– Supporting Information –

Transition Metal-Free, Iodide-Mediated Domino Carbonylation-Benzylation of Benzyl Chlorides with Arylboronic Acids under Ambient Pressure of Carbon Monoxide

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Table of Contents

1. General Information

Reagent Information. All the benzyl chlorides and the arylboronic acids were purchased from Alfa Aesar, Energy Chemical, Beijing InnoChem Science & Technology Co., Ltd., and Accela ChemBio Co., Ltd. and were used as received. PEG-400 (bought from Acros and Aladdin) was pre-dried (toluene azeotrope) and deoxygenated. The following NaI and base were used: NaI (Alfa Aesar), ultrapure NaI (99.999% based on trace metals, Across), anhydrous Na_3PO_4 (Alfa Aesar), K_2HPO_4 (Alfa Aesar).

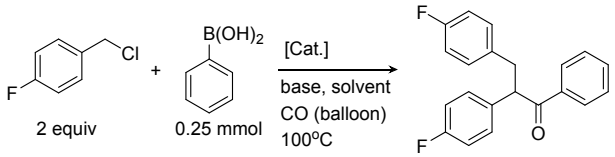
Physical Methods. ^1H and ^{13}C NMR spectra of solutions in $CDCl_3$ were recorded on a Bruker Avance 400 instrument. Chemical shifts were expressed in parts per million (ppm) downfield from tetramethylsilane and refer to the solvent signal ($CDCl_3$: H 7.24 and C 77.0 ppm). The signal of water was observed at about 1.58 ppm in $CDCl_3$. Abbreviations for signal couplings are: br, broad; s, singlet; d, doublet; t, triplet; m, multiplet; dd, doublet of doublets; dt, triplet of doublets; td, doublet of triplets; tt, triplet of triplets; tdd, doublet of doublet of triplets. Coupling constants, *J,* were reported in hertz unit (Hz). Infrared spectra of neat substances were recorded on a BRUKER TENSOR 27 FT-IR spectrometer. HRMS was performed on a Bruker's solarix 94 (ESI-FTICR-MS) mass spectrometer. Column chromatography was performed using silica gel 300-400 mesh (Yantai Jiangyou Silica Gel Co., Ltd., China) as the solid support.

2. General Procedure for NaI-Mediated Domino Carbonylation-Benzylation

A 25 mL flask equipped with a magnetic stir bar was charged with aryboronic acid (0.25 mmol), NaI (0.0375 mmol, 5.7 mg), Na_3PO_4 (1.0 mmol, 169 mg), K_2HPO_4 (0.05 mmol, 8.9 mg), PEG-400 (2 ml) before standard cycles of evacuation and back-filling with dry and pure carbon monoxide. Corresponding benzyl chloride (0.5 mmol) was added successively. The mixture was then stirred at 100^{o}C for the indicated time. After being allowed to cool to room temperature, the reaction mixture was diluted with 3 mL water and extracted with diethyl ether (4 × 5 mL). The organic phases were combined, and the volatile components were evaporated in a rotary evaporator. The residue was purified by column chromatography on silica gel (petroleum ether: diethyl ether = 100 : 1 to 10 : 1).

3. Optimization of reaction conditions

Table S1. Optimization of reaction conditions[a]



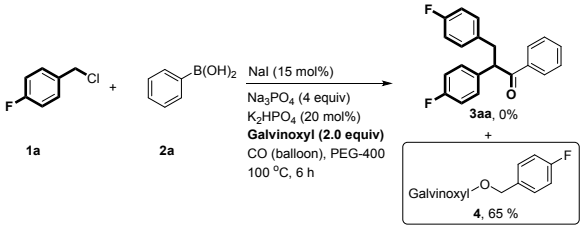
1a **2a** **3aa**

Entry	[Cat.] (%)	base	solvent	yield of **3aa**(%)[b]
1	-	Na_3PO_4	PEG-400	-
2	CuI (10)	Na_3PO_4	PEG-400	<5
3	NaI (10)	Na_3PO_4	PEG-400	71
4	NaI (15)	Na_3PO_4	PEG-400	80(62)[c]
5	KI (15)	Na_3PO_4	PEG-400	69
6	nBu$_4$NI (15)	Na_3PO_4	PEG-400	64
7	I_2 (15)	Na_3PO_4	PEG-400	52
8	NaI (15)	K_3PO_4	PEG-400	73
9	NaI (15)	K_2HPO_4	PEG-400	7
10	NaI (15)	Na_2CO_3	PEG-400	11
11	NaI (15)	K_2CO_3	PEG-400	10
12	NaI (15)	$NaHCO_3$	PEG-400	31
13	NaI (15)	KF	PEG-400	74
14	NaI (15)	NaF	PEG-400	<5
15	NaI (15)	NEt_3	PEG-400	trace
16	NaI (15)	DBU	PEG-400	trace
17[d]	**NaI (15)**	**Na_3PO_4 K_2HPO_4**	**PEG-400**	**91**
18[e]	NaI (15)	Na_3PO_4 K_2HPO_4	PEG-400	82
19[f]	NaI (15)	Na_3PO_4 K_2HPO_4	PEG-400	83
20[d]	-	Na_3PO_4 K_2HPO_4	PEG-400	-
21[d,g]	**NaI (15)**	**Na_3PO_4 K_2HPO_4**	**PEG-400**	**92**
22[d]	$Pd(OAc)_2$ (2)	Na_3PO_4 K_2HPO_4	PEG-400	-
23[d]	NaI (15)	Na_3PO_4 K_2HPO_4	PEG-200	84
24[d]	NaI (15)	Na_3PO_4 K_2HPO_4	NHD	<5
25[d]	NaI (15)	Na_3PO_4 K_2HPO_4	toluene	0

26[d]	NaI (15)	Na_3PO_4 K_2HPO_4	glycol	0
27[d]	NaI (15)	Na_3PO_4 K_2HPO_4	glycerol	0
28[d]	NaI (15)	Na_3PO_4 K_2HPO_4	DMF	<5
29[d]	NaI (15)	Na_3PO_4 K_2HPO_4	DME	0
30[d]	NaI (15)	Na_3PO_4 K_2HPO_4	H_2O	0
31[d]	NaI (15)	Na_3PO_4 K_2HPO_4	PEG-200	41
32[d]	NaI (15)	Na_3PO_4 K_2HPO_4	PEG-600	76

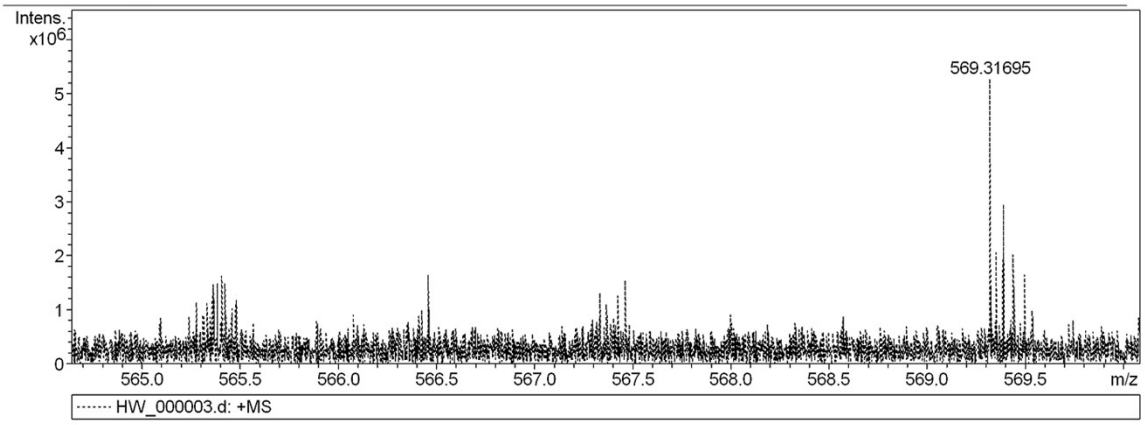
[a] *Reaction conditions*(unless otherwise stated): **1a** (0.5 mmol), **2a** (0.25 mmol), CO (1 atm), base (1.0 mmol), solvent (2.0 mL), 100 °C, and 6 h. [b] Isolated yields. [c] 80°C. [d] K_2HPO_4 (20 mol %). [e] K_2HPO_4 (50 mol %). [f] K_2HPO_4 (10 mol %). [g] Ultrapure NaI (99.999% based on trace metals, Across).

4. Effect of A Free-Radical Scavenger



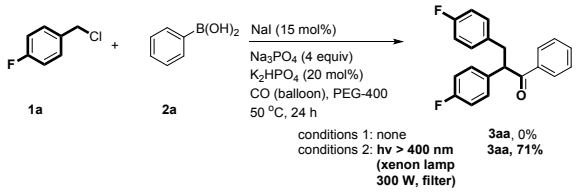
Following general procedure, the product **4** from the interception of benzyl radical by the radical scavenger Galvinoxyl was detected by HRMS (*m/z* calcd. for $C_{36}H_{47}O_2FK^+$ $[M + K]^+$ 569.3192, found 569.3170).

Acquisition Parameter	
Polarity	Positive
n/a	n/a
Broadband Low Mass	101.1 m/z
Broadband High Mass	1500.0 m/z
Acquisition Mode	Single MS
Pulse Program	basic
Source Accumulation	0.001 sec
Ion Accumulation Time	0.050 sec
Flight Time to Acq. Cell	0.001 sec



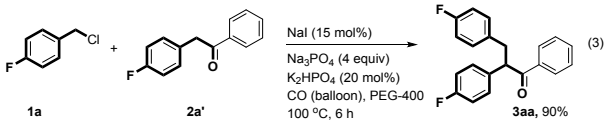
Meas. m/z	#	Formula	Score	m/z	err [ppm]	Mean err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
569.31695	1	C 36 H 47 F K O 2	100.00	569.31917	3.90	3.90	190.0	12.5	even	ok

5. Effect of Visible Light



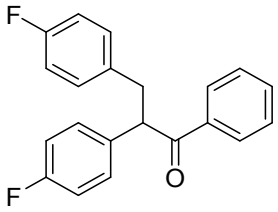
A 300 W xenon arc lamp through a UV-cutoff filter with a wavelength range of 420−800 nm, which was positioned 8 cm away from the reaction flask, was used as a visible light source to induce the model reaction at 50 °C.

6. Blank Experiment with 2-(4-Fluorophenyl)-1-phenylethanone

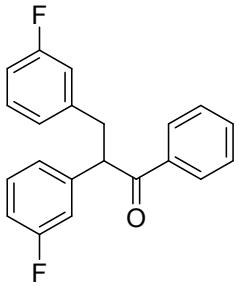


Following general procedure, the reaction of **1a** (0.25 mmol) and **2a'** (0.25 mmol) provided the desired product **3aa** in 90% yield under standard conditions.

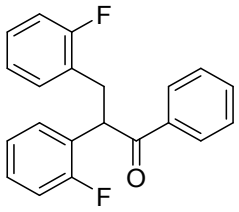
7. Analytical Data of Products



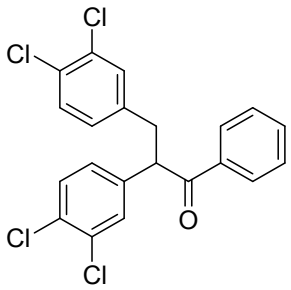
2,3-Bis-(4-fluoro-phenyl)-1-phenyl-propan-1-one (3aa): Following general procedure, **3aa** was isolated as a white solid (72.5 mg, 91%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90–7.81 (m, 2 H), 7.48–7.41 (m, 1 H), 7.39–7.31 (m, 2 H), 7.17–7.12 (m, 2 H), 7.00– 6.81 (m, 6 H), 4.71 (t, *J* = 7.4, 1 H), 3.45 (dd, *J* = 7.2, 13.6 Hz, 1 H), 3.00 (dd, *J*=7.6, 13.6 Hz, 1 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.1, 162.0 (d, *J* = 245, Hz), 161.5 (d, *J* = 243 Hz), 136.5, 135.1 (d, *J* = 3.1 Hz), 134.5 (d, *J* = 4.0 Hz), 133.1, 130.5 (d, *J* = 8.0 Hz), 129.8 (d, *J* = 8.0 Hz), 128.6, 128.9, 115.9 (d, *J* = 22 Hz), 115.1 (d, *J* = 21 Hz), 55.1, 39.3. HRMS (ESI) *m/z* calcd. for $C_{21}H_{16}OF_2Na^+$ $[M+Na]^+$ 345.1067, found 345.1070. IR (KBr, cm^{-1}) 3448, 3026, 2918, 2858, 1677, 1597, 1516, 1456, 1328, 1248, 1214, 1174, 1101, 946, 926, 879, 818, 792, 685. Mp: 91.2–91.7 °C.



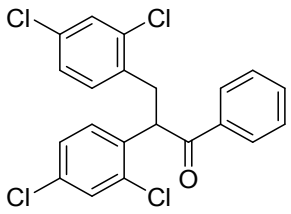
2,3-Bis-(3-fluoro-phenyl)-1-phenyl-propan-1-one (3ba): Following general procedure, **3ba** was isolated as a white solid (70.9 mg, 88%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88–7.86 (m, 2 H), 7.47 (tt, *J* = 7.6, 1.4, 1 H), 7.38–7.34 (m, 2 H), 7.18–7.11 (m, 2 H), 6.99–6.76 (m, 6 H), 4.77 (t, *J* = 7.3, 1 H), 3.51 (dd, *J* = 7.6, 13.6 Hz, 1 H), 3.04 (dd, *J* = 7.2, 13.6 Hz, 1 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.4, 163.0 (d, *J* = 245 Hz), 162.7 (d, *J* = 244 Hz), 141.8 (d, *J* = 7.0 Hz), 141.1 (d, *J* = 7.0 Hz), 136.3, 133.2, 130.4 (d, *J* = 8.0 Hz), 129.8 (d, *J* = 8.0 Hz), 128.63, 128.61, 124.8 (d, *J* = 2.0 Hz), 124.0 (d, *J* =2.0 Hz), 116.0 (d, *J* = 21 Hz), 115.1 (d, *J* = 22 Hz), 114.3 (d, *J* = 21 Hz), 113.2 (d, *J* = 20 Hz), 55.2, 39.6. HRMS (ESI) *m/z* calcd. for $C_{21}H_{16}OF_2Na^+$ $[M+Na]^+$ 345.1061, found 345.1070. IR (KBr, cm^{-1}) 3435, 3066, 2979, 2932, 1684, 1624, 1584, 1476, 1449, 1328, 1255, 1181, 1127, 1074, 933, 886, 809, 785, 691. Mp: 109.9–110.4 °C.



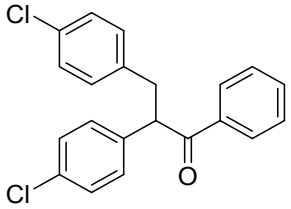
2,3-Bis-(2-fluoro-phenyl)-1-phenyl-propan-1-one (3ca): Following general procedure, **3ca** was isolated as a light yellow oil (72.5 mg, 90%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88–7.86 (m, 2 H), 7.47 (tt, *J* = 7.6, 1.2 Hz, 1 H), 7.38– 7.34 (m, 2 H), 7.22–7.11 (m, 2 H), 6.99– 6.76 (m, 6 H), 4.77 (t, *J* = 7.2 Hz, 1 H), 3.51 (dd, *J* = 7.6, 13.6 Hz, 1 H), 3.04 (dd, *J* = 7.2, 13.6 Hz, 1 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.8, 136.4, 136.3, 136.2, 134.5, 133.9, 133.1, 131.6, 129.8, 129.3, 129.1, 128.6, 128.5, 127.8, 127.4, 126.3, 48.4, 37.3. HRMS (ESI) *m/z* calcd. for $C_{21}H_{16}OF_2Na^+$ $[M+Na]^+$ 345.1061, found 345.1070. IR (KBr, cm^{-1}) 3417, 3071, 2918, 2842, 1673, 1597, 1466, 1439, 1404, 1293, 1259, 1210, 1183, 1127, 1037, 975, 941, 747, 712, 678.



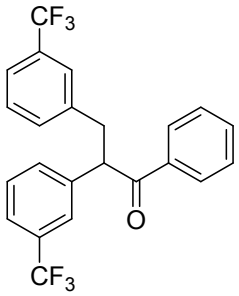
2,3-Bis-(3,4-dichloro-phenyl)-1-phenyl-propan-1-one (3da): Following general procedure, **3da** was isolated as a white solid (93.3 mg, 88%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87–7.81 (m, 2 H), 7.50 (tt, *J* = 7.2, 1.2 Hz, 1 H), 7.40–7.32 (m, 4 H), 7.25 (d, *J* = 8.4 Hz, 1 H), 7.21 (d, *J* = 2.0 Hz, 1 H), 7.05 (dd, *J* = 8.4, 2.0 Hz, 1 H), 6.87 (dd, *J* = 8.0, 2.0 Hz, 1 H), 4.71 (t, *J* = 7.6 Hz, 1 H), 3.46 (dd, *J* = 7.6, 13.6 Hz, 1 H), 2.96 (dd, *J* = 7.2, 13.6 Hz, 1 H).^{13}C NMR (100 MHz, $CDCl_3$) δ 197.7, 139.2, 138.5, 135.9, 133.5, 133.2, 132.3, 131.8, 131.0, 130.9,130.6, 130.3, 129.9, 128.8, 128.6,128.6,127.6, 54.4, 39.0. HRMS (ESI) *m/z* calcd. for $C_{21}H_{14}OCl_4Na^+$ $[M+Na]^+$ 444.9691, found 444.9695. IR (KBr, cm^{-1}) 3419, 3065, 2928, 2855, 1671, 1629, 1587, 1549, 1466, 1454, 1369, 1279, 1245, 1093, 1051, 878, 736, 671. Mp: 112.4–112.7 ^{o}C.



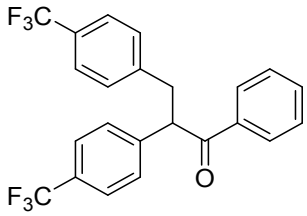
2,3-Bis-(2,4-dichloro-phenyl)-1-phenyl-propan-1-one (3ea): Following general procedure, **3ea** was isolated as a light yellow oil (94.4 mg, 89%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90–7.88 (m, 2 H), 7.48 (tt, *J* = 7.6, 1.2 Hz, 1 H), 7.38–7.34 (m, 2 H), 7.31 (d, *J* = 6.4 Hz, 1 H), 7.30 (d, *J* = 6.0 Hz, 1 H), 7.26 (d, *J* = 8.4 Hz, 1 H), 7.15 (dd, *J*=8.4, 2.0 Hz, 1 H), 6.99 (dd, *J*=8.0, 2.0 Hz, 1 H), 6.84 (d, *J* = 8.4 Hz, 1 H), 5.47 (t, *J* = 7.4 Hz, 1 H), 3.55 (dd, *J* = 6.8, 13.6 Hz, 1 H), 3.07 (dd, *J* = 8.0, 13.6 Hz, 1 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.2 , 135.8, 135.1, 134.8, 134.6, 134.5, 133.8 , 133.5, 133.0, 132.5, 129.8, 129.7, 129.2, 128.7, 128.5, 127.9, 126.8, 47.9, 36.7. HRMS (ESI) *m/z* calcd. for $C_{21}H_{14}C_{l4}ONa^+$ $[M+Na]^+$ 444.9709, found 444.9691. IR (KBr, cm^{-1}) 3417, 3068, 2926, 2855, 1681, 1619, 1577, 1549, 1466, 1455, 1369, 1279, 1245, 1093, 1051, 878, 809, 726, 671.



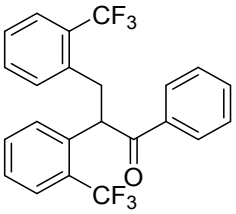
2,3-Bis-(4-chloro-phenyl)-1-phenyl-propan-1-one (3fa): Following general procedure, **3fa** was isolated as a white solid (78.1 mg, 88%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87–7.84 (m, 2 H), 7.46 (tt, *J* = 7.6, 1.2 Hz, 1 H), 7.37–7.33 (m, 2 H), 7.25 –7.18 (m, 2 H), 7.17–7.08 (m, 4 H), 6.97 (d, *J* = 8.4 Hz, 2 H), 4.71 (t, *J* = 7.3 Hz, 1 H), 3.46 (dd, *J* = 13.6, 7.2 Hz, 1 H), 3.00 (dd, *J* = 13.6, 7.6 Hz, 1 H).^{13}C NMR (100 MHz, $CDCl_3$) δ 198.6, 137.7, 137.1, 136.2, 133.2,133.1, 132.1, 130.5, 129.6, 129.1, 128.7, 128.5, 55.0, 39.9. HRMS (ESI) *m/z* calcd. for $C_{21}H_{16}OCl_2Na^+$ $[M+Na]^+$ 377.0476, found 377.0477; IR (KBr, cm^{-1}) 3435, 3079, 2965, 2925, 1671, 1603,1556, 1449, 1396, 1349, 1235, 1208, 1127, 1033, 939, 879, 826, 738, 685. Mp: 113.9–114.5 °C.



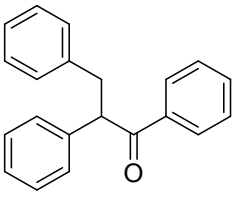
1-Phenyl-2,3-bis-(3-trifluoromethyl-phenyl)-propan-1-one (3ga): Following general procedure, **3ga** was isolated as a light yellow oil (85.5 mg, 81%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88–7.85 (m, 2 H), 7.50–7.44 (m, 2 H), 7.42 – 7.35 (m, 6 H), 7.29 (t, J = 7.6 Hz, 2 H), 7.25 (s, 1 H), 7.20 (d, J = 8.0 Hz, 1 H), 4.84 (t, J = 7.2 Hz, 1 H), 3.58 (dd, J = 13.6, 7.2 Hz, 1 H), 3.12 (dd, J = 13.6, 7.6 Hz, 1 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.2, 139.9, 139.4, 136.1, 133.4, 132.6, 131.5, 131.3 (q, J_2 = 31 Hz), 130.7 (q, J_2 = 32 Hz), 129.5, 128.8, 128.7, 128.6, 125.8 (q, J_3 = 3.8 Hz), 125.1 (q, J_3 = 3.8 Hz), 124.3 (q, J_3 = 3.7 Hz), 123.9 (q, J_1 = 271 Hz), 123.8 (q, J_1 = 271 Hz), 123.3 (q, J_3 = 3.7 Hz), 55.2, 39.9. HRMS (ESI) *m/z* calcd. for $C_{23}H_{16}OF_6Na^+$ $[M+Na]^+$ 445.0998, found 445.1007. IR (KBr, cm^{-1}) 3435, 3064, 2928, 2857, 1681, 1609, 1577, 1569, 1466, 1475, 1389, 1267, 1247, 1093, 1051, 878, 809, 716, 685.



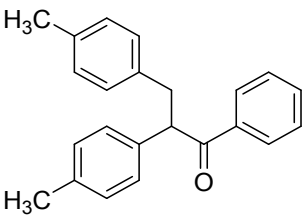
1-Phenyl-2,3-bis-(4-trifluoromethyl-phenyl)-propan-1-one (3ha): Following general procedure, **3ha** was isolated as a white solid (84.5 mg, 81%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87–7.85 (m, 2 H), 7.53–7.44 (m, 5 H), 7.39–7.33 (m, 4 H), 7.17 (d, J = 8.0 Hz, 2 H), 4.86 (t, J = 7.2 Hz, 1 H), 3.61 (dd, J = 7.6, 14 Hz, 1 H), 3.10 (dd, J = 7.2, 14 Hz, 1 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.0, 143.2, 142.5, 136.1, 133.4, 130.8, 129.7 (q, J_2=32 Hz), 129.4, 128.7, 128.6, 128.5, 126.0 (q, J_3= 3.7 Hz), 125.3 (q, J_3 = 3.8 Hz), 124.2 (q, J_1 = 270 Hz), 123.9 (q, J_1 = 270 Hz), 55.2, 39.7. HRMS (ESI) *m/z* calcd. for $C_{23}H_{16}OF_6Na^+$ $[M+Na]^+$ 445.1003, found 445.1012. IR (KBr, cm^{-1}) 3435, 3060, 2965, 2925, 1671, 1590, 1469, 1389, 1228, 1208, 1127, 1033, 939, 873, 799, 690. Mp: 64.5–65.2 °C.



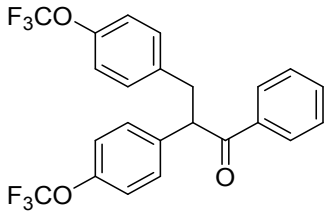
1-Phenyl-2,3-bis-(2-trifluoromethyl-phenyl)-propan-1-one (3ia): Following general procedure, **3ia** was isolated as a light yellow oil (89.3 mg, 85%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (dd, J = 8.8, 1.6 Hz, 2 H), 7.78 (d, J = 8.0 Hz, 1 H), 7.65 (d, J = 8.8 Hz, 2 H), 7.58 (t, J = 7.6 Hz, 1 H), 7.51 (tt, J = 7.2, 1.2 Hz, 1 H), 7.41–7.36 (m, 3 H), 7.28–7.23 (m, 2 H), 6.98–6.96 (m, 1 H), 5.56 (t, J = 7.2, 1 H), 3.81 (dd, J = 8.0, 14.8 Hz, 1H), 3.36 (q, J = 6.8, 14.8 Hz, 1 H). ^{13}C NMR (100MHz, $CDCl_3$) δ 199.4, 137.1, 136.8, 136.6, 133.2, 132.3, 131.4, 130.4, 130.0, 128.9 (q, J_2 = 30 Hz), 128.56, 128.53, 128.4 (q, J_2 = 30 Hz), 127.4, 126.5 (q, J_3 = 5.8 Hz), 126.49, 126.2 (q, J_3 = 5.8 Hz), 124.6 (q, J_1 = 272 Hz), 124.2 (q, J_1 = 272 Hz), 48.0, 37.2. HRMS (ESI) *m/z* calcd. for $C_{23}H_{16}OF_6Na^+$ $[M+Na]^+$ 445.1003, found: 445.1010; IR (KBr, cm^{-1}) 3435, 3060, 2925, 2855, 1671,1599, 1588, 1469, 1388, 1279, 1228,1093,1033,939,873, 809, 691.



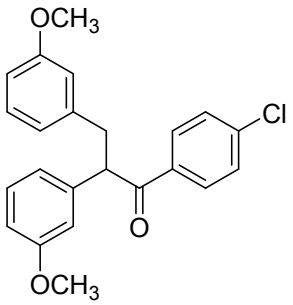
1,2,3-Triphenyl-propan-1-one (3ja): Following general procedure, **3ja** was isolated as a light yellow oil (65 mg, 91%), Known compound. the NMR spectroscopic data agree with those described in ref.[S1] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90–7.87 (m, 2 H), 7.43 (tt, J = 1.2, 7.6 Hz, 1 H), 7.35–7.21 (m, 2 H), 7.25–7.15 (m, 7 H), 7.12 (tt, J = 1.2, 7.2 Hz, 1 H), 7.08–7.06 (m, 2 H), 4.80 (t, J = 7.3 Hz, 1 H), 3.55 (dd, J = 7.6, 13.6 Hz, 1 H), 3.05 (dd, J = 7.6, 13.6 Hz, 1 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.2, 139.8, 139.1, 136.7, 132.8, 129.1, 128.9, 128.7, 128.5, 128.3, 128.2, 127.1, 126.1, 55.9, 40.1.



