

Supplementary Information

Rhodium-catalyzed [2+2+2] Cyclization of Various Fluorine-containing Alkynes –Novel Synthesis of Multi-substituted Fluoroalkylated Aromatic Compounds–

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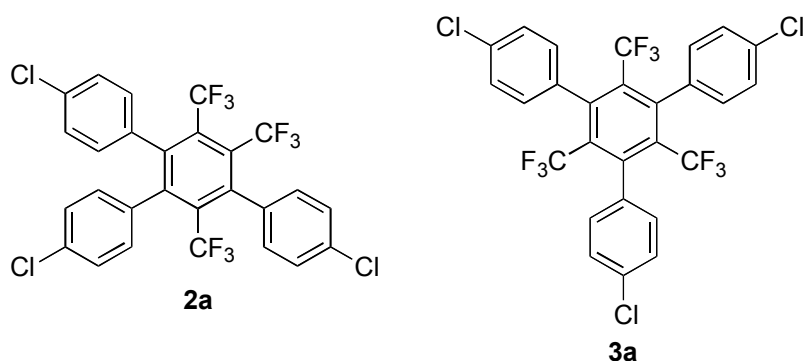
Infrared spectra (IR) were taken on a JASCO FT/IR-4100typeA spectrometer as film on a NaCl plate. ^1H and ^{13}C NMR spectra were measured with a JEOL JNM-AL 400 NMR spectrometer in a chloroform-*d* (CDCl_3) solution with tetramethylsilane (Me_4Si) as an internal reference. A JEOL JNM-EX90A (84.21 MHz) FT-NMR spectrometer and a JEOL JNM-AL 400 NMR spectrometer were used for determining the yield of the products with hexafluorobenzene (C_6F_6). ^{19}F NMR (376.05 MHz) spectra was measured with a JEOL JNM-AL 400 NMR spectrometer in a chloroform-*d* (CDCl_3) solution with trichlorofluoromethane (CFCl_3) as an internal standard. High-resolution mass spectra (HRMS) were taken on a Hitachi M-80B mass spectrometer by electron impact (EI), chemical ionization (CI), and fast atom bombardment (FAB) methods.

All chemicals were of reagent grade and, if necessary, were purified in the usual manner prior to use. Thin-layer chromatography (TLC) was done on aluminium sheets coated with Merck silica gel 60 F₂₅₄ plates, and column chromatography was carried out using Wacogel C-200 as adsorbent.

General procedure for the trimerization of fluoroalkylated alkynes

To a suspension of $\text{RhCl}_3 \cdot 3\text{H}_2\text{O}$ (13 mg, 0.05 mmol) in toluene (1.0 mL) were added *i*-Pr₂NEt (24 mg, 0.15 mmol) and fluoroalkylated alkynes (0.50 mmol), and then the mixture was stirred at the reflux temperature for 18 h. After being cooled to room temperature, the residue was purified by column chromatography on silica gel (EtOAc/Hexane) to afford the corresponding fluoroalkylated benzene derivatives.

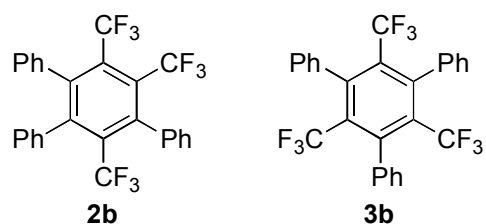
3,5,6-Tris(4-chlorophenyl)-1,2,4-tris(trifluoromethyl)benzene (**2a**) and 2,4,6-Tris(4-chlorophenyl)-1,3,5-tris(trifluoromethyl)benzene (**3a**)



Combined yield : 85% (^{19}F NMR yield), 75% (isolated yield); Isomeric ratio (**2a** : **3a**) = 84:16 (inseparable); HRMS (FAB) calcd for (M^+) $\text{C}_{27}\text{H}_{12}\text{ClF}_9$: 611.9874, Found 611.9861; ^1H NMR (CDCl_3) δ = 6.87 ~ 7.43 (m, 12H); **2a** : ^{19}F NMR (CDCl_3) δ = -51.52 (q, J = 16.2 Hz, 3F), -50.27 (q, J = 16.2 Hz, 3F), -48.36 (s, 3F); **3a** : ^{19}F NMR (CDCl_3) δ = -46.76 (s, 9F).

1,2,4-Tris(trifluoromethyl)-3,5,6-triphenylbenzene (2b) and

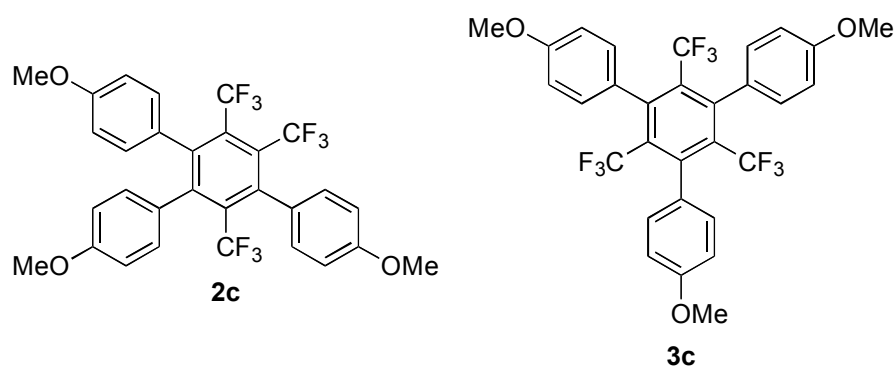
1,3,5-Tris(trifluoromethyl)-2,4,6-triphenylbenzene (3b)



Combined yield : 69% (^{19}F NMR yield), 65% (isolated yield); Isomeric ratio (**2b** : **3b**) = 81:19 (inseparable); HRMS (FAB) calcd for (M^+) $\text{C}_{27}\text{H}_{15}\text{F}_9$: 510.1042, Found 510.1030; ^1H NMR (CDCl_3) δ = 6.95 ~ 7.46 (m, 15H); **2b** : ^{19}F NMR (CDCl_3) δ = -51.48 (q, J = 16.2 Hz, 3F), -50.22 (q, J = 16.2 Hz, 3F), -48.42 (s, 3F); **3b** : ^{19}F NMR (CDCl_3) δ = 46.85 (s, 9F).

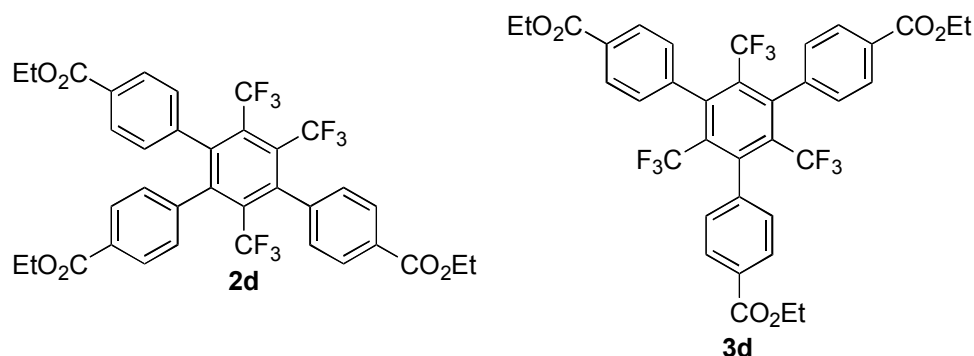
1,2,4-Tris(trifluoromethyl)-3,5,6-tris(4-methoxyphenyl)benzene (2c) and

1,3,5-Tris(trifluoromethyl)-2,4,6-tris(4-methoxyphenyl)benzene (3c)



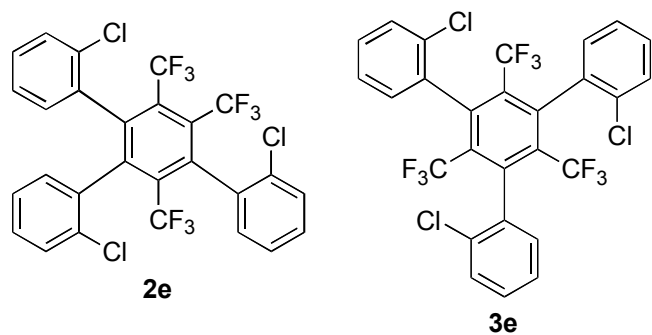
Combined yield : 92% (^{19}F NMR yield), 90% (isolated yield); Isomeric ratio (**2c** : **3c**) = 84:16 (inseparable); HRMS (FAB) calcd for (M^+) $\text{C}_{30}\text{H}_{21}\text{F}_9\text{O}_3$: 600.1337, Found 600.1347; **2c** : ^1H NMR (CDCl_3) δ = 3.73 (s, 3H), 3.74 (s, 3H), 3.84 (s, 3H), 3.87 (s, 3H), 6.34 ~ 6.67 (m, 4H), 6.85 ~ 6.96 (m, 8H); ^{19}F NMR (CDCl_3) δ = -51.63 (q, J = 16.3 Hz, 3F), -50.38 (q, J = 16.3 Hz, 3F), -48.64 (s, 3F); **3c** : ^1H NMR (CDCl_3) δ = 3.73 (s, 3H), 3.74 (s, 3H), 3.84 (s, 3H), 3.87 (s, 3H), 7.18 ~ 7.28 (m, 12H); ^{19}F NMR (CDCl_3) δ = -47.05 (s, 9F).

3,5,6-Tris(4-ethoxycarbonylphenyl)-1,2,4-tris(trifluoromethyl)benzene (2d) and
2,4,6-Tris(4-ethoxycarbonylphenyl)-1,3,5-tris(trifluoromethyl)benzene (3d)



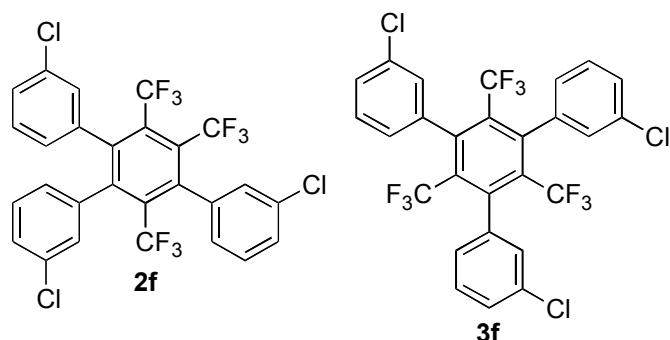
Combined yield : 68% (^{19}F NMR yield), 67% (isolated yield); Isomeric ratio (**2d** : **3d**) = 84:16 (inseparable); HRMS (FAB) calcd for ($\text{M}+\text{H}$) $\text{C}_{36}\text{H}_{28}\text{F}_9\text{O}_6$: 727.1732, Found 727.1742; **2d** : ^1H NMR (CDCl_3) δ = 1.35 ~ 1.45 (m, 9H), 4.30 ~ 4.43 (m, 6H), 7.06 ~ 7.11 (m, 4H), 7.46 ~ 7.48 (m, 2H), 7.80 ~ 7.83 (m, 4H), 8.12 ~ 8.14 (m, 2H); ^{19}F NMR (CDCl_3) δ = -51.43 (q, J = 16.2 Hz, 3F), -50.17 (q, J = 16.2 Hz, 3F), -48.29 (s, 3F); **3d** : ^1H NMR (CDCl_3) δ = 1.35 ~ 1.45 (m, 9H), 4.30 ~ 4.43 (m, 6H), 7.39 ~ 7.41 (m, 6H), 8.07 ~ 8.09 (m, 6H); ^{19}F NMR (CDCl_3) δ = -46.70 (s, 9F).

3,5,6-Tris(2-chlorophenyl)-1,2,4-tris(trifluoromethyl)benzene (2e) and
2,4,6-Tris(2-chlorophenyl)-1,3,5-tris(trifluoromethyl)benzene (3e)



Combined yield : 49% (^{19}F NMR yield), 40% (isolated yield); Isomeric ratio (**2e** : **3e**) = 88:12 (inseparable); HRMS (FAB) calcd for (M^+) $\text{C}_{27}\text{H}_{12}\text{ClF}_9$: 611.9867, Found 611.9861; **2e** : (atropisomer 1) : ^1H NMR (CDCl_3) δ = 7.05 ~ 7.50 (m, 12H); ^{19}F NMR (CDCl_3) δ = -53.04 (q, J = 17.5 Hz, 3F), -52.67 (s, 3F), -52.28 (q, J = 17.5 Hz, 3F); IR (KBr) 2963, 1638, 1435, 1323, 1261, 1235, 1173, 1096, 1022, 802 cm^{-1} ; (atropisomer 2) : ^1H NMR (CDCl_3) δ = 7.04 ~ 7.49 (m, 12H); ^{19}F NMR (CDCl_3) δ = -53.49 (q, J = 17.1 Hz, 3F), -52.77 (s, 3F), -52.35 (q, J = 17.1 Hz, 3F); (atropisomer 3) : ^1H NMR (CDCl_3) δ = 7.04 ~ 7.49 (m, 12H); ^{19}F NMR (CDCl_3) δ = -53.29 (q, J = 18.1 Hz, 3F), -52.80 (s, 3F), -52.24 (q, J = 18.1 Hz, 3F); **3e** : ^1H NMR (CDCl_3) δ = 7.04 ~ 7.49 (m, 12H); (atropisomer 1) : ^{19}F NMR (CDCl_3) δ = -51.29 (s, 9F); (atropisomer 2) : ^{19}F NMR (CDCl_3) δ = -51.21 (s, 9F).

2,4,6-Tris(2-chlorophenyl)-1,3,5-tris(trifluoromethyl)benzene (2f) and
2,4,6-Tris(2-chlorophenyl)-1,3,5-tris(trifluoromethyl)benzene (3f)

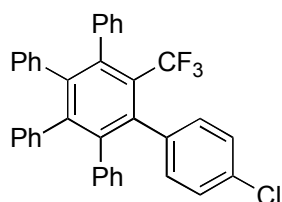


Combined yield : 93% (^{19}F NMR yield), 80% (isolated yield); Isomeric ratio (**2f** : **3f**) = 82:18 (inseparable); HRMS (FAB) calcd for (M^+) $\text{C}_{27}\text{H}_{12}\text{ClF}_9$: 611.9853, Found 611.9861; ^1H NMR (CDCl_3) $\delta = 6.80 \sim 7.47$ (m, 12H); **2f**: ^{19}F NMR (CDCl_3) $\delta = -51.48$ (q, $J = 16.2$ Hz, 3F), -50.26 (q, $J = 16.2$ Hz, 3F), -48.41 (s, 3F); **3f**: ^{19}F NMR (CDCl_3) $\delta = -46.80$ (s, 9F).

General procedure for [2+2+2] cyclization of fluoroalkylated alkynes and non-fluorinated alkynes

To a suspension of $\text{RhCl}_3 \cdot 3\text{H}_2\text{O}$ (13 mg, 0.05 mmol) in toluene (0.5 mL) were added *i*- Pr_2NEt (24 mg, 0.15 mmol) and fluoroalkylated alkynes (0.50 mmol), diphenylacetylene (1.0 mmol, 0.178 g), and then the mixture was stirred at the reflux temperature for 18 h. After being cooled to room temperature, the residue was purified by column chromatography on silica gel (EtOAc/Hexane) to afford the corresponding fluoroalkylated benzene derivatives.

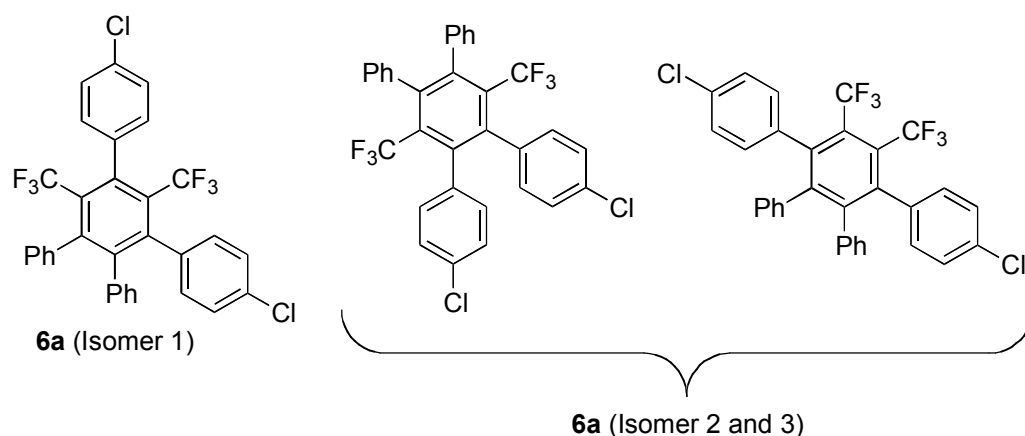
2-(4-Chlorophenyl)-1-trifluoromethyl-3,4,5,6-tetraphenylbenzene (5a)



Yield : 51% (^{19}F NMR yield), 50% (isolated yield); m.p. 176~178 °C; HRMS (FAB) calcd for (M^+) $\text{C}_{37}\text{H}_{24}\text{ClF}_3$: 560.1514, Found 560.1519; ^1H NMR (CDCl_3) $\delta = 6.77 \sim 6.87$ (m, 15H), 7.08 ~ 7.15 (m, 9H); ^{13}C NMR (CDCl_3) $\delta = 125.66, 125.76, 125.87, 126.50, 126.56, 126.63, 126.78, 127.00$ (q, $J = 25.6$

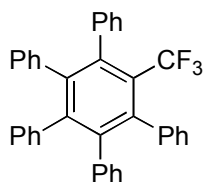
Hz), 127.06, 128.91 (q, $J = 285.9$ Hz), 130.04 (q, $J = 1.7$ Hz), 130.45, 130.79, 130.84, 131.42 (q, $J = 1.6$ Hz), 137.79, 138.78, 138.84 (q, $J = 1.6$ Hz), 138.93, 139.05, 139.25, 140.33 (q, $J = 1.6$ Hz), 142.50, 142.90, 144.51; ^{19}F NMR (CDCl_3) $\delta = -47.96$ (s, 3F); IR (KBr) 1493, 1441, 1407, 1342, 1235, 1176, 1121, 806, 745, 697, 564 cm^{-1} .

2,4-Bis(4-chlorophenyl)-1,3-bis(trifluoromethyl)-5,6-diphenylbenzene,
2,3-Bis(4-chlorophenyl)-1,4-bis(trifluoromethyl)-5,6-diphenylbenzene, and
3,6-Bis(4-chlorophenyl)-1,2-bis(trifluoromethyl)-4,5-diphenylbenzene (6a)



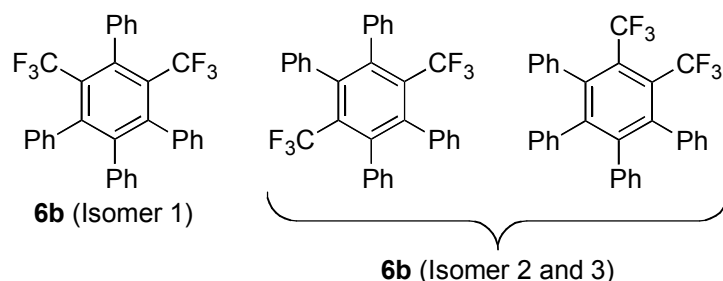
Combined yield : 41% (^{19}F NMR yield), Isomeric ratio = 38:15:47 (inseparable); HRMS (FAB) calcd for (M^+) $\text{C}_{32}\text{H}_{18}\text{Cl}_2\text{F}_6$: 586.0690, Found 586.0690; ^1H NMR (CDCl_3) $\delta = 6.87 \sim 7.43$ (m, 18H); (Isomer 1) : ^{19}F NMR (CDCl_3) $\delta = -47.61$ (s, 3F), -47.44 (s, 3F); (Isomer 2) : ^{19}F NMR (CDCl_3) $\delta = -50.33$ (s, 6F); (Isomer 3) : ^{19}F NMR (CDCl_3) $\delta = -49.01$ (s, 6F).

1-Trifluoromethyl-2,3,4,5,6-pentaphenylbenzene (5b)



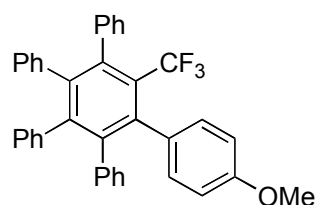
Yield : 47% (^{19}F NMR yield), 32% (isolated yield); m.p. 275-277 $^\circ\text{C}$; HRMS (FAB) calcd for (M^+) $\text{C}_{37}\text{H}_{25}\text{F}_3$: 526.1909, Found 526.1908; ^1H NMR (CDCl_3) $\delta = 6.78 \sim 6.85$ (m, 16H), 7.08 \sim 7.17 (m, 9H); ^{13}C NMR (CDCl_3) $\delta = 125.16, 125.58, 125.66, 126.42, 126.53, 126.55, 126.58, 126.74, 127.15$ (q, $J = 281.1$ Hz), 130.13 (q, $J = 1.7$ Hz), 130.54, 130.93, 131.39, 139.11, 140.15 (q, $J = 1.6$ Hz), 140.59, 142.52; ^{19}F NMR (CDCl_3) $\delta = -48.05$ (s, 3F); IR (KBr) 2962, 2925, 2371, 1735, 1442, 1261, 1097, 1029, 802, 741, 697 cm^{-1} .

**1,3-Bis(trifluoromethyl)-2,4,5,6-tetraphenylbenzene and
1,4-bis(trifluoromethyl)-2,3,5,6-tetraphenylbenzene,
1,2-bis(trifluoromethyl)-3,4,5,6-tetraphenylbenzene (6b)**



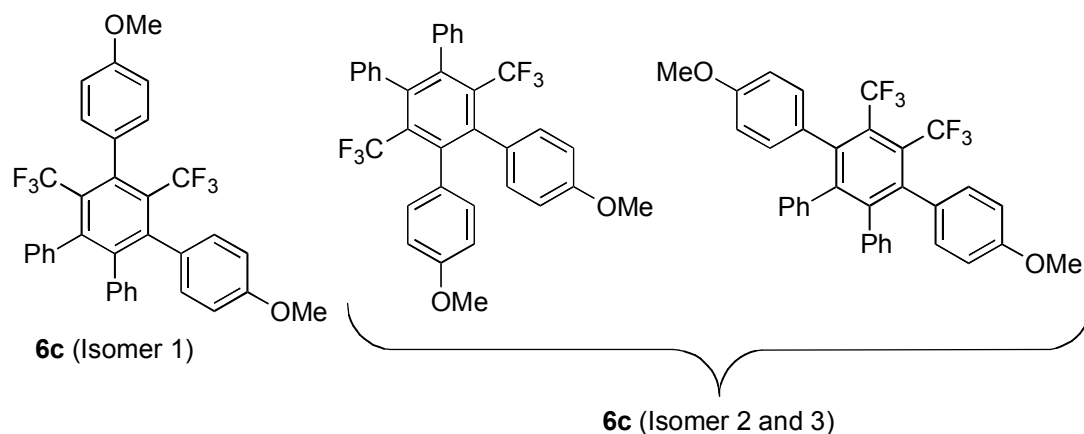
Combined yield : 36% (^{19}F NMR yield); Isomeric ratio = 33:15:52 (inseparable); HRMS (FAB) calcd for (M^+) $\text{C}_{32}\text{H}_{20}\text{F}_6$: 518.1475, Found 518.1469; ^1H NMR (CDCl_3) $\delta = 6.95 \sim 7.46$ (m, 20H); (Isomer 1) : ^{19}F NMR (CDCl_3) $\delta = -47.60$ (s, 6F); (Isomer 2) : ^{19}F NMR (CDCl_3) $\delta = -50.36$ (s, 6F); (Isomer 3) : ^{19}F NMR (CDCl_3) $\delta = -49.00$ (s, 6F).

1-Trifluoromethyl-2-(4-methoxyphenyl)-3,4,5,6-tetraphenylbenzene (5c)



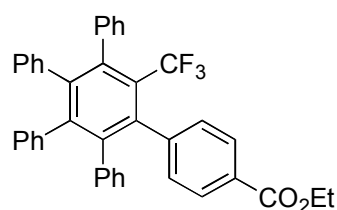
Yield : 45% (^{19}F NMR yield), 43% (isolated yield); m.p. 228~230 °C; HRMS (FAB) calcd for (M^+) $\text{C}_{38}\text{H}_{27}\text{F}_3\text{O}$: 556.2016, Found 556.2014; ^1H NMR (CDCl_3) $\delta = 6.65 \sim 6.67$ (m, 2H), 6.76 ~ 6.85 (m, 15H), 7.05 ~ 7.16 (m, 7H); ^{13}C NMR (CDCl_3) $\delta = 54.95, 124.4$ (q, $J = 277.7$ Hz), 125.54, 125.62, 126.36, 126.50, 126.55, 126.59, 126.73, 127.01 (q, $J = 26.4$ Hz), 130.08 (q, $J = 1.6$ Hz), 130.52, 130.88, 130.93, 131.15, 131.16 (q, $J = 1.7$ Hz), 131.54, 139.15, 139.25, 139.39, 139.54, 140.00 (q, $J = 1.7$ Hz), 140.11 (q, $J = 1.6$ Hz), 142.36, 142.93, 144.27; ^{19}F NMR (CDCl_3) $\delta = -48.04$ (s, 3F); IR (KBr) 1493, 1441, 1407, 1342, 1235, 1176, 1121, 806, 745, 697, 564 cm^{-1} .

**1,3-Bis(trifluoromethyl)-2,4-bis(4-methoxyphenyl)-5,6-diphenylbenzene and
1,4-Bis(trifluoromethyl)-2,3-bis(4-methoxyphenyl)-5,6-diphenylbenzene,
1,2-Bis(trifluoromethyl)-3,6-bis(4-methoxyphenyl)-4,5-diphenylbenzene (6c)**



Combined yield : 37% (^{19}F NMR yield); Isomeric ratio = 49:13:38 (inseparable); HRMS (FAB) calcd for (M^+) $\text{C}_{34}\text{H}_{24}\text{F}_6\text{O}_2$: 578.1678, Found 578.1680; (Isomer 1) : ^{19}F NMR (CDCl_3) $\delta = -47.79$ (s, 3F), -47.64 (s, 3F); (Isomer 2) : ^{19}F NMR (CDCl_3) $\delta = -50.50$ (s, 6F); (Isomer 3) : ^{19}F NMR (CDCl_3) $\delta = -49.05$ (s, 6F).

