Supporting Information For:

Iron-Catalyzed Selective Biaryl Coupling: Remarkable Suppression of Homo-Coupling by the Fluoride Anion

Takuji Hatakeyama and Masaharu Nakamura*

International Research Center of Elements Science,

Institute for Chemical Research, Kyoto University, Uji, Kyoto, 611-0011, Japan

General. All the reactions dealing with air- or moisture-sensitive compounds were carried out in a dry reaction vessel under a positive pressure of argon or nitrogen. Air- and moisture-sensitive liquids and solutions were transferred via a syringe or a stainless steel cannula. Analytical thin-layer chromatography (TLC) was performed on glass plates coated with 0.25 mm 230–400 mesh silica gel containing a fluorescent indicator (Merck, #1.05715.0009). TLC plates were visualized by exposure to ultraviolet light (254 nm) and/or by immersion in an acidic staining solution of *p*-anisaldehyde followed by heating on a hot plate. Organic solutions were concentrated by rotary evaporation at *ca.* 30 mmHg. Flash column chromatography was performed on Kanto silica gel 60 (spherical, neutral, 140–325 mesh) as described by Still et al.[1] Gel permeation chromatography was performed on a JAIGEL-1H and 2H (20 mm i.d.) with an LC-918 (Japan Analytical Industry Co., Ltd.).

Instrumentation. Proton nuclear magnetic resonance (^{1}H NMR) and carbon NMR (^{13}C NMR) spectra were recorded on JEOL EX-270 (270 MHz) NMR spectrometers. Proton chemical shift values are reported in parts per million (ppm, δ scale) downfield from tetramethylsilane and are referenced to the residual proton signal of $CDCl_3$ (δ 7.26). ^{13}C NMR spectra were recorded at 67.8 MHz: chemical shifts for carbons are reported in parts per million (ppm, δ scale) downfield from tetramethylsilane and are referenced to the carbon resonance of $CDCl_3$ (δ 77.0).

Solvent. Anhydrous tetrahydrofuran (THF) was purchased from Wako Chemical Co. and distilled from benzophenone ketyl at 760 mmHg under argon immediately before use. Anhydrous toluene was purchased from Wako Chemical Co. and dried over Molecular Sieves 4A and degassed before

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(1) Still, W. C.; Kahn, M.; Mitra, A. *J. Org. Chem.* **1978**, *43*, 2923–2925.

use. Water content of the solvent was determined with a Karl Fischer Moisture Titrator (MKC-210, Kyoto Electronics Company) to be less than 10 ppm.

Materials. Materials were purchased from Wako Chemical Co., Tokyo Kasei Kogyo Co., Aldrich Inc., and other commercial suppliers, and were used after appropriate purification unless otherwise noted. Florisil (100–200 mesh) was purchased from Nakarai Tesque Inc. $FeF_3 \cdot 3H_2O$ was purchased from Aldrich Inc. and ground with a mortar before use. Aryl magnesium bromides (ArMgBr) were prepared from the corresponding aryl bromides and magnesium (turnings) using a standard method and titrated before use. Aryl halides were purified by distillation and shown to be over 99.5% pure by GC analysis.

GC analyses. Yield (using undecane as an internal standard) was determined for a crude product by GC analyses on a Shimadzu GC-17A instrument equipped with an FID detector and a capillary column, HR-1 (Shinwa, 25 m × 0.25 mm i.d., 0.25 mm film thickness).

Screening of catalysts and additives (Tables S-1 and S-2: Table 1 and related data)

p-Tolylmagnesium bromide (0.98 mL, 1.02 M, 1.0 mmol) was added to a catalyst (0.02 mmol), an additive (0.02–0.06 mmol), chlorobenzene **1** (45.0 mg, 0.4 mmol), and undecane (42.2 μL, 0.2 mmol) at room temperature. The coupling reaction was carried out at 60 °C for 24 h. After cooling to ambient temperature, aqueous sodium potassium tartrate (saturated, 2.0 mL) was added. The aqueous layer was extracted five times with Et_2O. The combined organic extracts were filtered with a pad of Florisil (100–200 mesh, Nakarai Tesque Inc.). Yields of 4-methylbiphenyl **2**, biphenyl **3**, and 4,4'-dimethylbiphenyl **4** (0.50 mmol = 100% yield, based on the amount of *p*-TolMgBr) and recovery of chlorobenzene **1** were determined by GC analysis.

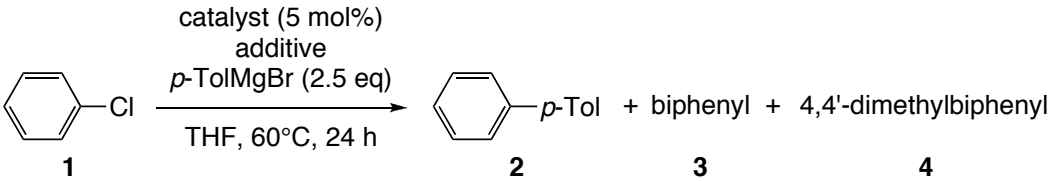


Table S-1

entry[a]	catalyst	additive (mol%)	yield[b] (%)			
			2	**3**	**1**	**4**[c]
1	$FeF_3{\cdot}3H_2O$	SIPr·HCl (15)	98	<1	0	4
2	$FeF_3{\cdot}3H_2O$	SIPr·HCl (10)	93	<1	5	4
3	$FeF_3{\cdot}3H_2O$	SIPr·HCl (5)	61	<1	36	5
4	$FeF_3{\cdot}3H_2O$	SIMes·HCl (15)	34	trace	64	5
5	$FeF_3{\cdot}3H_2O$	IPr·HCl (15)	25	trace	71	4
6	$FeF_3{\cdot}3H_2O$	PCy_3 (10)	5	trace	87	5
7	$FeF_3{\cdot}3H_2O$	none	6	trace	93	4
8	$FeF_2{\cdot}4H_2O$	SIPr·HCl (15)	96	<1	2	4
9	FeF_3	SIPr·HCl (15)	29	trace	69	2
10	FeF_2	SIPr·HCl (15)	18	trace	81	1
11[d]	FeF_3	H_2O (15), SIPr·HCl (15)	40	trace	57	1
12[d]	FeF_2	H_2O (20), SIPr·HCl (15)	20	trace	78	1
13	$FeCl_3$	SIPr·HCl (15)	32	2	10	32
14[e]	$FeCl_3$	KF (20), SIPr·HCl (15)	92	1	0	8
15	$FeCl_3$	TBAF (20), SIPr·HCl (15)	65	3	12	17
16	$FeCl_3$	none	17	2	17	33
17	$Fe(acac)_3$	SIPr·HCl (15)	26	2	18	29
18[f]	$CoF_2{\cdot}4H_2O$	SIPr·HCl (15)	91	1	2	10
19	$Co(acac)_3$	SIPr·HCl (15)	51	6	6	22
20[g]	$NiF_2{\cdot}4H_2O$	SIPr·HCl (5)	93	2	0	8
21	$Ni(acac)_2$	SIPr·HCl (5)	46	11	0	30

[a] Reactions were carried out on a 0.4–1.0 mmol scale. [b] The yield was determined by GC analysis using undecane as an internal standard. [c] The yield was based on the amount of *p*-TolMgBr. [d] Iron salt and additives were mixed in THF for 10 min at room temperature prior to the reaction. [e] $FeCl_3$ was treated with KF in MeOH/THF, which were then removed in vacuo. [f] Reaction was carried out at 60 °C for 24 h, then 80 °C for 8 h. [g] Reaction was carried out at 60 °C for 60 h.

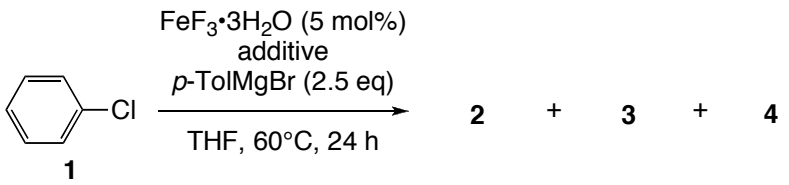
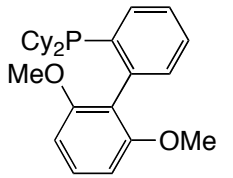
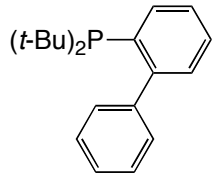


Table S-2

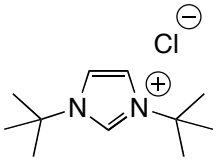
entry[a]	additive (mol%)	yield[b] (%)			
		2	**3**	**1**	**4**[c]
1	SIPr·HBF_4 (15)	39	trace	48	6
2	I-*t*-Bu·HCl (15)	51	<1	48	6
3	I-Adm·HBF_4 (15)	8	trace	71	25
4	I-MeBu·HBF_4 (15)	2	0	96	4
5	PPh_3 (10)	2	0	94	4
6	dppe (10)	0	0	100	3
7	dppf (10)	1	0	99	4
8	Cy_2P(MeO-biphenyl) (10)	2	trace	97	5
9	*t*-Bu_2P(biphenyl) (10)	2	trace	95	5
10	Phenanthoroline (5)	1	0	95	6
11	TMEDA (2.5 equiv)	1	0	95	8



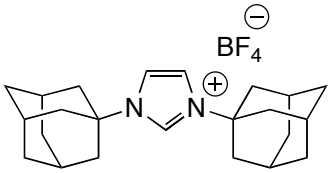
Cy_2P(MeO-biphenyl)



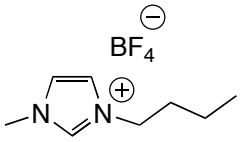
t-Bu_2P(biphenyl)



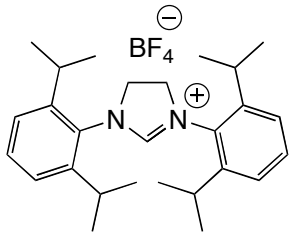
I-*t*-Bu·HCl



I-Adm·HBF_4



I-MeBu·HBF_4

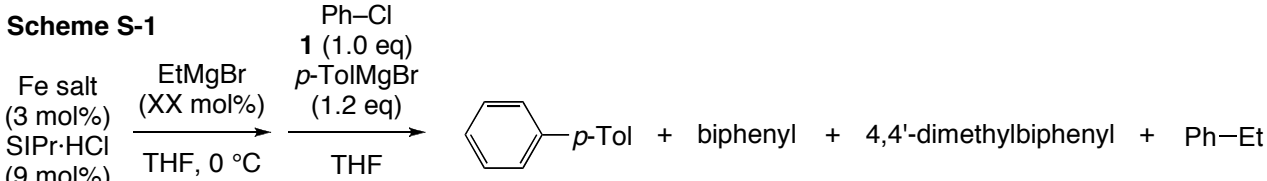


SIPr·HBF_4

[a] Reactions were carried out on a 0.4 mmol scale. [b] The yield was determined by GC analysis using undecane as an internal standard. [c] The yield was based on the amount of *p*-TolMgBr.

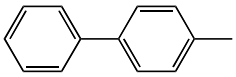
Preparation of active iron catalyst by using EtMgBr shown in equation 1 and additional data

Scheme S-1



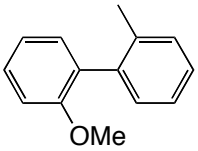
	2	**3**	**4**	
FeF_3•$3H_2O$, 18 mol%	98%	trace	3%	0%
FeF_3•$3H_2O$, 27 mol%	97%	trace	2%	3%
FeF_3, 9 mol%	98%	trace	3%	0%
FeF_3, 18 mol%	96%	trace	1%	4%

A representative procedure for the reaction shown in Table 2 and equation 1; Synthesis of 4-methylbiphenyl (2)



$FeF_3 \cdot 3H_2O$ (0.150 g, 0.90 mmol) and 1,3-bis(2,6-*i*-propylphenyl)imidazolinium hydrochloride (1.153 g, 2.70 mmol) was added to a THF solution of ethylmagnesium bromide (5.00 mL, 1.08 M, 5.40 mmol) at 0 °C. THF (1.0 mL) was added to rinse the vessel. After stirring at room temperature for 5 h, chlorobenzene (3.377 g, 30.0 mmol) and a THF solution of *p*-tolylmagnesium bromide (38.7 mL, 1.02 M, 36.0 mmol) was added at 0 °C. The reaction was carried out at 60 °C for 24 h. After cooling to ambient temperature, aqueous sodium potassium tartrate (saturated, 60.0 mL) was added. The aqueous layer was extracted five times with hexane. The combined organic extracts were filtered with a pad of Florisil (100–200 mesh, Nakarai Tesque Inc.). GC analysis was carried out (98% yield) using undecane as an internal standard. After the solvent was removed *in vacuo*, the crude product was purified by chromatography on silica gel (pentane) to obtain the title compound (4.97 g, 98% yield, > 98% pure on GC analysis) as a white solid. ^{1}H and ^{13}C NMR spectra have been attached. Analytical data for the title compound have been reported.[2]

Synthesis of 2-methoxy-2'-methylbiphenyl (S1)



A THF solution of ethylmagnesium bromide (0.167 mL, 1.08 M, 0.18 mmol) was added to $FeF_3 \cdot 3H_2O$ (5.01 mg, 0.03 mmol) and 1,3-bis(2,6-*i*-propylphenyl)imidazolinium hydrochloride (38.4 mg, 0.09 mmol) at 0 °C. THF (0.10 mL) was added to rinse the vessel. After stirring at room temperature for 5 h, 2-chloroanisole (142.6 mg, 1.0 mmol) and a THF solution of *o*-tolylmagnesium bromide (1.50 mL, 0.80 M, 1.2 mmol) was added at 0 °C. The reaction was carried out at 80 °C for 24 h. After cooling to ambient temperature, aqueous sodium potassium tartrate (saturated, 2.0 mL) was added. The aqueous layer was extracted five times with Et_2O. The combined organic extracts were filtered with a pad of Florisil (100–200 mesh, Nakarai Tesque Inc.). After the solvent was removed *in vacuo*, the crude product was purified by chromatography on silica gel (15, 30, and 50% toluene in hexane) to obtain the title compound **S1** (0.178 g, 90% yield, > 99% pure on GC analysis) as a colorless liquid. ^{1}H and ^{13}C NMR spectra have been attached. Analytical data for the title compound have been reported.[3]

Synthesis of 4-methoxy-4'-methylbiphenyl (S2)

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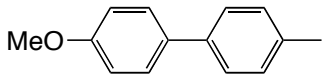
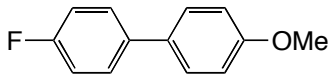
(2) Fairlamb, I. J. S.; Kapdi, A. R.; Lee, A. F. *Org. Lett.* **2004**, 6, 4435–4438.