1-Phenyl-2,3-di-(*p*-tolyl)-propan-1-one (3ka): Following general procedure, **3ka** was isolated as a white solid (62.9 mg, 80%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91– 7.89 (m, 2 H), 7.43 (tt, *J* = 1.2, 7.2 Hz, 1 H), 7.35–7.31 (m, 2 H), 7.15 (d, *J* = 8.4 Hz, 2 H), 7.07 (d, *J* = 8.0 Hz, 2 H), 7.02–6.98 (m, 4 H), 4.78 (t, *J* = 7.2 Hz, 1 H), 3.53 (dd, *J* = 8.0, 13.6 Hz, 1 H), 3.01 (dd, *J* = 6.8, 13.6 Hz, 1 H), 2.27 (s, 3 H), 2.265 (s, 3 H).^{13}C NMR (100 MHz, $CDCl_3$) δ 199.4, 136.8, 136.79, 136.7, 136.2, 135.4, 132.7, 129.6, 128.9, 128.8, 128.6, 128.4, 128.1, 55.5, 39.6, 21.0, 20.9. HRMS (ESI) *m/z* calcd. for $C_{23}H_{22}ONa^+$ $[M+Na]^+$ 337.1568, found 337.1562; IR (KBr, cm^{-1}) 3422, 3073, 2965, 2925, 1664, 1597, 1563, 1469, 1396, 1241, 1214, 1114, 1040, 939, 879, 818, 792, 685. Mp: 97.6–98.1 °C.

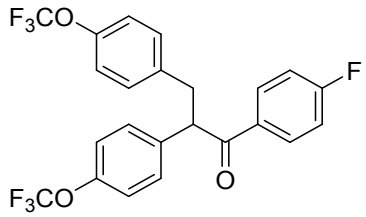


1-Phenyl-2,3-bis-(4-trifluoromethoxy-phenyl)-propan-1-one (3la): Following general procedure, **3la** was isolated as a light yellow oil (99.9 mg, 88%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87–7.84 (m, 2 H), 7.47 (tt, *J* = 1.2, 7.6 Hz, 1 H), 7.38–7.34 (m, 2 H), 7.23 (d, *J* = 8.8 Hz, 2 H), 7.10 (d, *J* = 8.8 Hz, 2 H), 7.06 (d, *J* = 8.8 Hz, 2 H), 7.02 (d, *J* = 8.8 Hz, 2 H), 4.78 (t, *J* = 7.3 Hz, 1 H), 3.51 (dd, *J* = 7.2, 13.6 Hz, 1 H), 3.03 (dd, *J* = 7.2, 13.6 Hz, 1 H).^{13}C NMR (100 MHz, $CDCl_3$) δ 198.6, 148.4, 147.8, 137.9, 137.3, 136.3, 133.3, 130.4, 129.5, 128.7, 128.6, 121.4, 120.8, 120.42 (q, *J* = 256 Hz), 120.4 (q, *J* = 255 Hz), 54.9, 39.4. HRMS (ESI) *m/z* calcd. for $C_{23}H_{16}O_3F_6Na^+$ $[M+Na]^+$ 477.0901, found 477.0914. IR (KBr, cm^{-1}) 3441, 3079, 2925, 2865, 1677, 1597,1516, 1456, 1328, 1248, 1214, 1174, 1101, 946, 926, 879, 818, 792, 685.

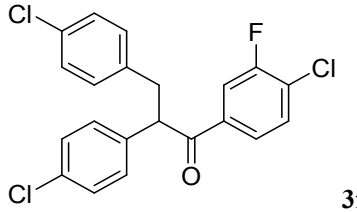


2,3-Bis-(3-methoxy-phenyl)-1-phenyl-propan-1-one (3ma): Following general procedure, **3ma** was

isolated as a transparent oil (85.5 mg, 90%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91–7.89 (m, 2 H), 7.44 (tt, *J* = 1.2, 7.2 Hz, 1 H), 7.36–7.32 (m, 2 H), 7.19–7.09 (m, 2 H), 6.82 (d, *J*=7.6 Hz, 1 H), 6.78– 6.60 (m, 5 H), 4.77 (t, *J* = 7.2, 1 H), 3.73 (s, 3 H), 3.69 (s, 3 H), 3.53 (dd, *J* = 7.6, 13.6 Hz, 1 H), 3.03 (dd, *J* = 6.8, 13.6 Hz, 1 H).^{13}C NMR (100 MHz, $CDCl_3$) δ 199.0, 159.7, 159.4, 141.4, 140.6, 136.7, 132.8, 129.8, 129.2, 128.7, 128.4, 121.5, 120.7, 114.7, 113.8, 112.6, 111.7, 55.8, 55.2, 55.0, 40.0. HRMS (ESI) *m/z* calcd. $C_{23}H_{21}ClO_3Na^+$ $[M+Na]^+$ 403.1077, found 403.1068. IR (KBr, cm^{-1}) 3430, 3079, 2935, 2825, 1671, 1603,1556, 1449, 1396, 1349, 1235, 1208, 1127, 1033, 939, 897, 822, 768, 746, 685.



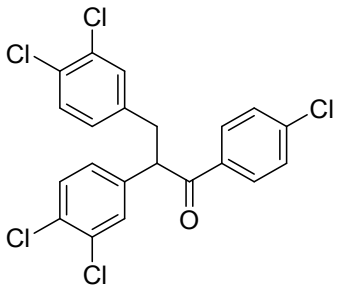
1-(4-Fluoro-phenyl)-2,3-bis-(4-trifluoromethoxy-phenyl)-propan-1-one (3lb): Following general procedure, **3lb** was isolated as a light yellow oil (103.9 mg, 88%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90–7.87 (m, 2 H), 7.21 (d, *J* = 8.8 Hz, 2 H), 7.11 (d, *J* = 8.8 Hz, 2 H), 7.07– 6.99 (m, 6 H), 4.72 (t, *J* = 7.3 Hz, 1H), 3.51 (dd, *J* = 7.6, 13.6 Hz, 1 H), 3.02 (dd, *J* = 7.2, 13.6 Hz, 1 H).^{13}C NMR (100 MHz, $CDCl_3$) δ 197.0, 165.7 (d, *J* = 254 Hz), 148.5, 147.8, 137.9, 137.2, 132.6 (d, *J* = 3.0 Hz), 131.3 (d, *J* = 9.3 Hz), 130.4, 129.5, 121.4,120.8, 120.3 (q, *J* = 256 Hz), 120.4 (q, *J* = 255 Hz), 115.8 (d, *J* = 22 Hz), 54.9, 39.4. HRMS (ESI) *m/z* calcd. for $C_{23}H_{16}O_3F_7^+$ $[M+H]^+$ 473.0988, found: 473.0982. IR (KBr, cm^{-1}) 3448, 3026, 2952, 2831, 1637, 1590, 1503, 1409, 1288, 1248, 1167, 1141, 1063, 1013, 933, 859, 852, 758, 685, 631.



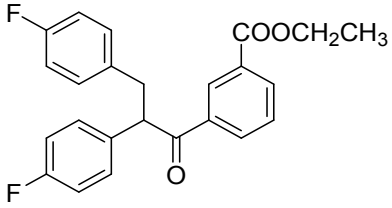
3fc

1-(4-Chloro-3-fluoro-phenyl)-2,3-bis-(4-chloro-phenyl)-propan-1-one (3fc): Following general procedure except using $NaHCO_3$ (0.5 mmol) instead of Na_3PO_4 (1.0 mmol) and K_2HPO_4 (0.05 mmol), **3fc** was isolated as a transparent oil (86.5 mg, 85%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (dd, *J* = 2.0,

7.2 Hz, 1 H), 7.87-7.83 (m, 1 H), 7.36 (d, J = 8.4 Hz, 2 H), 7.29 (d, J = 8.4 Hz, 2 H), 7.23-7.20 (m, 3 H), 7.08 (d, J = 8.4 Hz, 2 H), 4.73 (t, J = 7.3 Hz, 1 H), 3.56 (dd, J = 7.2, 13.6 Hz, 1 H), 3.10 (dd, J = 7.6, 13.6 Hz, 1 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.0, 161.0 (d, J_1 = 256 Hz), 137.3, 136.4, 133.6, 133.3 (d, J_4 = 3.6 Hz), 132.3, 131.6, 130.4, 129.4, 129.3, 128.9 (d, J_3 = 8.0 Hz), 128.5, 121.9 (d, J_2 = 18 Hz), 116.8 (d, J_2 = 22 Hz), 55.2, 39.2. HRMS (ESI) *m/z* calcd. for $C_{21}H_{14}Cl_3FONa^+$ $[M+Na]^+$ 428.999, found 428.9980. IR (KBr, cm^{-1}) 3435, 3079, 2965, 2925, 1671, 1603, 1556, 1449, 1396, 1349, 1235, 1208, 1127, 1033, 939, 879, 826, 738, 685.

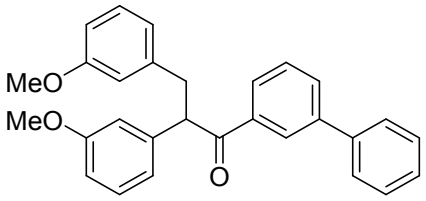


1-(4-Chloro-phenyl)-2,3-bis-(3,4-dichloro-phenyl)-propan-1-one (3dd): Following general procedure A, **3dd** was isolated as as a white solid (104.3 mg, 91%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, J = 8.8 Hz, 2 H), 7.35–7.29 (m, 4 H), 7.25–7.18 (m, 2 H), 7.01 (dd, J = 2.0, 8.4 Hz, 1 H), 6.85 (dd, J = 2.0, 8.4 Hz, 1 H), 4.63 (t, J = 7.3 Hz, 1 H), 3.42 (dd, J = 7.6, 13.6 Hz, 1 H), 2.94 (dd, J = 6.8, 13.6 Hz, 1 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.5, 140.1, 139.8, 138.9, 138.2, 134.1, 133.3, 132.3, 131.9, 131.1, 130.9, 130.7, 130.3, 130.0,129.1, 128.5, 127.5, 54.4, 38.9. HRMS (ESI) *m/z* calcd. for $C_{42}H_{26}O_2Cl_{10}K^+$ $[2M+K]^+$ 950.8474, found 950.8450. IR (KBr, cm^{-1}) 3437, 3064, 2928, 2857, 1681,1609, 1577, 1569, 1466, 1475, 1389, 1267, 1247, 1093, 1051, 878, 809, 716, 685. Mp: 122.9–110.4 °C.

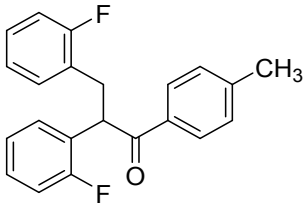


3-[2,3-Bis-(4-fluoro-phenyl)-propionyl]-benzoic acid ethyl ester (3ae): Following general procedure except using $NaHCO_3$ (0.5 mmol) instead of Na_3PO_4 (1.0 mmol) and K_2HPO_4 (0.05 mmol), **3ae** was isolated as a white oil (73.9 mg, 75%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.50 (s, 1 H), 8.13– 8.09 (m, 1H), 8.07– 8.00 (m, 1H), 7.43 (t, J = 7.8 Hz, 1 H), 7.19–7.11 (m, 2 H), 7.04–6.79 (m, 6 H), 4.74 (t, J = 7.3 Hz,

1H), 4.39– 4.29 (m, 2 H), 3.46 (q, J = 8 Hz, 1H), 3.01 (q, J = 8 Hz, 1 H), 1.37 (t, J = 7.1 Hz, 3H).^{13}C NMR (100 MHz, $CDCl_3$) δ 198.2, 165.6, 162.0 (d, J = 245 Hz), 161.5 (d, J = 243 Hz), 136.5, 134.8 (d, J = 3.3 Hz), 134.1 (d, J = 3.4 Hz), 133.8, 132.6, 130.1, 130.5 (d, J = 7.7 Hz), 129.8 (d, J = 8.0 Hz), 129.7, 128.8, 115.9 (d, J = 21 Hz), 115.1 (d, J = 21 Hz), 61.4, 55.3, 39.2, 14.3. HRMS (ESI) *m/z* calcd. for $C_{24}H_{20}O_3F_2Na^+$ $[M+Na]^+$ 417.1278, found 417.1289. IR (KBr, cm^{-1}) 3455, 3073, 3060, 2959, 2918, 2851, 1664, 1603, 1550, 1456, 1396, 1349, 1315, 1228, 1214, 1174, 1134, 1027, 939, 879, 812, 799, 731, 678.

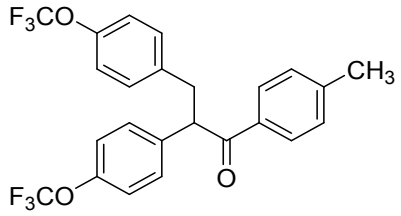


1-Biphenyl-3-yl-2,3-bis-(3-methoxy-phenyl)-propan-1-one (3mf): Following general procedure except adding TBAI instead of NaI, **3mf** was isolated as a white oil (79.2 mg, 75%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15 (t, J = 1.6 Hz, 1 H), 7.89–7.86 (m, 1 H), 7.68–7.66 (m, 1 H), 7.52–7.50 (m, 2 H), 7.45–7.34 (m, 4 H), 7.20 (t, J = 8.0 Hz, 1 H), 7.13 (t, J = 7.6 Hz, 1 H), 6.87 (d, J = 8.0 Hz, 1 H), 6.82 (t, J = 1.6 Hz, 1 H), 6.77– 6.68 (m, 3 H), 6.64 (t, J = 2.0 Hz, 1 H), 4.83 (t, J = 7.2 Hz, 1 H), 3.74 (s, 3 H), 3.70 (s, 3 H), 3.57 (dd, J = 7.6, 13.6 Hz, 1 H), 3.07 (dd, J = 6.8, 13.6 Hz, 1 H).^{13}C NMR (100 MHz, $CDCl_3$) δ 198.9, 159.9, 159.4, 141.5,142.2, 140.6, 140.1, 137.2, 131.4, 129.9, 129.2, 128.9,128.8, 127.7, 127.4, 127.1, 121.5, 120.8, 114.7, 113.8, 112.7, 111.7, 55.9, 55.2,55.0, 40.0. HRMS (ESI) *m/z* calcd. for $C_{29}H_{26}O_3Na^+$ $[M+Na]^+$ 445.1780, found 445.1768. IR (KBr, cm^{-1}) 3448, 3066,2932, 2851, 1684, 1597, 1510, 1449, 1262, 1208,1167, 1101, 1013, 960, 832, 752, 691.

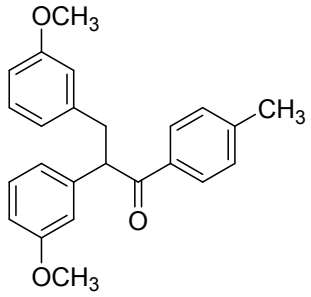


2,3-Bis-(2-fluoro-phenyl)-1-*p*-tolyl-propan-1-one (3cg): Following general procedure, **3cg** was isolated as a light yellow oil (68.9 mg, 82%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (d, J = 8.4 Hz, 2 H), 7.35 (dd, J = 7.6, 1.6 Hz, 1 H), 7.29 (dd, J = 8.0, 1.6 Hz, 1 H), 7.25 (dd, J = 8.0, 1.6 Hz, 1 H), 7.18–7.13

(m, 3 H), 7.11 (dd, *J* = 7.6, 1.6 Hz, 1 H), 7.07 (dd, *J* = 7.6, 1.6 Hz, 1 H), 6.97 (td, *J* = 7.6, 1.2 Hz, 1 H), 6.88 (dd, *J* = 7.6, 1.6 Hz, 1 H), 5.54 (t, *J* = 7.6 Hz, 1H), 3.62 (dd, *J* = 6.8, 13.6 Hz, 1 H), 3.11 (dd, *J* = 8.0, 13.6 Hz, 1 H), 2.31 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.4, 144.0, 136.6, 136.4, 134.5, 134.0, 133.7, 131.6, 129.8, 129.3, 129.1, 128.7, 128.4, 127.8, 127.3, 126.3, 48.3, 37.3, 21.6. HRMS (ESI) *m/z* calcd. for $C_{22}H_{18}OF_2K^+$ $[M+K]^+$ 375.0963, found 375.0957. IR (KBr, cm^{-1}) 3424, 3073, 2955, 2895, 1661, 1597, 1574, 1469, 1398, 1349, 1259, 1214, 1114, 1037, 939, 879, 818, 792, 687.

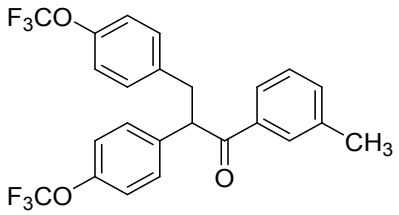


1-(*p*-Tolyl)-2,3-bis-(4-trifluoromethoxy-phenyl)-propan-1-one (3lg): Following general procedure, **3lg** was isolated as a white solid (103 mg, 87%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.3 Hz, 2 H), 7.24 (d, *J* = 8.4 Hz, 2 H), 7.16 (d, *J* = 7.6 Hz, 2 H), 7.11–7.02 (m, 6 H), 4.78 (t, *J* = 7.3 Hz, 1 H), 3.53 (dd, *J* = 7.6, 13.6 Hz, 1 H), 3.03 (dd, *J* = 7.2, 13.6 Hz, 1 H), 2.33 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.1, 148.4, 147.7, 144.2, 138.1, 137.6, 133.8 , 130.4, 129.5, 129.3, 128.8, 121.3, 120.8, 120.5 (q, *J* = 256 Hz), 120.4 (q, *J* = 255 Hz), 54.7, 39.4, 21.5. HRMS (ESI) *m/z* calcd. for $C_{24}H_{18}O_3F_6Na^+$ $[M+Na]^+$ 491.1058, found 491.1063. IR (KBr, cm^{-1}) 3422, 3060, 2972, 2905, 1664,1590, 1510, 1442, 1235, 1148,1093,1013,946,839, 738,671,524; mp:73.2–73.8 °C.

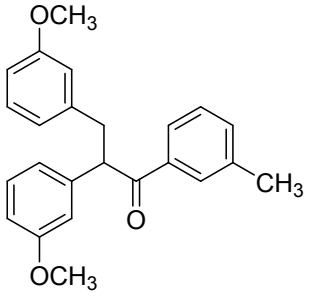


2,3-Bis-(3-methoxy-phenyl)-1-(*p*-tolyl)-propan-1-one (3mg): Following general procedure, **3mg** was isolated as a light yellow oil (79.3 mg, 88%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (d, *J* = 8.4 Hz, 2 H), 7.19–7.09 (m, 4 H), 6.83 (d, *J* = 7.6 Hz, 1 H), 6.79 (t, *J* = 1.6 Hz, 1 H), 6.62 (t, *J* = 2.0 Hz, 1 H), 4.77 (t, *J* = 7.2 Hz, 1 H), 3.72 (s, 3 H), 3.69 (s, 3 H), 3.54 (dd, *J* = 7.6, 13.6 Hz, 1 H), 3.04 (dd, *J* = 6.8, 13.6 Hz,

1 H), 2.31 (s, 3 H).^{13}C NMR (100 MHz, $CDCl_3$) δ 198.5, 159.8, 159.3, 143.6, 141.4, 140.8, 134.1, 129.7, 129.1, 128.7, 121.4, 120.7, 114.6, 113.8, 112.5, 111.6, 55.5, 55.1, 55.0, 40.0, 21.5. HRMS (ESI) *m/z* calcd. for $C_{24}H_{26}O_3^+$ $[M+H]^+$ 361.1804, found 361.1820. IR (KBr, cm^{-1}) 3488, 2999, 2965, 2838, 1677, 1603, 1496, 1429, 1262, 1154, 1040, 939, 879, 845, 778, 698.

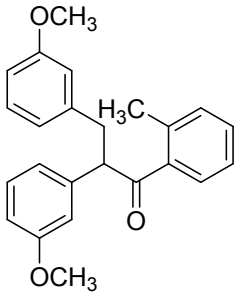


1-(*m*-Tolyl)-2,3-bis-(4-trifluoromethoxy-phenyl)-propan-1-one (3lh): Following general procedure, **3lh** was isolated as a transparent oil (103.0 mg, 88%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 (s, 1 H), 7.65 (d, *J* = 7.6 Hz, 1 H), 7.29 (d, *J* = 7.6 Hz, 1 H), 7.27–7.22 (m, 3 H), 7.12–7.02 (m, 6 H), 4.79 (t, *J* = 7.3, 1 H), 3.52 (dd, *J* = 7.6, 13.6 Hz, 1 H), 3.03 (dd, *J* = 7.2, 13.6 Hz, 1 H), 2.33 (s, 3 H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.8, 148.4, 147.7, 138.6, 138.1, 137.4 , 136.3, 134.1, 130.4, 129.6, 129.1, 128.5, 125.8, 121.3,120.8, 120.3 (q, *J* = 256 Hz), 120.4 (q, *J* = 255 Hz), 54.9, 39.4, 21.3. HRMS (ESI) *m/z* calcd. for $C_{24}H_{20}O_3F_6^+$ $[M+H]^+$ 469.1238, found, 469.1253. IR (KBr, cm^{-1}) 3422, 2965, 2925, 2872, 1684, 1643, 1510, 1402, 1268, 1221, 1154, 1107, 1006, 919, 845, 678.

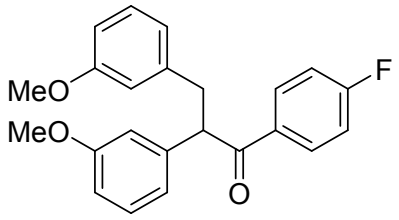


2,3-Bis-(3-methoxy-phenyl)-1-(*m*-tolyl)-propan-1-one (3mh): Following general procedure, **3mh** was isolated as a light yellow oil (68.5 mg, 76%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 (s, 1 H), 7.70 (d, *J* = 7.2 Hz, 1 H), 7.26 (d, *J* = 7.6 Hz, 1 H), 7.22 (d, *J* = 7.6 Hz, 1 H), 7.17 (t, *J* = 8.0 Hz, 1 H), 7.11 (t, *J* = 7.6 Hz, 1 H), 6.84 (d, *J* = 8.0 Hz, 1 H), 6.80 (t, *J* = 1.6 Hz, 1 H), 6.75– 6.67 (m, 3 H), 6.63 (t, *J* = 1.6 Hz, 1 H), 4.78 (t, *J* = 7.2 Hz, 1 H), 3.73 (s, 3 H), 3.69 (s, 3 H), 3.54 (dd, *J* = 7.6, 13.6 Hz, 1 H), 3.04 (dd, *J* = 7.2, 13.6 Hz, 1 H), 2.32 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.2, 159.8, 159.4, 141.4, 140.7,

138.2, 136.7, 133.6, 129.8, 129.1, 128.3, 125.9, 121.4, 120.7, 114.7, 113.7, 112.5, 111.6, 55.7, 55.1 , 55.0, 40.0, 21.3. HRMS (ESI) *m/z* calcd. for $C_{24}H_{25}O_3Na^+$ $[M+Na]^+$ 383.1623, found 383.1628. IR (KBr, cm^{-1}) 3415, 3033, 2952, 2831, 1677, 1597, 1483, 1442, 1396, 1255, 1141, 1040, 879, 845, 691.

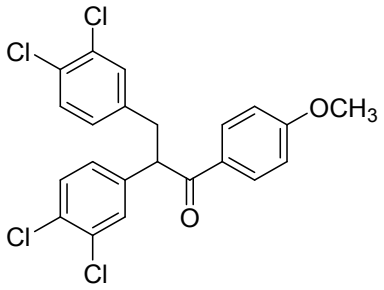


2,3-Bis-(3-methoxy-phenyl)-1-(*o*-tolyl)-propan-1-one (3mi): Following general procedure, **3mi** was isolated as a light yellow oil (70.3 mg, 78%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 (d, *J* = 7.6 Hz, 1 H), 7.25–7.21 (m, 1 H), 7.17 (t, *J* = 8.0 Hz, 1 H), 7.13 (t, *J* = 6.8 Hz, 1 H), 7.11 (s, 1 H), 7.09 (s, 1 H), 6.82 (d, *J* = 7.6 Hz, 1 H), 4.62 (t, *J* = 7.2 Hz, 1 H), 3.73 (s, 3 H), 3.71 (s, 3 H), 3.57 (dd, *J* = 8.4, 13.6 Hz, 1 H), 3.01 (dd, *J* = 6.0, 13.6 Hz, 1 H), 2.23 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.2, 159.8, 159.5, 141.4, 140.0, 138.7, 137.9, 131.5, 130.8, 129.7, 129.2, 127.8, 125.3, 121.5, 120.8, 114.7, 113.7, 112.7, 111.8, 58.9, 55.2,55.1, 39.7, 20.5. HRMS (ESI) *m/z* calcd. for $C_{24}H_{25}O_3Na^+$ $[M+Na]^+$ 383.1623, found 383.1633. IR (KBr, cm^{-1}) 3488, 2999, 2965, 2838, 1677, 1603, 1496, 1429, 1262, 1154, 1040, 939, 879, 845, 778, 698.

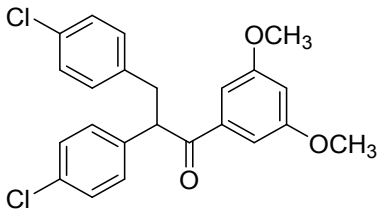


1-(4-fluorophenyl)-2,3-bis(3-methoxyphenyl)propan-1-one (3lj): Following general procedure, **3lj** was isolated as a white oil (77 mg, 85%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (dd, *J* = 8.8, 5.6 Hz, 2 H), 7.17 (t, *J* = 7.6 Hz, 1 H), 7.11 (d, *J* = 8.0 Hz, 1 H), 6.99 (t, *J* = 8.8 Hz, 2 H), 6.80 (d, *J* = 7.6 Hz, 1 H), 6.76– 6.72 (m, 2 H), 6.68 (dd, *J* = 8.0, 2.0 Hz, 2 H), 6.60 (t, *J* = 2.0 Hz, 1 H), 4.71 (t, *J* = 7.2 Hz, 1 H), 3.72 (s, 3 H), 3.69 (s, 3 H), 3.52 (dd, *J* = 7.6, 13.6 Hz, 1 H), 3.02 (dd, *J* = 6.8, 13.6 Hz, 1 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.4, 165.5 (d, *J* = 254 Hz), 159.9, 159.4, 141.2, 140.4, 133.0 (d, *J* = 3 Hz), 131.3

(d, J = 9.2 Hz), 129.9, 129.2, 121.4, 120.6, 115.5 (d, J = 22 Hz), 114.7, 113.8, 112.6, 111.7, 55.8, 55.1, 55.0, 40.0. HRMS (ESI) *m/z* calcd. for $C_{23}H_{21}O_3FNa^+$ $[M+Na]^+$ 387.1372, found 387.1375. IR (KBr, cm^{-1}) 3435, 3073, 2972, 2851, 1684, 1597,1584, 1489, 1456, 1315, 1255, 1154, 1101, 1114, 1061, 946, 758, 698, 651.