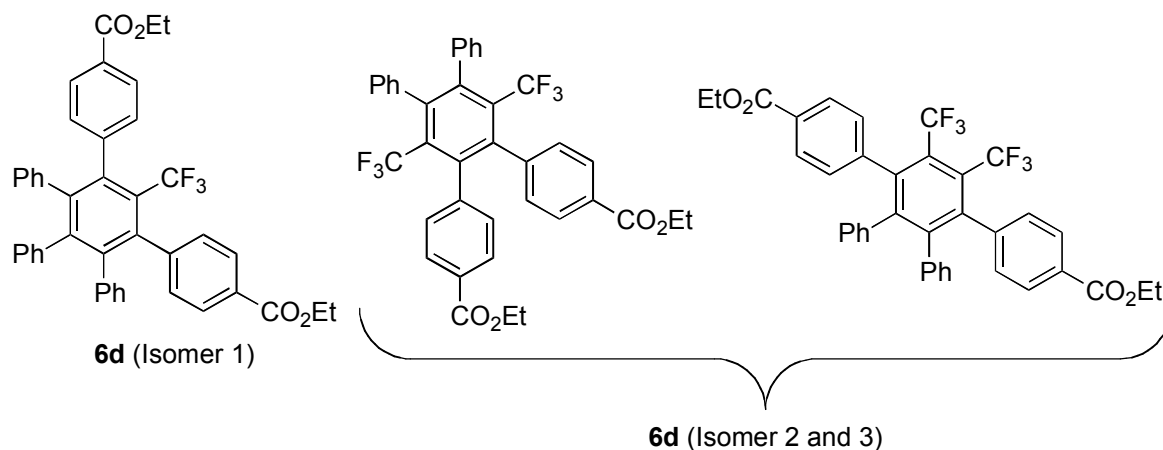
2-(4-ethoxycarbonylphenyl)-1-trifluoromethyl-3,4,5,6-tetraphenylbenzene (5d)



Combined yield : m.p. 228~230 °C; 45%; HRMS (FAB) calcd for (M^+) $\text{C}_{40}\text{H}_{29}\text{F}_3\text{O}_2$: 598.2116, Found 598.2120; ^1H NMR (CDCl_3) $\delta = 1.36$ (t, $J = 7.6$ Hz, 3H), 4.31 (q, $J = 7.2$ Hz, 2H), 6.77 ~ 6.84 (m, 16H), 7.10 ~ 7.14 (m, 4H), 7.25 ~ 7.26 (m, 2H), 7.81 ~ 7.83 (m, 2H); ^{13}C NMR (CDCl_3) $\delta = 14.23$, 60.80, 124.22 (q, $J = 276.9$ Hz), 125.66, 125.76, 125.90, 126.39, 126.51, 126.61, 126.73, 126.77, 128.08, 128.46, 130.05 (q, $J = 1.7$ Hz), 130.17 (q, $J = 1.6$ Hz), 130.44, 130.77, 130.82, 131.36, 138.59, 138.94, 139.04 (q, $J = 1.7$ Hz), 139.17, 140.34 (q, $J = 1.7$ Hz), 142.12, 142.99, 144.27, 144.53, 166.44; ^{19}F NMR (CDCl_3) $\delta = -48.00$ (s, 3F); IR (KBr) 3057, 2962, 2371, 1719, 1610, 1497, 1442, 1343, 1273, 1175, 1123, 1023

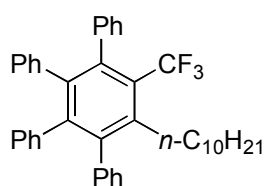
cm⁻¹.

2,4-Bis(4-ethoxycarbonylphenyl)-1,3-bis(trifluoromethyl)-5,6-diphenylbenzene,
2,4-Bis(4-ethoxycarbonylphenyl)-1,3-bis(trifluoromethyl)-5,6-diphenylbenzene, and
2,4-Bis(4-ethoxycarbonylphenyl)-1,3-bis(trifluoromethyl)-5,6-diphenylbenzene (6d)



Combined yield : 32%; Isomeric ratio = 37:16:47; HRMS (FAB) calcd for (M+H) C₃₈H₂₉F₆O₄: 663.1967, Found 663.1970; (Isomer 1) : ¹⁹F NMR (CDCl₃) δ = -47.57 (s, 3F), -47.37 (s, 3F); (Isomer 2) : ¹⁹F NMR (CDCl₃) δ = -50.24 (s, 6F); (Isomer 3) : ¹⁹F NMR (CDCl₃) δ = -48.97 (s, 6F).

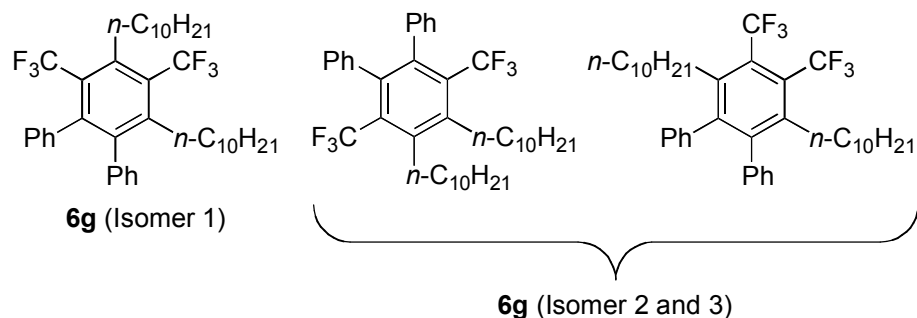
2-(Decyl)-1-trifluoromethyl-3,4,5,6-tetraphenylbenzene (5g)



Yield : 41% (¹⁹F NMR yield), 33% (isolated yield); m.p. 106~108 °C; HRMS (FAB) calcd for (M⁺) C₄₁H₄₁F₃: 590.3157, Found 590.3160; ¹H NMR (CDCl₃) δ = 0.88 (t, *J* = 7.0 Hz, 3H), 1.03 ~ 1.47 (m, 16H), 2.68 ~ 2.75 (m, 2H), 6.66 ~ 6.81 (m, 9H), 7.04 ~ 7.17 (m, 11H); ¹³C NMR (CDCl₃) δ = 14.10, 22.67, 28.77, 29.28, 29.31, 29.49, 29.92, 31.29 (q, *J* = 1.5 Hz), 31.79 (q, *J* = 2.8 Hz), 31.89, 125.45, 126.25 (q, *J* = 23.5 Hz), 126.41, 126.41 (q, *J* = 275.9 Hz), 126.47, 126.69, 127.28, 129.87 (q, *J* = 1.8 Hz), 130.02, 130.38, 130.42, 130.56, 131.01, 131.23, 131.50, 139.31, 139.55, 140.24 (q, *J* = 1.4 Hz), 140.33 (q, *J* = 1.5 Hz), 140.68, 143.06, 144.61; ¹⁹F NMR (CDCl₃) δ = -49.77 (s, 3F); IR (KBr) 2371, 1654, 1542,

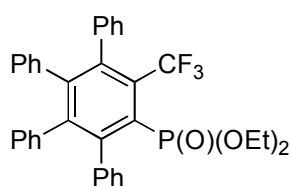
1509, 1117 cm^{-1} .

**2,6-Didodecyl-1,3-bis(trifluoromethyl)-4,5-diphenylbenzene,
5,6-didodecyl-1,4-bis(trifluoromethyl)-2,3-diphenylbenzene, and
3,6-didodecyl-1,2-bis(trifluoromethyl)-3,4-diphenylbenzene (6g)**



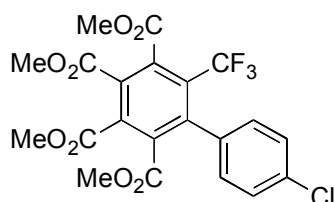
Combined yield : 32% (^{19}F NMR yield); Isomeric ratio = 31:13:56 (inseparable); ^{19}F NMR (CDCl_3) δ = -51.11 (s, 3F), -49.05 (s, 3F); (Isomer 2) : ^{19}F NMR (CDCl_3) δ = -52.49 (s, 3F); (Isomer 3) : ^{19}F NMR (CDCl_3) δ = -50.44 (s, 3F).

Diethyl (1-trifluoromethyl-2,3,4,5-tetraphenyl)phenylphosphonate (5i)



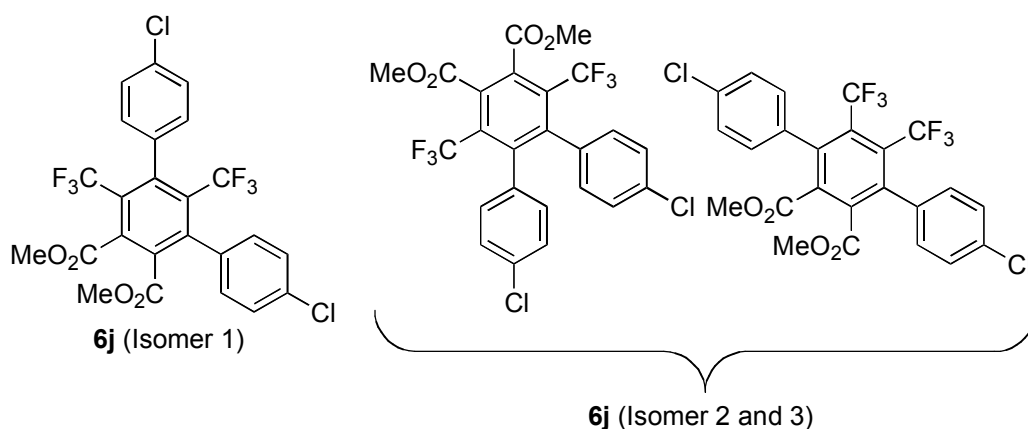
Yield : 16% (^{19}F NMR yield); HRMS (FAB) calcd for (M+H) $\text{C}_{35}\text{H}_{31}\text{F}_3\text{O}_3\text{P}$: 587.1965, Found 587.1963; ^1H NMR (CDCl_3) δ = 1.17 (t, J = 6.8 Hz, 6H), 3.54 ~ 3.61 (m, 2H), 3.88 ~ 3.96 (m, 2H), 6.54 ~ 6.85 (m, 4H), 6.79 ~ 6.85 (m, 6H), 7.02 ~ 7.14 (m, 10H); ^{13}C NMR (CDCl_3) δ = 16.02, 16.09, 61.90, 61.97, 125.75, 125.80, 126.49 (q, J = 271.9 Hz), 126.71, 126.74, 126.82, 126.87, 126.93, 130.52, 130.75, 131.34, 138.41, 138.52, 140.06, 140.11, 141.61, 141.72, 144.54, 144.68, 145.83, 145.87, 146.34, 146.42; ^{19}F NMR (CDCl_3) δ = -49.09 (s, 3F); ^{31}P NMR (CDCl_3) δ = -109.30; IR (KBr) 2985, 2371, 1637, 1492, 1443, 1393, 1334, 1254, 1227, 1161, 1130, 1058, 1021, 966, 872 cm^{-1} .

6-(4-Chlorophenyl)-1-trifluoromethyl-2,3,4,5-tetramethoxycarbonylbenzene (5j)



Yield : 17% (^{19}F NMR yield), 11% (isolated yield); HRMS (FAB) calcd for (M^+) $\text{C}_{21}\text{H}_{16}\text{F}_3\text{ClO}_8$: 488.0482, Found 488.0486; ^1H NMR (CDCl_3) δ = 3.50 (s, 3H), 3.87 (s, 3H), 3.90 (s, 3H), 3.94 (s, 3H), 7.17 (d, J = 8.4 Hz, 2H), 7.37 (d, J = 8.8 Hz, 2H); ^{13}C NMR (CDCl_3) δ = 52.74, 53.52, 122.09 (q, J = 276.0 Hz), 127.76, 129.14 (q, J = 30.6 Hz), 130.81, 131.62, 132.41, 133.05, 133.86, 133.88, 135.19, 137.62, 141.30, 141.31, 164.93, 165.19, 165.52, 165.81; ^{19}F NMR (CDCl_3) δ = -53.88 (s, 3F); IR (KBr) 2345, 1736, 1638, 1442, 1257, 1227 cm^{-1} .

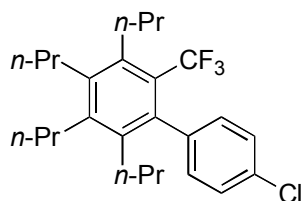
2,6-Bis(4-chlorophenyl)-1,3-bis(trifluoromethyl)-4,5-dimethoxycarbonylbenzene, 5,6-Bis(4-chlorophenyl)-1,4-bis(trifluoromethyl)-2,3-dimethoxycarbonylbenzene, and 3,6-Bis(4-chlorophenyl)-1,2-bis(trifluoromethyl)-4,5-dimethoxycarbonylbenzene (6j)



Combined yield : 17% (^{19}F NMR yield); Isomeric ratio = 35:24:41 (inseparable); HRMS (FAB) calcd for (M^+) $\text{C}_{24}\text{H}_{14}\text{F}_6\text{Cl}_2\text{O}_4$: 550.0176, Found 550.0173; ^1H NMR (CDCl_3) δ = 1.25 ~ 1.39 (m, 4H), 3.49 ~ 3.96 (m, 6H), 5.72 ~ 7.43 (m, 18H); (Isomer 1) : ^{19}F NMR (CDCl_3) δ = -52.67 (s, 3F), -48.46 (s, 3F); (Isomer

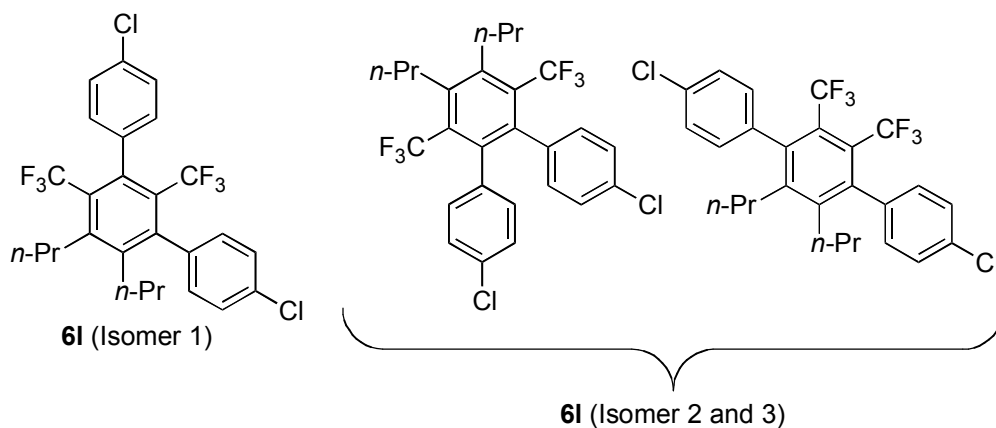
2) : ^{19}F NMR (CDCl_3) $\delta = -53.88$ (s, 3F); (Isomer 3) : ^{19}F NMR (CDCl_3) $\delta = -50.84$ (s, 3F).

2-(4-Chlorophenyl)-1-trifluoromethyl-3,4,5,6-tetrapropylbenzene (5I)



Yield : 21% (^{19}F NMR yield), 11% (isolated yield); HRMS (FAB) calcd for (M^+) $\text{C}_{25}\text{H}_{32}\text{ClF}_3$: 424.2149, Found 424.2145; ^1H NMR (CDCl_3) $\delta = 1.06$ (t, $J = 7.6$ Hz, 12H), 1.50 ~ 1.60 (m, 10H), 2.46 ~ 2.50 (m, 6H), 7.09 (d, $J = 8.8$ Hz, 2H), 7.33 (d, $J = 8.4$ Hz, 2H); ^{19}F NMR (CDCl_3) $\delta = -49.41$ (s, 3F).

**2,6-Bis(4-chlorophenyl)-1,3-trifluoromethyl-4,5-dipropylbenzene,
2,3-Bis(4-chlorophenyl)-1,4-trifluoromethyl-5,6-dipropylbenzene, and
3,6-Bis(4-chlorophenyl)-1,2-trifluoromethyl-4,5-dipropylbenzene (6I)**



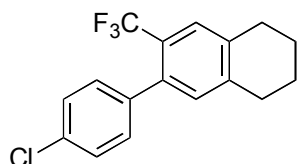
Combined yield : 69% (^{19}F NMR yield); ^{19}F NMR (CDCl_3) $\delta = -49.18$ (s, 3F), -47.25 (s, 3F); (Isomer 2) : ^{19}F NMR (CDCl_3) $\delta = -50.56$ (s, 3F); (Isomer 3) : ^{19}F NMR (CDCl_3) $\delta = -50.44$ (s, 3F).

General procedure for [2+2+2] cycloaddition of fluoroalkylated alkynes and non-fluorinated diynes

To a suspension of $\text{RhCl}_3 \cdot 3\text{H}_2\text{O}$ (13 mg, 0.05 mmol) in toluene (0.5 mL) were added *i*- Pr_2NEt (24 mg, 0.15 mmol) and fluoroalkylated alkynes (0.50 mmol), non-fluorinated diynes (0.5 mmol). The

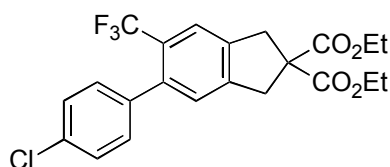
mixture was stirred at the reflux temperature for 18 h. After being cooled to room temperature, the residue was purified by column chromatography on silica gel (EtOAc/Hexane) to afford the corresponding fluoroalkylated benzene derivatives.

7-Trifluoromethyl-8-(4-chlorophenyl)-1,2,3,4-tetrahydronaphthalene (7a)



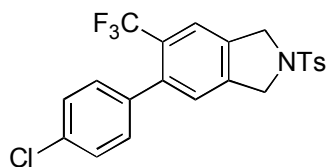
Yield : 15% (^{19}F NMR yield); ^1H NMR (CDCl_3) δ = 1.78 ~ 1.86 (m, 4H), 2.80 ~ 2.84 (m, 4H), 6.98 (s, 1H), 7.22 ~ 7.25 (m, 2H), 7.33 ~ 7.37 (m, 2H), 7.43 (s, 1H); ^{19}F NMR (CDCl_3) δ = -57.03 (s, 3F).

Diethyl 6-(4-chlorophenyl)-5-trifluoromethyl-1H-indene-2,2(3H)-dicarboxylate (8a)



Yield : 20% (^{19}F NMR yield); HRMS (FAB) calcd for (M+H) $\text{C}_{22}\text{H}_{21}\text{F}_3\text{ClO}_4$: 441.1080, Found 440.1071; ^1H NMR (CDCl_3) δ = 1.30 (t, J = 7.2 Hz, 6H), 3.65 (s, 2H), 3.69 (s, 2H), 4.25 (q, J = 6.8 Hz, 4H), 7.13 (s, 1H), 7.24 (d, J = 8.4 Hz, 2H), 7.37 (d, J = 8.4 Hz, 2H), 7.58 (s, 1H); ^{13}C NMR (CDCl_3) δ = 13.97, 40.08, 40.26, 60.30, 61.98, 121.96 (q, J = 5.8 Hz), 124.10 (q, J = 273.6 Hz), 127.37 (q, J = 30.6 Hz), 127.67, 127.87; ^{19}F NMR (CDCl_3) δ = -57.02 (s, 3F); IR (KBr) 3467, 2982, 2318, 1906, 1732, 1597, 1485, 1297, 1135, 911 cm^{-1} .

7-(4-Chlorophenyl)-6-trifluoromethyl-2-p-toluenesulfonylisindoline (9a)



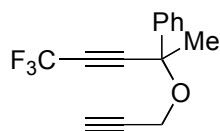
Yield : 21% (^{19}F NMR yield); m.p. 192~193 °C; HRMS (FAB) calcd for (M+H) $\text{C}_{22}\text{H}_{18}\text{ClF}_3\text{NO}_2\text{S}$: 452.0696, Found 452.0699; ^1H NMR (CDCl_3) δ = 2.42 (s, 3H), 4.67 (s, 2H), 4.69 (s, 2H), 7.10 (s, 1H),

7.17 (d, $J = 8.4$ Hz, 2H), 7.33 ~ 7.36 (m, 4H), 7.55 (s, 1H), 7.79 (d, $J = 8.4$ Hz, 2H); ^{13}C NMR (CDCl_3) $\delta = 21.78, 53.64, 53.73, 120.94$ (q, $J = 5.7$ Hz), 124.19 (q, $J = 273.6$ Hz), $126.50, 127.87, 128.34, 130.24, 130.49, 130.50, 133.65, 133.34, 136.32, 137.78, 140.21, 140.43, 144.32$; ^{19}F NMR (CDCl_3) $\delta = -57.26$ (s, 3F); IR (KBr) $3449, 2370, 1638, 1342, 1158, 1102$ cm^{-1} .

General procedure for the synthesis of fluoroalkylated diynes

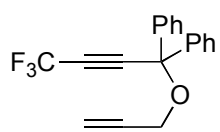
To stirred and cooled (0 °C) solution of γ -fluoroalkylated propargyl alcohol (1.0 mmol), propargyl bromide (0.179 g, 1.5 mmol), and tetrabutylammonium iodide (0.018 g, 0.05 mmol) in CH_2Cl_2 (2 mL) was added slowly a solution of NaOH (0.006 g, 1.5 mmol) in water (1 mL). The cooling bath was removed and the mixture was vigorously stirred at room temperature until completion of the reaction (5 d). The organic layer was extracted with CH_2Cl_2 , and the combined CH_2Cl_2 extracts were washed with brine, dried (Na_2SO_4) and filtered. The filtrate was concentrated under reduced pressure and the residue was purified by column chromatography on silica gel (EtOAc/Hexane) to afford the corresponding fluoroalkylated diynes.

4,4,4-Trifluoro-1-methyl-1-phenyl-2-butyn-1-yl 2-propyn-1-yl ether (10A)



Yield : 80% (^{19}F NMR yield), 64% (isolated yield); HRMS (EI) calcd for (M-CH₃) $\text{C}_{13}\text{H}_8\text{F}_3\text{O}$: 237.0521, Found 237.0527; ^1H NMR (CDCl_3) $\delta = 1.83$ (s, 3H), 2.45 (t, $J = 2.4$ Hz, 1H), 3.86 (dd, $J = 2.4, 15.2$ Hz, 1H), 4.16 (dd, $J = 2.4, 14.4$ Hz, 1H), 7.37 ~ 7.44 (m, 3H), 7.53 ~ 7.55 (m, 2H); ^{13}C NMR (CDCl_3) $\delta = 31.98, 31.99, 53.84, 74.46, 76.30, 79.26, 86.58$ (q, $J = 5.8$ Hz), 114.03 (q, $J = 257.9$ Hz), $125.55, 128.84, 128.87, 139.54$; ^{19}F NMR (CDCl_3) $\delta = -50.82$ (s, 3F); IR (neat) $3312, 2963, 2271, 1703, 1492, 1448, 1375, 1279, 1149, 1096, 1046, 804, 765, 700, 501$ cm^{-1} .

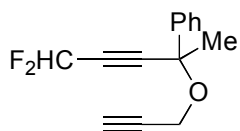
4,4,4-Trifluoro-1,1-diphenyl-2-butyn-1-yl 2-propyn-1-yl ether (10B)



Yield : 40% (^{19}F NMR yield), 21% (isolated yield); m.p. $31\sim 32$ °C; HRMS (FAB) calcd for (M⁺)

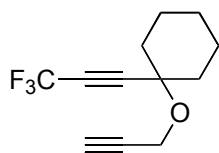
C₁₉H₁₃F₃O: 314.0912, Found 314.0918; ¹H NMR (CDCl₃) δ = 2.46 (t, *J* = 2.4 Hz, 1H), 4.16 (d, *J* = 2.4 Hz, 2H), 7.31 ~ 7.39 (m, 6H), 7.47 ~ 7.50 (m, 4H); ¹³C NMR (CDCl₃) δ = 53.91, 74.59, 76.45, 79.15, 80.27 (q, *J* = 1.7 Hz), 85.96 (q, *J* = 5.8 Hz), 114.08 (q, *J* = 257.8 Hz), 126.59, 128.60, 128.66, 140.40; ¹⁹F NMR (CDCl₃) δ = -50.83 (s, 3F); IR (neat) 3300, 3063, 3031, 2925, 2867, 2258, 1957, 1726, 159, 1492, 1450, 1375, 1270, 1216, 1146, 1063, 826 cm⁻¹.

4,4-Difluoro-1-methyl-1-phenyl-2-butyn-1-yl 2-propyn-1-yl ether (10C)



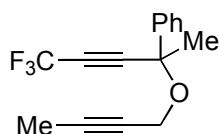
Yield : 68% (¹⁹F NMR yield), 50% (isolated yield); HRMS (EI) calcd for (M⁺) C₁₄H₁₂F₂O: 234.0863, Found 234.0856; ¹H NMR (CDCl₃) δ = 1.70 (s, 3H), 2.32 (t, *J* = 2.4 Hz, 1H), 3.73 (dd, *J* = 2.4, 14.4 Hz, 1H), 4.08 (dd, *J* = 2.4, 14.8 Hz, 1H), 6.24 (t, *J* = 54.4 Hz, 1H), 7.23 ~ 7.31 (m, 3H), 7.45 ~ 7.47 (m, 2H); ¹³C NMR (CDCl₃) δ = 32.06 (t, *J* = 1.6 Hz), 53.51, 74.18, 76.31 (t, *J* = 1.6 Hz), 79.15 (t, *J* = 33.9 Hz), 79.52, 88.09 (t, *J* = 7.4 Hz), 103.50 (t, *J* = 232.2 Hz), 125.58, 128.52, 128.64, 140.13 (t, *J* = 1.6 Hz); ¹⁹F NMR (CDCl₃) δ = -106.68 (d, *J* = 53.8 Hz, 2F); IR (neat) 3306, 3064, 3030, 2993, 2934, 2866, 2260, 2131, 1602, 1494, 1447, 1191, 1117, 1013 cm⁻¹.

1-(3,3-Trifluoropropynyl)-1-prop-2-ynyloxy-cyclohexane (10D)



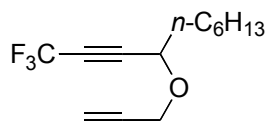
Yield : 70% (¹⁹F NMR yield), 15% (isolated yield); ¹H NMR (CDCl₃) δ 1.25 ~ 1.34 (m, 1H), 1.44 ~ 1.55 (m, 3H), 1.68 ~ 1.74 (m, 4H), 1.95 ~ 1.98 (m, 2H), 2.45 (t, *J* = 2.4 Hz, 2H), 4.26 (d, *J* = 2.4 Hz, 2H); ¹³C NMR (CDCl₃) δ 22.30, 24.84, 24.85, 36.19, 52.20, 52.22, 73.55 (q, *J* = 2.5 Hz), 74.07 (q, *J* = 2.5 Hz), 74.24, 79.93, 87.91 (q, *J* = 6.6 Hz), 114.02 (q, *J* = 257.1 Hz); ¹⁹F NMR (CDCl₃) δ -50.55 (s, 3F); IR (neat) 3314, 2942, 2864, 2267, 1451, 1362, 1298, 1266, 1217, 1145, 1081, 1027, 938, 906, 861, 813 cm⁻¹.

4,4,4-Trifluoro-1-methyl-1-phenyl-2-butyn-1-yl 2-butyn-1-yl ether (10E)



Yield : 48% (^{19}F NMR yield) (This compound could not be given in a pure form. A small amount of unidentified materials was contaminated.); HRMS (CI) calcd for (M+H) $\text{C}_{15}\text{H}_{14}\text{F}_3\text{O}$: 267.0993, Found 267.0997; ^1H NMR (CDCl_3) δ = 1.82 (s, 3H), 1.85 (t, J = 2.4 Hz, 3H), 3.81 (dd, J = 2.4, 14.4 Hz, 1H), 4.12 (dd, J = 2.0, 14.2 Hz, 1H), 7.33 ~ 7.43 (m, 3H), 7.52 ~ 7.54 (m, 2H); ^{19}F NMR (CDCl_3) δ = -50.71 (s, 3F).

