

# Krüss DSA-30 User Manual

Version of 2024-11-20.

## 1. Introduction

This user manual explains how to operate the Krüss DSA-30 contact angle measurement system in standard mode.

## 2. Login & System Initialization

- Login for “Krüss DSA-30” with CAE on zone 16 (CMi+1 corridor) pc/accounting computer.

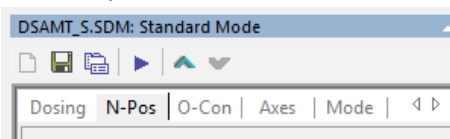


- Switch on the DSA-30 main power. (The lamp should light up and the dosing system should initialize at this point. Call the STAFF if it is not the case.)
- Double-click on the DSA4 icon to start the software.

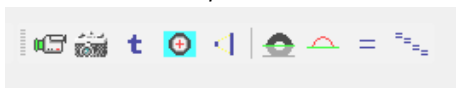


- The live image is displayed in “ImageView”. Place your hand in between the lamp and the objective to make sure that the camera is recording. Call the STAFF if it is not the case.
- Go into “Tools” and “Run Mode”. Verify that “Standard Mode” is checked. If it is not the case, select “Standard Mode”, exit and restart the DSA4 software.
- The machine is operated using the control panel and the measurement panel. See images below.

Control panel:

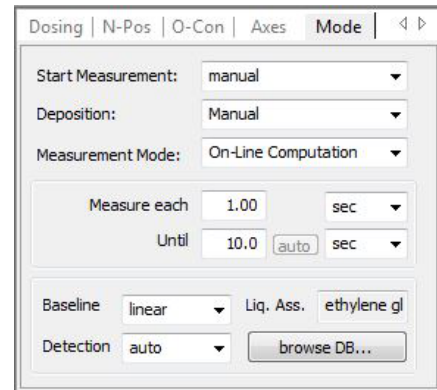


Measurement panel:

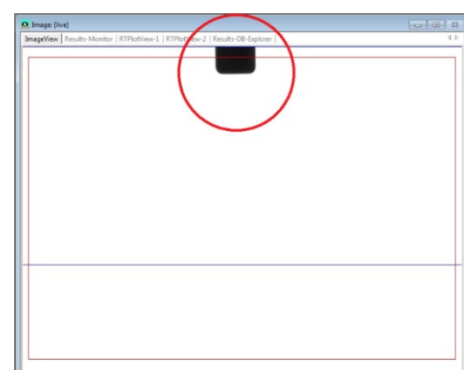


## 3. Measurements Setup

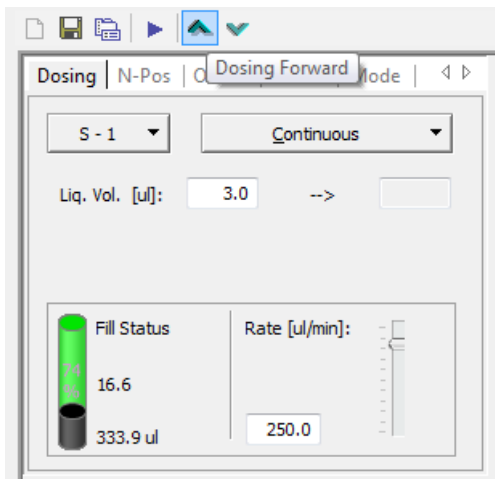
- In the control panel, select the “Mode” tab. Use the standard settings shown here:



- In the control panel, select the “Dosing” tab. Select the syringe you are planning to use: *S-1 = water / S-2 = diiodo-methane / S-3 = ethylene glycol / S-4 = custom / S-5 = manual syringe.*
- Syringe preparation :
  - For manual dispense unit: Please refer to the separate section of the manual.
  - For automatic dispense unit: Refill the syringe using the “Refill → Refill Now!” command.
- Move the sample table down using the manual knob.
- In the control panel, select the “N-Pos” tab. Use the PageDown (fast) and ArrowDown (slow) keys to move the dosing system down until the tip is visible on the screen. See image below.