(3) Limmert, M. E.; Roy, A. H.; Hartwig, J. F. *J. Org. Chem.* **2005**, *70*, 9364–9370.



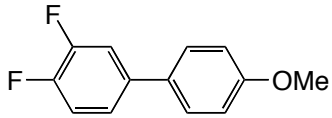
The reaction was carried out according to the typical procedure on a 1.0 mmol scale by using a THF solution of phenylmagnesium bromide (1.34 mL, 1.01 M, 1.35 mmol) and *p*-anisyl chloride (142.6 mg, 1.0 mmol). Conditions: 60 °C, 24 h. The title compound **S2** (0.182 g, 92% yield, > 99% pure on GC analysis) was obtained as a white solid after silica gel column chromatography (15, 30, and 50% toluene in hexane). ^{1}H and ^{13}C NMR spectra have been attached. Analytical data for the title compound have been reported.[4]

Synthesis of 4-fluoro-4'-methoxybiphenyl (S3)



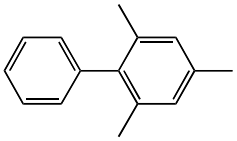
The reaction was carried out according to the typical procedure on a 1.0 mmol scale by using a THF solution of 4-methoxyphenylmagnesium bromide (1.88 mL, 0.64 M, 1.2 mmol) and 1-chloro-4-fluorobenzene (130.6 mg, 1.0 mmol). Conditions: 60 °C, 24 h. The title compound **S3** (0.184 g, 91% yield, > 99% pure on GC analysis) was obtained as a white solid after silica gel column chromatography (15, 30, and 50% toluene in hexane). ^{1}H and ^{13}C NMR spectra have been attached. Analytical data for the title compound have been reported.[4]

Synthesis of 3,4-difluoro-4'-methoxybiphenyl (S4)



The reaction was carried out according to the typical procedure on a 1.0 mmol scale by using a THF solution of 4-methoxyphenylmagnesium bromide (2.34 mL, 0.64 M, 1.5 mmol), 4-chloro-1,2-difluorobenzene (148.6 mg, 1.0 mmol), $FeF_3{\cdot}3H_2O$ (8.34 mg, 0.05 mmol), and 1,3-bis(2,6-*i*-propylphenyl)imidazolinium hydrochloride (64.1 mg, 0.15 mmol). Conditions: 60 °C, 24 h, 80 °C, 12 h. The title compound **S4** (0.178 g, 81% yield, > 99% pure on GC analysis) was obtained as a white solid after silica gel column chromatography (15, 30, and 50% toluene in hexane). ^{1}H and ^{13}C NMR spectra have been attached. Analytical data for the title compound have been reported.[5]

Synthesis of 2,4,6-trimethylbiphenyl (S5)



$FeF_3{\cdot}3H_2O$ (5.01 mg, 0.03 mmol), 1,3-bis(2,6-*i*-propylphenyl)imidazolinium hydrochloride (38.4 mg, 0.09 mmol) was added to a THF solution of ethylmagnesium bromide (0.167 mL, 1.08 M, 0.18 mmol) at 0 °C. THF (0.10 mL) was added to rinse the vessel. After 10 h, a THF solution of

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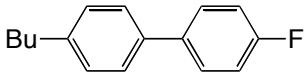
(4) Ackermann, L.; Althammer, A. *Org. Lett.* **2006**, *8*, 3457–3460.

(5) Hagiwara, E.; Gouda, K-i.; Hatanaka, Y.; Hiyama, T. *Tetrahedron Lett.* **1997**, *38*, 439–442.

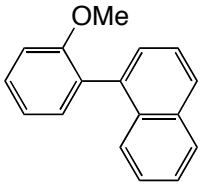
mesitylmagnesium bromide (1.02 mL, 1.18 M, 1.2 mmol) was added at 0 °C. The solvent was removed *in vacuo*. The resulting viscous liquid was dissolved in toluene (1.0 mL) and then chlorobenzene (112.6 mg, 1.0 mmol) was added at 0 °C. The reaction was carried out at 120 °C for 24 h. After cooling to ambient temperature, aqueous sodium potassium tartrate (saturated, 2.0 mL) was added. The aqueous layer was extracted five times with Et_2O. The combined organic extracts were filtered with a pad of Florisil (100–200 mesh, Nakarai Tesque Inc.). GC analysis was carried out (93% yield) using undecane (42.2 μL, 0.2 mmol) as an internal standard. After the solvent was removed *in vacuo*, the crude product was purified by GPC to obtain the title compound **S5** (0.174 g, 89% yield, > 99% pure on GC analysis) as a colorless liquid. ^{1}H and ^{13}C NMR spectra have been attached. Analytical data for the title compound have been reported.[6]

Synthesis of 4-butyl-4'-fluorobiphenyl (S6)



The reaction was carried out according to the typical procedure on a 1.0 mmol scale by using a THF solution of 4-fluorophenylmagnesium bromide (1.46 mL, 1.03 M, 1.5 mmol), 4-butylchlorobenzene (168.7 mg, 1.0 mmol), $FeF_3{\cdot}3H_2O$ (6.68 mg, 0.04 mmol), and 1,3-bis(2,6-*i*-propylphenyl)imidazolinium hydrochloride (51.2 mg, 0.12 mmol). Conditions: 60 °C, 24 h. The title compound **S6** (0.199 g, 87% yield, > 97% pure on GC analysis) was obtained as a white solid after silica gel column chromatography (pentane). ^{1}H and ^{13}C NMR spectra have been attached. Analytical data for the title compound have been reported.[7]

Synthesis of 1-(2-methoxyphenyl)naphthalene (S7)



The reaction was carried out according to the typical procedure on a 1.0 mmol scale by using a THF solution of 1-naphthylmagnesium bromide (4.62 mL, 0.26 M, 1.2 mmol), 2-chloroanisole (142.6 mg, 1.0 mmol), $FeF_3{\cdot}3H_2O$ (8.34 mg, 0.05 mmol), and 1,3-bis(2,6-*i*-propylphenyl)imidazolinium hydrochloride (64.1 mg, 0.15 mmol). Conditions: 70 °C, 48 h. The title compound **S7** (0.215 g, 92% yield, > 99% pure on GC analysis) was obtained as a white solid after silica gel column chromatography (10, 20, and 40% toluene in hexane). ^{1}H and ^{13}C NMR spectra have been attached. Analytical data for the title compound have been reported.[8]

Synthesis of 2-(2-methoxyphenyl)naphthalene (S8)

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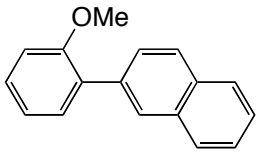
(6) Liang, L.-C.; Chien, P.-S.; Huang, M.-H. *Organometallics* **2005**, *24*, 353–357.

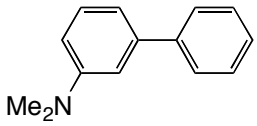
(7) Blakey, S. B.; MacMillan, D. W. C. *J. Am. Chem. Soc.* **2003**, 125, 6046–6047.

(8) Song, C.; Ma, Y.; Chai, Q.; Ma, C.; Jiang, W.; Andrus, M. B. *Tetrahedron* **2005**, *61*, 7438–7446.



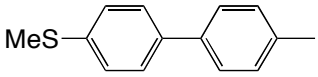
The reaction was carried out according to the typical procedure on a 1.0 mmol scale by using a THF solution of 2-naphthylmagnesium bromide (1.47 mL, 1.02 M, 1.5 mmol) and 2-chloroanisole (142.6 mg, 1.0 mmol). Conditions: 70 °C, 48 h. The title compound **S8** (0.224 g, 96% yield, > 99% pure on GC analysis) was obtained as a white solid after silica gel column chromatography (10, 20, and 40% toluene in hexane). ^{1}H and ^{13}C NMR spectra have been attached. Analytical data for the title compound have been reported.[9]

Synthesis of biphenyl-3-yl-dimethylamine (S9)



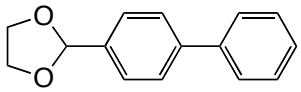
The reaction was carried out according to the typical procedure on a 1.0 mmol scale by using a THF solution of phenylmagnesium bromide (1.19 mL, 1.01 M, 1.2 mmol) and 3-chloro-*N*, *N*-dimethylaniline (155.6 mg, 1.0 mmol). Conditions: 60 °C, 24 h. The title compound **S9** (0.185 g, 94% yield, > 98% pure on GC analysis) was obtained as a colorless liquid after silica gel column chromatography (50 and 100% toluene in hexane). ^{1}H and ^{13}C NMR spectra have been attached. Analytical data for the title compound have been reported.[10]

Synthesis of 4'-methyl-4-methylsulfanylbiphenyl (S10)



The reaction was carried out according to the typical procedure on a 1.0 mmol scale by using a THF solution of *p*-tolylmagnesium bromide (1.323 mL, 1.02 M, 1.35 mmol) and 4-chlorothioanisole (158.7 mg, 1.0 mmol). Conditions: 60 °C, 24 h. The title compound **S10** (0.171 g, 80% yield, > 99% pure on GC analysis) was obtained as a white solid after silica gel column chromatography (10, 20, 40, and 100% toluene in hexane). ^{1}H and ^{13}C NMR spectra have been attached. Analytical data for the title compound have been reported.[11]

Synthesis of 2-(biphenyl-4-yl)-[1,3]dioxolane (S11)



The reaction was carried out according to the typical procedure on a 1.0 mmol scale by using a THF solution of phenylmagnesium bromide (1.34 mL, 1.01 M, 1.35 mmol) and 2-(4-chlorophenyl)-

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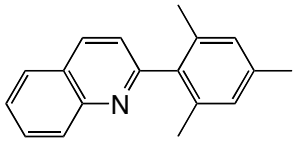
(9) Schneider, S.; Bannwarth, W. *Angew. Chem., Int. Ed.* **2000**, *39*, 4142–4145.

(10) Becht, J.-M.; Gissot, A.; Wagner, A.; Mioskowski, C. *Chem. Eur. J.* **2003**, *9*, 3209–3215.

(11) Yamada, Y.; Takeda, K.; Takahashi, H.; Ikegami, S. *Org. Lett.* **2002**, *4*, 3371–3374.

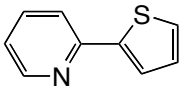
[1,3]dioxolane (184.6 mg, 1.0 mmol). Conditions: 60 °C, 24 h, 80 °C, 12 h. The title compound **S11** (0.199 g, 88% yield, > 99% pure on GC analysis) was obtained as a white solid after silica gel column chromatography (5, 10, and 20% Et_2O in hexane). ^1H and ^{13}C NMR spectra have been attached. Analytical data for the title compound have been reported.[12]

Synthesis of 2-(2,4,6-trimethylphenyl)quinoline (S12)



$FeF_3 \cdot 3H_2O$ (5.01 mg, 0.03 mmol), 1,3-bis(2,6-*i*-propylphenyl)imidazolinium hydrochloride (38.4 mg, 0.09 mmol) was added to a THF solution of ethylmagnesium bromide (0.167 mL, 1.08 M, 0.18 mmol) at 0 °C. After stirring at 0 °C for 12 h, a THF solution of mesitylmagnesium bromide (1.144 mL, 1.18 M, 1.35 mmol) was added at 0 °C. The solvent was removed *in vacuo*. The resulting viscous liquid was dissolved in toluene (1.0 mL) and then 2-chloroquinoline (163.6 mg, 1.0 mmol) was added at 0 °C. The reaction was carried out at 100 °C for 8 h. After cooling to ambient temperature, aqueous sodium potassium tartrate (saturated, 2.0 mL) was added. The aqueous layer was extracted five times with Et_2O and then once with EtOAc. The combined organic extracts were filtered with a pad of Florisil (100–200 mesh, Nakarai Tesque Inc.). After the solvent was removed *in vacuo*, the crude product was purified by chromatography on silica gel (10, 20, and 40% EtOAc in hexane) to obtain the title compound **S12** (0.203 g, 82% yield, > 99% pure on GC analysis) as a yellow solid. ^1H and ^{13}C NMR spectra have been attached. Analytical data for the title compound have been reported.[13]

Synthesis of 4-thiophen-2-yl-pyridine (S13)



The reaction was carried out according to the typical procedure on a 1.0 mmol scale by using a THF solution of 2-thienylmagnesium bromide (1.50 mL, 1.00 M, 1.5 mmol), 2-bromopyridine (158.0 mg, 1.0 mmol), $FeF_3 \cdot 3H_2O$ (10.01 mg, 0.06 mmol), and 1,3-bis(2,6-*i*-propylphenyl)imidazolinium hydrochloride (76.9 mg, 0.18 mmol). Conditions: 80 °C, 24 h. The title compound (0.119 g, 74% yield, > 95% pure on NMR analysis) was obtained as a yellow solid after silica gel column chromatography. Recrystallization gave the title compound **S13** (0.101 g, 63% yield, > 98% pure on NMR analysis) as a white solid. ^1H and ^{13}C NMR spectra have been attached. Analytical data for the title compound have been reported.[14]

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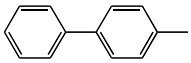
(12) Alonso, D. A.; Najera, C.; Pacheco, M. C. *J. Org. Chem.* **2002**, *67*, 5588–5594.

