform (1) supported on four rubber feet. The tested part is placed on a specimen holder (2). The height of the specimen holder can be adjusted by rotating the knurled knob (3). The specimen holder has an axial micrometer adjustment (4) for image focusing with 20mm of travel within the focal range of the optics

The micrometer syringe (5) for applying droplets onto the tested surface is held in place with a set screw with a large knurled knob. The distance between the tip and the surface can be adjusted by moving the syringe up or down within the holder and locking it in place with a holding set screw.

A fiber optic illuminator (6) is routed near end of the platform. It produces a columniated horizontal light beam across the tested surface and through the projection lens (7) and through an inversion prism (8) located on the opposite side of the specimen holder. The lens position can be adjusted by moving the plate (9). The image of the droplet is projected onto the circular screen (10) located at the other end of the platform. The screen is mounted on a movable plate allowing for horizontal adjustment. A protractor with a calibrated scale and a dial are mounted on the screen.

### III. ASSEMBLY INSTRUCTIONS

- 1) Remove and inspect contents of the shipment.

The following items have been shipped in this container:

No. of Pcs.	Part No.	Description
1	25000XX	Contact Angle Meter CAM-PLUS MICRO
1	9990099	Instruction Manual
1	2000066	Fiber Optic Power Supply, 110V
1	2000067	Fiber Optic Cable with Right Angle Connection
1	1850172D	Needle, blunt 22 Ga., 1"

- 2) Plug the fiber optic cable into the power supply (cylindrical larger end). Tighten the setscrew to secure it into place.
- 3) Plug the power supply into 120 VAC/60Hz (or specified voltage) wall outlet.
- 4) Assemble the micrometer syringe assembly as outlined on page 15.
- 5) Rotate the power supply intensity knob clockwise in order to begin measuring.
- 6) If you are planning to evaluate small-diameter tubing, mount the two spring clamps to the specimen holder using the two plastic screws.

#### IV. OPERATING INSTRUCTIONS

1. To recharge the syringe assembly, follow the steps as outlined below:
  - a. Rotate the micrometer until it is all the way out.
  - b. Remove syringe holder from unit.
  - c. Remove syringe from holder.
  - d. Immerse needle tip in water (or test liquid) and fully depress plunger – allow spring to retract plunger.
  - e. Hold syringe so the needle points up. Put syringe back in holder while still pointing needle up. After the syringe is in the holder, if no liquid has come out, rotate micrometer head in until all air is removed – remove excess liquid.
  - f. Replace holder in unit and ensure the bottom of the needle is at the top of the grid and in focus.

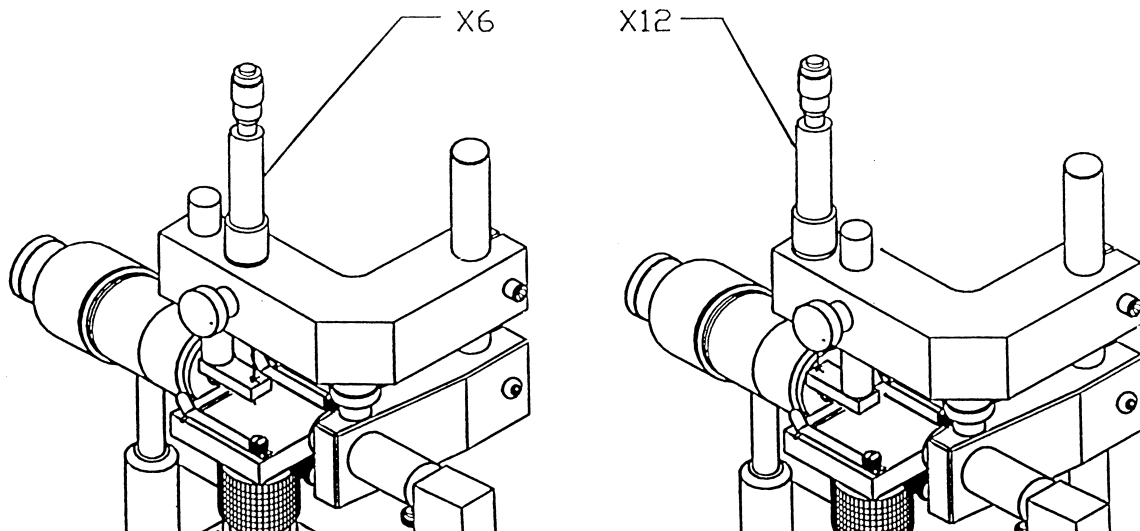


Figure 4



2. Plug the fiber optic cable into the power supply; tighten the holding lock knob in place. Plug the input cable of the power supply into the correct input voltage. Slowly turn the intensity (potentiometer up clockwise rotation) up to  $\frac{3}{4}$  power. You will now see the projected image on the screen.

