Supplementary information

Metal-free DDQ-mediated oxidative C-O coupling of acetalic sp^3 C-H bonds with carboxylic acids

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Contents

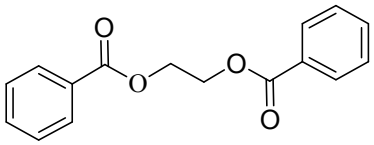
General information

All solvents and reagents were purchased from the suppliers and used without further purification. Yields reported are for isolated yields unless otherwise stated. ^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz) spectra were recorded in $CDCl_3$ at room temperature on Bruker Avance III 400 spectrometer. The chemical-shift scale is based on internal TMS. IR spectra were recorded by Thermo Nicolet Avatar 360. MS spectra were performed on a Agilent 1100 series mass spectrometer. Elemental analyses were measured on a Perkin Elmer 2400 series analyzer. TLC analyses were performed on silica gel plates and column chromatography was conducted over silica gel (mesh 200-300) at increased pressure.

General procedure

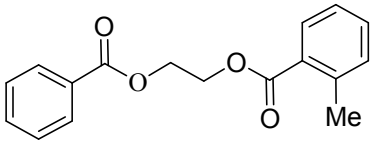
A reaction sealed tube was charged with DDQ (1.2 mmol), acid (1.0 mmol) and dry CH_2Cl_2 (4 mL), then purged with N_2. After the addition of acetal (2.0 mmol) under N_2, the reaction mixture was stirred at 80 ^{o}C (oil bath) for 12 h. The mixture was allowed to cool at room temperature, ethyl acetate (2 mL) added, and the insoluble residue filtered. The filtrate was diluted with ethyl acetate (20 mL), subsequently washed with aqueous saturated $NaHCO_3$ (10 mL × 3), and dried over anhydrous Na_2SO_4. To the organic phase little silica gel was added. The solvent was roto-evaporated under the reduced pressure, the resulting powder added to the top of a short silica-gel column and purified using petroleum ether/ethyl acetate in a 20:1~10:1 (v/v) ratio as the eluent to afford the desired product.

Ethane-1,2-diyl dibenzoate (**3aa & 3al**)



White solid, mp 75~76 ^{o}C (lit.[1] 73 ^{o}C); ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.08-8.05 (m, 4 H), 7.59-7.55 (m, 2 H), 7.47-7.43 (m, 4 H), 4.67(s, 4 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 166.43 (2 C), 133.20 (2 C), 129.81 (2 C), 129.74 (4 C), 128.46 (4 C), 62.78 (2 C); IR (KBr) ν_{max}/cm^{-1} 1727, 1707, 1257, 1114, 701; MS (EI) m/z: 270.0, 149.0, 105.1, 77.0; Anal. Calcd for $C_{16}H_{14}O_4$ C 71.10, H 5.22; Found: C 71.18, H 5.31%.

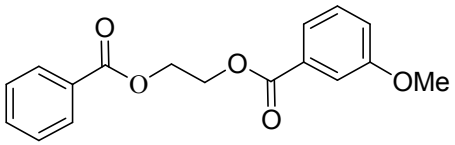
2-(Benzoyloxy)ethyl 2-methylbenzoate (**3ab**)



Orange liquid; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.07 (d, *J* = 8.0 Hz, 2 H),7.94 (d, *J* = 8.0 Hz, 1 H), 7.57 (t, *J* = 7.2 Hz,1 H), 7.46-7.37 (m, 4 H), 7.25-7.22 (m, 1 H), 4.66 (s, 4 H), 2.60 (s, 3 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 167.37, 166.40, 140.35, 133.19, 132.22, 131.75, 130.78, 129.83, 129.73 (2 C), 129.22, 128.44 (2 C), 125.80, 62.84, 62.49, 21.83; IR (KBr) ν_{max}/cm^{-1} 1723, 1275, 1251, 1112,

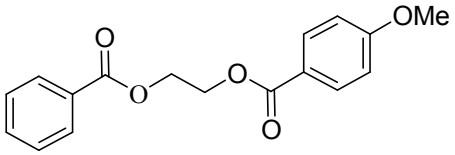
739, 711; MS (EI) m/z: 284.0, 162.0, 149.0, 119.0, 105.1, 91.0; Anal. Calcd for $C_{17}H_{16}O_4$ C 71.82, H 5.67; Found: C 71.70, H 5.80%.

2-(Benzoyloxy)ethyl 3-methoxybenzoate (**3ac**)



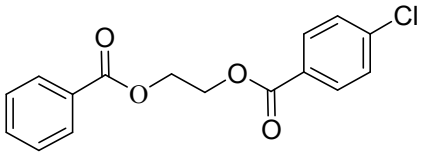
White solid, mp 49~50 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.07 (dd, *J* = 8.5, 1.4 Hz, 2 H), 7.66 (dt, *J* = 7.7, 1.3 Hz, 1 H), 7.59-7.55 (m, 2 H), 7.44 (t, *J* = 7.9 Hz, 2 H), 7.35 (t, *J* = 7.9 Hz, 1 H), 7.11 (ddd, *J* = 8.3, 2.7, 0.9 Hz, 1 H), 4.67 (s, 4 H), 3.83 (s, 3 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 166.43, 166.31, 159.55, 133.23, 131.08, 129.79, 129.75 (2 C), 129.51, 128.47 (2 C), 122.15, 119.80, 114.03, 62.88, 62.72, 55.46; IR (KBr) ν_{max}/cm^{-1} 1721, 1269, 1099, 755, 712; MS (EI) m/z: 300.0, 179.0, 149.0, 135.1, 105.1, 77.1; Anal. Calcd for $C_{17}H_{16}O_5$ C 67.99, H 5.37; Found: C 67.90, H 5.48%.

2-(Benzoyloxy)ethyl 4-methoxybenzoate (**3ad**)



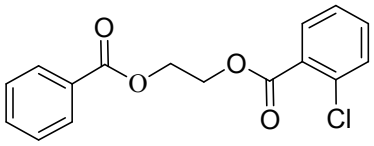
White solid, mp 71~73 °C (lit.[2] 96 °C); ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.06 (dd, *J* = 8.8, 1.6 Hz, 2 H), 8.01 (d, *J* = 8.8 Hz, 2 H), 7.56 (t, *J* = 7.6 Hz, 1 H), 7.44 (t, *J* = 8.0 Hz, 2 H), 6.91 (d, *J* = 8.8 Hz, 2 H), 4.67-4.62 (m, 4 H), 3.85 (s, 3 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 166.42, 166.13, 163.54, 133.15, 131.79 (2 C), 129.88, 129.73 (2 C), 128.43 (2 C), 122.23, 113.69 (2 C), 62.87, 62.48, 55.46; IR (KBr) ν_{max}/cm^{-1} 1733, 1716, 1254, 1106, 846, 770, 714; MS (EI) m/z: 300.0, 179.0, 149.0, 135.1, 105.1, 77.0; Anal. Calcd for $C_{17}H_{16}O_5$ C 67.99, H 5.37; Found: C 67.82, H 5.47%.

2-(Benzoyloxy)ethyl 4-chlorobenzoate (**3ae & 3cl**)



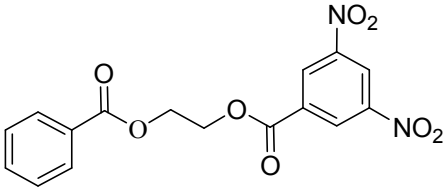
White solid, mp 66~68 °C (lit.[2] 88 °C); ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.06 (dd, *J* = 8.4, 1.2 Hz, 2 H), 7.99 (dt, *J* = 8.4, 1.6 Hz, 2 H), 7.60-7.56 (m,1 H), 7.47-7.41 (m, 4 H), 4.67 (s, 4 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 166.41, 165.58, 139.68, 133.28, 131.15 (2 C), 129.73 (2 C), 128.83 (2 C), 128.49 (2 C), 128.22, 63.04, 62.66; IR (KBr) ν_{max}/cm^{-1} 1718, 1266, 1120, 851, 758, 710; MS (EI) m/z: 304.0, 185.0, 182.9, 149.0, 141.0, 138.9, 105.1; Anal. Calcd for $C_{16}H_{13}ClO_4$ C 63.06, H 4.30; Found: C 63.18, H 4.44%.

2-(Benzoyloxy)ethyl 2-chlorobenzoate (**3af & 3am**)



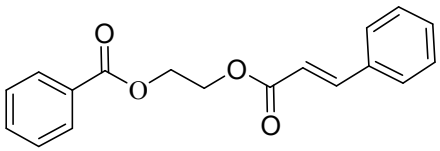
Colorless liquid; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.07 (d, *J* = 8.0 Hz, 2 H), 7.85 (d, *J* = 7.6 Hz, 1 H), 7.57 (t, *J* = 7.6 Hz, 1 H), 7.45 (t, *J* = 8.0 Hz, 4 H), 7.32 (t, *J* = 7.2 Hz, 1 H), 4.69-4.68 (m, 4 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 166.39, 165.51, 133.90, 133.22, 132.80, 131.56, 131.16, 129.77 (2 C), 129.73, 128.46 (2 C), 126.66, 63.18, 62.63; IR (KBr) ν_{max}/cm^{-1} 1720, 1247, 1109, 749, 711; MS (EI) m/z: 304.0, 185.0, 183.0, 149.0, 141.0, 138.9, 105.1; Anal. Calcd for $C_{16}H_{13}ClO_4$ C 63.06, H 4.30; Found: C 63.19, H 4.42%.

2-(Benzoyloxy)ethyl 3,5-dinitrobenzoate (**3ag**)



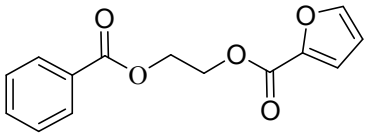
White solid, mp 122~124 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 9.23 (t, *J* = 1.6 Hz, 1 H), 9.17 (d, *J* = 1.6 Hz, 2 H), 8.06 (d, *J* = 7.6 Hz, 2 H), 7.58 (t, *J* = 7.2 Hz, 1H), 7.46 (t, *J* = 7.6 Hz, 2 H), 4.82-4.79 (m, 2 H), 4.75-4.72 (m, 2 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 166.34, 162.41, 148.72 (2 C), 133.50, 133.45, 129.73 (2 C), 129.59 (2 C), 129.48, 128.56 (2 C), 122.60, 64.65, 62.16; IR (KBr) ν_{max}/cm^{-1} 1739, 1717, 1541, 1286, 1260, 1170, 731, 716; MS (EI) m/z: 360.0, 239.0, 195.0, 166.0, 149.0, 105.1; Anal. Calcd for $C_{16}H_{12}N_2O_8$ C 53.34, H 3.36, N 7.78; Found: C 53.22, H 3.27, N 7.69%.

(E)-2-(cinnamoyloxy)ethyl benzoate (**3ah**)



White solid, mp 56~58 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.07 (dd, *J* = 8.0, 1.2 Hz, 2 H), 7.73 (d, *J* = 16.0 Hz, 1 H), 7.59-7.52 (m, 3 H), 7.45 (t, *J* = 8.0 Hz, 2 H), 7.40-7.38 (m, 3 H), 6.48 (d, *J* = 16.0 Hz, 1 H), 4.62-4.60 (m, 2 H), 4.57-4.55 (m, 2 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 166.76, 166.42, 145.50, 134.29, 133.10, 130.47, 129.85, 129.76 (2 C), 128.93 (2 C), 128.44 (2 C), 128.19 (2 C), 117.55, 62.85, 62.33; IR (KBr) ν_{max}/cm^{-1} 1711, 1450, 1270, 1128, 765, 712; MS (EI) m/z: 296.0, 149.0, 131.0, 105.0, 77.0; Anal. Calcd for $C_{18}H_{16}O_4$ C 72.96, H 5.44; Found: C 73.06, H 5.53%.

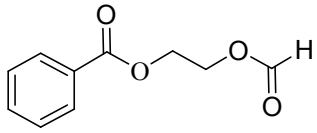
2-(Benzoyloxy)ethyl furan-2-carboxylate (**3ai**)



White solid, mp 65~66 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.06 (d, *J* = 7.6 Hz, 2 H), 7.59 (s, 1 H),

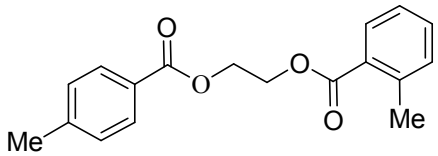
7.57 (t, J = 8.0 Hz, 1 H), 7.44 (t, J = 8.0 Hz, 2 H), 7.21 (d, J = 3.6 Hz, 1 H), 6.51 (dd, J = 3.2, 1.6 Hz, 1 H), 4.67-4.62 (m, 4 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 166.36, 158.44, 148.64, 144.26, 133.19, 129.77, 129.74 (2 C), 128.43 (2 C), 118.48, 111.94, 62.69, 62.58; IR (KBr) ν_{max}/cm^{-1} 1709, 1477, 1318, 1279, 1116, 947, 714; MS (EI) m/z: 260.0, 149.0, 139.0, 105.0, 95.0, 77.0; Anal. Calcd for $C_{14}H_{12}O_5$ C 64.61, H 4.65; Found: C 64.70, H 4.78%.

2-(Formyloxy)ethyl benzoate (**3aj**)



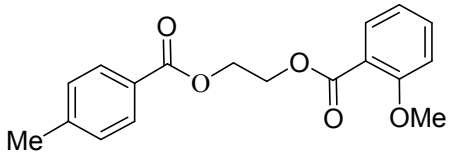
Colorless liquid; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.11 (s, 1 H), 8.05 (d, J = 7.6 Hz, 2 H), 7.58 (t, J = 7.6 Hz, 1 H), 7.45 (t, J = 7.6 Hz, 2 H), 4.57-4.51 (m, 4 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 166.29, 160.68, 133.27, 129.73 (2 C), 129.64, 128.46 (2 C), 62.42, 61.63; IR (KBr) ν_{max}/cm^{-1} 3431, 1719, 1276, 1114, 712; MS (EI) m/z:194.0, 148.0, 123.0, 105.0, 77.0; Anal. Calcd for $C_{10}H_{10}O_4$ C 61.85, H 5.19; Found: C 61.97, H 5.30%.

2-((4-Methylbenzoyl)oxy)ethyl 2-methylbenzoate (**3bb**)



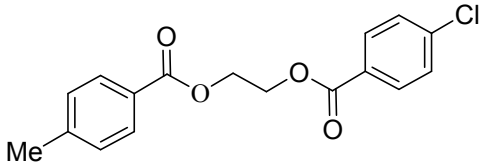
White solid, mp 55~56 ^{o}C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.96-7.93 (m, 3 H), 7.40 (t, J = 7.6 Hz, 1 H), 7.26-7.23 (m, 4 H), 4.64 (s, 4 H), 2.60 (s, 3 H), 2.40 (s, 3 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 167.40, 166.48, 143.93, 140.35, 132.23, 131.75, 130.80, 129.77 (2 C), 129.24, 129.18 (2 C), 127.07, 125.81, 62.67, 62.56, 21.87, 21.73; IR (KBr) ν_{max}/cm^{-1} 1724, 1283, 1254, 1086, 751, 738; MS (EI) m/z: 298.0, 163.0, 119.0, 91.0; Anal. Calcd for $C_{18}H_{18}O_4$ C 72.47, H 6.08; Found: C 72.56, H 6.20%.

2-((4-Methylbenzoyl)oxy)ethyl 2-methoxybenzoate (**3bk**)



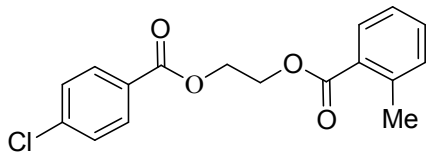
Yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.96 (d, J = 8.4 Hz, 2 H), 7.81 (dd, J = 8.0, 1.6 Hz, 1 H), 7.47 (td, J = 8.0, 1.6 Hz, 1 H), 7.28-7.23 (m, 2 H), 6.99-6.95 (m, 2 H), 4.632 (s, 4 H), 3.86 (s, 3 H), 2.40 (s, 3 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 166.51, 165.93, 159.36, 143.86, 133.80, 131.76, 129.79 (2 C), 129.14 (2 C), 127.14, 120.15, 119.62, 112.03, 62.66, 62.55, 55.95, 21.73; IR (KBr) ν_{max}/cm^{-1} 1716, 1277, 1251, 1112, 1084, 754; MS (EI) m/z: 314.0, 179.0, 163.0, 135.1, 119.0, 91.0. Anal. Calcd for $C_{18}H_{18}O_5$ C 68.78, H 5.77; Found: C 68.89, H 5.88%.

2-((4-Chlorobenzoyl)oxy)ethyl 4-methylbenzoate (**3be**)



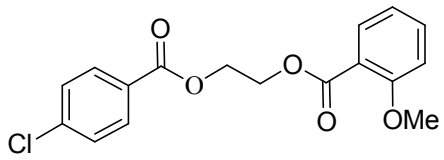
White solid, mp 134~135 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.98 (d, *J* = 8.4 Hz, 2 H), 7.94 (d, *J* = 8.0 Hz, 2 H), 7.41 (d, *J* = 8.4 Hz, 2 H), 7.24 (d, *J* = 8.0 Hz, 2 H), 4.65 (s, 4 H), 2.40 (s, 3 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 166.44, 165.55, 143.97, 139.64, 131.13 (2 C), 129.75 (2 C), 129.18 (2 C), 128.80 (2 C), 128.29, 127.03, 63.07, 62.45, 21.71; IR (KBr) ν_{max}/cm^{-1} 1716, 1269, 1091, 752; MS (EI) m/z: 318.0, 185.0, 182.9, 163.0, 141.0, 138.9, 119.0, 91.0; Anal. Calcd for $C_{17}H_{15}ClO_4$ C 64.06, H 4.74; Found: C 64.13, H 4.63%.

2-((4-Chlorobenzoyl)oxy)ethyl 2-methylbenzoate (**3cb**)



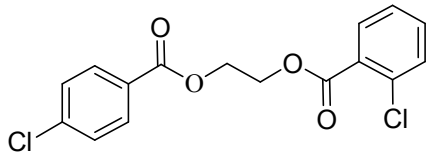
Pale yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.99 (d, *J* = 8.4 Hz, 2 H), 7.93 (d, *J* = 8.0 Hz, 1 H), 7.41 (d, *J* = 8.0 Hz, 2 H), 7.39-7.34 (m, 2 H), 7.26-7.22 (m, 1 H), 4.65 (s, 4 H), 2.59 (s, 3 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 167.30, 165.53, 140.36, 139.68, 132.28, 131.78, 131.12 (2 C), 130.74, 129.14, 128.82 (2 C), 128.26, 125.82, 63.09, 62.35, 21.83; IR (KBr) ν_{max}/cm^{-1} 1724, 1254, 1090, 854, 758, 740; MS (EI) m/z: 318.0, 185.0, 182.9, 162.0, 141.0, 138.9, 118.0, 111.0, 91.0; Anal. Calcd for $C_{17}H_{15}ClO_4$ C 64.06, H 4.74; Found: C 64.19, H 4.87%.

2-((4-Chlorobenzoyl)oxy)ethyl 2-methoxybenzoate (**3ck**)



Pale yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.00 (d, *J* = 8.8 Hz, 2 H), 7.80 (dd, *J* = 8.0, 2.0 Hz, 1 H), 7.50-7.46 (m, 1 H), 7.41 (d, *J* = 8.4 Hz, 2 H), 6.99-6.96 (m, 2 H), 4.64 (s, 4 H), 3.86 (s, 3 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 165.87, 165.56, 159.36, 139.62, 133.86, 131.71, 131.16 (2 C), 128.79 (2 C), 128.33, 120.17, 119.53, 112.05, 63.09, 62.34, 55.95; IR (KBr) ν_{max}/cm^{-1} 1725, 1274, 1247, 1091, 850, 758; MS (EI) m/z: 334.0, 185.0, 182.9, 179.0, 141.0, 138.9, 135.1, 111.0; Anal. Calcd for $C_{17}H_{15}ClO_5$ C 61.00, H 4.52; Found: C 61.12, H 4.65%.

