Borate Esters as Convenient Reagents for Direct Amidation of Carboxylic Acids and Transamidation of Primary Amides

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1 General

All chemicals were used as supplied. Amidations and transamidations were performed on 0.5–3.0 mmol scale. Chromatographic separations were performed on silica gel (VWR/BDH Prolabo® (40–63 μm) and Merck Silica gel 60 (40–63 μm). Thin-layer chromatography was performed on Merck TLC Silica gel 60 F254 and visualised by UV (254 nm) and/or 10% PMA ethanolic solution (PMA = phosphomolybdic acid). Melting points were determined using a Gallenkamp apparatus and are uncorrected. Infrared spectra were recorded on Perkin–Elmer Spectrum 100 FTIR ATR spectrometer and are quoted in cm^{-1}. ^{1}H and ^{13}C NMR spectra were recorded on Bruker Avance 600, ^{11}B NMR on Bruker Avance 500 and ^{19}F on Bruker 300 spectrometers. Residual solvent peak was used as an internal standard[1]. Chemical shifts are quoted in ppm using the following abbreviations: s, singlet; d, doublet; t, triplet; q, quartet; qn quintet; sext sextet; non nonet, m multiplet; br, broad; or a combination thereof. The coupling constants *J* are measured in Hz. Mass spectra were recorded in the Department of Chemistry, University College London.

2 Procedures

2.1 Tris(2,2,2-trifluoroethyl) Borate

$$B(OCH_2CF_3)_3$$

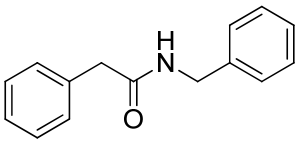
2,2,2-Trifluroethanol (42.7 mL, 586 mmol, 3.0 equiv) was added via syringe pump over 30 min to neat BBr_3 (48.9 g, 195 mmol, 1.0 equiv) at –78 °C and the mixture was allowed to warm up to RT overnight under argon flow. The mixture was heated for 1 h at 70 °C under argon before being distilled (120–123 °C; 760 Torr) to give the product as a colourless liquid (54.0 g, 90%).

Tris(2,2,2-trifluoroethyl)borate can also be prepared from BCl_3,[2,3] B_2O_3,[4] $B(OH)_3$[5] and BH_3·THF.[6]

Tris(2,2,2-trifluoroethyl) borate:[2,3]
Colourless liquid. Lit Bp 77 ºC (200 Torr)[3]
^{1}H NMR ($CDCl_3$, 600 MHz) δ 4.22 (q, 6H, J = 8.4, CH_2CF_3).
^{13}C NMR ($CDCl_3$, 150 MHz) δ 123.3 (q, J = 278), 61.9 (q, J = 36).
^{19}F NMR ($CDCl_3$, 282 MHz) δ –77.2.
^{11}B NMR ($CDCl_3$, 160 MHz) δ 15.3.
IR ν 2974, 1429, 1390, 1264, 1161, 1079, 964, 907, 840, 731.
HRMS for $C_6H_7O_3F_9B$ $[M]^+$ found 309.03461, calc. 309.03445.

2.2 Direct Carboxamidation

Representative Procedure: Borate (2 equiv) was added to a solution/suspension of carboxylic acid (1 equiv) and amine (1 equiv) in MeCN (0.5 M) and the mixture was heated at 80 °C. After 15 h, the solvent was removed under reduced pressure.The residue was redissolved in DCM and washed with $NaHCO_3$ (1 M) and HCl (1 M) aqueous solutions, dried over $MgSO_4$, filtered and concentrated under reduced pressure to give the clean amide product.



N-Benzyl-2-phenylacetamide:[7]

Colourless solid. Mp 118–120 °C (DCM). Lit Mp 118–119 °C (PE/EtOAc).[7]

^{1}H NMR ($CDCl_3$, 600 MHz) δ 7.36–7.32 (m, 2H, Ar*H*), 7.32–7.22 (m, 6H, Ar*H*), 7.19–7.15 (d, 2H, *J* = 7.1, Ar*H*), 5.79 (br s, 1H, N*H*), 4.41 (d, 2H, *J* = 5.8, CH_2NH), 3.63 (s, 2H, CH_2CO).

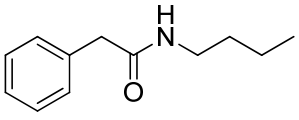
^{13}C NMR ($CDCl_3$, 150 MHz) δ 171.1, 138.2, 134.9, 129.6, 129.2, 128.8, 127.61, 127.56, 127.55, 43.9, 43.7.

^{1}H NMR (DMSO-d_6, 600 MHz) δ 8.56 (br t, 1H, *J* = 5.5, N*H*), 7.33–7.26 (m, 6H, Ar*H*), 7.25–7.20 (m, 4H, Ar*H*), 4.26 (d, 2H, *J* = 6.0, CH_2NH), 3.47 (s, 2H, CH_2CO).

^{13}C NMR (DMSO-d_6, 150 MHz) δ 170.1, 139.5, 136.4, 129.0, 128.3, 128.2, 127.2, 126.8, 126.4, 42.4, 42.2.

IR ν 3286, 1637, 1551.

HRMS for $C_{15}H_{15}NO$ $[M]^+$ found 225.11483, calc. 225.11482.



N-Butyl-2-phenylacetamide:[8]

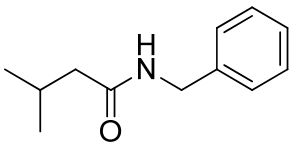
Colourless solid. Mp 49–50 °C (DCM). Lit Mp 49 °C.[8]

^{1}H NMR ($CDCl_3$, 600 MHz) δ 7.34–7.30 (m, 2H, Ar*H*), 7.30–7.21 (m, 3H, Ar*H*), 5.71 (br s, 1H, N*H*), 3.53 (s, 2H, CH_2CO), 3.17 (q, 2H, *J* = 6.5, NHCH_2), 1.38 (qn, 2H, *J* = 7.3, CH_2Et), 1.23 (sx, 2H, *J* = 7.3, CH_2CH$_3$), 0.85 (t, 3H, *J* = 7.3, CH_3).

^{13}C NMR ($CDCl_3$, 150 MHz) δ 171.2, 135.2, 129.5, 129.1, 127.4, 43.9, 39.5, 31.6, 20.1, 13.8.

IR ν 3294, 1642, 1549.

HRMS for $C_{12}H_{17}NO$ $[M]^+$ found 191.12961, calc. 191.13047.



N-Benzyl-3-methylbutanamide:[9]

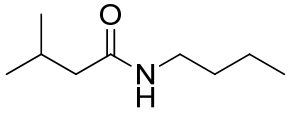
Colourless solid. Mp 58–59 °C (DCM). Lit Mp 58–60 °C.[9]

^{1}H NMR ($CDCl_3$, 600 MHz) δ 7.34–7.29 (m, 2H, Ar*H*), 7.28–7.23 (m, 3H, Ar*H*), 5.97 (br s , 1H, N*H*), 4.41 (d, 2H, *J* = 5.7, C*H*$_2$NH), 2.13 (non, 1H, *J* = 6.7, C*H*Me$_2$), 2.06 (d, 2H, *J* = 7.1, C*H*$_2$CO), 0.94 (d, 6H, *J* = 6.6, C*H*$_3$).

^{13}C NMR ($CDCl_3$, 150 MHz) δ 172.5, 138.5, 128.8, 127.9, 127.6, 46.2, 43.6, 26.3, 22.6.

IR ν 3289, 1635, 1543.

HRMS for $C_{12}H_{17}NO$ $[M]^+$ found 191.13126, calc. 191.13047.



N-Butyl-3-methylbutanamide:[10]

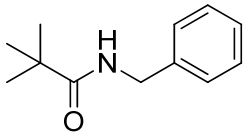
Colourless oil.

^{1}H NMR ($CDCl_3$, 600 MHz) δ 5.79 (br s, 1H. CON*H*), 3.20 (q, 2H, *J* = 6.7, NHC*H*$_2$), 2.07 (non, 1H, *J* = 6.7, C*H*(CH$_3$)$_3$), 1.99 (d, 2H, *J* = 6.7, C*H*$_2$CO), 1.44 (qn, 2H, *J* = 7.2, NHCH$_2$C*H*$_2$), 1.30 (sx, 2H, *J* = 7.2, C*H*$_2$CH$_3$), 0.90 (d, 6H, *J* = 6.7, CH(C*H*$_3$)$_2$), 0.88 (t, 3H, *J* = 7.2, CH$_2$C*H*$_3$).

^{13}C NMR ($CDCl_3$, 150 MHz) δ 172.7, 46.3, 39.2, 31.9, 26.3, 22.5, 20.2, 13.9.

IR ν 3284, 2957, 2871, 1641, 1550, 1465, 1368.

HRMS for $C_9H_{20}NO$ $[M+H]^+$ found 158.15521, calc. 157.15449.



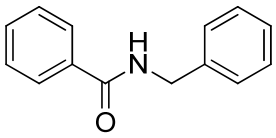
N-Benzylpivalamide:[11] Colourless solid. Mp 80–81 °C (DCM). Lit Mp 80–82 °C.[11]

^{1}H NMR ($CDCl_3$, 600 MHz) δ 7.36–7.30 (m, 2H, Ar*H*), 7.30–7.23 (m, 3H, Ar*H*), 5.92 (br s, 1H, N*H*), 4.43 (d, 2H, J = 5.6, C*H*$_2$NH), 1.22 (s, 9H, C*H*$_3$).

^{13}C NMR ($CDCl_3$, 150 MHz) δ 178.5, 138.7, 128.8, 127.8, 127.6, 43.7, 38.8, 27.7.

IR ν 3293, 1634, 1540.

HRMS for $C_{12}H_{17}NO$ $[M]^+$ found 191.12954, calc. 191.13047.



N-Benzylbenzamide:[7]

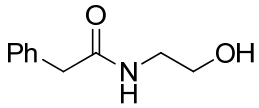
Colourless solid.Mp 100–101 °C (DCM). Lit Mp 98–100 °C (H_2O/EtOH).[7]

^{1}H NMR ($CDCl_3$, 600 MHz) δ 7.90–7.76 (m, 2H, Ar*H*), 7.52–7.46 (m, 1H, Ar*H*), 7.45–7.39 (m, 2H, Ar*H*), 7.37–7.32 (m, 4H, Ar*H*), 7.32–7.26 (m, 1H, Ar*H*), 6.56 (br s, 1H, N*H*), 4.63 (d, 2H, *J* = 5.1, CH_2CO).

^{13}C NMR ($CDCl_3$, 150 MHz) δ 167.5, 138.3, 134.5, 131.7, 128.9, 128.7, 128.0, 127.7, 127.1, 44.2.

IR ν 3318, 1639, 1540.

HRMS for $C_{14}H_{14}NO$ $[M+H]^+$ found 212.10846, calc. 212.10754.



N-(2-Hydroxyethyl)-2-phenylacetamide:[12]

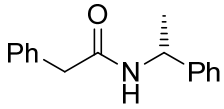
Colourless solid. Mp 64–66 °C. Lit Mp 65–66 °C.[12]

^{1}H NMR ($CDCl_3$, 600 MHz) δ 7.38–7.25 (m, 5H, Ar*H*), 5.98 (br s, 1H, CON*H*), 3.68 (t, 2H, *J* = 5.0, CH_2OH), 3.61 (s, 2H, PhCH_2), 3.38 (q, 2H, *J* = 5.0, NHCH_2), 2.46 (br s, 1H, O*H*).

^{13}C NMR ($CDCl_3$, 150 MHz) δ 172.8, 134.6, 129.6, 129.2, 127.6, 62.5, 43.7, 42.9.

IR ν 3397, 3278, 3090, 2931, 1635, 1540, 1495, 1454, 1429, 1345, 1061.

HRMS for $C_{10}H_{14}NO_2$ $[M+H]^+$ found 180.10266, calc. 180.10245.



(R)-2-Phenyl-N-(1-phenylethyl)acetamide:[7]

Colourless solid. Mp 116–117 °C (DCM). Lit Mp 115-116 °C (H_2O/EtOH).[7]

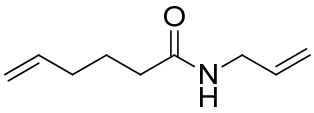
α_D^{25} +3.3 (*c* 1.0, $CHCl_3$). Lit α_D^{25} +3.3 (*c* 1.0, $CHCl_3$).[7]

^{1}H NMR ($CDCl_3$, 600 MHz) δ 7.37–7.33 (m, 2H, Ar*H*), 7.32–7.27 (m, 3H, Ar*H*), 7.27–7.21 (m, 3H, Ar*H*), 7.20–7.16 (m, 2H, Ar*H*), 5.66 (br s, 1H, CON*H*), 5.12 (qn, 1H, *J* = 7.1, C*H*CH_3), 3.60(d, 1H, *J* = 16.3, PhC*H*H), 3.58 (d, 1H, *J* = 16.3, PhCH*H*), 1.39 (d, 3H, *J* = 7.1, CH_3).

^{13}C NMR ($CDCl_3$, 150 MHz) δ 170.2, 143.1, 134.9, 129.5, 129.2, 128.7, 127.5, 127.4, 126.1, 48.9, 43.9, 21.9.

IR ν 3284, 3062, 3030, 2974, 1641, 1543, 1495, 1453.

HRMS for $C_{16}H_{18}NO$ $[M+H]^+$ found 240.13757, calc. 240.13884.



N-Allylhex-5-enamide:

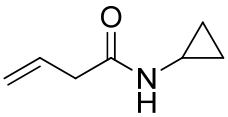
Pale yellow oil.

^{1}H NMR ($CDCl_3$, 600 MHz) δ 6.09 (bs s, 1H, CON*H*), 5.83–5.66 (m, 2H, 2×C*H*=CH_2), 5.12 (d, 1H, *J* = 17.2, $NHCH_2CH$=CH*H-trans*), 5.07 (d, 1H, *J* = 10.4, $NHCH_2CH$=CH*H-cis*), 4.97 (d, 1H, *J* = 17.2, $(CH_2)_2CH$=CH*H-trans*), 4.92 (d, 1H, *J* = 10.4, $(CH_2)_2CH$=CH*H-cis*), 3.81 (t, 2H, *J* = 5.6, NHCH_2), 2.16 (t, 2H, *J* = 7.6, COCH_2), 2.04 (q, 2H, *J* = 7.2, CH_2CH=CH_2), 1.70 (qn, 2H, *J* = 7.5, $CH_2CH_2CH_2$).

^{13}C NMR ($CDCl_3$, 150 MHz) δ 173.0, 138.0, 134.4, 116.3, 115.4, 41.9, 35.9, 33.3, 24.9.

IR ν 3289, 3077, 2927, 1640, 1543, 911.

HRMS for $C_9H_{14}NO$ $[M–H]^+$ found 152.10643, calc. 152.10699.



N-Cyclopropylbut-3-enamide:

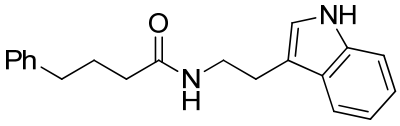
Yellow oil.

^{1}H NMR ($CDCl_3$, 600 MHz) δ 6.53 (br s, 1H, CON*H*), 5.82 (ddt, 1H, *J* = 17.1, 10.7, 7.1, C*H*=CH_2), 5.10–5.05 (m, 2H, CH=CH_2), 2.88 (d, 2H, *J* = 7.1, CH_2CO), 2.59 (tq, *J* = 7.3, 3.7, 1H, C*H*N), 0.66–0.62 (m, 2H, ^{c}Pr–*H*), 0.40 (m, 2H, ^{c}Pr–*H*).

^{13}C NMR ($CDCl_3$, 150 MHz) δ 172.5, 131.6, 119.2, 41.4, 22.7, 6.4.

IR ν 3275, 3081, 1647, 1537, 913.

HRMS for $C_7H_{11}NO$ $[M]^+$ found 125.08291, calc. 125.08352.



