

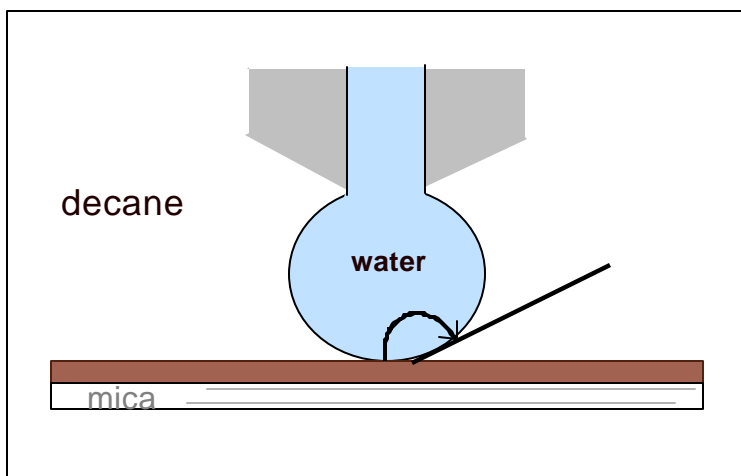
Contact Angle Measurement

A. Sample Preparation

1. Cleave mica – Prepare thin, rectangular mica samples (~1cm x 2 cm). Peel off external layers of mica using scotch tape. Care must be taken not to contaminate the freshly exposed mica surfaces.
2. Clean mica – If further cleaning is prescribed, prepare a wash solution containing 9 parts H₂O₂ (30% solution) and 1 part NH₄OH (20% solution). Place slides in solution and agitate with sonicator for 30 minutes. Remove and let set in hood overnight. Then remove mica and rinse extensively in double distilled water.
3. Condition mica – Place each piece of mica in prescribed brine to wet surface. Then, drain each piece and transfer to a second container of brine. Allow mica to soak for 24 hours.
4. Drain mica – Remove each piece of mica from brine using tweezers. Touch the edge to a clean Kimwipe to remove the excess brine. (Do not dry mica.)
5. Age mica – Place each piece in a sample vial with making sure the mica is completely covered with oil. (If adding precipitant or solvent to the oil, premix before contacting with mica.) Allow mica to soak for the prescribed aging time at prescribed temperature.
6. Rinse mica – After the prescribed aging time, remove the mica from the oil and rinse in prescribed wash solvent until the rinsing solvent comes off clear. The solvent used for washing is usually toluene, but other cyclic hydrocarbons (e.g., cyclohexane) may be substituted. Rinse with purified decane and transfer the mica to a contact angle cell containing about 8-10 ml of purified decane. (It is often useful to place the mica on a teflon stage inside the cell to increase the visibility near the 3 phase contact line.)

B. Advancing Angle Measurement

1. Apply a single drop of double distilled water to surface of mica using Gilmont® buret. Increase the size of the drop slightly causing the drop to advance.
2. Zero the cross hairs and take measurements of the angles on each side of the drop through the water phase after 2 minutes of contact as shown below.
3. If a drop is allowed to rest on the surface, the contact angle normally will change little with time, but if such changes are noted, additional measurements should be made of the advancing angle as a function of contact time.



C. Receding Angle Measurement

1. Retract drop paying close attention to the three-phase line. If that line moves, it is possible to measure the receding angle.
2. Measure this angle as described previously.
3. If the drop does not recede, it is pinned. In that case, receding angles can be measured with a drop of decane brought into contact with a piece of mica that has been dried after the toluene rinse (step A6) and immersed in water.

Repeat parts B and C for about 10 to 12 drops on each piece of mica.