

Advanced
**Synthesis &
Catalysis**

Supporting Information

© Copyright Wiley-VCH Verlag GmbH & Co. KGaA, 69451 Weinheim, 2005

A General and Efficient Method for the Preparation of β -Enamino ketones and Esters Catalyzed by Indium Tribromide

Zhan-Hui Zhang, Liang Yin, Yong-Mei Wang*

Department of Chemistry and the State Key Laboratory of Elemento-Organic Chemistry, Nankai University, Tianjin 300071, China

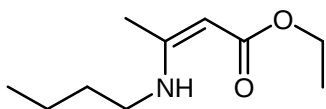
Fax: +86(22)23502654

E-mail: zhanhui@mail.nankai.edu.cn

Supporting Information

General Information. Melting points were measured using a X-4 apparatus and are uncorrected. NMR spectra were taken with a Bruker 300 spectrometer in a CDCl_3 solution with tetramethylsilane as an internal standard. IR spectra were obtained using Bruker-TENSOR 27 spectrometer instrument. Mass spectra were performed on a ThermoFinnigan LCQ Advantage instrument with an ESI source (4.5 KeV). Elemental analyses were carried out on MT-3 analyzer.

Ethyl 3-(butylamino)but-2-enoate (Table 2, entry 1)^[1]

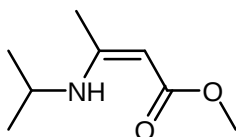


A yellowish oil.

IR (neat): $\nu = 3283, 2959, 2873, 1651, 1609, 1503, 1272, 1173, 1059, 783 \text{ cm}^{-1}$.

^1H NMR (CDCl_3 , 300 MHz): $\delta = 0.93$ (t, $J = 6.9$ Hz, 3H), 1.25 (t, $J = 6.9$ Hz, 3H), 1.36-1.57 (m, 4H), 1.91 (s, 3H), 3.20 (q, $J = 6.3$ Hz, 2H), 4.08 (q, $J = 6.9$ Hz, 2H), 4.42 (s, 1H), 8.56 (br s, 1H, NH).

Methyl 3-(isopropylamino)but-2-enoate (Table 2, entry 2)^[2]

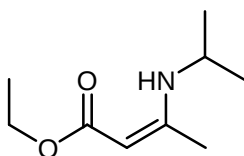


A yellowish oil.

IR (neat): ν = 3274, 2972, 2875, 1656, 1606, 1500, 1444, 1269, 1157, 1057, 1029, 784 cm^{-1} .

^1H NMR (CDCl_3 , 300 MHz): δ = 1.21 (d, J = 6.3 Hz, 6H), 1.94 (s, 3H), 3.61 (s, 3H), 3.64-3.74 (m, 1H), 4.39 (s, 1H), 8.50 (br s, 1H, NH).

Ethyl 3-(isopropylamino)but-2-enoate (Table 2, entry 3)^[3,4]

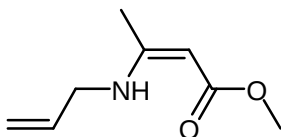


A yellowish oil.

IR (neat): ν = 3272, 2974, 2933, 1651, 1607, 1499, 1444, 1268, 1156, 1059, 784 cm^{-1} .

^1H NMR (CDCl_3 , 300 MHz): δ = 1.20-1.27 (m, 9H), 1.94 (s, 3H), 3.60-3.74 (m, 1H), 4.08 (q, J = 7.2 Hz, 2H), 4.39 (s, 1H), 8.50 (br s, 1H, NH).

Methyl 3-(allylamino)but-2-enoate (Table 2, entry 4)^[2]



A yellowish oil.

IR (neat): ν = 3294, 3084, 1655, 1606, 1501, 1285, 1173, 1060, 929, 785 cm^{-1} .

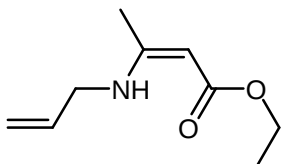
^1H NMR (CDCl_3 , 300 MHz): δ = 1.90 (s, 3H), 3.63 (s, 3H), 3.81-3.85 (m, 2H), 4.98 (s, 1H), 5.14-5.25 (m, 2H), 5.82-5.93 (m, 1H), 8.64 (br s, 1H, NH).

^{13}C NMR (CDCl_3 , 300 MHz): δ = 18.8, 44.9, 49.7, 82.3, 115.6, 134.8, 161.8, 170.7.

ESI-MS: m/z = 156 ($\text{M}+1$)⁺.

Anal. Calcd for $\text{C}_8\text{H}_{13}\text{NO}_2$: C, 61.91; H, 8.44; N, 9.03. Found: C, 62.08; H, 8.26; N, 8.88.

Ethyl 3-(allylamino)but-2-enoate (Table 2, entry 5)^[5]

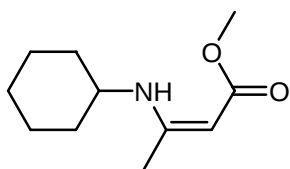


A yellowish oil.

IR (neat): ν = 3290, 3084, 2902, 1743, 1717, 1652, 1607, 1500, 1235, 1148, 1097, 927, 784 cm^{-1} .

^1H NMR (CDCl_3 , 300 MHz): δ = 1.25 (t, J = 6.9 Hz, 3H), 1.91 (s, 3H), 3.82-3.87 (m, 2H), 4.09 (q, J = 6.9 Hz, 2H), 4.48 (s, 1H), 5.15 (d, J = 10.8 Hz, 1H), 5.21 (d, J = 17.4 Hz, 1H), 5.80-5.92 (m, 1H), 8.66 (br s, 1H, NH).

Methyl 3-(cyclohexylamino)but-2-enoate (Table 2, entry 6)^[6]

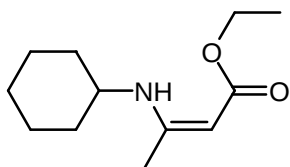


A yellowish oil.

IR (neat): ν = 3278, 2932, 1654, 1608, 1498, 1447, 1276, 1172, 1059, 784 cm^{-1} .

^1H NMR (CDCl_3 , 300 MHz): δ = 1.22-1.42 (m, 6H), 1.75-1.88 (m, 4H), 1.94 (s, 3H), 3.26-3.37 (m, 1H), 3.61 (s, 3H), 4.39 (s, 1H), 8.64 (br s, 1H, NH).

Ethyl 3-(cyclohexylamino)but-2-enoate (Table 2, entry 7)^[3,5]



A yellowish oil.

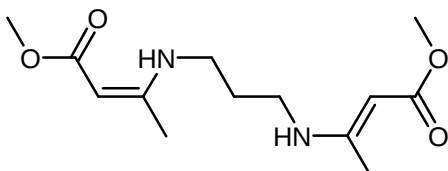
IR (neat): ν = 3275, 2932, 1651, 1608, 1497, 1448, 1274, 1172, 1060, 783 cm^{-1} .

^1H NMR (CDCl_3 , 300 MHz): δ = 1.24 (t, J = 6.9 Hz, 3H), 1.29-1.88 (m, 10H), 1.93 (s, 3H), 3.29-3.33 (m, 1H), 4.08 (q, J = 6.9 Hz, 2H), 4.38 (s, 1H), 8.62 (br s, 1H, NH).

ESI-MS: m/z = 212 ($\text{M}+1$)⁺.

Anal. Calcd for $\text{C}_{12}\text{H}_{21}\text{NO}_2$: C, 68.21; H, 10.02; N, 6.63. Found: C, 68.50; H, 10.28; N, 6.41.

Methyl 3-[3-(2-methoxycarbonyl-1-methyl-vinylamino)-propylamino]-but-2-enoate (Table 2, entry 8)



A pale yellow solid, mp 69-70 $^{\circ}\text{C}$.

IR (KBr): ν = 3443, 2947, 1655, 1602, 1269, 1170, 1053, 786 cm^{-1} .

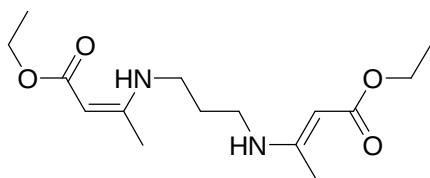
^1H NMR (CDCl_3 , 300 MHz): δ = 1.83 (quin, J =6.3 Hz, 2H), 1.92 (s, 6H), 3.31 (q, J =6.3 Hz, 4H), 3.62 (s, 6H), 4.47 (s, 2H), 8.55 (br s, 2H, NH).

^{13}C NMR (CDCl_3 , 75 MHz): δ = 19.2, 31.0, 39.7, 49.8, 82.4, 161.8, 170.9.

ESI-MS: 271 ($\text{M}+1$)⁺.

Anal. Calcd for $\text{C}_{13}\text{H}_{22}\text{N}_2\text{O}_4$: C, 57.76; H, 8.20; N, 10.36. Found: C, 57.58; H, 8.42; N, 10.20.

Ethyl 3-[3-(2-ethoxycarbonyl-1-methyl-vinylamino)-propylamino]-but-2-enoate (Table 2, entry 9)^[7]



A pale yellow solid, mp 39-40 °C.

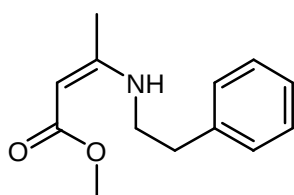
IR (KBr): ν = 3291, 2981, 1647, 1605, 1500, 1441, 1360, 1276, 1171, 1054, 978 cm^{-1} .

^1H NMR (CDCl_3 , 300 MHz): δ = 1.25 (t, J = 7.2 Hz, 6H), 1.82 (quin, J = 6.6 Hz, 2H), 1.91 (s, 6H), 3.31 (q, J = 6.6 Hz, 4H), 4.08 (q, J = 7.2 Hz, 4H), 4.46 (s, 2H), 8.56 (br s, 2H, NH).

^{13}C NMR (CDCl_3 , 75 MHz): δ = 14.6, 19.2, 31.0, 39.8, 58.3, 82.7, 161.7, 170.6.

ESI-MS: 299 ($\text{M}+1$) $^+$.

Methyl 3-(phenethylamino)but-2-enoate (Table 2, entry 10)^[8]

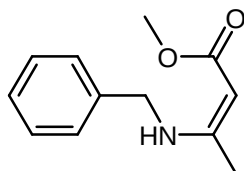


A yellowish oil.

IR (neat): ν = 3287, 2946, 1653, 1601, 1498, 1440, 1266, 1171, 1059, 784 cm^{-1} .

^1H NMR (CDCl_3 , 300 MHz): δ = 1.81 (s, 3H), 2.84 (t, J = 7.2 Hz, 2H), 3.40-3.47 (m, 2H), 3.61 (s, 3H), 4.42 (s, 1H), 7.18-7.25 (m, 3H), 7.28-7.33 (m, 2H), 8.63 (br s, 1H, NH).

Methyl 3-benzylamino-but-2-enoate (Table 2, entry 11)^[2,8]



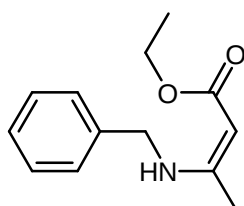
A pale yellow solid, mp 36-37 °C.

IR (KBr): ν = 3442, 3133, 1646, 1605, 1505, 1451, 1400, 1316, 1284, 1238, 1172, 1117, 1068, 1013, 951, 847, 783 cm^{-1} .

^1H NMR (CDCl_3 , 300 MHz): δ = 1.92 (s, 3H), 3.63 (s, 3H), 4.43 (d, J = 6.3 Hz, 2H), 4.53 (s, 1H), 7.24-7.28 (m, 3H), 7.32-7.36 (m, 2H), 8.95 (br s, 1H, NH).

Anal. Calcd for $\text{C}_{12}\text{H}_{15}\text{NO}_2$: C, 70.22; H, 7.37; N, 6.82. Found: C, 70.01; H, 7.46; N, 6.65.

Ethyl 3-benzylamino-but-2-enoate (Table 2, entry 12)^[3,5]

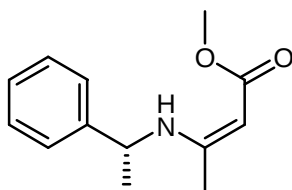


A yellowish oil.

IR (neat): $\nu = 3292, 2978, 1651, 1608, 1496, 1235, 1171, 1059, 784 \text{ cm}^{-1}$.

$^1\text{H NMR}$ (CDCl_3 , 300 MHz): $\delta = 1.25$ (t, $J = 6.9 \text{ Hz}$, 6H), 1.91 (s, 3H), 4.09 (q, $J = 6.9 \text{ Hz}$, 2H), 4.42 (d, $J = 6.3 \text{ Hz}$, 2H), 4.53 (s, 1H), 7.27-7.33 (m, 5H), 8.95 (br s, 1H, NH).