*N-(2-(1H-Indol-3-yl)ethyl)-4-phenylbutanamide***:**

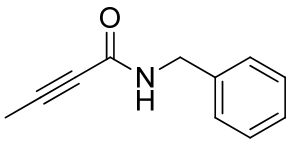
Pale yellow solid. Mp 110–111 °C (PE/Et_2O). Lit Mp 112–113 °C (MeOH)[13]

^{1}H NMR ($CDCl_3$, 600 MHz) δ 8.59 (br s, 1H, indole-N*H*), 7.60 (d, 1H, *J* = 7.9, indole-C*H*), 7.37 (d, 1H, *J* = 7.9, indole-C*H*), 7.29–7.24 (m, 2H, Ar*H*), 7.24–7.17 (m, 2H, indole-C*H*), 7.15–7.10 (m, 3H, Ar*H*), 6.97 (s, 1H, indole-C*H*), 5.69 (br s, 1H, CON*H*), 3.59 (q, 2H, *J* = 6.6, NHCH_2), 2.98 (t, 2H, *J* = 6.6, $NHCH_2CH_2$), 2.60 (t, 2H, *J* = 7.6, PhCH_2), 2.11 (t, 2H, *J* = 7.6, CH_2CO), 1.94 (qn, 2H, *J* = 7.6, $CH_2CH_2CH_2$).

^{13}C NMR ($CDCl_3$, 150 MHz) δ 173.2. 141.6, 136.5, 128.6, 128.5, 127.5, 126.1, 122.4, 122.2, 119.5, 118.8, 112.8. 111.6, 40.0, 36.1, 35.3, 27.3, 25.4.

IR ν 3407, 3284, 2924, 1644, 1526, 1455.

HRMS for $C_{20}H_{22}N_2ONa$ $[M+Na]^+$ found 329.1628, calc. 329.1630.



N-Benzylbut-2-ynamide:

Pale yellow solid. Mp 114–115 °C (DCM).

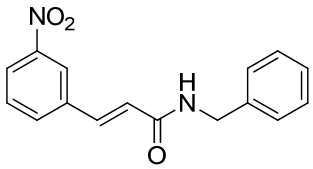
Mixture of rotamers in a ratio 1:10.

^{1}H NMR ($CDCl_3$, 600 MHz) δ 7.37–7.31 (m, 2H, Ar*H*), 7.31–7.26 (m, 3H, Ar*H*), 6.02 (br s, 1H, CON*H*), 4.60/4.47 (minor/major, d, 2H, J_{minor} = 6.5, J_{major} = 5.9, CH_2Ph), 2.01/1.93 (minor/major, s, 3H, CH_3).

^{13}C NMR ($CDCl_3$, 150 MHz, major rotamer) δ 153.4, 137.4, 128.9, 128.0, 127.9, 83.9, 74.8, 43.9, 3.8.

IR ν 3266, 3062, 2253, 1631, 1532, 1287.

HRMS for $C_{11}H_{11}NO$ $[M]^+$ found 173.08273, calc. 173.08352.



(E)-N-Benzyl-3-(3-nitrophenyl)acrylamide:[14]

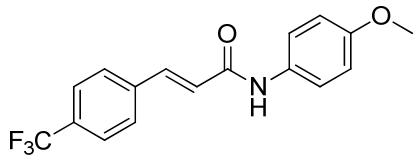
Pale yellow solid. Mp 185–186 °C (DCM). Lit Mp 184–185 °C.[14]

^{1}H NMR ($CDCl_3$, 600 MHz) δ 8.31 (s, 1H, Ar*H*), 8.15 (d, 1H, *J* = 8.2, Ar*H*), 7.71 (d, 1H, *J* = 7.7, Ar*H*), 7.66 (d, 1H, *J* = 15.6, ArC*H*=CH), 7.52 (t, 1H, *J* = 8.2, Ar*H*), 7.34–7.24 (m, 5H, Ar*H*), 6.60 (d, 1H, *J* = 15.6, ArCH=C*H*), 6.48 (br s, 1H, N*H*), 4.57 (d, 2H, *J* = 5.8, NHCH_2).

^{13}C NMR ($CDCl_3$, 150 MHz) δ 165.1, 148.7, 138.8, 138.0, 136.7, 134.1, 130.0, 128.9, 128.0, 127.8, 124.1, 123.7, 121.8, 44.8.

IR ν 3283, 1656, 1619, 1525, 1349, 1221.

HRMS for $C_{16}H_{15}NO$ $[M]^+$ found 282.09909, calc. 282.09989.



Purified by column chromatography (PE/EtOAc 1:2).

(E)-N-(4-Methoxyphenyl)-3-(4-(trifluoromethyl)phenyl)acrylamide:

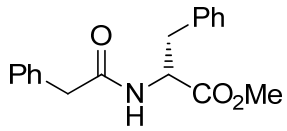
Colourless solid. Mp 191–192 °C (EtOAc/PE).

^{1}H NMR (DMSO-d_6, 600 MHz) δ 10.2 (s, 1H, CON*H*), 7.84 (d, 2H, *J* = 8.5, Ar*H*), 7.80 (d, 2H, *J* = 8.5, Ar*H*), 7.66–7.60 (m, 3H, PMP*H* and ArC*H*=CH), 6.96–6.90 (m, 3H, PMP*H* and ArCH=C*H*), 3.74 (s, 3H, OCH_3).

^{13}C NMR (DMSO-d_6, 150 MHz) δ 162.6, 155.5, 138.9, 137.9, 132.3, 129.4 (*J* = 32.2), 128.3, 125.9 (*J* = 3.9), 125.3, 124.2 (*J* = 271.8), 120.7, 114.0, 55.2.

IR ν 3294, 1658, 1622, 1537, 1511, 1326, 1125, 1070.

HRMS for $C_{17}H_{15}F_3NO_2$ $[M+H]^+$ found 322.1057, calc. 322.1055.



(R)-Methyl 3-phenyl-2-(2-phenylacetamido)propanoate:[15]

Colourless solid. Mp 93–94 °C (DCM). Lit Mp 92–94 °C (EtOAc/hexane).[15]

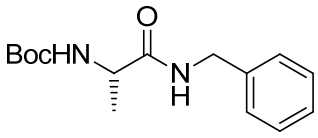
α_D^{25} –49.1 (*c* 1.0, $CHCl_3$). Lit α_D^{25} –49.5 (*c* 1.0, $CHCl_3$).[15]

^{1}H NMR ($CDCl_3$, 600 MHz) δ 7.36.–7.27 (m, 3H, Ar*H*), 7.21–7.16 (m, 5H, Ar*H*), 6.90–6.85 (m, 2H, Ar*H*), 5.80 (d, 1H, *J* = 6.9, CON*H*), 4.85 (dt, 1H, *J* = 7.9, 5.7, NHC*H*), 3.70 (s, 3H, CH_3), 3.56 (d, 1H, *J* = 15.9, PhC*H*HCO), 3.53 (d, 1H, *J* = 15.9, PhCH*H*CO), 3.06 (dd, 1H, *J* = 13.8, 5.8, CH*H*Ph), 2.99 (dd, 1H, *J* = 13.8, 5.8, CH*H*Ph).

^{13}C NMR ($CDCl_3$, 150 MHz) δ 171.9, 170.6, 135.6, 134.5, 129.5 129.3, 129.1, 128.7, 127.5, 127.2, 53.1, 52.46, 43.8, 37.7.

IR ν 3287, 3063, 3029, 2951, 1744, 1651, 1537, 1496, 1217.

HRMS for $C_{18}H_{20}NO_3$ $[M+H]^+$ found 298.14468, calc. 298.14431.



tert-Butyl (1-(benzylamino)-1-oxopropan-2-yl)carbamate:[16]

Colourless solid. Mp 100–102 °C (DCM). Lit Mp 104–106 °C (EtOAc/hexane).[16]

α_D^{22} (*c* 1.9, $CHCl_3$) = – 23.9. Lit α_D^{22} (*c* 1.9, $CHCl_3$) = – 24.5.[16]

^{1}H NMR ($CDCl_3$, 600 MHz) δ 7.33–7.28 (m, 2H, Ar*H*), 7.28–7.22 (m, 3H, Ar*H*), 6.63 (br s, 1H, CON*H*Bn), 5.07 (m, 1H, C*H*CH_3), 4.43 (br s, 2H, CH_2Ph), 4.20 (br s, 1H, BocN*H*), 1.40 (s, 9H, C(CH_3)$_3$), 1.37 (d, 3H, *J* = 6.8, CHCH_3).

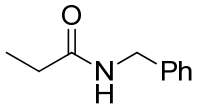
^{13}C NMR ($CDCl_3$, 150 MHz) δ 172.7, 155.7, 138.1, 128.8, 127.7, 127.6, 80.3, 50.3, 43.5, 28.4, 18.3.

IR ν 3304, 1695, 1951, 1497, 1365, 1162.

HRMS for $C_{15}H_{22}N_2O_3Na$ $[M+Na]^+$ found 301.15319, calc. 301.15280.

2.3 Transamidations

Borate (2 equiv) was added to a solution/suspension of amide (1 equiv) and amine (1 equiv) in MeCN (0.5 M) and the mixture was heated at 100 °C in carousel tube. After 15 h, solvent was removed under reduced pressure. The residue was purified by column chromatography (EtOAc/PE 1:1) to give the product.



N-Benzylpropionamide:[17]

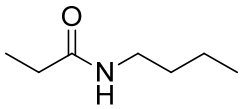
Colourless solid. Mp 51 °C (EtOAc/PE). Lit Mp 49–50 ºC (EtOAc/hexane)[17]

^{1}H NMR (DMSO-d_6, 600 MHz) δ 8.28 (s, 1H, CONH), 7.34–7.29 (m, 2H, Ar*H*), 7.25–7.21 (m, 3H, Ar*H*), 4.25 (d, 2H, J = 6.0, NHCH_2), 2.14 (q, 2H, J = 7.7, CH_2CH$_3$), 1.02 (t, 3H, J = 7.7, CH_3).

^{13}C NMR (DMSO-d_6, 150 MHz) δ 172.9, 139.8, 128.3, 127.2, 126.7, 42.0, 28.5, 10.0.

IR ν 3282, 3066, 3031, 2977, 2938, 1642, 1541, 1454, 1234, 1029.

HRMS for $C_{10}H_{13}NO$ $[M]^+$ found 163.09931, calc. 163.09917.



N-Butylpropionamide:[18]

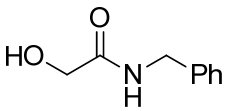
Colourless oil.

^{1}H NMR ($CDCl_3$, 600 MHz) δ 5.55 (br s, 1H, CON*H*), 3.23 (q, 2H, J = 6.5, NHCH_2), 2.18 (q, 2H, J = 7.5, CH_2CO), 1.46 (qn, 2H, J = 7.5, NHCH$_2$CH_2), 1.32 (sx, 2H, J = 7.5, CH$_2$CH_2CH$_3$), 1.13 (t, 3H, J = 7.5, CH_3CH$_2$CO), 0.90 (t, 3H, J = 7.5, NH(CH$_2$)$_3$CH_3).

^{13}C NMR ($CDCl_3$, 150 MHz) δ 174.2, 39.3, 31.7, 29.7, 20.1, 13.8, 10.1.

IR ν 3292, 2960, 2933, 1644, 1550, 1464, 1236.

HRMS for $C_7H_{15}NO$ $[M]^+$ found 129.11468, calc. 129.11468.



N-Benzyl-2-hydroxyacetamide:[19]

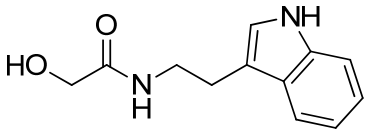
Colourless solid. Mp 102–103 °C (EtOAc/hexane). Lit Mp 102–103 °C (DCM). [19]

^{1}H NMR ($CDCl_3$, 600 MHz) δ 7.35–7.30 (m, 2H, Ar*H*), 7.29–7.24 (m, 3H, Ar*H*), 6.99 (br s, 1H, CON*H*), 4.45 (d, 2H, J = 5.9, NHCH_2), 4.09 (s, 2H, HOCH_2), O*H* not observed.

^{13}C NMR ($CDCl_3$, 150 MHz) δ 172.0, 137.8, 128.9, 127.9, 127.8 , 62.2, 43.1.

IR ν 3317, 3208, 3058, 3031, 2933, 2857, 1633, 1562, 1453, 1424, 1342, 1082.

HRMS for $C_9H_{11}NO_2$ $[M]^+$ found 165.07859, calc. 165.07843.



Purified by column chromatography (PE/EtOAc/MeOH 5:5:1).

N-((1H-indol-3-yl)methyl)-2-hydroxyacetamide

Colourless solid. Mp 141–142 °C (PE/MeOH).

^{1}H NMR (DMSO-d_6, 600 MHz) δ 10.81 (s, 1H, indole-N*H*), 7.81 (t, 1H, *J* = 5.8, CON*H*), 7.56 (d, 1H, *J* = 7.9, Ar*H*), 7.33 (d, 1H, *J* = 7.9, Ar*H*), 7.16 (d, 1H, *J* = 2.2, Ar*H*), 7.06 (td, 1H, *J* = 7.4, 0.9, Ar*H*), 6.97 (td, 1H, *J* = 7.4, 0.9, Ar*H*), 5.49 (t, 1H, *J* = 5.8, CH_2O*H*), 3.79 (d, 2H, *J* = 5.8, CH_2OH), 3.39 (q, 2H, *J* = 7.1, NHCH_2), 2.83 (t, 2H, *J* = 7.1, ArCH_2)

^{13}C NMR (DMSO-d_6, 150 MHz) δ 171.6, 136.3, 127.2, 122.6, 121.0, 118.4, 118.2, 111.7, 111.4, 61.5, 38.8, 25.4.

IR ν 3391, 3301, 3260, 1644, 1619, 1543, 1455, 1353, 1223, 1072.

HRMS for $C_{12}H_{13}N_2O_2$ $[M+H]^+$ found 217.0992, calc. 217.0977.