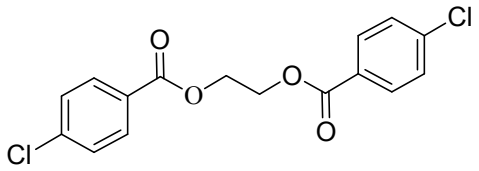
2-((4-Chlorobenzoyl)oxy)ethyl 2-chlorobenzoate (**3cf & 3cm**)



Pale pink solid, mp 53~54 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.05-7.99 (m, 2 H), 7.88-7.83 (m, 1 H), 7.46-7.40 (m, 4 H), 7.35-7.29 (m, 1H) , 4.70-

4.65 (m, 4 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 165.52, 165.49, 139.70, 133.86, 132.83, 131.52, 131.17 (2 C), 129.70, 128.85, 128.82 (2 C), 128.20, 126.68, 63.04, 62.87; IR (KBr) ν_{max}/cm^{-1} 1735, 1718, 1290, 1249, 1109, 758, 749; MS (EI) m/z: 337.9, 183.0, 141.0, 138.9, 111.0, 75.0; Anal. Calcd for $C_{16}H_{12}Cl_2O_4$ C 56.66, H 3.57; Found: C 56.58, H 3.67%.

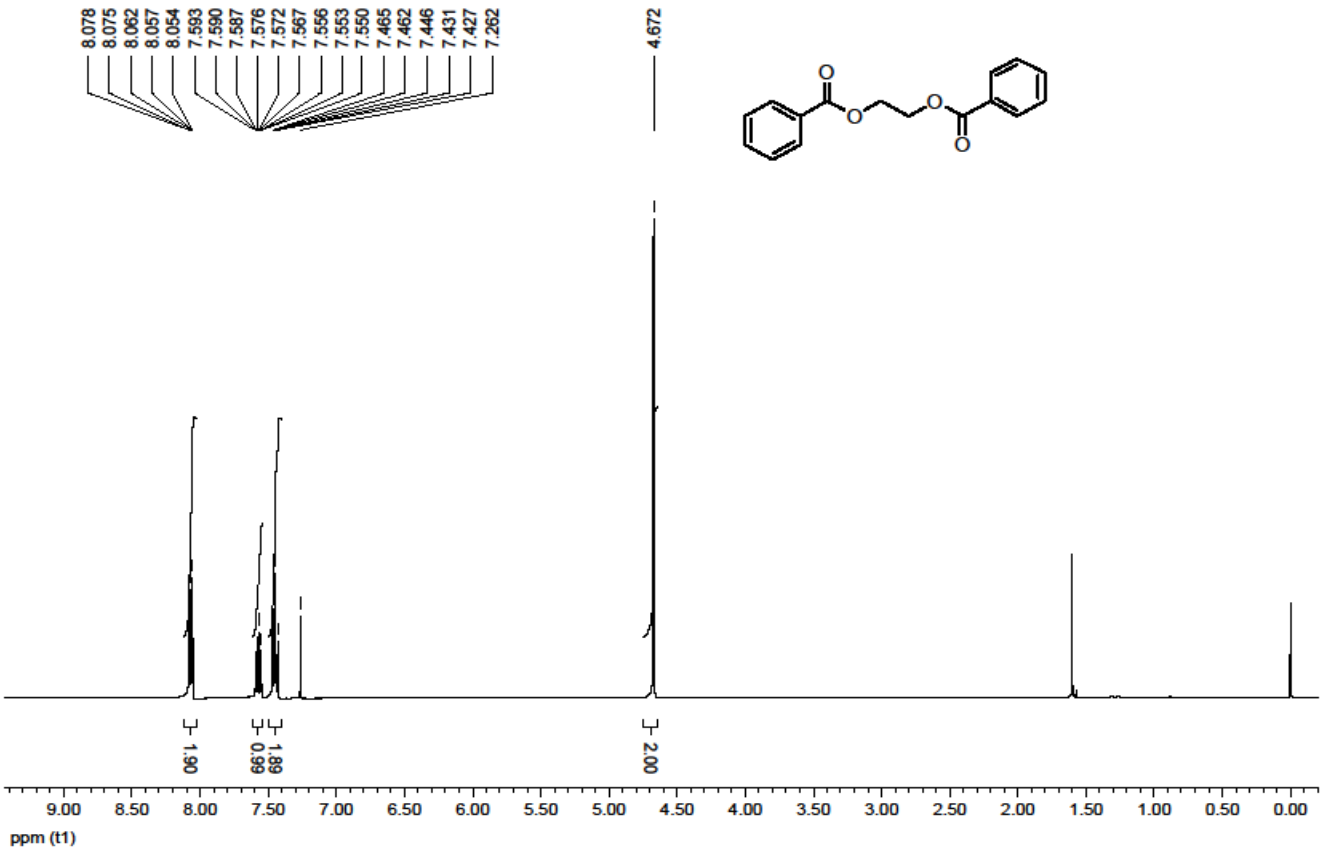
Ethane-1,2-diyl bis(4-chlorobenzoate) (**3ce**)

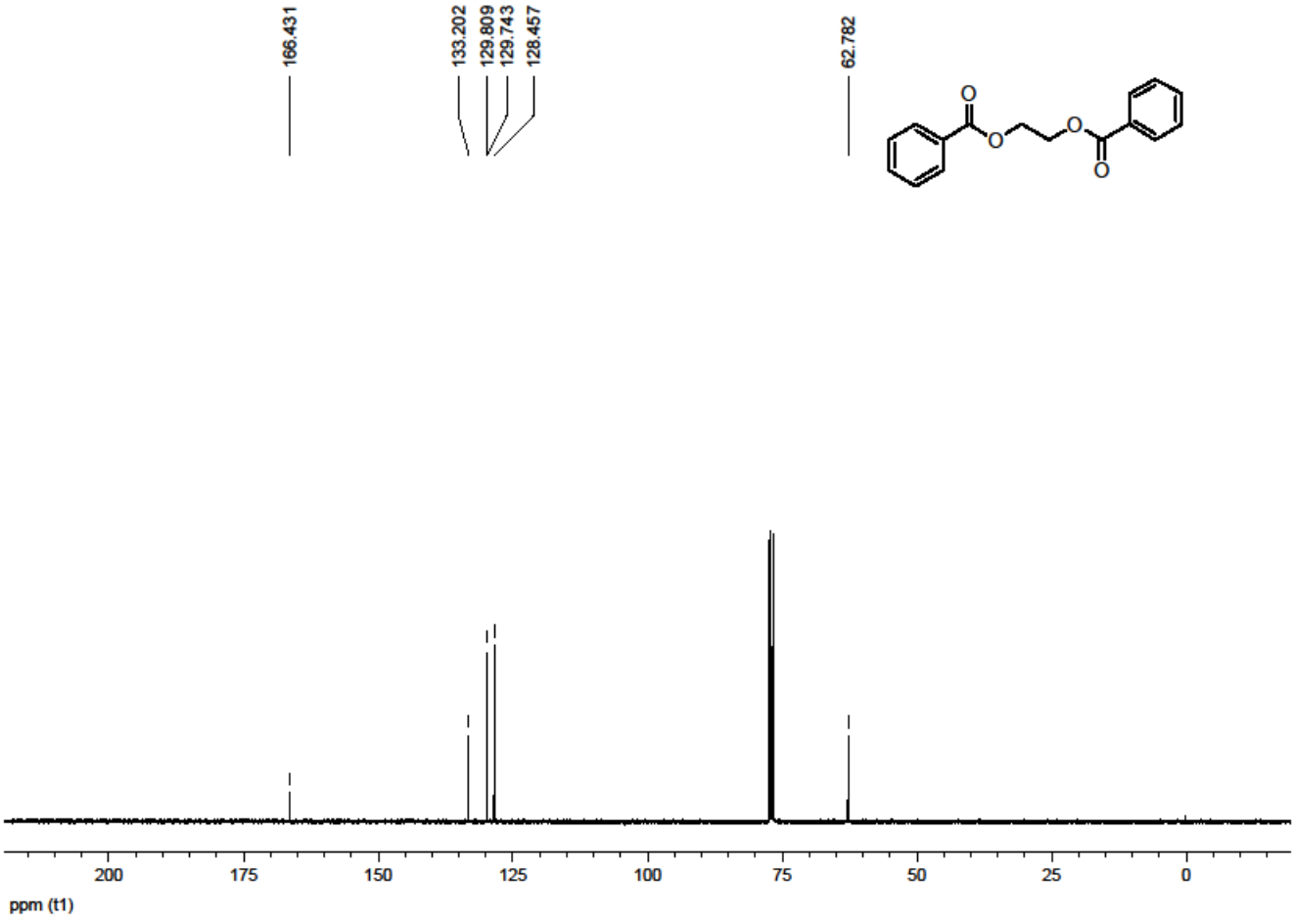


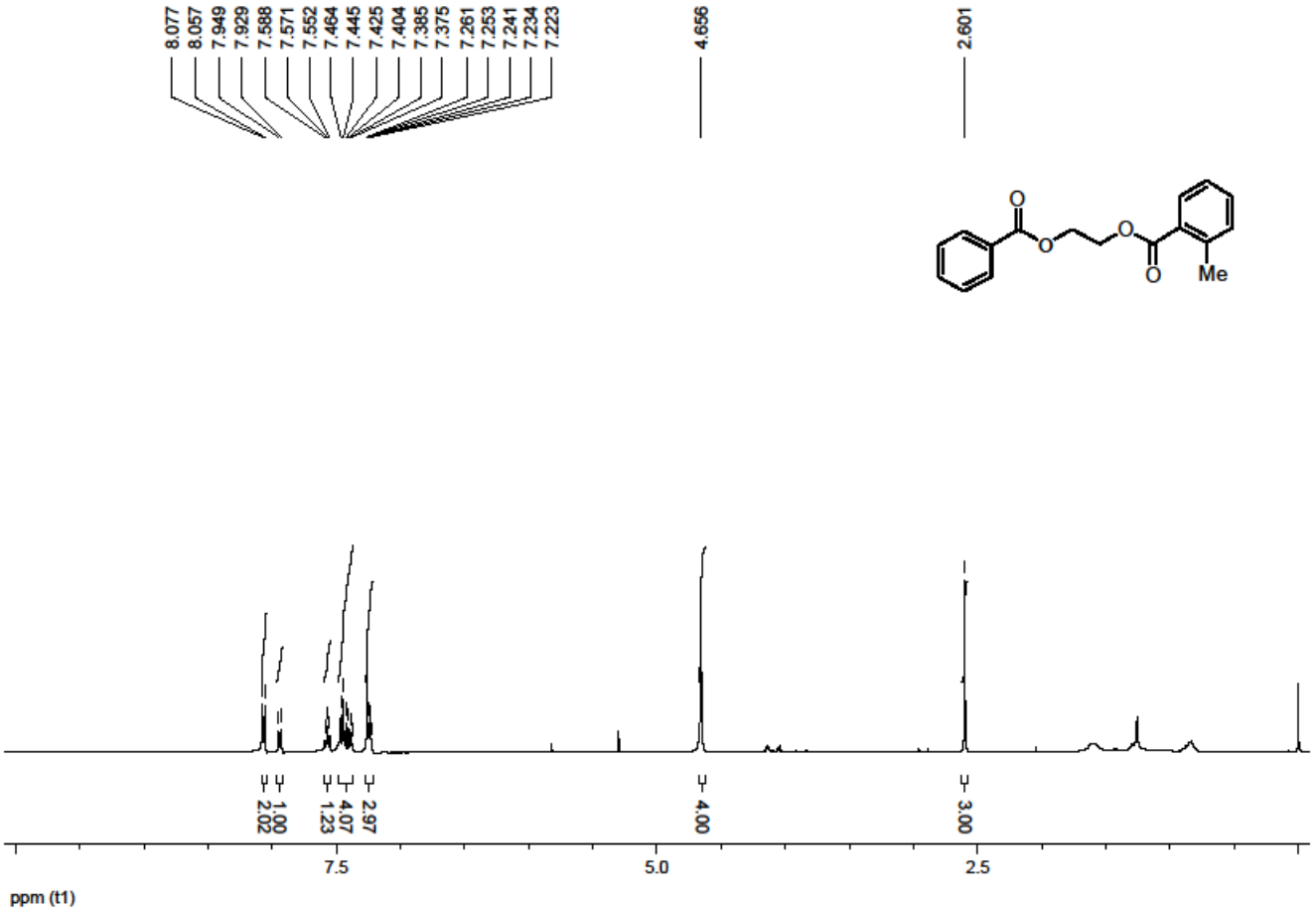
White solid, mp 152~153 °C (lit.[1] 140 °C); ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.98 (d, *J* = 8.8 Hz, 4 H), 7.42 (d, *J* = 8.4 Hz, 4 H), 4.657 (s, 4 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 165.52 (2 C), 139.74 (2 C), 131.11 (4 C), 128.84 (4 C), 128.19 (2 C), 62.88 (2 C); IR (KBr) ν_{max}/cm^{-1} 1717, 1270, 1113, 860, 760; MS (EI) m/z: 337.9, 185.0, 182.9, 141.0, 138.9, 111.0; Anal. Calcd for $C_{16}H_{12}Cl_2O_4$ C 56.66, H 3.57; Found: C 56.54, H 3.68%.

[1] Heim, Harold C.; J. Org. Chem., 1944, 9, 299-301.
[2] Safiev, O. G.; Doklady Akademii Nauk SSSR, 1990, 310(4), 889-893

2) PDF files of copies of ^{1}H NMR and ^{13}C NMR spectra for all coupled compounds



