Supporting Information

for

Nickel-Catalyzed Decarbonylative Alkylation of Aroyl Fluorides Assisted by Lewis-Acidic Organoboranes

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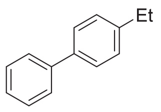
1. *General*

High resolution mass spectra (HRMS) were obtained by fast atom bombardment (FAB) using a double focusing magnetic sector mass spectrometer.

Representative Procedure for Decarbonylative Ethylation of 1a. Synthesis of 4-ethylbiphenyl (4aa).[1] To a 20 mL Schlenk tube, were added Ni(cod)$_2$ (10 mol %, 3.4 mg, 0.0125 mmol), DPPE (10 mol %, 5.0 mg, 0.0125 mmol), and toluene (500 μL) were added. After short minutes stirring, BEt_3 (**2a**) (1.0 M hexane solution, 125 μL, 0.125 mmol) and biphenyl-4-carbonyl fluoride (**1a**) (25.0 mg, 0.125 mmol) were added, and resulting mixture was heated at 130 ºC. After 24 h, the reaction was quenched with 1 M HCl solution, extracted with EtOAc, washed by brine, and dried over $MgSO_4$. After the volatiles were removed under vacuum, the crude product was purified by column chromatography (hexane:Et_2O = 10:1) to afford **4aa** (17.7 mg, 0.0971 mmol) in 78% yield.

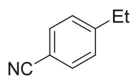


4aa

^{1}H NMR ($CDCl_3$, 600 MHz, rt): δ 1.29 (t, J = 7.8 Hz, 3H), 2.71 (q, J = 7.8 Hz, 2H), 7.29 (d, J = 7.8 Hz, 2H), 7.32-7.35 (m, 1H), 7.42-7.45 (m, 2H), 7.52-7.54 (m, 2H), 7.59-7.60 (m, 2H); ^{13}C{^{1}H} NMR ($CDCl_3$, 151 MHz, rt) δ 15.7, 28.7, 127.10, 127.16, 127.22, 128.4, 128.8, 138.8, 141.3, 143.5.

4-Ethylbenzonitrile (4ba).[2]

Yield was 68% (11.1 mg).

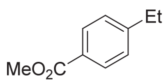


4ba

^{1}H NMR ($CDCl_3$, 600 MHz, rt): δ 1.25 (t, J = 7.8 Hz, 3H), 2.71 (q, J = 7.6 Hz, 2H), 7.29 (d, J = 9.0 Hz, 2H), 7.57 (d, J = 9.0 Hz, 2H); ^{13}C{^{1}H} NMR ($CDCl_3$, 151 MHz, rt) δ 15.2, 29.2, 109.6, 119.3, 128.8, 132.3, 149.9.

Methyl 4-ethylbenzoate (4ca).[2]

Yield was 54% (11.0 mg).

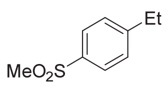


4ca

^{1}H NMR ($CDCl_3$, 600 MHz, rt): δ 1.25 (t, J = 7.8 Hz, 3H), 2.70 (q, J = 7.4 Hz, 2H), 3.90 (s, 3H), 7.26 (d, J = 8.4 Hz, 2H), 7.96 (d, J = 8.4 Hz, 2H); ^{13}C{^{1}H} NMR ($CDCl_3$, 151 MHz, rt) δ 15.4, 29.1, 52.1, 127.6, 128.0, 129.8, 149.9, 167.4.

1-Ethyl-4-(methylsulfonyl)benzene (4da).[3]

Yield was 51% (11.8 mg).

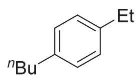


4da

[1]H NMR ($CDCl_3$, 600 MHz, rt): δ 1.27 (t, J = 7.8 Hz, 3H), 2.75 (q, J = 7.6 Hz, 2H), 3.04 (s, 3H), 7.39 (d, J = 9.0 Hz, 2H), 7.85 (d, J = 8.4 Hz, 2H); $^{13}C\{^1H\}$ NMR ($CDCl_3$, 151 MHz, rt) δ 15.3, 29.0, 44.8, 127.6, 129.0, 138.0, 150.9.

1-*n*-Butyl-4-ethylbenzene (4ea).[4]

Yield was 61% (12.4 mg).

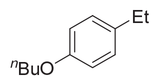


4ea

^1H NMR ($CDCl_3$, 600 MHz, rt): δ 0.92 (t, J = 7.2 Hz, 3H), 1.23 (t, J = 7.5 Hz, 3H), 1.36 (sext, J = 7.4 Hz, 2H), 1.55-1.63 (m, 2H), 2.58 (t, J = 7.8 Hz, 2H), 2.62 (q, J = 7.6 Hz, 2H), 7.09-7.12 (m, 4H); $^{13}C\{^1H\}$ NMR ($CDCl_3$, 151 MHz, rt) δ 14.1, 15.8, 22.6, 28.6, 33.9, 35.4, 127.8, 128.5, 140.2, 141.5.

1-*n*-Butoyl-4-ethylbenzene (4fa).[5]

Yield was 52% (11.6 mg).

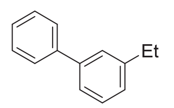


4fa

^1H NMR ($CDCl_3$, 600 MHz, rt): δ 0.97 (t, J = 7.5 Hz, 3H), 1.21 (t, J = 7.8 Hz, 3H), 1.43-1.51 (m, 2H), 1.74-1.77 (m, 2H), 2.59 (q, J = 7.6 Hz, 2H), 3.94 (t, J = 6.6 Hz, 2H), 6.83 (d, J = 9.0 Hz, 2H), 7.10 (d, J = 9.0 Hz, 2H); $^{13}C\{^1H\}$ NMR ($CDCl_3$, 151 MHz, rt) δ 14.0, 16.0, 19.4, 28.1, 31.6, 67.8, 114.5, 128.8, 136.3, 157.3.

3-Ethylbiphenyl (4ga).[1]

Yield was 71% (16.2 mg).

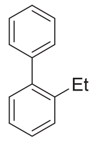


4ga

^1H NMR ($CDCl_3$, 600 MHz, rt): δ 1.30 (t, J = 7.8 Hz, 3H), 2.73 (q, J = 7.6 Hz, 2H), 7.20 (d, J = 7.2 Hz, 1H), 7.33-7.38 (m, 2H), 7.41-7.46 (m, 4H), 7.60 (dd, J = 8.4, 1.2 Hz, 2H); $^{13}C\{^1H\}$ NMR ($CDCl_3$, 151 MHz, rt) δ 15.8, 29.1, 124.7, 127.0, 127.28, 127.31, 127.35, 127.39, 128.83, 128.87, 128.89, 141.4, 141.6, 144.9.

2-Ethylbiphenyl (4ha).[1]

Yield was 36% (8.2 mg).

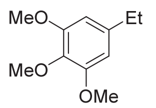


4ha

^1H NMR ($CDCl_3$, 600 MHz, rt): δ 1.10 (t, J = 7.8 Hz, 3H), 2.60 (q, J = 7.6 Hz, 2H), 7.18-7.42 (m, 9H); $^{13}C\{^1H\}$ NMR ($CDCl_3$, 151 MHz, rt) δ 15.8, 26.3, 125.7, 126.9, 127.3, 127.4, 127.6, 128.1, 128.7, 128.9, 129.4, 130.1, 141.75, 141.77.

5-Ethyl-1,2,3-trimethoxybenzene (4ia).[6]

Yield was 68% (16.6 mg).

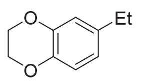


4ia

^{1}H NMR ($CDCl_3$, 600 MHz, rt): δ 1.24 (t, J = 7.5 Hz, 3H), 2.60 (q, J = 7.6 Hz, 2H), 3.82 (s, 3H), 3.86 (s, 6H), 6.42 (s, 2H); ^{13}C{^{1}H} NMR ($CDCl_3$, 151 MHz, rt) δ 15.8, 29.4, 56.2, 61.0, 104.8, 128.9, 136.0, 153.2.

6-Ethyl-1,4-benzodioxane (4ja).[7]

Yield was 69% (14.2 mg).

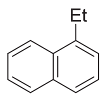


4ja

^{1}H NMR ($CDCl_3$, 600 MHz, rt): δ 1.20 (t, J = 7.8 Hz, 3H), 2.55 (q, J = 7.6 Hz, 2H), 4.24 (m, 4H), 6.67 (dd, J = 8.1, 2.1 Hz, 1H), 6.71 (d, J = 1.8 Hz, 1H), 6.78 (d, J = 7.8 Hz, 1H); ^{13}C{^{1}H} NMR ($CDCl_3$, 151 MHz, rt) δ 15.9, 28.3, 64.5, 64.6, 116.6, 117.1, 120.9, 137.8, 141.6, 143.4.

1-Ethylnaphthalene (4ka).[1]

Yield was 70% (13.7 mg).

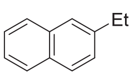


4ka

^{1}H NMR ($CDCl_3$, 600 MHz, rt): δ 1.39 (t, J = 7.5 Hz, 3H), 3.12 (q, J = 7.2 Hz, 2H), 7.34 (d, J = 6.6 Hz, 1H), 7.41 (t, J = 6.9 Hz, 1H), 7.46-7.53 (m, 2H), 7.71 (d, J = 8.4 Hz, 1H), 7.86 (d, J = 7.8 Hz, 1H), 8.06 (d, J = 8.4 Hz, 1H); ^{13}C{^{1}H} NMR ($CDCl_3$, 151 MHz, rt) δ 15.2, 26.0, 123.9, 125.0, 125.5, 125.8 (2C), 126.5, 128.9, 131.9, 133.9, 140.4.

2-Ethylnaphthalene (4la).[1]

Yield was 74% (14.5 mg).

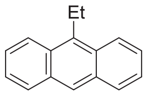


4la

^{1}H NMR ($CDCl_3$, 600 MHz, rt): δ 1.33 (t, J = 7.5 Hz, 3H), 2.82 (q, J = 7.6 Hz, 2H), 7.35 (dd, J = 8.4, 1.8 Hz, 1H), 7.39-7.46 (m, 2H), 7.63 (s, 1H), 7.76-7.81 (m, 3H); ^{13}C{^{1}H} NMR ($CDCl_3$, 151 MHz, rt) δ 15.7, 29.2, 123.6, 125.1, 125.7, 126.0, 127.2, 127.5, 127.7, 127.9, 131.0, 133.8, 141.9.

9-Ethylanthracene (4ma).[8]

Yield was 56% (14.4 mg).

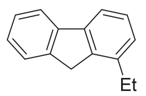


4ma

^{1}H NMR ($CDCl_3$, 600 MHz, rt): δ 1.45 (t, J = 7.8 Hz, 3H), 3.65 (q, J = 7.8 Hz, 2H), 7.45-7.47 (m, 2H), 7.49-7.52 (m, 2H), 8.01 (d, J = 7.8 Hz, 2H), 8.28 (d, J = 8.4 Hz, 2H), 8.34 (s, 1H); ^{13}C{^{1}H} NMR ($CDCl_3$, 151 MHz, rt) δ 15.7, 21.3, 124.5, 124.9, 125.5, 125.6, 129.3, 129.4, 131.8, 136.8.

1-Ethyl-9*H*-fluorene (4na).[9]

Yield was 78% (19.0 mg).

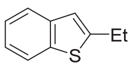


4na

^{1}H NMR ($CDCl_3$, 600 MHz, rt): δ 1.32 (t, J = 7.5 Hz, 3H), 2.79 (q, J = 7.6 Hz, 2H), 3.85 (s, 2H), 7.17 (d, J = 7.8 Hz, 1H), 7.30 (td, J = 7.2, 1.2 Hz, 1H), 7.34-7.38 (m, 2H), 7.56 (d, J = 7.2 Hz, 1H), 7.65 (d, J = 7.2 Hz, 1H), 7.78 (d, J = 7.8 Hz, 1H); ^{13}C{^{1}H} NMR ($CDCl_3$, 151 MHz, rt) δ 14.4, 26.4, 35.6, 117.6, 120.1, 125.1, 126.1, 126.7, 126.8, 127.4, 140.5, 141.5, 141.6, 142.2, 143.2.

2-Ethylbenzo[*b*]thiophene (4pa).[10]

Yield was 70% (14.1 mg).

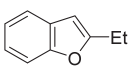


4pa

^{1}H NMR ($CDCl_3$, 600 MHz, rt): δ 1.38 (t, J = 7.5 Hz, 3H), 2.94 (qd, J = 7.6, 1.2 Hz, 2H), 7.01 (d, J = 0.6 Hz, 1H), 7.23-7.26 (m, 1H), 7.28-7.32 (m, 1H), 7.66 (d, J = 7.8 Hz, 1H), 7.76 (dd, J = 7.8, 0.6 Hz, 1H); ^{13}C{^{1}H} NMR ($CDCl_3$, 151 MHz, rt) δ 15.6, 24.3, 119.8, 122.3, 122.8, 123.5, 124.2, 139.3, 140.4, 148.5.

2-Ethylbenzofuran (4qa).[7]

Yield was 28% (5.1 mg).

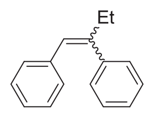


4qa

^{1}H NMR ($CDCl_3$, 600 MHz, rt): δ 1.34 (t, J = 7.8 Hz, 3H), 2.80 (qd, J = 7.5, 0.9 Hz, 2H), 6.38 (d, J = 0.6 Hz, 1H), 7.16-7.22 (m, 2H), 7.41 (dd, J = 7.8, 0.6 Hz, 1H), 7.48 (dd, J = 6.3, 0.9 Hz, 1H); ^{13}C{^{1}H} NMR ($CDCl_3$, 151 MHz, rt) δ 12.0, 21.9, 101.1, 110.8, 120.3, 122.5, 123.2, 129.1, 154.8, 161.1.

(*E*)- and (*Z*)-1,2-Diphenyl-1-butene (4ra).[11(for (*E*)-isomer), 12(for (*Z*)-isomer)]

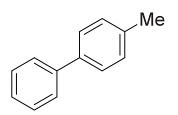
Yield was 66% (*E*:*Z* = 0.93:1) (17.2 mg).



4ra

^{1}H NMR ($CDCl_3$, 600 MHz, rt): δ 1.05-1.08 (m, (*E*) and (*Z*)), 2.51 (qd, J = 7.4, 1.5 Hz, (*Z*)), 2.75 (q, J = 7.6 Hz, (*E*)), 6.43 (s, (*Z*)), 6.69 (s, (*E*)), 6.92 (d, J = 7.2 Hz, (*Z*)), 7.04-7.10 (m, (*Z*)), 7.15 (d, J = 7.2 Hz, (*Z*)), 7.24-7.48 (m, (*E*) and (*Z*)); ^{13}C{^{1}H} NMR ($CDCl_3$, 151 MHz, rt) δ 13.0 (*Z*), 13.6 (*E*), 23.4 (*E*), 33.7 (*Z*), 125.2 (*Z*), 126.2 (*Z*), 126.7 (*E*), 126.8 (*Z*), 126.9 (*E*), 127.3 (*E*), 127.7 (*Z*), 127.9 (*E*), 128.4 (*Z*), 128.5 (*E*), 128.6 (*E*), 128.7 (*Z*), 128.9 (*E*), 129.1 (*Z*), 137.7 (*Z*), 138.4 (*E*), 141.6 (*Z*), 142.8 (*E*), 144.6 (*E*), 145.1 (*Z*).

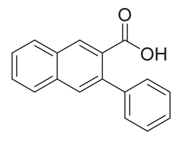
Representative Procedure for Decarbonylative C(aroyl)–F Bond Alkylation of 1a with Trimethylboroxine: Synthesis of 4-Methylbiphenyl (4ab).[13] To a 20 mL Schlenk tube, were added Ni(cod)$_2$ (20 mol %, 6.9 mg, 0.025 mmol), DPPE (20 mol %, 10.0 mg, 0.025 mmol), and toluene (500 μL). Subsequently, CsF (38.0 mg, 0.25 mmol, 2 equiv), $(MeBO)_3$ (**2b**) (34.9 μL, 0.25 mmol, 2 equiv), and biphenyl-4-carbonyl fluoride (**1a**) (25.0 mg, 0.125 mmol) were added, and the resulting mixture was heated at 130 ºC. After 24 h, the reaction mixture was quenched with 1 M HCl, extracted with EtOAc, washed by brine, and dried over $MgSO_4$. After the volatiles were removed under vacuum, the crude product was purified by column chromatography (hexane:Et_2O = 10:1) to afford **4ab** (10 mg, 0.061 mmol) in 49% yield.



4ab

^{1}H NMR ($CDCl_3$, 600 MHz, rt): δ 2.40 (s, 3H), 7.26 (d, J = 7.8 Hz, 2H), 7.31-7.34 (m, 1H), 7.43 (t, J = 7.5 Hz, 2H), 7.50 (d, J = 8.4 Hz, 2H), 7.59 (dd, J = 8.4, 1.2 Hz, 2H); ^{13}C{^{1}H} NMR ($CDCl_3$, 151 MHz, rt) δ 21.2, 127.12, 127.14, 127.3, 128.9, 129.6, 137.2, 138.5, 141.3.

Representative Procedure for the Palladium-Catalyzed *ortho*-C–H Arylation of 2-Naphthoic Acid (5): Synthesis of 3-Phenyl-2-naphthoic Acid (6a).[14] To an oven-dried 20 mL Schlenk tube, were added $Pd(OAc)_2$ (11.2 mg, 0.05 mmol, 5 mol %), Ag_2CO_3 (358 mg, 1.3 mmol, 1.3 equiv), 2-naphthoic acid (**5**) (172 mg, 1.0 mmol), iodobenzene (336 μL, 3.0 mmol, 3.0 equiv), and AcOH (100 μL) under an argon atmosphere. After the reaction mixture was stirred at 145 ºC for 6 h, it was cooled to room temperature and quenched with 1 M HCl. The mixture was filtrated by Celite®, and extracted with CH_2Cl_2, washed with brine, and dried over $MgSO_4$. After the volatiles were evaporated, the crude mixture was purified by column chromatography (CH_2Cl_2:EtOAc = 10:1), and the high-boiling starting material 2-naphthoic acid (**5**) was removed by bulb-to-bulb distillation (2 mmHg, 200 ºC) afforded **6a** (154 mg, 0.62 mmol) in 62% yield.

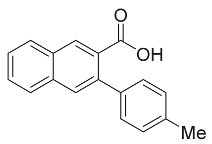


6a

^{1}H NMR ($CDCl_3$, 600 MHz, rt): δ 7.41-7.46 (m, 5H), 7.56 (td, J = 7.5, 1.2 Hz, 1H), 7.62 (td, J = 7.6, 1.4 Hz, 1H), 7.82 (s, 1H), 7.87 (d, J = 7.8 Hz, 1H), 7.96 (d, J = 7.8 Hz, 1H), 8.55 (s, 1H), The signal of carboxylic acid was not observed.; ^{13}C{^{1}H} NMR ($CDCl_3$, 151 MHz, rt) δ 126.9, 127.3, 127.5, 127.8, 128.1, 128.78, 128.81, 128.9, 130.3, 131.5, 132.6, 134.8, 139.2, 141.2, 173.9.

3-(*p*-Tolyl)-2-naphthoic acid (6b).[15]

Yield was 49% (129 mg).

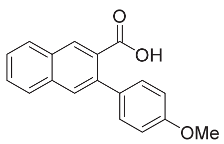


6b

^{1}H NMR ($CDCl_3$, 600 MHz, rt): δ 2.44 (s, 3H), 7.25 (d, J = 7.8 Hz, 2H), 7.37 (d, J = 7.8 Hz, 2H), 7.55 (td, J = 7.5, 1.2 Hz, 1H), 7.61 (td, J = 7.5, 1.2 Hz, 1H), 7.82 (s, 1H), 7.87 (d, J = 7.8 Hz, 1H), 7.96 (d, J = 8.4 Hz, 1H), 8.56 (s, 1H), The signal of carboxylic acid was not observed.; ^{13}C{^{1}H} NMR ($CDCl_3$, 151 MHz, rt) δ 21.4, 126.8, 127.7, 127.9, 128.7, 128.8, 128.9, 129.0, 130.3, 131.5, 132.5, 134.9, 137.0, 138.3, 139.1, 173.9.

3-(*p*-Anisoyl)-2-naphthoic acid (6c).[15]

Yield was 45% (125 mg).

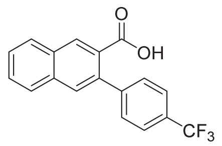


6c

^{1}H NMR ($CDCl_3$, 600 MHz, rt): δ 3.78 (s, 3H), 6.99 (d, J = 8.4 Hz, 2H), 7.40 (d, J = 8.4 Hz, 2H), 7.55 (td J = 7.5, 0.8 Hz, 1H), 7.61 (td, J = 7.6, 1.0 Hz, 1H), 7.81 (s, 1H), 7.86 (d, J = 8.4 Hz, 1H), 7.95 (d, J = 8.4 Hz, 1H), 8.56 (s, 1H), The signal of carboxylic acid was not observed.; ^{13}C{^{1}H} NMR ($CDCl_3$, 151 MHz, rt) δ 55.3, 113.7, 126.8, 127.7, 127.8, 128.7, 128.9, 129.9, 130.2, 131.3, 132.5, 133.6, 134.9, 138.8, 159.0, 174.0.

3-(*p*-Trifluoromethylphenyl)-2-naphthoic acid (6d).[15]

Yield was 59% (187 mg).

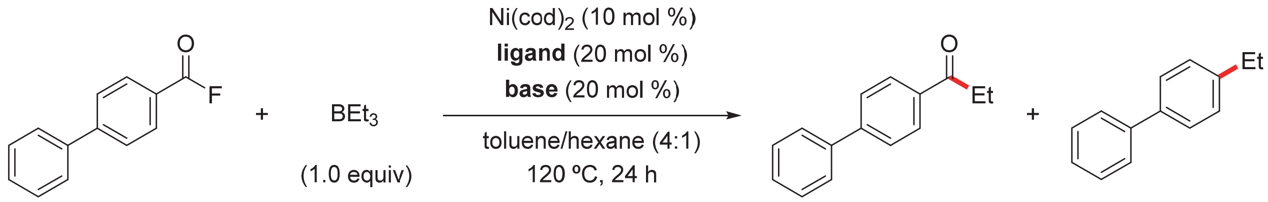


6d

^{1}H NMR ($CDCl_3$, 600 MHz, rt): δ 7.53 (d, J = 7.8 Hz, 2H), 7.60 (td, J = 7.5, 1.2 Hz, 1H), 7.66 (td, J = 8.4 Hz, 1.2 Hz, 1H), 7.68 (d, J = 7.8 Hz, 2H), 7.78 (s, 1H), 7.88 (d, J = 7.8 Hz, 1H), 7.99 (d, J = 8.4 Hz, 1H), 8.64 (s, 1H), The signal of carboxylic acid was not observed.; ^{13}C{^{1}H} NMR ($CDCl_3$, 151 MHz, rt) δ 124.5 (q, $^{1}J_{C–F}$ = 272 Hz), 125.0 (q, $^{2}J_{C–F}$ = 4 Hz), 126.7, 127.5, 128.0, 129.1, 129.2, 129.3, 129.5, 130.7, 131.9, 133.3, 134.9, 138.1, 145.2, 172.6.; ^{19}F{^{1}H} NMR ($CDCl_3$, 564 MHz, rt) δ –62.3.

2. *Optimization of the Reaction Conditions*

Table S1. Screening of ligand and reaction temperature

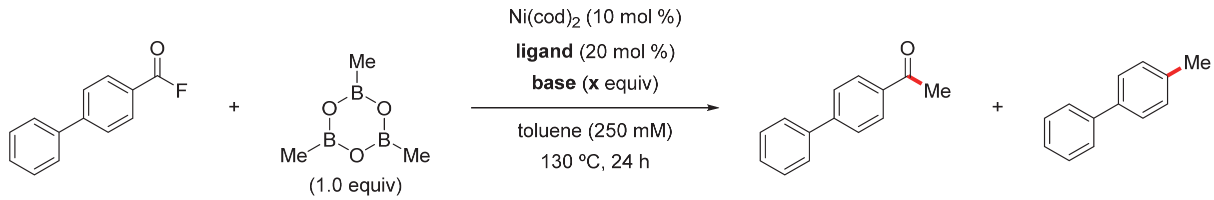


Entry	ligand	base	NMR yield (%) 3aa	NMR yield (%) 4aa
1	none	none	0	0
2	PPh_3	none	0	21
3	PCy_3	none	22	26
4	$P(C_6F_5)_3$	none	0	0
5	P(mesityl)$_3$	none	0	0
6[a]	DPPM	none	0	36
7[a]	DPPE	none	0	83 (78)[b]
8[a]	DPPP	none	0	28
9[a]	DPPB	none	0	25
10[a]	DCPE	none	trace	38
11[a]	DPPBz	none	0	45
12[a]	DPPF	none	0	38
13[a]	Xantphos	none	0	16
14	Xphos	none	0	18
15[a]	dtbpy	none	12	23
16	IPr•HCl	pyridine	27	trace
17	ICy•HCl	pyridine	27	trace
18[a,c]	DPPE	none	0	85 (85)[b]
19[a,d]	DPPE	none	0	77 (73)[b]
20[a,e]	DPPE	none	0	28

DCPE = 1,2-bis(diclohexylphosphino)ethane, DPPBz = 1,2-bis(diphenylphosphino)benzene, IPr•HCl = 1,3-bis(2,6-diisopropylphenyl)imidazolium chloride, ICy•HCl = 1,3-dicyclohexylimidazolium chloride

[a] 10 mol % of ligand was used. [b] An isolated yield. [c] Run at 130 ºC for 24 h. [d] Run at 140 °C for 24 h. [e] Run at 110 ºC for 24 h.

Table S2. Effect of ligand, temperature and base in methylation

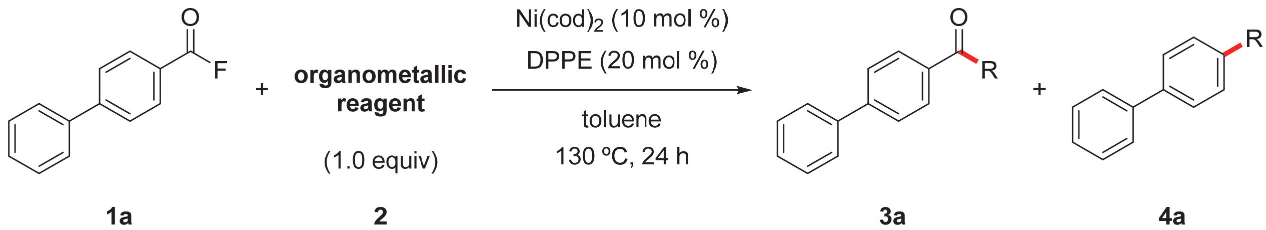


Entry	ligand	base	NMR yield (%) 3aa	NMR yield (%) 4ab
1	none	CsF (1 equiv)	0	1
2	PPh_3	CsF (1 equiv)	0	4
3	PCy_3	CsF (1 equiv)	0	13
4	$P(C_6F_5)$	CsF (1 equiv)	0	0
5	P(mesit	CsF (1 equiv)	0	0
6[a]	DPPM	CsF (1 equiv)	6	15
7[a]	DPPE	CsF (1 equiv)	trace	30
8[a]	DPPP	CsF (1 equiv)	3	9
9[a]	DPPB	CsF (1 equiv)	3	<3
10[a]	DCPE	CsF (1 equiv)	4	20
11[a]	DPPBz	CsF (1 equiv)	trace	29
12[a]	DPPF	CsF (1 equiv)	trace	4
13[a]	Xantph	CsF (1 equiv)	2	7
14	Xphos	CsF (1 equiv)	3	<9
15[a]	dtbpy	CsF (1 equiv)	6	29
16	IPr•HCl	CsF (1 equiv) + pyridine	2	2
17	ICy•HC	CsF (1 equiv) + pyridine	3	2
18[a,b]	DPPE	CsF (1 quiv)	trace	30
19[a,c]	DPPE	CsF (2 equiv)	trace	50(49)[d]
20[a,c]	DPPE	TBAF in THF (1.0 M, 2	0	trace
21[a,c]	DPPE	KF (2 equiv)	0	32
22[a,c]	DPPE	KOH (2 equiv)	0	21
23[a,c]	DPPE	K_2CO_3 (2 equiv)	0	21
24[a,c]	DPPE	K_3PO_4 (2 equiv)	0	18
25[a,c]	DPPE	pyridine (2 equiv)	0	9
26[a,c]	DPPE	NEt_3 (2 equiv)	0	22

DCPE = 1,2-bis(diclohexylphosphino)ethane, DPPBz = 1,2-bis(diphenylphosphino)benzene, IPr•HCl = 1,3-bis(2,6-diisopropylphenyl)imidazolium chloride, ICy•HCl = 1,3-dicyclohexylimidazolium chloride

[a] 10 mol % of ligand was used. [b] $Ni(cod)_2$ (20 mol %), DPPE (20 mol %). [c] $Ni(cod)_2$ (20 mol %), DPPE (20 mol %), $(MeBO)_3$ (2 equiv). [d] An isolated yield.

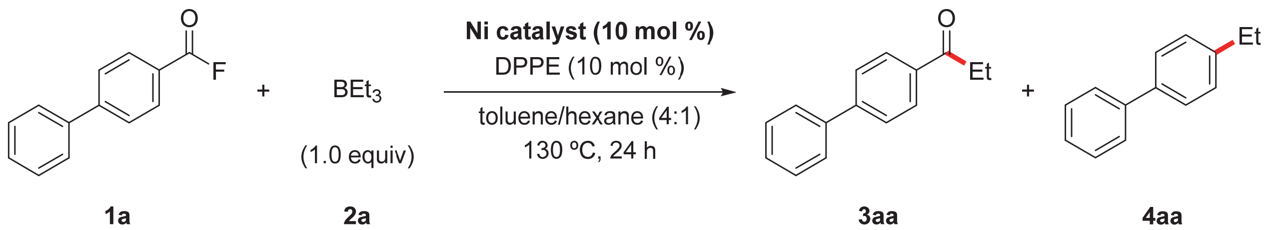
Table S3. Effect of organometallic reagents



Entry	organometallic reagent	NMR yield (%)	
		3a	**4a**
1	$SnMe_4$	0	0
2	$SiMe_4$	0	0
3	$(MeBO)_3$ (**2b**)	0	2
4[a]	$(MeBO)_3$ (**2b**)	3	20
5[b]	$(MeBO)_3$ (**2b**)	trace	30
6[c]	$(MeBO)_3$ (**2b**)	0	50 (49)[d]
7	$^nBuB(OH)_2$	0	0
8[a]	$^nBuB(OH)_2$	0	0
9	$^nC_{10}H_{21}$–(9-BBN)	0	0

[a] CsF (3 equiv). [b] CsF (1 equiv). [c] $Ni(cod)_2$ (20 mol %), DPPE (20 mol %), **2b** (2 equiv), CsF (2 equiv). [d] An isolated yield.

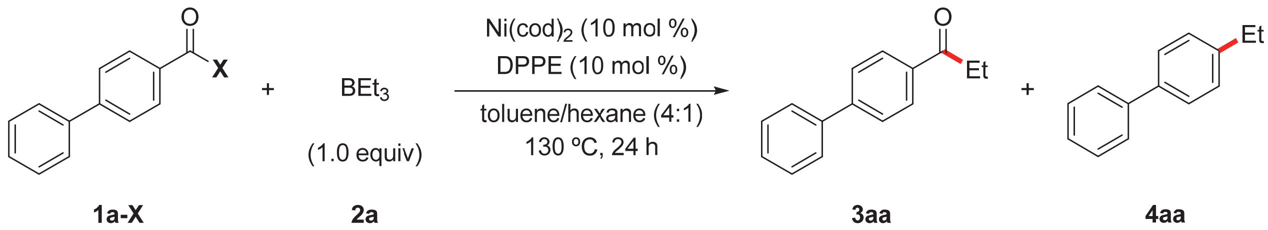
Table S4. Effect of nickel precatalyst



Entry	Ni catalyst	NMR yield (%)	
		3aa	**4aa**
1	$NiCl_2$	0	0
2	$Ni(OAc)_2$•$4H_2O$	0	80
3[a,b]	$Ni(OAc)_2$	0	30

[a] BEt_3 (**2a**) (1.2 equiv). [b] $Ni(OAc)_2$•$4H_2O$ was heated with a heatgun under reduced pressure for 1-2 minutes before the reaction.

Table S5. Scope of acid derivatives

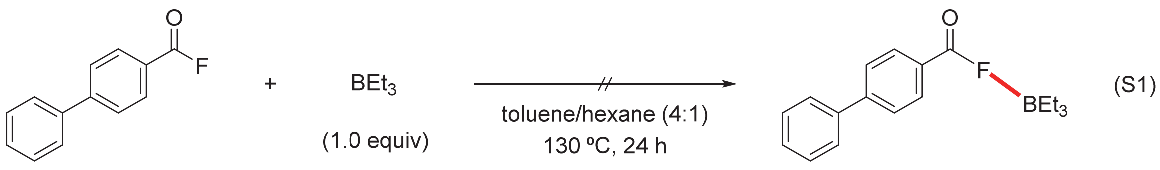


Entry	X	NMR yield (%)	
		3aa	**4aa**
1	Cl (distilled)	0	0
2	OPh	0	0
3[a]	SPh	0	0

[a] Decarbonylation of thioester through a C–S bond cleavage and following reductive elimination was only occured.

A Stoichiometric Reaction without a Nickel Catalyst

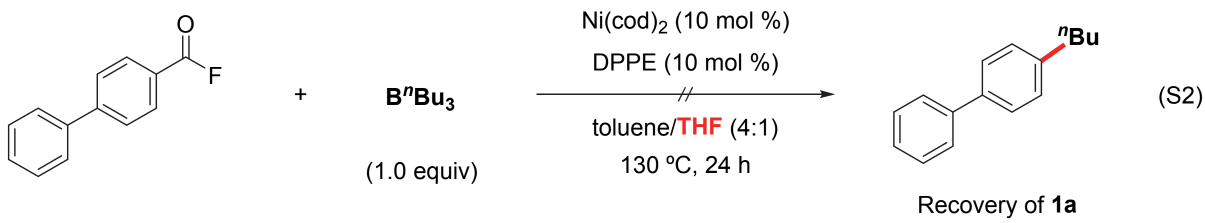
To an NMR tube, were added biphenyl-4-carbonyl fluoride (**1a**) (10.0 mg, 0.05 mmol), BEt_3 (**2a**) (1.0 M hexane solution, 50 μL, 0.05 mmol) in toluene-d_8 (1 mL) under an argon atmosphere. After the solution mixture was heated at 130 ºC for 24 h, $^{11}B\{^1H\}$ and $^{19}F\{^1H\}$ NMR were measured. The result clearly indicates that the preactivation of **1a** with **2a** did not occur under the standard conditions.



1a **2a**

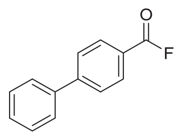
Reaction with B^nBu_3: Scope of Trialkylboranes

Decarbonylative alkylation with commercially available B^nBu_3 (**2c**) in THF solution was conducted. Unfortunately, the target 4-*n*-butylbiphenyl (**4ac**) was not detected at all. Based on this result, we assumed that Lewis acidity of B^nBu_3 would be drastically decreased by the formation of trialkylborane–THF complex.

