Supplementary Information

for

Phosphorus Oxychloride as an Efficient Coupling Reagent for the Synthesis of Ester, Amide and Peptide under Mild Conditions

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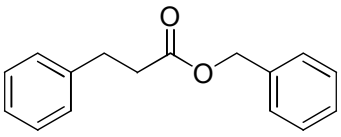
General:

All reactions were carried out under N_2 atmosphere. All reagents were purchased and used without further purification. The solvent was freshly distilled. All new compounds were further characterized by HRMS(ESI).

Typical Procedure for Ester/Amide Synthesis. A solution of acid (0.5 mmol), alcohol or amine (0.6 mmol) and triethylamine (1.2 mmol) in anhydrous CH_2Cl_2 (1.5 mL) under N_2 atmosphere was added a solution of DMAP (0.15 mmol) in anhydrous CH_2Cl_2 (0.5 mL). After stirring for 5 min, $POCl_3$ (0.5 mmol) in anhydrous CH_2Cl_2 (1 mL) was added into the above reaction. After completion of reaction at room temperature (ca. 2 h, monitored through TLC), the reaction mixture was diluted with CH_2Cl_2 (30 mL). The organic phase was washed sequentially with ice-cooled water(10 mL), 10% aqueous HCl (10 mL), saturated aqueous $NaHCO_3$ solution (10 mL), and brine. Drying over anhydrous Na_2SO_4, removal of solvent under vacuum, and purification of the residue by silica gel column chromatography (petroleum ether/ethyl acetate 9/1~5/1) afforded the pure product.

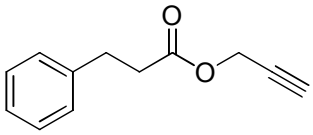
Synthesis of Dipeptide: A solution of the *N*-protected amino acid (0.5 mmol), methyl ester of amino acid hydrochloride (0.6 mmol) and triethylamine (1.8 mmol) in anhydrous CH_2Cl_2 (1.5 mL) under N_2 atmosphere was added a solution of 4-(dimethylamino) pyridine (0.15 mmol) in anhydrous CH_2Cl_2 (0.5 mL). After stirring for 5 min, the reaction mixture was added a solution of the $POCl_3$ (0.5 mmol) in anhydrous CH_2Cl_2 (1 mL), and stirring was continued at room temperature. After completion of reaction (ca. 2 h, TLC monitoring), the reaction mixture was diluted with CH_2Cl_2(30 mL). The organic phase was washed sequentially with ice-cooled water(10 mL), 10% aqueous HCl (10 mL), saturated aqueous $NaHCO_3$ solution (10 mL), and brine. Drying over anhydrous Na_2SO_4, removal of solvent under vacuum, and purification of the residue by silica gel column chromatography (petroleum ether/ethyl acetate 9/1~5/1) afforded the pure product. Enantiomeric purity of the products formed was verified by HPLC analysis. HPLC conditions: column, CHIRAL-CEL (OD); mobile phase, 2~10% *i*-PrOH in *n*-hexane; flowrate, 1mL/min; UV detection at 225 nm.

*Benzyl 3-phenylpropanoate (**3a**) (CAS no: 22767-96-0)*



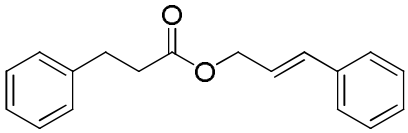
115 mg, 96% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.23 (m, 7H), 7. 21 – 7.17 (m, 3H), 5.10 (s,2H), 2.97 (t, *J* = 7.8 Hz, 2H), 2.68 (t, *J* = 7.8 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.7, 140.4, 135.9, 128.6, 128.5, 128.4, 128.3, 128.2, 126.2, 66.2, 35.9, 30.9. ESI-MS: *m/z* 241.0 ($[M+H]^+$).

*Prop-2-yn-1-yl 3-phenylpropanoate (**3b**) (CAS no: 23522-64-7)*



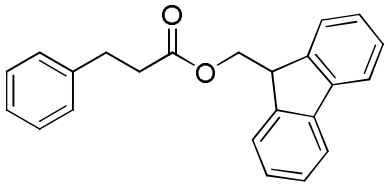
89 mg, 92% yield.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 – 7.26 (m, 2H), 7. 21 – 7.18 (m, 3H), 4.67 (d, *J* = 2.5 Hz, 2H), 2.96 (t, *J* = 7.8 Hz, 2H), 2.69 – 2.65 (m, 2H), 2.46 (t, *J* = 2.5 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.9, 140.1, 128.5, 128.2, 126.3, 77.6, 74.8, 51.9, 35.5, 30.7. ESI-MS: *m/z* 189.1 ($[M+H]^+$).

*Cinnamyl 3-phenylpropanoate (**3c**) (CAS no: 28048-98-8)*



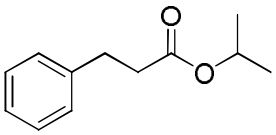
120 mg, 90% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.36 (m, 2H), 7.33 – 7.24 (m, 5H), 7.23 – 7.17 (m, 3H), 6.61 (d, *J* = 15.9 Hz, 1H), 6.24 (dt, *J* = 15.9, 6.4 Hz, 1H), 4.72 (dd, *J* = 6.5, 1.2 Hz, 2H), 2.97 (t, *J* = 7.8 Hz, 2H), 2.67 (dd, *J* = 9.3, 6.4 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.6, 140.4, 136.1, 134.1, 128.6, 128.5, 128.3, 128.0, 126.6, 126.2, 123.1, 65.0, 35.9, 30.9. ESI-MS: *m/z* 267.1 ($[M+H]^+$).

*(9H-fluoren-9-yl)Methyl 3-phenylpropanoate (**3d**)*



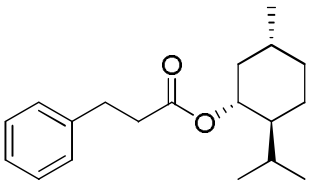
154 mg, 94% yield. IR (KBr, film), υ_{max} (cm^{-1}): 3063, 3027, 2948, 2359, 1736, 1450, 1291, 1250, 1159, 758, 740, 699; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 7.6 Hz, 2H), 7.52 (dd, *J* = 7.5, 0.7 Hz, 2H), 7.38 (t, *J* = 7.3 Hz, 2H), 7.30 – 7.26 (m, 4H), 7.21 – 7.18 (m, 3H), 4.38 (d, *J* = 7.1 Hz, 2H), 4.16 (t, *J* = 7.1 Hz, 1H), 2.95 (t, *J* = 7.8 Hz, 2H), 2.72 – 2.68 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.8, 143.7, 141.3, 140.4, 128.5, 128.2, 127.7, 127.1, 126.3, 125.0, 112.0, 66.3, 46.8, 35.9, 30.8. HRMS(ESI): *m/z* 329.1549 ($[M+H]^+$, $C_{23}H_{21}O_2^+$ calcd. 329.1542).

*Isopropyl 3-phenylpropanoate (**3e**) (CAS no: 22767-95-9)*



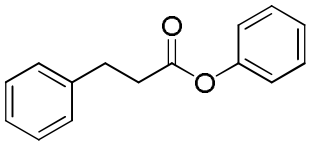
90 mg, 94% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 – 7.25 (m, 2H), 7.21 – 7.17 (m, 3H), 5.00 (hept, *J* = 6.3 Hz, 1H), 2.94 (t, *J* = 8.0 Hz, 2H), 2.59 (t, *J* = 8.0 Hz, 2H), 1.20 (d, *J* = 6.3 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.4, 140.6, 128.4, 128.3, 126.1, 67.6, 36.2, 31.0, 21.8. ESI-MS: *m/z* 193.3 ($[M+H]^+$).

*(1R, 2S, 5R)-2-Isopropyl-5-methylcyclohexyl 3-phenylpropanoate (**3f**) (CAS no: 101023-15-8)*



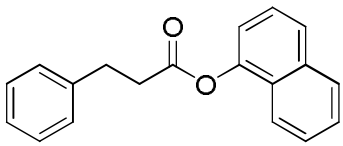
137 mg, 95% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 – 7.25 (m, 2H), 7.21 – 7.17 (m, 3H), 4.67 (td, *J* = 10.9, 4.4 Hz, 1H), 2.97 – 2.93 (m, 2H), 2.61 (dd, *J* = 8.6, 7.0 Hz, 2H), 1.96 – 1.90 (m, 1H), 1.76 – 1.70 (m, 1H), 1.69 – 1.62 (m, 2H), 1.53 – 1.40 (m, 1H), 1.37 – 1.29 (m, 1H), 1.08 – 0.98 (m, 1H), 0.96 – 0.91 (m, 1H), 0.90 – 0.88 (m, 3H), 0.86 – 0.84 (m, 4H), 0.70 (d, *J* = 7.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.5, 140.5, 128.4, 128.3, 126.2, 74.1, 46.9, 40.9, 36.2, 34.2, 31.3, 31.1, 26.1, 23.4, 22.0, 20.7, 16.2. ESI-MS: *m/z* 289.3 ($[M+H]^+$).

*Phenyl 3-phenylpropanoate (**3g**) (CAS no: 726-26-1)*



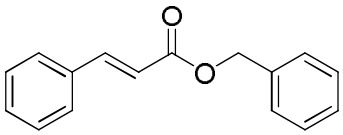
103 mg, 91% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.30 (m, 4H), 7.27 – 7.18 (m, 4H), 7.01 – 6.98 (m, 2H), 3.07 (t, *J* = 7.7 Hz, 2H), 2.90 – 2.86 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.4, 150.6, 140.1, 129.4, 128.6, 128.4, 126.4, 125.8, 121.5, 35.9, 30.9. ESI-MS: *m/z* 227.1($[M+H]^+$).

*Naphthalen-1-yl 3-phenylpropanoate (**3h**)*



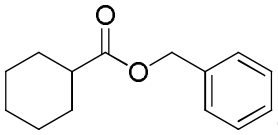
121 mg, 88% yield. IR (KBr, film), υ_{max} (cm^{-1}): 3061, 3028, 2927, 1759, 1598, 1496, 1454, 1390, 1258, 1221, 1125, 794, 771, 698; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 – 7.82 (m, 1H), 7.71 (d, *J* = 8.3 Hz, 1H), 7.57 (dt, *J* = 8.5, 4.3 Hz, 1H), 7.49 – 7.39 (m, 3H), 7.38 – 7.25 (m, 5H), 7.16 (dd, *J* = 7.5, 1.0 Hz, 1H), 3.19 – 3.14 (m, 2H), 3.09 – 3.04 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.4, 146.5, 140.1, 134.6, 128.7, 128.5, 127.9, 126.7, 126.5, 126.4, 126.3, 126.0, 125.3, 121.1, 118.0, 35.9, 31.0. HRMS(ESI): *m/z* 277.1240 ($[M+H]^+$, $C_{19}H_{17}O_2^+$ calcd. 277.1229).

*Benzyl cinnamate (**3i**) (CAS no: 103-41-3)*



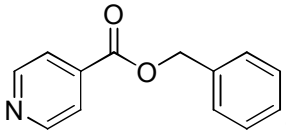
107 mg, 90% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 (d, *J* = 16.0 Hz, 1H), 7.53 – 7.49 (m, 2H), 7.43 – 7.31 (m, 8H), 6.48 (d, *J* = 16.0 Hz, 1H), 5.25 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.8, 145.1, 136.0, 134.3, 130.3, 128.8, 128.6, 128.2, 128.2, 128.1, 117.8, 66.3. ESI-MS: *m/z* 239.3($[M+H]^+$).

*Benzyl cyclohexanecarboxylate (**3j**) (CAS no: 22733-94-4)*



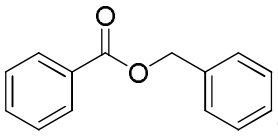
98 mg, 90% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.28 (m, 5H), 5.10 (s, 2H), 2.39 – 2.31 (m, 1H), 1.95 – 1.90 (m, 2H), 1.78 – 1.71 (m, 2H), 1.65 – 1.60 (m, 1H), 1.51 – 1.41 (m, 2H), 1.32 – 1.16 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 175.9, 136.3, 128.5, 128.0, 127.9, 65.8, 43.2, 28.9, 25.7, 25.4. ESI-MS: *m/z* 219.2($[M+H]^+$).

*Benzyl isonicotinate (**3k**) (CAS no: 21182-01-4)*



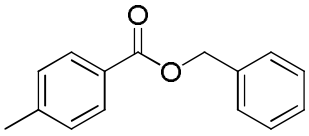
99 mg, 96% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.76 (dd, *J* = 4.4, 1.6 Hz, 2H), 7.86 (dd, *J* = 4.4, 1.6 Hz, 2H), 7.46 – 7.32 (m, 5H), 5.38 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.9, 150.5, 137.2, 135.2, 128.6, 128.5, 128.4, 128.3, 127.4, 126.9, 122.9, 67.4. ESI-MS: *m/z* 214.0($[M+H]^+$).

*Benzyl benzoate (**3l**) (CAS no: 120-51-4)*



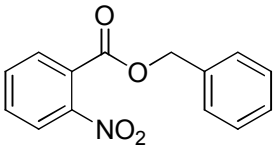
95 mg, 90% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09 – 8.07 (m, 2H), 7.57 – 7.52 (m, 1H), 7.46 – 7.31 (m, 7H), 5.36 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.4, 136.0, 133.0, 130.1, 129.7, 128.6, 128.3, 128.2, 128.1, 66.6. ESI-MS: *m/z* 213.0($[M+H]^+$).

*Benzyl 4-methylbenzoate (**3m**) (CAS no: 5467-99-2)*



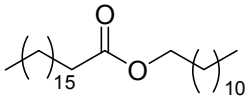
96 mg, 85% yield.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 – 7.95 (m, 2H), 7.45 – 7.42 (m, 2H), 7.39 – 7.30 (m, 3H), 7.23 – 7.21 (m, 2H), 5.34 (s, 2H), 2.39 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.5, 143.7, 136.1, 129.7, 129.0, 128.5, 128.1, 128.0, 127.3, 66.4, 21.6. ESI-MS: *m/z* 227.1($[M+H]^+$).

*Benzyl 2-nitrobenzoate (**3n**) (CAS no: 7579-38-6)*



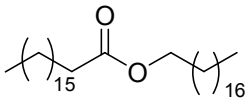
123 mg, 96% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 – 7.88 (m, 1H), 7.75 – 7.73 (m, 1H), 7.67 – 7.58 (m, 2H), 7.42 – 7.32 (m, 5H), 5.35 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.2, 154.8, 148.1, 134.7, 132.9, 131.7, 129.8, 128.7, 128.6, 128.5, 127.5, 123.9, 89.7, 68.3. ESI-MS: *m/z* 258.2($[M+H]^+$).

*Dodecyl stearate (**3o**) (CAS no: 5303-25-3)*



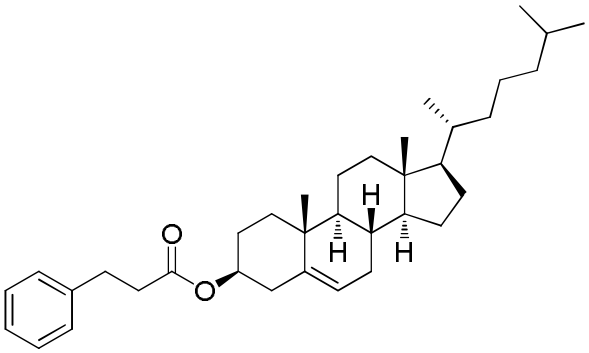
193 mg, 90% yield. ^{1}H NMR (400MHz, $CDCl_3$) δ 4.06 (t, *J* = 6.7Hz, 2H), 2.30 (t, *J* = 7.5Hz, 2H), 1.62 (t, *J* = 7.4Hz, 4H), 1.26 (br s, 45H), 0.89 (t, *J* = 6.8Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.03, 64.39, 34.41, 31.92, 29.70, 29.66, 29.63, 29.61, 29.57, 29.53, 29.48, 29.36, 29.35, 29.27, 29.25, 29.16, 28.65, 25.93, 25.03, 22.68, 14.11. ESI-MS: *m/z* 453.4($[M+H]^+$).

*Octadecyl stearate (**3p**) (CAS no: 2778-96-3)*



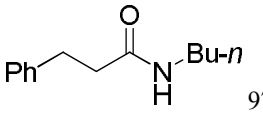
236 mg, 88% yield.^{1}H NMR (400MHz, $CDCl_3$) δ 4.05 (t, *J* = 5.7Hz, 2H), 2.29 (t, *J* = 7.5Hz, 2H), 1.63 (t, *J* = 6.8Hz, 4H), 1.26 (br s, 58H), 0.88 (t, *J* = 6.9Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.04, 64.40, 34.42, 31.92, 29.70, 29.65, 29.60, 29.58, 29.53, 29.48, 29.36, 29.27, 29.26, 29.16, 28.65, 25.94, 25.04, 22.69, 14.11. ESI-MS: *m/z* 537.8($[M+H]^+$).

*Cholest-5-en-3-ol (3β)-, 3-benzenepropanoate (**3q**) (CAS no: 14914-99-9)*



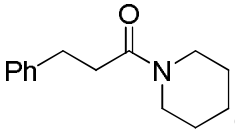
197 mg, 76% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23 – 7.18 (m, 2H), 7.13 – 7.10 (m, 3H), 5.29 (d, *J* = 4.0 Hz, 1H), 4.57 – 4.50 (m, 1H), 2.87 (t, *J* = 7.8 Hz, 2H), 2.52 (t, *J* = 7.8 Hz, 2H), 2.21 (d, *J* = 7.8 Hz, 2H), 1.96 – 1.86 (m, 2H), 1.80 – 1.71 (m, 3H), 1.54 – 1.36 (m, 8H), 1.34 – 1.13 (m, 5H), 1.11 – 0.95 (m, 8H), 0.94 (s, 3H), 0.84 (d, *J* = 6.5 Hz, 3H), 0.80 (d, *J* = 1.7 Hz, 3H), 0.79 (d, *J* = 1.7 Hz, 3H), 0.60 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.3, 140.6, 139.7, 128.4, 128.3, 126.2, 122.6, 74.0, 56.7, 56.2, 50.1, 42.3, 39.8, 39.5, 38.1, 37.0, 36.6, 36.3, 36.2, 35.8, 31.9, 31.8, 31.1, 28.2, 28.0, 27.8, 24.3, 23.8, 22.8, 22.5, 21.0, 19.3, 18.7, 11.9. ESI-MS: *m/z* 519.4($[M+H]^+$).

*N-Butyl-3-phenylpropanamide (**5a**) (CAS no: 10264-11-6)*



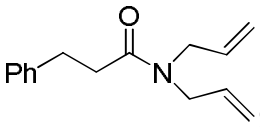
97 mg, 95% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 – 7.26 (m, 2H), 7.23 – 7.17 (m, 3H), 5.39 (s, 1H), 3.20 (td, *J* = 7.1, 5.8 Hz, 2H), 2.96 (t, *J* = 8.0 Hz, 2H), 2.45 (t, *J* = 8.0 Hz, 2H), 1.43 – 1.36 (m, 2H), 1.30 – 1.21 (m, 2H), 0.88 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.0, 140.9, 128.5, 128.3, 126.2, 39.2, 38.6, 31.8, 31.6, 19.9, 13.7. ESI-MS: *m/z* 228.1([M+Na]$^+$).

*3-Phenyl-1-(piperidin-1-yl)propan-1-one (**5b**) (CAS no: 21924-11-8)*



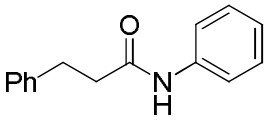
95 mg, 88% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 – 7.25 (m, 2H), 7.22 – 7.17 (m, 3H), 3.55 (t, *J* = 5.6 Hz, 2H), 3.32 (t, *J* = 5.6 Hz, 2H), 2.96 (t, *J* = 8.0 Hz, 2H), 2.61 (dd, *J* = 9.0, 7.0 Hz, 2H), 1.63 – 1.57 (m, 2H), 1.54 – 1.42 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.4, 141.4, 128.4, 128.3, 125.9, 46.5, 42.6, 35.0, 31.5, 26.3, 25.4, 24.4. ESI-MS: *m/z* 240.2([M+Na]$^+$).

*N, N-Diallyl-3-phenylpropanamide (**5c**) (CAS no: 101104-56-7)*



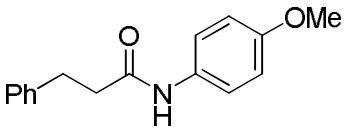
94 mg, 82% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22 – 7.17 (m, 2H), 7.14 – 7.08 (m, 3H), 5.72-57 (m, 2H), 5.10 – 4.98 (m, 4H), 3.91 (d, *J* = 6.0 Hz, 2H), 3.72 (dt, *J* = 4.6, 1.7 Hz, 2H), 2.91 (t, *J* = 8.0 Hz,, 2H), 2.56 – 2.52 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.1, 141.3, 133.3, 132.8, 128.4, 126.0, 117.1, 116.5, 49.0, 47.9, 34.8, 31.4. ESI-MS: *m/z* 252.0([M+Na]$^+$).

*N, 3-Diphenylpropanamide (**5d**) (CAS no: 3271-81-6)*



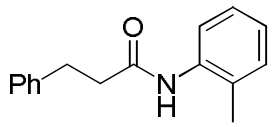
101 mg, 90% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 (s, 1H), 7.34 (d, *J* = 7.8 Hz, 2H), 7.19 – 7.14 (m, 4H), 7.11 – 7.07 (m, 3H), 6.97 (t, *J* = 7.4 Hz, 1H), 2.91 (t, *J* = 7.7 Hz, 2H), 2.52 (t, *J* = 7.7 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.7, 140.6, 137.8, 128.8, 128.5, 128.3, 126.3, 124.2, 120.1, 39.2, 31.5. ESI-MS: *m/z* 248.1([M+Na]$^+$).

*N-(4-methoxyphenyl)-3-phenylpropanamide (**5e**) (CAS no: 97754-31-9)*



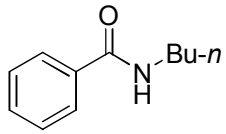
117 mg, 92% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 (s, 1H), 7.23 – 7.17 (m, 4H), 7.13 – 7.10 (m, 3H), 6.71 (dd, *J* = 12.0, 2.8 Hz, 2H), 3.69 (s, 3H), 2.93 (t, *J* = 7.7 Hz, 2H), 2.52 (t, *J* = 7.7 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.4, 156.4, 140.7, 130.9, 128.5, 128.3, 126.3, 122.0, 114.0, 55.4, 39.1, 31.6. ESI-MS: *m/z* 278.0([M+Na]$^+$).

*3-Phenyl-N-(o-tolyl)propanamide (**5f**) (CAS no: 71231-24-8)*



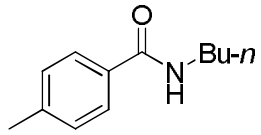
104 mg, 87% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (d, *J* = 7.9 Hz, 1H), 7.23 – 7.11 (m, 5H), 7.10 – 7.03 (m, 2H), 6.96 (t, *J* = 7.3 Hz, 1H), 6.88 (s, 1H), 2.96 (t, *J* = 7.5 Hz, 2H), 2.60 (t, *J* = 7.5 Hz, 2H), 1.96 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.4, 140.5, 135.5, 130.4, 129.4, 128.6, 128.4, 126.6, 126.4, 125.2, 123.4, 39.1, 31.7, 17.5. ESI-MS: *m/z* 262.0([M+Na]$^+$).

*N-Butylbenzamide (**5g**) (CAS no: 2782-40-3)*



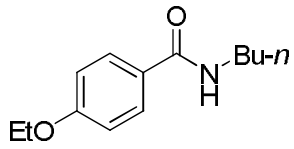
84 mg, 95% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 – 7.67 (m, 2H), 7.40 – 7.35 (m, 1H), 7.32 – 7.28 (m, 2H), 6.44 (s, 1H), 3.34 (dd, *J* = 13.5, 6.6 Hz, 2H), 1.53 – 1.46 (m, 2H), 1.35 – 1.26 (m, 2H), 0.85 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.6, 134.8, 131.1, 128.4, 126.8, 39.7, 31.6, 20.1, 13.7. ESI-MS: *m/z* 200.2([M+Na]$^+$).

*N-Butyl-4-methylbenzamide (**5h**) (CAS no: 5456-97-3)*



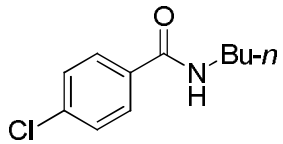
88 mg, 92% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 – 7.58 (m, 2H), 7.11 (d, *J* = 7.9 Hz, 2H), 6.32 (s, 1H), 3.34 (td, *J* = 7.2, 5.8 Hz, 2H), 2.29 (s, 3H), 1.53 – 1.46 (m, 2H), 1.33 – 1.28 (m, 2H), 0.86 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.5, 141.5, 131.9, 129.0, 126.8, 39.7, 31.7, 21.3, 20.1, 13.7. ESI-MS: *m/z* 214.1([M+Na]$^+$).