(13) Korn, T. J.; Schade, M. A.; Cheemala, M. N.; Wirth, S.; Guevara, S. A.; Cahiez, G.; Knochel, P. *Synthesis* **2006**, *21*, 3547–3574.

(14) Li, J.-H.; Zhu, Q.-M.; Xie, Y.-X. *Tetrahedron* **2006**, *62*, 10888–10895.

^{1}H and ^{13}C NMR spectra for isolated compounds (2) and (S1)–(S13)



(2)

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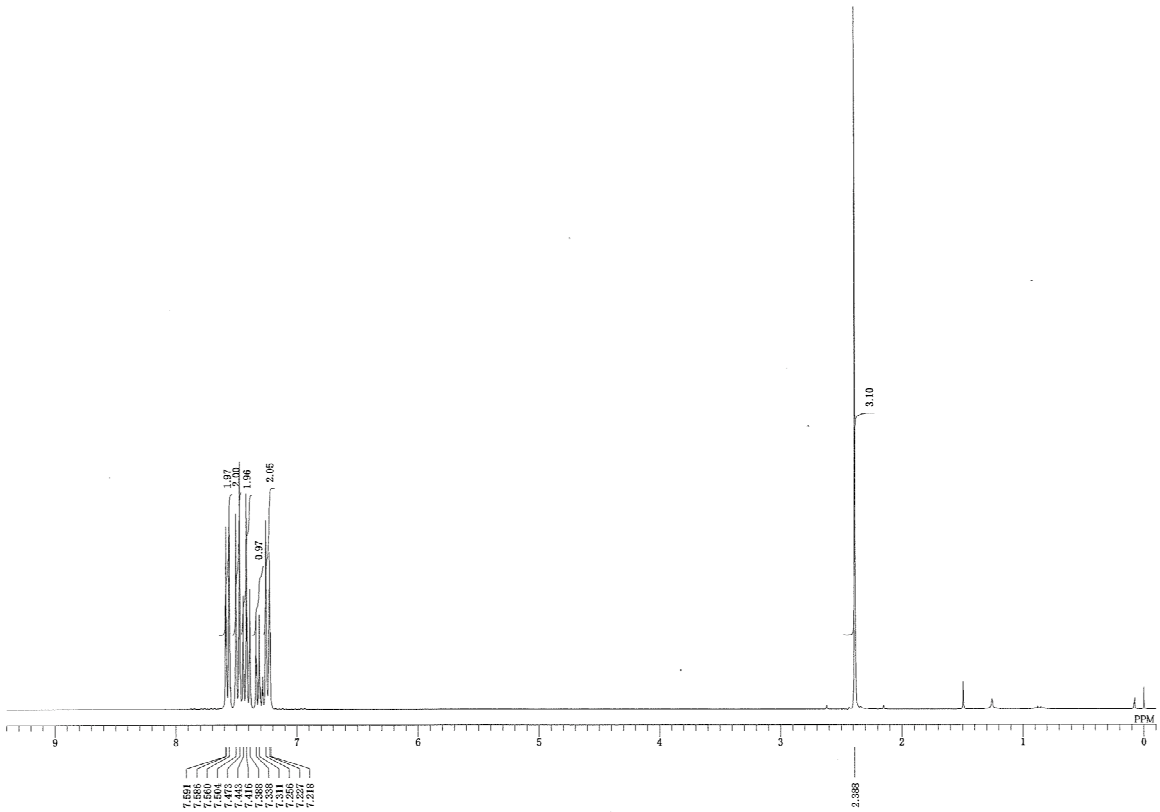

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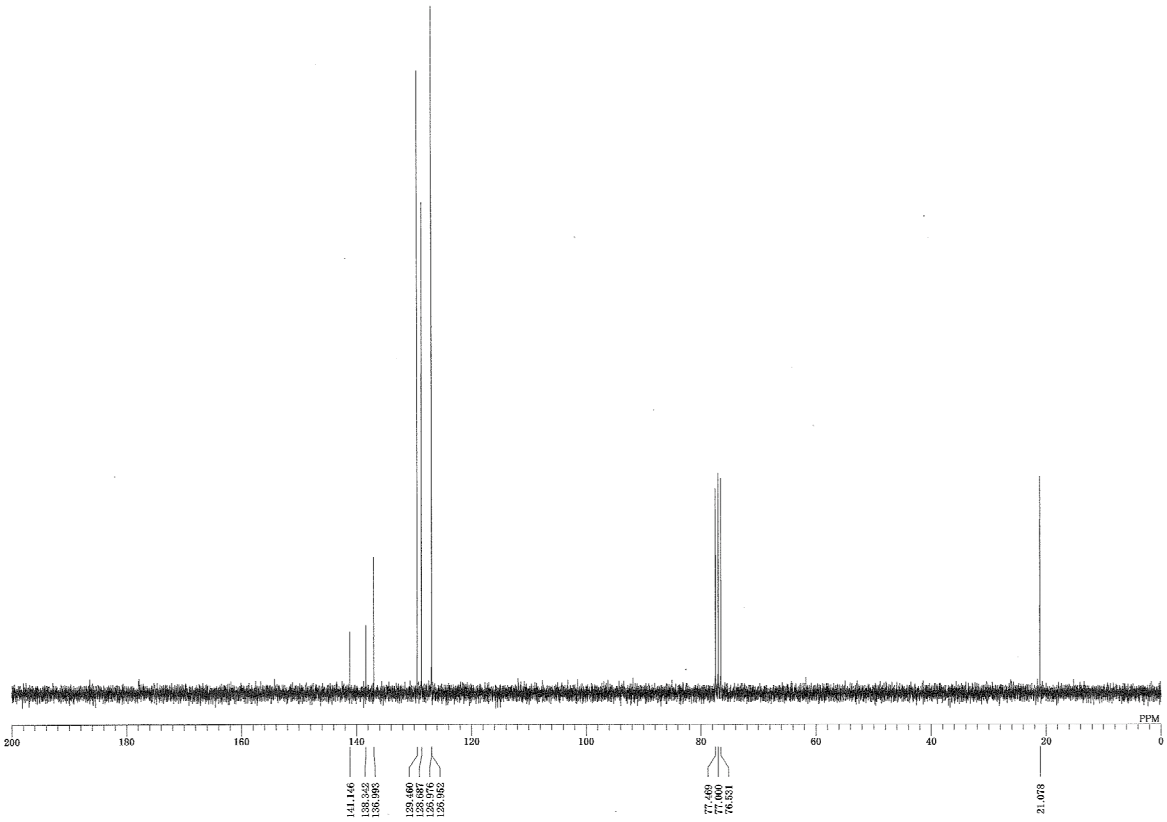
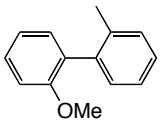
4MePh-Ph-13C





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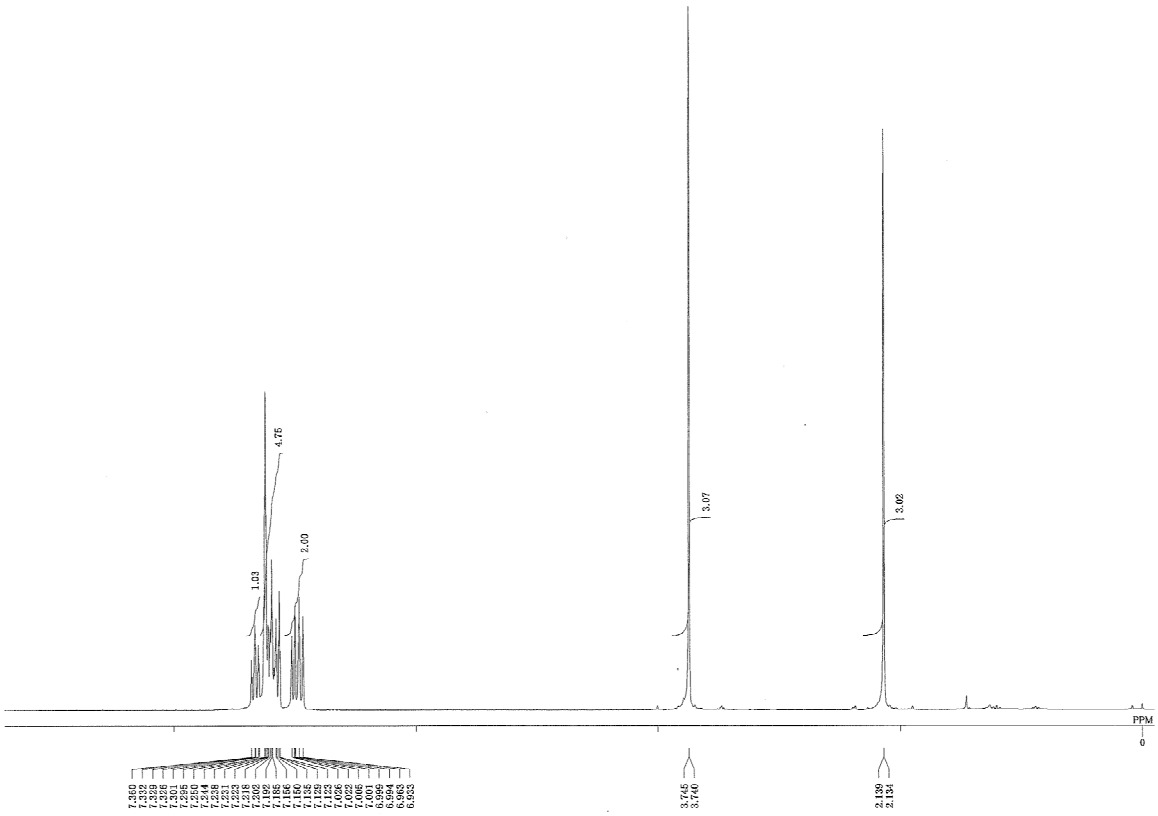
2MeOPh-2MePh-1H



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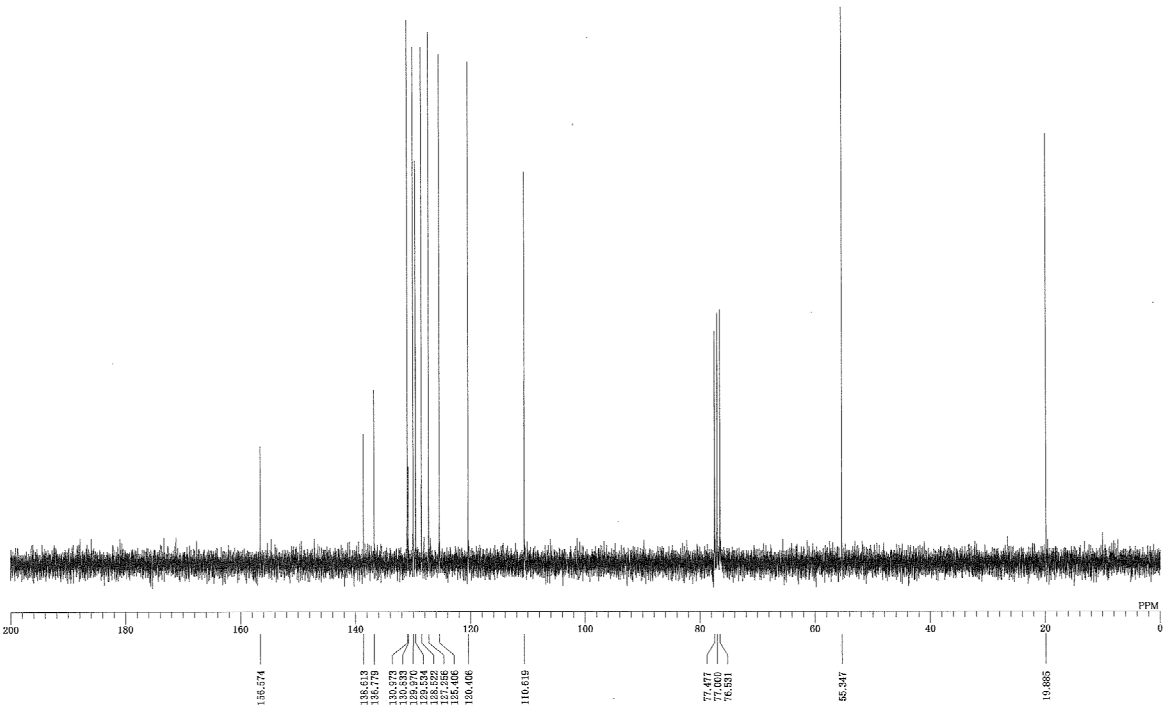
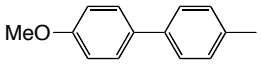
2MeOPh-2MePh-13C





(S2)