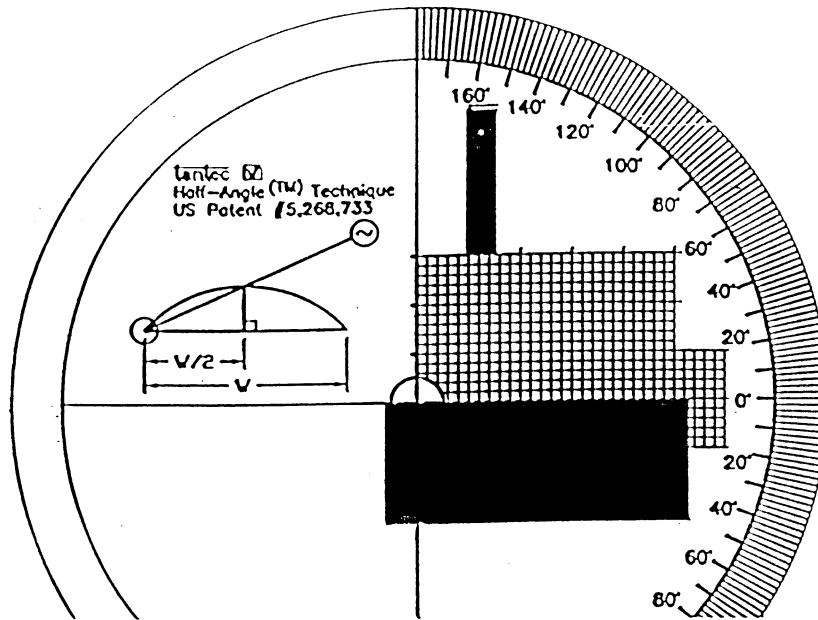


Figure 5

3. Move the sample stage to the “load” position – this is to the far left when looking at the screen (Figure 5).
4. Place a sample onto the specimen holder under the syringe needle. If the sample is a tube, place it into the groove in the specimen holder and secure it with spring clamps. Move the sample stage to the “measure” position.
5. Locate the tested surface precisely under the tip of the syringe needle. If front/back adjustments are required, then adjust the micrometer assembly. Left/right adjustments can be done via the slide on the rail or by repositioning the part on the specimen table.
6. Rotate the knob of the micrometer syringe clockwise to release the desired amount of the testing liquid. Observe the droplet on the screen. The recommended droplet diameter is 10 divisions on the screen. If the sample is a tube, the droplet diameter should not exceed the radius of the measured tubing.

## MICRO STAGE

- Bring the specimen holder up until the droplet touches the surface (Figure 6). Lower the specimen holder to complete transfer of the droplet. Wait for the droplet to stabilize. Make sure that the footprint of the droplet is round on a flat specimen and round or slightly oval on a tube.

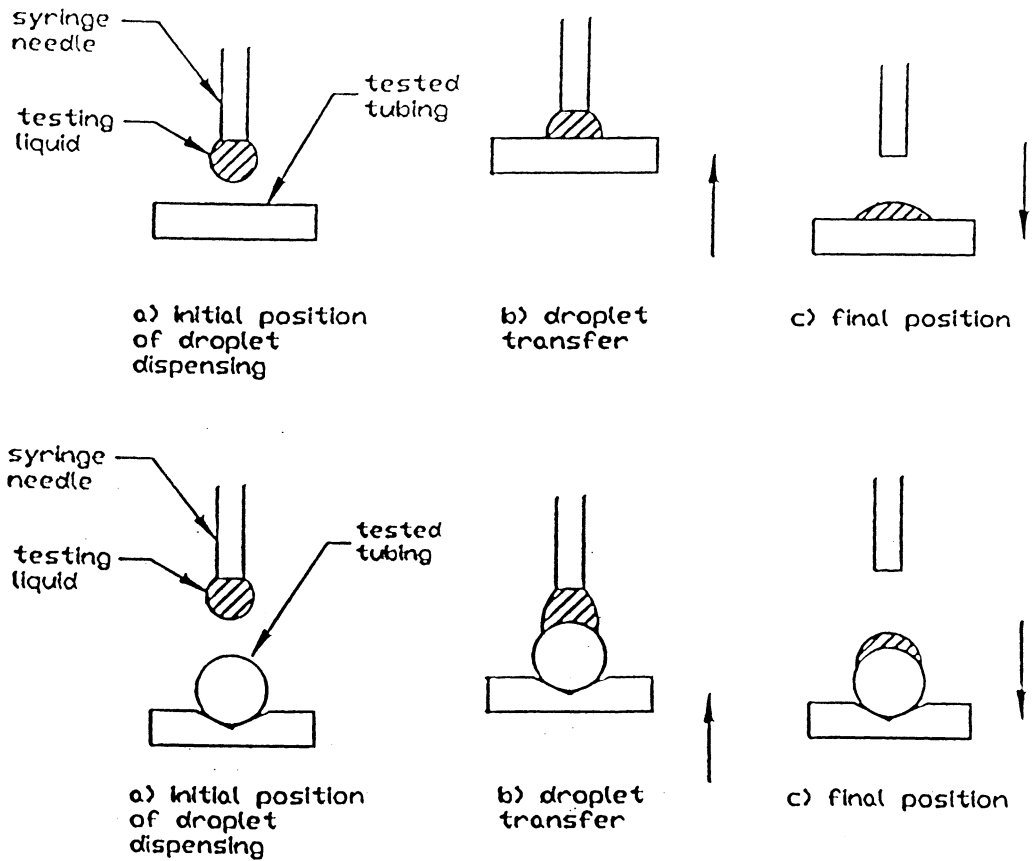


Figure 6

## FILM STAGE

### Changing Sample Strip Loading Procedure

1. To load a sample strip, pull the rollers straight outward and down to lock in open position. Place long end of the strip between the back roller and the plate. Place the other end between the front roller and the plate. Release (close) both rollers. Take up slack by rotating the knob forward (Figure 8).

Note: For rigid materials and thick films (over 10 mils) prepare a 3/8" x 1-1/2" sample. Place a sample along the flat portion of the specimen holder. Move the sample left or right to take several measurements.

2. Move the sample stage to the "measure" position.

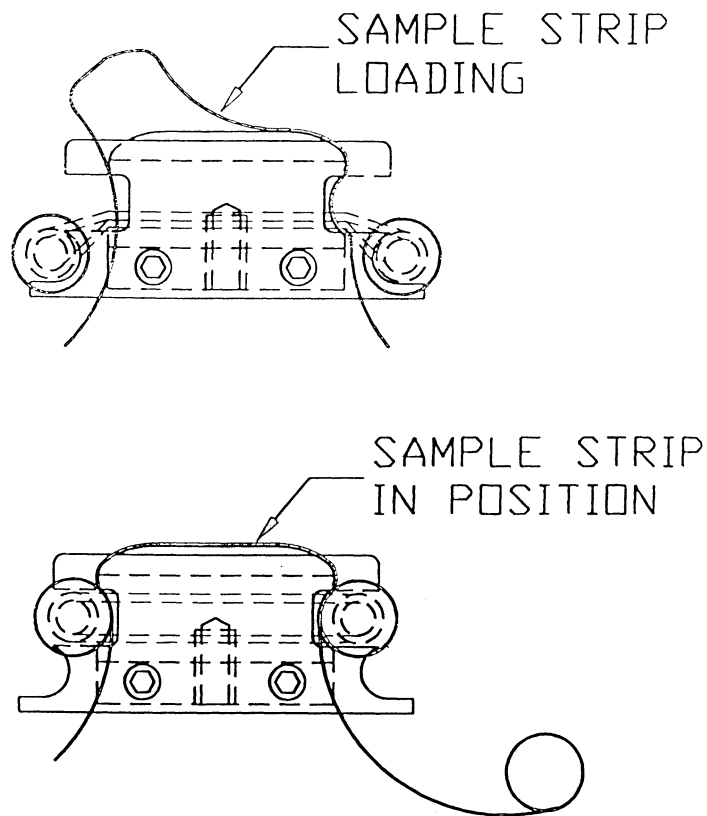


Figure 8

### Lens Attachments

To change from one lens magnification to the other, simply unscrew the projection lens assembly of one (rotate counter-clockwise) and replace it with the other.