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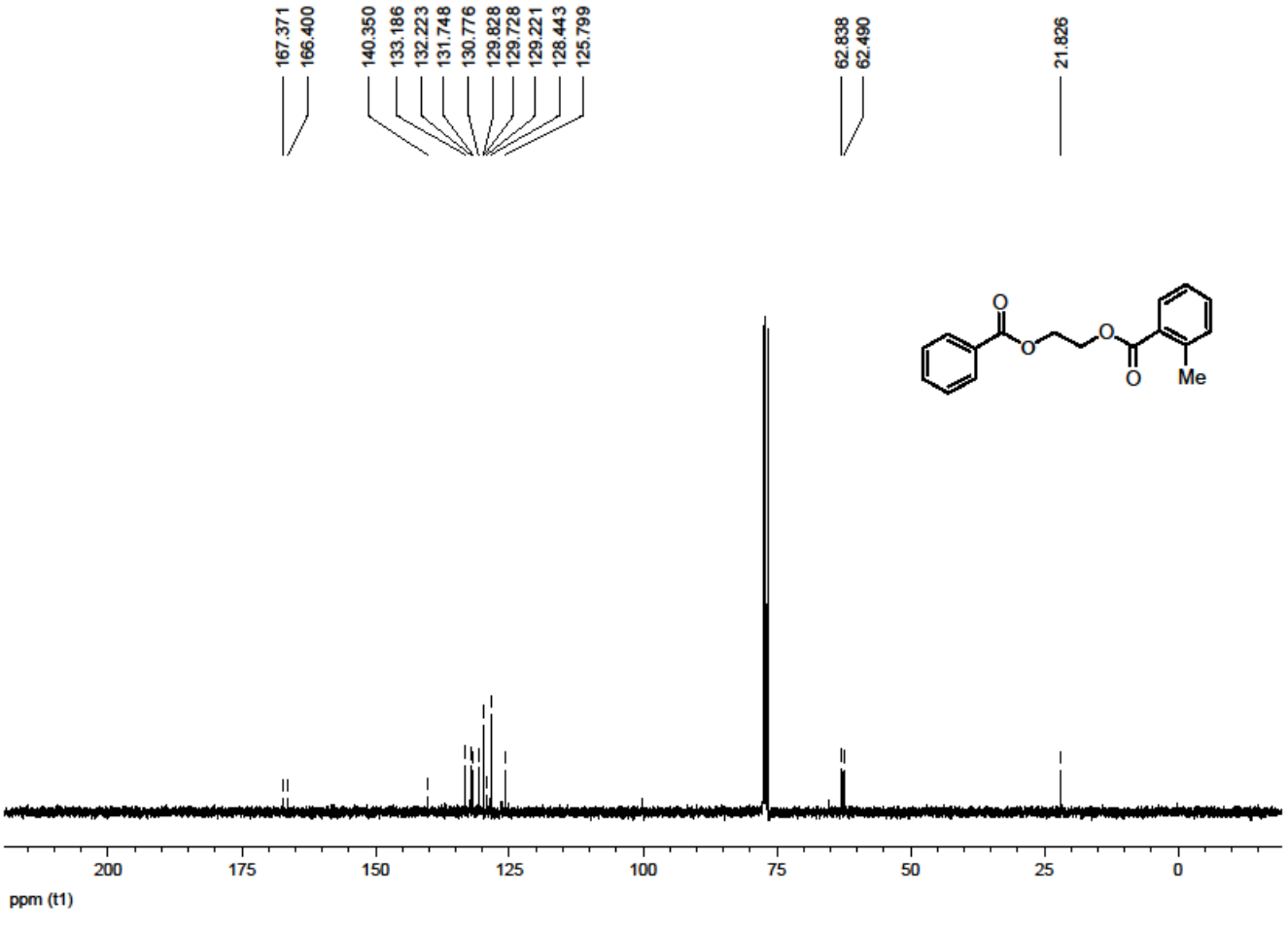
7.929
7.588
7.571
7.552
7.464
7.445
7.425
7.404
7.385
7.375
7.261
7.253
7.241
7.234
7.223
4.656
2.601
O
O
O
O
Me
2.02
1.00
1.23
4.07
2.97
4.00
3.00
7.5
5.0
2.5
ppm (t1)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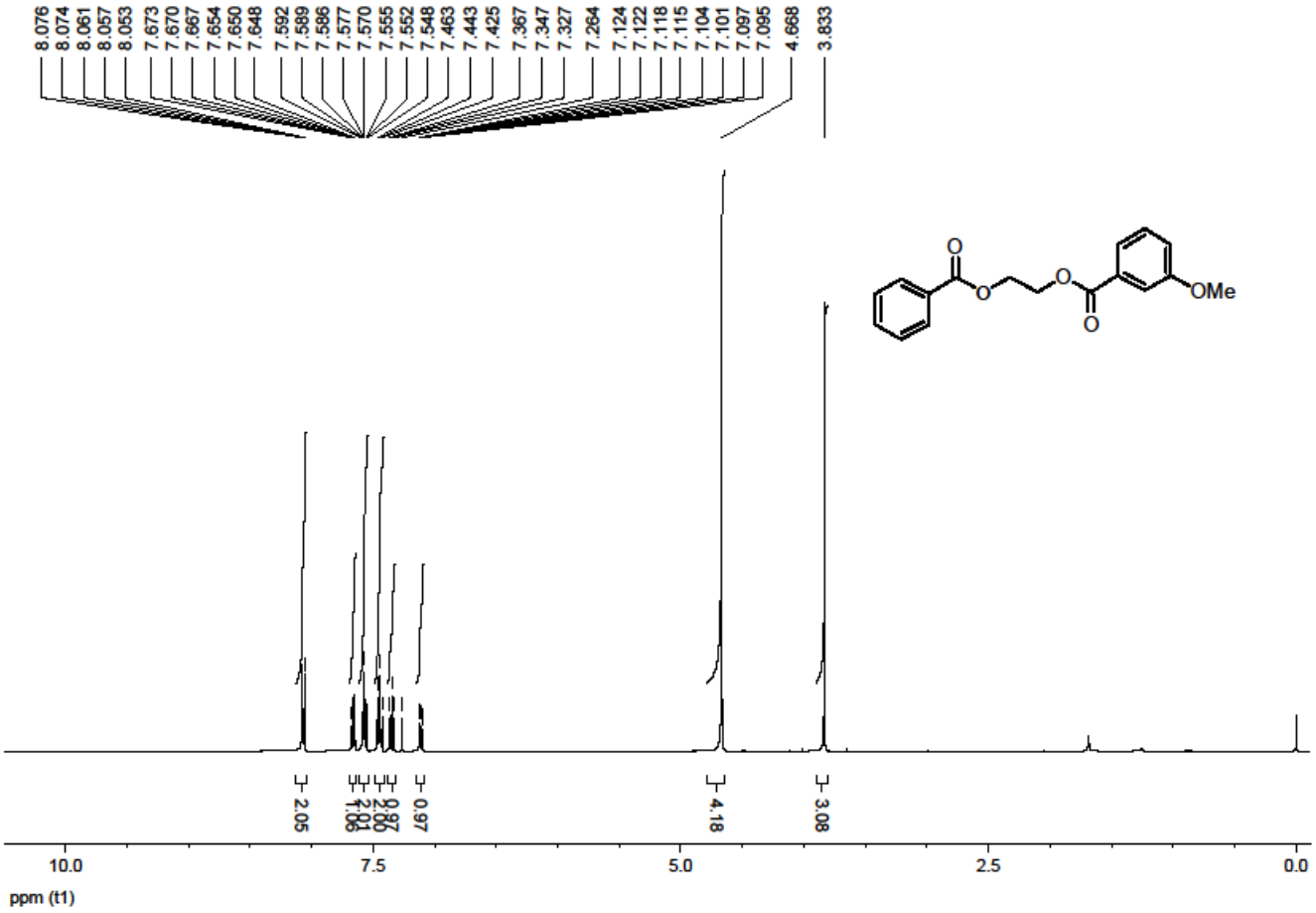
140.350
133.186
132.223
131.748
130.776
129.828
129.728
129.221
128.443
125.799
62.838
62.490
21.826
O
O
O
O
Me
200
175
150
125
100
75
50
25
0
ppm (t1)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7.670
7.667
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7.650
7.648
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7.589
7.586
7.577
7.570
7.555
7.552
7.548
7.463
7.443
7.425
7.367
7.347
7.327
7.264
7.124
7.122
7.118
7.115
7.104
7.101
7.097
7.095
4.668
3.833
OMe
2.05
1.06
1.01
2.00
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0.97
4.18
3.08
10.0
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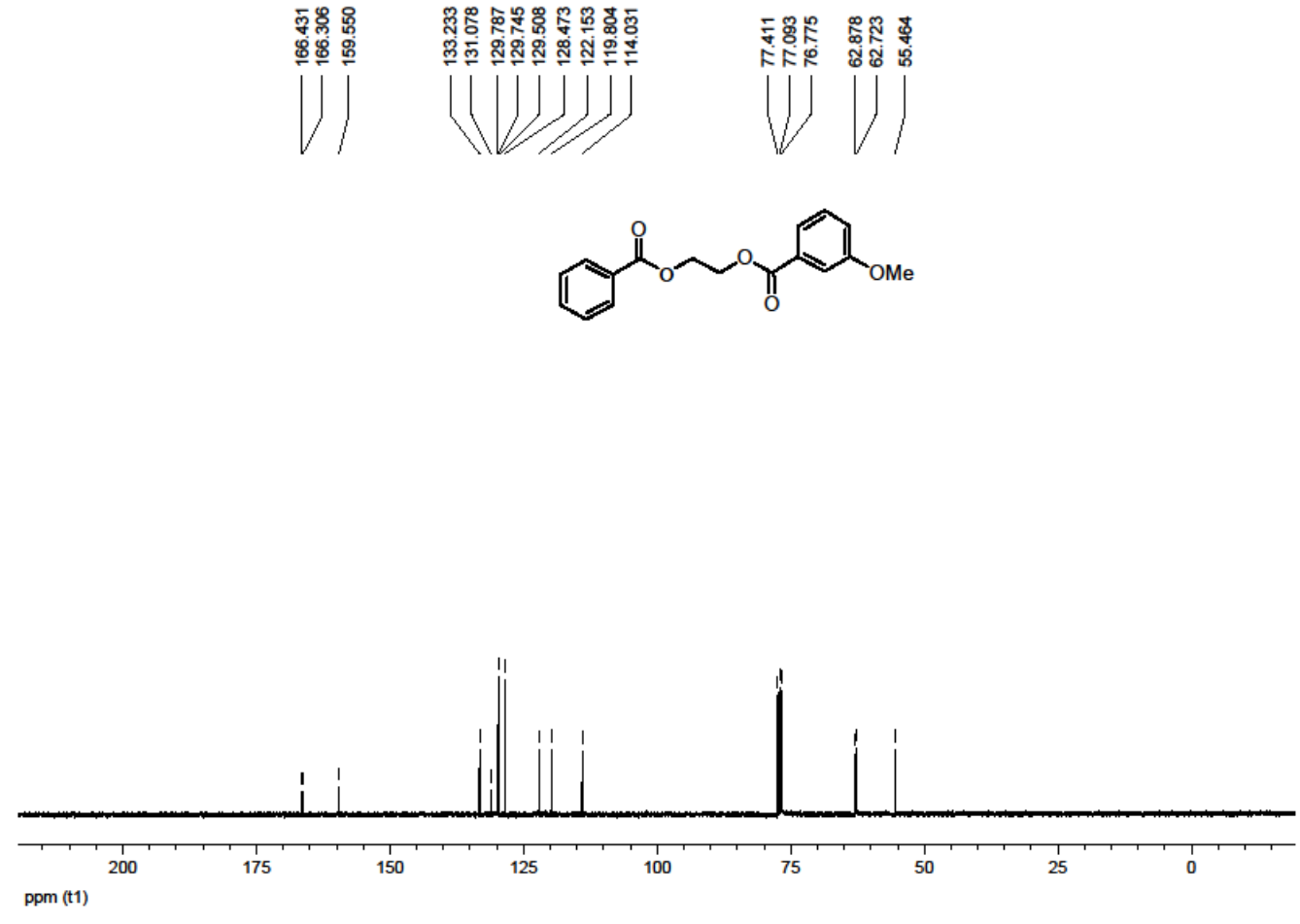
ppm (t1)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159.550
133.233
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129.745
129.508
128.473
122.153
119.804
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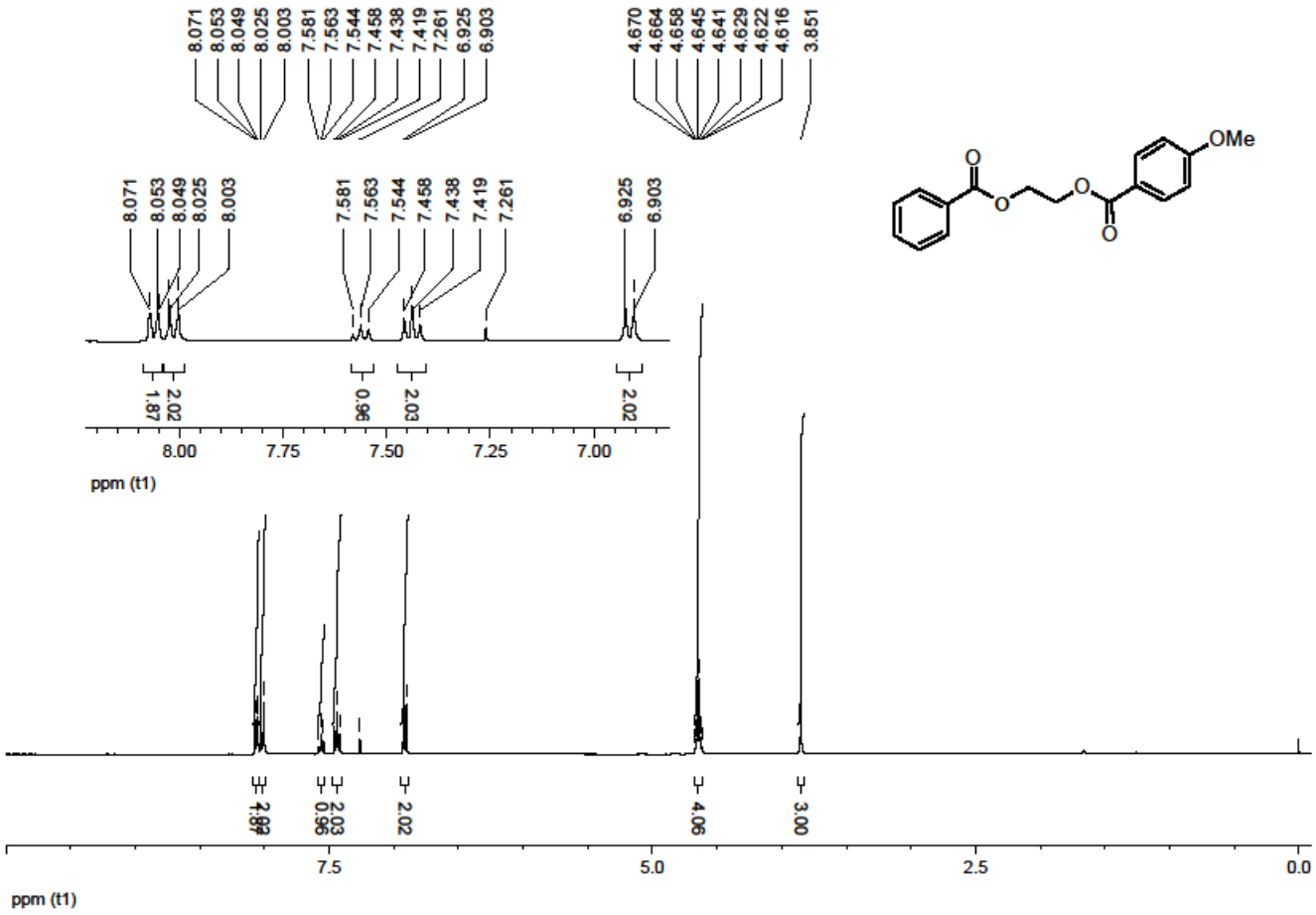
77.411
77.093
76.775
62.878
62.723
55.464
OMe
200
175
150
125
100
75
50
25
0
ppm (t1)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8.049
8.025
8.003
7.581
7.563
7.544
7.458
7.438
7.419
7.261
6.925
6.903
4.670
4.664
4.658
4.645
4.641
4.629
4.622
4.616
3.851
OMe
1.87
2.02
0.96
2.03
2.02
4.06
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7.25
7.00
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5.0
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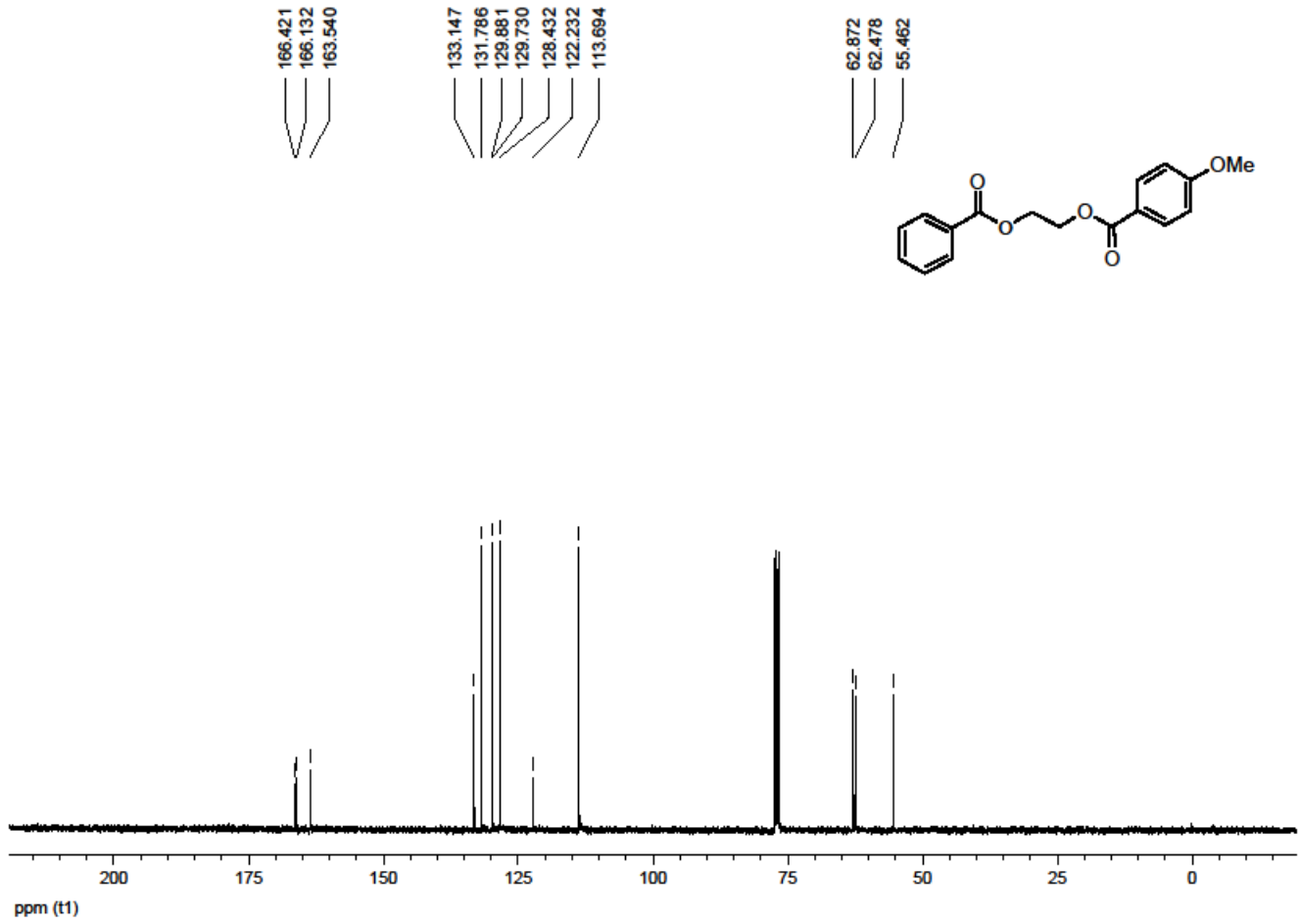
ppm (t1)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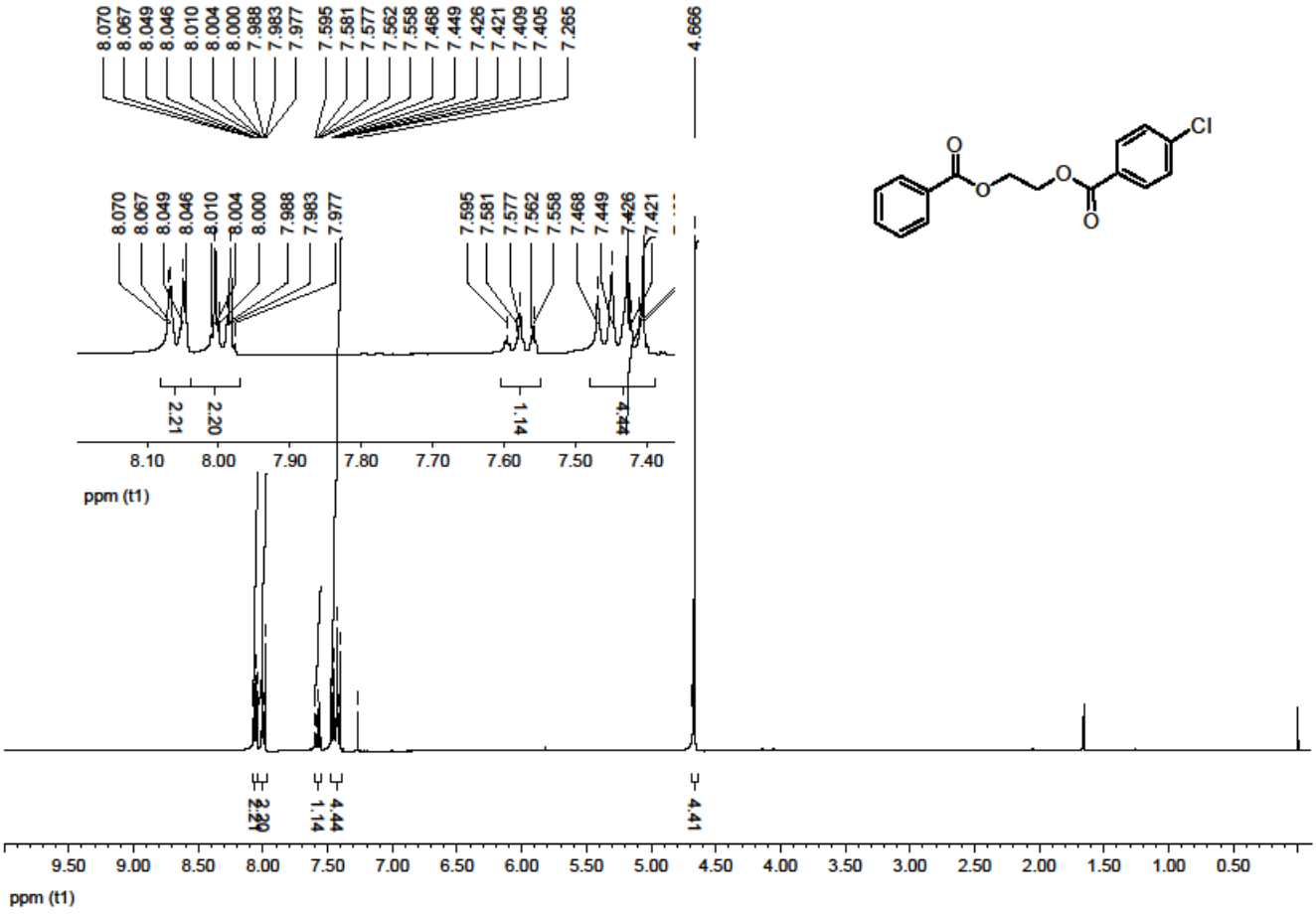
163.540
133.147
131.786
129.881
129.730
128.432
122.232
113.694
62.872
62.478
55.462
OMe
200
175
150
125
100
75
50
25
0
ppm (t1)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8.067
8.049
8.046
8.010
8.004
8.000
7.988
7.983
7.977
7.595
7.581
7.577
7.562
7.558
7.468
7.449
7.426
7.421
7.409
7.405
7.265
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Cl