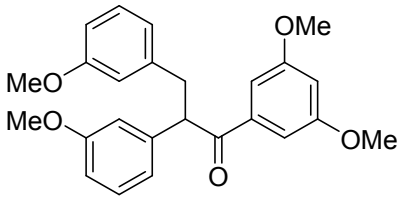


2,3-Bis-(3,4-dichloro-phenyl)-1-(4-methoxy-phenyl)-propan-1-one (3dk): Following general procedure except using TBAI instead of NaI, **3dk** was isolated as a light yellow oil (79.5 mg, 70%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (d, J = 9.2 Hz, 2 H), 7.33–7.31 (m, 2 H), 7.24 (d, J = 8.0 Hz, 1 H), 7.19 (d, J = 2.0, 1 H), 7.04 (dd, J = 8.4, 2.0 Hz, 1 H), 6.89– 6.80 (m, 3 H), 4.65 (t, J = 7.3 Hz, 1 H), 3.80 (s, 3H), 3.44 (dd, J = 7.6, 13.6 Hz, 1 H), 2.93 (dd, J = 7.2, 13.6 Hz, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 196.1, 163.8, 139.4, 139.1, 133.1, 132.3, 131.6, 131.0, 130.93, 130.9, 130.5, 130.3, 129.9, 128.8, 128.6 127.5, 113.9, 55.5, 53.9, 39.0. HRMS (ESI) *m/z* calcd. for $C_{22}H_{16}Cl_4O_2Na^+$ $[M+Na]^+$ 474.9802, found 474.9811. IR (KBr, cm^{-1}) 3428, 3066, 2925, 2851, 1677, 1603, 1577, 1469, 1416, 1355, 1288, 1248, 1167, 973, 953, 886, 752, 671.

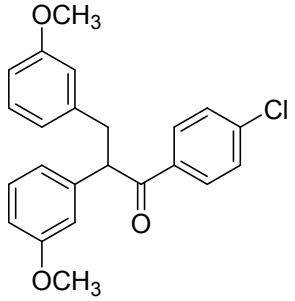


2,3-Bis-(4-chloro-phenyl)-1-(3,5-dimethoxy-phenyl)-propan-1-one (3fl): Following general procedure, **3fl** was isolated as a white solid (84.9 mg, 82%). ^{1}H NMR (400 MHz, CDCl3) δ 7.22 (d, J = 8.4 Hz, 2 H), 7.15 (d, J = 8.4 Hz, 2 H), 7.11 (d, J = 8.4 Hz, 2 H), 6.97 (d, J = 2.4 Hz, 2 H), 6.96 (d, J = 8.4 Hz, 2 H), 6.54 (t, J = 2.4 Hz, 1 H), 4.64 (t, J = 7.3 Hz, 1 H), 3.74 (s, 6 H), 3.43 (dd, J = 7.2, 14 Hz, 1 H), 2.97 (dd, J = 7.2, 14 Hz, 1 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.3, 160.8, 138.2, 137.7, 137.1, 133.3, 132.1, 130.4, 129.5, 129.2, 128.4, 106.5, 105.3, 55.5, 55.1, 39.3. HRMS (ESI) *m/z* calcd. for

$C_{23}H_{20}Cl_2O_3Na^+$ $[M+Na]^+$ 437.0687, found: 437.0681. IR (KBr, cm^{-1}) 3440, 3050, 2940, 2830, 1681, 1600, 1490, 1450, 1370, 1320, 1235, 1208, 1127, 1033, 957, 874, 756, 698. Mp: 112.3–113.1 °C



1-(3,5-Dimethoxy-phenyl)-2,3-bis-(3-methoxy-phenyl)-propan-1-one (3mm): Following general procedure A, **3mm** was isolated as a transparent oil (81.3 mg, 80%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.16 (t, *J* = 8.0 Hz, 1 H), 7.10 (t, *J* = 8.0 Hz, 1 H), 7.03 (s, 1 H), 7.02 (s, 1 H), 6.81 (d, *J* = 7.6 Hz, 1 H), 6.76 (t, *J* = 1.6 Hz, 1 H), 6.74–6.71 (m, 1 H), 6.68 (dd, *J* = 8.4, 2.4 Hz, 2 H), 6.60 (t, *J* = 1.6 Hz, 1 H), 6.52 (t, *J* = 2.4 Hz, 1 H), 4.70 (t, *J* = 7.2 Hz, 1 H), 3.73 (s, 6 H), 3.72 (s, 3 H), 3.69 (s, 3 H), 3.50 (dd, *J* = 7.6, 13.6 Hz, 1 H), 3.01 (dd, *J* = 6.8, 13.6 Hz, 1 H).^{13}C NMR (100 MHz, $CDCl_3$) δ 198.7, 160.6, 159.9, 159.4, 141.3, 140.6, 138.6, 129.8, 129.2, 121.4, 120.7, 114.7, 113.7, 112.6, 111.7, 106.5, 105.2, 55.9, 55.4, 55.2, 55.0, 40.1. HRMS (ESI) *m/z* calcd. for $C_{25}H_{27}O_5Na^+$ $[M+Na]^+$ 430.1751, found 430.1723. IR (KBr, cm^{-1}) 3440, 3120, 2965, 2925, 1671, 1603, 1556, 1449, 1396, 1349, 1235, 1208, 1127, 1033, 939, 879, 854, 738, 698.



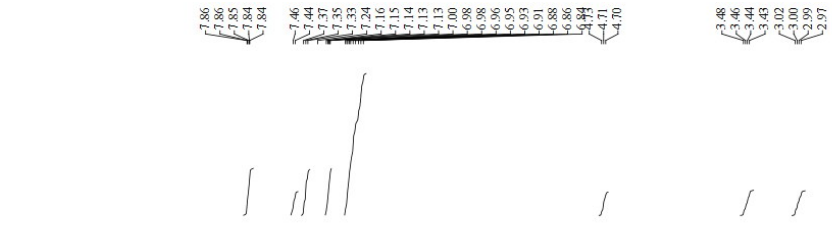
1-(4-Chloro-phenyl)-2,3-bis-(3-methoxy-phenyl)-propan-1-one (3md): Following general procedure except adding 3 equiv **1m** instead of 1.5 equiv **1m**, **3md** was isolated as a transparent oil (77.9 mg, 82%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (d, *J* = 8.8 Hz, 2 H), 7.29 (d, *J* = 8.8 Hz, 2 H), 7.15 (td, *J* = 7.6, 2.0 Hz, 1 H), 7.11 (t, *J* = 7.6 Hz, 1 H), 6.79 (d, *J* = 7.6 Hz, 1 H), 6.75–6.67 (m, 4 H), 6.60 (t, *J* = 2.0 Hz, 1 H), 4.70 (t, *J* = 7.2 Hz, 1 H), 3.72 (s, 3 H), 3.69 (s, 3 H), 3.52 (dd, *J* = 7.6, 13.6 Hz, 1 H), 3.02 (dd, *J* = 6.8, 13.6 Hz, 1 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.7, 159.9, 159.4, 141.1, 140.2, 139.2, 134.9, 130.0, 129.9, 129.2, 128.7, 121.4, 120.6, 114.7, 113.8, 112.6, 111.6, 55.8, 55.1, 55.0, 39.9. HRMS (ESI)

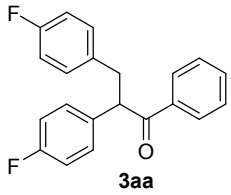
m/z calcd. for $C_{23}H_{21}ClO_3Na^+$ $[M+Na]^+$ 403.1077, found 403.1065. IR (KBr, cm^{-1}) 3450, 3001, 2930, 2925, 1671, 1603, 1556, 1449, 1396, 1349, 1235, 1208, 1090, 1033, 924,879, 826,752, 584.

8. References

(S1) S.-M. Lu, C. Bolm, *Angew. Chem., Int. Ed.,* 2008, **47**, 8920.

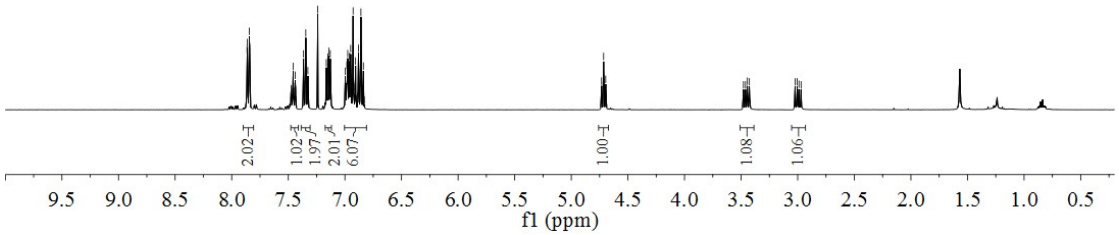
9. NMR Spectra for Products

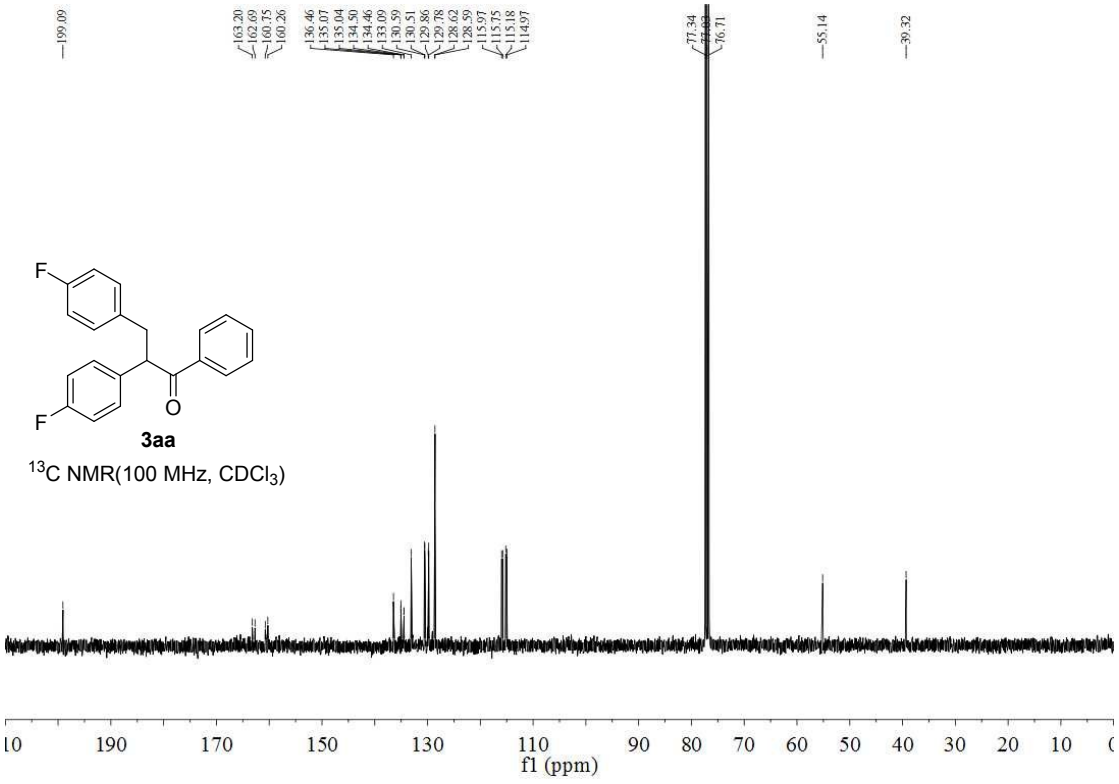




3aa

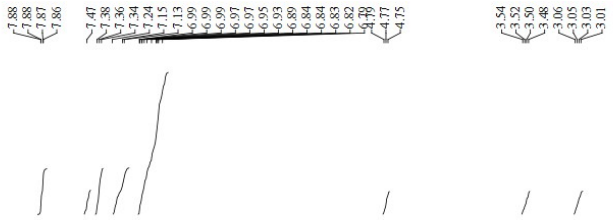
^{1}H NMR (400 MHz, $CDCl_3$)

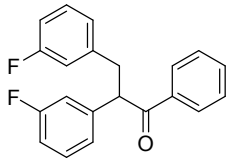




3aa

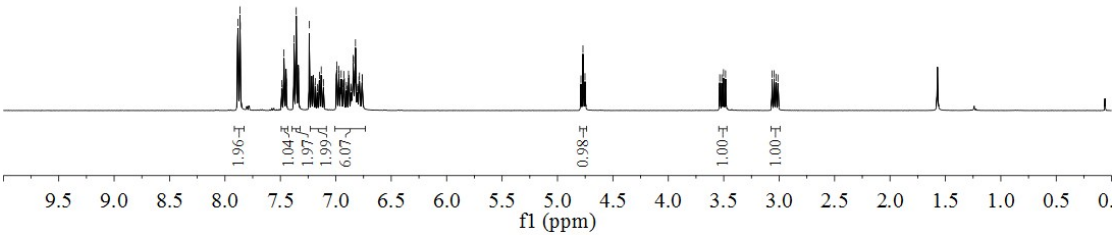
^{13}C NMR(100 MHz, $CDCl_3$)

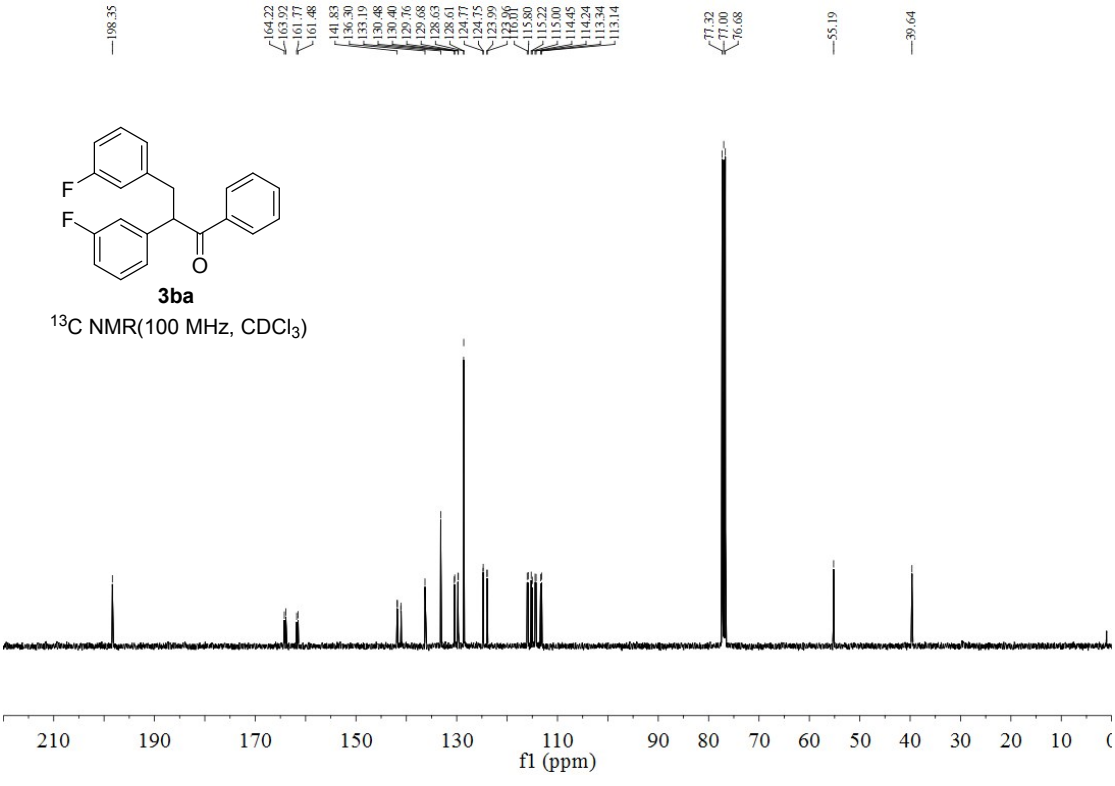




3ba

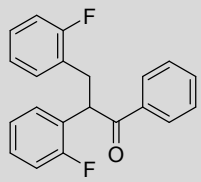
^{1}H NMR (400 MHz, $CDCl_3$)





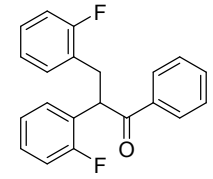
3ba

^{13}C NMR(100 MHz, $CDCl_3$)



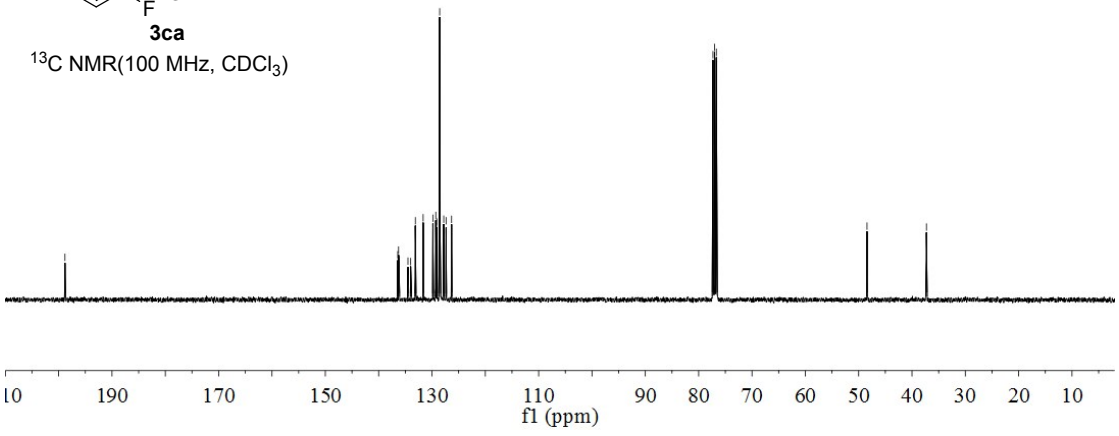
3ca

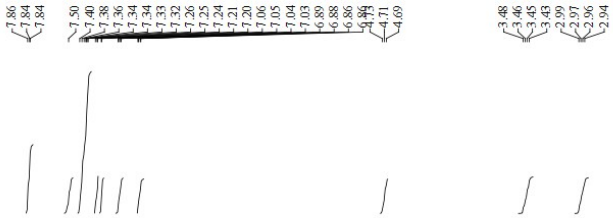
^{1}H NMR (400 MHz, $CDCl_3$)

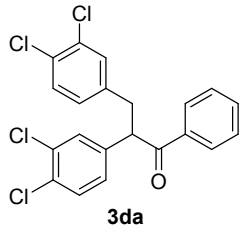


3ca

^{13}C NMR(100 MHz, $CDCl_3$)

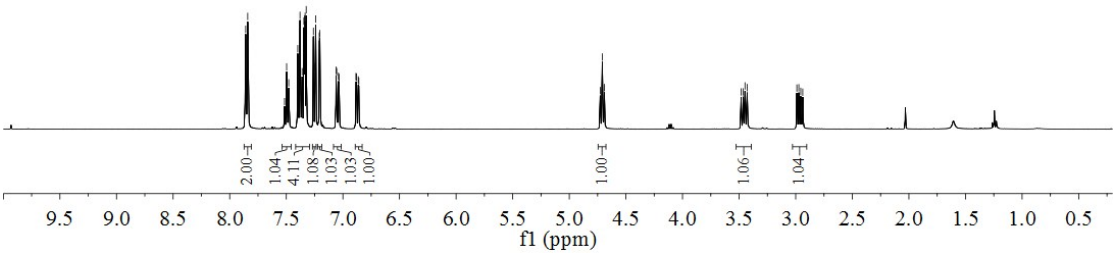


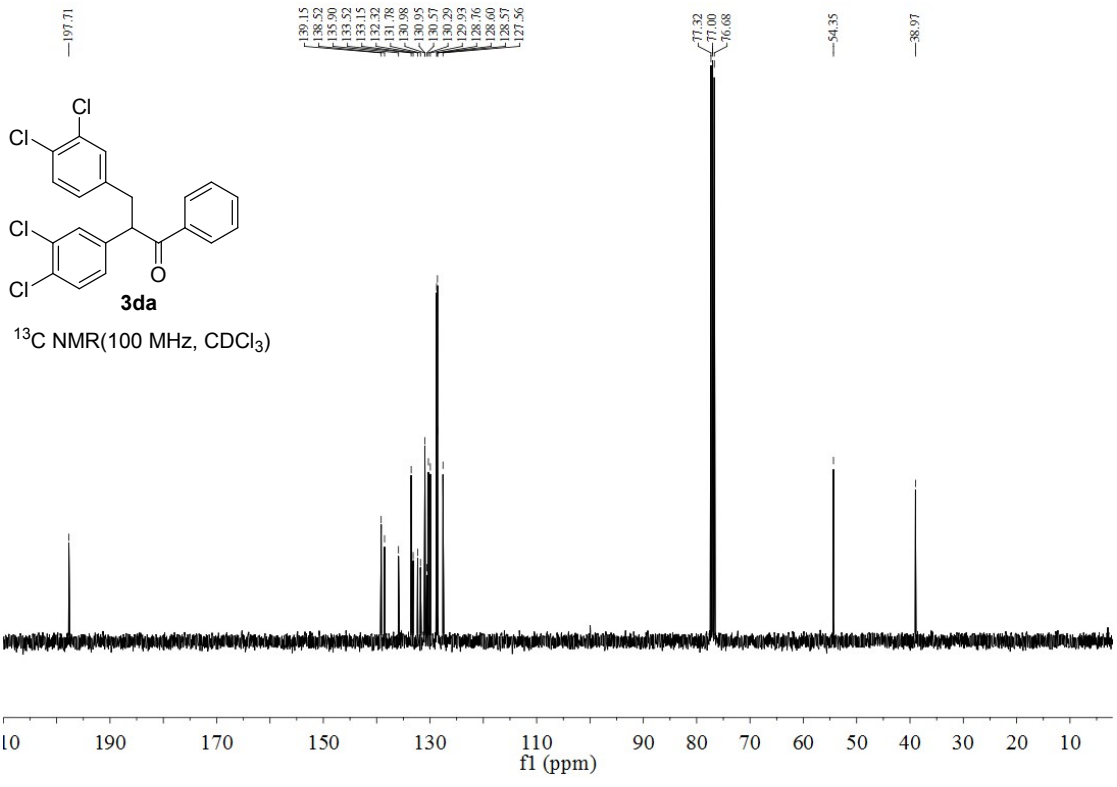




3da

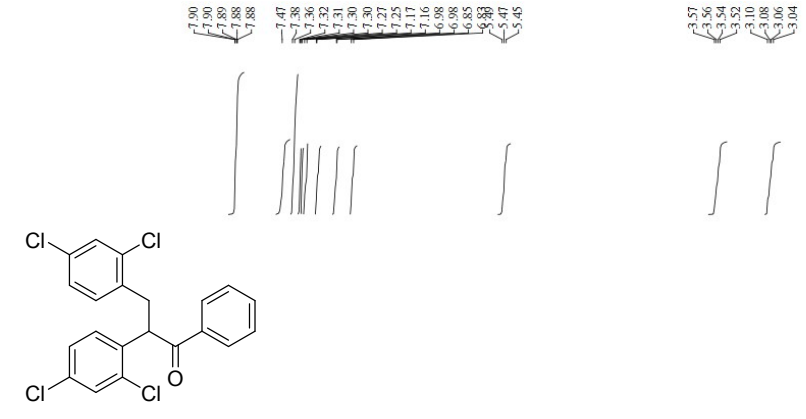
^{1}H NMR (400 MHz, $CDCl_3$)





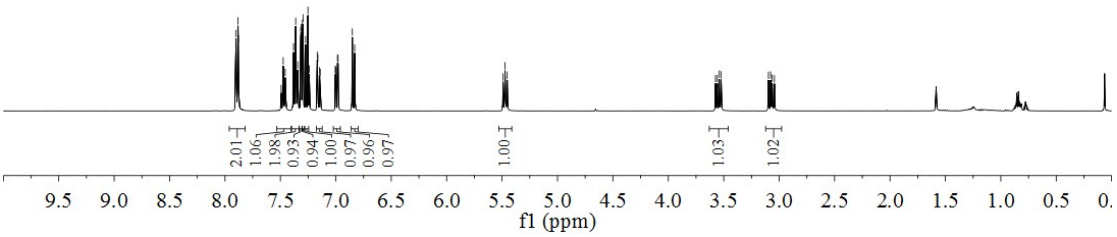
3da

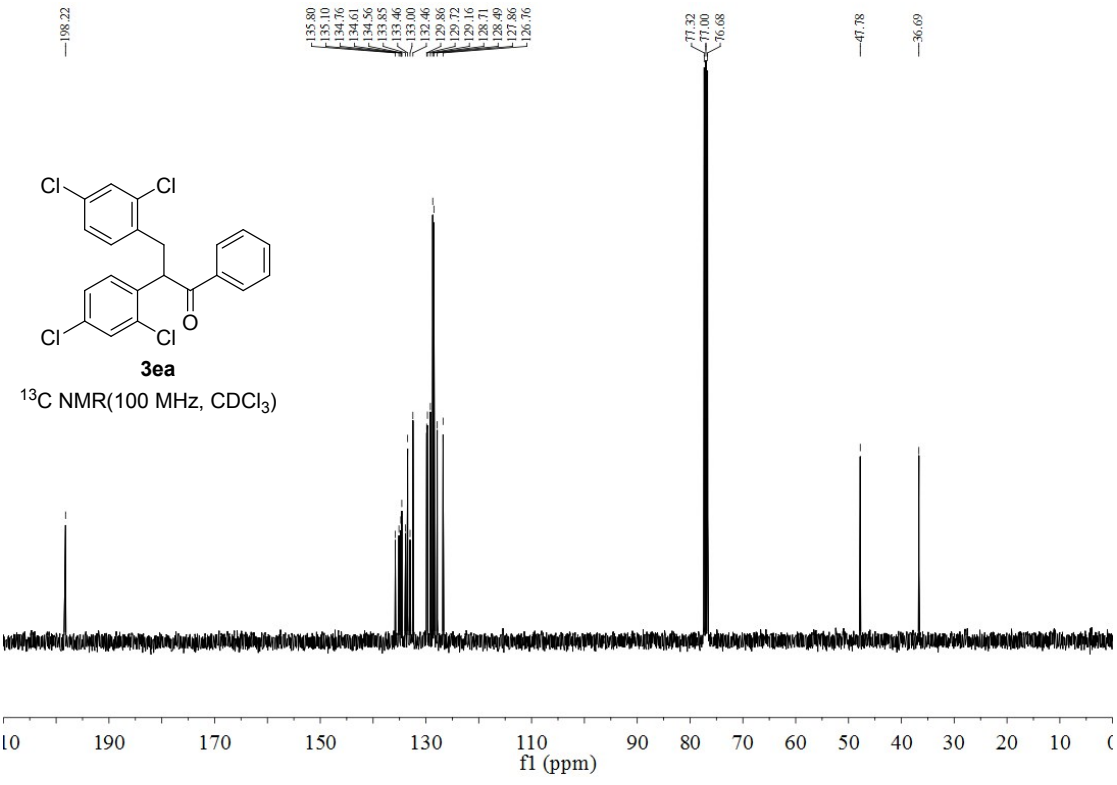
^{13}C NMR(100 MHz, $CDCl_3$)