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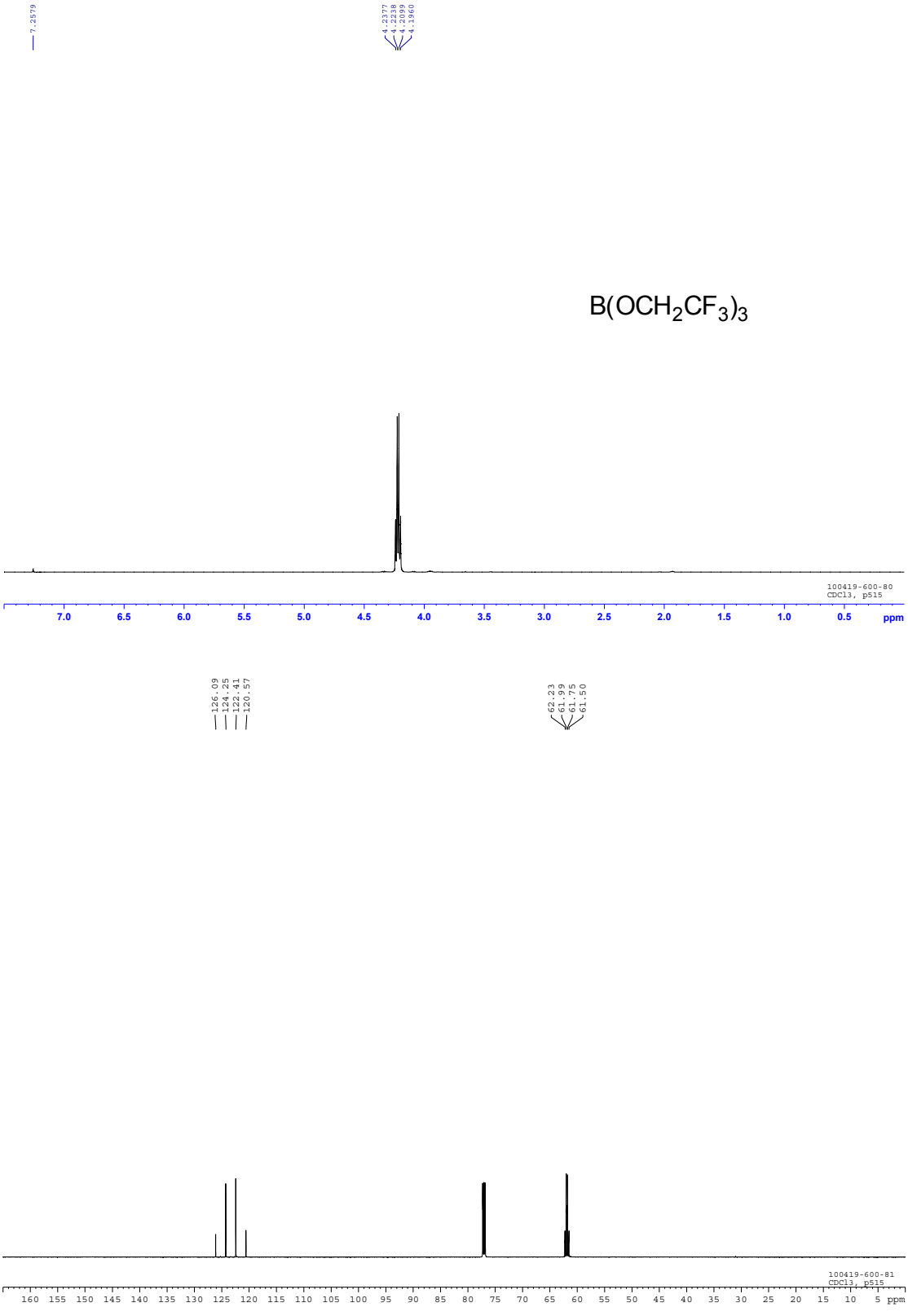
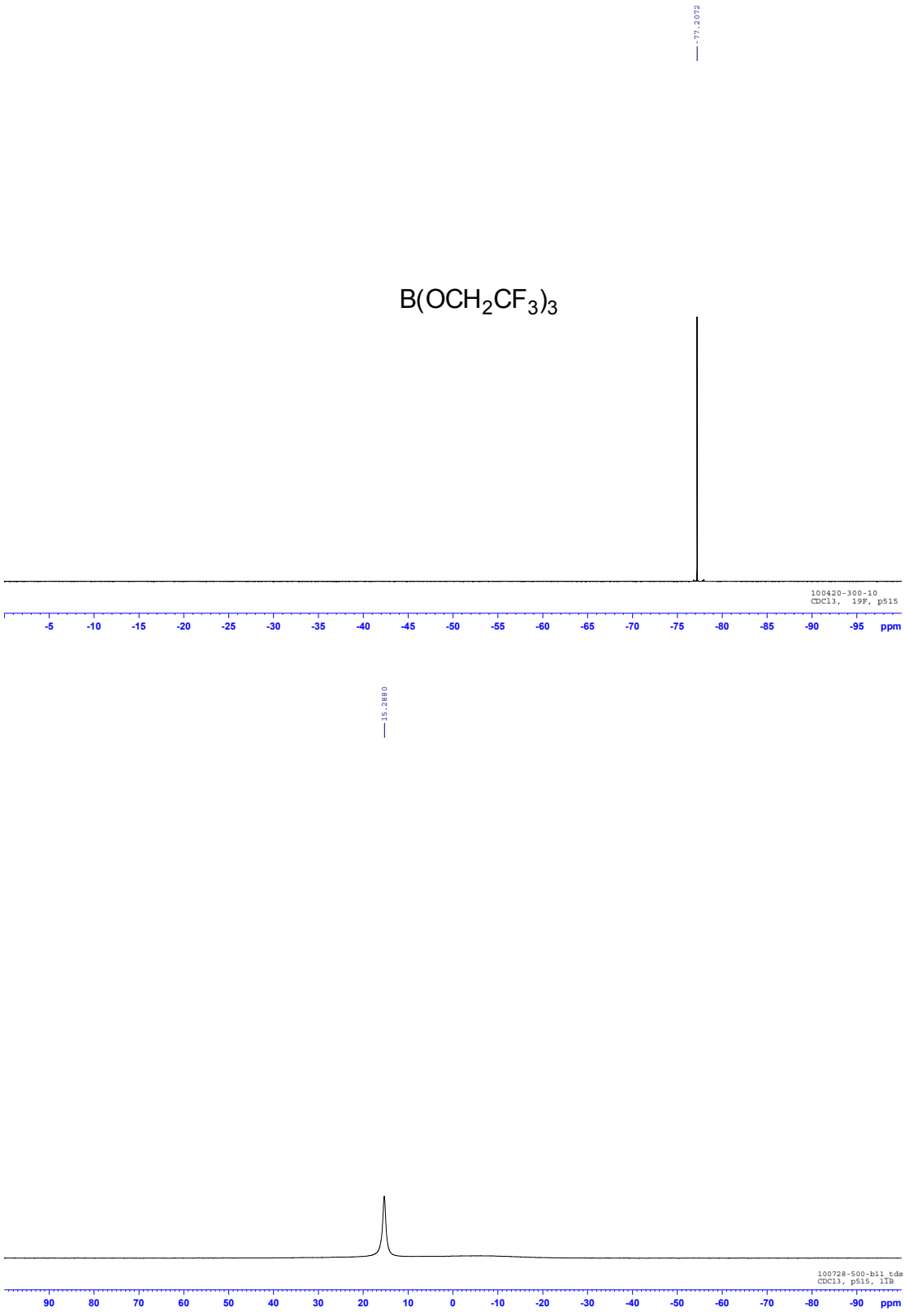
http://dx.doi.org/10.1080/00304940009356751

4 Spectra

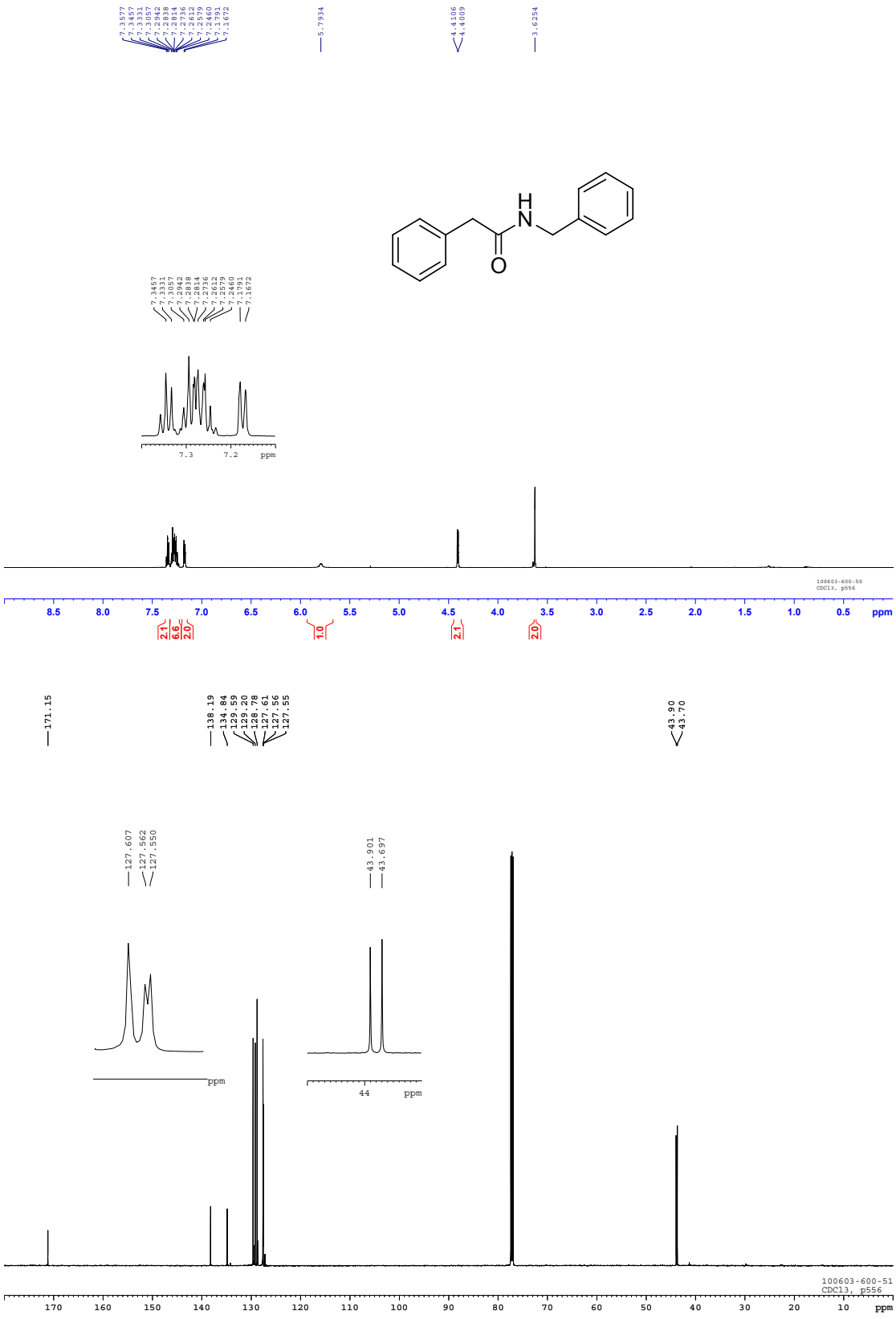
Tris(2,2,2-trifluoroethyl) borate



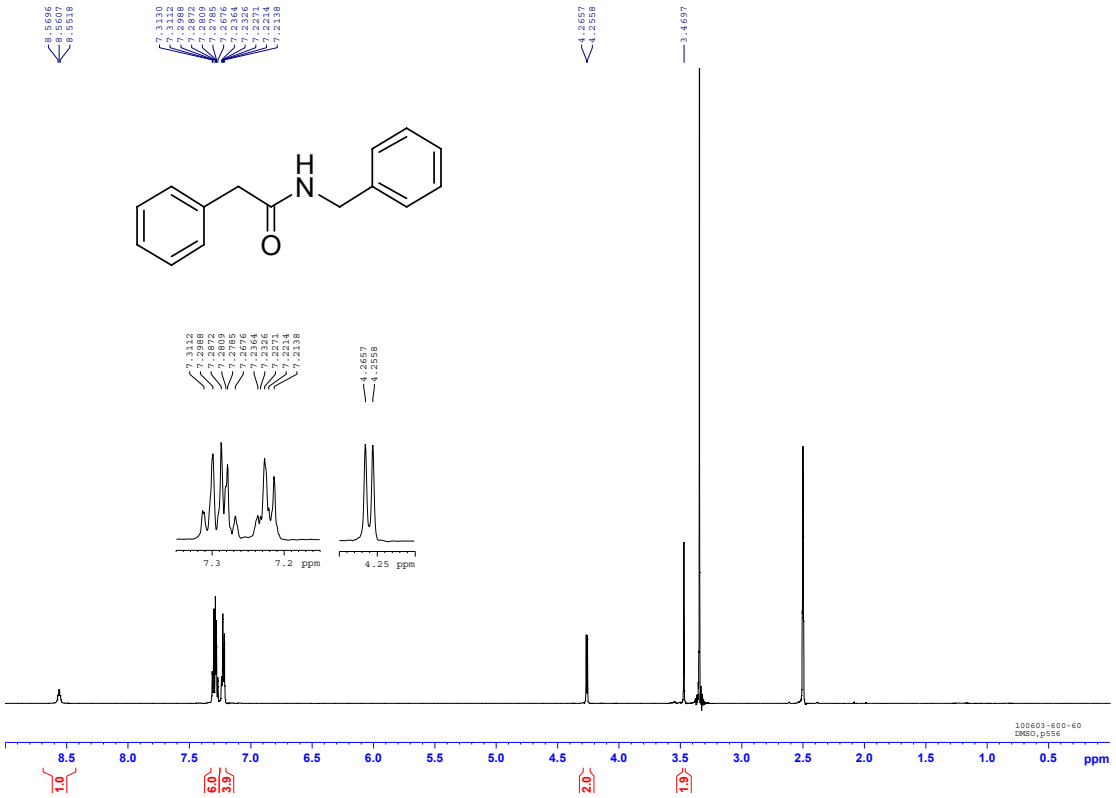
Tris(2,2,2-trifluoroethyl) borate (^{19}F and ^{11}B NMR)

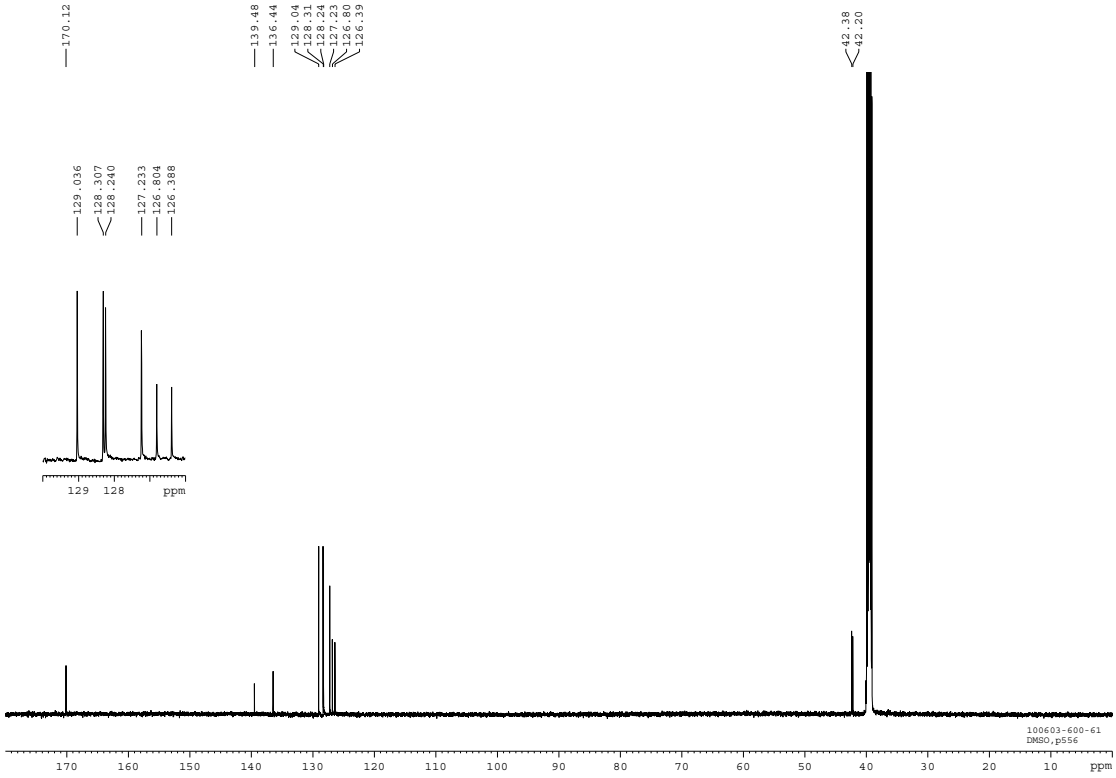


N-Benzyl-2-phenylacetamide ($CDCl_3$)