4,4,4-Trifluoro-1-hexyl-2-butyn-1-yl 2-propyn-1-yl ether (10F)

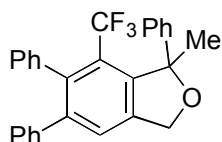


Yield : 57% (^{19}F NMR yield) (This compound could not be given in a pure form. A small amount of unidentified materials was contaminated.); HRMS (EI) calcd for (M+) $\text{C}_{13}\text{H}_{17}\text{F}_3\text{O}$: 246.1228, Found 246.1231; ^1H NMR (CDCl_3) δ = 1.27 ~ 1.36 (m, 7H), 1.43 ~ 1.50 (m, 3H), 1.73 ~ 1.85 (m, 3H), 2.46 (t, J = 2.5 Hz, 1H), 4.22 (dd, J = 2.5, 15.8 Hz, 1H), 4.33 (dd, J = 2.5, 15.8 Hz, 1H), 4.38 ~ 4.42 (m, 1H); ^{19}F NMR (CDCl_3) δ = -50.80 (s, 3F).

General procedure for fluoroalkylated 1,3-dihydroisobenzofuran derivatives

To a suspension of $\text{RhCl}_3 \cdot 3\text{H}_2\text{O}$ (8 mg, 0.03 mmol) were added *i*- Pr_2NEt (14 mg, 0.09 mmol) and fluoroalkylated diynes (0.30 mmol), diphenylacetylene (0.530 g, 3.0 mmol). The mixture was stirred at 130 °C (bath temp.) for 2 h. After being cooled to room temperature, the residue was purified by column chromatography on silica gel to afford corresponding fluoroalkylated 1,3-dihydroisobenzofuran derivatives.

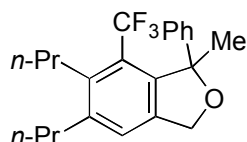
5-Trifluoromethyl-3-methyl-3,6,7-triphenyl-1,3-dihydroisobenzofuran (13a)



Yield : 73% (^{19}F NMR yield), 68% (isolated yield); m.p. 165-166 °C; HRMS (FAB) calcd for (M+Na) $\text{C}_{28}\text{H}_{21}\text{F}_3\text{NaO}$: 453.1449, Found 453.1442; ^1H NMR (CDCl_3) δ = 2.13 (s, 3H), 5.11 (d, J = 12.8 Hz, 1H), 5.16 (d, J = 12.4 Hz, 1H), 6.99 ~ 7.01 (m, 3H), 7.03 ~ 7.17 (m, 7H), 7.33 ~ 7.35 (m, 5H), 7.46 (s, 1H);

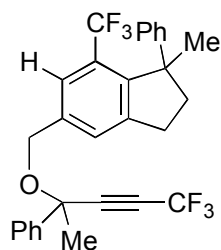
^{13}C NMR (CDCl_3) $\delta = 24.62$ (q, $J = 4.1$ Hz), 69.91, 91.27, 123.78 (q, $J = 276.9$ Hz), 124.73 (q, $J = 29.7$ Hz), 125.72, 126.07, 126.67, 126.94, 127.50, 127.52, 128.04, 129.56, 130.51 (q, $J = 1.7$ Hz), 130.63 (q, $J = 1.6$ Hz), 137.91, 140.15 (q, $J = 2.4$ Hz), 140.41, 141.61, 142.89 (q, $J = 2.2$ Hz), 143.87 (q, $J = 1.6$ Hz), 144.06; ^{19}F NMR (CDCl_3) $\delta = -49.94$ (s, 3F); IR (KBr) 2963, 2345, 1654, 1261, 1095 cm^{-1} .

5-Trifluoromethyl-3-methyl-3-phenyl-6,7-propyl-1,3-dihydroisobenzofuran (13b)



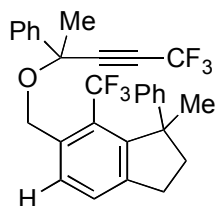
Yield : 49% (^{19}F NMR yield), 31% (isolated yield); HRMS (FAB) calcd for (M-H) $\text{C}_{26}\text{H}_{24}\text{F}_3\text{O}_9$: 361.1773, Found 361.1777; ^1H NMR (CDCl_3) $\delta = 0.97$ (q, $J = 8.8$ Hz, 6H), 1.43 ~ 1.48 (m, 2H), 1.56 ~ 1.66 (m, 2H), 1.93 (s, 3H), 2.61 (q, $J = 7.6$ Hz, 4H), 4.92 (d, $J = 12.0$ Hz, 1H), 4.96 (d, $J = 12.0$ Hz, 1H), 7.12 ~ 7.23 (m, 6H); ^{13}C NMR (CDCl_3) $\delta = 14.37$, 14.73, 24.49, 24.65 (q, $J = 7.4$ Hz), 24.94 (q, $J = 1.7$ Hz), 32.07 (q, $J = 2.4$ Hz), 34.56, 70.06, 91.42, 125.10, 125.80, 127.25, 127.43 (q, $J = 276.1$ Hz), 127.98, 139.64, 140.02 (q, $J = 2.5$ Hz), 141.41 (q, $J = 1.7$ Hz), 143.14, 144.54 (q, $J = 1.6$ Hz); ^{19}F NMR (CDCl_3) $\delta = -52.35$ (s, 3F); IR (neat) 2962, 2873, 1730, 1447, 1358, 1311, 1179, 1151, 1114 cm^{-1} .

7-(4,4,4-Trifluoro-1-methyl-1-phenyl)but-2-ynyl-5-Trifluoromethyl-3-methyl-3-phenyl-1,3-dihydroisobenzofuran (15a)



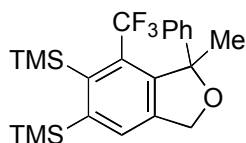
Yields are shown in Table 5. HRMS (FAB) calcd for (M^+) $\text{C}_{28}\text{H}_{22}\text{F}_6\text{O}_2$: 504.1528, Found 504.1524; ^1H NMR (CDCl_3) $\delta = 1.91$ (s, 3H), 2.01 (s, 3H), 4.32 (d, $J = 11.2$ Hz, 1H), 4.63 (d, $J = 11.6$ Hz, 1H), 5.07 (d, $J = 12.8$ Hz, 1H), 5.12 (d, $J = 12.8$ Hz, 1H), 7.21 ~ 7.31 (m, 5H), 7.38 ~ 7.49 (m, 5H), 7.59 ~ 7.61 (m, 2H); ^{19}F NMR (CDCl_3) $\delta = -58.18$ (s, 3F), -50.63 (s, 3F); IR (neat) 3062, 3032, 2989, 2961, 2936, 2855, 2270, 1729, 1602, 1495, 1349, 1323, 1278, 1194, 1144, 1076 cm^{-1} .

6-(4,4,4-Trifluoro-1-methyl-1-phenyl)but-2-ynyl-5-Trifluoromethyl-3-methyl-3-phenyl-1,3-dihydroisobenzofuran (15b)



Yields are shown in Table 5. HRMS (FAB) calcd for (M^+) $C_{28}H_{22}F_6O_2$: 504.1522, Found 504.1524; 1H NMR ($CDCl_3$) δ = 1.88 (d, J = 1.6 Hz, 3H), 2.01 (d, J = 4.8 Hz, 3H), 4.41 (t, J = 11.2 Hz, 1H), 4.74 (t, J = 11.2 Hz, 1H), 5.07 (d, J = 6.0 Hz, 2H), 7.18 ~ 7.59 (m, 11H), 7.75 (t, J = 8.4 Hz, 1H); ^{19}F NMR ($CDCl_3$) δ = -53.32 (d, J = 19.6 Hz, 3F), -50.79 (d, J = 12.4 Hz, 3F); IR (neat) 3031, 2932, 2853, 2270, 1727, 1592, 1495, 1448, 1375, 1353, 1302, 1277, 1222, 1148, 1079 cm^{-1} .

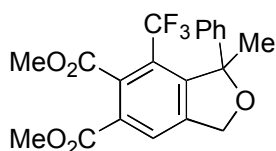
5-Trifluoromethyl-3-methyl-6,7-bis(trimethylsilyl)-3-phenyl-1,3-dihydroisobenzofuran (13c)



This compound could not be given in a pure form.

Yield : 10% (^{19}F NMR yield); HRMS (FAB) calcd for ($M+Na$) $C_{22}H_{29}F_3NaOSi_2$: 445.1606, Found 445.1607; 1H NMR ($CDCl_3$) δ = 0.08 (s, 9H), 0.38 (s, 9H), 0.43 (s, 9H), 2.07 (s, 3H), 4.94 (d, J = 12.4 Hz, 1H), 5.01 (d, J = 12.8 Hz, 1H), 7.15 ~ 7.55 (m, 7H), 7.72 (s, 1H); ^{19}F NMR ($CDCl_3$) δ = -50.20 (s, 3F).

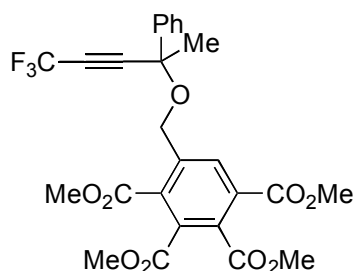
5-Trifluoromethyl-6,7-dimethoxycarbonyl-3-methyl-3-phenyl-1,3-dihydroisobenzofuran (13d)



Yield : 47% (^{19}F NMR yield), 35% (isolated yield); HRMS (FAB) calcd for ($M+H$) $C_{20}H_{18}F_3O_5$: 395.1101, Found 395.1106; 1H NMR ($CDCl_3$) δ = 2.04 (s, 3H), 3.94 (s, 6H), 5.06 (d, J = 13.2 Hz, 1H), 5.12 (d, J = 13.2 Hz, 1H), 7.16 ~ 7.19 (m, 2H), 7.26 ~ 7.32 (m, 3H), 8.11 (s, 1H); ^{13}C NMR ($CDCl_3$) δ = 24.07 (q, J = 3.2 Hz), 53.00, 53.13, 69.49, 90.94, 122.57 (q, J = 276.1 Hz), 123.78 (q, J = 33.0 Hz), 126.17, 126.34, 127.99, 128.12, 129.73, 134.36 (q, J = 2.5 Hz), 142.30 (q, J = 1.6 Hz), 144.21, 148.49 (q, J = 1.6 Hz), 164.74, 167.36; ^{19}F NMR ($CDCl_3$) δ = -54.26 (s, 3F); IR (neat) 2954, 2849, 1739, 1610, 1570, 1496, 1436,

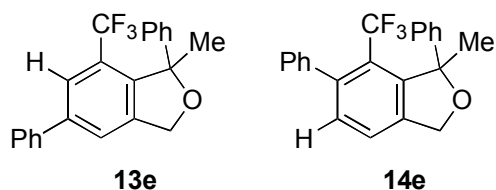
1362, 1323, 1238, 1180, 1144, 1055, 1010, 972 cm^{-1} .

4,4,4-Trifluoro-1-methyl-1-phenylbut-2-ynyl 2,3,4,5-tetramethoxycarbonylbenzyl ether (16)



Yield : 41% (^{19}F NMR yield); HRMS (FAB) calcd for (M+H) $\text{C}_{26}\text{H}_{24}\text{F}_3\text{O}_9$: 537.1364, Found 537.1372; ^1H NMR (CDCl_3) δ = 1.84 (s, 3H), 3.81 (s, 3H), 3.85 (s, 3H), 3.92 (s, 3H), 3.93 (s, 3H), 4.32 (d, J = 12.8 Hz, 1H), 4.70 (d, J = 12.4 Hz, 1H), 7.34 ~ 7.43 (m, 3H), 7.50 ~ 7.52 (m, 2H), 8.14 (s, 1H); ^{13}C NMR (CDCl_3) δ = 23.16, 31.68, 52.74, 52.97, 52.99, 53.13, 64.37, 75.25 (q, J = 52.9 Hz), 76.38, 86.78 (q, J = 5.7 Hz), 113.99 (q, J = 257.9 Hz), 128.38, 128.85, 130.34, 130.41, 132.29, 134.46, 135.56, 138.08, 139.62, 164.93, 165.65, 166.59, 167.26; ^{19}F NMR (CDCl_3) δ = -50.71 (s, 3F); IR (neat) 2954, 2270, 1738, 1600, 1442, 1372, 1334, 1278, 1149, 1112, 1025, 998 cm^{-1} .

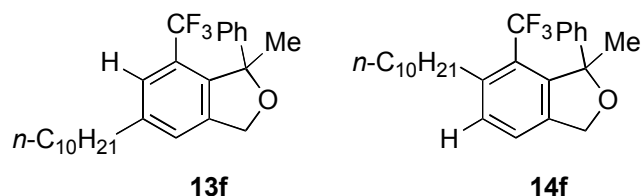
5-Trifluoromethyl-3-methyl-3,7-diphenyl-1,3-dihydroisobenzofuran (13e) and 5-Trifluoromethyl-3-methyl-3,6-diphenyl-1,3-dihydroisobenzofuran (14e)



Combined yield : 99% (^{19}F NMR yield), 86% (isolated yield); Isomeric ratio (**13e** : **14e**) = 71 : 29 (inseparable); **13e** : HRMS (FAB) calcd for (M^+) $\text{C}_{22}\text{H}_{17}\text{F}_3\text{O}$: 354.1230, Found 354.1231; ^1H NMR (CDCl_3) δ = 2.06 (s, 3H), 5.13 (d, J = 12.8 Hz, 1H), 5.18 (d, J = 12.8 Hz, 1H), 7.27 ~ 7.51 (m, 8H), 7.61 ~ 7.63 (m, 2H), 7.67 (s, 1H), 7.78 (s, 1H); ^{13}C NMR (CDCl_3) δ = 24.62 (q, J = 4.1 Hz), 70.30, 89.78, 123.13, 124.86 (q, J = 245.5 Hz), 124.87 (q, J = 5.0 Hz), 125.62 (q, J = 32.2 Hz), 126.45, 127.16, 127.69, 127.93, 128.17, 129.04, 139.26, 141.91, 141.94, 143.47, 143.53; ^{19}F NMR (CDCl_3) δ = -58.11 (s, 3F); **14e** : HRMS (FAB) calcd for (M^+) $\text{C}_{22}\text{H}_{17}\text{F}_3\text{O}$: 354.1230, Found 354.1231; ^1H NMR (CDCl_3) δ = 2.11 (s, 3H), 5.08 (d, J = 12.8 Hz, 1H), 5.14 (d, J = 13.2 Hz, 1H), 7.26 ~ 7.79 (m, 12H); ^{19}F NMR (CDCl_3) δ =

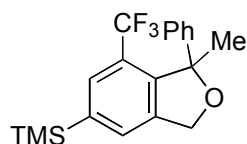
-50.21 (s, 3F).

5-Trifluoromethyl-3-methyl-3-phenyl-7-decyl-1,3-dihydroisobenzofuran (13f) and
5-Trifluoromethyl-3-methyl-3-phenyl-6-decyl-1,3-dihydroisobenzofuran (14f)



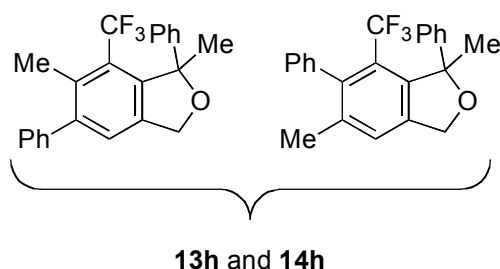
Combined yield : 87% (^{19}F NMR yield), 80% (isolated yield); Isomeric ratio (**13f** : **14f**) = 79 : 21 (inseparable); **13f** : HRMS (FAB) calcd for (M+Na) $\text{C}_{26}\text{H}_{33}\text{F}_3\text{ONa}$: 441.2378, Found 441.2381; ^1H NMR (CDCl_3) δ = 0.89 (t, J = 7.6 Hz, 4H), 1.27 ~ 1.34 (m, 13H), 1.62 ~ 1.67 (m, 2H), 2.01 (s, 3H), 2.69 (t, J = 8.4 Hz, 2H), 5.03 (d, J = 12.0 Hz, 1H), 5.09 (d, J = 12.4 Hz, 1H), 7.21 ~ 7.31 (m, 6H), 7.36 (s, 1H); ^{13}C NMR (CDCl_3) δ = 14.07, 22.66, 24.66 (q, J = 3.3 Hz), 29.30, 29.42, 29.52, 29.58, 31.29, 31.88, 35.50, 70.22, 89.66, 123.77 (q, J = 273.6 Hz), 124.47, 124.93 (q, J = 32.2 Hz), 125.84 (q, J = 5.7 Hz), 126.39, 127.52, 127.83, 140.35 (q, J = 2.5 Hz), 142.79, 143.65, 143.75 (q, J = 1.7 Hz); ^{19}F NMR (CDCl_3) δ = -58.03 (s, 3F); IR (neat) 2926, 2855, 1726, 1602, 1496, 1466, 1364, 1321, 1251, 1193, 1163, 1127, 1057 cm^{-1} ; **14f** : HRMS (FAB) calcd for (M+Na) $\text{C}_{26}\text{H}_{33}\text{F}_3\text{ONa}$: 441.2392, Found 441.2381; ^1H NMR (CDCl_3) δ = 0.88 (t, J = 6.8 Hz, 4H), 1.20 ~ 1.40 (m, 13H), 1.55 ~ 1.63 (m, 2H), 2.02 (s, 3H), 2.75 (t, J = 8.0 Hz, 2H), 5.04 (d, J = 5.2 Hz, 2H), 7.20 ~ 7.36 (m, 7H); ^{19}F NMR (CDCl_3) δ = -52.76 (s, 3F).

5-Trifluoromethyl-3-methyl-7-trimethylsilyl-3-phenyl-1,3-dihydroisobenzofuran (13g)



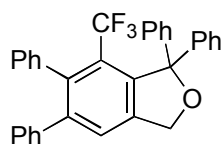
Yield : 16% (^{19}F NMR yield); HRMS (FAB) calcd for (M^+) $\text{C}_{19}\text{H}_{21}\text{F}_3\text{OSi}$: 350.1321, Found 350.1314; ^1H NMR (CDCl_3) δ = 0.01 (s, 9H), 1.69 (s, 3H), 4.75 (d, J = 13.2 Hz, 1H), 4.80 (d, J = 12.8 Hz, 1H), 6.89 ~ 6.99 (m, 5H), 7.28 (s, 1H), 7.34 (s, 1H); ^{19}F NMR (CDCl_3) δ = -57.82 (s, 3F).

5-Trifluoromethyl-3,6-dimethyl-3,7-diphenyl-1,3-dihydroisobenzofuran (13h) and
5-Trifluoromethyl-3,7-dimethyl-3,6-diphenyl-1,3-dihydroisobenzofuran (14h)



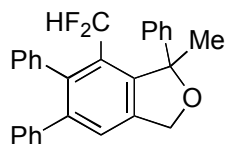
Combined yield : 48% (¹⁹F NMR yield); Isomeric ratio (**13h** : **14h**) = 60 : 40 (inseparable); (Isomer 1) HRMS (FAB) calcd for (M⁺) C₂₃H₁₉F₃O: 368.1378, Found 368.1388; ¹H NMR (CDCl₃) δ = 2.07 (s, 3H), 2.33 (s, 3H), 5.06 (d, *J* = 12.4 Hz, 1H), 5.10 (d, *J* = 12.0 Hz, 1H), 7.26 ~ 7.48 (m, 11H); ¹⁹F NMR (CDCl₃) δ = -54.13 (s, 3F); (Isomer 2) HRMS (FAB) calcd for (M⁺) C₂₃H₁₉F₃O: 368.1383, Found 368.1388; ¹H NMR (CDCl₃) δ = 2.02 (s, 3H), 2.07 (s, 3H), 5.07 (d, *J* = 12.8 Hz, 1H), 5.12 (d, *J* = 12.8 Hz, 1H), 7.14 ~ 7.61 (m, 11H); ¹⁹F NMR (CDCl₃) δ = -50.57 (s, 3F).

5-Trifluoromethyl-3,3,6,7-tetraphenyl-1,3-dihydroisobenzofuran (17)



Yield : 55% (¹⁹F NMR yield); HRMS (FAB) calcd for (M+H) C₃₃H₂₄F₃O: 493.1778, Found 493.1779; ¹H NMR (CDCl₃) δ = 4.96 (s, 2H), 7.00 ~ 7.07 (m, 4H), 7.13 ~ 7.15 (m, 6H), 7.35 (s, 10H), 7.49 (s, 1H); ¹⁹F NMR (CDCl₃) δ = -49.23 (s, 3F).

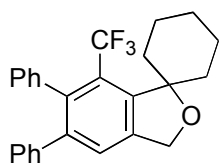
5-Difluoromethyl-3-methyl-3,6,7-triphenyl-1,3-dihydroisobenzofuran (18)



Yield : 53% (¹⁹F NMR yield), 39% (isolated yield); HRMS (FAB) calcd for (M⁺) C₂₈H₂₂F₂O: 412.1635, Found 412.1639; ¹H NMR (CDCl₃) δ = 2.17 (s, 3H), 5.16 (d, *J* = 12.0 Hz, 1H), 5.20 (d, *J* = 12.4 Hz, 1H), 6.29 (t, *J* = 54.0 Hz, 1H), 6.86 (s, 1H), 7.00 ~ 7.06 (m, 3H), 7.12 ~ 7.39 (m, 11H), 7.43 (s, 1H); ¹³C NMR

(CDCl₃) δ = 25.67 (t, J = 1.6 Hz), 70.35, 90.16, 113.50 (t, J = 238.9 Hz), 124.78, 126.43, 126.54 (t, J = 139.7 Hz), 126.62, 127.20, 127.44, 127.48, 127.61, 128.03, 129.65, 130.74, 131.06, 131.39, 137.46, 140.60, 141.17, 142.91, 144.20; ¹⁹F NMR (CDCl₃) δ = -108.38 (dd, J = 53.8, 309.9 Hz, 1F), -103.58 (dd, J = 53.8, 300.1 Hz, 1F); IR (neat) 2345, 1655, 1638, 1037, 766 cm⁻¹.

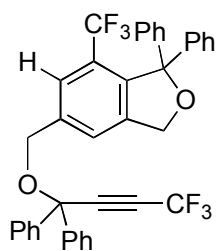
5-Trifluoromethyl-6,7-diphenyl-1,3-dihydroisobenzofuran-3-spiro-cyclohexane (19)



Yield : 71% (¹⁹F NMR yield), 53% (isolated yield); m.p. 139~140 °C; HRMS (FAB) calcd for (M+Na) C₂₆H₂₃F₃NaO: 431.1606, Found 431.1599; ¹H NMR (CDCl₃) δ = 1.26 ~ 1.34 (m, 2H), 1.68 ~ 1.91 (m, 6H), 2.13 ~ 2.21 (m, 2H), 5.06 (s, 2H), 6.91 ~ 6.94 (m, 2H), 7.02 ~ 7.05 (m, 2H), 7.10 ~ 7.12 (m, 3H), 7.13 ~ 7.16 (m, 3H), 7.36 (s, 1H); ¹³C NMR (CDCl₃) δ = 22.78, 24.90, 33.28 (q, J = 4.1 Hz), 68.83, 90.35, 124.15 (q, J = 29.1 Hz), 124.45 (q, J = 276.1 Hz), 125.92, 126.50, 126.75, 126.96, 127.43, 129.53, 130.43 (q, J = 1.6 Hz), 138.64, 139.56 (q, J = 2.5 Hz), 140.64, 141.56, 143.38, 144.26 (q, J = 2.4 Hz); ¹⁹F NMR (CDCl₃) δ = -48.00 (s, 3F); IR (KBr) 2954, 2812, 2345, 1655, 1543, 1509, 1165, 1120 cm⁻¹.

The compound **22** could not be isolated in a pure form. ¹H NMR (CDCl₃) δ = 4.22 (1H), 4.63 (1H), 4.94 ~ 4.96 (2H), 6.84 ~ 7.35 (m, 22H).

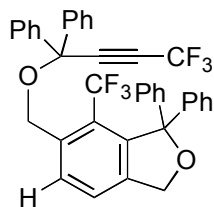
7-(4,4,4-Trifluoro-1,1-diphenyl)but-2-ynyl-5-Trifluoromethyl-3,3-phenyl-1,3-dihydroisobenzofuran (22)



The determination of the chemical structure was carried out mainly on the basis of the analogy of the chemical shift of **15a** due to their structural similarities.

Yield : 19% (¹⁹F NMR yield); ¹⁹F NMR (CDCl₃) δ = -57.61 (s, 3F), -50.63 (s, 3F).

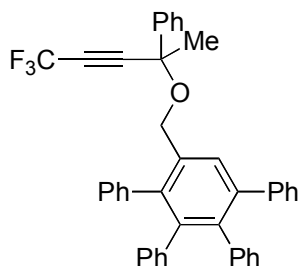
6-(4,4,4-Trifluoro-1,1-diphenyl)but-2-ynyl-5-Trifluoromethyl-3,3-phenyl-1,3-dihydroisobenzofuran (23)



This compound could not be isolated in a pure form. The determinations of the chemical structures were carried out on the basis of the analogy of the chemical shift of **15b** due to their structural similarities.

Yield : 8% (^{19}F NMR yield); ^{19}F NMR (CDCl_3) $\delta = -52.60$ (s, 3F), -50.71 (s, 3F).

4,4,4-Trifluoro-1,1-phenylbut-2-ynyl 2,3,4,5-tetraphenylbenzyl ether (24)

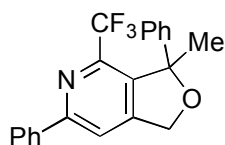


Yield : 16% (^{19}F NMR yield); HRMS (FAB) calcd for (M^+) $\text{C}_{47}\text{H}_{33}\text{F}_3\text{O}$: 670.2485, Found 670.2484; ^1H NMR (CDCl_3) $\delta = 4.36$ (s, 2H), 6.75 ~ 7.39 (m, 30H), 7.63 (s, 1H); ^{19}F NMR (CDCl_3) $\delta = -50.51$ (s, 3F); IR (neat) 2345, 1638, 1449, 1263, 1093, 803 cm^{-1} .

General procedure for fluoroalkylated 1,3-dihydrofuro[3,4-c]pyridine derivatives

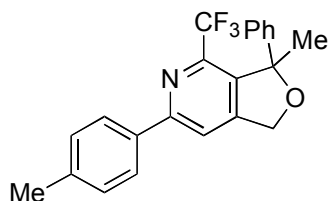
To a suspension of $\text{RhCl}_3 \cdot 3\text{H}_2\text{O}$ (8 mg, 0.03 mmol) were added *i*- Pr_2NEt (14 mg, 0.09 mmol) and fluoroalkylated diynes (0.30 mmol), nitriles (6.0 mmol). The mixture was stirred at 130 $^\circ\text{C}$ (bath temp.) for 2 h. After being cooled to room temperature, the residue was purified by column chromatography on silica gel (EtOAc/Hexane) to afford corresponding fluoroalkylated 1,3-dihydrofuro[3,4-c]pyridine derivatives.