8. Using the specimen holder axial micrometer adjustment, bring the image into focus if necessary.
9. Using the knurled vertical adjustment knob, adjust the height of the tested surface so that the image is aligned with the horizontal cross-line.
10. Slide the round measuring screen on the vertical back panel of the meter left/right until the vertical cross-line (Fig. 7) touches the left edge of the droplet's image.
11. Determine the coordinates of the droplet's apex by dividing the width of the base of the droplet's image by two and finding the apex of the intersection of the corresponding vertical line with the edge of the image.

For example, in Figure 7, the width of the droplet's base is 13 divisions. Therefore, the apex is located at the intersection of the vertical gridline corresponding to 6.5 divisions and the outer border of the image.

12. Rotate the clear protractor with a hairline on the measuring screen until the hairline crosses the apex.
13. Read the contact angle on the scale.

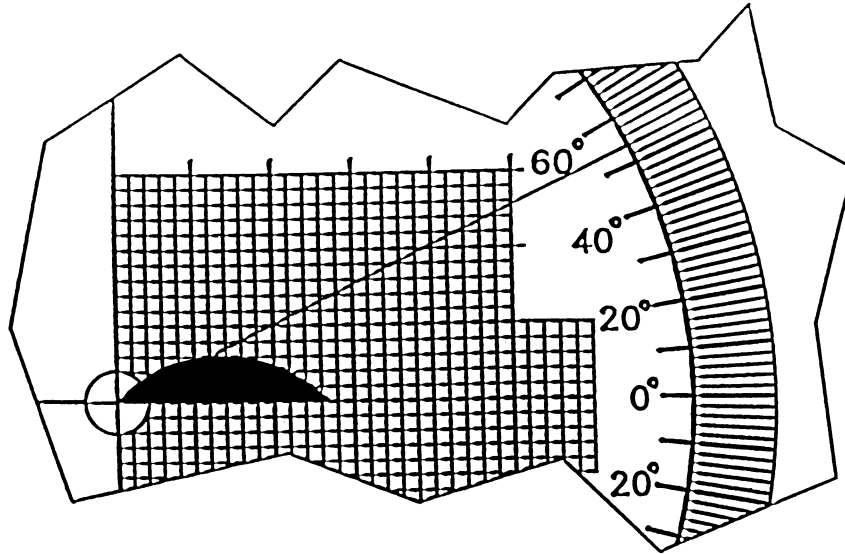


Figure 7

### **Changing Magnification of the CAM-PLUS MICRO Series**

In order to change the magnification of the CAM-PLUS MICRO from 6 to 12 or vice versa, two elements need to be in place, the syringe location and the focusing lens.

#### **Syringe Location**

As Figure 8 below points out, the X6 location of the syringe is closer to the operator side while the X12 magnification is closer to the screen.

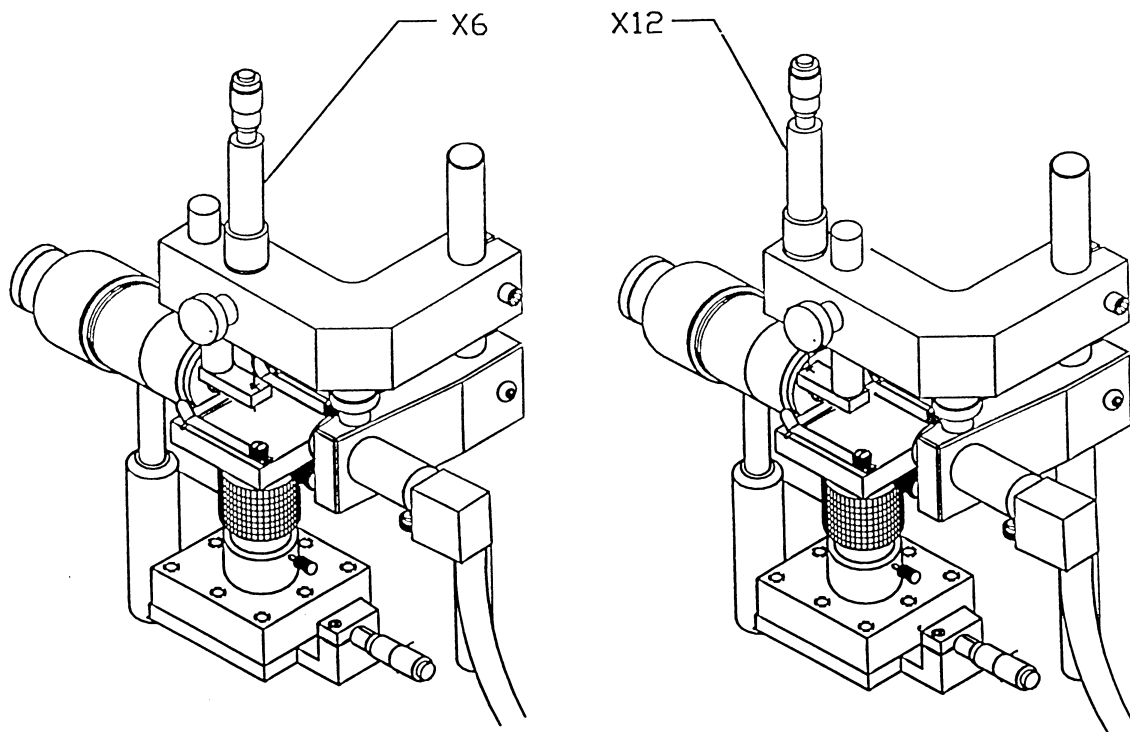


Figure 8

#### **Lens Attachments**

To change from one lens magnification to the other, simply unscrew the projection lens assembly of one (rotate counter-clockwise) and replace it with the other.