3ea

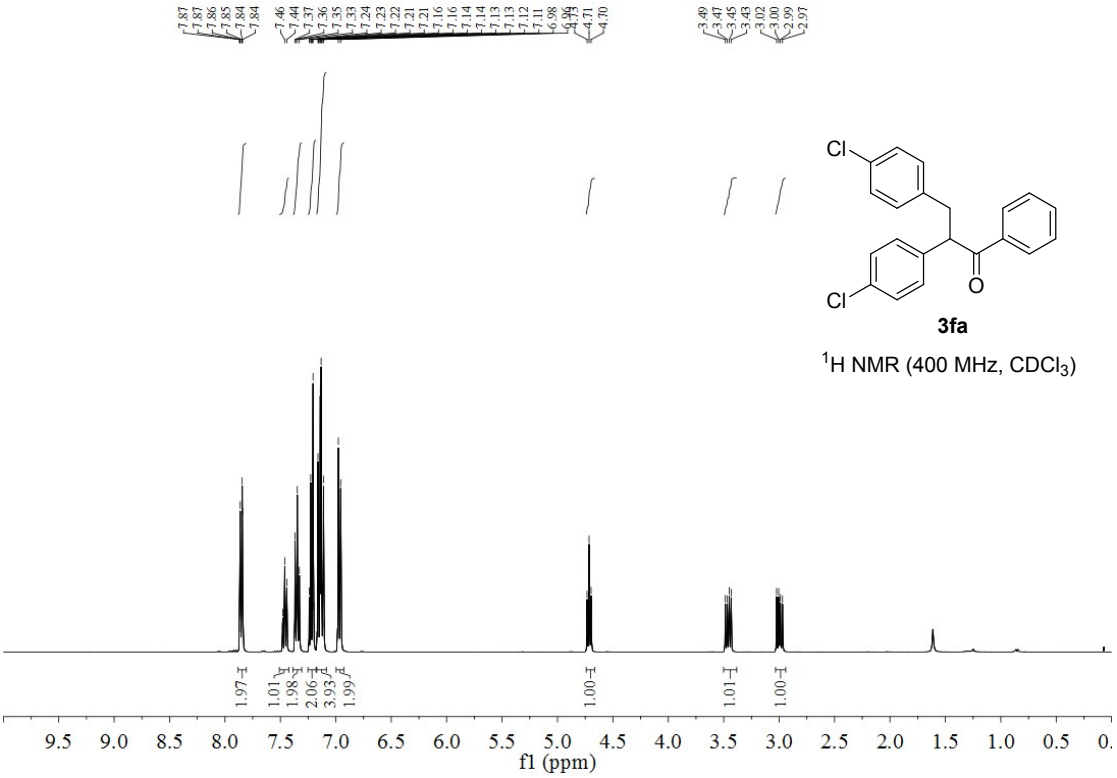
^{1}H NMR (400 MHz, $CDCl_3$)





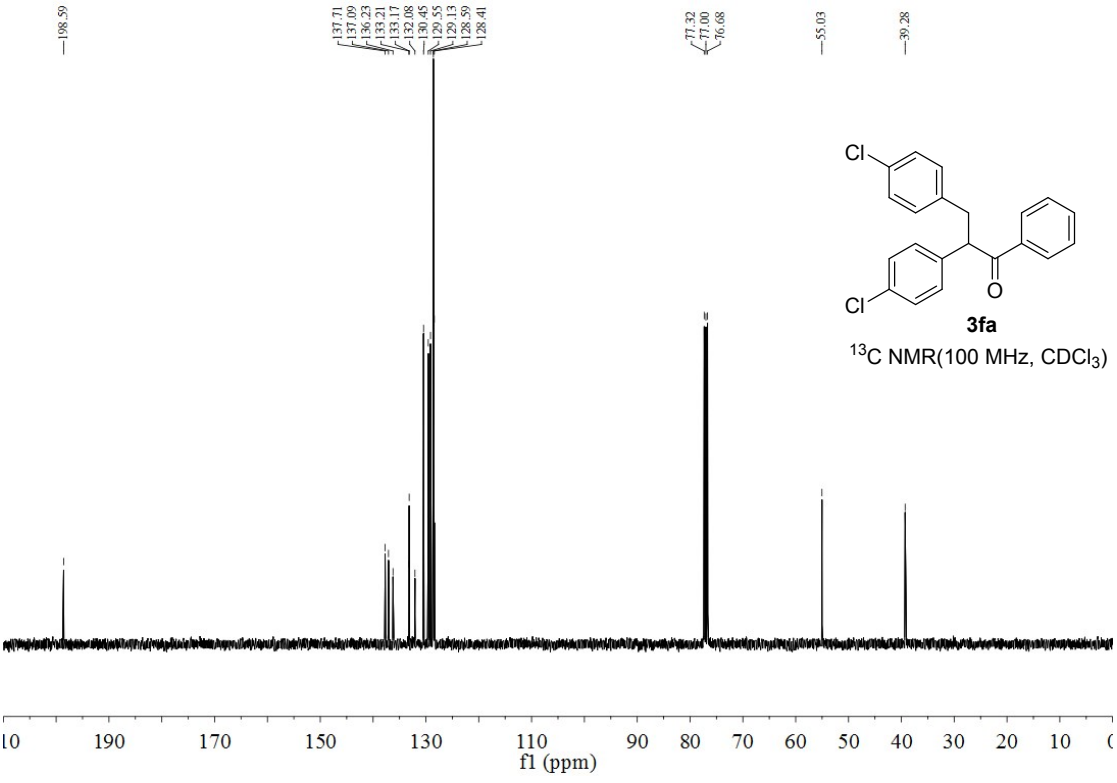
3ea

^{13}C NMR(100 MHz, $CDCl_3$)



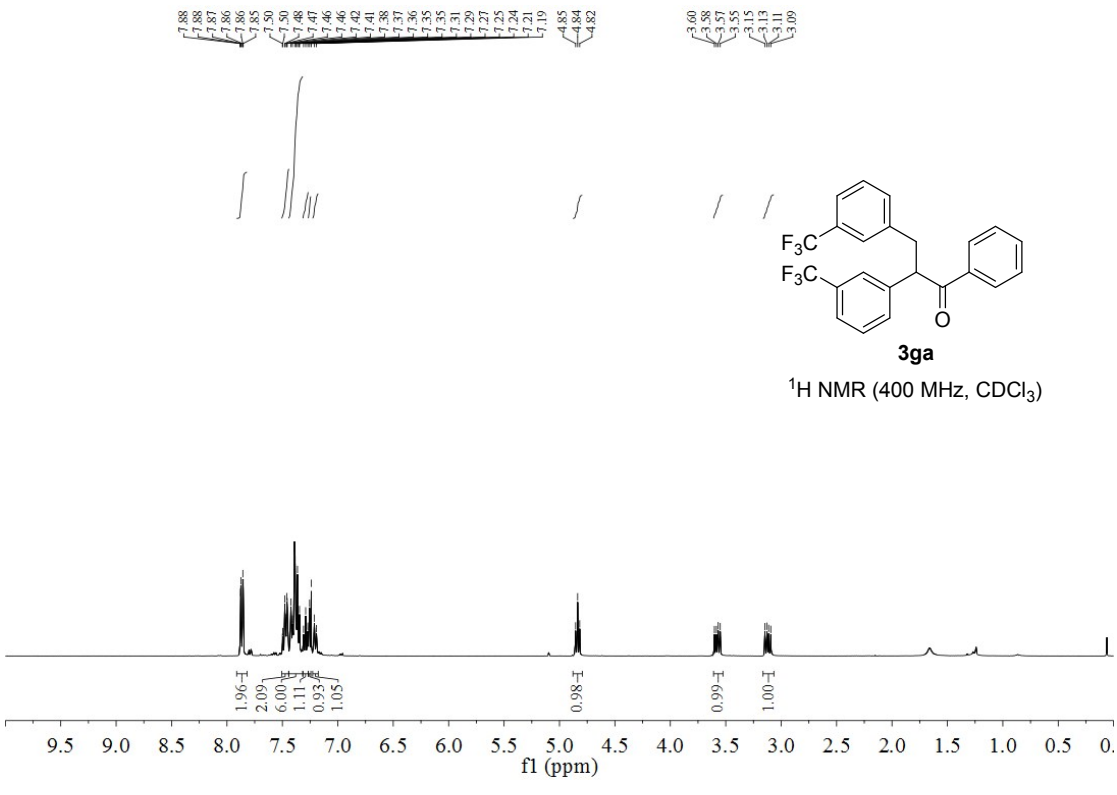
3fa

^{1}H NMR (400 MHz, $CDCl_3$)



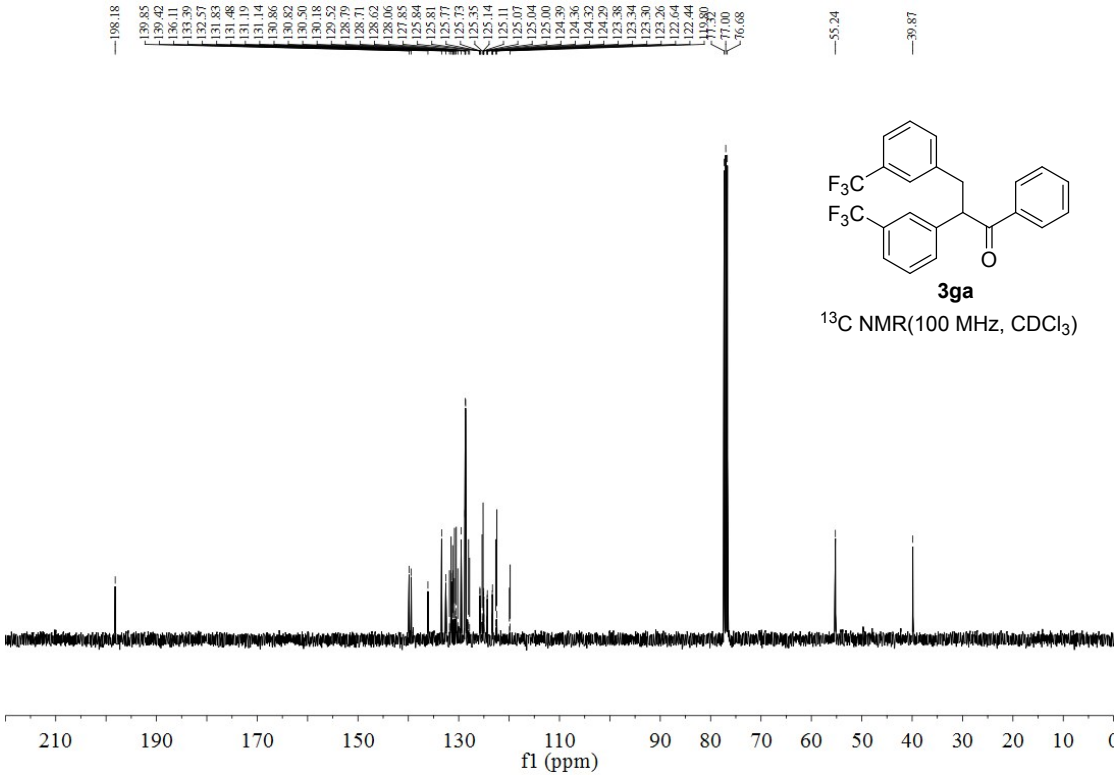
3fa

^{13}C NMR(100 MHz, $CDCl_3$)



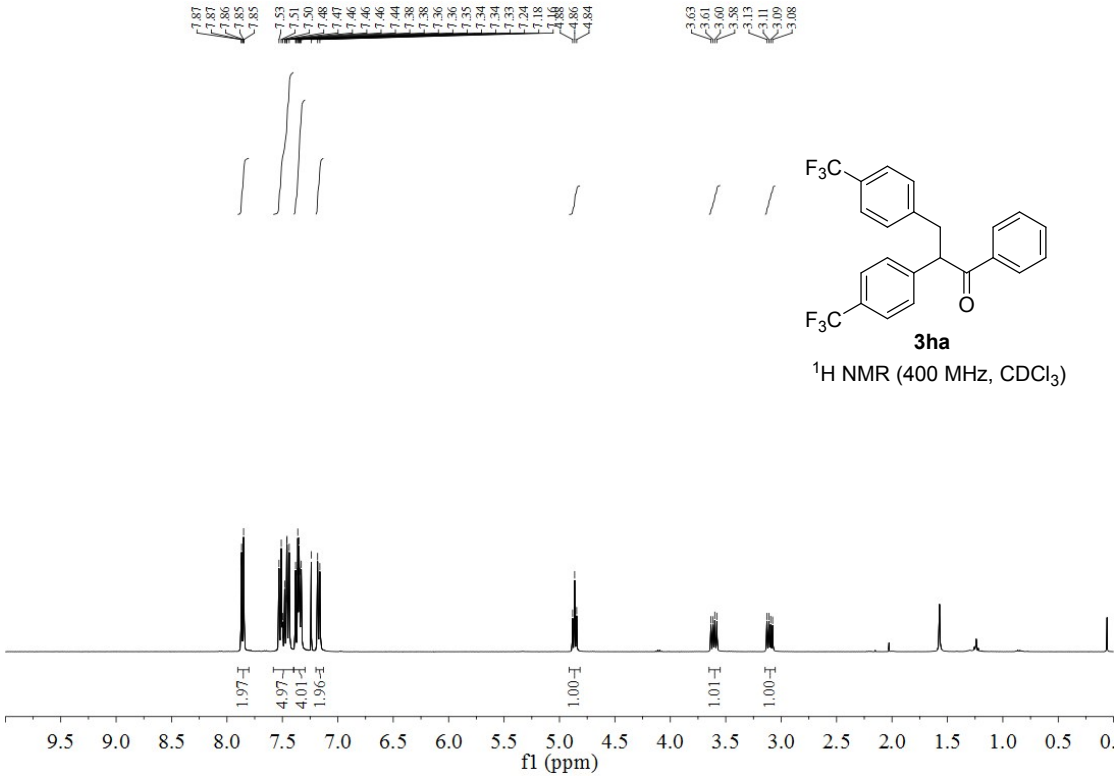
3ga

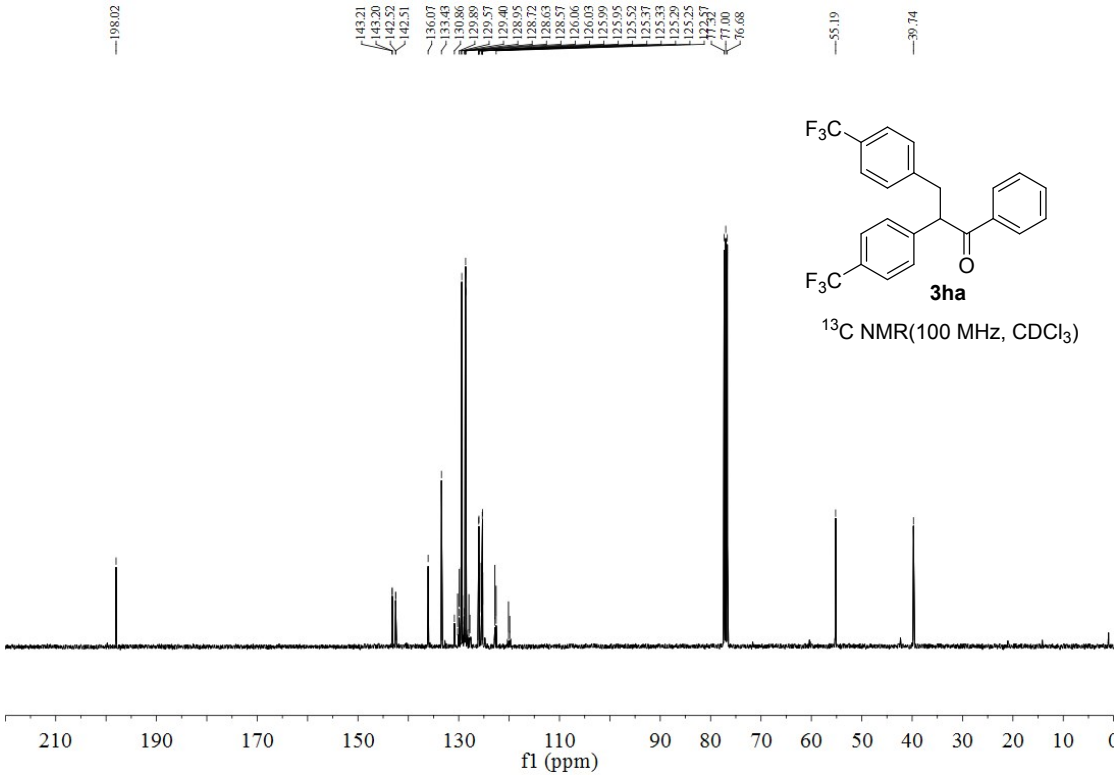
^{1}H NMR (400 MHz, $CDCl_3$)

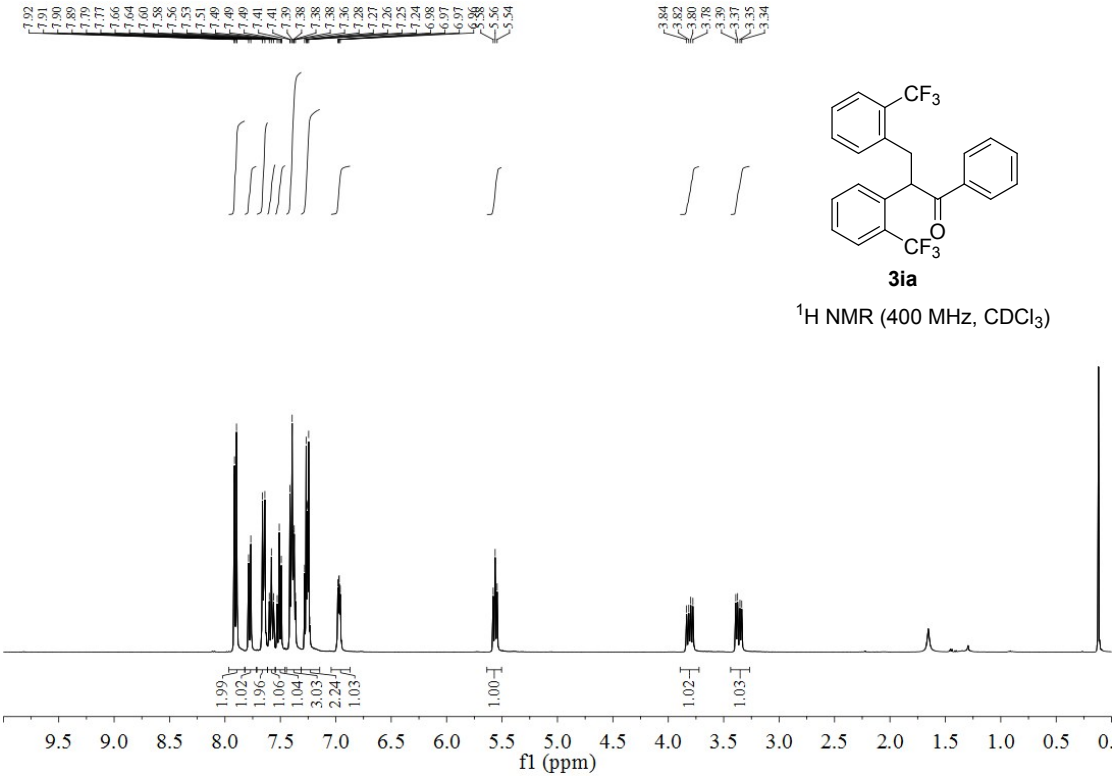


3ga

^{13}C NMR(100 MHz, $CDCl_3$)

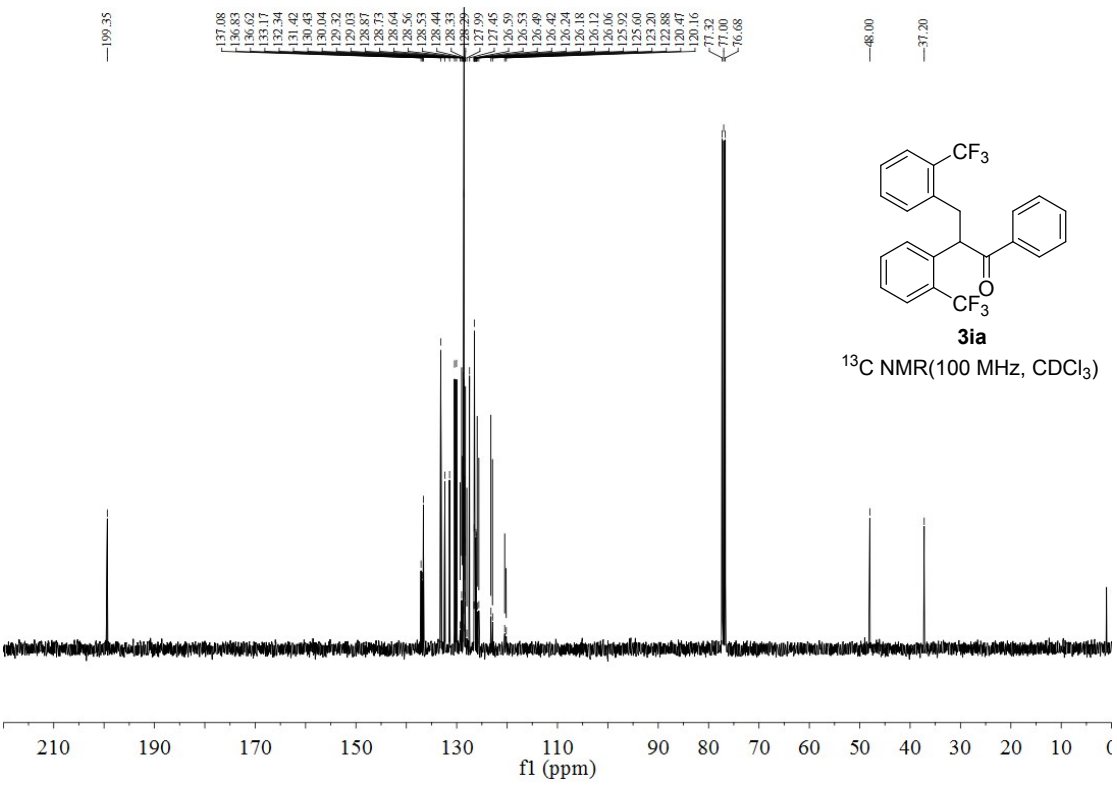

7.87
7.87
7.86
7.85
7.85
7.53
7.51
7.50
7.48
7.47
7.46
7.46
7.46
7.44
7.38
7.38
7.36
7.36
7.35
7.34
7.34
7.33
7.24
7.18
4.86
4.84
3.63
3.61
3.60
3.58
3.13
3.11
3.09
3.08
F3C
O
F3C
3ha
1H NMR (400 MHz, CDCl3)
1.97
4.97
4.01
1.96
1.00
1.01
1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)


198.02
143.21
143.20
142.52
142.51
136.07
133.43
130.86
129.89
129.57
129.40
128.95
128.72
128.63
128.57
126.06
126.03
125.99
125.95
125.52
125.37
125.33
125.29
125.25
77.37
77.00
76.68
55.19
39.74
F3C
O
F3C
3ha
13C NMR(100 MHz, CDCl3)
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10
f1 (ppm)



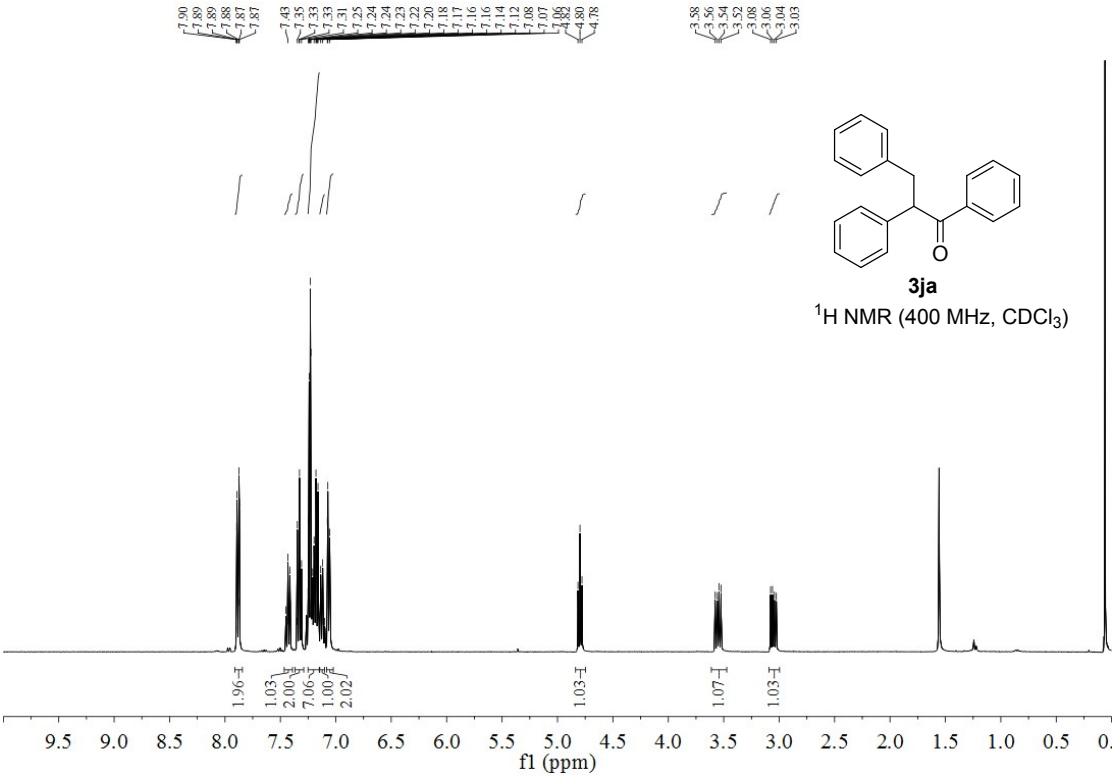
3ia

^{1}H NMR (400 MHz, $CDCl_3$)



