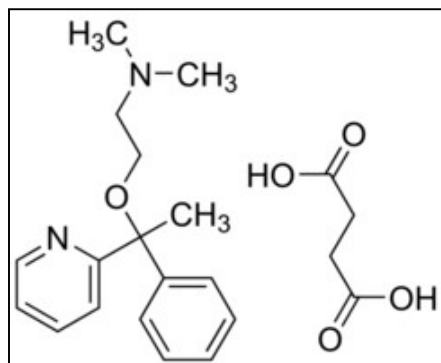# Doxylamine Succinate
25 mg Tablet

Structure:

Molecular Formula and Mass: $C_{17}H_{22}N_2O \cdot C_4H_6O_4$ – 388.46

Category: Antihistamine and sleep aid

Sample:

Grind one tablet and dissolve in 30.0 mL ethanol. Shake at least 10 min and filter. Final concentration of sample solution = 25.0 mg/30.0 mL = 0.833 mg/mL, which is the required concentration representing 100%.

Standards:

High Standard:

The high limit is 115%; therefore the concentration of the high standard = (0.833 mg/mL X 1.15 = 0.958 mg/mL. Weigh approximately 23.0 mg of standard. If you weighed 23.1 mg of standard, dissolve it in: (23.1 mg)/(0.958mg/mL) = 24.1 mL of ethanol. This makes the high standard solution concentration equal to 0.958 mg/mL.

Low Standard:

The low limit is 85%; therefore the concentration of the low standard = (0.833 mg/mL X 0.85 = 0.708 mg/mL. Dilute 1.00 mL of high standard to 1.35 mL by adding 0.35 mL of ethanol (1.15/0.85 = 1.35).

Spotting:

Spot on the 5 X 10 cm silica gel TLC aluminium plate with 3.00 µL aliquots as follows:

        Left spot   low standard (85%) = 2.12 µg
        Center Spot   100% sample = 2.50 µg
        Right Spot   high standard (115%) = 2.88 µg

Development:

Mix 24.0 mL of ethyl acetate, 3.00 mL of methanol, and 1.00 mL of ammonium hydroxide. Develop the plate in a small glass chamber with approximately 20.0 mL of this solution until the solvent front reaches within 1 cm of the top of the TLC plate.

                                                        (R_f = 0.54)

Detection:

UV:

Dry the plate and observe under ultraviolet light at 254 nm. Observe the intensities and the sizes of the spots.
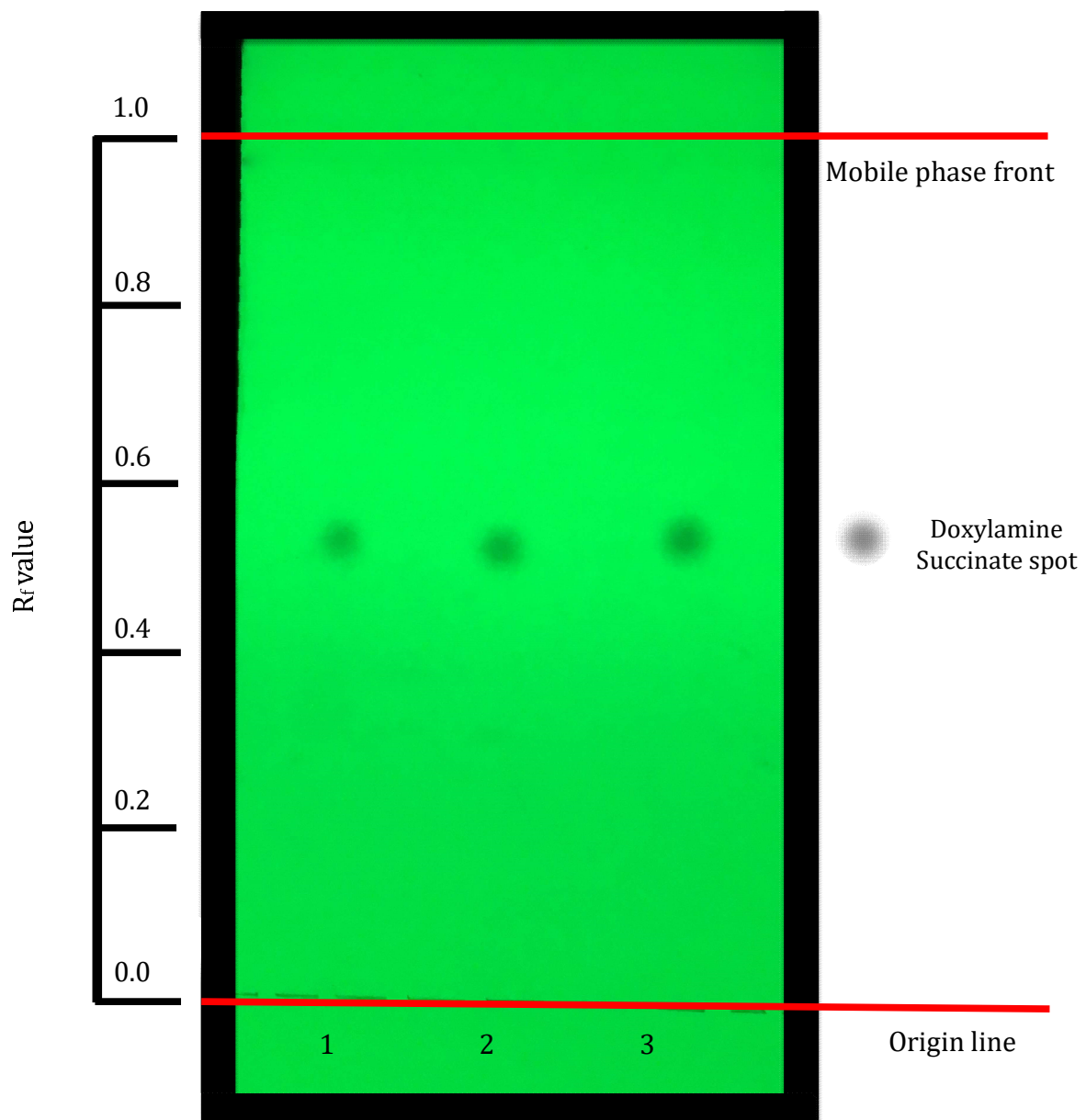
Plate observed under ultraviolet light at 254 nm

Lane 1: Low standard (85%) = 2.12 µg

Lane 2: 100% sample = 2.50 µg

Lane 3: High standard (115%) = 2.88 µg