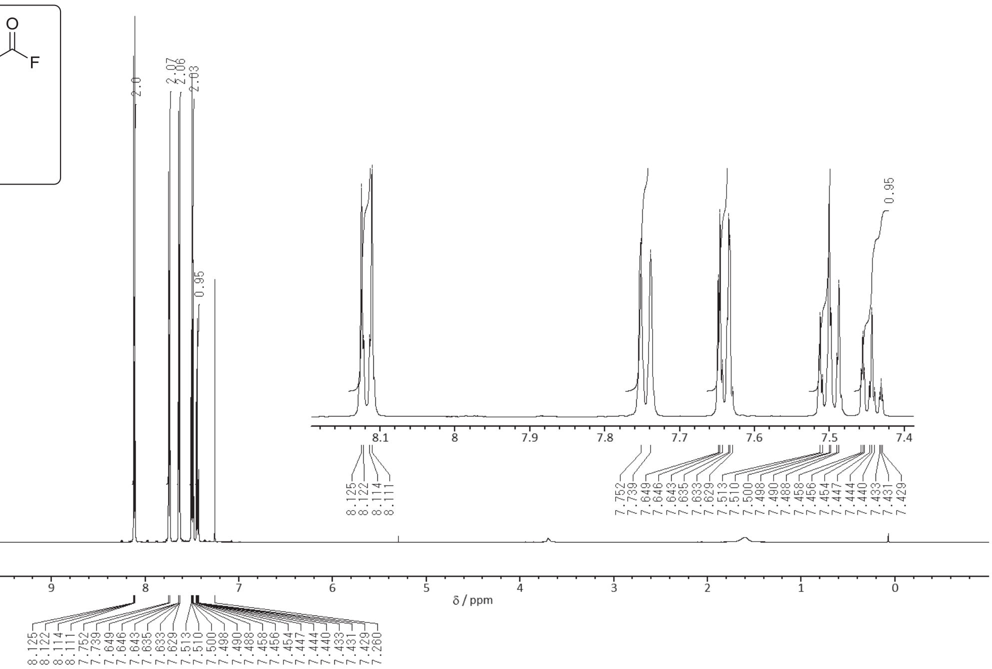


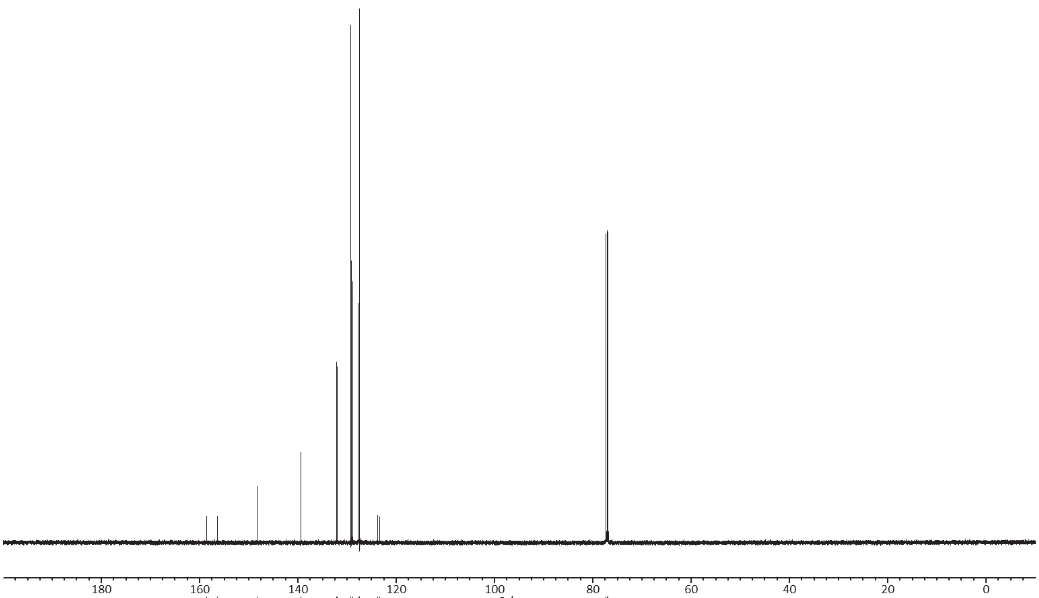
1a **2c** **4ac**

3. *Copies of ^{1}H and $^{13}C\{^{1}H\}$ NMR Charts for the New Compounds*



1a





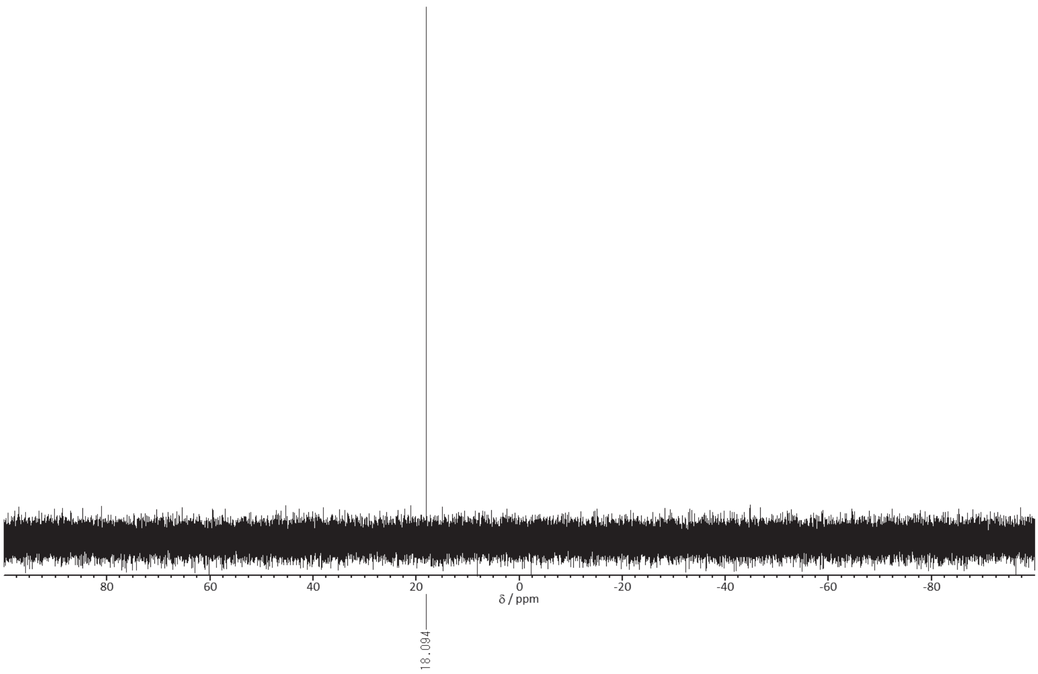
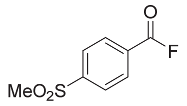
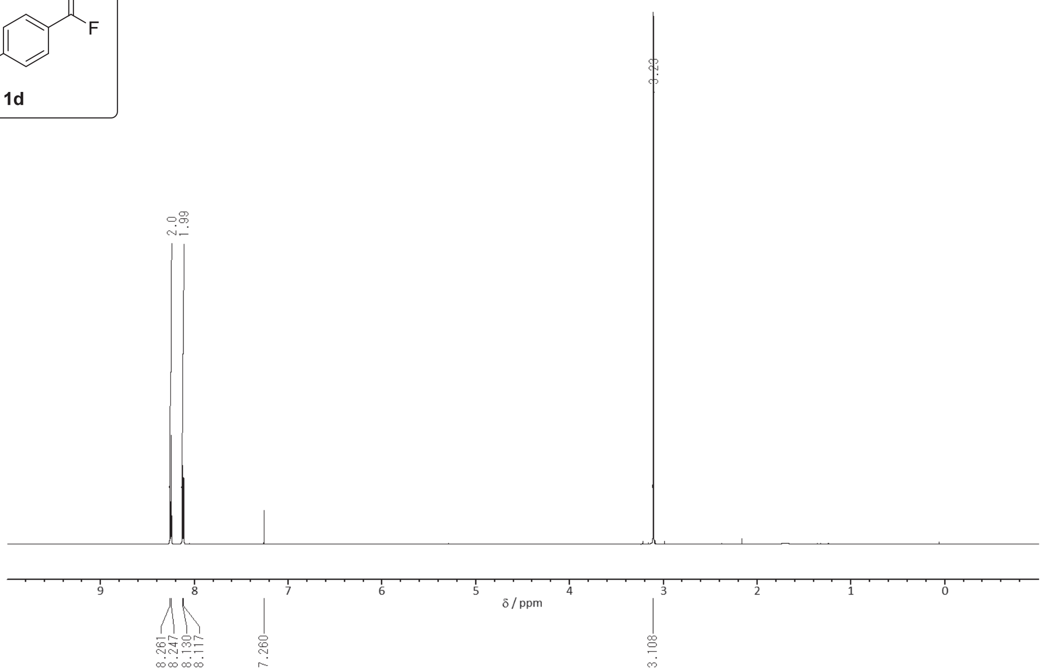
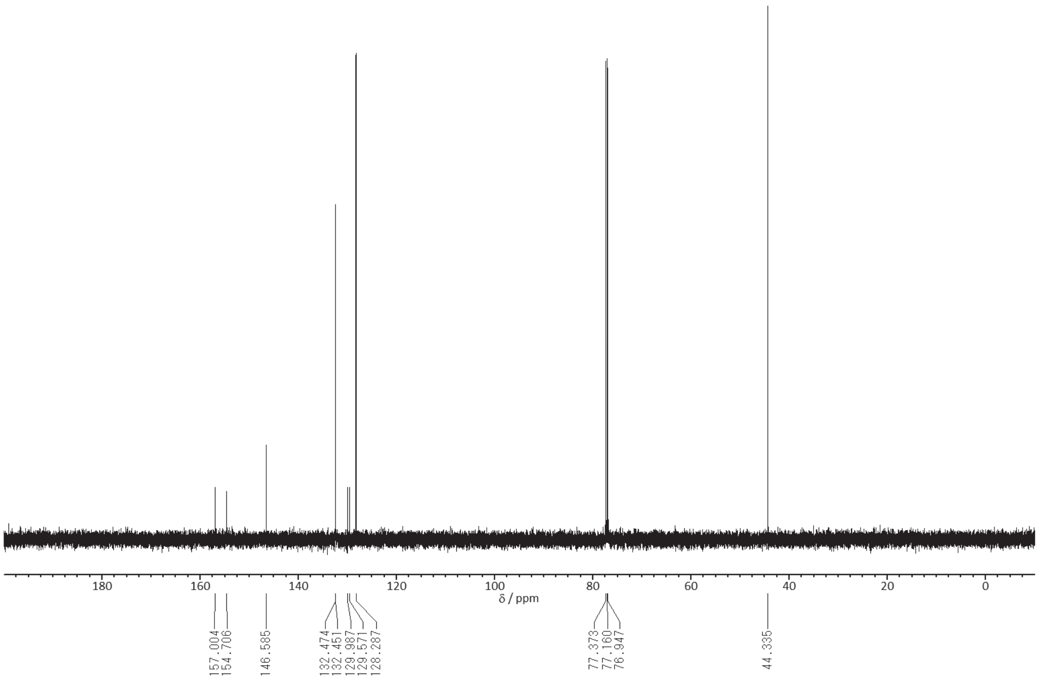


Figure S1. ^{1}H, $^{13}C\{^{1}H\}$ and $^{19}F\{^{1}H\}$ NMR spectra of compound **1a** (in $CDCl_3$, rt)


O
F
MeO2S

1d


2.0
1.99
3.23
δ / ppm
8.261
8.247
8.130
8.117
7.260
3.108


δ / ppm
157.004
154.706
146.585
132.474
132.451
129.987
129.571
128.287
77.373
77.160
76.947
44.335

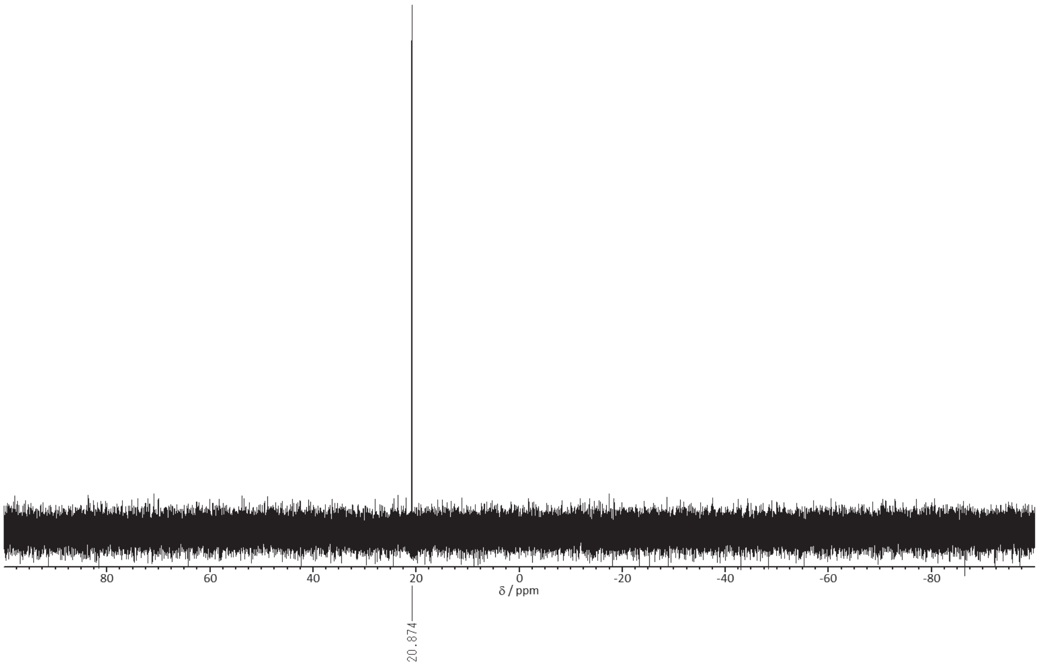
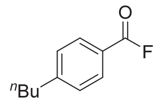
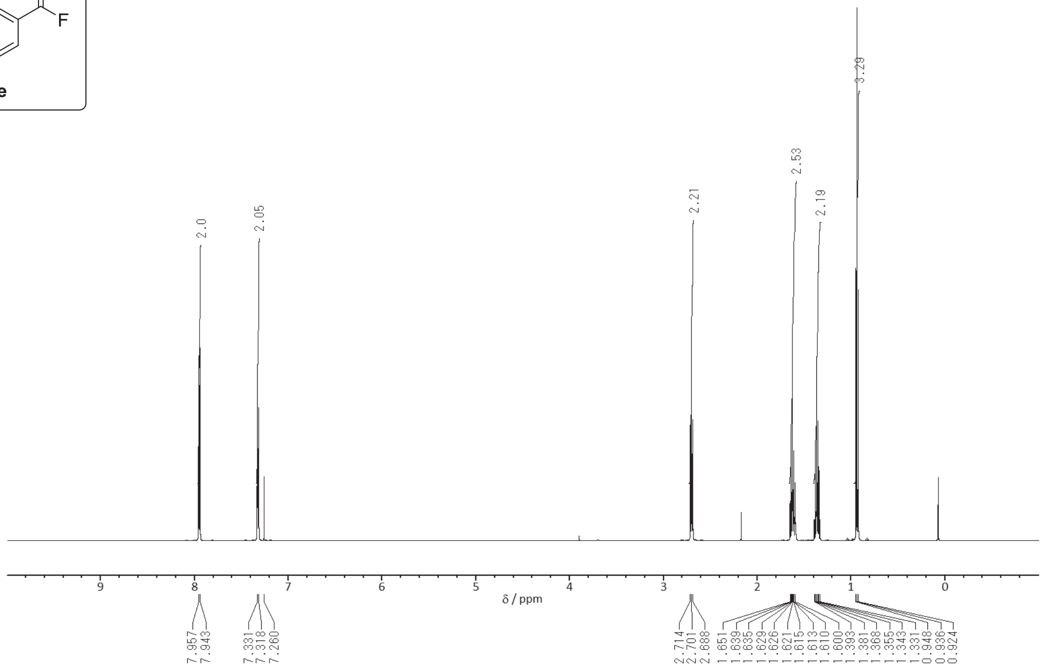
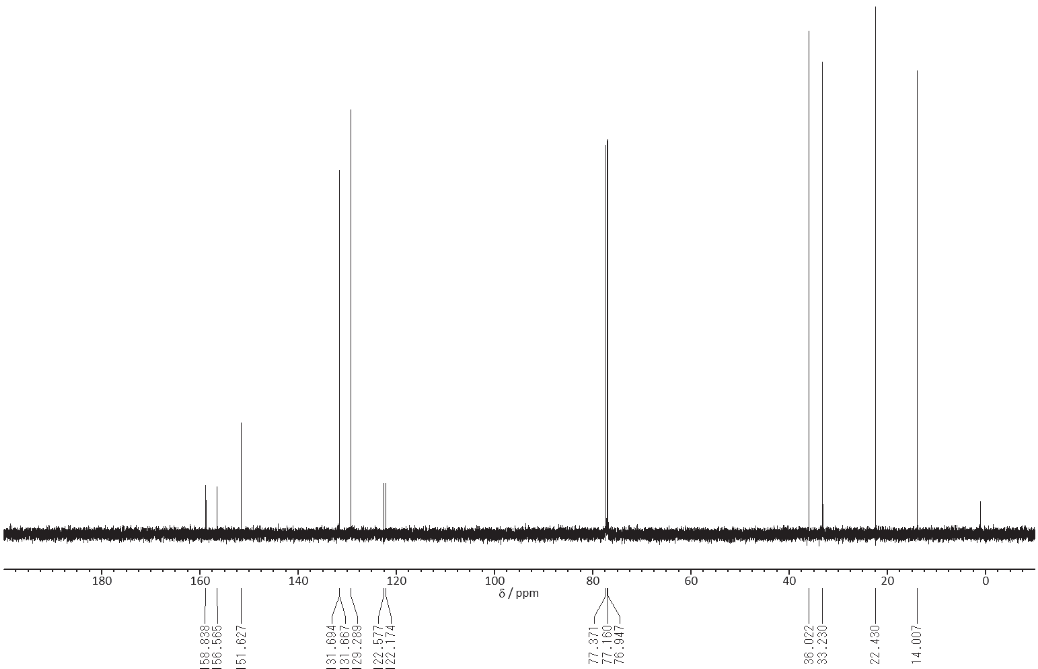


Figure S2. ^{1}H, $^{13}C\{^{1}H\}$ and $^{19}F\{^{1}H\}$ NMR spectra of compound **1d** (in $CDCl_3$, rt)


O
F
ⁿBu

1e


2.0
2.05
2.21
2.53
2.19
3.29
δ / ppm
7.957
7.943
7.331
7.318
7.260
2.714
2.701
2.688
1.651
1.639
1.635
1.629
1.626
1.621
1.615
1.613
1.610
1.600
1.393
1.381
1.368
1.355
1.343
1.331
0.948
0.936
0.924


δ / ppm
158.838
156.565
151.627
131.694
131.667
129.289
122.577
122.174
77.371
77.160
76.947
36.022
33.230
22.430
14.007

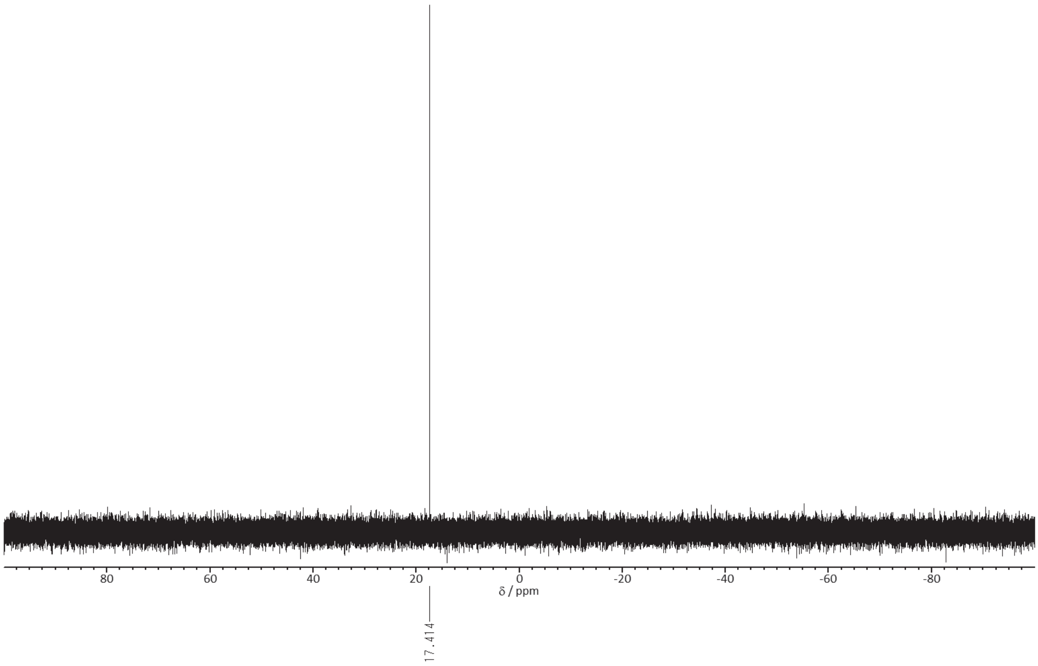
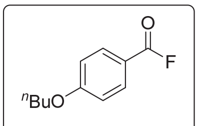
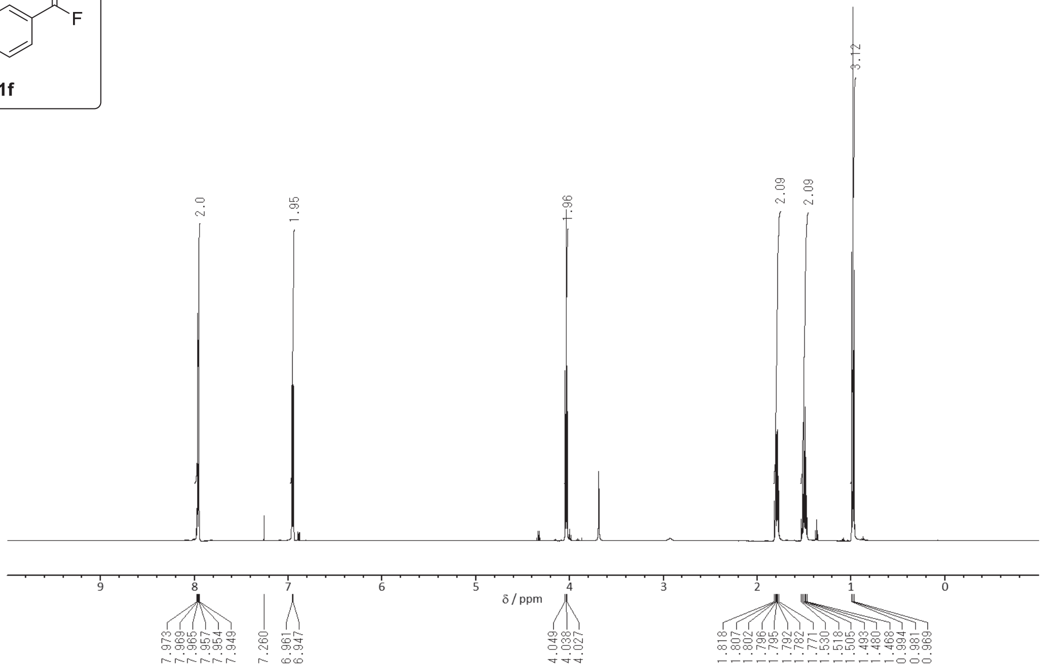
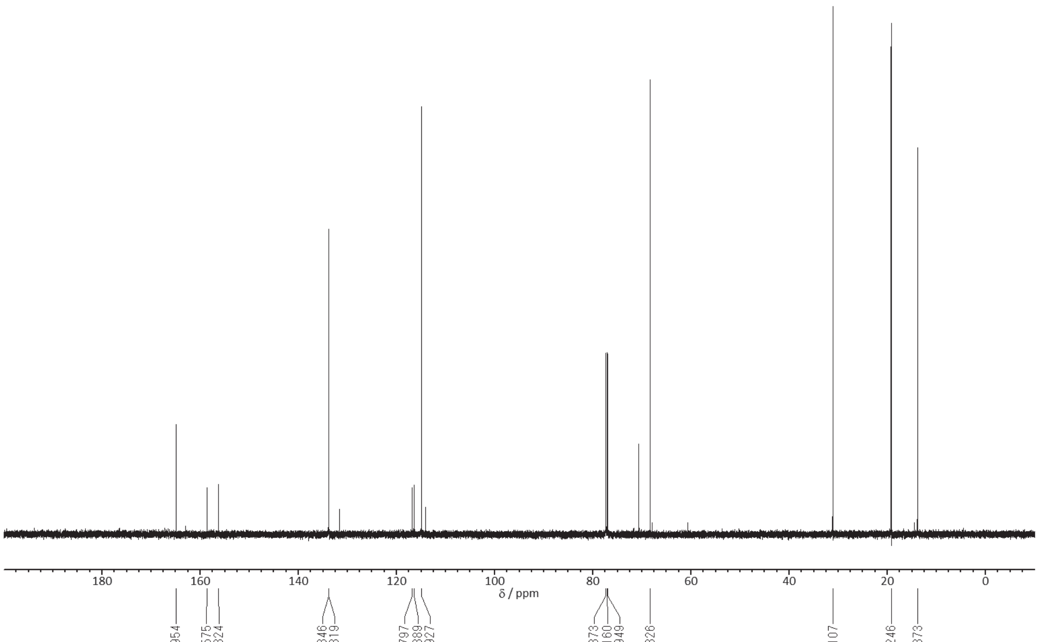


Figure S3. ^{1}H, $^{13}C\{^{1}H\}$ and $^{19}F\{^{1}H\}$ NMR spectra of compound **1e** (in $CDCl_3$, rt)



1f





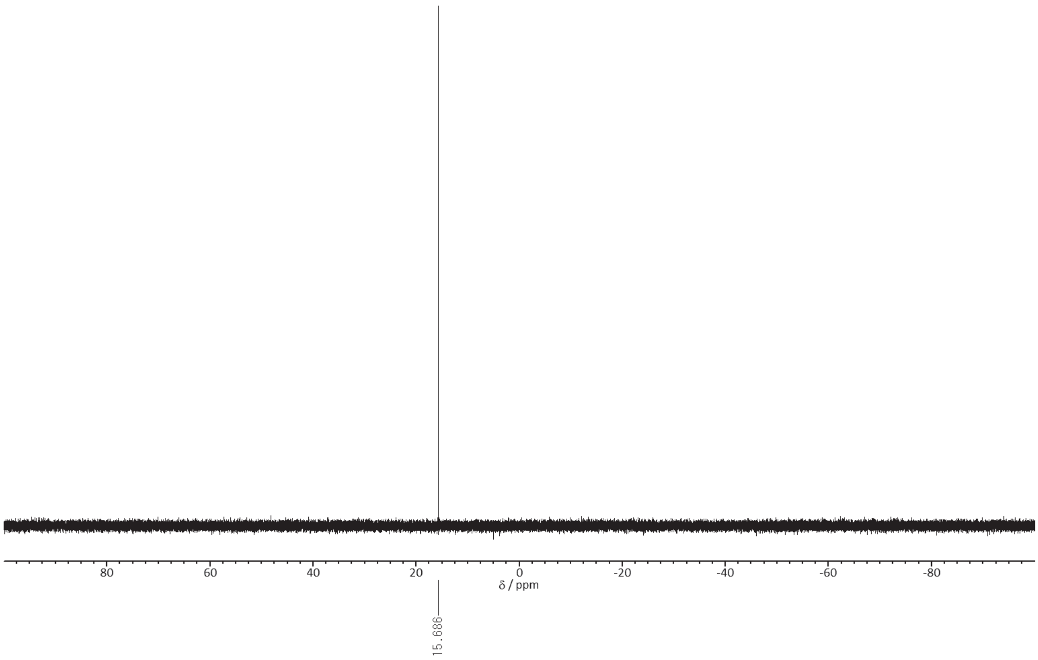
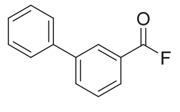
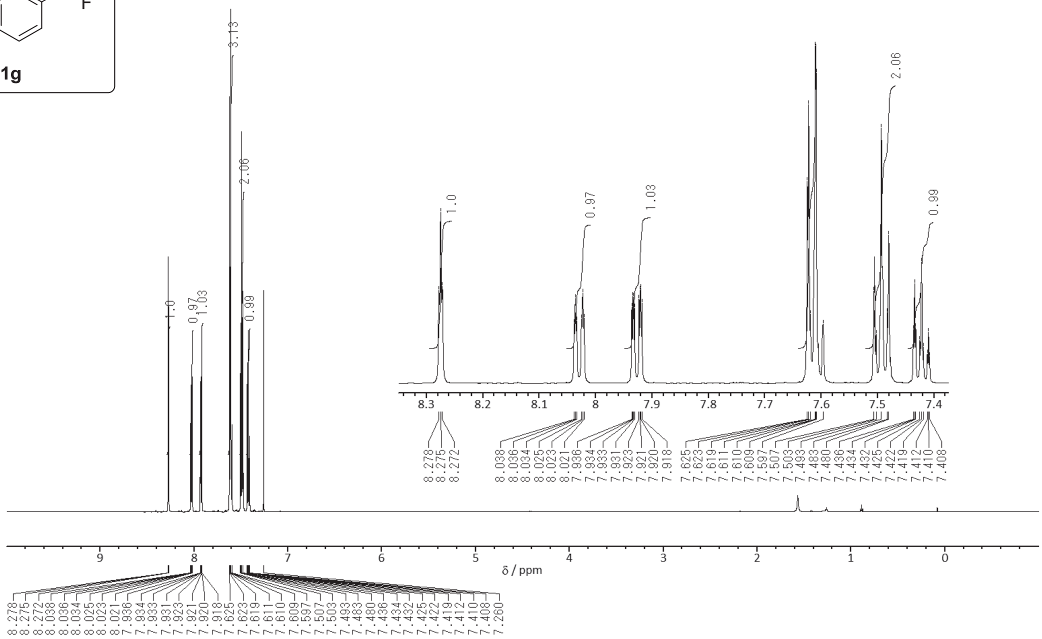
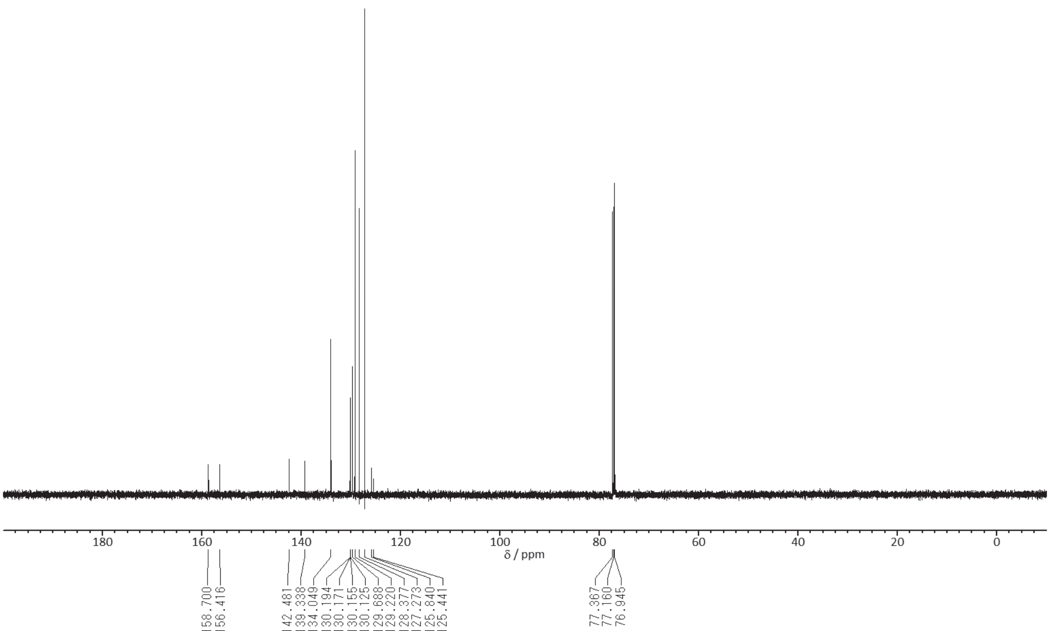


Figure S4. ^{1}H, $^{13}C\{^{1}H\}$ and $^{19}F\{^{1}H\}$ NMR spectra of compound **1f** (in $CDCl_3$, rt)


O
F

1g


1.0
0.97
1.03
3.13
2.06
0.99
δ / ppm


δ / ppm
158.700
156.416
142.481
139.338
134.049
130.194
130.171
130.155
130.125
129.688
129.220
128.377
127.273
125.840
125.441
77.367
77.160
76.945

