Electronic Supplementary Material (ESI) for Green Chemistry

ELECTRONIC SUPPORTING INFORMATION (ESI)

Heterogeneously catalysed Strecker-type reactions using supported Co(II) catalysts: microwave vs conventional heating

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General procedure for the Strecker reaction of aldehydes using Co-catalyst

To the mixture of aldehyde (1 mmol), amine (1 mmol), and Co-catalyst **(**1mol%) in sealed tube, trimethylsilyl cyanide (1.1 mmol) was added and the mixture was stirred at room temperature and the appropriate time as indicated in Table 1. The reaction was monitored by TLC. After the completion of the reaction, the reaction mixture was diluted with ethyl acetate and filtrated to obtain the crude product. In some cases the crude product was purified by column chromatography on silica gel to afford the pure related α-amino nitrile in good to excellent yields (Table 1).

General procedure for the Strecker reaction of Ketimines and ketiminum salts using Co-catalyst

To the mixture of ketimine or ketiminum salt (1 mmol) and Co-catalyst (1mol%) in sealed tube, trimethylsilyl cyanide (1.2 mmol) was added at 40 °C and the mixture was stirred at appropriate time as indicated in Table 2. The reaction was monitored by TLC. After the completion of the reaction, the reaction mixture was diluted with ethyl acetate and filtrated to obtain the crude product. In some cases the crude product was purified by column chromatography on silica gel to afford the pure related α-amino nitrile in good to excellent yields (Table 2).

Characterisation of selected compounds

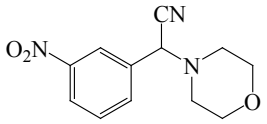
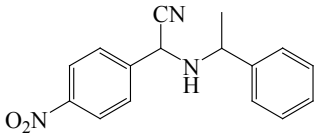
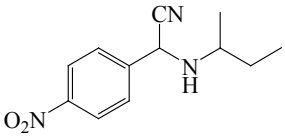
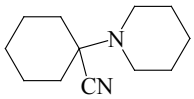
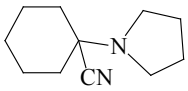
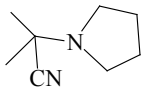
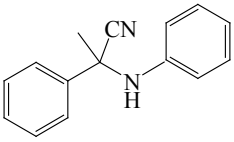
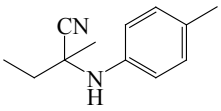
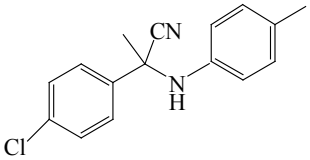
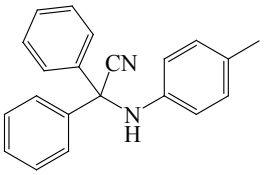
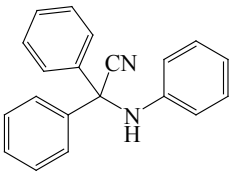
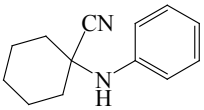
Table	Entry	□	□	Product
1	8			
1	10			
1	11			
2	1			
2	3			
2	5			
2	7			
2	11			
4	7			
4	8			
4	9			
4	10			

Table 1 (Entry 8): ^{1}H-NMR (500 MHz, $CDCl_3$, 25 ºC, TMS): δ = 8.25-8.27 (m, 2H), 7.72-7.74(d, 1H, J= 7.7Hz), 7.60-7.63 (m, 1H), 4.92(s, 1H) ,3.70-3.72 (t, 4H, J=4.8 Hz), 3.21-3.23 (t, 4H, J=4.8 Hz); ^{13}C-NMR(125 MHz,$CDCl_3$, 25 ºC, TMS): δ = 148.5, 137.3, 133.5, 130.3, 125.0, 122.7, 114.5, 67.1, 66.0, 43.1.

Table 1 (Entry 10): ^{1}H-NMR (500 MHz, $CDCl_3$, 25 ºC, TMS): δ = 7.37-7.51 (m, 9H), 4.39 (m, 1H), 4.25-4.29 (m, 1H), 1.63-1.65 (m, 1H), 1.42-1.44 (d, 3H, J=6.5 Hz); ^{13}C-NMR(125 MHz,$CDCl_3$, 25 ºC, TMS): δ = 144.1, 135.3, 134.2, 131.3, 129.4, 129.2, 128.9, 128.3, 127.2, 127.1, 118.9, 57.3, 52.2, 25.2.

Table 1 (Entry 11): ^{1}H-NMR (500 MHz, $CDCl_3$, 25 ºC, TMS): δ = 8.36-8.38 (d, 2H, J=8.3 Hz), 8.21-8.23 (d, 2H, J=8.3 Hz), 4.02-4.05 (m, 1H), 2.20 (s, 2H), 1.75-1.81(m, 2H), 1.38-1.40 (d, 3H, J=5.7Hz), 0.94-0.98(m, 3H); ^{13}C-NMR (125 MHz,$CDCl_3$, 25 ºC, TMS): δ= 138.3, 128.9, 126.3, 124.4, 113.2, 66.8, 66.6, 31.2, 31.1, 21.8, 11.2.

