Practical Contact Angle Measurement (3)

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Industry: all
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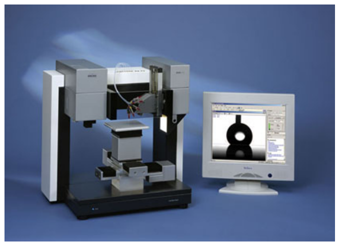
Method: 



Contact Angle Instrument DSA100

The eye also measures

Keywords: methods, contact angle, sessile drop,

The optical system with illumination, camera and video image evaluation is the heart of a contact angle measuring instrument. Its increasing precision is primarily due to advances in video technology and drop shape analysis. This third part of our series on practical contact angle measurement helps you to utilize the possibilities of the optics to the full and measure accurately and reproducibly.

In comparison to dosing, in which the volume and volume flow together with the dosing procedure can be exactly controlled, setting the optical parameters is apparently concerned with more subjective criteria: The drop image should be “well” illuminated and “sharp” and “as large as possible”. In fact the optical parameters can also be controlled exactly and matched to suit the measuring task.

Preparations

The creation of optimal optical conditions starts with the selection of the measuring location. External light influences negatively affect the shadow image of the drop created by using the instrument illumination. The result is a broader gray level distribution of the drop shape and the surrounding white region; this affects the accuracy of contour recognition. In extreme cases – for example direct sunlight – light reflexes can result in the drop not being recognized at all.

The objective or prism and the glass panel in front of the light source should not be touched with the fingers and never with sharp-edged objects. If marks are seen on optical components these should be carefully removed using a solvent-impregnated (e.g. isopropanol), lint-free cotton swab.

All adjustable optical components should have been adjusted before the dosing process; afterwards only minor corrections at the most should be made so that as little time as possible elapses between the drop contacting the sample and the measurement itself. Otherwise incorrect results could be obtained, for example as a result of evaporation of the liquid.

Illumination

“Bright lights cast dark shadows” – this saying only has a limited validity for drop shapes, as the drop liquid is normally light-permeable, so that a bright illumination increases the gray levels not only outside but also inside the drop and possibly produces an unfavorably broad gray level distribution. In addition, if the illumination is too bright then diffraction effects can make the shape appear smaller – the drop is “over-illuminated”. When you have gained some experience in measuring contact angles you will realize that a drop image that is optimal for evaluation frequently seems to be too dark.

The gray level values of the image can easily be read off in all three in KRÜSS programs for Drop Shape Analysis – DSA1, DSA2 and DSA3. The value for the current video image position of the mouse cursor is shown in the information bar at the lower margin of the program window.

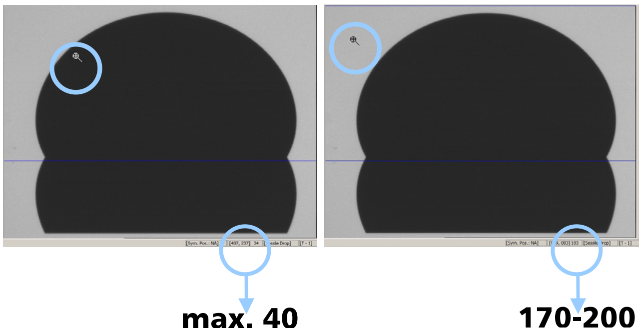


Fig.1: Gray level measurement for a contact angle standard

For the optical drop shape standards, which can be acquired for drop shape calibration, KRÜSS gives gray level guide values of max. 40 for the interior of the drop shape and 170-200 for the outer region. With real drops the value should be measured as close to the drop margin as possible.

Image size

Drop shape analysis increases in reliability as the number of pixels that the shape image contains increases – this means that the drop image must not be too small. The width of the drop should amount to between 2/3 and 3/4 of the total image width. In automatic measurement procedures it must be remembered that the middle of the drop is not always exactly beneath the needle, but is frequently slightly displaced to the left or right of the dosing position. This means that in order for every drop to be shown completely some space must remain at the left and right-hand image margins.