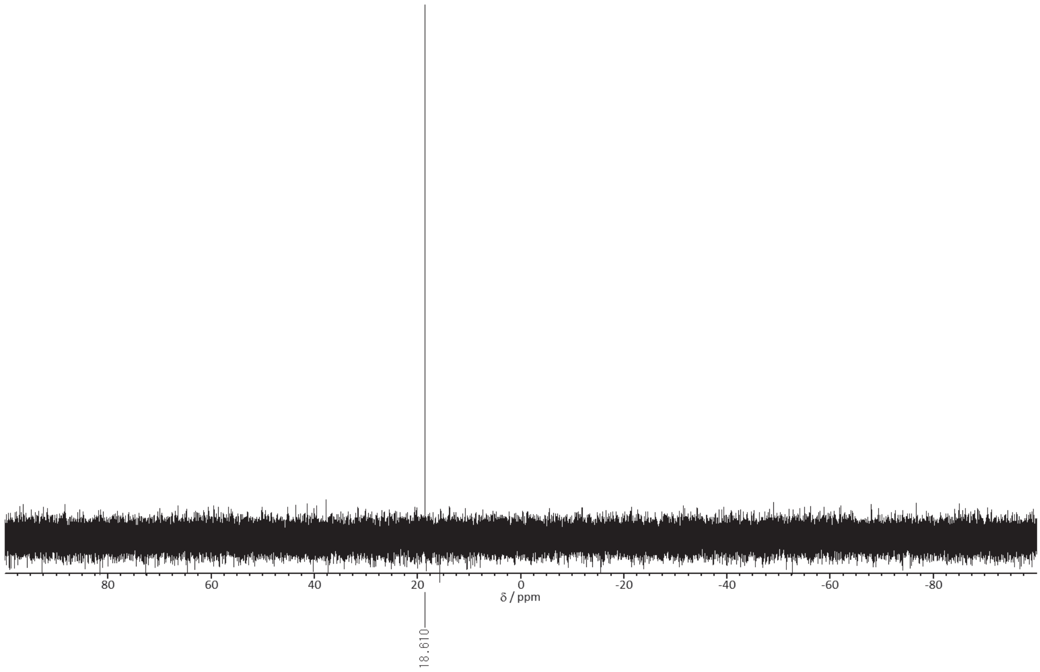
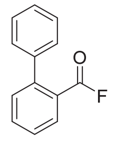
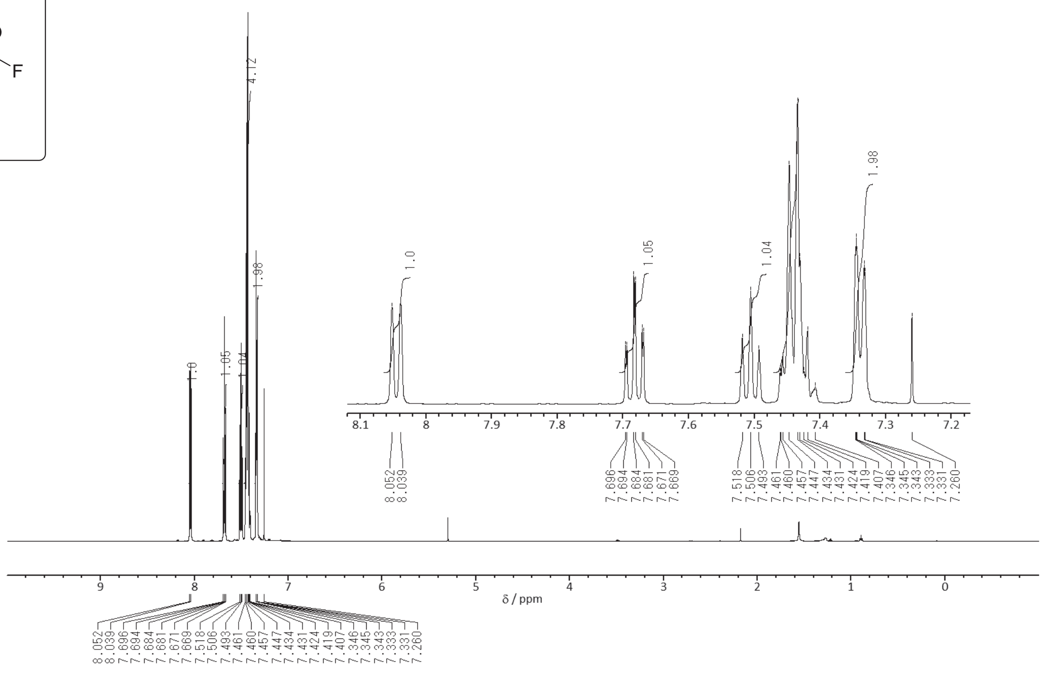
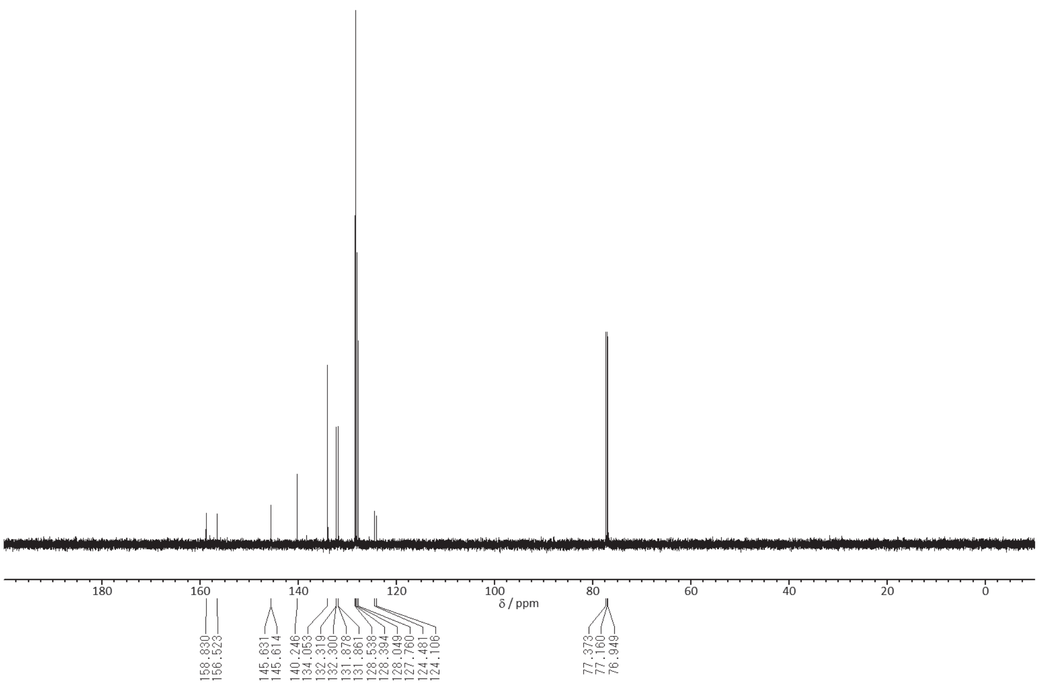


Figure S5. ^{1}H, ^{13}C{^{1}H} and ^{19}F{^{1}H} NMR spectra of compound **1g** (in $CDCl_3$, rt)


O
F

1h


1.0
1.05
1.04
4.12
1.98
δ / ppm
8.052
8.039
7.696
7.694
7.684
7.681
7.671
7.669
7.518
7.506
7.493
7.461
7.460
7.457
7.447
7.434
7.431
7.424
7.419
7.407
7.346
7.345
7.343
7.333
7.331
7.260


δ / ppm
158.830
156.523
145.631
145.614
140.246
134.053
132.319
132.300
131.878
131.861
128.538
128.394
128.049
127.760
124.481
124.106
77.373
77.160
76.949

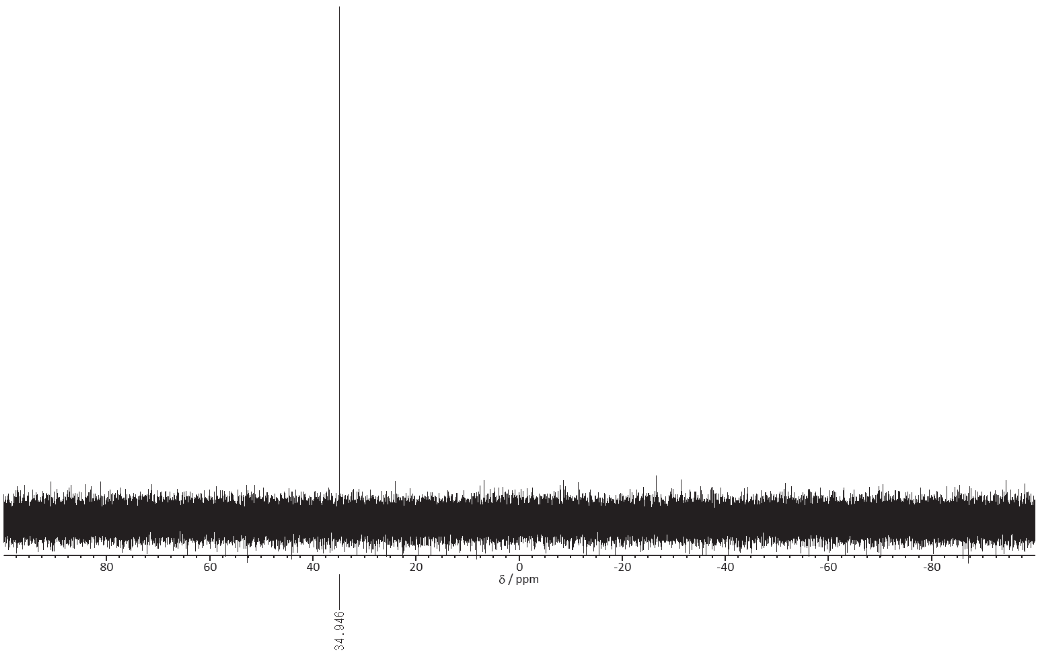
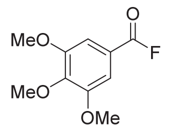
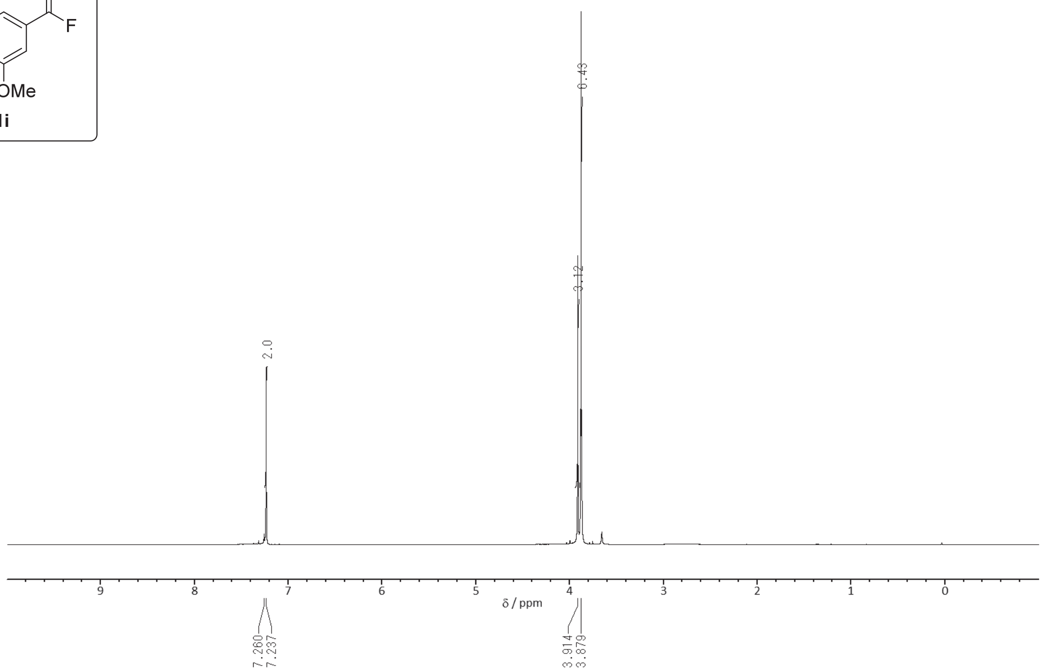
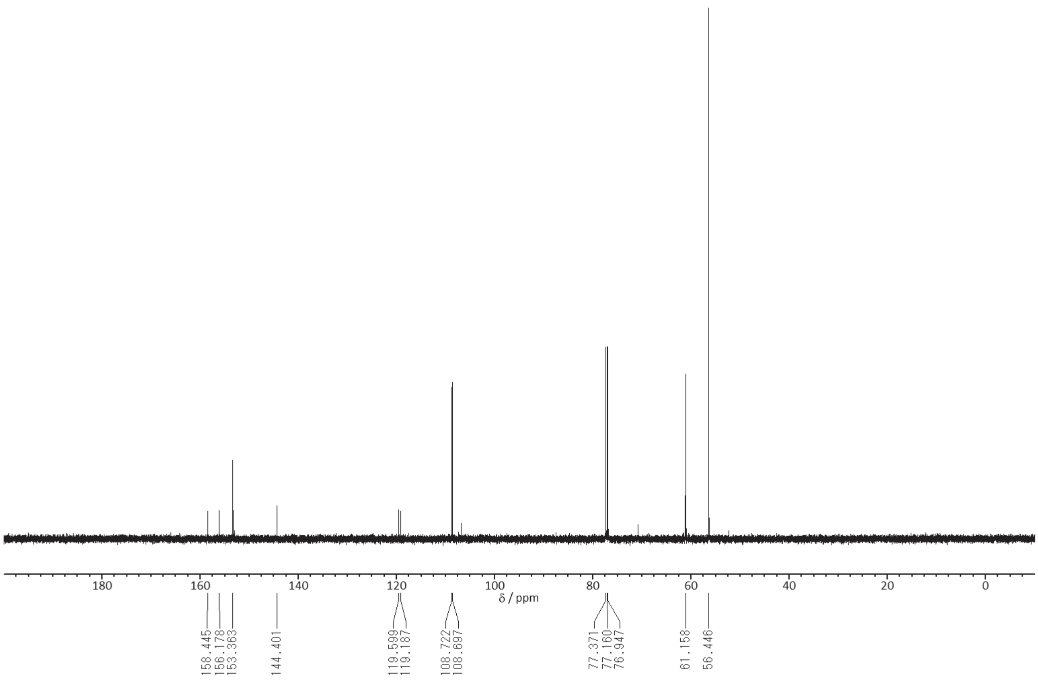


Figure S6. ^{1}H, $^{13}C\{^{1}H\}$ and $^{19}F\{^{1}H\}$ NMR spectra of compound **1h** (in $CDCl_3$, rt)


MeO
MeO
OMe
O
F

1i


2.0
3.12
6.43
9
8
7
6
5
4
3
2
1
0
δ / ppm
7.260
7.237
3.914
3.879


180
160
140
120
100
80
60
40
20
0
δ / ppm
158.445
156.178
153.363
144.401
119.599
119.187
108.722
108.697
77.371
77.160
76.947
61.158
56.446

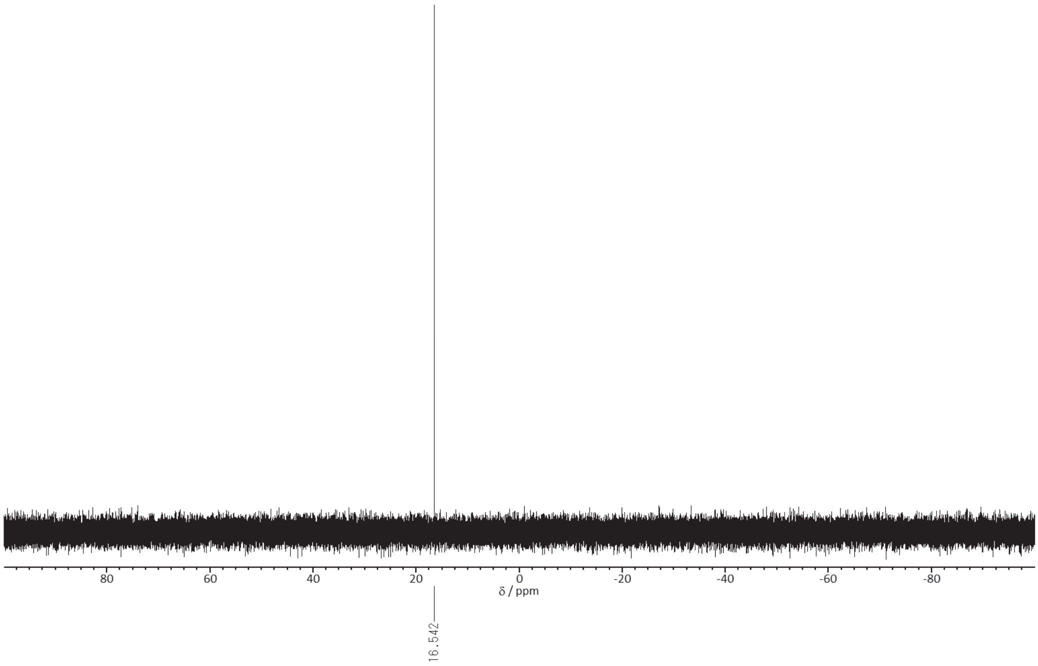
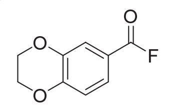
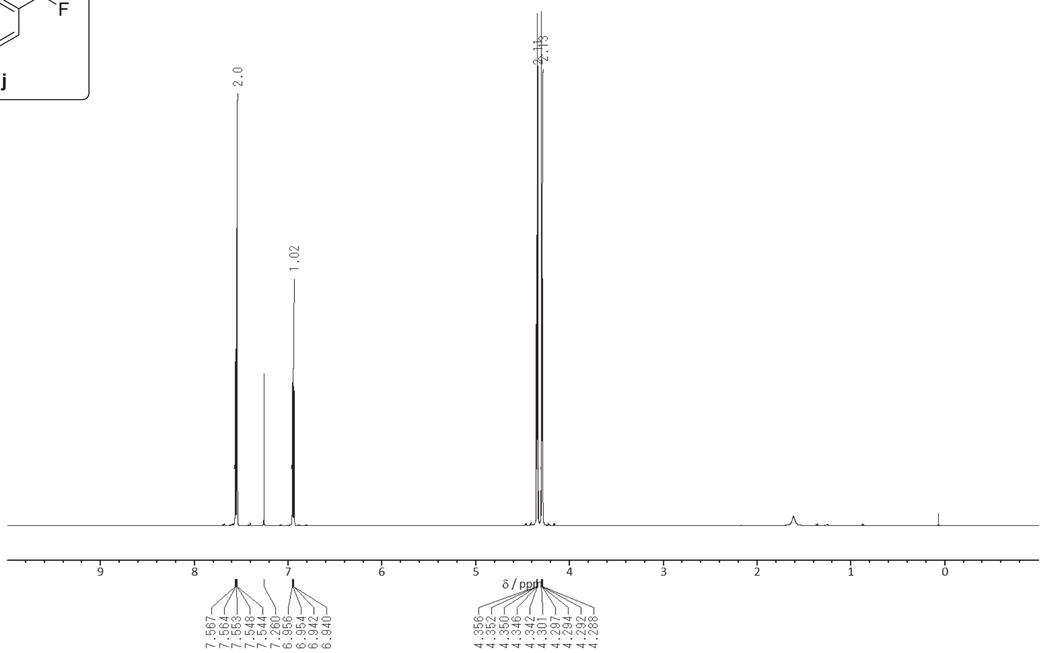
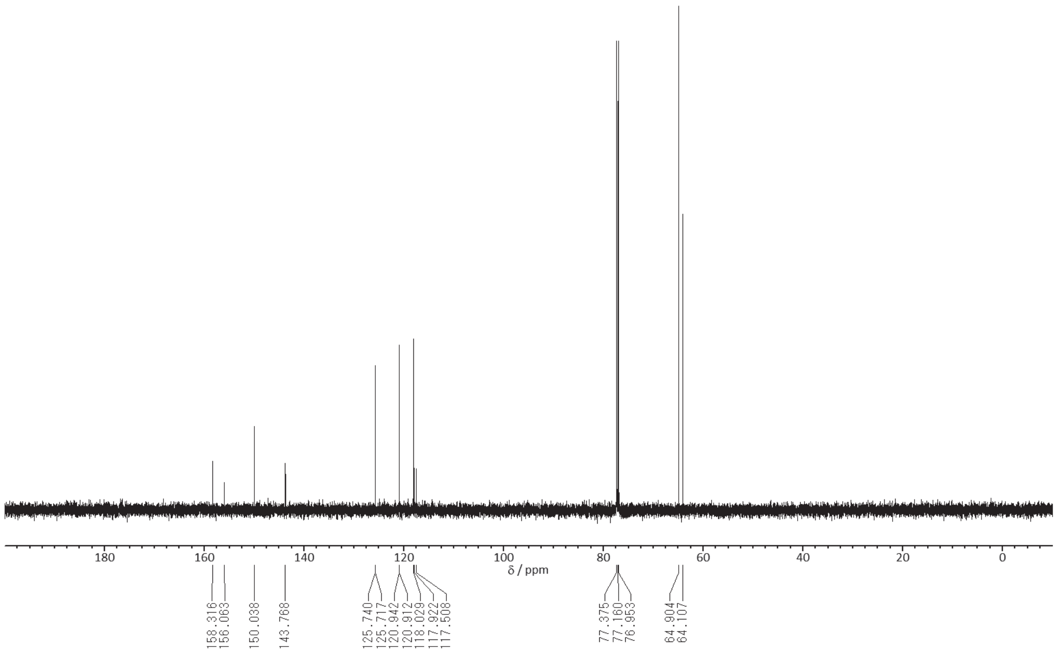


Figure S7. ^{1}H, $^{13}C\{^{1}H\}$ and $^{19}F\{^{1}H\}$ NMR spectra of compound **1i** (in $CDCl_3$, rt)


O
O
O
F

1j


2.0
1.02
2.11
2.13
9
8
7
6
5
4
3
2
1
0
δ / ppm
7.567
7.564
7.553
7.548
7.544
7.260
6.956
6.954
6.942
6.940
4.356
4.352
4.350
4.346
4.342
4.301
4.297
4.294
4.292
4.288


180
160
140
120
100
80
60
40
20
0
δ / ppm
158.316
156.063
150.038
143.768
125.740
125.717
120.942
120.912
118.029
117.922
117.508
77.375
77.160
76.953
64.904
64.107

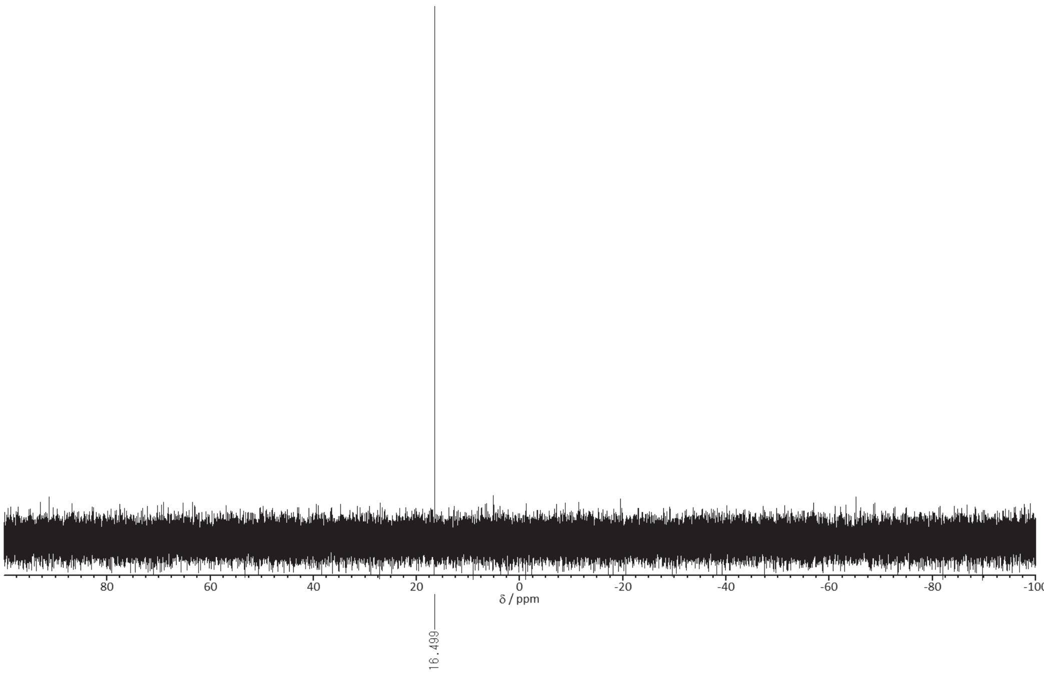
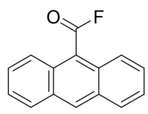
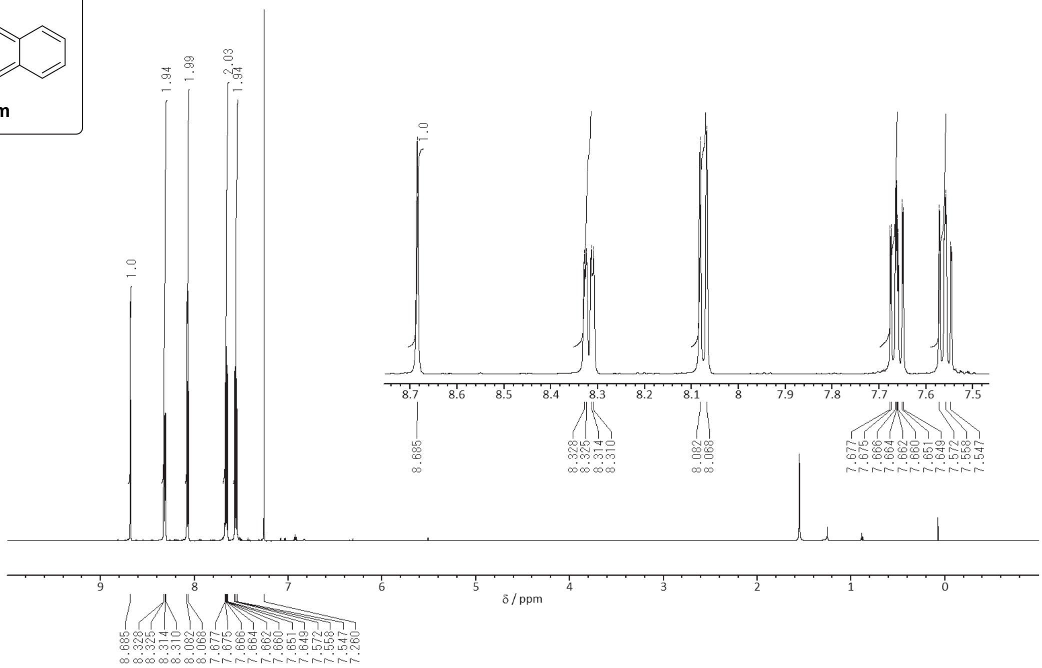
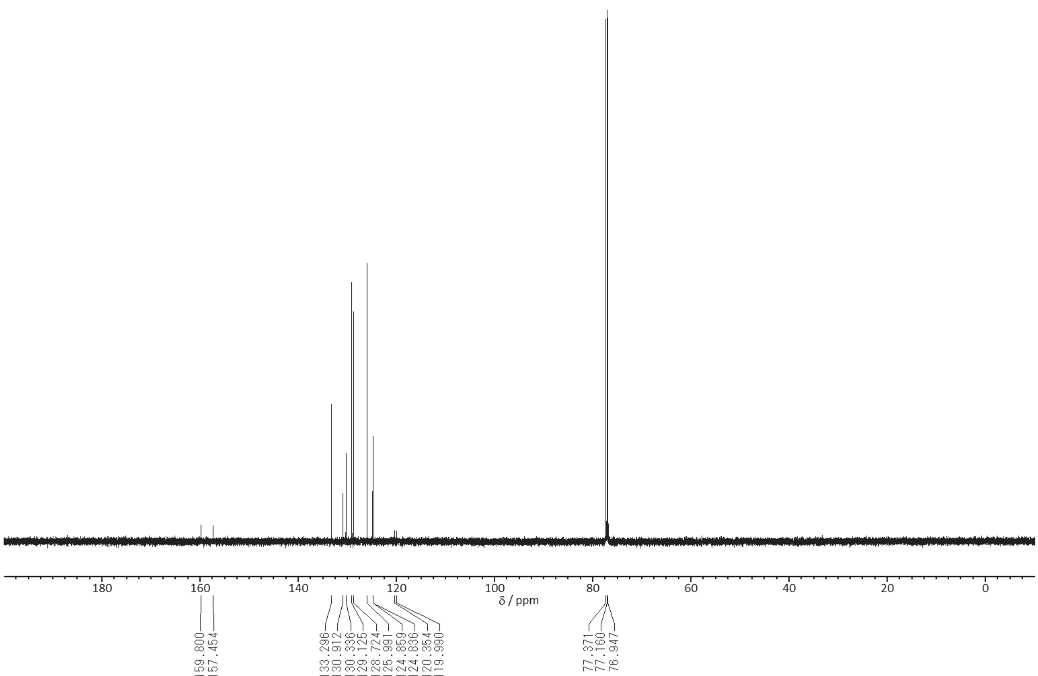


Figure S8. ${}^{1}H$, ${}^{13}C\{{}^{1}H\}$ and ${}^{19}F\{{}^{1}H\}$ NMR spectra of compound **1j** (in $CDCl_3$, rt)


O
F

1m


1.0
1.94
1.99
2.03
1.94
8.685
8.328
8.325
8.314
8.310
8.082
8.068
7.677
7.675
7.666
7.664
7.662
7.660
7.651
7.649
7.572
7.558
7.547
7.260
δ / ppm


159.800
157.454
133.296
130.912
130.336
129.125
128.724
125.991
124.859
124.836
120.354
119.990
77.371
77.160
76.947
δ / ppm

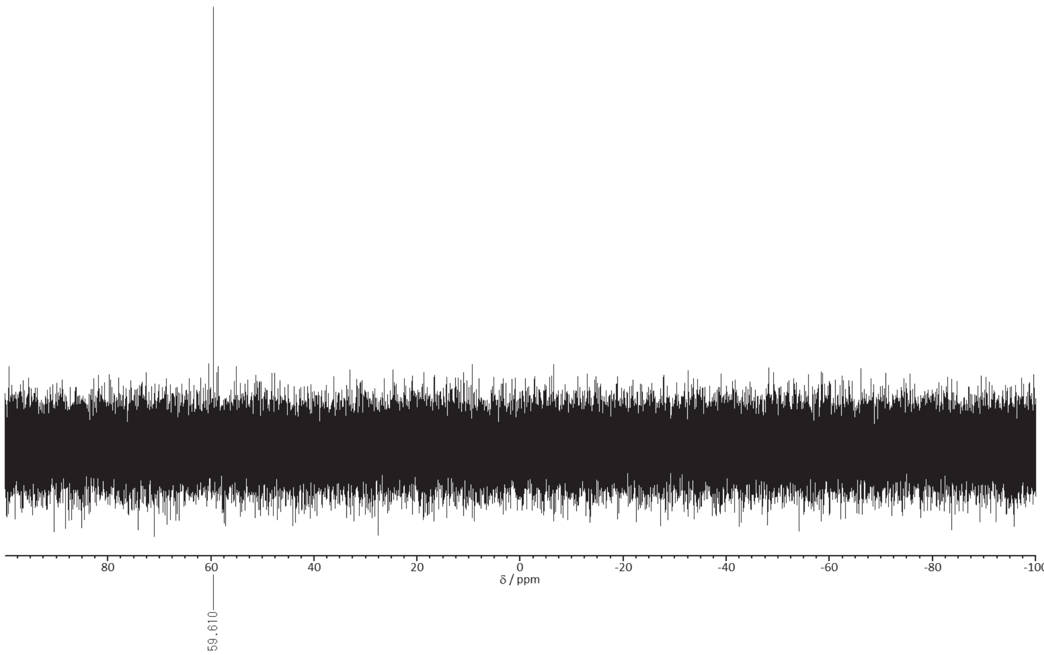
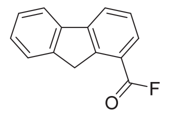
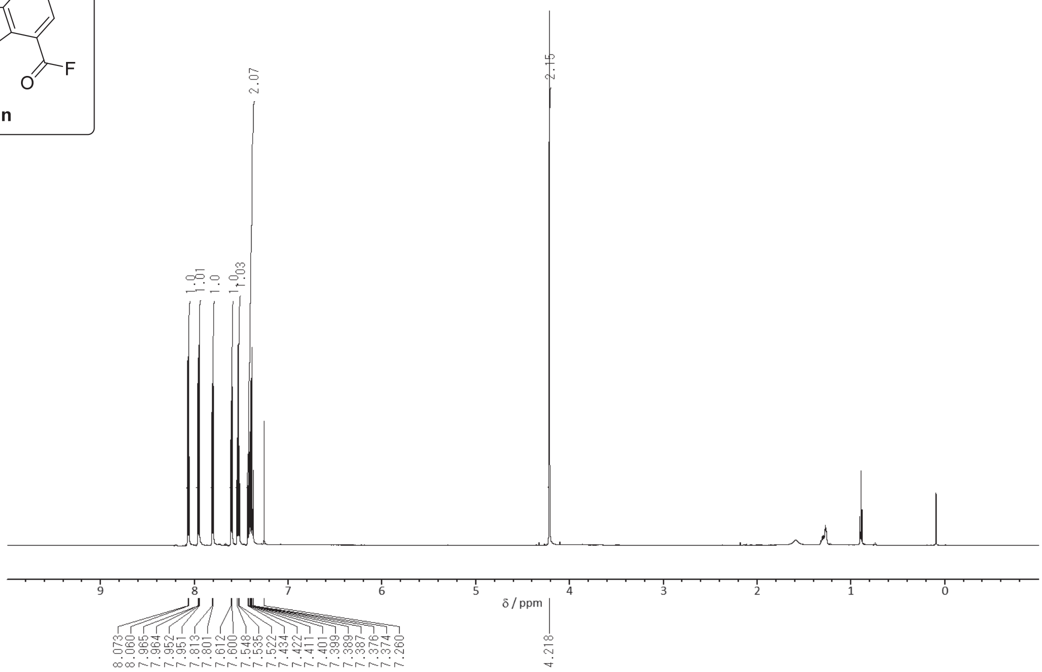
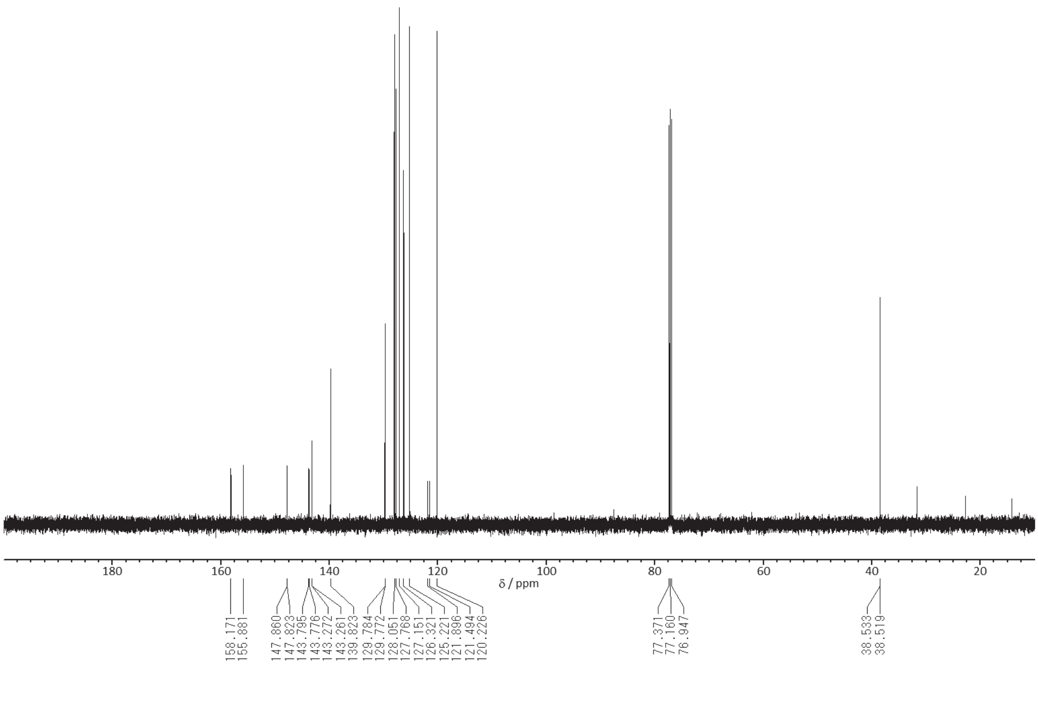


Figure S9. ^{1}H, $^{13}C\{^{1}H\}$ and $^{19}F\{^{1}H\}$ NMR spectra of compound **1m** (in $CDCl_3$, rt)