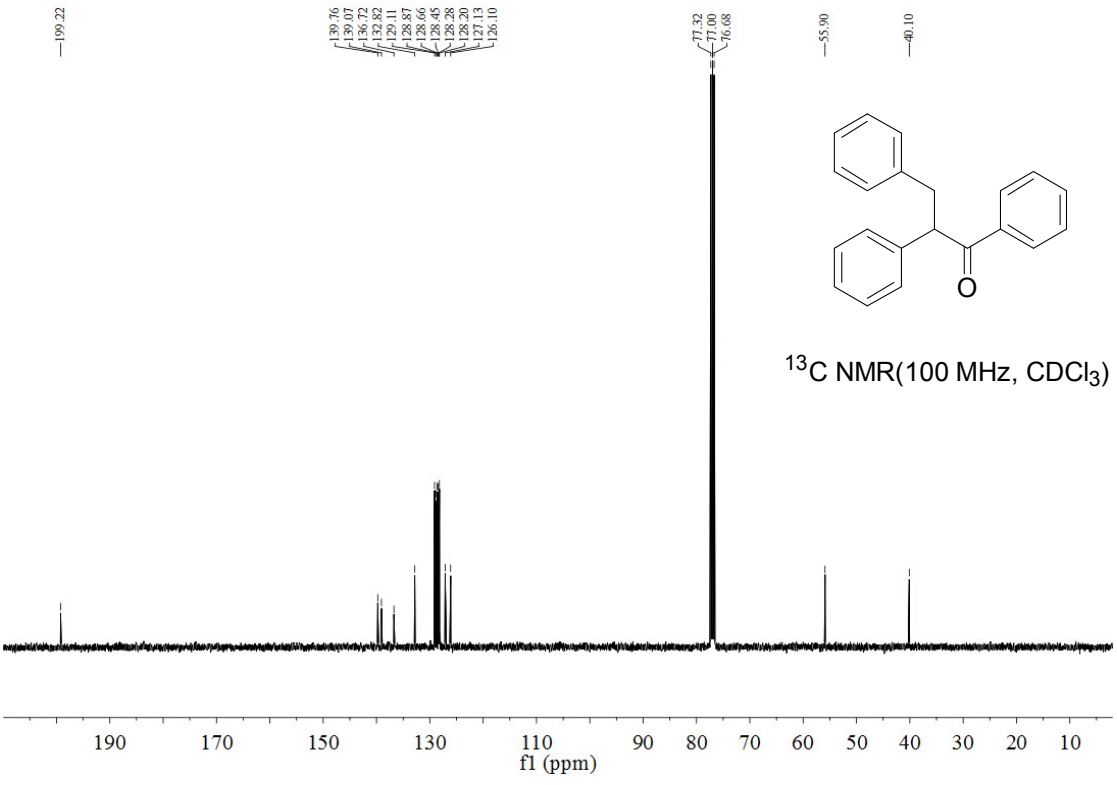
3ia

^{13}C NMR(100 MHz, $CDCl_3$)

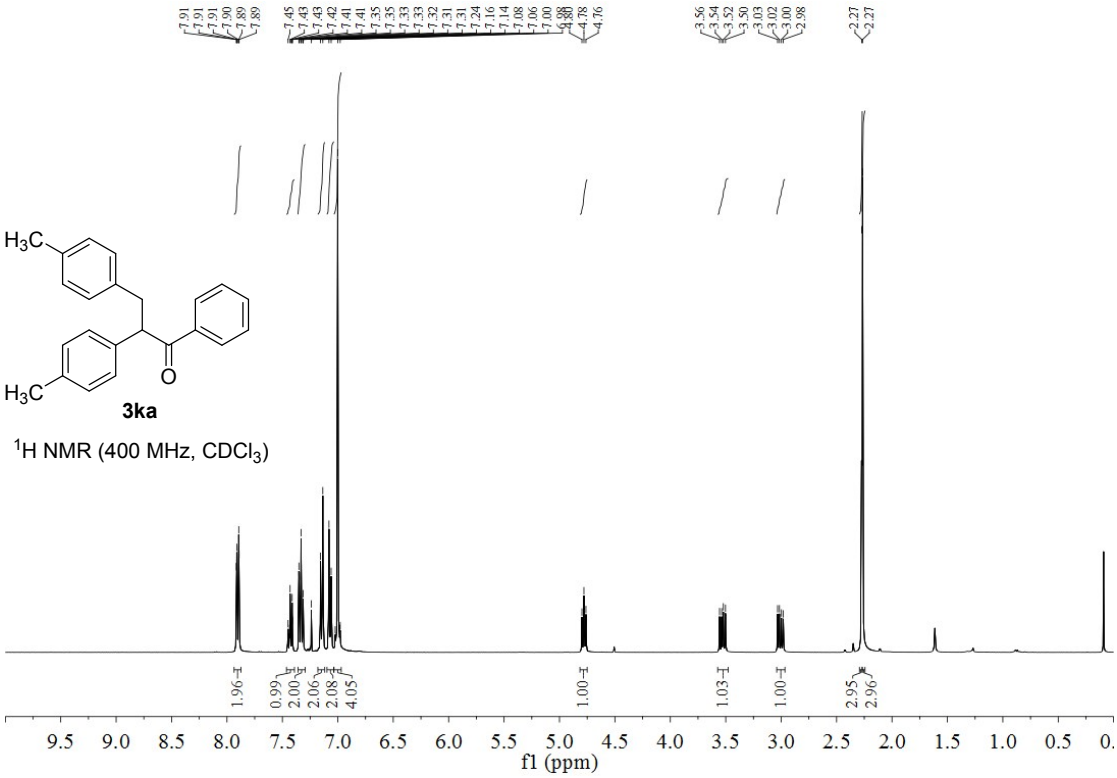


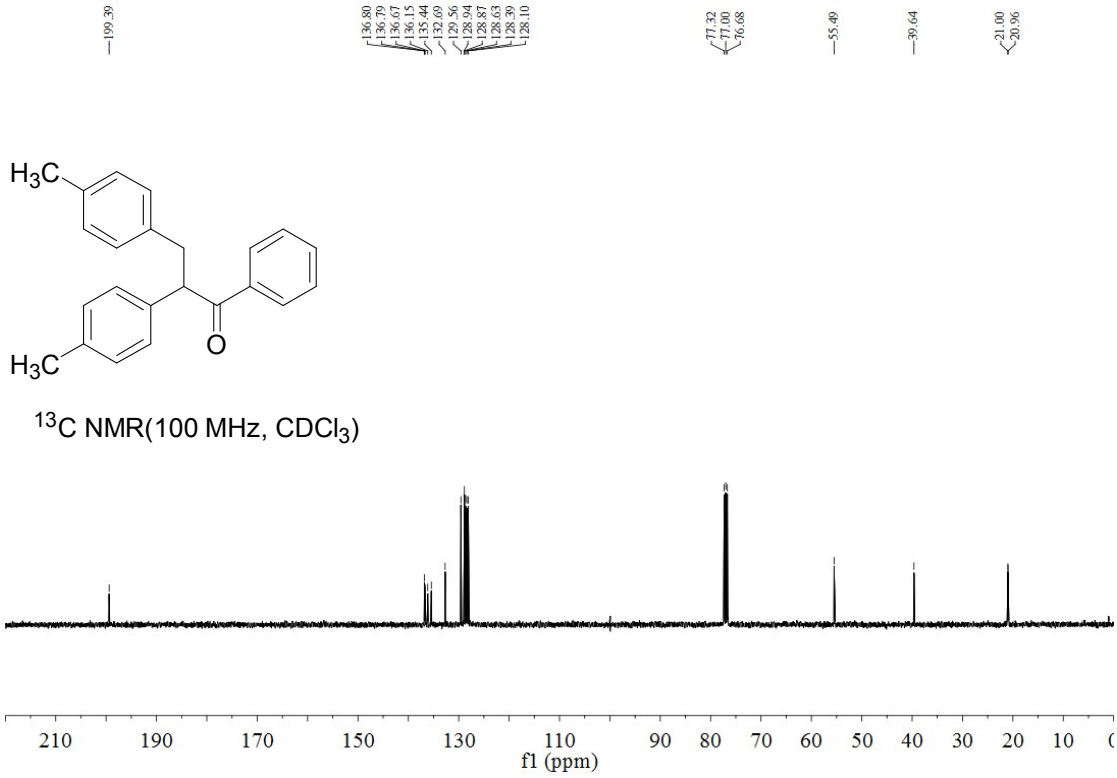
3ja

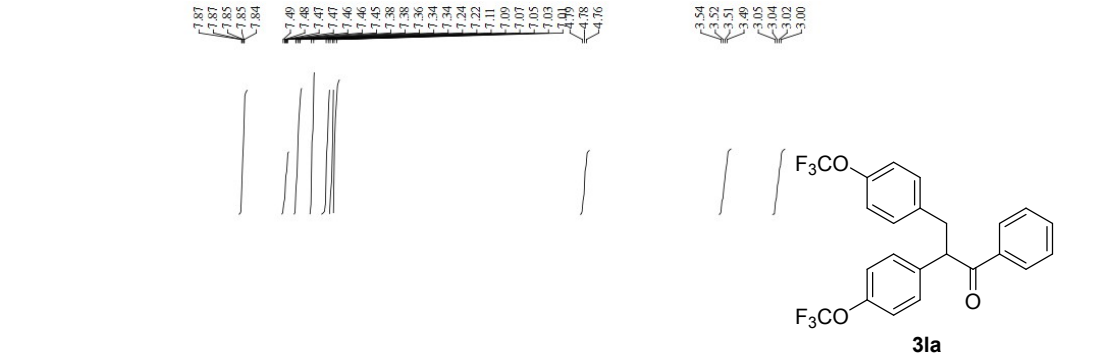
^{1}H NMR (400 MHz, $CDCl_3$)



^{13}C NMR(100 MHz, $CDCl_3$)

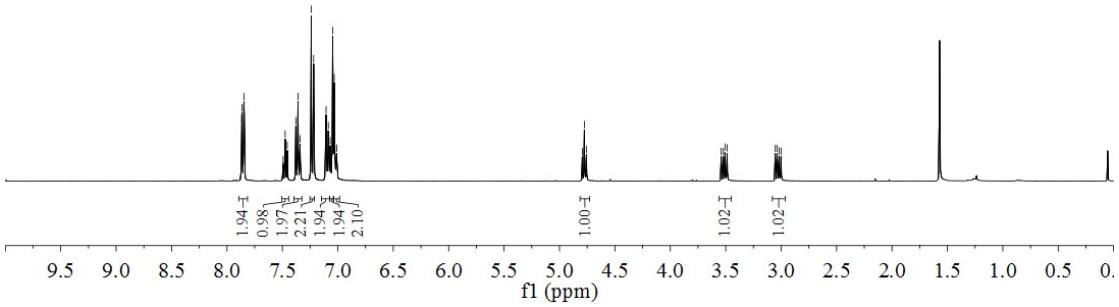

H3C
H3C
O
3ka
1H NMR (400 MHz, CDCl3)
f1 (ppm)

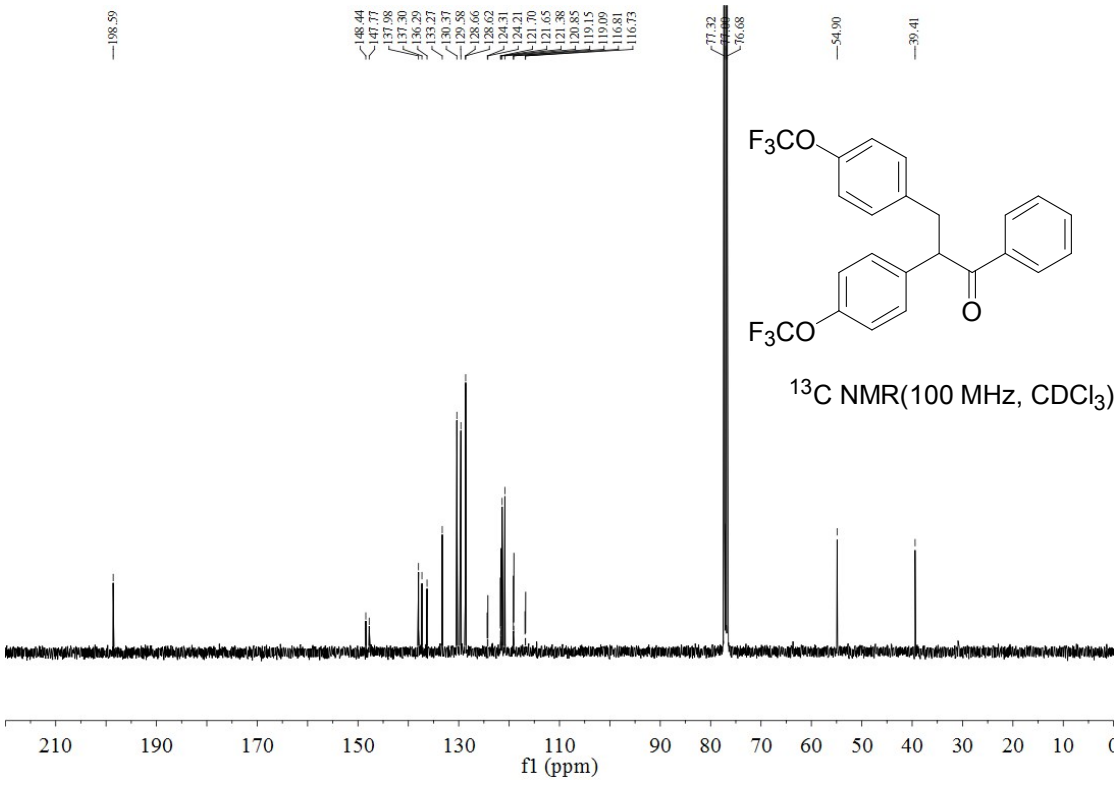

H3C
H3C
O
13C NMR(100 MHz, CDCl3)
f1 (ppm)



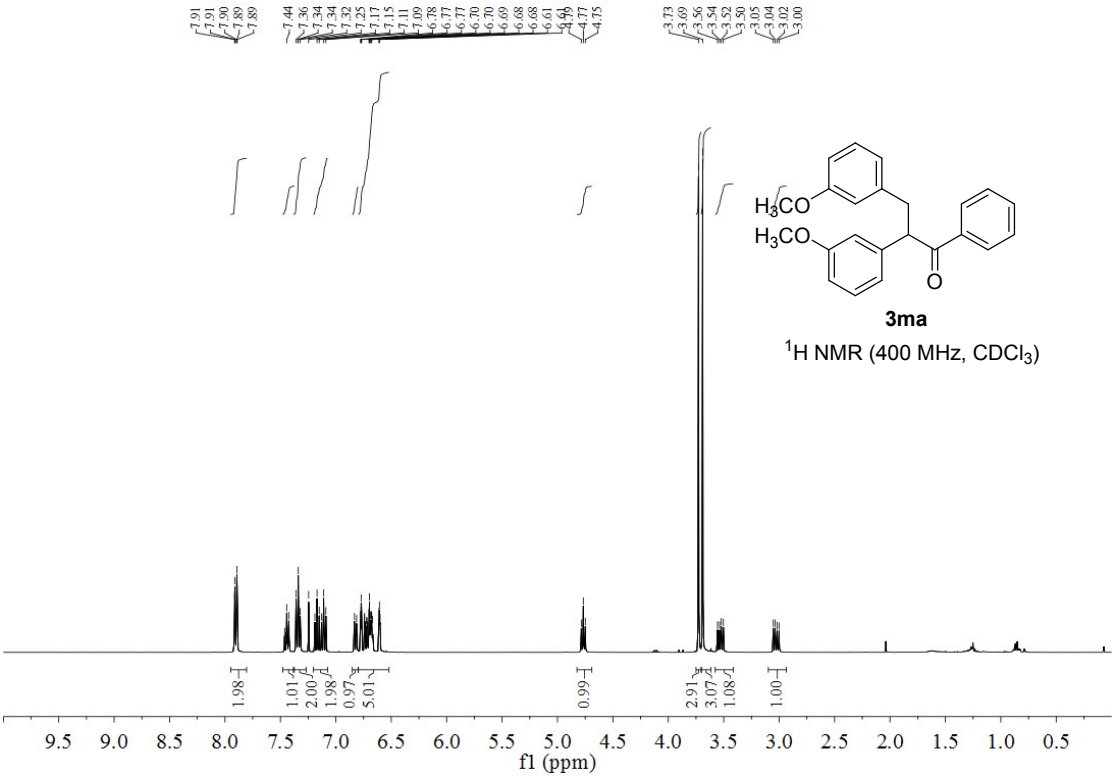
3la

^{1}H NMR (400 MHz, $CDCl_3$)



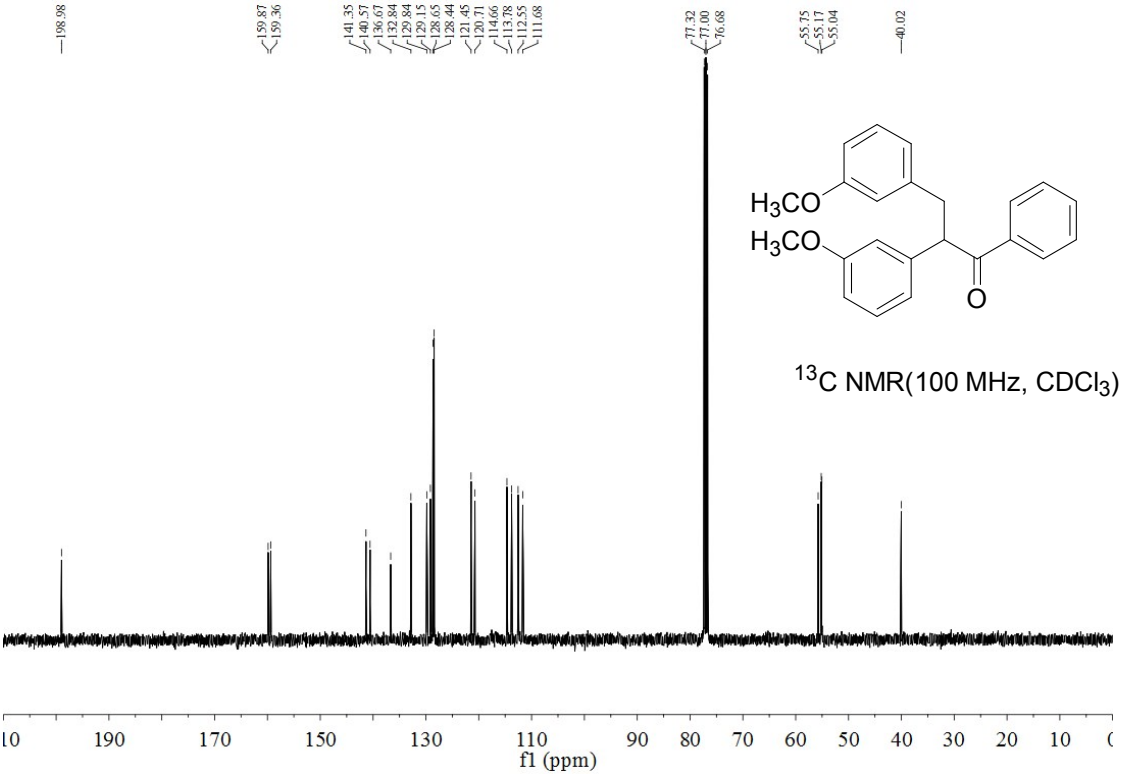


^{13}C NMR(100 MHz, $CDCl_3$)

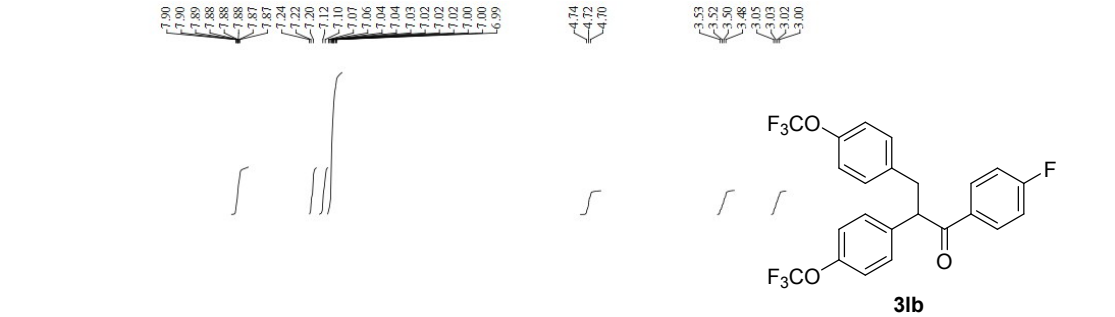


3ma

^{1}H NMR (400 MHz, $CDCl_3$)

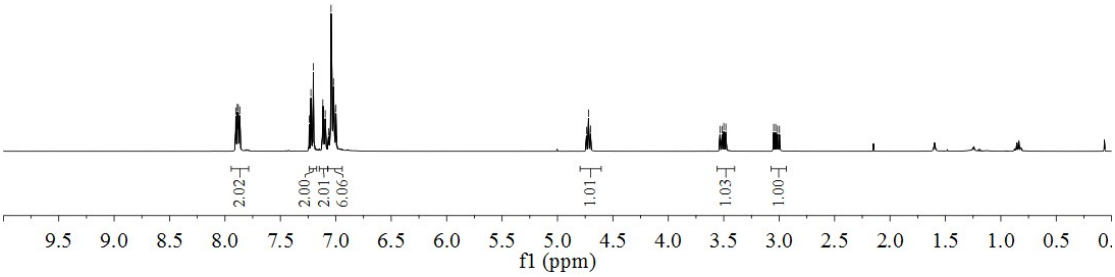


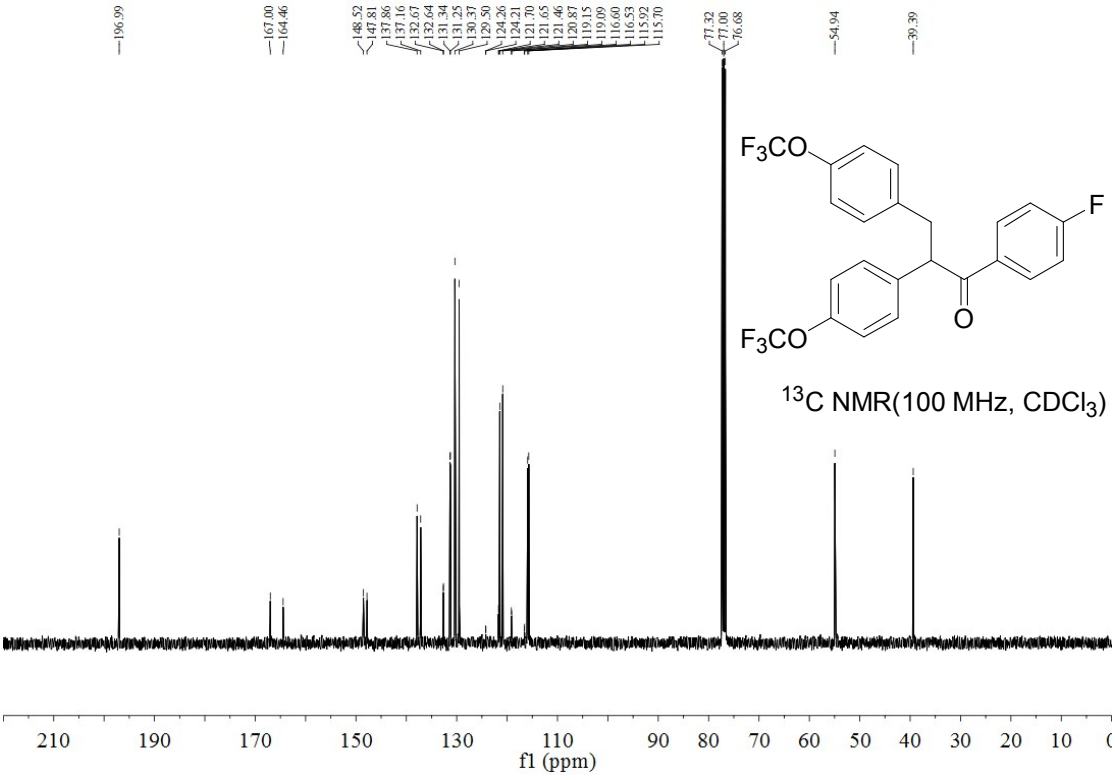
^{13}C NMR(100 MHz, $CDCl_3$)



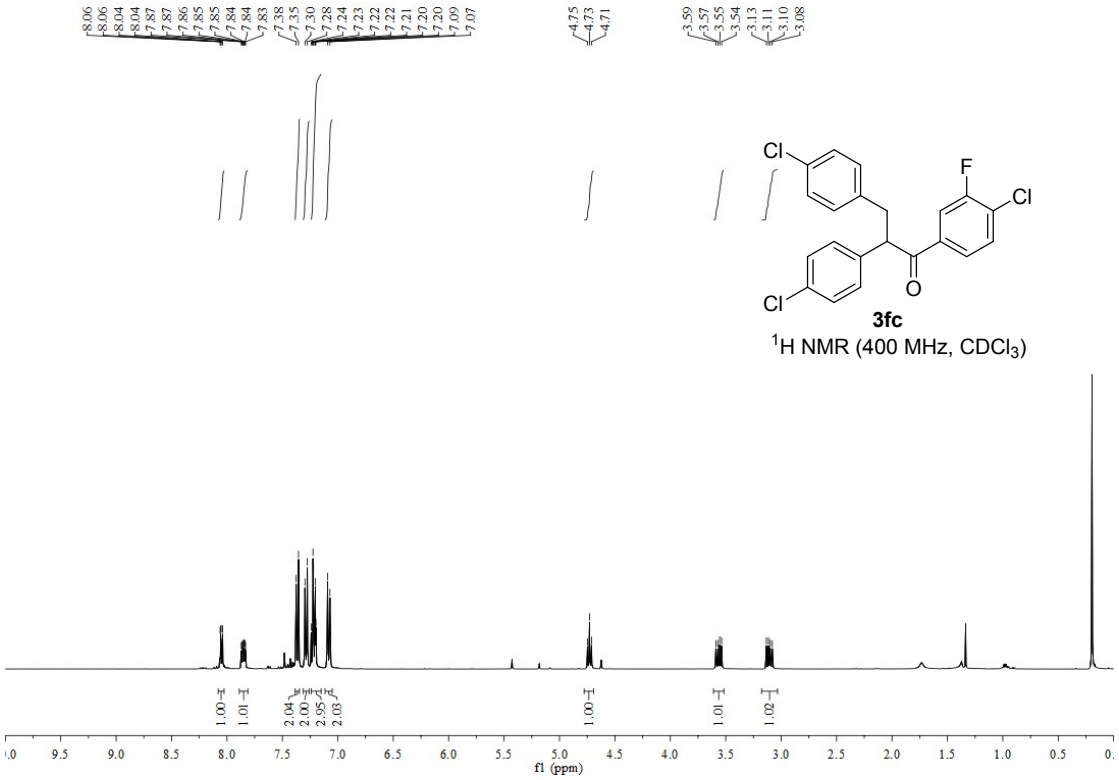
3lb

^{1}H NMR (400 MHz, $CDCl_3$)