4-Trifluoromethyl-2-methyl-2,6-diphenyl-1,3-dihydrofuro[3,4-c]pyridine (25)



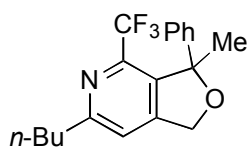
Yield : 39% (^{19}F NMR yield), 25% (isolated yield); HRMS (FAB) calcd for (M+H) $\text{C}_{21}\text{H}_{17}\text{F}_3\text{NO}$: 356.1266, Found 356.1262; ^1H NMR (CDCl_3) δ = 2.07 (s, 3H), 5.15 (d, J = 14.0 Hz, 1H), 5.20 (d, J = 14.0 Hz, 1H), 7.26 ~ 7.33 (m, 5H), 7.47 ~ 7.53 (m, 3H), 7.86 (s, 1H), 8.08 ~ 8.11 (m, 2H); ^{13}C NMR (CDCl_3) δ = 24.72 (q, J = 3.3 Hz), 69.95, 89.28, 115.59, 121.25 (q, J = 274.4 Hz), 126.31, 127.12, 128.01, 128.13, 128.93, 129.89, 137.37, 138.03, 142.76, 154.12, 156.60; ^{19}F NMR (CDCl_3) δ = -63.68 (s, 3F); IR (neat) 2927, 2345, 1719, 1610, 1446, 1375, 1261, 1224, 1191, 1136, 1049 cm^{-1} .

6-(4-methylphenyl)-4-trifluoromethyl-2-methyl-2-diphenyl-1,3-dihydrofuro[3,4-c]pyridine (26)



Yield : 44% (^{19}F NMR yield), 43% (isolated yield); ^1H NMR (CDCl_3) δ = 2.07 (s, 3H), 2.44 (s, 3H), 5.13 (d, J = 14.0 Hz, 1H), 5.18 (d, J = 14.0 Hz, 1H), 7.26 ~ 7.36 (m, 7H), 7.83 (s, 1H), 8.00 (d, J = 8.0 Hz, 2H); ^{13}C NMR (CDCl_3) δ = 21.33, 24.74 (q, J = 2.4 Hz), 69.95, 89.26, 115.19, 121.28 (q, J = 275.2 Hz), 1256.31, 127.00, 127.99, 128.11, 129.66, 134.62, 137.67, 140.08, 141.83, 142.83, 153.99, 156.62; ^{19}F NMR (CDCl_3) δ = -63.68 (s, 3F); IR (neat) 2923, 2372, 1609, 1560, 1446, 1376, 1325, 1224, 1190, 1136, 1047 cm^{-1} .

6-Buthyl-4-trifluoromethyl-2-methyl-2-diphenyl-1,3-dihydrofuro[3,4-c]pyridine (27)

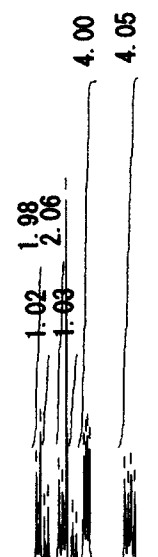


This compound could not be isolated in a pure form. The mixture of **27** and **15b** was obtained.

Yield : 10% (^{19}F NMR yield); HRMS (FAB) calcd for (M+H) $\text{C}_{19}\text{H}_{21}\text{F}_3\text{NO}$: 336.1576, Found 336.1575; ^1H NMR (CDCl_3) δ = 0.98 (t, J = 7.2 Hz, 3H), 1.41 ~ 1.47 (m, 3H), 1.73 ~ 1.80 (m, 3H), 2.02 (t, J = 1.6

Hz, 3H), 2.91 (t, $J = 8.4$ Hz, 2H), 7.18 ~ 7.49 (m, 6H); ^{19}F NMR (CDCl_3) $\delta = -63.49$ (s, 3F).

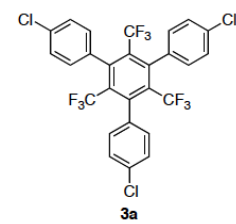
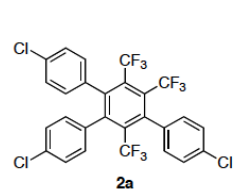
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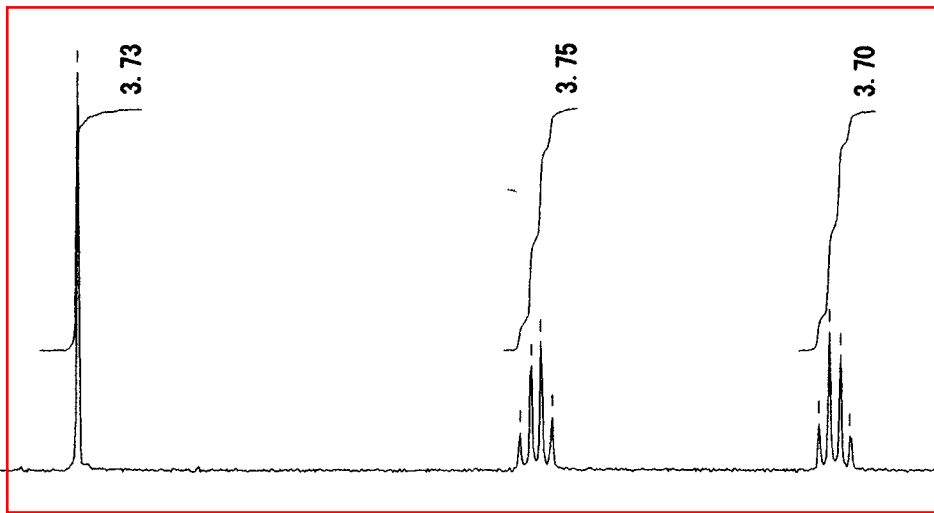
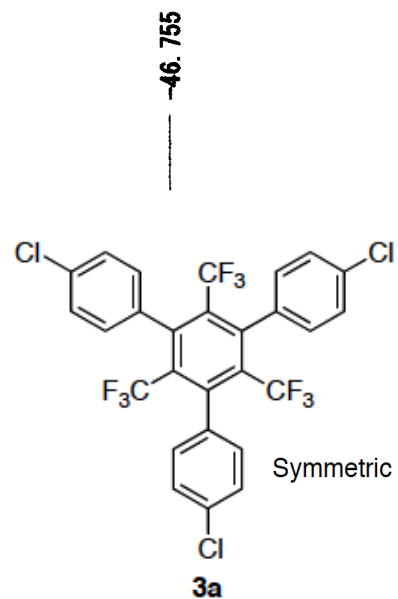


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FILDF
  
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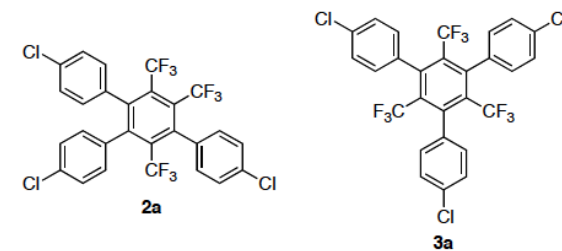


-50.0

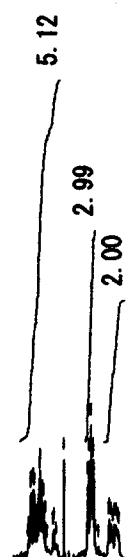
PPM

```

MENUF 19F
OBNUC 19F
OFR 376.05 MHz
OBSET 139.60 KHz
OBFIN 36.10 Hz
PW1 6.00 usec
DEADT 10.00 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 32768
SPO 32768
TIMES 16
DUMMY 1
FREQU 80000.00 Hz
FLT 40000 Hz
DELAY 5.00 usec
ACQTM 0.4096 sec
PD 4.9500 sec
ADBIT 16
RGAIN 14
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM
IRNUC 1H
IFR 399.65 MHz
IRSET 124.00 KHz
IRFIN 10500.00 Hz
IRRPW 45 usec
IRATN 511
DFILE 3 量体-p-Cl (19F).als
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 23
LKPHS 219
LKSIG 1003
CSPED 15 Hz
FILDC
FILDF
  
```

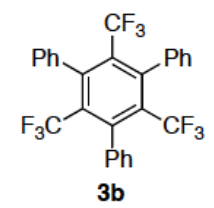
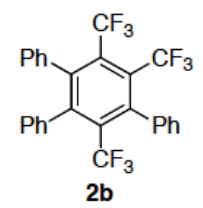


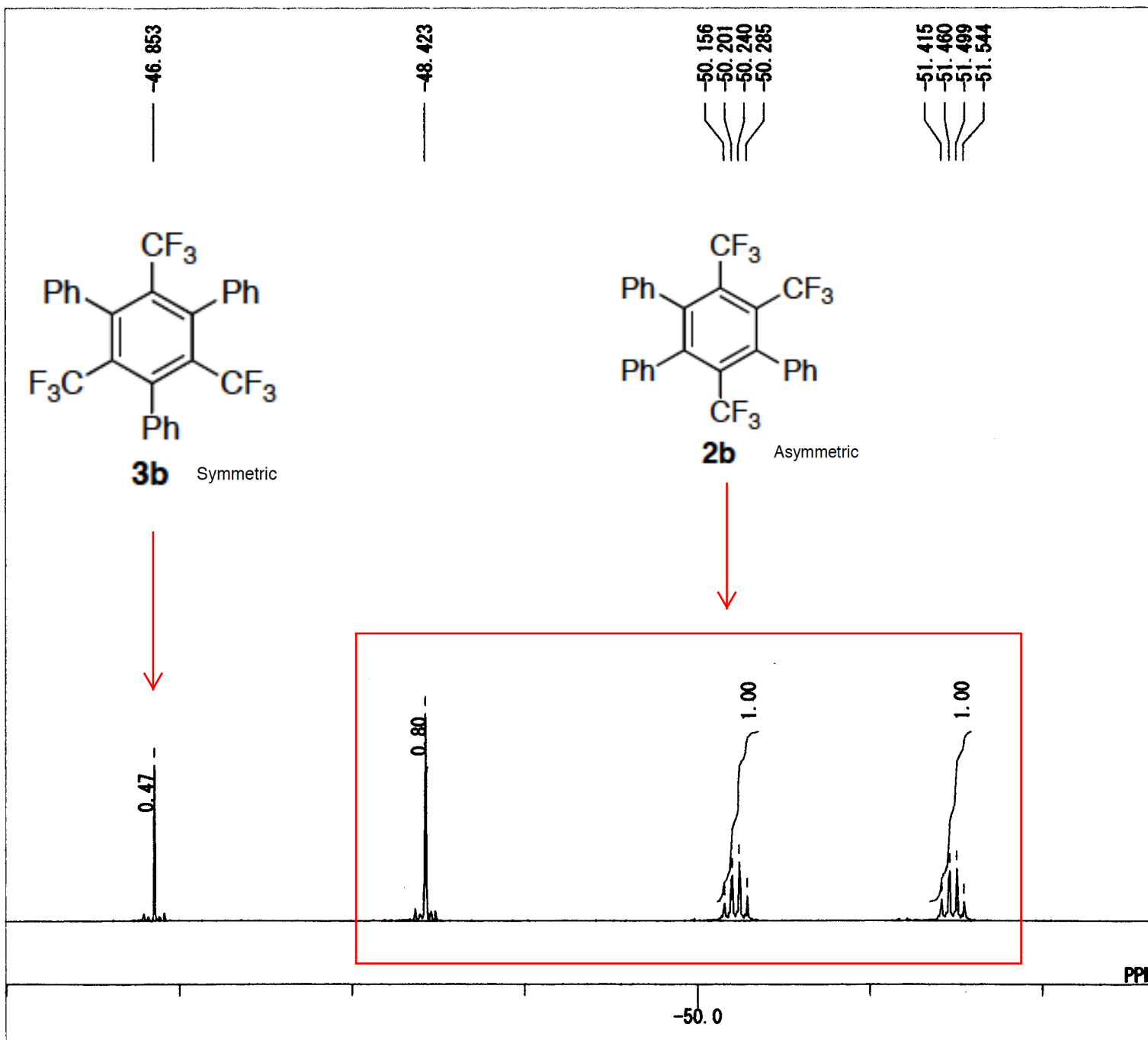
7.464
7.460
7.452
7.449
7.447
7.441
7.436
7.431
7.428
7.421
7.417
7.413
7.409
7.399
7.395
7.391
7.384
7.381
7.378
7.372
7.366
7.358
7.321
7.316
7.303
7.260
7.126
7.120
7.109
7.104
7.100
7.093
7.083
7.065
7.009
7.004
6.995
6.984
6.973
6.966
6.956
6.949



```

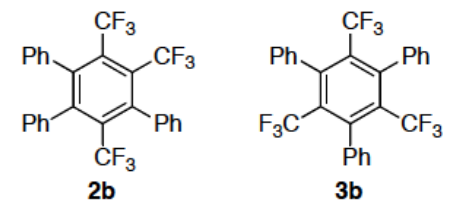
MENUF 1H
OBNUC 1H
OFR      399.65 MHz
OBSET    135.40 KHz
OBF IN   24.90 Hz
PW1      5.20 usec
DEADT    72.40 usec
PREDL    0.20000 msec
IWT      1.0000 msec
POINT    16384
SPO      16384
TIMES    8
DUMMY    1
FREQU    7992.01 Hz
FLT      4000 Hz
DELAY    50.00 usec
ACQTM    2.0501 sec
PD       4.9500 sec
ADBIT    16
RGAIN    17
BF       0.10 Hz
T1       0.00
T2       0.00
T3       90.00
T4       100.00
EXMOD    NON
EXPCM    NON:Single.coupled:PW1_ACQTM
IRNUC    1H
IFR      399.65 MHz
IRSET    136.90 KHz
IRF IN   97.50 Hz
IRRPW    45 usec
IRATN    511
DFILE    3置体-Ph(1H).als
SF       TH5ATFG2
LKSET    61.60 KHz
LKFIN    79.0 Hz
LKLEV    180
LGAIN    24
LKPHS    219
LKSIG    1638
CSPED    11 Hz
FILDC
FILDF
  
```

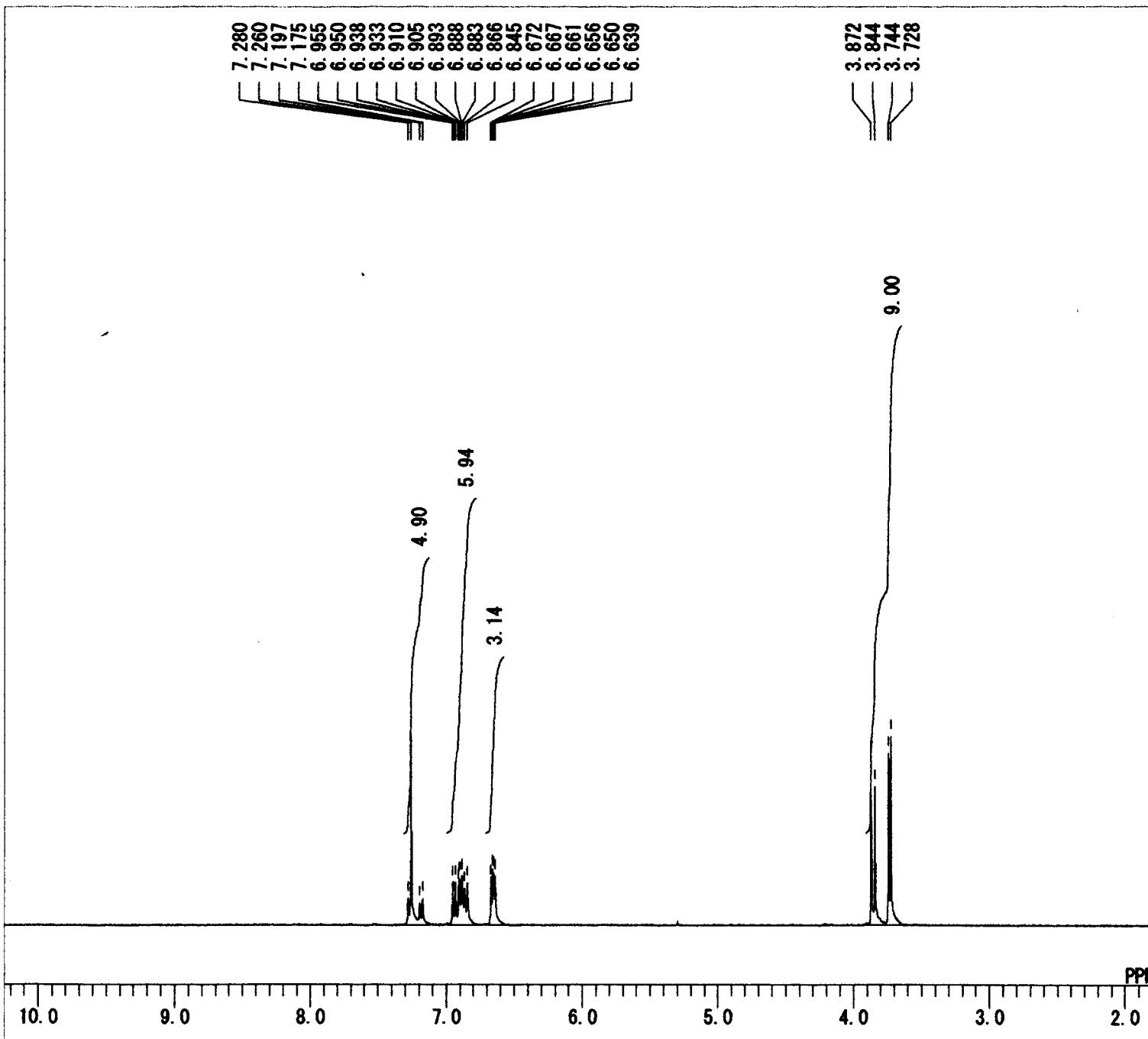




```

MNUF 19F
OBNUC 19F
OFR 376.05 MHz
OBSET 139.60 KHz
OBF IN 36.10 Hz
PW1 6.00 usec
DEADT 10.00 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 32768
SPO 32768
TIMES 16
DUMMY 1
FREQU 80000.00 Hz
FLT 40000 Hz
DELAY 5.00 usec
ACQTM 0.4096 sec
PD 4.9500 sec
ADBIT 16
RGAIN 14
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM
IRNUC 1H
IFR 399.65 MHz
IRSET 124.00 KHz
IRFIN 10500.00 Hz
IRRPW 45 usec
IRATN 511
DFILE 3-量体-Ph(19F).als
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 24
LKPHS 219
LKSIG 1675
CSPED 12 Hz
FILDC
FILDF
  
```

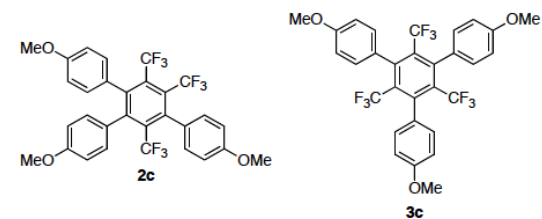


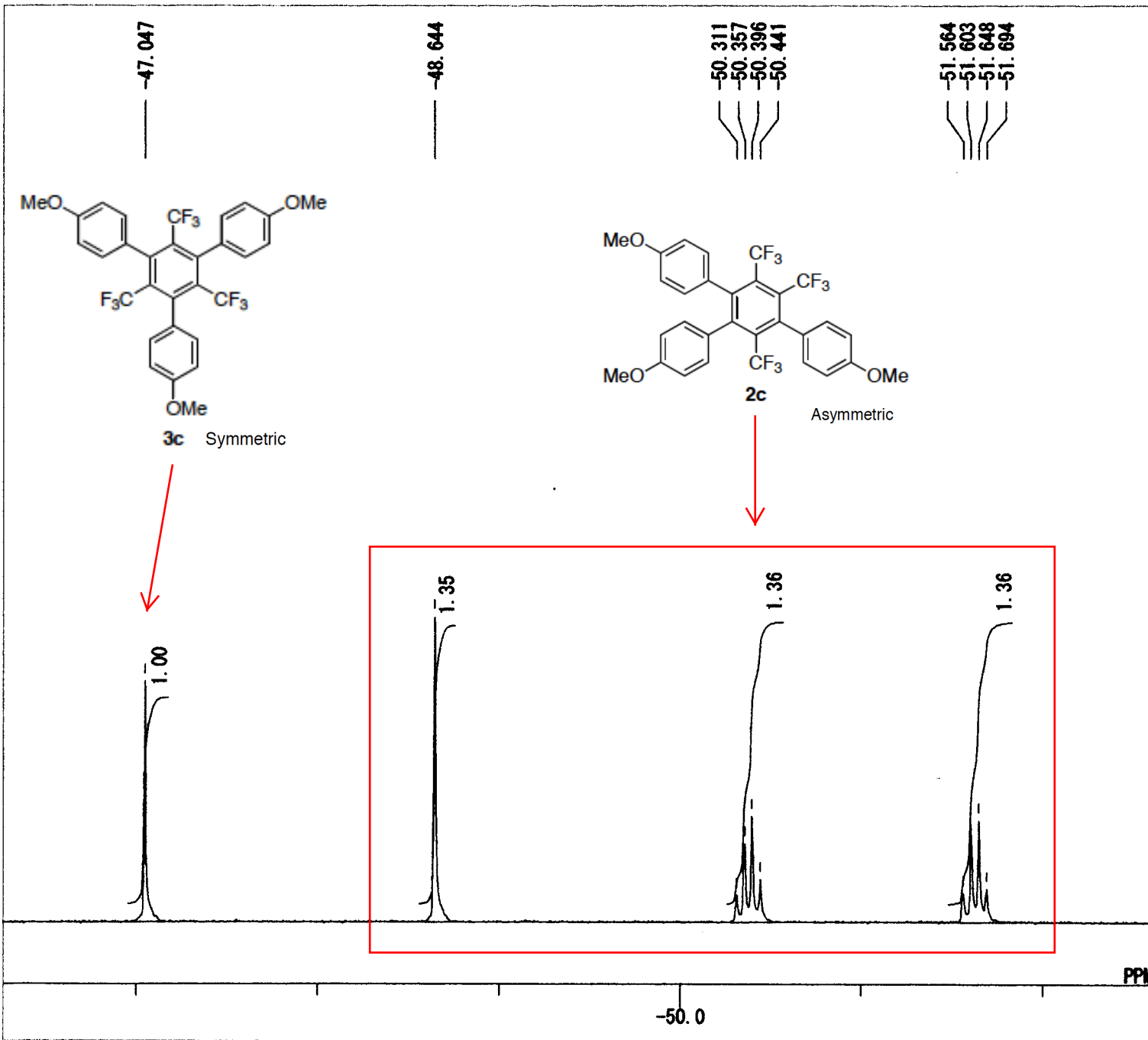


```

MENUMF 1H
OBNUC 1H
OFR 399.65 MHz
OBSET 135.40 KHz
OBFIN 24.90 Hz
PW1 5.20 usec
DEADT 72.40 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7992.01 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 2.0501 sec
PD 4.9500 sec
ADBIT 16
RGAIN 21
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM
IRNUC 1H
IFR 399.65 MHz
IRSET 136.90 KHz
IRFIN 97.50 Hz
IRRPW 45 usec
IRATN 511
DFILE 3置体-Ph-OMe(1H).als
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 22
LKPHS 219
LKSIG 588
CSPED 13 Hz
FILDC
FILDF

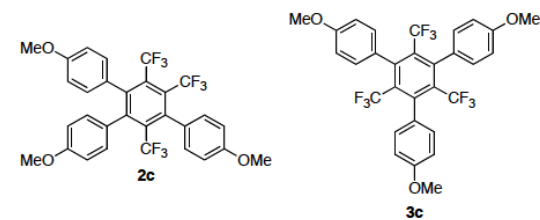
```





```

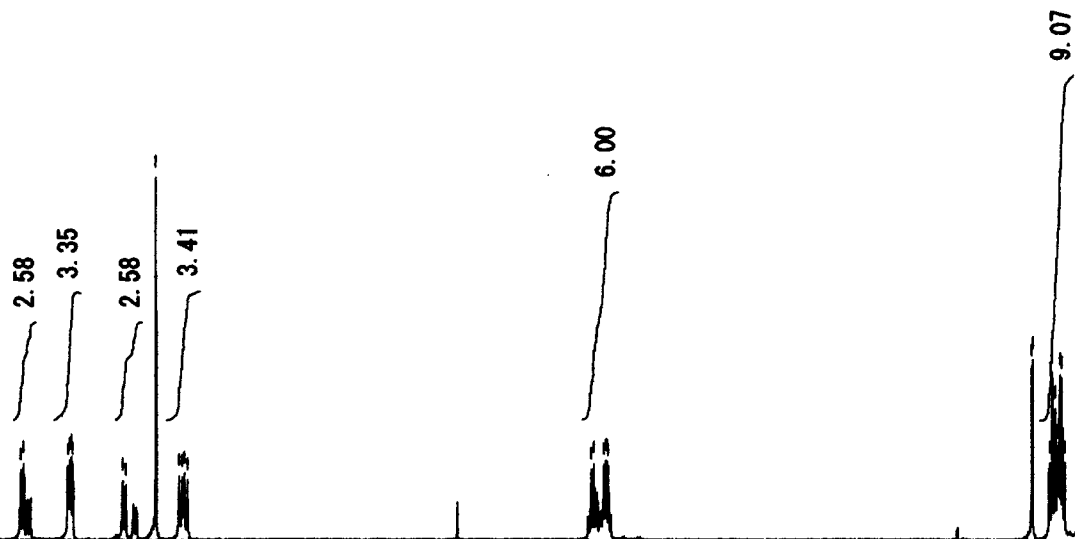
MENUF 19F
OBNUC 19F
OFR 376.05 MHz
OBSET 139.60 KHz
OBFIN 36.10 Hz
PW1 6.00 usec
DEADT 10.00 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 32768
SPO 32768
TIMES 16
DUMMY 1
FREQU 80000.00 Hz
FLT 40000 Hz
DELAY 5.00 usec
ACQTM 0.4096 sec
PD 4.9500 sec
ADBIT 16
RGAIN 14
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM
IRNUC 1H
IFR 399.65 MHz
IRSET 124.00 KHz
IRFIN 10500.00 Hz
IRRPW 45 usec
IRATN 511
DFILE 3-置体-Ph-OMe(19F).als
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 22
LKPHS 219
LKSIG 588
CSPED 13 Hz
FILDC
FILDF
  
```



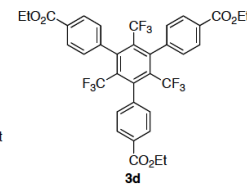
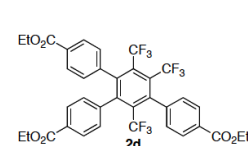
8.139
8.119
7.833
7.820
7.812
7.800
7.478
7.458
7.260
7.259
7.108
7.088
7.075
7.054