1n





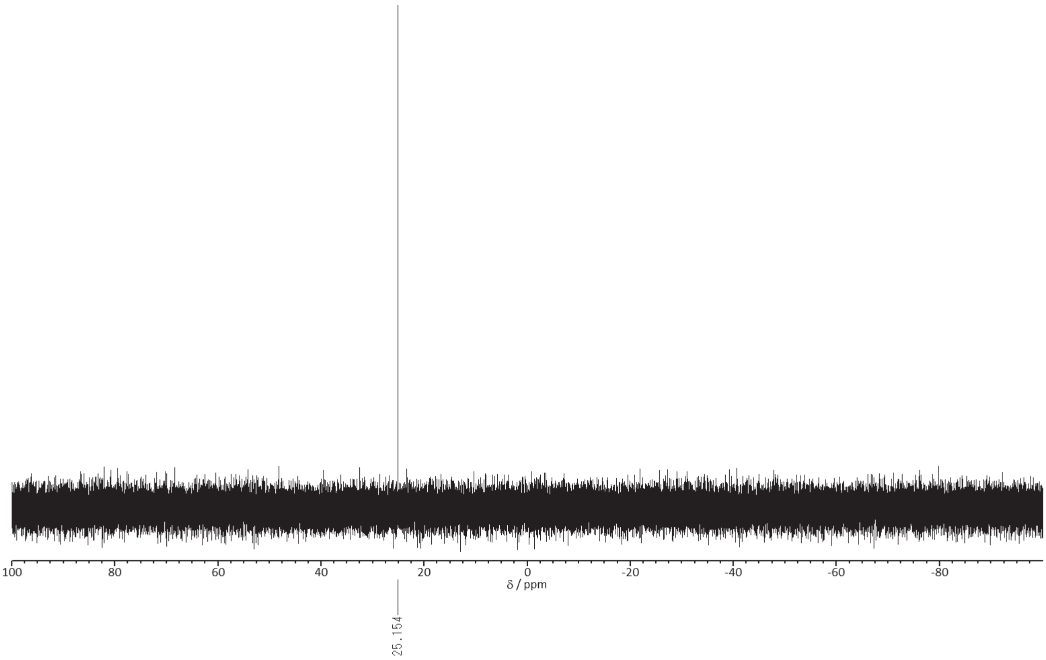
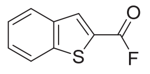
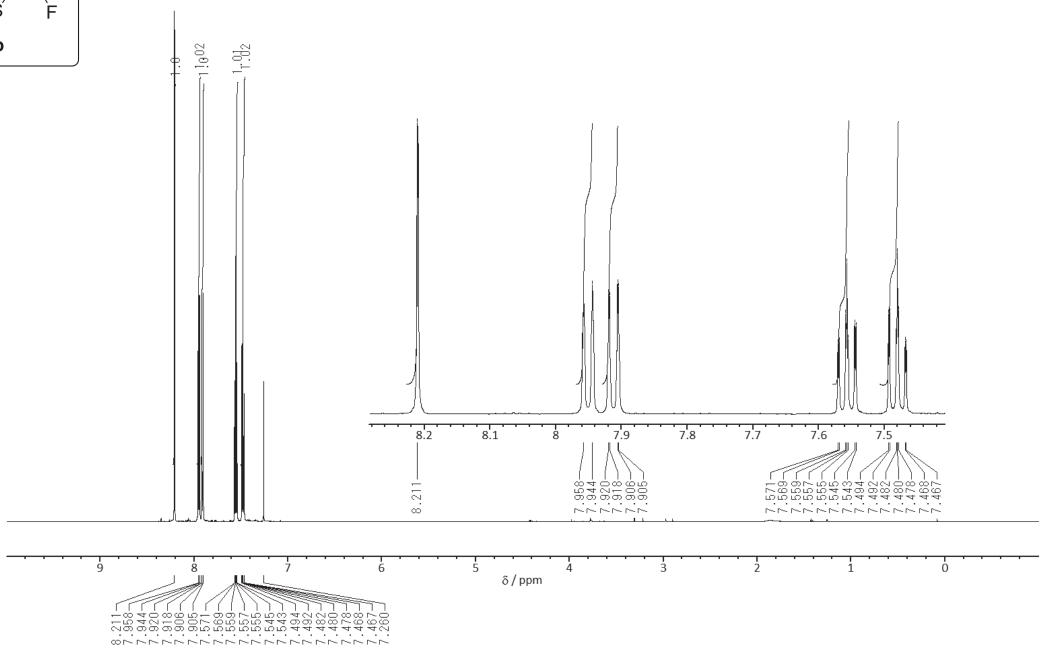
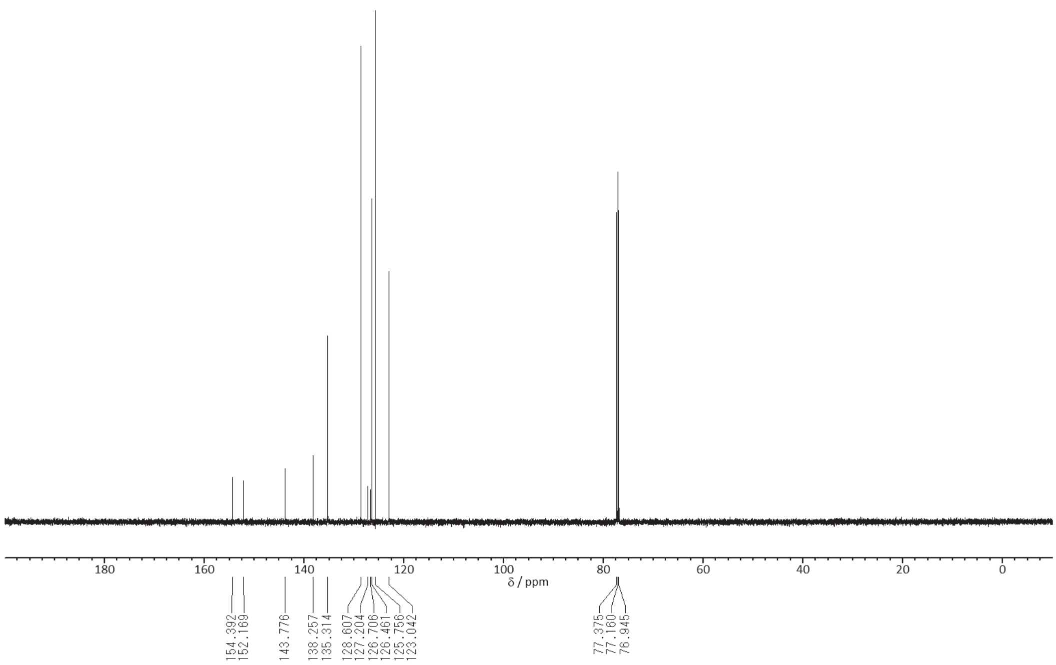


Figure S10. ^{1}H, $^{13}C\{^{1}H\}$ and $^{19}F\{^{1}H\}$ NMR spectra of compound **1n** (in $CDCl_3$, rt)


O
S
F

1p


1.0
1.02
1.01
1.02
8.211
7.958
7.944
7.920
7.918
7.906
7.905
7.571
7.569
7.559
7.557
7.555
7.545
7.543
7.494
7.492
7.482
7.480
7.478
7.468
7.467
7.260
δ / ppm


154.392
152.169
143.776
138.257
135.314
128.607
127.204
126.706
126.461
125.756
123.042
77.375
77.160
76.945
δ / ppm

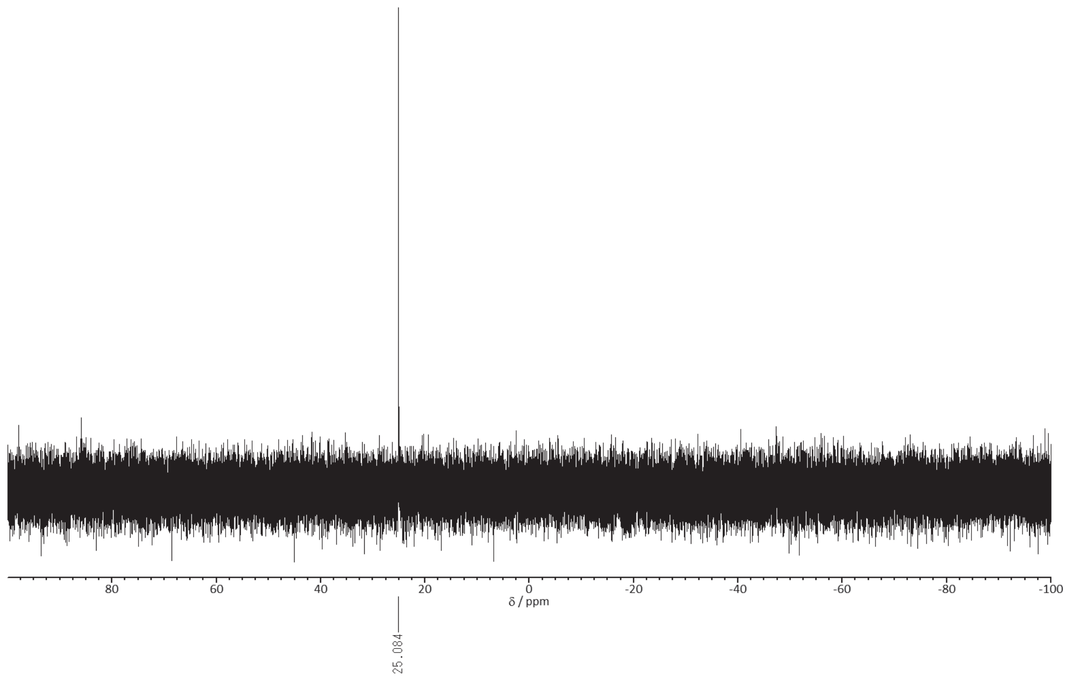
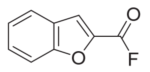
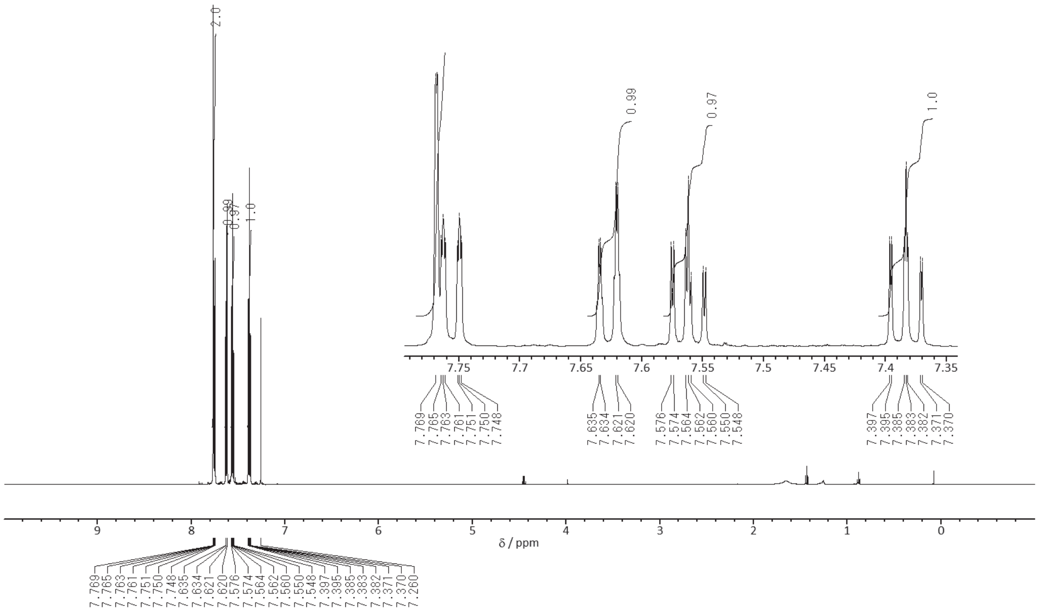
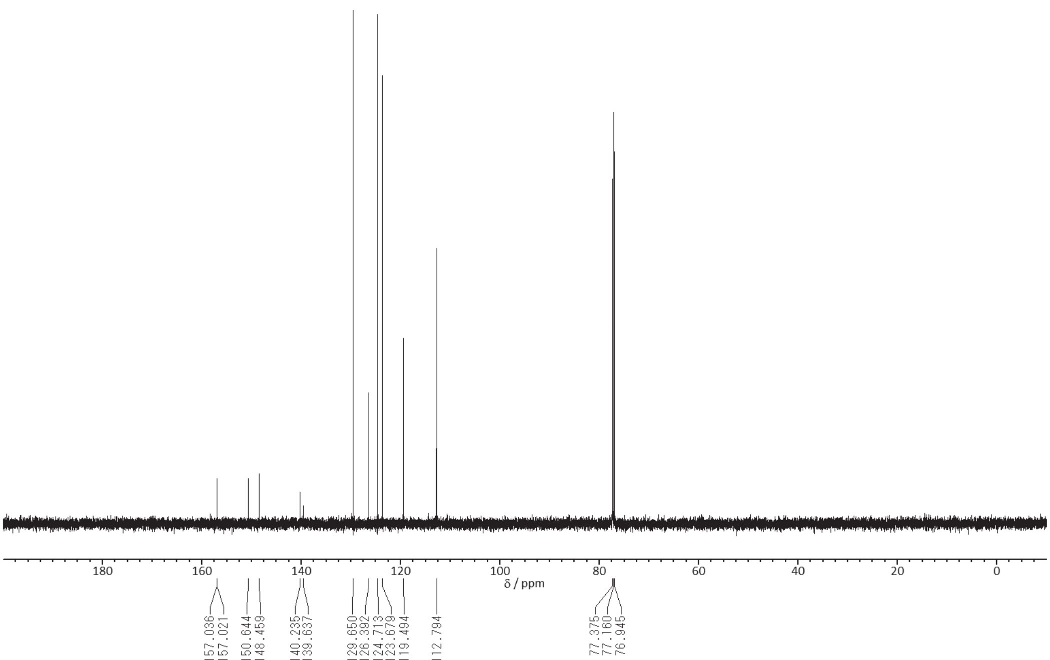


Figure S11. ^{1}H, $^{13}C\{^{1}H\}$ and $^{19}F\{^{1}H\}$ NMR spectra of compound **1p** (in $CDCl_3$)



1q


δ / ppm
7.769
7.765
7.763
7.761
7.751
7.750
7.748
7.635
7.634
7.621
7.620
7.576
7.574
7.564
7.562
7.560
7.550
7.548
7.397
7.395
7.385
7.383
7.382
7.371
7.370
7.260


δ / ppm
157.036
157.021
150.644
148.459
140.235
139.637
129.650
126.392
124.713
123.679
119.494
112.794
77.375
77.160
76.945

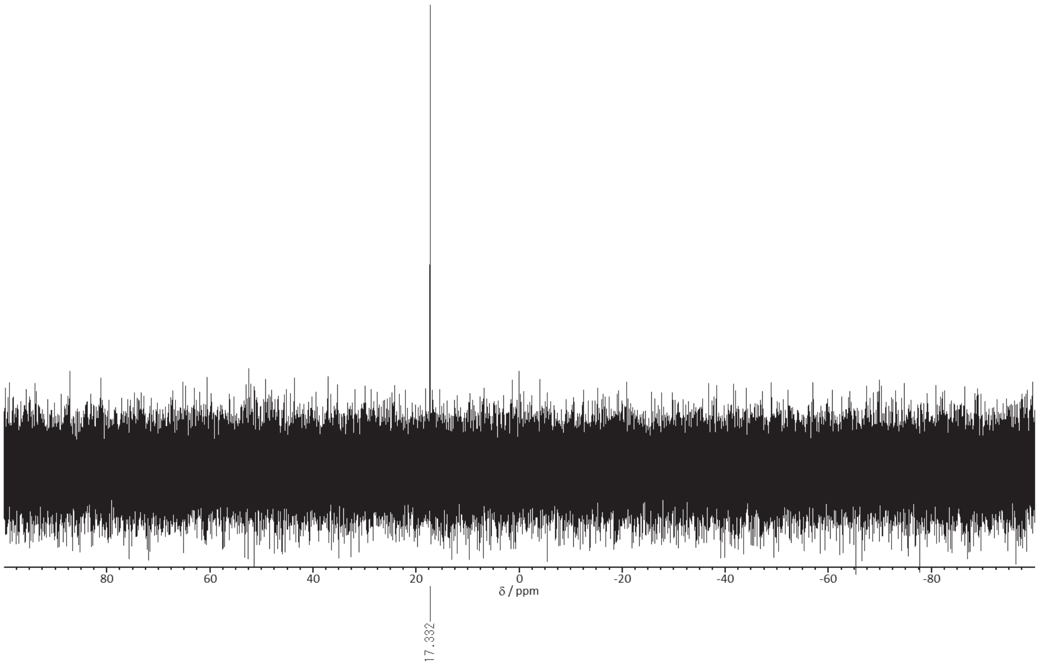
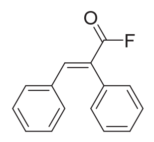
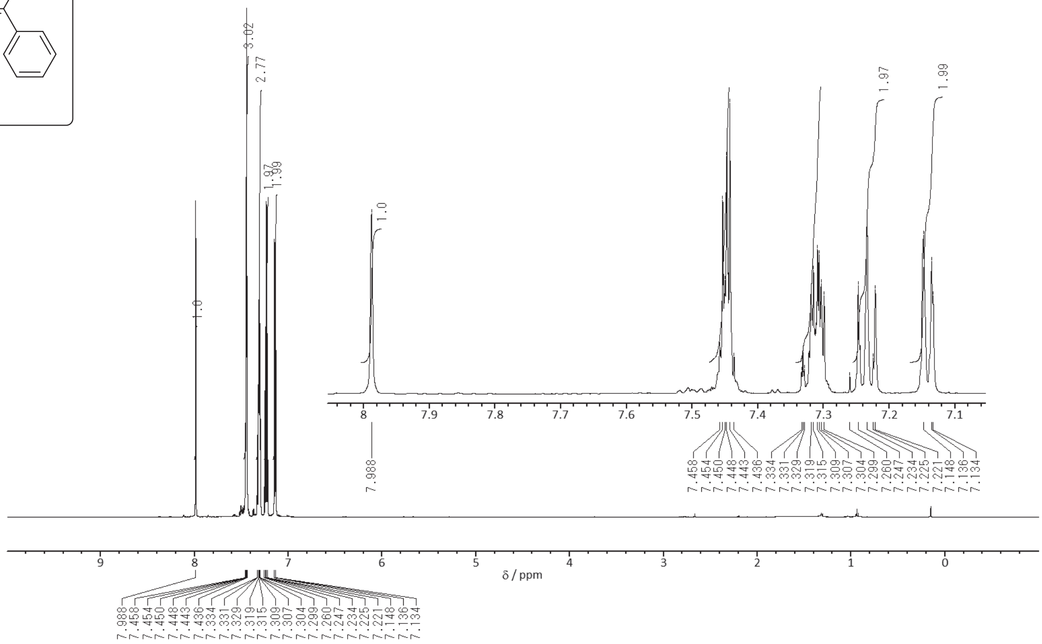
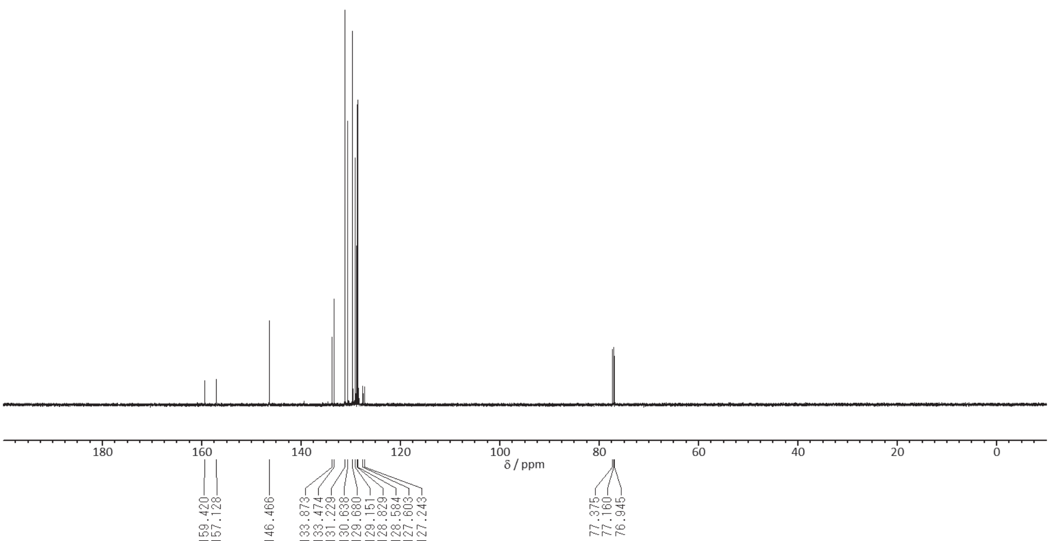


Figure S12. ^1H, $^{13}C\{^1H\}$ and $^{19}F\{^1H\}$ NMR spectra of compound **1q** (in $CDCl_3$)


O
F

1r


1.0
3.02
2.77
1.97
1.99
7.988
7.458
7.454
7.450
7.448
7.443
7.436
7.334
7.331
7.329
7.319
7.315
7.309
7.307
7.304
7.299
7.260
7.247
7.234
7.225
7.221
7.148
7.136
7.134
δ / ppm


159.420
157.128
146.466
133.873
133.474
131.229
130.638
129.680
129.151
128.829
128.584
127.603
127.243
77.375
77.160
76.945
δ / ppm

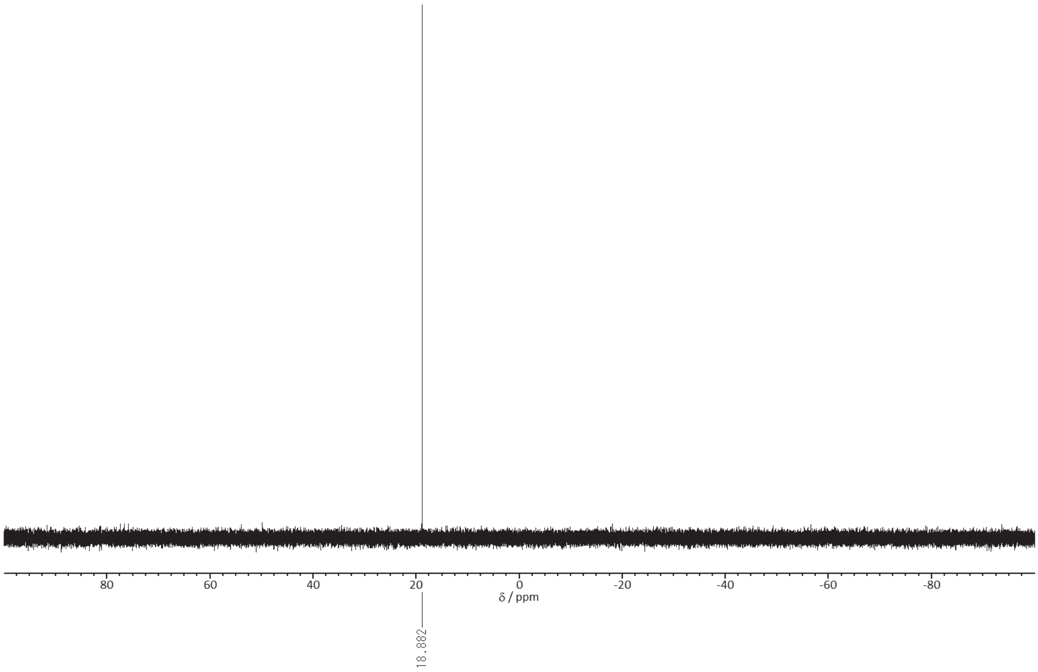
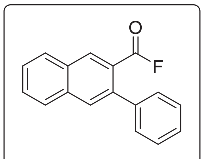
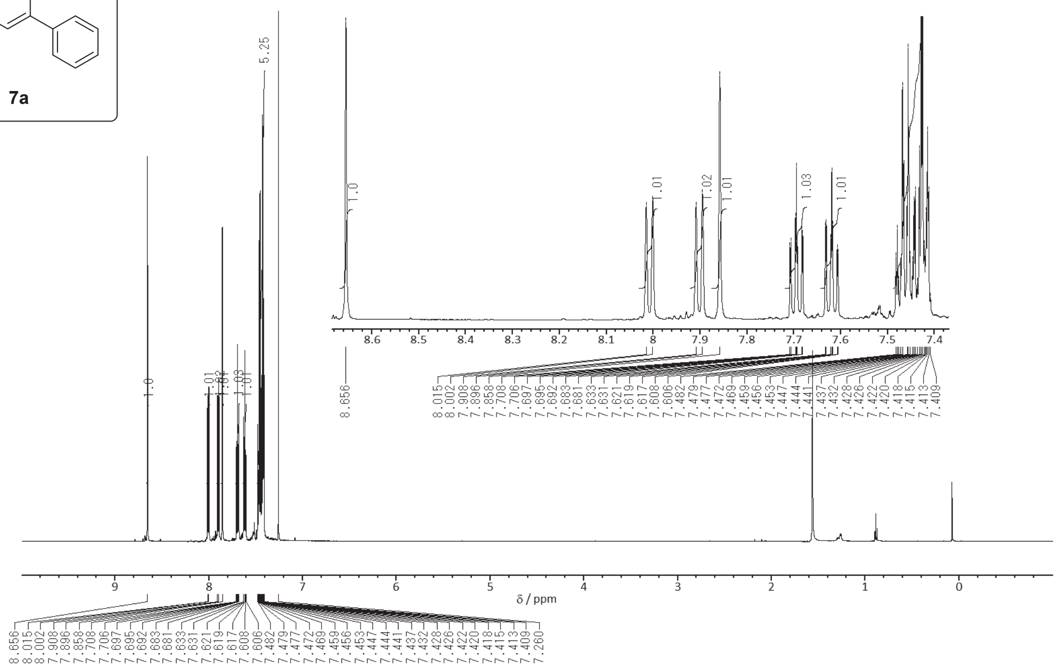
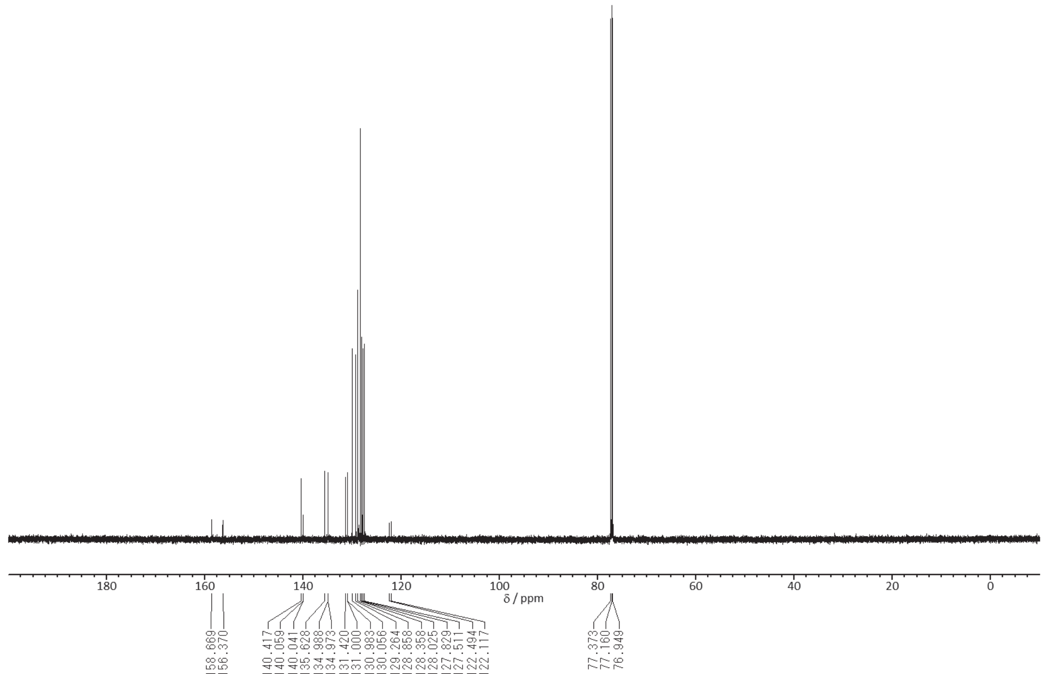


Figure S13. ^{1}H, $^{13}C\{^{1}H\}$ and $^{19}F\{^{1}H\}$ NMR spectra of compound **1r** (in $CDCl_3$)



7a





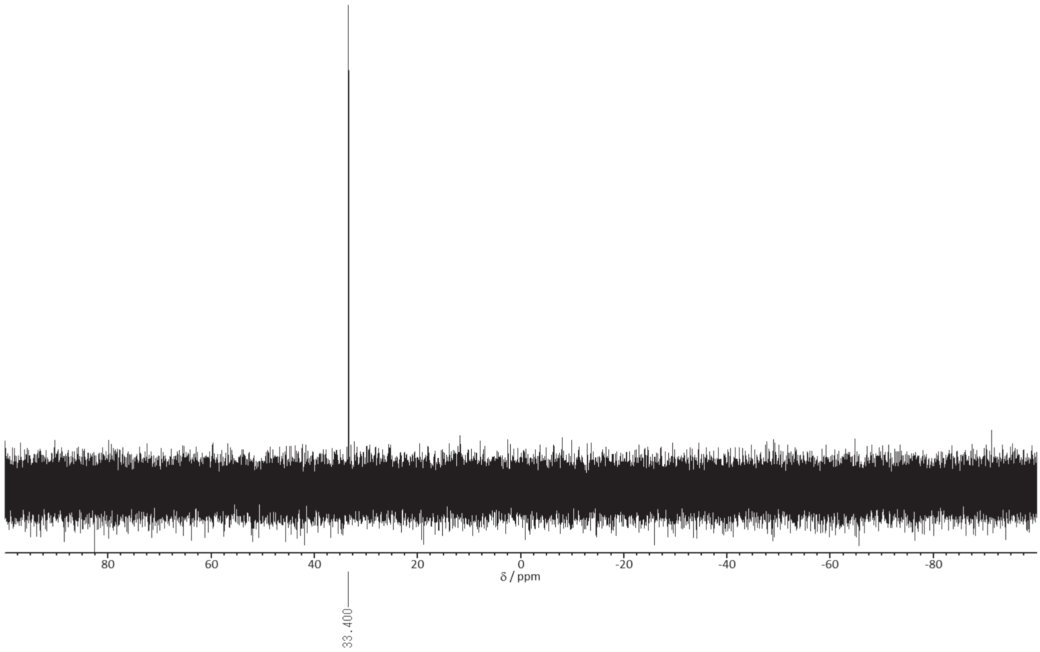
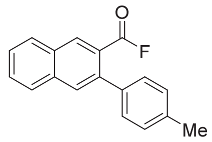
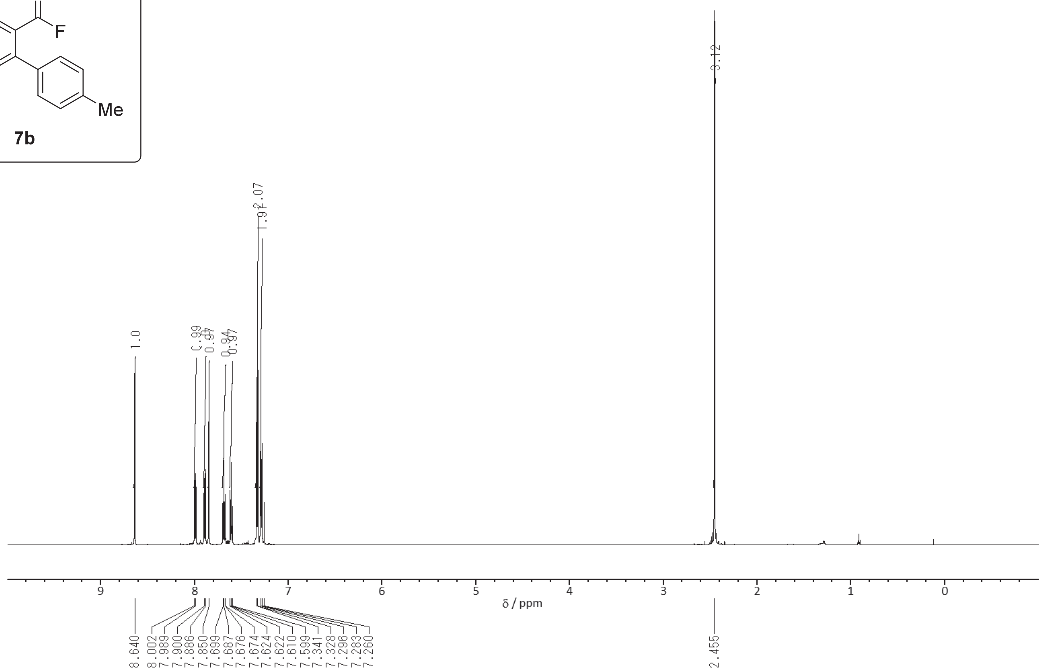
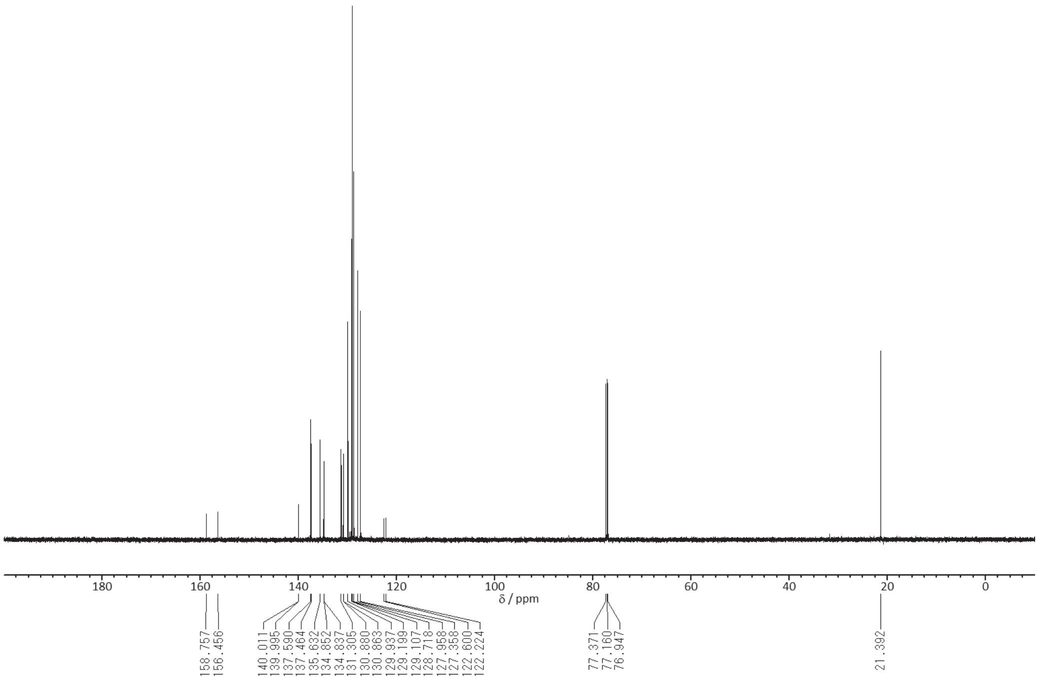


Figure S14. ^{1}H, $^{13}C\{^{1}H\}$ and $^{19}F\{^{1}H\}$ NMR spectra of compound **7a** (in $CDCl_3$)


O
F
Me

7b


δ / ppm
8.640
8.002
7.989
7.900
7.886
7.850
7.699
7.687
7.676
7.674
7.624
7.622
7.610
7.599
7.341
7.328
7.296
7.283
7.260
2.455