*N-Butyl-4-ethoxybenzamide (**5i**) (CAS no: 6283-99-4)*



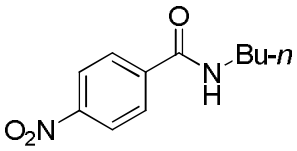
93 mg, 84% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 – 7.62 (m, 2H), 6.84 – 6.80 (m, 2H), 5.99 (s, 1H), 4.00 (q, *J* = 7.0 Hz, 2H), 3.36 (td, *J* = 7.1, 5.8 Hz, 2H), 1.55 – 1.48 (m, 2H), 1.38 – 1.29 (m, 5H), 0.91 – 0.86 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.1, 161.5, 128.6, 127.0, 114.2, 63.6, 39.7, 31.8, 20.2, 14.7, 13.8. ESI-MS: *m/z* 244.2([M+Na]$^+$).

*N-Butyl-4-chlorobenzamide (**5j**) (CAS no: 1455-99-8)*



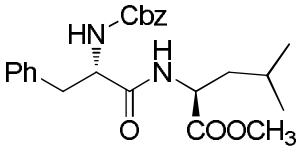
101 mg, 96% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 (d, *J* = 8.5 Hz, 2H), 7.28 (d, *J* = 8.5 Hz, 2H), 6.43 (s, 1H), 3.33 (dd, *J* = 13.2, 6.9 Hz, 2H), 1.54 – 1.46 (m, 2H), 1.35 – 1.26 (m, 2H), 0.86 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.5, 137.4, 133.2, 128.6, 128.3, 39.9, 31.6, 20.1, 13.7. ESI-MS: *m/z* 244.2([M+Na]$^+$). ESI-MS: *m/z* 234.1([M+Na]$^+$).

*N-Butyl-4-nitrobenzamide (**5k**) (CAS no: 51207-98-8)*



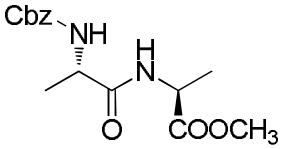
107 mg, 96% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.19 – 8.16 (m, 2H), 7.87 – 7.84 (m, 2H), 6.46 (s, 1H), 3.39 (td, *J* = 7.2, 5.8 Hz, 2H), 1.58 – 1.51 (m, 2H), 1.38 – 1.29 (m, 2H), 0.88 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.5, 149.4, 140.4, 128.1, 123.7, 40.1, 31.5, 20.1, 13.7. ESI-MS: *m/z* 245.3([M+Na]$^{+}$).

*Cbz-Phe-Leu-OMe (**5l**) (CAS no: 3580-45-1)*



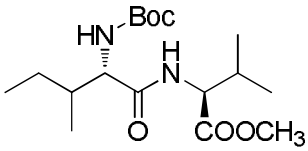
203 mg, 95% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.28 (m, 5H), 7.27 – 7.22 (m, 3H), 7.21 – 7.17 (m, 2H), 6.30 (d, *J* = 7.6 Hz, 1H), 5.39 (d, *J* = 7.6 Hz, 1H), 5.08 (s, 2H), 4.58 – 4.53 (m, 1H), 4.50 – 4.46 (m, 1H), 3.69 (s, 3H), 3.13 – 3.02 (m, 2H), 1.60 – 1.41 (m, 3H), 0.88 (t, *J* = 6.2 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.8, 170.6, 155.9, 136.2, 136.0, 129.3, 128.6, 128.5, 128.2, 128.0, 127.0, 67.0, 55.9, 52.2, 50.7, 41.4, 38.3, 24.6, 22.6, 21.8; ESI-MS: *m/z* 449.2([M+Na]$^{+}$). HPLC retention time = 12.0 min (mobile phase, 7% *i*-PrOH in *n*-hexane).

*Cbz-Ala-Ala-OMe (**5m**), (CAS no: 2483-51-4)*



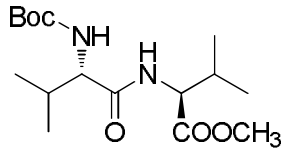
145 mg, 94% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.28 (m, 5H), 6.79 (d, *J* = 6.3 Hz, 1H), 5.56 (d, *J* = 7.4 Hz, 1H), 5.14 – 5.07 (m, 2H), 4.56 (p, *J* = 7.2 Hz, 1H), 4.33 – 4.29 (m, 1H), 3.74 (s, 3H), 1.38 (d, *J* = 7.0 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.1, 171.9, 155.9, 136.1, 128.5, 128.1, 128.0, 66.9, 52.4, 50.3, 48.0, 18.7, 18.1. ESI-MS: *m/z* 331.1([M+Na]$^{+}$). HPLC retention time = 15.8 min (mobile phase, 10% *i*-PrOH in *n*-hexane).

*Boc-Ile-Val-OMe (**5n**) (CAS no: 33911-17-0)*



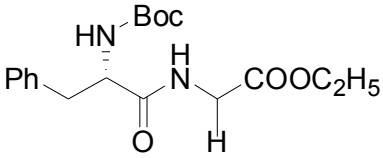
143 mg, 83% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.45 (d, *J* = 8.3 Hz, 1H), 5.11 (d, *J* = 8.6 Hz, 1H), 4.55 (dd, *J* = 8.7, 4.9 Hz, 1H), 3.96 (dd, *J* = 16.6, 8.7 Hz, 1H), 3.74 (s, 3H), 2.24 – 2.12 (m, 1H), 1.88 – 1.86 (m, 1H), 1.57 – 1.51 (m, 1H), 1.44 (s, 9H), 1.21 – 1.10 (m, 1H), 0.95 – 0.90 (m, 12H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.1, 171.6, 155.7, 79.8, 59.3, 57.0, 52.1, 36.9, 31.2, 28.2, 24.7, 18.9, 17.7, 15.4, 11.3. ESI-MS: *m/z* 367.4([M+Na]$^{+}$). HPLC retention time = 10.4 min (mobile phase, 2% *i*-PrOH in *n*-hexane).

*Boc-Val-Val-OMe (**5o**) (CAS no: 33857-88-4)*



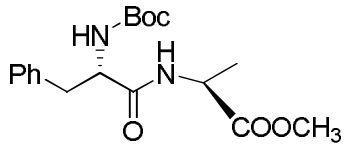
159 mg, 96% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.47 (d, *J* = 8.1 Hz, 1H), 5.12 (d, *J* = 8.6 Hz, 1H), 4.55 (dd, *J* = 8.7, 4.9 Hz, 1H), 3.93 (dd, *J* = 10.6, 4.6 Hz, 1H), 3.74 (s, 3H), 2.22 – 2.10 (m, 2H), 1.45 (s, 9H), 0.98 – 0.90 (m, 12H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.1, 171.9, 171.6, 162.9, 155.8, 79.8, 60.1, 57.0, 52.1, 31.1, 30.6, 28.2, 19.2, 18.9, 17.9, 17.7. ESI-MS: *m/z* 353.2([M+Na]$^+$). HPLC retention time = 6.6 min (mobile phase, 2% *i*-PrOH in *n*-hexane).

*Boc-Phe-Gly-OEt (**5p**) (CAS no: 30189-51-6)*



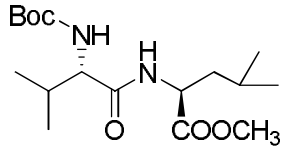
159 mg, 91% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ(ppm) 1.27 (t, *J* = 7.1 Hz, 3H), 1.39 (s, 9H), 3.05 (m, 1H), 3.13 (dd, *J* = 6.5, 13.9 Hz, 1H), 3.92 (dd, *J* = 4.9, 18.3 Hz, 1H), 4.02 (dd, *J* = 5.4, 18.2 Hz, 1H), 4.19 (q, J=7.1 Hz, 2H), 4.43 (d, *J* = 6.2 Hz, 1H), 5.11 (br d, *J* = 7.2 Hz, 1H), 6.57 (t, *J* = 5.0 Hz, 1H), 7.20-7.31(m,5H) ; ^{13}C-NMR (100 MHz, $CDCl_3$) δ(ppm) 171.5, 169.4, 155.4, 136.6, 129.2, 128.6, 126.9, 80.2, 61.5, 55.5, 41.3, 38.3, 28.2, 14.1, 1.0; ESI-MS: *m/z* 373.2 ([M+Na]$^+$); HPLC retention time = 13.6 min (mobile phase, 7% i-PrOH in n-hexane);

*Boc-Phe-Ala-OMe (**5q**) (CAS no: 15136-29-5)*



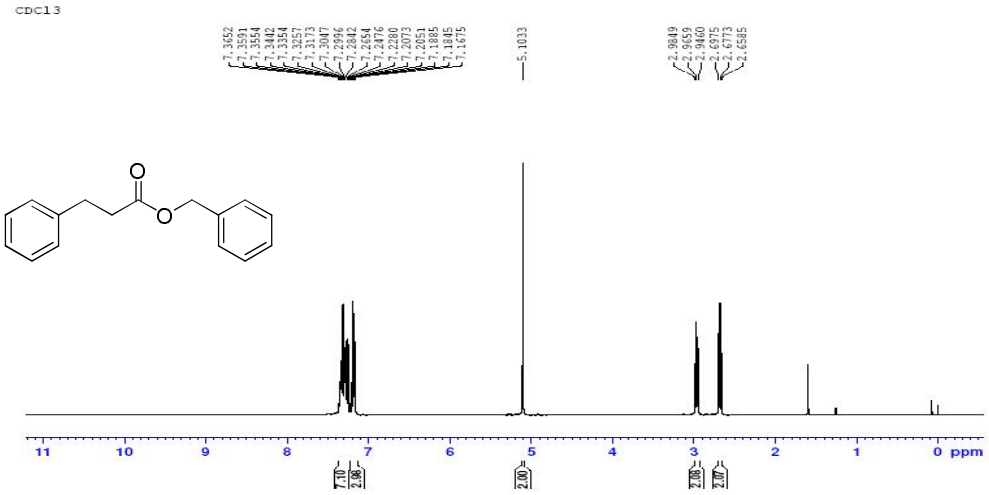
159 mg, 91% yield. ^{1}H-NMR (400 MHz, $CDCl_3$) δ(ppm) 1.35 (d, *J* = 7.2 Hz, 3H), 1.40 (s, 9H) , 3.07 (d, *J* = 6.5 Hz, 2H), 3.71 (s, 3H), 4.39 (d, *J* = 6.2 Hz, 1H), 4.52 (p, *J* = 7.1 Hz, 1H), 5.08 (br d, *J* = 5.9 Hz, 1H), 6.55 (br d, *J* = 5.6 Hz, 1 H) , 7.20-7.31 (m,5H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ(ppm) 172.8, 170.8, 155.3, 136.5, 129.3, 128.6, 126.9, 80.1, 55.5, 52.4, 48.0, 38.3, 28.2, 18.3; ESI-MS: *m/z*: 373.2([M+Na]$^+$); HPLC retention time = 11.7 min (mobile phase, 7% i-PrOH in n-hexane);

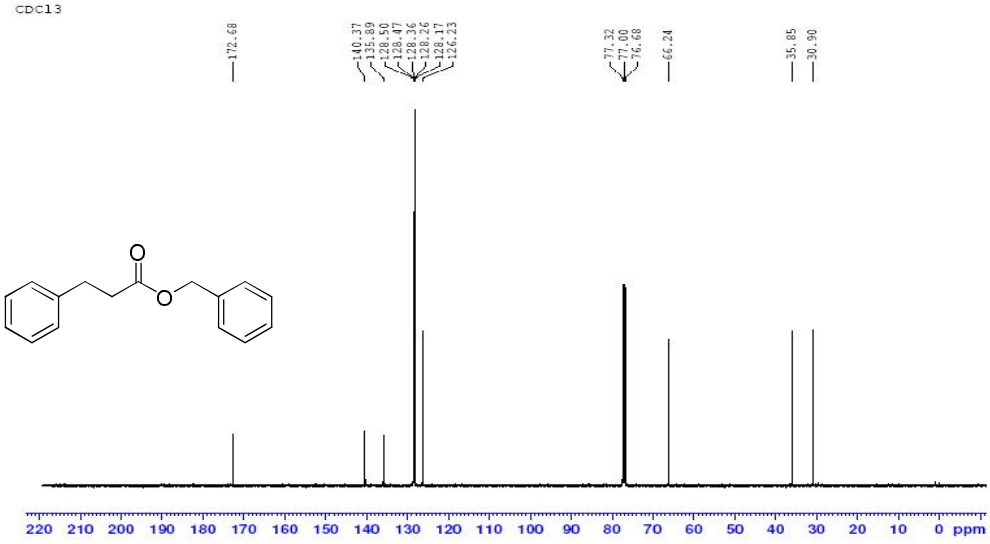
*Boc-Val-Leu-OMe (**5r**) (CAS no: 15215-73-3)*



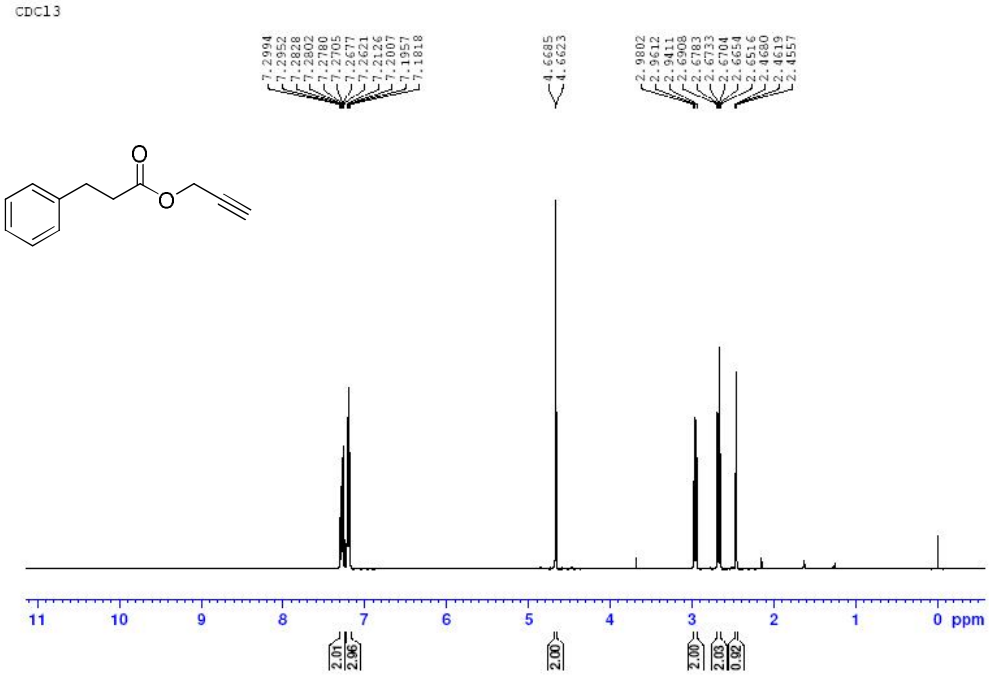
151 mg, 88% yield.^{1}H-NMR (400 MHz, $CDCl_3$) δ(ppm) 0.92-0.98 (m, 12H), 1.44 (s, H), 1.54-1.58 (m, 1H), 1.60-1.69 (m, 1H), 2.08-2.13 (m, 1H), 3.73 (s, 3H), 3.92 (dd, *J* = 6.8, 8.6 Hz, 1H), 4.59-4.65 (m, 1H), 5.13 (br d, *J* = 8.8, 1 H), 6.41 (br d, *J* = 7.8Hz, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ(ppm) 173.1, 171.5, 155.8, 79.8, 59.8, 52.2, 50.6, 41.3, 30.8, 28.2, 24.7, 22.7, 21.8, 19.1, 17.8; ESI-MS: *m/z* 367.3([M+Na]$^+$); HPLC retention time = 10.3 min (mobile phase, 2% i-PrOH in n-hexane).

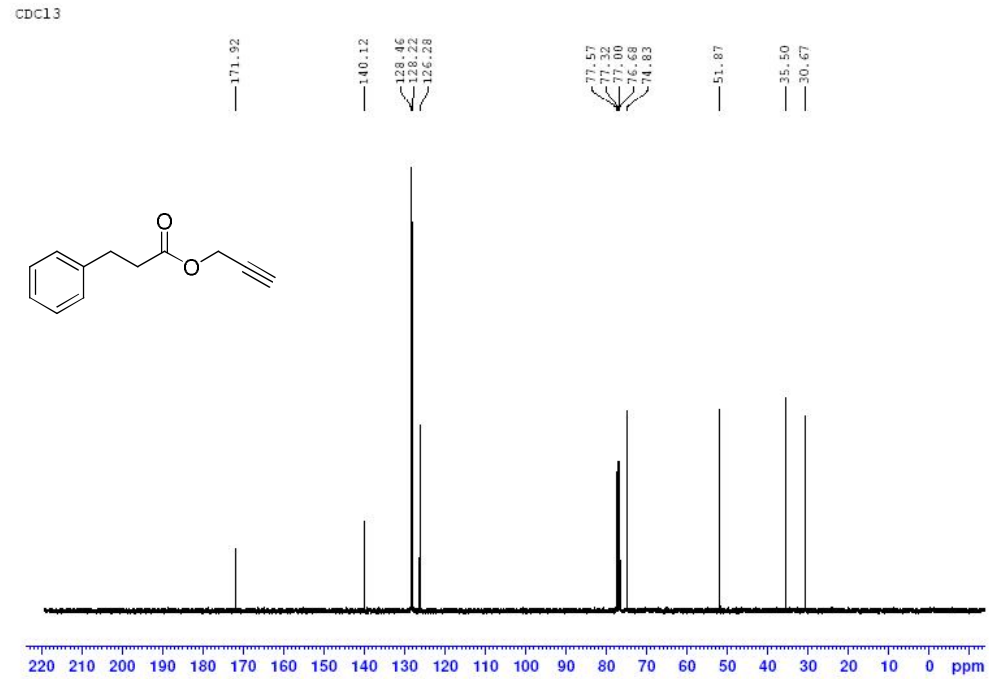
3a


CDCl3
7.3652
7.3591
7.3554
7.3442
7.3354
7.3257
7.3173
7.3047
7.2996
7.2842
7.2654
7.2476
7.2280
7.2073
7.2051
7.1885
7.1845
7.1675
5.1033
2.9849
2.9659
2.9460
2.6975
2.6773
2.6585
11 10 9 8 7 6 5 4 3 2 1 0 ppm
7.10
2.08
2.00
2.08
2.07

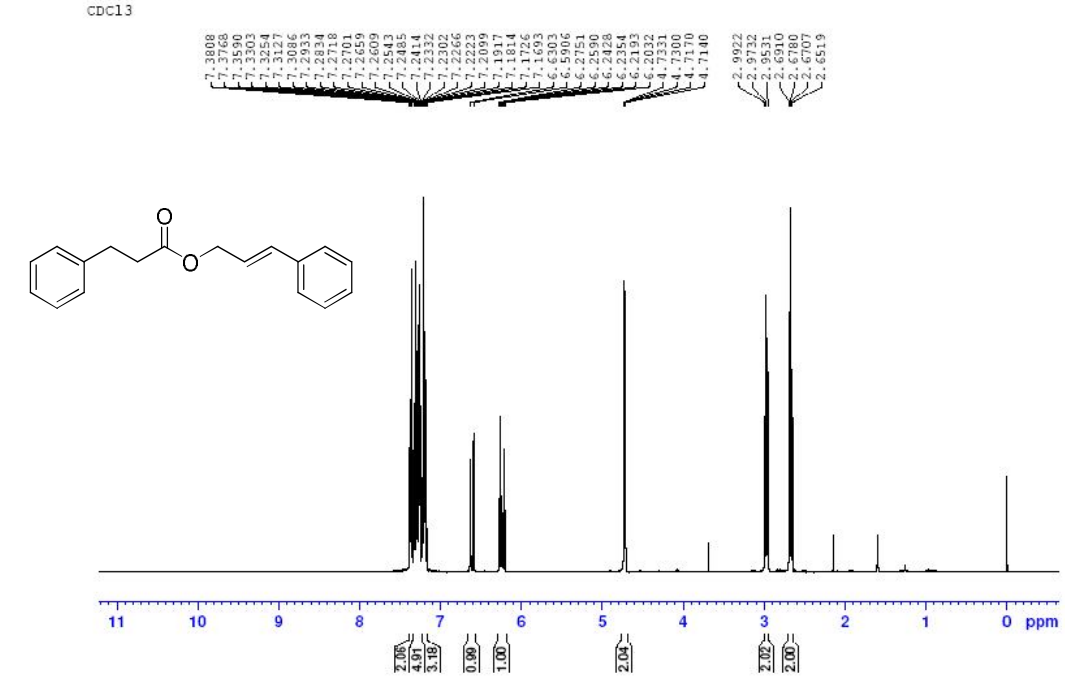

CDCl3
172.68
140.37
135.89
128.50
128.47
128.36
128.26
128.17
126.23
77.32
77.00
76.68
66.24
35.85
30.90
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

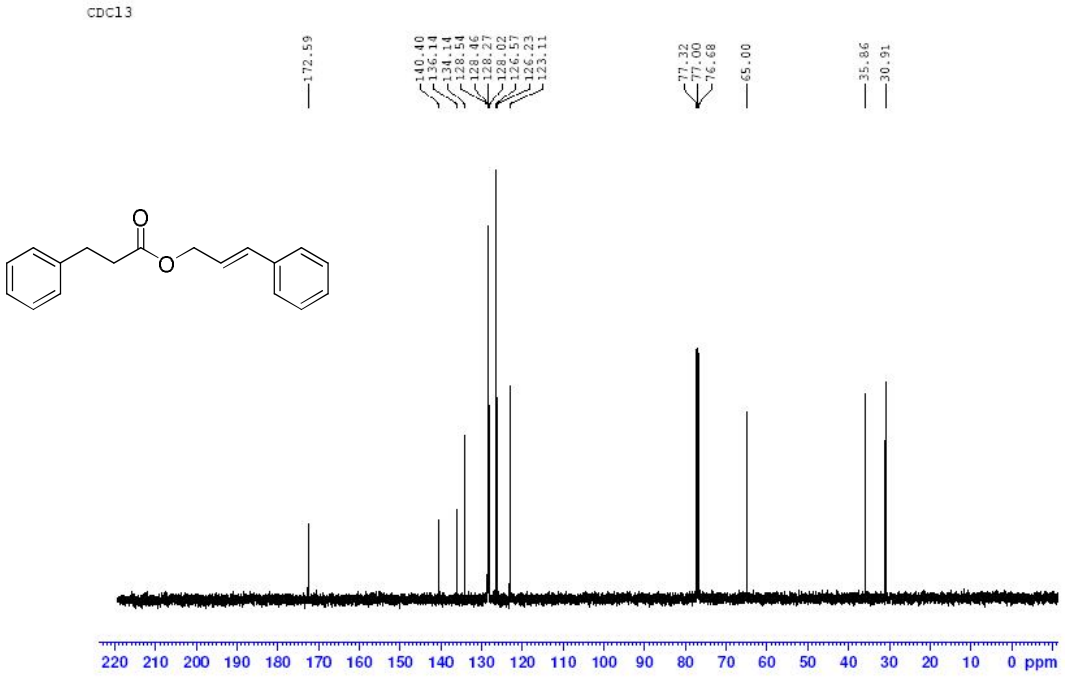
3b


CDCl3
7.2994
7.2952
7.2828
7.2802
7.2780
7.2705
7.2677
7.2621
7.2126
7.2007
7.1957
7.1818
4.6685
4.6623
2.9802
2.9612
2.9411
2.6908
2.6783
2.6733
2.6704
2.6654
2.6516
2.4680
2.4619
2.4557
11 10 9 8 7 6 5 4 3 2 1 0 ppm
2.01
2.06
2.00
2.00
2.03
0.92