### **DETERMINING CONTACT ANGLE MEASUREMENTS OF APPROX. LESS THAN**

The contact angle of a water droplet using the Half-Angle™ technique can also be determined by calculating the ARCTANGENT of the height: radius ratio according to the following:

$$\text{Contact Angle} = 2 \times \text{ARCTAN} (H/R)$$

where: H = height of the droplet

R = radius of the droplet's base

Practically, this is accomplished in the following manner:

- A) Measure the height of the droplet (=H)  
(Units can be either cm/inches/mm or divisions)
- B) Measure the radius of the droplet's base (=R)  
(units can be either cm/inches/mm or divisions)
- C) Calculate H/R rounding this value to three digits
- D) Take the ARCTANGENT of the H/R ratio
- E) Multiply the result by 2

This will give you the contact angle.

## V. LIGHT SOURCE MAINTENANCE PROCEDURES

The CAM-PLUS MICRO uses a fiber optic illuminator which has been engineered to give a cool, intense white light source for easy measurements and to make sure the sample itself is affected as little as possible.

The light source provides up to 150 W of light from a tungsten halogen lamp. A variable 5-21 VAC controls the output intensity. A removable lamp assembly contains the lamp holder, which contains a type DDL lamp. Cooling the entire assembly is a high output fan with a specially designed heat fin assembly.

### A. Cleaning

Clean the finish of the enclosure as needed with any commercial glass cleaner. DO NOT use detergents, harsh spray or chemicals. Use any cleaning solution sparingly being careful not to let the fluid spill into the interior of the enclosure. If a spill does incur, unplug the line chord and let the power supply sufficiently dry before using again.

### B. Lamp Replacement

1. Turn the power OFF
2. Remove the fiber optic component
3. Allow the lamp to cool
4. Loosen the captive thumbscrew until the head springs forward
5. Pull the top of the lamp assembly forward and lift the unit out of the recess. CAUTION! Lamp surfaces may be hot! Do not pull the assembly beyond the length of the cable and connector assembly.
6. Push down on the lamp holder release lever to raise the lamp
7. Gently unplug the ceramic socket
8. Insert a new bulb into the lamp socket. BE CAREFUL NOT TO TOUCH THE BULB ON THE INSIDE OF THE REFLECTOR. FINGERPRINTS MAY AFFECT THE LAMP OUTPUT!  
(Newark P/N 96F4330, phone: (630) 317-1000 or call WIKO lamps)
9. Push the ceramic socket on the two lamp pins until flush with the reflector.

### C. To Remove Lamp Assembly

1. Make sure switch is OFF
2. Loosen the captive thumbscrew
3. Pull lamp assembly out of the housing
4. Reach into the opening and depress the release tab on the connector
5. Slide the connector apart and remove the lamp assembly

### D. To Replace the Lamp Assembly

1. Slide the connector into the mating receptacle until the release tab clicks into place
2. Place the assembly into the recess with the 2-bottom "ears" riding over the bottom edge of the recess first.
3. Slide the assembly into place and depress the captive thumbscrew while turning clockwise. TIGHTEN FIRMLY!

## LAMP ASSEMBLY

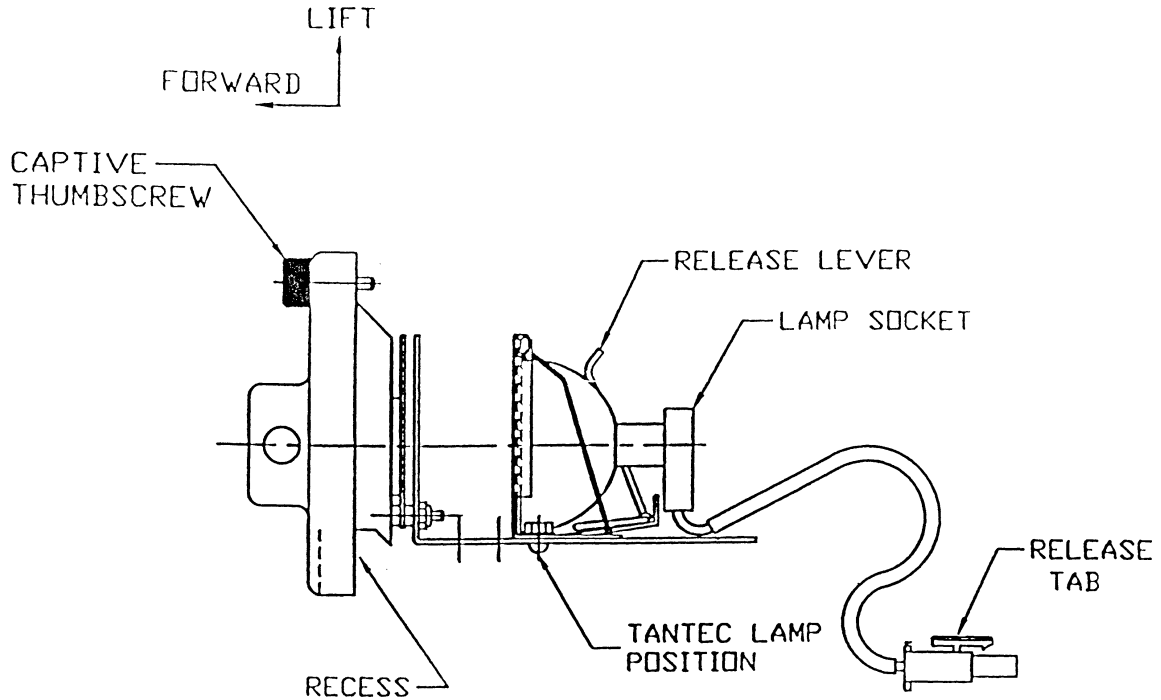


Figure 9

### **E. Troubleshooting**

If Fan operates but no light, check following:

1. Turn the intensity control knob completely clockwise
2. Change the bulb
3. Examine lamp socket for damage. Check continuity of wiring. If contacts are blackened or if the ceramic base cracked or the wiring are frayed, replace the bulb.
4. Make sure the two-pin modular connector is properly engaged in the locking position. Check the contacts.

