Electronic Supplementary Material (ESI) for Chemical Communications

Carboxylic Acid Anhydrides via Pd-Catalyzed Carbonylation of Aryl Halides at Atmospheric CO Pressure

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1. General information

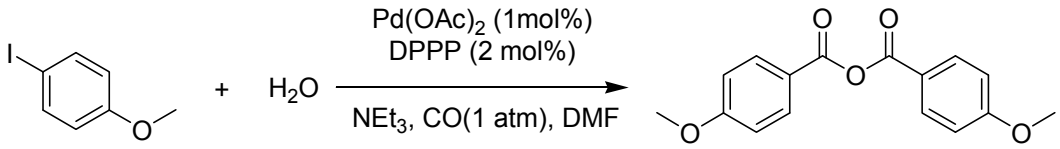
Flash chromatography was performed with freshly distilled solvents. ^{1}H NMR (300 MHz) and ^{13}C NMR (75 MHz) spectra were recorded using $CDCl_3$ as solvent. Chemical shifts (δ) are reported in ppm, using TMS as an internal standard. Data are presented as follows: chemical shift (ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet). The amount of water was determined with an 831 KF Coulometer (Metrohm, Switzerland). Solvents were purified using the following method. DMF, DMSO and DMAc were dried over CaH_2 for 24 h and distilled under reduced pressure. Toluene and dioxane were dried over sodium for 4 h, and distilled under N_2 atmosphere.

2. The effect of water on carbonylation of 4-iodoanisole

The concentration of water in DMF was determined to be 25 ppm after refluxing over CaH_2 for 24 h. From the background reaction, we found that water also came from the reaction system, including the CO gas and autoclave.

The actual amount of water in the reaction was determined in a blank experiment that mimicked the carbonylation reactions via the following procedure: an autoclave containing a 10 mL Teflon reaction tube was charged with a magnetic stir bar, which was then capped with a stopper and flushed with argon. 4-Iodoanisole (1 mmol), NEt_3 (2.5 mmol), DMF (2 mL) were added to the tube with a syringe. The tube was placed in the autoclave. Once sealed, the autoclave was purged several times with CO, then pressurized to 1 atm at room temperature and heated in an oil bath at 115 °C for 6 h. The autoclave was then cooled to room temperature and vented to discharge the CO. The amount of water in the DMF solution was determined by an 831 KF Coulometer; 4.9 mg (0.27 mmol) water was found, and this was assumed to be the background water. The effect of different amount of additionally-added water is shown in Table S1.

Table S1 The effect of added water on the reaction.

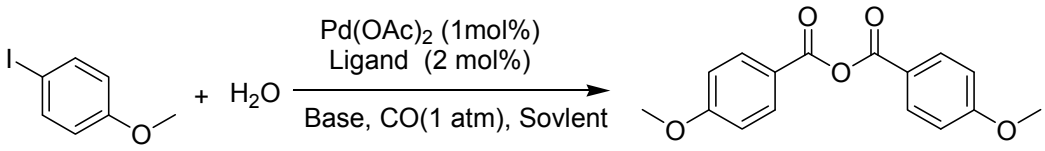


Entry	Water added	Water concentration (mol%)	Isolated yield (%)
1	0 mg	27	55
2	3 mg	44	80
3	4 mg	49	90
4	6 mg	61	77
5	8 mg	72	49
6	18 mg	127	0

3. Optimization of reaction conditions for carbonylation of 4-iodoanisole

The reaction was carried out in an autoclave containing a 10 mL Teflon reaction tube. $Pd(OAc)_2$ (0.01 mmol), ligand (0.02 mmol) and a magnetic stir bar were placed in the tube, which was then capped with a stopper and flushed with argon. Then, 4-iodoanisole (1 mmol), base (2.5 mmol), solvent (2 mL, a certain amount of water added; see Table S2) were added to the tube with a syringe. The tube was placed in the autoclave. Once sealed, the autoclave was purged several times with CO, then pressurized to 1 atm at room temperature and heated in an oil bath at 115 °C for 6 h. The autoclave was then cooled to room temperature and vented to discharge the excess CO. Water (10 mL) was added, and the product was extracted with DCM (3×3 mL). The organic layers were washed with brine, dried over Na_2SO_4, and evaporated. The crude product was purified by column chromatography on silica gel using a mixture of ethyl acetate and petroleum ether as eluent to give 4-meothoxy-benzoic anhydride. The isolated yields are given in Table S2. Structures of ligands are shown in Scheme S1.

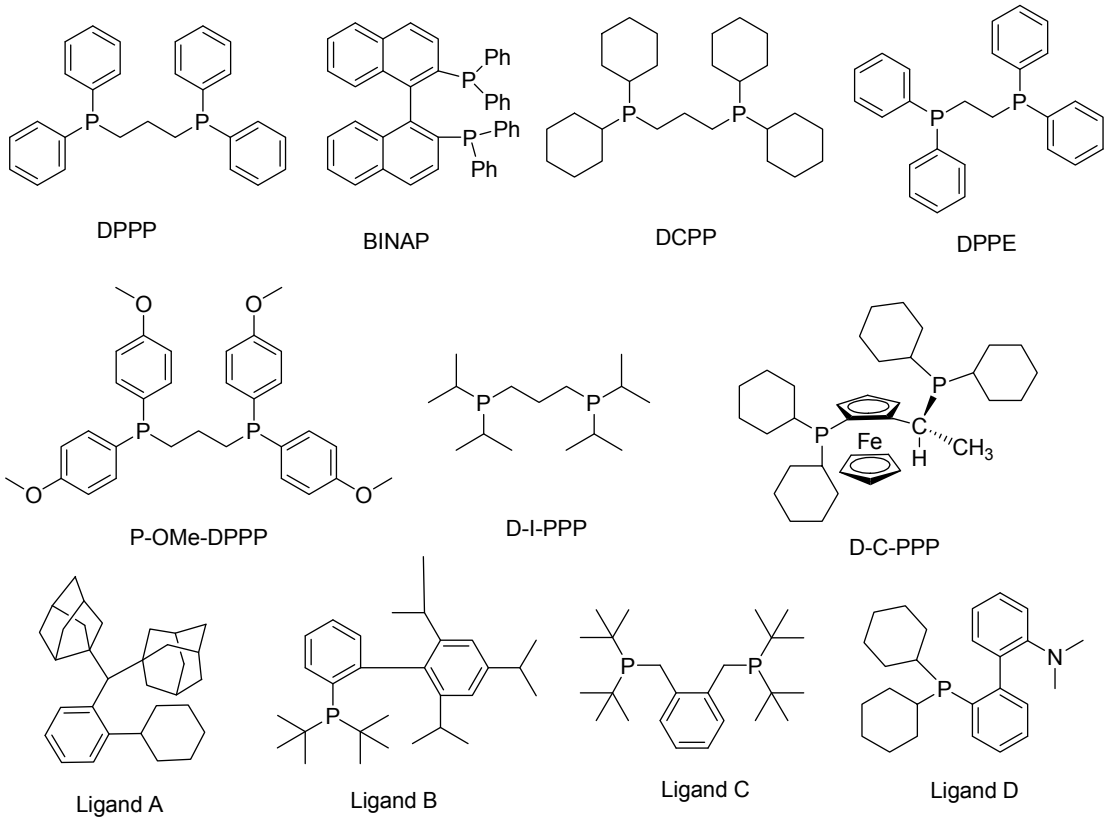
Table S2 Optimization of carbonylation of 4-iodoanisole with water at 1 atm CO[a]



Entry	Ligand	Base	Water contents (mol%)	Solvent	Isolated Yield (%)
1	DPPP	Et_3N	27%	DMF	55
2	DPPP	Et_3N	44%	DMF	80
3	DPPP	Et_3N	50%	DMF	90
4	DPPP	Et_3N	72%	DMF	49
5	DPPP	Et_3N	94%	DMF	2
6	DPPP	Et_3N	50%	DMF	90
7	DPPP	Et_3N	50%	DMF	45
8	DPPF	Et_3N	50%	DMF	12
9	DPPE	Et_3N	50%	DMF	40
10	BINAP	Et_3N	50%	DMF	24
11	P-OMe-DPPP	Et_3N	50%	DMF	53
12	D-I-PPP	Et_3N	50%	DMF	52
13	D-C-PPP	Et_3N	50%	DMF	53
14	PPh_3	Et_3N	50%	DMF	32
15	Ligand A	Et_3N	50%	DMF	48
16	Ligand B	Et_3N	50%	DMF	70
17	Ligand C	Et_3N	50%	DMF	69
18	Ligand D	Et_3N	50%	DMF	56
19	Ligand free	Et_3N	50%	DMF	0
20	DPPP	KOH	50%	DMF	0
21	DPPP	KOBu	50%	DMF	0
22	DPPP	K_2CO_3	50%	DMF	65
23	DPPP	DBU	50%	DMF	0
24	DPPP	DIPEA	50%	DMF	0
25	DPPP	Et_3N	50%	Toluene	56

26	DPPP	Et_3N	50%	DMSO	0
27	DPPP	Et_3N	50%	DMAc	0
28	DPPP	Et_3N	50%	Dioxane	0

Scheme S1 Structures of ligands



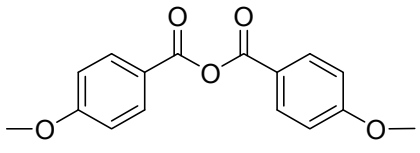
4. General procedure for carbonylation of aryl iodides

The reaction was carried out in an autoclave containing a 10 mL Teflon reaction tube. $Pd(OAc)_2$ (0.01 mmol), dppp (0.02 mmol) and a magnetic stir bar were placed in the tube which was then capped with a stopper and flushed with argon. Then, an aryl iodide (1 mmol), NEt_3 (2.5 mmol), DMF (2 mL, 0.22 mmol additional water added) were added to the tube with a syringe. The tube was placed in the autoclave. Once sealed, the autoclave was purged several times with CO, then pressurized to 1 atm at room temperature and heated in an oil bath at 115 °C for 6 h. The autoclave was then cooled to room temperature and vented to discharge CO. Water (10 mL) was added, and the product was extracted with DCM (3×3 mL). The organic layers were washed with brine, dried over Na_2SO_4, and evaporated. The crude product was purified by column chromatography on

silica gel using a mixture of ethyl acetate and petroleum ether as eluent to give the following compounds.

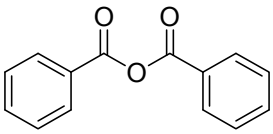
5. Experimental data for products

(2a) 4-Methoxybenzoic anhydride



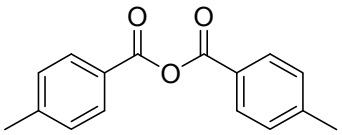
R_f = 0.3 (petroleum ether/ethyl acetate 12:1), White solid; m.p. = 88-93 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm): 8.10 (d, *J* = 7. 4Hz, 4H), 6.98 (d, *J* = 7.4 Hz, 4H), 3.90 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm): 164.9, 162.3, 132.8, 121.3, 114.2, 55.6; IR (KBr): 3010, 1790, 1720, 1620, 1300, 1220, 1180 cm^{-1}; HRMS (ESI) calc. for (M + Na^+) 309.0739; found 309.0733.

(2b) benzoic anhydride



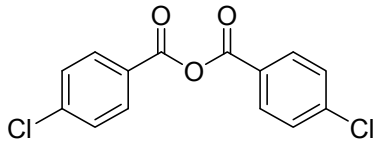
R_f = 0.3 (petroleum ether/ethyl acetate 15:1), White solid. m.p = 42-44°C, ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm): 8.16 (d, *J* = 7.5 Hz, 4H), 7.68 (d, *J* = 7.5 Hz, 2H), 7.49-7.44 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm): 162.4, 134.5, 128.8; IR (KBr) 3064, 1788, 1713, 1609, 1212,1167, 1000 cm^{-1}; HRMS (ESI) calc. for (M + Na^+) 249.0522 found 249.0520.

(2c) 4-methylbenzoic anhydride



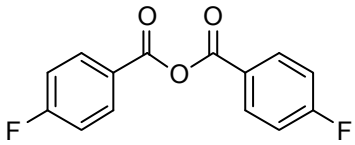
R_f = 0.2 (petroleum ether/ethyl acetate 14:1), White solid. m.p = 78-80°C, ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm): 8.04 (dd, *J* =7.8 Hz, *J* =5.1 Hz , 4H), 7.32 (d, *J* = 7.8 Hz, 4H), 2.46 (s, 6H); ^{13}C NMR (75 MHz, CDCl3) δ (ppm): 162.6, 145.5, 130.6, 129.6, 126.3, 21.8; IR (KBr) 3010, 1775, 1717, 1616, 1229, 1175, 1043, 1000 cm^{-1}; HRMS(ESI) calc. for (M + Na^+) 277.0835 found 277.0836.

(2d) 4-chlorobenzoic anhydride (2d)



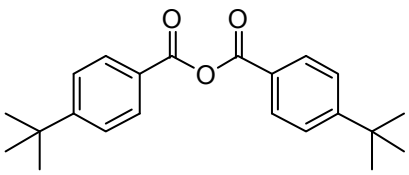
R_f = 0.3 (petroleum ether/ethyl acetate 9:1), White solid. m.p = 184-185°C, ^{1}H NMR (300 MHz, CD_3OD) δ (ppm): 7.51 (d, *J* = 8.4 Hz 4H), 8.07 (d, *J* = 8.4 Hz 4H); ^{13}C NMR (75 MHz, CD_3OD) δ (ppm): 167.4, 138.8, 130.9, 128.3; IR (KBr) 3010, 1800, 1720,1600, 1220, 1180, 1060, 1000 cm^{-1}; HRMS(ESI) calc. for (M + Na$^+$) 316.9748 found 316.9745.

(2e) 4-fluorobenzoic anhydride



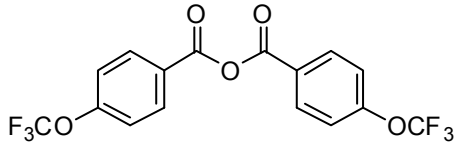
R_f = 0.2 (petroleum ether/ethyl acetate 20:1), White solid. m. p = 112-114°C, ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm): 8.19-8.16 (m, 4H), 7.23-7.19 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm): 167.8, 165.3, 161.2, 133.4, 133.3, 125.1, 116.4, 116.2; IR (KBr) 2967, 1885, 1857, 1784, 1585, 1286, 1239, 1177, 1029 cm^{-1}; HRMS(ESI) calc. for (M + Na$^+$) 285.0339 found 285.0334.

(2f) 4-tert-butylbenzoic anhydride



R_f = 0.3 (petroleum ether/ethyl acetate 15:1), colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm): 8.08 (d, *J* = 8.4 Hz, 4H), 7.53 (d, *J* = 8.4 Hz, 4H), 1.36 (s, 18H); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm): 162.5, 158.5, 130.5, 126.2, 125.9, 35.3, 31.0; IR (KBr); 2966，1783, 1722, 1607, 1409, 1224, 1042, 999 cm^{-1}; HRMS(ESI) calc. for (M + Na$^+$) 361.1780 found 361.1778.