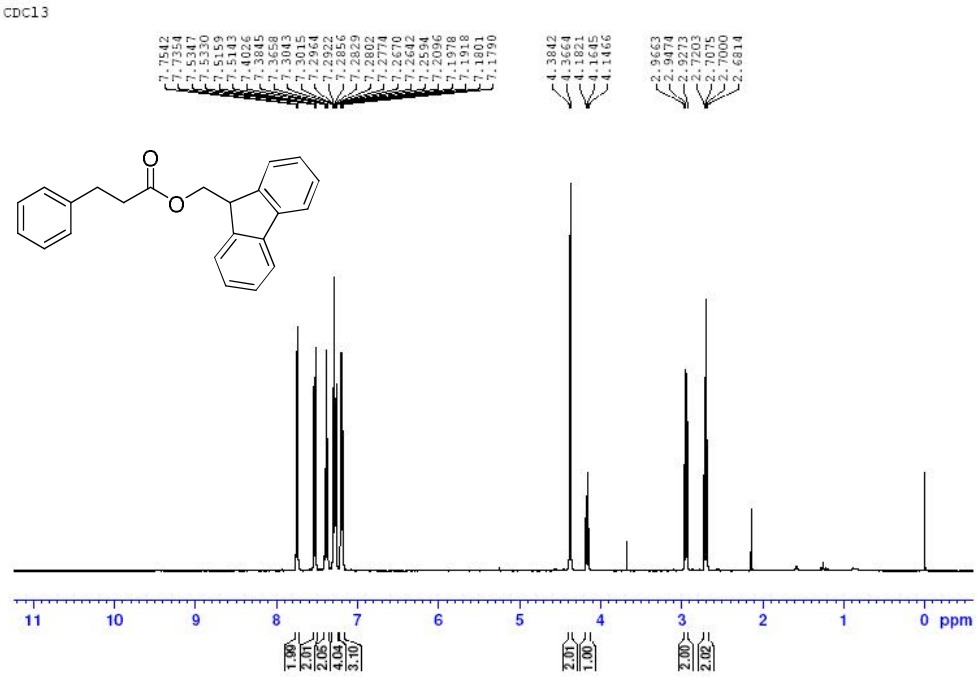

CDCl3
171.92
140.12
128.46
128.22
126.28
77.57
77.32
77.00
76.68
74.83
51.87
35.50
30.67
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

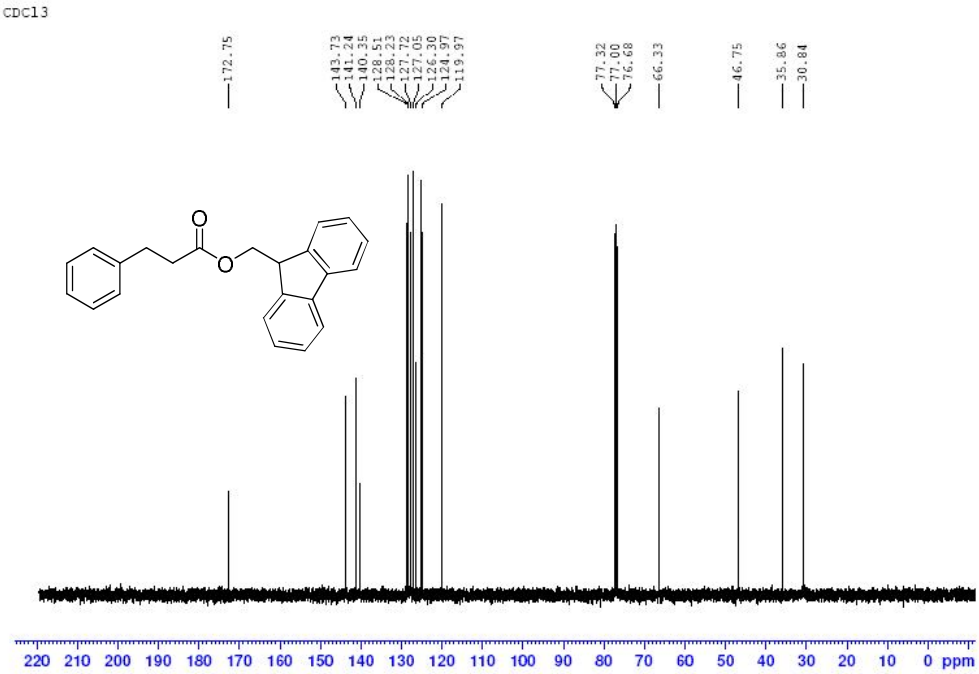
3c


CDCl3
7.3808
7.3768
7.3590
7.3303
7.3254
7.3127
7.3086
7.2933
7.2834
7.2718
7.2701
7.2659
7.2609
7.2543
7.2485
7.2414
7.2332
7.2302
7.2266
7.2223
7.2099
7.1917
7.1814
7.1726
7.1693
6.6303
6.5906
6.2751
6.2590
6.2428
6.2354
6.2193
6.2032
4.7331
4.7300
4.7170
4.7140
2.9922
2.9732
2.9531
2.6910
2.6780
2.6707
2.6519
11 10 9 8 7 6 5 4 3 2 1 0 ppm
2.06
4.91
3.18
0.90
1.00
2.04
2.02
2.00

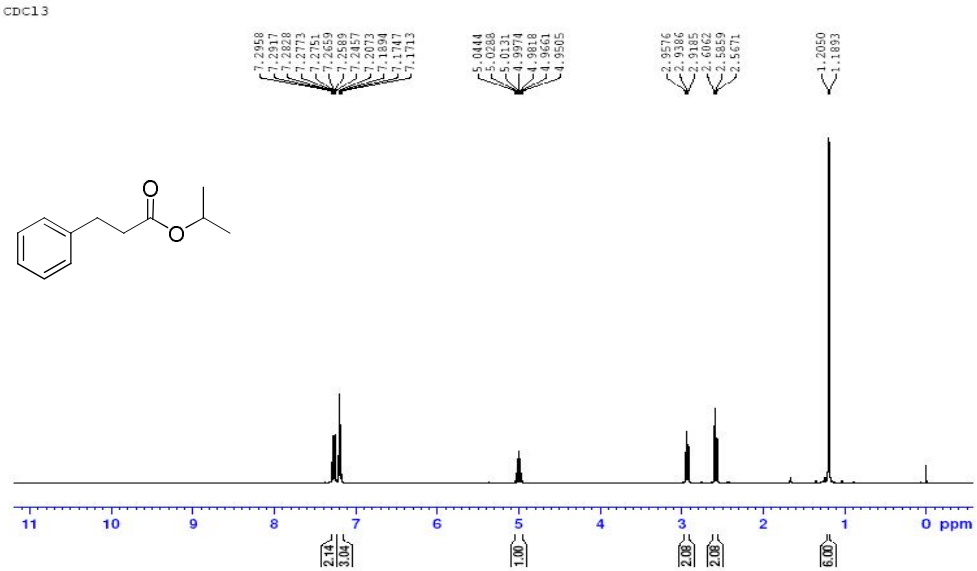

CDCl3
172.59
140.40
136.14
134.14
128.54
128.46
128.27
128.02
126.57
126.23
123.11
77.32
77.00
76.68
65.00
35.86
30.91
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

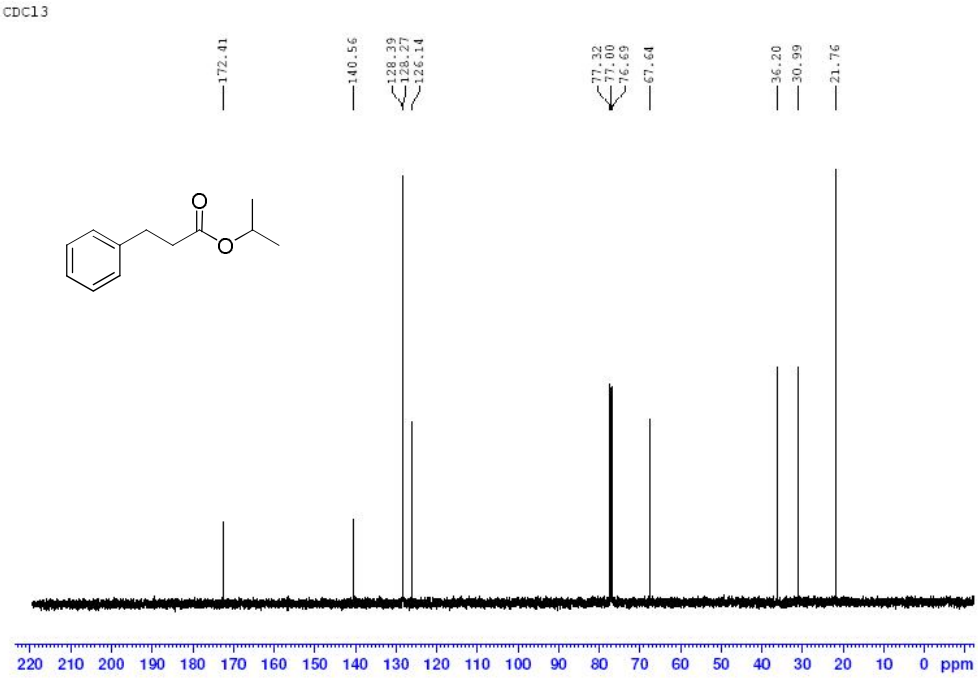
3d


CDCl3
7.7542
7.7354
7.5347
7.5330
7.5159
7.5143
7.4026
7.3845
7.3658
7.3043
7.3015
7.2964
7.2922
7.2856
7.2829
7.2802
7.2774
7.2670
7.2642
7.2594
7.2096
7.1978
7.1918
7.1801
7.1790
4.3842
4.3664
4.1821
4.1645
4.1466
2.9663
2.9474
2.9273
2.7203
2.7075
2.7000
2.6814
O
O
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.00 2.01 2.05 4.04 3.10
2.01 1.00
2.00 2.02

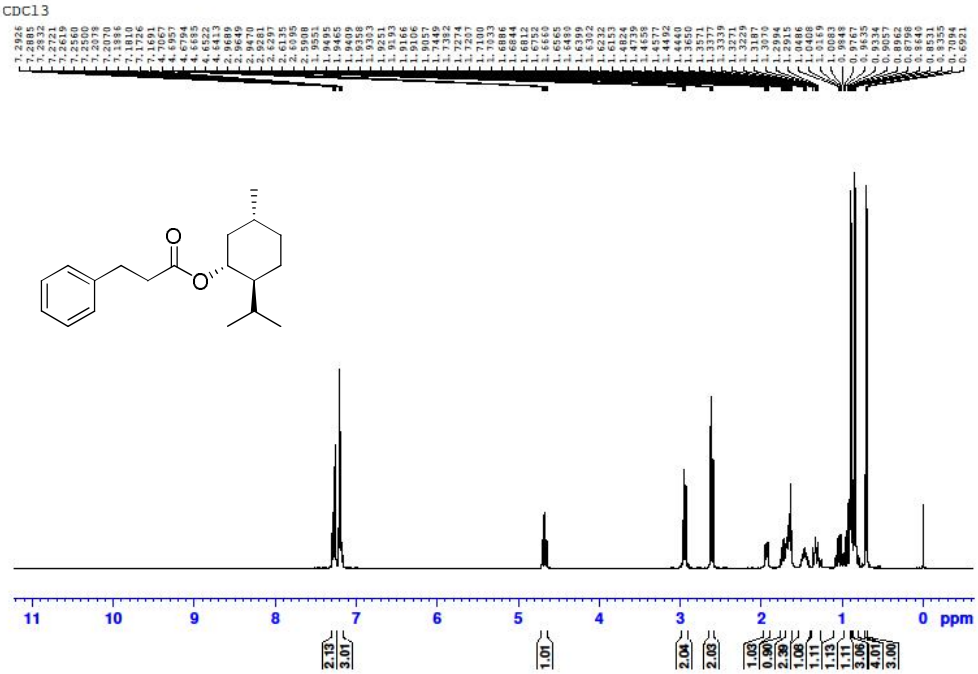

CDCl3
172.75
143.73
141.24
140.35
128.51
128.23
127.72
127.05
126.30
124.97
119.97
77.32
77.00
76.68
66.33
46.75
35.86
30.84
O
O
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

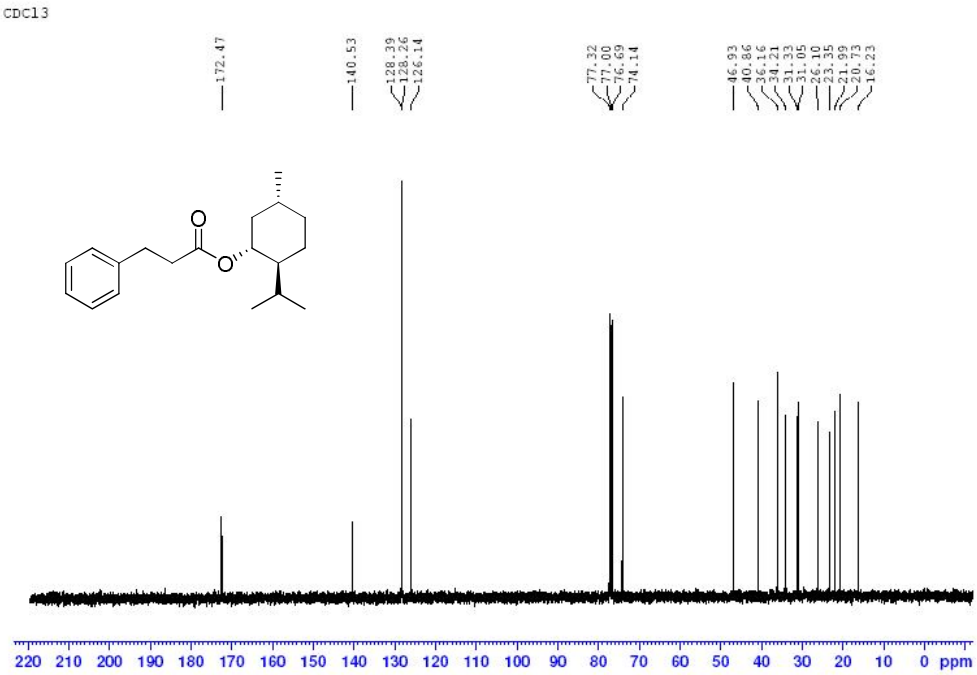
3e


CDCl3
7.2958
7.2917
7.2828
7.2773
7.2751
7.2659
7.2589
7.2457
7.2073
7.1894
7.1747
7.1713
5.0444
5.0288
5.0131
4.9974
4.9818
4.9661
4.9505
2.9576
2.9386
2.9185
2.6062
2.5859
2.5671
1.2050
1.1893
11 10 9 8 7 6 5 4 3 2 1 0 ppm
2.14
3.04
1.00
2.08
2.08
6.00

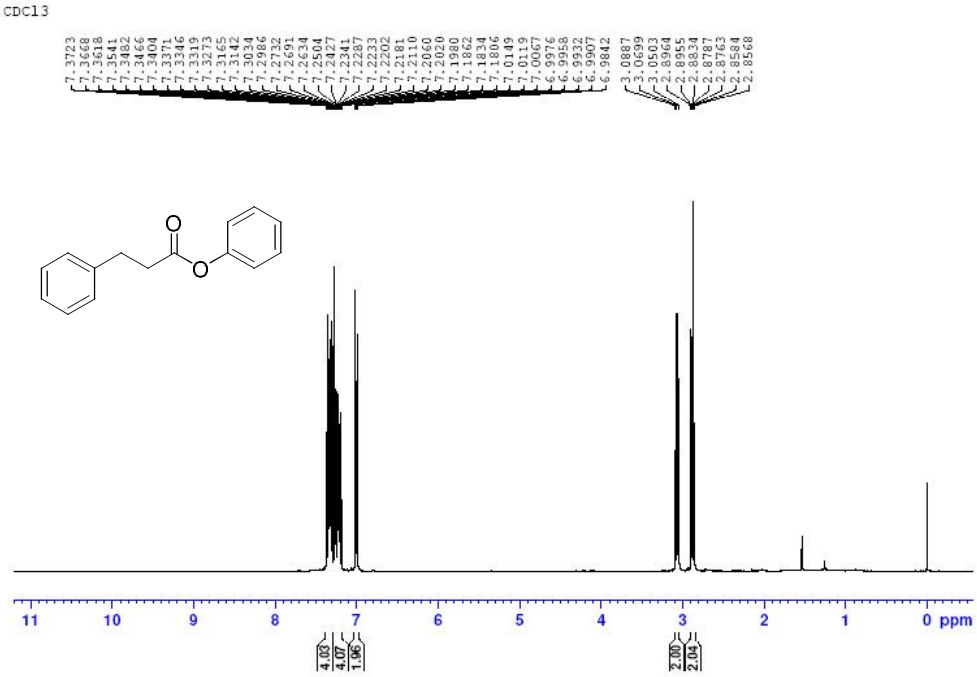

CDCl3
172.41
140.56
128.39
128.27
126.14
77.32
77.00
76.69
67.64
36.20
30.99
21.76
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

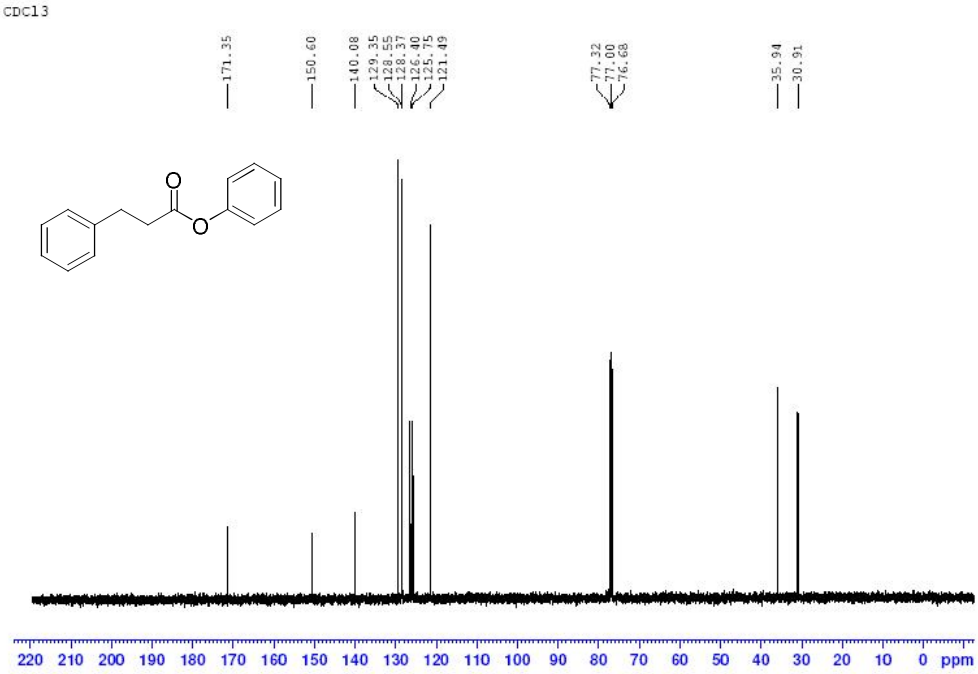
3f


CDCl3
11 10 9 8 7 6 5 4 3 2 1 0 ppm

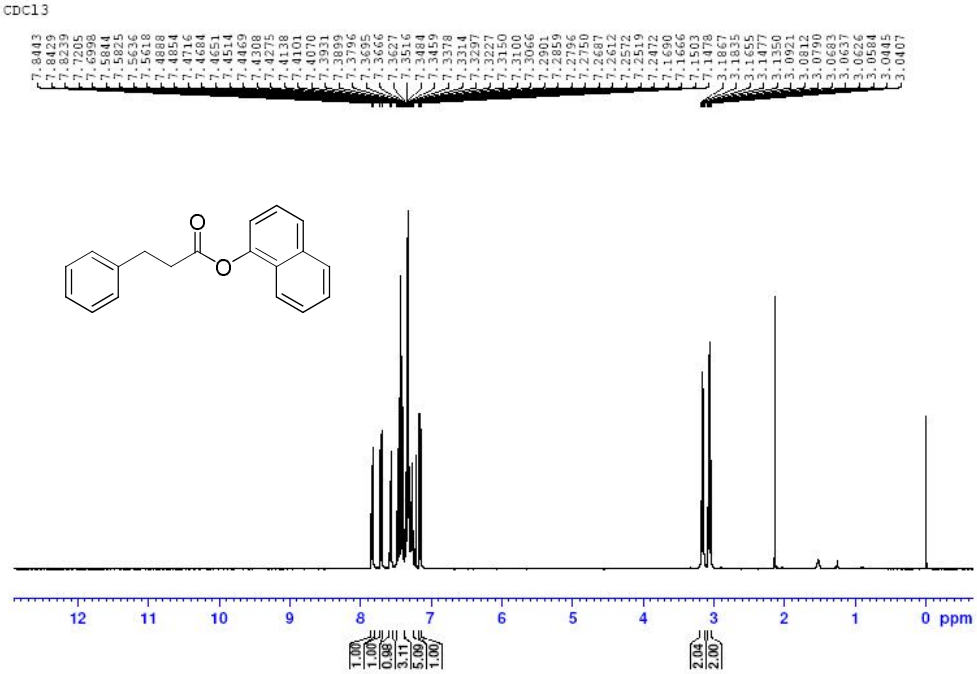

CDCl3
172.47
140.53
128.39
128.26
126.14
77.32
77.00
76.69
74.14
46.93
40.86
36.16
34.21
31.33
31.05
26.10
23.35
21.99
20.73
16.23
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

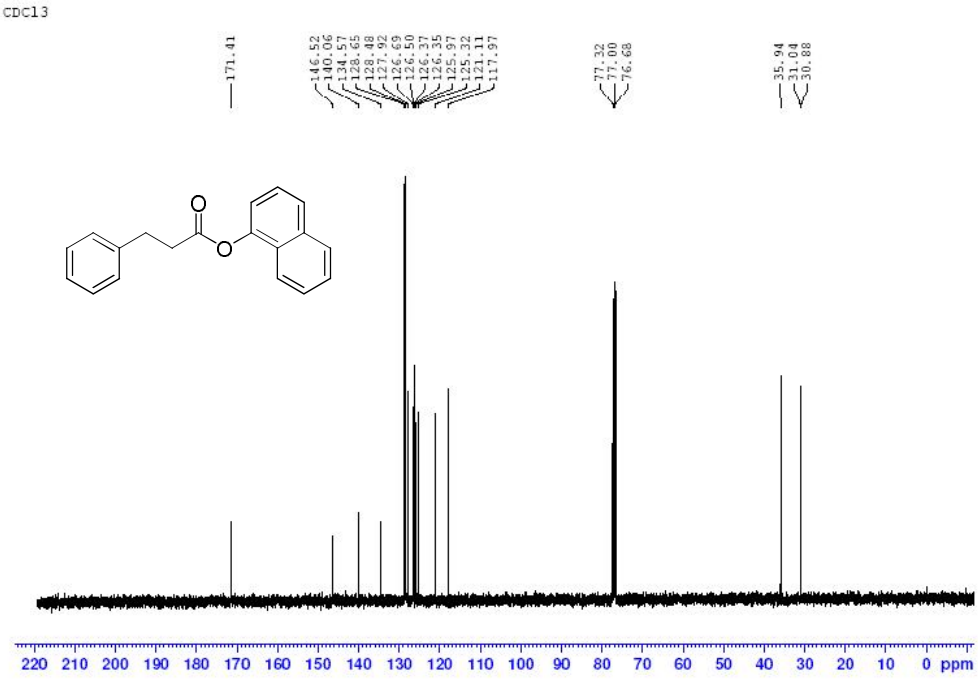
3g


CDCl3
7.3723
7.3668
7.3618
7.3441
7.3402
7.3466
7.3404
7.3371
7.3346
7.3319
7.3273
7.3165
7.3142
7.3034
7.2986
7.2732
7.2691
7.2634
7.2504
7.2427
7.2341
7.2287
7.2233
7.2202
7.2181
7.2110
7.2060
7.2020
7.1980
7.1862
7.1834
7.1806
7.0149
7.0119
7.0067
6.9976
6.9958
6.9932
6.9907
6.9842
3.0887
3.0699
3.0503
2.8964
2.8955
2.8834
2.8787
2.8763
2.8584
2.8568
11
10
9
8
7
6
5
4
3
2
1
0 ppm
4.03
4.07
1.96
2.00
2.04

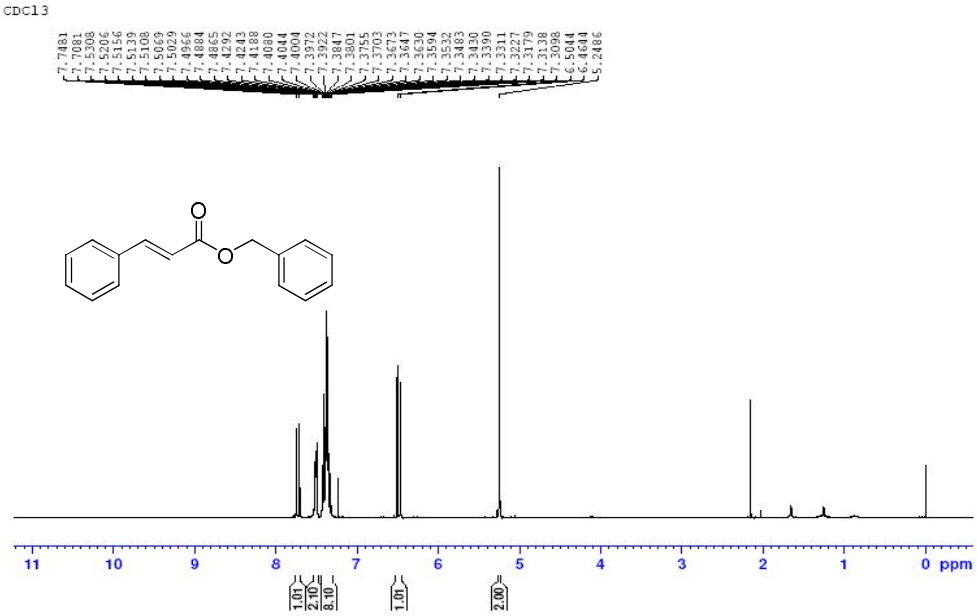

CDCl3
171.35
150.60
140.08
129.35
128.55
128.37
126.40
125.75
121.49
77.32
77.00
76.68
35.94
30.91
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm

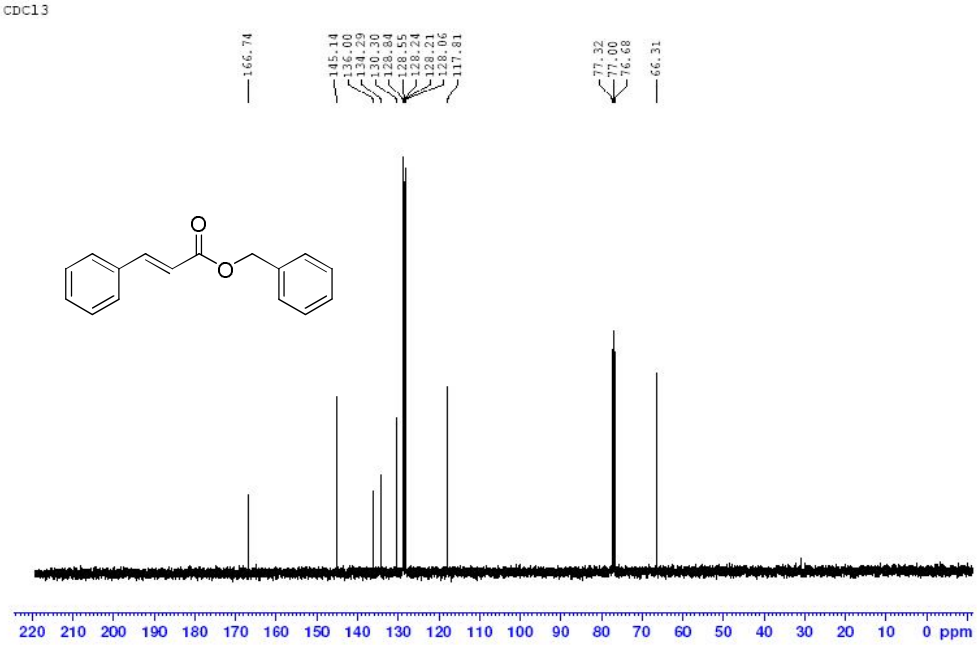
3h


CDCl3
12 11 10 9 8 7 6 5 4 3 2 1 0 ppm

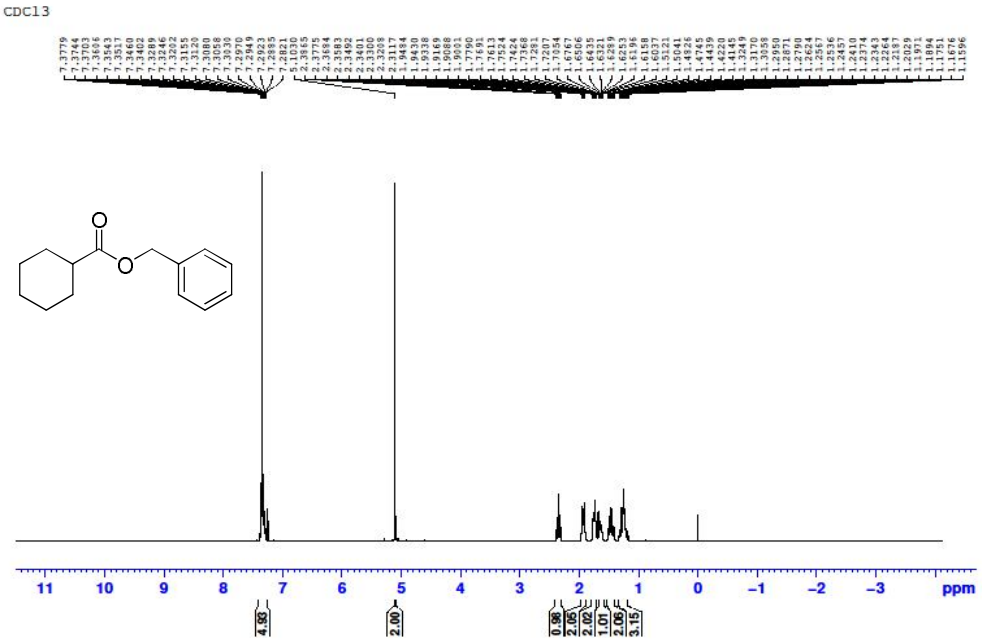

CDCl3
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

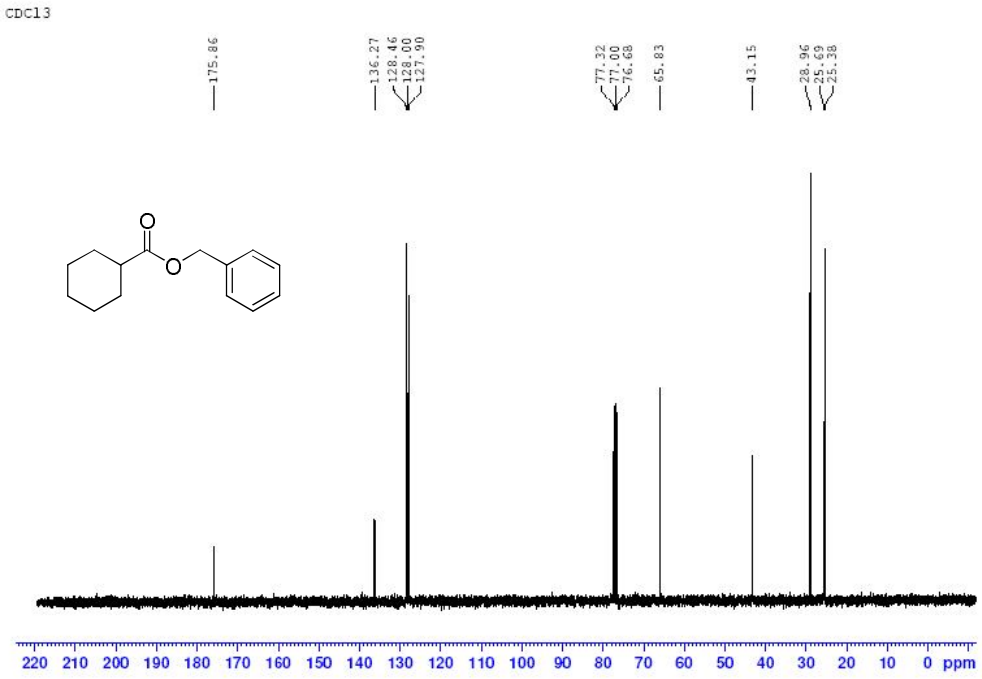
3i


CDCl3
7.7481
7.7081
7.5308
7.5206
7.5156
7.5139
7.5108
7.5069
7.5029
7.4966
7.4884
7.4865
7.4292
7.4243
7.4188
7.4080
7.4044
7.4004
7.3972
7.3922
7.3847
7.3801
7.3755
7.3703
7.3673
7.3647
7.3630
7.3594
7.3532
7.3483
7.3430
7.3390
7.3311
7.3227
7.3179
7.3138
7.3098
6.5044
6.4644
5.2486
O
O
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.01
2.10
8.10
1.01
2.00

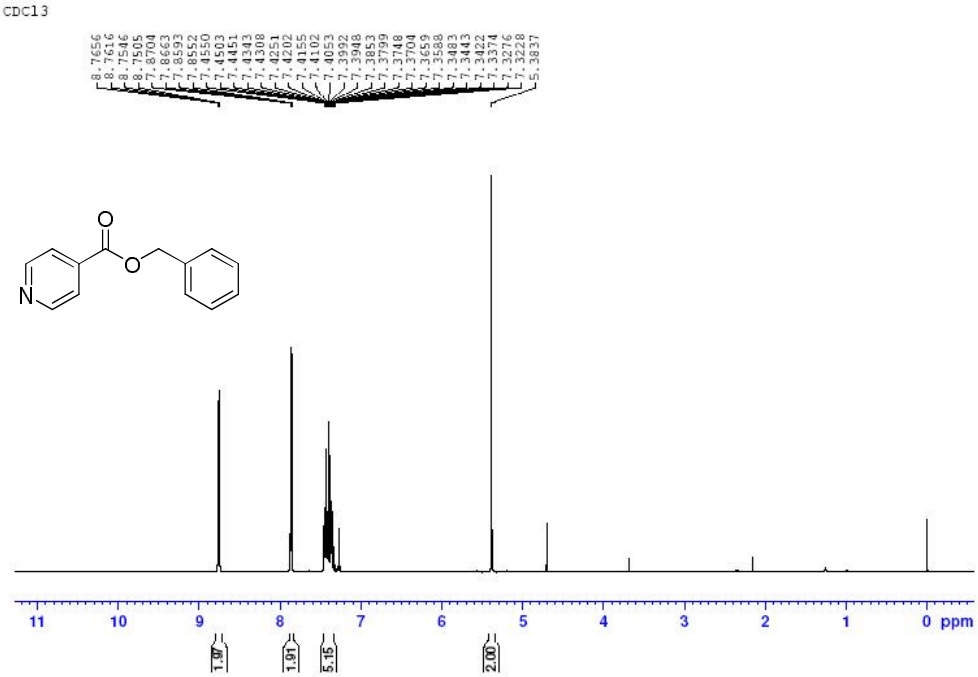

CDCl3
166.74
145.14
136.00
134.29
130.30
128.84
128.55
128.24
128.21
128.06
117.81
77.32
77.00
76.68
66.31
O
O
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

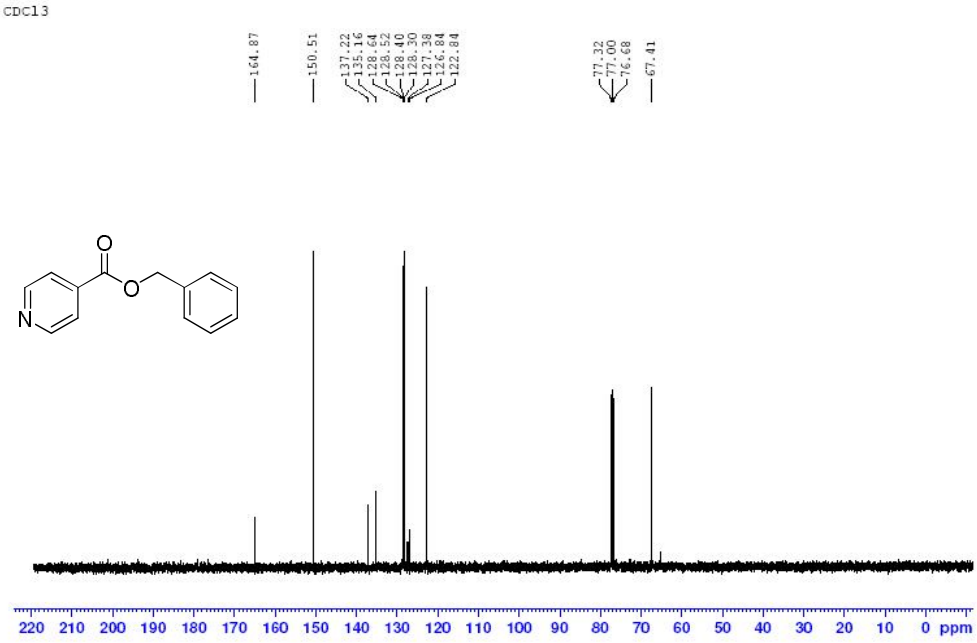
3j


CDCl3
11 10 9 8 7 6 5 4 3 2 1 0 −1 −2 −3 ppm
4.66
2.00
0.98
2.05
2.02
1.01
2.06
9.15

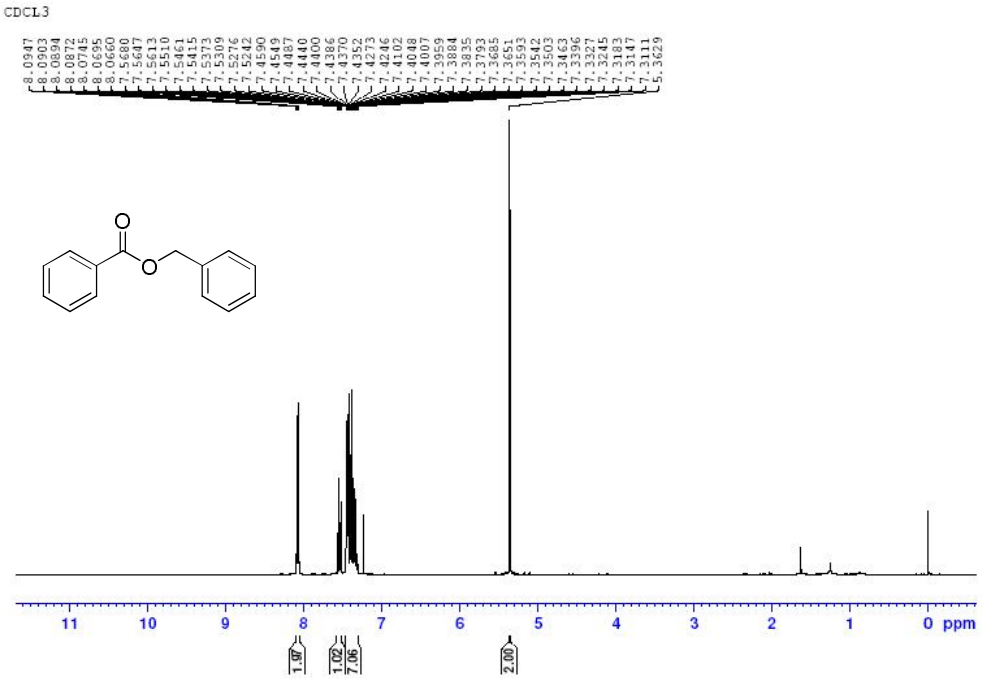

CDCl3
175.86
136.27
128.46
128.00
127.90
77.32
77.00
76.68
65.83
43.15
28.96
25.69
25.38
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

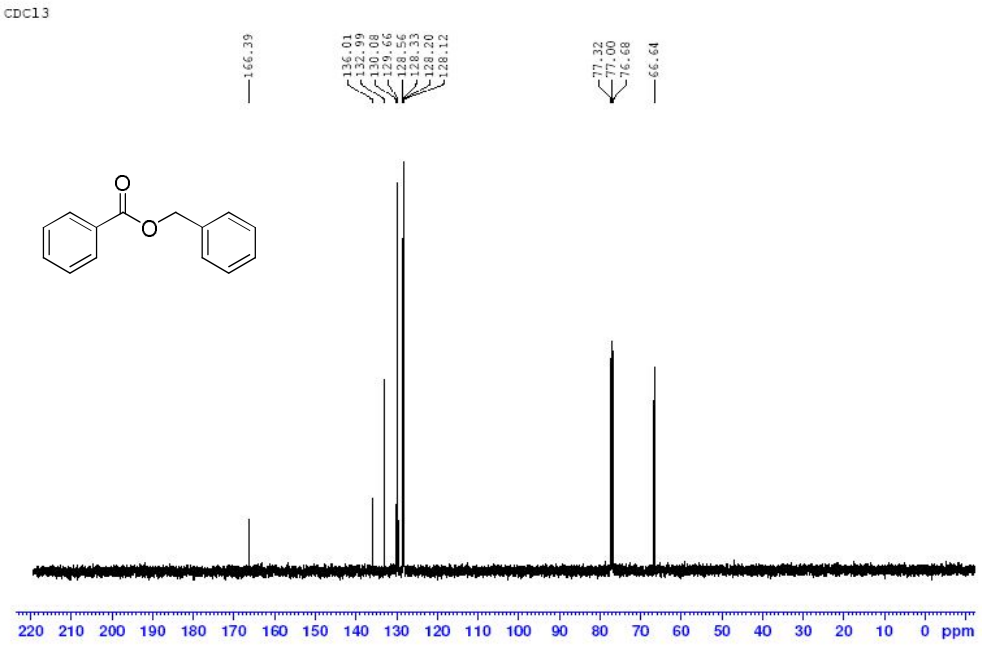
3k


CDCl3
8.7656
8.7616
8.7546
8.7505
7.8704
7.8663
7.8593
7.8552
7.4550
7.4503
7.4451
7.4343
7.4308
7.4251
7.4202
7.4155
7.4102
7.4053
7.3992
7.3948
7.3853
7.3799
7.3748
7.3704
7.3659
7.3588
7.3483
7.3443
7.3422
7.3374
7.3276
7.3228
5.3837
O
O
N
11
10
9
8
7
6
5
4
3
2
1
0 ppm
1.97
1.91
5.15
2.00

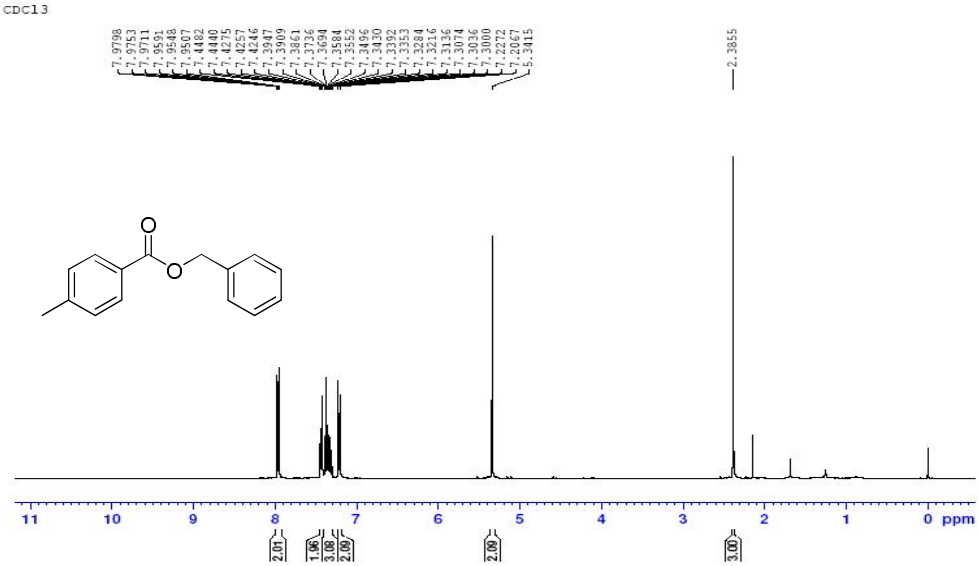

CDCl3
164.87
150.51
137.22
135.16
128.64
128.52
128.40
128.30
127.38
126.84
122.84
77.32
77.00
76.68
67.41
O
O
N
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm

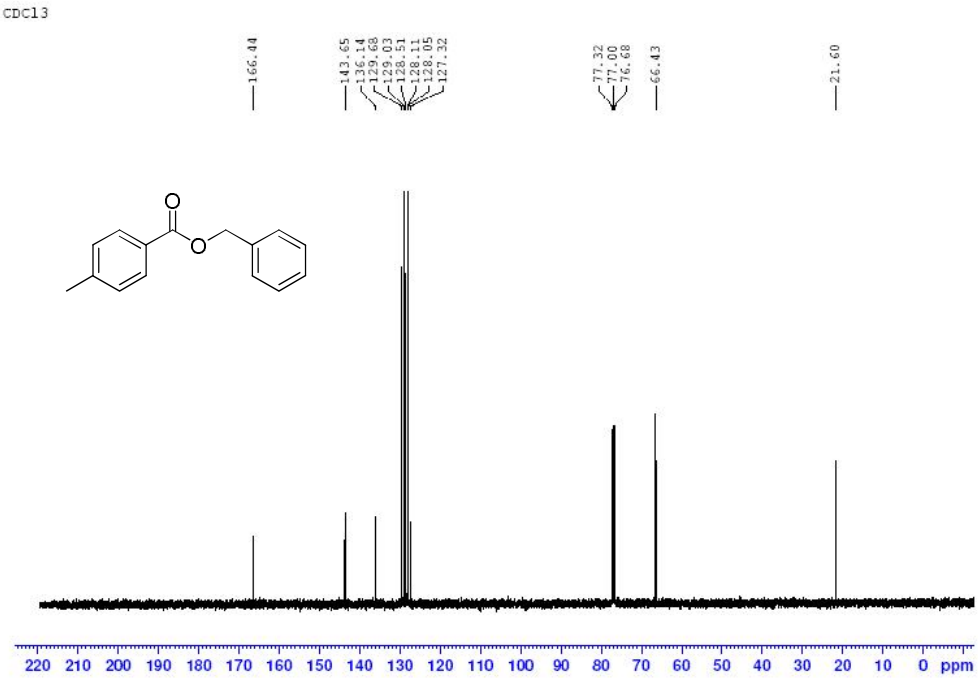
3l


CDCL3
8.0947
8.0903
8.0894
8.0872
8.0745
8.0695
8.0660
7.5680
7.5647
7.5613
7.5510
7.5461
7.5415
7.5373
7.5309
7.5276
7.5242
7.4590
7.4549
7.4487
7.4440
7.4400
7.4386
7.4370
7.4352
7.4273
7.4246
7.4102
7.4048
7.4007
7.3959
7.3884
7.3835
7.3793
7.3685
7.3651
7.3593
7.3542
7.3503
7.3463
7.3396
7.3327
7.3245
7.3183
7.3147
7.3111
5.3629
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.97
1.02
7.06
2.00

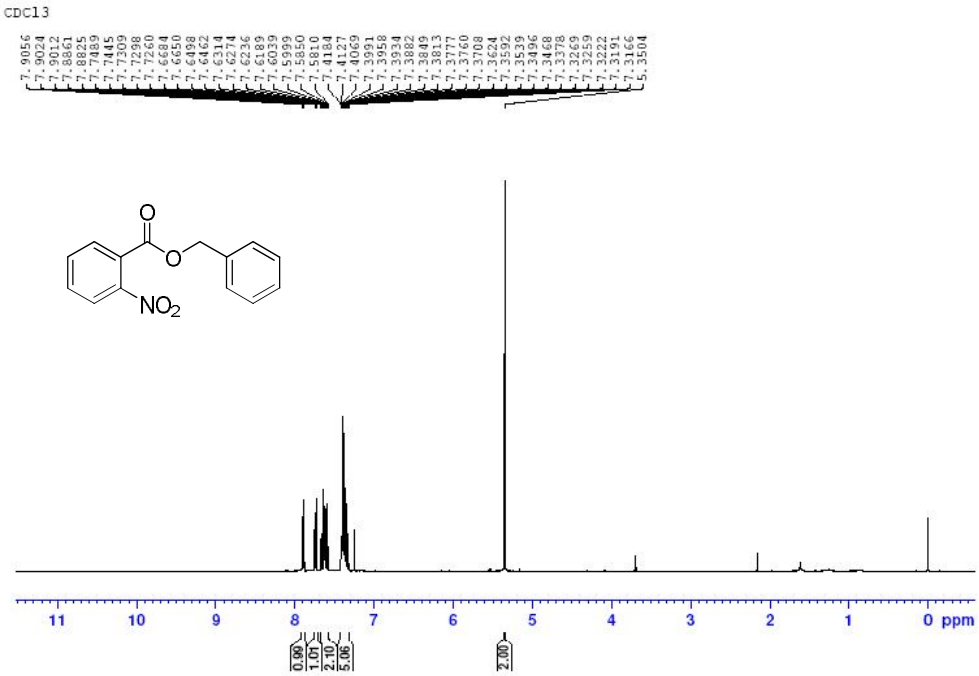

CDCl3
166.39
136.01
132.99
130.08
129.66
128.56
128.33
128.20
128.12
77.32
77.00
76.68
66.64
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