O
O
O
O
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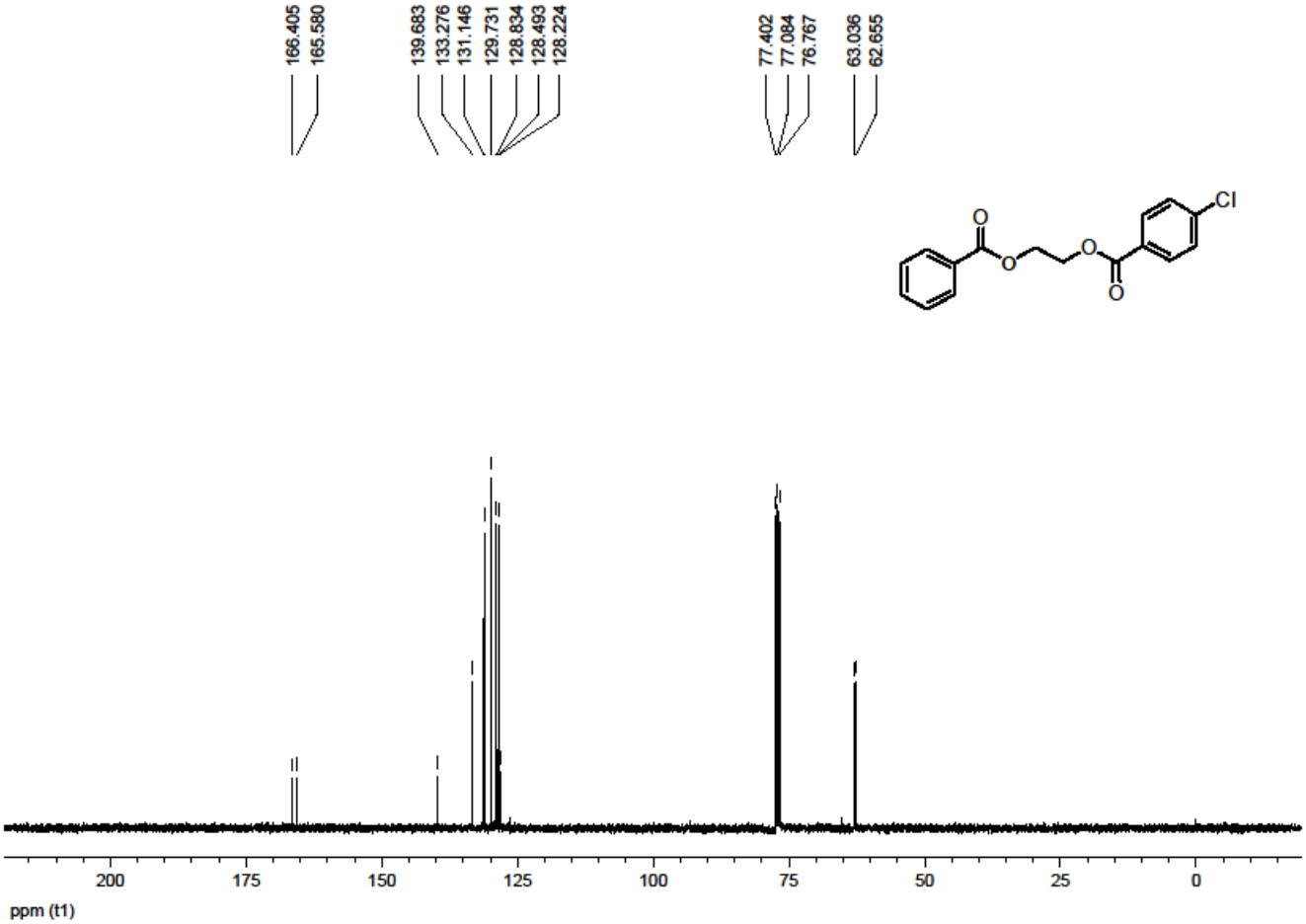
2.20
1.14
4.44
4.41
ppm (t1)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139.683
133.276
131.146
129.731
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128.493
128.224
77.402
77.084
76.767
63.036
62.655
Cl
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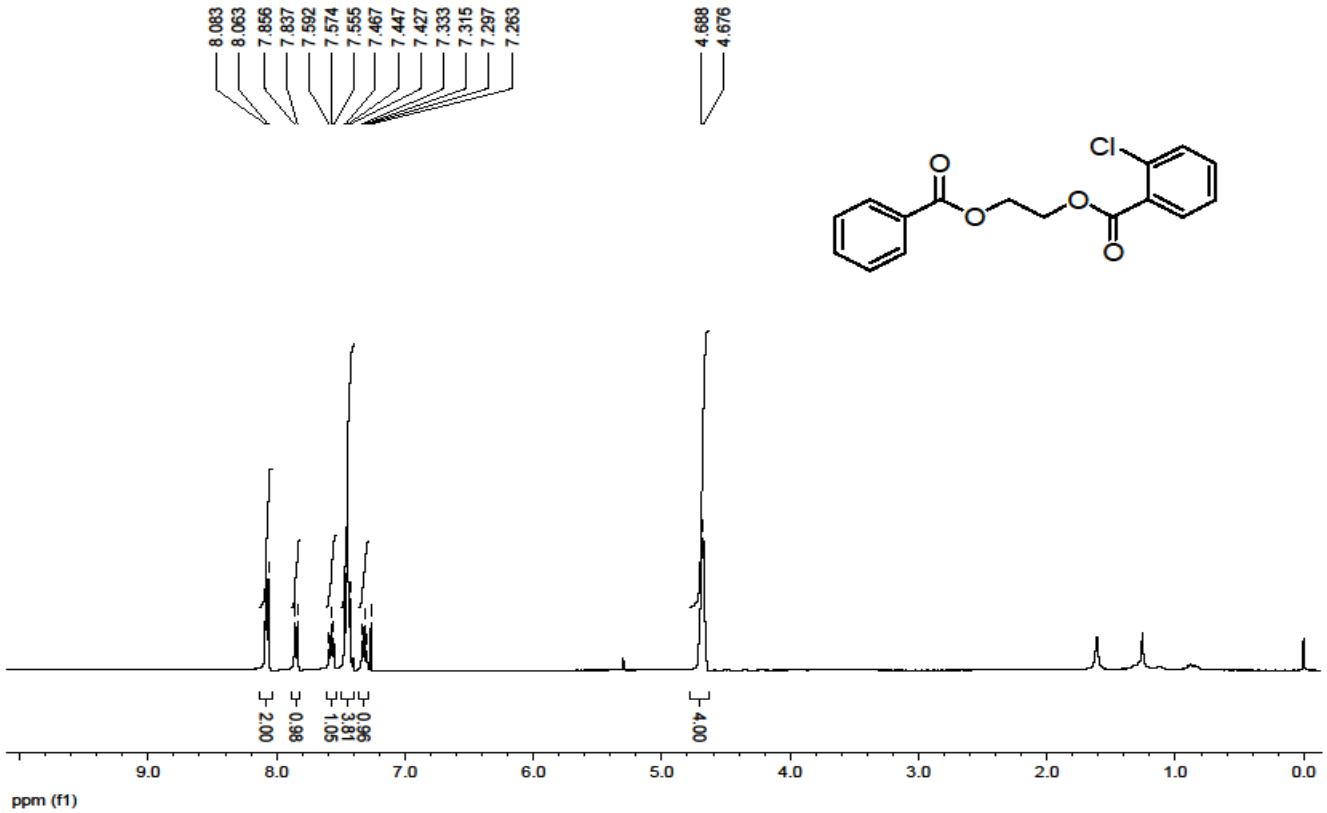
O
O
O
ppm (t1)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7.837
7.592
7.574
7.555
7.467
7.447
7.427
7.333
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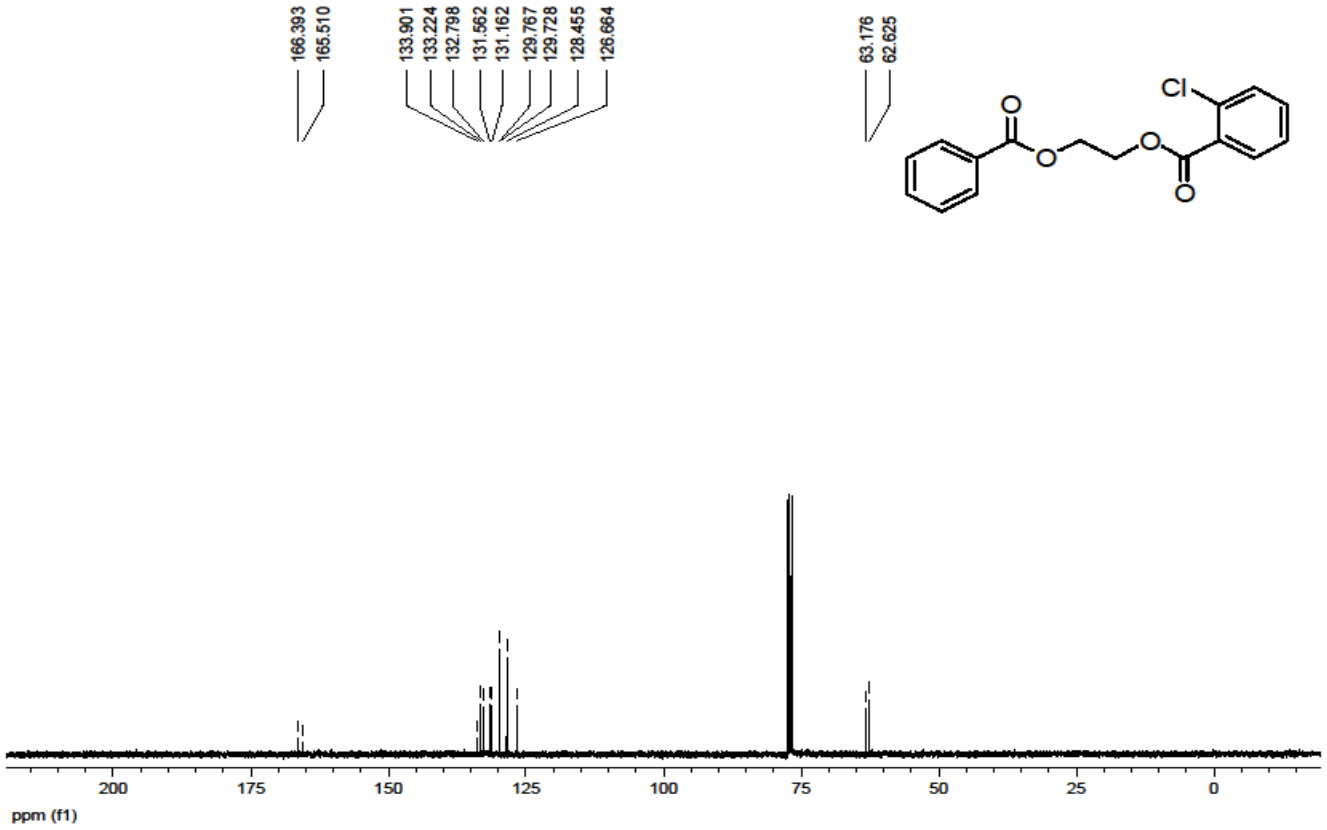
7.297
7.263
4.688
4.676
Cl
O
O
O
O
2.00
0.98
1.06
3.81
0.96
0.90
4.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (f1)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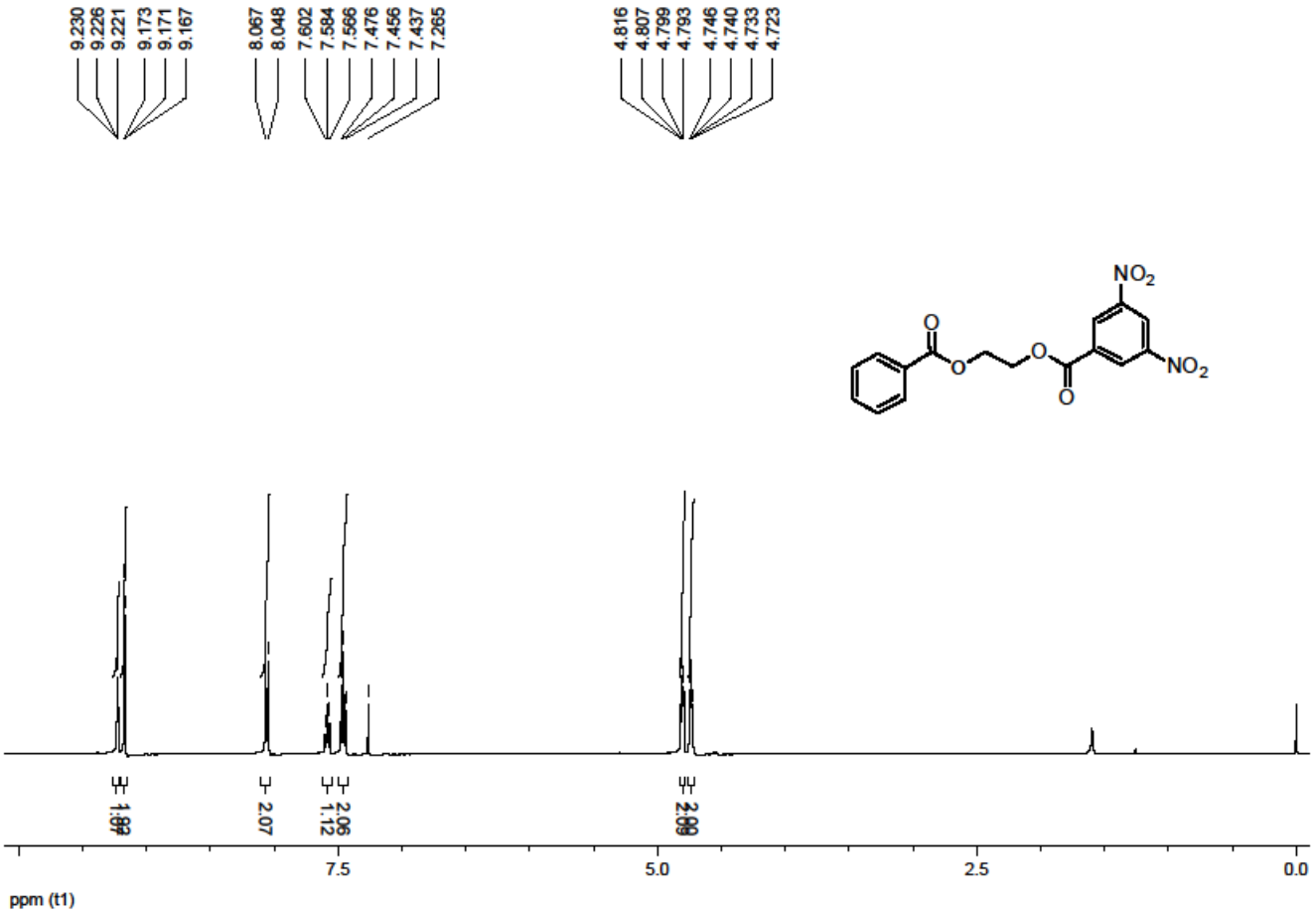
133.901
133.224
132.798
131.562
131.162
129.767
129.728
128.455
126.664
63.176
62.625
Cl
O
O
O
O
200
175
150
125
100
75
50
25
0
ppm (f1)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9.221
9.173
9.171
9.167
8.067
8.048
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7.584
7.566
7.476
7.456
7.437
7.265
4.816
4.807
4.799
4.793
4.746
4.740
4.733
4.723
NO2
NO2
O
O
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2.06
2.00
2.00
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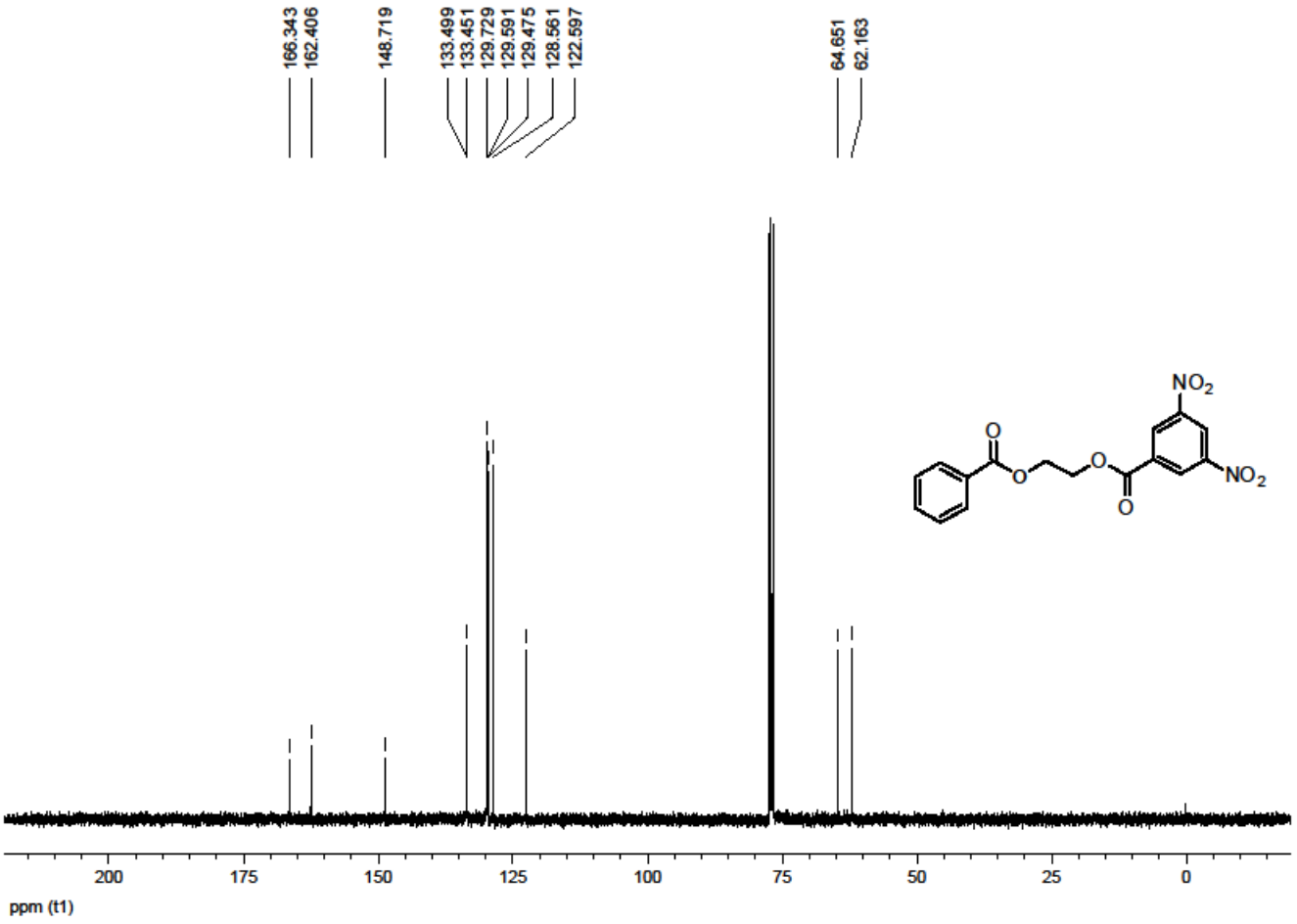
ppm (t1)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133.451
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129.591
129.475
128.561
122.597
64.651
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NO2
O
O
O
O
200
175
150
125
100
75
50
25
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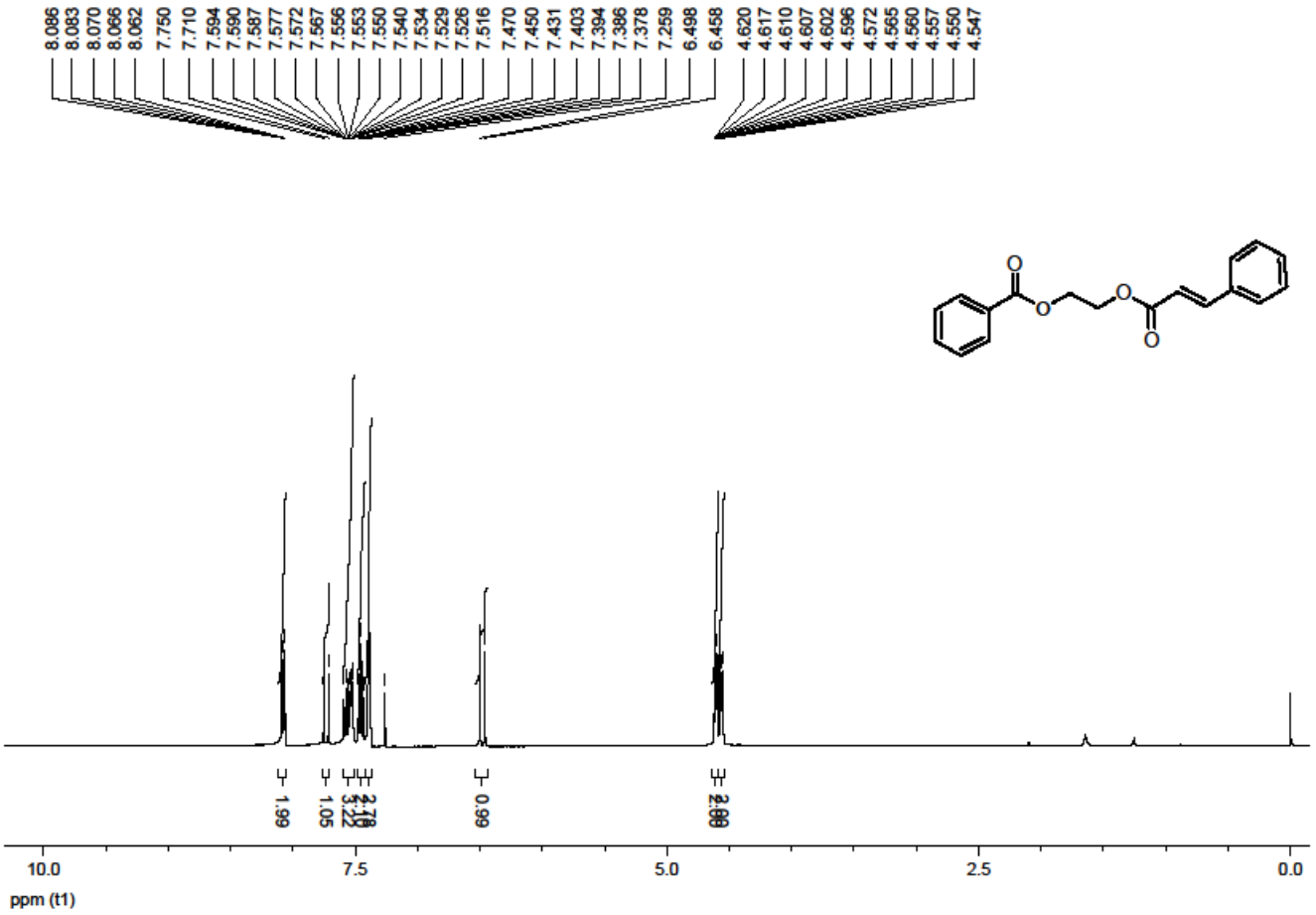
ppm (t1)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8.083
8.070
8.066
8.062
7.750
7.710
7.594
7.590
7.587
7.577
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7.567
7.556
7.553
7.550
7.540
7.534
7.529
7.526
7.516
7.470
7.450
7.431
7.403
7.394
7.386
7.378
7.259
6.498
6.458
4.620
4.617
4.610
4.607
4.602
4.596
4.572
4.565
4.560
4.557
4.550
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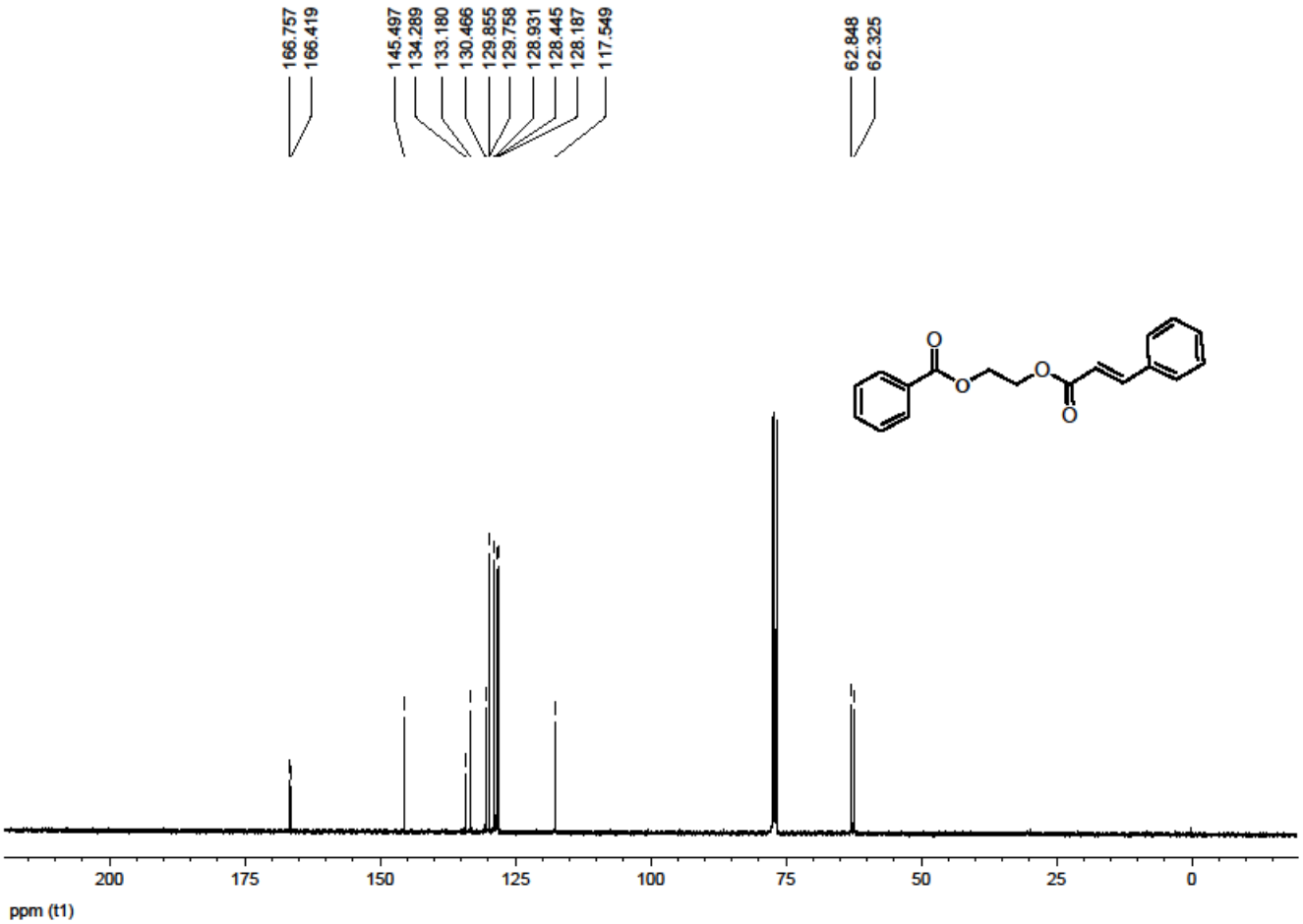
ppm (t1)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130.466