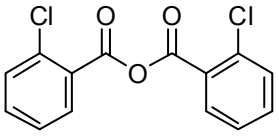
(2g) 4-(trifluoromethoxy)benzoic anhydride



R_f = 0.3 (petroleum ether/ethyl acetate 11:1), White solid. m.p = 68-69°C, ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm): 8.19 (d, *J* = 8.4Hz, 4H), 7.36 (d, *J* = 8.1Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 160.9, 153.9, 132.7, 126.8, 121.3, 120.6, 119.2; IR (KBr): 3010, 1791, 1725, 1609, 1508, 1225, 1171 cm^{-1};

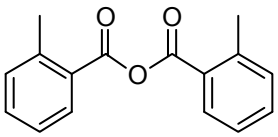
HRMS (ESI) calc. for (M + Na^+) 417.0174 found 417.0172.

(2h) 2-chlorobenzoic anhydride



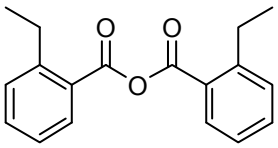
R_f = 0.3 (petroleum ether/ethyl acetate 15:1), White solid. m.p = 63-68°C, ^{1}H NMR (300 MHz, $CDCl_3$,) δ (ppm): 8.03 (d, *J* = 7.5 Hz, 2H), 7.54-7.49 (m, 4H), 7.42-7.36 (m, 2H); ^{13}CNMR (75 MHz, $CDCl_3$) δ (ppm): 160.4, 135.1, 134.1, 132.6, 131.6, 126.9; IR (KBr): 3010, 1783, 1721, 1593, 1442, 1209, 1074, 1008cm^{-1}; HRMS (ESI) calc. for (M + Na^+) 316.9748 found 316.9745.

(2i) 2-methylbenzoic anhydride



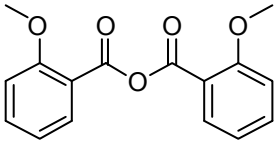
R_f = 0.3 (petroleum ether/ethyl acetate 15:1), White solid. m.p = 87-89°C,^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm): 7.96 (d, *J* = 7.7 Hz, 2H), 7.40-7.45 (m, 2H), 7.24 (d, *J* = 8.8 Hz, 4H), 2.62 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm): 162.9, 142.5, 133.6, 132.3, 131.4, 127.8, 126.1, 21.9; IR (KBr); 2973, 1787, 1729, 1601, 1460, 1193, 1055, 980 cm^{-1}; HRMS(ESI) calc. for (M + Na^+) 277.0841 found 277.0839.

(2j) 2-ethylbenzoic anhydride



R_f = 0.3 (petroleum ether/ethyl acetate 10:1), Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$,) δ (ppm): 8.02 (d, *J* = 8.1Hz, 2H), 7.56-7.51 (m, 2H), 7.37-7.25 (m, 4H), 3.0 (q, *J* = 7.2Hz, 4H), 1.29 (t, *J* = 7.2Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm): 162.8, 148.4, 133.7, 131.4, 130.7, 127.4, 126.0, 27.6, 15.6; IR (KBr); 2964, 1782, 1723, 1604, 1465, 1229, 1052, 993cm^{-1}; HRMS (ESI) calc. for (M + Na^+) 305.1154 found 305.1157.

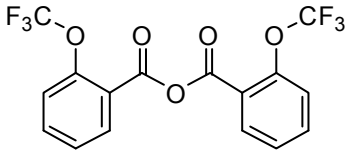
(2k) 2-methoxybenzoic anhydride



R_f = 0.3 (petroleum ether/ethyl acetate 3:1), White solid. m.p = 72-74°C, ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm): 8.00 (d, *J* = 6.9Hz, 2H), 7.55 (d, *J* = 6.7Hz, 2H), 7.06-6.92 (m, 4H), 3.84 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm): 161.9, 160.1, 135.3, 133.0, 120.3,

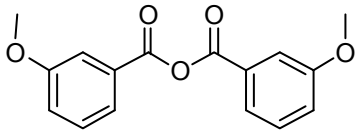
118.1, 112.1, 55.8; IR (KBr); 2950, 1773, 1710, 1604, 1474, 1279, 1171, 1013 cm^{-1}; HRMS (ESI) calc. for (M + Na^+) 309.0739 found 309.0733.

(2l) 2-(trifluoromethoxy)benzoic anhydride



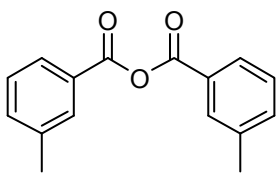
R_f = 0.3 (petroleum ether/ethyl acetate 10:1), white solid. m.p = 96-98°C, ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm): 8.1 (d, *J* = 6.6 Hz, 2H), 7.71-7.64 (m, 4H, 2H), 7.48-7.26 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm): 159.2, 148.4, 135.3, 134.3, 132.9, 127.2, 127.0, 122.8, 122.6, 122.4, 121.9, 118.5; IR(KBr) : 2976, 1690, 1601, 1496, 1457, 1413, 1260, 1154cm^{-1}; HRMS (ESI) calc. for (M + Na^+) 417.0174 found 417.0171.

(2m) 3-methoxybenzoic anhydride



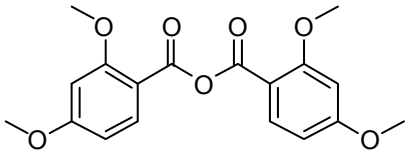
R_f = 0.3 (petroleum ether/ethyl acetate 8:1), White solid. m.p = 65-66 °C, ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm): 7.73 (d, *J* = 7.8 Hz, 2H), 7.67 (s, 2H), 7.4 (t, *J* = 7.8 Hz, 2H), 7.21 (d, *J* = 7.8 Hz, 2H), 3.87 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm): 162.3, 159.8, 130.1, 122.9, 120.9, 115.1, 55.6; IR (KBr): 3008, 1771, 1725, 1609, 1225, 1171, 1035, 1004cm^{-1} ; HRMS (ESI) calc. for (M + Na^+) 309.0739 found 309.0736.

(2n) 3-methylbenzoic anhydride



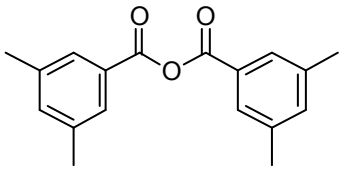
R_f = 0.3 (petro ether/ethyl acetate 10:1), White solid. m.p = 64-66°C, ^{1}H NMR (300 MHz, $CDCl_3$,) δ (ppm): 7.8 (s, 4H), 7.39-7.32 (m, 6H), 2.36 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm): 162.7, 138.8, 135.3, 131.1, 128.8, 127.7, 21.3; IR (KBr): 3010, 1791, 1717, 1589, 1256, 1175, 1039 cm^{-1}; HRMS (ESI) calc. for (M + Na^+) 277.0841 found 277.0839.

(2o) 2,4-dimethoxybenzoic anhydride



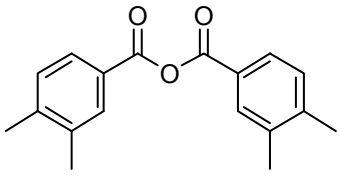
$R_{f=}$ 0.3 (petroleum ether/ethyl acetate 1.5:1), White solid. m.p = 104-106 °C, ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm): 8.0 (d, J = 8.7Hz, 2H), 6.65-46 (m, 4H), 3.89 (s, 6H), 3.82 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm): 165.5, 162.3, 161.6, 135.2, 111.0, 105.2, 98.8, 55.8, 55.5; IR (KBr): 3009, 1775, 1713, 1609, 1225, 1167, 1039, 1000 cm^{-1}; HRMS (ESI) calc. for ($M + Na^{+}$) 369.0950 found 309.0945.

(2p) 3,5-dimethylbenzoic anhydride



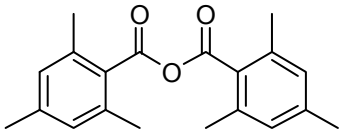
R_f = 0.3 (petroleum ether/ethyl acetate 15:1), White solid. m.p = 173-176 °C, ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm): 7.68 (s, 4H), 7.24 (s, 2H), 2.33 (s, 12H); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm): 163.0, 138.6, 136.2, 128.8, 128.3, 21.2, 21.1; IR (KBr): 3010, 1779, 1717, 1609, 1225, 1171, 1043, 1000 cm^{-1}; HRMS (ESI) calc. for ($M + Na^{+}$) 305.1154 found 305.1153.

(2q) 3,4-dimethylbenzoic anhydride



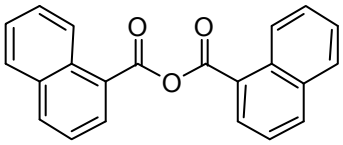
R_f = 0.3 (petroleum ether/ethyl acetate 15:1), White solid. m.p =120-121 °C, ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm): 7.8 (m, 4H), 7.2 (s, 2H), 2.3 (s, 12H); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm): 162.9, 144.2, 137.3, 131.5, 130.1, 128.1, 126.5, 20.2, 19.7; IR (KBr): 3010, 1771, 1713, 1612, 1244, 1206, 1120, 1051cm^{-1}; HRMS (ESI) calc. for ($M + Na^{+}$) 305.1154 found 305.1150.

(2r) 2,4,6-trimethylbenzoic anhydride



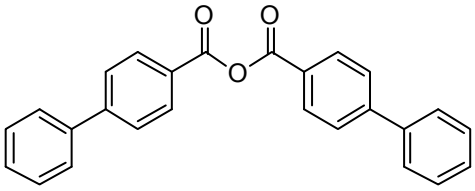
R_f= 0.3 (petroleum ether/ethyl acetate 20:1), White solid. m.p = 102-104 °C, ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm): 6.87 (s, 4H) 2.39 (s, 12 H).2.28 (s, 6 H), ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm): 166.3, 140.7, 136.3, 128.8, 21.1, 20.1; IR (KBr): 3010, 1779, 1713, 1609, 1221, 1171, 1039, 1004 cm^{-1}; HRMS (ESI) calc. for ($M + Na^{+}$) 333.1467 found 333.1462.

1-Naphthoic Anhydride (2s)



R_f = 0.3 (petro ether/ethyl acetate 6:1), white solid. m.p = 142-144 °C, ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 9.1 (d, *J* = 8.6Hz, 2 H), 8.4 (d, *J* = 4.5Hz, 2 H), 8.1 (d, *J* = 8.1Hz, 2 H), 7.9 (d, *J* = 7.9Hz, 2 H), 7.5–7.7 (m, 6 H); ^{13}C NMR (100MHz, $CDCl_3$) δ (ppm): 162.9, 135.5, 134.0, 132.1, 128.8, 129.3, 127.2, 126.8, 126.2, 125.2, 124.9; IR (KBr): 3054, 1771, 1705, 1593, 1225, 1171, 1054, 958 cm^{-1}; HRMS (ESI) calc. for (M + Na^+) 349.0841 found 349.0838.

4-Phenylbenzoic anhydride (2t)



R_f = 0.3 (petroleum ether/ethyl acetate 5:1), white solid. m.p = 137-139 °C, ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 8.2 (d, *J* = 7.4 Hz, 4H), 7.7(d, *J* = 7.9Hz, 4H), 7.6 (d, *J* = 7.2Hz, 4H), 7.4 (m, *J* = 7.3Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 162.4, 147.4, 139.6, 131.2, 129.1, 128.6, 127.7, 127.6, 127.4; IR (KBr): 3010, 1770, 1730, 1600, 1244, 1175, 1050, 961 cm^{-1}; HRMS (ESI) calc. for (M + Na^+) 401.1154 found 401.1152.

6. References available for known products

No.	*Products*	*Table and entry*	*Reference*
1	benzoic anhydride		[S 1]
			[S 7]
2	4-methoxybenzoic anhydride		[S 1]
			[S 5]
			[S 7]
3	4-methylbenzoic anhydride		[S 1]
4	4-fluorobenzoic anhydride		[S 3]
5	4-chlorobenzoic anhydride		[S 5]
6	2-methylbenzoic anhydride		[S 4]
7	2-methoxybenzoic anhydride		[S 2]
8	2-chlorobenzoic anhydride		[S 4]
			[S 7]
9	2-ethylbenzoic anhydride		NA
10	3-methylbenzoic anhydride		NA

11	3-methoxybenzoic anhydride	[S 2]
12	2-(trifluoromethoxy)benzoic anhydride	NA
13	4-(trifluoromethoxy)benzoic anhydride	NA
14	3,5-dimethylbenzoic anhydride	[S 6]
15	3,4-dimethylbenzoic anhydride	NA
16	2,4-dimethoxybenzoic anhydride	NA
17	2,4,6-trimethylbenzoic anhydride	[S 4]
18	4-Phenylbenzoic anhydride	[S 5]
19	1-Naphthoic Anhydride	[S 4]

NA: Not available;

NO: No reference was found via SCIfinder.

References:

[S1] Ilirian, D.; SantaLucia, J.; *Org. Lett.*, **2006**, *8*, 47–50.

[S2] Warren, J. L.; Wood, A. W.; Patterson, H. T.; Rishi K. J.; Jonathan A. E.; *J. Am. Chem. Soc.* **2005**, *127*, 15521–15527.

[S3] Neves F.; Ricardo A. W.; *J. Braz. Chem. Soc.* **2007**, *18,* 1410-1414.

