Organocatalysis by a multidentate halogen-bond donor: An alternative for hydrogen- bond based catalysis

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Supporting information

Experimental

All the chemicals were purchased from available commercial sources and used without further purification. $^1$H NMR was reported using a Varian Unity plus 400 MHz spectrophotometer in CDCl$_3$. IR Spectroscopy was carried out on a Nicolet 380 FT-IR. Melting points were measured on Fisher-Johns melting point apparatus.

Synthesis of N,N'-(cyclohexane-1,2-diyl)bis(1-phenylmethanimine) (L1-phenyl)

To 1,2-diaminocyclohexane (0.23 g, 2.0 mmol) 100 mL of ethanol was added and heated to 70oC for 10 min. To the reaction mixture, 0.42 g, 4.0 mmol of benzaldehyde was added and heated under reflux for 2 hrs. The volume was reduced to half using a rotary evaporator and to the reaction mixture was then added 200 mL of water to induce precipitation. The resulted solid was redissolved in 120 mL of methylene chloride, washed with water and brine. After concentrating the organic layer, the product was obtained in 80% yield as a white solid. M.p 115-120°C ($^1$HNMR DMSO-d6 $\delta$ H) 8.18 (s 1H) 7.60 (m 3H) 7.35 (m 2H) 3.41 (s 1H) 1.45-1.85 (m 4H)

Synthesis of N,N'-(cyclohexane-1,2-diyl)bis(1-(3-chlorophenyl)methanimine) (L1-Chloro)

To 1,2-diaminocyclohexane (0.11 g, 1.0 mmol) 100 mL of ethanol was added and heated to 70oC for 10 min. To the reaction mixture 0.25 g, 2.0 mmol of 3-chlorobenzaldehyde was added and heated under reflux for 2 hrs. The volume was reduced to half using a rotary evaporator and to the reaction mixture was then added 200 mL of water was added to induce precipitation. The resulting solid was re-dissolved in 120 mL of methylene chloride, washed with water and brine. After concentrating the organic layer product
was obtained in 75% yield as a white solid. M.p 95-100°C ($^1$HNMR DMSO-d6 δ H) 8.18 (s 1H) 7.61 (d 2H) 7.39 (d 2H) 3.34 (s 1H) 1.42-1.77 (m 4H)

Figure 1. $^1$HNMR spectrum of L1

Figure 2. IR spectrum of L1
Figure 3. $^1$HNMR spectrum of L1-phenyl

Figure 4. $^1$HNMR spectrum of L1- chloro
Figure 5. $^1$HNMR spectrum of the reaction without any catalyst after 96 hrs

Figure 6. Reaction progress with different catalytic loading
Figure 7. Appearance of additional peaks in the NMR spectrum of L1-Chloro after the reaction