Methyl (*R*)-3-(1-Phenyl-ethylamino)-but-2-enoate (Table 2, entry 13)^[9]



A colorless liquid, $[\alpha]_{\text{D}}^{20}$: - 546 (c 1.20, EtOH).

IR (neat): $\nu = 3279, 2972, 2928, 1652, 1607, 1493, 1445, 1377, 1266, 1054, 852, 763, 701 \text{ cm}^{-1}$.

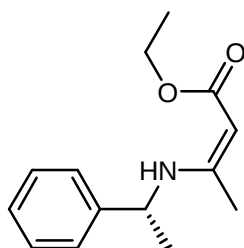
$^1\text{H NMR}$ (CDCl_3 , 300 MHz): $\delta = 1.51$ (d, $J = 6.6 \text{ Hz}$, 3H), 1.77 (s, 3H), 3.66 (s, 3H), 4.48 (s, 1H), 4.63 (q, $J = 6.6 \text{ Hz}$, 1H), 7.21-7.36 (m, 5H), 8.96 (br s, 1H, NH).

$^{13}\text{C NMR}$ (CDCl_3 , 75 MHz): $\delta = 19.6, 24.9, 49.9, 52.8, 82.8, 125.3, 127.0, 128.7, 144.9, 161.5, 170.9$.

ESI-MS: 220 ($\text{M}+1$)⁺.

Anal. Calcd. for $\text{C}_{13}\text{H}_{17}\text{NO}_2$: C, 71.21; H, 7.81; N, 6.39. Found: C, 71.48; H, 8.01; N, 6.11.

Ethyl (*R*)-3-(1-Phenyl-ethylamino)-but-2-enoate (Table 2, entry 14)^[4,8,10]

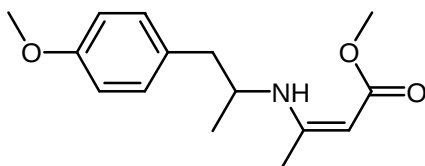


A colorless liquid, $[\alpha]_{\text{D}}^{20}$: - 628 (c 1.21, EtOH).

IR (neat): $\nu = 3278, 2978, 2929, 1651, 1608, 1493, 1444, 1384, 1264, 1053, 843, 785, 701 \text{ cm}^{-1}$.

$^1\text{H NMR}$ (CDCl_3 , 300 MHz): $\delta = 1.29$ (t, $J = 7.2 \text{ Hz}$, 3H), 1.53 (d, $J = 6.6 \text{ Hz}$, 3H), 1.79 (s, 3H), 4.14 (q, $J = 7.2 \text{ Hz}$, 2H), 4.89 (s, 1H), 4.65 (m, 1H), 7.22-7.37 (m, 5H), 8.99 (br s, 1H, NH).

Methyl 3-[2-(4-Methoxy-phenyl)-1-methyl-ethylamino]-but-2-enoate (Table 2, entry 15)



A pale yellow solid, mp 49-51 °C.

IR (KBr): $\nu = 3112, 2964, 2839, 1649, 1600, 1511, 1273, 1169, 1035, 951, 783, 713 \text{ cm}^{-1}$.

$^1\text{H NMR}$ (CDCl_3 , 300 MHz): $\delta = 1.23$ (d, $J = 6.6 \text{ Hz}$, 3H), 1.67 (s, 3H), 2.69 (d, $J = 6.6 \text{ Hz}$, 2H),

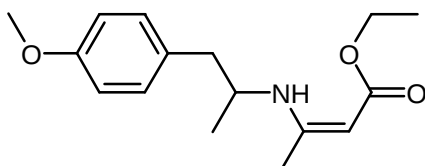
3.63 (s, 3H), 3.66-3.71 (m, 1H), 3.78 (s, 3H), 4.33 (s, 1H), 6.82 (d, $J = 8.4$ Hz, 2H), 7.07 (d, $J = 8.4$ Hz, 2H), 8.60 (br s, 1H, NH).

^{13}C NMR (CDCl_3 , 75 MHz): $\delta = 19.2, 21.9, 44.1, 49.8, 50.8, 55.2, 81.4, 113.8, 130.2, 130.4, 158.2, 161.1, 170.8$.

ESI-MS: 264 ($\text{M}+1$)⁺.

Anal. Calcd. for $\text{C}_{15}\text{H}_{21}\text{NO}_3$: C, 68.42; H, 8.04; N, 5.32; Found: C, 68.68; H, 8.26; N, 5.16.

Ethyl 3-[2-(4-Methoxy-phenyl)-1-methyl-ethylamino]-but-2-enoate (Table 2, entry 16)



A colorless liquid.

IR (neat): $\nu = 3271, 2973, 2835, 1650, 1608, 1513, 1443, 1268, 1174, 1056, 976, 784, 702\text{ cm}^{-1}$.

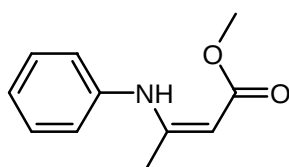
^1H NMR (CDCl_3 , 300 MHz): $\delta = 1.21$ (t, $J = 7.2$ Hz, 3H), 1.28 (d, $J = 7.2$ Hz, 3H), 1.68 (s, 3H), 2.69 (d, $J = 7.2$ Hz, 2H), 3.61-3.70 (m, 1H), 3.78 (s, 3H), 4.09 (q, $J = 7.2$ Hz, 2H), 4.33 (s, 1H), 6.81 (d, $J = 8.4$ Hz, 2H), 7.06 (d, $J = 8.4$ Hz, 2H), 8.60 (d, $J = 9.0$ Hz, 1H, NH).

^{13}C NMR (CDCl_3 , 75 MHz): $\delta = 14.6, 19.1, 21.8, 44.0, 50.6, 55.0, 58.0, 81.9, 113.7, 130.2, 130.3, 158.2, 160.8, 170.4$.

ESI-MS: $m/z = 278$ ($\text{M}+1$)⁺.

Anal. Calcd for $\text{C}_{16}\text{H}_{23}\text{NO}_3$: C, 69.29; H, 8.36; N, 5.05. Found: C, 69.11; H, 8.46; N, 5.28.

Methyl 3-(phenylamino)but-2-enoate (Table 2, entry 17)^[11]



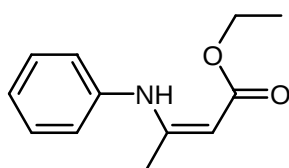
A pale yellow solid, mp 46-47 °C (Lit.^[11] mp 42-45 °C).

IR (KBr): $\nu = 3247, 2992, 1651, 1589, 1483, 1435, 1356, 1260, 1162, 1055, 786\text{ cm}^{-1}$.

^1H NMR (CDCl_3 , 300 MHz): $\delta = 1.99$ (s, 3H), 3.68 (s, 3H), 4.69 (s, 1H), 7.07-7.18 (m, 3H), 7.30-7.35 (m, 2H), 10.36 (br s, 1H, NH).

^{13}C NMR (CDCl_3 , 75 MHz): $\delta = 20.2, 50.2, 85.6, 124.4, 125.0, 129.0, 139.3, 159.0, 170.0$.

Ethyl 3-(phenylamino)but-2-enoate (Table 2, entry 18)^[5,12]

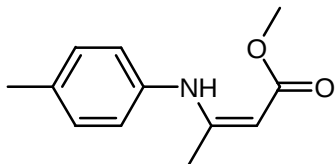


A yellowish oil.

IR (neat): ν = 3256, 2979, 1653, 1596, 1503, 1440, 1359, 1272, 1164, 1059, 788 cm^{-1} .

^1H NMR (CDCl_3 , 300 MHz): δ = 1.29 (t, J = 7.2 Hz, 3H), 2.00 (s, 3H), 4.15 (q, J = 7.2 Hz, 2H), 4.69 (s, 1H), 7.07-7.17 (m, 3H), 7.29-7.35 (m, 2H), 10.38 (br s, 1H, NH).

Methyl 3-(p-tolylamino)but-2-enoate (Table 2, entry 19)



A pale yellow solid, mp 57-58 $^{\circ}\text{C}$.

IR (KBr): ν = 3263, 2949, 1651, 1598, 1489, 1384, 1360, 1275, 1187, 1162, 1058, 913, 787 cm^{-1} .

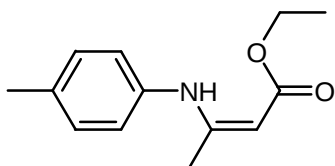
^1H NMR (CDCl_3 , 300 MHz): δ = 1.95 (s, 3H), 2.33 (s, 3H), 3.68 (s, 3H), 4.66 (s, 1H), 6.97 (d, J = 8.1 Hz, 2H), 7.12 (d, J = 8.1 Hz, 2H), 10.25 (br s, 1H, NH).

^{13}C NMR (CDCl_3 , 75 MHz): δ = 20.1, 20.8, 50.1, 85.0, 124.7, 129.6, 130.8, 136.6, 159.4, 170.7.

ESI-MS: m/z = 206 ($\text{M}+1$) $^{+}$.

Anal. Calcd for $\text{C}_{12}\text{H}_{15}\text{NO}_2$: C, 70.22; H, 7.37; N, 6.82. Found: C, 70.28; H, 7.43; N, 6.68.

Ethyl 3-(p-tolylamino)but-2-enoate (Table 2, entry 20)^[13]



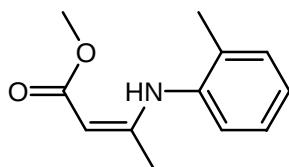
A yellowish oil.

IR (neat): ν = 3256, 2978, 1654, 1608, 1518, 1270, 1161, 1059, 787 cm^{-1} .

^1H NMR (CDCl_3 , 300 MHz): δ = 1.28 (t, J = 6.9 Hz, 3H), 1.95 (s, 3H), 2.32 (s, 3H), 4.14 (q, J = 6.9 Hz, 2H), 4.66 (s, 1H), 6.97 (d, J = 8.1 Hz, 2H), 7.12 (d, J = 8.1 Hz, 2H), 10.27 (br s, 1H, NH).

^{13}C NMR (CDCl_3 , 75 MHz): δ = 14.8, 20.1, 20.8, 58.6, 85.3, 124.7, 129.6, 134.8, 136.7, 159.3, 170.4.

Methyl 3-(o-tolylamino)but-2-enoate (Table 2, entry 21)^[14]



A pale yellow solid, mp 27-28 $^{\circ}\text{C}$.

IR (KBr): ν = 3442, 3111, 1598, 1402, 1269, 1164, 1058, 916, 786 cm^{-1} .

^1H NMR (CDCl_3 , 300 MHz): δ = 1.85 (s, 3H), 2.28 (s, 3H), 3.68 (s, 3H), 4.70 (s, 1H), 7.02-7.23 (m, 4H), 10.12 (br s, 1H, NH).

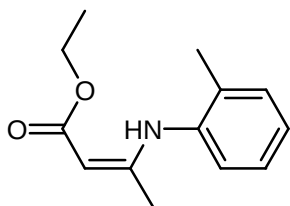
^{13}C NMR (CDCl_3 , 75 MHz): δ = 17.9, 19.9, 50.0, 84.8, 126.0, 126.3, 130.8, 133.9, 137.9, 159.9,

170.9.

ESI-MS: $m/z = 206$ ($M+1$)⁺.

Anal. Calcd for C₁₂H₁₅NO₂: C, 70.22; H, 7.37; N, 6.82. Found: C, 70.02; H, 7.56; N, 6.68.

Ethyl 3-(*o*-tolylamino)but-2-enoate (Table 2, entry 22)^[15]

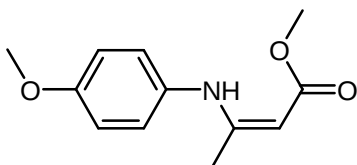


A yellowish oil.

IR (neat): $\nu = 3255, 2978, 1618, 1598, 1475, 1269, 1162, 1059, 978, 787$ cm⁻¹.

¹H NMR (CDCl₃, 300 MHz): $\delta = 1.29$ (t, $J = 6.9$ Hz, 3H), 1.85 (s, 3H), 2.28 (s, 3H), 4.15 (q, $J = 6.9$ Hz, 2H), 4.68 (s, 1H), 7.05-7.26 (m, 4H), 10.17 (br s, 1H, NH).

Methyl 3-(4-methoxyphenylamino)but-2-enoate (Table 2, entry 23)^[16]



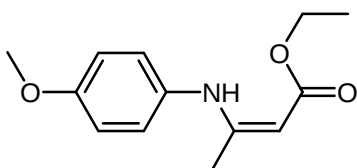
A yellowish oil.