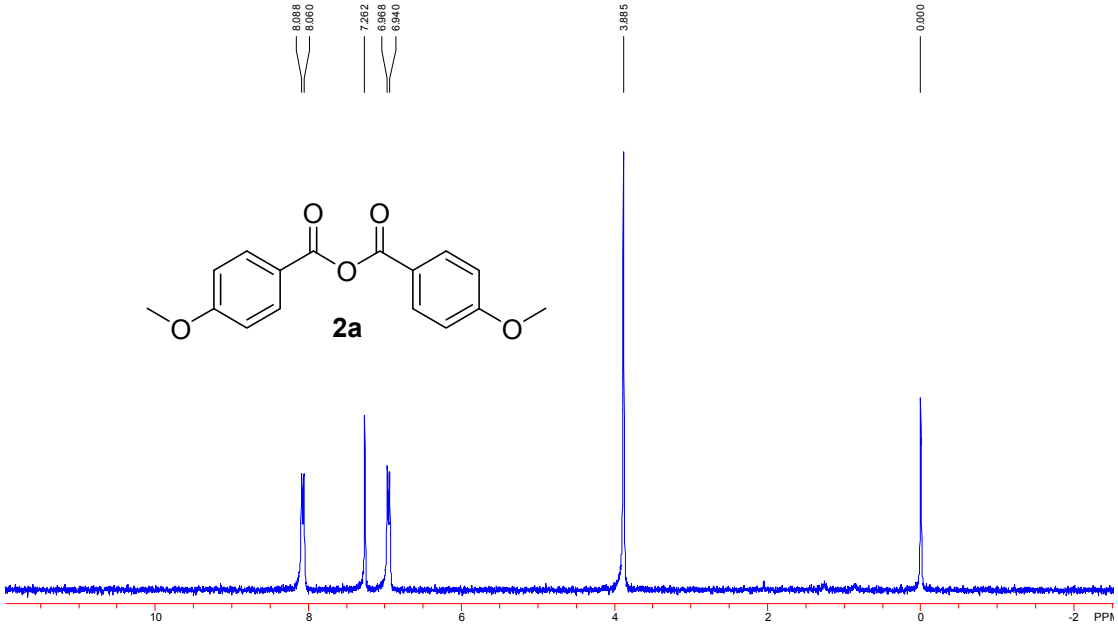
[S4] Foad. k.; Hashem S.; Mohammad A. N.; *Synthesis,* **2004**, *2*, 205–207.Sy

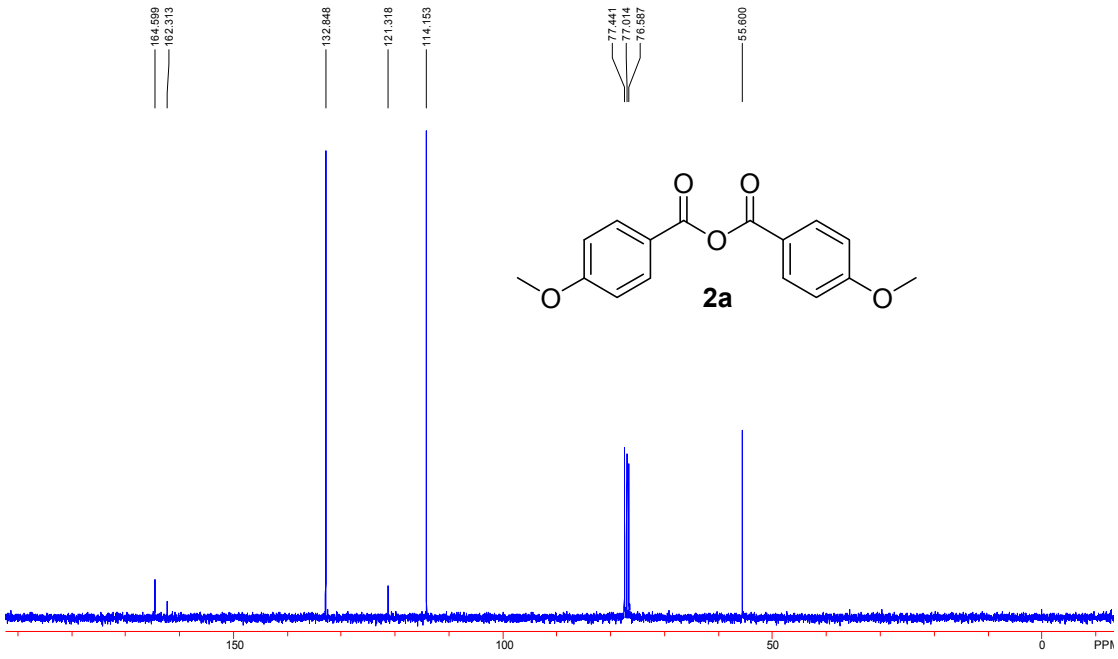
[S5] Yong, D. P.;Jeum-Jong K.; Ho-Kyun K.; *Synth. Commun.*, **2005**, *35*, 371–378.

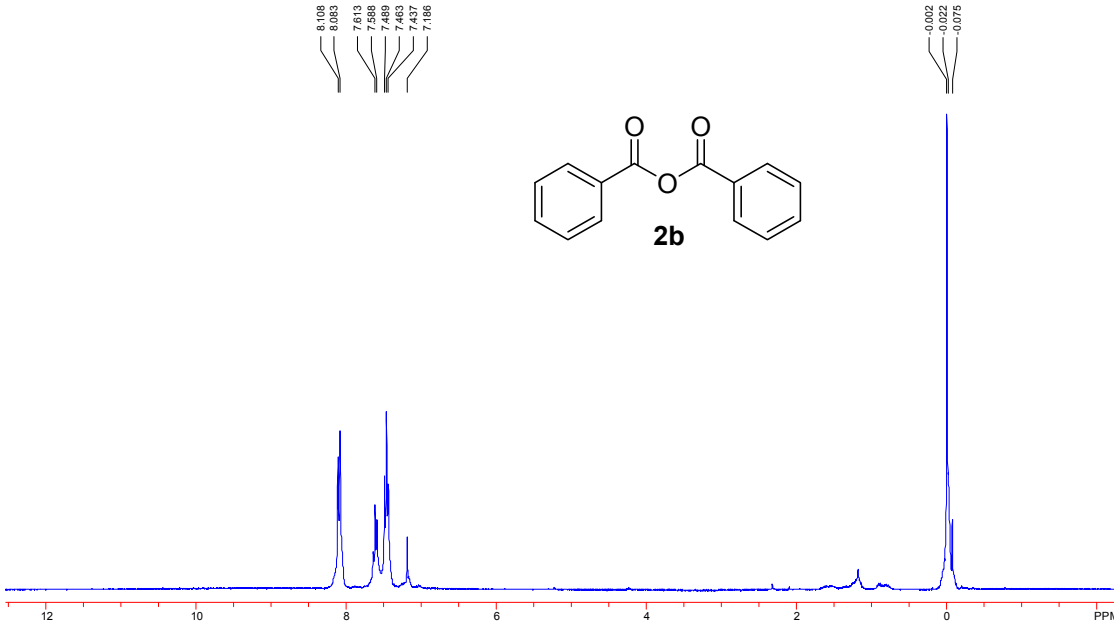
[S6] Yuya T.; Hiroaki K.; Nobuhiro K.; Toshikazu, T.; *J. Org. Chem.*, **2006**, *71*, 5093–5104.

[S7] Zbigniew J. K.; Beata K.; Marcinkowska M.; *Synth. Commun.*, **2004**, *34,* 3349–3358.

7. Traces of the ^{1}H NMR and ^{13}C NMR spectra of products



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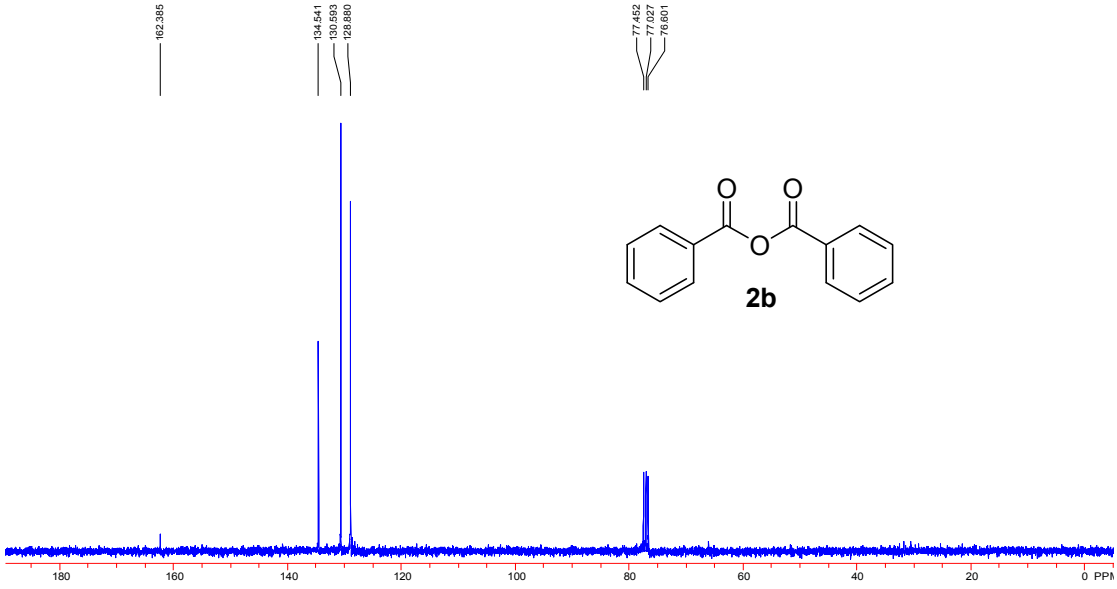
8.083
7.613
7.588
7.489
7.463
7.437
7.186
-0.002
-0.022
-0.075
O
O
O
2b
12
10
8
6
4
2
0
PPM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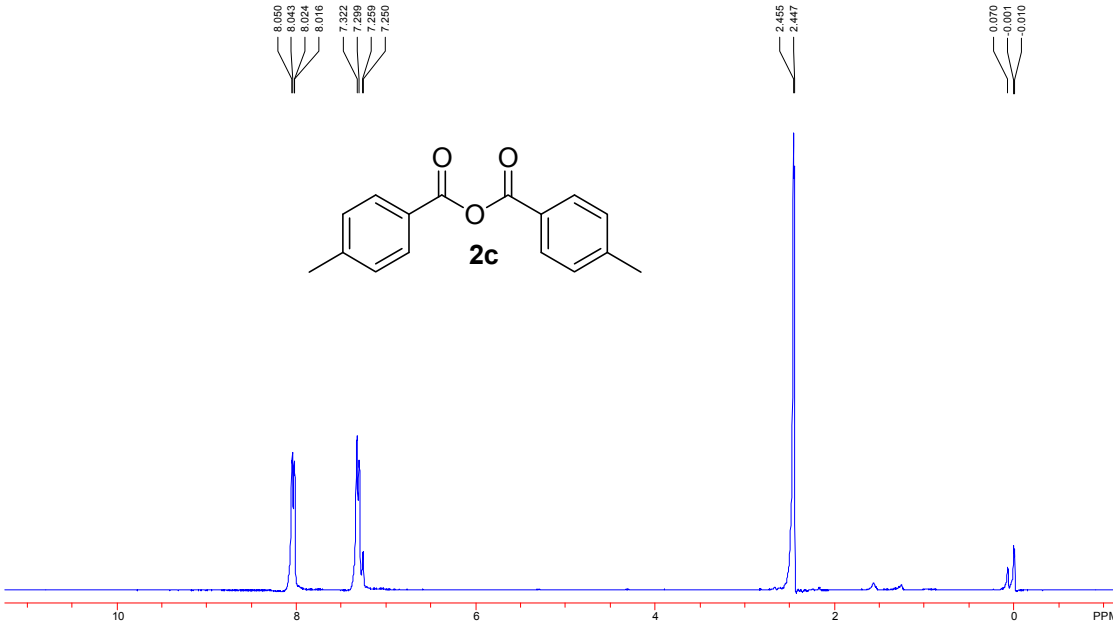
134.541
130.593
128.880
77.452
77.027
76.601
O
O
O
2b
180
160
140
120
100
80
60
40
20
0 PPM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8.024
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7.322
7.299
7.259
7.250
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2.447
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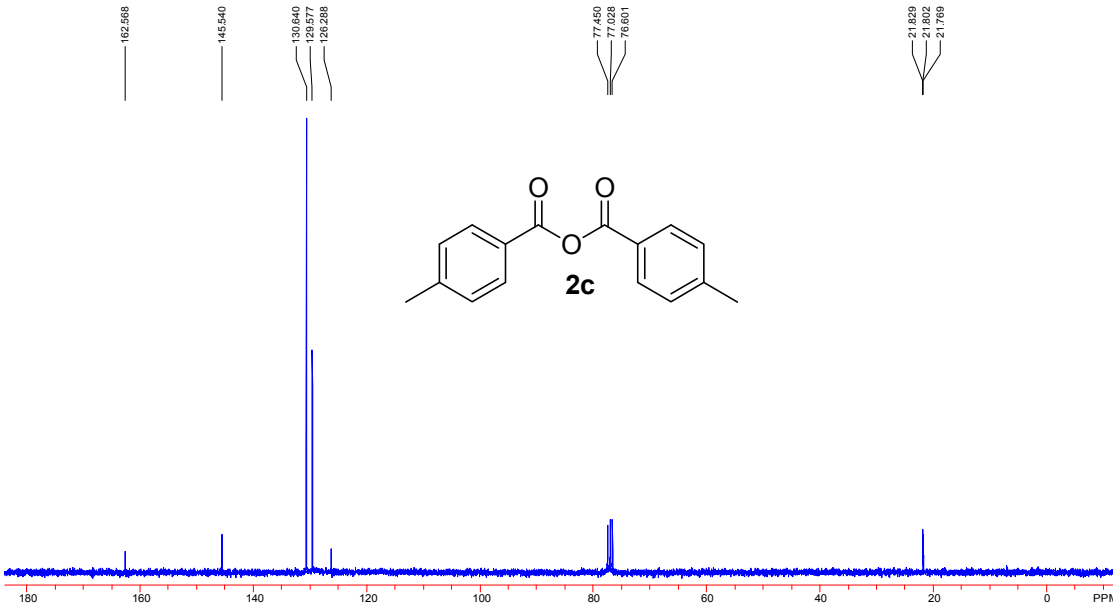
0.001
-0.010
2c
10
8
6
4
2
0
PPM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145.540
130.640
129.577
126.288
77.450
77.028
76.601
21.829
21.802
21.769
2c
180
160
140
120
100
80
60
40
20
0
PPM


