Thin Layer Chromatography of Crude Caffeine

Reference:

Mohrig et al., *Techniques in Organic Chemistry*, p. 221-235 (3rd ed.) or p. 255-269 (4th ed.)

Supplies needed:

Two 15 x 125 mm test tube
Weighing paper
150-mL beaker
Watch glass
Two capillary tubes (open-ended)
One TLC Plate (only touch the TLC plate on the sides on the upper part to prevent contaminations)

Procedure:

• Weigh approximately 4 mg of your crude caffeine

• Dissolve the crude caffeine in approximately 5 mL of distilled water in one of the test tubes and mix well

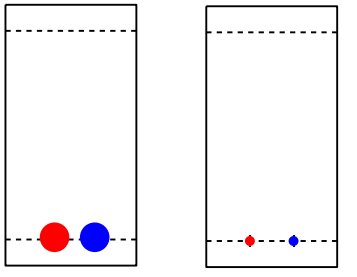
• Pour about ¼ mL of the pure caffeine solution prepared by your TA into the other test tube (You should share this solution with your "hood mate")

• Lightly mark a straight line with a pencil approximately 1 cm from the bottom of the TLC plate (even better are two small notches on the side)

• Spot the TLC plate

• Use a capillary tube to put a small (~ 3-4 mm diameter) spot of crude caffeine solution on the line on the TLC plate about 7-8 mm from the edge

• Use the second capillary tube to put a similar sized spot of pure caffeine solution on the line on the TLC plate approximately 7-8 mm away from the other edge. Make sure that the spots are not too large and that you do not place the two spots too close together. Also, be careful that the spots do not smear on the TLC plate. Make sure you remember which spot is pure caffeine and which one is the crude caffeine.



Red (left): sample
Blue (right): reference

IMPORTANT: After spotting, use the UV Lamp (select the SHORT wavelength, 254 nm) and check to see if you see **TWO** dark spots on the TLC plate. Make sure that the two spots do not overlap. If they do, repeat the spotting procedures with a new TLC plate. If you do not see the spots, you will need to re-spot the TLC plate by adding more sample to each of the spots.

• Fill the bottom of a 150-mL beaker with about 1 cm of distilled water

• Place the TLC plate in the beaker and cover the beaker with a watch glass (see figures 17.1 and 17.5 (3rd ed.) or 18.1 and 18.5 (4th ed.)). Make sure that the solvent level is **BELOW** the line on which you spotted the samples. The solvent (water) will slowly move up the TLC plate by capillary motion.

• Remove the TLC plate from the beaker when the solvent level is about 1 cm from the **TOP** of the TLC plate (mark this location on the TLC plate with a pencil)

• Allow the plate to air dry

• Place the TLC plate under the UV lamp and circle any **DARK** spots in the plate

Do not look directly into the UV lamp!