

Synthesis of 2-hydroxymalonamides from carbamoylsilane and α -keto carboxamides

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3h: yield 95%, mp 135–136 °C. ^1H NMR δ : 7.27–6.87 (m, 4H, C_6H_4), 3.82 (s, 1H, OCH_3), 3.05 (s, 6H, 2 NCH_3), 2.94 (s, 6H, 2 NCH_3), 0.05 (s, 9H, Me_3Si). ^{13}C NMR (151MHz, CDCl_3) δ : 170.4, 159.8, 141.0, 129.4, 119.7, 113.7, 113.1, 81.8, 55.3, 39.0, 37.7, 1.95. IR (KBr) ν : 1620, 1350, 1120, 550 cm^{-1} . Calc. for $\text{C}_{17}\text{H}_{28}\text{N}_2\text{O}_4\text{Si}$ (%): C 57.92, H 8.01, N 7.95; found (%): C 57.72, H 8.15, N 7.98.

3i: yield 86%, mp 130–131 °C. ^1H NMR δ : 7.21–7.15 (q, 4H, C_6H_4), 3.05 (s, 6H, 2 NCH_3), 2.93 (s, 6H, 2 NCH_3), 2.37 (s, 1H, CH_3) 0.01 (s, 9H, Me_3Si). ^{13}C NMR (151MHz, CDCl_3) δ : 170.7, 138.2, 136.5, 129.2, 127.1, 81.8, 39.0, 37.6, 21.2, 2.0. IR (KBr) ν : 1605, 1408, 1132, 785 cm^{-1} . Calc. for $\text{C}_{17}\text{H}_{28}\text{N}_2\text{O}_3\text{Si}$ (%): C 60.68, H 8.39, N 8.32; found (%): C 60.65, H 8.45, N 8.10.

4b: yield 69%, mp 66–68 °C. ^1H NMR δ : 4.99 (s, 1H, OH), 3.01 (s, 12H, 4 NCH_3), 2.69 (m, J = 6.6 Hz, 1H, CH), 0.89 (d, J = 6.6 Hz, 6H, 2 CCH_3). ^{13}C NMR (151MHz, CDCl_3) δ : 170.5, 80.9, 37.8, 37.4, 32.2, 17.2. IR (KBr) ν : 3503, 1665, 1438, 1370, 1134 cm^{-1} . Calc. for $\text{C}_{10}\text{H}_{20}\text{N}_2\text{O}_3$ (%): C 55.53, H 9.32, N 12.95; found (%): C 55.43, H 9.23, N 12.88.

4d: yield 80%, mp 100–101 °C. ^1H NMR δ : 5.03 (s, 1H, OH), 3.02 (s, 12H, 4 NCH_3), 2.36 (m, 1H, CH), 1.76 (m, 2H, CH_2), 1.64–1.13 (m, 8H, 4 CH_2). ^{13}C NMR (151MHz, CDCl_3) δ : 170.4, 81.1, 42.3, 37.8, 37.4, 27.1, 26.6, 26.4. IR (KBr) ν : 3490, 1701, 1361, 861 cm^{-1} . Calc. for $\text{C}_{13}\text{H}_{24}\text{N}_2\text{O}_3$ (%): C 60.91, H 9.44, N 10.43; found (%): C 60.75, H 9.16, N 10.78.

4e: yield 86%, mp 100–102 °C. ^1H NMR δ : 7.40–7.31 (m, 5H, C_6H_5), 6.60 (s, 1H, OH), 3.08 (s, 6H, 2NCH₃), 2.84 (s, 6H, 2NCH₃). ^{13}C NMR (151MHz, CDCl_3) δ : 170.5, 139.5, 128.6, 128.3, 127.3, 82.0, 38.9, 37.7. IR (KBr) ν : 3475, 1658, 1470, 685 cm^{-1} . Calc. for $\text{C}_{13}\text{H}_{18}\text{N}_2\text{O}_3$ (%): C 62.38, H 7.25, N 11.20; found (%): C 62.63, H 7.21, N 11.42.

4f: yield 81%, mp 133–134 °C. ^1H NMR δ : 7.45–7.27 (m, 4H, C_6H_4), 6.43 (s, 1H, OH), 3.08 (s, 6H, 2NCH₃), 2.87 (s, 6H, 2NCH₃). ^{13}C NMR (151MHz, CDCl_3) δ : 170.1, 136.5, 134.1, 132.0, 129.9, 129.5, 126.9, 82.3, 39.0, 37.8. IR (KBr) ν : 3469, 1615, 1400, 1125, 806 cm^{-1} . Calc. for $\text{C}_{13}\text{H}_{17}\text{N}_2\text{O}_3\text{Cl}$ (%): C 54.84, H 6.02, N 9.84; found (%): C 54.63, H 5.99, N 9.63.

4j: yield 70%, mp 128–129 °C. ^1H NMR δ : 7.57–7.32 (m, 5H, C_6H_5), 6.96 (d, 1H, =CH), 6.64 (d, 1H, =CH), 5.54 (s, 1H, OH), 3.07 (s, 6H, 2NCH₃), 3.04 (s, 6H, 2NCH₃). ^{13}C NMR (151MHz, CDCl_3) δ : 191.1, 144.2, 133.4, 132.4, 129.1, 128.8, 123.4, 77.4, 37.2, 34.4. IR (KBr) ν : 3485, 1656, 1432, 1250, 994, 728 cm^{-1} . Calc. for $\text{C}_{15}\text{H}_{20}\text{N}_2\text{O}_3$ (%): C 65.20, H 7.29, N 10.14; found (%): C 65.35, H 7.52, N 10.03.

4k: yield 80%, mp 107–108 °C. ^1H NMR δ : 7.42 (d, 1H, 5- H_{Furyl}), 6.47 (m, 1H, 4- H_{Furyl}), 6.40 (d, 1H, 3- H_{Furyl}), 6.11 (br s, 1H, OH), 3.06 (s, 6H, 2NCH₃), 2.93 (s, 6H, 2NCH₃). ^{13}C NMR (151MHz, CDCl_3) δ : 168.5, 151.8, 142.5, 110.8, 109.3, 77.3, 37.8, 37.6. IR (KBr) ν : 3486, 1685, 1413, 758 cm^{-1} . Calc. for $\text{C}_{11}\text{H}_{16}\text{N}_2\text{O}_4$ (%): C 54.99, H 6.71, N 11.66; found (%): C 54.76, H 6.98, N 11.40.