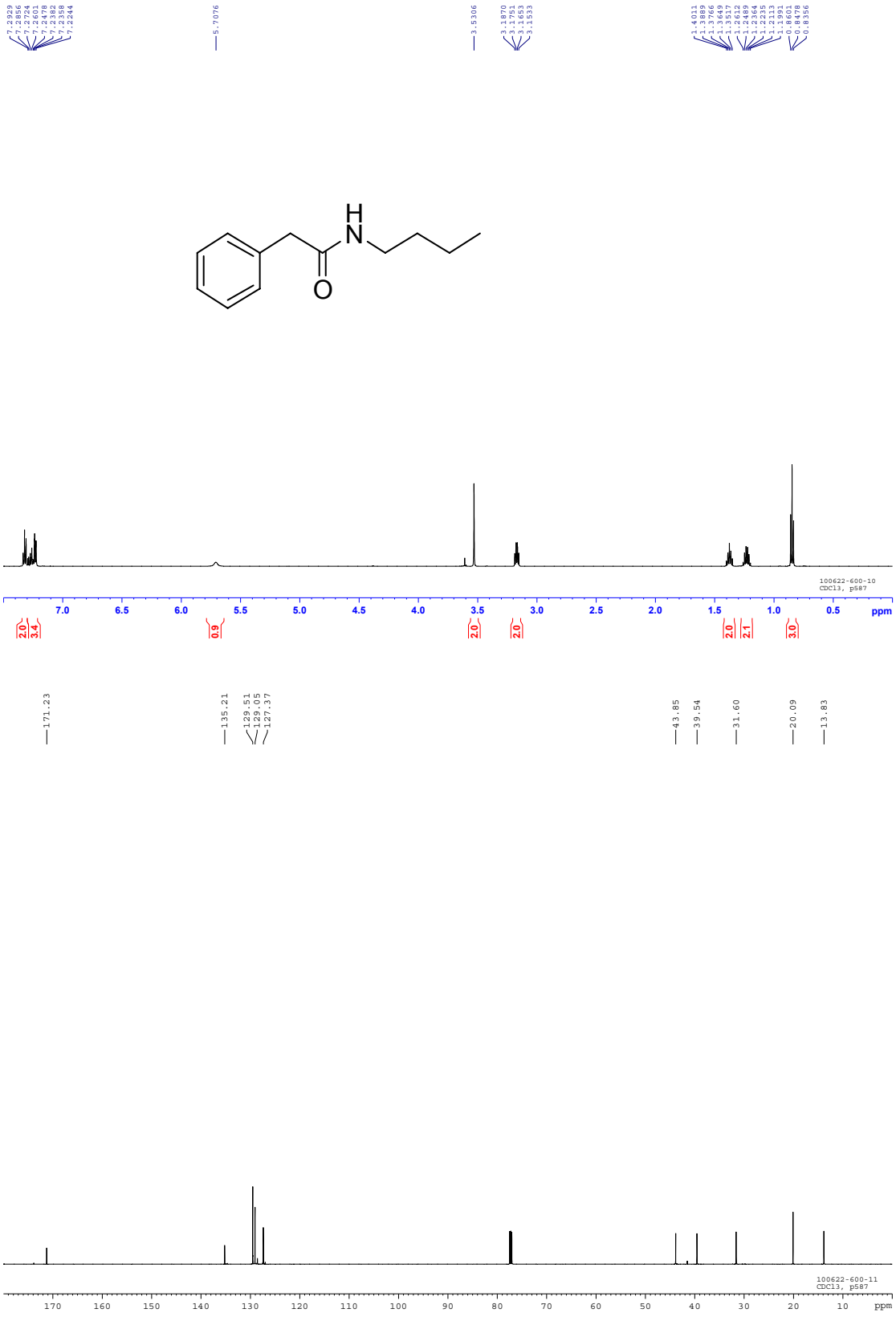


N-Benzyl-2-phenylacetamide (DMSO-d_6)

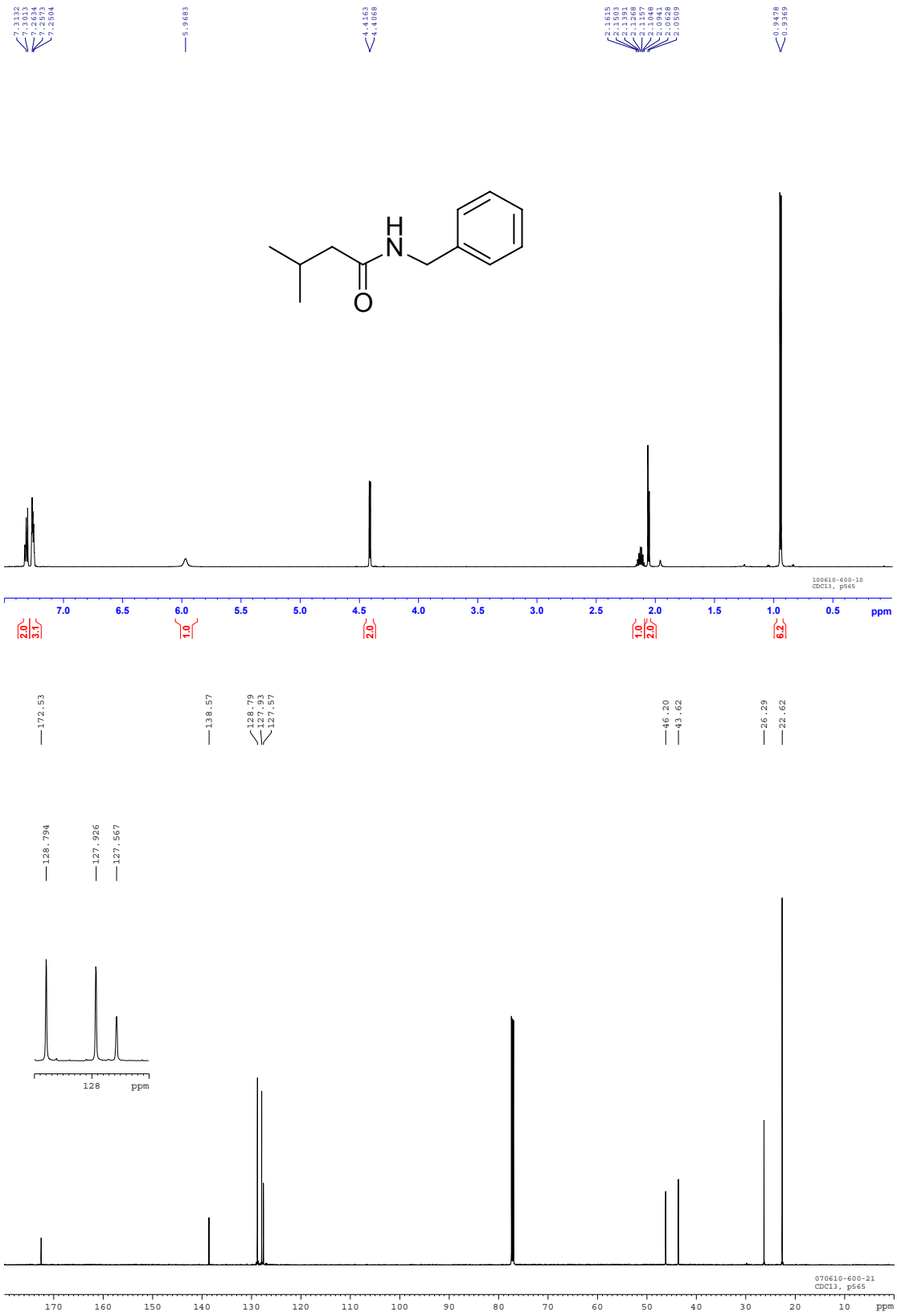




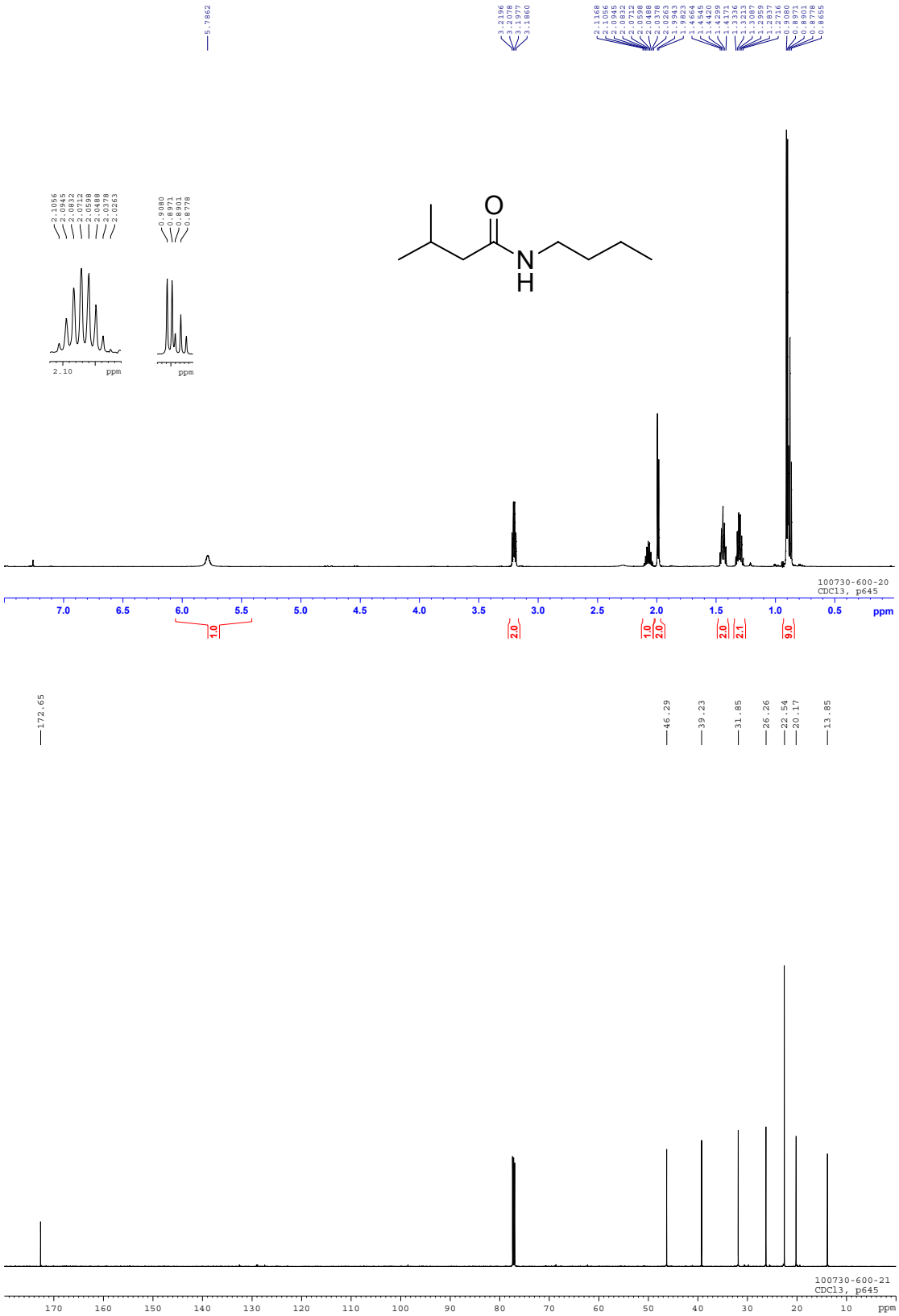
N-Butyl-2-phenylacetamide



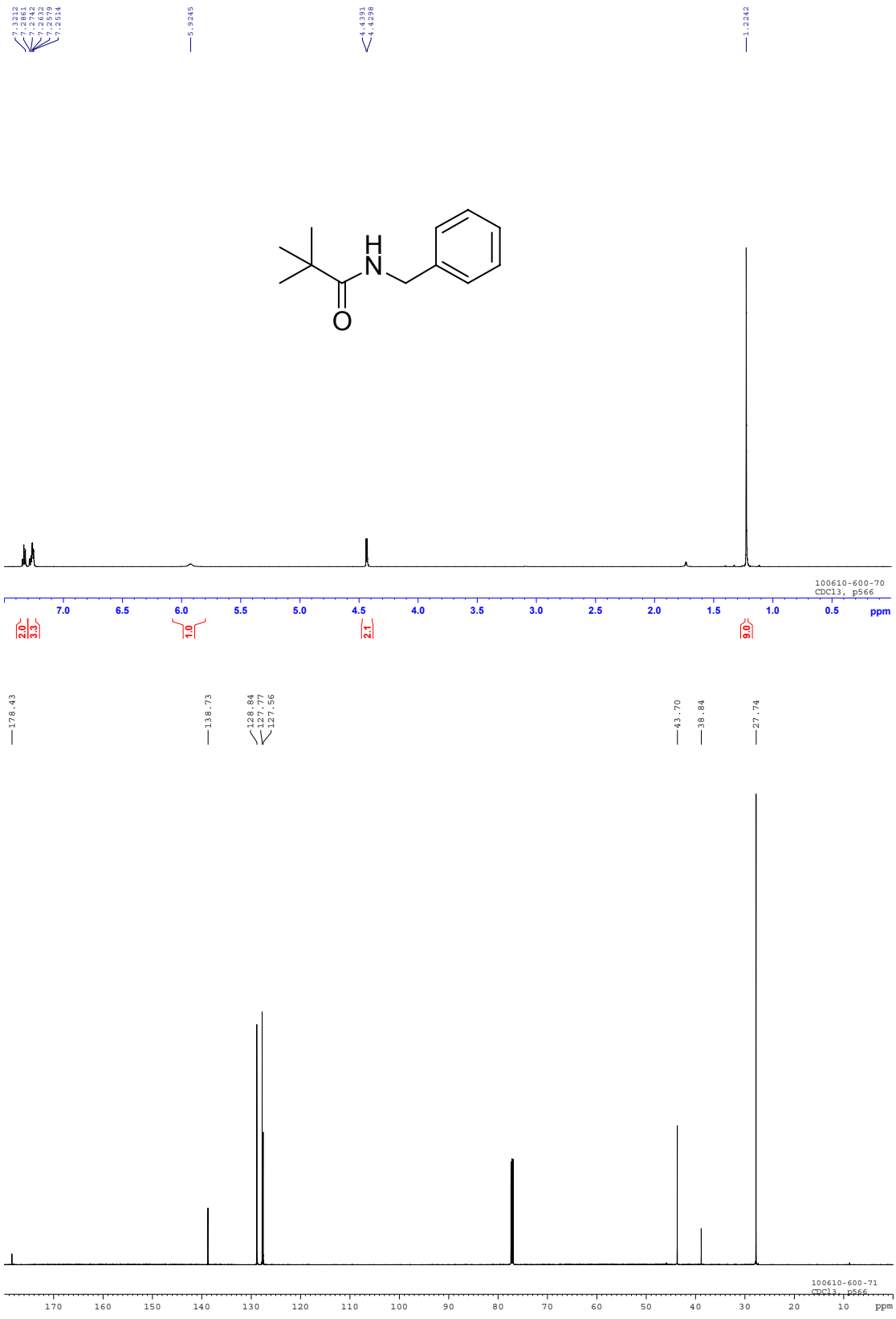
N-Benzyl-3-methylbutanamide ($CDCl_3$)