IR (KBr): $\nu = 3270, 2943, 2844, 1701, 1597, 1515, 1486, 1435, 1243, 1162, 1029, 1008, 785$ cm⁻¹.

¹H NMR (CDCl₃, 300 MHz): $\delta = 1.88$ (s, 3H), 3.67 (s, 3H), 3.80 (s, 3H), 4.65 (s, 1H), 6.86 (d, $J = 8.7$ Hz, 2H), 7.02 (d, $J = 8.7$ Hz, 2H), 10.12 (br s, 1H, NH).

¹³C NMR (CDCl₃, 75 MHz): $\delta = 20.0, 50.1, 55.4, 84.3, 114.2, 128.8, 132.0, 157.5, 160.1, 170.7$.

Ethyl 3-(4-methoxyphenylamino)but-2-enoate (Table 2, entry 24)^[15,16]

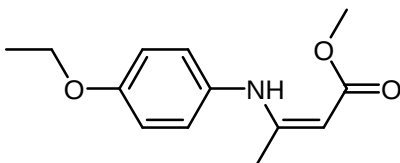


A pale yellow solid, mp 43-44 °C (Lit.^[15] mp 44.5-45 °C).

IR (KBr): $\nu = 32464, 2835, 1700, 1650, 1615, 1513, 1486, 1439, 1245, 1159, 1032, 977, 786$ cm⁻¹.

¹H NMR (CDCl₃, 300 MHz): $\delta = 1.28$ (t, $J = 6.9$ Hz, 3H), 1.88 (s, 3H), 3.80 (s, 3H), 4.14 (q, $J = 6.9$ Hz, 2H), 4.64 (s, 1H), 6.85 (d, $J = 8.7$ Hz, 2H), 7.03 (d, $J = 8.7$ Hz, 2H), 10.15 (br s, 1H, NH).

Methyl 3-(4-Ethoxy-phenylamino)-but-2-enoate (Table 2, entry 25)



A pale yellow solid, mp: 61-62 °C.

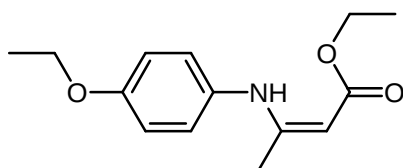
IR (KBr): ν = 3443, 3111, 2941, 1649, 1596, 1516, 1487, 1399, 1357, 1242, 1162, 1057, 1006, 951, 911 cm^{-1} .

^1H NMR (CDCl_3 , 300 MHz): δ = 1.43 (t, J = 7.2 Hz, 3H), 1.91 (s, 3H), 3.70 (s, 3H), 4.03 (q, J = 7.2 Hz, 2H), 4.66 (s, 1H), 6.86 (d, J = 8.7 Hz, 2H), 7.03 (d, J = 8.7 Hz, 2H), 10.14 (br s, 1H, NH).

^{13}C NMR (CDCl_3 , 75 MHz): δ = 14.8, 20.0, 50.1, 63.6, 84.2, 114.7, 126.8, 131.9, 156.9, 160.1, 170.7. ESI-MS: m/z = 236 ($\text{M}+1$)⁺.

Anal. Calcd for $\text{C}_{13}\text{H}_{17}\text{NO}_3$: C, 66.36; H, 7.28; N, 5.95. Found: C, 66.50; H, 7.02; N, 5.82.

Ethyl 3-(4-ethoxyphenylamino)but-2-enoate (Table 2, entry 26)^[17]



A pale yellow solid, mp 53-54 °C (Lit.^[17] mp 54-54.5 °C).

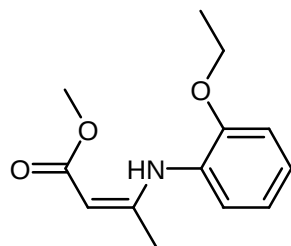
IR (KBr): ν = 3417, 2977, 1651, 1612, 1513, 1438, 1390, 1246, 1159, 1116, 1047, 923, 785 cm^{-1} .

^1H NMR (CDCl_3 , 300 MHz): δ = 1.28 (t, J = 6.9 Hz, 3H), 1.41 (t, J = 6.9 Hz, 3H), 1.88 (s, 3H), 4.01 (q, J = 6.9 Hz, 2H), 4.14 (q, J = 6.9 Hz, 2H), 4.64 (s, 1H), 6.84 (d, J = 8.7 Hz, 2H), 7.01 (d, J = 8.7 Hz, 2H), 10.14 (br s, 1H, NH).

^{13}C NMR (CDCl_3 , 75 MHz): δ = 14.6, 14.8, 20.0, 58.6, 63.6, 84.6, 114.7, 126.8, 132.0, 156.8, 160.0, 170.5.

ESI-MS: m/z = 250 ($\text{M}+1$)⁺.

Methyl 3-(2-ethoxyphenylamino)but-2-enoate (Table 2, entry 27)



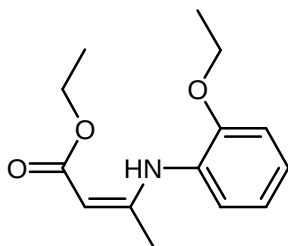
A pale yellow solid, mp 46-47 °C.

IR (KBr): ν = 3413, 3184, 2947, 1658, 1622, 1582, 1514, 1483, 1453, 1271, 1241, 1209, 1165, 1039, 1008, 925, 848, 747 cm^{-1} .

^1H NMR (CDCl_3 , 300 MHz): δ = 1.45 (t, J = 6.9 Hz, 3H), 2.01 (s, 3H), 3.68 (s, 3H), 4.08 (q, J = 6.9 Hz, 2H), 4.70 (s, 1H), 6.87-6.92 (m, 2H), 7.06-7.11 (m, 2H), 10.28 (br s, 1H, NH).

^{13}C NMR (CDCl_3 , 75 MHz): δ = 14.7, 20.4, 50.1, 64.3, 85.8, 112.5, 120.3, 124.3, 125.3, 129.1, 151.9, 159.0, 170.3.

Anal. Calcd for $\text{C}_{13}\text{H}_{17}\text{NO}_3$: C, 66.36; H, 7.28; N, 5.95. Found: C, 66.23; H, 7.42; N, 5.81.

Ethyl 3-(2-ethoxyphenylamino)but-2-enoate (Table 2, entry 28)

A pale yellow solid, mp 42-43 °C.

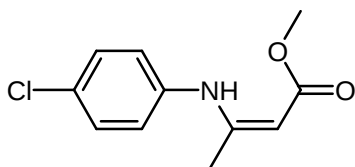
IR (KBr): ν = 3417, 2980, 1652, 1621, 1581, 1510, 1475, 1272, 1166, 1117, 1039, 917, 792 cm^{-1} .

^1H NMR (CDCl_3 , 300 MHz): δ = 1.28 (t, J = 6.9 Hz, 3H), 1.44 (t, J = 6.9 Hz, 3H), 2.01 (s, 3H), 4.07 (q, J = 6.9 Hz, 2H), 4.15 (q, J = 6.9 Hz, 2H), 4.69 (s, 1H), 6.85-6.90 (m, 2H), 7.05-7.10 (m, 2H), 10.29 (br s, 1H, NH).

^{13}C NMR (CDCl_3 , 75 MHz): δ = 14.6, 14.7, 20.4, 58.5, 64.3, 86.3, 112.5, 120.3, 124.2, 125.2, 129.1, 151.9, 158.8, 170.0.

ESI-MS: 250 ($\text{M}+1$) $^+$.

Anal. Calcd. for $\text{C}_{14}\text{H}_{19}\text{NO}_3$: C, 67.45; H, 7.68; N, 5.62. Found: C, 67.59; H, 7.48; N, 5.71.

Methyl 3-(4-chlorophenylamino)but-2-enoate (Table 2, entry 29)

A pale yellow solid, mp 60-61 °C.

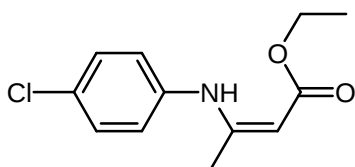
IR (KBr): ν = 3273, 2949, 1653, 1592, 1487, 1352, 1270, 1166, 1059, 941, 787, 702 cm^{-1} .

^1H NMR (CDCl_3 , 300 MHz): δ = 1.98 (s, 3H), 3.68 (s, 3H), 4.72 (s, 1H), 7.01 (d, J = 8.4 Hz, 2H), 7.28 (d, J = 8.4 Hz, 2H), 10.33 (br s, 1H, NH).

^{13}C NMR (CDCl_3 , 75 MHz): δ = 20.1, 50.3, 86.4, 125.5, 129.1, 137.9, 158.5, 162.3, 170.6.

ESI-MS: 226 ($\text{M}+1$) $^+$.

Anal. Calcd. for $\text{C}_{11}\text{H}_{12}\text{ClNO}_2$: C, 58.54; H, 5.36; N, 6.21. Found: C, 58.38; H, 5.51; N, 6.38.

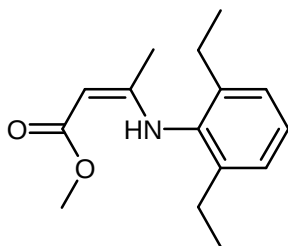
Ethyl 3-(4-chlorophenylamino)but-2-enoate (Table 2, entry 30)^[13]

A pale yellow solid, mp 54-55 °C (Lit.^[13] mp 53-54 °C).

IR (KBr): ν = 3451, 2984, 1652, 1608, 1499, 1481, 1387, 1267, 1161, 1057, 971, 788, 681 cm^{-1} .

^1H NMR (CDCl_3 , 300 MHz): δ = 1.28 (t, J = 6.9 Hz, 3H), 1.98 (s, 3H), 4.14 (q, J = 6.9 Hz, 2H), 4.71 (s, 1H), 7.01 (d, J = 8.7 Hz, 2H), 7.28 (d, J = 8.7 Hz, 2H), 10.35 (br s, 1H, NH).

Methyl 3-(2,6-Diethyl-phenylamino)-but-2-enoate (Table 3, entry 31)



A pale yellow solid, mp 26-27 °C.

IR (KBr): ν = 3263, 2959, 1658, 1607, 1464, 1271, 1160, 1057, 912, 782 cm^{-1} .

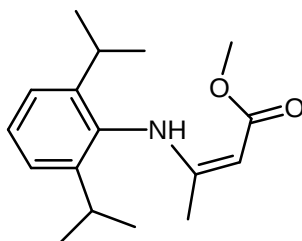
^1H NMR (CDCl_3 , 300 MHz): δ = 1.18 (t, J = 7.5 Hz, 6H), 1.61 (s, 3H), 2.51 (q, J = 7.5 Hz, 2H), 2.62 (q, J = 7.5 Hz, 2H), 3.70 (s, 3H), 4.86 (s, 1H), 7.12 (d, J = 7.8 Hz, 2H), 7.12-7.19 (m, 1H), 9.86 (br s, 1H, NH).

^{13}C NMR (CDCl_3 , 75 MHz): δ = 14.5, 19.4, 24.7, 50.0, 82.9, 126.4, 127.8, 135.5, 142.5, 161.7, 171.0.

ESI-MS: 248 ($\text{M}+1$) $^+$.

Anal. Calcd. for $\text{C}_{15}\text{H}_{21}\text{NO}_2$: C, 72.84; H, 8.56; N, 5.66. Found: C, 72.99; H, 8.47; N, 5.48.

Methyl 3-(2,6-diisopropylphenylamino)but-2-enoate (Table 2, entry 32)^[18]



A white crystalline solid, mp 129-130 °C.

IR (KBr): ν = 3246, 2958, 1657, 1606, 1489, 1443, 1316, 1266, 1154, 1056, 913, 806, 697 cm^{-1} .

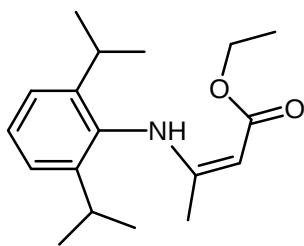
^1H NMR (CDCl_3 , 300 MHz): δ = 1.15 (d, J = 6.6 Hz, 6H), 1.22 (d, J = 6.6 Hz, 6H), 1.61 (s, 3H), 3.04-3.14 (m, 2H), 3.70 (s, 3H), 4.68 (s, 1H), 7.16 (d, J = 7.8 Hz, 2H), 7.29-7.32 (m, 1H), 9.83 (br s, 1H, NH).

^{13}C NMR (CDCl_3 , 75 MHz): δ = 19.6, 22.6, 24.6, 28.3, 50.1, 82.7, 123.4, 128.1, 133.8, 147.0, 162.0, 171.0.

ESI-MS: 276 ($\text{M}+1$) $^+$.