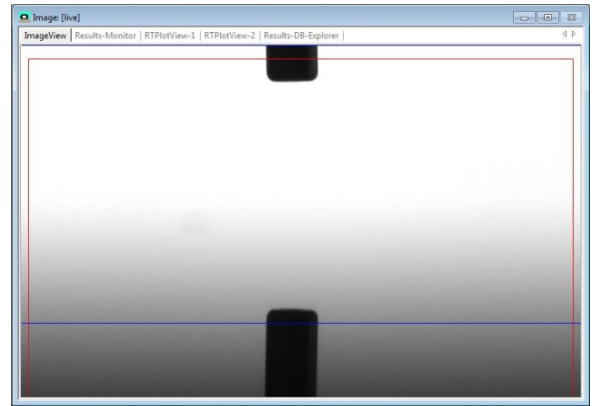
- Droplets & nozzle purge: Place a cleanroom paper on the sample holder.
  - For manual dispense unit: Dispense the liquid with the micrometer knob until droplets are continuously flowing.
  - For automatic dispense unit: In the “Dosing” tab again, switch to “continuous” and press the “dosing forward” command until droplets are continuously flowing. Stop de dosing by pressing the “ESC” key or pressing the “dosing forward” again. See image below. Refill the syringe if necessary.



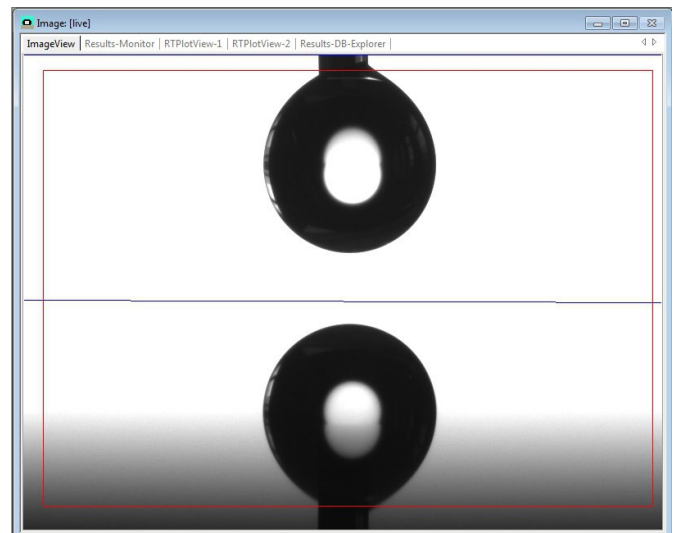
- Adjust the dosing unit Y-axis so that the syringe is at the center of the live image. Set the objective zoom at 2x. **The optimal view angle is +2°. Do not change it!**
- Clean the tip of the nozzle with a cleanroom paper.

#### 4. Taking measurements

- Place your wafer on the table. Center the wafer and the table (X: manual axis / Y: motorized axis at « 50 » in the “Axes” tab. Move the table up until the surface is visible at the bottom of the live image. In most case, the reflection of the nozzle will be visible on the surface. See image below.

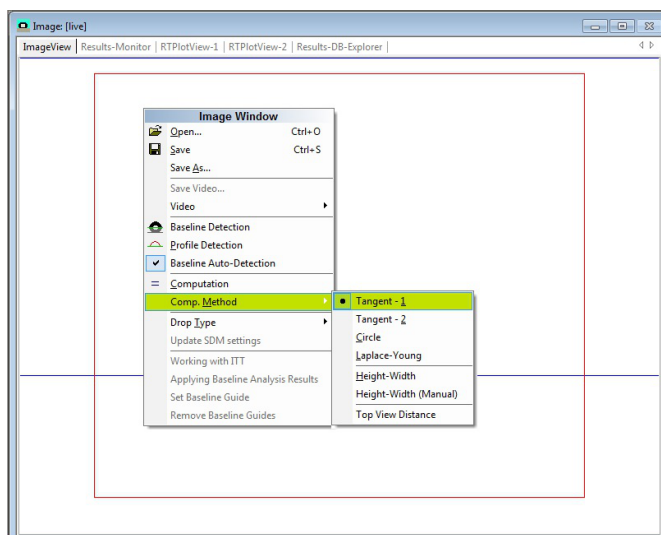


- Generate the drop:
  - For manual dispense unit: Dispense the liquid with the micrometer knob until drop reaches an optimal size.
  - For automatic dispense unit: In the “Dosing” tab again, switch to “volume”. Select the volume and rate, make sure to press the “ENTER” key to validate. Press the “dosing forward” button. **Recommended : S-1 (water) : 3 uL @ 150 uL/min, S-2 (diiodomethane) : 2 uL @ 75 uL/min, S-3 (ethylene glycol) : 2.5 uL @ 75 uL/min**