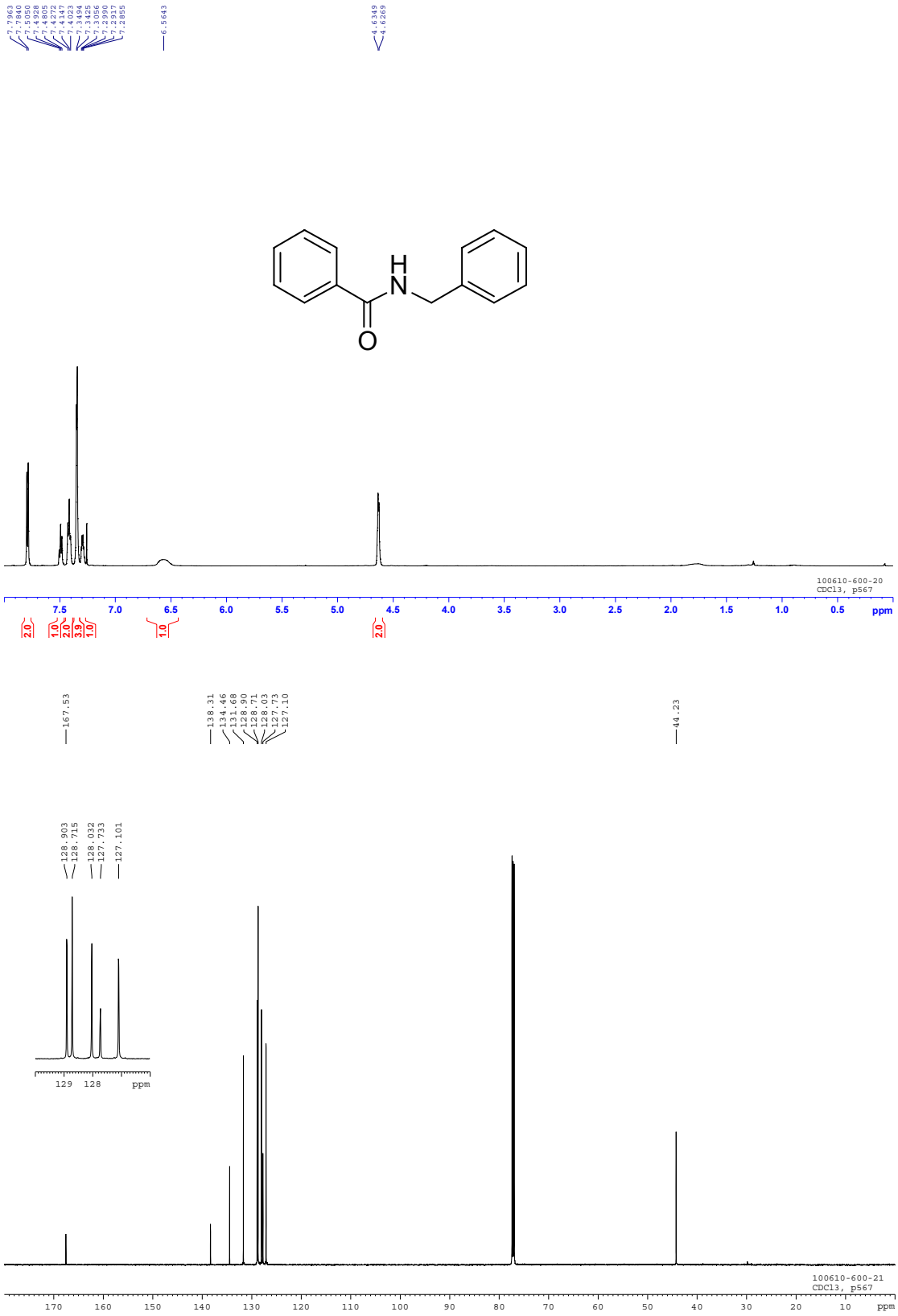
N-Butyl-3-methylbutanamide



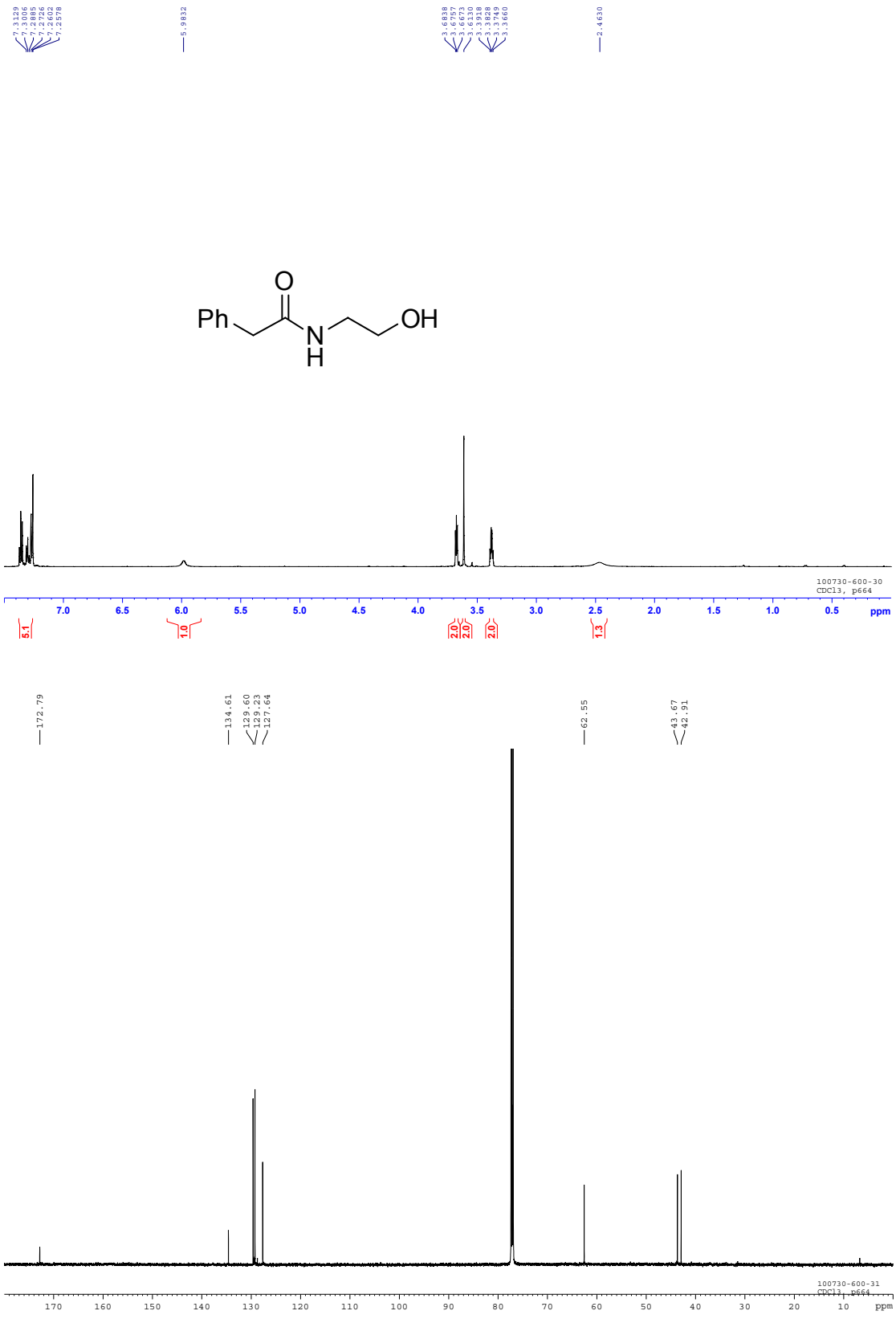
N-Benzylpivalamide



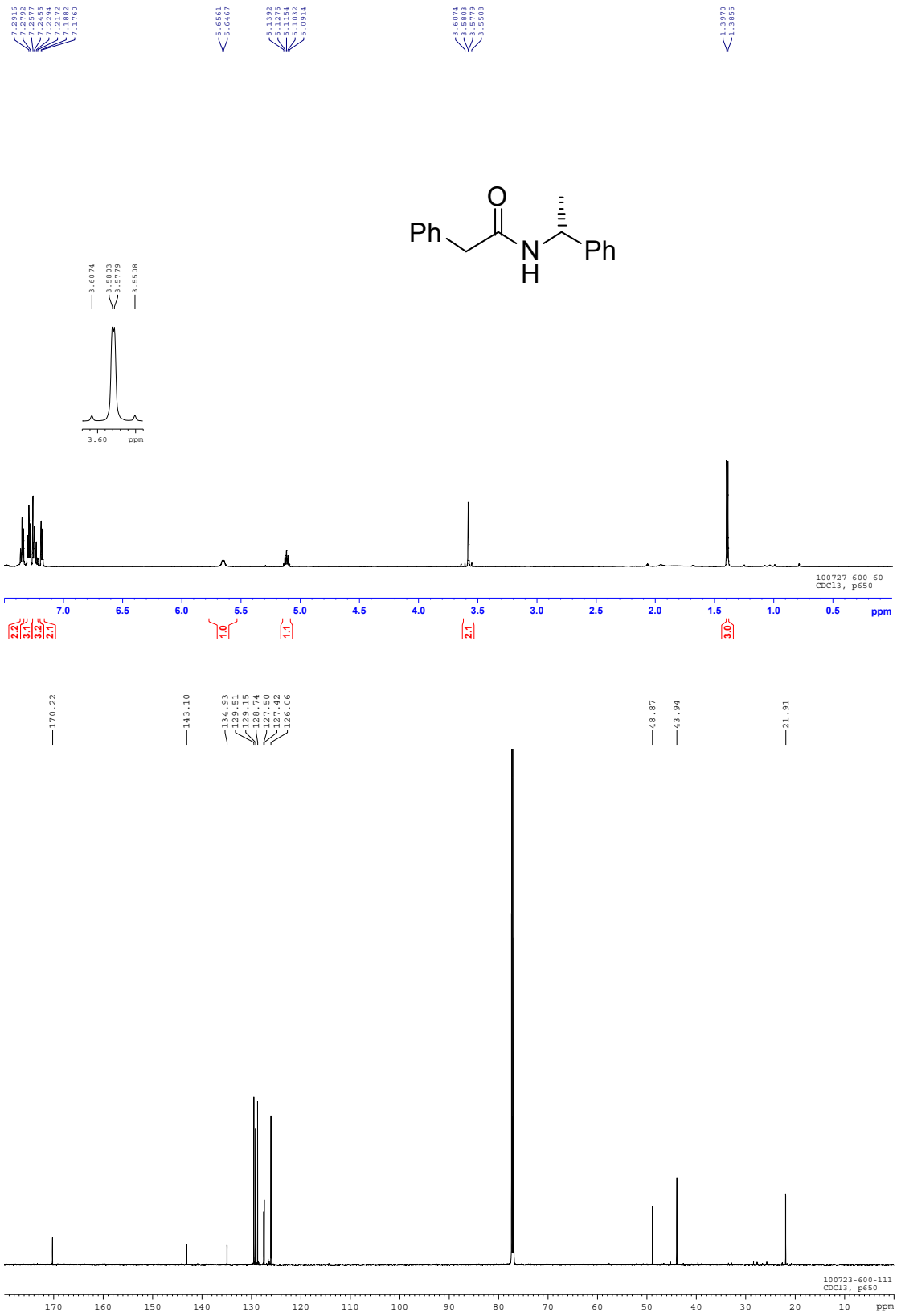
N-Benzylbenzamide



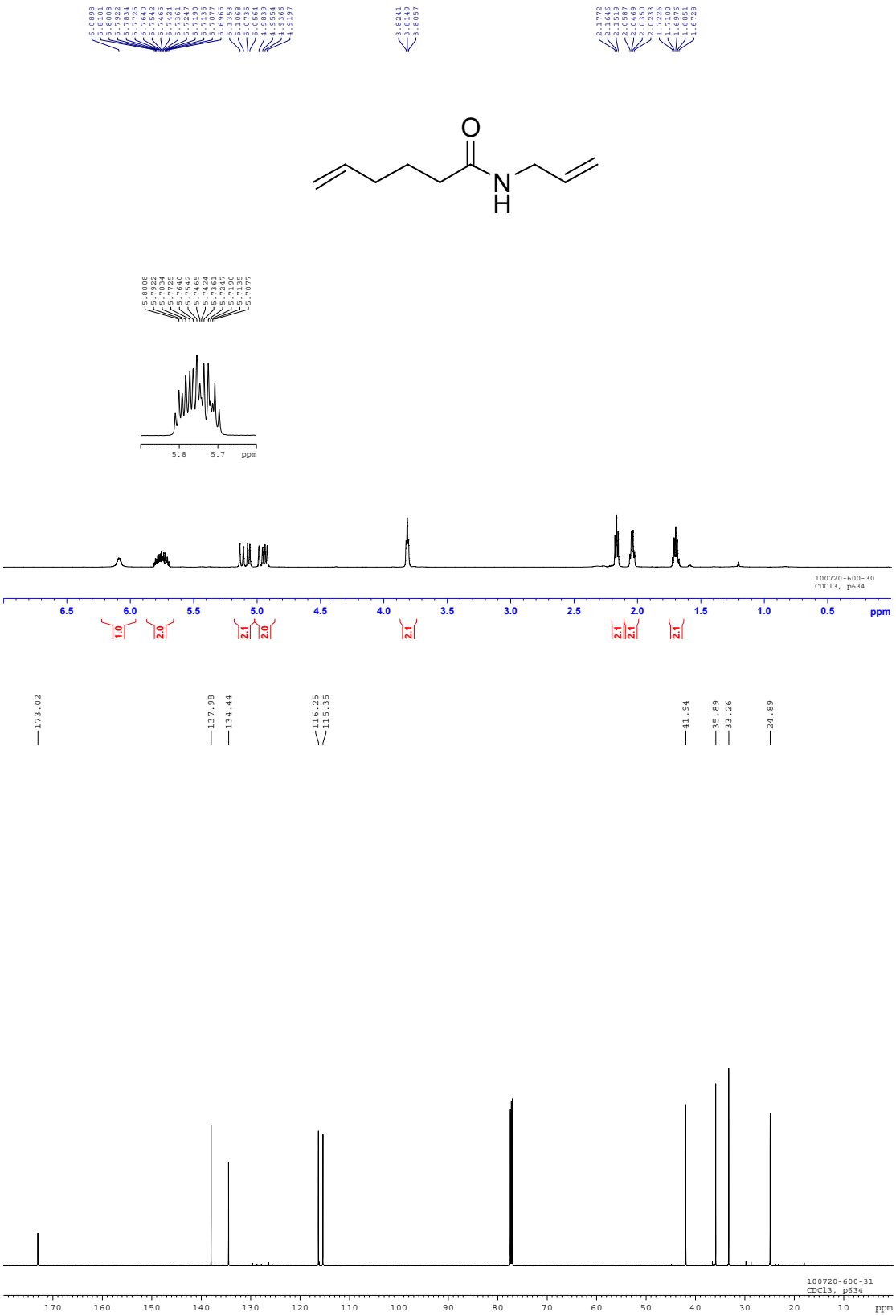
N-(2-Hydroxyethyl)-2-phenylacetamide



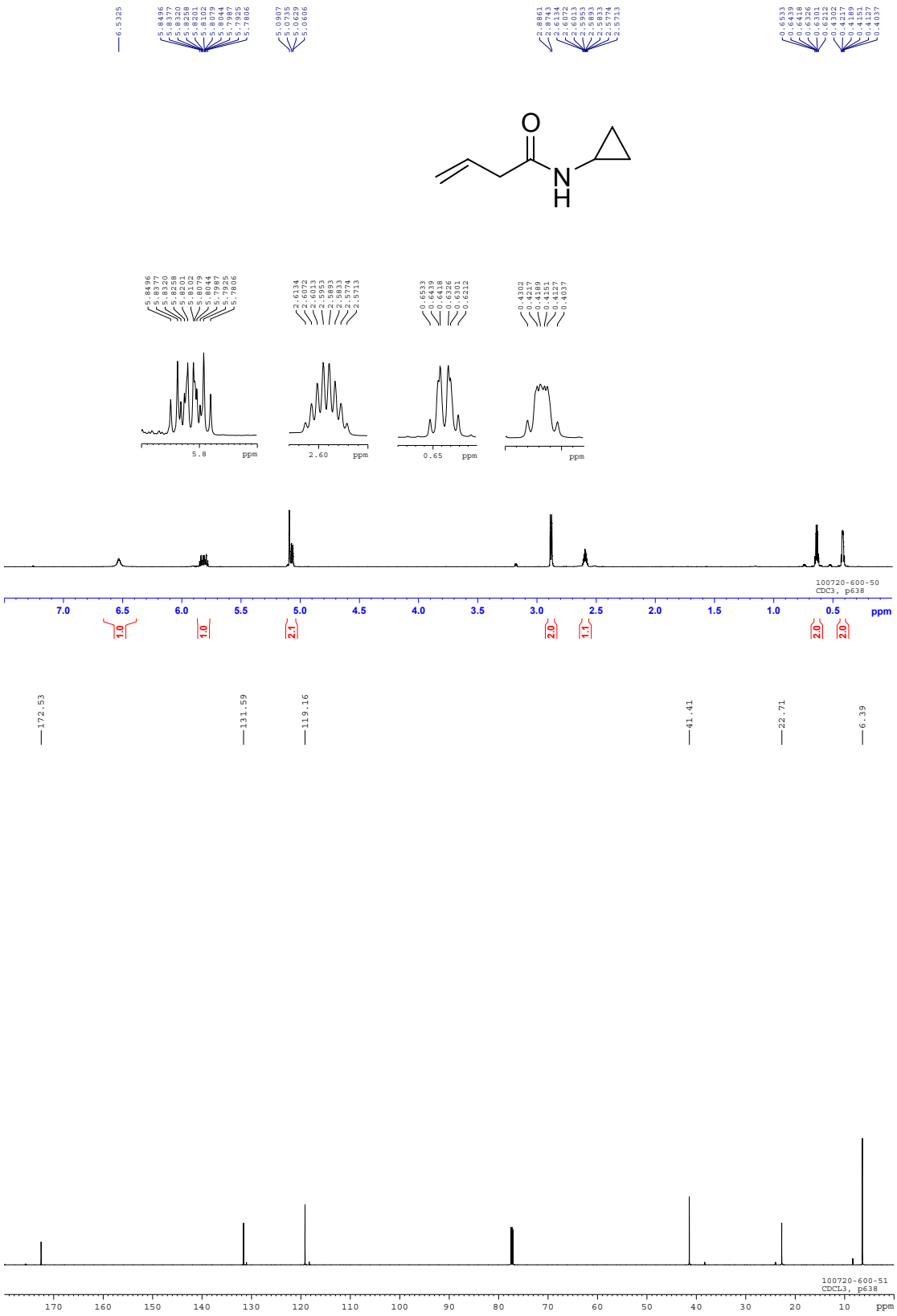
(*R*)-2-Phenyl-*N*-(1-phenylethyl)acetamide