Anal. Calcd. for $\text{C}_{17}\text{H}_{25}\text{NO}_2$: C, 74.14; H, 9.15; N, 5.09. Found: C, 74.35; H, 9.27; N, 4.96.

Ethyl 3-(2,6-diisopropylphenylamino)but-2-enoate (Table 2, entry 33)



A white crystalline solid, mp 81-82 °C.

IR (KBr): ν = 3263, 3064, 2962, 1612, 1488, 1456, 1380, 1320, 1061, 977, 782 cm^{-1} .

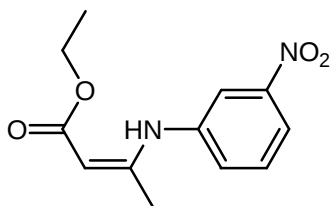
^1H NMR (CDCl_3 , 300 MHz): δ = 1.15 (d, J = 6.6 Hz, 6H), 1.22 (d, J = 6.6 Hz, 6H), 1.30 (t, J = 7.2 Hz, 3H), 1.63 (s, 3H), 3.04-3.13 (m, 2H), 4.17 (q, J = 7.2 Hz, 2H), 4.68 (s, 1H), 7.16 (d, J = 7.8 Hz, 2H), 7.29-7.32 (m, 1H), 9.99 (br s, 1H, NH).

^{13}C NMR (CDCl_3 , 75 MHz): δ = 14.6, 19.5, 22.5, 24.6, 28.3, 58.5, 83.1, 123.4, 128.1, 133.9, 147.0, 162.3, 170.7.

ESI-MS: 290 ($\text{M}+1$) $^+$.

Anal. Calcd. for $\text{C}_{18}\text{H}_{27}\text{NO}_2$: C, 74.70; H, 9.40; N, 4.84. Found: C, 74.91; H, 9.57; N, 4.62.

Ethyl 3-(3-nitrophenylamino)but-2-enoate (Table 2, entry 34)^[16]

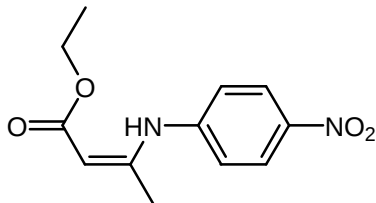


A yellow solid, mp 52-53 °C (Lit.^[16] mp 48-50 °C).

IR (KBr): ν = 3320, 3104, 2980, 1650, 1613, 1531, 1347, 1286, 1234, 1174, 1092, 1055, 1025, 981, 880, 784 cm^{-1}

^1H NMR (CDCl_3 , 300 MHz): δ = 1.28 (t, J = 6.9 Hz, 3H), 2.11 (s, 3H), 4.16 (q, J = 6.9 Hz, 2H), 4.82 (s, 1H), 7.35-7.51 (m, 2H), 7.94-7.98 (m, 2H), 10.67 (br s, 1H, NH).

Ethyl 3-(4-nitrophenylamino)but-2-enoate (Table 2, entry 35)^[16]

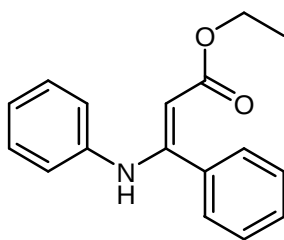


A yellow solid, mp 111-112 °C (Lit.^[16] mp 109-110 °C).

IR (KBr): ν = 3306, 3097, 2992, 1652, 1592, 1509, 1334, 1287, 1179, 1111, 1020, 836, 791 cm^{-1}

^1H NMR (CDCl_3 , 300 MHz): δ = 1.30 (t, J = 7.2 Hz, 3H), 2.21 (s, 3H), 4.21 (q, J = 7.2 Hz, 2H), 4.87 (s, 1H), 7.12 (d, J = 9.0 Hz, 2H), 8.19 (d, J = 9.0 Hz, 2H), 10.93 (br s, 1H, NH).

Ethyl 3-phenyl-3-(phenylamino)acrylate (Table 2, entry 36)^[19]

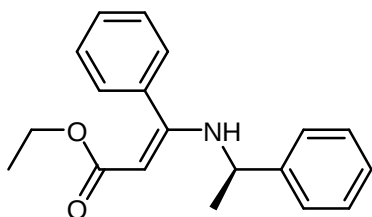


A pale yellow solid, mp 65-66 °C (Lit.^[19] mp 64-69 °C).

IR (neat): ν = 3260, 2994, 1654, 1592, 1497, 1437, 1314, 1173, 1033, 918, 700 cm^{-1} .

¹HNMR(CDCl₃, 300 MHz): δ = 1.31 (t, J = 7.2 Hz, 3H), 4.21 (q, J = 7.2 Hz, 2H), 4.99 (s, 1H), 6.64-6.67 (m, 2H), 6.87-6.93 (m, 2H), 7.04-7.10 (m, 1H), 7.28-7.35 (m, 5H), 10.30 (br s, 1H, NH).

Ethyl 3-((R)-1-phenylethylamino)-3-phenylacrylate (Table 2, entry 37)^[4]

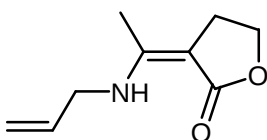


A colorless liquid. $[\alpha]_D^{20}$: - 332 (c 1.21, EtOH).

IR (neat): ν = 3276, 2976, 1653, 1613, 1480, 1363, 1174, 1040, 920, 769, 699 cm^{-1} .

¹HNMR(CDCl₃, 300 MHz): δ = 1.29 (t, J = 7.2 Hz, 3H), 1.46 (d, J = 6.9 Hz, 3H), 4.17 (q, J = 7.2 Hz, 2H), 4.38-4.48 (dq, J = 9.3, 6.9 Hz, 1H), 4.62 (s, 1H), 7.01-7.25 (m, 7H), 7.28-7.37 (m, 3H), 8.92 (br, d, J = 9.3 Hz, 1H).

3-(1-Allylamino-ethylidene)-dihydro-furan-2-one (Table 2, entry 38)^[20]



A pale yellow solid, mp 59-60 °C.

IR (KBr): ν = 3519, 3004, 2866, 1680, 1620, 1477, 1374, 1229, 1152, 1096, 1025, 977, 768, 706 cm^{-1} .

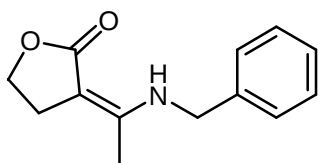
¹HNMR(CDCl₃, 300 MHz): δ = 1.92 (s, 3H), 2.84 (t, J = 7.8 Hz, 2H), 3.80-3.84 (m, 2H), 4.28 (t, J = 7.8 Hz, 2H), 5.10-5.25 (m, 2H), 5.79-5.92 (m, 1H), 8.36 (br s, 1H, NH).

¹³C NMR: δ = 16.0, 26.4, 45.1, 65.1, 85.5, 115.9, 134.9, 156.9, 174.0.

ESI-MS: m/z = 168 ($M+1$)⁺.

Anal. Calcd for C₉H₁₃NO₂: C, 64.65; H, 7.84; N, 8.38. Found: C, 64.40; H, 7.98; N, 8.21.

3-(1-(Benzylamino)ethylidene)-dihydrofuran-2(3H)-one (Table 2, entry 39)^[12,21]

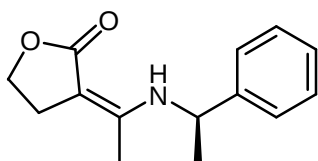


A pale yellow solid, mp 106-107 °C (Lit.^[12] mp 119-120 °C).

IR (KBr): ν = 3305, 2985, 1687, 1625, 1477, 1452, 1367, 1237, 1139, 1082, 979, 767 cm^{-1} .

¹H NMR (CDCl_3 , 300 MHz): δ = 1.90 (s, 3H), 2.84 (t, J = 7.8 Hz, 2H), 4.29 (t, J = 7.8 Hz, 2H), 4.43 (d, J = 6.0 Hz, 2H), 7.25-7.33 (m, 5H), 8.67 (br s, 1H, NH).

(R)-3-[1-(1-Phenyl-ethylamino)-ethylidene]-dihydro-furan-2-one (Table 2, entry 40)^[22]



A pale yellow solid, mp 71-73 °C (Lit.^[22] mp 73-74 °C); $[\alpha]_D^{20}$: -506 (c 1.22, EtOH).

IR (KBr): ν = 3435, 2957, 1687, 1607, 1454, 1408, 1371, 1223, 1100, 961, 748 cm^{-1} .

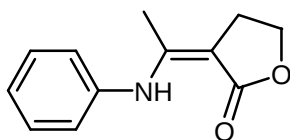
¹H NMR (CDCl_3 , 300 MHz): δ = 1.51 (d, J = 6.6 Hz, 3H), 1.77 (s, 3H), 2.79 (t, J = 7.8 Hz, 2H), 4.27 (t, J = 7.8 Hz, 2H), 4.62 (quin, J = 6.9 Hz, 1H), 7.23-7.35 (m, 5H), 8.62 (br s, 1H, NH).

¹³C NMR (CDCl_3 , 75 MHz): δ = 16.7, 24.8, 26.3, 52.9, 65.1, 86.1, 125.4, 127.1, 128.8, 145.0, 156.4, 174.0.

ESI-MS: 232 ($M+1$)⁺.

Anal. Calcd. for $\text{C}_{14}\text{H}_{17}\text{NO}_2$: C, 72.70; H, 7.41; N, 6.06. Found: C, 72.94; H, 7.63; N, 5.95.

3-(1-(Phenylamino)ethylidene)-dihydrofuran-2(3H)-one (Table 2, entry 41)^[10,12]

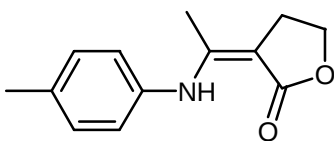


A pale yellow solid, mp 93-94 °C (Lit.^[10] mp 94-95 °C).

IR (KBr): ν = 3278, 2982, 1684, 1639, 1505, 1480, 1437, 1297, 1118, 1025, 947, 761 cm^{-1} .

¹H NMR (CDCl_3 , 300 MHz): δ = 2.02 (s, 3H), 2.91 (t, J = 7.8 Hz, 2H), 4.35 (t, J = 7.8 Hz, 2H), 7.04-7.17 (m, 3H), 7.30-7.35 (m, 2H), 9.98 (br s, 1H, NH).

3-(1-(4-Toluidino)ethylidene)-dihydrofuran-2(3H)-one (Table 2, entry 42)^[21]



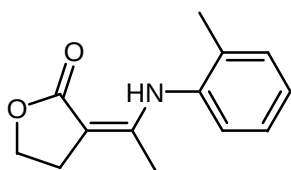
A pale yellow solid, mp 90-92 °C (Lit.^[21] mp 92-94 °C).

IR (KBr): ν = 3473, 2983, 1679, 1643, 1519, 1372, 1299, 1120, 1025, 962, 764 cm^{-1} .

¹H NMR (CDCl_3 , 300 MHz): δ = 1.97 (s, 3H), 2.33 (s, 3H), 2.90 (t, J = 7.8 Hz, 2H), 4.34 (t, J = 7.8

Hz, 2H), 6.95 (d, $J = 7.8$ Hz, 1H), 7.12 (d, $J = 7.8$ Hz, 1H), 9.89 (br s, 1H, NH).

3-(1-2-Tolylamino-ethylidene)-dihydro-furan-2-one (Table 2, entry 43)^[23]



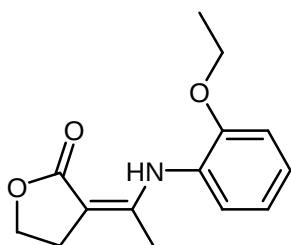
A pale yellow solid, mp 71-72 °C.

IR (KBr): $\nu = 3417, 2909, 1682, 1637, 1597, 1509, 1457, 1370, 1287, 1255, 1122, 1028, 968, 746$ cm^{-1} .

^1H NMR (CDCl_3 , 300 MHz): $\delta = 1.89$ (s, 3H), 2.28 (s, 3H), 2.91 (t, $J = 7.8$ Hz, 2H), 4.36 (t, $J = 7.8$ Hz, 2H), 7.01-7.23 (m, 4H), 9.78 (br s, 1H, NH).

Anal. Calcd for $\text{C}_{13}\text{H}_{15}\text{NO}_2$: C, 71.87; H, 6.96; N, 6.45. Found: C, 71.98; H, 6.66; N, 6.35.

3-(1-(2-Ethoxyphenylamino)ethylidene)-dihydrofuran-2(3H)-one (Table 2, entry 44)



A pale yellow solid, mp 115-116 °C.