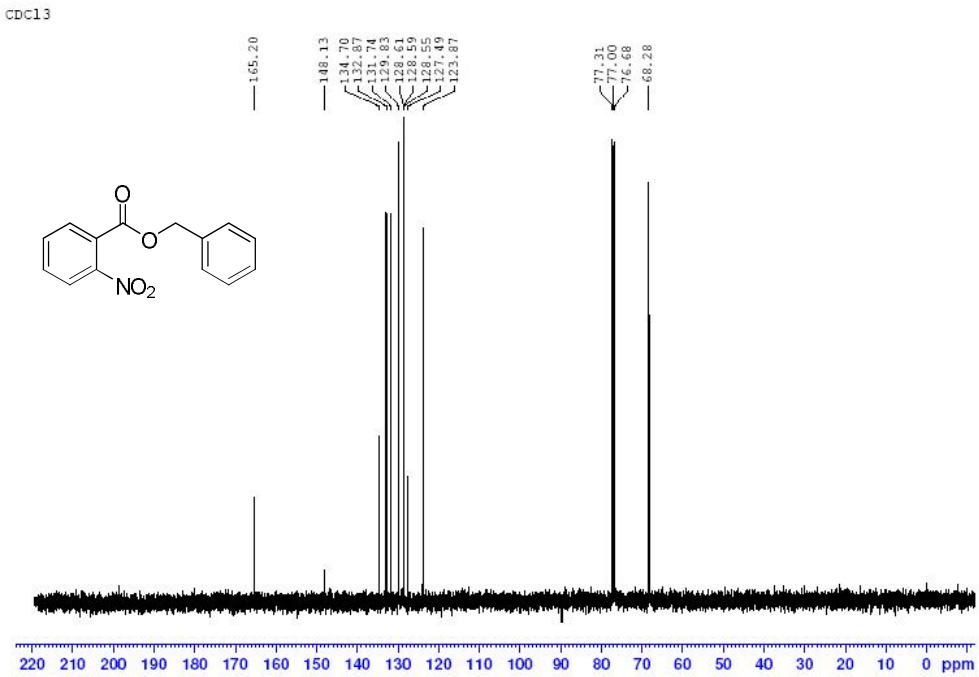
3m


CDCl3
7.9798
7.9753
7.9711
7.9591
7.9548
7.9507
7.4482
7.4440
7.4275
7.4257
7.4246
7.3947
7.3909
7.3861
7.3736
7.3694
7.3584
7.3552
7.3496
7.3436
7.3392
7.3353
7.3284
7.3216
7.3136
7.3074
7.3036
7.3000
7.2272
7.2067
5.3415
2.3855
11 10 9 8 7 6 5 4 3 2 1 0 ppm
2.01
1.96
3.08
2.00
2.00
3.00

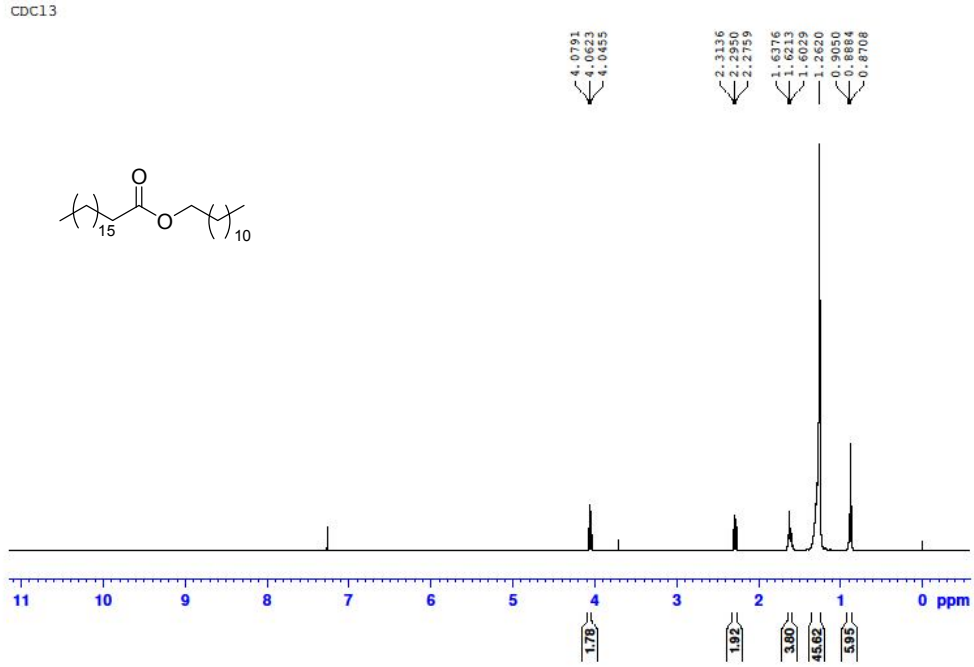

CDCl3
166.44
143.65
136.14
129.68
129.03
128.51
128.11
128.05
127.32
77.32
77.00
76.68
66.43
21.60
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

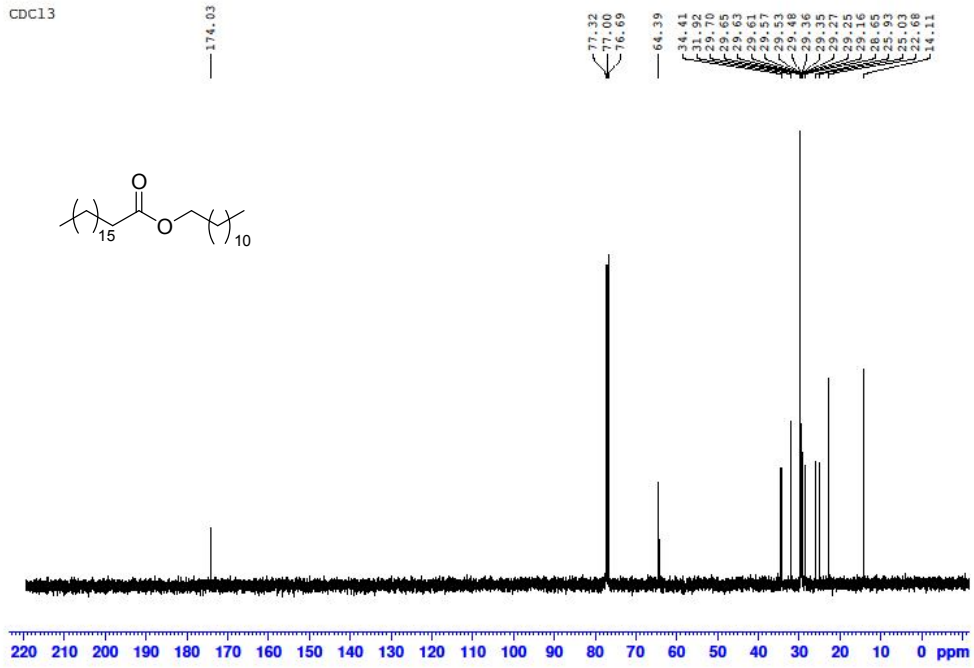
3n


CDCl3
7.9056
7.9024
7.9012
7.8861
7.8825
7.7489
7.7445
7.7309
7.7298
7.7260
7.6684
7.6650
7.6498
7.6462
7.6314
7.6274
7.6236
7.6189
7.6039
7.5999
7.5850
7.5810
7.4184
7.4127
7.4069
7.3991
7.3958
7.3934
7.3882
7.3849
7.3813
7.3777
7.3760
7.3708
7.3624
7.3592
7.3539
7.3496
7.3468
7.3378
7.3269
7.3222
7.3191
7.3166
5.3504
O
O
NO2
11
10
9
8
7
6
5
4
3
2
1
0 ppm
0.98
1.01
2.10
5.06
2.00

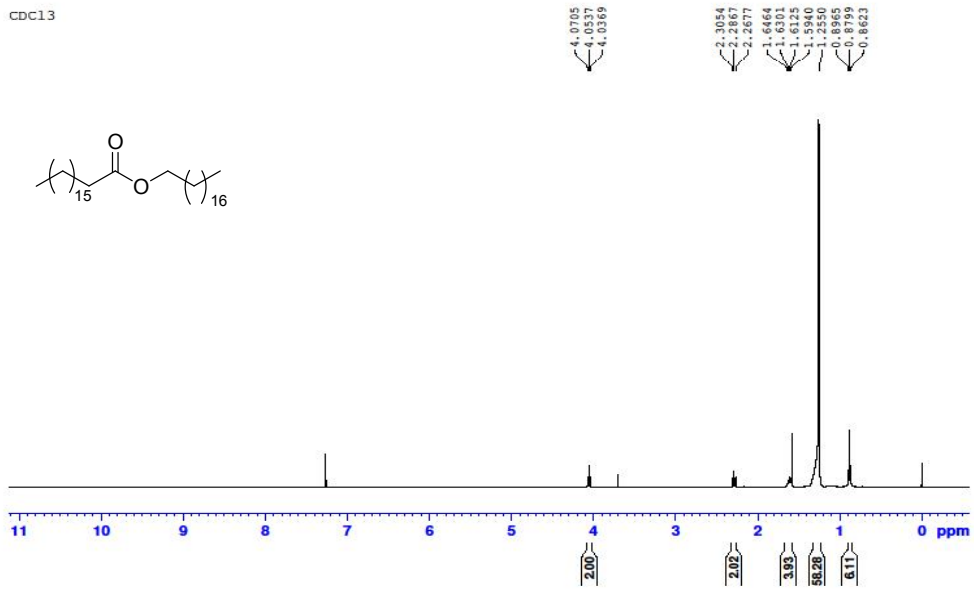

CDCl3
165.20
148.13
134.70
132.87
131.74
129.83
128.61
128.59
128.55
127.49
123.87
77.31
77.00
76.68
68.28
O
O
NO2
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm

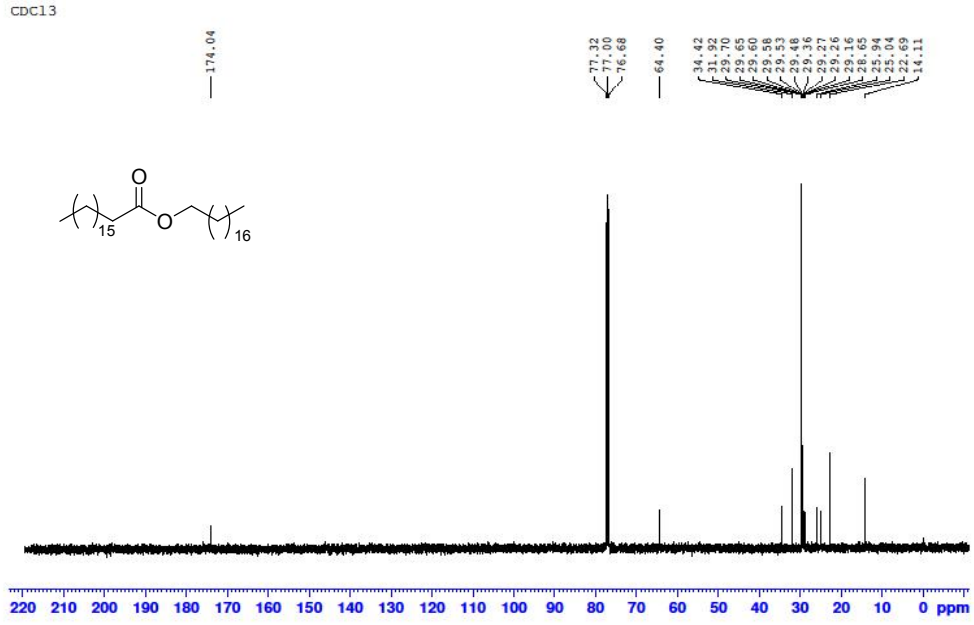
3o


CDCl3
4.0791
4.0623
4.0455
2.3136
2.2950
2.2759
1.6376
1.6213
1.6029
1.2620
0.9050
0.8864
0.8708
O
15
O
10
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.78
1.92
3.80
45.62
5.95

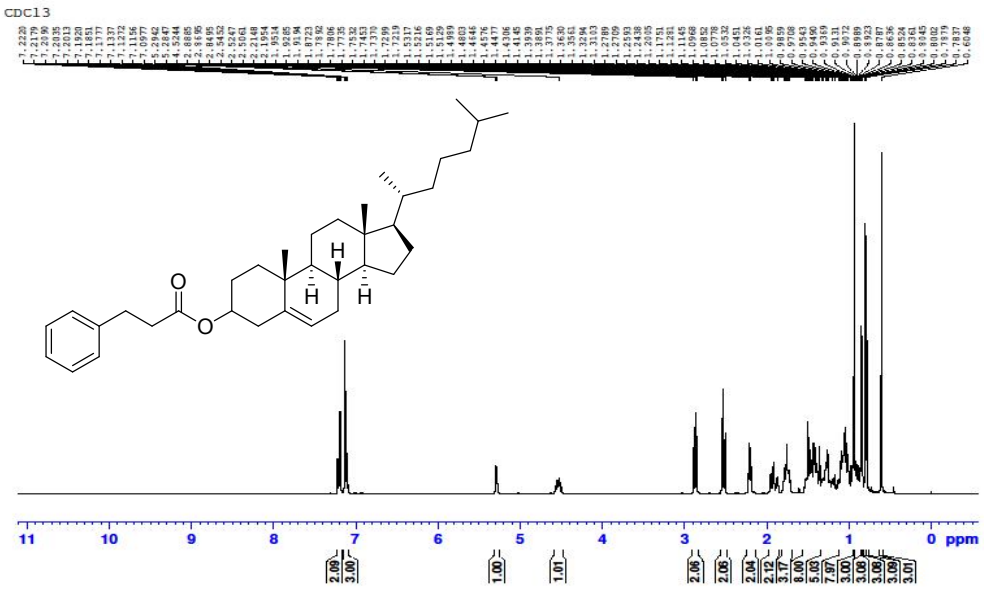

CDCl3
174.03
77.32
77.00
76.69
64.39
34.41
31.92
29.70
29.65
29.63
29.61
29.57
29.53
29.48
29.36
29.35
29.27
29.25
29.16
28.65
25.93
25.03
22.68
14.11
O
15
O
10
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

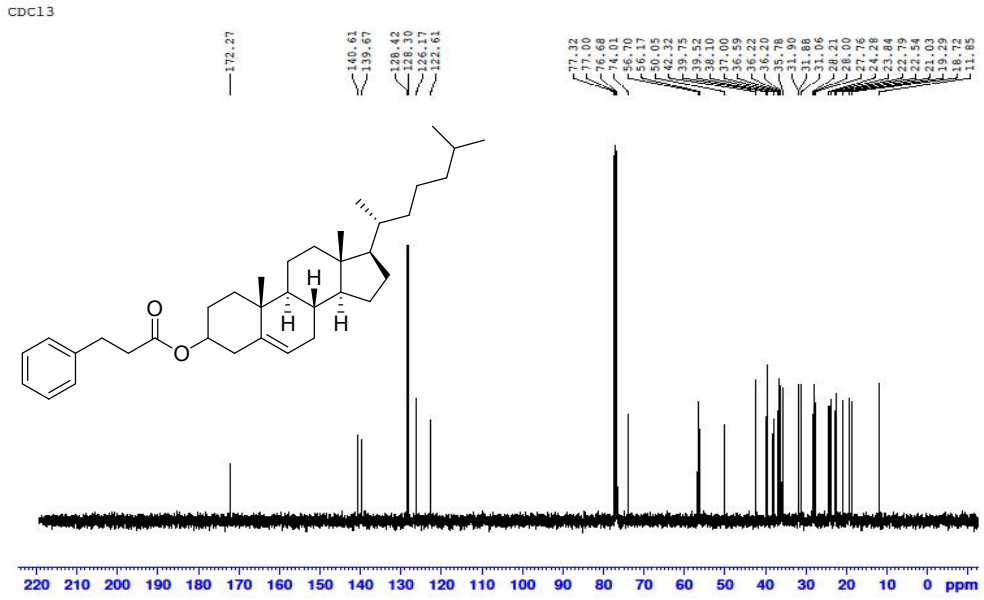
3p


CDCl3
4.0705
4.0537
4.0369
2.3054
2.2867
2.2677
1.6464
1.6301
1.6125
1.5940
1.2550
0.8965
0.8799
0.8623
O
15
O
16
11 10 9 8 7 6 5 4 3 2 1 0 ppm
2.00
2.02
3.93
58.28
6.11

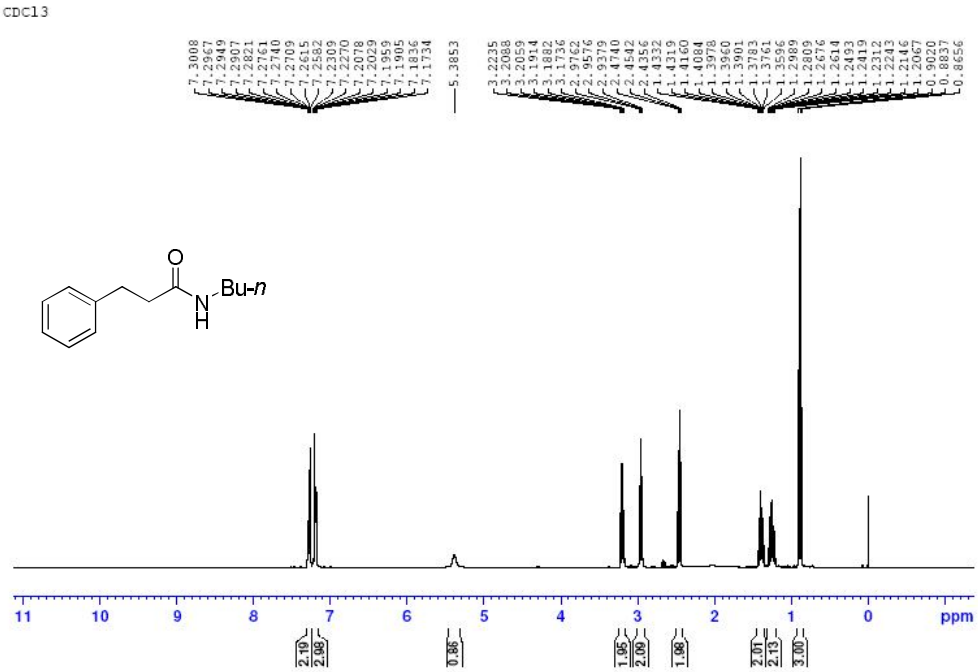

CDCl3
174.04
77.32
77.00
76.68
64.40
34.42
31.92
29.70
29.65
29.60
29.58
29.53
29.46
29.36
29.27
29.16
29.06
28.95
25.94
25.04
22.69
14.11
O
15
O
16
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

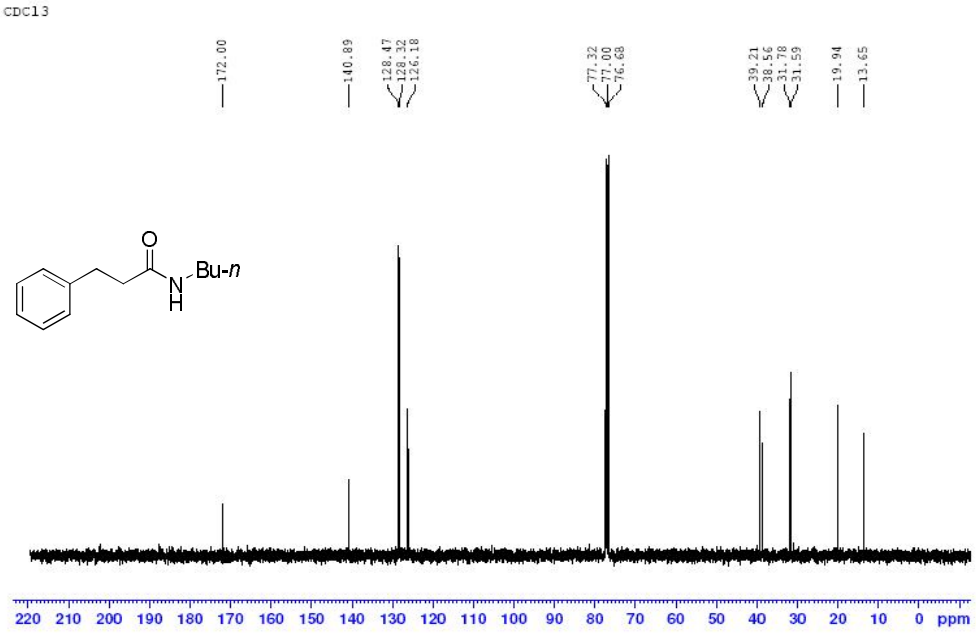
3q


CDCl3
O
O
H
H
H
11 10 9 8 7 6 5 4 3 2 1 0 ppm

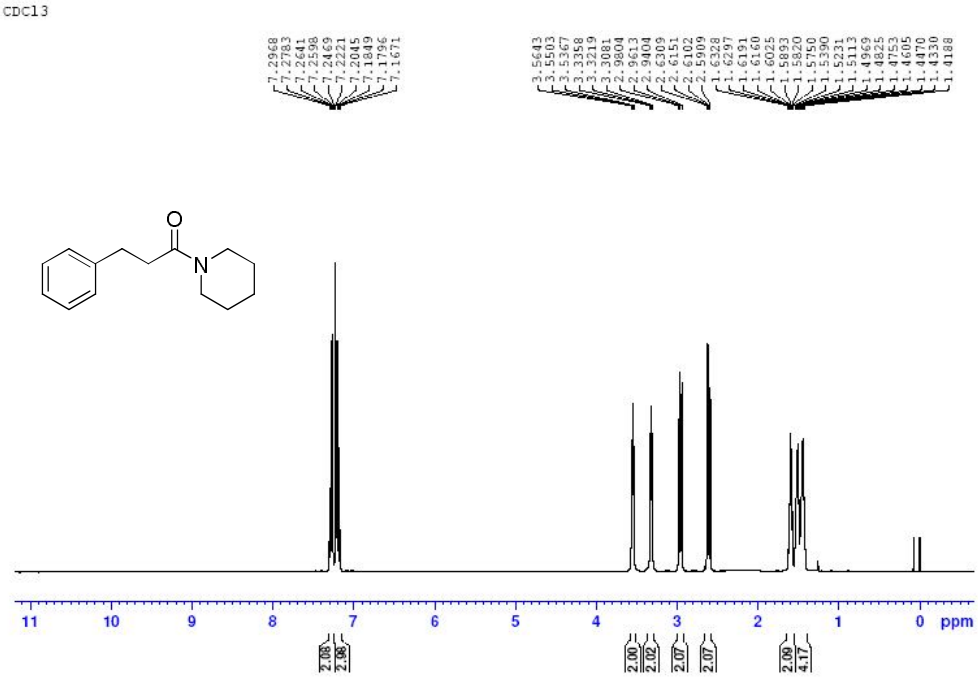

CDCl3
172.27
140.61
139.67
128.42
128.30
126.17
122.61
77.32
77.00
76.68
74.01
56.70
56.17
50.05
42.32
39.75
39.52
38.10
37.00
36.59
36.22
36.20
35.76
31.90
31.88
31.06
28.21
28.00
27.76
24.28
23.84
22.79
22.54
21.03
19.29
18.72
11.85
O
O
H
H
H
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

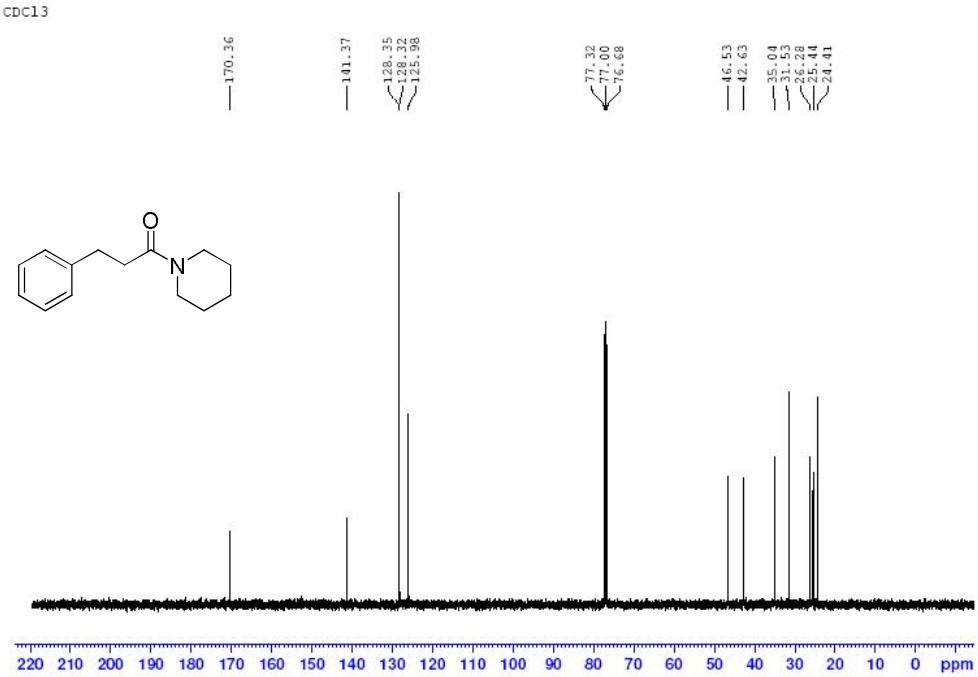
5a


CDCl3
O
N
H
Bu-n
11 10 9 8 7 6 5 4 3 2 1 0 ppm

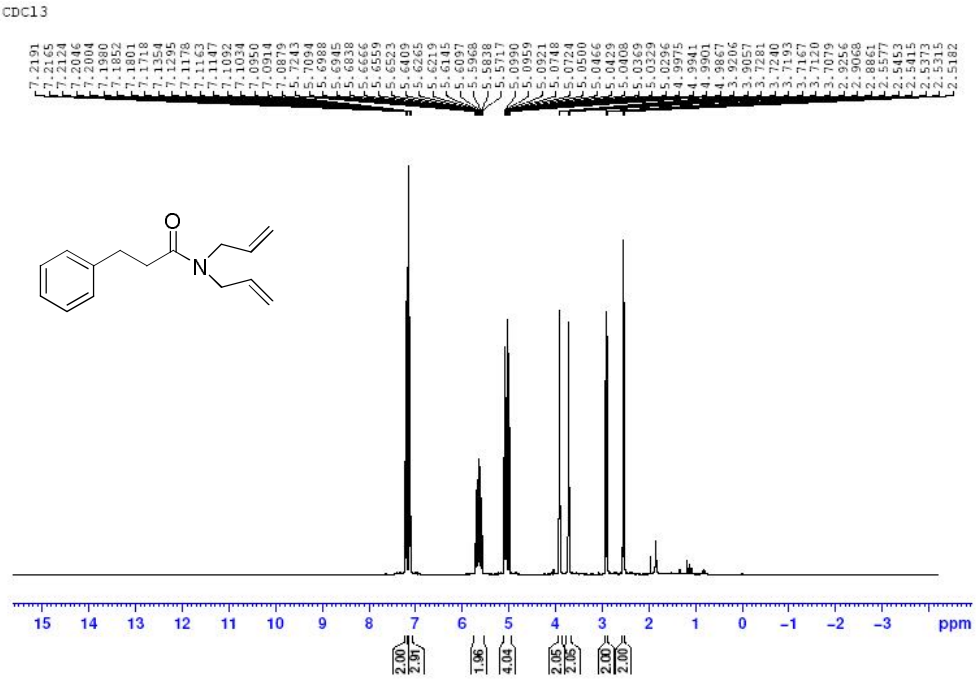

CDCl3
172.00
140.89
128.47
128.32
126.18
77.32
77.00
76.68
39.21
38.56
31.78
31.59
19.94
13.65
O
N
H
Bu-n
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