4.431
4.413
4.348
4.335
4.330
4.318

1.561
1.555
1.447
1.429
1.409
1.395
1.390
1.383
1.378
1.366
1.360
1.348

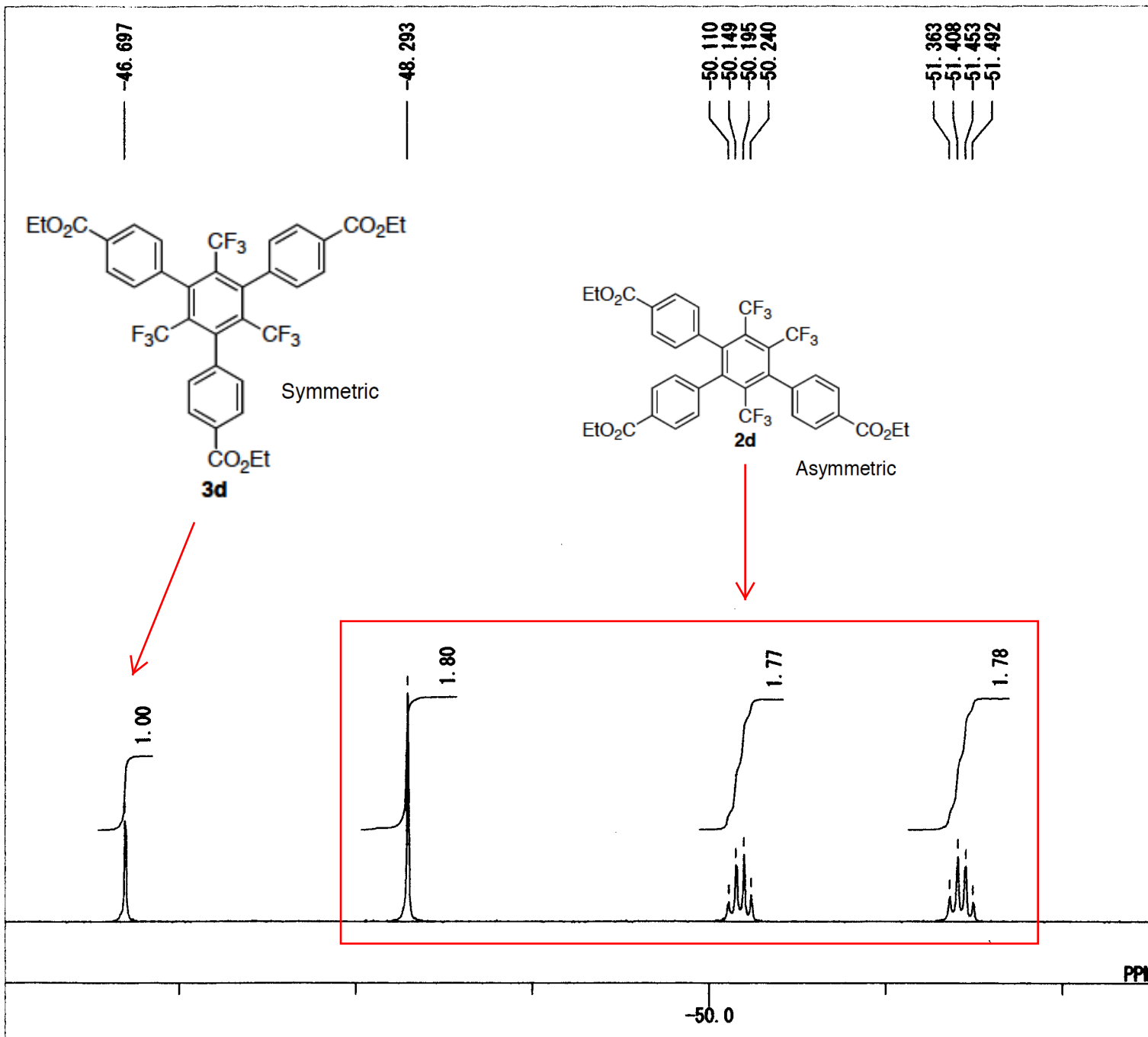


MENUF 1H
OBNUC 1H
OFR 399.65 MHz
OBSET 135.40 KHz
OBFIN 24.90 Hz
PW1 5.20 usec
DEADT 72.40 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 65536
SPO 16384
TIMES 8
DUMMY 1
FREQU 7992.01 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 2.0501 sec
PD 4.9500 sec
ADBIT 16
RGAIN 22
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM
IRNUC 1H
IFR 399.65 MHz
IRSET 136.90 KHz
IRFIN 97.50 Hz
IRRPW 45 usec
IRATN 511
DFILE 3-量体-Ph-CO2Et(1H).als
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 24
LKPHS 219
LKSIG 1088
CSPED 9 Hz
FILDC
FILDF



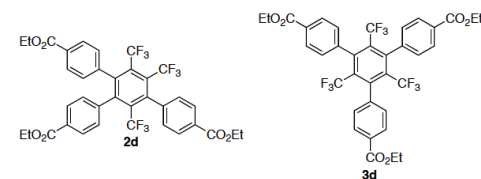
10.0

PPM

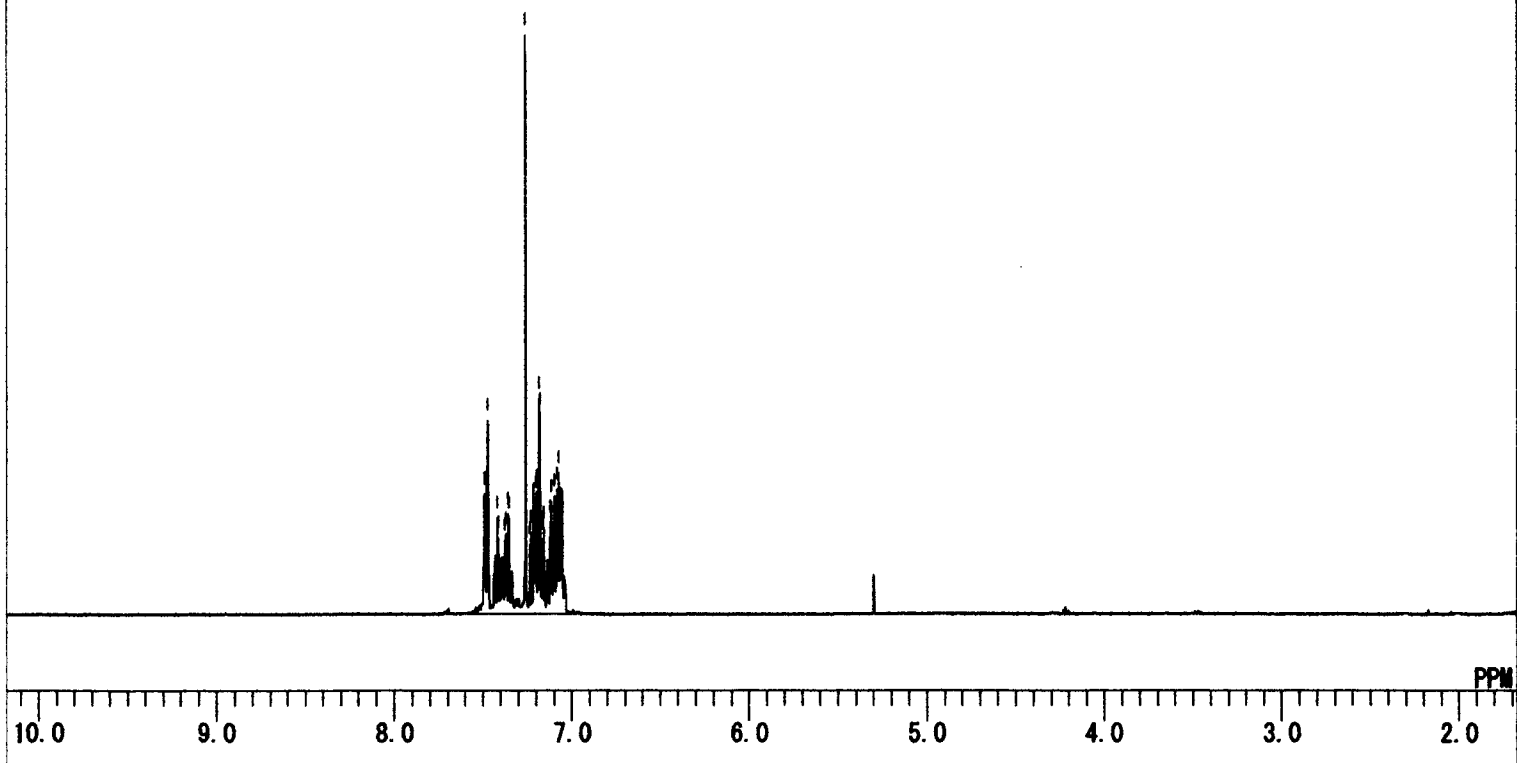


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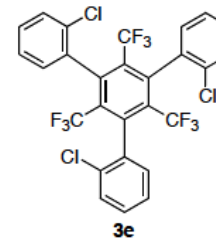
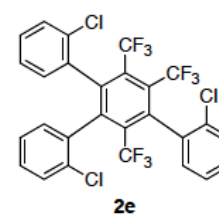
MENUF 19F
OBNUC 19F
OFR 376.05 MHz
OBSET 139.60 KHz
OBFIN 36.10 Hz
PW1 6.00 usec
DEADT 10.00 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 32768
SPO 32768
TIMES 16
DUMMY 1
FREQU 80000.00 Hz
FLT 40000 Hz
DELAY 5.00 usec
ACQTM 0.4096 sec
PD 4.9500 sec
ADBIT 16
RGAIN 14
BF 1.00 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM
IRNUC 1H
IFR 399.65 MHz
IRSET 124.00 KHz
IRFIN 10500.00 Hz
IRRPW 45 usec
IRATN 511
DFILE 3-置体-Ph-CO2Et(19F).als
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGA1N 24
LKP1S 219
LKS1G 1150 Hz
CSPED 13 Hz
FILDC
FILDF
  
```

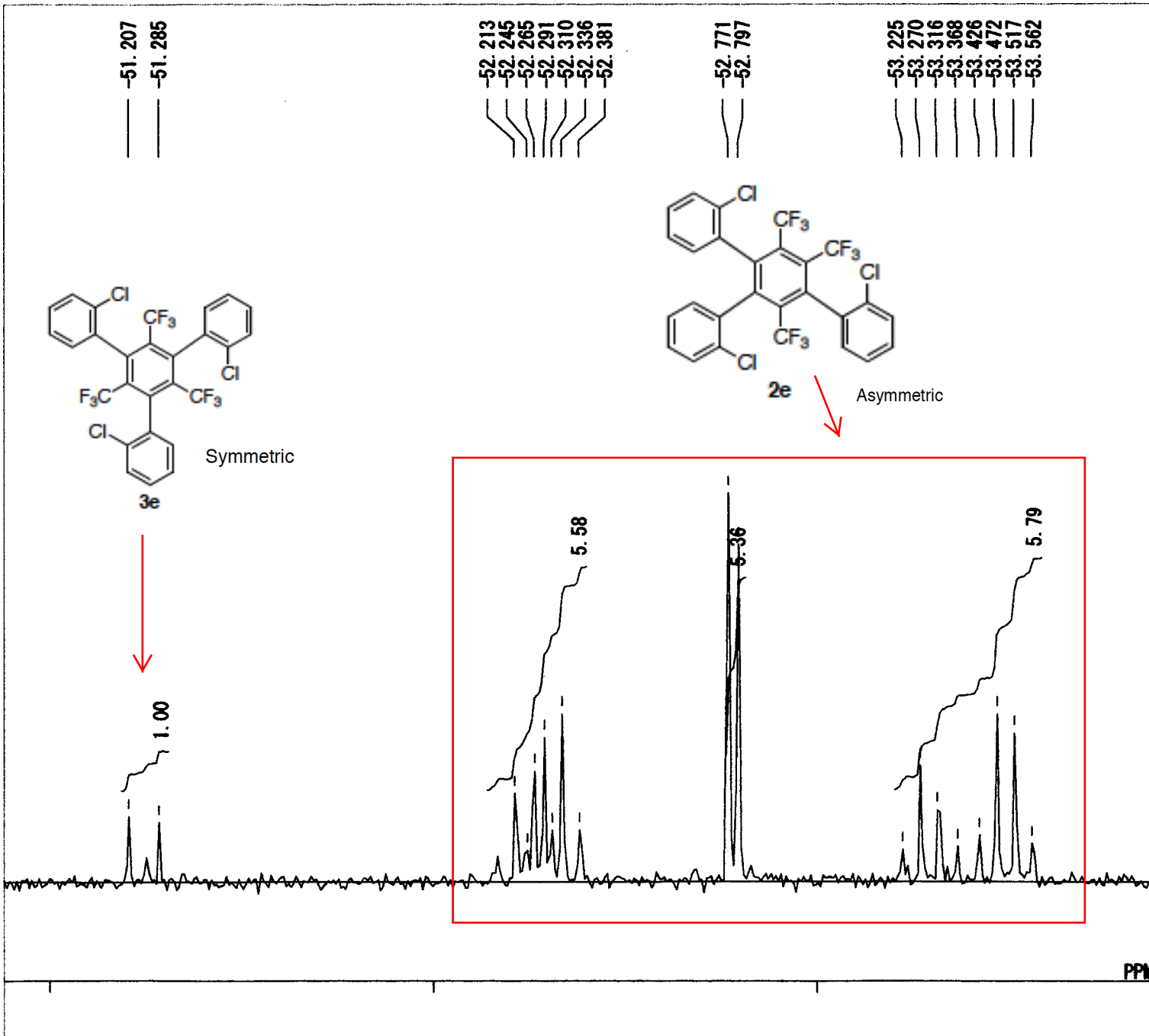


7.495
7.492
7.472
7.419
7.414
7.376
7.372
7.358
7.354
7.260
7.236
7.232
7.215
7.212
7.203
7.199
7.183
7.181
7.164
7.159
7.122
7.118
7.100
7.095
7.081
7.076
7.072
7.065
7.061
7.057
7.054



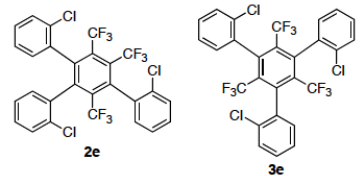
MENUF 1H
 OBNUC 1H
 OFR 399.65 MHz
 OBSSET 135.40 KHz
 OBF IN 24.90 Hz
 PW1 5.20 usec
 DEADT 72.40 usec
 PREDL 0.20000 msec
 IWT 1.0000 msec
 POINT 16384
 SPO 16384
 TIMES 8
 DUMMY 1
 FREQU 7992.01 Hz
 FLT 4000 Hz
 DELAY 50.00 usec
 ACQTM 2.0501 sec
 PD 4.9500 sec
 ADBIT 16
 RGAIN 17
 BF 0.00 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD NON
 EXPCM NON:Single.coupled:PW1_ACQTM
 IRNUC 1H
 IFR 399.65 MHz
 IRSET 136.90 KHz
 IRF IN 97.50 Hz
 IRRPW 45 usec
 IRATN 511
 DF FILE 3量体-o-ClPh(1H)-Part2.als
 SF TH5ATFG2
 LKSET 61.60 KHz
 LKF IN 79.0 Hz
 LKLEV 180
 LGAIN 22
 LKPHS 221
 LKSIG 860
 CSPED 14 Hz
 FILDC
 FILDF





```

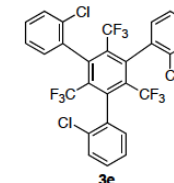
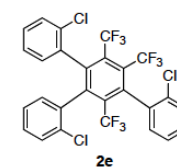
MENUF 19F
OBNUC 19F
OFR 376.05 MHz
OBSET 139.60 KHz
OBFIN 36.10 Hz
PW1 6.00 usec
DEADT 10.00 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 32768
SPO 32768
TIMES 16
DUMMY 1
FREQU 80000.00 Hz
FLT 40000 Hz
DELAY 5.00 usec
ACQTM 0.4096 sec
PD 4.9500 sec
ADBIT 16
RGAIN 14
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM.
IRNUC 1H
IFR 399.65 MHz
IRSET 124.00 KHz
IRFIN 10500.00 Hz
IRRPW 45 usec
IRATN 511
DFILE 3重体-o-ClPh(19F)-Part1.als
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 21
LKPHS 221
LKSIG 715
CSPED 11 Hz
FILDC
FILDF
  
```



7.492
7.472
7.469
7.426
7.414
7.409
7.391
7.386
7.371
7.367
7.352
7.332
7.317
7.314
7.260
7.183
7.181
7.175
7.171
7.153
7.123
7.118
7.115
7.109
7.089
7.084
7.077
7.070
7.066
7.059
7.054
7.037



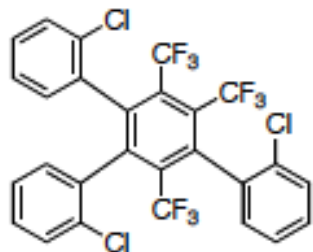
MENUF 1H
OBNUC 1H
OFR 399.65 MHz
OBSET 135.40 KHz
OBFIN 24.90 Hz
PW1 5.20 usec
DEADT 72.40 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7992.01 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 2.0501 sec
PD 4.9500 sec
ADBIT 16
RGAIN 18
BF 0.00 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM
IRNUC 1H
IFR 399.65 MHz
IRSET 136.90 KHz
IRFIN 97.50 Hz
IRRPW 45 usec
IRATN 511
DFILE 3量体-o-CIPh(1H)-Part1.als
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 21
LKPHS 221
LKSIG 666
CSPED 14 Hz
FILDC
FILDF



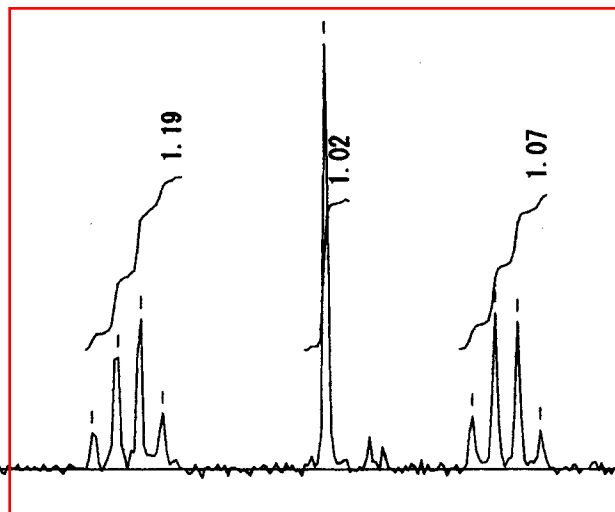
-52.206
-52.258
-52.304
-52.349

-52.673

-52.972
-53.017
-53.063
-53.108



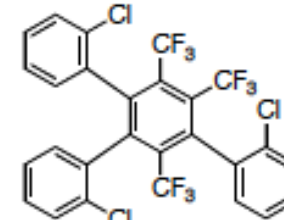
2e Asymmetric



PPM

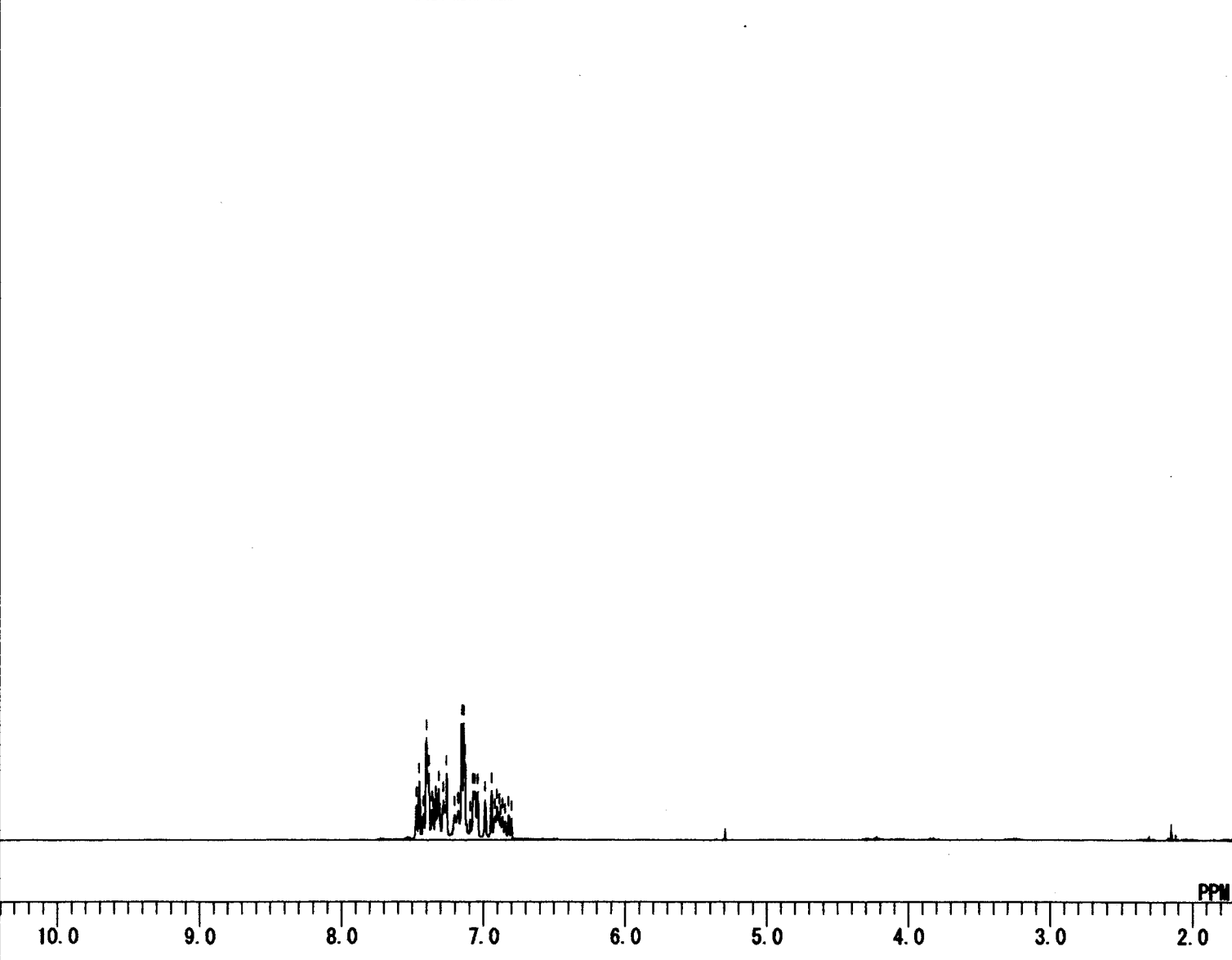
```

MENUF 19F
OBNUC 19F
OFR      376.05 MHz
OBSET    139.60 KHz
OBFIN    36.10 Hz
PW1       6.00 usec
DEADT    10.00 usec
PREDL    0.20000 msec
IWT      1.0000 msec
POINT    32768
SPO      32768
TIMES    16
DUMMY    1
FREQU    80000.00 Hz
FLT      40000 Hz
DELAY    5.00 usec
ACQTM    0.4096 sec
PD       4.9500 sec
ADBIT    16
RGAIN    14
BF       0.10 Hz
T1       0.00
T2       0.00
T3       90.00
T4       100.00
EXMOD    NON
EXPCM    NON:Single.coupled:PW1_ACQTM
IRNUC    1H
IFR      399.65 MHz
IRSET    124.00 KHz
IRFIN    10500.00 Hz
IRRPW    45 usec
IRATN    511
DFILE    3量体-o-CIPh(19F)-Part2.als
SF       TH5ATFG2
LKSET    61.60 KHz
LKFIN    79.0 Hz
LKLEV    180
LGAIN    23
LKPHS    221
LKSIG    1050
CSPED    10 Hz
FILDC
FILDF
  
```

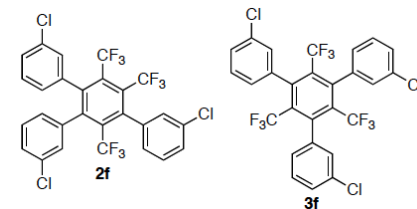


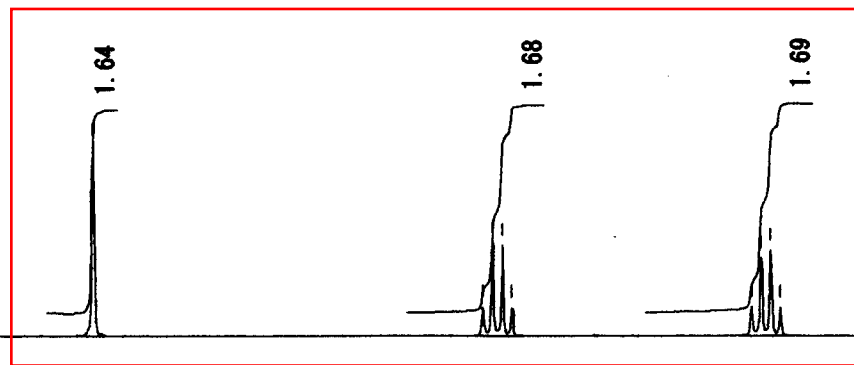
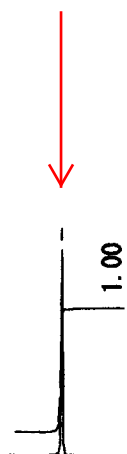
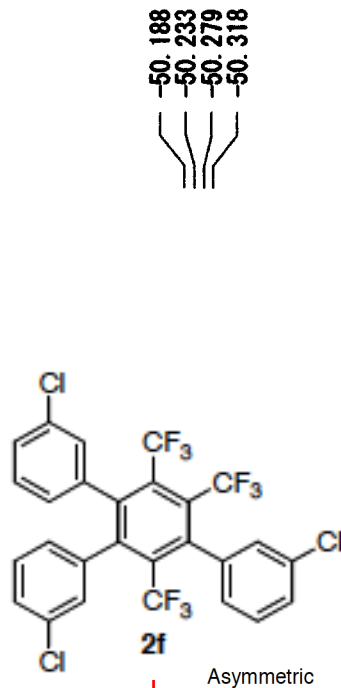
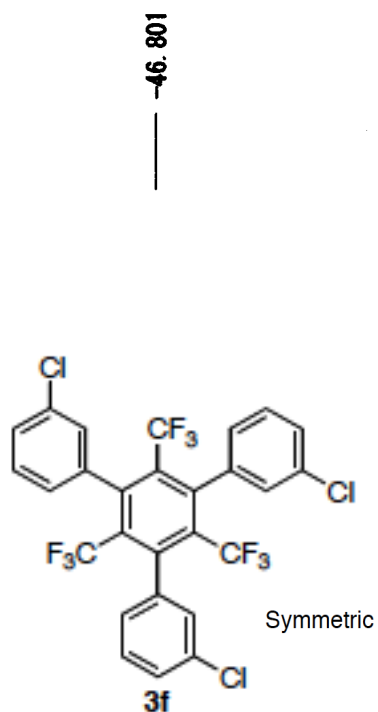
2e

7.471
7.450
7.422
7.402
7.388
7.383
7.362
7.354
7.349
7.341
7.334
7.330
7.315
7.282
7.260
7.204
7.179
7.172
7.154
7.148
7.140
7.134
7.129
7.092
7.075
7.072
7.060
7.040
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6.944
6.922
6.902
6.889
6.881
6.866
6.846
6.822
6.802



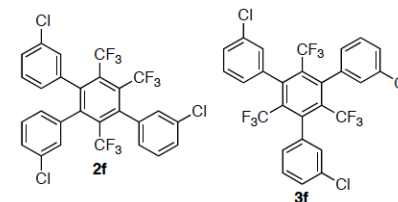
MENUF 1H
OBNUC 1H
OFR 399.65 MHz
OBSET 135.40 KHz
OBFIN 24.90 Hz
PW1 5.20 usec
DEADT 72.40 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7992.01 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 2.0501 sec
PD 4.9500 sec
ADBIT 16
RGAIN 14
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM
IRNUC 1H
IFR 399.65 MHz
IRSET 136.90 KHz
IRFIN 97.50 Hz
IRRPW 45 usec
IRATN 511
DFILE 3重体-m-ClPh(1H).als
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 22
LKPHS 221
LKSIG 727
CSPED 12 Hz
FILDC
FILDF





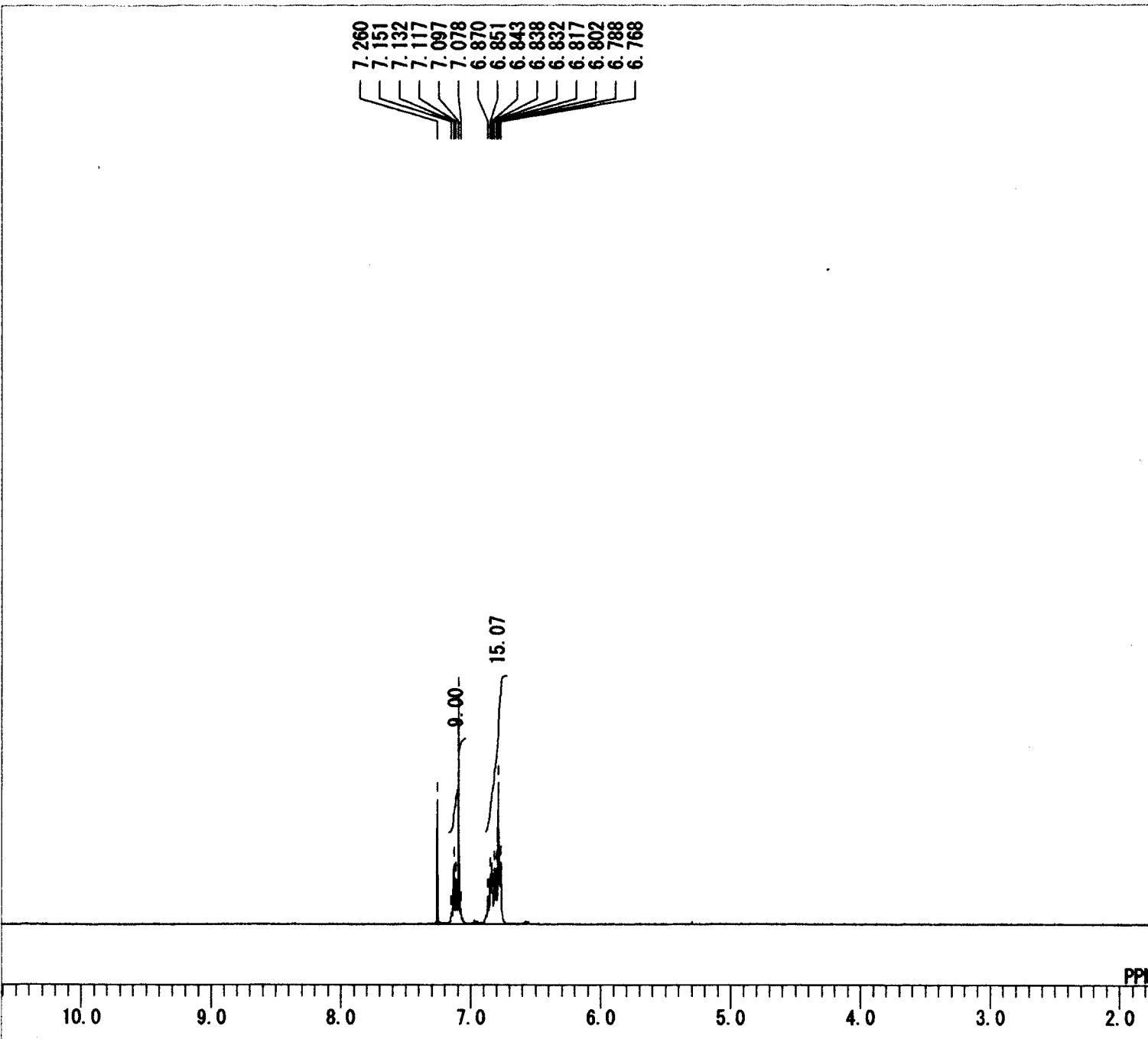
```