IR (KBr): $\nu = 3517, 2989, 1689, 1624, 1513, 1285, 1117, 1024, 965, 766$ cm^{-1} .

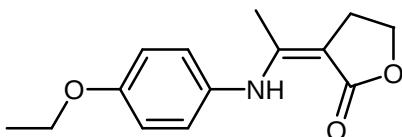
^1H NMR (CDCl_3 , 300 MHz): $\delta = 1.44$ (t, $J = 6.9$ Hz, 3H), 2.04 (s, 3H), 2.91 (t, $J = 7.8$ Hz, 2H), 4.07 (q, $J = 6.9$ Hz, 2H), 4.34 (t, $J = 7.8$ Hz, 2H), 6.86-7.09 (m, 4H), 10.00 (br s, 1H, NH).

^{13}C NMR (CDCl_3 , 75 MHz): $\delta = 14.7, 17.8, 26.6, 64.3, 65.1, 89.3, 112.5, 120.3, 123.7, 125.0, 128.9, 151.8, 153.4, 173.6$.

ESI-MS: 248 ($\text{M}+1$)⁺.

Anal. Called. for $\text{C}_{14}\text{H}_{17}\text{NO}_3$: C, 68.00; H, 6.93; N, 5.66. Found: C, 68.20; H, 7.02; N, 5.51.

3-(1-(4-ethoxyphenylamino)ethylidene)-dihydrofuran-2(3H)-one (Table 2, entry 45)



A pale yellow solid, mp 66-68 °C.

IR (KBr): $\nu = 3526, 2976, 1683, 1628, 1513, 1475, 1228, 1114, 1029, 947, 822, 763$ cm^{-1} .

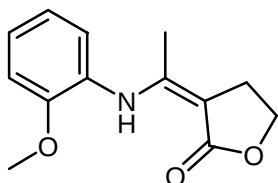
^1H NMR (CDCl_3 , 300 MHz): $\delta = 1.41$ (t, $J = 6.9$ Hz, 3H), 1.91 (s, 3H), 2.89 (t, $J = 7.8$ Hz, 2H), 4.02 (q, $J = 6.9$ Hz, 2H), 4.34 (t, $J = 7.8$ Hz, 2H), 6.84 (d, $J = 7.8$ Hz, 1H), 6.99 (d, $J = 7.8$ Hz, 1H), 9.77 (br s, 1H, NH).

^{13}C NMR (CDCl_3 , 75 MHz): δ = 14.7, 17.4, 26.4, 63.7, 65.2, 87.6, 114.8, 126.6, 131.8, 154.6, 156.8, 173.9.

ESI-MS: 248 ($\text{M}+1$) $^+$.

Anal. Calcd. for $\text{C}_{14}\text{H}_{17}\text{NO}_3$: C, 68.00; H, 6.93; N, 5.66. Found: C, 68.12; H, 7.05; N, 5.79.

3-(1-(2-methoxyphenylamino)ethylidene)-dihydrofuran-2(3H)-one (Table 2, entry 46)



A pale yellow solid, mp 92-93 $^{\circ}\text{C}$.

IR (KBr): ν = 3455, 2932, 1678, 1636, 1517, 1481, 1294, 1173, 1034, 962, 844, 767 cm^{-1} .

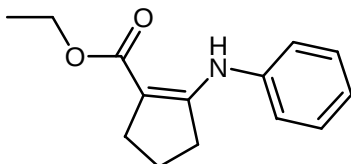
^1H NMR (CDCl_3 , 300 MHz): δ = 1.91 (s, 3H), 2.89 (t, J = 7.8 Hz, 2H), 3.80 (s, 3H), 4.34 (t, J = 7.8 Hz, 2H), 6.86 (d, J = 7.8 Hz, 1H), 7.00 (d, J = 7.8 Hz, 1H), 9.78 (br s, 1H, NH).

^{13}C NMR (CDCl_3 , 75 MHz): δ = 17.4, 26.4, 55.4, 65.3, 87.8, 114.3, 126.6, 131.9, 154.6, 157.4, 173.9.

ESI-MS: 234 ($\text{M}+1$) $^+$.

Anal. Calcd. for $\text{C}_{13}\text{H}_{15}\text{NO}_3$: C, 66.94; H, 6.48; N, 6.00. Found: C, 66.81; H, 6.36; N, 5.91.

Ethyl 2-(phenylamino)cyclopent-1-enecarboxylate (Table 2, entry 47)^[12]



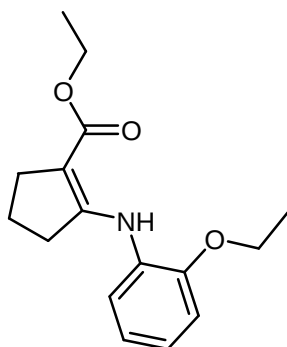
A yellowish oil.

IR (KBr): ν = 3284, 2956, 1655, 1623, 1503, 1479, 1263, 1171, 1048, 753 cm^{-1} .

^1H NMR (CDCl_3 , 300 MHz): δ = 1.30 (t, J = 7.2 Hz, 3H), 1.82-1.91 (m, 2H), 2.56 (t, J = 7.5 Hz, 2H), 2.80 (t, J = 7.2 Hz, 2H), 4.20 (q, J = 7.2 Hz, 2H), 7.02-7.29 (m, 5H), 9.59 (br s, 1H, NH).

^{13}C NMR (CDCl_3 , 75 MHz): δ = 14.6, 21.7, 28.7, 33.6, 58.9, 97.7, 120.6, 123.0, 129.1, 140.7, 160.2, 168.4.

Ethyl 2-(2-ethoxyphenylamino)cyclopent-1-enecarboxylate (Table 2, entry 48)^[24]



A pale yellow solid, mp 69-70 °C

IR (KBr): ν = 3413, 3184, 2975, 1658, 1622, 1582, 1514, 1483, 1272, 1120, 1008, 925, 747 cm^{-1} .

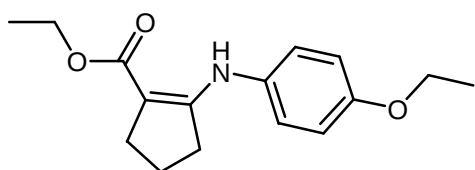
^1H NMR (CDCl_3 , 300 MHz): δ = 1.30 (t, J = 7.2 Hz, 3H), 1.49 (t, J = 6.9 Hz, 3H), 1.83-1.90 (m, 2H), 2.57 (t, J = 7.5 Hz, 2H), 2.87 (t, J = 7.2 Hz, 2H), 4.10 (q, J = 6.9 Hz, 2H), 4.22 (q, J = 7.2 Hz, 2H), 6.82-6.96 (m, 3H), 7.11 (d, J = 6.9 Hz, 1H), 9.73 (br s, 1H, NH).

^{13}C NMR (CDCl_3 , 75 MHz): δ = 14.7, 14.8, 21.8, 28.9, 33.9, 58.8, 64.3, 98.5, 112.6, 118.9, 120.5, 122.5, 130.9, 159.0, 162.3, 167.9.

ESI-MS: 276 ($\text{M}+1$) $^+$.

Anal. Calcd. for $\text{C}_{16}\text{H}_{21}\text{NO}_3$: C, 69.79; H, 7.69; N, 5.09. Found: C, 69.51; H, 7.83; N, 5.21.

Ethyl 2-(4-ethoxy-phenylamino)-cyclopent-1-enecarboxylate (Table 2, entry 49)



A pale yellow solid, mp: 50-52 °C

IR (KBr): ν = 3266, 2973, 2924, 1651, 1620, 1578, 1516, 1470, 1388, 1361, 1308, 1270, 1171, 1117, 1092, 1048, 922, 771 cm^{-1} .

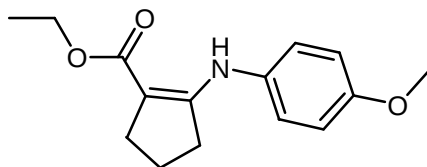
^1H NMR (CDCl_3 , 300 MHz): δ = 1.30 (t, J = 7.2 Hz, 3H), 1.40 (t, J = 7.2 Hz, 3H), 1.78-1.89 (m, 2H), 2.56 (t, J = 6.9 Hz, 2H), 2.64 (t, J = 6.9 Hz, 2H), 4.00 (q, J = 7.2 Hz, 2H), 4.19 (q, J = 7.2 Hz, 2H), 6.82 (d, J = 8.7 Hz, 2H), 6.97 (d, J = 8.7 Hz, 2H), 9.28 (br s, 1H, NH).

^{13}C NMR (CDCl_3 , 75 MHz): 14.7, 21.6, 28.9, 33.2, 55.4, 58.7, 63.6, 95.1, 114.2, 123.6, 133.7, 156.3, 161.6, 168.4.

ESI-MS: m/z = 276 ($\text{M}+1$) $^+$.

Anal. Calcd for $\text{C}_{16}\text{H}_{21}\text{NO}_3$: C, 69.79; H, 7.69; N, 5.09. Found: C, 69.68; H, 7.56; N, 5.32.

Ethyl 2-(4-methoxy-phenylamino)-cyclopent-1-enecarboxylate (Table 2, entry 50)^[25]

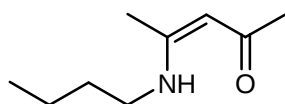


A pale yellow solid, mp 52-53 °C (Lit.^[25] mp 54 °C).

IR (KBr): ν = 3266, 2954, 1623, 1579, 1516, 1461, 1245, 1173, 1148, 1046, 822, 691 cm^{-1} .

^1H NMR (CDCl_3 , 300 MHz): δ = 1.30 (t, J = 7.2 Hz, 3H), 1.80-1.88 (m, 2H), 2.56 (t, J = 7.2 Hz, 2H), 2.64 (t, J = 7.2 Hz, 2H), 3.78 (s, 3H), 4.19 (q, J = 7.2 Hz, 2H), 6.83 (d, J = 8.7 Hz, 2H), 6.99 (d, J = 8.7 Hz, 2H), 9.30 (br s, 1H, NH).

4-(Butylamino)pent-3-en-2-one (Table 3, entry 1)^[1]

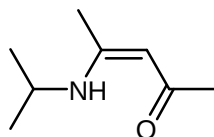


A yellowish oil.

IR (neat): ν = 3291, 2959, 1611, 1578, 1516, 1439, 1298, 736 cm^{-1} .

^1H NMR (CDCl_3 , 300 MHz): δ = 0.94 (t, J = 7.2 Hz, 3H), 1.37-1.45 (m, 2H), 1.56-1.60 (m, 2H), 1.92 (s, 3H), 2.08 (s, 3H), 3.20-3.27 (m, 2H), 4.95 (s, 1H), 10.88 (br s, 1H, NH).

4-(isopropylamino)pent-3-en-2-one (Table 3, entry 2)^[26]

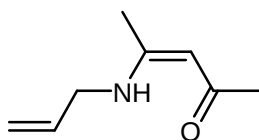


A yellowish oil.

IR (neat): ν = 3443, 2972, 1610, 1578, 1513, 1440, 1305, 1167, 738 cm^{-1} .

^1H NMR (CDCl_3 , 300 MHz): δ = 1.23 (d, J = 6.6 Hz, 6H), 1.95 (s, 3H), 1.99 (s, 3H), 3.68-3.75 (m, 1H), 4.91 (s, 1H), 10.83 (br s, 1H, NH).

4-(Allylamino)pent-3-en-2-on (Table 3, entry 3)^[5]

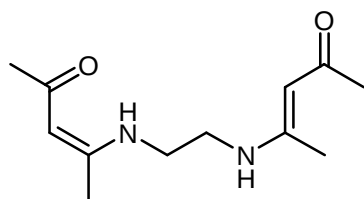


A yellowish oil.

IR (neat): ν = 3166, 3083, 2920, 1610, 1575, 1515, 1439, 1301, 1239, 1027, 742 cm^{-1} .

^1H NMR (CDCl_3 , 300 MHz): δ = 1.91 (s, 3H), 2.01 (s, 3H), 3.85-3.89 (m, 2H), 5.00 (s, 1H), 5.15-5.25 (m, 2H), 5.80-5.92 (m, 1H), 10.87 (br s, 1H, NH).

4-[2-(1-Methyl-3-oxo-but-1-enylamino)-ethylamino]-pent-3-en-2-one (Table 3, entry 4)^[5]



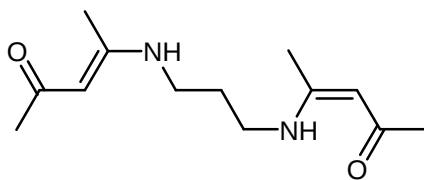
A pale yellow crystalline solid, mp: 112-113 $^{\circ}\text{C}$ (Lit.^[5] mp 105 $^{\circ}\text{C}$).