### **F. If neither the Lamp nor the Fan Operates**

1. Make sure the power chord is inserted completely into the IEC connector on the rear panel and also into the correct power source.
2. Check the power chord for damage
3. Check the fuse
4. If the unit feels warm to the touch, check for any air obstruction to air intake or exhaust area. The light source has an automatic reset thermal cutout device as a safety precaution. Let the unit cool down for 10 minutes and turn it back ON.

**DO NOT ATTEMPT TO REPAIR THE UNIT ON YOUR OWN! CALL TANTEC FOR AUTHORIZED REPAIRS!**



**VI. SPARE PARTS AND ACCESSORIES FOR MODEL CAM-PLUS MICRO**

Part No.	Description
2400012	X12 Projection Lens Assembly
2400013	X6 Projection Lens Assembly
1850172	Micro Droplet/Syringe Assembly
1850141	Glass Syringe Assembly
1850179	Needle, Blunt, 22Ga. 1" Lg.
2000066	Fiber Optic Power Supply (for 230V use P/N 2000070)
2000068	Replacement Lamp (Newark P/N 96F4330)
2400117	Tubing Holder Kit (optional)
8800051	Static Eliminator Package (optional)
2400007	Recently Advanced – Recently Receded Angle Measuring Package- includes: (5) small 30 Ga. Needles, Adv./Recede. Measuring screen

**VII. MICROSYPHINGE/DROPLET REGULATOR SPECIFICATIONS**

1. Assemble the glass syringe as shown in the drawing below:

**ASSEMBLY OF GLASS SYRINGE**

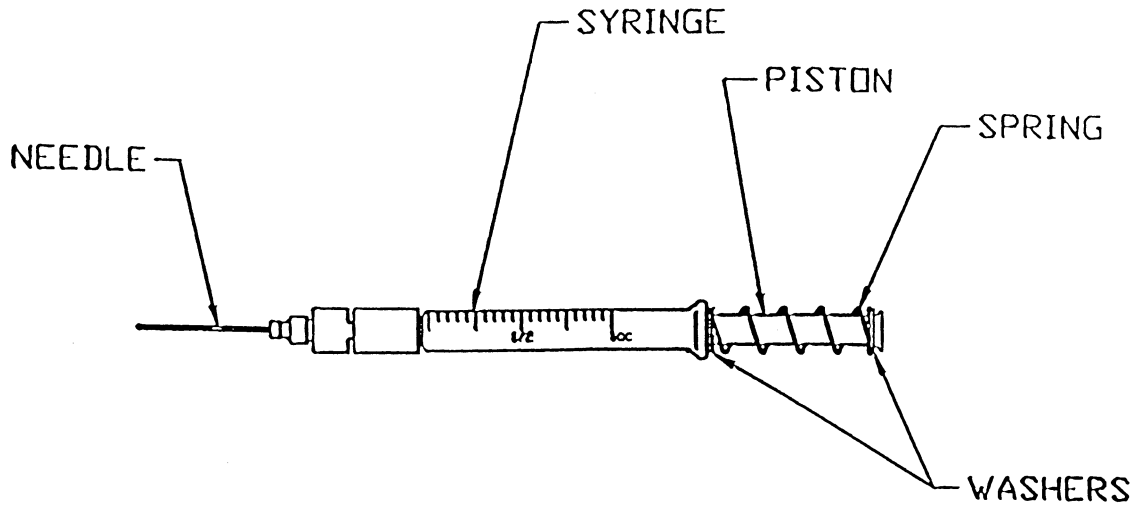


Figure 10

2. Affix the needle to the hub, turn the needle clockwise until it is locked into position
3. Check that the piston can move smoothly after the assembly of the micro syringe assembly.
4. Depress the piston completely, inserting the needle into the liquid slowly. Pull enough liquid into the syringe until the spring is extended.

If bubbles are sucked up into the syringe, point needle upwards depressing the piston so as to get rid of the air bubbles.

5. Assemble the droplet regulator assembly as shown below (See assembly in Figure 11 below).
6. Turn the micrometer and adjust the scale until it is nearly completely retracted. Now set the droplet regulator (syringe) assembly into the CAM-PLUS MICRO.