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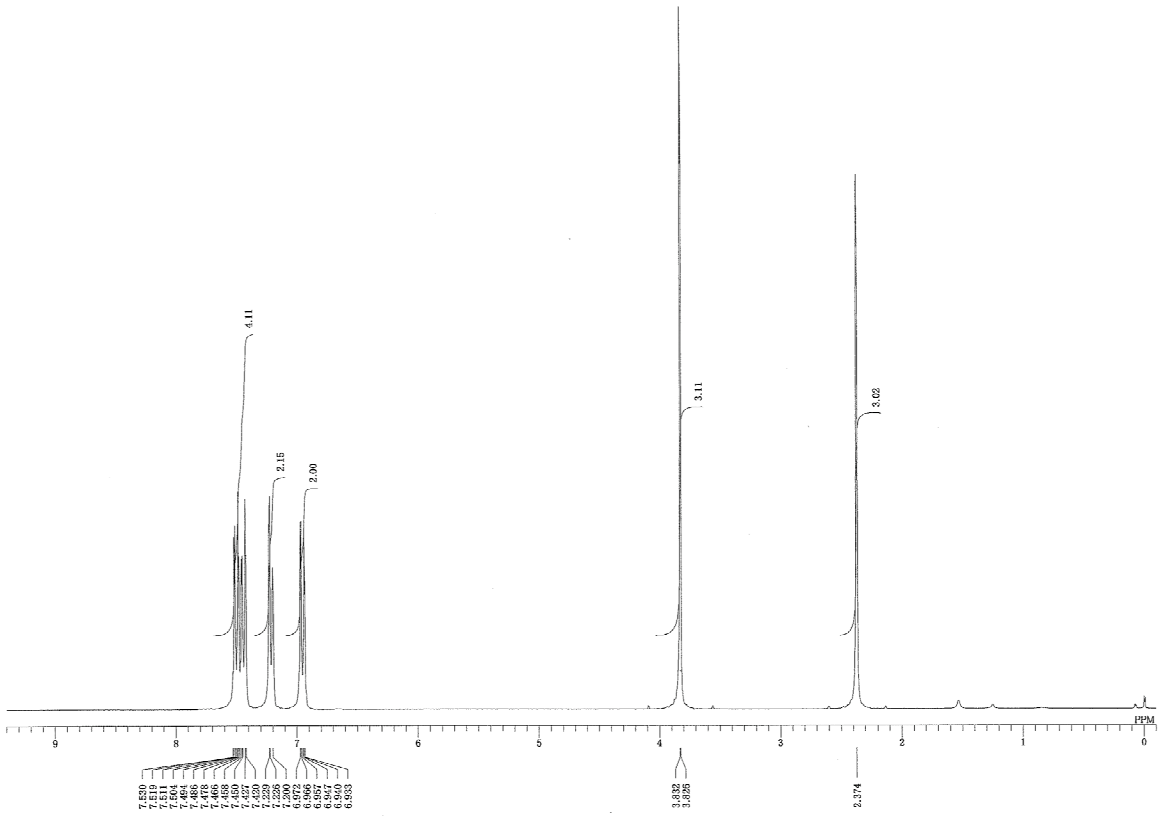
4MePh-4MeOPh-1H



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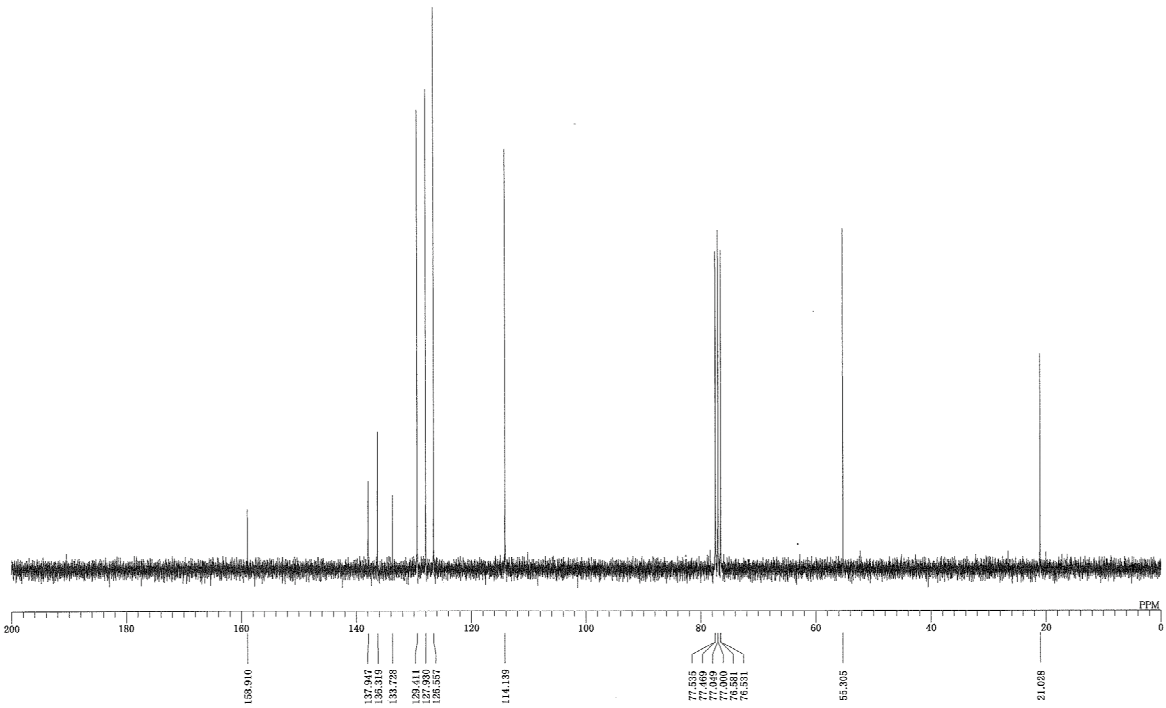
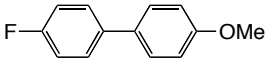
4MePh-4MeOPh-13C





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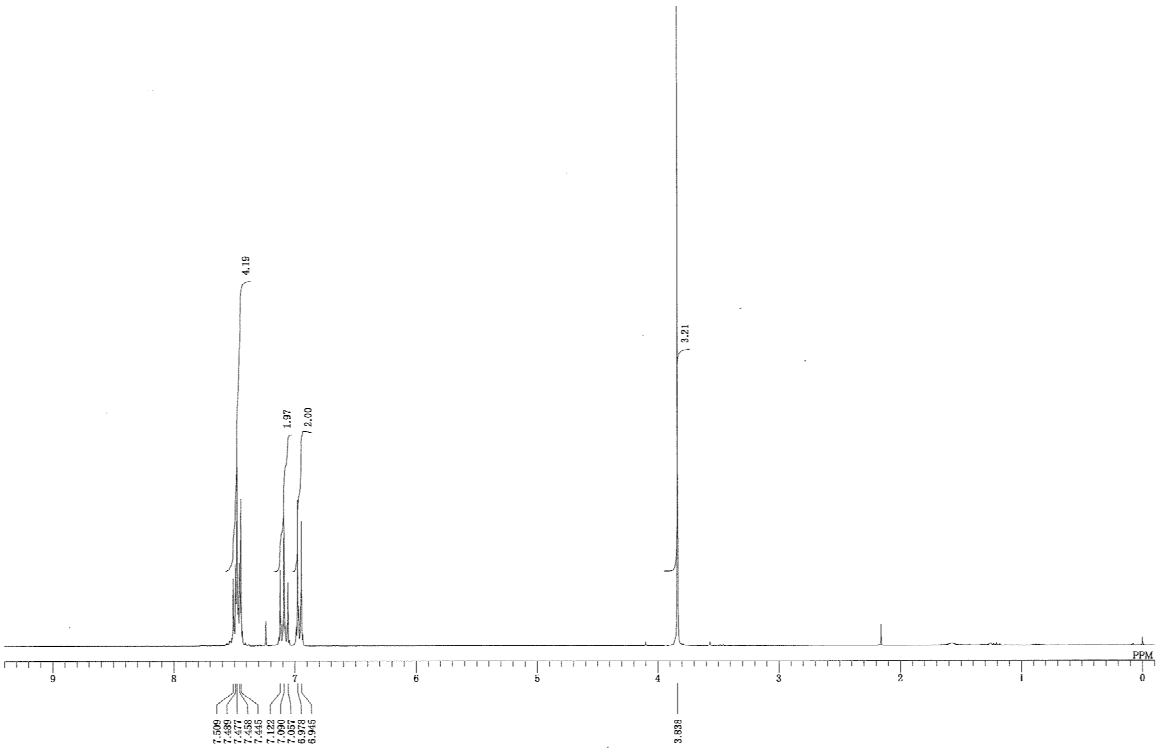
4MeOPh-4FPh



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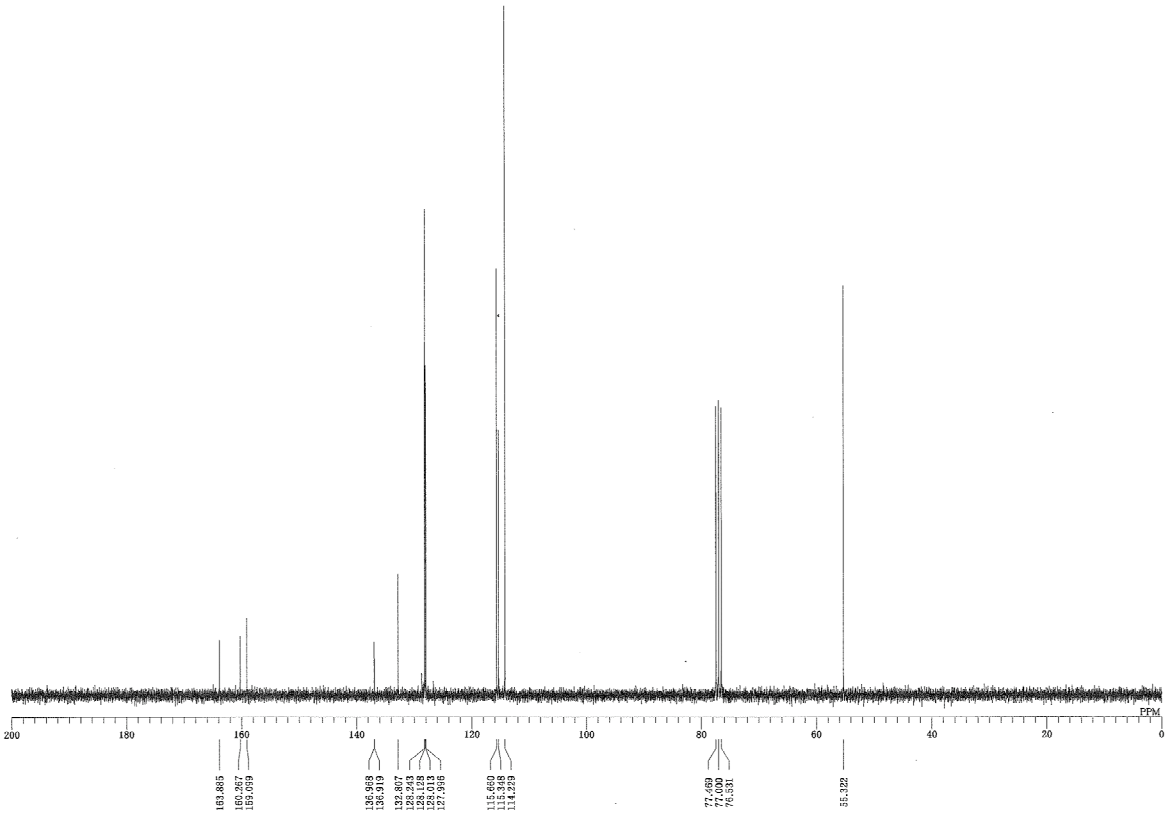
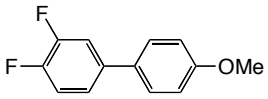
4MeOPh-4FPh-13C





(S4)

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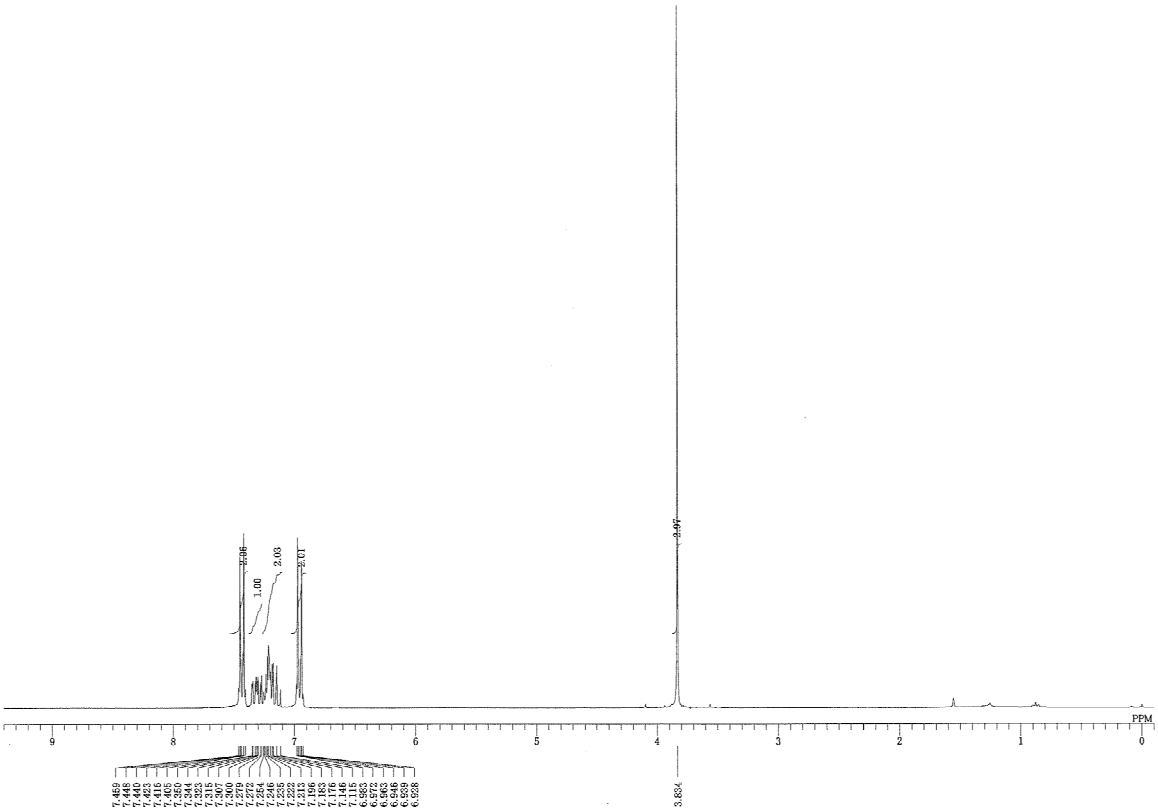
4MeOPh-3,4FPh-1H