MENUF 19F
OBNUC 19F
OFR      376.05 MHz
OBSET    139.60 KHz
OBFIN    36.10 Hz
PW1      6.00 usec
DEADT    10.00 usec
PREDL    0.20000 msec
IWT      1.0000 msec
POINT    32768
SPO      32768
TIMES    16
DUMMY    1
FREQU    80000.00 Hz
FLT      40000 Hz
DELAY    5.00 usec
ACQTM    0.4096 sec
PD        4.9500 sec
ADBIT    16
RGAIN    14
BF        0.10 Hz
T1        0.00
T2        0.00
T3        90.00
T4        100.00
EXMOD    NON
EXPCM    NON:Single.coupled:PW1_ACQTM
IRNUC    1H
IFR      399.65 MHz
IRSET    124.00 KHz
IRFIN    10500.00 Hz
IRRPW    45 usec
IRATN    511
DFILE    3量体-m-ClPh(19F).als
SF        TH5ATFG2
LKSET    61.60 KHz
LKFIN    79.0 Hz
LKLEV    180
LGAIN    22
LKPHS    221
LKSIG    753
CSPED    11 Hz
FILDG
FILDF
  
```



PPM

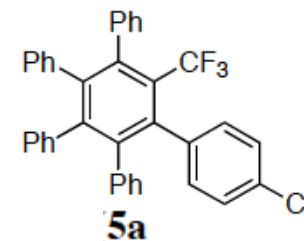
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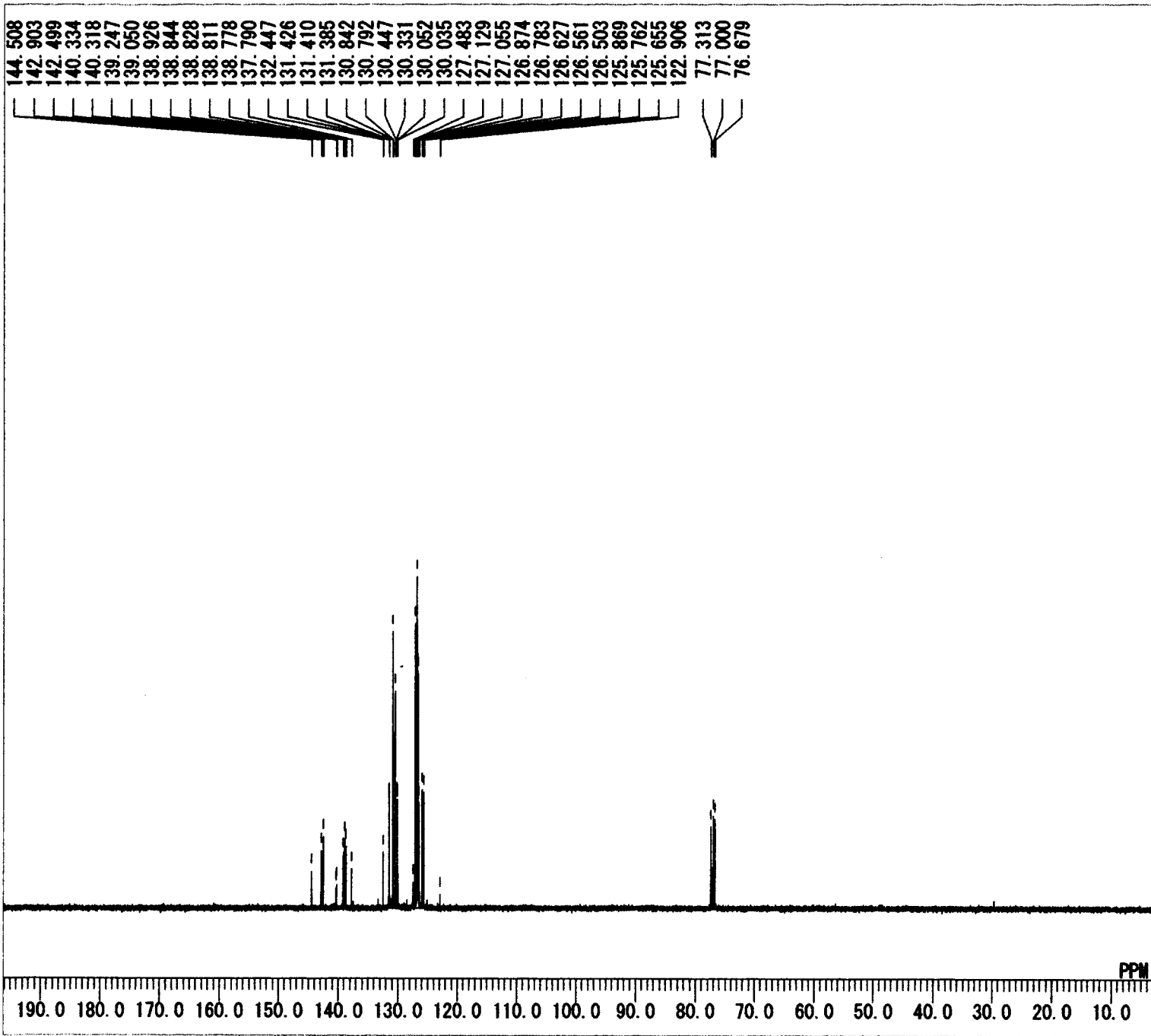


```

MENUF 1H
OBNUC 1H
OFR 399.65 MHz
OBSET 135.40 KHz
OBFIN 24.90 Hz
PW1 5.20 usec
DEADT 72.40 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7992.01 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 2.0501 sec
PD 4.9500 sec
ADBIT 16
RGAIN 19
BF 0.00 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM
IRNUC 1H
IFR 399.65 MHz
IRSET 136.90 KHz
IRFIN 97.50 Hz
IRRPW 45 usec
IRATN 511
DFILE CF3-PhCl+2diphenylacetylene(
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 23
LKPHS 221
LKSIG 949
CSPED 13 Hz
FILDC
FILDF

```

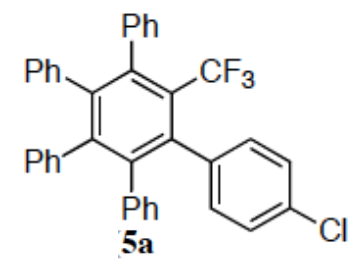


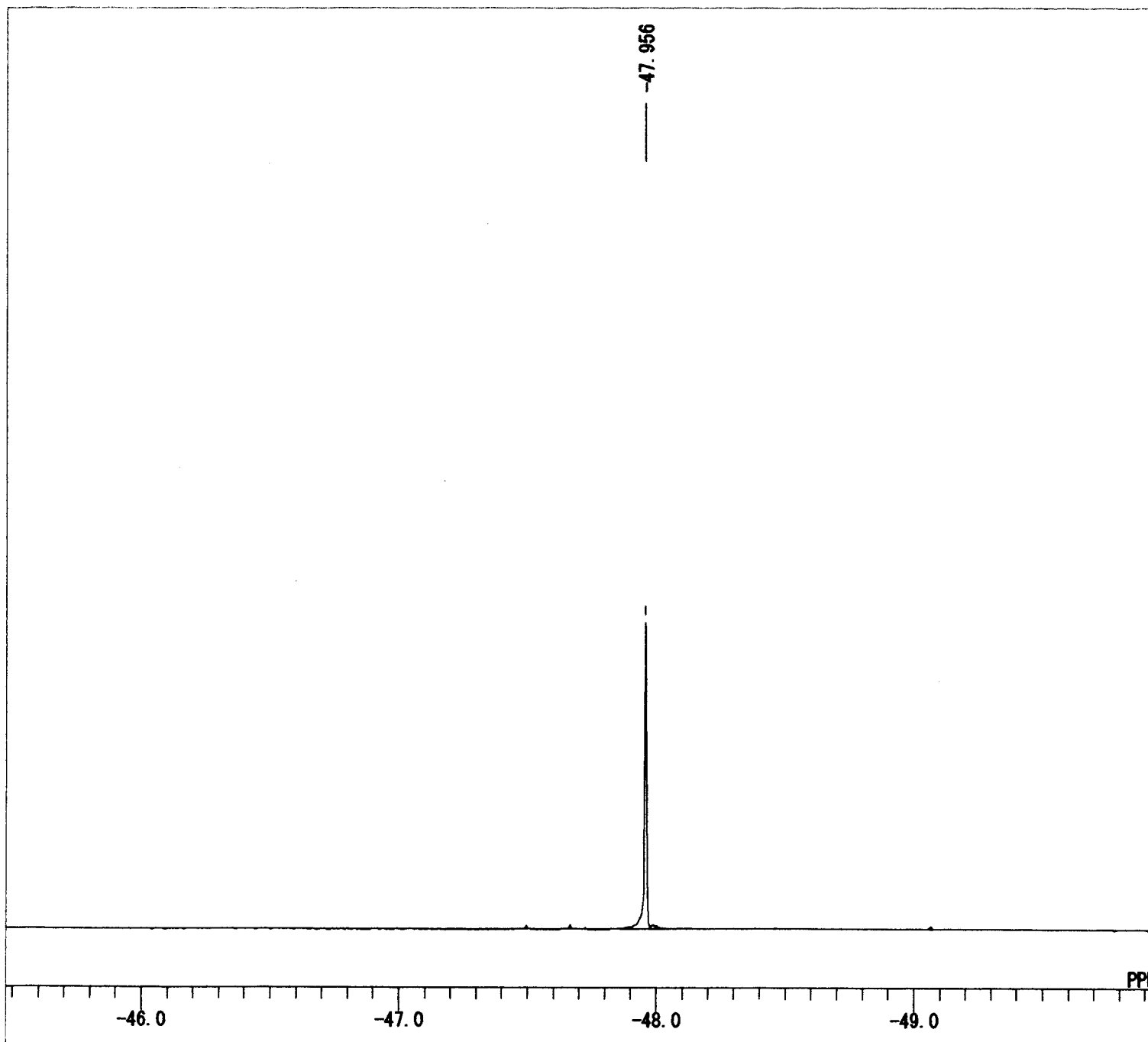


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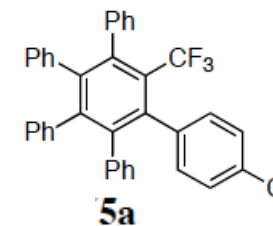
MENUF 13C
OBNUC 13C
OFR 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
PW1 6.00 usec
DEADT 19.10 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 32768
SPO 32768
TIMES 256
DUMMY 1
FREQU 27118.64 Hz
FLT 13550 Hz
DELAY 14.80 usec
ACQTM 1.2083 sec
PD 1.7920 sec
ADBIT 16
RGAIN 24
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD BCM
EXPCM Bilevel. complete. decoupling:
IRNUC 1H
IFR 399.65 MHz
IRSET 124.00 KHz
IRFIN 10500.00 Hz
IRRPW 45 usec
IRATN 511
DFILE CF3-PhCl+2diphenylacetylene (
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 24
LKPHS 221
LKSIG 894
CSPED 14 Hz
FILDC
FILDF

```

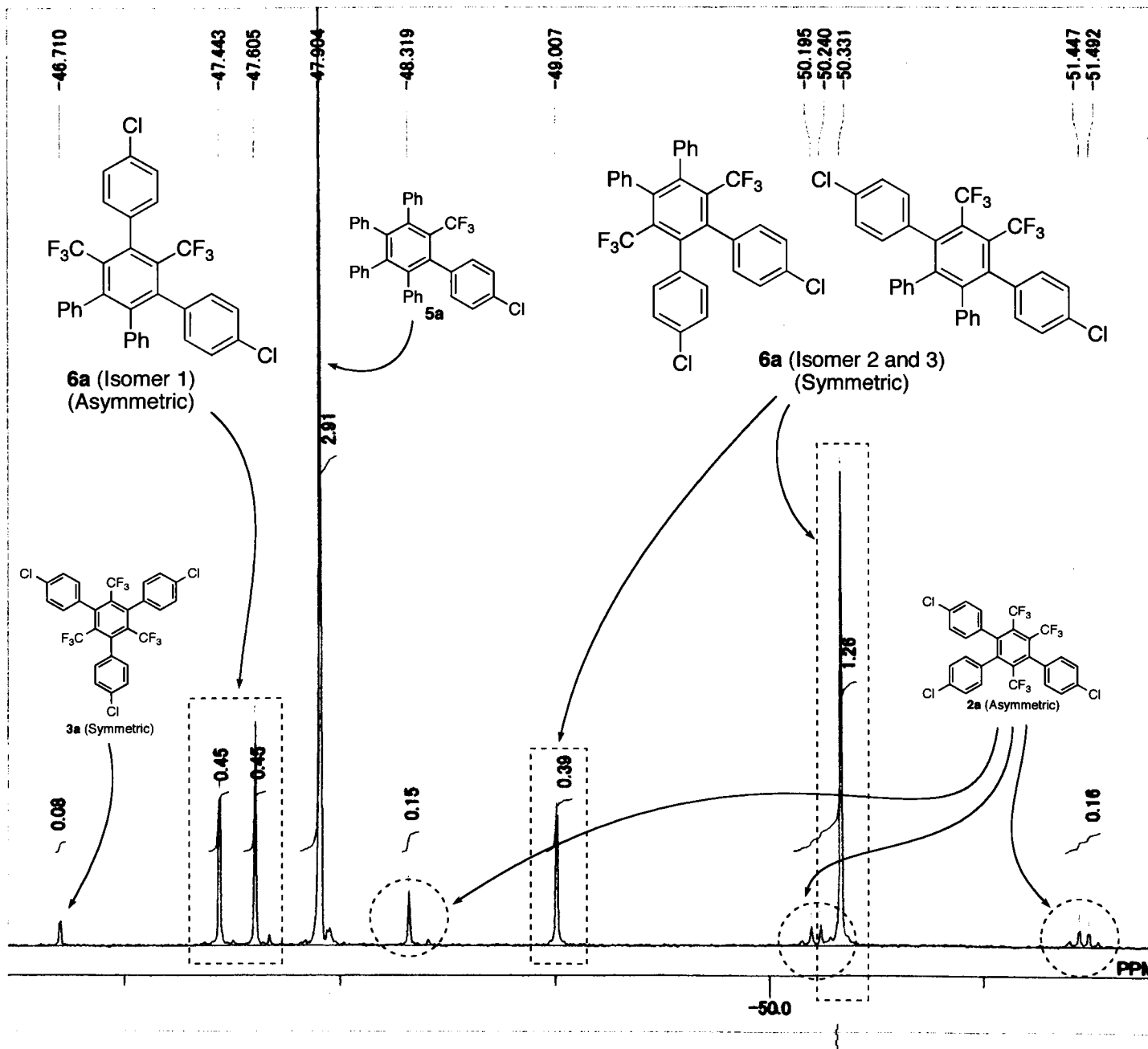




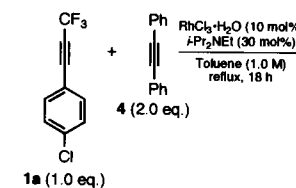
MENUF 19F
 OBNUC 19F
 OFR 376.05 MHz
 OBSET 139.60 KHz
 OBFIN 36.10 Hz
 PW1 6.00 usec
 DEADT 10.00 usec
 PREDL 0.20000 msec
 IWT 1.0000 msec
 POINT 32768
 SPO 32768
 TIMES 16
 DUMMY 1
 FREQU 80000.00 Hz
 FLT 40000 Hz
 DELAY 5.00 usec
 ACQTM 0.4096 sec
 PD 4.9500 sec
 ADBIT 16
 RGAIN 14
 BF 0.00 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD NON
 EXPCM NON:Single.coupled:PW1_ACQTM.
 IRNUC 1H
 IFR 399.65 MHz
 IRSET 124.00 KHz
 IRFIN 10500.00 Hz
 IRRPW 45 usec
 IRATN 511
 DFILE CF3-PhCl+2diphenylacetylene (TH5ATFG2
 SF TH5ATFG2
 LKSET 61.60 KHz
 LKFIN 79.0 Hz
 LKLEV 180
 LGAIN 25
 LKPHS 221
 LKSIG 1136
 GSPED 14 Hz
 FILDC
 FILDF



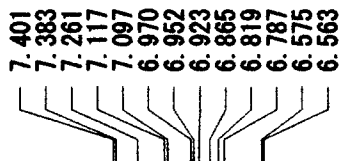
¹⁹F NMR Analysis of the reaction mixture (Total yield for all CF₃-containing compounds : 100% yield)



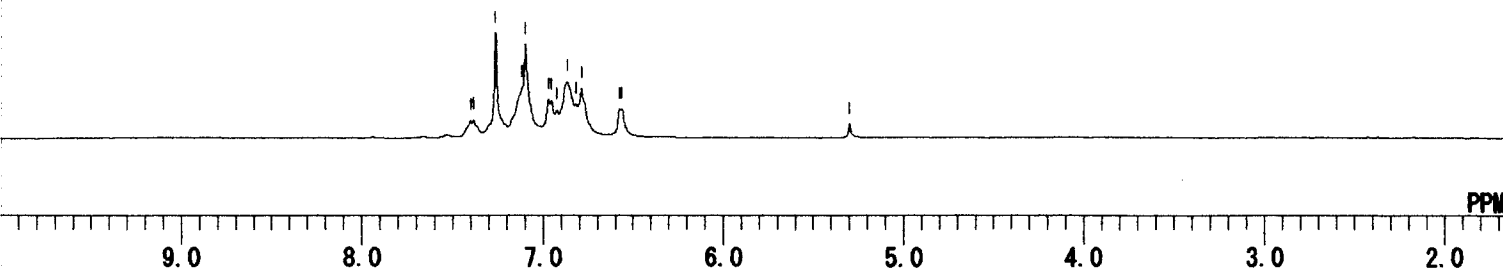
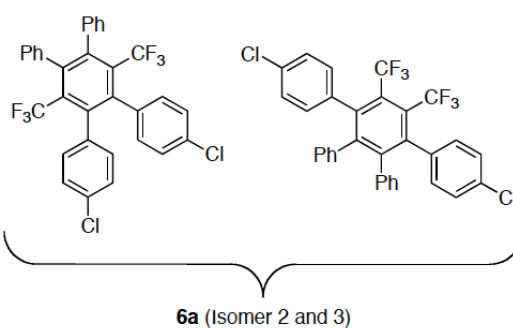
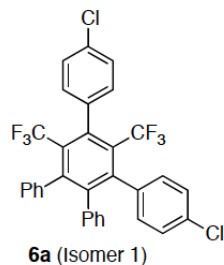
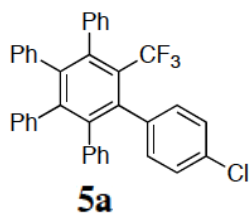
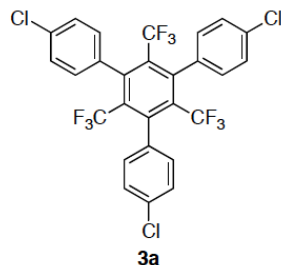
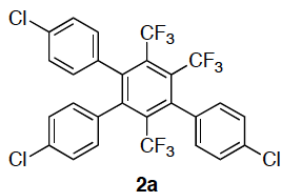
MENU	19F
OBNUC	19F
OFR	376.05 MHz
OBSET	139.80 KHz
OBFIN	38.10 Hz
PW1	6.00 usec
DEADT	10.00 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	16
DUMMY	1
FREQU	80000.00 Hz
FLT	40000 Hz
DELAY	5.00 usec
ACQTM	0.4096 sec
PD	4.9500 sec
ADBIT	16
RGAIN	14
BF	0.60 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM_PD:
IRNUC	1H
IFR	399.65 MHz
IRSET	124.00 KHz
IRFIN	10500.00 Hz
IRFPW	45 usec
IRATN	511
DFILE	DEFAULT.ALS
SF	TH5ATFG2
LKSET	61.90 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	23
LKPHS	222
LKSIG	827
CSPED	14 Hz
FILDC	
FILDF	



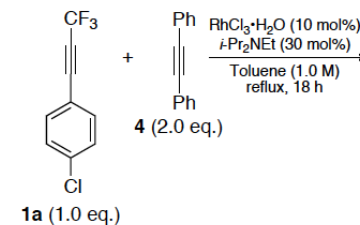
After *one* silica gel column chromatography



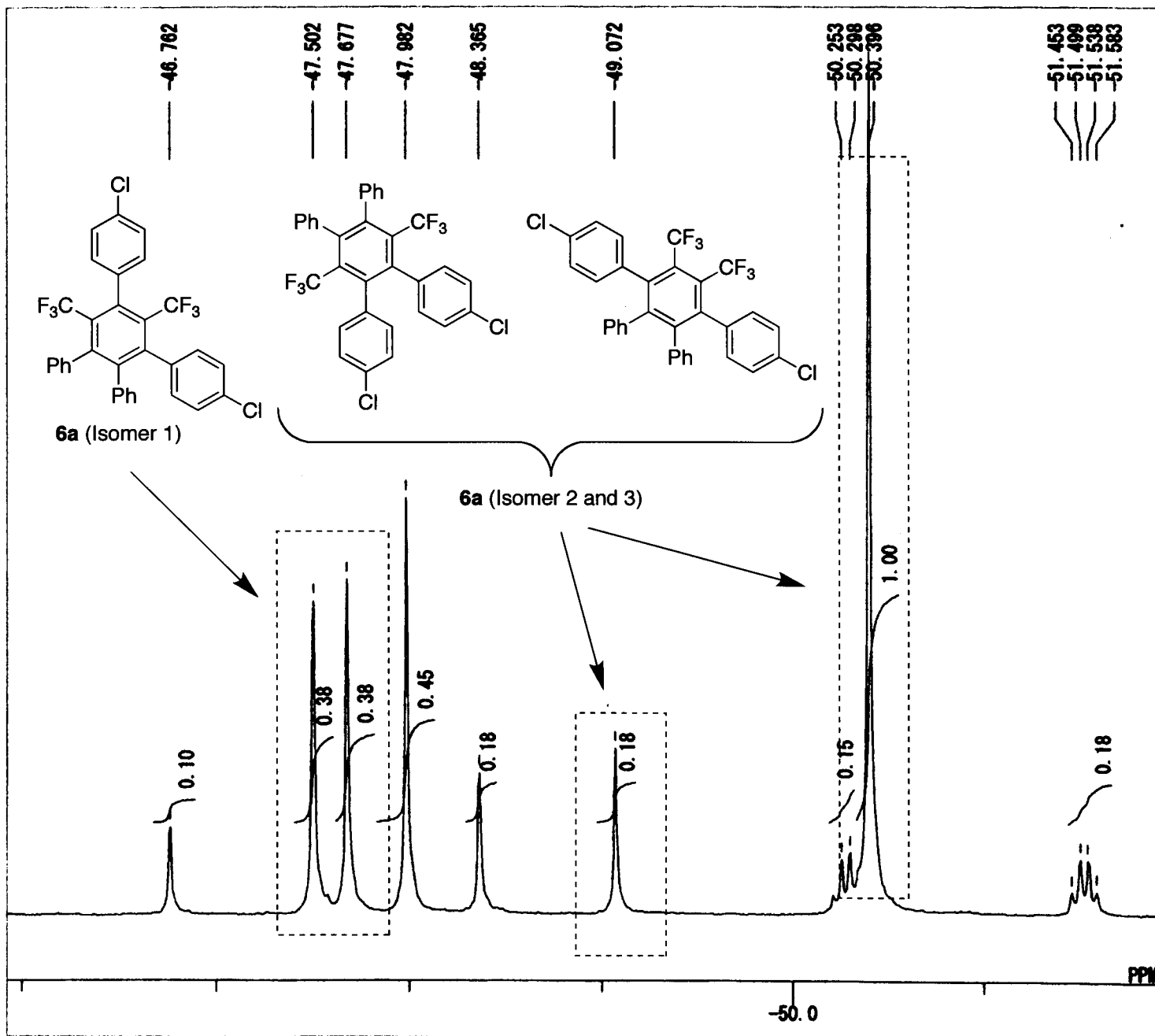
5.300



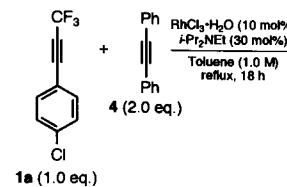
MENUF	1H
OBNUC	1H
OFR	399.65 MHz
OBSET	135.40 KHz
OBFIN	24.90 Hz
PW1	5.80 usec
DEADT	72.10 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	16384
SPO	16384
TIMES	64
DUMMY	1
FREQU	7992.01 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	2.0501 sec
PD	4.9500 sec
ADBIT	16
RGAIN	20
BF	5.00 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON: Single. coupled:PW1_ACQTM
IRNUC	1H
IFR	399.65 MHz
IRSET	136.90 KHz
IRFIN	97.50 Hz
IRRPW	45 usec
IRATN	511
DFILE	bypro-p-Cl-diPh(1H).als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	25
LKPHS	250
LKSIG	1831
CSPED	14 Hz
FILDC	
FILDF	