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129.758
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62.848
62.325
200
175
150
125
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75
50
25
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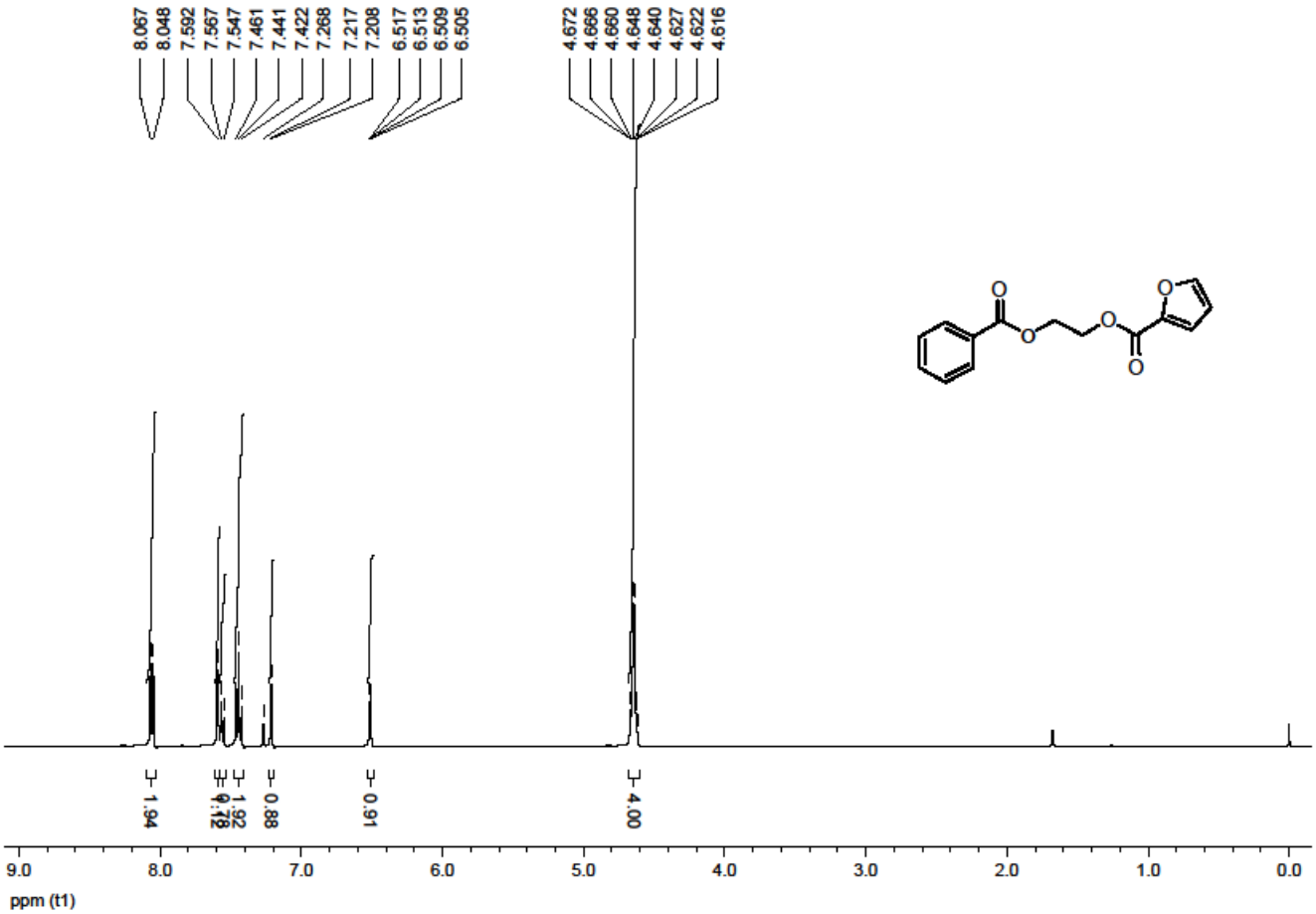
ppm (t1)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7.547
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7.422
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7.217
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6.513
6.509
6.505
4.672
4.666
4.660
4.648
4.640
4.627
4.622
4.616
1.94
1.92
0.88
0.91
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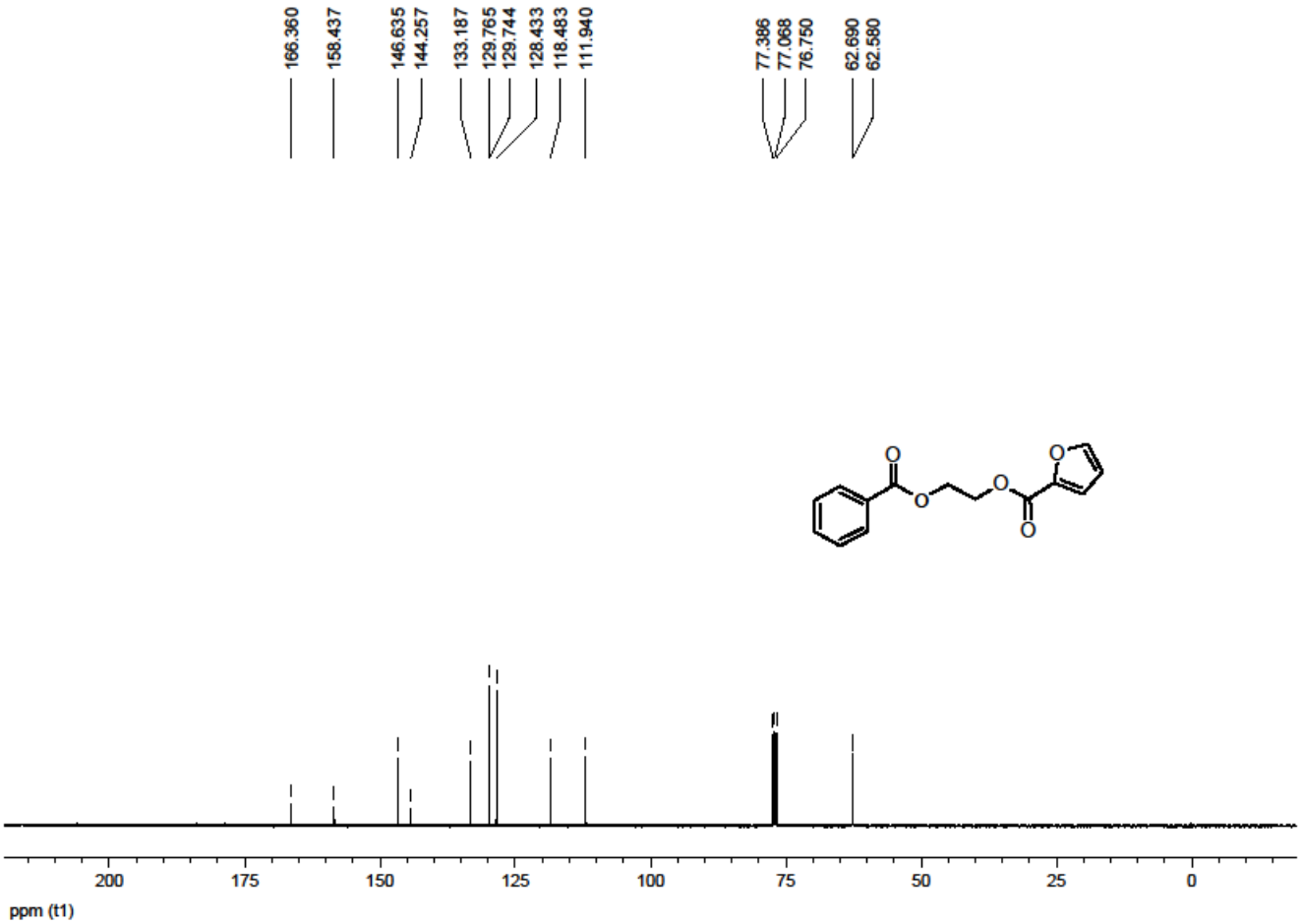
ppm (t1)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144.257
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129.744
128.433
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200
175
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125
100
75
50
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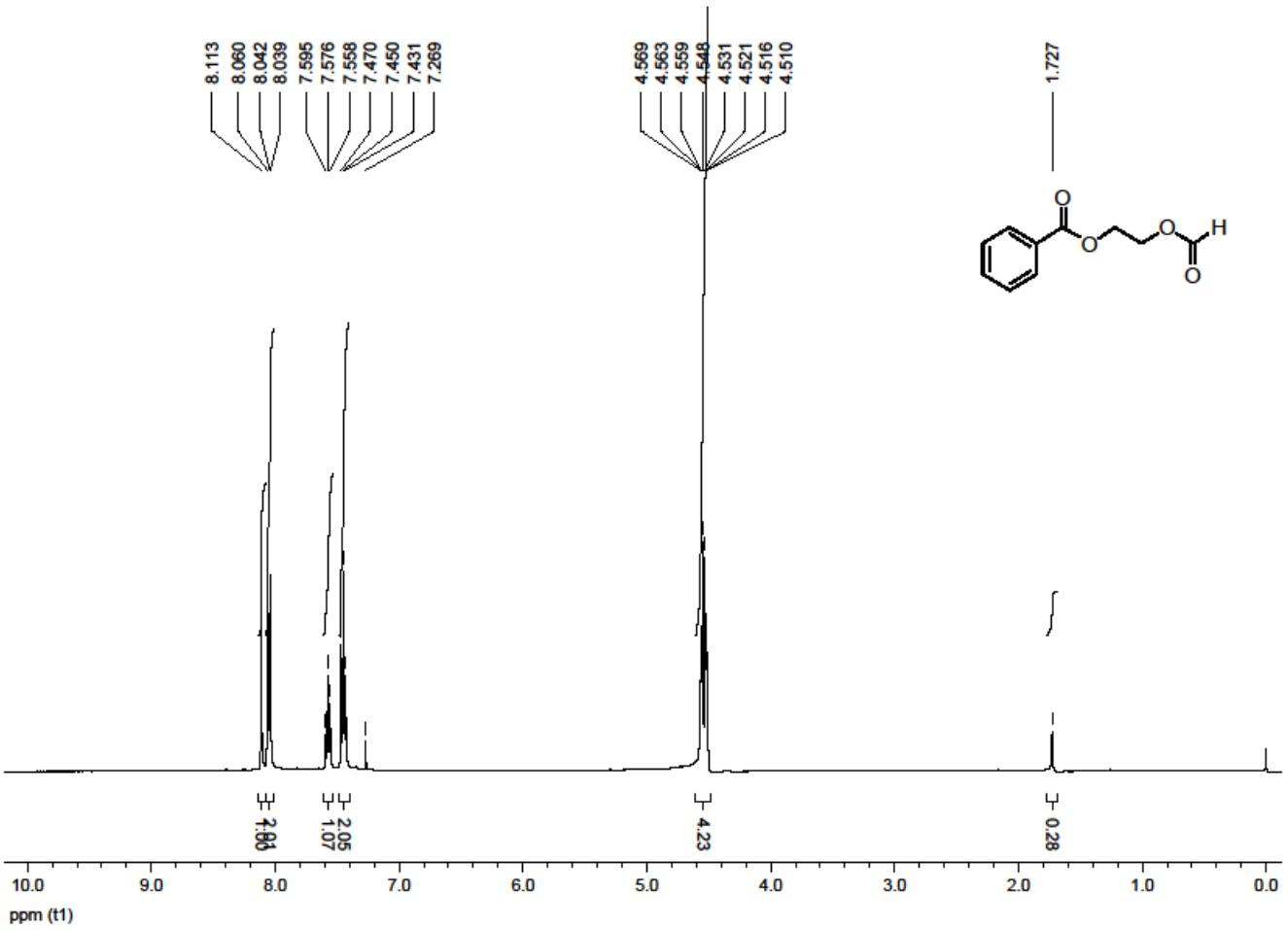
ppm (t1)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7.470
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7.269
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4.563
4.559
4.548
4.531
4.521
4.516
4.510
1.727
2.00
2.07
2.05
4.23
0.28
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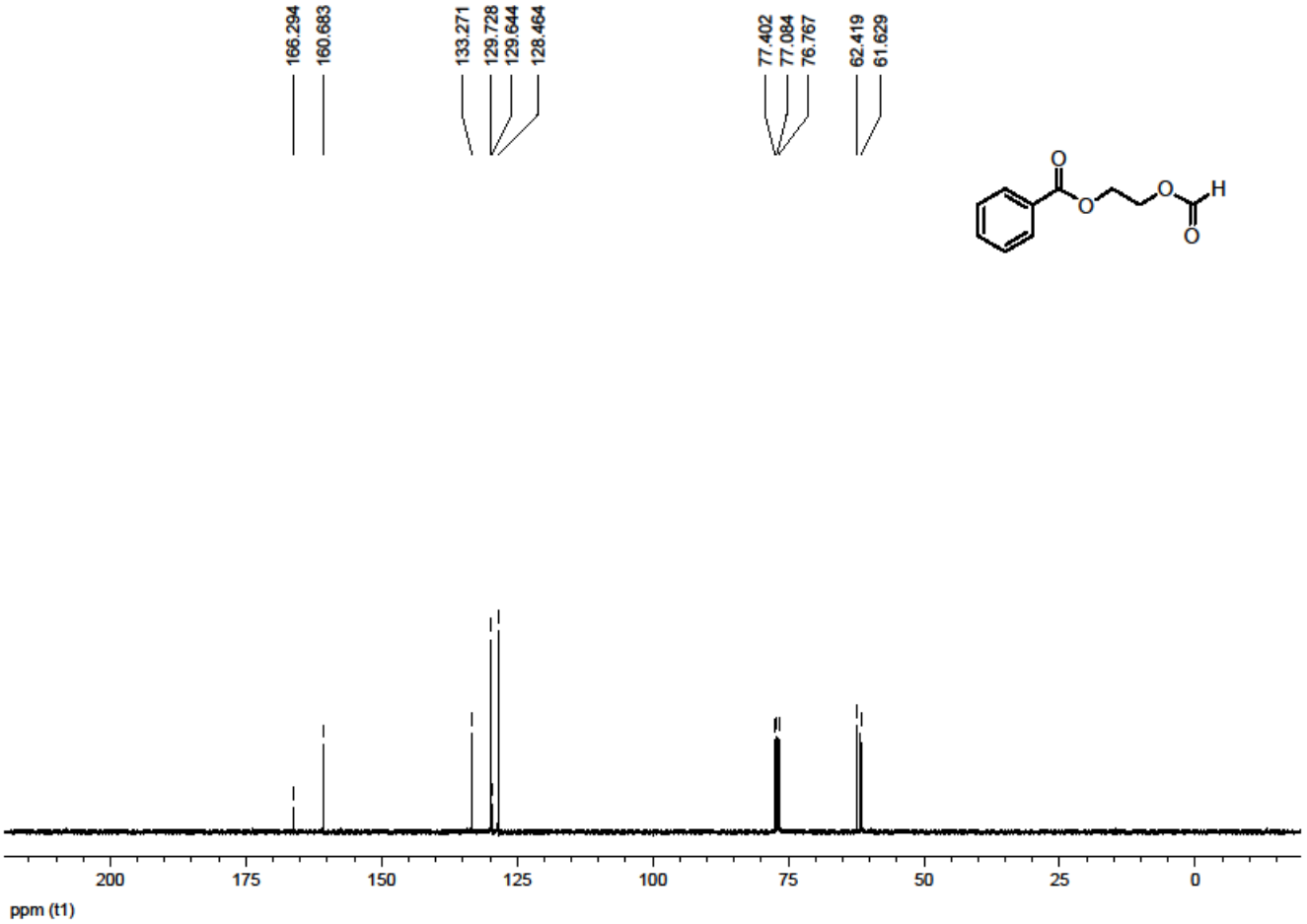
ppm (t1)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175
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125
100
75
50
25
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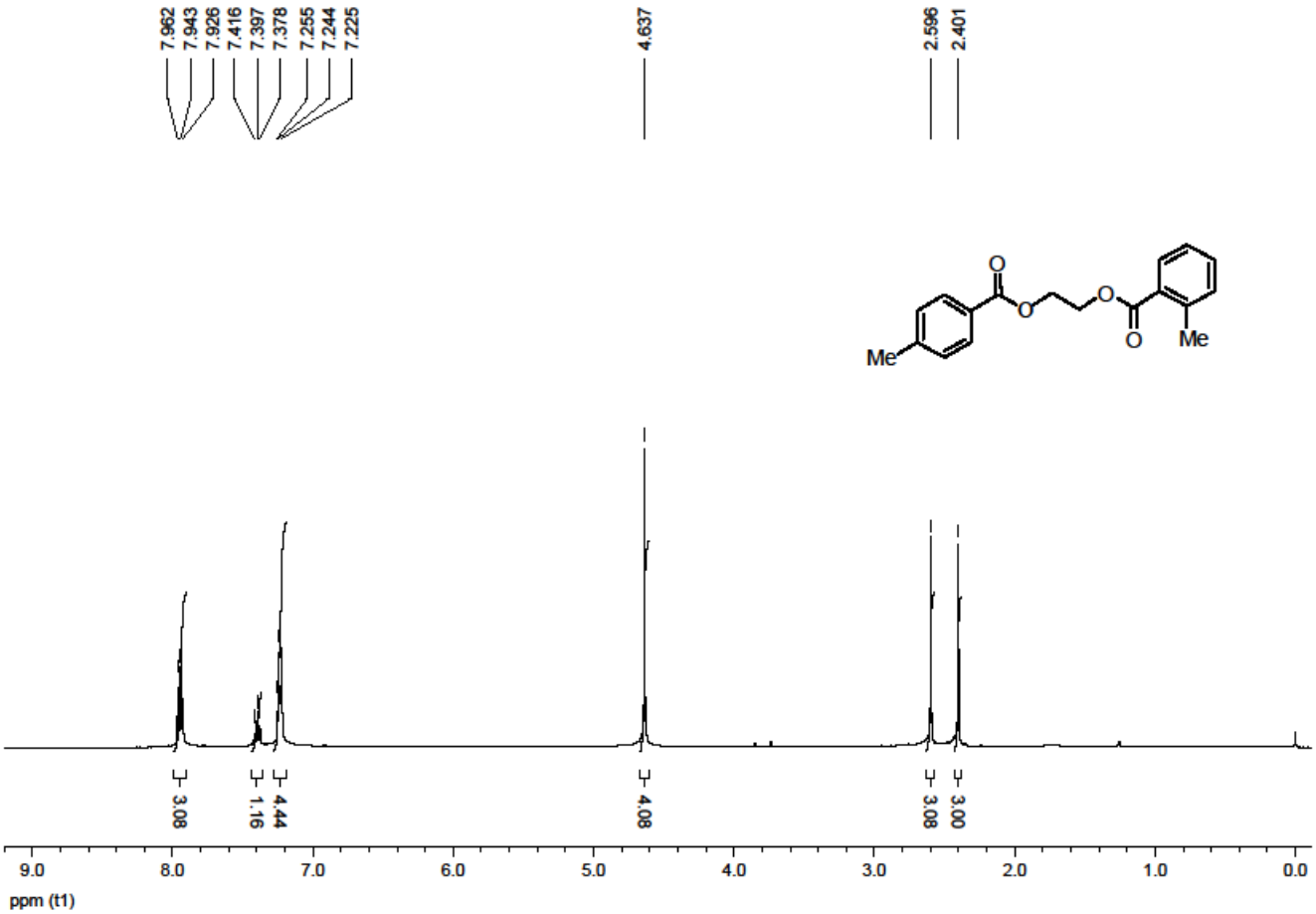
ppm (t1)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2.401
Me
Me
3.08
1.16
4.44
4.08
3.08
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7.0
6.0
5.0
4.0
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2.0
1.0
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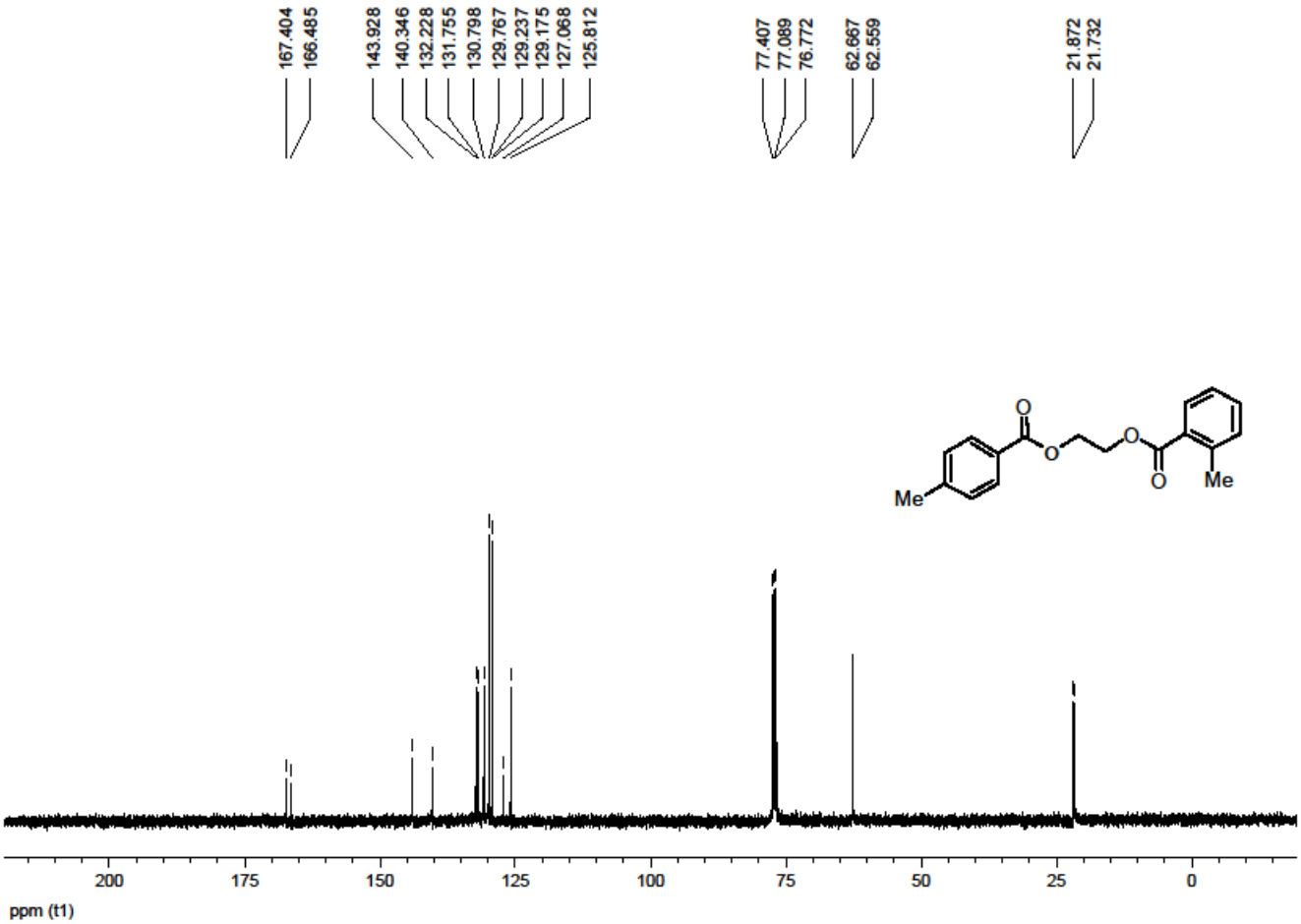
ppm (t1)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130.798
129.767
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129.175
127.068
125.812
77.407
77.089
76.772
62.667
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21.732
Me
Me