IR (KBr): ν = 3399, 1608, 1570, 1438, 1356, 1296, 1136, 1020, 746 cm^{-1} .

^1H NMR (CDCl_3 , 300 MHz): δ = 1.91 (s, 6H), 2.00 (s, 6H), 3.40-3.43 (m, 4H), 5.00 (s, 2H), 10.90 (br s, 2H, NH).

^{13}C NMR (CDCl_3 , 75 MHz): δ = 18.6, 28.8, 43.5, 96.1, 162.7, 195.5.

4-[3-(1-Methyl-3-oxo-but-1-enylamino)-propylamino]-pent-3-en-2-one (Table 3, entry 5)^[27]



A straw-colored solid, mp 51-52 °C (Lit.^[27] mp 51 °C).

IR (KBr): ν = 3399, 2956, 1608, 1570, 1438, 1366, 1296, 1138, 1020, 746 cm^{-1} .

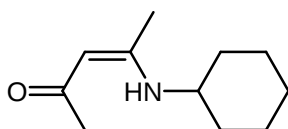
^1H NMR (CDCl_3 , 300 MHz): δ = 1.88 (t, J = 6.3 Hz, 2H), 1.93 (s, 6H), 2.00 (s, 6H), 3.37 (q, J = 6.3 Hz, 4H), 4.99 (s, 2H), 10.89 (br s, 1H, NH).

^{13}C NMR (CDCl_3 , 75 MHz): δ = 18.7, 28.8, 30.3, 39.6, 95.8, 163.2, 195.3.

ESI-MS: 239 ($\text{M}+1$)⁺.

Anal. Calcd. for $\text{C}_{13}\text{H}_{22}\text{N}_2\text{O}_2$: C, 65.51; H, 9.30; N, 11.75. Found: C, 65.78; H, 9.52; N, 11.60.

4-Cyclohexylamino-pent-3-en-2-one (Table 3, entry 6)^[6]



A yellowish oil.

IR (neat): ν = 3444, 2931, 2854, 1610, 1578, 1511, 1443, 1376, 736 cm^{-1} .

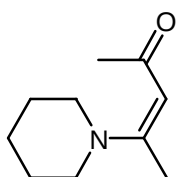
^1H NMR (CDCl_3 , 300 MHz): δ = 1.31-1.59 (m, 6H), 1.75-1.98 (m, 4H), 1.94 (s, 3H), 1.99 (s, 3H), 3.36 (t, J = 4.5 Hz, 1H), 4.91 (s, 1H), 10.99 (br s, 1H, NH).

^{13}C NMR (CDCl_3 , 75 MHz): δ = 18.4, 24.2, 25.2, 28.5, 33.6, 51.3, 94.8, 161.6, 194.1.

ESI-MS: 182 ($\text{M}+1$)⁺.

Anal. Calcd. for $\text{C}_{11}\text{H}_{19}\text{NO}$: C, 72.88; H, 10.56; N, 7.73. Found: C, 72.95; H, 10.70; N, 7.61.

4-(Piperidin-1-yl)pent-3-en-2-one (Table 3, entry 7)^[28]



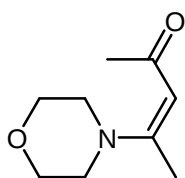
A pale yellow solid, mp: 45-46 °C (Lit.^[28] mp 47-48 °C)

IR (KBr): ν = 2931, 2857, 1536, 1436, 1254, 1181, 1054, 1019, 956, 784 cm^{-1} .

^1H NMR (CDCl_3 , 300 MHz): δ = 1.58-1.66 (m, 6H), 2.07 (s, 3H), 2.50 (s, 3H), 3.34 (t, J = 4.8 Hz, 4H), 5.21 (s, 1H).

^{13}C NMR (CDCl_3 , 75 MHz): δ = 15.6, 24.3, 25.5, 31.8, 47.3, 95.4, 160.8, 195.0.

4-Morpholinopent-3-en-2-one (Table 3, entry 8)^[28]

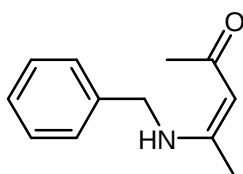


A pale yellow solid, mp 45-47 °C (Lit.^[28] mp 46-47 °C).

IR (KBr): ν = 2963, 2956, 1539, 1431, 1256, 1194, 1122, 1002, 956, 785 cm^{-1} .

¹H NMR (CDCl_3 , 300 MHz): δ = 2.09 (s, 3H), 2.47 (s, 3H), 3.30 (t, J = 4.5 Hz, 4H), 3.73 (t, J = 4.5 Hz, 4H), 5.24 (s, 1H).

4-(Benzylamino)pent-3-en-2-one (Table 3, entry 9)^[12]

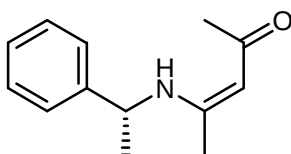


A pale yellow solid, mp 23-24 °C.

IR (KBr): ν = 3431, 3029, 1610, 1574, 1514, 1437, 1354, 1294, 1102, 1027, 986, 735, 697 cm^{-1} .

¹H NMR (CDCl_3 , 300 MHz): δ = 1.91 (s, 3H), 2.03 (s, 3H), 4.45 (d, J = 6.0 Hz, 2H), 5.04 (s, 1H), 7.31-7.36 (m, 5H), 11.17 (br s, 1H, NH).

(R)-4-(1-phenylethylamino)pent-3-en-2-one (Table 3, entry 10)^[29]



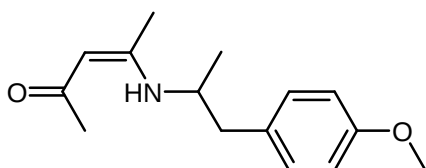
A yellowish oil; $[\alpha]_D^{20}$: -848 (c 0.68, EtOH).

IR (neat): ν = 3443, 2970, 2927, 1611, 1576, 1507, 1439, 1354, 1294, 1139, 1018, 850, 744, 701 cm^{-1} .

¹H NMR (CDCl_3 , 300 MHz): δ = 1.53 (d, J = 6.9 Hz, 3H), 1.78 (s, 3H), 2.03 (s, 3H), 4.61-4.70 (m, 1H), 4.98 (s, 1H), 7.23-7.35 (m, 5H), 11.25 (br s, 1H, NH).

¹³C NMR (CDCl_3 , 75 MHz): δ = 18.7, 24.4, 28.5, 52.6, 95.6, 125.2, 126.9, 128.6, 144.0, 162.2, 194.6.

4-[2-(4-Methoxy-phenyl)-1-methyl-ethylamino]-pent-3-en-2-one (Table 3, entry 11)



A yellowish oil.

IR (neat): ν = 3422, 3031, 2967, 2930, 2835, 1609, 1575, 1513, 1440, 1377, 1247, 809, 736 cm^{-1} .

¹H NMR (CDCl_3 , 300 MHz): δ = 1.23 (d, J = 6.3 Hz, 3H), 1.65 (s, 3H), 1.99 (s, 3H), 2.71 (d, J =

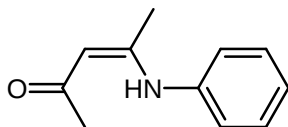
6.6 Hz, 2H), 3.64-3.73 (m, 1H), 3.80 (s, 3H), 4.83 (s, 1H), 6.81 (d, $J = 7.8$ Hz, 2H), 7.07 (d, $J = 7.8$ Hz, 2H), 10.96 (br s, 1H, NH).

^{13}C NMR (CDCl_3 , 75 MHz): $\delta = 18.5, 21.7, 28.7, 43.7, 51.0, 55.1, 94.9, 130.2, 158.2, 162.0, 194.4$.

ESI-MS: 248 ($\text{M}+1$) $^+$.

Anal. Calcd. for $\text{C}_{15}\text{H}_{21}\text{NO}_2$: C, 72.84; H, 8.56; N, 5.66. Found: C, 72.99; H, 8.70; N, 5.41.

4-(Phenylamino)pent-3-en-2-one (Table 3, entry 12)^[5,13]

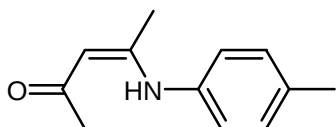


A pale yellow crystalline solid, mp: 48-49 °C (Lit.^[5] mp 47 °C).

IR (KBr): $\nu = 3442, 2997, 1595, 1572, 1509, 1437, 1356, 1281, 1190, 1019, 905, 749, 695\text{ cm}^{-1}$.

^1H NMR (CDCl_3 , 300 MHz): $\delta = 1.99$ (s, 3H), 2.10 (s, 3H), 5.19 (s, 1H), 7.09-7.21 (m, 3H), 7.31-7.37 (m, 2H), 12.49 (br s, 1H, NH).

4-(p-Tolylamino)pent-3-en-2-one (Table 3, entry 13)^[13]

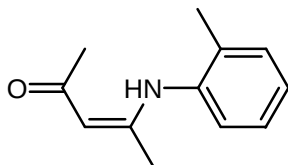


A pale yellow solid, mp 66-68 °C (Lit.^[13] mp 65-67 °C).

IR (KBr): $\nu = 3424, 3026, 1609, 1565, 1519, 1355, 1282, 1186, 1017, 942, 762\text{ cm}^{-1}$.

^1H NMR (CDCl_3 , 300 MHz): $\delta = 1.96$ (s, 3H), 2.09 (s, 3H), 2.34 (s, 3H), 5.16 (s, 1H), 6.99 (d, $J = 8.7$ Hz, 2H), 7.14 (d, $J = 8.7$ Hz, 2H), 12.39 (br s, 1H, NH).

4-o-Tolylamino-pent-3-en-2-one (Table 3, entry 14)



A pale yellow solid, mp 37-38 °C.

IR (KBr): $\nu = 3448, 2971, 1595, 1560, 1278, 1176, 1027, 823, 755\text{ cm}^{-1}$.

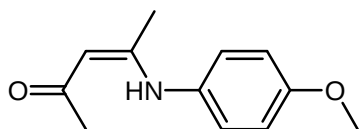
^1H NMR (CDCl_3 , 300 MHz): $\delta = 1.87$ (s, 3H), 2.11 (s, 3H), 2.28 (s, 3H), 5.20 (s, 1H), 7.06-7.23 (m, 4H), 12.35 (br s, 1H, NH).

^{13}C NMR (CDCl_3 , 75 MHz): $\delta = 18.0, 19.5, 29.0, 97.0, 126.2, 126.3, 130.7, 133.7, 137.5, 161.1, 196.0$.

ESI-MS: $m/z = 190$ ($\text{M}+1$) $^+$.

Anal. Calcd. for $\text{C}_{12}\text{H}_{15}\text{NO}$: C, 76.16; H, 7.99; N, 7.40. Found: C, 76.32; H, 7.91; N, 7.56.

4-(4-Methoxyphenylamino)pent-3-en-2-one (Table 3, entry 15)^[30]

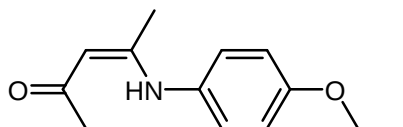


A pale yellow solid, mp 41-43 °C (Lit.^[30] mp 40.5-42.1 °C).

IR (KBr): ν = 3430, 3000, 2829, 1601, 1568, 1355, 1279, 1243, 1194, 1035, 921, 835, 763 cm^{-1} .

¹HNMR (CDCl_3 , 300 MHz): δ = 1.90 (s, 3H), 2.08 (s, 3H), 3.80 (s, 3H), 5.15 (s, 1H), 6.86 (d, J = 8.7 Hz, 2H), 7.04 (d, J = 8.7 Hz, 2H), 12.35 (br s, 1H, NH).

4-(4-Ethoxyphenylamino)pent-3-en-2-one (Table 3, entry 16)^[31]

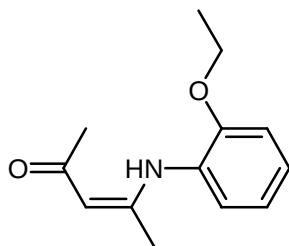


A pale yellow solid, mp 39-40 °C (Lit.^[31] mp 40.5-41.5 °C).

IR (KBr): ν = 3414, 2980, 1611, 1515, 1437, 1247, 1187, 1115, 922, 844, 744 cm^{-1} .

¹HNMR (CDCl_3 , 300 MHz): δ = 1.42 (t, J = 6.9 Hz, 3H), 1.90 (s, 3H), 2.08 (s, 3H), 4.02 (q, J = 6.9 Hz, 2H), 5.15 (s, 1H), 6.85 (d, J = 9.0 Hz, 2H), 7.03 (d, J = 9.0 Hz, 2H), 12.28 (br s, 1H, NH).