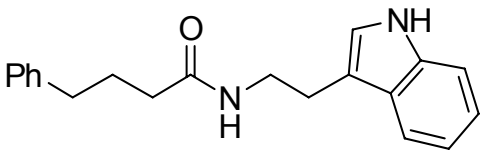
N-Allylhex-5-enamide

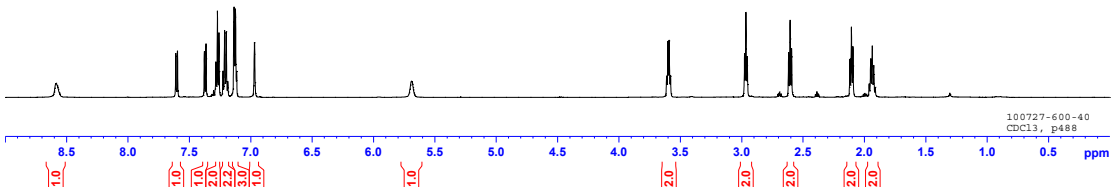


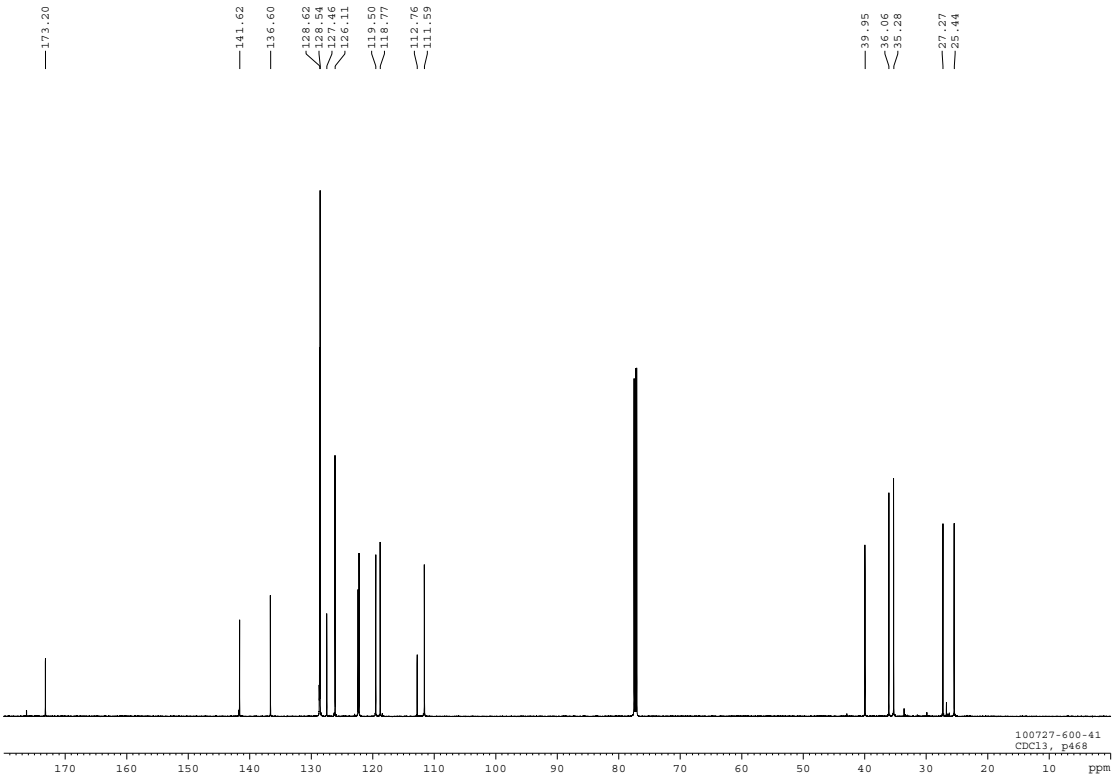
N-Cyclopropylbut-3-enamide



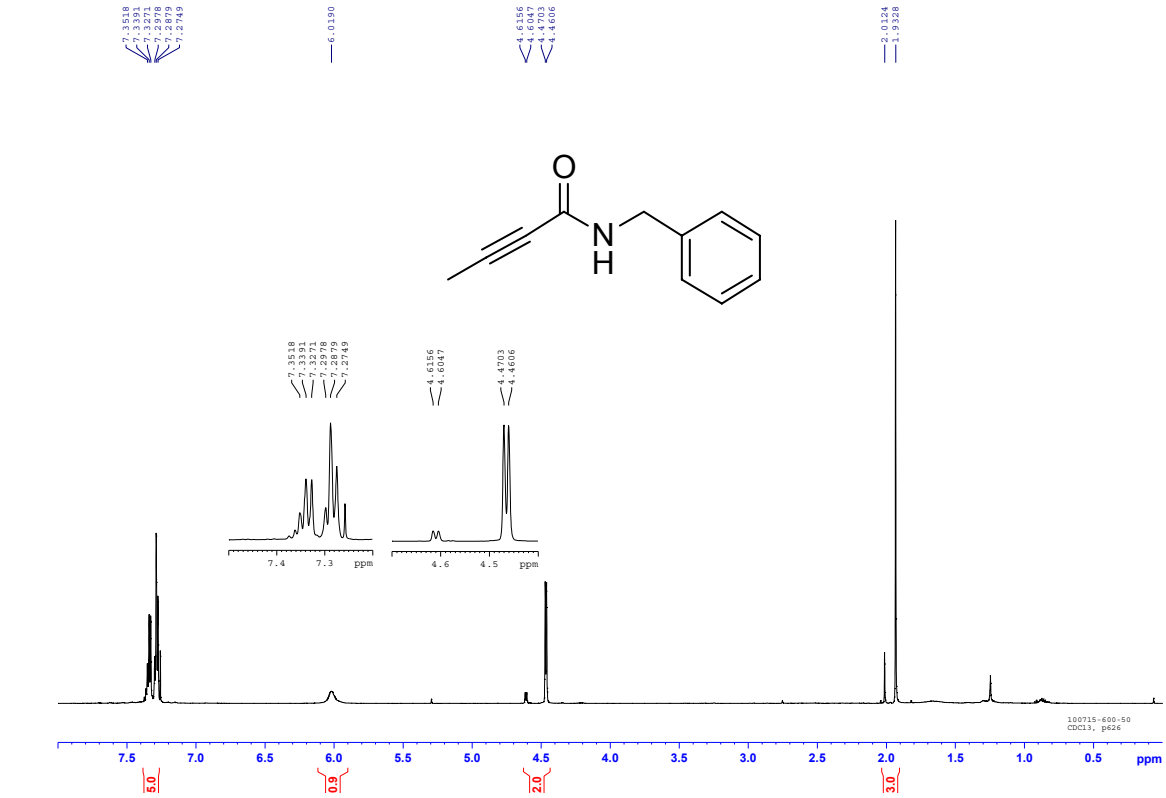
N-(2-(1*H*-Indol-2-yl)ethyl)-5-phenylbutanamide