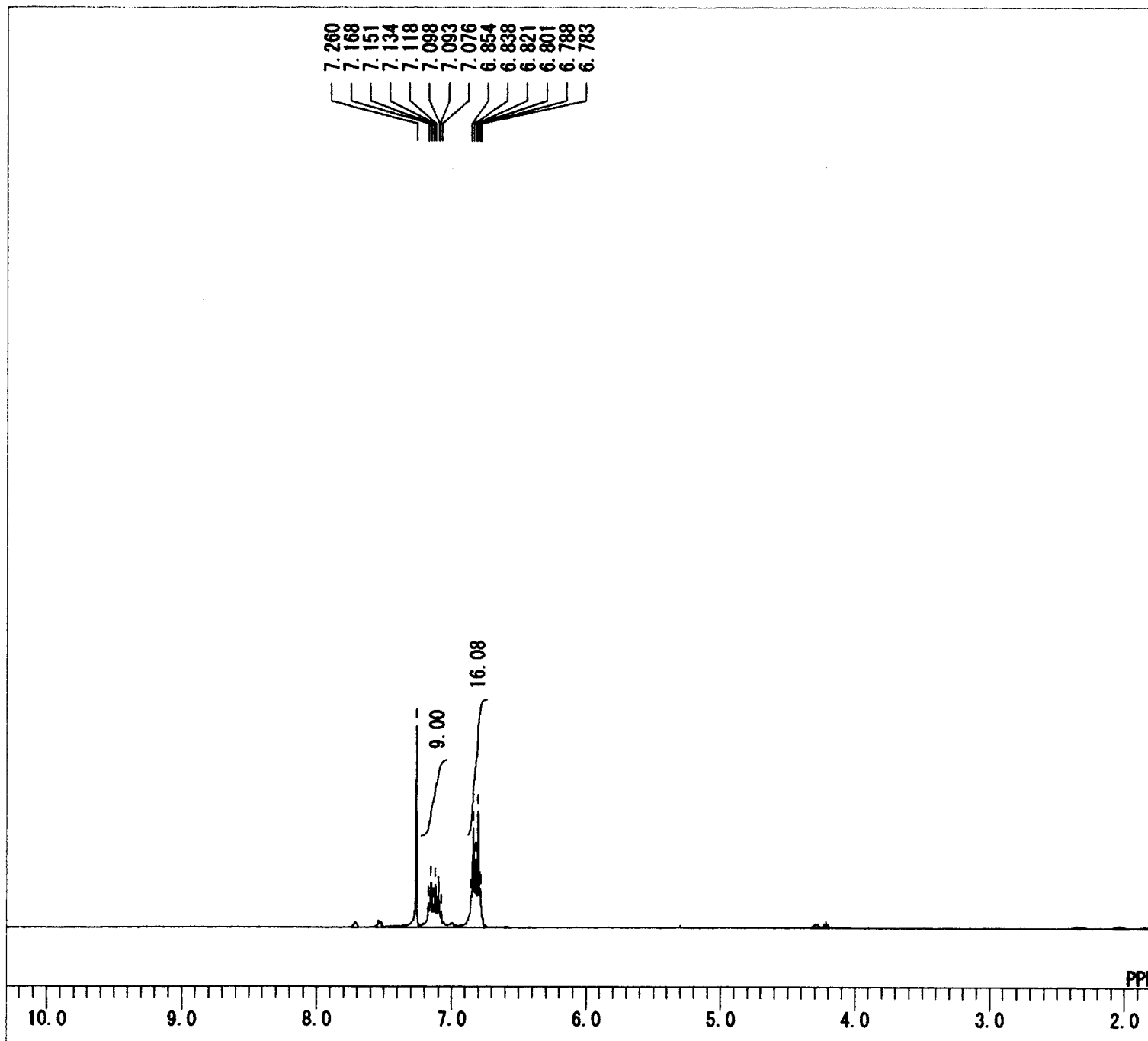


After one silica gel column chromatography

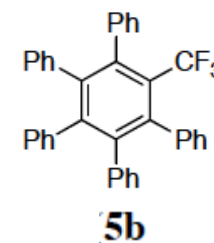


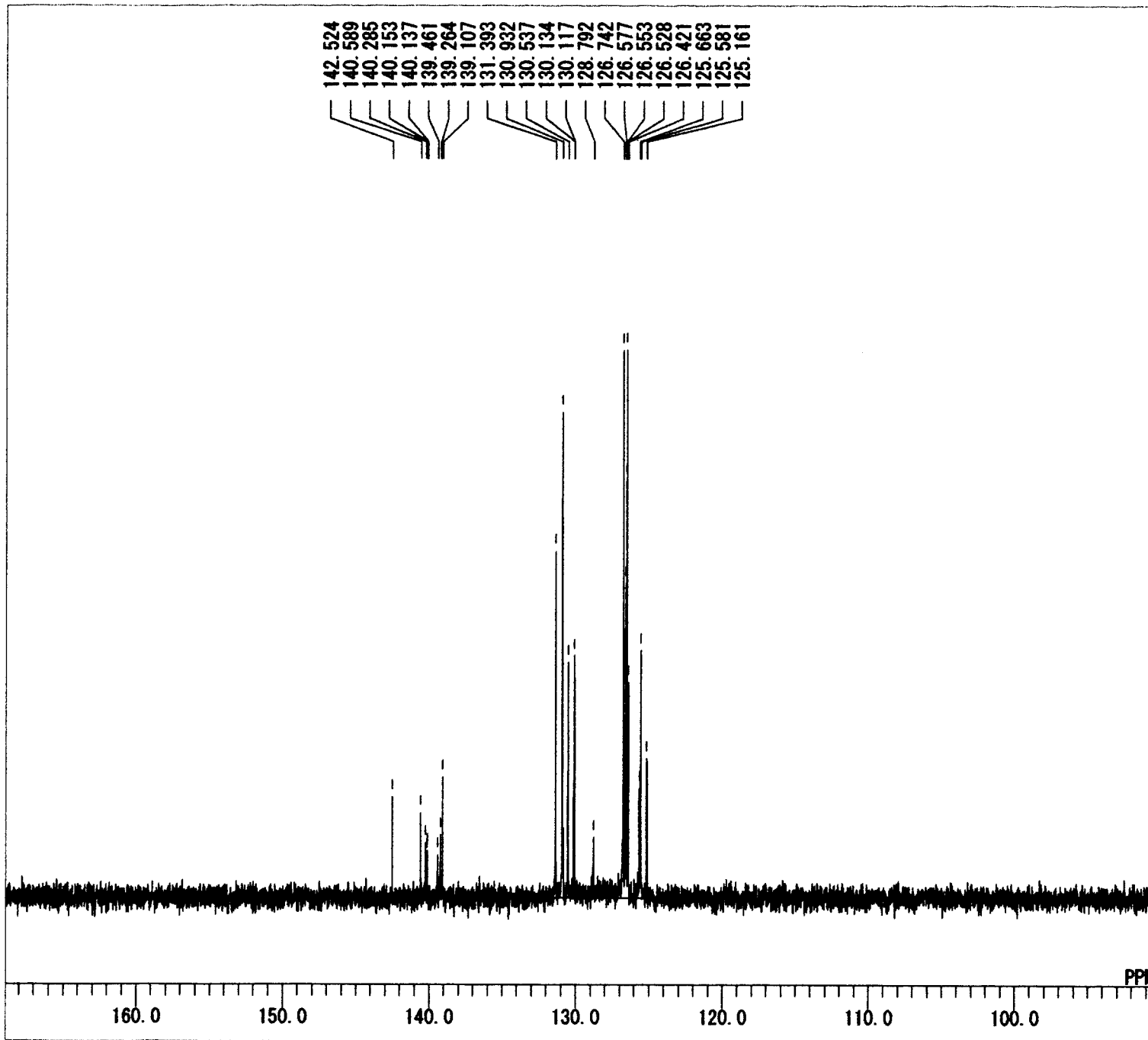
MENUF	19F
OBNUC	19F
OFR	376.05 MHz
OBSET	139.60 KHz
OBFIN	36.10 Hz
PW1	6.00 usec
DEADT	10.00 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	128
DUMMY	1
FREQU	80000.00 Hz
FLT	40000 Hz
DELAY	5.00 usec
ACQTM	0.4096 sec
PD	4.9500 sec
ADBIT	16
RGAIN	14
BF	5.00 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM
IRNUC	1H
IFR	399.65 MHz
IRSET	124.00 KHz
IRFIN	10500.00 Hz
IRRPW	45 usec
IRATN	511
DFILE	DEFAULT.ALS
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	25
LKPHS	250
LKSIG	1814
CSPED	13 Hz
FILDC	
FILDF	





MENUF	1H
OBNUC	1H
OFR	399.65 MHz
OBSET	135.40 KHz
OBFIN	24.90 Hz
PW1	5.20 usec
DEADT	72.40 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7992.01 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	2.0501 sec
PD	4.9500 sec
ADBIT	16
RGAIN	19
BF	0.60 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM
IRNUC	1H
IFR	399.65 MHz
IRSET	136.90 KHz
IRFIN	97.50 Hz
IRRPW	45 usec
IRATN	511
DFILE	CF3-Ph+2diphenylacetylene (1H)
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	27
LKPHS	222
LKSIG	2703
CSPED	14 Hz
FILDC	
FILDF	

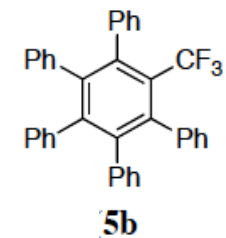


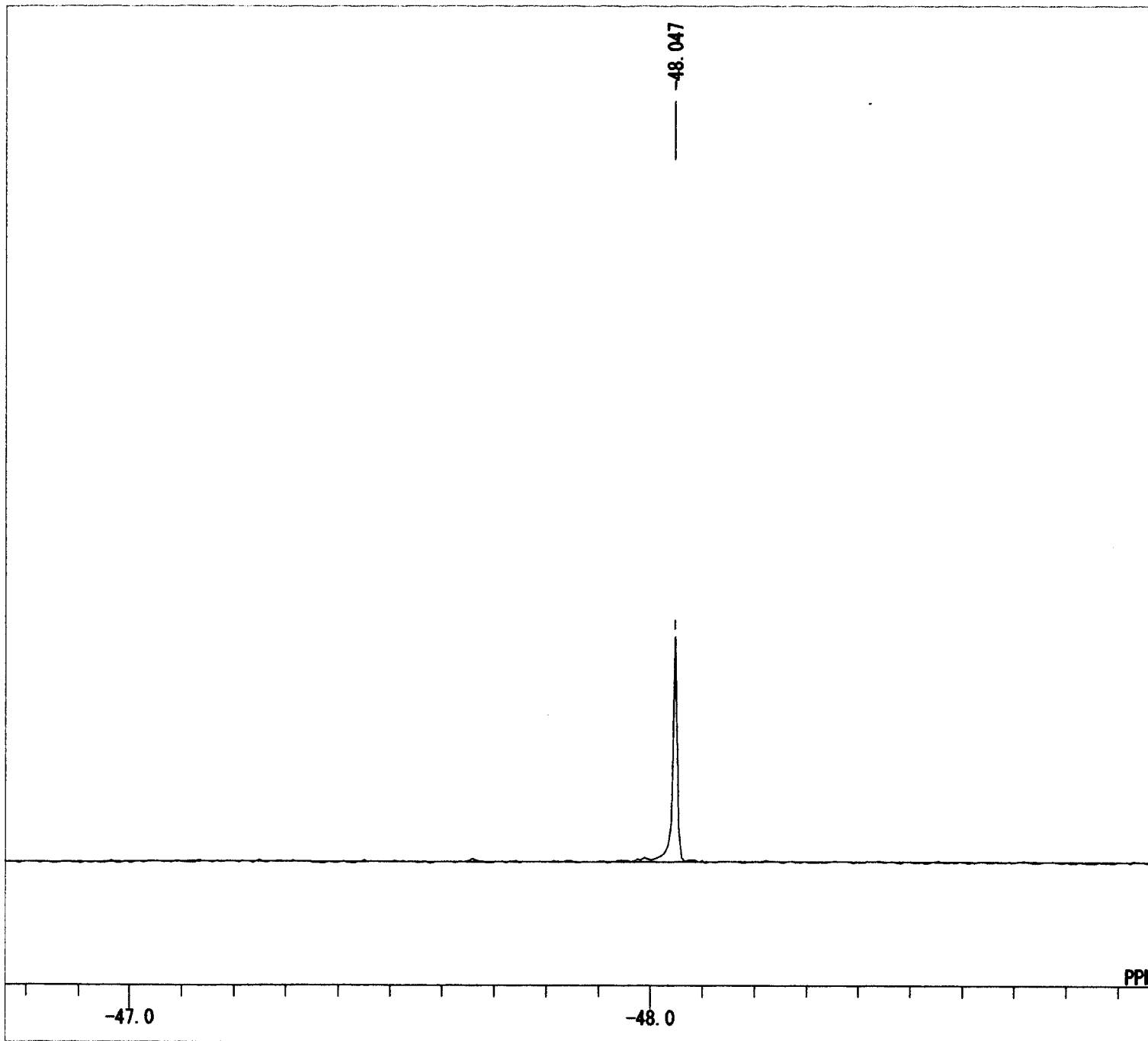


```

MNUF 13C
OBNUC 13C
OFR 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
PW1 6.00 usec
DEADT 19.10 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 32768
SPO 32768
TIMES 512
DUMMY 1
FREQU 27118.64 Hz
FLT 13550 Hz
DELAY 14.80 usec
ACQTM 1.2083 sec
PD 1.7920 sec
ADBIT 16
RGAIN 25
BF 0.60 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD BCM
EXPCM Bilevel. complete. decoupling:
IRNUC 1H
IFR 399.65 MHz
IRSET 124.00 KHz
IRFIN 10500.00 Hz
IRRPW 45 usec
IRATN 511
DFILE CF3-Ph+2diphenylacetylene (13C
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 24
LKPHS 222
LKSIG 1049
CSPED 14 Hz
FILDC
FILDF