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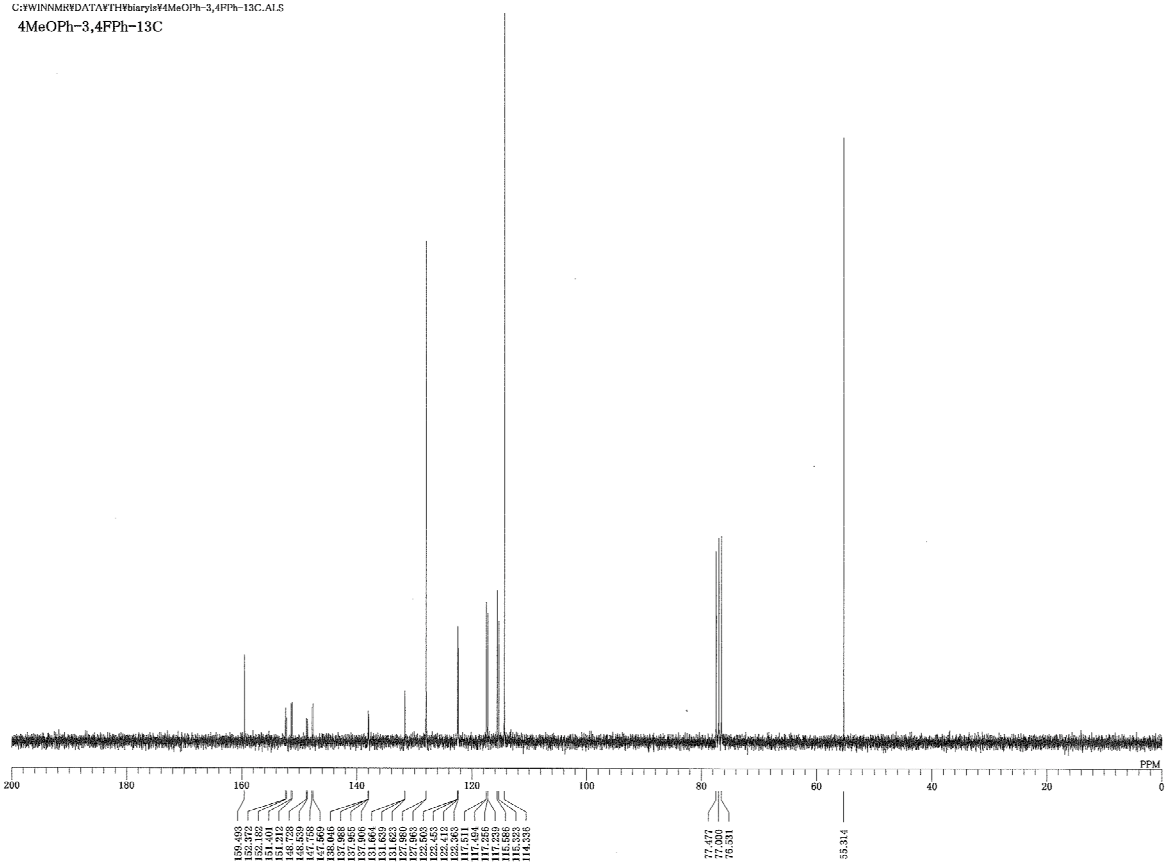
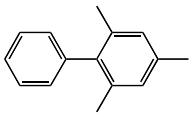
4MeOPh-3,4FPh-13C





(S5)

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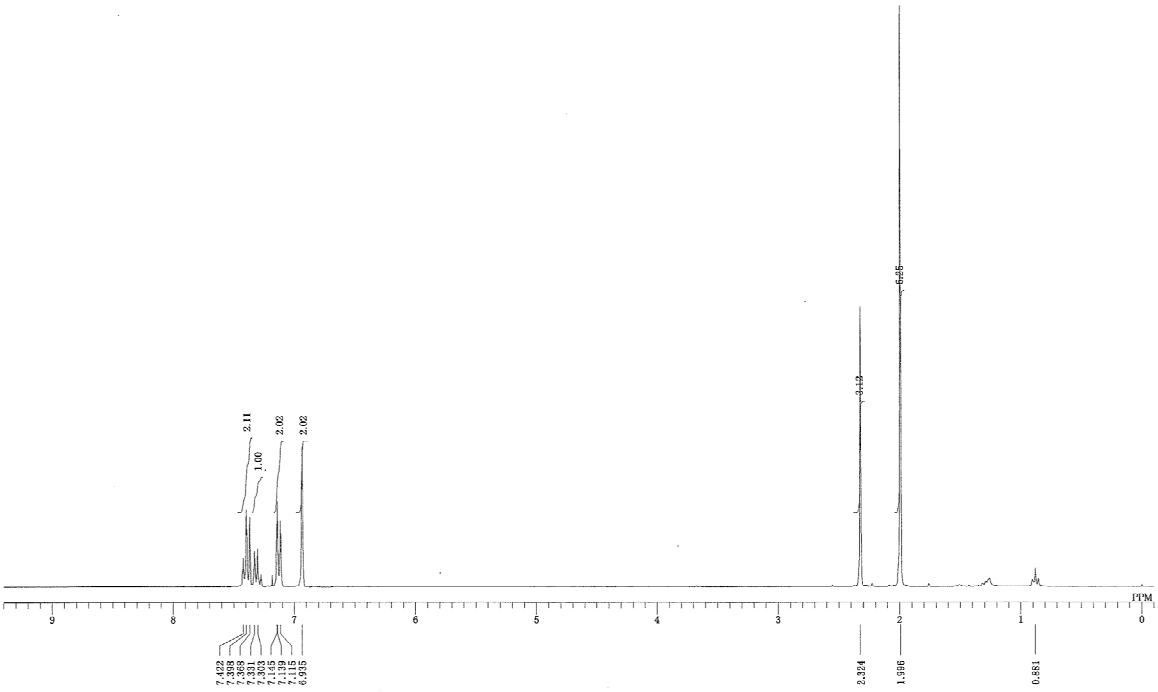
Ph-Mes-1H



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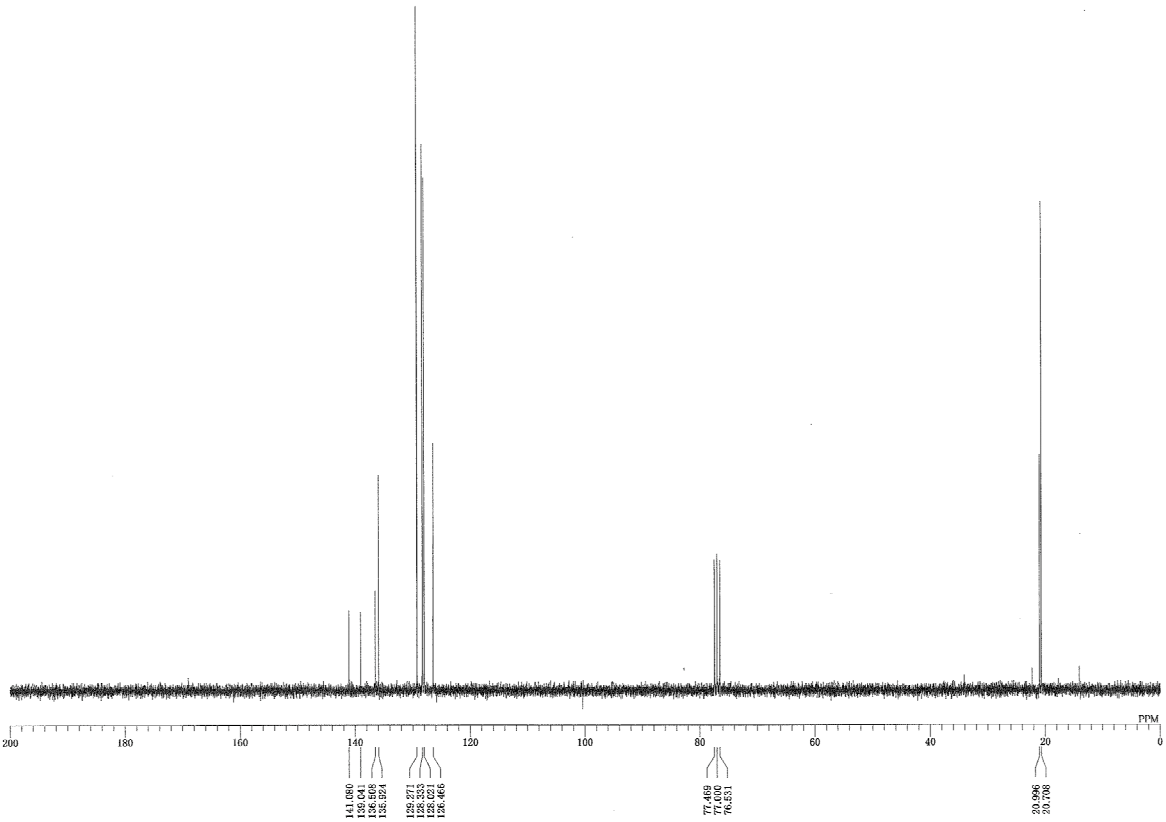
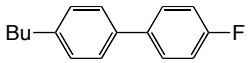
Ph-Mes-13C





(S6)

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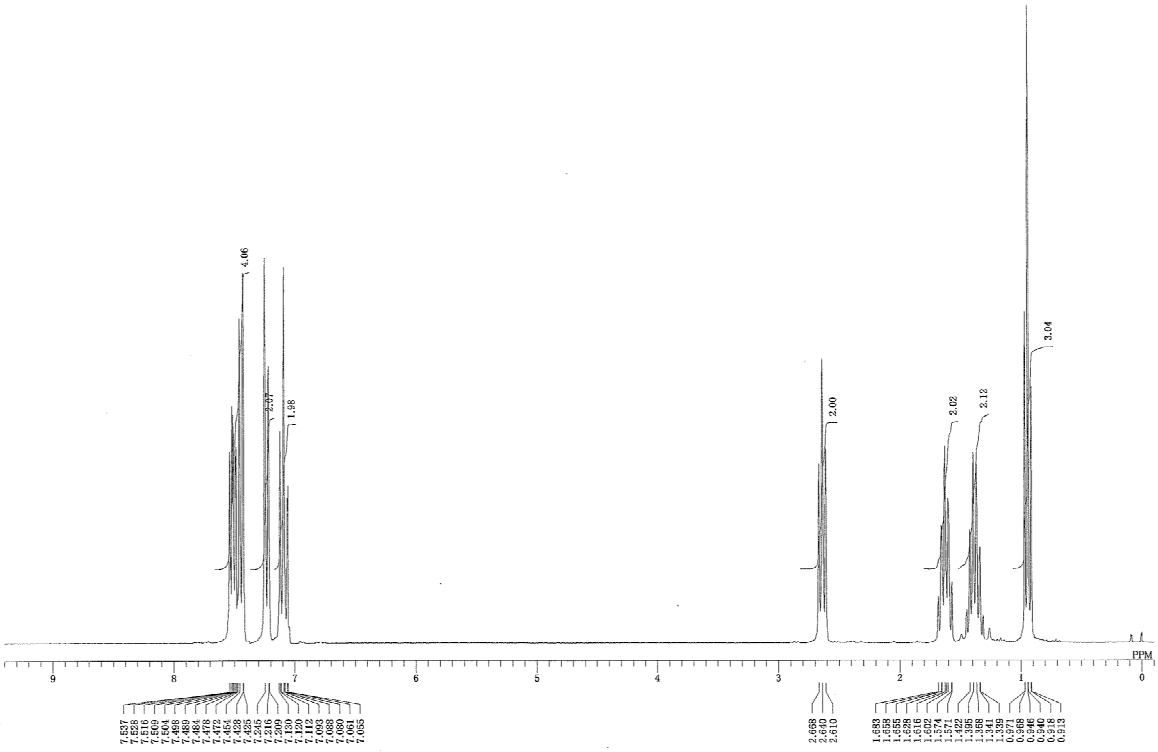
4BuPh-4FPh-1H