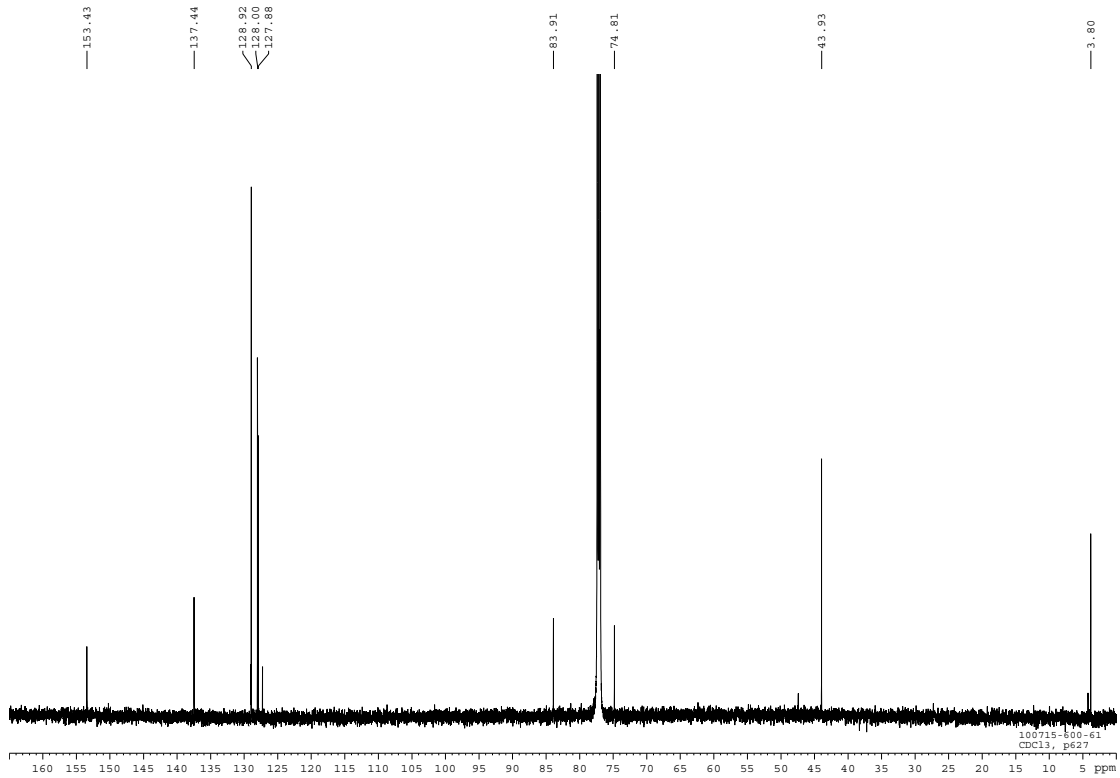




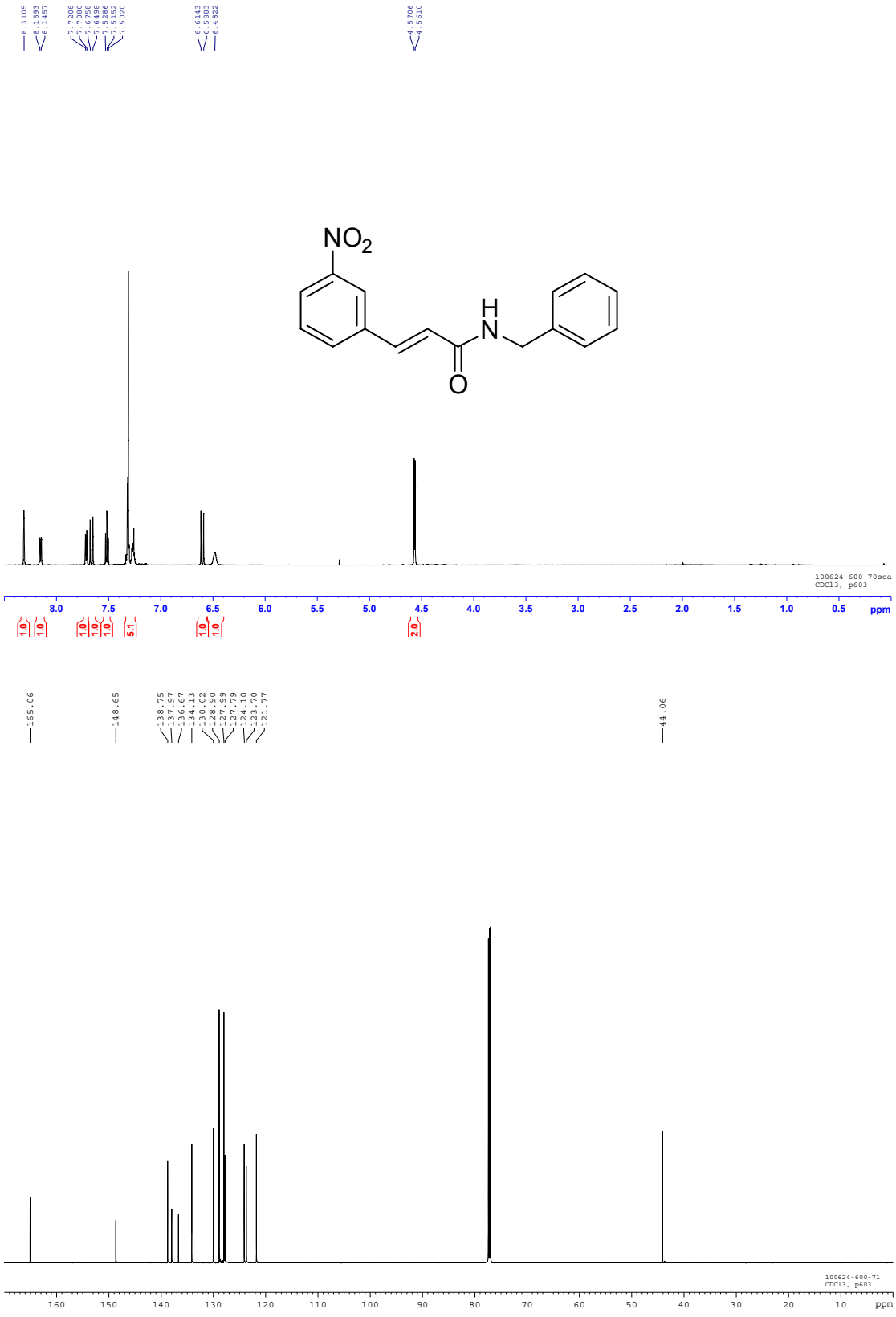


N-Benzylbut-2-ynamide

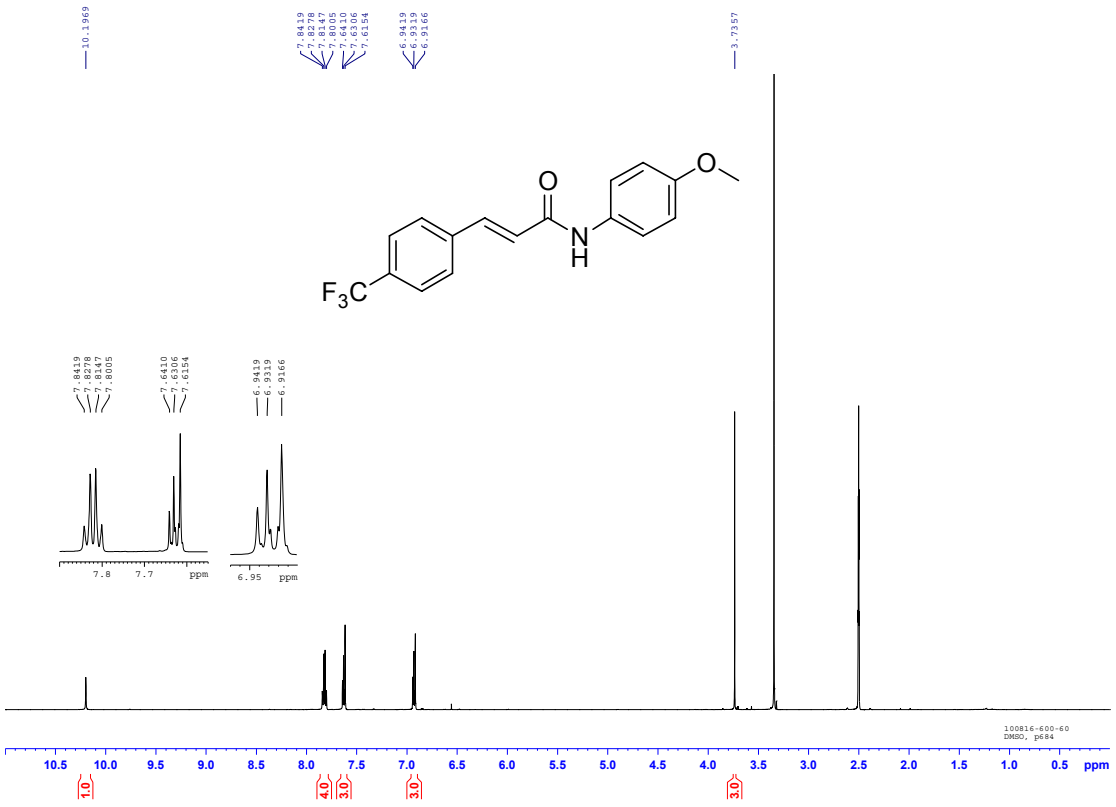


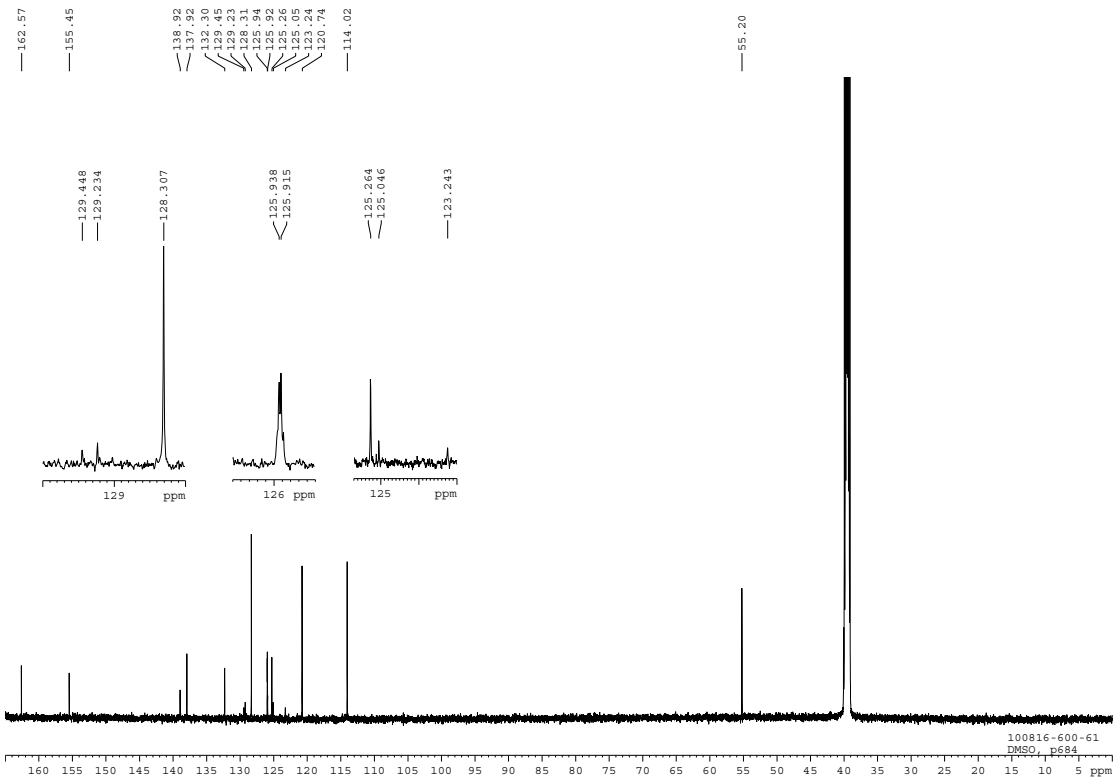


(*E*)-*N*-Benzyl-3-(3-nitrophenyl)acrylamide

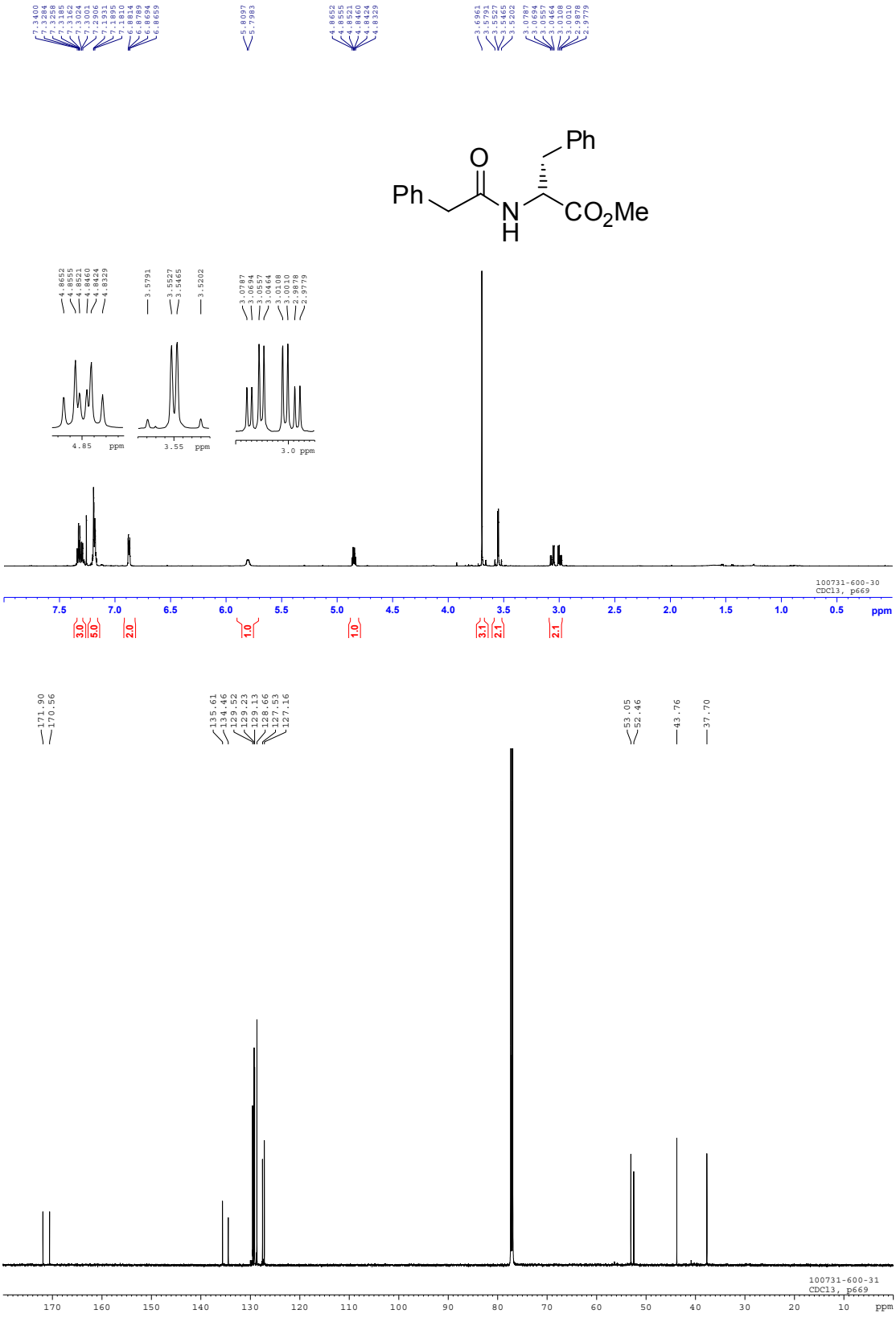


(*E*)-*N*-(4-Methoxyphenyl)-3-(4-(trifluoromethyl)phenyl)acrylamide

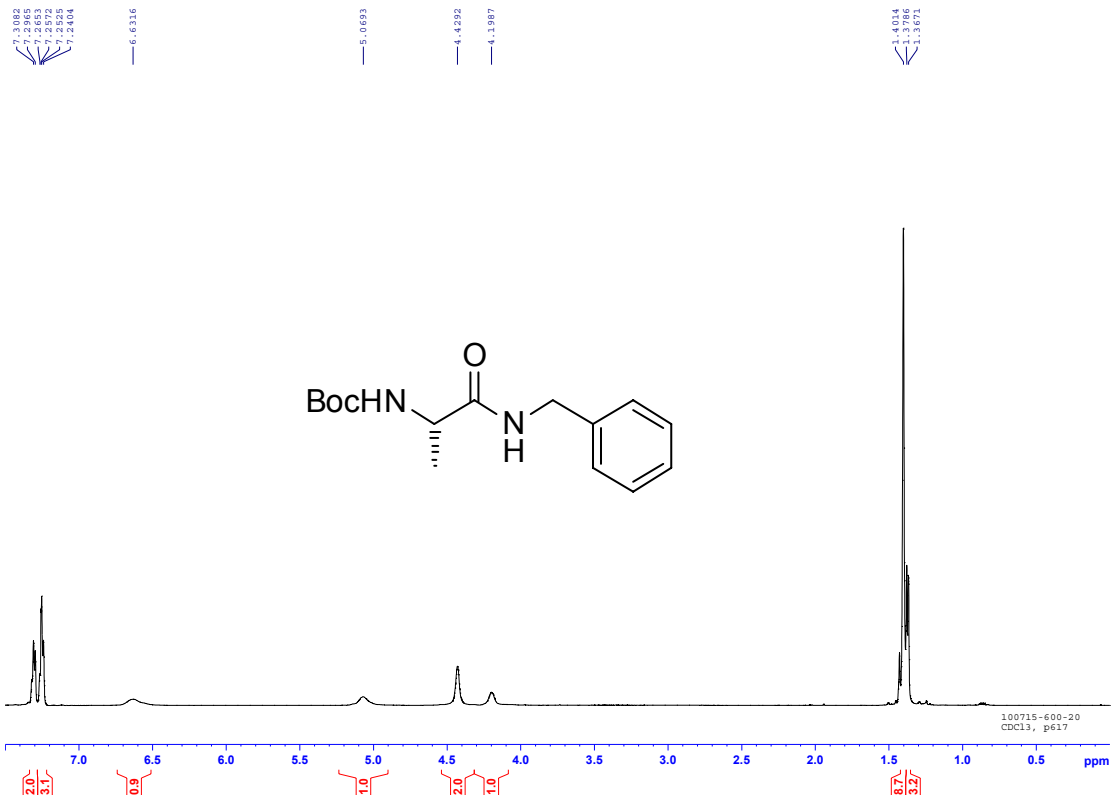


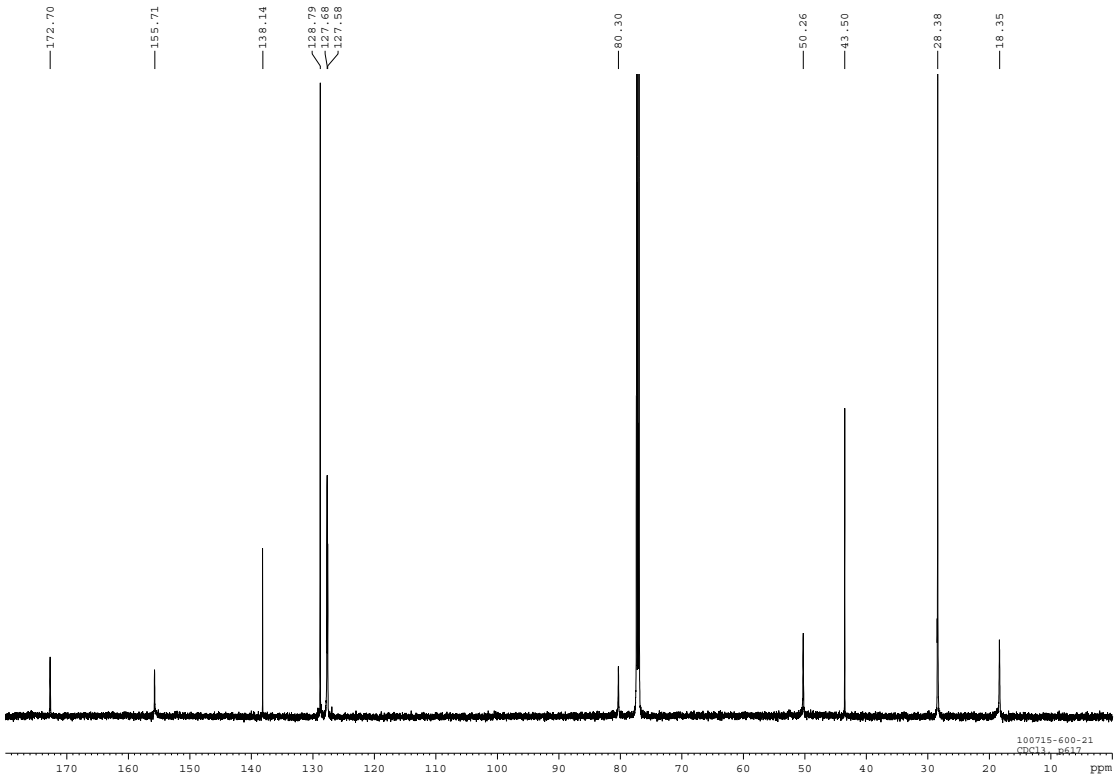


(R)-Methyl 3-phenyl-2-(2-phenylacetamido)propanoate



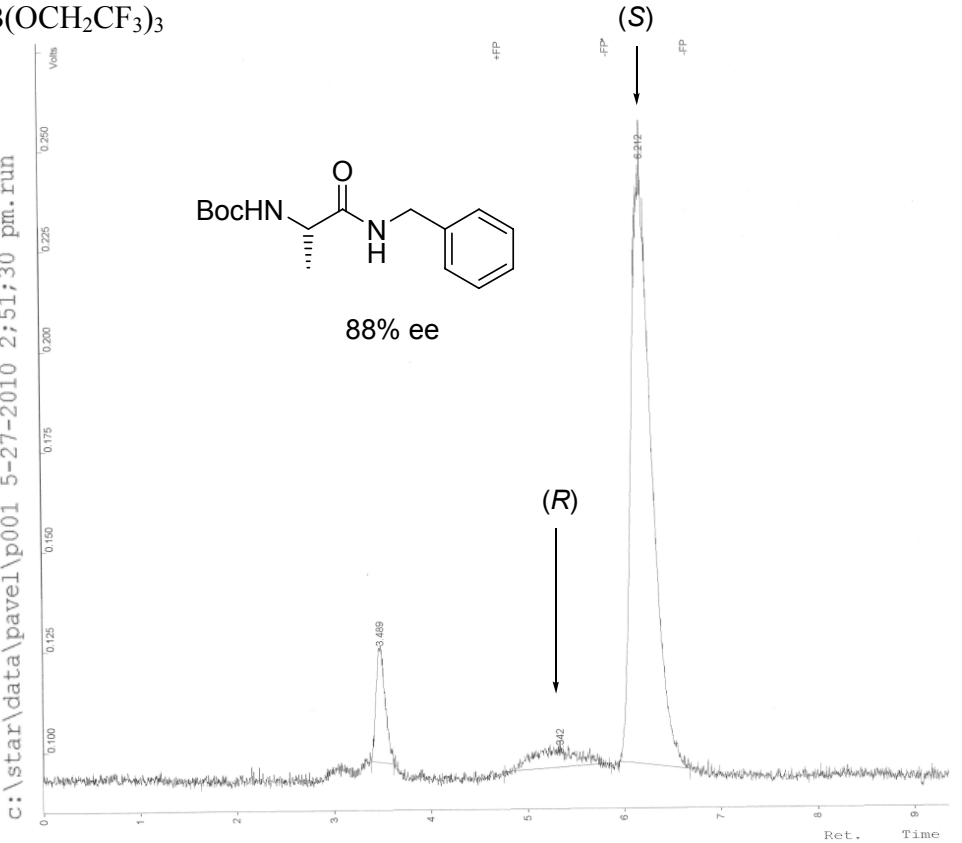
tert-Butyl (1-(benzylamino)-1-oxopropan-2-yl)carbamate