### **ASSEMBLY OF DROPLET REGULATOR**

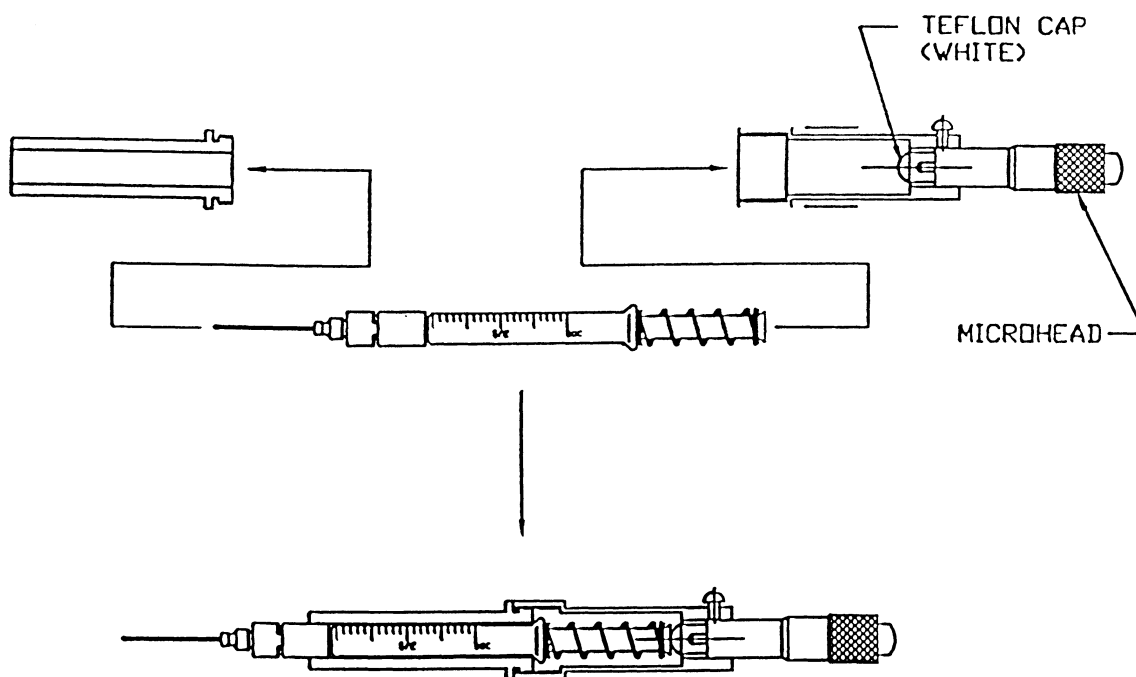


Figure 11

NOTE: To clean remaining fluids in the syringe, depress the plunger until the liquid has been squirted out. Refill 2-3 times with solvent cleaner or alcohol, squirting out the cleaner in-between.

## VIII. LIQUID SAMPLES & SOLID SAMPLES

### Liquid Samples:

#### 1. Proper Use of Needles:

Except for the standard stainless needle of 22G (gauge) several different needle sizes are available as an option. Use them according to the characteristics of liquid samples, which will be used to measure the contact angle.

a) Proper selection of the needle size depends on the test liquids' surface tension:

The liquid sample of low surface tension will wet the stainless needle well. Sometimes, the liquid can climb the needle and not form the droplet correctly. In that case a Teflon™ needle in smaller gauge size is required.

Example: Ethanol with a surface tension of (22.3 mN/m @ 20° C) - use an 18 gauge Teflon needle.

b) Proper needle selection depends on the liquid viscosity:

A highly viscous liquid is difficult to control as it is pushed out from the needle. Use a smaller gauge needle.

Example: Glycerin with a viscosity of (1412 mPas @ - 20° C) - use a 18 gauge stainless

c) Proper needle selection depends on the contact angle to be measured:

In the case of a high contact angle, measurements depositing the droplet from needle onto the solid sample will be difficult. Use a large gauge Teflon™ needle in this case.

Example: Water/Teflon interface yields a contact angle of about (108°) - use a 28 gauge Teflon needle.

d) Proper needle selection depends on the droplet volume:

In the case of very low contact angles the droplet end points will be off the scale. Use a larger gauge needle (28 gauge) for controlling small volumes.

#### 2. Proper Use of Syringes:

The standard syringe attached is a glass syringe with a Luer-lock stainless steel needle.

a) Proper syringe material selection depends on the characteristics of the liquid:

Acidic or alkaline liquid samples will corrode the materials of metals or glass.

b) Proper syringe material selection depends on the water solubility of the sample:

The liquid samples, which are not water-soluble like inks, oil and fats are difficult to clean up. Extra syringes should be purchased for these kinds of liquids.

### **Solid Samples:**

Proper care and precaution should be taken when making contact angle measurements on a variety of substrates. The following needs to be considered:

1. Surface conditions:

Irregular surface:

Irregular surface flatness, cleanliness, etc. will not lead to reproducible results.

2. Surface roughness:

The roughness of the surface will make the contact angle smaller if the original material of the contact angle is smaller than  $90^\circ$  (and vice versa).

3. Samples with high surface energy (glass, metal, etc.):

Generally, contact angles measured, where the liquid's surface tension is less than the solid's surface energy will be very close to 0 degrees.

4. Absorbing samples (paper, powder, cloth, etc.):

Prompt measuring after adhering the droplet on sample is needed for accurate reproducibility, as the droplet will be absorbed quickly. Video printer, automatic image analyzing systems are recommended. (Call Tante applications engineers).

## **IX. DETERMINATION OF WETTING TENSION FROM CONTACT ANGLE DATA (for Polymers)**

Since the surface energy of a solid polymer cannot be measured directly, its value can be estimated from contact angle data. Wetting tension of a surface is a reliable measure of its surface energy. The wetting tension equals the surface tension of the liquid, which exhibits a zero contact angle on that surface.

Treated polymers, whose surfaces do not contain additives, can be considered a homologous polymer series. The Tantec R&D department has found a relationship between  $\cos \theta$  for water and wetting tension for such polymer series. This relationship can be used to estimate values of wetting tension of treated polymers from the water contact angle data. A chart for conversion of contact angle values to wetting tension in dynes/cm is shown in Table I. (Page 25).

The proper measuring procedure is as follows:

1. Take 10 contact angle measurements on your surface. Do not convert each measurement into a dyne/cm value.
2. Calculate the mean value of the samples as well as the standard deviation.
3. Use dyne conversion software to calculate the surface energy.

The conversion of contact angle data to wetting tension is not necessary since the value of the contact angle of water would by itself be sufficient to characterize the surface of treated polymers.

### Accuracy and Repeatability of Measurements

There are some important rules one must follow to assure accuracy of surface measurements:

1. Never handle a sample with bare hands, as oils from your skin can affect the measured surface tension by as much as 10 dynes/cm or more. This rule also applies to all other kinds of contamination.
2. Use ultra-filtered deionized water and keep it in very clean containers. The use of pure water allows for more repeatable and reproducible results.
3. Keep samples free from static electricity buildup.
4. Keep the droplets as round in topographical shape as possible. Dispense liquid droplets to same volume each time when comparing results.
5. Wait the same amount of time to stabilize liquid on surface prior to measurement.

### Effect of Static Charges on Measurements

Polymer surfaces are known to generate static charges when they are processed. A sample for surface measurement may carry the static charge generated in processing or may acquire a charge by friction against the specimen holder surface.

Static charges may cause errors in contact angle measurements. These errors occur as the electrostatic field generated by the charged sample pulls the water droplet to the surface stronger than it would in the absence of charge. You can observe the effect of this static field when you see the droplet suspended from the needle being pulled toward the sample or to the side.

### Static Control Measures

To avoid measurement errors, the sample must be kept free of static buildup before and during measurements. This can be accomplished by using air ionizers, also known as static eliminators. These devices produce air ions of positive and negative polarity. The charged object in the vicinity of the ionizer will attract ions of the opposite polarity and the surface will be neutralized. Among the appropriate antistatic devices are “shockless” electrical static eliminators and radioactive static eliminators. (Contact Tantec Inc. about our static eliminators).