δ / ppm
158.757
156.456
140.011
139.995
137.590
137.464
135.632
134.852
134.837
131.305
130.880
130.863
129.937
129.199
129.107
128.718
127.958
127.358
122.600
122.224
77.371
77.160
76.947
21.392

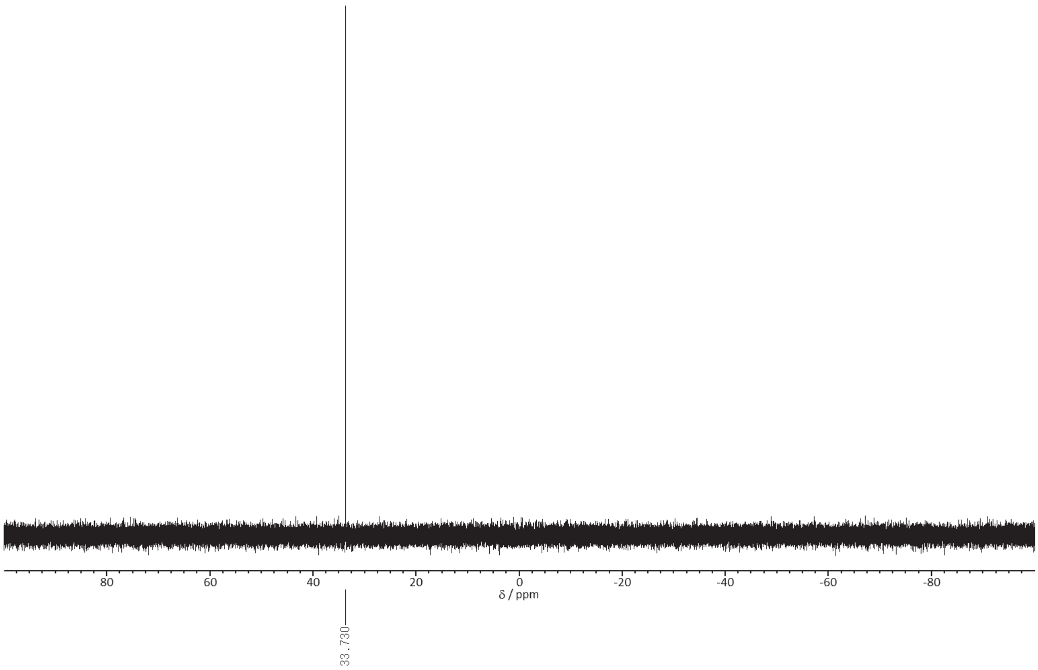
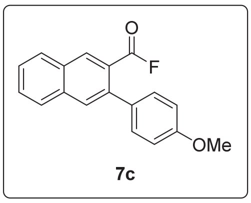
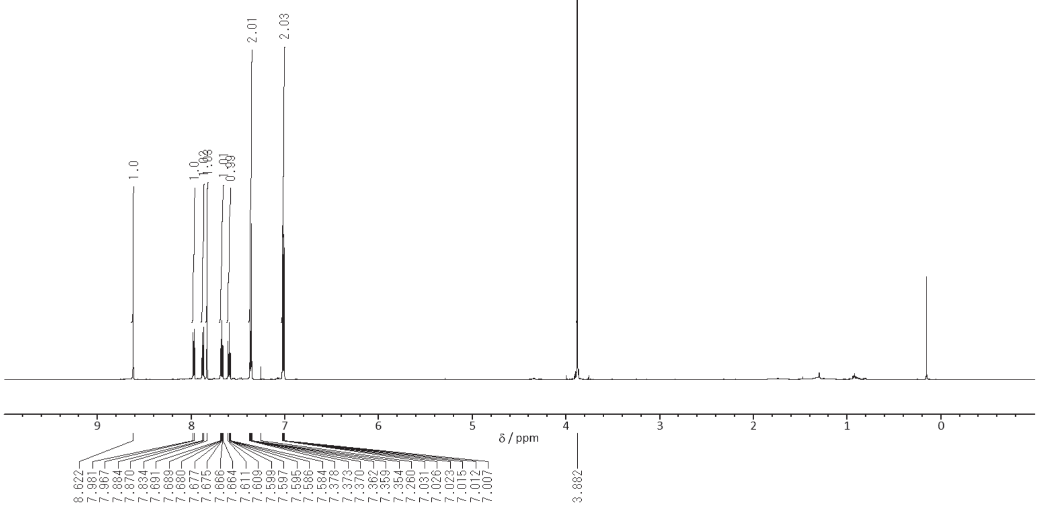
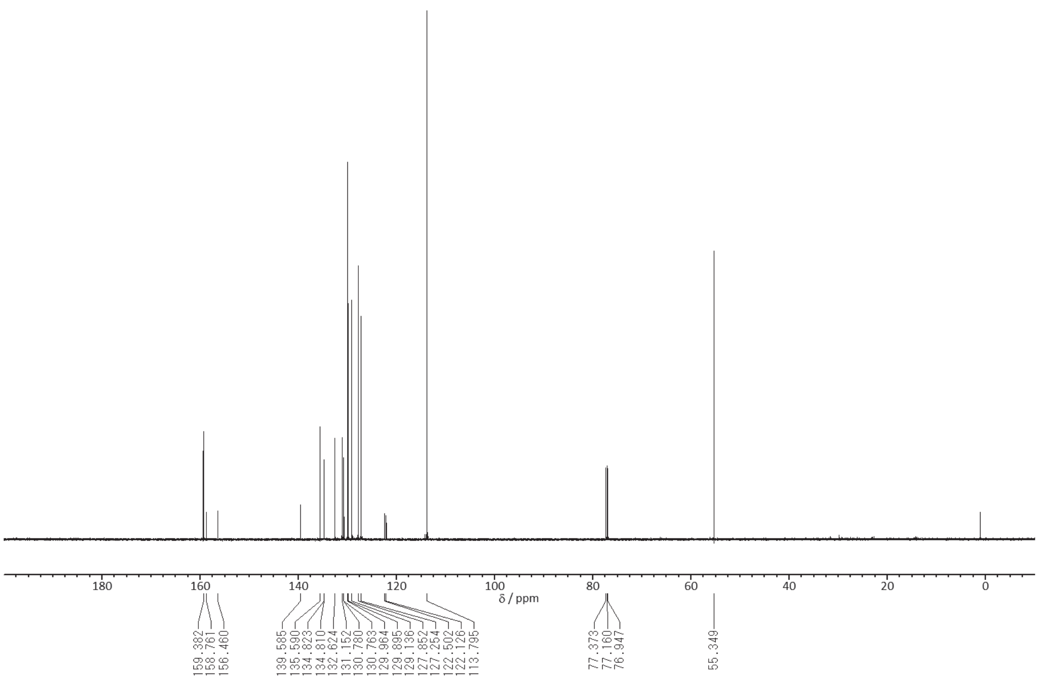


Figure S15. ^{1}H, $^{13}C\{^{1}H\}$ and $^{19}F\{^{1}H\}$ NMR spectra of compound **7b** (in $CDCl_3$)




2.94
2.01
2.03
1.0
1.03
1.08
1.01
0.99
9
8
7
6
5
4
3
2
1
0
δ / ppm
8.622
7.981
7.967
7.884
7.870
7.834
7.691
7.689
7.680
7.677
7.675
7.666
7.664
7.611
7.609
7.599
7.597
7.595
7.586
7.584
7.378
7.373
7.370
7.362
7.359
7.354
7.260
7.031
7.026
7.023
7.015
7.012
7.007
3.882


180
160
140
120
100
80
60
40
20
0
δ / ppm
159.382
158.761
156.460
139.585
135.590
134.823
134.810
132.624
131.152
130.780
130.763
129.964
129.895
129.136
127.852
127.254
122.502
122.126
113.795
77.373
77.160
76.947
55.349

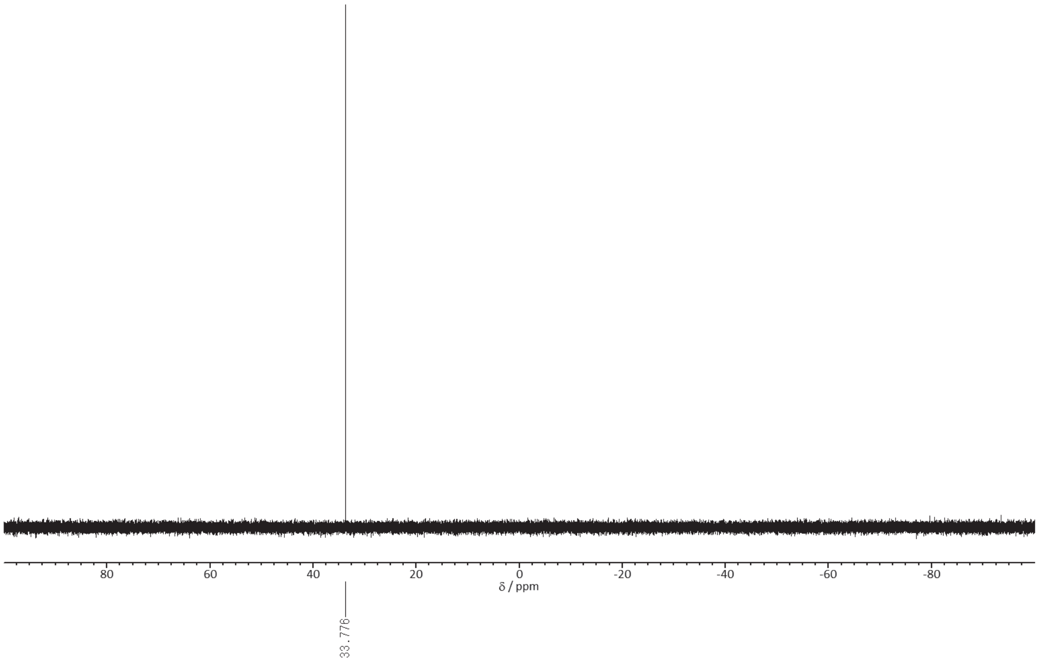
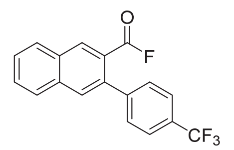
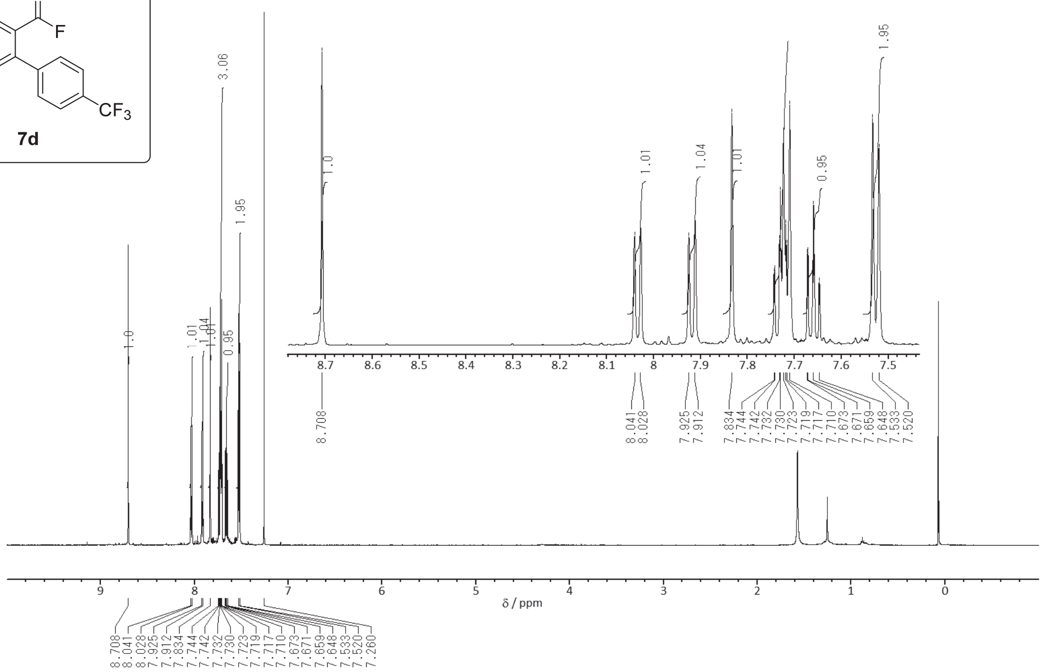
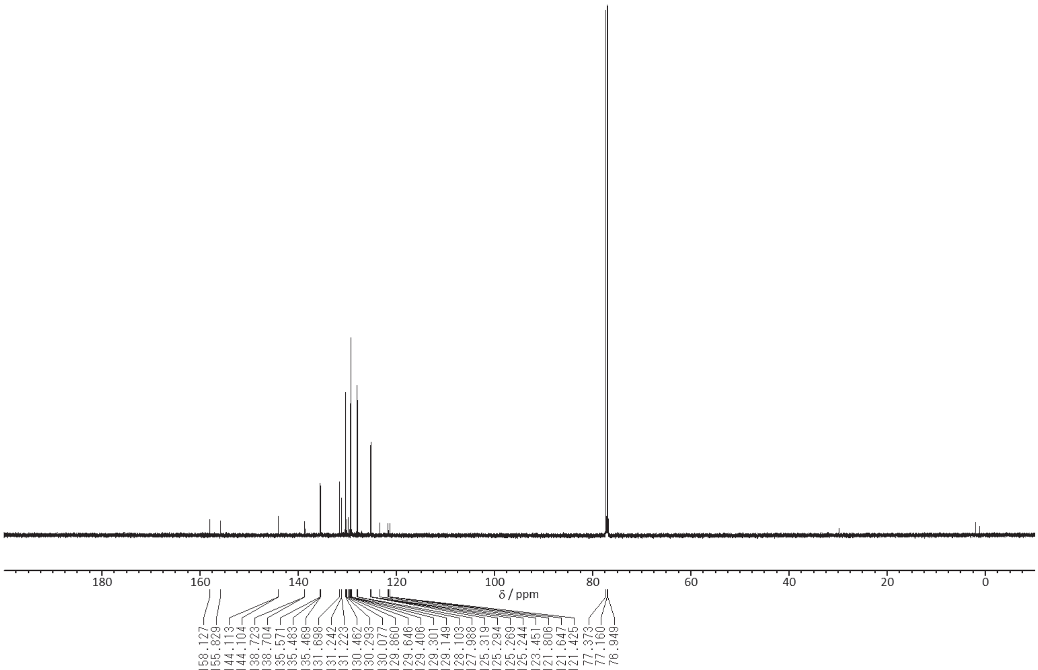


Figure S16. ^{1}H, $^{13}C\{^{1}H\}$ and $^{19}F\{^{1}H\}$ NMR spectra of compound **7c** (in $CDCl_3$)


O
F
CF3

7d


δ / ppm
1.0
1.01
1.04
1.01
0.95
3.06
1.95
8.708
8.041
8.028
7.925
7.912
7.834
7.744
7.742
7.732
7.730
7.723
7.719
7.717
7.710
7.673
7.671
7.659
7.648
7.533
7.520
7.260


δ / ppm
158.127
155.829
144.113
144.104
138.723
138.704
135.571
135.483
135.469
131.698
131.242
131.223
130.462
130.293
130.077
129.860
129.646
129.406
129.301
129.149
128.103
127.988
125.319
125.294
125.269
125.244
123.451
121.806
121.647
121.425
77.373
77.160
76.949

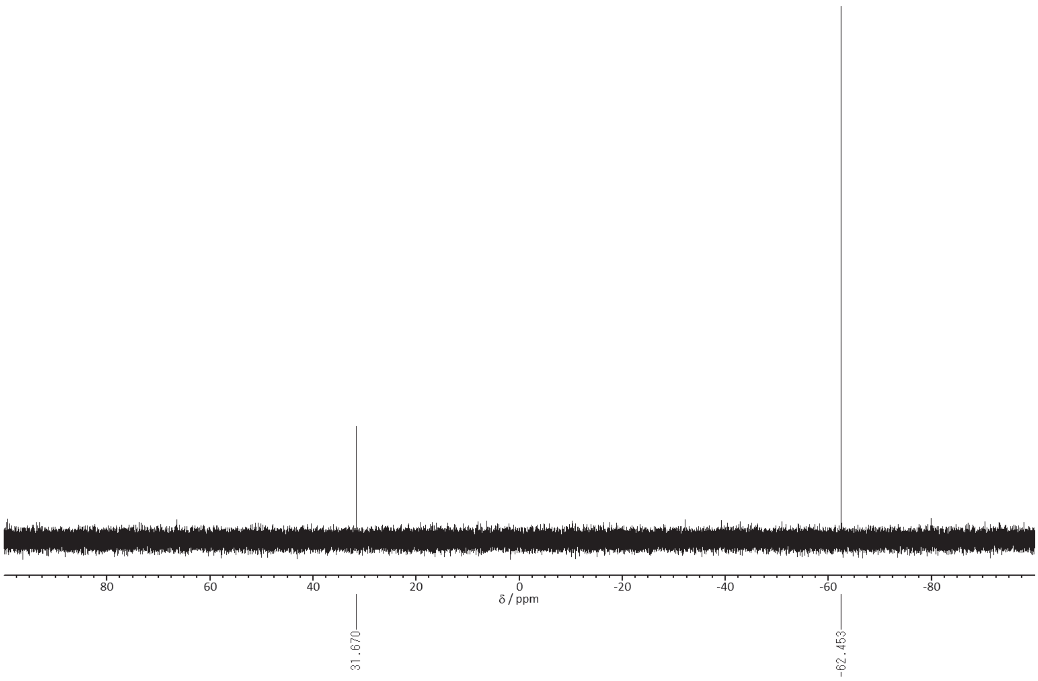
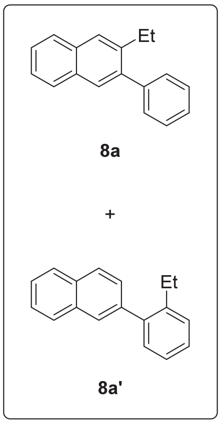
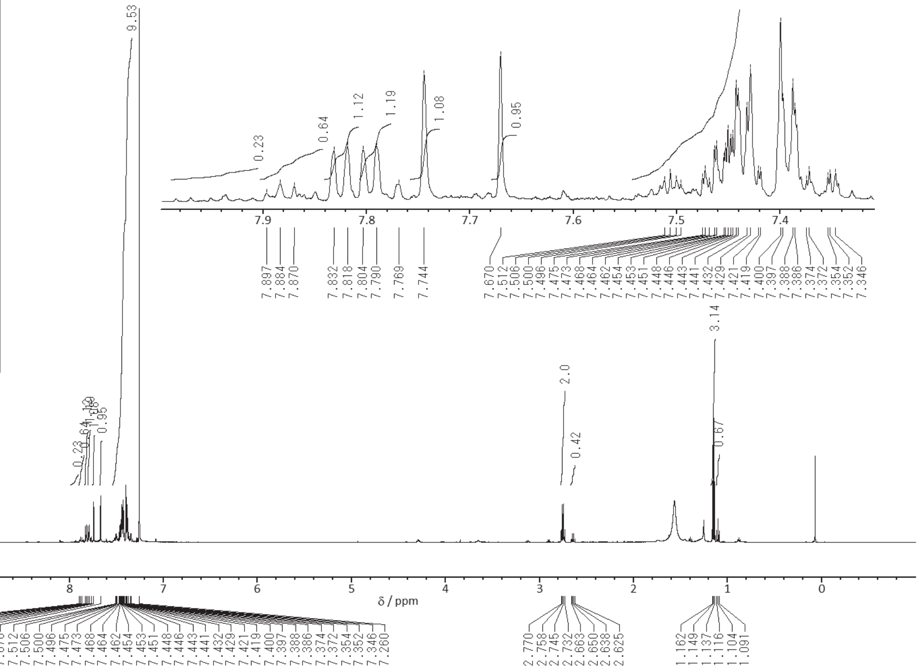


Figure S17. ^{1}H, $^{13}C\{^{1}H\}$ and $^{19}F\{^{1}H\}$ NMR spectra of compound **7d** (in $CDCl_3$)





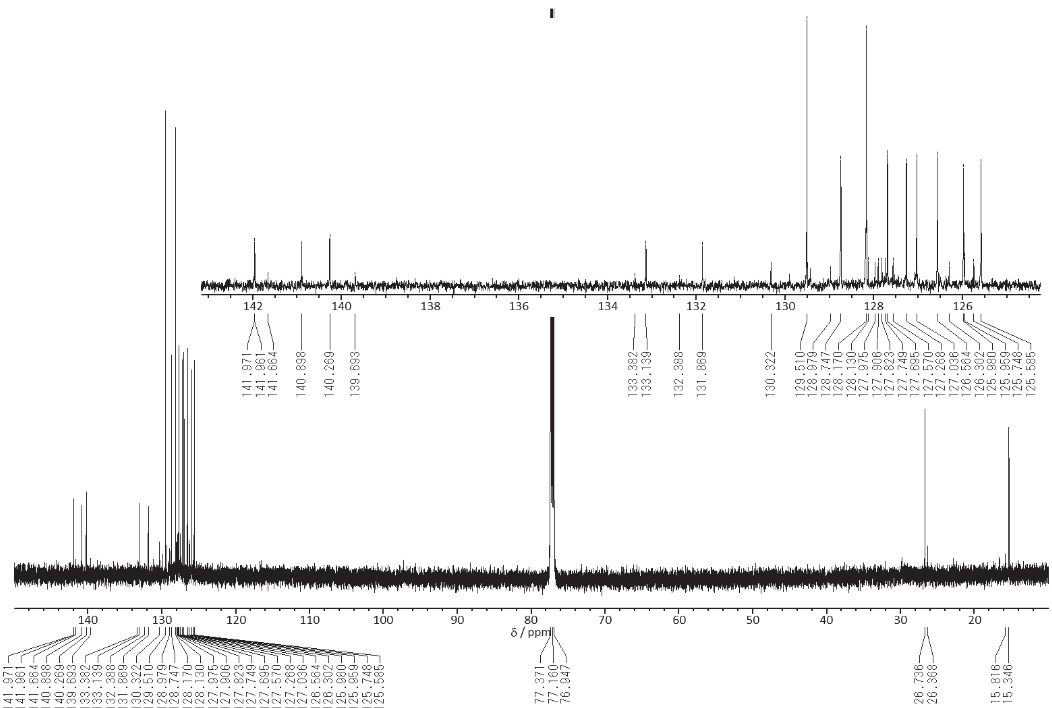
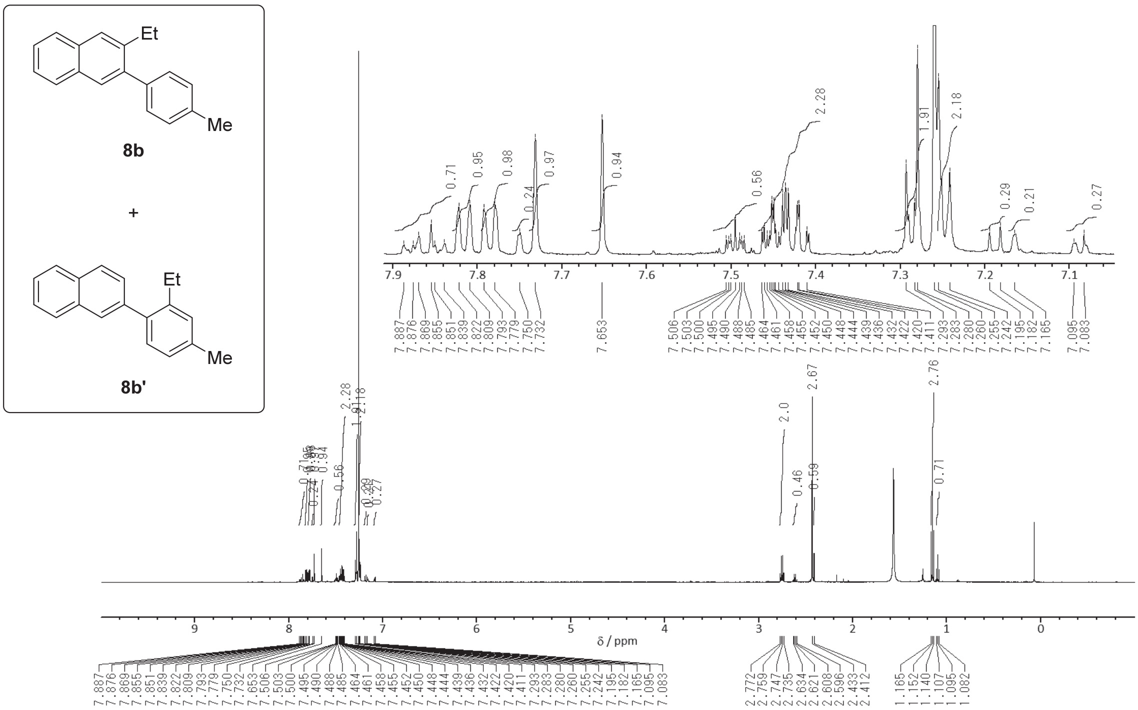


Figure S18. ^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of mixture of **8a** and **8a'** (in $CDCl_3$)



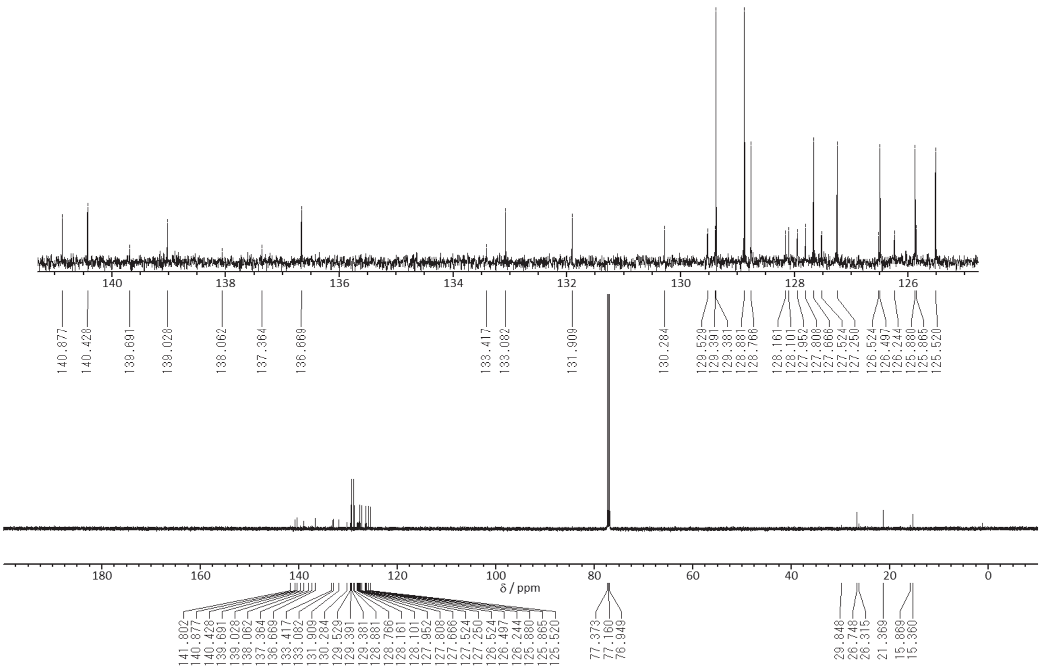


Figure S19. ^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of mixture of **8b** and **8b'** (in $CDCl_3$)

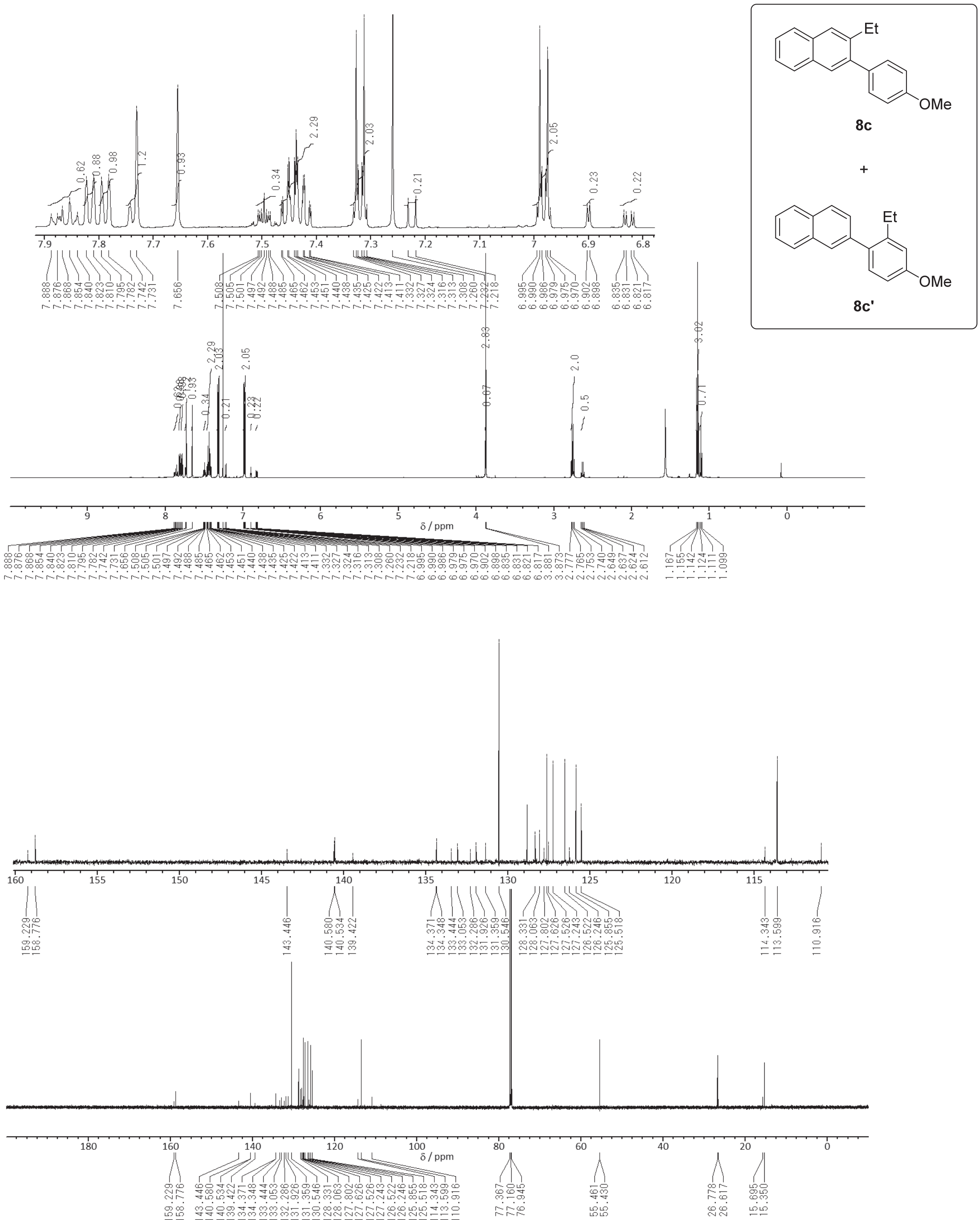
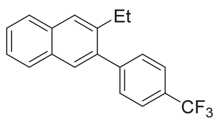
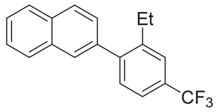


Figure S20. ^{1}H and ^{13}C{^{1}H} NMR spectra of mixture of **8c** and **8c'** (in $CDCl_3$)

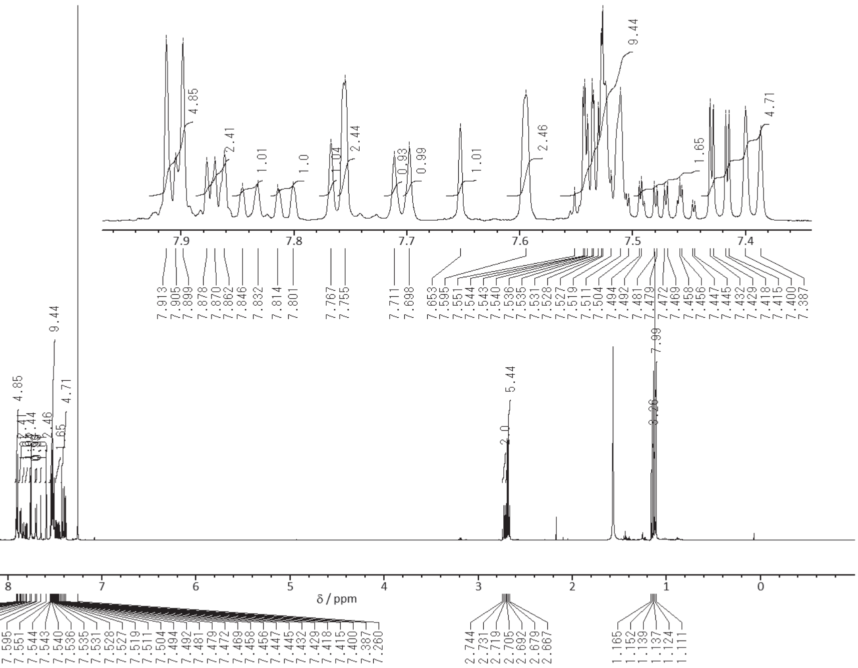


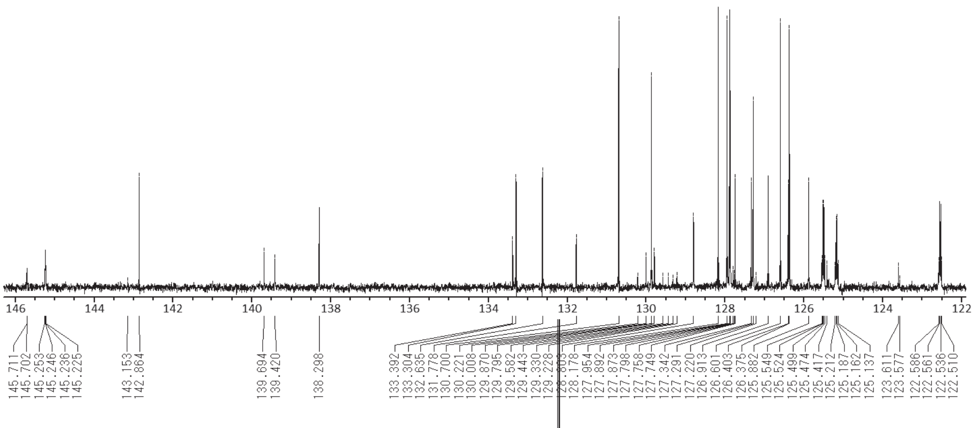
8d

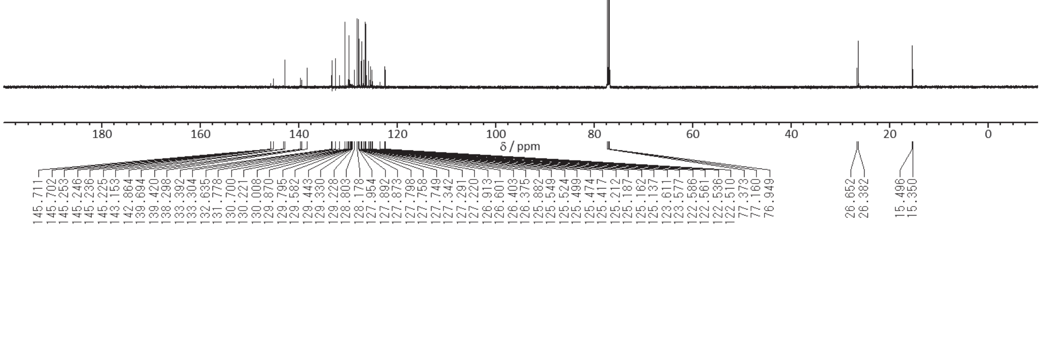
+



8d'