tert-Butyl (1-(benzylamino)-1-oxopropan-2-yl)carbamate

$B(OCH_2CF_3)_3$



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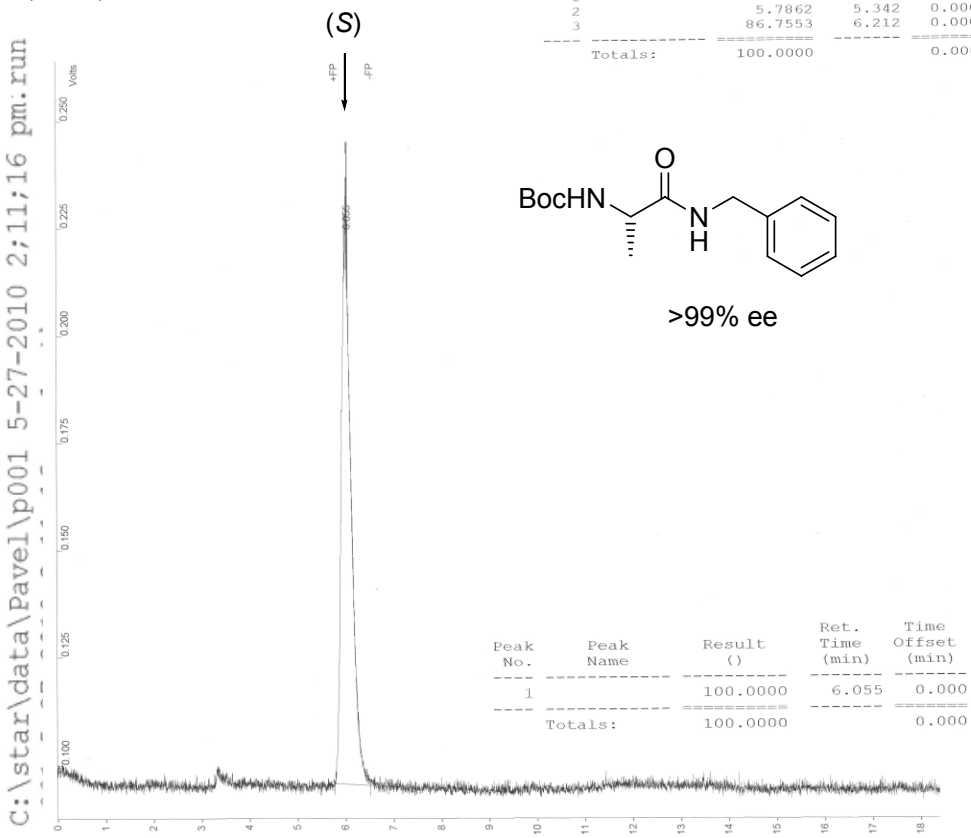
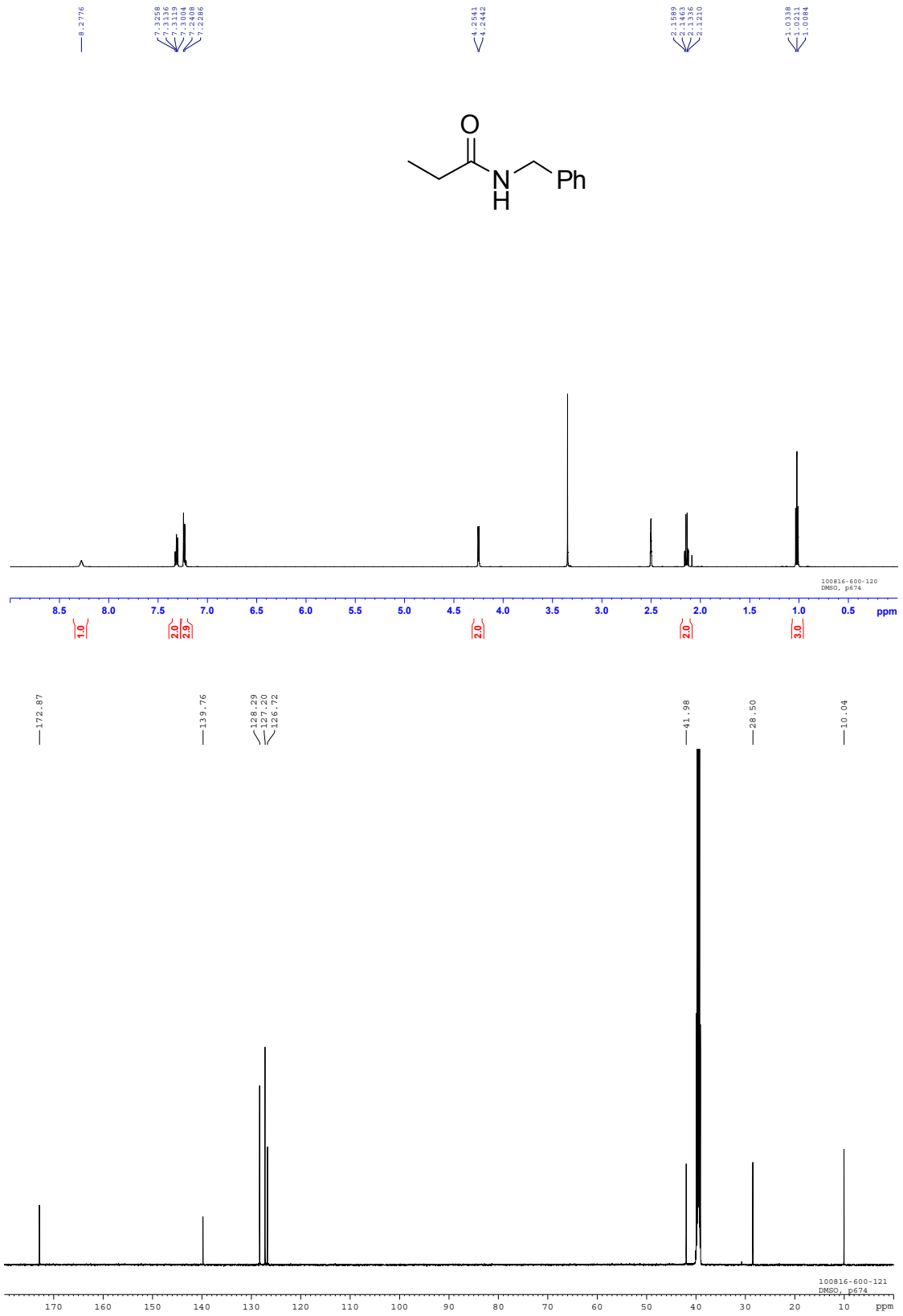
3		86.7553	6.212	0.000	2173177	BB	12.5	
	Totals:	100.0000		0.000	2504951			

$B(OMe)_3$

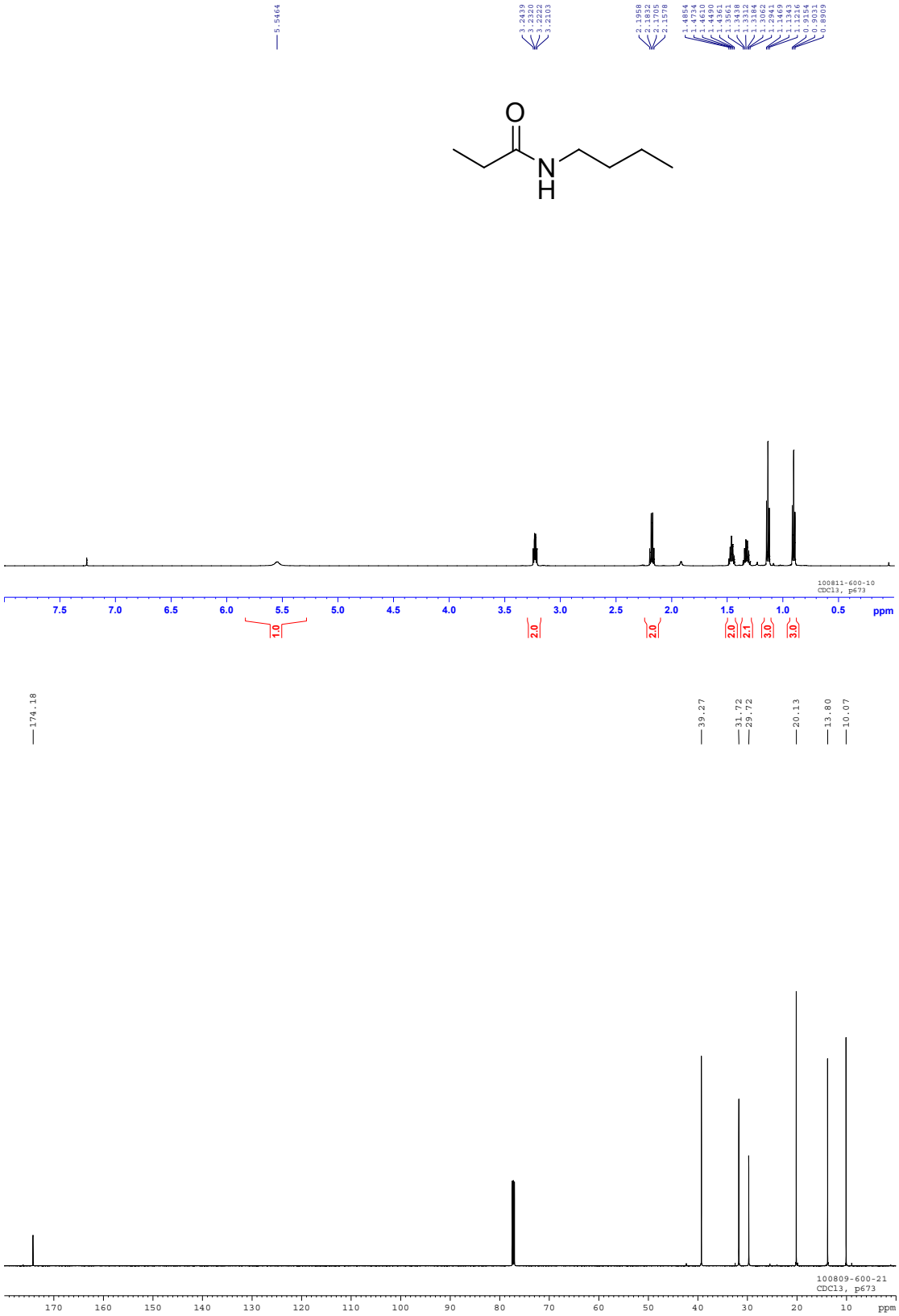


Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		100.0000	6.055	0.000	1858101	BB	11.4	
	Totals:	100.0000		0.000	1858101			

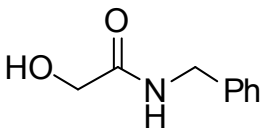
N-Benzylpropionamide

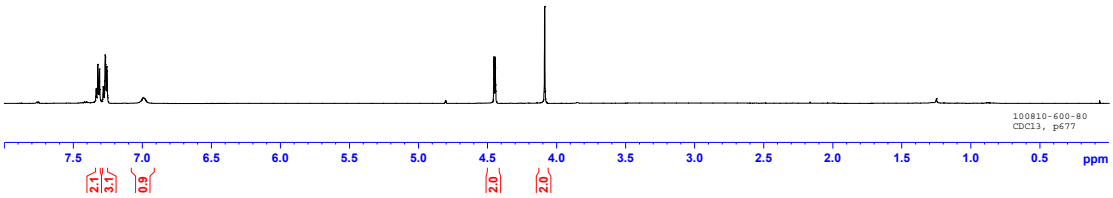


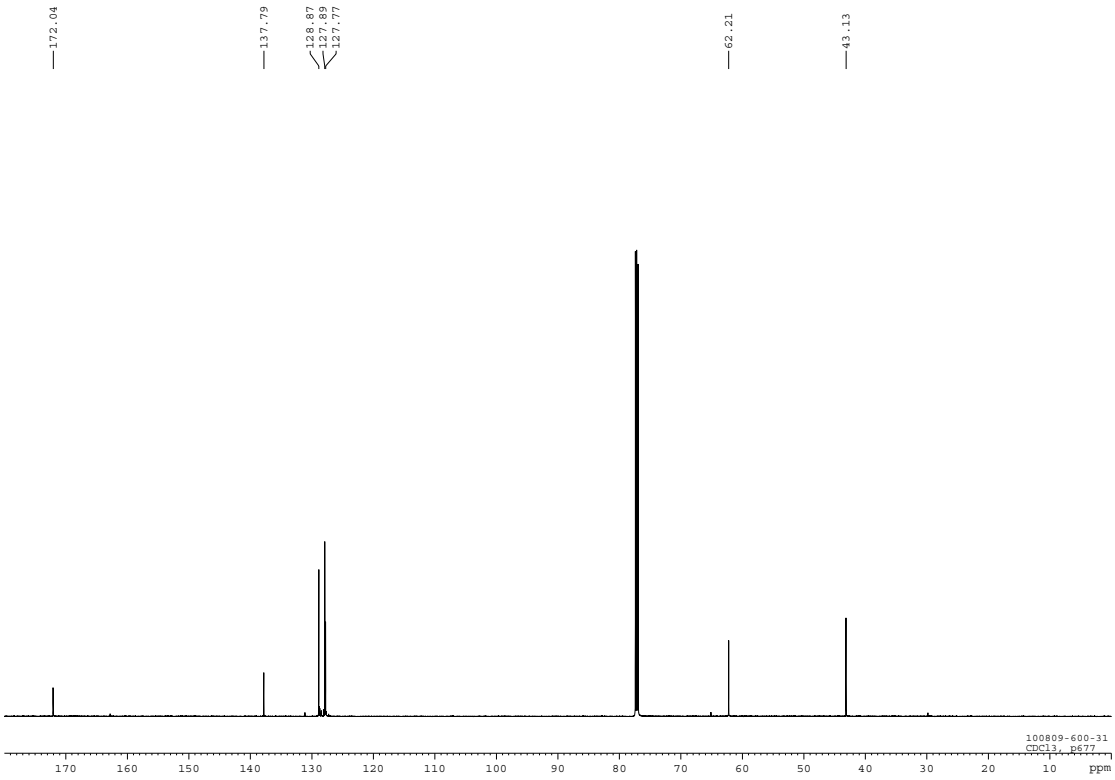
N-Butylpropionamide



N-Benzyl-2-hydroxyacetamide







_N_-((1_H_-indol-3-yl)methyl)-2-hydroxyacetamide