### Effect of Polymer Additive

Additives and plasticizers add significant uncertainty to any surface measurements.

## **OPTIONAL EQUIPMENT**

### **I. Surface-Free Energy (Polar & Dispersive Elements) Determination (WU Method)**

Determining the polar and dispersive elements of surface-free energy. This optional software kit is available for the CAM-PLUS Series.

## **THEORETICAL DISCUSSION**

The surface free energy of a solid material  $Y_s$  consists of two components, dispersion (non-polar) and polar, each reflecting the type of molecular interaction between two phases. <sup>1</sup>

$$Y_s = Y_{sd} + Y_{sp}$$

where  $Y_{sd}$  - dispersion component,  
 $Y_{sp}$  - polar component.

The higher the polar component the more actively the surface reacts with different polar interfaces. High polarity improves surface wettability and helps adhesive bonding.

Polymers used in making films, polyethylene and polypropylene, have a near zero polarity, and therefore, poor wettability and ink adhesion. When surface treatment, such as flame, corona or plasma treatments are applied to a polymer, its surface polarity increases. The stronger the treatment the higher the polarity.

Practically speaking, this method consists of measuring the contact angle at the 3-phase gas/liquid/solid interface point using at least 2 different liquids (depending upon the type of calculations used) with defined surface tensions. Using the measured contact angles with these different liquids, the surface free energy of the solid, including the polar and dispersive elements, can be calculated using either commercially available software or by simultaneous solving of several equations in several unknowns.

## **PRACTICAL MEASUREMENTS**

In order to determine the surface free energy using the equation of states methods proposed by WU, the following "Kit" can be ordered from Tanteq:

EXCEL Worksheet (Version 5.0 or higher) with formula and calculation  
You will need to acquire Trycresyl Phosphate or Methylene Iodide locally.

Determining the surface free energy, polar and dispersive elements using the “Equation of States Method”.

1. Prepare a multiple column/row table such as outlined below.

Measurement Number	Measured Contact Angle Using Water	Measured Contact Angle Using Phosphate Methylene Iodide	Measured Contact Angle Using Trycresyl Phosphate
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			

2. Prepare sample, make sure syringe is loaded with deionized water and take contact angle measurements as per instruction steps (1)-(19). Transpose the measurements to the table prepared in (1) above.
3. Loosen the locking knob on the droplet regulator assembly. Raise the assembly, remove the syringe and carefully set aside. Label the assembly “Water”.
4. Load the second syringe assembly with the second liquid (for example methylene iodide) as per instructions 2) through 4) on page 7. Follow the instructions numbered 5) through 19) on pages 7-10. Take the contact angle measurements. Transpose the contact angles of the sample(s) to the table prepared in (1) above.
5. Loosen the set screw on the dispenser assembly. Raise the assembly, remove the syringe and carefully set aside. Label the syringe “MI” (for methylene iodide).
6. Repeat the above steps for any two pair of liquids noting their respective contact angle measurements.
7. Boot up Microsoft EXCEL Version 5.0 or higher. Insert 3.5” diskette labeled **Tantec Inc. Surface Free Energy Calculator** into diskette drive. Open file MI which contains the calculation software.
8. Insert contact angle values into columns “C” and “D” of spreadsheet. The spreadsheet will calculate polar and dispersive components of surface free energy and their sum. Repeat these steps for other liquids using the measured contact angles and known surface tensions of the respective liquids.

## **THEORETICAL DISCUSSION**

### **II. Recently Advanced and Recently Receded Angles**

This optional kit determines the recently advanced and recently receded angles on materials. This optional kit will work with the CAM-PLUS MICRO model.

Many real surfaces are rough and practically speaking heterogeneous for industrial polymers. A liquid droplet resting on such a surface can be in the metastable state where an energy trough separates the droplet from neighboring states or it resides in a state of equilibrium where it is in the lowest energy state. Often in the real world, the system resides in a metastable state, exhibiting a metastable contact angle. In these cases the advancing and receding contact angles differ from this “static” contact angle and the result is known as hysteresis.

#### **Water’s Reaction To Treated Surfaces**

How will water interact with treated surfaces? Water generally does not react with treated polymers, although, there are exceptions. Also, when water is applied to a treated surface, polar and nonpolar (dispersive) interactions take place. Therefore, as far as water is concerned, it sees a polymer surface only as a combination of polar and dispersive forces acting upon it.

#### **Summary:**

With the proliferation of materials requiring surface treatment, techniques for determining the “level” of treatment have advanced. From simple destructive peel testing to dyne liquid testing to the WCAT technique the measurement techniques have become more sophisticated. Several types of even more advanced testing advancements to these techniques are currently under review by the converting industry.

The Tantec CAM recently advanced/receded measuring option is one of these methods.

In order to modify the existing CAM-PLUS MICRO and convert it to a contact angle meter, which is capable of measuring advancing and receding angle, the projection screen has to be replaced with the one supplied in the retrofit kit. In order to remove the projection screen and install the new one, perform the following:

1. Loosen two holding screws on the back of measuring screen. Remove the screen.
2. Replace the measuring screen with the advanced and receded angle screen.  
Retighten the screws. Make sure the screen can still move freely.

Measuring recently advanced and recently receded angles using the CAM Series.

- 1) Install the 28-gauge needle onto the syringe tip (as supplied in the retrofit kit).
- 2) Recharge/refill the syringe as in page 11.
- 3) Move the needle approximately (1) needle diameter from the surface. Slowly dispense the droplet until it makes contact with the surface. Move the stage down slowly such that the droplet is approximately (2) needle diameters from the surface. The droplet should still be attached to the surface and be immersed by the needle tip. (See Figure 12 below).