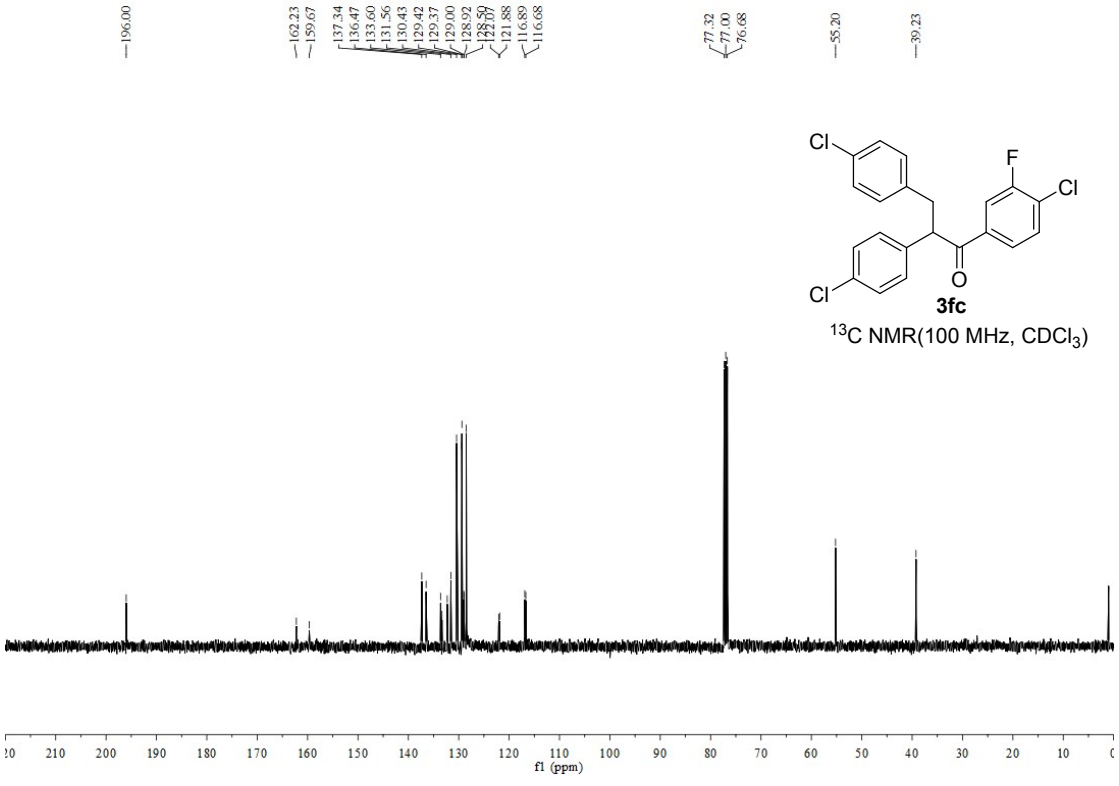


^{13}C NMR(100 MHz, $CDCl_3$)



3fc

^{1}H NMR (400 MHz, $CDCl_3$)

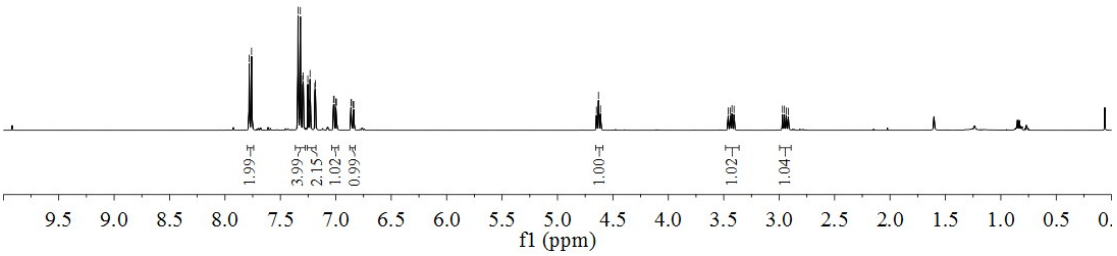


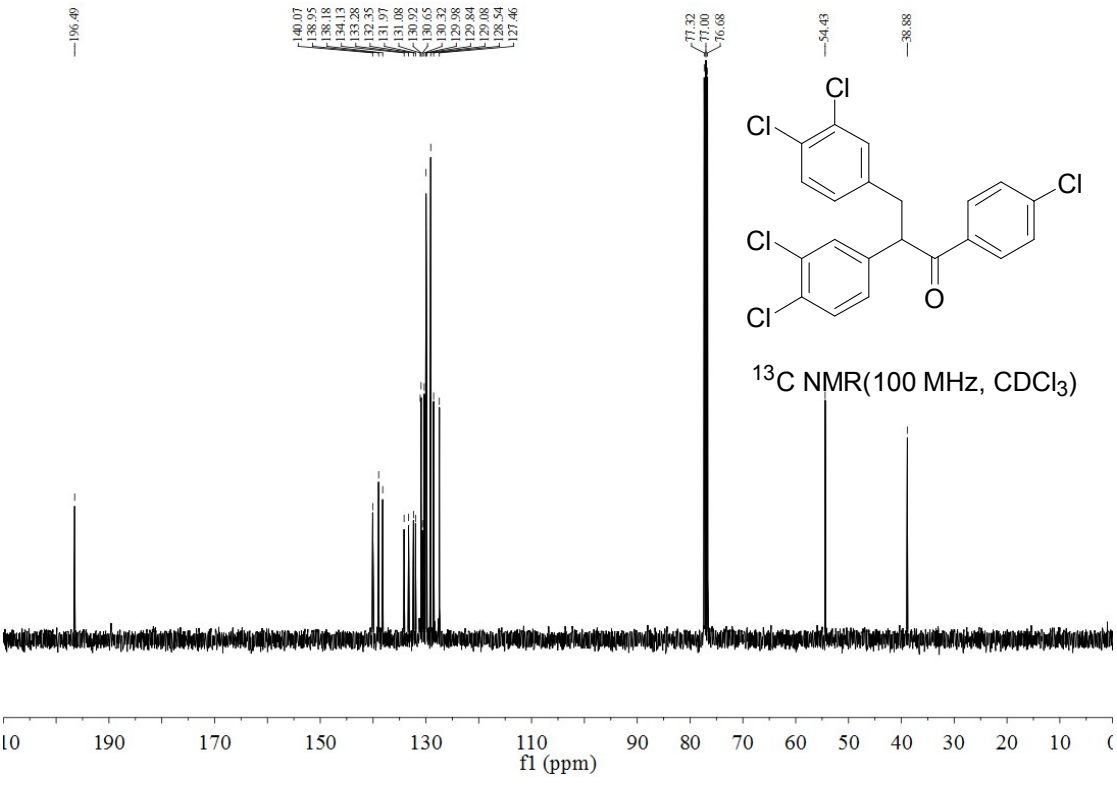
3fc

^{13}C NMR(100 MHz, $CDCl_3$)

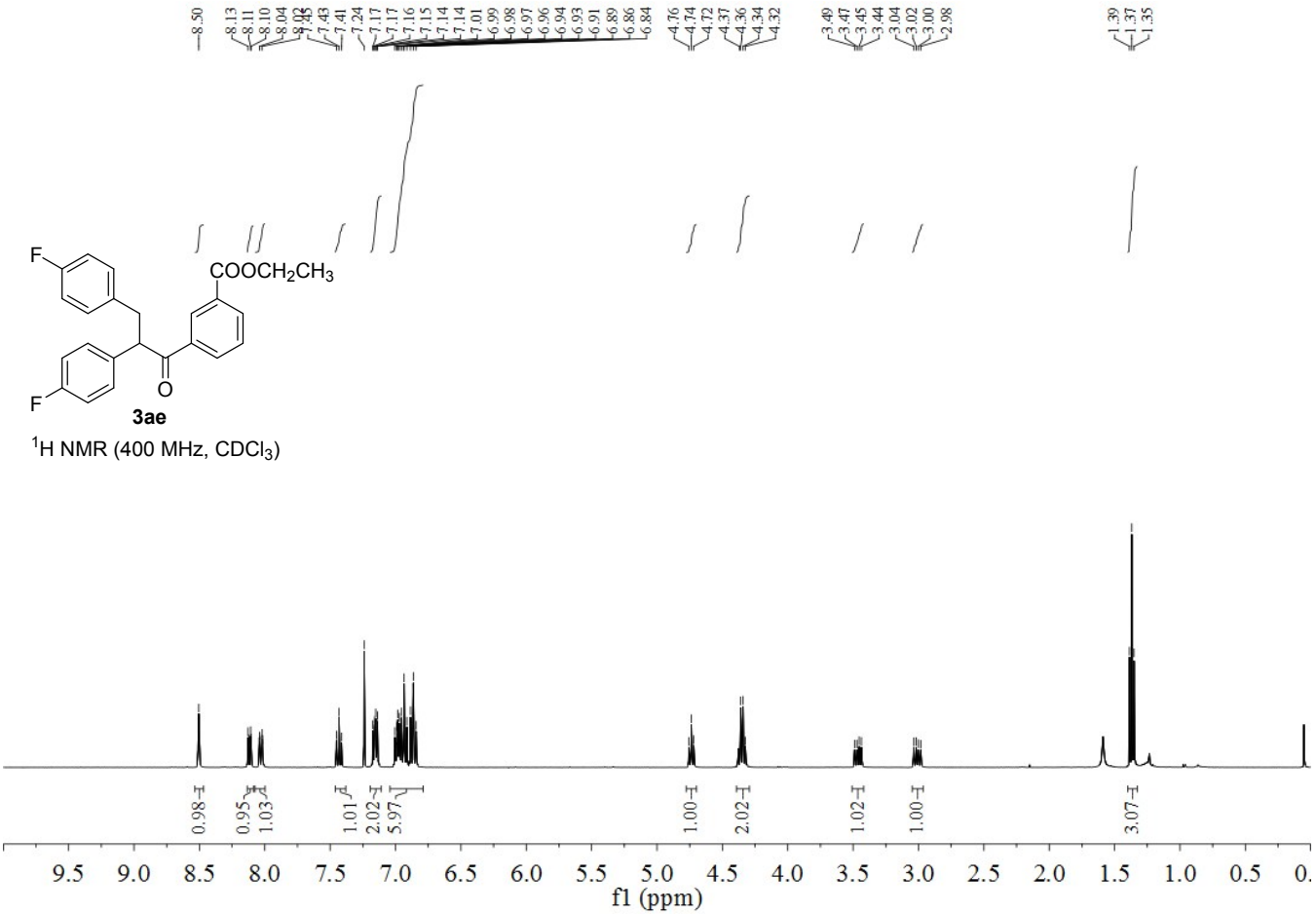
3dd

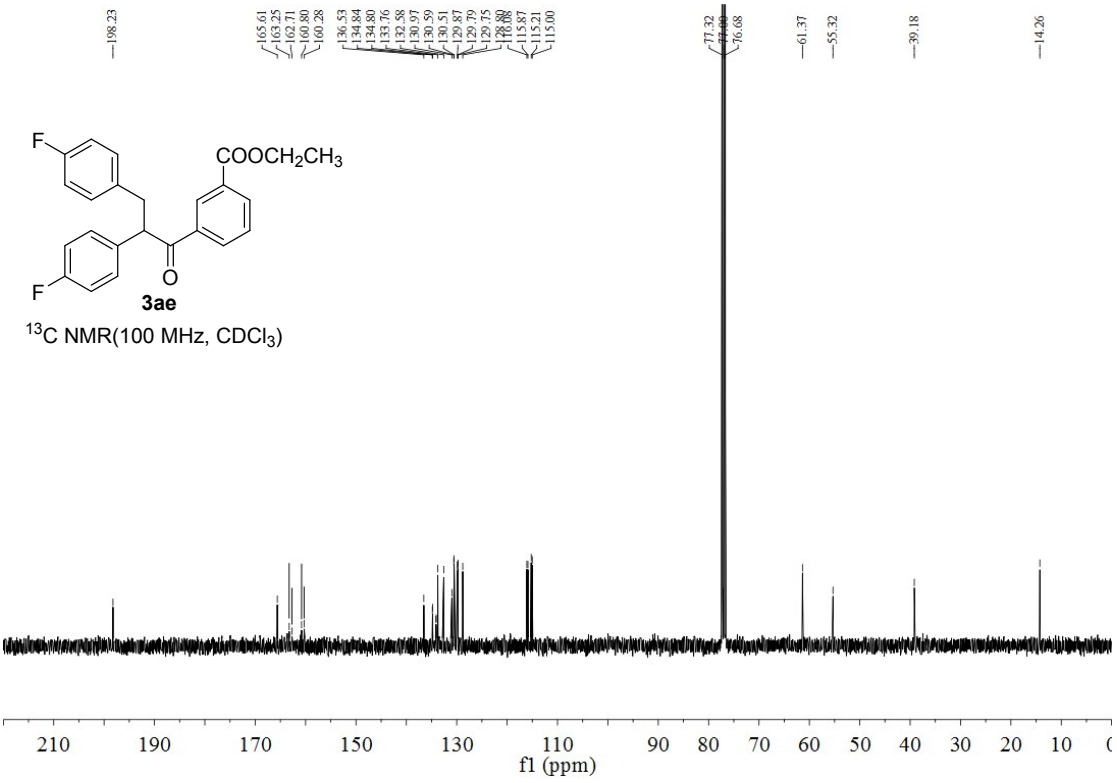
^{1}H NMR (400 MHz, $CDCl_3$)

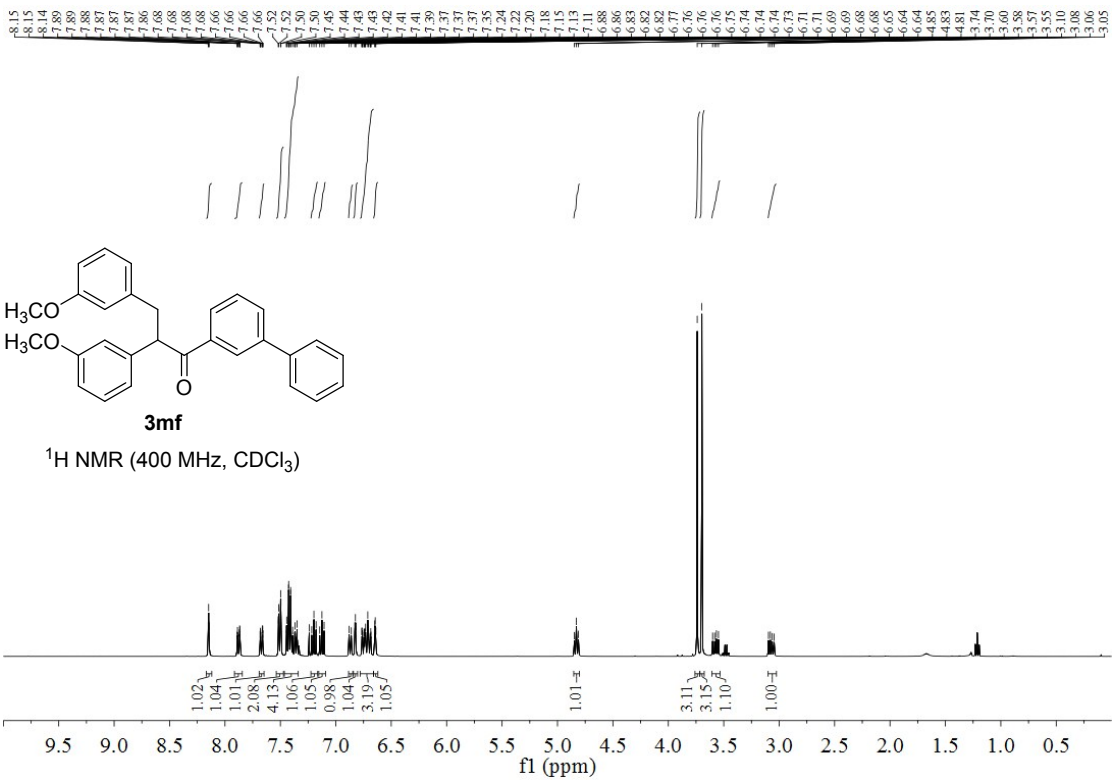


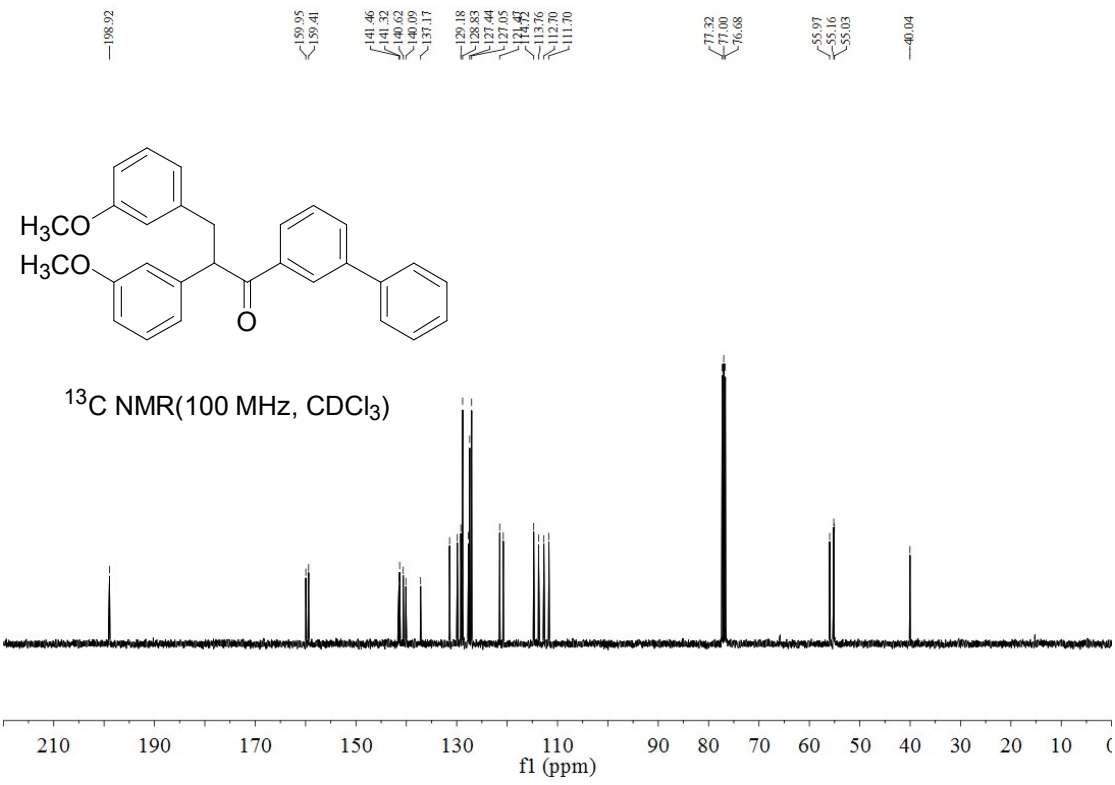


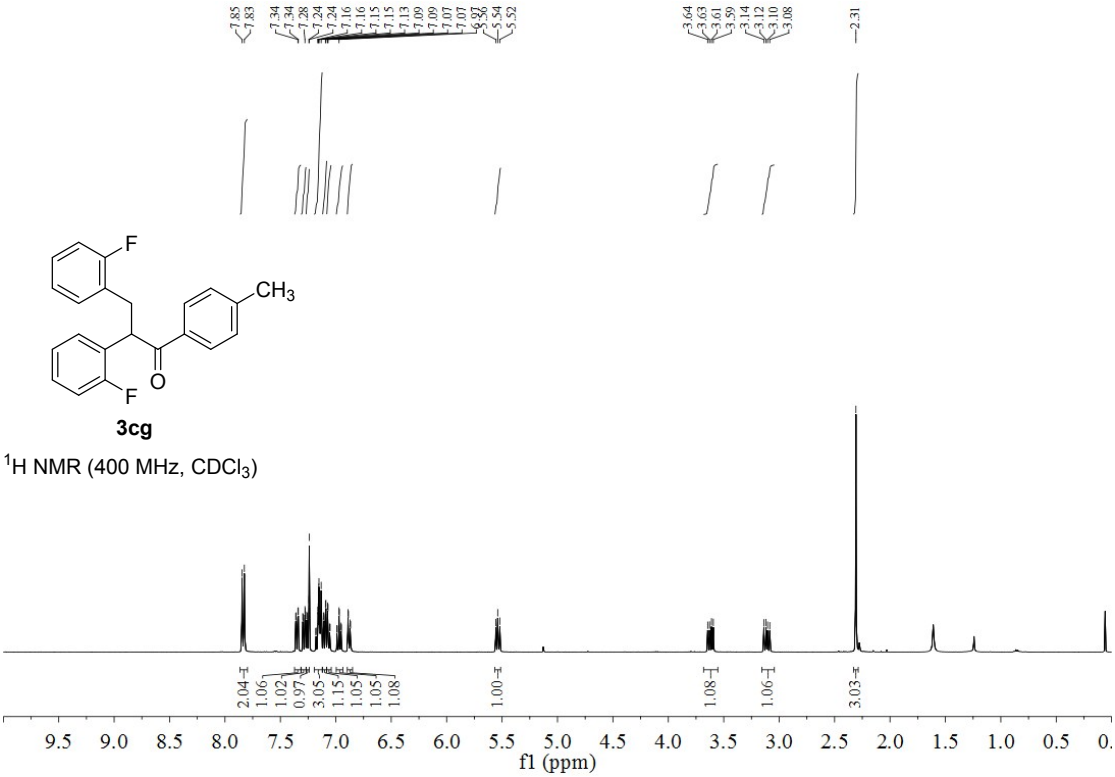
^{13}C NMR(100 MHz, $CDCl_3$)


F
COOCH2CH3
O
F
3ae
1H NMR (400 MHz, CDCl3)
f1 (ppm)


F
COOCH2CH3
O
F
3ae
13C NMR(100 MHz, CDCl3)
f1 (ppm)

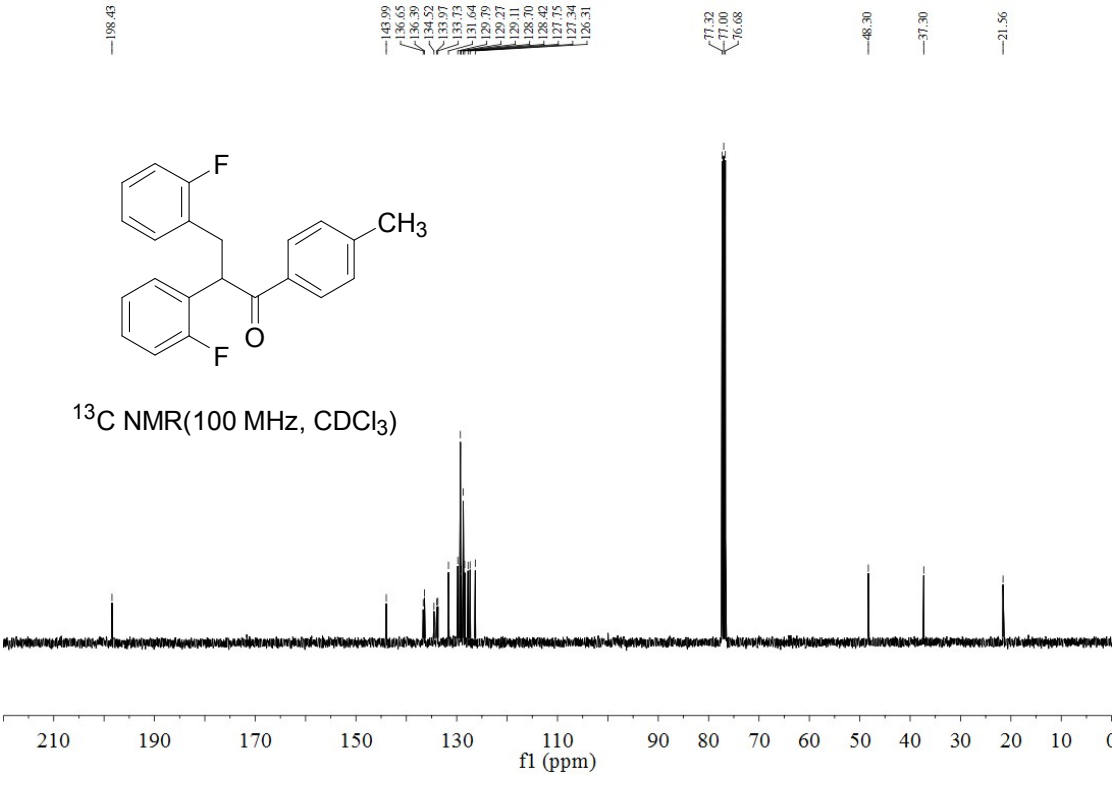

H3CO
H3CO
O
3mf
1H NMR (400 MHz, CDCl3)
f1 (ppm)


H3CO
H3CO
O
13C NMR(100 MHz, CDCl3)
f1 (ppm)