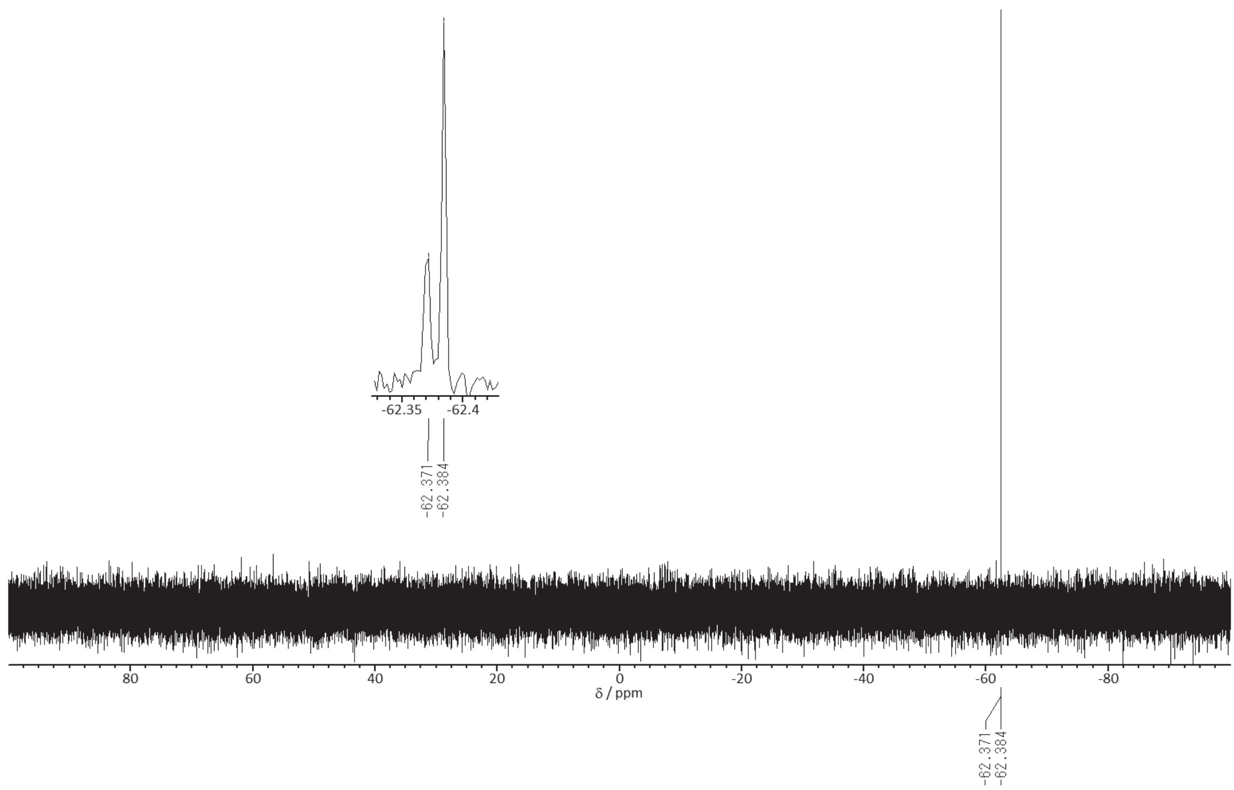
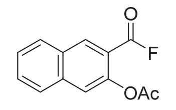
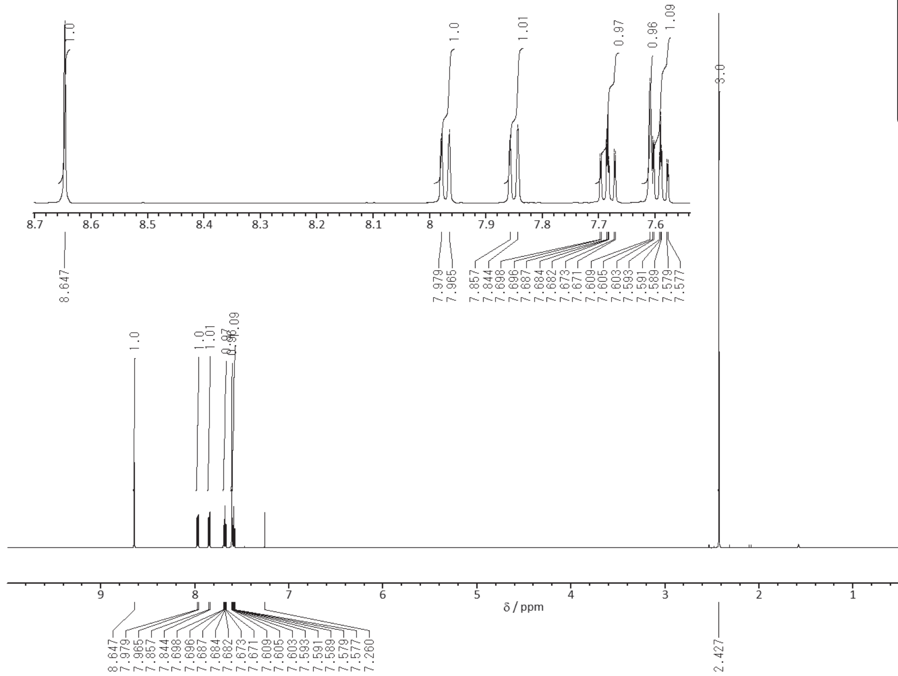


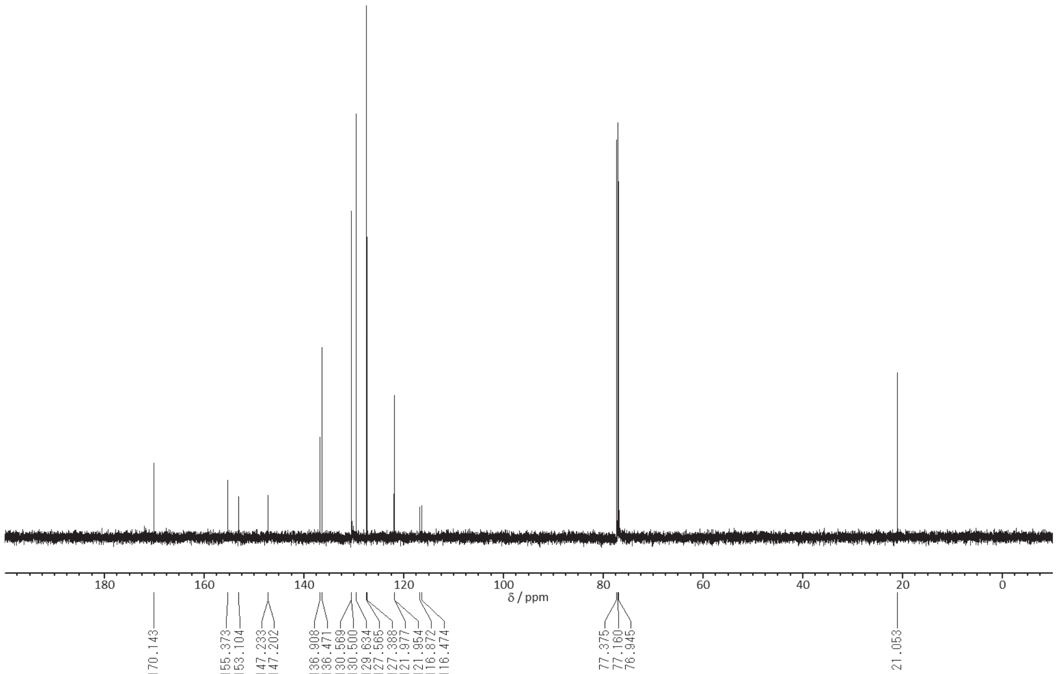
Figure S21. ^{1}H, ^{13}C{^{1}H} and ^{19}F{^{1}H} NMR spectra of mixture of **8d** and **8d'** (in $CDCl_3$)



11



1.0
1.0
1.01
0.97
0.96
1.09
3.0
8.647
7.979
7.965
7.857
7.844
7.698
7.696
7.687
7.684
7.682
7.673
7.671
7.609
7.605
7.603
7.593
7.591
7.589
7.579
7.577
7.260
2.427
δ / ppm



170.143
155.373
153.104
147.233
147.202
136.908
136.471
130.569
130.500
129.634
127.565
127.388
121.977
121.954
116.872
116.474
77.375
77.160
76.945
21.053
δ / ppm

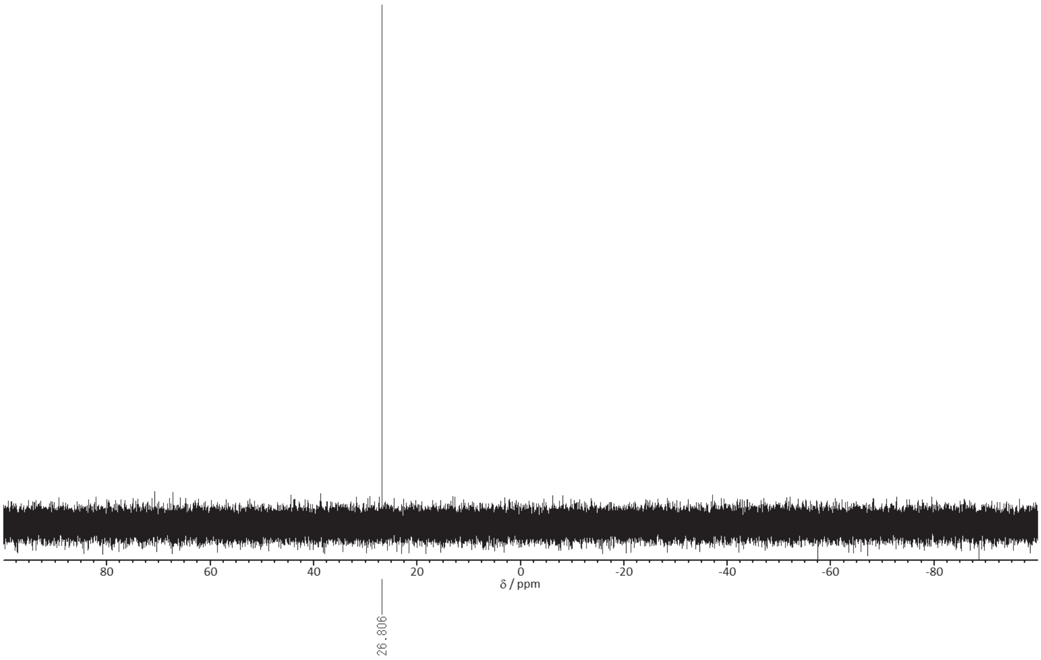


Figure S22. ^{1}H, $^{13}C\{^{1}H\}$ and $^{19}F\{^{1}H\}$ NMR spectra of **11** (in $CDCl_3$)

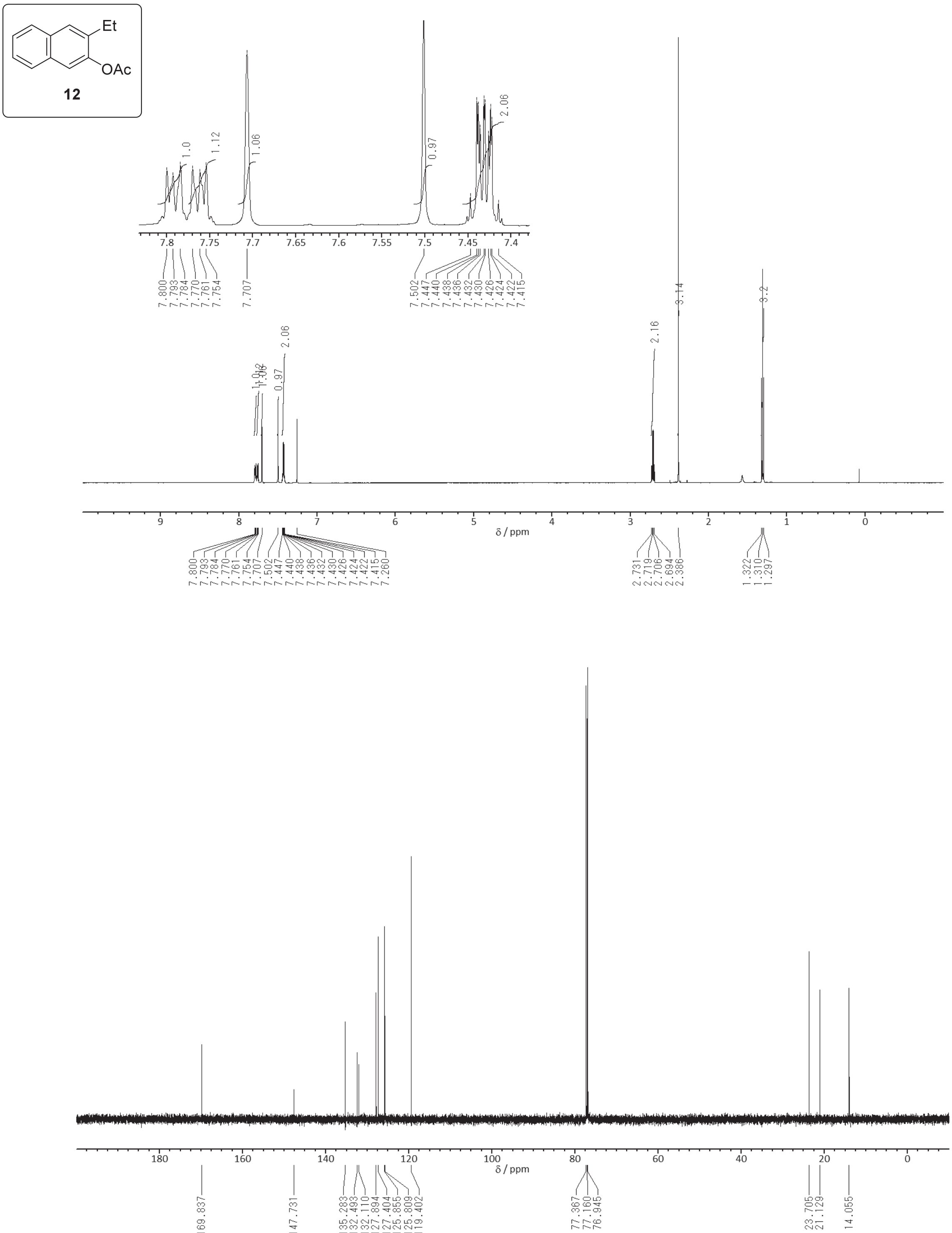


Figure S23. ^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of **12** (in $CDCl_3$)

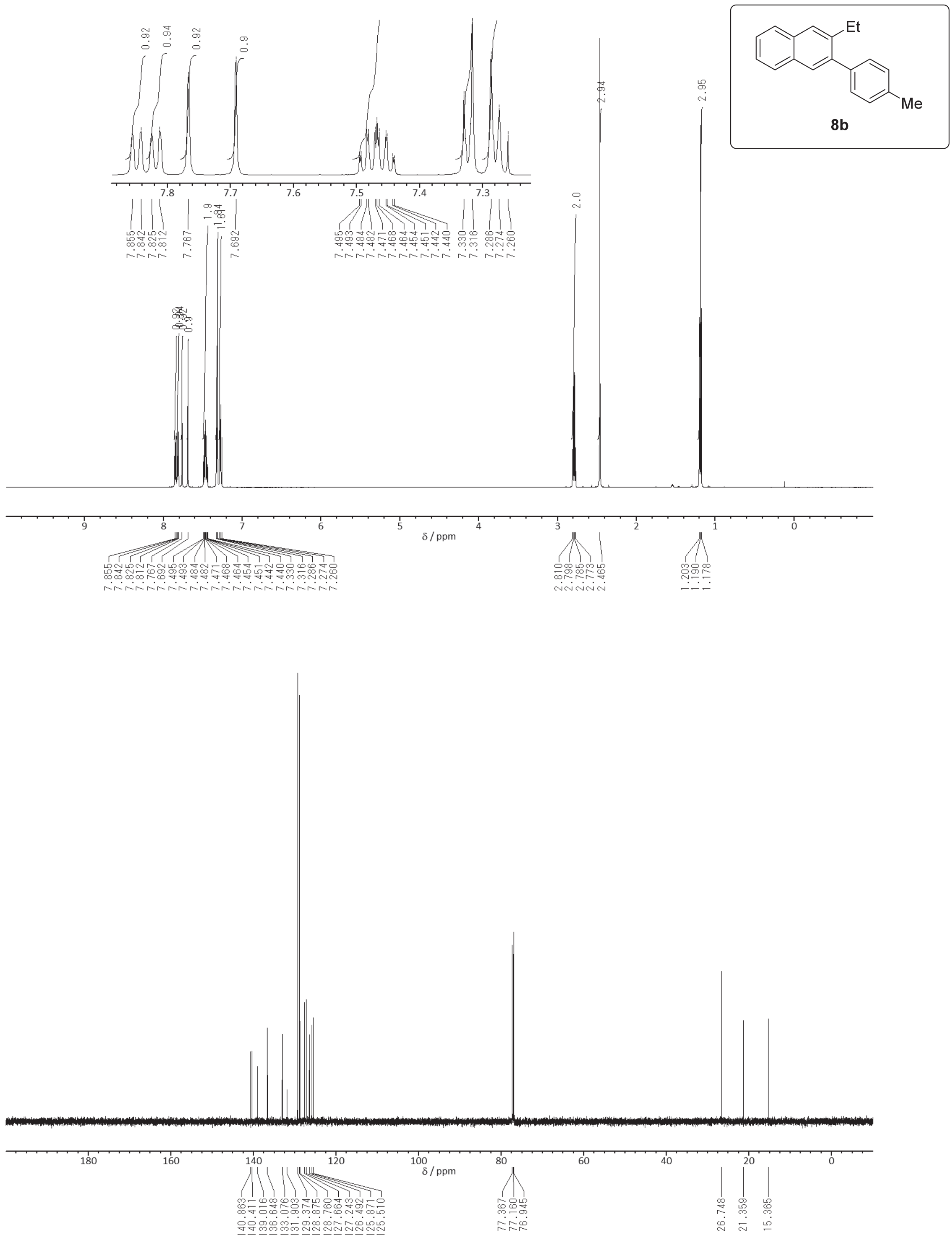
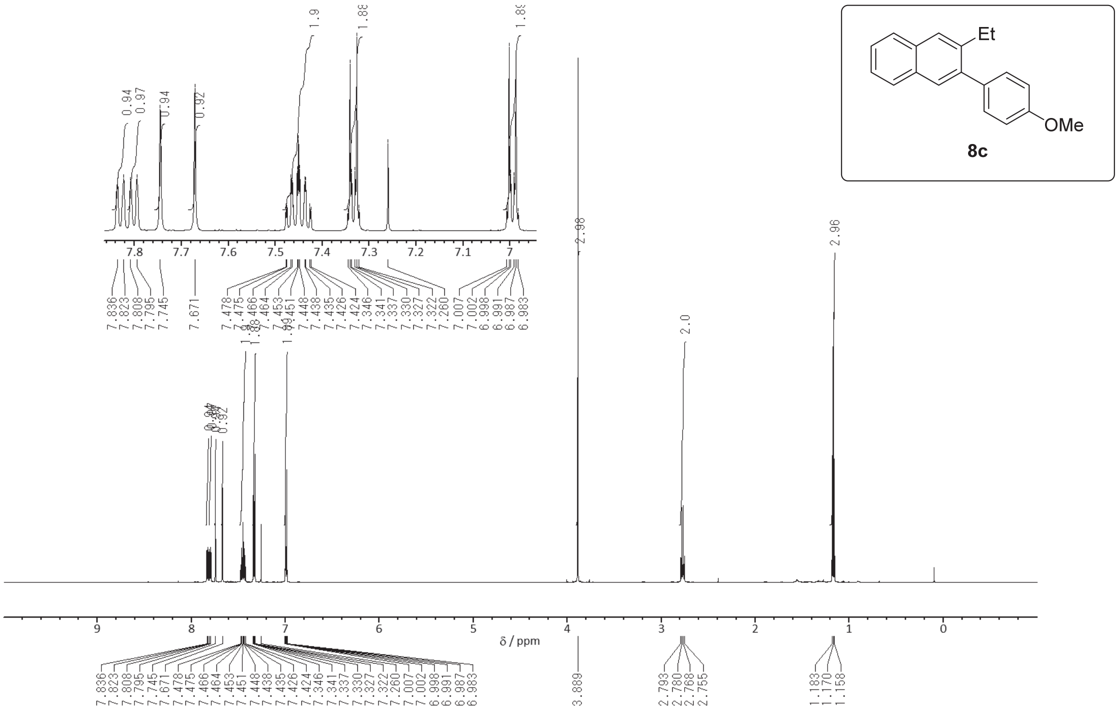


Figure S24. ^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of **8b** (in $CDCl_3$)



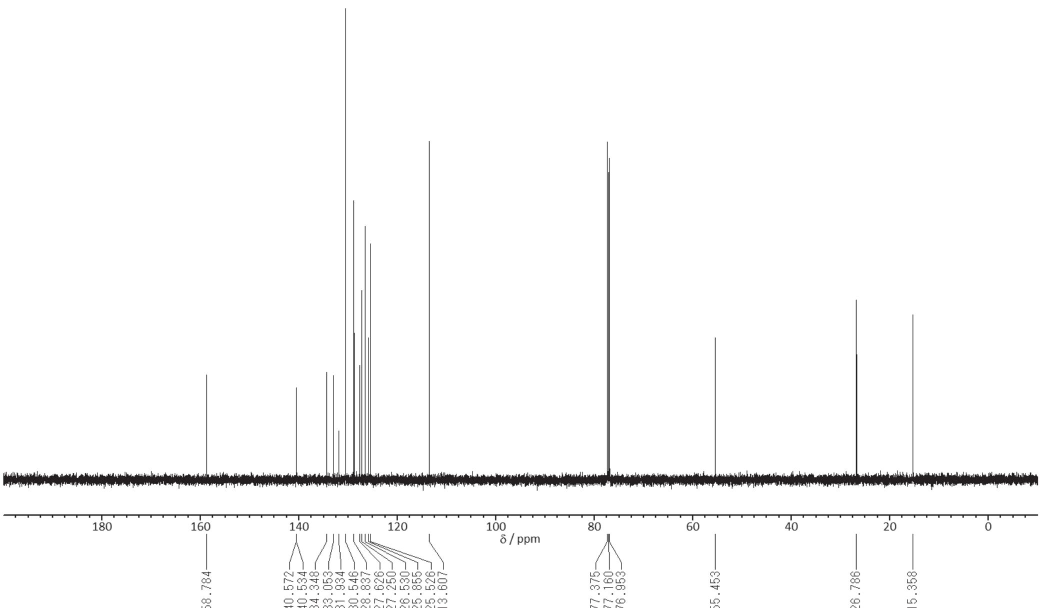
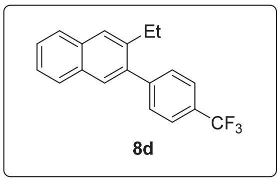
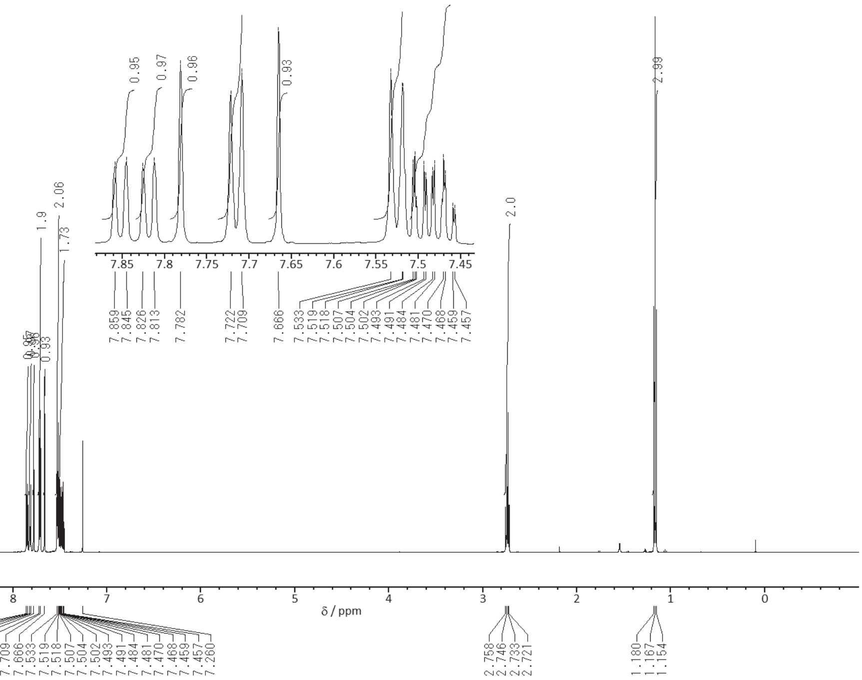
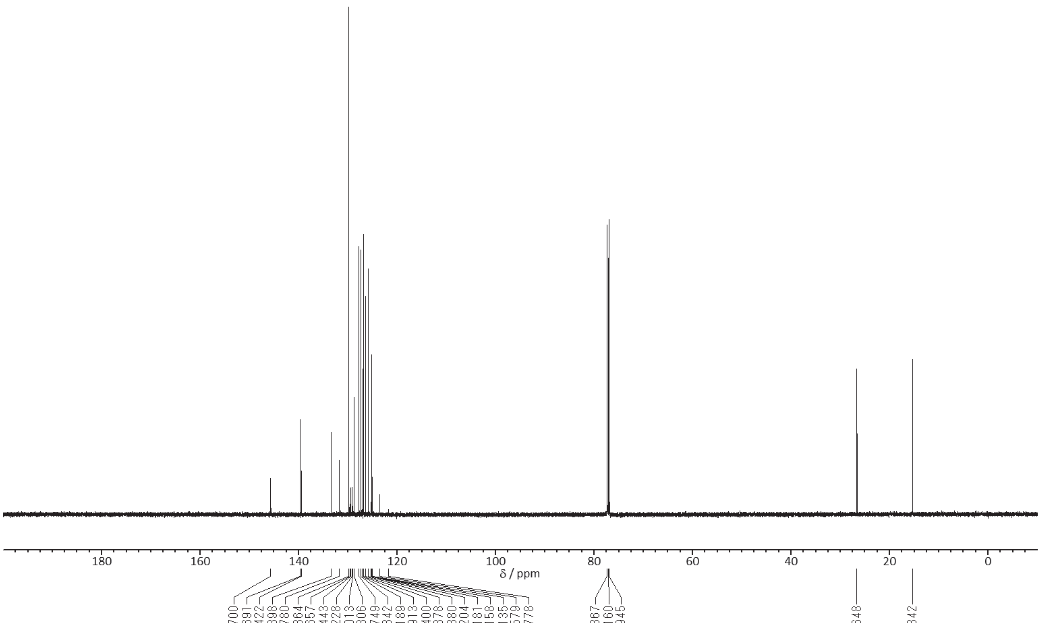


Figure S25. ^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of **8c** (in $CDCl_3$)


Et
CF3
8d





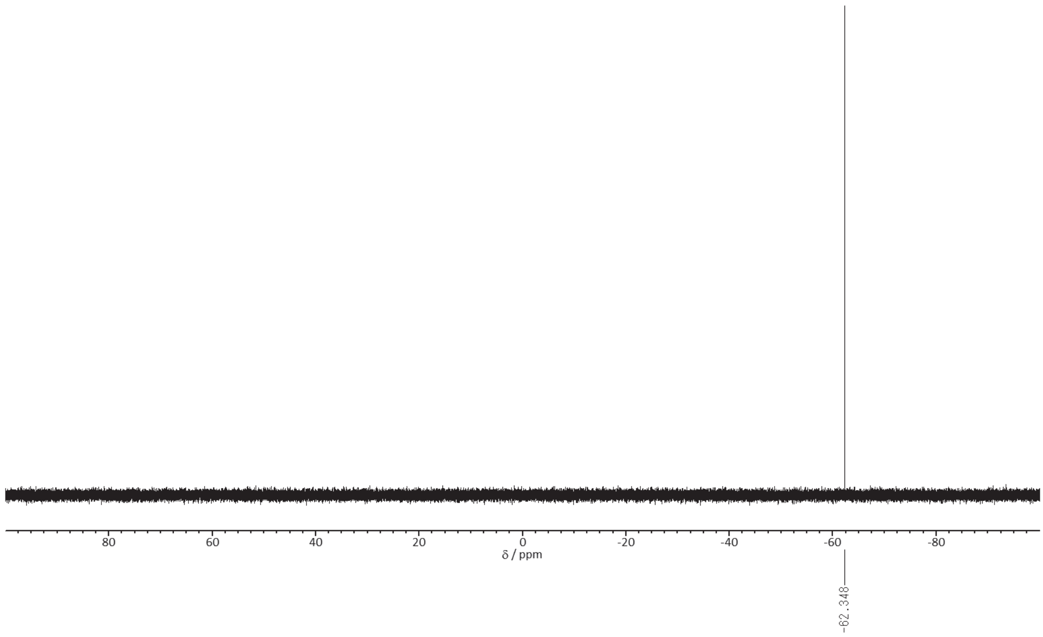


Figure S26. ^1H, $^{13}C\{^1H\}$ and $^{19}F\{^1H\}$ NMR spectra of **8d** (in $CDCl_3$)

4. *Copies of ¹H NMR Charts for the Known Compounds*

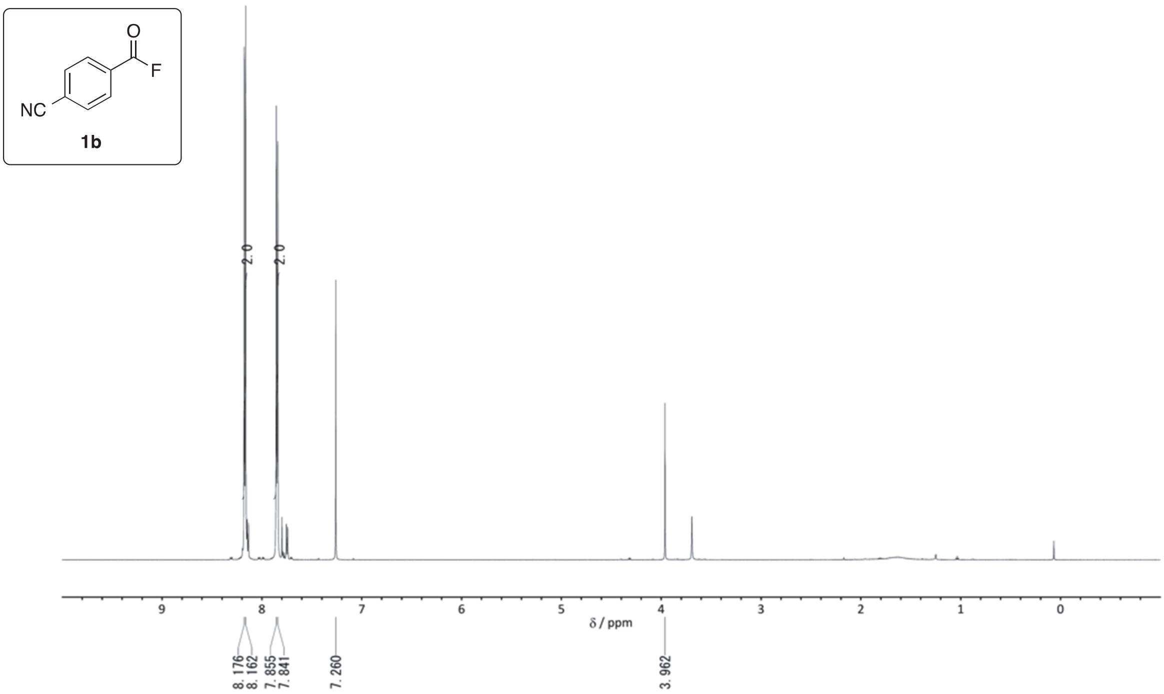


Figure S27. ¹H NMR spectra of **1b** (in $CDCl_3$)

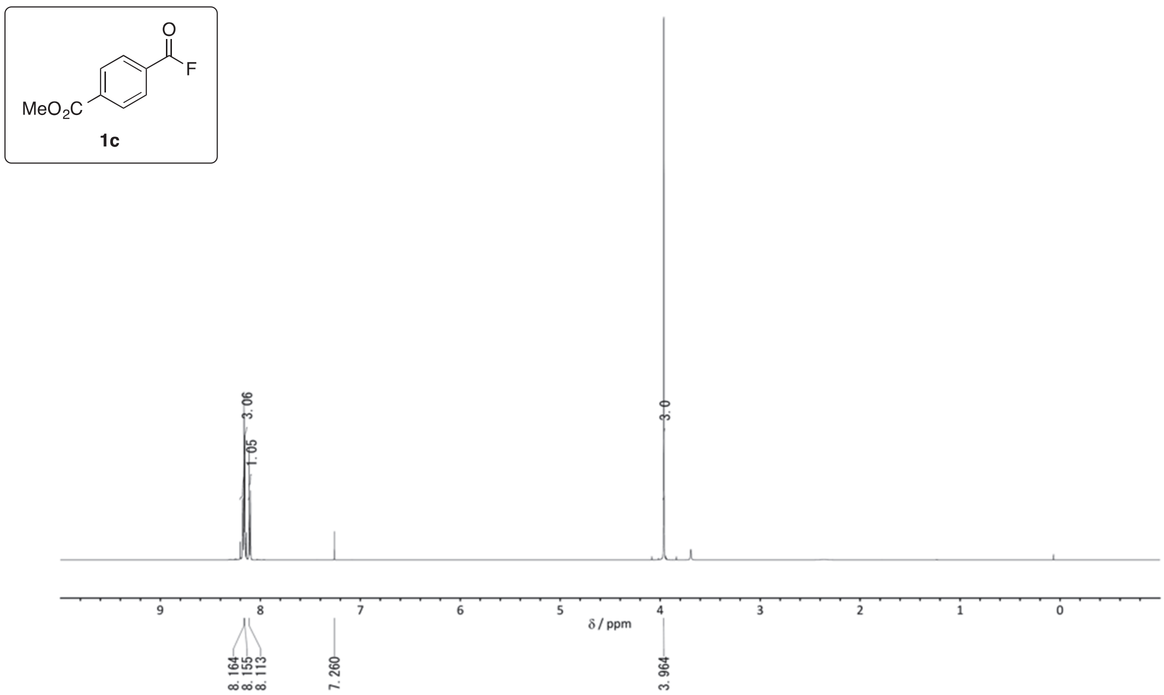


Figure S28. ¹H NMR spectra of **1c** (in $CDCl_3$)