4-(2-Ethoxyphenylamino)pent-3-en-2-one (Table 3, entry 17)



A pale yellow solid, mp 56-57 °C.

IR (KBr): ν = 3441, 2988, 1608, 1568, 1441, 1272, 1182, 1121, 1042, 919, 843, 752 cm^{-1} .

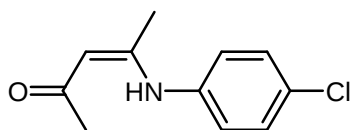
¹HNMR (CDCl_3 , 300 MHz): δ = 1.45 (t, J = 6.9 Hz, 3H), 2.01 (s, 3H), 2.10 (s, 3H), 4.09 (q, J = 6.9 Hz, 2H), 5.19 (s, 1H), 6.89-6.92 (m, 2H), 7.10-7.13 (m, 2H), 12.30 (br s, 1H, NH).

¹³C NM(CDCl_3 , 75 MHz): δ = 14.7, 19.9, 29.1, 64.3, 97.8, 112.6, 120.3, 124.6, 125.9, 128.4, 151.9, 160.0, 195.5.

ESI-MS: m/z = 220 ($M+1$)⁺.

Anal. Calcd. for $\text{C}_{13}\text{H}_{17}\text{NO}_2$: C, 71.21; H, 7.81; N, 6.39. Found: C, 71.48; H, 7.94; N, 6.21.

4-(4-Chlorophenylamino)pent-3-en-2-one (Table 3, entry 18)^[13,30]



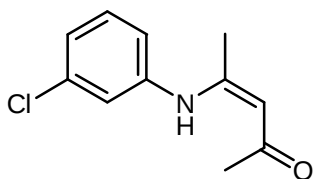
A pale yellow solid, mp 61-62 °C (Lit.^[13] mp 60-62 °C).

IR (KBr): ν = 3456, 2994, 1613, 1567, 1503, 1434, 1278, 1189, 1090, 1011, 915, 840, 755 cm^{-1} .

¹HNMR (CDCl_3 , 300 MHz): δ = 1.98 (s, 3H), 2.10 (s, 3H), 5.20 (s, 1H), 7.03 (d, J = 8.7 Hz, 2H),

7.30 (d, J = 8.7 Hz, 2H), 12.43 (br s, 1H, NH).

4-(3-Chlorophenylamino)pent-3-en-2-one (Table 3, entry 19)^[13,30]

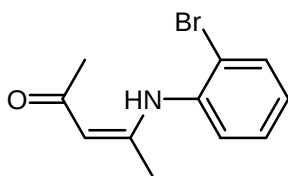


A pale yellow solid, mp 39-40 °C (lit.^[13] mp 39-40 °C).

IR (KBr): ν = 3443, 1618, 1567, 1507, 1437, 1277, 1188, 1094, 1025, 943, 844, 781 cm^{-1} .

¹HNMR (CDCl_3 , 300 MHz): δ = 2.01(s, 3H), 2.10 (s, 3H), 5.21 (s, 1H), 7.00-7.28 (m, 4H), 12.46 (br s, 1H, NH).

4-(2-Bromophenylamino)pent-3-en-2-one (Table 3, entry 20)^[32]

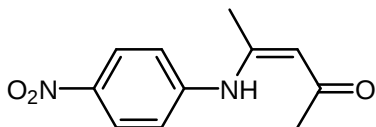


A pale yellow solid, mp 72-73 °C (Lit.^[32] mp 72-74 °C).

IR (KBr): ν = 3440, 3067, 1612, 1582, 1506, 1272, 1186, 1021, 923, 757, 532 cm^{-1} .

¹HNMR (CDCl_3 , 300 MHz): δ = 1.93 (s, 3H), 2.15 (s, 3H), 5.27 (s, 1H), 7.07-7.30 (m, 3H), 7.63 (dd, J = 8.7 Hz, 1.2 Hz, 1H), 12.34 (br s, 1H, NH).

4-(4-nitrophenylamino)pent-3-en-2-one (Table 3, entry 21)^[30]

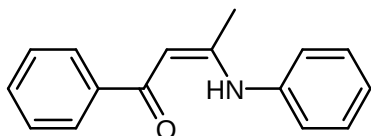


A yellow solid, mp 142-143 °C (Lit.^[30] mp 143.6-144.1 °C).

IR (KBr): ν = 3405, 3097, 2970, 1632, 1585, 1503, 1482, 1327, 1277, 1192, 1127, 1030, 924, 838, 790 cm^{-1} .

¹HNMR (CDCl_3): δ = 2.05 (s, 3H), 2.19 (s, 3H), 5.33 (s, 1H), 7.19 (d, J = 9.0 Hz, 2H), 8.21 (d, J = 9.0 Hz, 2H), 12.78 (br s, 1H, NH).

1-Phenyl-3-phenylamino-but-2-en-1-one (Table 3, entry 22)^[33]



A yellow solid, mp 109-110 °C (Lit.^[33] mp 110.5-111.5 °C)

IR (KBr): ν = 3416, 3064, 1619, 1547, 1520, 1432, 1378, 1323, 1284, 1194, 1067, 1022, 854, 809, 751, 696 cm^{-1} .

¹HNMR (CDCl_3): δ = 2.15 (s, 3H), 5.91 (s, 1H), 7.16-7.23 (m, 3H), 7.36-7.49 (m, 5H), 7.90-7.94

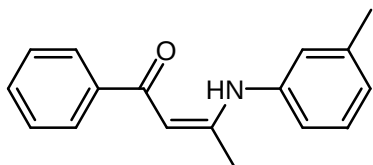
(m, 2H), 13.11 (br s, 1H, NH).

^{13}C NM(CDCl_3 , 75 MHz): δ = 20.4, 94.2, 124.7, 125.8, 127.0, 128.2, 129.1, 130.9, 138.6, 140.0, 162.2, 188.7.

ESI-MS: m/z = 238 ($\text{M}+1$) $^+$.

Anal. Calcd. for $\text{C}_{16}\text{H}_{15}\text{NO}$: C, 80.98; H, 6.37; N, 5.90. Found: C, 80.81; H, 6.52; N, 6.04.

3-(3-Tolylamino)-1-phenylbut-2-en-1-one (Table 3, entry 23)^[34]



A yellow solid, mp 55-56 °C.

IR (KBr): ν = 3056, 1571, 1430, 1371, 1282, 1157, 1089, 1067, 1024, 862, 739 cm^{-1} .

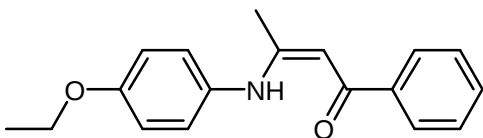
^1H NMR (CDCl_3): δ = 2.15 (s, 3H), 2.36 (s, 3H), 5.88 (s, 1H), 6.98-7.05 (m, 3H), 7.23-7.27 (m, 1H), 7.42-7.46 (m, 3H), 7.92 (dd, J = 7.8, 1.5 Hz, 2H), 13.09 (br s, 1H, NH).

^{13}C NMR (CDCl_3 , 75 MHz): δ = 20.4, 21.3, 94.1, 121.7, 125.4, 126.5, 127.0, 128.2, 128.9, 130.8, 138.5, 139.1, 140.1, 188.5.

ESI-MS: 252 ($\text{M}+1$) $^+$.

Anal. Calcd. for $\text{C}_{17}\text{H}_{17}\text{NO}$: C, 81.24; H, 6.82; N, 5.57. Found: C, 80.98; H, 6.96; N, 5.69.

3-(4-Ethoxy-phenylamino)-1-phenylbut-2-en-1-one (Table 3, entry 24)



A yellow solid, mp 84-85 °C.

IR (KBr): ν = 3414, 2979, 1599, 1504, 1473, 1432, 1372, 1322, 820, 744 cm^{-1} .

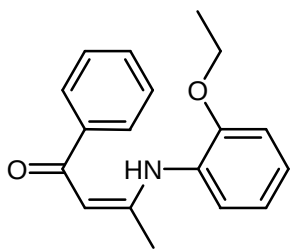
^1H NMR (CDCl_3 , 300 MHz): δ = 1.42 (t, J = 6.9 Hz, 3H), 2.06 (s, 3H), 4.03 (q, J = 6.9 Hz, 2H), 5.86 (s, 1H), 6.88 (d, J = 9.0 Hz, 2H), 7.09 (d, J = 9.0 Hz, 2H), 7.42-7.45 (m, 3H), 7.89-7.92 (m, 2H), 12.92 (br s, 1H, NH).

^{13}C NMR (CDCl_3 , 75 MHz): δ = 14.8, 20.2, 63.7, 93.5, 114.8, 126.5, 127.0, 128.2, 130.7, 131.3, 140.1, 157.2, 163.1, 188.3.

ESI-MS: 282 ($\text{M}+1$) $^+$.

Anal. Calcd. for $\text{C}_{18}\text{H}_{19}\text{NO}_2$: C, 76.84; H, 6.81; N, 4.98. Found: C, 75.59; H, 6.92; N, 5.02.

3-(2-Ethoxy-phenylamino)-1-phenylbut-2-en-1-one (Table 3, entry 25)



A yellow solid, mp 104 °C.

IR (KBr): ν = 3413, 2978, 1613, 1591, 1577, 1492, 1437, 1376, 1282, 1248, 1121, 1067, 1040, 921, 831, 742 cm^{-1} .

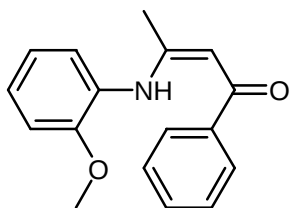
^1H NMR (CDCl_3) δ = 1.42 (t, J = 6.9 Hz, 3H), 2.06 (s, 3H), 4.03 (q, J = 6.9 Hz, 2H), 5.86 (s, 1H), 6.88 (d, J = 8.7 Hz, 2H), 7.15 (d, J = 8.7 Hz, 2H), 7.43-7.45 (m, 3H), 7.89-7.93 (m, 2H), 12.93 (br s, 1H, NH).

^{13}C NMR (CDCl_3 , 75 MHz): δ = 14.7, 20.5, 64.4, 94.3, 112.8, 120.4, 125.3, 126.5, 127.1, 128.1, 128.3, 130.6, 140.4, 152.4, 162.3, 188.4.

ESI-MS: 282 ($\text{M}+1$) $^+$.

Anal. Calcd for $\text{C}_{18}\text{H}_{19}\text{NO}_2$: C, 76.84; H, 6.81; N, 4.98. Found: C, 76.68; H, 6.70; N, 4.11.

3-(2-Methoxyphenylamino)-1-phenylbut-2-en-1-one (Table 3, entry 26)^[35]



A yellow solid, mp 92-93°C.

IR (KBr): ν = 3006, 1608, 1576, 1477, 1461, 1436, 1373, 1284, 1245, 1166, 1119, 1089, 1023, 748 cm^{-1} .

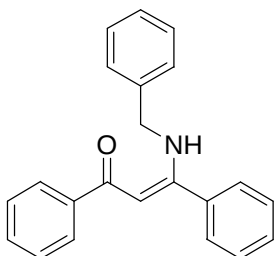
^1H NMR (CDCl_3 , 300 MHz): δ = 2.14 (s, 3H), 3.90 (s, 3H), 5.91 (s, 1H), 6.92-6.97 (m, 2H), 7.17-7.22 (m, 2H), 7.41-7.45 (m, 3H), 7.91-7.94 (m, 2H), 12.87 (br s, 1H, NH).

^{13}C NMR (CDCl_3 , 75 MHz): δ = 20.4, 55.8, 94.4, 111.4, 120.4, 125.3, 126.6, 127.1, 128.1, 130.7, 140.2, 153.0, 162.3, 188.5.

ESI-MS: 268 ($\text{M}+1$) $^+$.

Anal. Calcd for $\text{C}_{17}\text{H}_{17}\text{NO}_2$: C, 76.38; H, 6.41; N, 5.24. Found: C, 76.56; H, 6.28; N, 5.35.

3-(benzylamino)-1,3-diphenylprop-2-en-1-one (Table 3, entry 27)^[36]



A yellow-orange solid, mp 100-101 °C (Lit.^[36] mp 100 °C).

IR (KBr): ν = 3053, 1601, 1556, 1482, 1438, 1336, 1146, 1059, 1025, 961, 925, 851, 691 cm^{-1} .