8.006
7.977
7.491
7.464
4.872
3.308
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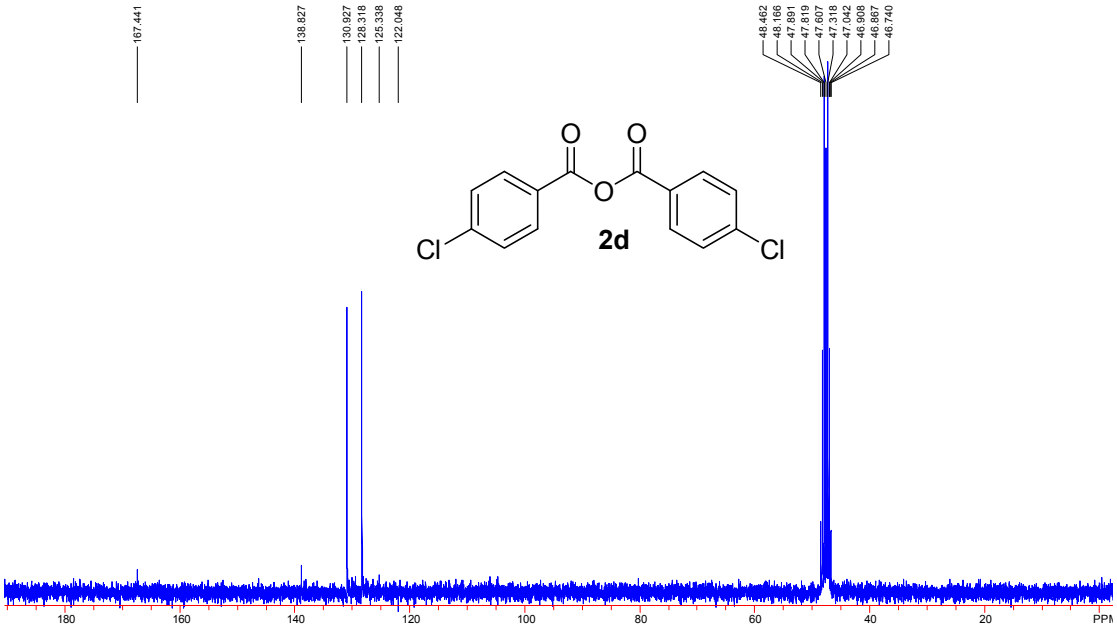
O
O
O
Cl
Cl
2d
10
8
6
4
2
0
-2
PPM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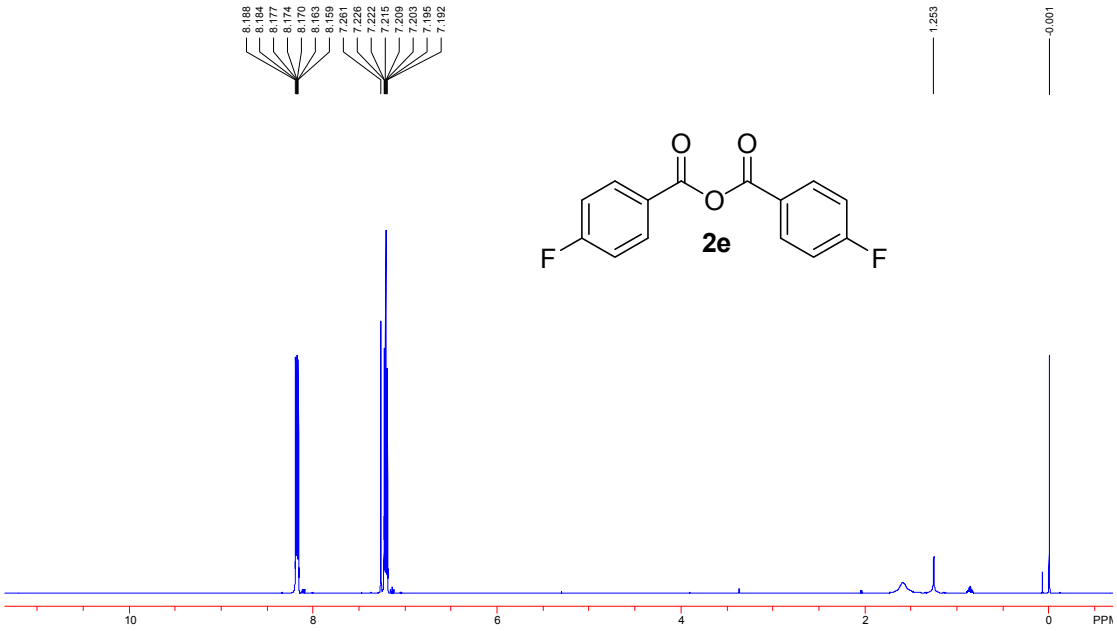
138.827
130.927
128.318
125.338
122.048
48.462
48.166
47.891
47.819
47.607
47.318
47.042
46.908
46.867
46.740
O
O
O
Cl
Cl
2d
180
160
140
120
100
80
60
40
20
PPM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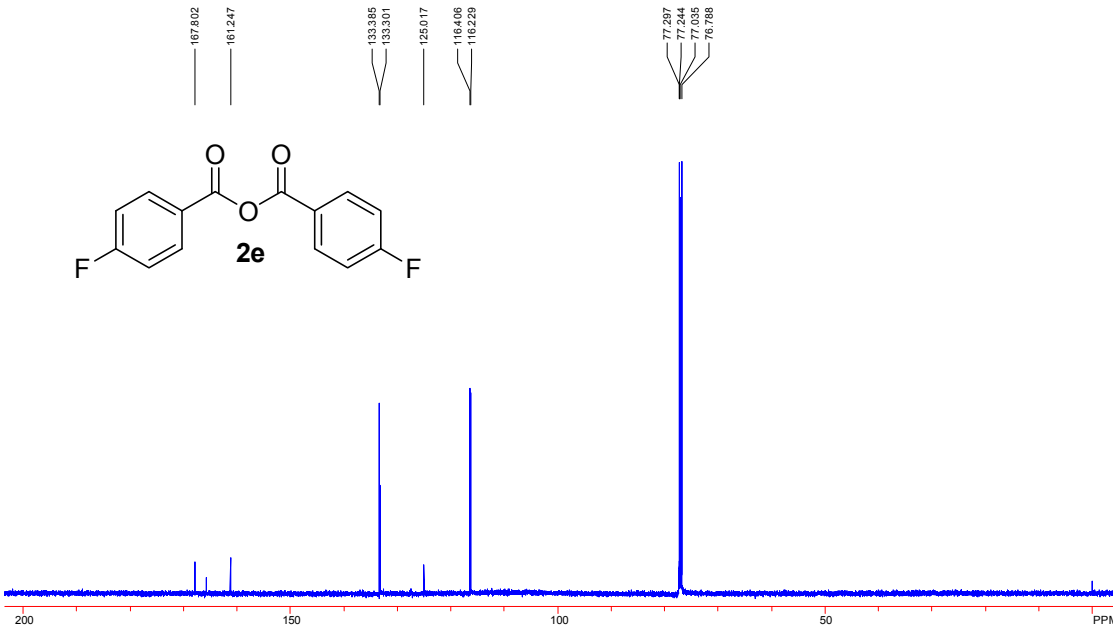
8.184
8.177
8.174
8.170
8.163
8.159
7.261
7.226
7.222
7.215
7.209
7.203
7.195
7.192
1.253
0.001
O
O
O
F
F
2e
10
8
6
4
2
0
PPM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161.247
133.385
133.301
125.017
116.406
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77.244
77.035
76.788
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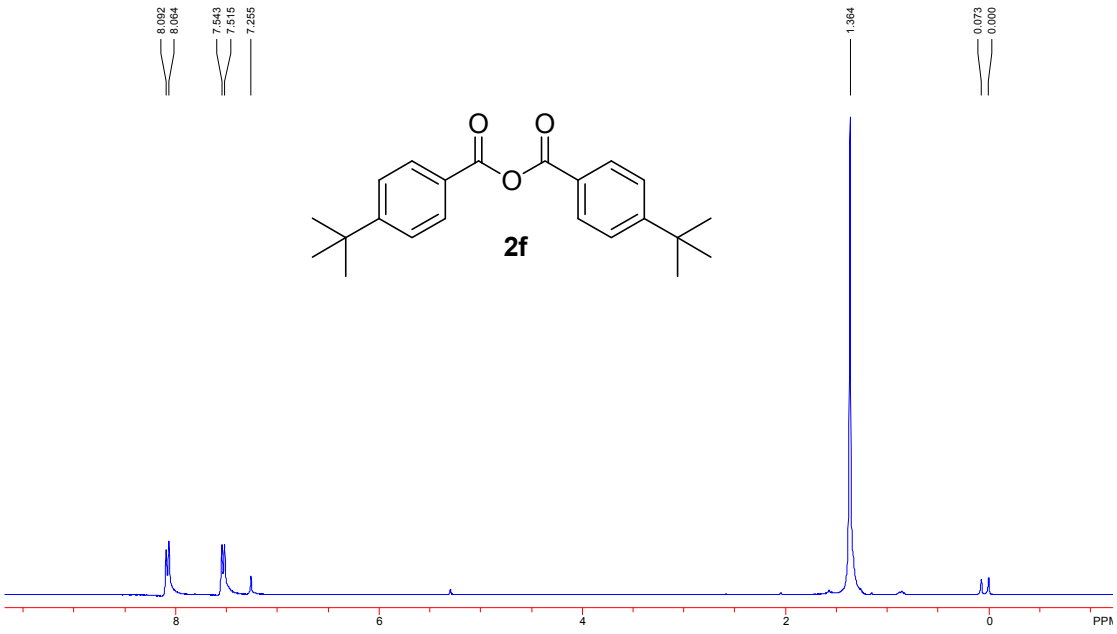
O
O
F
F
2e
200
150
100
50
PPM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0.073
0.000
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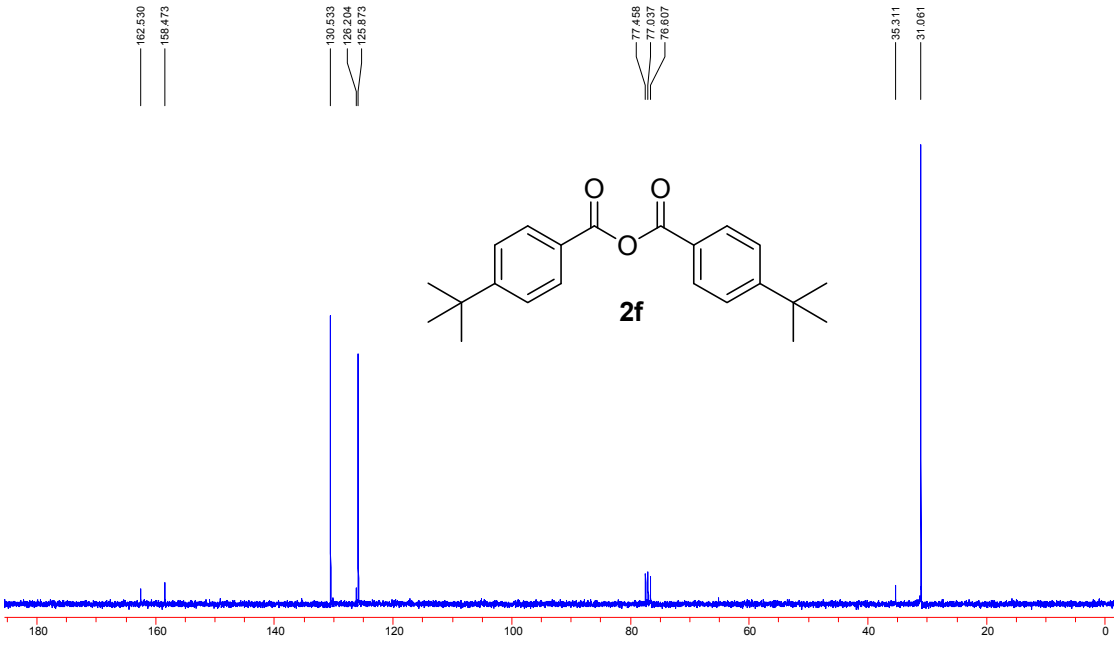
2f
8
6
4
2
0
PPM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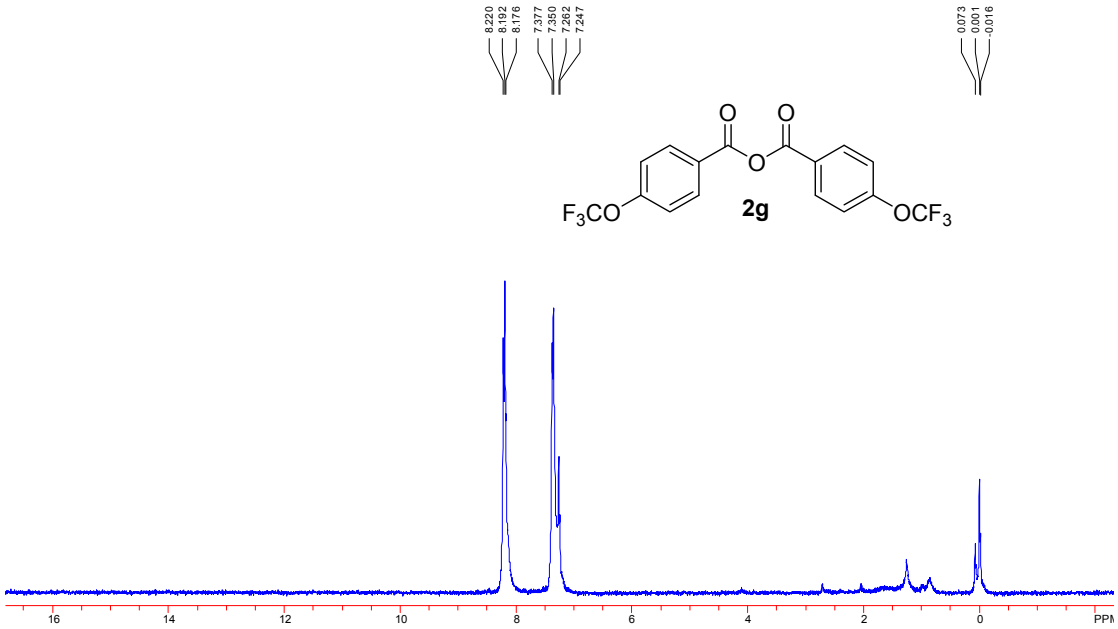
130.533
126.204
125.873
77.458
77.037
76.607
35.311
31.061
O
O
O
2f
180
160
140
120
100
80
60
40
20