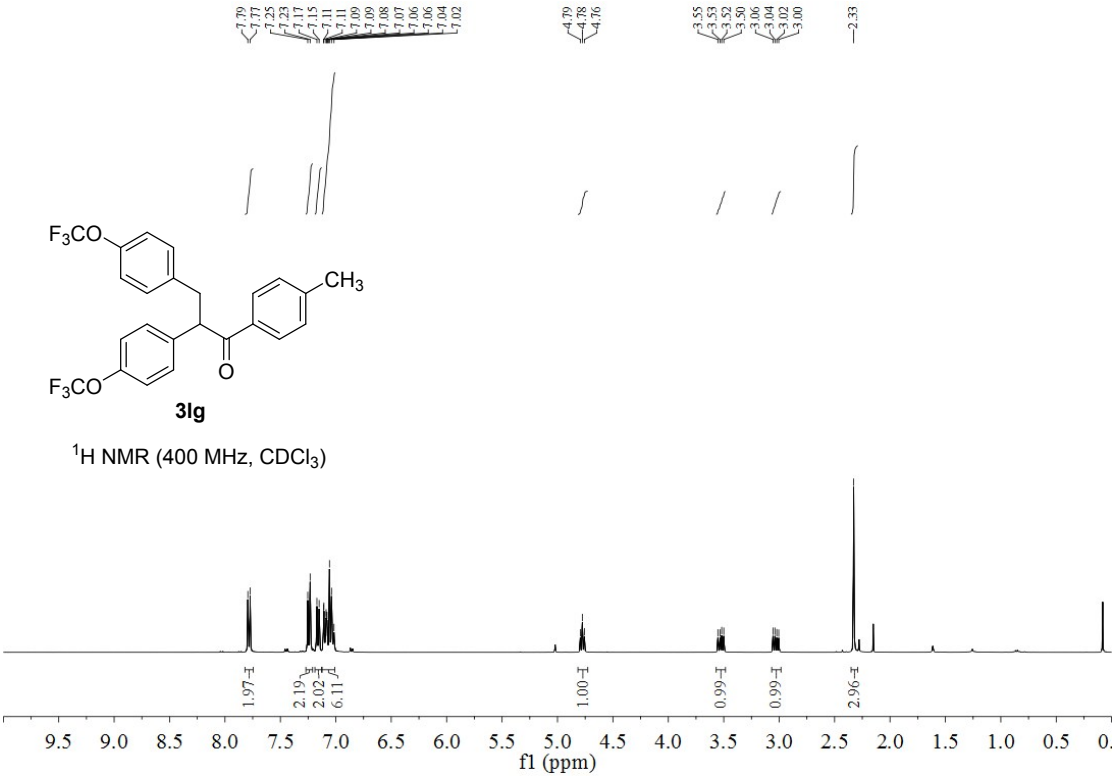


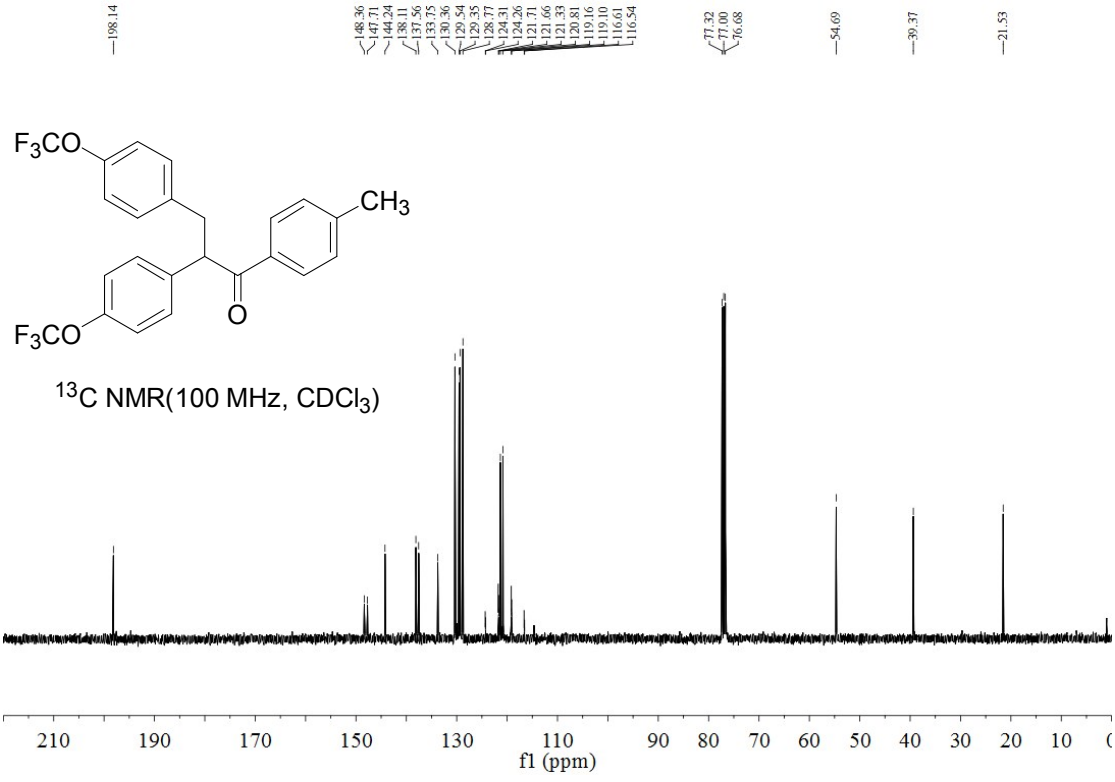
3cg

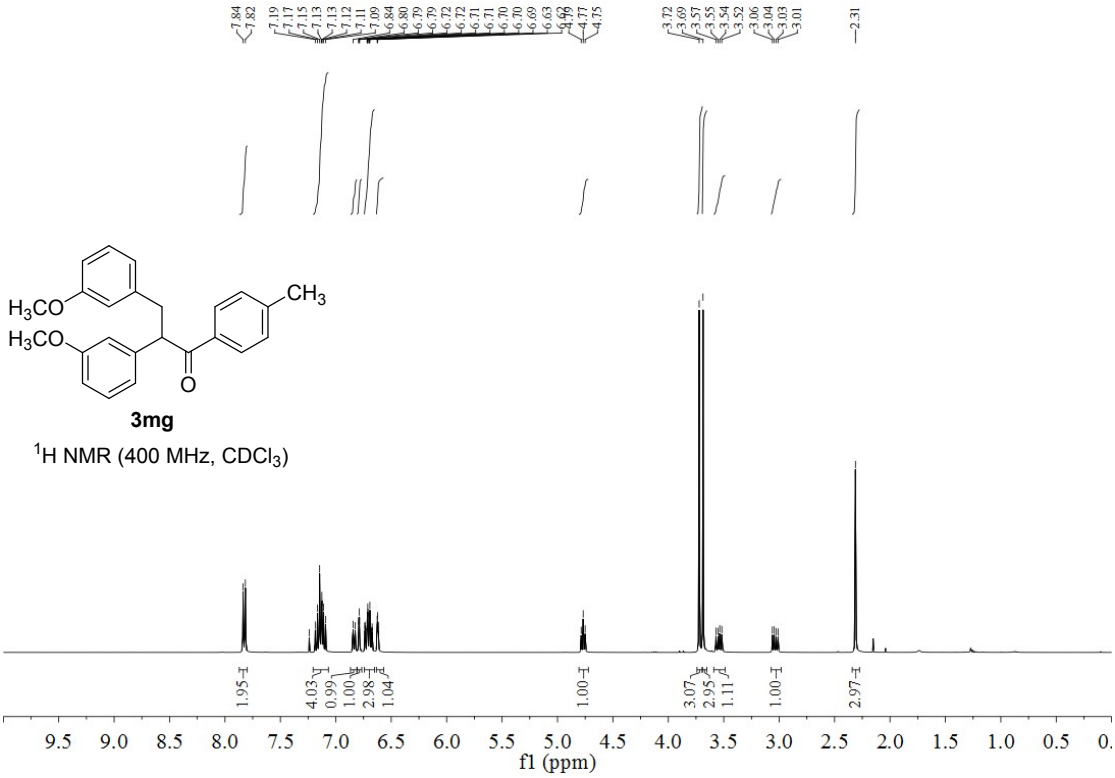
^{1}H NMR (400 MHz, $CDCl_3$)

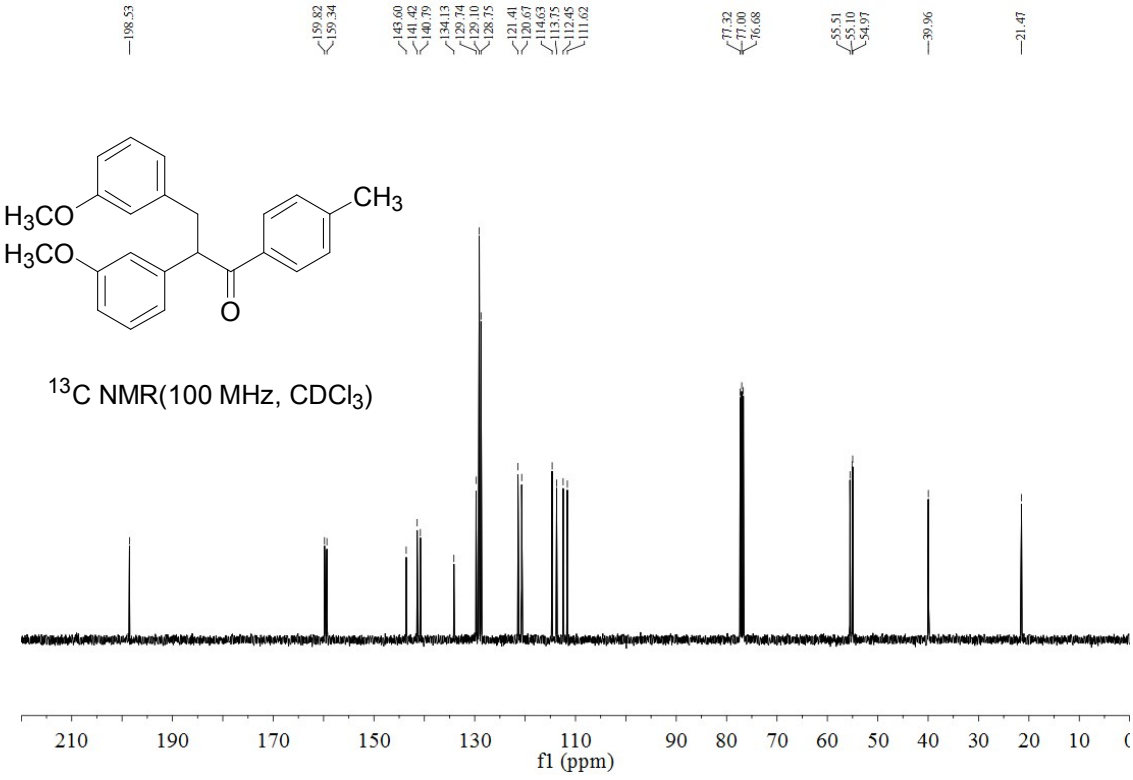


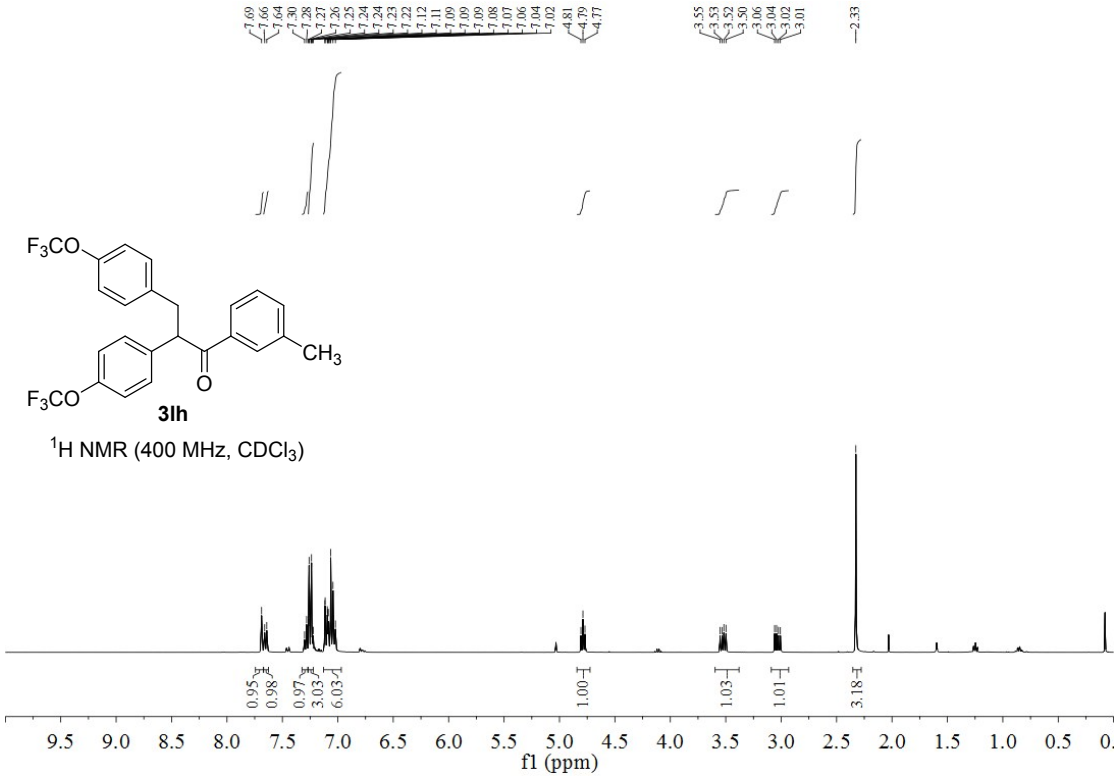
^{13}C NMR(100 MHz, $CDCl_3$)

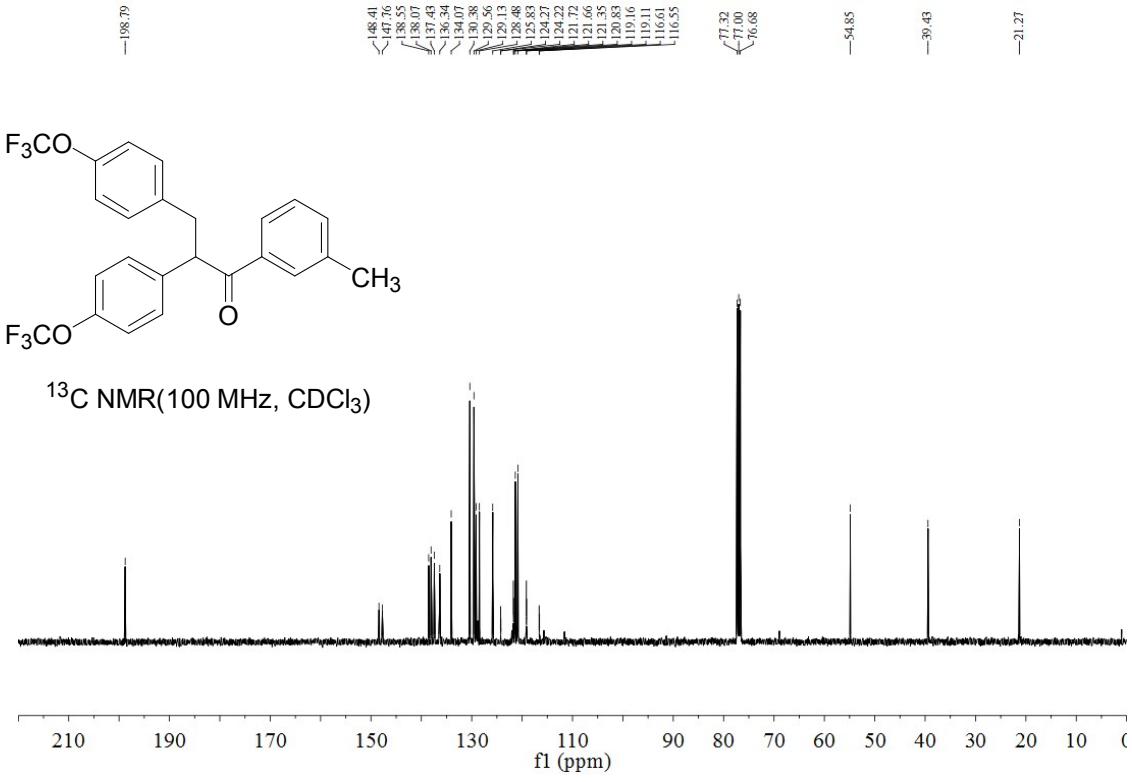

7.79
7.77
7.25
7.23
7.17
7.15
7.11
7.11
7.09
7.09
7.08
7.07
7.06
7.06
7.04
7.02
4.79
4.78
4.76
3.55
3.53
3.52
3.50
3.06
3.04
3.02
3.00
2.33
F3CO
CH3
O
F3CO
3lg
1H NMR (400 MHz, CDCl3)
1.97
2.19
2.02
6.11
1.00
0.99
0.99
2.96
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.
f1 (ppm)

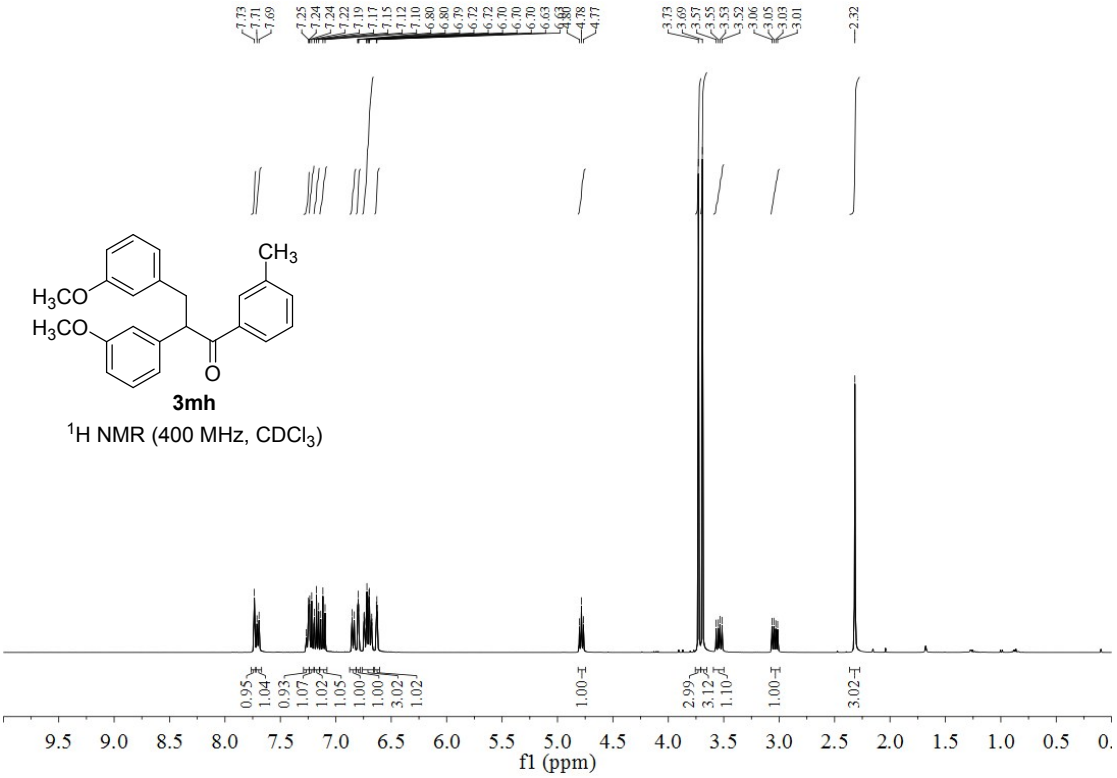

198.14
148.36
147.71
144.24
138.11
137.56
133.75
130.36
129.54
129.35
128.77
124.31
124.26
121.71
121.66
121.33
120.81
119.16
119.10
116.61
116.54
77.32
77.00
76.68
54.69
39.37
21.53
F3CO
CH3
O
F3CO
13C NMR(100 MHz, CDCl3)
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 (
f1 (ppm)

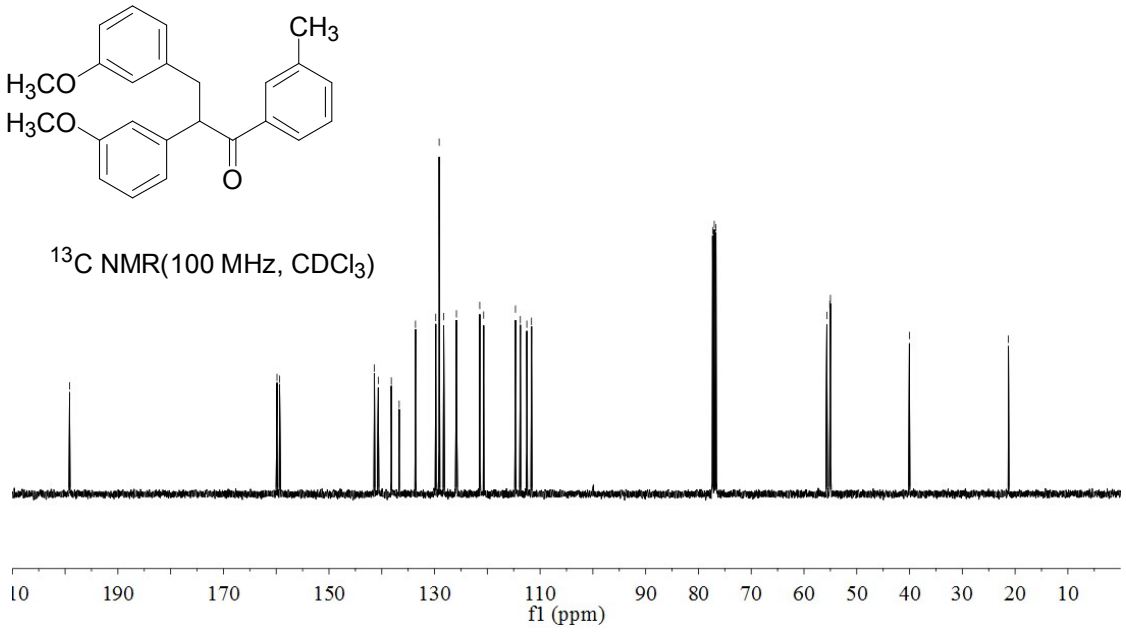

H3CO
H3CO
CH3
O
3mg
1H NMR (400 MHz, CDCl3)
f1 (ppm)

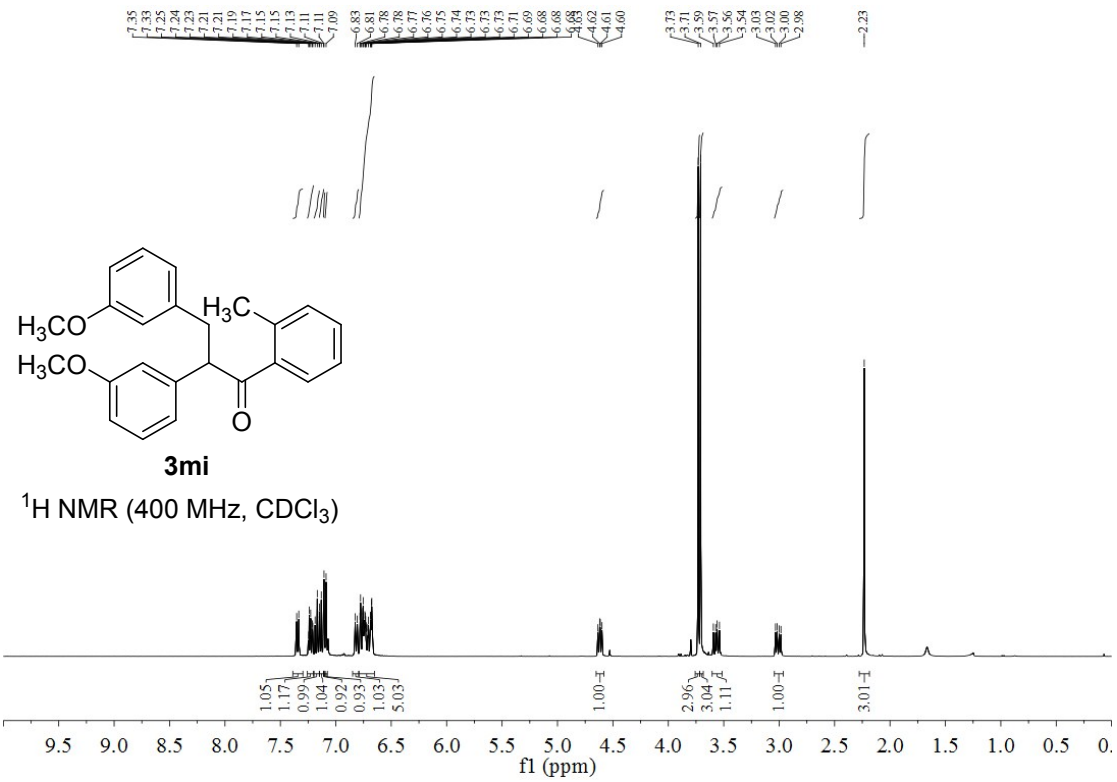

H3CO
H3CO
CH3
O
13C NMR(100 MHz, CDCl3)
f1 (ppm)

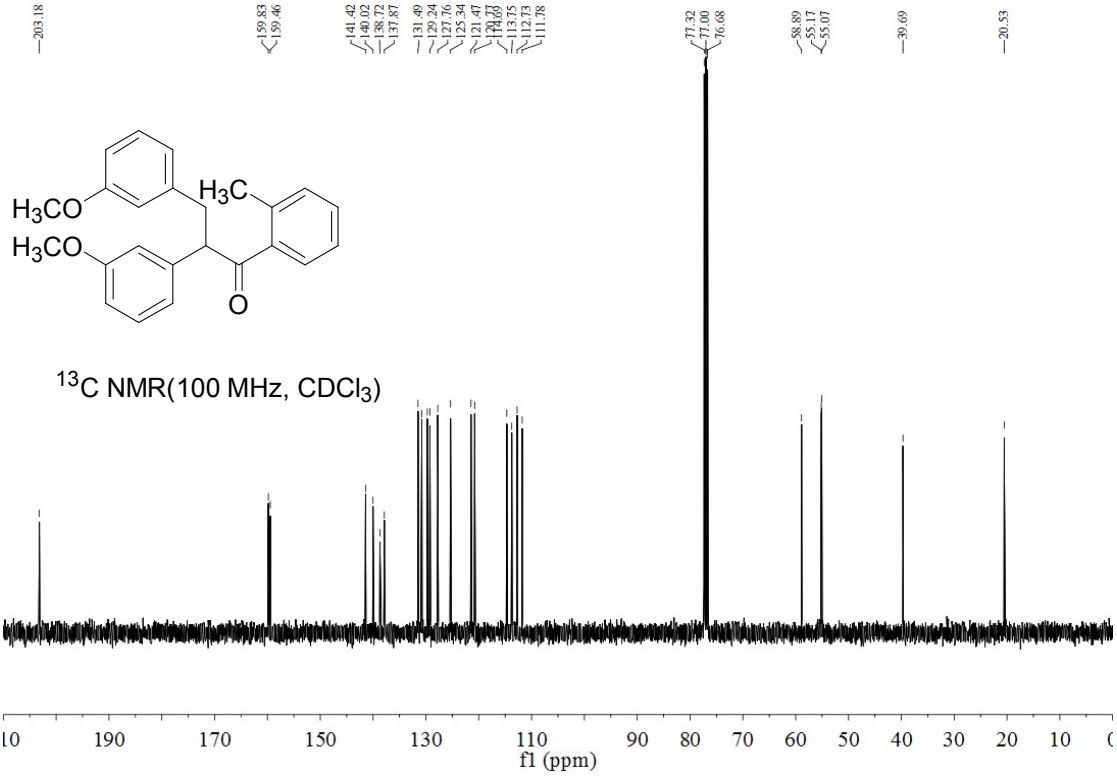

F3CO
CH3
O
F3CO
3Ih
1H NMR (400 MHz, CDCl3)
f1 (ppm)

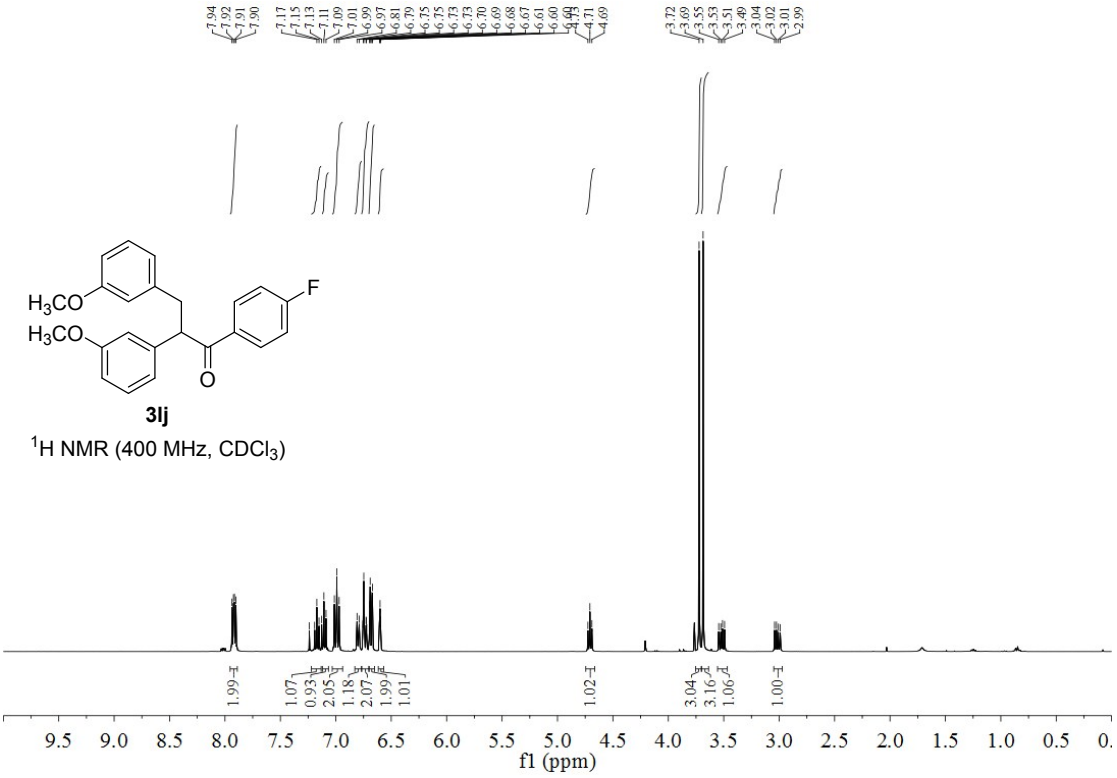

F3CO
CH3
O
F3CO
13C NMR(100 MHz, CDCl3)
f1 (ppm)