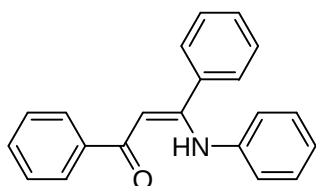
¹HNMR (CDCl_3 , 300 MHz): δ = 4.41 (d, J = 6.6 Hz, 2H), 5.84 (s, 1H), 7.21-7.26 (m, 3H), 7.29-7.43 (m, 10H), 7.90 (dd, J = 7.8, 1.5 Hz, 2H), 11.71 (br s, 1H, NH).

¹³C NMR (CDCl_3 , 75 MHz): δ = 48.4, 94.0, 126.9, 127.1, 127.4, 127.8, 128.2, 128.6, 128.7, 129.6, 130.8, 135.5, 138.4, 140.2, 166.7, 188.7.

ESI-MS: 314 ($\text{M}+1$)⁺.

Anal. Calcd for $\text{C}_{22}\text{H}_{19}\text{NO}$: C, 84.31; H, 6.11; N, 4.47. Found: C, 84.42; H, 6.23; N, 4.25.

1,3-diphenyl-3-(phenylamino)prop-2-en-1-one (Table 3, entry 28)^[36]



A yellow-orange solid, mp 96-97 °C (Lit.^[36] mp 103 °C).

IR (KBr): ν = 3053, 1592, 1568, 1475, 1441, 1323, 1212, 1079, 1053, 1022, 904, 841, 697 cm^{-1} .

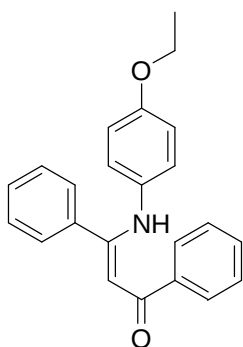
¹HNMR (CDCl_3 , 300 MHz): δ = 6.09 (s, 1H), 6.78 (d, J = 7.8 Hz, 2H), 6.96-7.00 (m, 1H), 7.09-7.14 (m, 2H), 7.26-7.50 (m, 8H), 7.96 (d, J = 6.9 Hz, 2H), 12.90 (br s, 1H, NH).

¹³C NMR (CDCl_3 , 75 MHz): δ = 97.0, 123.1, 124.2, 127.2, 128.2, 128.5, 128.7, 129.6, 131.3, 135.8, 139.4, 139.8, 161.4, 189.6.

ESI-MS: 300 ($\text{M}+1$)⁺.

Anal. Calcd for $\text{C}_{21}\text{H}_{17}\text{NO}$: C, 84.25; H, 5.72; N, 4.68. Found: C, 84.48; H, 5.82; N, 4.45.

3-(4-ethoxyphenylamino)-1,3-diphenylprop-2-en-1-one (Table 3, entry 29)



A yellow solid, mp 122-124 °C

IR (KBr): ν = 3065, 2974, 1591, 1562, 1515, 1476, 1327, 1251, 1121, 1169, 1112, 1044, 1025, 920, 852, 823, 775, 698 cm^{-1} .

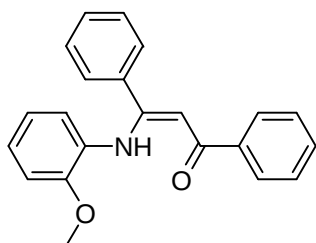
¹HNMR (CDCl_3 , 300 MHz): δ = 1.36 (t, J = 6.9 Hz, 3H), 3.93 (q, J = 6.9 Hz, 2H), 6.04 (s, 1H), 6.66 (d, J = 8.1 Hz, 2H), 6.74 (d, J = 8.4 Hz, 2H), 7.34-7.45 (m, 8H), 7.95 (d, J = 7.2 Hz, 2H), 12.90 (br s, 1H, NH).

^{13}C NMR (CDCl_3 , 75 MHz): δ = 14.8, 63.5, 96.0, 114.5, 124.9, 127.2, 128.3, 128.4, 129.5, 131.1, 132.3, 135.9, 140.0, 155.9, 162.1, 189.2.

ESI-MS: 344 ($\text{M}+1$) $^+$.

Anal. Calcd for $\text{C}_{23}\text{H}_{21}\text{NO}_2$: C, 80.44; H, 6.16; N, 4.08. Found: C, 80.56; H, 6.25; N, 3.96.

3-(2-methoxyphenylamino)-1,3-diphenylprop-2-en-1-one (Table 3, entry 30)



A yellow solid, mp 100-101 °C

IR (KBr): ν = 3452, 3047, 2835, 1596, 1461, 1326, 1287, 1212, 1206, 1173, 1133, 1105, 1052, 1023, 926, 845, 755, 710 cm^{-1} .

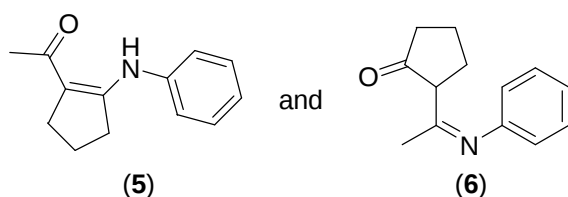
^1H NMR (CDCl_3 , 300 MHz): δ = 3.91 (s, 3H), 6.08 (s, 1H), 6.38 (dd, J = 8.1, 1.5 Hz, 1H), 6.56 (td, J = 8.1, 1.5 Hz, 1H), 6.86 (dd, J = 8.1, 1.5 Hz, 1H), 6.95 (td, J = 8.1, 1.5 Hz, 1H), 7.31-7.50 (m, 8H), 7.98 (dd, J = 7.8, 1.5 Hz, 2H), 12.73 (br s, 1H, NH).

^{13}C NMR (CDCl_3 , 75 MHz): δ = 55.7, 97.4, 110.9, 119.9, 123.4, 124.5, 127.3, 128.1, 128.3, 129.5, 131.1, 136.3, 140.1, 151.3, 160.9, 189.4.

ESI-MS: 330 ($\text{M}+1$) $^+$.

Anal. Calcd for $\text{C}_{22}\text{H}_{19}\text{NO}_2$: C, 80.22; H, 5.81; N, 4.25. Found: C, 80.31; H, 5.66; N, 4.17.

1-[2-(phenylamino)-1-cyclopenten-1-yl]-ethanone (5) and 2-[1-(phenylamino)ethylene]-cyclopentanone (6)



A pale yellow solid, the mixture of **5** and **6**, mp 39-40 °C (lit.^[37] mp 41 °C).

IR (KBr): ν = 3055, 2970, 2941, 2846, 1623, 1598, 1569, 1500, 1431, 1364, 1310, 1271, 1078, 1003, 930, 879, 829, 796, 757 cm^{-1} .

^1H NMR (CDCl_3 , 300 MHz): δ = 1.90-1.92 (m, 2H), 2.01 (s, 1.65 H), 2.16 (s, 1.35 H), 2.40 (t, J = 6.9 Hz, 1.1 H), 2.60-2.64 (m, 2H), 2.80 (t, J = 6.9 Hz, 0.9 H), 7.09-7.17 (m, 3H), 7.26-7.32 (m, 2H), 11.48 (br s, 0.45 H, NH), 11.94 (br s, 0.55 H, NH).

ESI-MS: 202 ($\text{M}+1$) $^+$.

References

- [1] T. Potešil, *J. Chromatogr.* **1982**, 249, 131-137.
- [2] K. Morita, Y. Haruta, T. Tomita, M. Tsuda, K. Kandori, M. Kise, K. Kimura, *Chem. Pharm. Bull.* **1987**, 35, 4819-4828.
- [3] C. A. Brandt, A. C. M. P. Da Silva, C. G. Pancote, C. L. Brito, M. A. B. Da Silveira, *Synthesis* **2004**, 1557-1559.
- [4] G. Bartoli, C. Cimagelli, R. Dalpozzo, G. Palmieri, *Tetrahedron* **1995**, 51, 8613-8622.
- [5] H. A. Stefani, I. M. Costa, D. De O. Silva, *Synthesis* **2000**, 1526-1528.
- [6] P. Siegle, K. Sasse, P. Roessler, *DE Patent* 2414456, **1975**; *Chem. Abstr.* **1976**, 84, 30462.
- [7] K.-U. Schoening, M. Studer, J. Hans, S. Burkhardt, *EP Patent* 1318139, **2003**; *Chem. Abstr.* **2003**, 138, 451809.
- [8] D. S. Reddy, T. V. Rajale, K. Shivakumar, J. Iqbal, *Tetrahedron Lett.* **2005**, 46, 979-982.
- [9] S. Jolidon, T. Meul, *EP Patent* 144980, **1985**; *Chem. Abstr.* **1986**, 105, 43325.
- [10] G. Revial, S. Lim, B. Viossat, P. Lemoine, A. Tomas, A. F. Duprat, M. Pfau, *J. Org. Chem.* **2000**, 65, 4593-4600.
- [11] H. L. Yale, E. R. Spitzmiller, *J. Heterocyclic Chem.* **1977**, 14, 1419-1421.
- [12] Y.-H. Gao, Q.-H. Zhang, J.-X. Xu, *Synth. Commun.* **2004**, 34, 909-916.
- [13] T. Potešil, *J. Chromatogr.* **1984**, 312, 387-393.
- [14] S. M. Pitzenberger, B. M. Trost, *EP Patent* 234776, **1987**; *Chem. Abstr.* **1988**, 108, 167304.
- [15] W. Werner, *Tetrahedron* **1971**, 27, 1755-1760.
- [16] D. Adams, J. Dominguez, V. Lo Russo, N. Morante De Rekowski, *Gazz. Chim. Ital.* **1989**, 119, 281-284.
- [17] S. Coffey, J. K. Thomson, F. J. Wilson, *J. Chem. Soc.* **1936**, 856-859.
- [18] L. K. Johnson, A. M. A. Bennett, S. D. Ittel, L. Wang, A. Parthasarathy, E. Hauptman, R. D. Simpson, J. Feldman, E. B. Coughlin, *WO Patent* 9830609, **1998**; *Chem. Abstr.* **1998**, 129, 149362.
- [19] T. Sano, Y. Horiguchi, J. Toda, K. Imafuku, Y. Tsuda, *Chem. Pharm. Bull.* **1984**, 32, 497-503.
- [20] T. A. Favorskaya, S. I. Yakimovich, L. N. Ignatyuk, A. M. Kutnevich, *Russ. J. Org. Chem.* **1969**, 5, 1011-1014.
- [21] M. M. Khodaei, A. R. Khosropour, M. Kookhazadeh, *Synlett* **2004**, 1980-1984.
- [22] A. Felk, G. Revial, B. Viossat, P. Lemoine, M. Pfau, *Tetrahedron: Asymm.* **1994**, 8, 1459-1462.
- [23] T. H. Brown, R. J. Ife, D. J. Keeling, S. M. Laing, C. A. Leach, M. E. Parsons, C. A. Price, D. R. Reavill, K. J. Wiggall, *J. Med. Chem.* **1990**, 33, 527-533.
- [24] M. S. Chadha, K. K. Chakravarti, S. Sidiqi, *J. Sci. Ind. Res.* **1951**, 10B, 1-3.
- [25] R. J. Brown, F. W. S. Carver, B. L. Hollingsworth, *J. Org. Chem.* **1967**, 32, 2624-2626.
- [26] R. V. Singh, J. P. Tandon, *J. Indin Cem. Soc.* **1980**, 57, 864-867.

- [27] R. H. McDougall, S. H. Malik, *J. Chem. Soc. (C)* **1969**, 2044-2051.
- [28] P. W. Hickmott, G. Sheppard, *J. Chem. Soc., Perkin Trans. 1* **1972**, 1038-1041.
- [29] C. Cimarrelli, G. Palmieri, *Tetrahedron* **1998**, 54, 915-926.
- [30] J.-C. Zhou, *Magn. Reson. Chem.* **1997**, 35, 311-322.
- [31] G.-Z. Zeng, J.-S. Yan, D.-Z. Shen, J.-H. Cheng, *Acta Chim. Sinica* **1982**, 40, 720-729.
- [32] T. Sakamoto, T. Nagano, Y. Kondo, H. Yamanaka, *Synthesis* **1990**, 215-218.
- [33] P. Šimunek, M. Pešková, V. Bertolasi, V. Macháček, A. Lycka, *Tetrahedron* **2005**, 61, 8130-8137.
- [34] A. A. Bakra, A. G. Mehta, *J. Inst. Chem.* **1987**, 59, 133-134.
- [35] M. D. Santana, A. Rufete, G. Garcia, G. Lopez, J. Casabo, A. Cabrero, E. Molins, C. Miravittles, *Polyhedron* **1997**, 16, 3713-3721.
- [36] N. H. Coromwell, *Chem. Rev.* **1946**, 38, 83-127.
- [37] A. A. H. Saeed, *J. Chem. Eng. Data* **1984**, 29, 358-361.