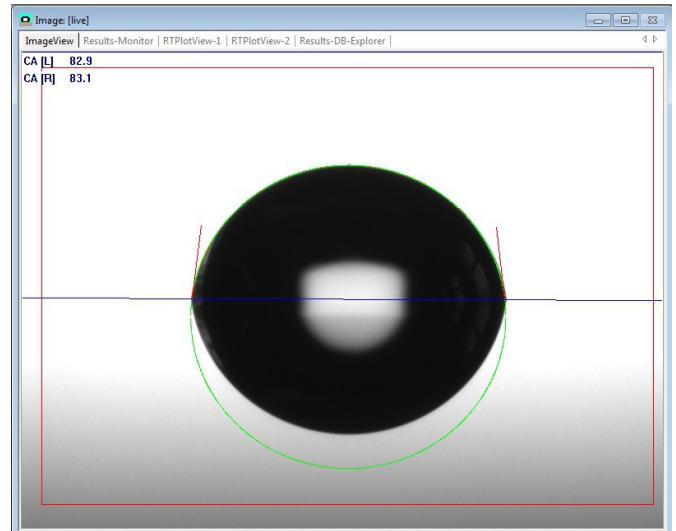


- Depositing the drop: Move the table up until the droplet touches the surface. The droplets will be transferred to the sample.
- Adjust the zoom and focus so that the drop fits nicely in the live image window. The red square defines the region of interest where the droplet computation will be done. It can be moved by shift-clicking on its boundaries.

- In the control panel, select the “O-Con” tab. Use the “ArrowUP” and “ArrowDown” keys to adjust the Intensity. The intensity should be set so that elements on the wafer are not visible.
- The computation can be done on the live image or a captured frame (“Freeze image” or “snap” options).
- In the measurement panel, click on “Baseline Determination”. The blue line should detect the triple-point (droplet corners). If it is not the case, adjust the intensity of the illumination again and the focus of the objective. For small contact angles, to reduce reflections, please use the plastic cache available near the machine to cover the top hemisphere of the illumination.
- Click on “Profile Extraction”. Make sure that the red line correctly detects the droplet shape.
- Change the computation method by clicking on the “ImageView” window and selecting “Comp. Method”. Recommended: Circle for angles below 20°, Tangent - 1 for angles between 20° and 120°, Tangent - 2 for angles above 120°.



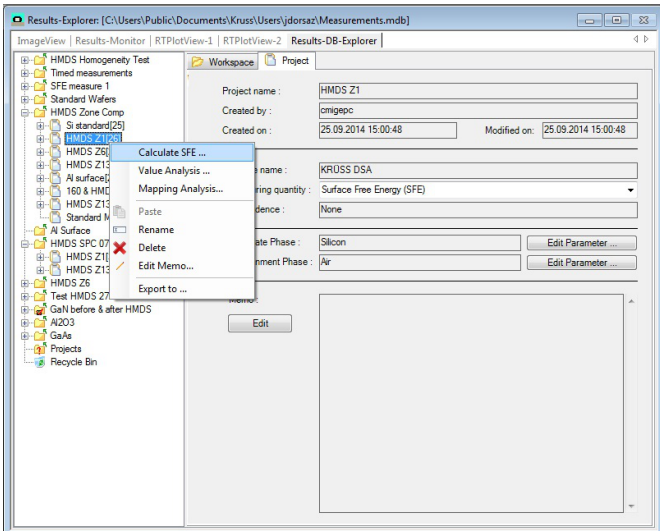
- Take the measurement by clicking on “Computation” or “Run Computation Series”. The series of measurements is set in the “Mode” tab of the control panel.