Table 2 (Entry 1): ^{1}H-NMR (500 MHz, $CDCl_3$, 25 ºC, TMS): δ = 7.54-7.56 (m, 2H), 7.37-7.43 (m, 3H), 5.07 (s, 1H), 2.65-2.73 (m, 4H), 1.86-1.89 (m, 4H); ^{13}C-NMR (125 MHz, $CDCl_3$, 25 ºC, TMS): δ = 119.9, 68.6, 61.9, 48.0, 46.1, 42.4, 34.5, 27.4, 26.7, 25.4, 24.7, 23.0, 22.7, 22.4.

Table 2 (Entry 3): ^{1}H-NMR (500 MHz, $CDCl_3$, 25 ºC, TMS): δ = 8.19-8.20 (m, 1H), 7.70-7.73(m, 1H), 7.45-7.50 (m, 1H), 4.82(s, 1H) ,3.47-3.50 (t, 4H, J= Hz), 1.76-1.86 (t, 4H); ^{13}C-NMR(125 MHz,$CDCl_3$, 25ºC, TMS): δ = 148.2, 139.0, 133.5, 129.9, 124.7, 122.6, 117.1, 66.1, 49.8, 46.8, 26.7, 24.7.

Table 2 (Entry 5): ^{13}C-NMR (125 MHz, CDCl3, 25 ºC, TMS): δ = 116.9, 57.8, 22.8, 31.3, 2.3.

Table 2 (Entry 7): ^{1}H-NMR (500 MHz, $CDCl_3$, 25 ºC, TMS): δ = 7.07-7.09 (d, 2H, J=8.4Hz), 6.86-6.87 (d, 2H, J=8.4Hz), 3.74 (brs, 1H), 2.33 (s, 3H), 1.99-2.02 (m, 1H), 1.88-1.91 (m, 1H), 1.62 (s, 3H), 1.18-1.21 (t, 3H, J= 7.5 Hz); ^{13}C-NMR(125 MHz,$CDCl_3$, 25 ºC, TMS): δ = 141.5, 131.1, 130.2,130.1, 121.6, 119.9, 115.6, 54.5, 34.0, 25.5, 21.0, 9.0.

Table 2 (Entry 11): ^{1}H-NMR (500 MHz, $CDCl_3$, 25 ºC, TMS): δ = 7.66-7.68 (d, 2H, J=7.3Hz), 7.42-7.44 (m, 2H), 7.36-7.39 (m, 1H), 6.94-6.95 (d, 2H, J=8.3Hz), 6.47-6.48 (d, 2H, J=8.3Hz), 2.25 (s, 3H) ,1.97 (s, 3H); ^{13}C-NMR(125 MHz,$CDCl_3$, 25 ºC, TMS): δ = 130.0, 129.6, 128.9, 125.4, 116.8, 57.8, 33.7, 20.9.

Table 4 (Entry 7): ^{1}H-NMR (500 MHz, $CDCl_3$, 25 ºC, TMS): δ = 8.08-8.11 (d, J=8.5 Hz, 2H), 7.87-7.90 (d, J=8.5 Hz, 2H), 7.77 (s, 1H), 7.20-7.24 (d, J=8.75 Hz, 2H), 7.02-7.06 (d, J=8.75 Hz, 1H), 4.47-4.49 (bs, 1H), 2.32 (s, 3H), 2.40 (s, 3H); ^{13}C-NMR(125 MHz,$CDCl_3$, 25 ºC, TMS): δ = 154.3, 138.7, 136.8, 134.5, 129.7, 129.3, 126.6, 118.5, 114.5, 57.7, 35.5, 32.9.

Table 4 (Entry 8): ^{1}H-NMR (500 MHz, $CDCl_3$, 25 ºC, TMS): δ = 8.15-8.17 (m, 4H), 7.83-7.93 (m, 6H), 7.77 (m, 1H), 7.22-7.26 (d, J=8.75 Hz, 2H), 7.07-7.11 (d, J=9 Hz, 1H), 2.72 (s, 1H), 2.35 (s, 3H); ^{13}C-NMR(125 MHz,$CDCl_3$, 25 ºC, TMS): δ = 153.9, 139.8, 137.4, 129.1, 128.7, 126.3, 120.1, 118.0, 114.4, 65.8, 34.5.

Table 4 (Entry 9): ^{1}H-NMR (500 MHz, $CDCl_3$, 25 ºC, TMS): δ= 8.14-8.17 (m, 4H), 7.87-7.95 (m, 4H), 7.77 (m, 2H), 7.34-7.71 (m, 2H), 7.33-7.39 (m, 1H), 7.12-7.15 (m, 2H), 4.93-5.00 (s, 1H); ^{13}C-NMR (62.9 MHz, CDCl3; TMS): δ = 143.5, 139.6, 129.2, 128.96, 128.93, 126.3, 120.2, 119.8, 116.1, 66.4.

Table 4 (Entry 10): ^{1}H-NMR (500 MHz, $CDCl_3$, 25 ºC, TMS): δ = 7.21-7.27 (m, 2H), 6.87-6.92 (m, 3H), 3.60 (bs, 1H), 2.31-2.35 (m, 2H), 1.63-1.76 (m, 8H), 1.32 (bs, 1H);

^{13}C-NMR(125 MHz,$CDCl_3$, 25 ºC, TMS): δ = 143.6, 129.2, 121.2, 120.5, 117.5, 54.4, 36.6, 24.9, 22.3.