0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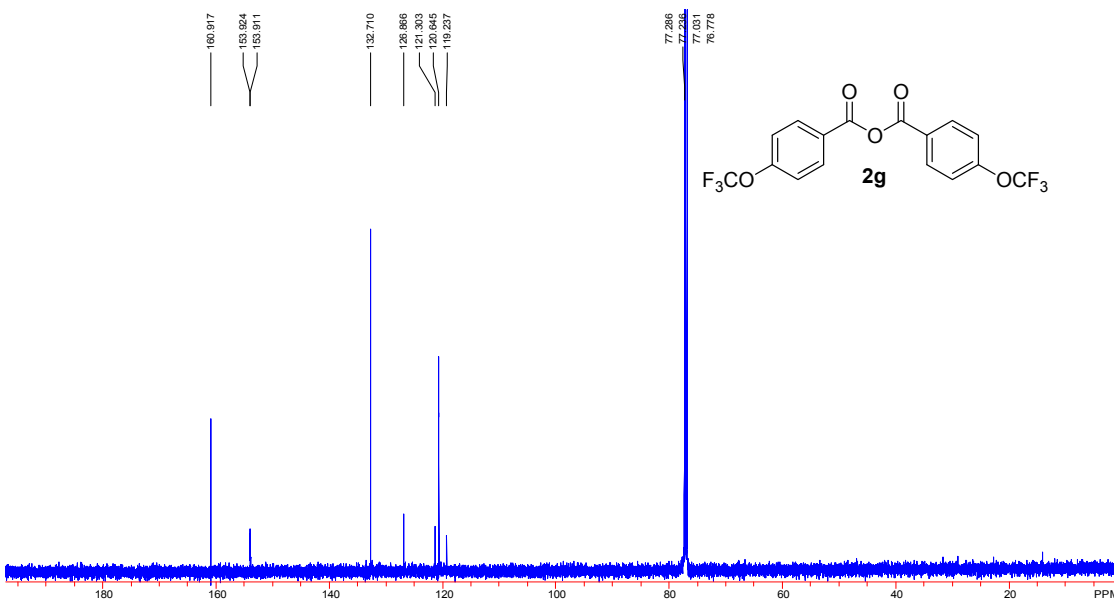
8.192
8.176
7.377
7.350
7.262
7.247
0.073
0.001
-0.016
O
O
O
F3CO
OCF3
2g
16
14
12
10
8
6
4
2
0
PPM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153.924
153.911
132.710
126.866
121.303
120.645
119.237
77.286
77.236
77.031
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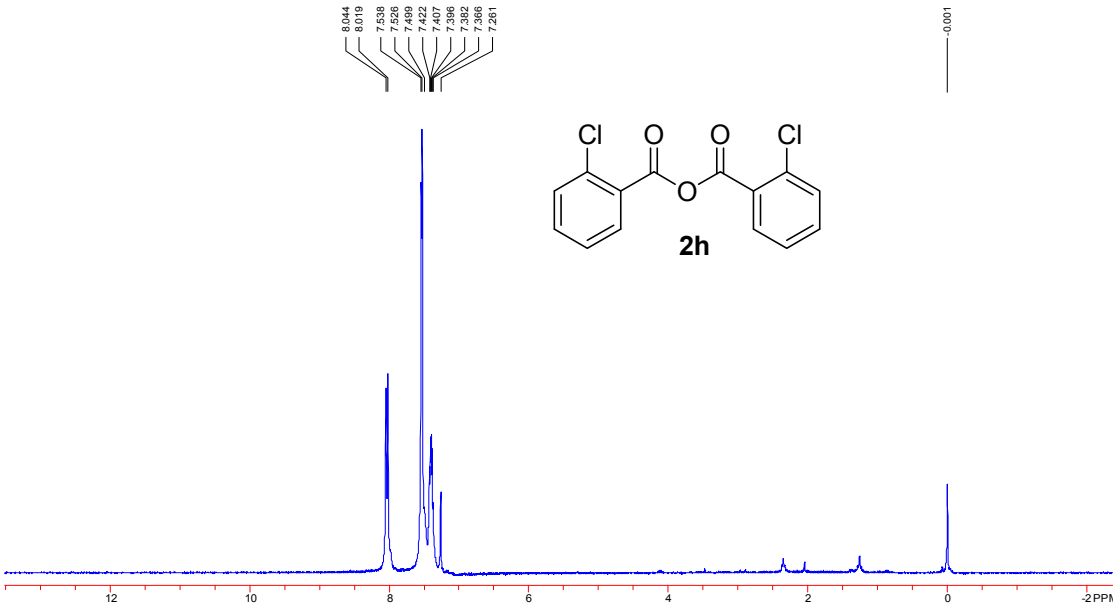
O
O
F3CO
OCF3
2g
180
160
140
120
100
80
60
40
20
PPM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Cl
O
O
Cl
O
2h
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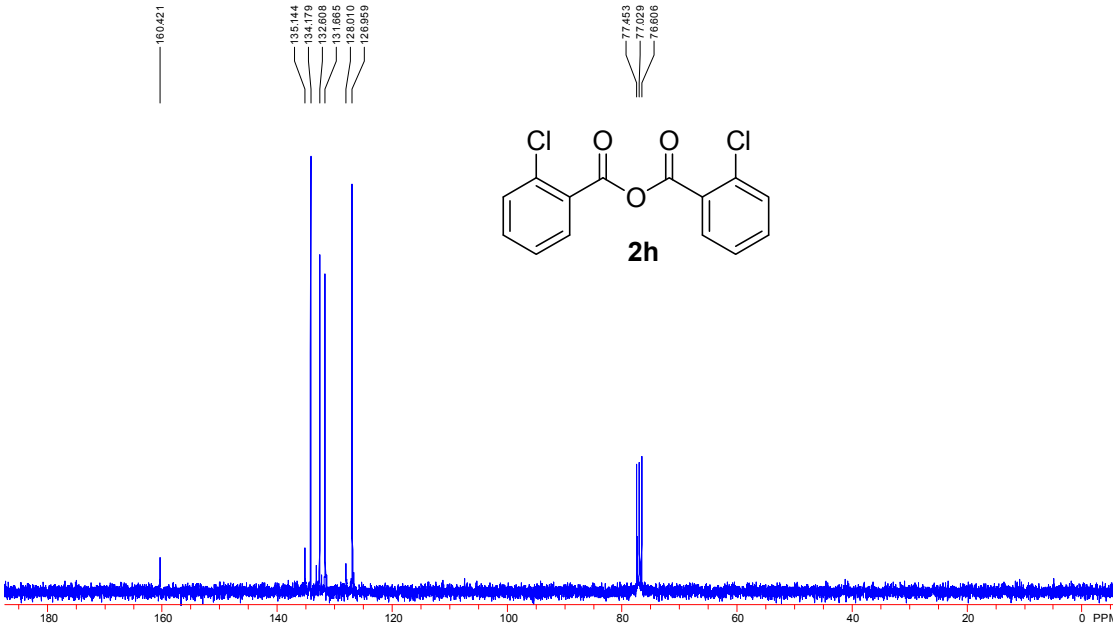
10
8
6
4
2
0
-2 PPM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128.010
126.959
77.453
77.029
76.606
Cl
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O
Cl
O
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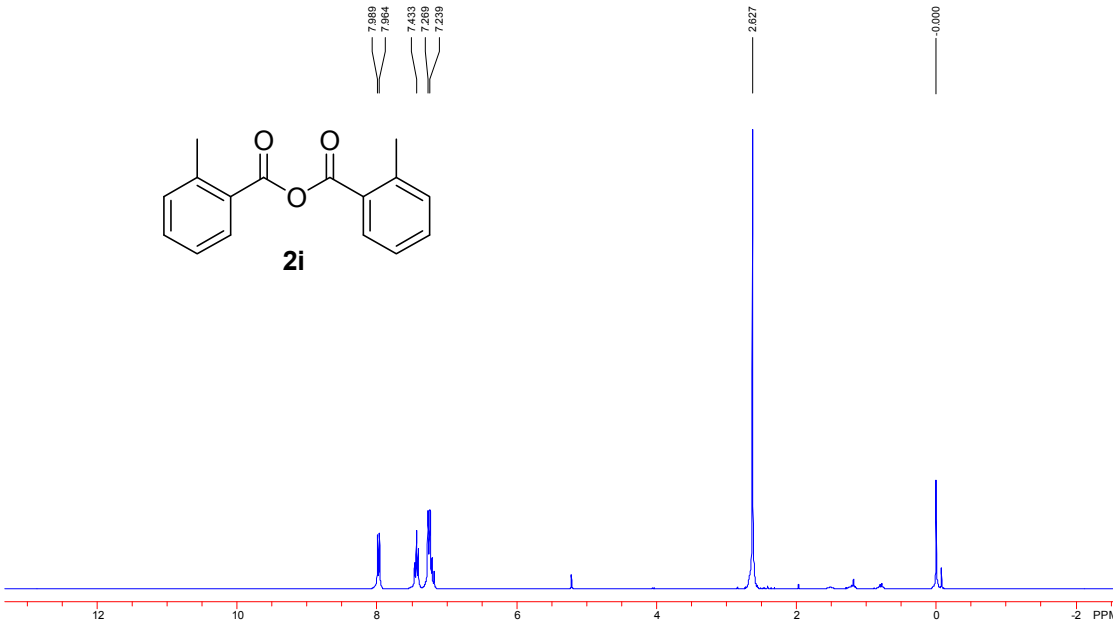
140
120
100
80
60
40
20
0 PPM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8
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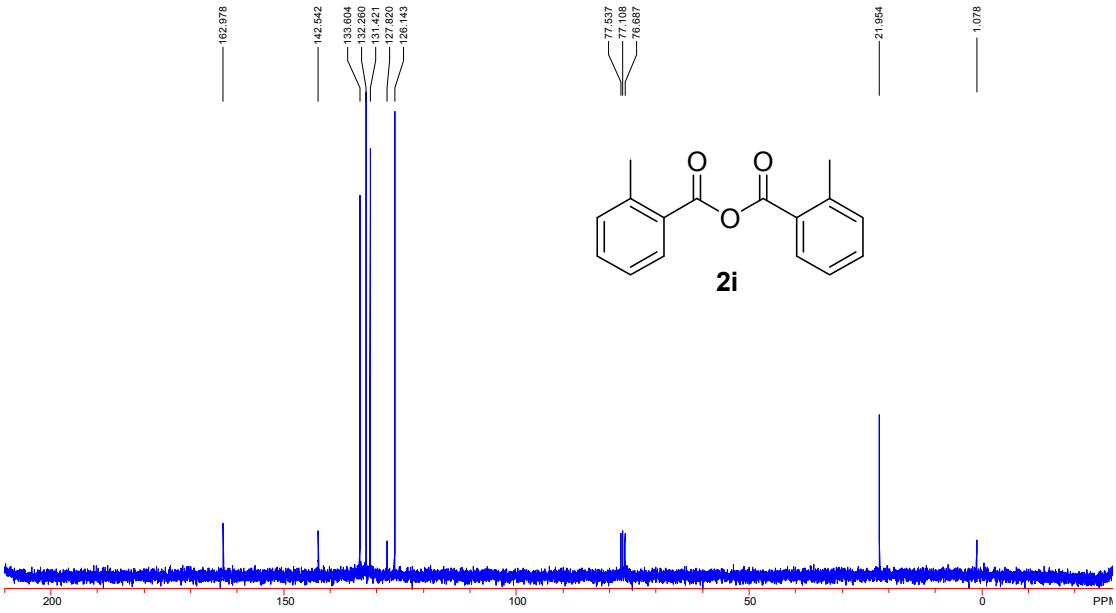
PPM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126.143
77.537
77.108
76.687
21.954
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O
O
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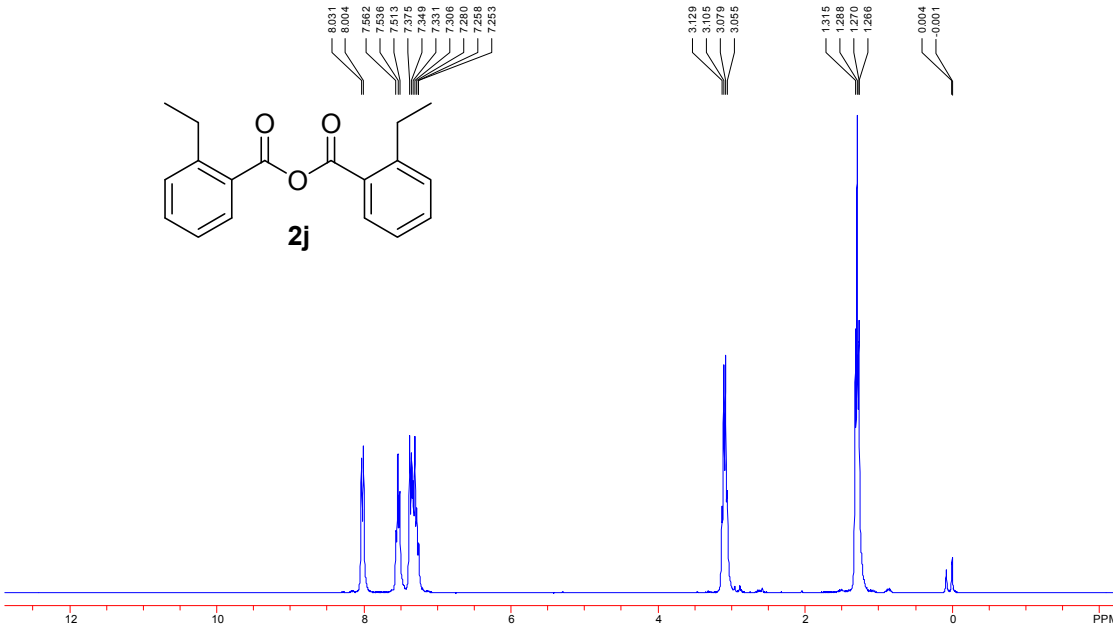
200
150
100
50
0
PPM