CH3
H3CO
H3CO
O
3mh
1H NMR (400 MHz, CDCl3)
f1 (ppm)


CH3
H3CO
H3CO
O
13C NMR(100 MHz, CDCl3)
f1 (ppm)

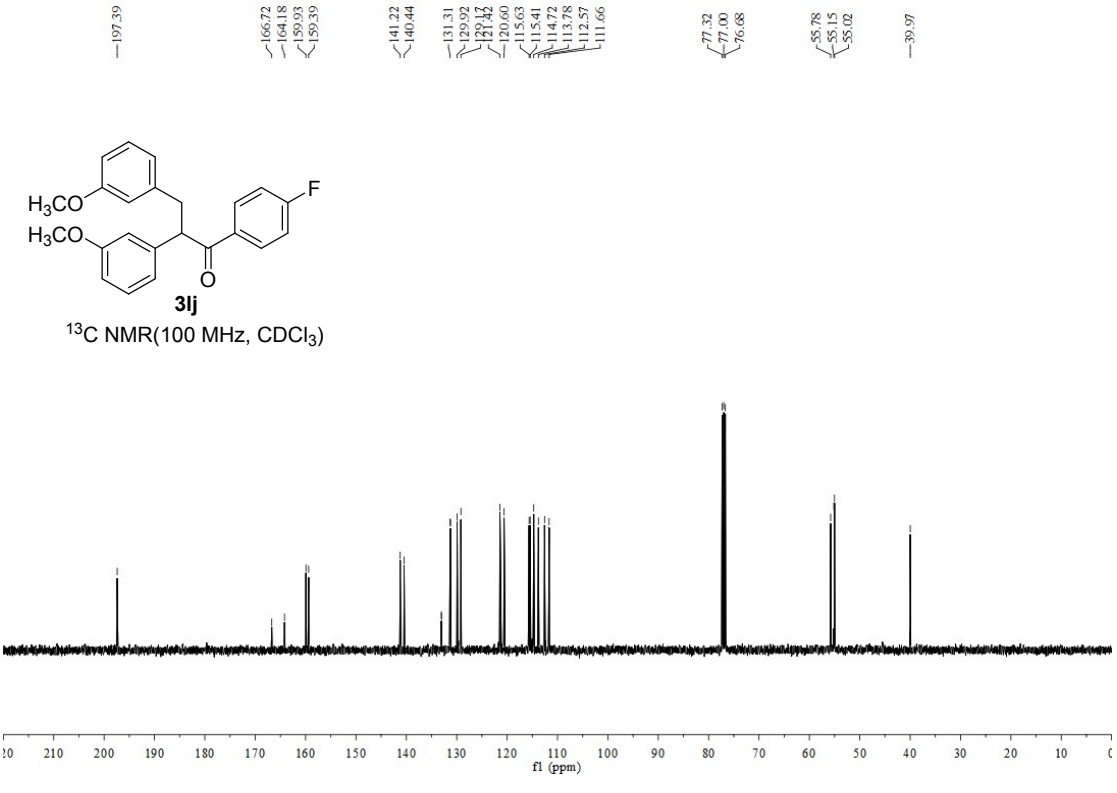

H3CO
H3CO
H3C
O
3mi
1H NMR (400 MHz, CDCl3)
f1 (ppm)


H3CO
H3CO
H3C
O
13C NMR(100 MHz, CDCl3)
f1 (ppm)



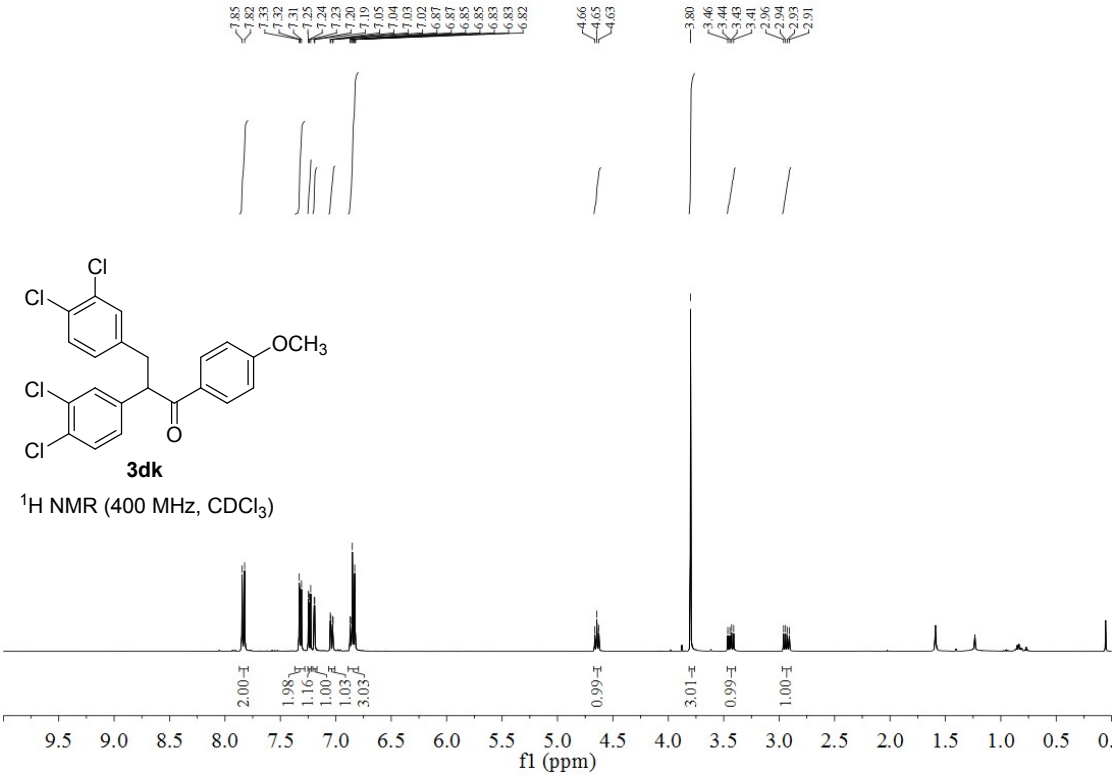
3lj

^{1}H NMR (400 MHz, $CDCl_3$)



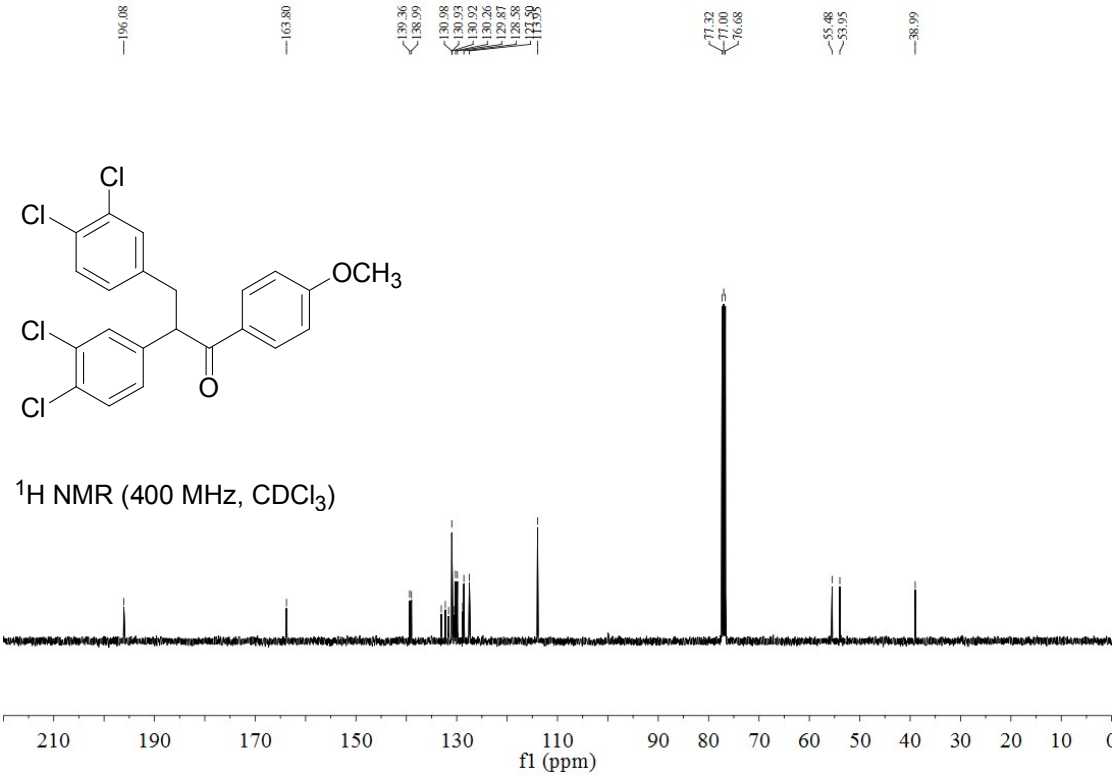
3lj

^{13}C NMR(100 MHz, $CDCl_3$)

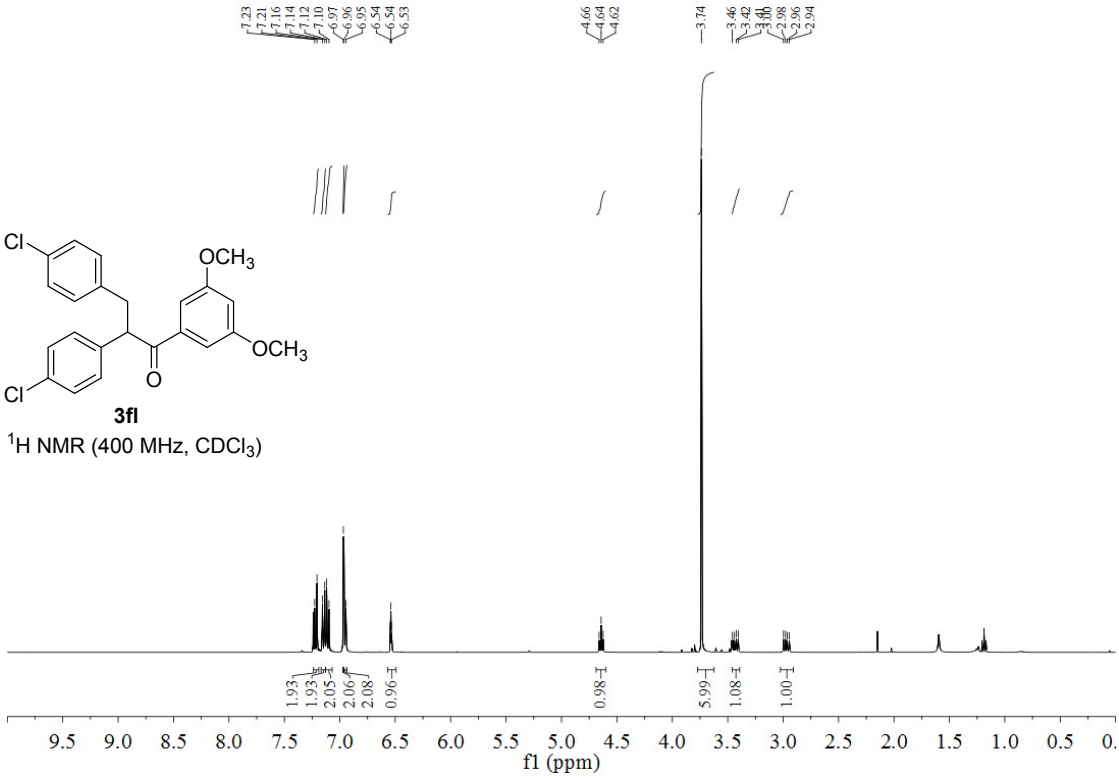


3dk

^{1}H NMR (400 MHz, $CDCl_3$)

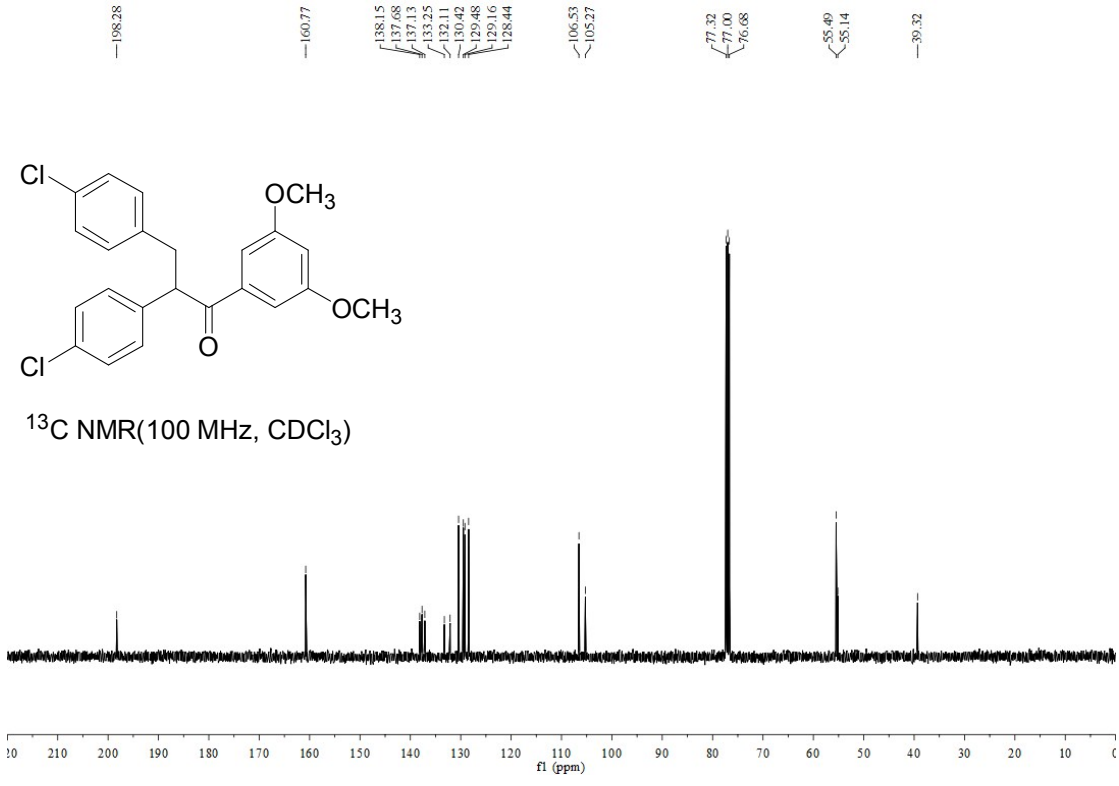


^{1}H NMR (400 MHz, $CDCl_3$)

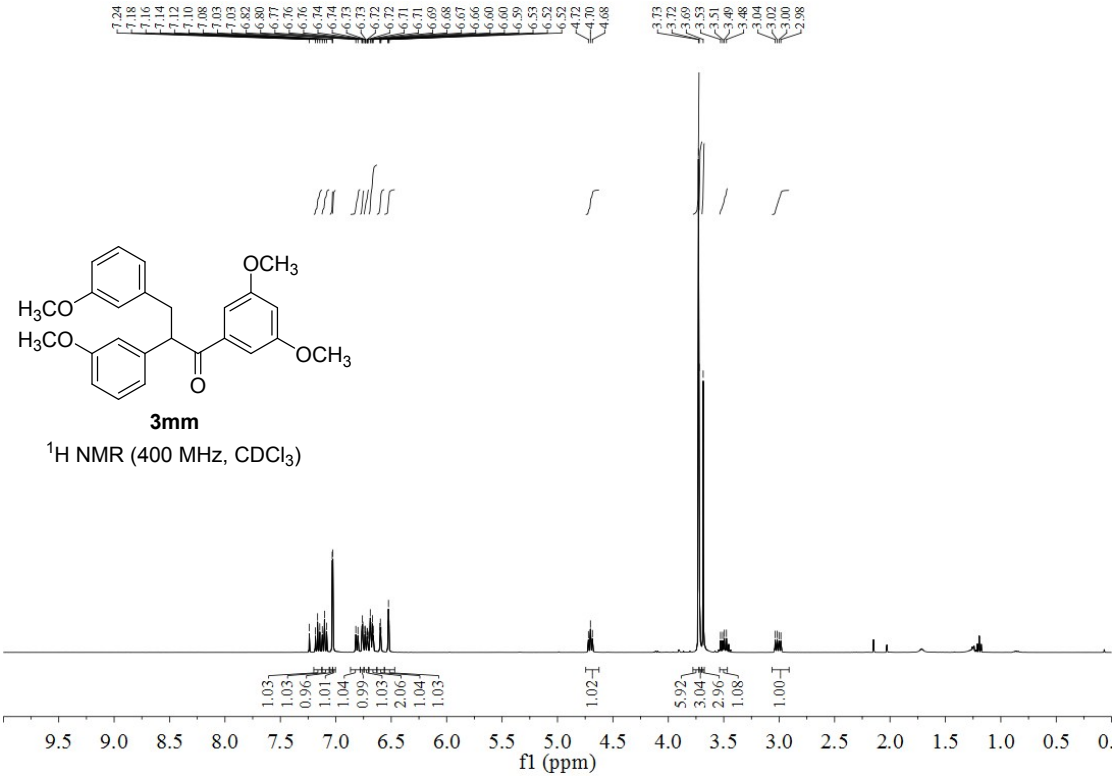


3fl

^{1}H NMR (400 MHz, $CDCl_3$)

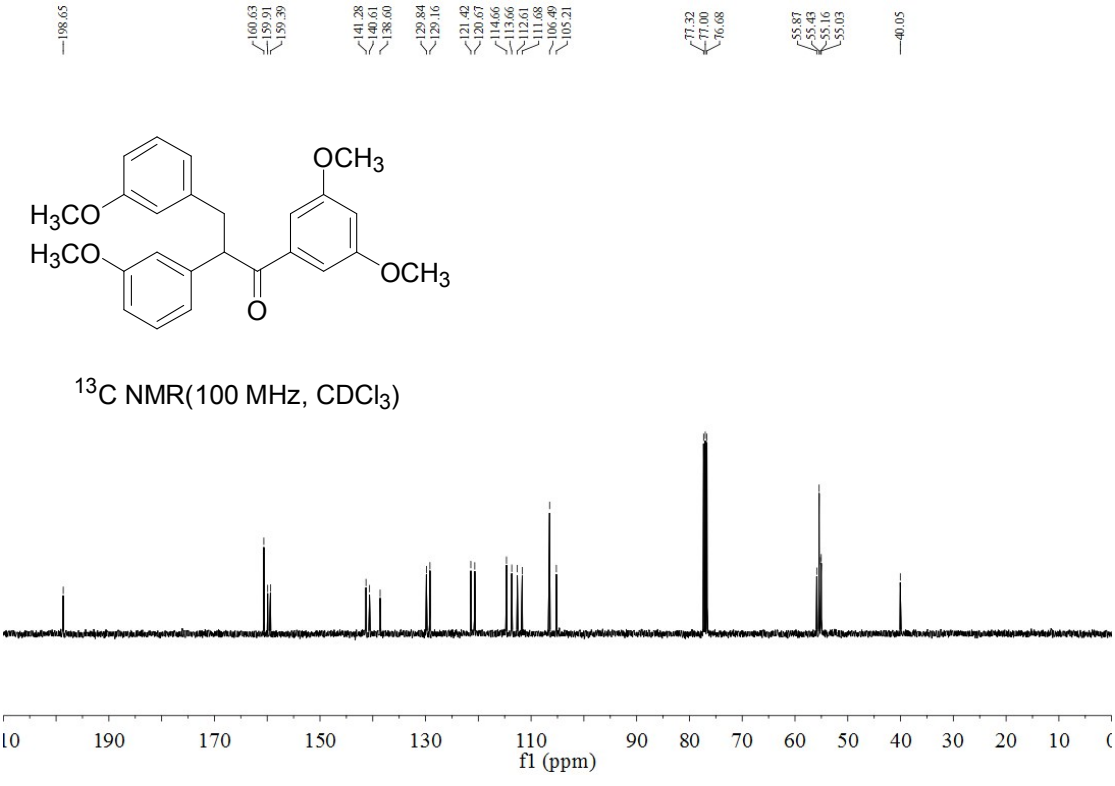


^{13}C NMR(100 MHz, $CDCl_3$)

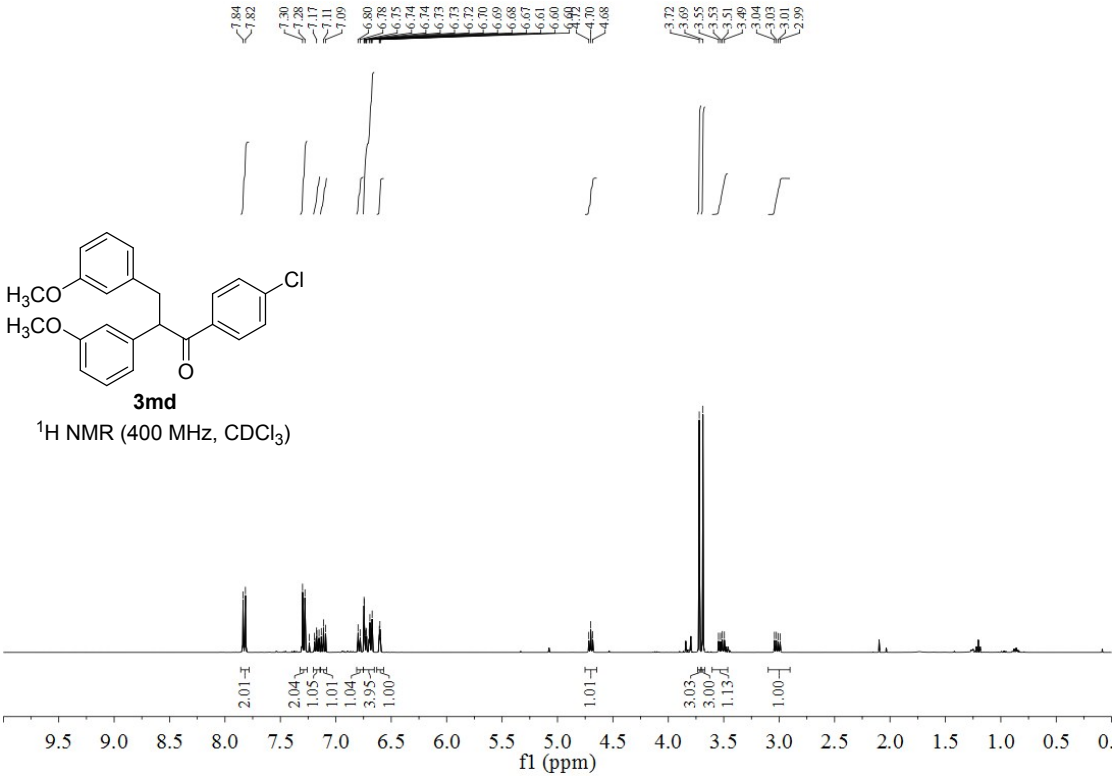


3mm

^{1}H NMR (400 MHz, $CDCl_3$)

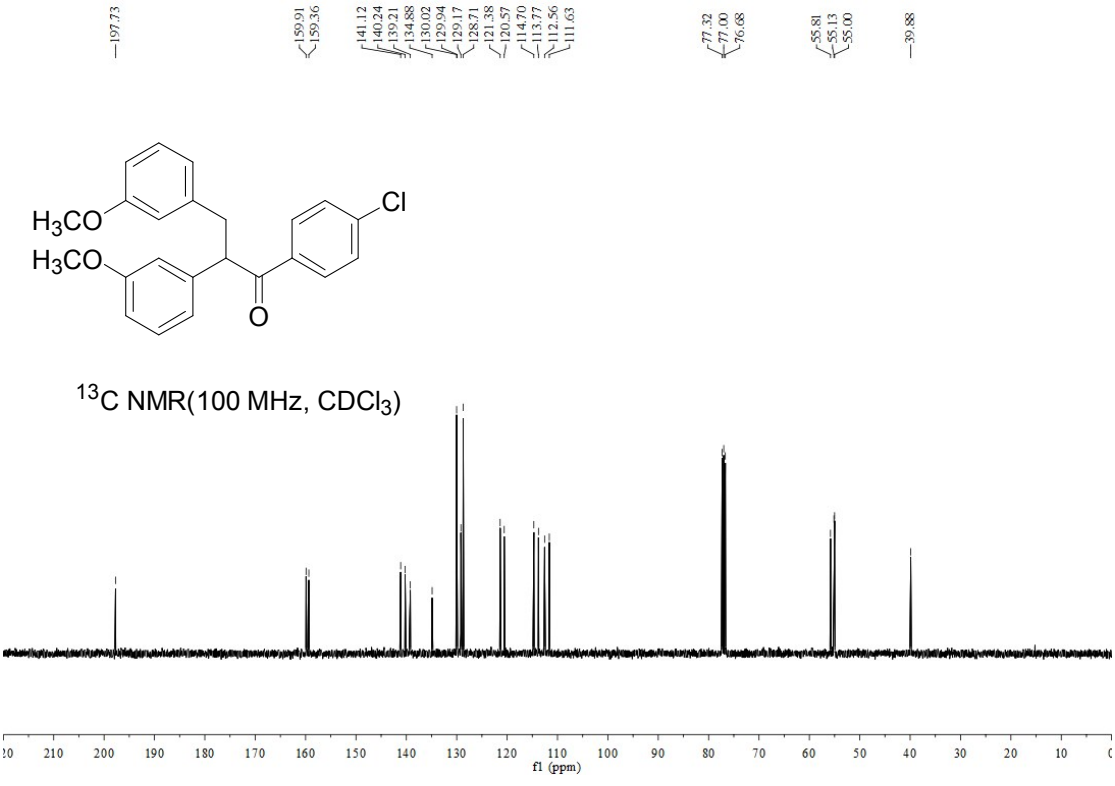


^{13}C NMR(100 MHz, $CDCl_3$)



3md

^{1}H NMR (400 MHz, $CDCl_3$)



^{13}C NMR(100 MHz, $CDCl_3$)