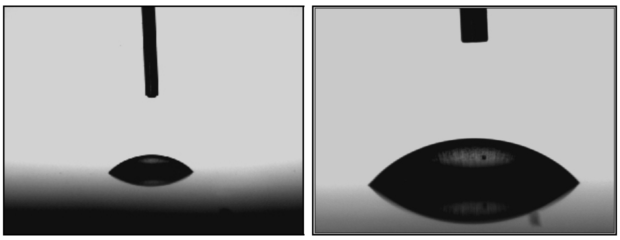


Fig. 2: Drop too small; adequately sized drop

Please also remember that the contact angle itself makes a contribution to the drop width – smaller contact angles result in broader drops. As a result, for example, if the zoom is set for water drops then a diiodomethane drop could extend beyond the image limits. In order to take this alteration into consideration either the dosing volume must be reduced or the programmed zoom adapted to suit the corresponding test liquid.

Image sharpness

The sharpness is the only truly subjective factor in the optical adjustments – which does not really have to present a problem. The eye is extremely reliable in assessing the sharpness and is better at this than the resolution of current cameras and monitors – this means that there is no “concealed” unsharpness. In addition, a slight degree of unsharpness frequently has no effect at all or only a slight one on the precision of the contact angle measurement.

In principle, focusing can be carried out on the needle or drop image. For non-programmed single measurements, which involve or demand adjustment of the optics for each drop, focusing should take place on the drop image.

In contrast, with automatic measurement focusing should be carried out on the needle. The same applies as for the horizontal position: the center of the drop may be slightly in front of or behind the needle. On average – assuming a Gaussian distribution of the position inaccuracy – focusing on the needle results in the smallest variation from the maximum sharpness. The positioning error often results from electrostatic charges on the sample – which is why sample preparation (see part 1 of this series) can also influence image sharpness.

Baseline

The baseline – the border between drop shape and sample surface – is a sensitive factor in shape recognition. A slight displacement of its height can result in an alteration of the measured contact angle by a few degrees. In order to avoid systematic errors during multiple measurements on a drop the baseline should be determined not just once, but separately for each drop shape analysis.

The DSA programs determine the baseline automatically by using the peak or inflection points in the drop shape. These are formed by either the termination of the drop shape line itself or – with reflecting drops – by the transition between the drop image and its mirror image. The second case is the more favorable one. If the surface provides a clear mirror image of the drop shape then the baseline will be recognized exactly.

With non-reflecting samples a max. 4° observation angle can be set for easier recognition of the baseline. At this angle the distortion of the shape is so small that it hardly affects the measured value; the transition between the drop shape and the sample is much clearer. The DSA100 is equipped with specially constructed prism optics allowing the inclination angle to be adjusted.

If the video system uses a frame-grabber then the contrast can be additionally increased by using the software; this also makes it easier to recognize the border region.

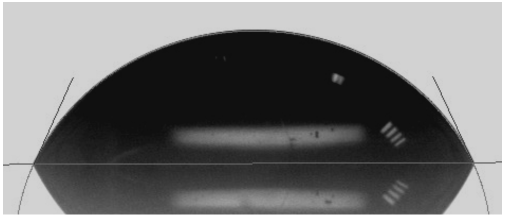


Fig. 3: Drop with baseline

In principle the user should make use of automatic baseline recognition wherever possible. The baseline should only be set manually when no recognition is possible after the measures described above. Setting the baseline becomes even more difficult when the sample not only reflects poorly, but is also inhomogeneous. In this case a large number of measured values with a manually set baseline is frequently necessary. In such a case it is better not to measure online, but to make the measurements on stored drop images or videos.

Small contact angles

With small contact angles and correspondingly flat drops the light meets the drop at an obtuse angle. The result is total reflection of the light from the upper part of the light source, the drop shape may then be impossible to recognize. For this reason the illumination of the DSA100 is fitted with a sliding diaphragm with which the upper part of the illumination can be covered. On the basic instruments the user can tape over this region, but should avoid contaminating the light source.

Summary

The optical components of a contact angle measuring instrument provide many different ways of influencing the quality of the results obtained and of standardizing the measuring procedure. By making good preparations and selectively setting the illumination, zoom, sharpness and observation angle the reproducibility of the drop shape analysis can be improved and even difficult samples mastered.