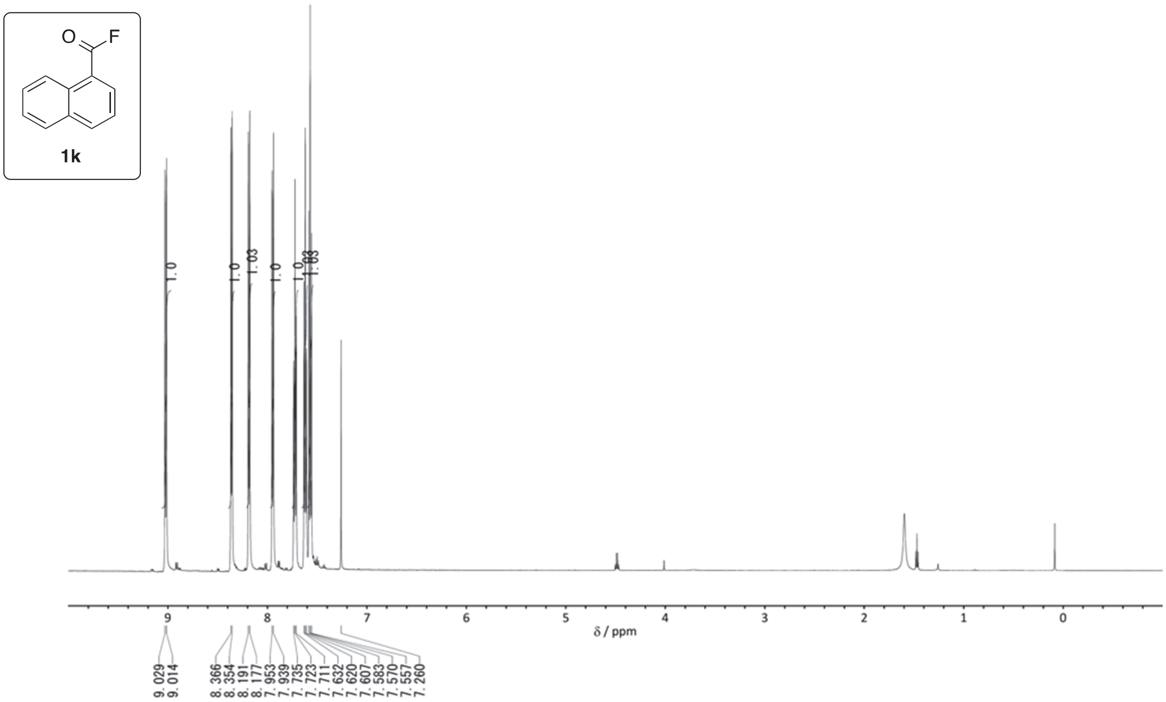


Figure S29. ^{1}H NMR spectra of **1k** (in $CDCl_3$)

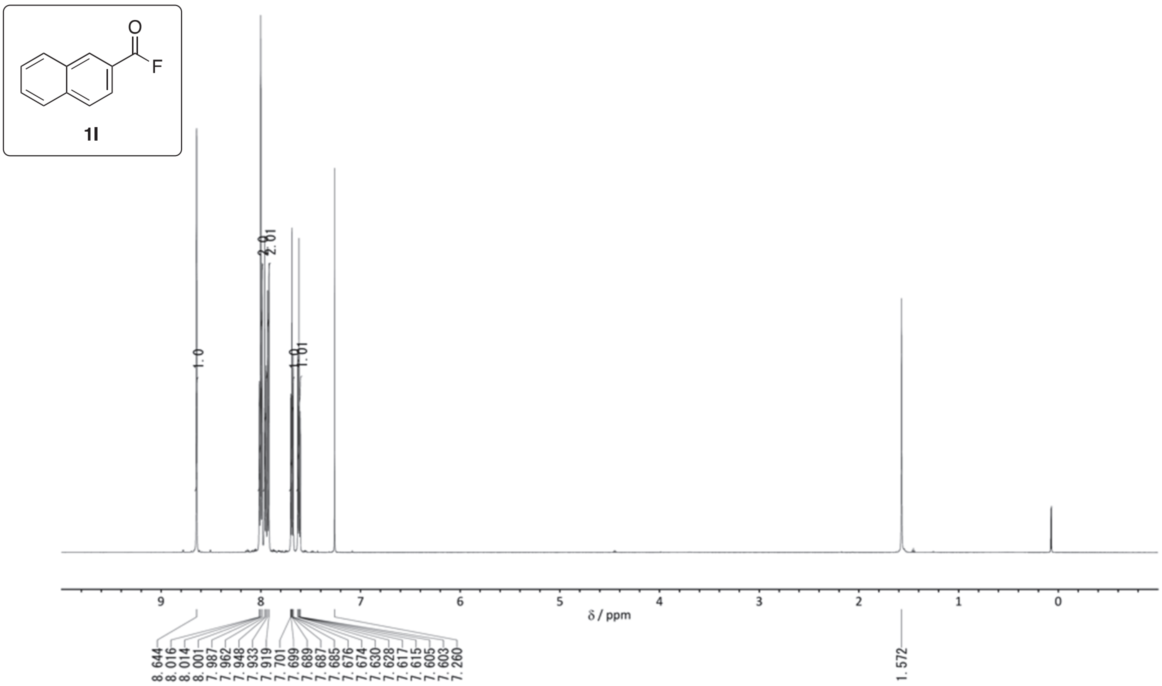
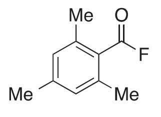


Figure S30. ^{1}H NMR spectra of **1l** (in $CDCl_3$)



1o

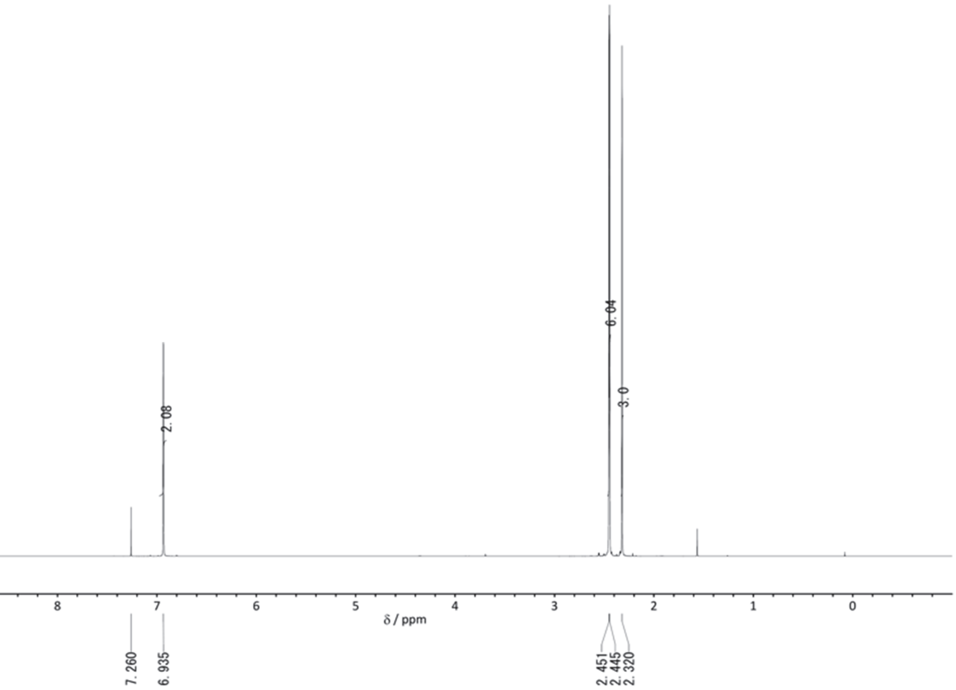


Figure S31. ^{1}H NMR spectra of **1o** (in $CDCl_3$)

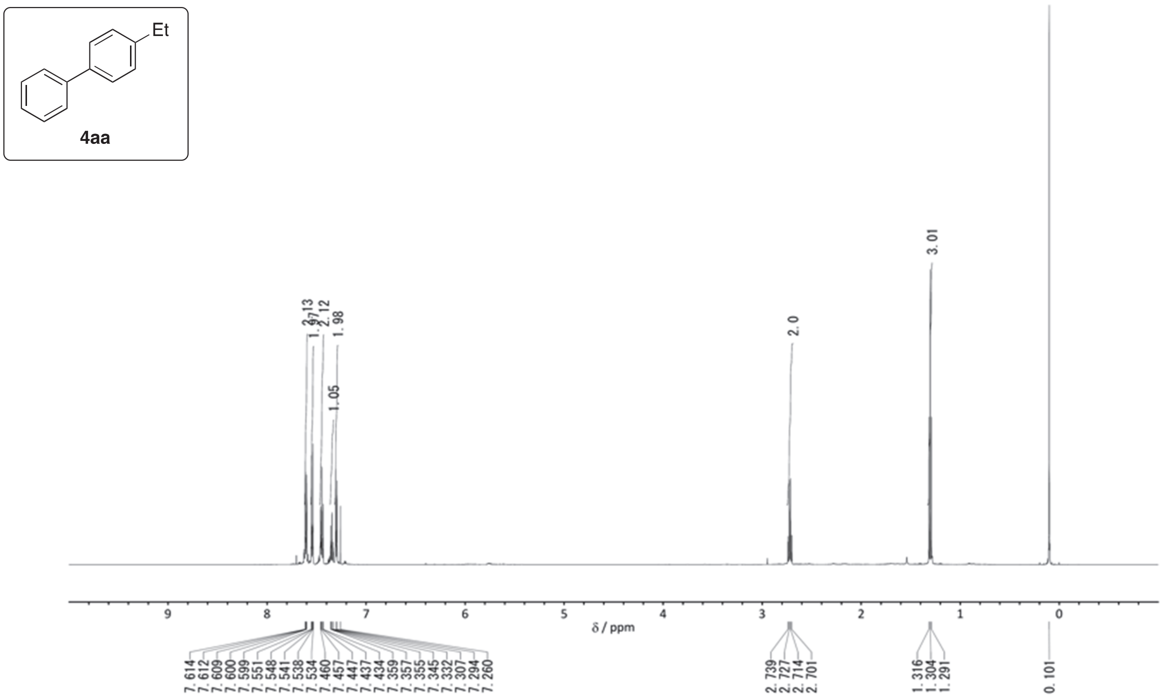


Figure S32. ^{1}H NMR spectra of **4aa** (in $CDCl_3$)

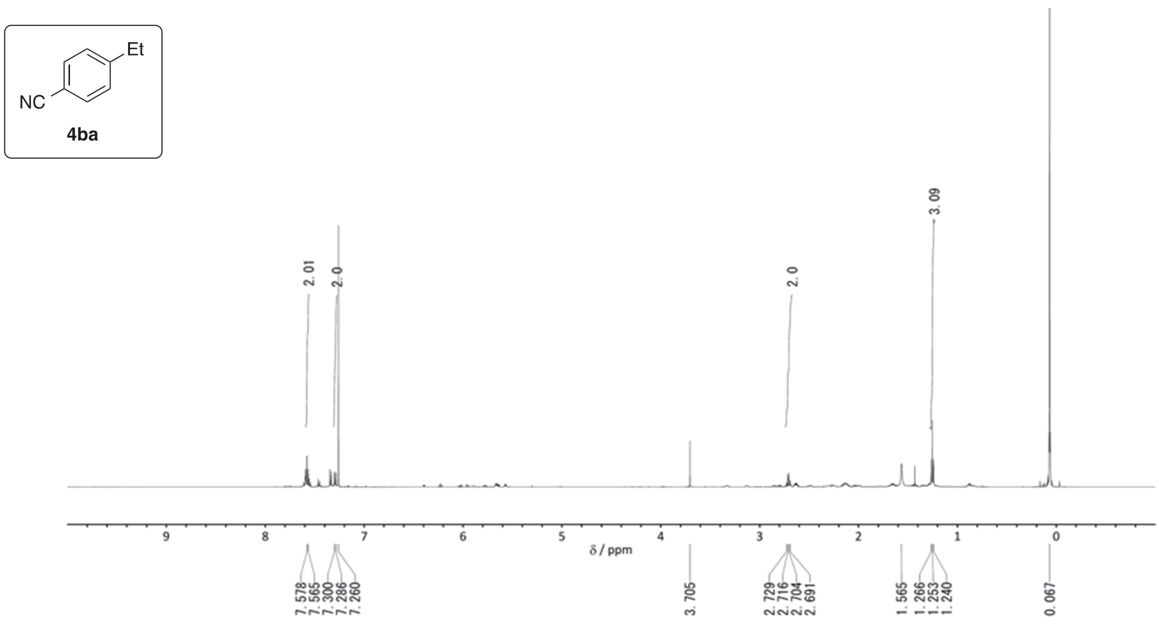


Figure S33. ^{1}H NMR spectra of **4ba** (in $CDCl_3$)

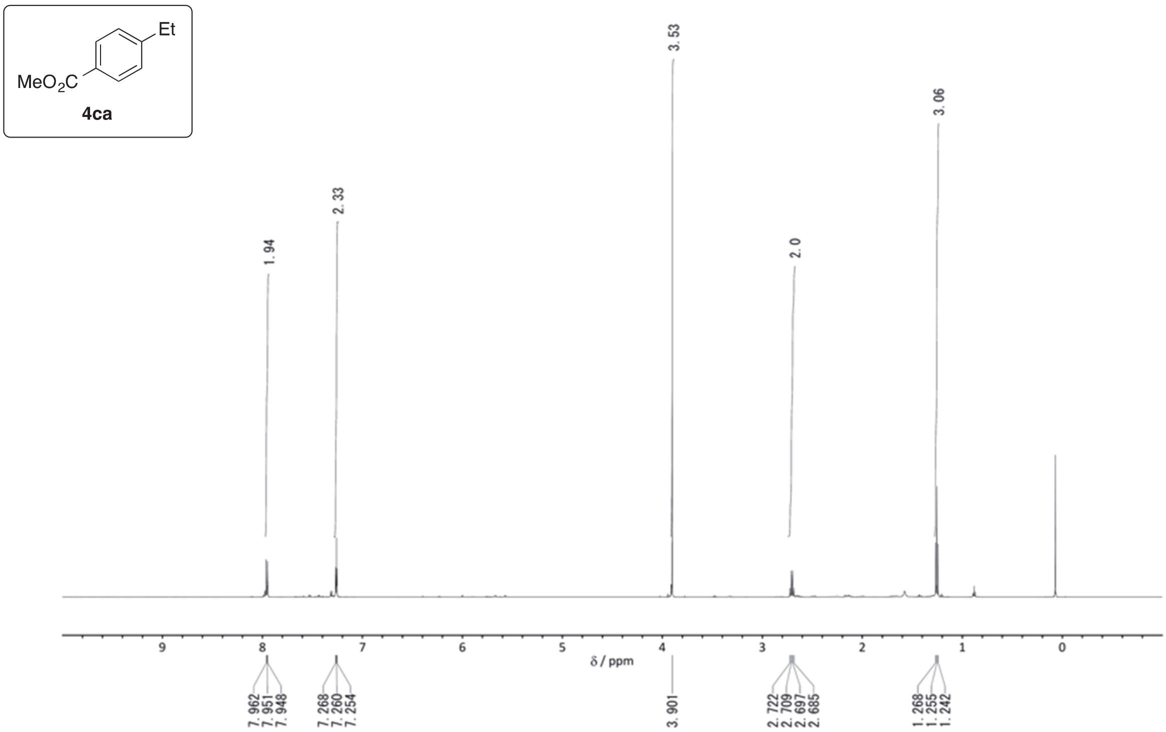


Figure S34. ^{1}H NMR spectra of **4ca** (in $CDCl_3$)

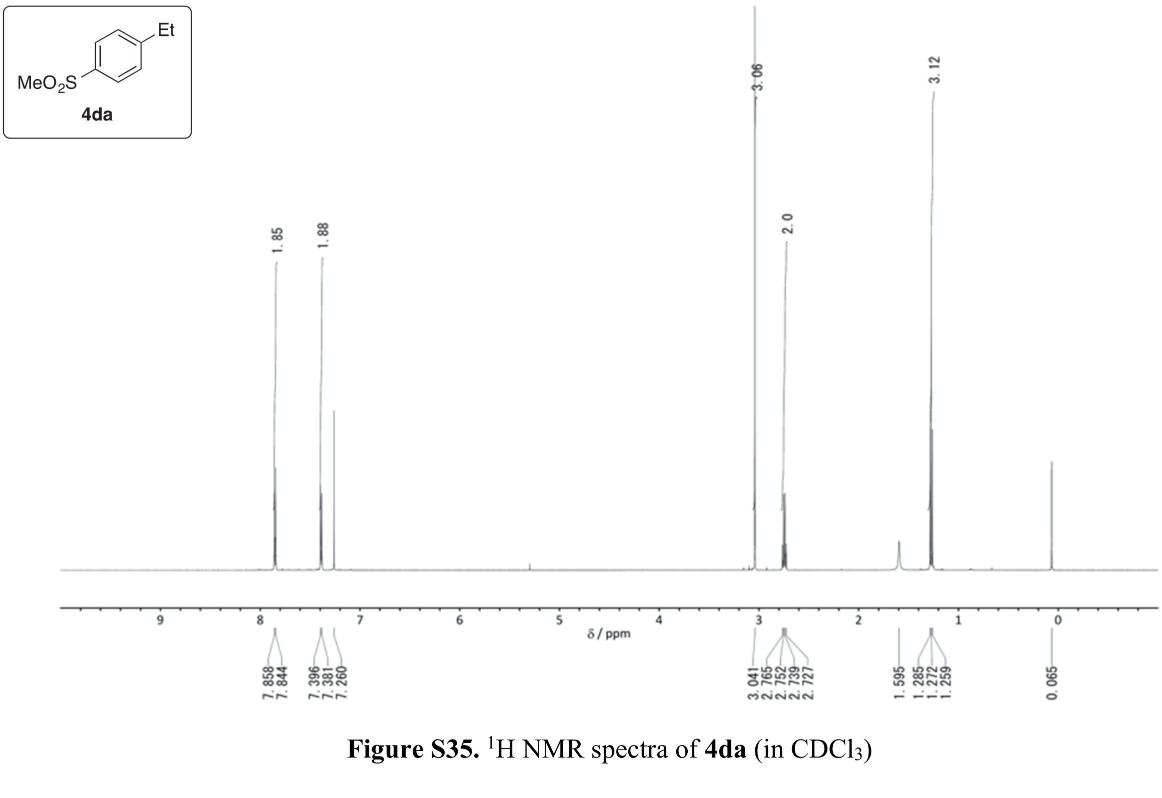


Figure S35. ^{1}H NMR spectra of **4da** (in $CDCl_3$)

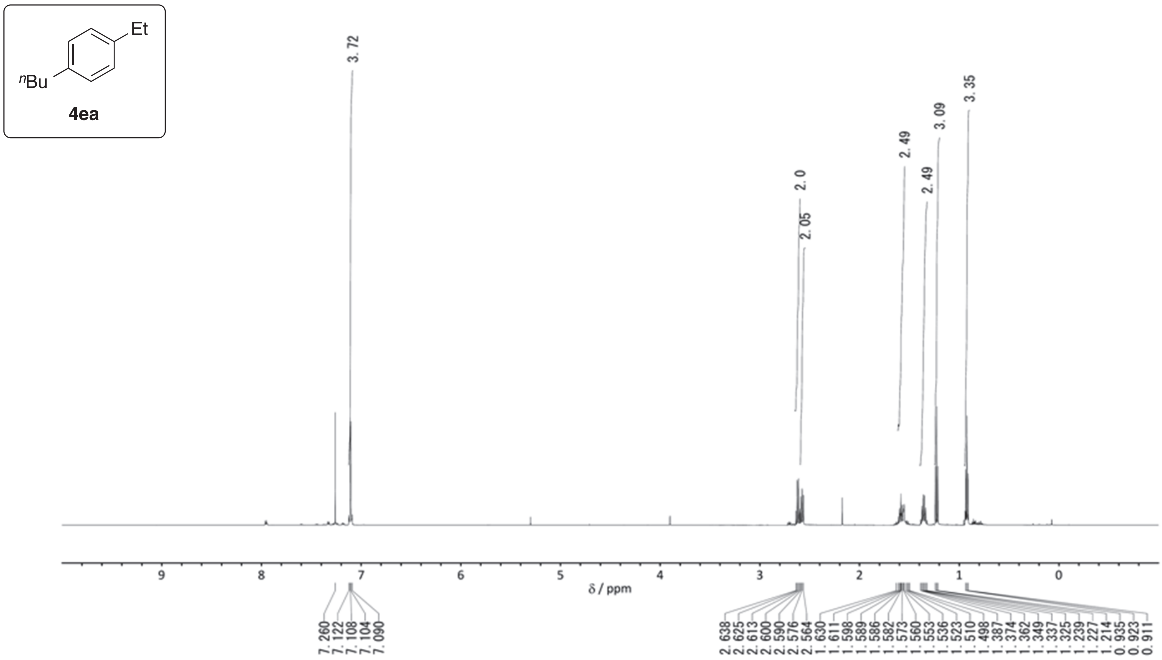


Figure S36. ^{1}H NMR spectra of **4ea** (in $CDCl_3$)

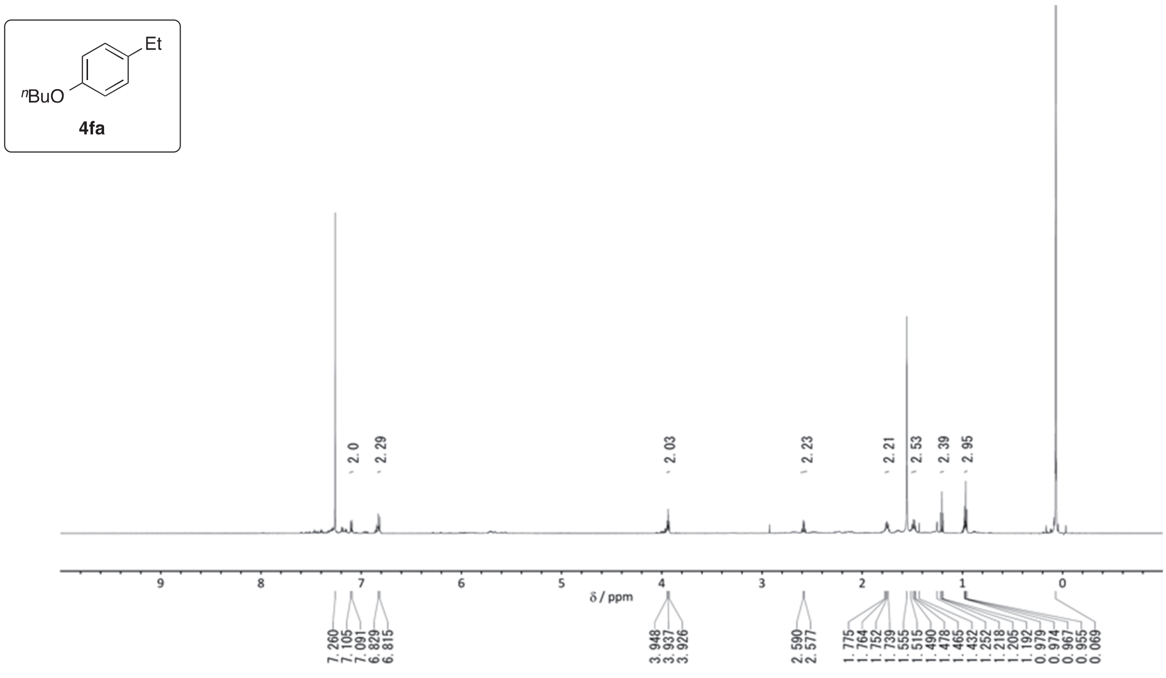


Figure S37. ^{1}H NMR spectra of **4fa** (in $CDCl_3$)

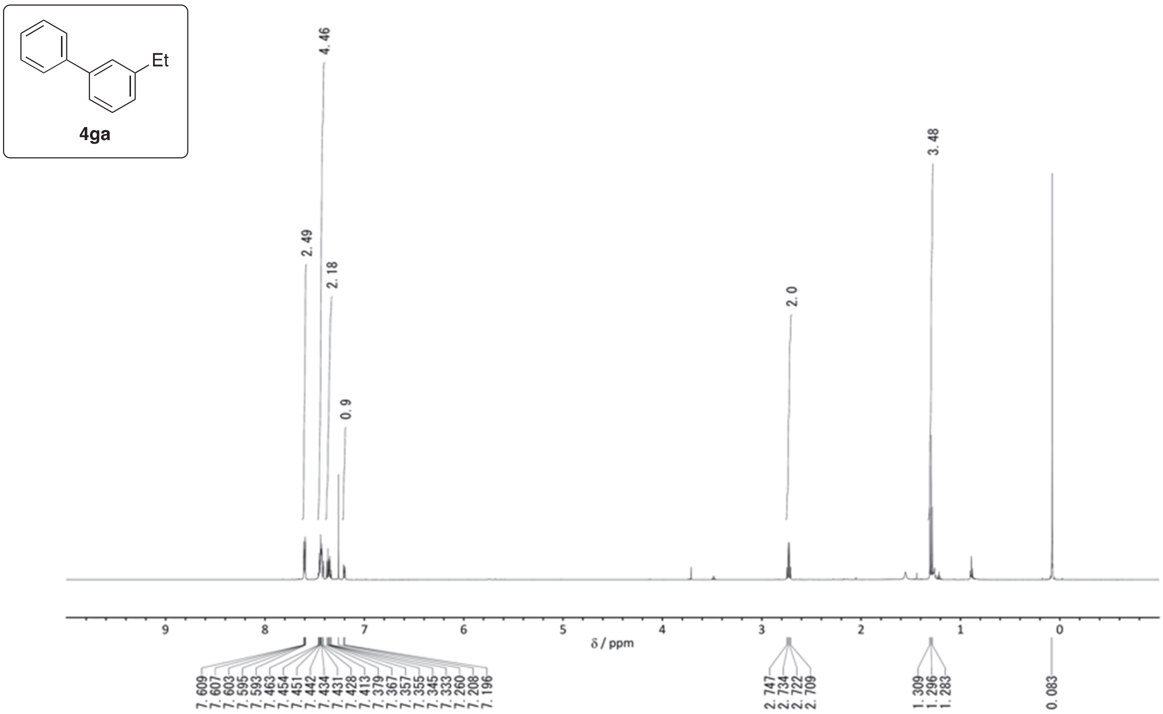


Figure S38. ^{1}H NMR spectra of **4ga** (in $CDCl_3$)

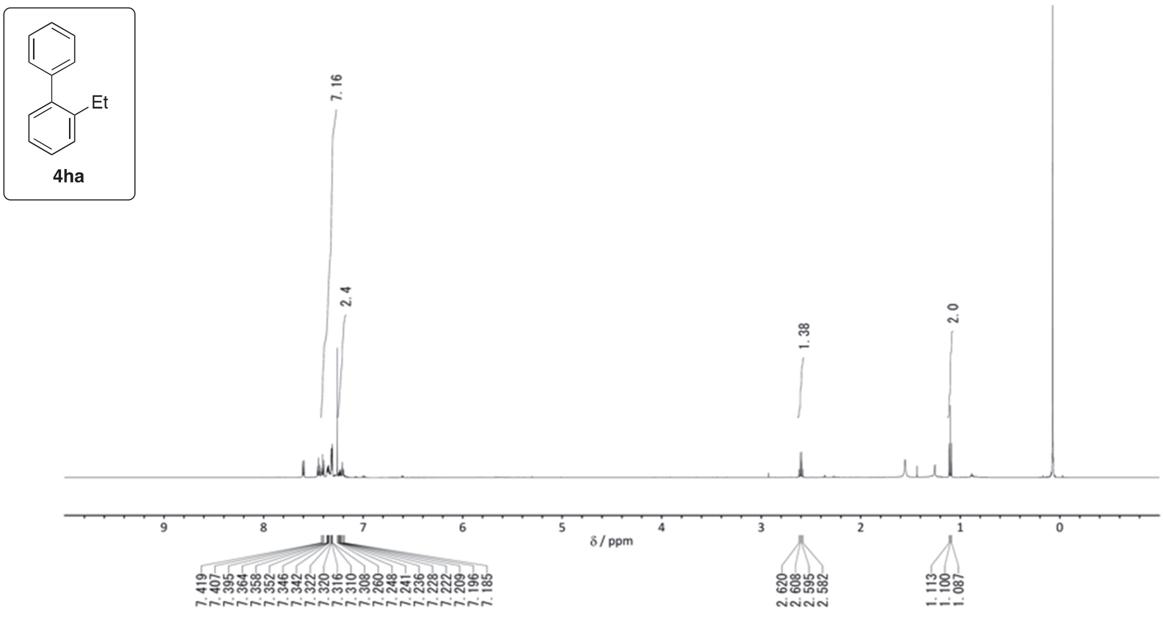


Figure S39. ^{1}H NMR spectra of **4ha** (in $CDCl_3$)

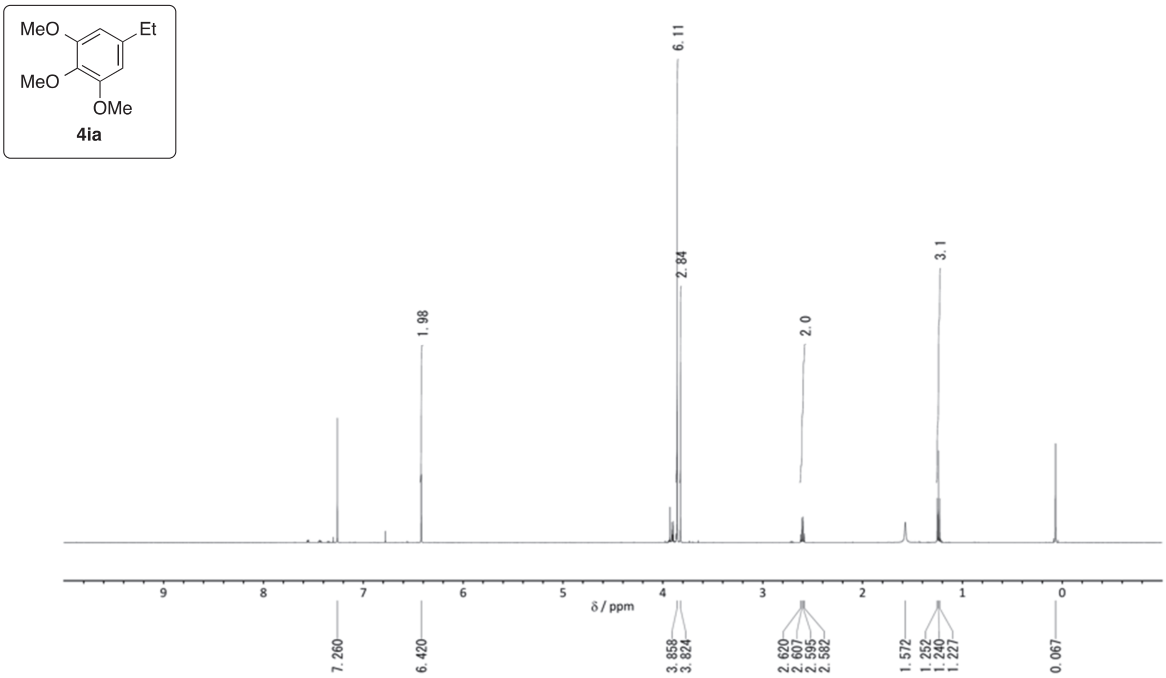


Figure S40. ^{1}H NMR spectra of **4ia** (in $CDCl_3$)

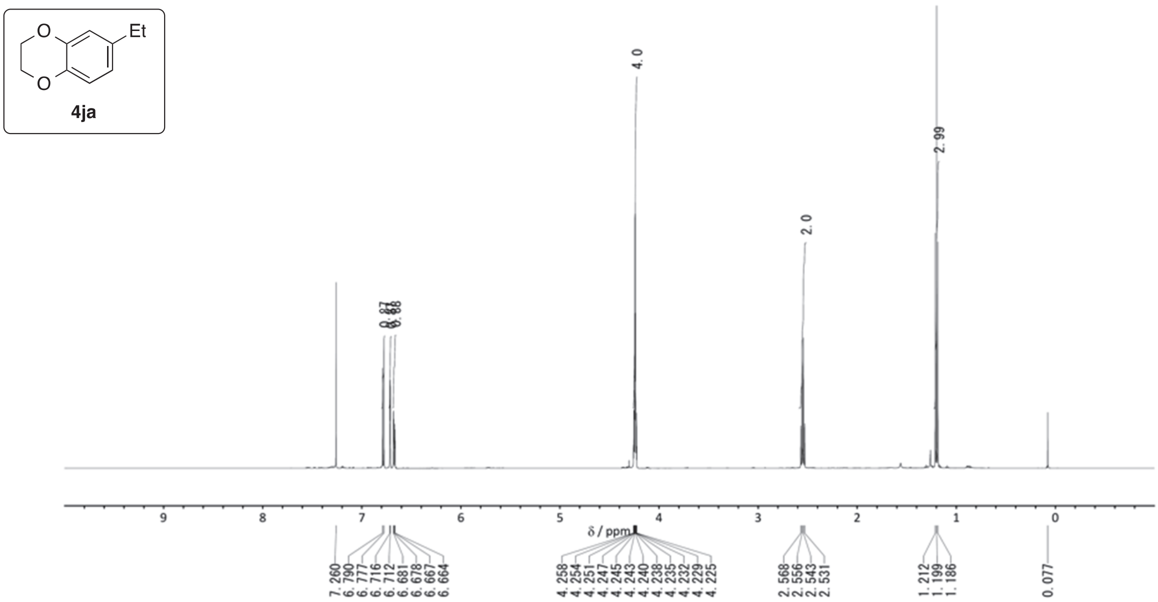


Figure S41. ^{1}H NMR spectra of **4ja** (in $CDCl_3$)

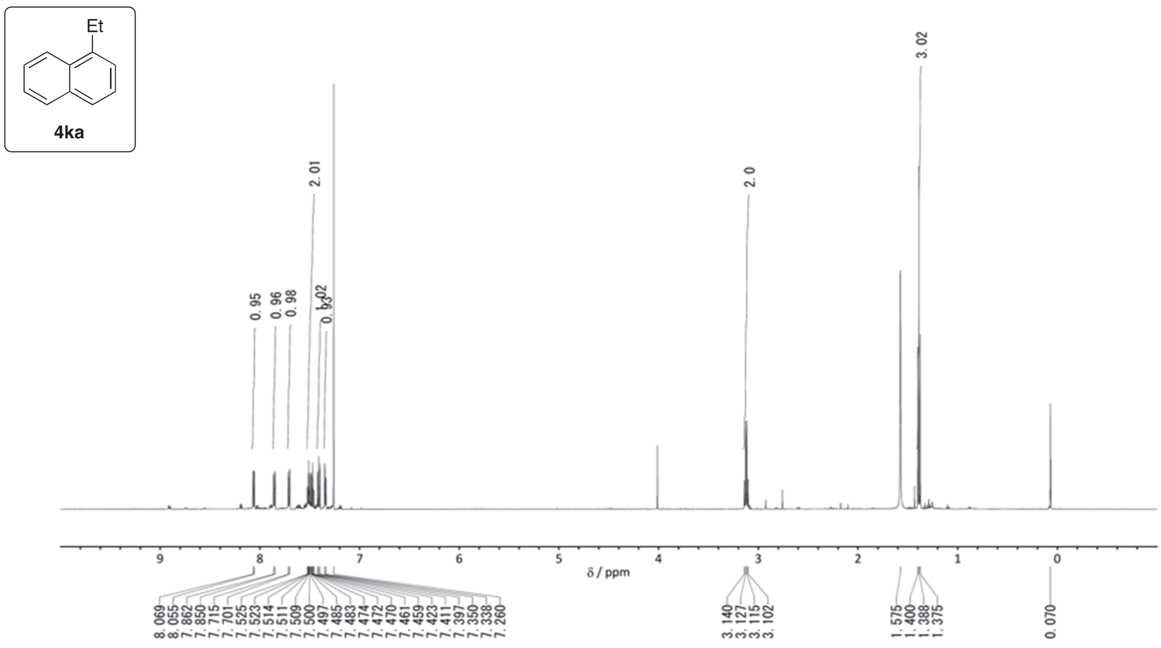


Figure S42. ^{1}H NMR spectra of **4ka** (in $CDCl_3$)