200
175
150
125
100
75
50
25
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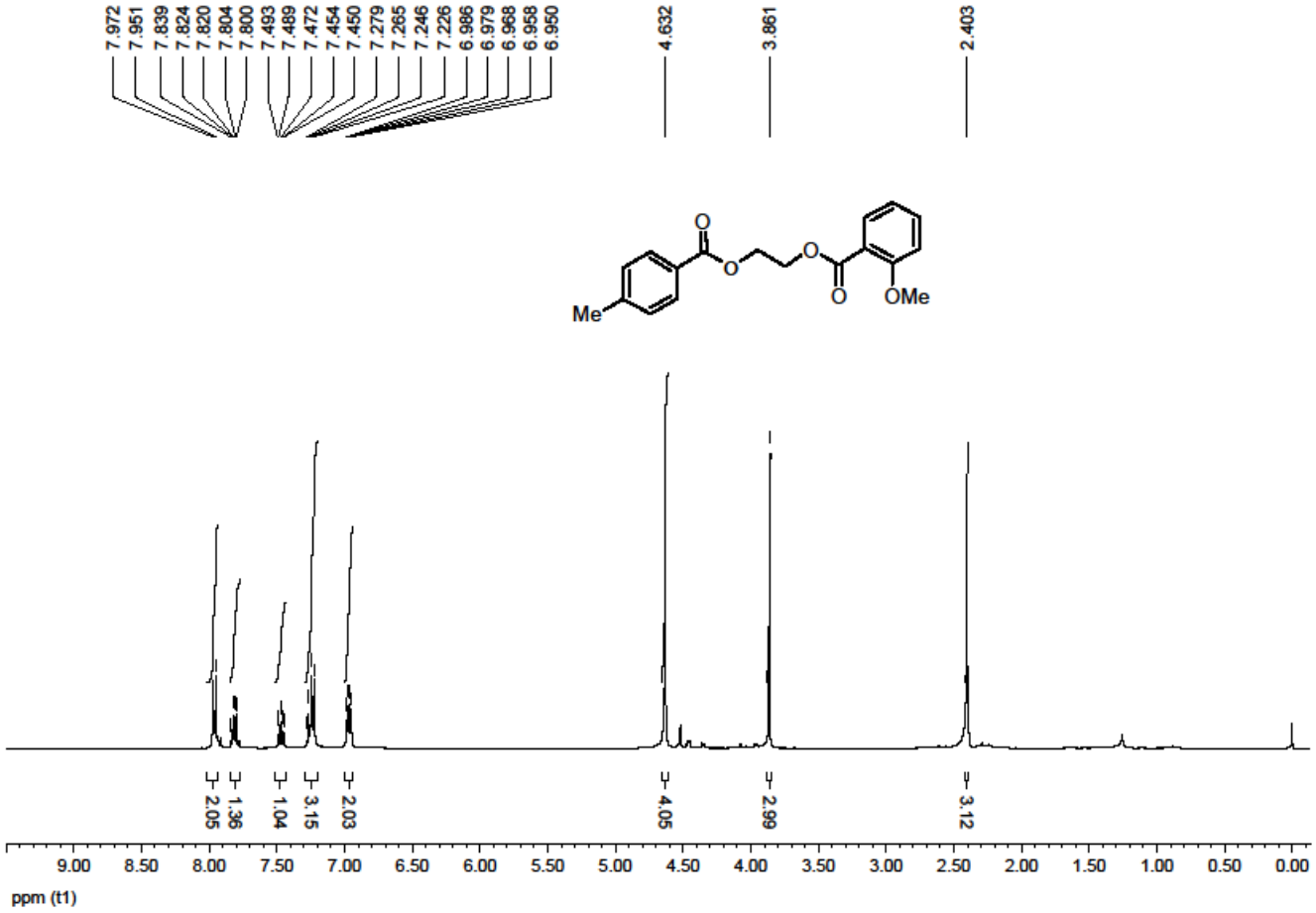
ppm (t1)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7.450
7.279
7.265
7.246
7.226
6.986
6.979
6.968
6.958
6.950
4.632
3.861
2.403
O
O
O
O
Me
OMe
2.05
1.96
1.04
3.15
2.03
4.05
2.99
3.12
9.00
8.50
8.00
7.50
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6.50
6.00
5.50
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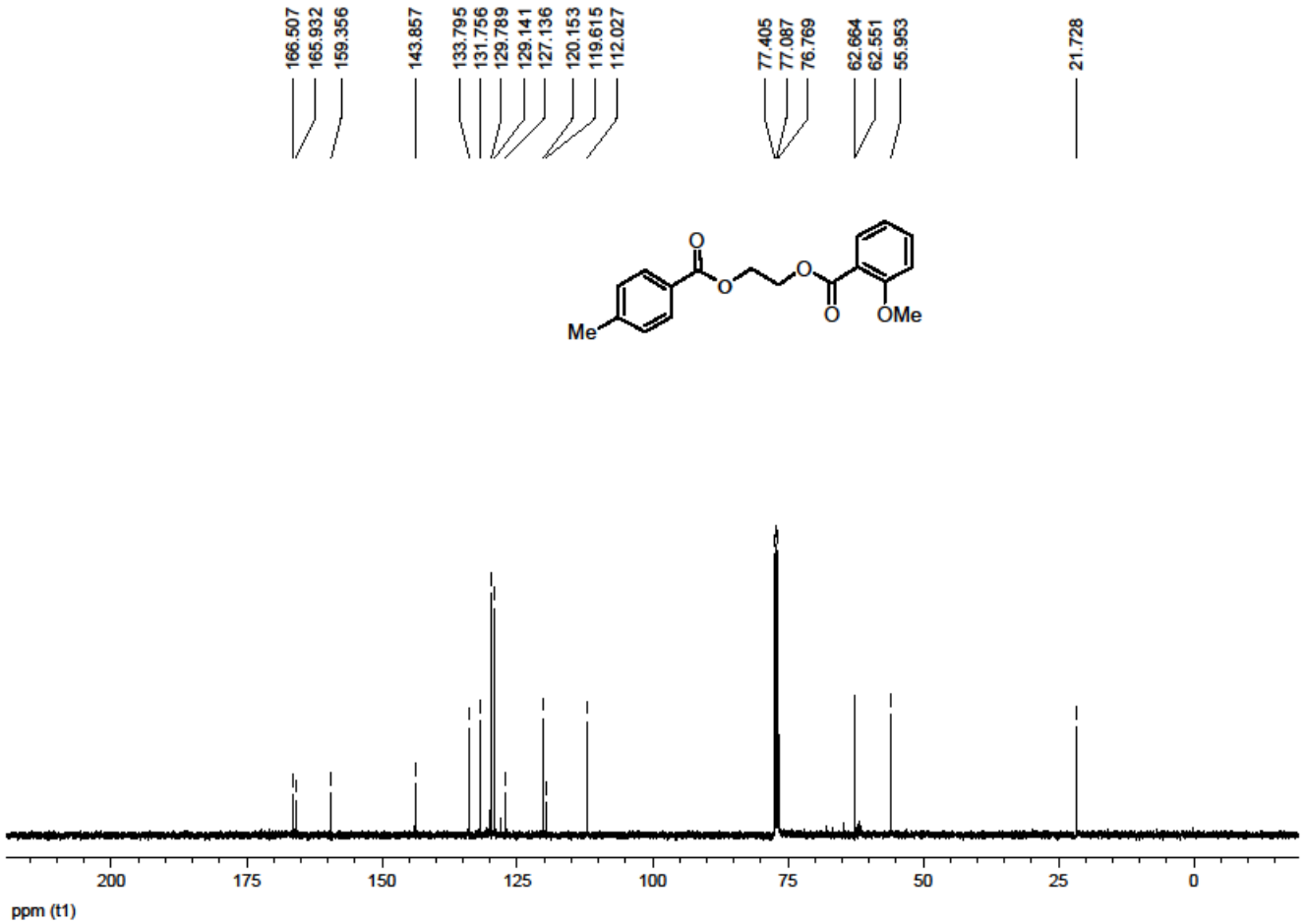
ppm (t1)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129.141
127.136
120.153
119.615
112.027
77.405
77.087
76.769
62.664
62.551
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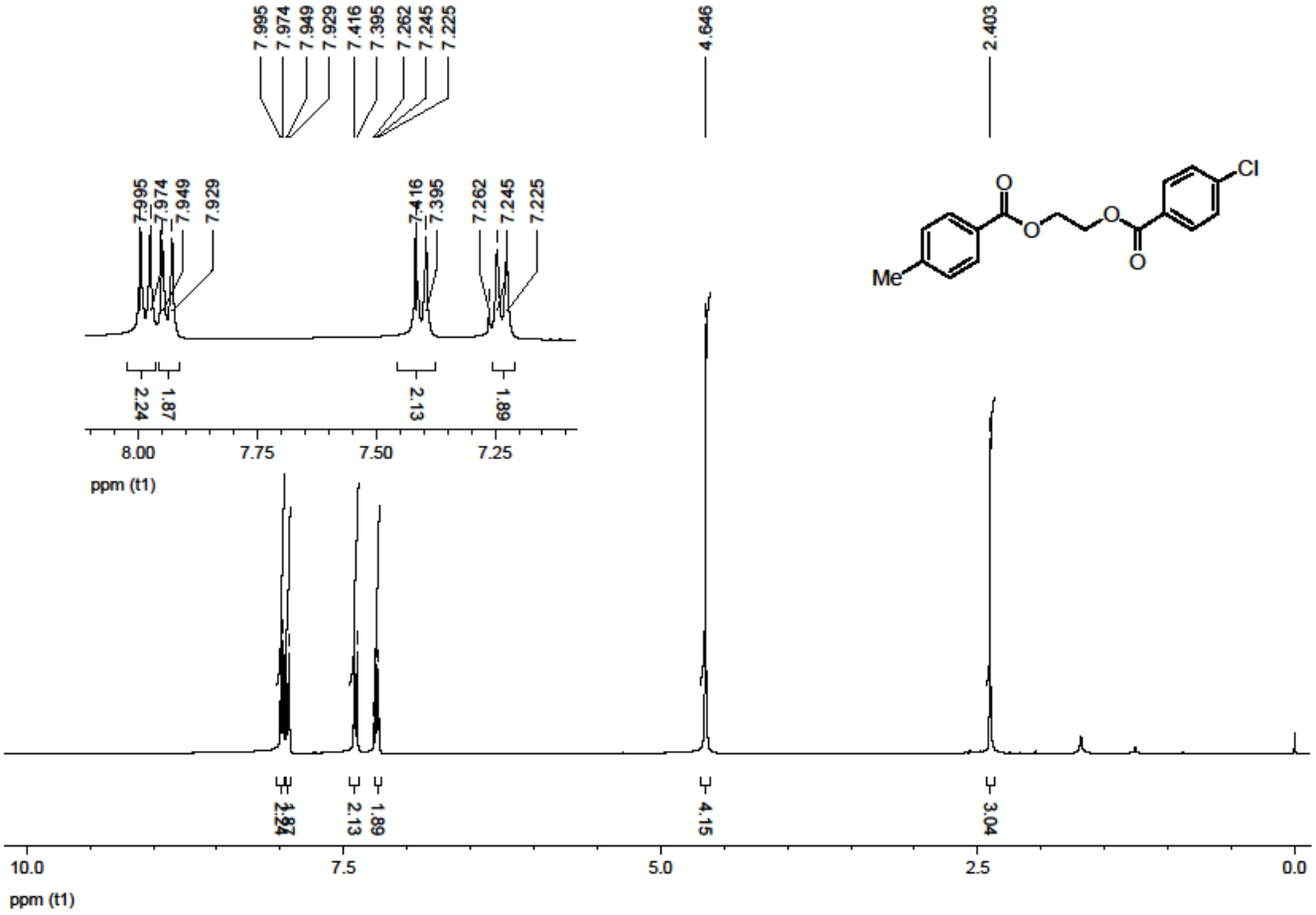
21.728
O
O
O
O
Me
OMe
200
175
150
125
100
75
50
25
0
ppm (t1)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Cl
Me
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1.87
2.13
1.89
8.00
7.75
7.50
7.25
ppm (t1)
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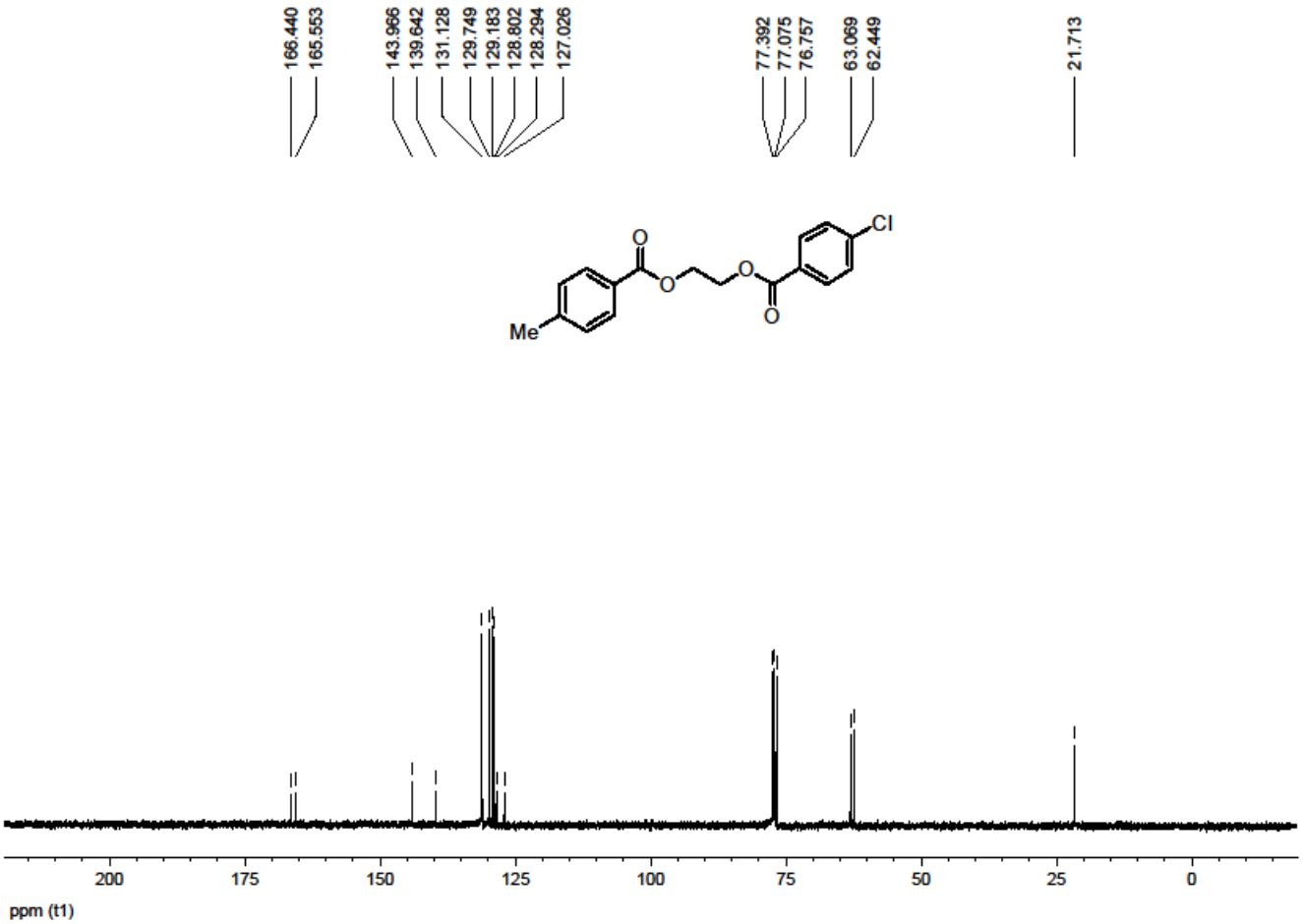
ppm (t1)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21.713
Cl
Me
200
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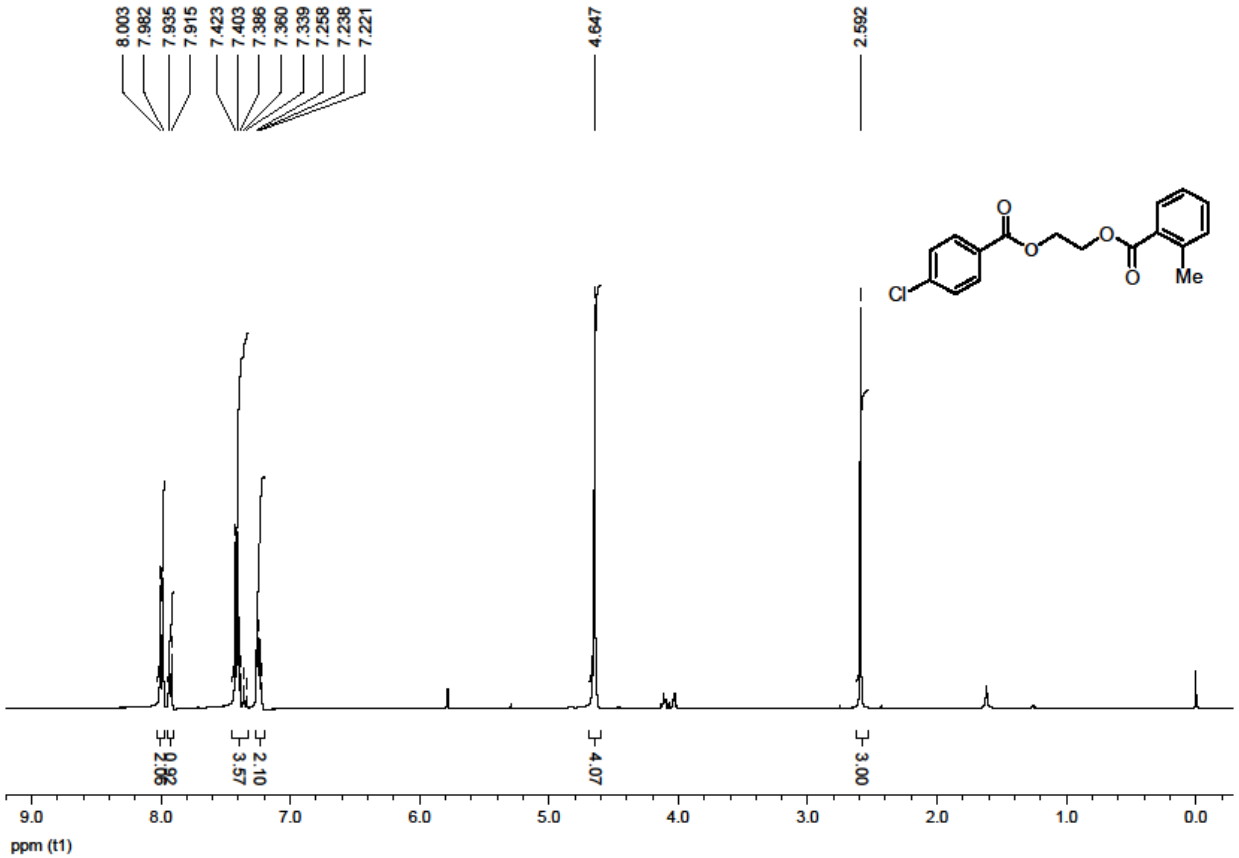
125
100
75
50
25
0
ppm (t1)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7.221
4.647
2.592
Cl
O
O
O
O
Me
2.06
2.00
3.57
2.10
4.07
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2.0
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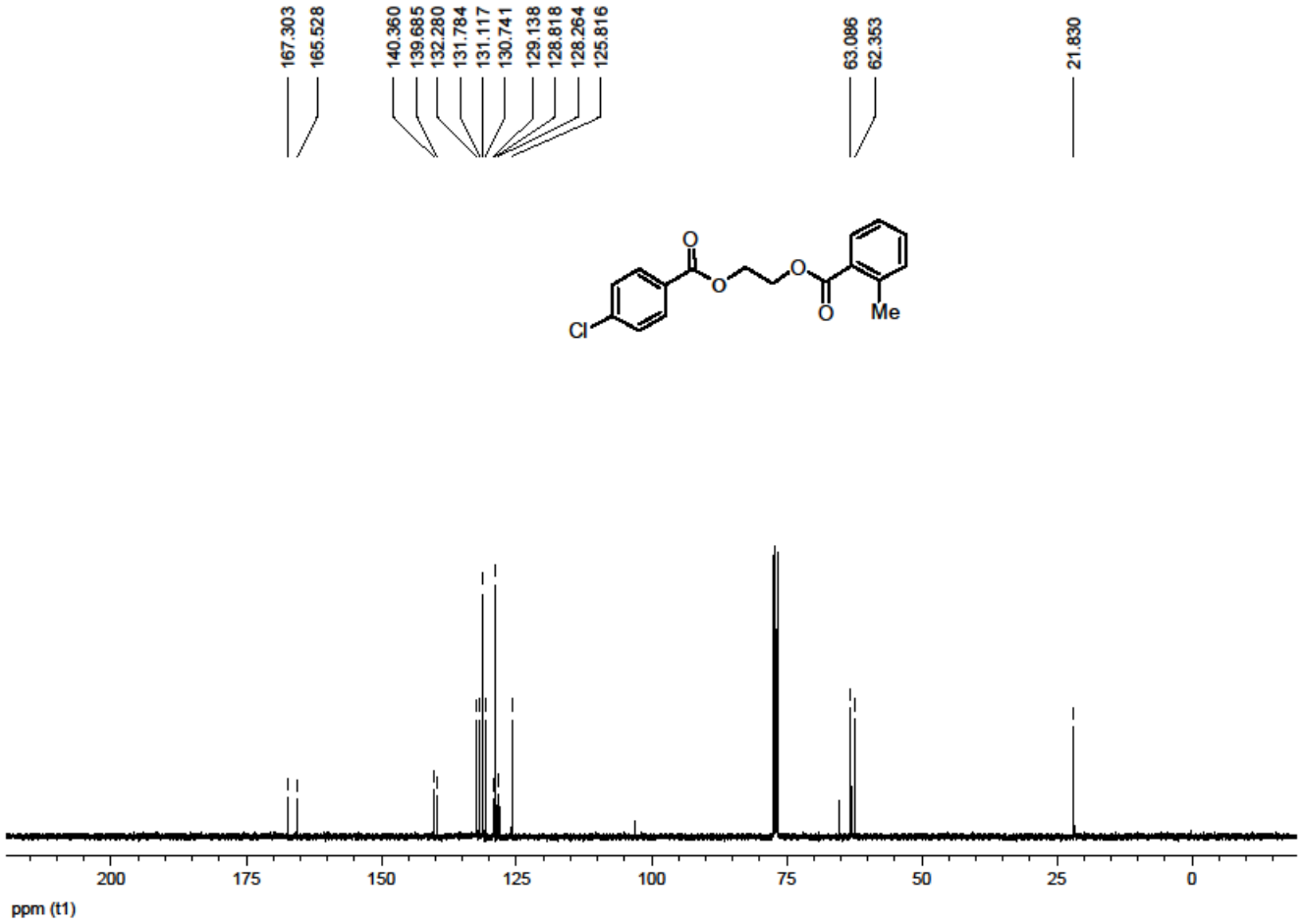
ppm (t1)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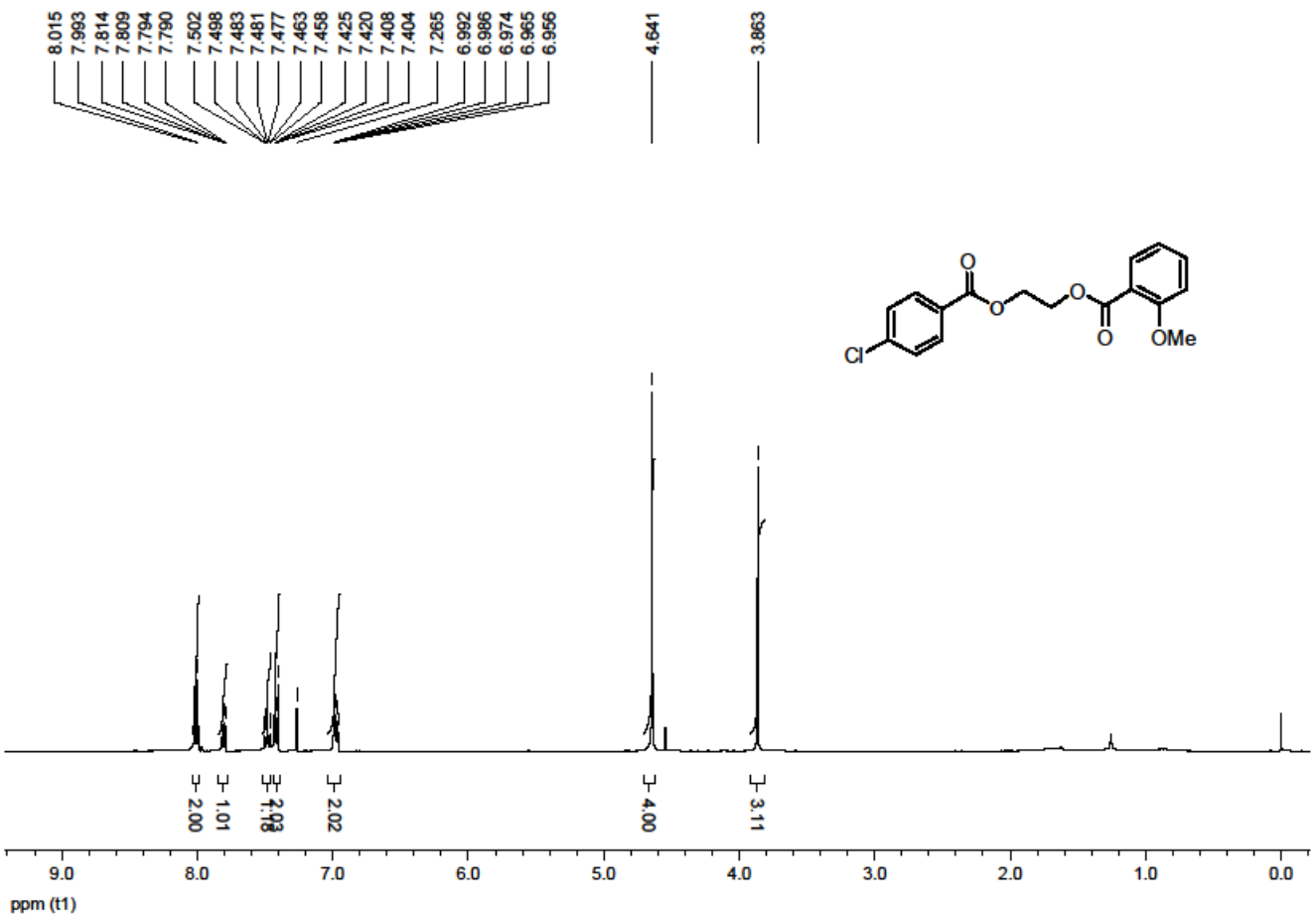
128.818
128.264
125.816
63.086
62.353
21.830
Cl
O
O
O
O
Me
200
175
150
125
100
75
50
25
0
ppm (t1)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7.993
7.814
7.809
7.794
7.790
7.502
7.498
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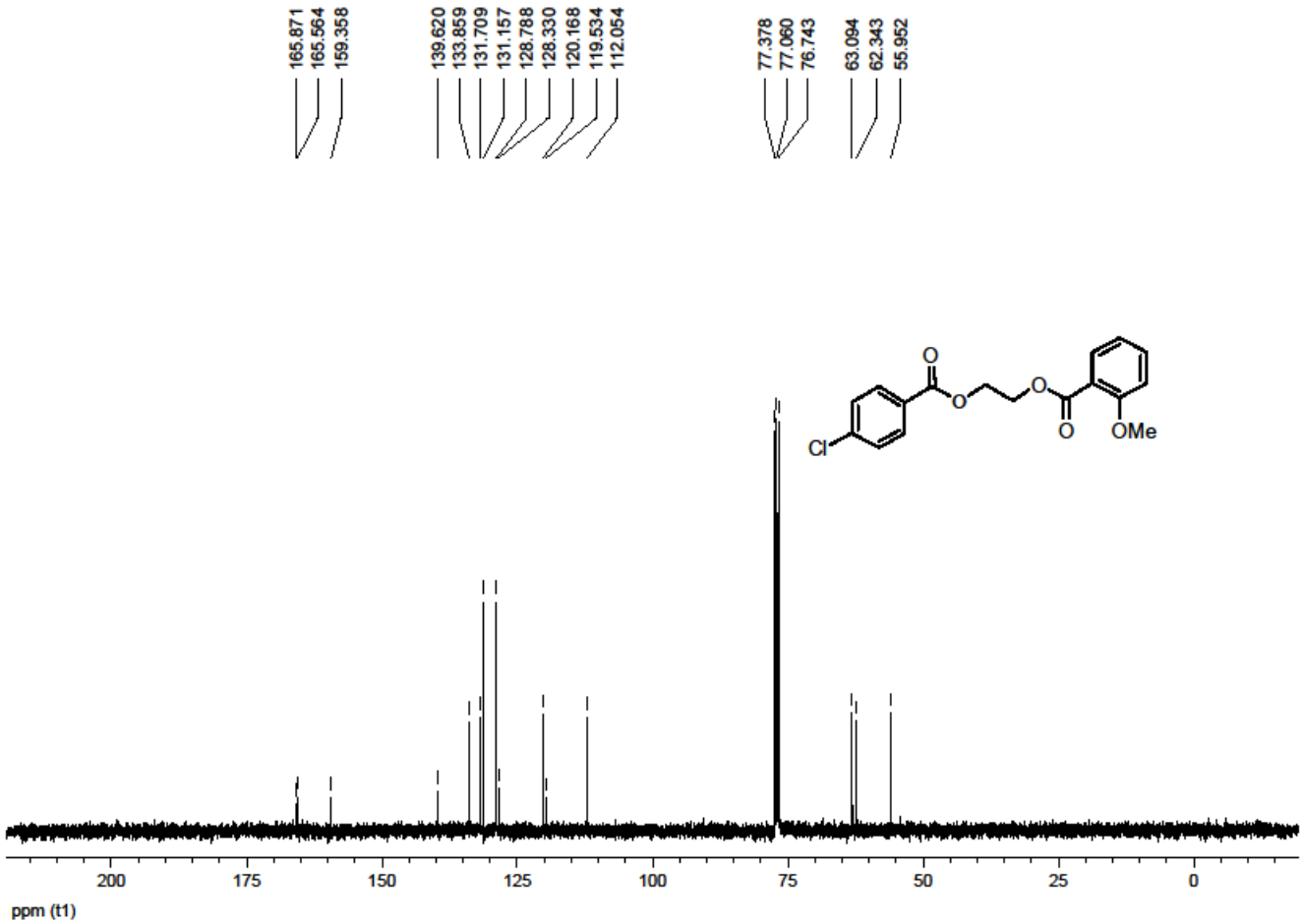
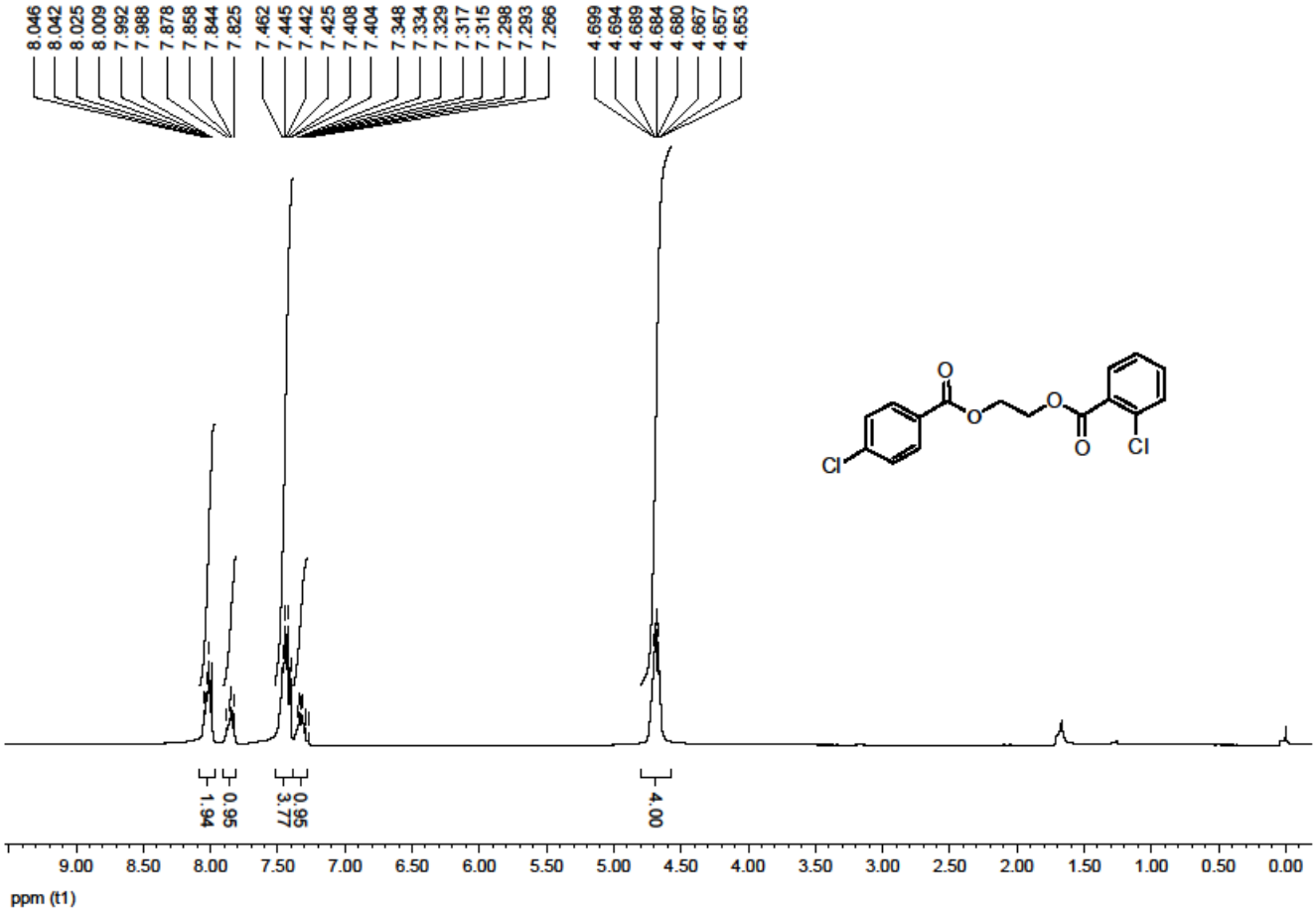
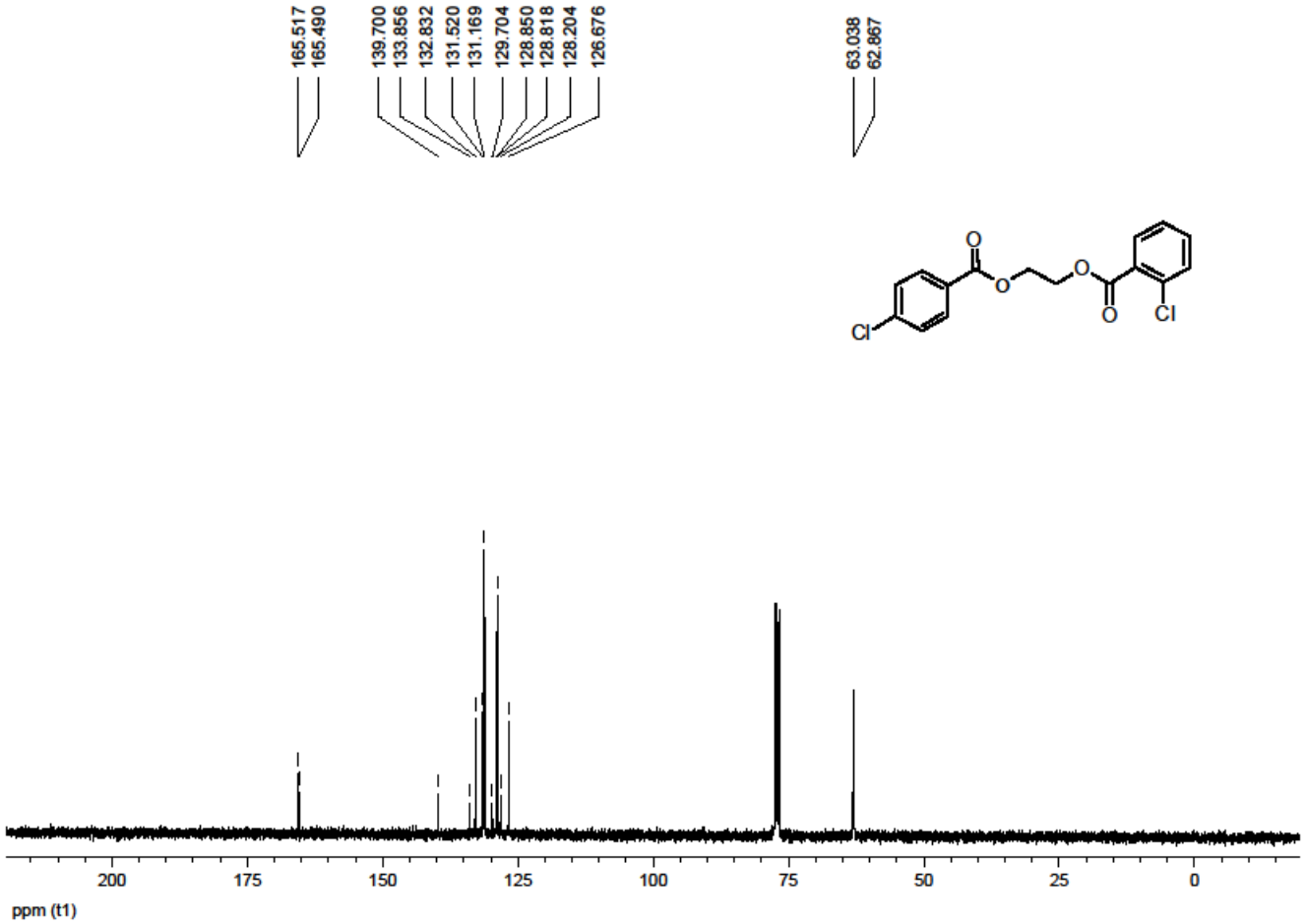
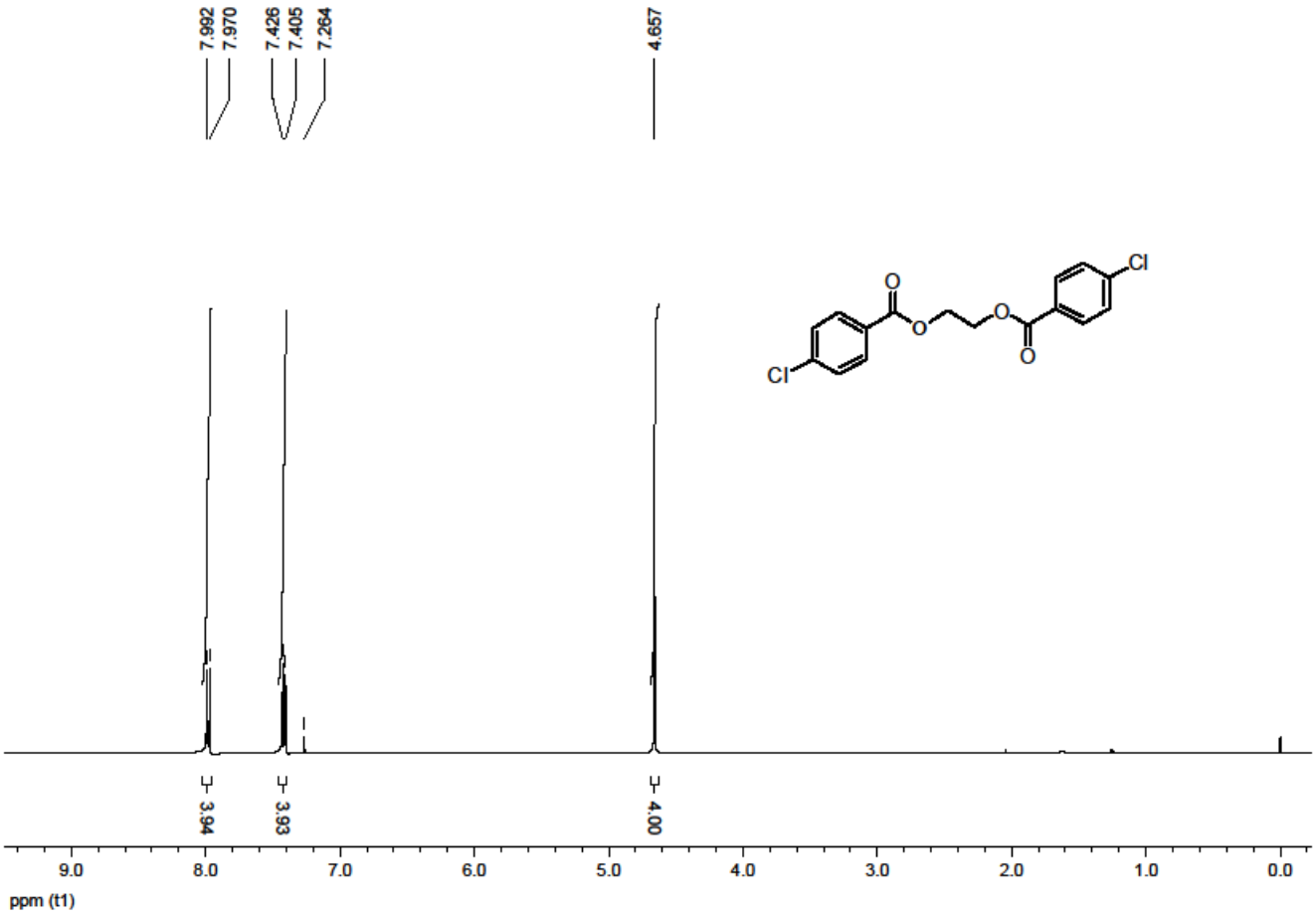
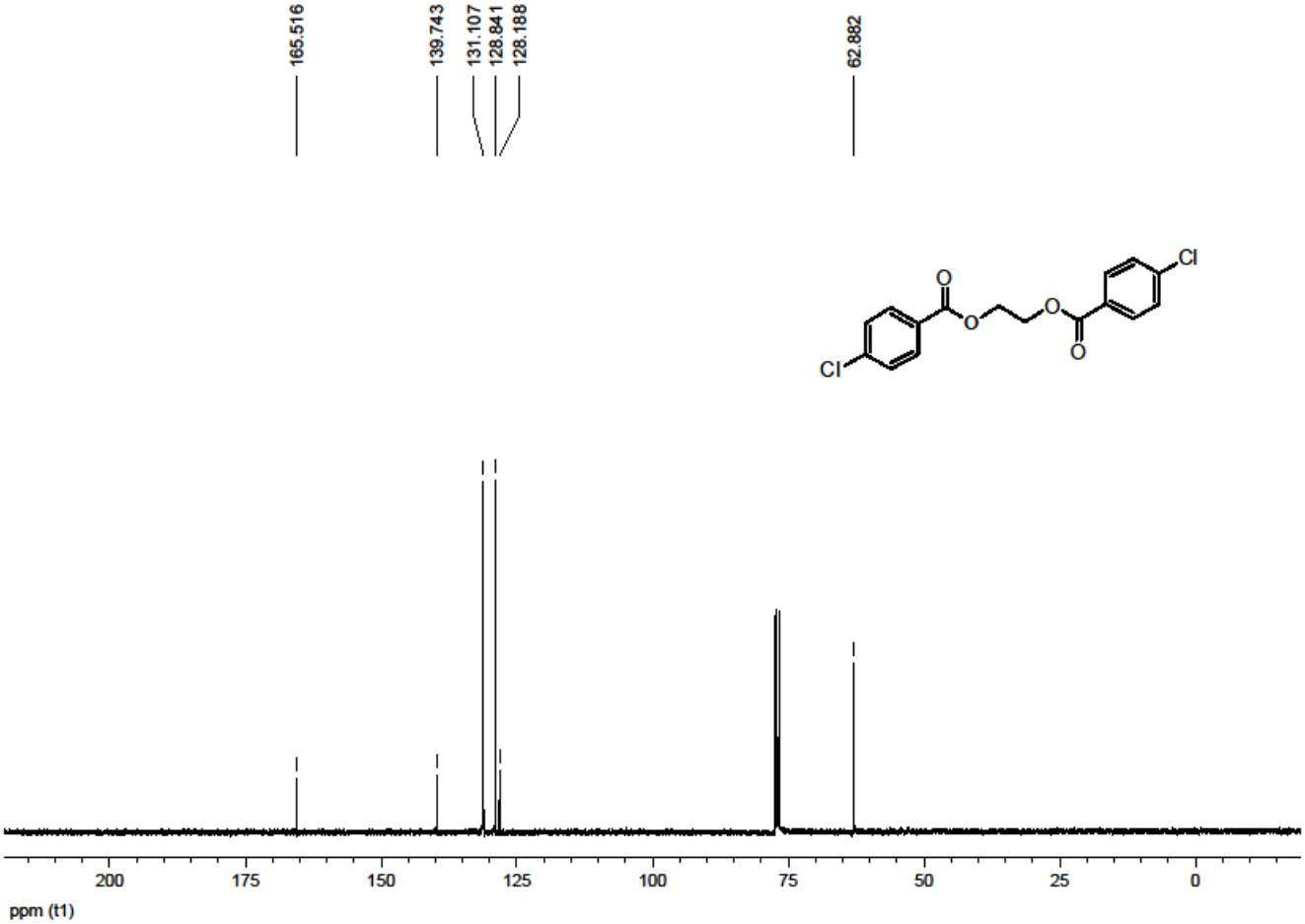
7.481
7.477
7.463
7.458
7.425
7.420
7.408
7.404
7.265
6.992
6.986
6.974
6.965
6.956
4.641
3.863
Cl
O
O
O
O
OMe
2.00
1.01
2.02
4.00
3.11
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


