2.4 synthesis of 2-methyl-1-(prop-2-yn-1-yl)-2,3-dihydro-1H-indole (AZ-1)

Procedure 1:

To 2-methylindoline (1.75 g, 13.2 mmol) and K_2CO_3 (2.18 g, 15.8 mmol) in acetonitrile (20 ml) was added 3-bromorpop-1-yne (1.88 g, 15.8 mmol) in acetonitrile (10 ml) with continuous stirring. The mixture was refluxed for 80 min.

After cooling, the insoluble residue was removed by filtration and the filtrate then concentrated in vacuo. The resulting mixture was extracted with chloroform and distilled water using separatory funnel. The organic layer was treated with Magnesium sulphate anhydrous, filtered and concentrated in vacuo to afford the desired brown crystals, recrystallization from diethylether to afford compound (1.6 g, 93%), **IR (NaCl, Cm^{-1}):** 3020, 2970, (ArH, stretch), 3300 (C≡ C-H, strech), 2300 (C ≡ C, strech), 1610 (Ar, C = C, strech), 1250, 1100, 1010 (Ar, C = C, bending).

^{1}H-NMR (DMSO-d6): δ 1.22 (d, 3H, C-CH_3), 3.06 (d, 1H, C<u>H2</u>-CH-N), 3.15 (s, 1H, C ≡ CH), 3.53, 3.92 (s, 2H, CH_2 - C≡CH), 3.66 (d, 1H, C<u>H_2</u>-CH-N), 3.86 (m, 1H, *J* = 6.15 Hz, N-C<u>H</u>-CH_3), 6.81-7.28 (m, 4H, ArH). **Anal. Calcd, ($C_{12}H_{13}N$):** C (84.3%); H (7.6%); N (8.1%). Found C (84.27%); H (7.58%); N (8.15%). **DSC:** melting point = 58 C° .

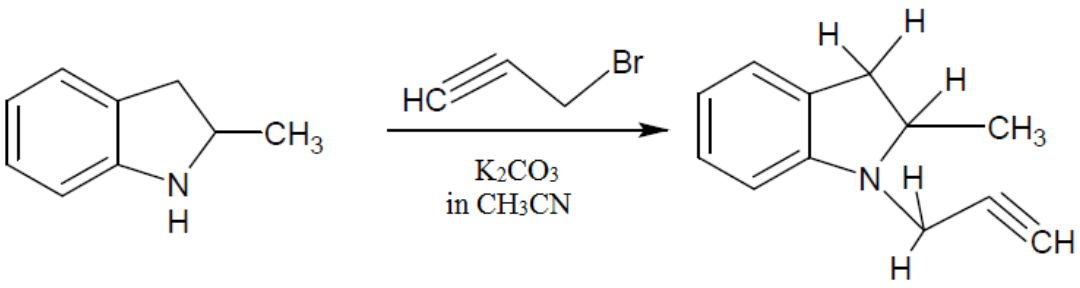

Figure 19: Synthesis of 2-methyl-1-(prop-2-yn-1-yl)-2,3-dihydro-1H-indole.