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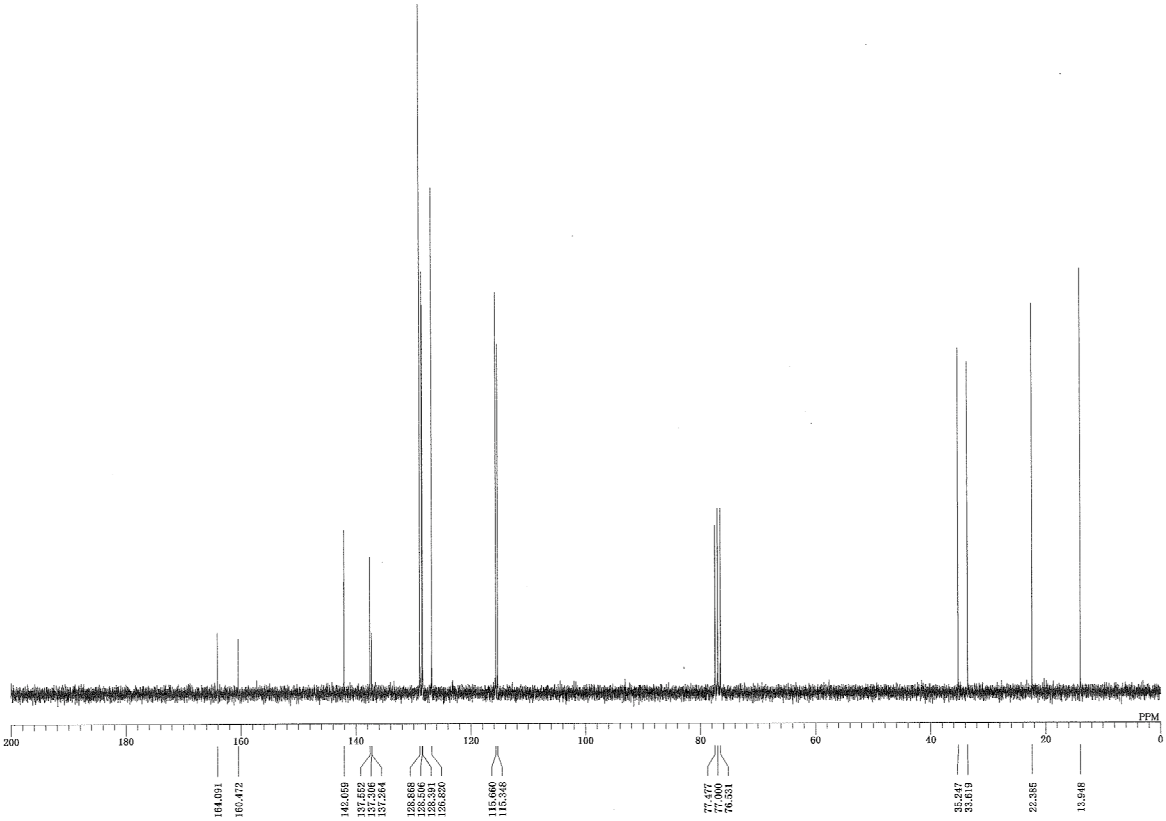
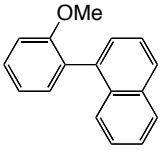
4BuPh-4FPh-13C





(S7)

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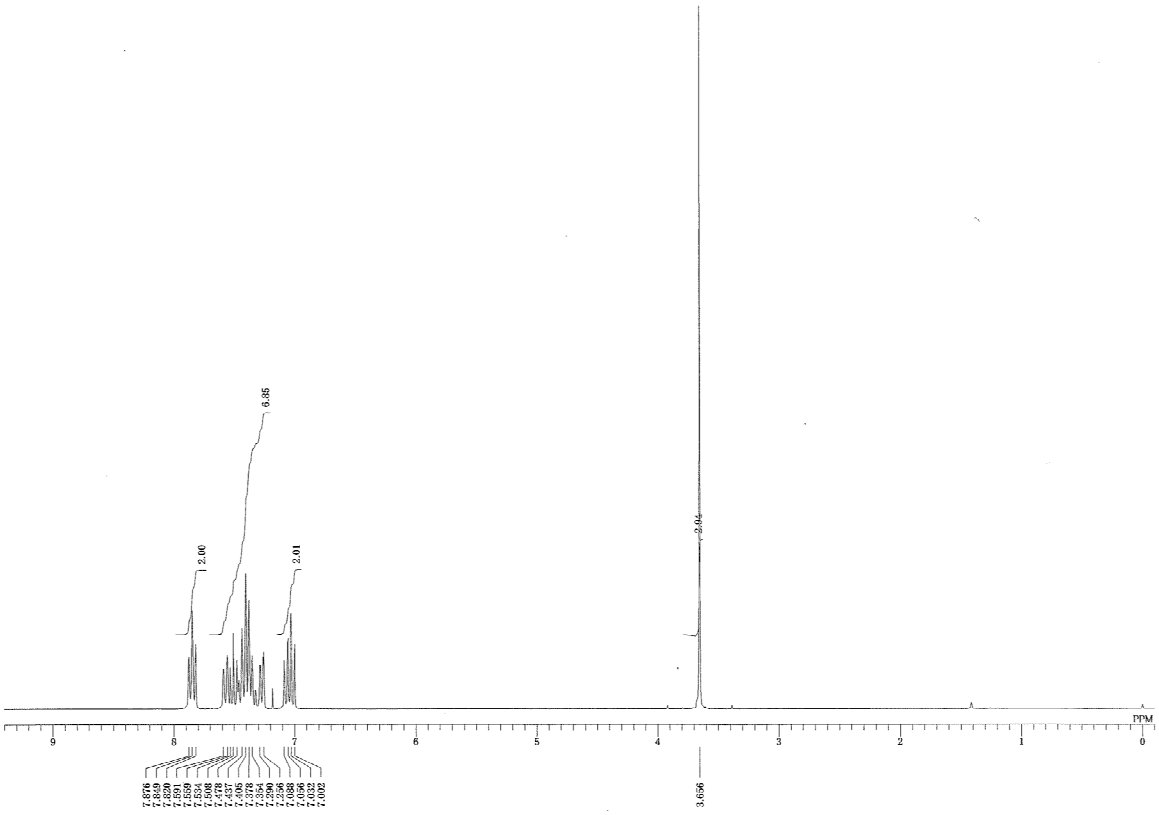
1Nap-2MeOPh-1H



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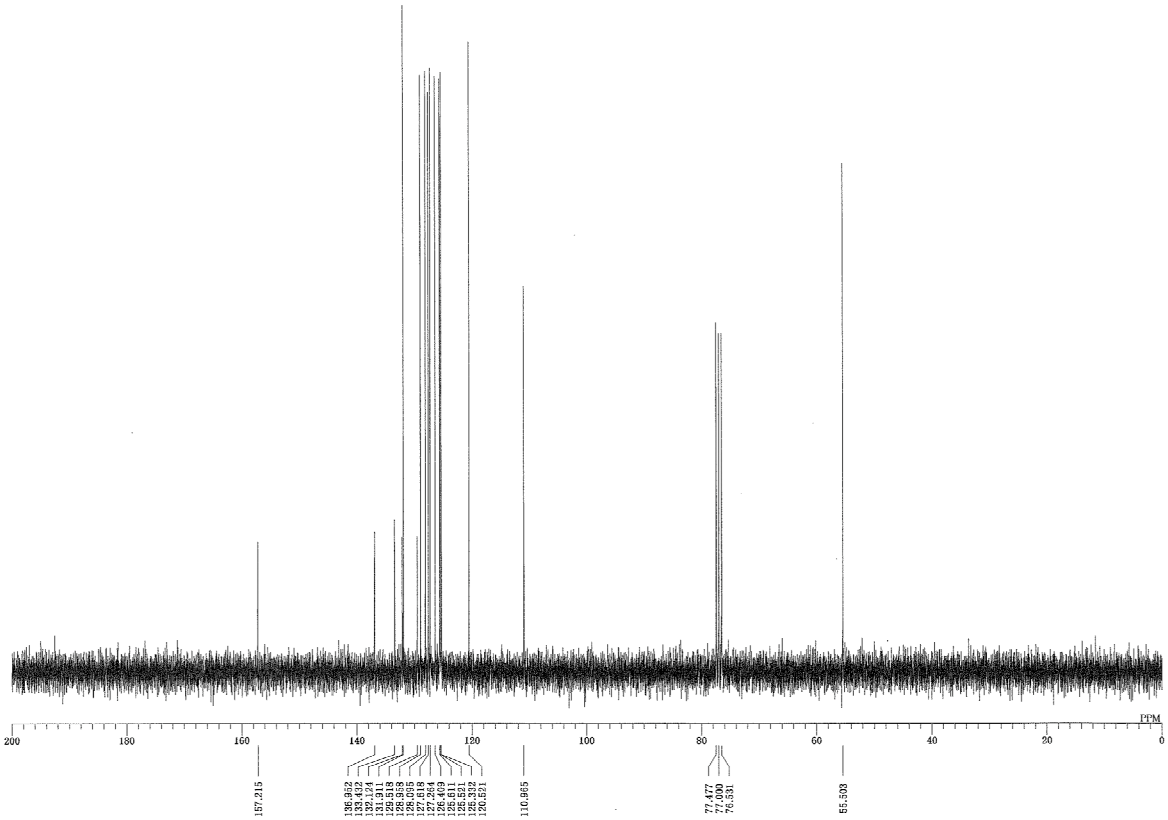
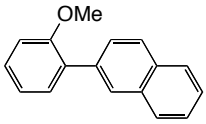
1Nap-2MeOPh-13C





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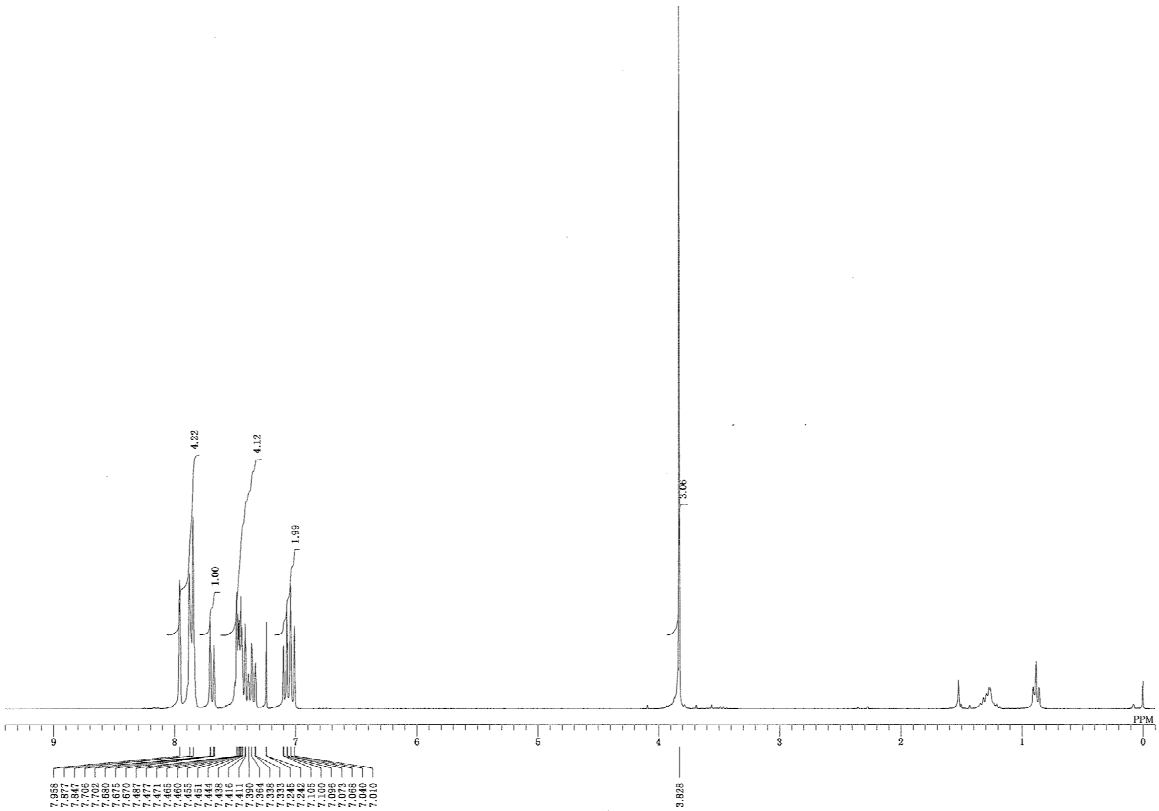
2Nap-2MeOPh-1H



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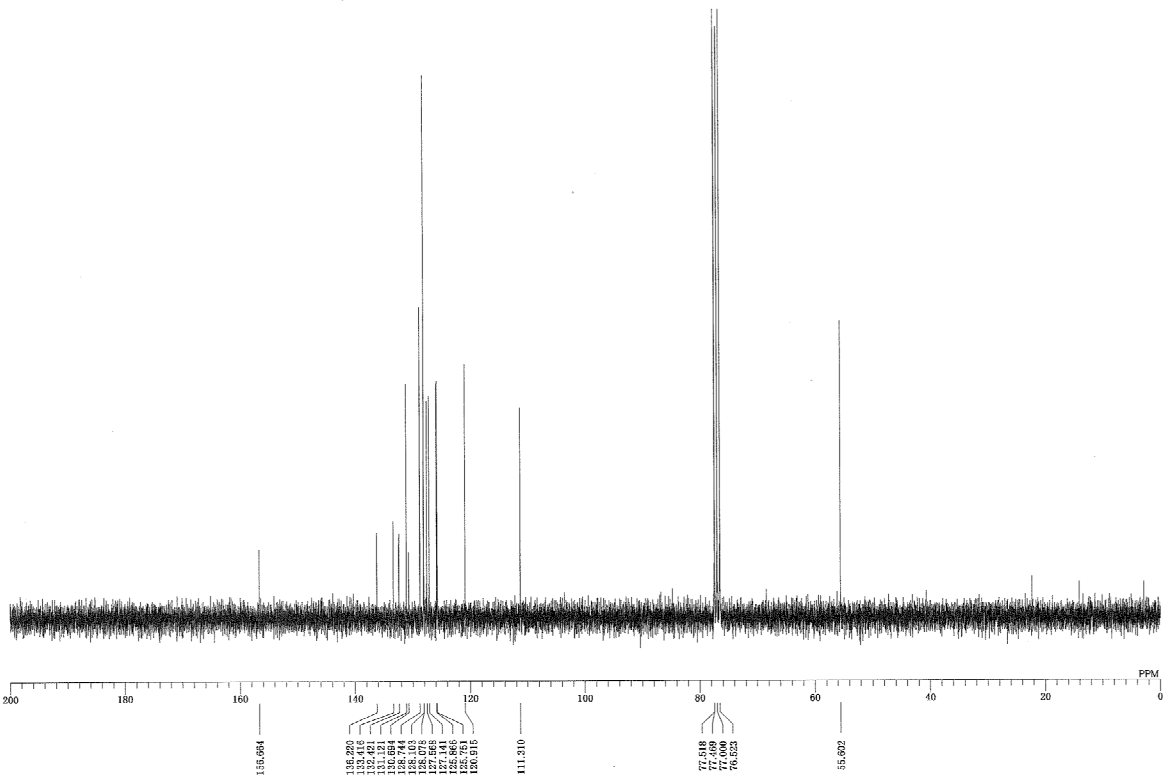
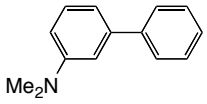
2Nap-2MeOPh-13C