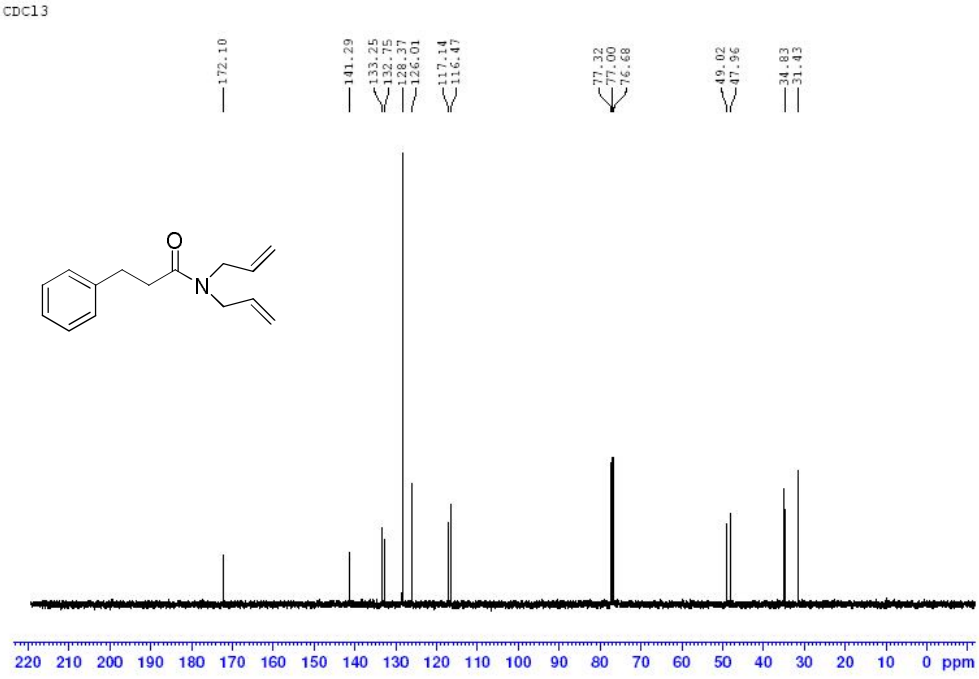
5b


CDCl3
7.2968
7.2783
7.2641
7.2598
7.2469
7.2221
7.2045
7.1849
7.1796
7.1671
3.5643
3.5503
3.5367
3.3358
3.3219
3.3081
2.9804
2.9613
2.9404
2.6309
2.6151
2.6102
2.5909
1.6228
1.6097
1.6041
1.6160
1.6025
1.5893
1.5820
1.5750
1.5390
1.5231
1.5113
1.4969
1.4825
1.4753
1.4605
1.4470
1.4330
1.4188
O
N
11 10 9 8 7 6 5 4 3 2 1 0 ppm
2.08
2.08
2.00
2.02
2.07
2.07
2.09
4.17

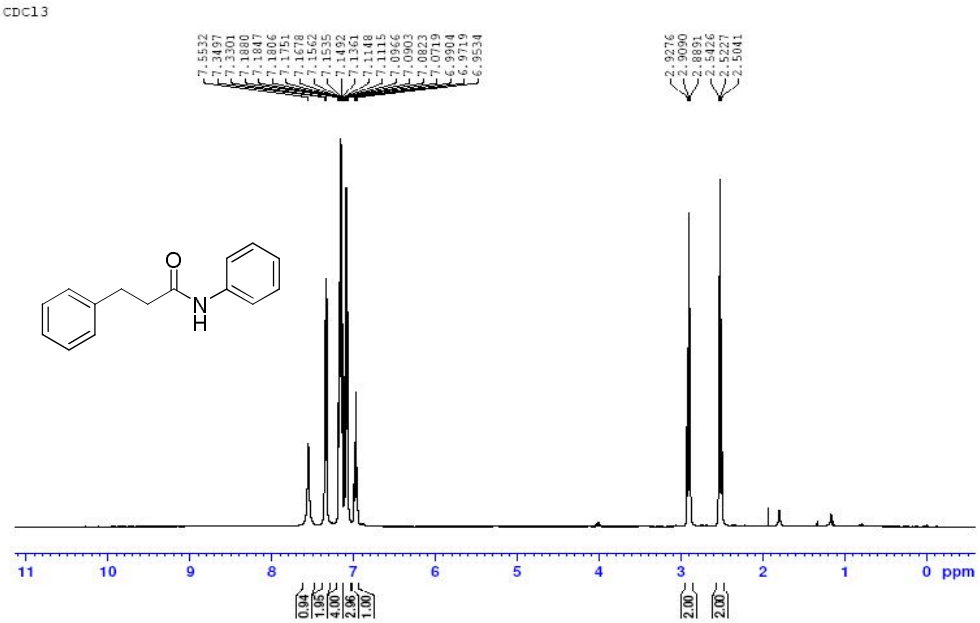

CDCl3
170.36
141.37
128.35
128.32
125.98
77.32
77.00
76.68
46.53
42.63
35.04
31.53
26.28
25.44
24.41
O
N
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

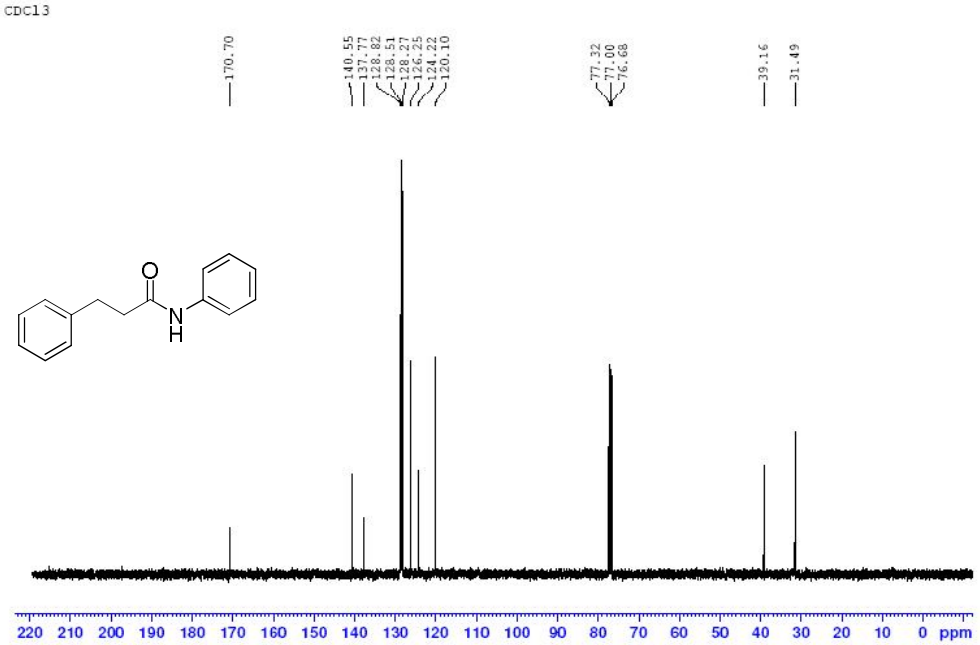
5c


CDCl3
7.2191
7.2165
7.2124
7.2046
7.2004
7.1980
7.1852
7.1801
7.1718
7.1354
7.1295
7.1178
7.1163
7.1147
7.1092
7.1034
7.0950
7.0914
7.0879
5.7243
5.7094
5.6988
5.6945
5.6838
5.6666
5.6559
5.6523
5.6409
5.6405
5.6219
5.6145
5.6097
5.5968
5.5838
5.5717
5.0990
5.0959
5.0921
5.0748
5.0724
5.0500
5.0466
5.0429
5.0408
5.0369
5.0329
5.0296
4.9975
4.9941
4.9901
4.9867
3.9206
3.9057
3.7281
3.7240
3.7193
3.7167
3.7120
3.7079
2.9256
2.9068
2.8861
2.5577
2.5453
2.5415
2.5373
2.5315
2.5182
O
N
15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 −1 −2 −3 ppm
2.00
2.01
1.96
4.04
2.05
2.05
2.00
2.00

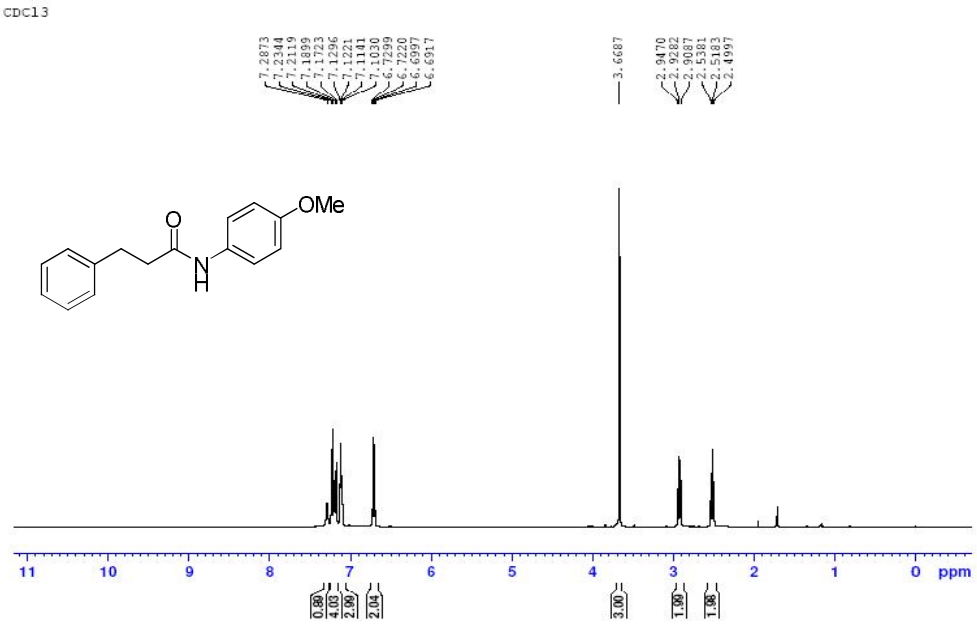

CDCl3
172.10
141.29
133.25
132.75
128.37
126.01
117.14
116.47
77.32
77.00
76.68
49.02
47.96
34.83
31.43
O
N
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

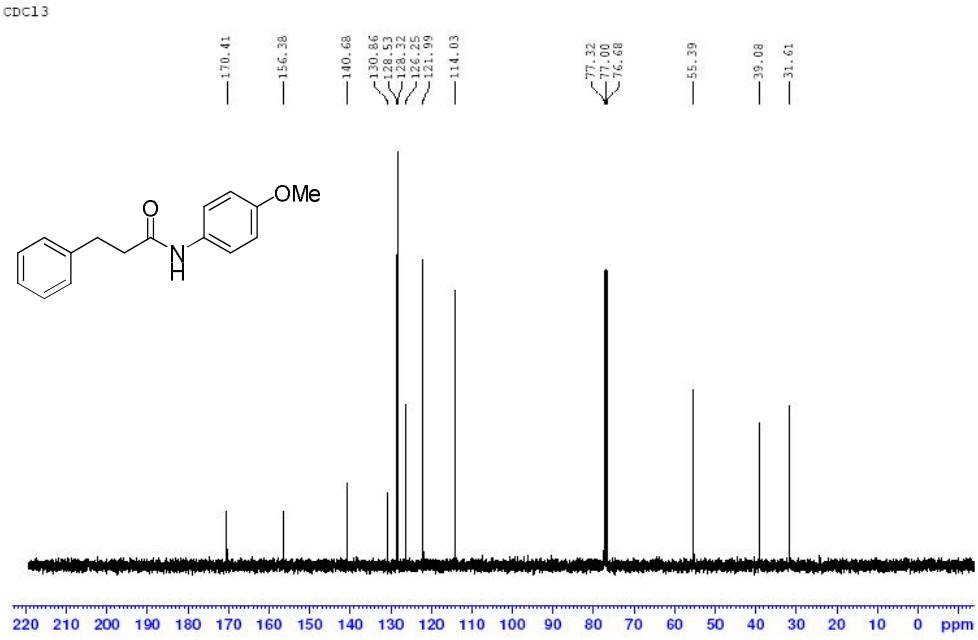
5d


CDCl3
7.5532
7.3497
7.3301
7.1880
7.1847
7.1806
7.1751
7.1678
7.1562
7.1535
7.1492
7.1361
7.1148
7.1115
7.0966
7.0903
7.0823
7.0719
6.9904
6.9719
6.9534
2.9276
2.9090
2.8891
2.5426
2.5227
2.5041
O
N
H
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.94
1.95
4.00
2.06
1.00
2.00
2.00

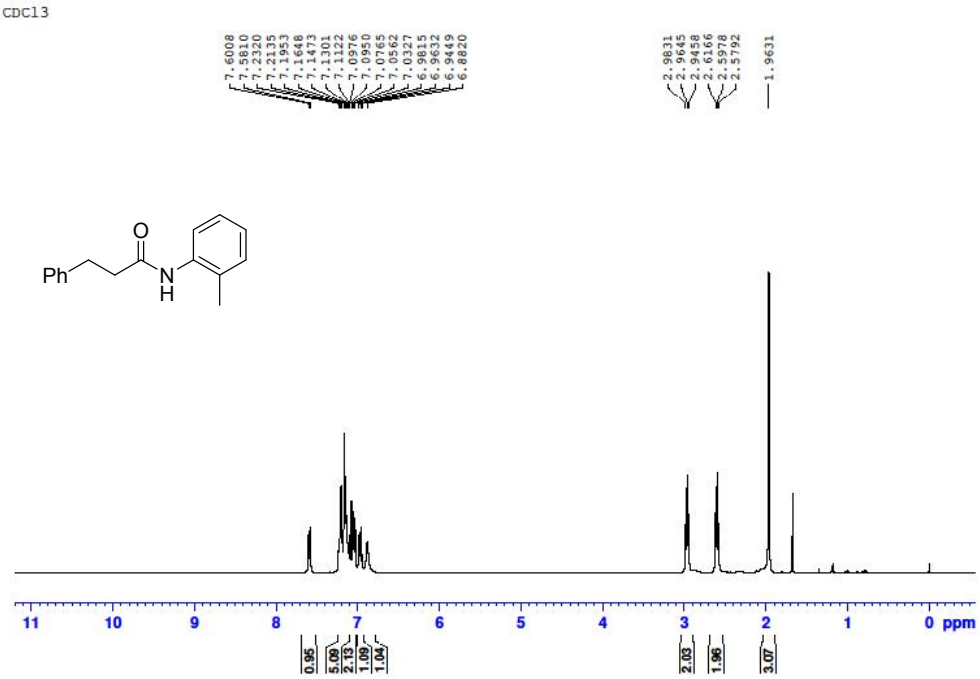

CDCl3
170.70
140.55
137.77
128.82
128.51
128.27
126.25
124.22
120.10
77.32
77.00
76.68
39.16
31.49
O
N
H
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

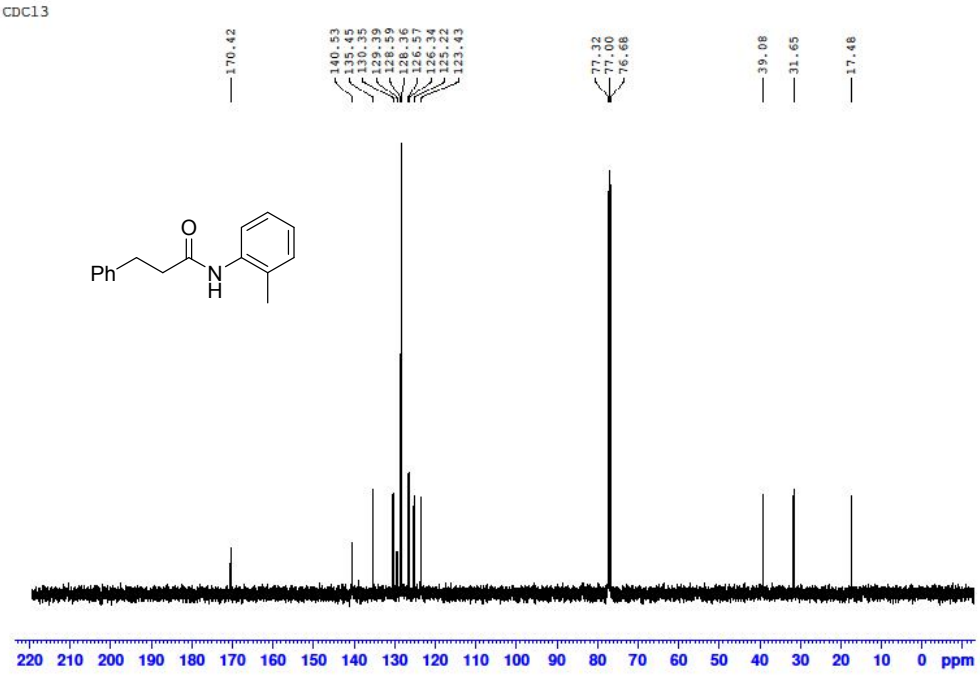
5e


CDCl3
7.2873
7.2344
7.2119
7.1899
7.1723
7.1296
7.1221
7.1141
7.1030
6.7299
6.7220
6.6997
6.6917
3.6687
2.9470
2.9282
2.9087
2.5381
2.5183
2.4997
OMe
O
N
H
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.80
4.03
2.99
2.04
3.00
1.99
1.06

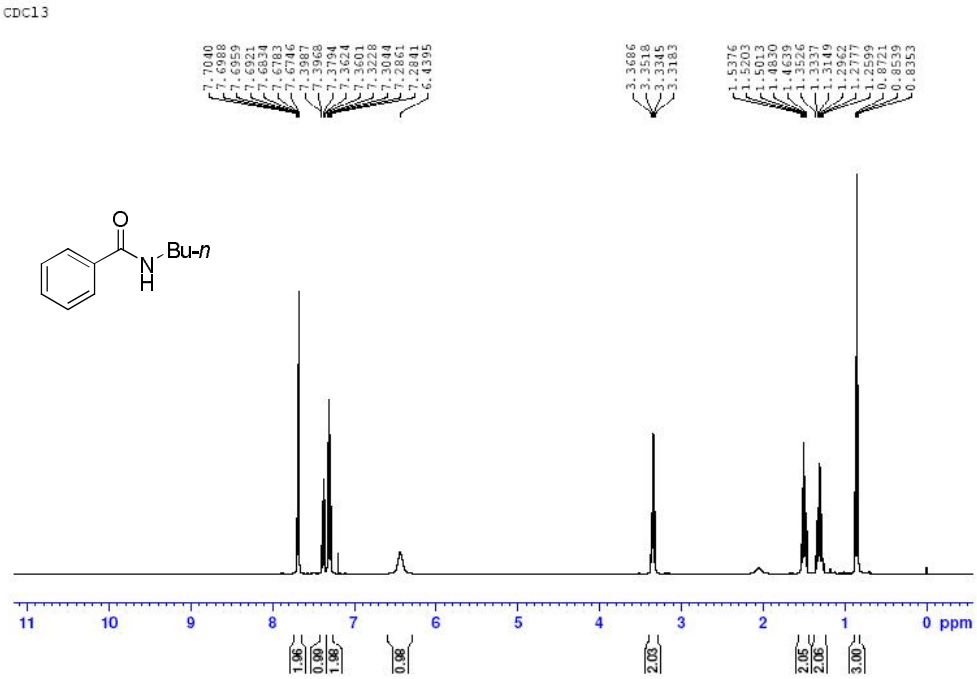

CDCl3
170.41
156.38
140.68
130.86
128.53
128.32
126.25
121.99
114.03
77.32
77.00
76.68
55.39
39.08
31.61
OMe
O
N
H
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

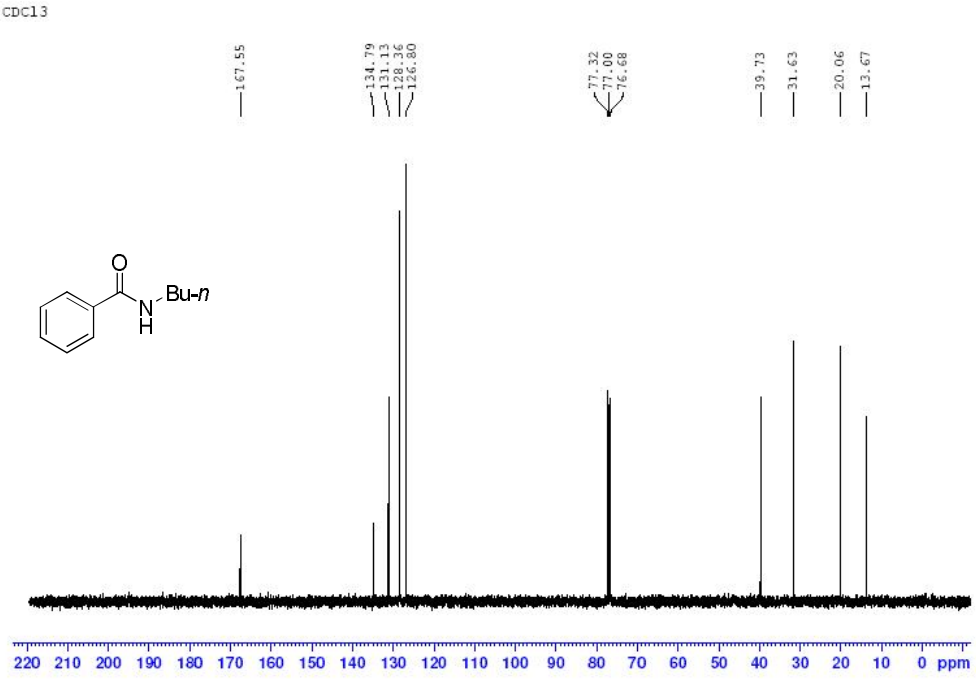
5f


CDCl3
7.6008
7.5810
7.2320
7.2135
7.1953
7.1648
7.1473
7.1301
7.1122
7.0976
7.0950
7.0765
7.0562
7.0327
6.9815
6.9632
6.9449
6.8820
2.9831
2.9645
2.9458
2.6166
2.5978
2.5792
1.9631
O
N
H
Ph
11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.95
5.09
2.13
1.09
1.04
2.03
1.96
3.07