165.871
165.564
159.358
139.620
133.859
131.709
131.157
128.788
128.330
120.168
119.534
112.054
77.378
77.060
76.743
63.094
62.343
55.952
Cl
O
O
O
O
OMe
200
175
150
125
100
75
50
25
0
ppm (t1)


8.046
8.042
8.025
8.009
7.992
7.988
7.878
7.858
7.844
7.825
7.462
7.445
7.442
7.425
7.408
7.404
7.348
7.334
7.329
7.317
7.315
7.298
7.293
7.266
4.699
4.694
4.689
4.684
4.680
4.667
4.657
4.653
1.94
0.95
3.77
0.95
4.00
9.00
8.50
8.00
7.50
7.00
6.50
6.00
5.50
5.00
4.50
4.00
3.50
3.00
2.50
2.00
1.50
1.00
0.50
0.00
ppm (t1)
O
O
O
O
Cl
Cl


165.517
165.490
139.700
133.856
132.832
131.520
131.169
129.704
128.850
128.818
128.204
126.676
63.038
62.867
200
175
150
125
100
75
50
25
0
ppm (t1)
O
O
O
O
Cl
Cl


7.992
7.970
7.426
7.405
7.264
4.657
Cl
O
O
O
O
Cl
3.94
3.93
4.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