(S9)

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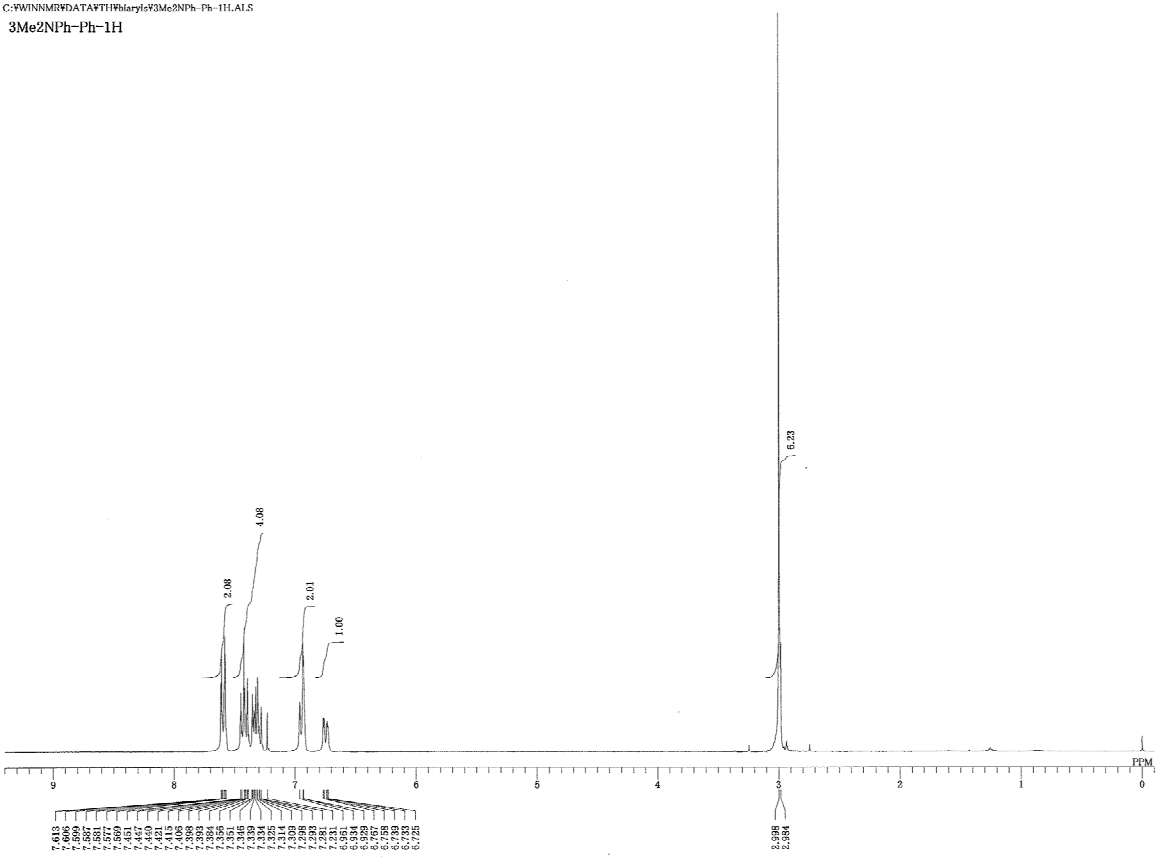
3Me2NPh-Ph-1H



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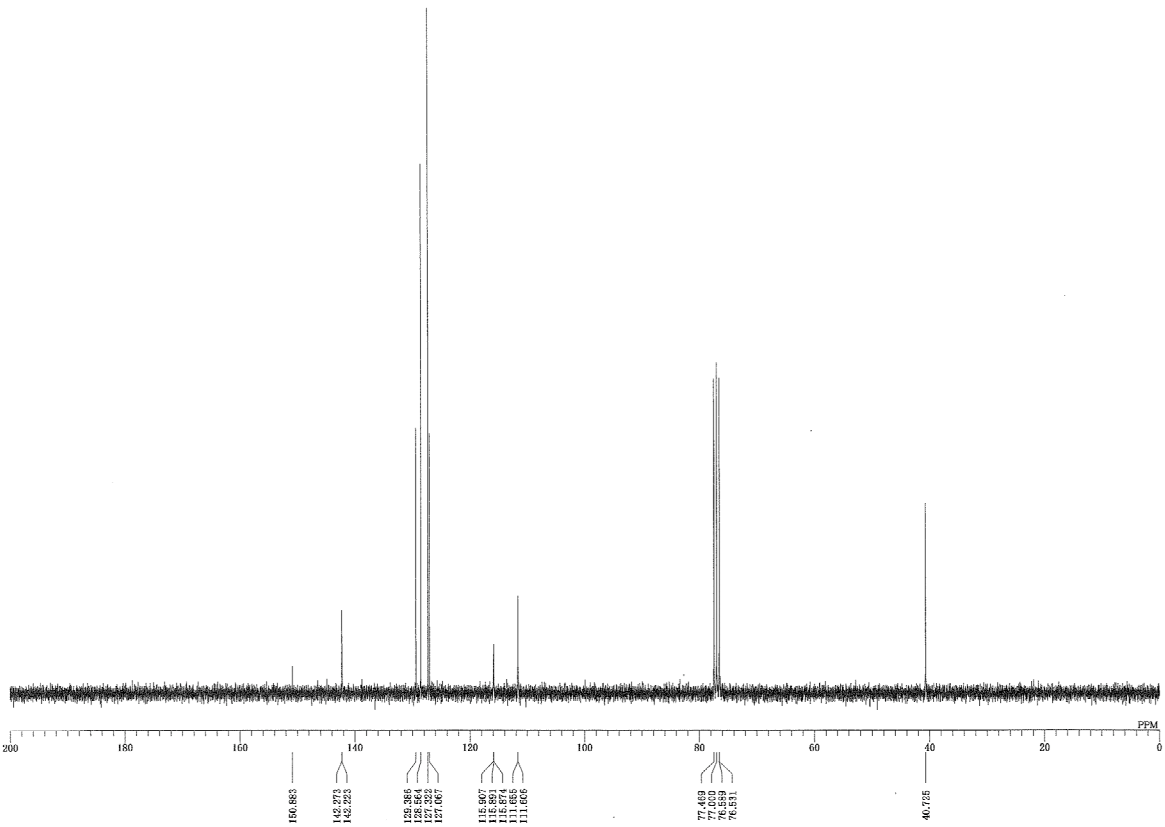
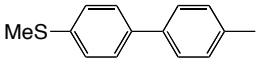
3Me2NPh-Ph-13C





(S10)

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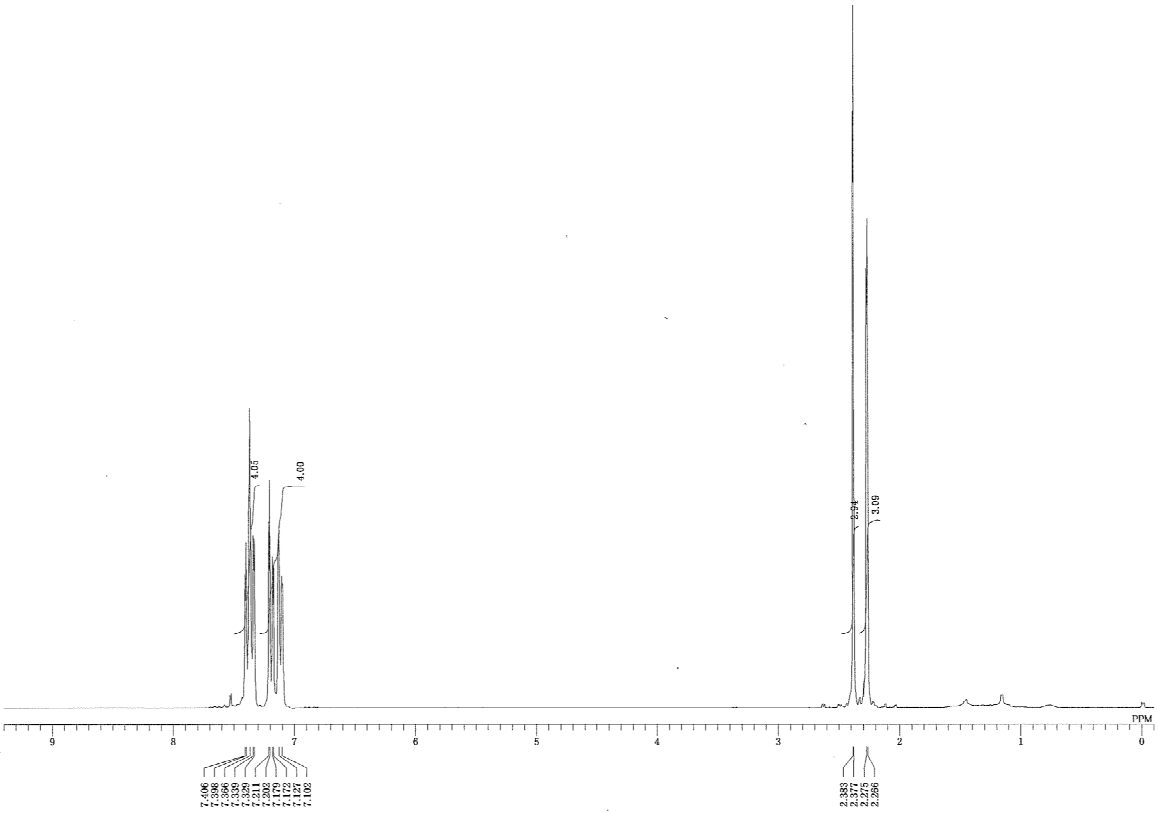
4MeSPh-4MePh-1H



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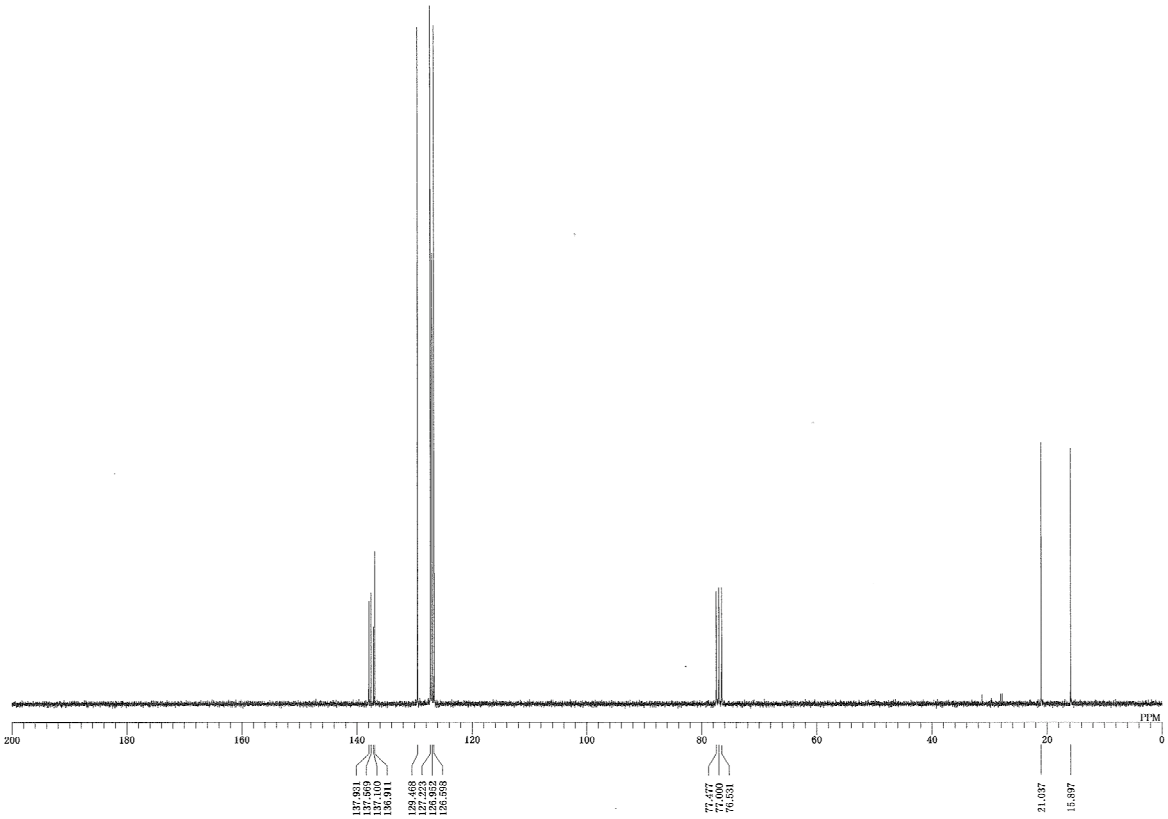
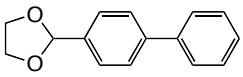
4MeSPh-4MePh-13C





(S11)