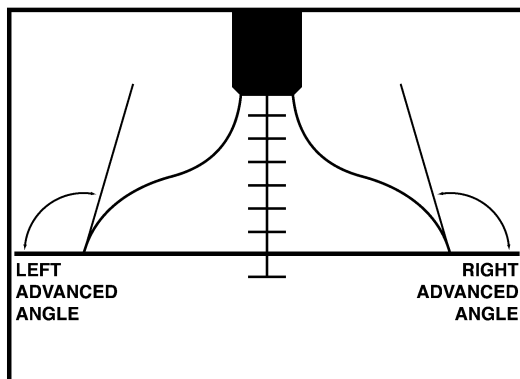


Figure 12

- 4) Verify that the syringe needle's image on the screen is in focus. If it is in focus, go to Step 8.
- 5) Loosen a thumbscrew, which holds the plate 12 with the projection lens stand.
- 6) Move the plate with the lens forward or back until you achieve a crisp focused image of the droplet.
- 7) Tighten the screw to secure the plate in that position.
- 8) Slowly add more liquid to the droplet until the front begins moving. Wait 10 seconds (apparent lateral motion of the front has now ceased).
- 9) Measure the left side of the droplet's contact angle using the newly installed pointer/projection screen combination. Remember to measure the tangent of the droplet at the interface point between the liquid droplet and the solid surface. Record this number.
- 10) Measure the right side of the droplet's contact angle. Record this number.
- 11) Make a minimum of (5) measurements, advancing the droplet in each case until the average contact angle stabilizes with (2) identical readings.

Example Data:

	Contact Angle Left (Degrees)	Contact Angle Right (Degrees)	Average Contact Advanced Angle (Degrees)
1.	60	64	62
2.	62	65	63.5
3.	64	65	64.5
4.	65	66	65.5
5.	66	67	66.5
6.	67	68	67.5
7.	68	69	68.5 ] Recently Advanced
8.	68	69	68.5 ] Angle = 68.5 degrees

Retreating Angle Measurements:

- 12) With the syringe needle still in the droplet slowly retract droplet volume by rotating the micrometer knob counter-clockwise. After 10 seconds measure the left and right edges of the liquid's contact angle. Record the left, right and average contact angles. Again make a minimum of (5) measurements, receding the droplet in each case until the average contact angle stabilizes with (2) identical readings. (See Figure 13 below).

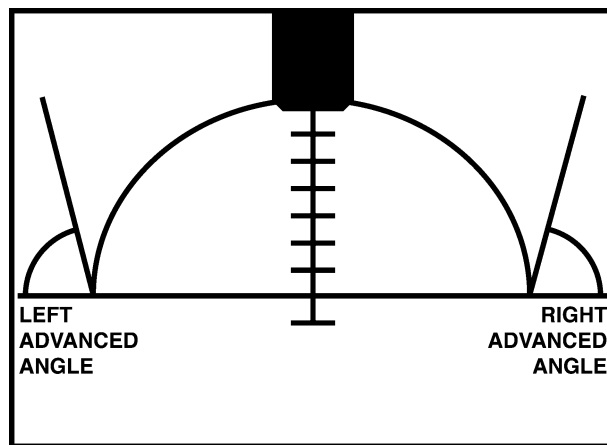


Fig. 13

- 12) Continue retracting, then measuring the contact angle taking careful watch of retreat of the lateral front movement.

See "Surface and Colloid Science", Robert J. Good and Robert Stromberg, Plenum Press 1979 for a more detailed description.

## **TANTEC**

### Contact angle to Dyne Conversion Worksheet

Insert your contact angle here (G8)	28.46	Degrees
Insert the Surface tension of your test liquid here (G9)	72.8	Dynes/cm
Here is the calculated surface tension for your solid	65.14	Dynes/cm

A	B	x'n	x'n+1	Contact Angle
0.939575009	0.6094816	1	0.939575009	<b><u>28.46</u></b>
			0.94570497	
			0.945929664	
			0.945934769	
				Liquid Surface Tension
				72.8

## LIMITED WARRANTY

Every instrument has been carefully inspected and its accuracy checked.

ChemInstruments warrants this equipment to be free from manufacturing defects for a period of one year from the date of original shipment from its factory. ChemInstruments shall repair or replace at its discretion, at no charge to the customer, any part which upon examination by ChemInstruments is determined to be defective in material or workmanship, providing such claimed defective material is returned upon written authorization to ChemInstruments freight prepaid.

Perishable parts are excluded from this warranty.

ChemInstruments warrants that this equipment will meet listed specifications if the equipment is operated by trained operators and in accordance with ChemInstruments operating instructions.

ChemInstruments assumes no responsibility for losses of materials, labor, production time, direct or consequential, resulting from the operation of, or use, or inability to use the product other than specifically covered in this warranty.

Damage or breakage through misuse or negligence is not covered by this warranty. The warranty will be void if the equipment has been altered in any way.

ChemInstruments shall make the final determination of validity of all claims against this warranty.

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Website at: <http://www.cheminstruments.com>

## RETURNING EQUIPMENT

Before returning any equipment to the factory, the following steps should be taken:

1. Contact ChemInstruments customer service department. Give a full description of the difficulty and include the model and serial numbers of the unit. On receipt of this information, we will give you service information or shipping instructions.
2. Equipment returned to ChemInstruments for service must be insured and packed in the original packing carton and inserts. You must request a "Return Goods Authorization Number". No returns will be accepted without a preauthorized RGA number.
3. For non-warranty repairs, ChemInstruments will submit a cost estimate for your approval before proceeding.

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### **Bibliography**

1. Wu S., "Polymer interface and adhesion", Marcel Dekker, Inc., New York, 1982.
2. ASTM Standard Test Method for Wetting Tension of Polyethylene and Polypropylene Films, D2578-84.
3. Ronald J. Caimi, Leighton K. Derr, Thomas J. Dunn, David Rudd, Precision of the Surface Energy Test, Converting Magazine, June 1992.
4. Good R.J., Contact angle, wetting, and adhesion: a critical review, in Contact Angle, Wettability and Adhesion, Ed., K. L. Mittal, pp. 3-36, VSP, 1993.
5. Strobel M. et al., Analysis of air-corona-treated polypropylene and poly (ethylene terephthalate) films by contact-angle measurements and X-ray photoelectron spectroscopy, in Contact Angle, Wettability and Adhesion, Ed. K.L. Mittal, pp. 493-507, VSP, 1993.
6. Bassemir R.W. and Krishnan R., Practical applications of surface energy measurements in flexography, FLEXO, July 1990.