165.516
139.743
131.107
128.841
128.188
62.882
Cl
O
O
O
O
Cl
200
175
150
125
100
75
50
25
0
ppm (t1)

3) A checkCIF report regarding compound **3cm**

checkCIF/PLATON (standard)

You have not supplied any structure factors. As a result the full set of tests cannot be run.
THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.
No syntax errors found.
CIF dictionary
Please wait while processing ....
Interpreting this report
Datablock: r131217f1

Bond precision: C-C = 0.0023 A Wavelength=0.71073
Cell: a=7.7369(15) b=7.9449(16) c=12.788(3)
alpha=104.35(3) beta=97.85(3) gamma=92.83(3)
Temperature: 173 K

	Calculated	Reported
Volume	751.6(3)	751.6(3)
Space group	P -1	P-1
Hall group	-P 1	?
Moiety formula	C16 H12 Cl2 O4	?
Sum formula	C16 H12 Cl2 O4	C16 H12 Cl2 O4
Mr	339.16	339.16
Dx,g cm-3	1.499	1.499
Z	2	2
Mu (mm-1)	0.446	0.446
F000	348.0	348.0
F000'	348.76	
h,k,lmax	10,10,17	10,10,17
Nref	4078	3946
Tmin,Tmax	0.915,0.948	0.916,0.948
Tmin'	0.915	

Correction method= MULTI-SCAN
Data completeness= 0.968 Theta(max)= 29.200
R(reflections)= 0.0445(2477) wR2(reflections)= 0.1075(3946)
S = 1.010 Npar= Npar = 200

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

PLAT029_ALERT_3_C
_diffrn_measured_fraction_theta_full Low 0.968
Note

Alert level G
PLAT005_ALERT_5_G No
_iucr_refine_instructions_details in the CIF Please
Do !
PLAT066_ALERT_1_G Predicted and Reported
Tmin&Tmax Range Identical ? Check
PLAT154_ALERT_1_G The su's on the Cell Angles are
Equal ..... 0.03000 Degree

---

0 **ALERT level A** = Most likely a serious problem - resolve or explain
0 **ALERT level B** = A potentially serious problem, consider carefully
1 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
3 **ALERT level G** = General information/check it is not something unexpected

2 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
0 ALERT type 2 Indicator that the structure model may be wrong or deficient
1 ALERT type 3 Indicator that the structure quality may be low
0 ALERT type 4 Improvement, methodology, query or suggestion
1 ALERT type 5 Informative message, check

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important

in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

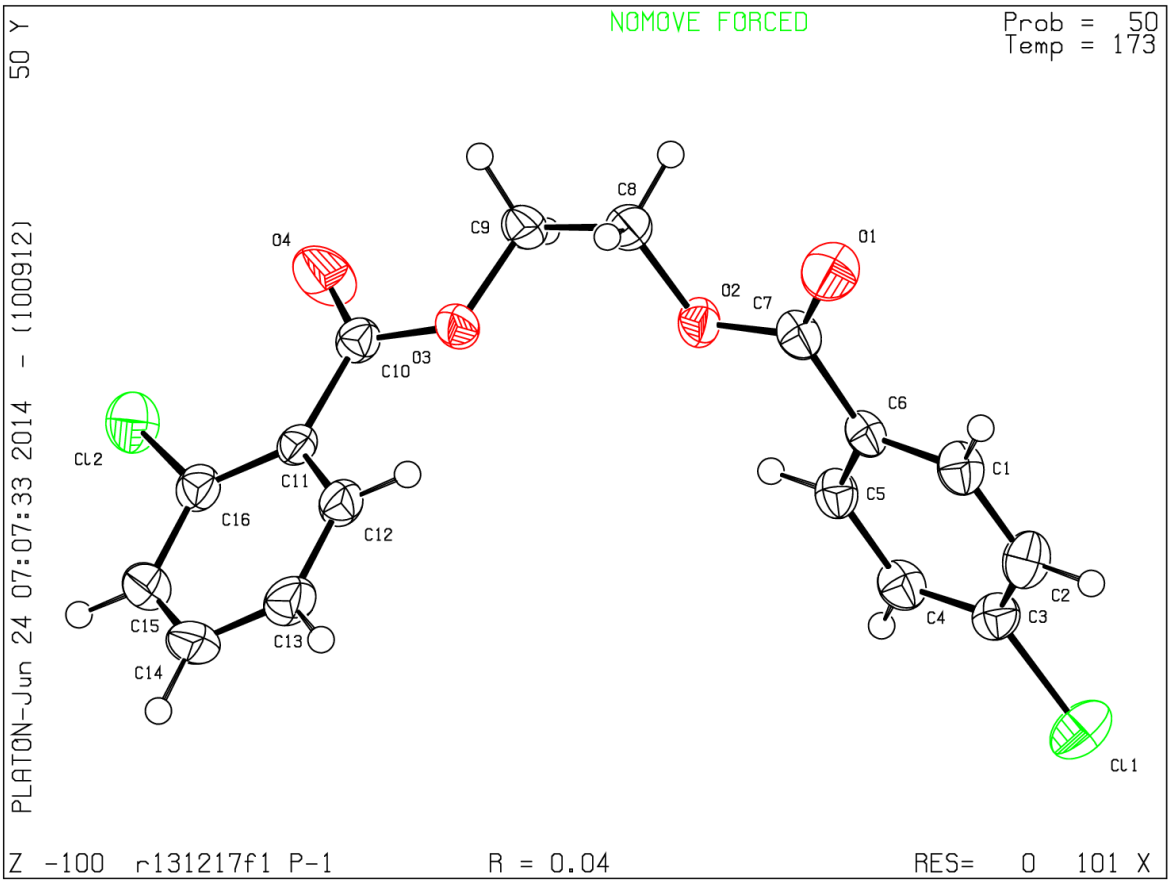
Publication of your CIF in IUCr journals

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*,*Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 05/02/2014; check.def file version of 05/02/2014
Datablock r131217f1 - ellipsoid plot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Temp = 173
50 Y
PLATON-Jun 24 07:07:33 2014 - (100912)
O4
C9
C8
O1
O2
C7
O3
C10
C6
CL2
C11
C1
C5
C16
C12
C2
C4
C3
C15
C13
C14
CL1
Z -100 r131217f1 P-1
R = 0.04
RES= 0 101 X