\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7.375
7.349
7.331
7.306
7.280
7.258
7.253
3.129
3.105
3.079
3.055
1.315
1.288
1.270
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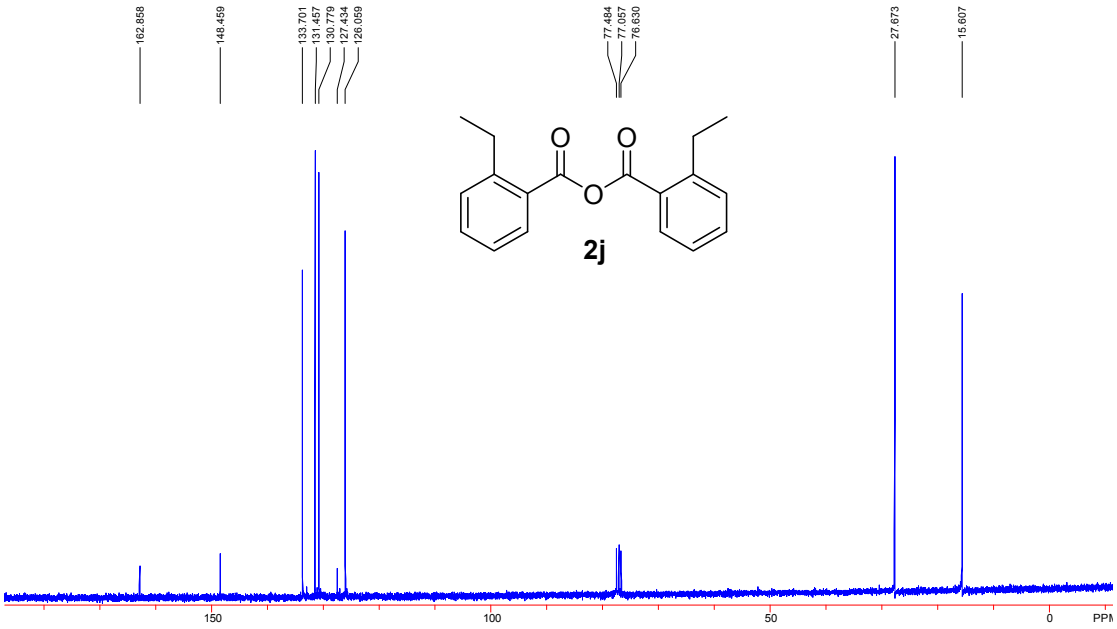
0.004
-0.001
O
O
O
2j
12
10
8
6
4
2
0
PPM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O
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50
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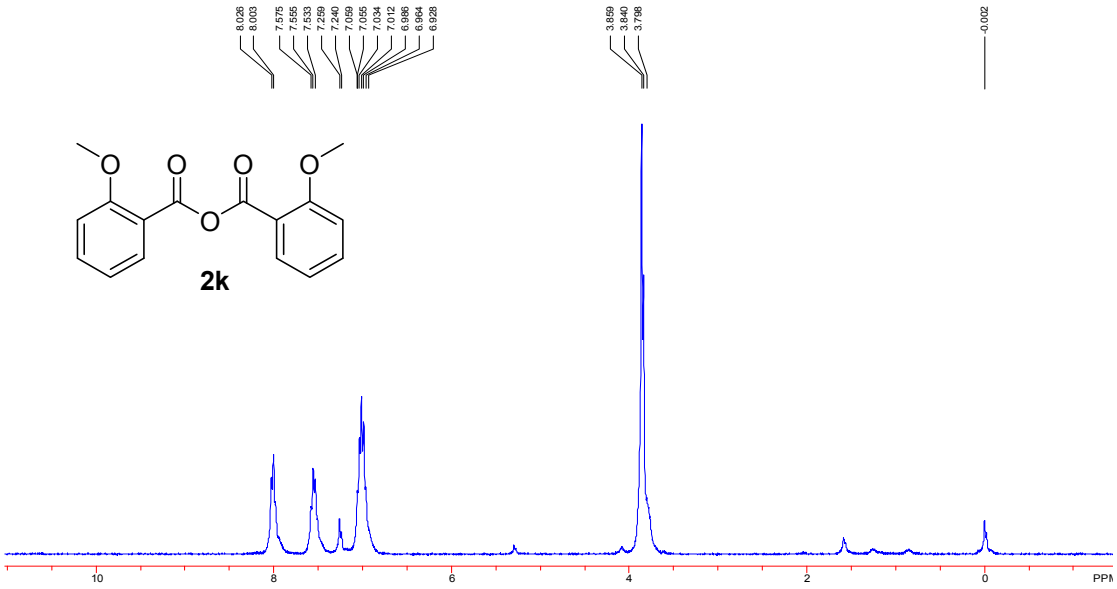
PPM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7.553
7.239
7.240
7.099
7.095
7.034
7.012
6.986
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3.798
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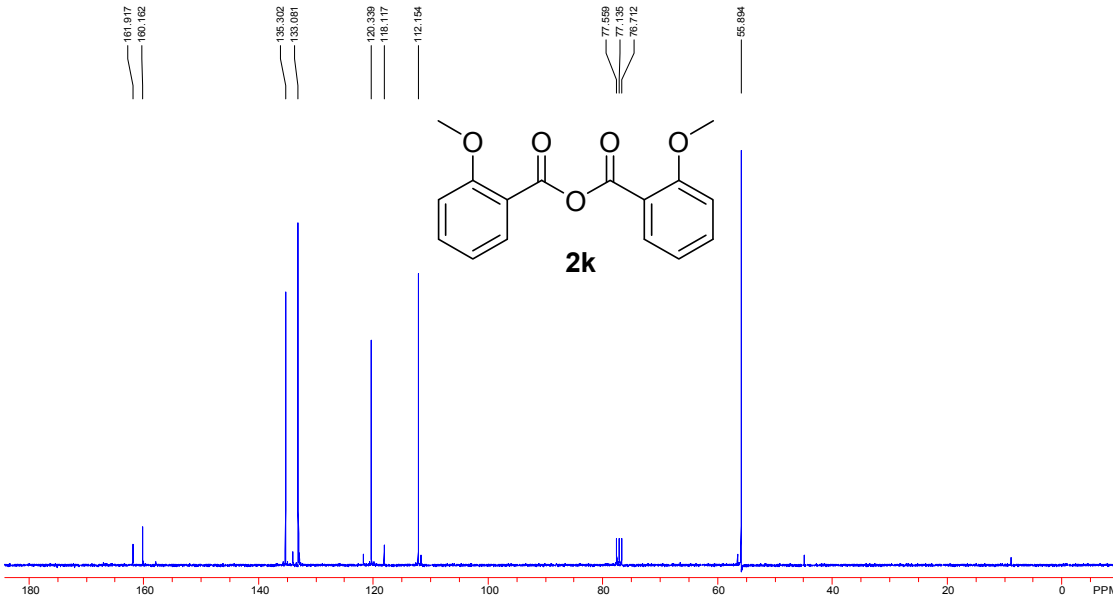
10
8
6
4
2
0
PPM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76.712
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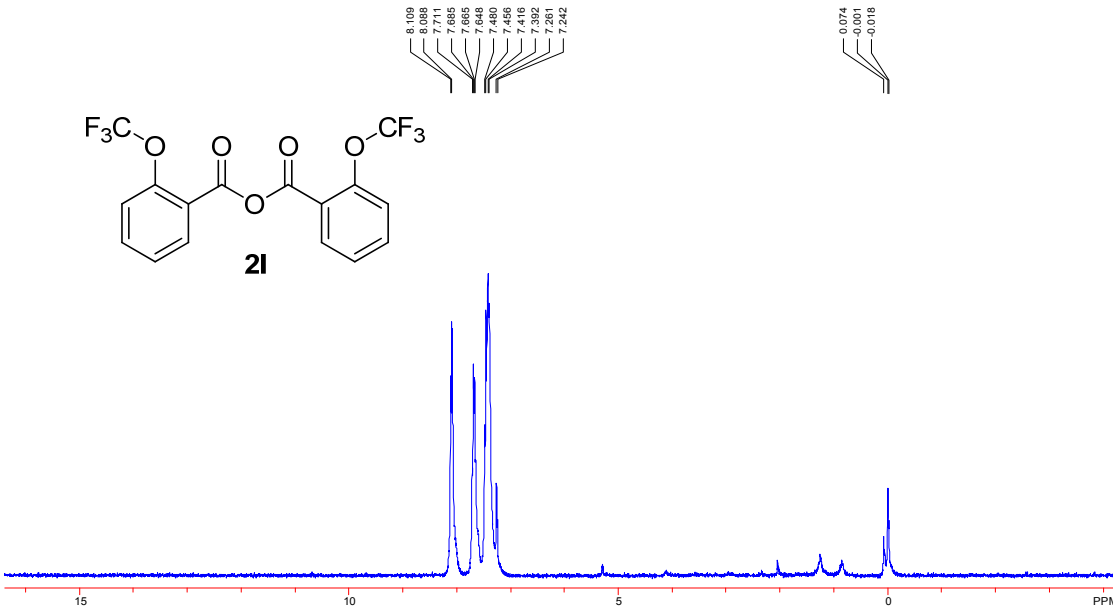
120
100
80
60
40
20
0
PPM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7.456