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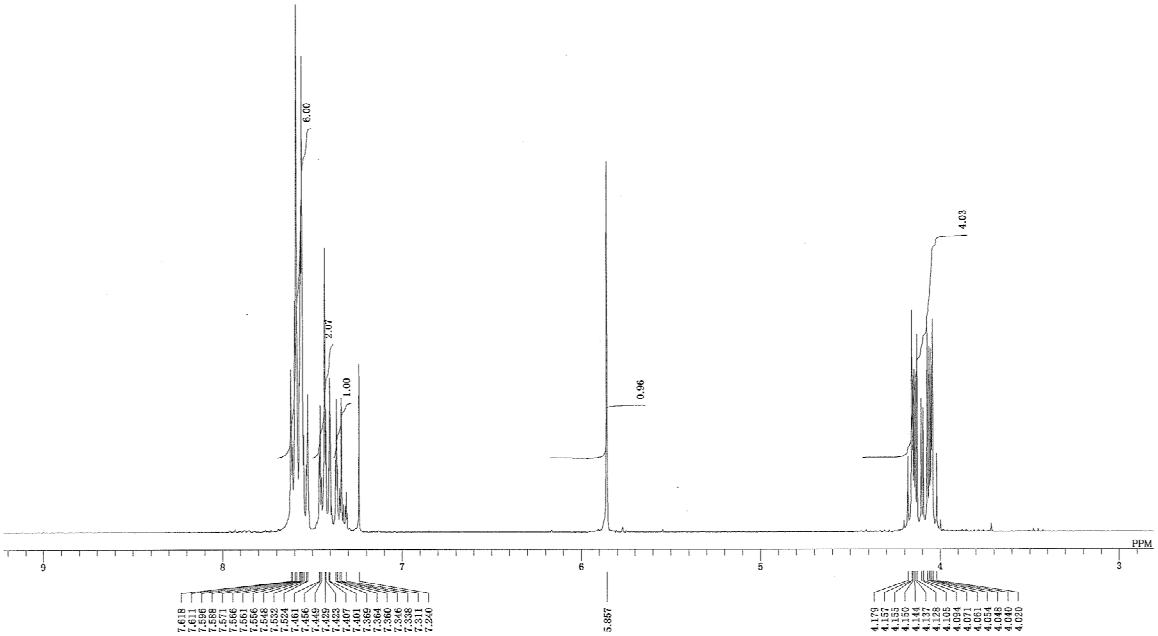
dioxPh-Ph-1H



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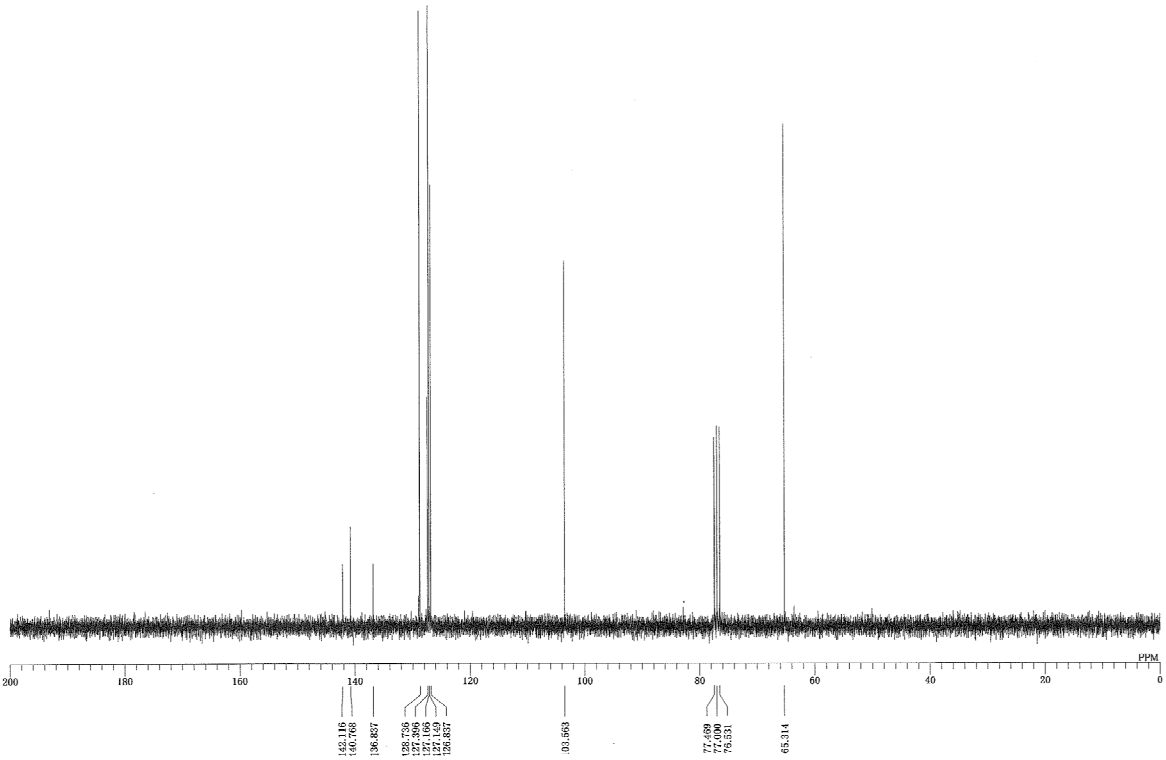

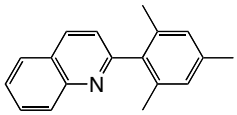

(S12)

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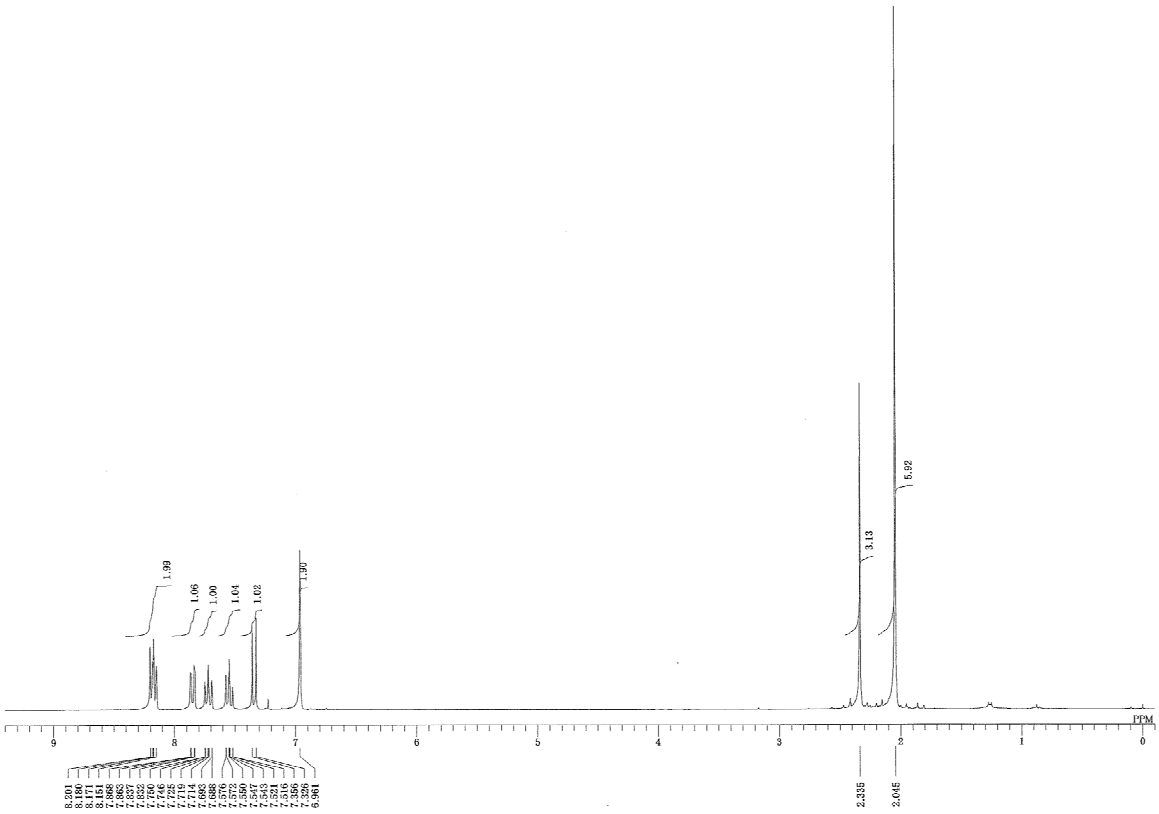
Mes-Quin-1H



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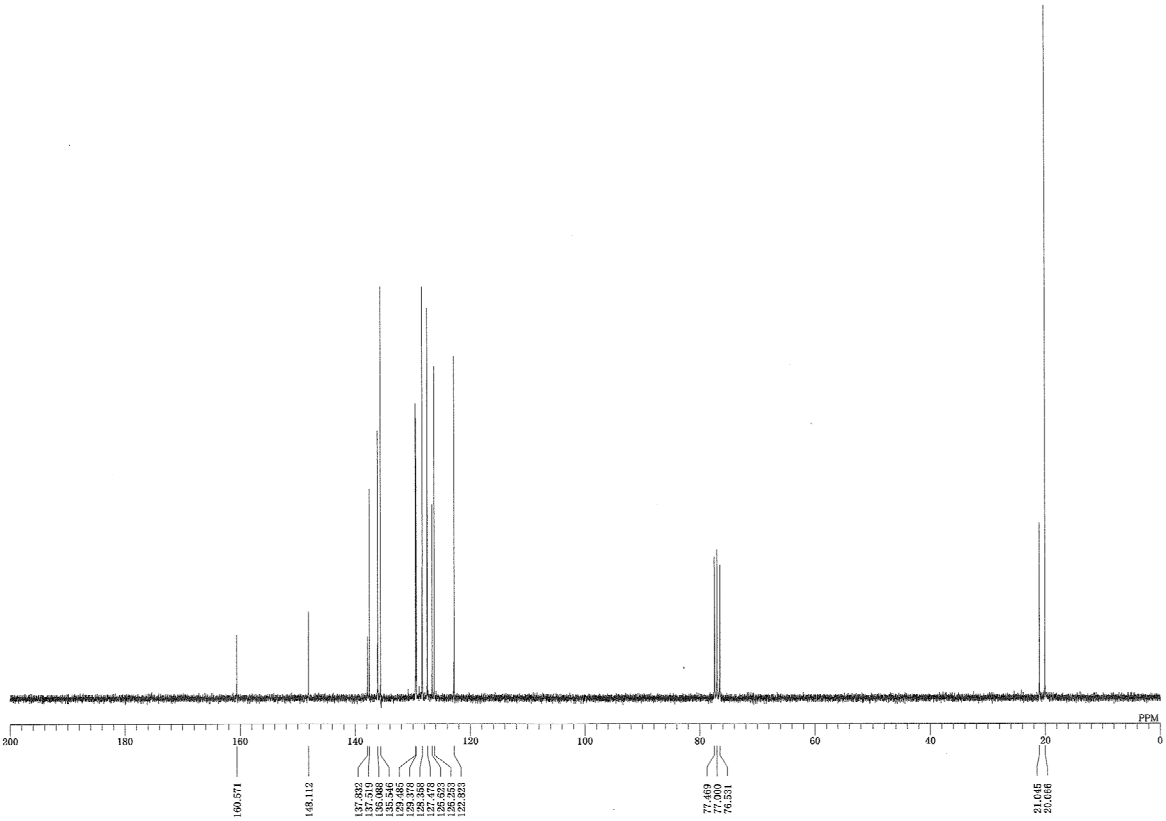
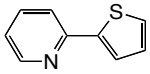
Mes-Quin-13C





(S13)

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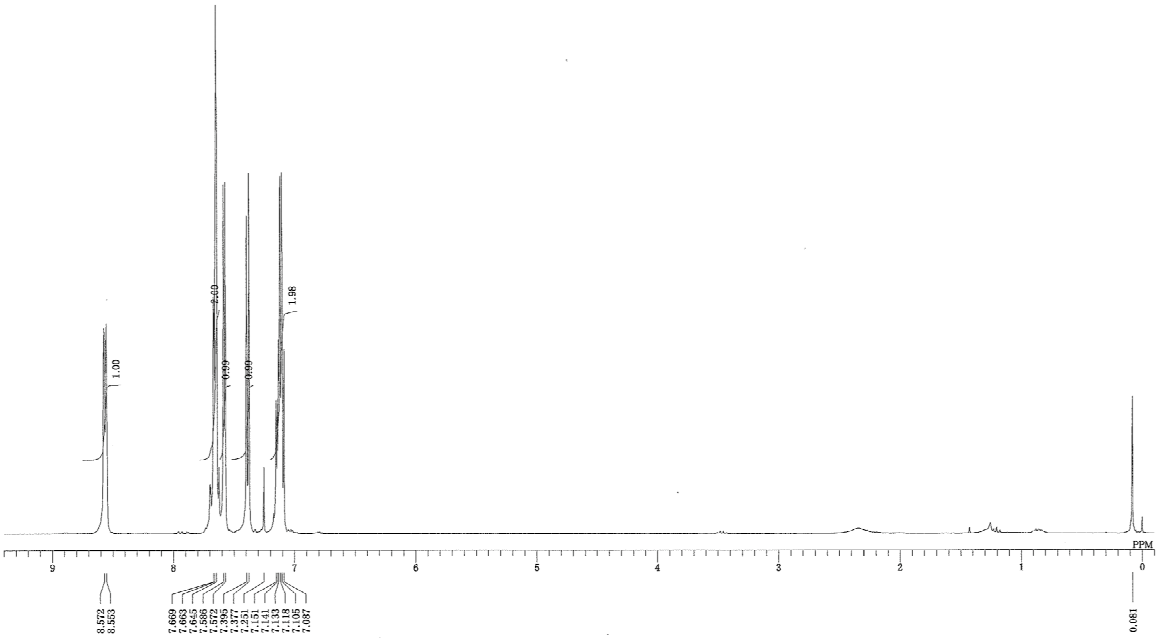
Pyr-Thio-1H



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Pyr-Thio-13C