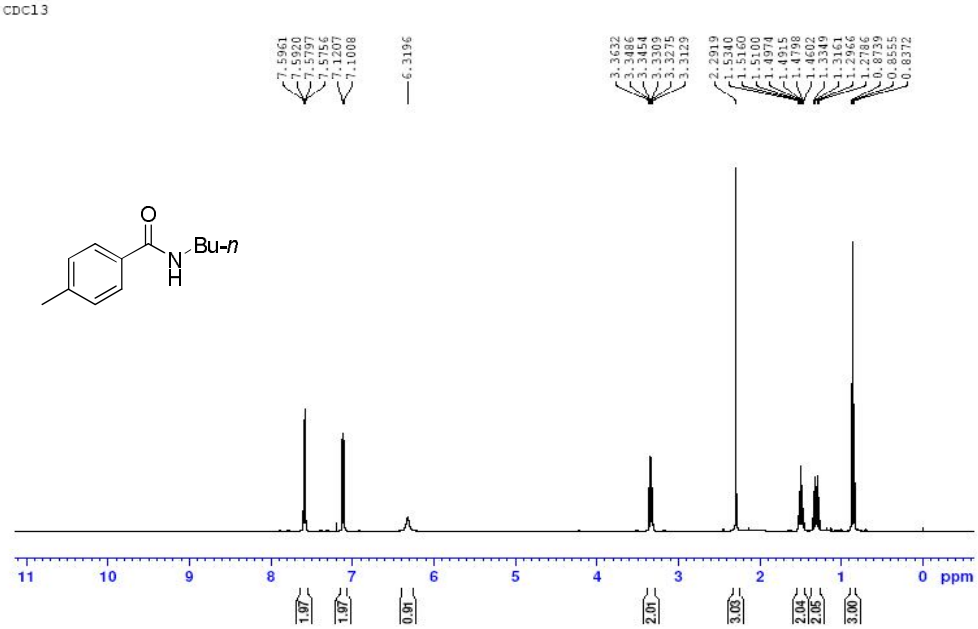

CDCl3
170.42
140.53
135.45
130.35
129.39
128.59
128.36
126.57
126.34
125.22
123.43
77.32
77.00
76.68
39.08
31.65
17.48
O
N
H
Ph
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

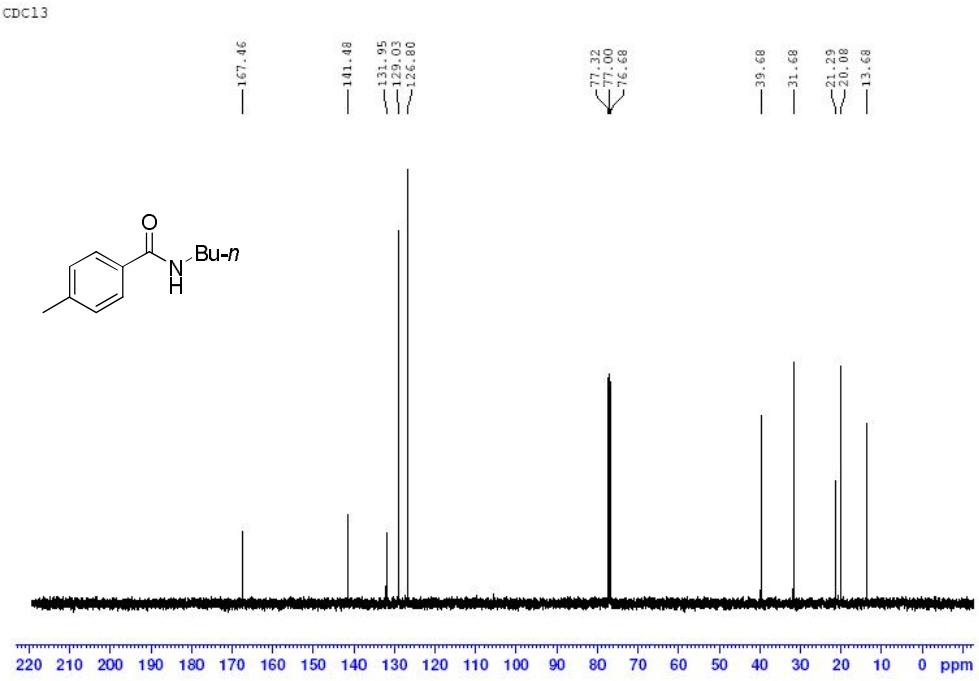
5g


CDCl3
7.7040
7.6988
7.6959
7.6921
7.6834
7.6783
7.6746
7.3987
7.3968
7.3794
7.3624
7.3601
7.3228
7.3044
7.2861
7.2841
6.4395
3.3686
3.3518
3.3345
3.3183
1.5376
1.5203
1.5013
1.4830
1.4639
1.3526
1.3337
1.3149
1.2962
1.2777
1.2599
0.8721
0.8559
0.8353
O
N
H
Bu-n
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.96
0.90
1.98
0.98
2.03
2.05
2.06
3.00

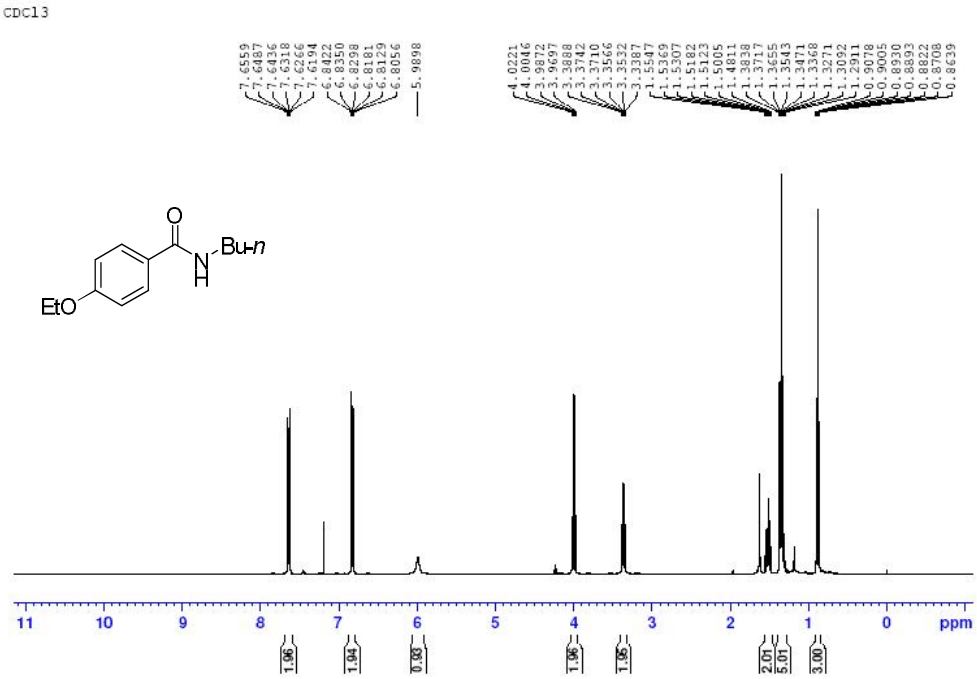

CDCl3
167.55
134.79
131.13
128.36
126.80
77.32
77.00
76.68
39.73
31.63
20.06
13.67
O
N
H
Bu-n
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

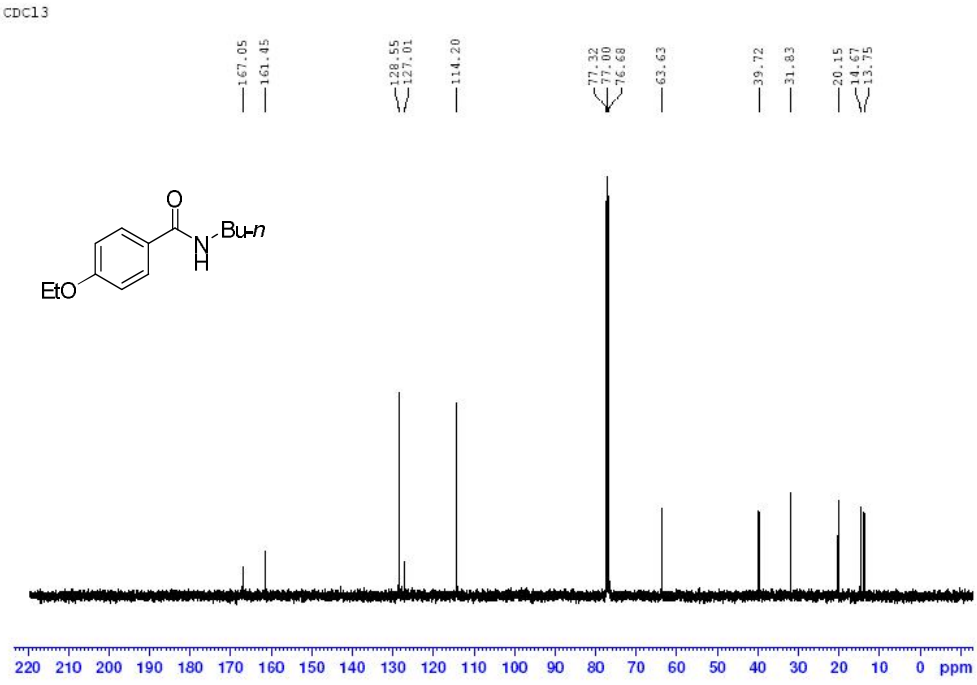
5h


CDCl3
7.5961
7.5920
7.5797
7.5756
7.1207
7.1008
6.3196
3.3632
3.3486
3.3454
3.3309
3.3275
3.3129
2.2919
1.5340
1.5160
1.5100
1.4974
1.4915
1.4798
1.4602
1.3349
1.3161
1.2966
1.2786
0.8739
0.8555
0.8372
Bu-n
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.97
1.97
0.91
2.01
3.03
2.04
2.05
3.00

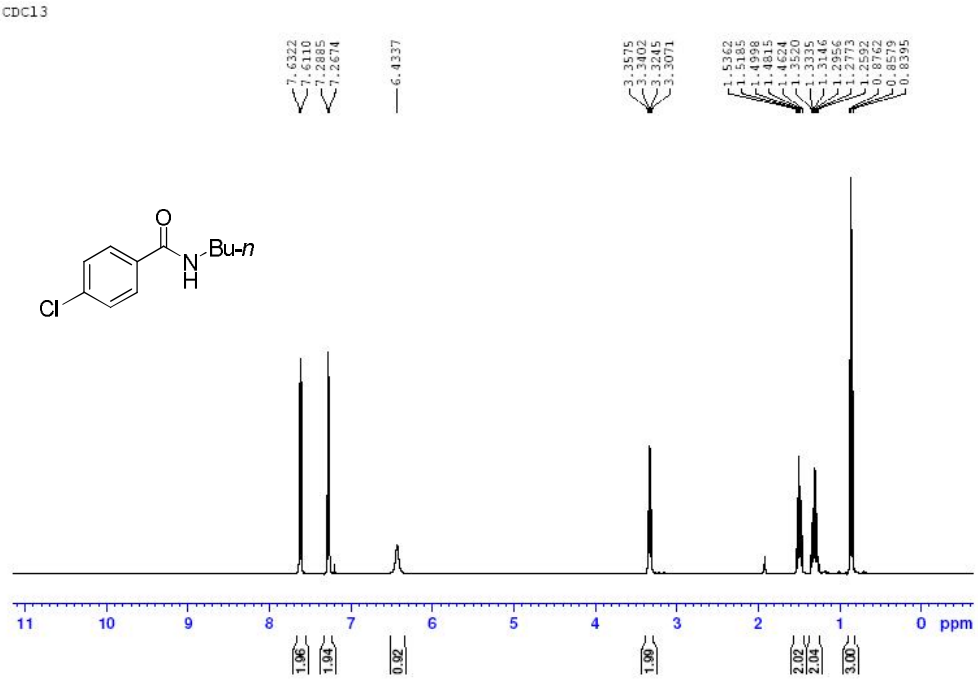

CDCl3
167.46
141.48
131.95
129.03
126.80
77.32
77.00
76.68
39.68
31.68
21.29
20.08
13.68
Bu-n
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

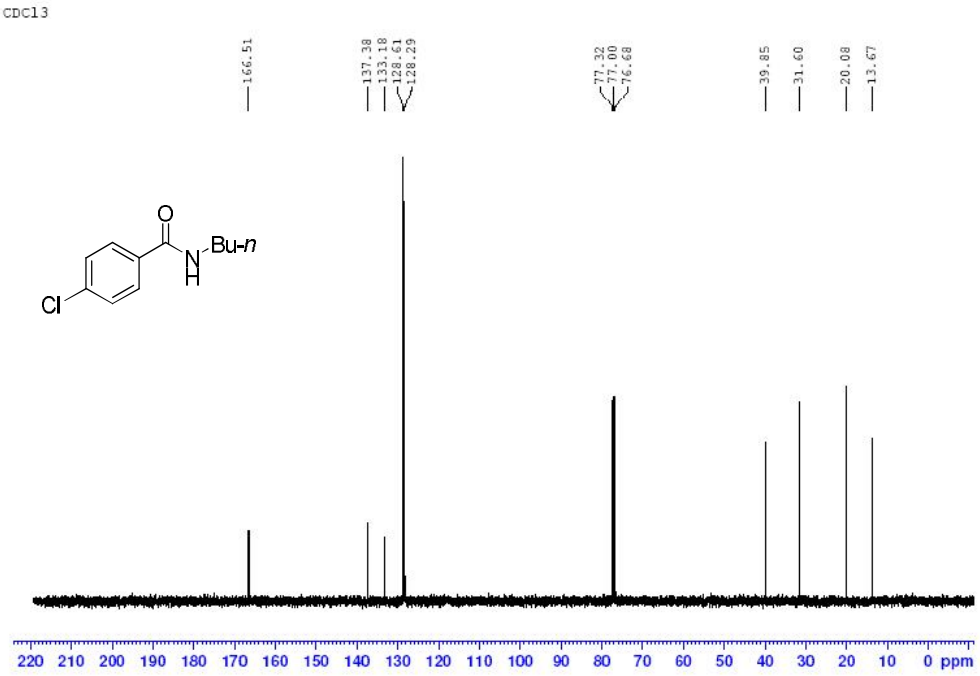
5i


CDCl3
7.6559
7.6487
7.6436
7.6318
7.6266
7.6194
6.8422
6.8350
6.8298
6.8181
6.8129
6.8056
5.9898
4.0221
4.0046
3.9872
3.9697
3.3888
3.3742
3.3710
3.3566
3.3532
3.3387
1.5547
1.5369
1.5307
1.5182
1.5123
1.5005
1.4811
1.3838
1.3717
1.3655
1.3543
1.3471
1.3368
1.3271
1.3092
1.2911
0.9078
0.9005
0.8930
0.8883
0.8822
0.8768
0.8639
O
N
H
Bu-n
EtO
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.96
1.84
0.88
1.96
1.85
2.01
5.01
3.00

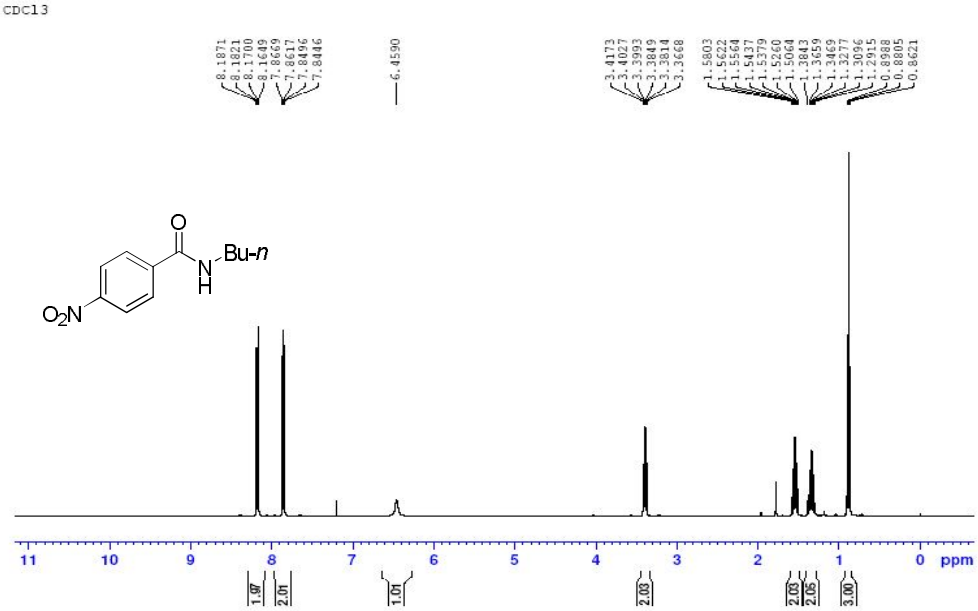

CDCl3
167.05
161.45
128.55
127.01
114.20
77.32
77.00
76.68
63.63
39.72
31.83
20.15
14.67
13.75
O
N
H
Bu-n
EtO
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

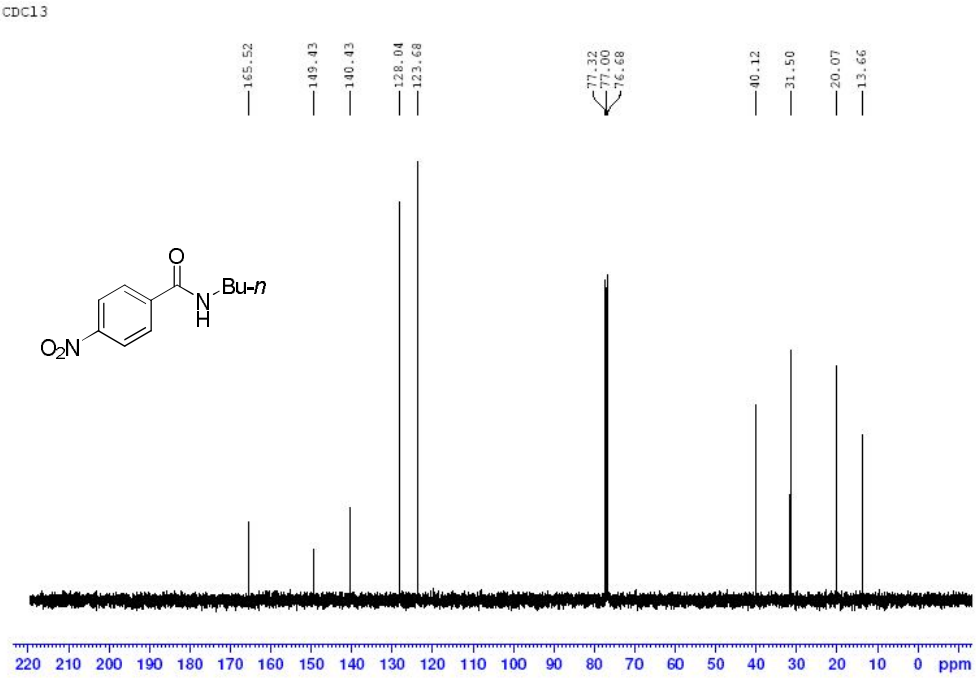
5j


CDCl3
7.6322
7.6110
7.2885
7.2674
6.4337
3.3575
3.3402
3.3245
3.3071
1.5362
1.5185
1.4998
1.4811
1.4624
1.3520
1.3335
1.3146
1.2956
1.2773
1.2592
0.8762
0.8579
0.8395
O
N
H
Bu-n
Cl
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.96
1.04
0.92
1.00
2.02
2.04
3.00

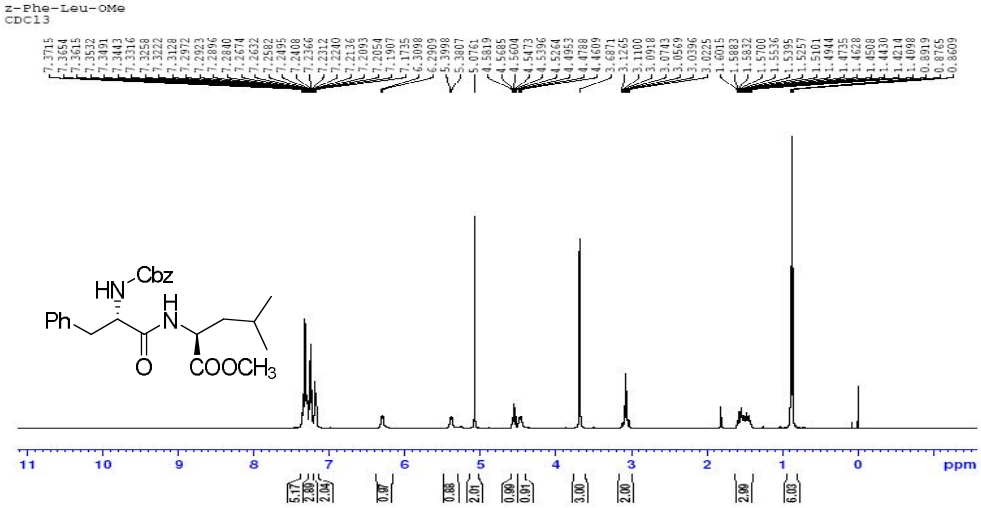

CDCl3
166.51
137.38
133.18
128.61
128.29
77.32
77.00
76.68
39.85
31.60
20.08
13.67
O
N
H
Bu-n
Cl
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

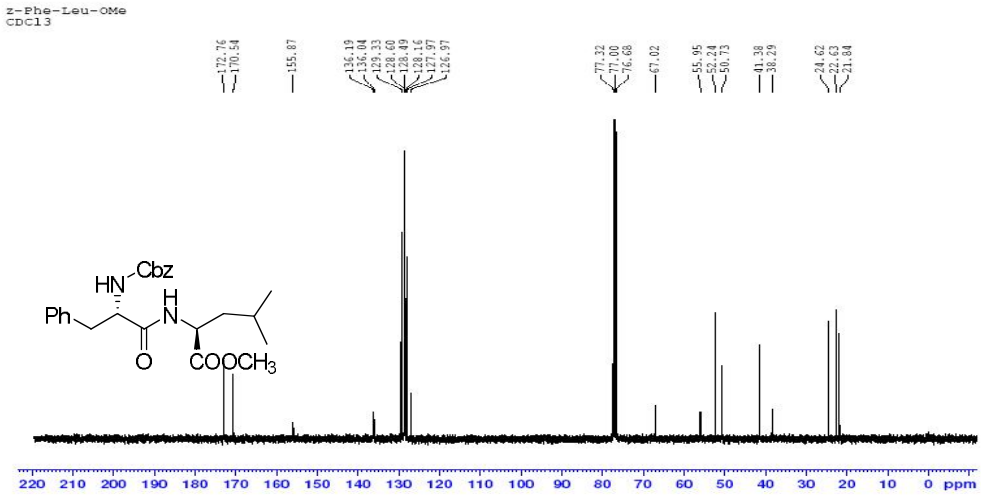
5k

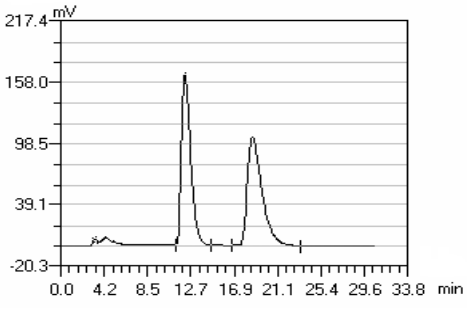

CDCl3
8.1871
8.1821
8.1700
8.1649
7.8669
7.8617
7.8496
7.8446
6.4590
3.4173
3.4027
3.3993
3.3849
3.3814
3.3668
1.5803
1.5622
1.5564
1.5437
1.5379
1.5260
1.5064
1.3843
1.3659
1.3469
1.3277
1.3096
1.2915
0.8988
0.8805
0.8621
Bu-n
O2N
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.97
2.01
1.01
2.03
2.03
2.05
3.00

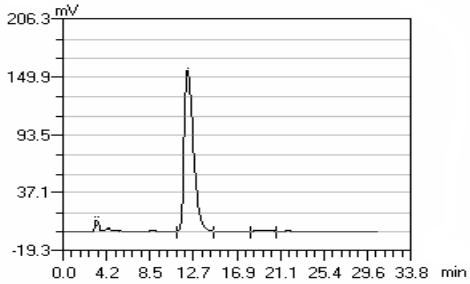

CDCl3
165.52
149.43
140.43
128.04
123.68
77.32
77.00
76.68
40.12
31.50
20.07
13.66
Bu-n
O2N
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