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7.392
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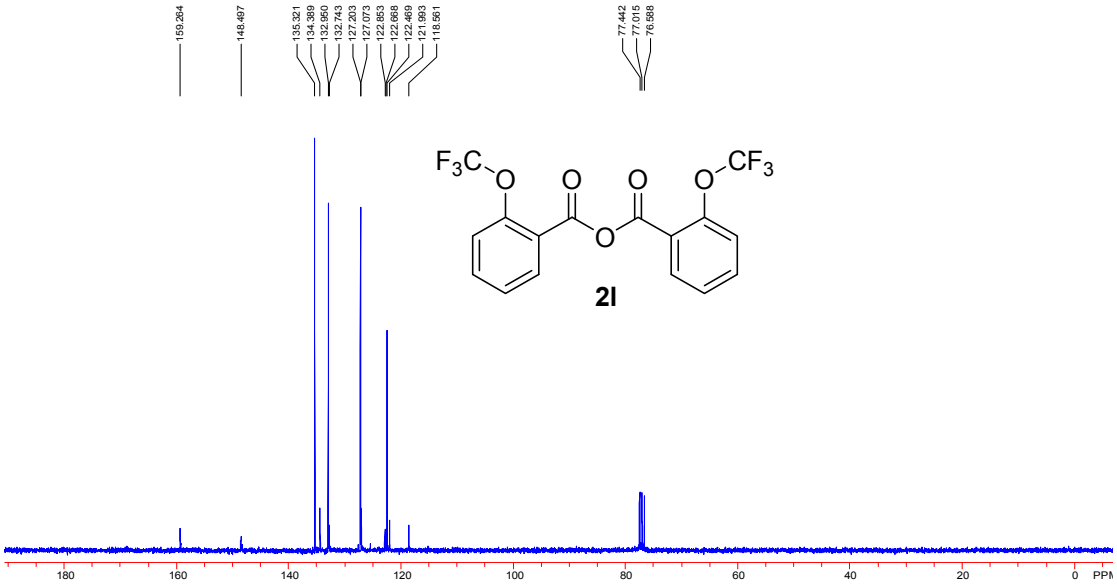
PPM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77.015
76.588
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CF3
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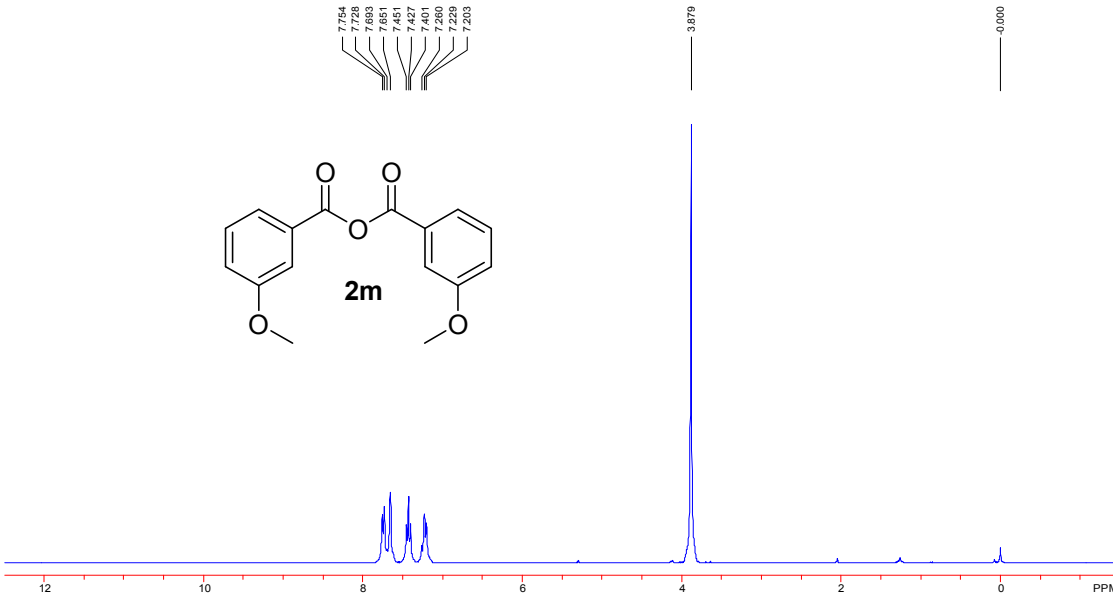
0
PPM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2
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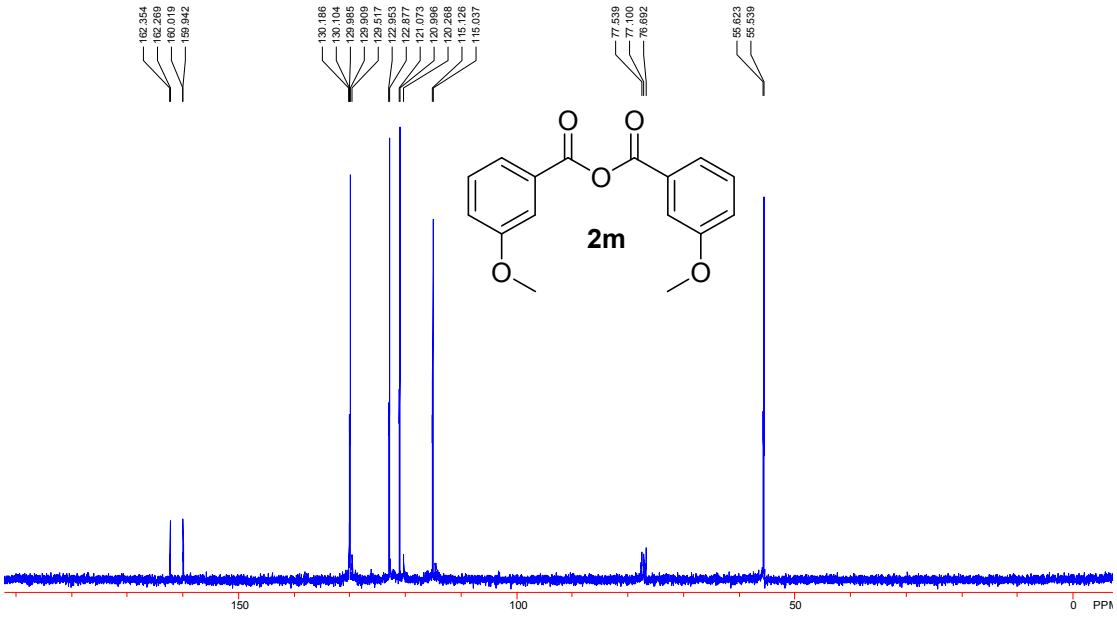
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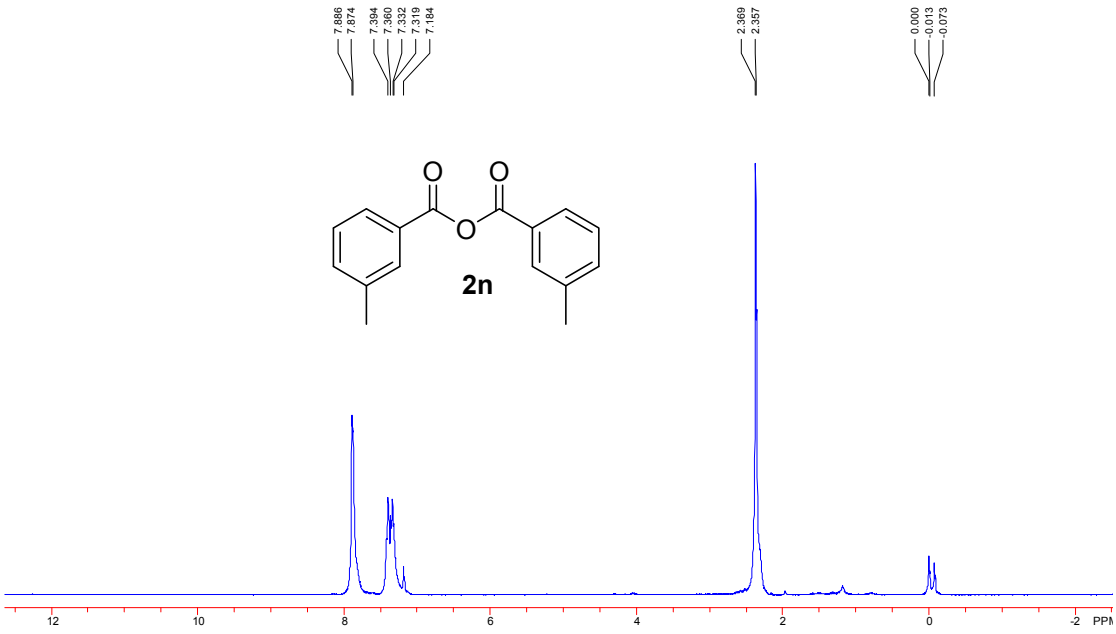
0
PPM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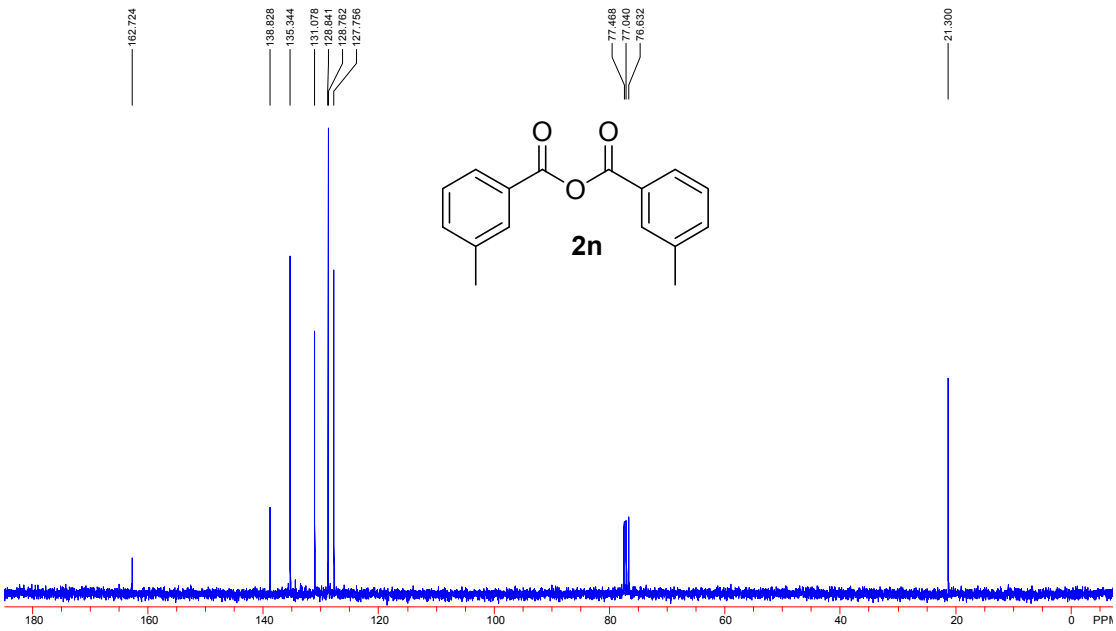
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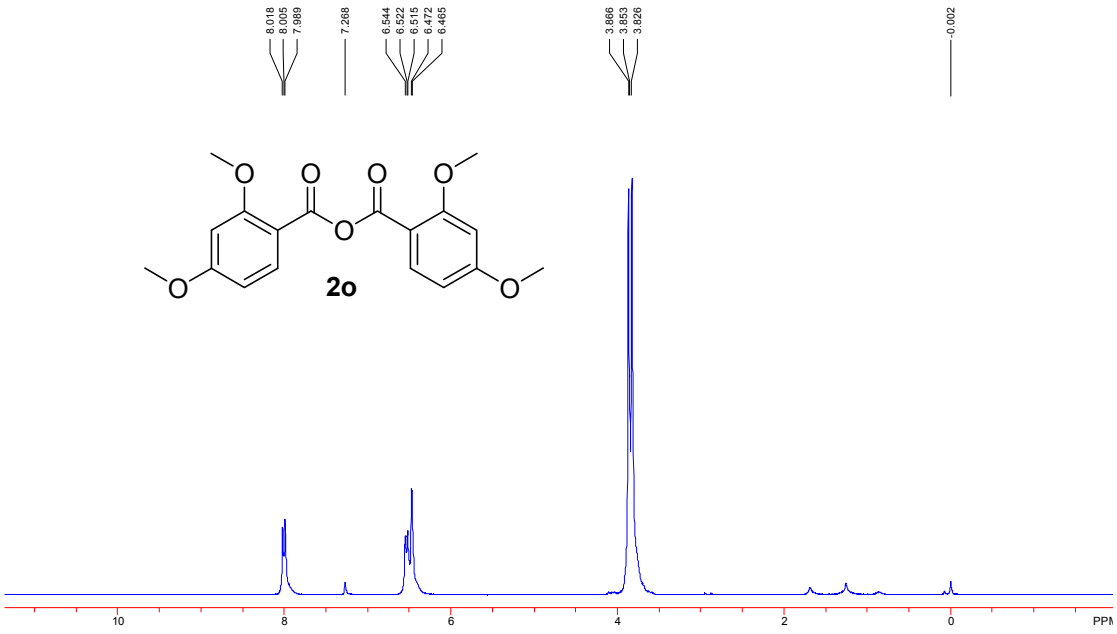
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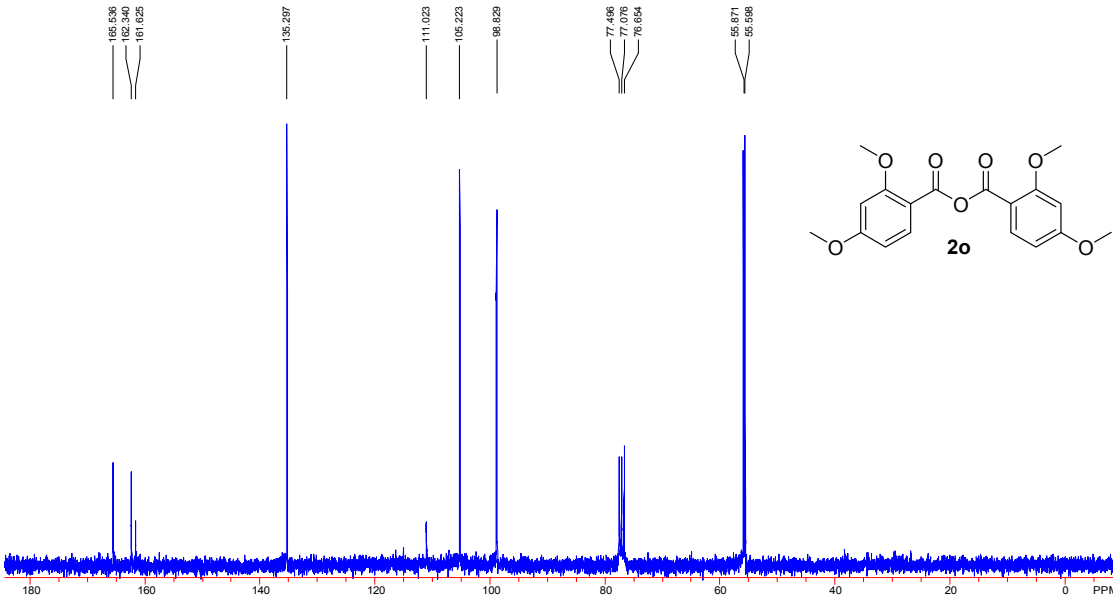
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120
100
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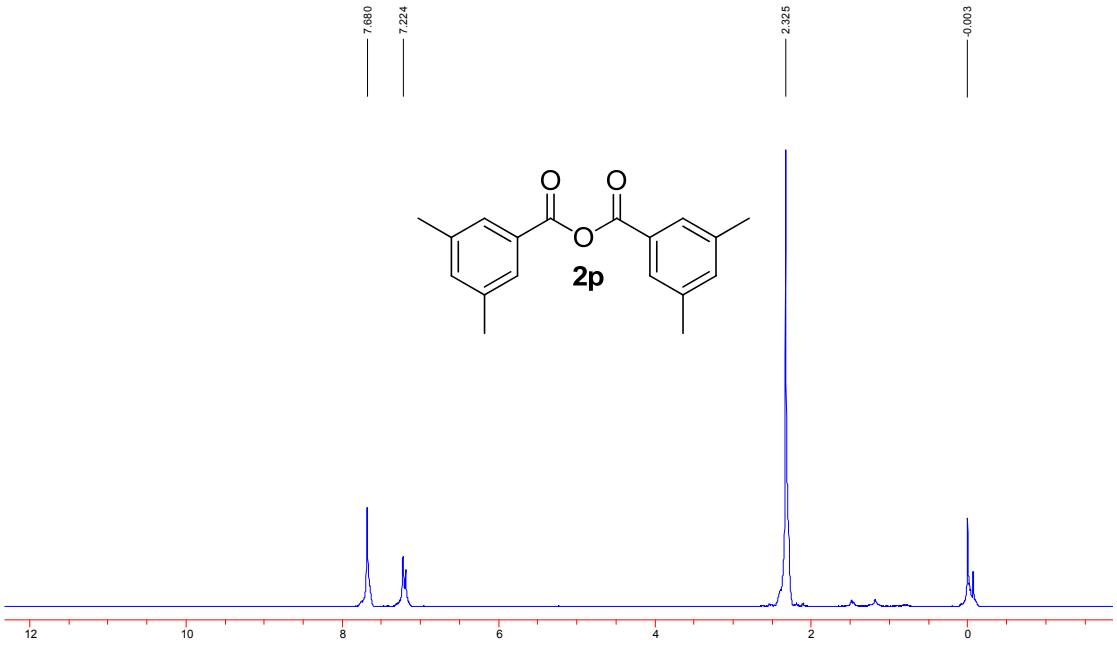
