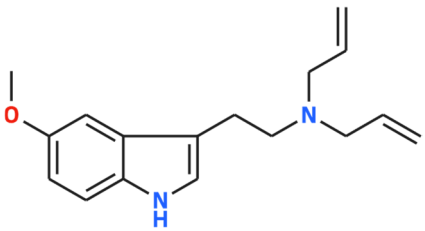
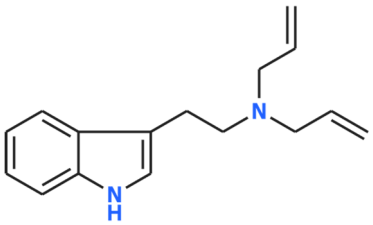
stripped of solvent on the rotary evaporator to yield a white solid residue with a brown oil contamination. This was dissolved in dilute aqueous HCl, washed with 2×50 mL methylene chloride (which removed the color), made basic with dilute aqueous NaOH, and extracted with 2×50 mL methylene chloride. The extracts were pooled and taken to dryness on the rotary evaporator to give 0.67 g of a tan oil that crystallized. This was distilled at the KugelRohr to give a colorless fraction with bp 165–175 °C at 40 microns which weighed 0.53 g and set up immediately as a white crystalline mass with a mp 98–101 °C. This was dissolved in 10 mL warm isopropanol and treated with 14 drops of concentrated HCl. With scratching and stirring, crystals began to form slowly. With vigorous stirring, 10 mL of anhydrous ether were added dropwise, producing a heavy crystalline mass. This was removed by filtration, washed with ether, and air dried to constant weight. There was thus obtained 0.51 g 5-MeO-DALT hydrochloride with a mp of 163–164 °C. The infra-red spectrum of the free base contained the following major peaks (cm^{-1}): 796, 923, 939, 1009, 1033, 1061, 1115. For the hydrochloride salt (cm^{-1}): 809, 829, 837, 946, 1002, 1083, 1180. The GCMS indicated a purity >99% with the following fragments (*m/z*): 110 (diallylaminomethylene fragment, 100%) 117 (26%), 145 (35%), 160 (5-methoxyindolemethylene fragment, 60%), 174 (8%), 229 (2%) and 270 (parent, 4%).

The solids that remained after the above decantation and ether washing were thoroughly ground up under 10 mL methylene chloride and removed by filtration to give 2.73 g of fine white crystals of 5-methoxy-*N,N,N*-triallyltryptammonium iodide. The infra-red spectrum contained the following major peaks (cm^{-1}): 818, 860, 952, 998, 1085, 1188. To a solution of 1.1 g phenyl mercaptan (10 mM) in 20 mL acetone there was added 0.4 g 50% W/W aqueous NaOH followed by 2.2 g of this quaternary salt, and the mixture was held at reflux on the steam-bath for 18 h. After removal of the volatiles on the rotary evaporator, the residue brown solids were suspended in 75 mL water which was made acidic by the addition of aqueous HCl. This was washed with 3×40 mL methylene chloride (the washings were saved), made basic with 5% aqueous NaOH and extracted with 3×40 mL methylene chloride. The combined extracts were stripped of solvent on the rotary evaporator yielding 0.12 g of crude 5-MeO-DALT as a light brown oil that crystallized. The methylene chloride washings (saved above) was extracted with dilute H_2SO_4 (leaving the diphenyldisulfide reaction product in the organic phase), made basic, extracted with methy-



seven drops required) and diluted with 10 mL anhydrous ether which produced, with scratching, fine powdery crystals. These were removed by filtration, washed with ether, and allowed to air-dry to constant weight. There was thus obtained 0.174 g of the first crop of *N,N*-diallyltryptamine hydrochloride (DALT). GCMS indicated a purity of >99%, and fragments (*m/z*): 110 (diallylaminomethylene fragment, 100%), 130 (3-indolemethylene fragment, 15%), 199 (1%) and 240 (parent, 1%). The infra-red spectrum contained the following major peaks (cm^{-1}): 750, 767, 801, 940, 952 and 1106. The mp was 142–143 °C.



The solids that remained following the above decantations were thoroughly triturated under 25 mL methylene chloride and the remaining white powder removed by filtration, washed generously with methylene chloride and air-dried to constant weight. The white solid weight (3.74 g) had an infra-red spectrum consistent with the quaternary salt *N,N,N*-triallyltryptammonium iodide (cm^{-1}): 753, 950, 980, 991, 1011, 1090, 1100. LCMS indicated a cation with a molecular weight of 281. Removal of the solvent from the mother liquors provided 5.10 g of a yellow-tan solid that had an infra-red spectrum identical to that of a reference sample of diisopropylethylamine hydroiodide.

To a solution of 1.1 g. phenylmercaptan (10 mM) in 20 mL acetone, there was added 0.4 g 50% W/W NaOH (5 mM) followed by 2.04 g of *N,N,N*-triallyltryptammonium iodide (5 mM) and the mixture held at reflux on a steam bath for 12 hr. After removal of all volatiles on the rotary evaporator, the residue was suspended in 75 mL water and made acidic with the addition of aq. HCl. This was washed with 2×40 mL methylene chloride (the washings were saved), made basic with 5% NaOH, and extracted with 3×40 mL methylene chloride. The combined extracts were stripped of solvent on the rotary evaporator yielding 0.354 g of a pale yellow oil which crystallized. Distillation at the KugelRohr yielded 0.13 g of a white oil which as converted to the DALT hydrochloride salt (as above) weighing 0.10 g. The methylene chloride washings above, on removal of the solvent, yielded a small amount of a brown oil (additional diallyltryptamine) and a clear colorless fluid that set to a spectacular crystalline mass of diphenyldisulfide.