5l





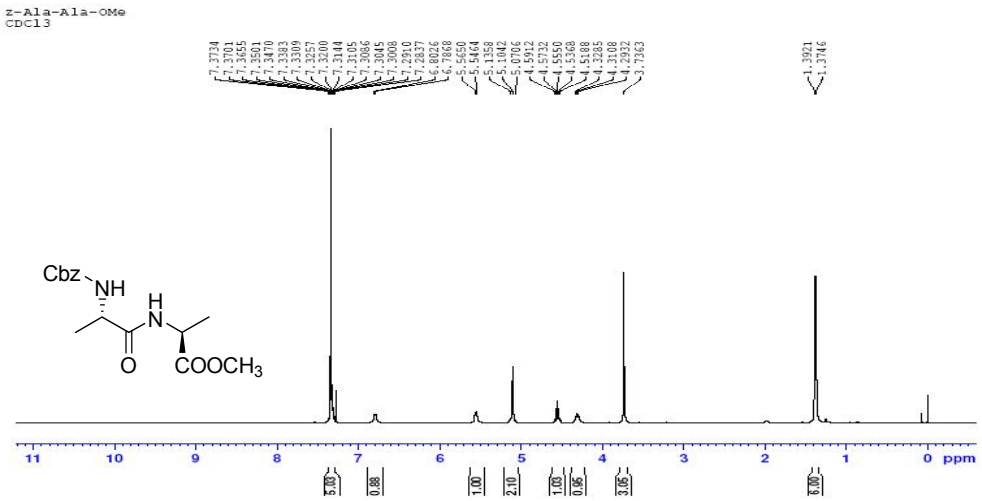


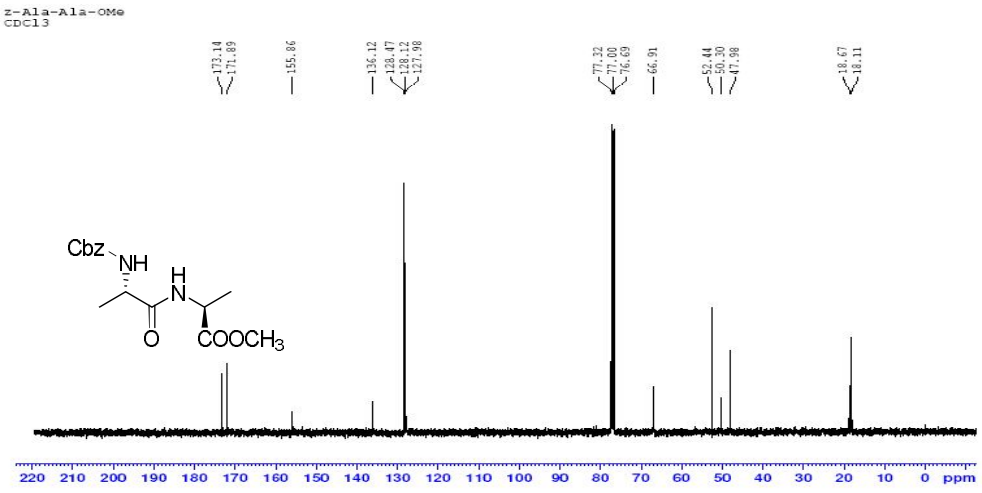


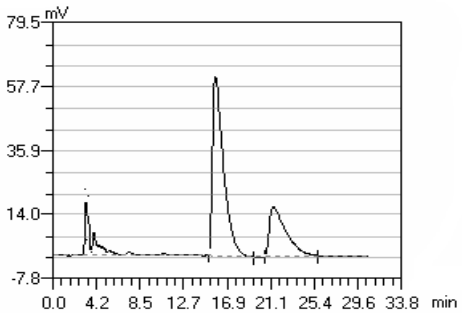
PEAK#	RT(min)	AREA	AREA%
1	11.97	10263	49.7
2	18.50	10397	50.3

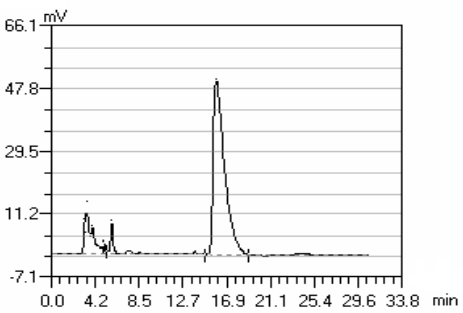
PEAK#	RT(min)	AREA	AREA%
1	12.03	9801	99.5
2	19.03	54	0.5

5m





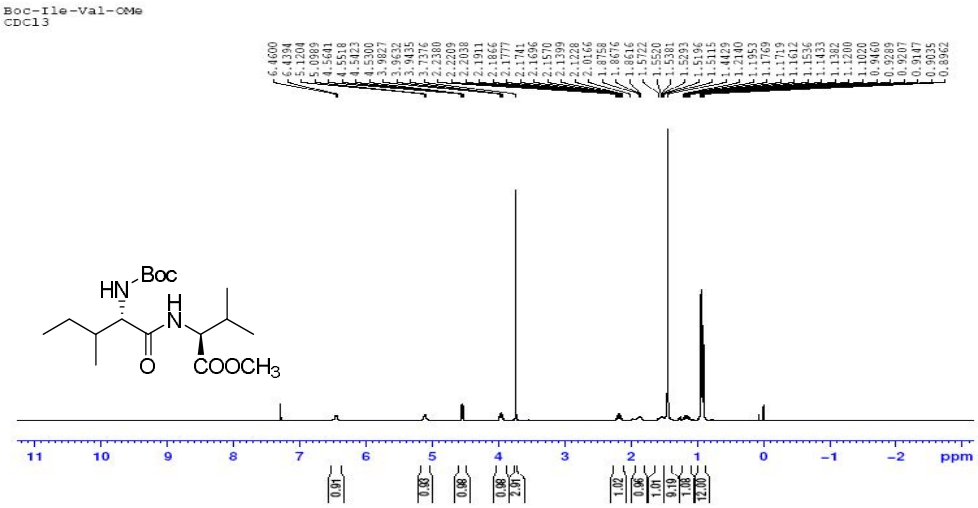


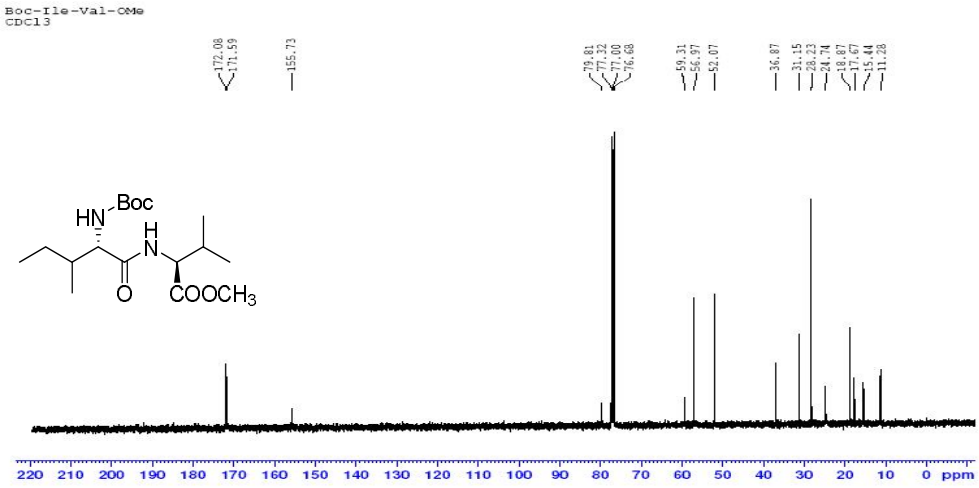


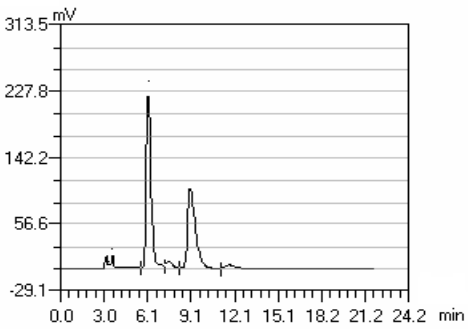
PEAK#	RT(min)	AREA	AREA%
1	15.63	4464	70.8
2	21.30	1840	29.2

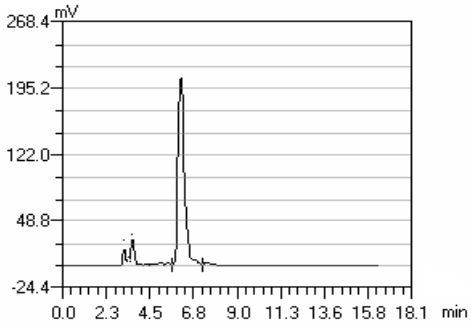
PEAK#	RT(min)	AREA	AREA%
1	15.77	3796	99.5
2	24.63	17	0.5

5n





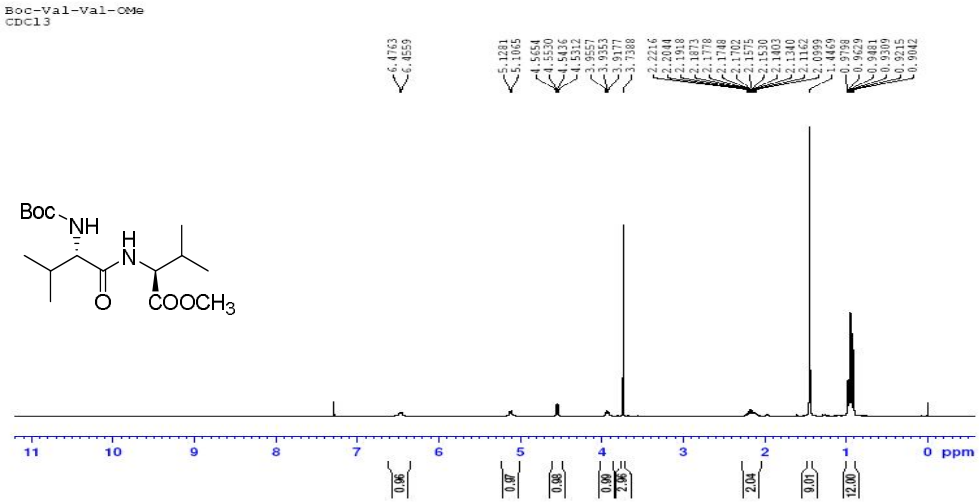


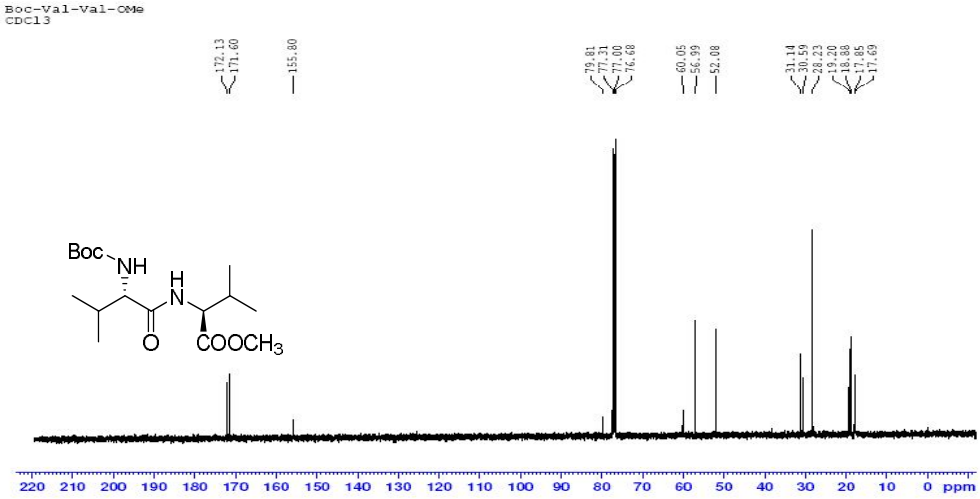


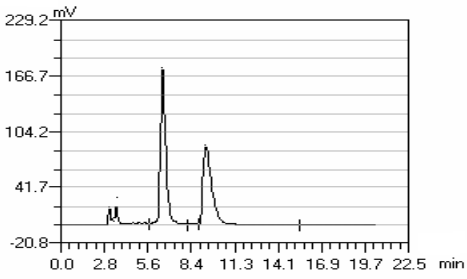
PEAK#	RT(min)	AREA	AREA%
1	6.03	5306	57.7
2	8.97	3890	42.3

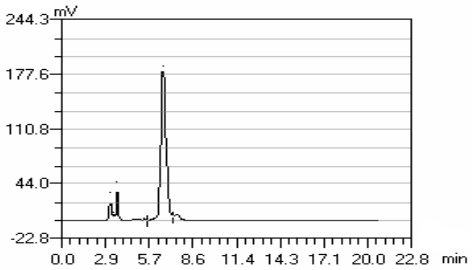
PEAK#	RT(min)	AREA	AREA%
1	6.10	4828	99.8
2	9.23	8	0.2

5o





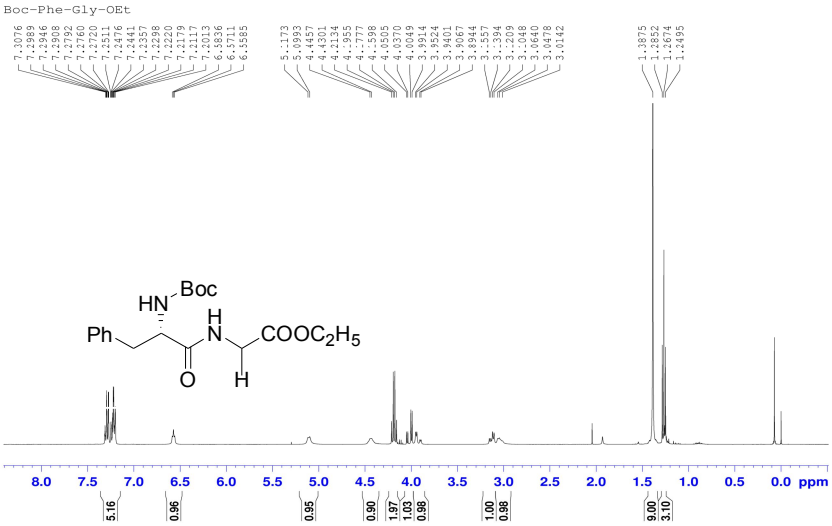


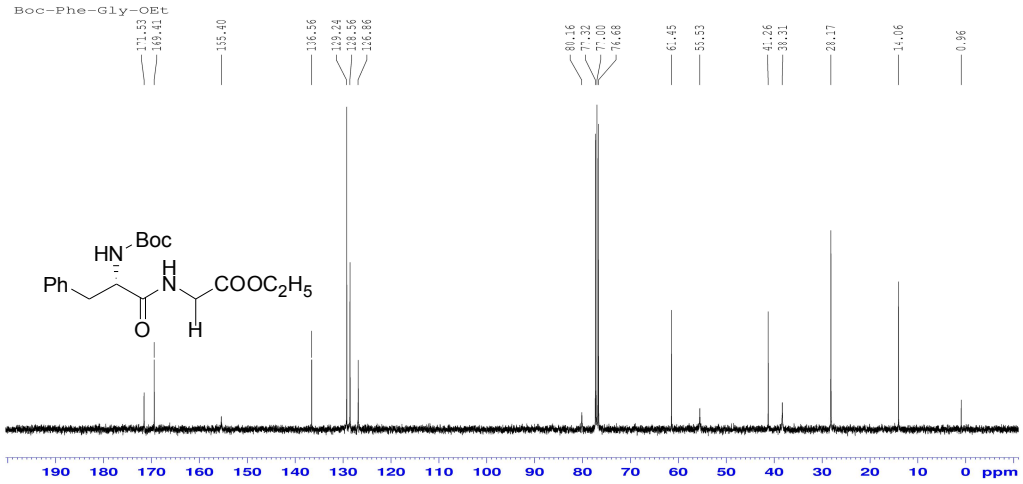


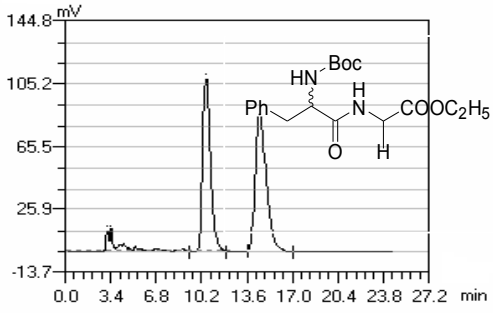
PEAK#	RT(min)	AREA	AREA%
1	6.50	4372	56.4
2	9.30	3384	43.6

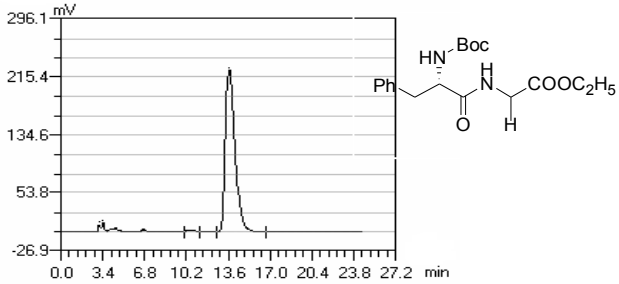
PEAK#	RT(min)	AREA	AREA%
1	6.57	4340	99.9
2	9.77	4	0.1

5p





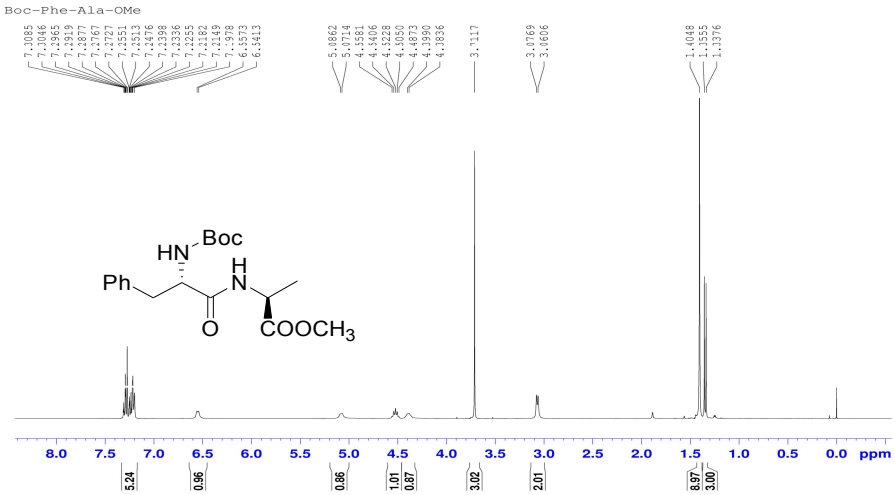


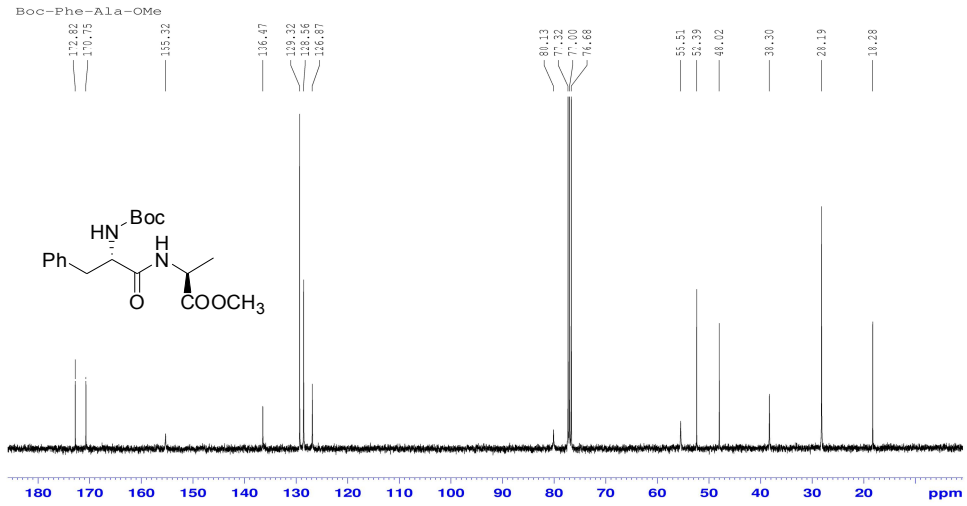


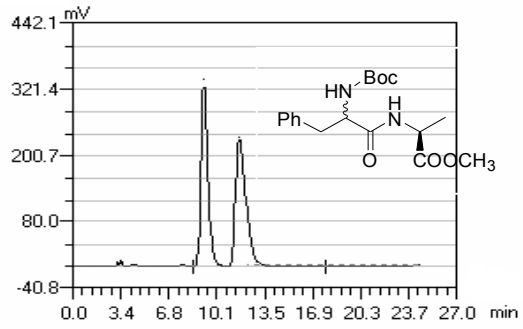
PEAK#	RT(min)	AREA	AREA%
1	10.43	4346	46.9
2	14.43	4915	53.1

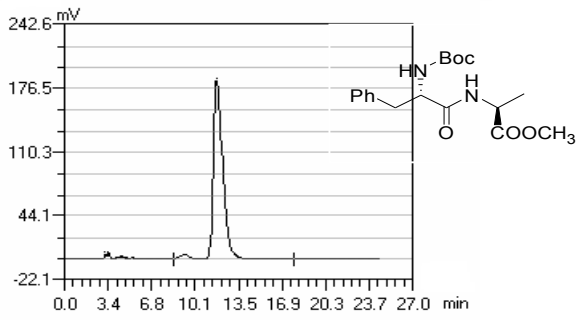
PEAK#	RT(min)	AREA	AREA%
1	10.50	54	0.4
2	13.57	12034	99.6

5q





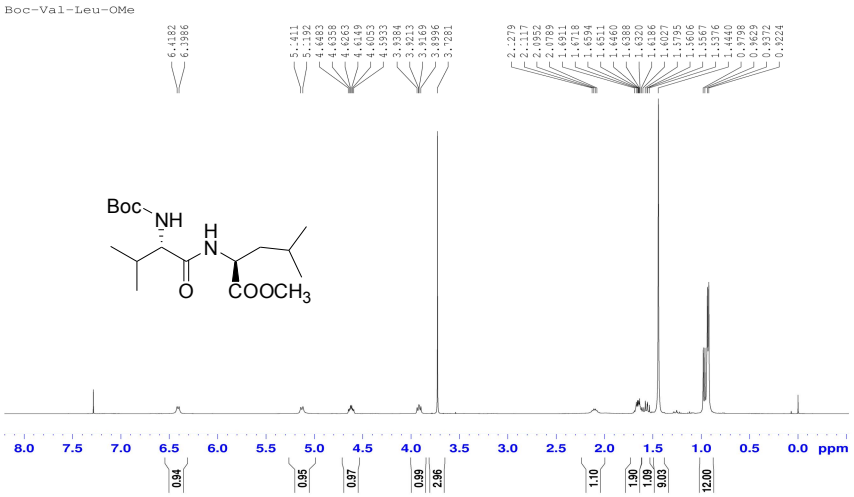


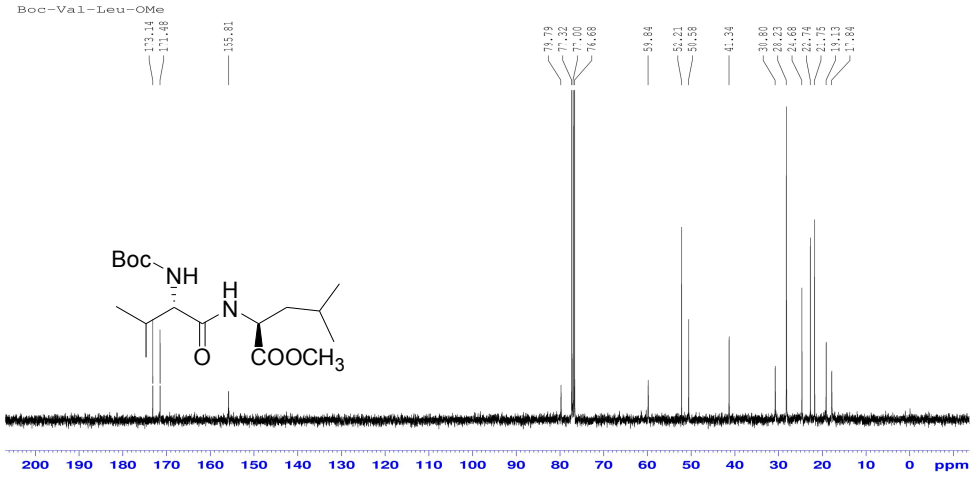


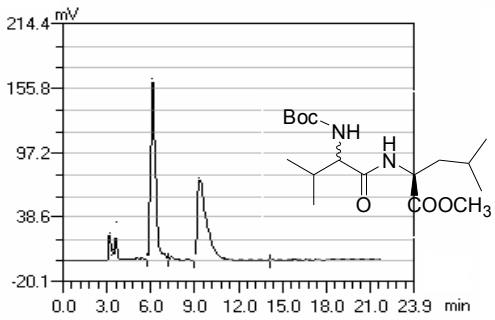
PEAK#	RT(min)	AREA	AREA%
1	9.17	11465	50.9
2	11.63	11075	49.1

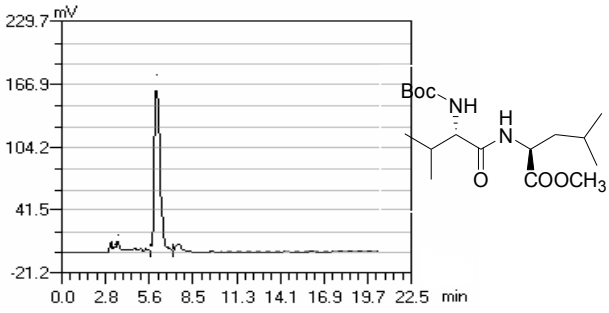
PEAK#	RT(min)	AREA	AREA%
1	9.23	100	1.1
2	11.70	9280	98.9

5r









PEAK#	RT(min)	AREA	AREA%
1	6.03	3868	54.3
2	9.23	3252	45.7

PEAK#	RT(min)	AREA	AREA%
1	6.03	4199	100.0