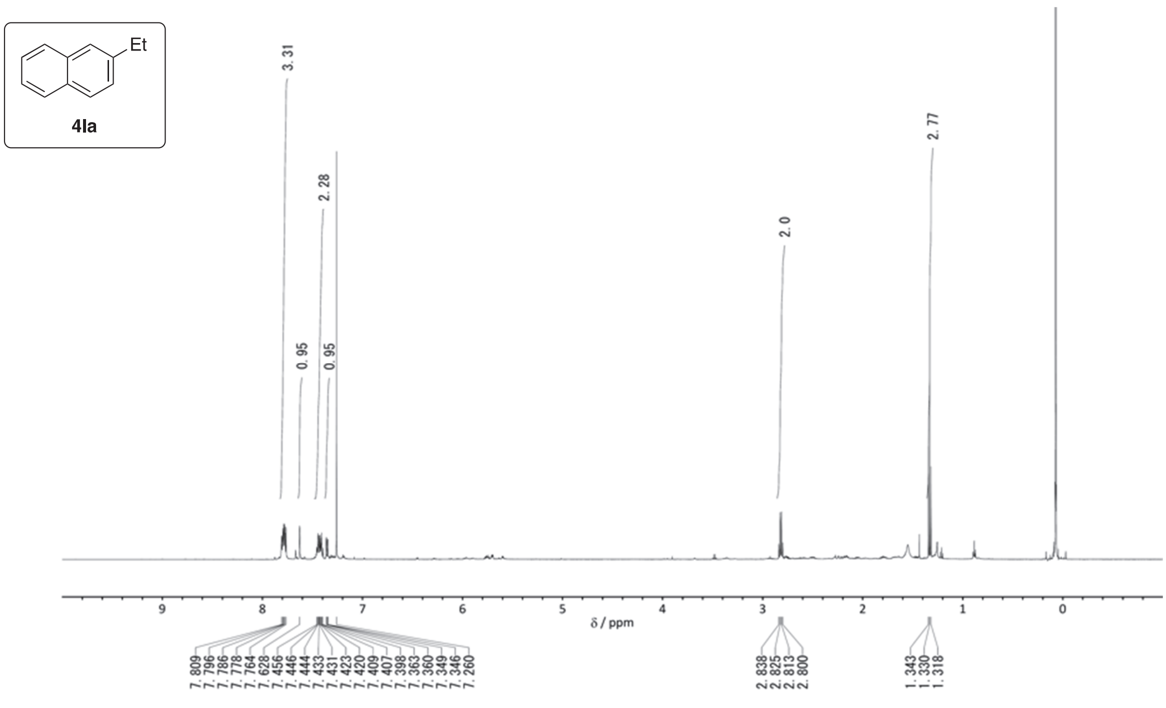


Figure S43. ^{1}H NMR spectra of **4la** (in $CDCl_3$)

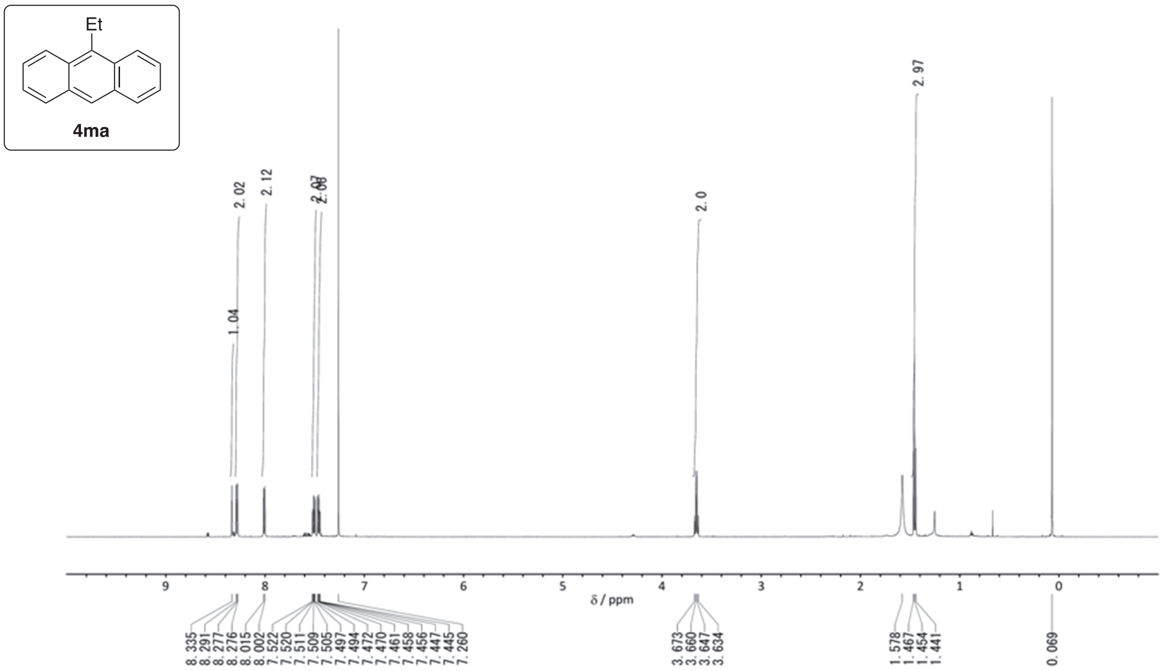


Figure S44. ^{1}H NMR spectra of **4ma** (in $CDCl_3$)

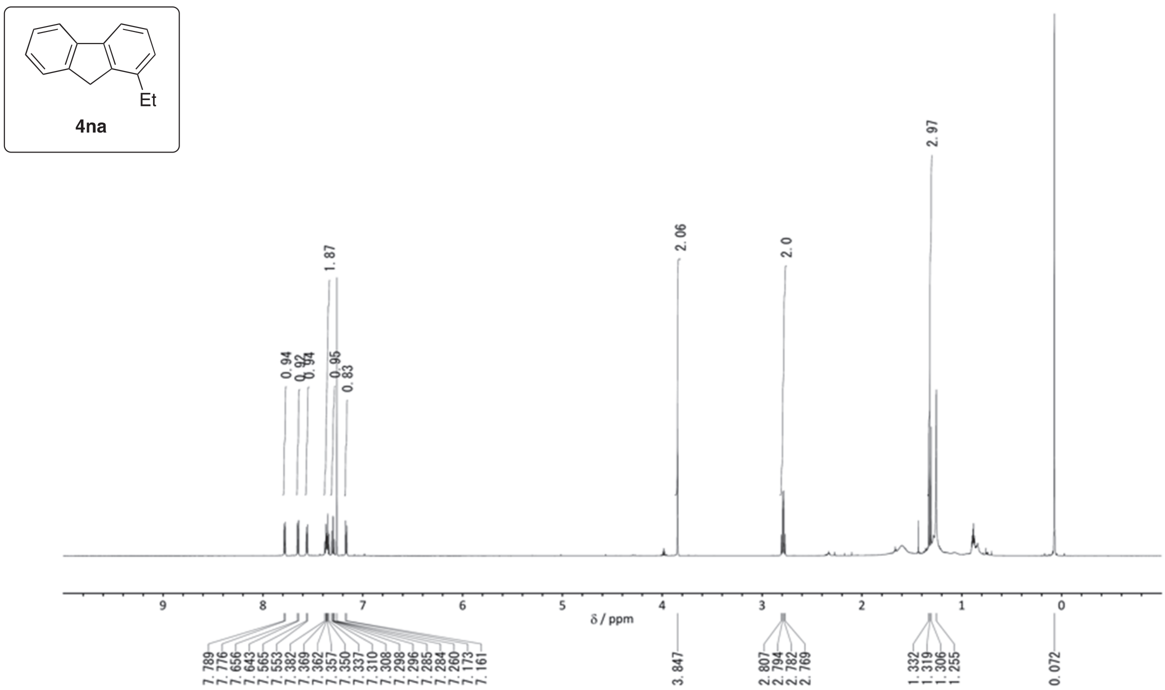


Figure S45. ^{1}H NMR spectra of **4na** (in $CDCl_3$)

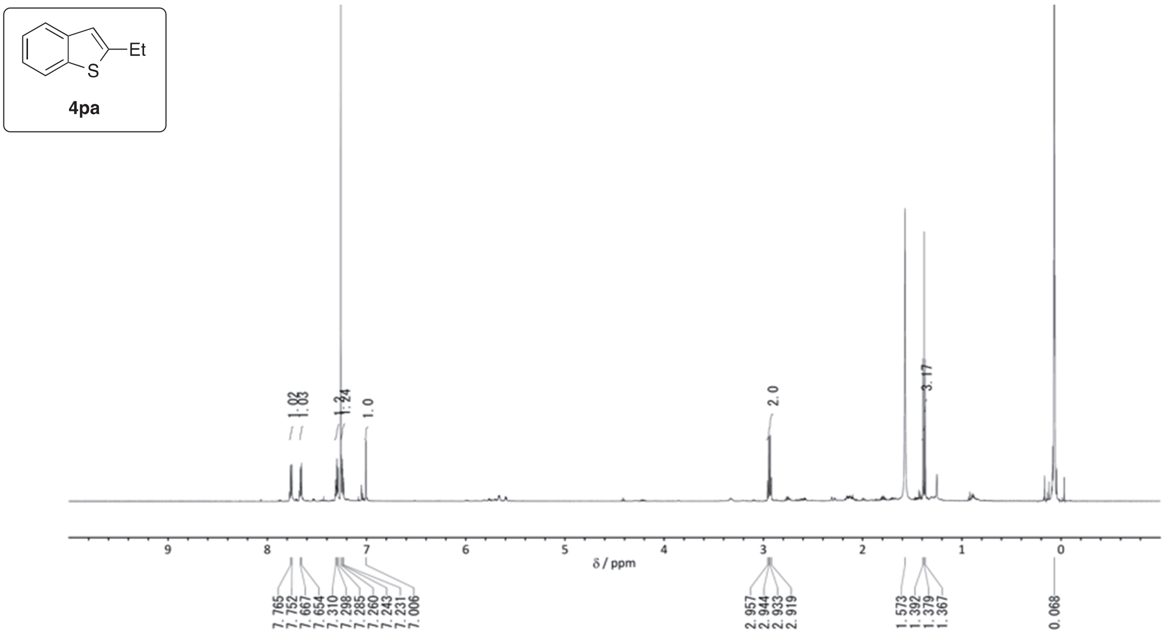


Figure S46. ^{1}H NMR spectra of **4pa** (in $CDCl_3$)

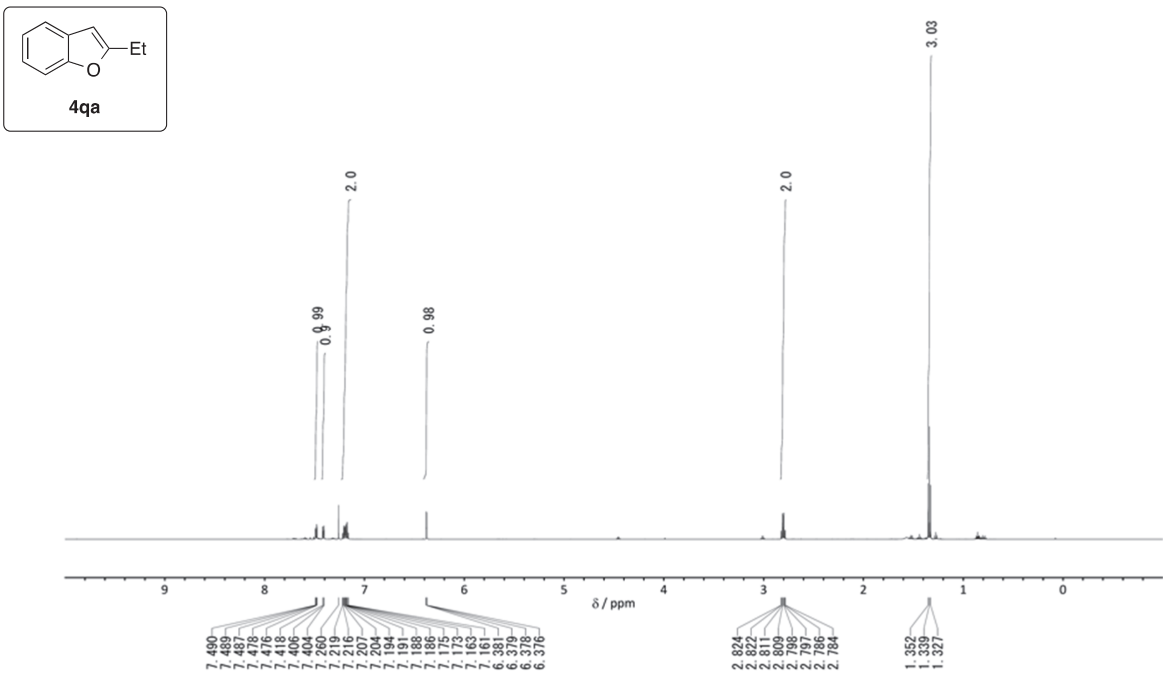


Figure S47. ^{1}H NMR spectra of **4qa** (in $CDCl_3$)

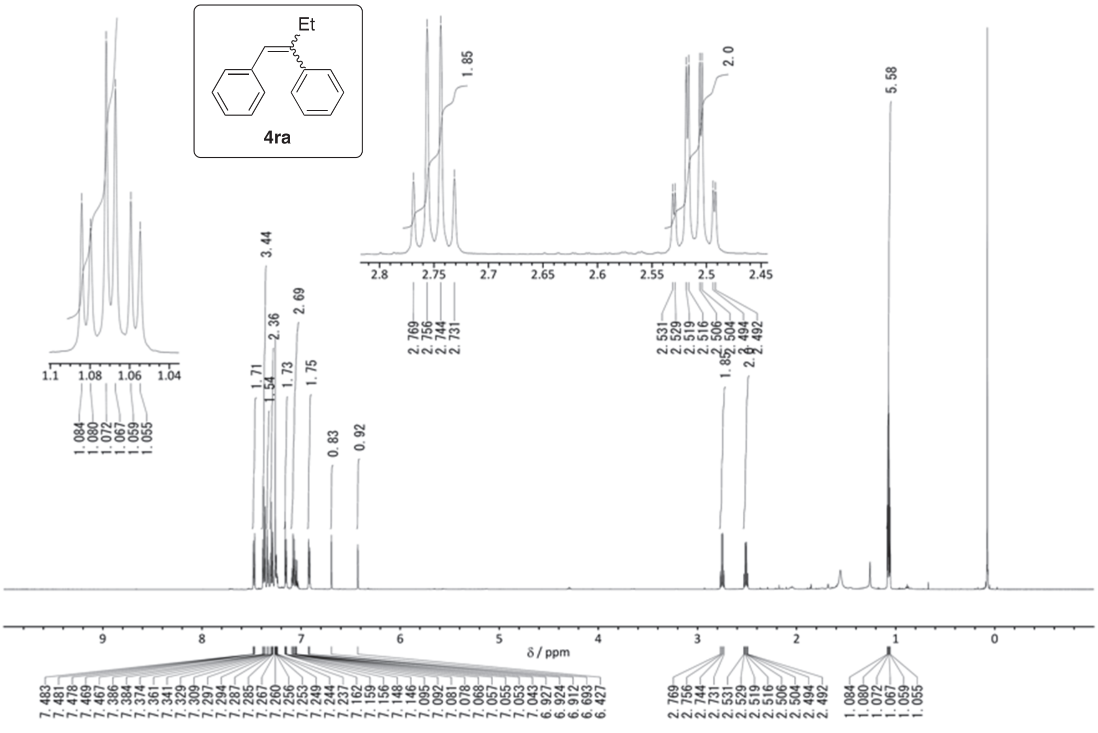


Figure S48. ^{1}H NMR spectra of **4ra** (in $CDCl_3$)

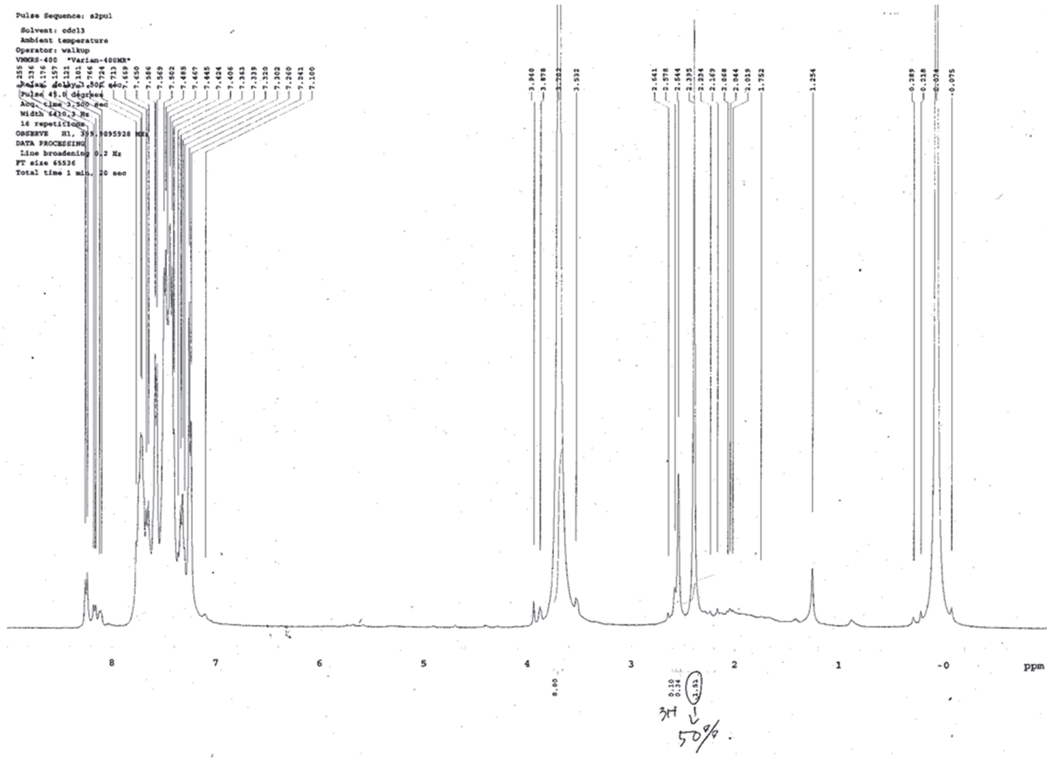


Figure S49. Calculation of NMR yield for the crude mixture of **4ab** (in $CDCl_3$)

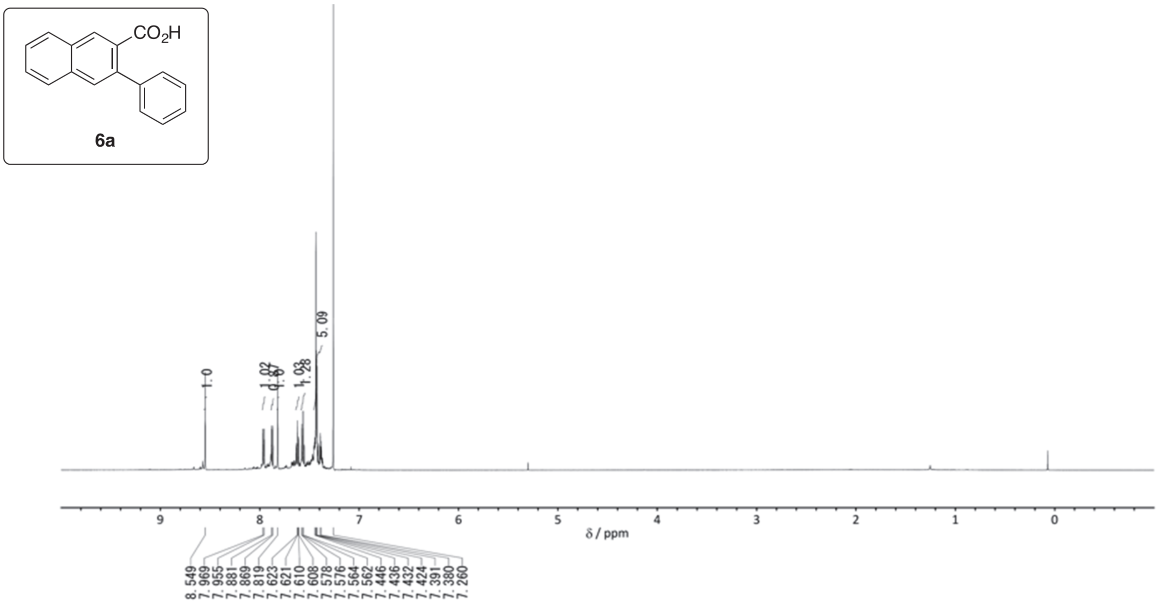


Figure S50. ^{1}H NMR spectra of **6a** (in $CDCl_3$)

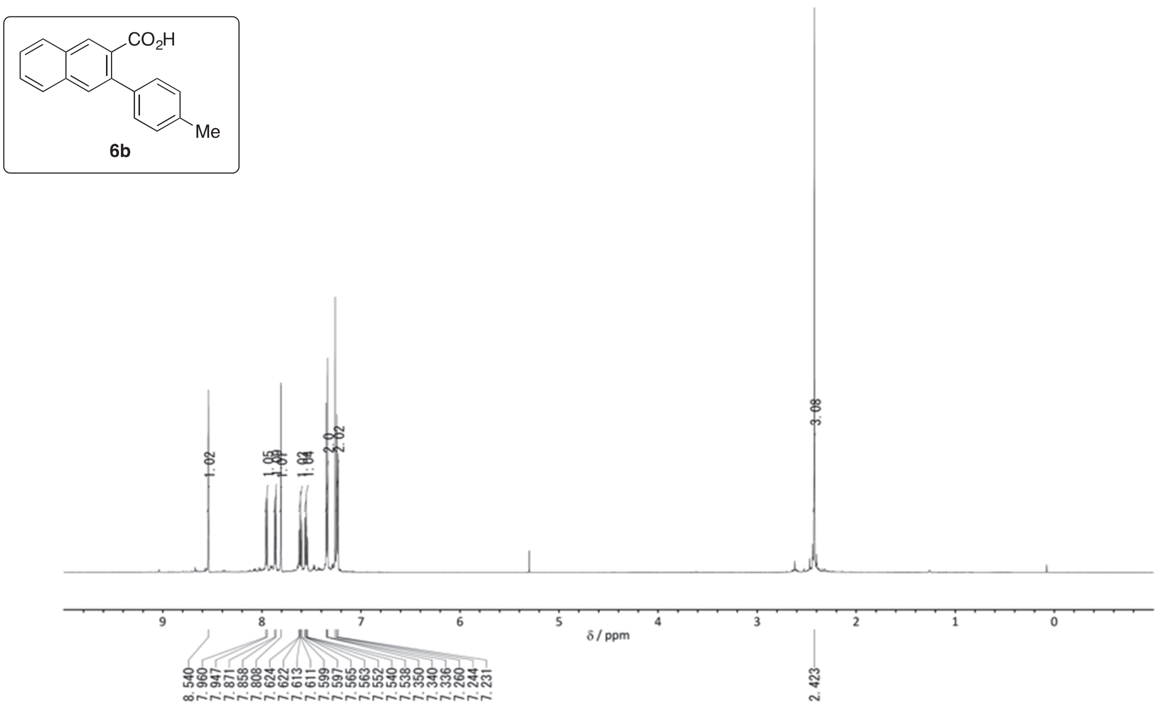


Figure S51. ^{1}H NMR spectra of **6b** (in $CDCl_3$)

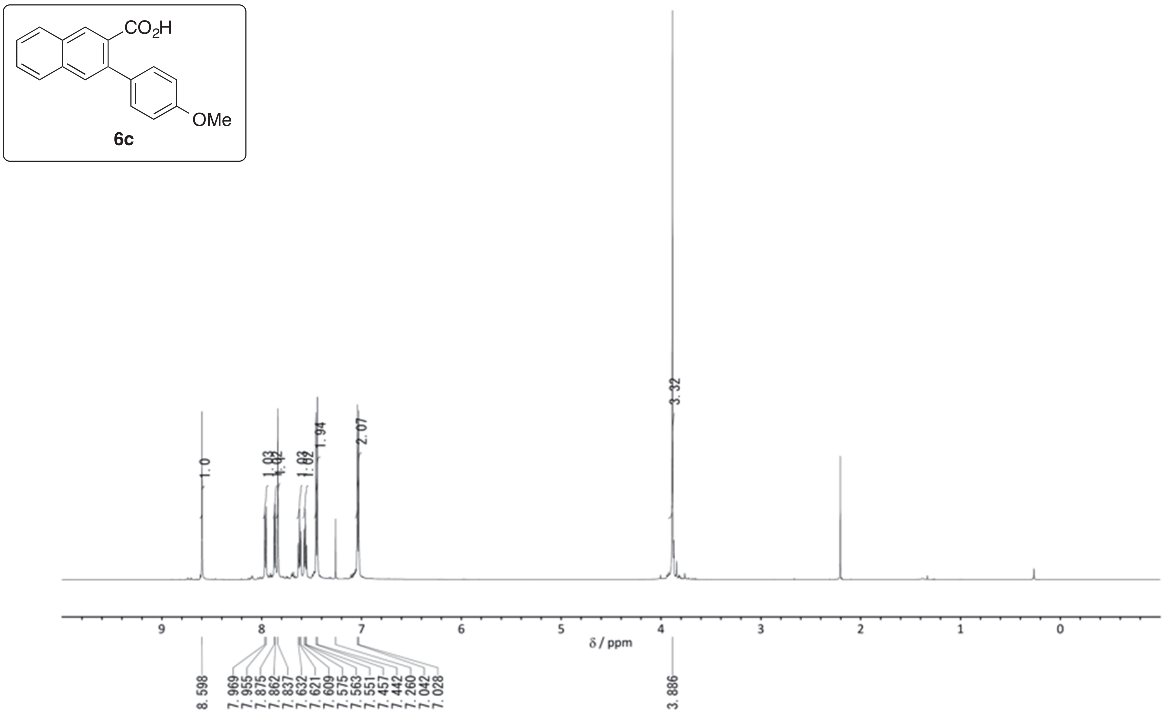


Figure S52. ^{1}H NMR spectra of **6c** (in $CDCl_3$)

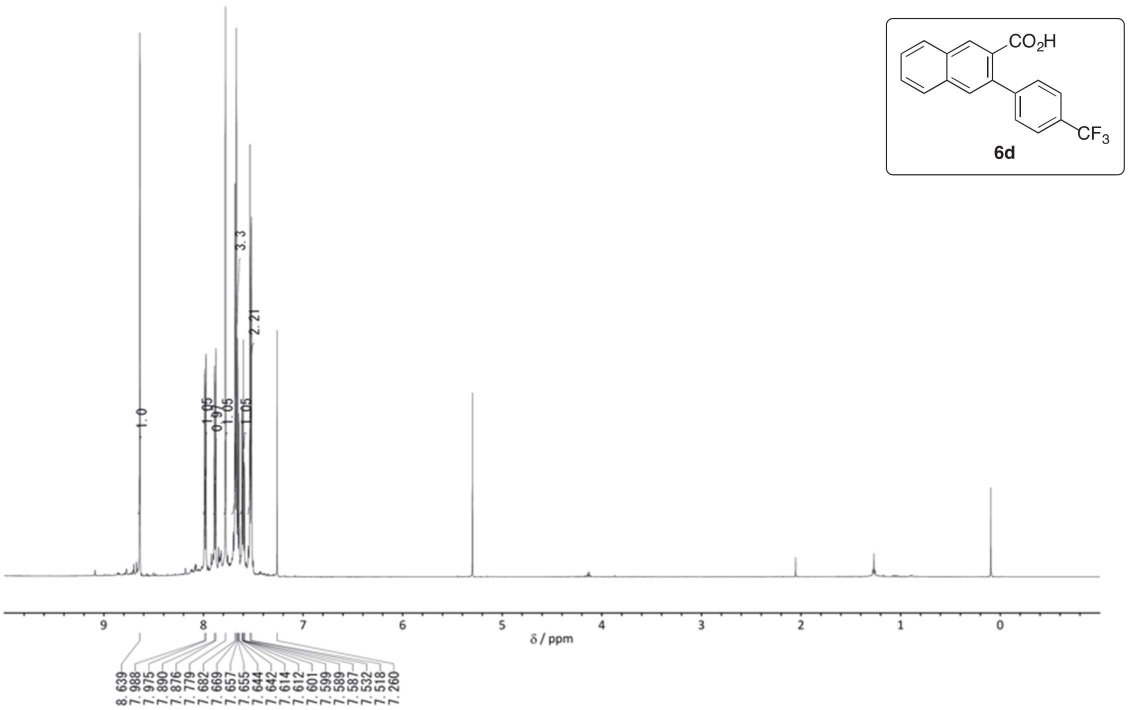


Figure S53. ^{1}H NMR spectra of **6d** (in $CDCl_3$)

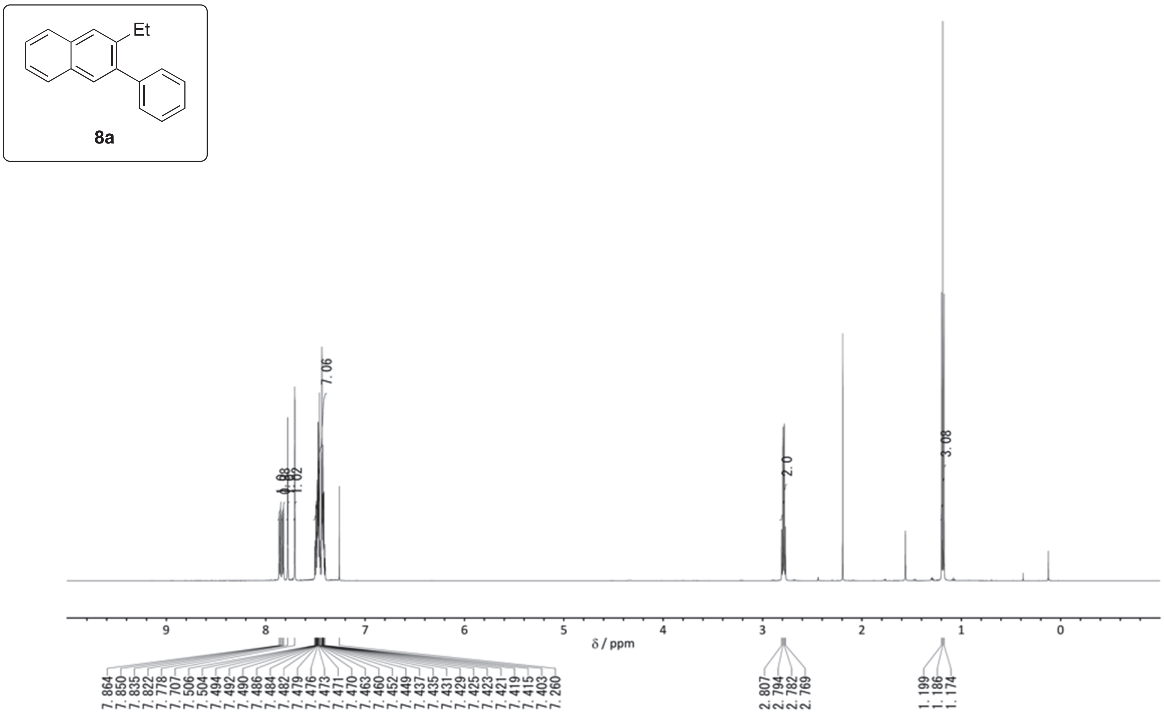


Figure S54. ^{1}H NMR spectra of **8a** (in $CDCl_3$)

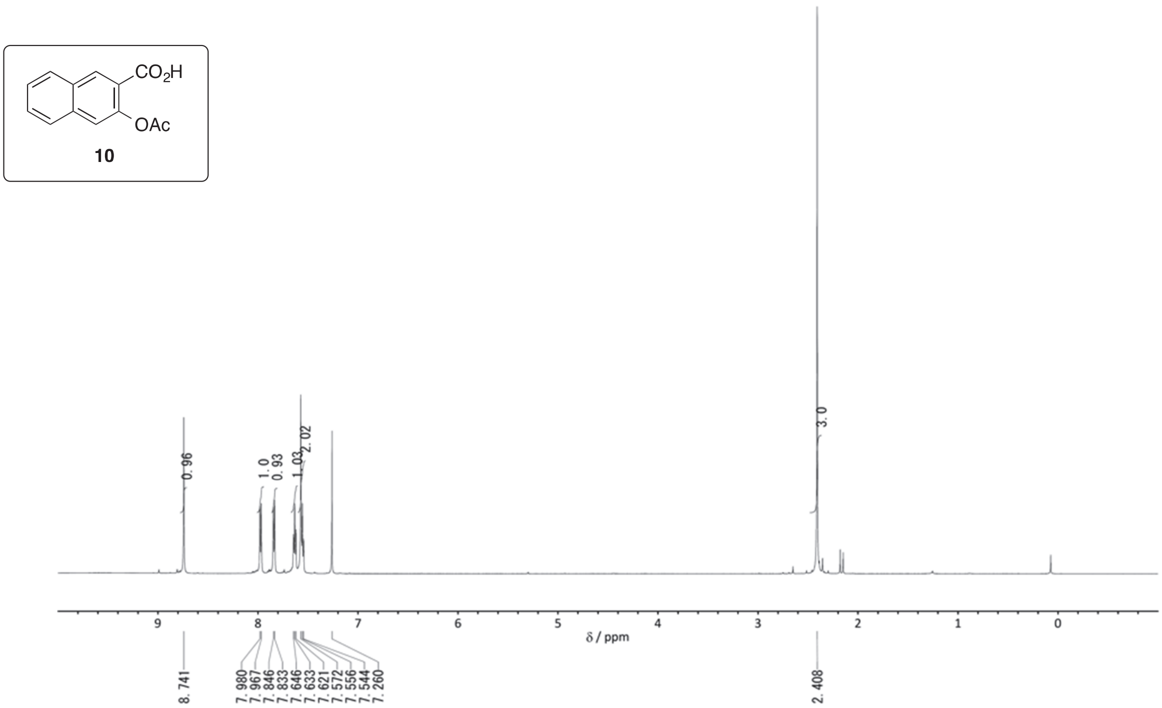


Figure S55. ^{1}H NMR spectra of **10** (in $CDCl_3$)

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[9]) NMR data of this product has not been reported, but synthesis was reported; Cairns, J. F.; Hickinbottom, W. J. *J. Chem. Soc.* **1962**, 867–870.

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