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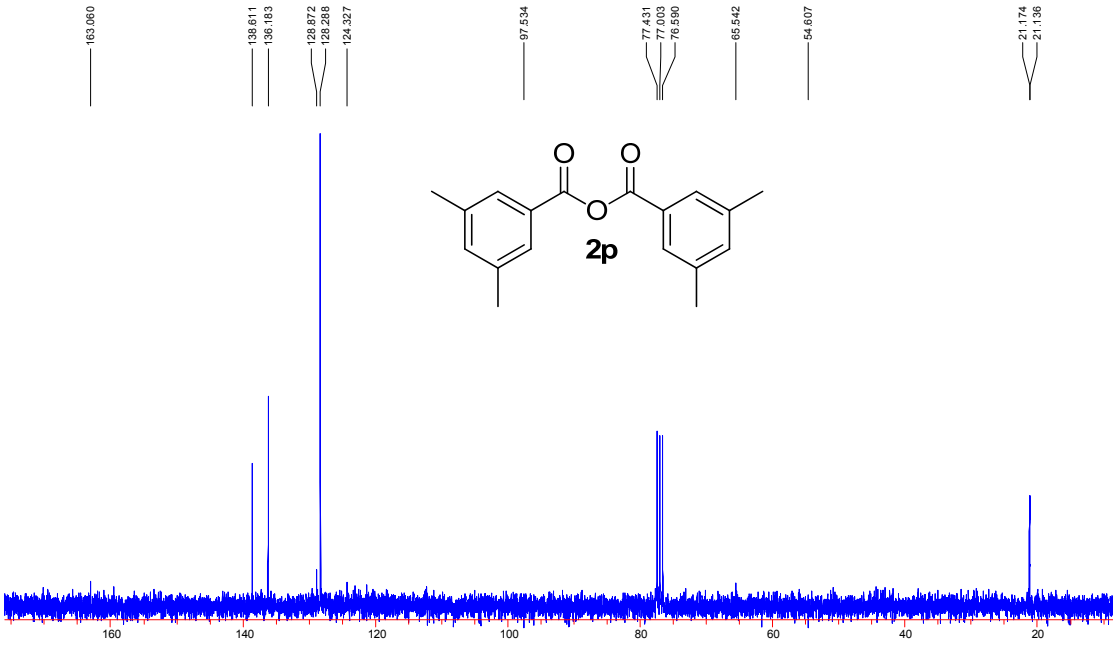
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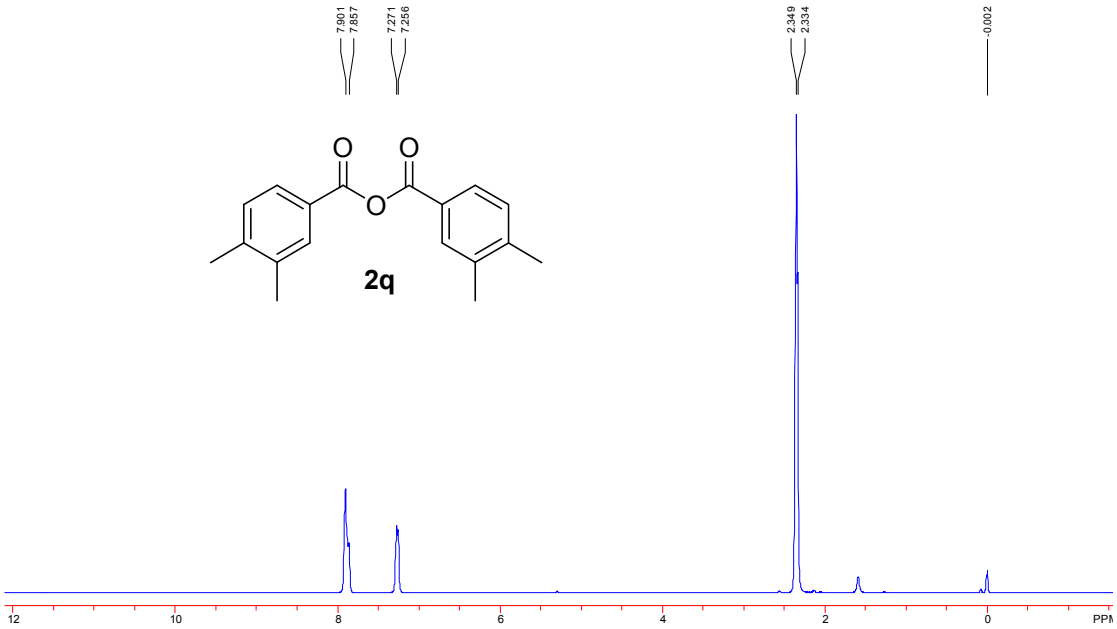
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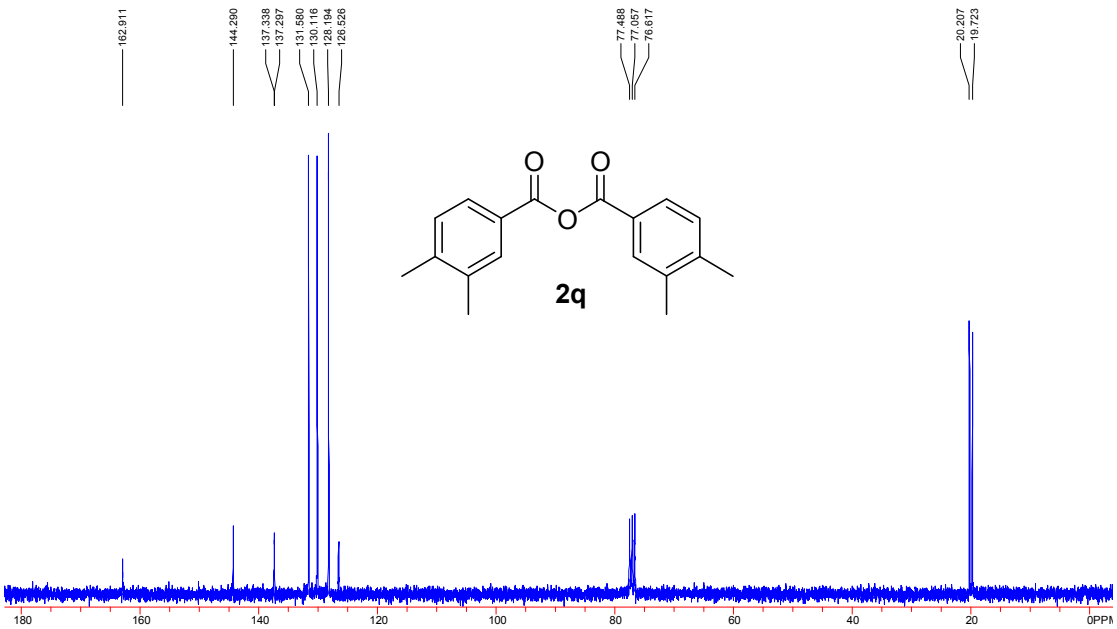
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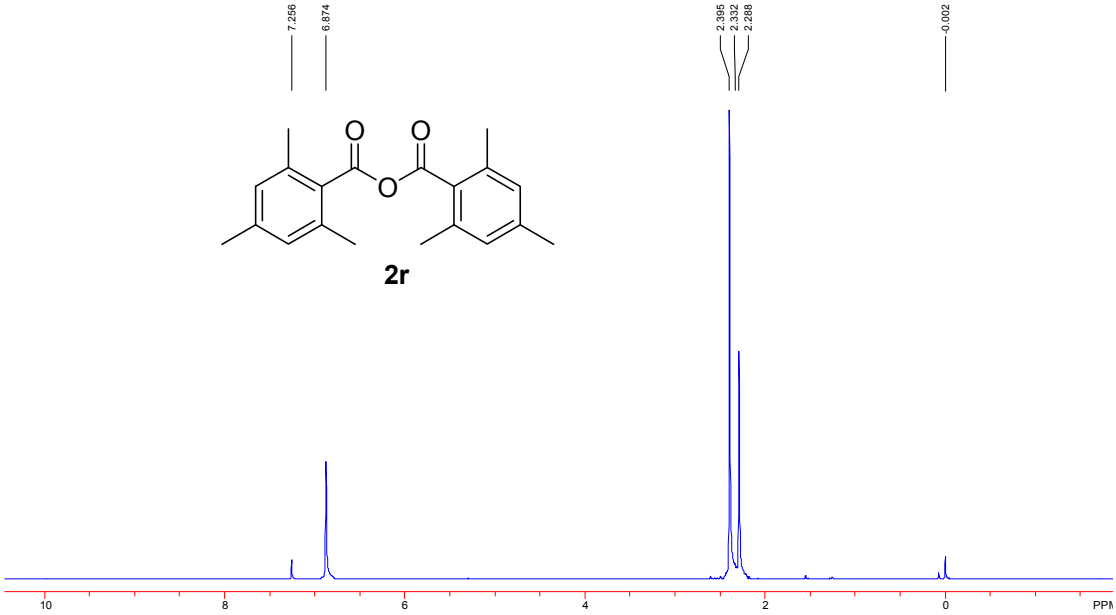
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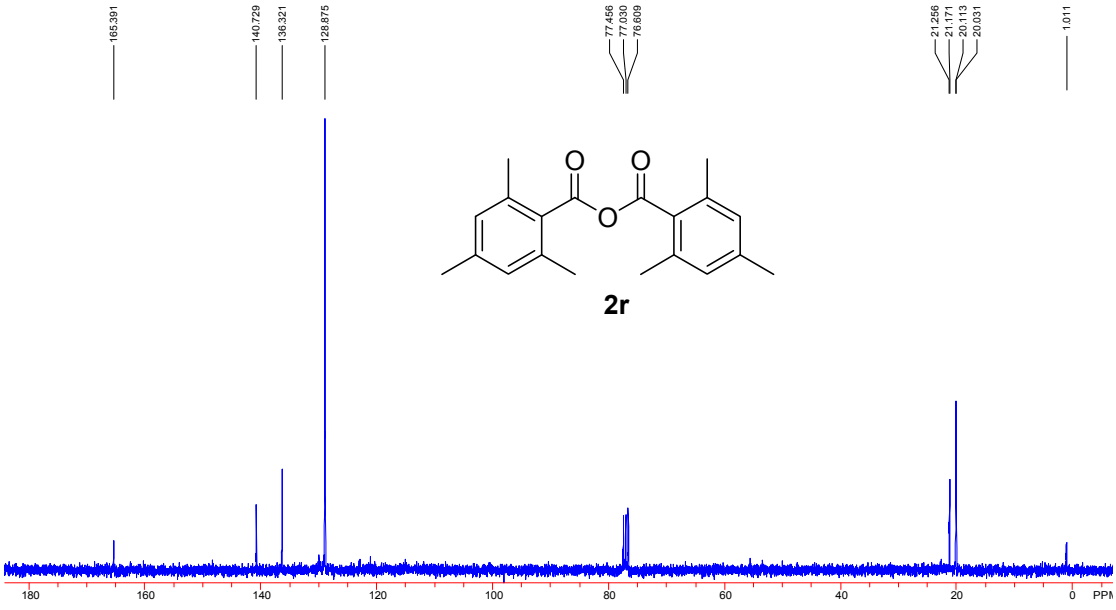
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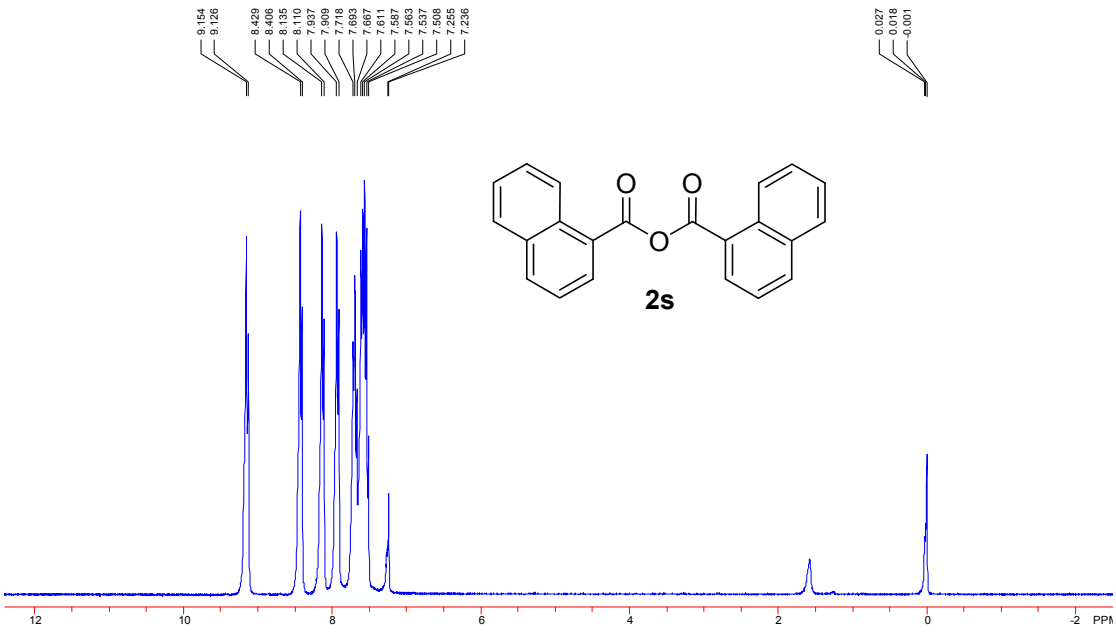
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7.587
7.563
7.537
7.508
7.295
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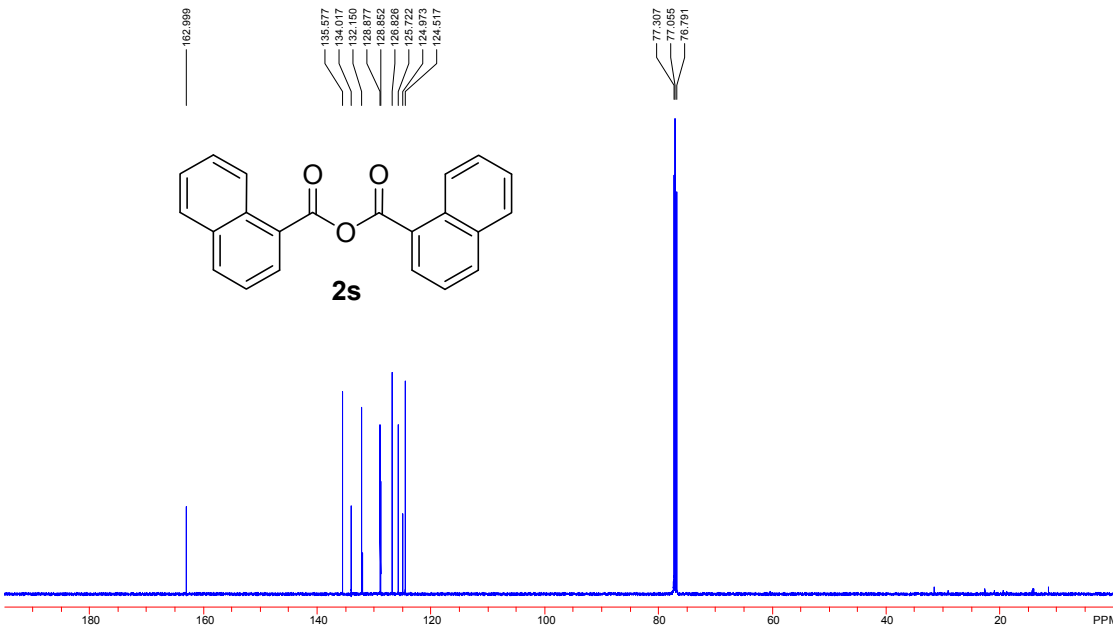
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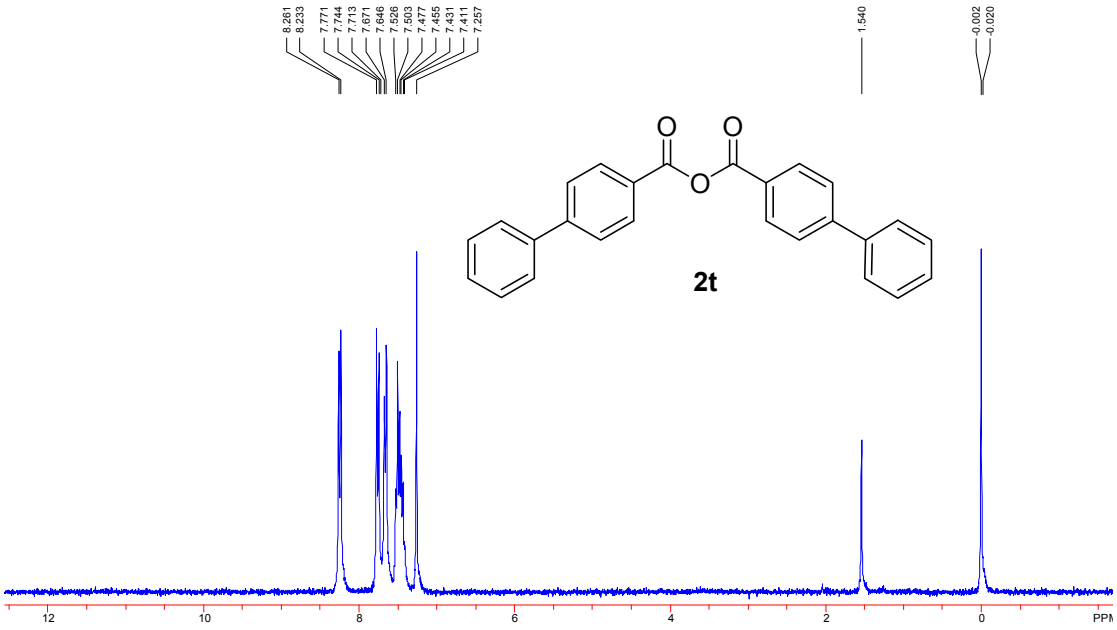
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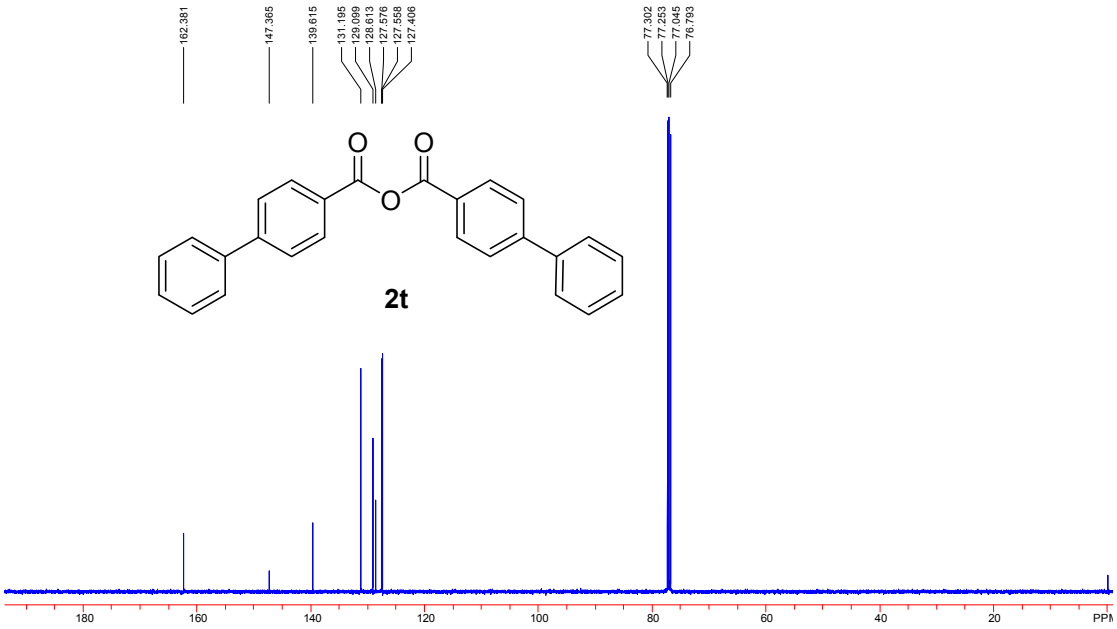
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