- You can take several sets of measurements with different liquids. All measurements can be seen in the “Results Monitor” (main window). Please use the motorized Y-Axis (“Axes” tab) to move the wafer to a new position for a new dispense. Minimum distance between droplets should be 5mm.

## 5. SFE Calculation & Saving Data

- Create a database to store your data: Go to “File” → “New” and create a database “your name”. If you already have a database, just open it in the “Results-DB-Explorer” tab.
- In the “Results-Monitor”, check all relevant data and choose “Store into DB”. The program will ask for a workspace, which can be the name of the sample/wafer, date, experiment, etc.... All data will be accessible in the “Results-DB-Explorer” tab.
- To export your data to .csv, .pdf or .txt format, right click on the current set of measurements, and select “Export to”.
- The Surface Free Energy (SFE) of the sample can be calculated in the “Results-DB-Explorer” tab. Right click on the current set of measurements, and select “Calculate SFE”.



- Select the relevant data, the recommended model for SFE and click on “Calculate”.  
**Recommendations:**
  - Silicon, SiO<sub>2</sub>, SiN, III-V  
 Semiconductors: *Extended FOWKES* (3 liquids), *FOWKES* (2 liquids), *OWRK* (2 liquids)
  - Photoresists, polymers: *Wu* (2 liquids minimum), *Zisman* (1 liquid minimum)
  - Metals: *GvOK* (3 liquids minimum)
- A report will show up with the calculated surface free energy value of the sample. The wetting envelope of the wafer can also be displayed in the report.
- Click on “Show Report”. Exporting and printing options will be displayed. Press “Start” and save your files.
- Press “Create and Save Report”. Check the preview. When closing the report will appear in the “Results-DB-Explorer” workspace.

## 6. System shutdown

- Emptying the syringe:
  - For manual dispense unit: Unclip the plunger, push the liquid back into its bottle or appropriate waste. The syringe can be rinsed with IPA, dried and reused several times.
  - For automatic dispense unit: When measurements with a single liquid in finished, the syringe should be emptied in the container. Go into the Dosing tab, select Tools → « Toggle Switch Syringe valve ». The green LED

near the syringe should be off meaning dispense will be done in the container. Switch to « continuous » and press the « dosing forward » command until the syringe is completely empty.

- Close the DSA4 program. Save the changes in the database.
- Switch off the DSA-30 main power.
- Logout with CAE on the Zone PC in Zone 16.

## 7. Manual Syringe Preparation

**Warning:** Test liquids for the Krüss need to be approved by the CMi Staff before being used with the manual-dispense syringes! These liquids are handled in the CMi cleanroom following a generic [SOP](https://cmiaccess.epfl.ch/restricted/files/SOP_Krüss_Chemicals.pdf). Check here for details: [https://cmiaccess.epfl.ch/restricted/files/SOP\\_Krüss\\_Chemicals.pdf](https://cmiaccess.epfl.ch/restricted/files/SOP_Krüss_Chemicals.pdf)

This section describes the steps in the preparation of a manual syringe to use with position S-5 of the dosing unit.

- Always wear appropriate gloves, googles and clothing when manipulating liquids!**
- Make sure to prepare the syringe in a well-ventilated area. Zone 13 wet benches with proper exhaust are recommended.**
- Manual, disposable syringes are available in the drawer of the worktable.
- Remove the plastic plunger from the syringe.
- Transfer the rubber from the plastic plunger to the metallic micrometer dispense plunger. See image below.



- Push the plunger into the disposable syringe. Rotate the holding clip to the top of the plunger. See image below.



- Pump the liquid from its bottle or an appropriate beaker.
- Rotate the holding clip towards its clipped position. Push some liquid and air bubbles back into the tank.
- Clip the micrometer plunger to the disposable syringe.
- Select the appropriate needle and insert it into the syringe. **Red: stainless steel needle.**  
**Purple: teflon needle.** See image below.



- Insert the syringe into the S-5 slot of the dosing unit. **Take care with the tubes of the automatic-dispense syringes!**

## 8. Known Issues

- Motorized Axis failure: Close the DSA4 software, turn-off and back on the Krüss DSA-30 main power. Open the DSA4 software, go into the control panel “Axes” tab, select Y-Axis and activate the “Reset Zero Position” option in the second drop-down menu.