APPENDIX 1
GENERAL EXPERIMENTAL

1. All melting points and boiling points are uncorrected and expressed in °C.

2. Optical rotations were recorded on a Rudolph polarimeter, Autopol III using sodium 589 nm wavelength.

3. Infrared spectra were recorded on Perkin Elmer Spectrometer RXI/FT and are reported in wavenumbers (cm⁻¹). Only the principle peaks of interest are reported.

4. Chemical ionization (CI) mass spectrum was analysed on a single quadrupole mass spectrometer, Hewlett Packard model 5989A HP engine. The sample was introduced into the source with the help of a particle beam interface, model 5980B. The source manifold and quadrupole temperatures were maintained at 250 °C and 100 °C respectively. Chemical ionization was performed using isobutene as chemical ionization gas to enhance the molecular ion.

5. ¹H and ¹³C NMR spectra were recorded in CDCl₃. ¹H NMR spectra were recorded on Mercury Plus (Varian 400 MHz) or Brucker AC 300F (300 MHz) or Gemini 2000 (Varian 200 MHz) spectrometer. Chemical shifts are expressed as δ values in ppm, downfield from tetramethylsilane (TMS) used as internal standard. J values are measured in Hertz. ¹³C NMR spectra were recorded on Brucker AC 300F (75 MHz) or Gemini 2000 (Varian 50 MHz) spectrometer. Chemical shifts are reported in ppm from tetramethylsilane with the solvent as internal reference (deuterchloroform : δ 77.0 ppm).

6. Analytical thin layer chromatography was performed using TLC grade silica gel 'G' or on 0.25 mm aluminium plates precoated with Merck silica gel 60F₂₅₄. It was visualized by ultraviolet light or iodine vapours. Preparative TLC was performed using Merck Kieselgel 60F₂₅₄, 2mm precoated preparative plates (20X20 cm). Flash chromatography was done using Merck Kieselgel 60 silica gel (230-400 mesh) or silica gel (Acme Synthetic Chemicals, 230-400 mesh).
7. All the non-aqueous reactions were performed in glassware that was flame dried and was under atmosphere of dry nitrogen or argon. Air and moisture sensitive liquids and solutions were transferred via syringe or stainless steel cannula.

8. Solvents used for extraction were LR grade. When necessary, the solvents and reagents were dried prior to use by standard methods. Diethyl ether, THF, benzene, toluene hexane and pentane were distilled from sodium benzophenone ketyl under nitrogen atmosphere prior to use. Benzaldehyde was distilled from zinc dust under reduced pressure. Hexamethyldisilazane, chlororotimethylsilane and diisopropyl amine were distilled from calcium hydride. Dichloromethane was distilled from phosphorous pentoxide.

9. Cul was purified by dissolving an appropriate quantity in boiling saturated aqueous Nal over 30 min. Pure Cul was obtained by cooling and diluting the solution with water, followed by filtration and washing sequentially with H2O, EtOH, EtOAc and ether, pentane and then drying in vacuum for 24 h.

10. CeCl3.7H2O was crushed to fine powder and was made anhydrous by heating under vacuum at 140 °C overnight.

11. Temperature of 0 °C was attained using ice-water bath. Temperature of −78 °C was attained using liquid nitrogen and ethyl acetate slush bath or dry ice-acetone bath or by using cryocool (Julabo FT 901). Temperature of −40 °C was attained by using cryocool (Julabo FT 901).

12. IUPAC nomenclature reported in this thesis has been deduced using CS Chem Draw Ultra 5.0.