```

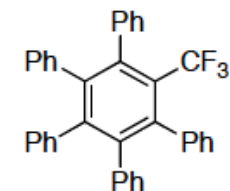




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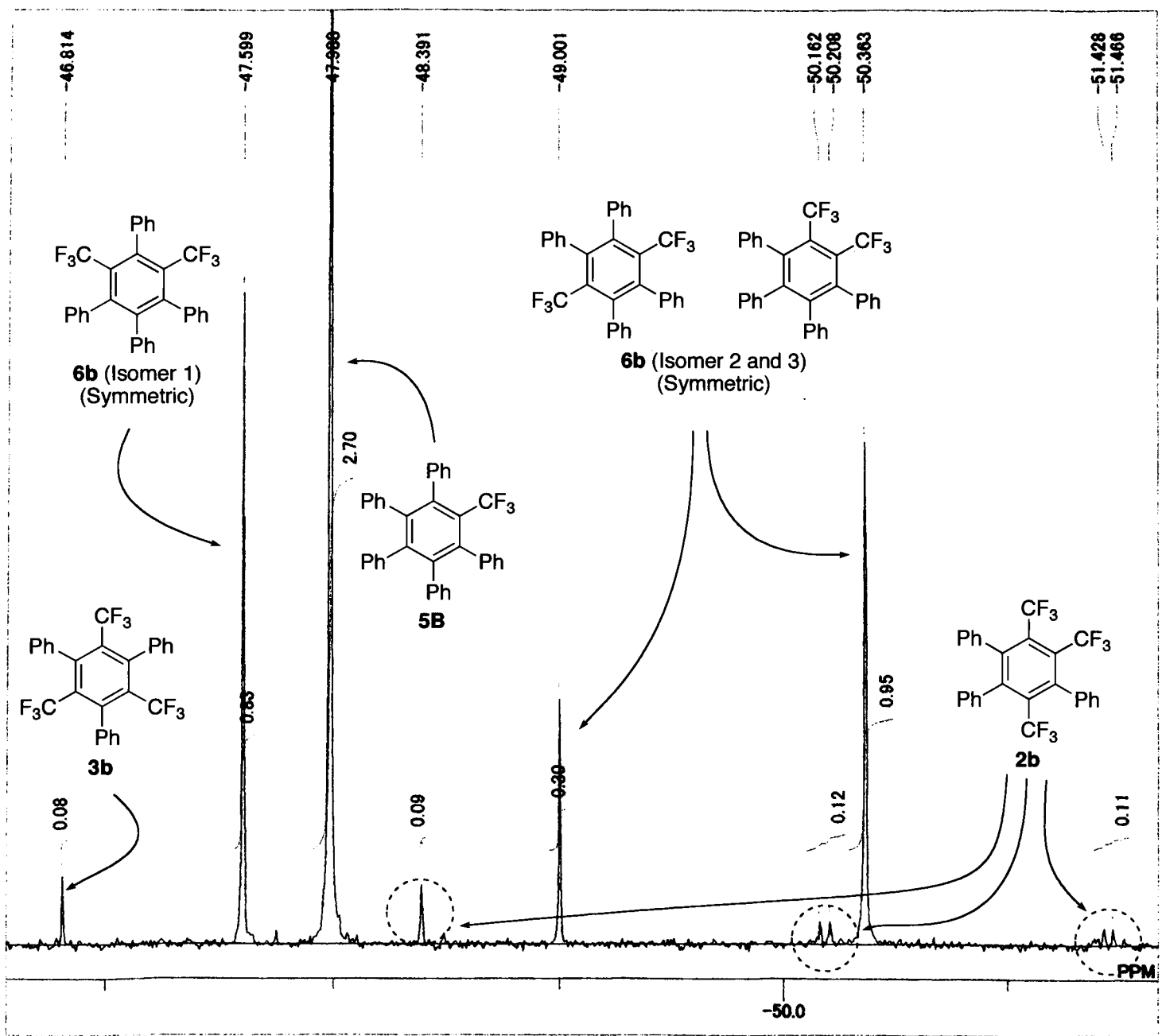
MENUF 19F
OBNUC 19F
OFR      376.05 MHz
OBSET    139.60 KHz
OBFIN    36.10 Hz
PW1      6.00 usec
DEADT    10.00 usec
PREDL    0.20000 msec
IWT      1.0000 msec
POINT    32768
SPO      32768
TIMES    16
DUMMY    1
FREQU    80000.00 Hz
FLT      40000 Hz
DELAY    5.00 usec
ACQTM    0.4096 sec
PD       4.9500 sec
ADBIT    16
RGAIN    14
BF       0.60 Hz
T1       0.00
T2       0.00
T3       90.00
T4       100.00
EXMOD    NON
EXPCM    NON:Single.coupled:PW1_ACQTM
IRNUC    1H
IFR      399.65 MHz
IRSET    124.00 KHz
IRFIN    10500.00 Hz
IRRPW    45 usec
IRATN    511
DFILE    CF3-Ph+2diphenylacetylene (19f
SF        TH5ATFG2
LKSET    61.60 KHz
LKFIN    79.0 Hz
LKLEV    180
LGA1N    27
LKPHS    222
LKSIG    2830
CSPED    14 Hz
FILDC
FILDF

```

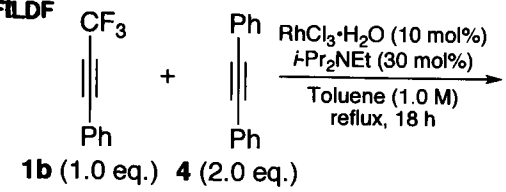


5b

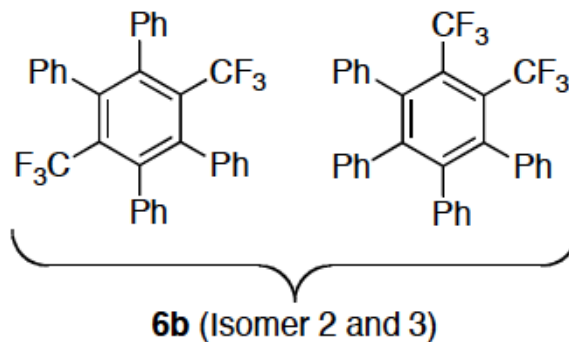
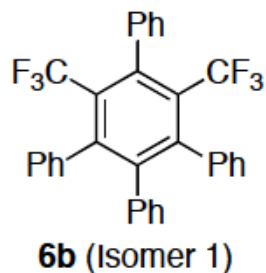
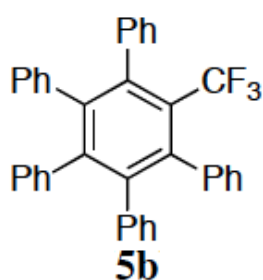
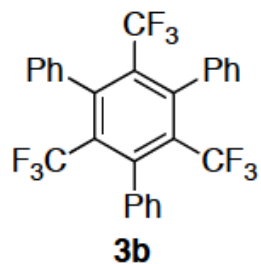
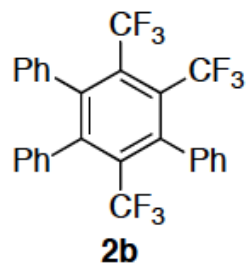
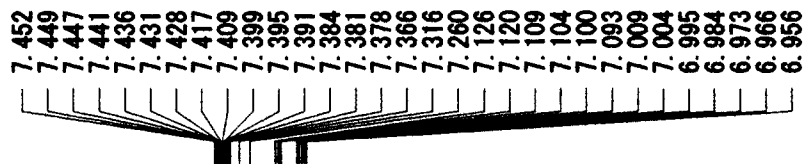
¹⁹F NMR Analysis of the reaction mixture (Total yield for all CF₃-containing compounds : 90%)



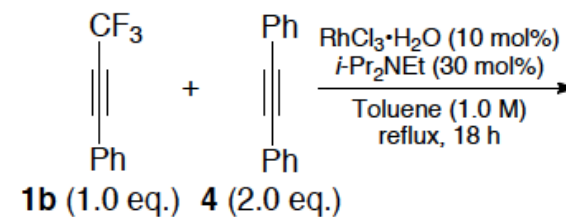
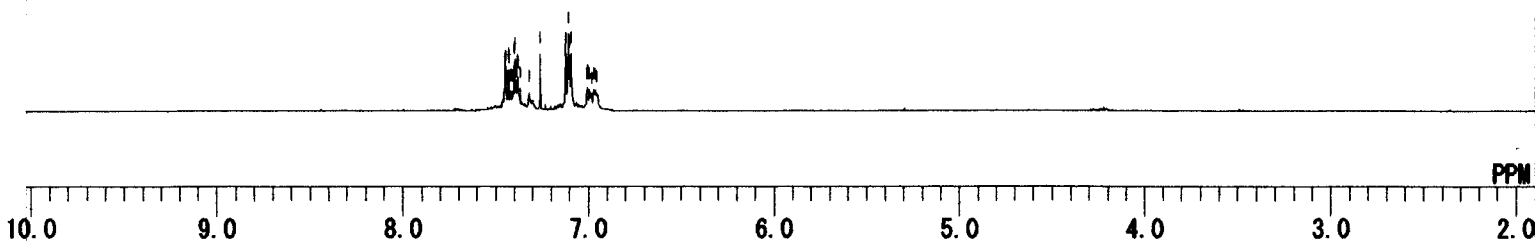
MENUF	19F
OBNUC	19F
OFR	376.05 MHz
OBSET	139.60 KHz
OBFIN	36.10 Hz
PW1	6.00 usec
DEADT	10.00 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	16
DUMMY	1
FREQU	80000.00 Hz
FLT	40000 Hz
DELAY	5.00 usec
ACQTM	0.4096 sec
PD	4.9500 sec
ADBIT	16
RGAIN	14
BF	0.60 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM_PD:
IRNUC	1H
IFR	399.85 MHz
IRSET	124.00 KHz
IRFIN	10500.00 Hz
IRFPW	45 usec
IRATN	511
DFILE	_DEFAULT.ALS
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	24
LKPHS	222
LKSIG	1194
CSPED	14 Hz
FILDC	
FILDF	

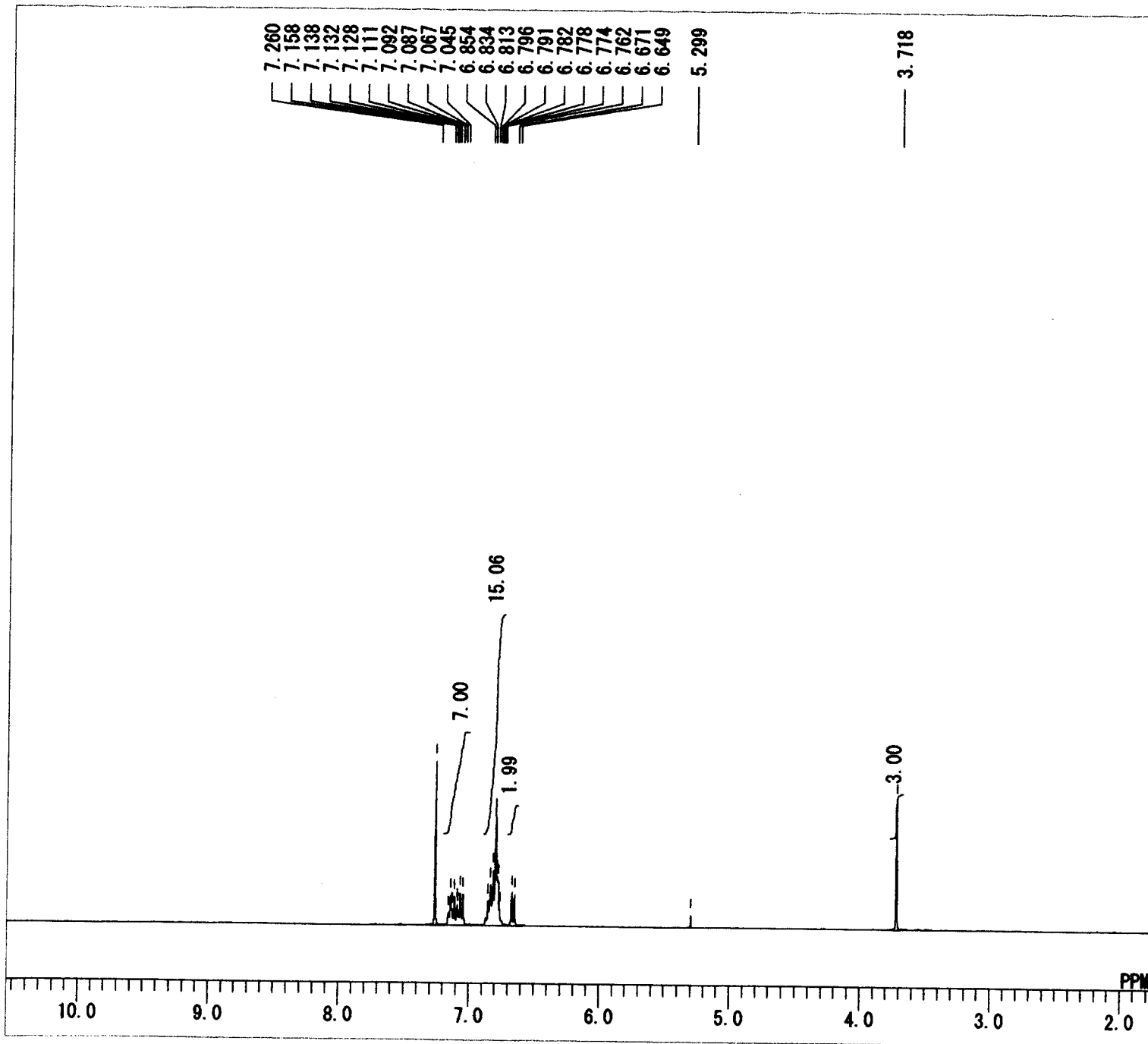


After *one* silica gel column chromatography

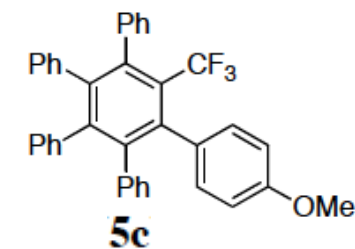


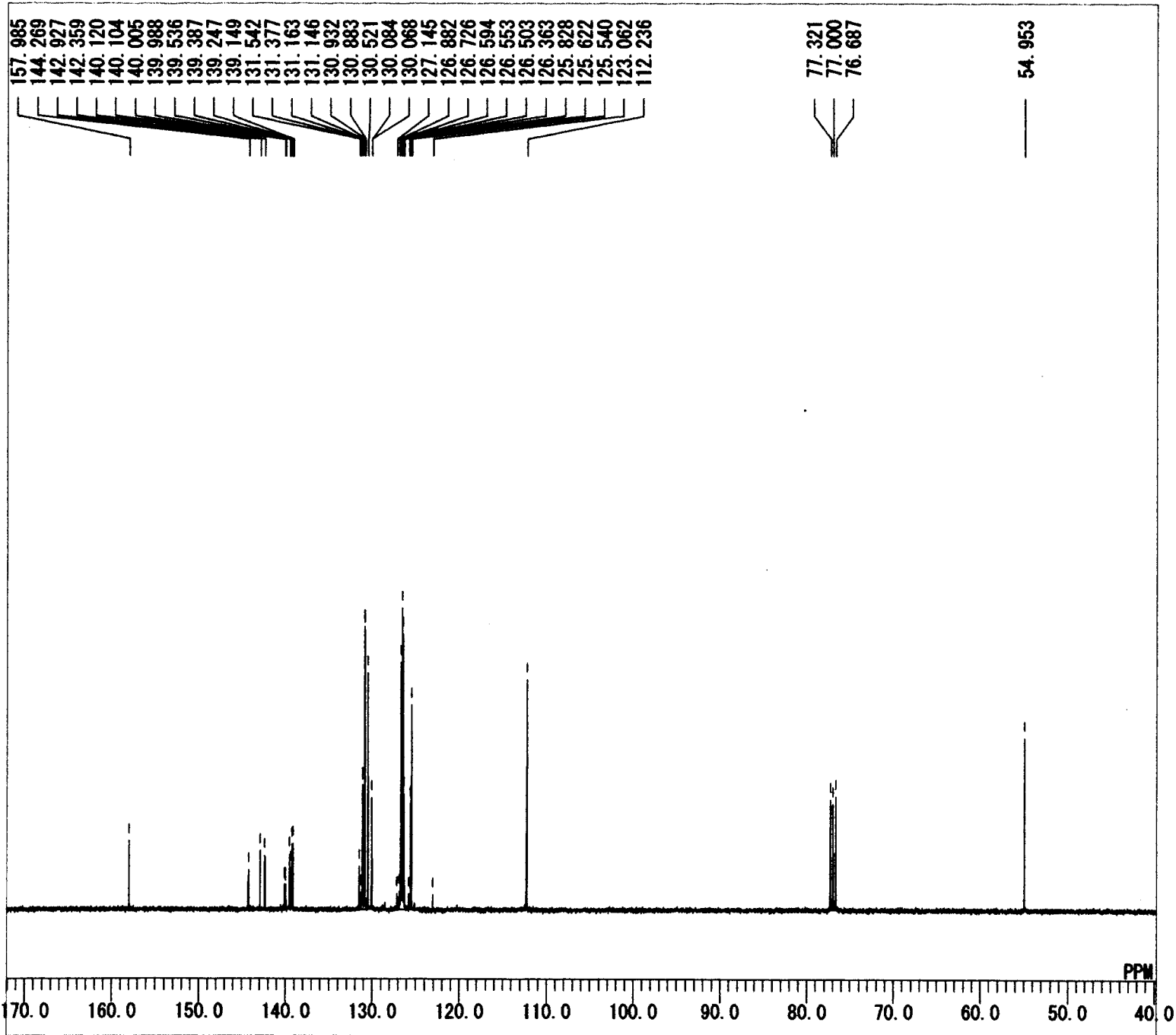
MENUF	1H
OBNUC	1H
OFR	399.65 MHz
OBSET	135.40 KHz
OBFIN	24.90 Hz
PW1	5.20 usec
DEADT	72.40 usec
PREDL	0.2000 msec
IWT	1.0000 msec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7992.01 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	2.0501 sec
PD	4.9500 sec
ADBIT	16
RGAIN	17
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM.
IRNUC	1H
IFR	399.65 MHz
IRSET	136.90 KHz
IRFIN	97.50 Hz
IRRPW	45 usec
IRATN	511
DFILE	bypro-CF3-Ph-diPh(1H).als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	24
LKPHS	219
LKSIG	1638
CSPED	11 Hz
FILDC	
FILDF	



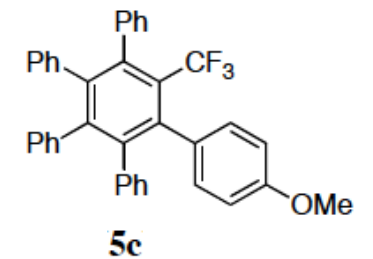


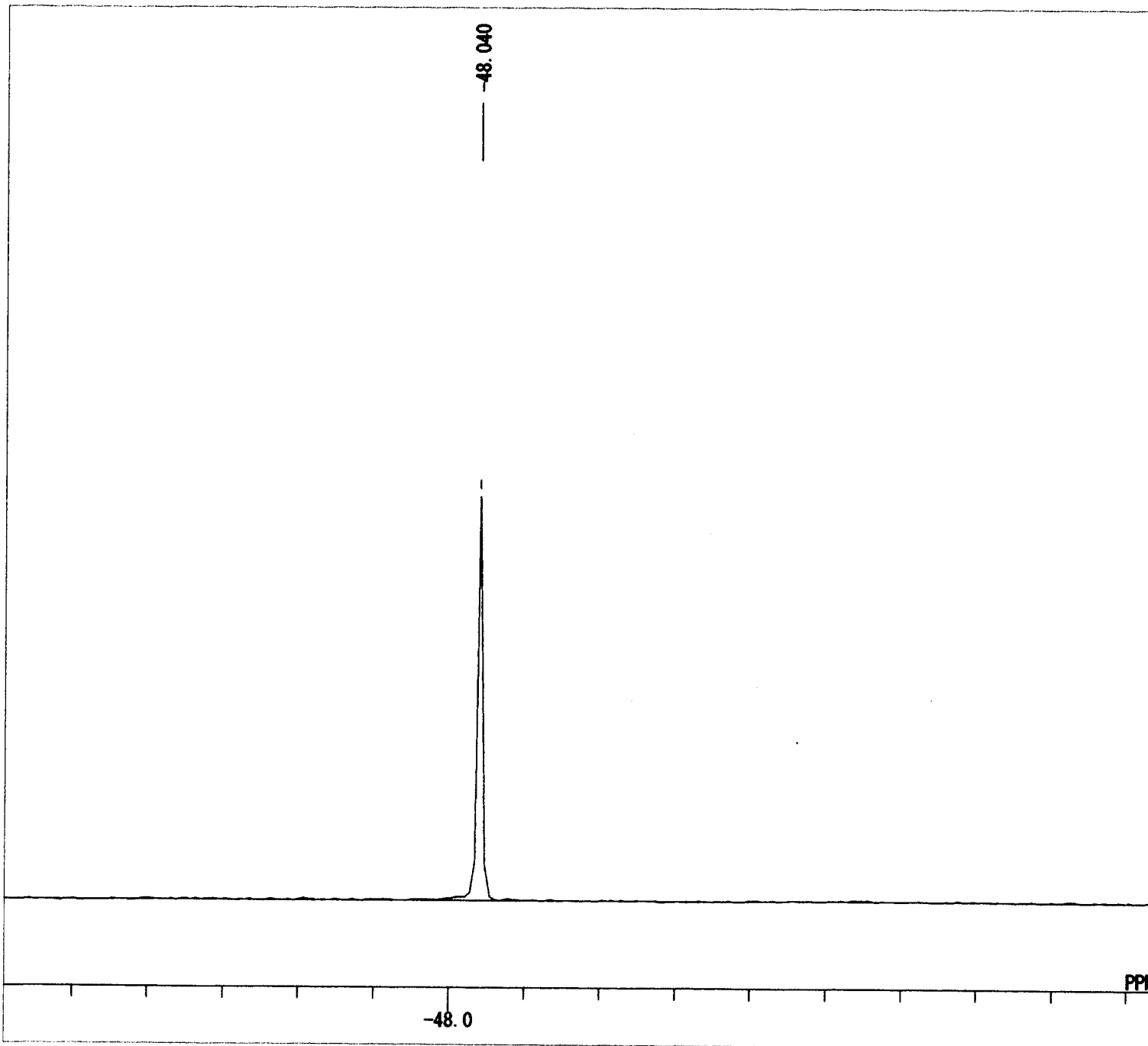
MENUF	1H
OBNUC	1H
OFR	399.65 MHz
OBSET	135.40 KHz
OBFIN	24.90 Hz
PW1	5.20 usec
DEADT	72.40 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7992.01 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	2.0501 sec
PD	4.9500 sec
ADBIT	16
RGAIN	21
BF	0.60 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM
IRNUC	1H
IFR	399.65 MHz
IRSET	136.90 KHz
IRFIN	97.50 Hz
IRRPW	45 usec
IRATN	511
DFILE	CF3-PhOMe+2diphenylacetylene
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	22
LKPHS	222
LKSIG	823
CSPED	13 Hz
FILDC	
FILDF	





MENUF 13C
 OBNUC 13C
 OFR 100.40 MHz
 OBSET 125.00 KHz
 OBFIN 10500.00 Hz
 PW1 6.00 usec
 DEADT 19.10 usec
 PREDL 0.20000 msec
 IWT 1.0000 msec
 POINT 32768
 SPO 32768
 TIMES 512
 DUMMY 1
 FREQU 27118.64 Hz
 FLT 13550 Hz
 DELAY 14.80 usec
 ACQTM 1.2083 sec
 PD 1.7920 sec
 ADBIT 16
 RGA IN 25
 BF 0.60 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD BCM
 EXPCM Bilevel. complete. decoupling:
 IRNUC 1H
 IFR 399.65 MHz
 IRSET 124.00 KHz
 IRFIN 10500.00 Hz
 IRRPW 45 usec
 IRATN 511
 DFILE CF3-PhOMe+2diphenylacetylene
 SF TH5ATFG2
 LKSET 61.60 KHz
 LKFIN 79.0 Hz
 LKLEV 180
 LGAIN 23
 LKPHS 222
 LKSIG 732
 CSPED 13 Hz
 FILDC
 FILDF

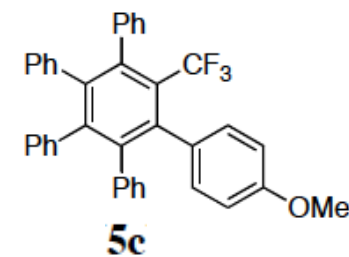




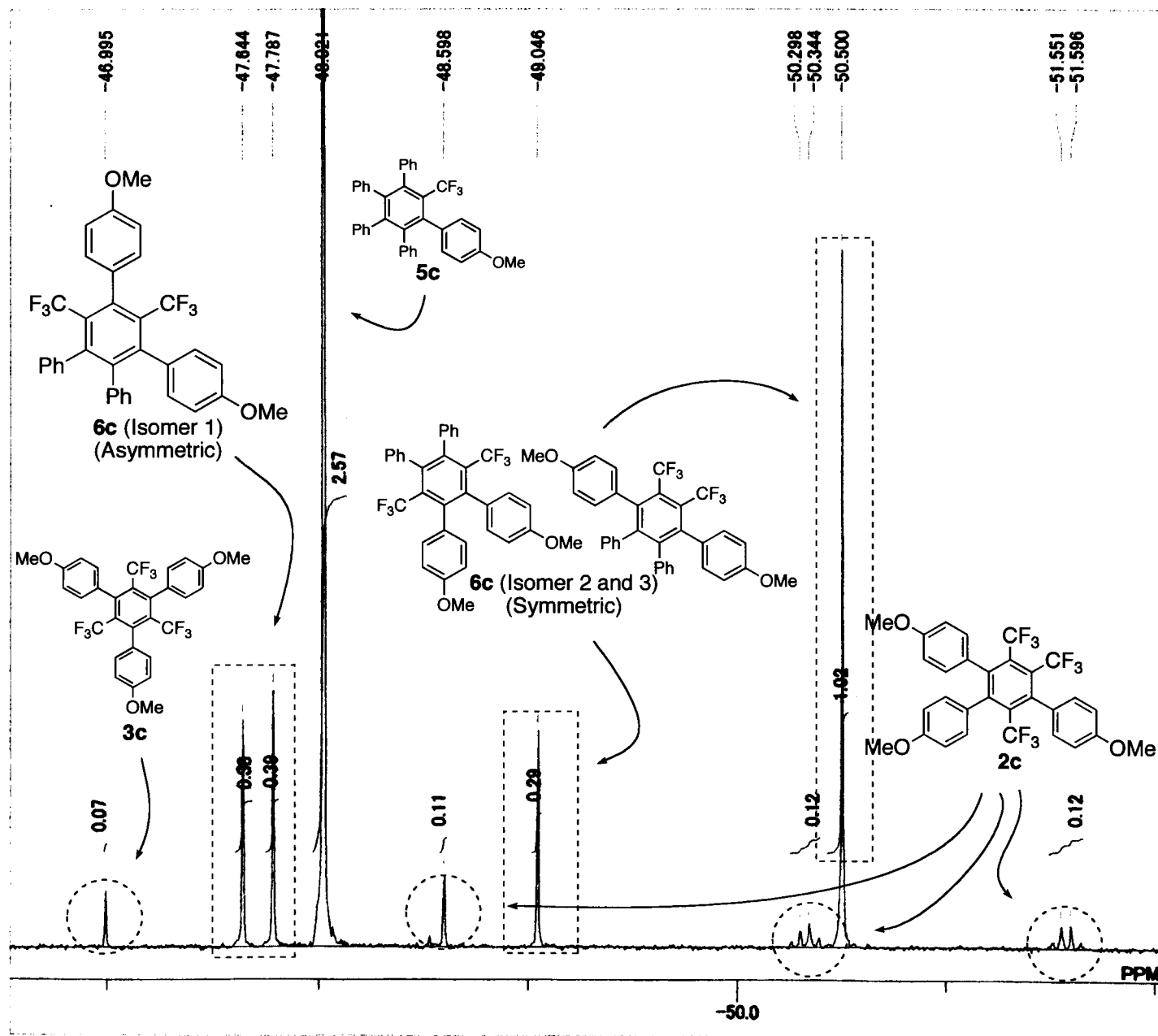
```

MENUF 19F
OBNUC 19F
OFR 376.05 MHz
OBSET 139.60 KHz
OBFIN 36.10 Hz
PW1 6.00 usec
DEADT 10.00 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 32768
SPO 32768
TIMES 16
DUMMY 1
FREQU 80000.00 Hz
FLT 40000 Hz
DELAY 5.00 usec
ACQTM 0.4096 sec
PD 4.9500 sec
ADBIT 16
RGAIN 14
BF 0.60 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM
IRNUC 1H
IFR 399.65 MHz
IRSET 124.00 KHz
IRFIN 10500.00 Hz
IRRPW 45 usec
IRATN 511
DFILE CF3-PhOMe+2diphenylacetylene
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 22
LKPHS 222
LKSIG 842
CSPED 13 Hz
FILDC
FILDF

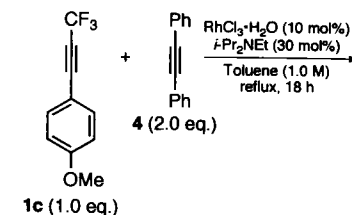
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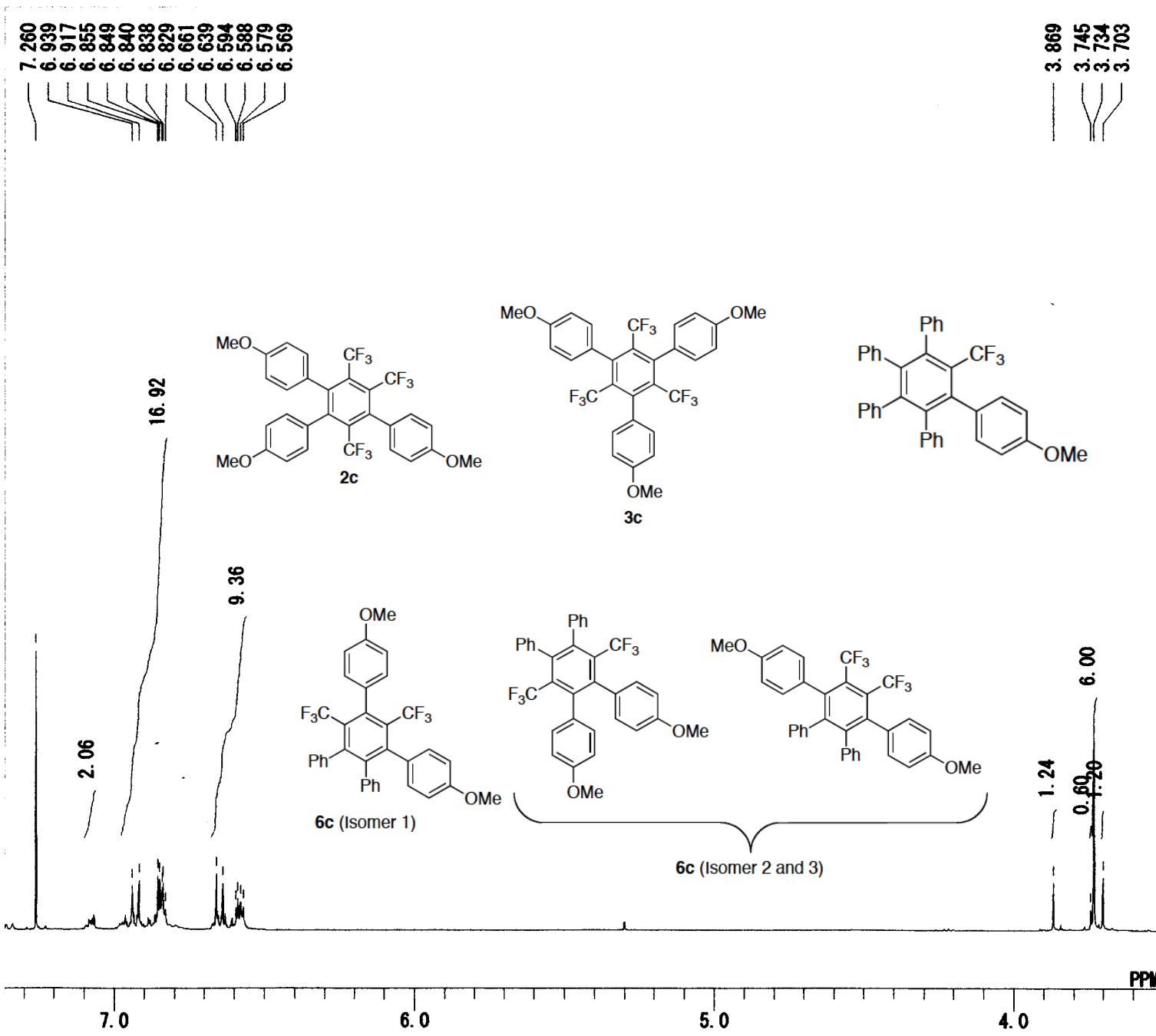
¹⁹F NMR Analysis of the reaction mixture (Total yield for all CF₃-containing compounds : 89%)



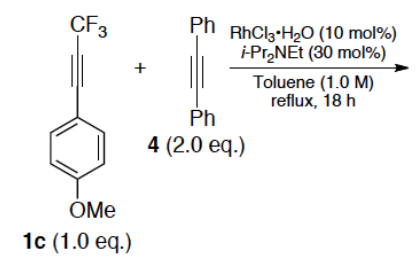
MENUF	19F
OBNUC	19F
OFR	376.05 MHz
OBSET	139.60 KHz
OBFIN	36.10 Hz
PW1	6.00 usec
DEADT	10.00 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	16
DUMMY	1
FREQU	80000.00 Hz
FLT	40000 Hz
DELAY	5.00 usec
ACQTM	0.4086 sec
PD	4.9500 sec
ADBIT	16
RGAIN	14
BF	0.60 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM_PD:
IRNUC	1H
IFR	399.65 MHz
IRSET	124.00 KHz
IRFIN	10500.00 Hz
IRPW	45 usec
IRATN	511
DFILE	_DEFAULT.ALS
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	24
LKPHS	222
LKSIG	1243
CSPED	13 Hz
FILDC	
FILDF	



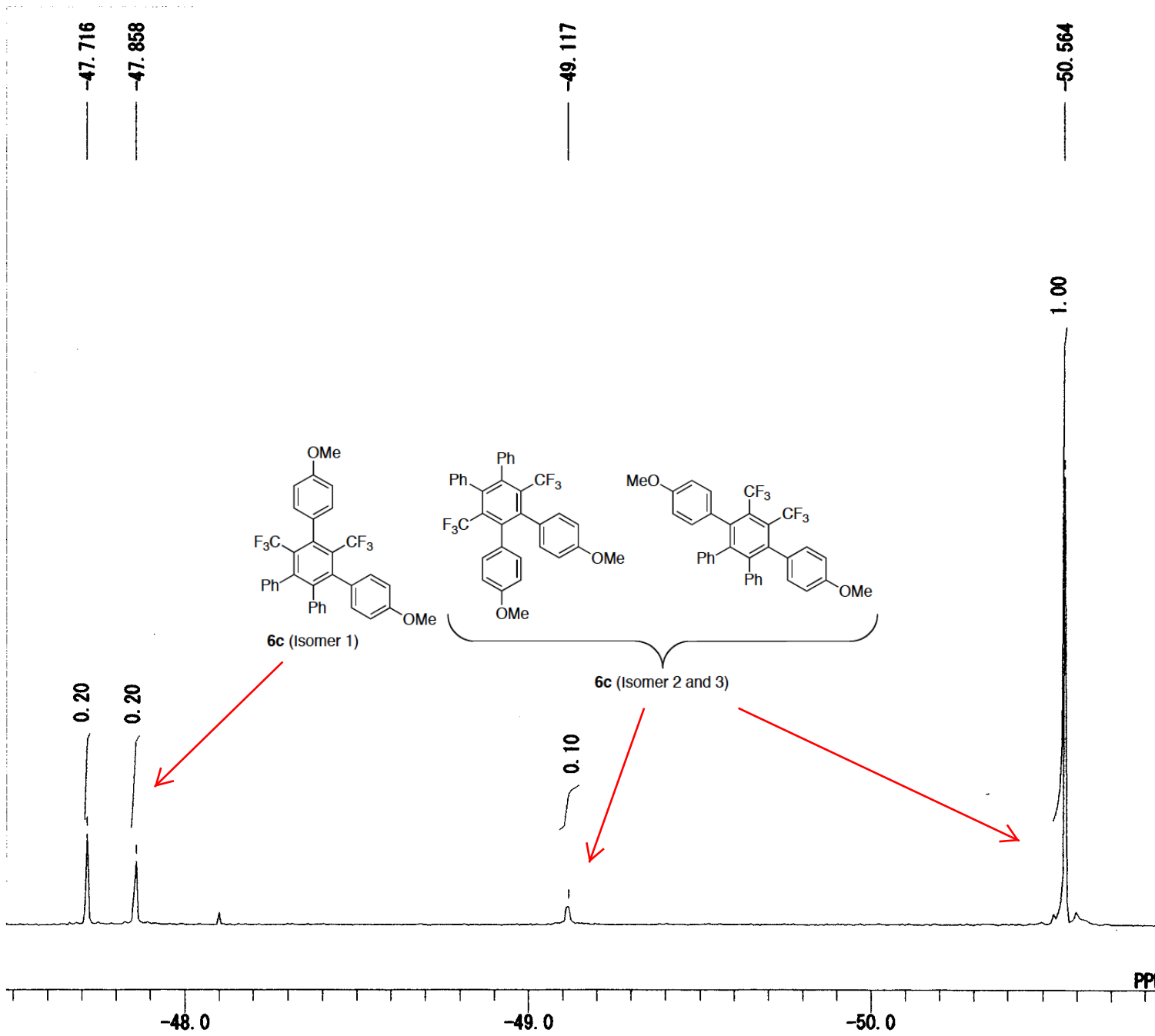
After *one* silica gel column chromatography



MENUF	1H
OBNUC	1H
OFR	399.65 MHz
OBSET	135.40 KHz
OBFIN	24.90 Hz
PW1	5.80 usec
DEADT	72.10 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	16384
SPO	16384
TIMES	32
DUMMY	1
FREQU	7992.01 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	2.0501 sec
PD	4.9500 sec
ADBIT	16
RGAIN	21
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM
IRNUC	1H
IFR	399.65 MHz
IRSET	136.90 KHz
IRFIN	97.50 Hz
IRRPW	45 usec
IRATN	511
DFILE	bypro-CF3-p-MeO-diPh(1H).als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	23
LKPHS	250
LKSIG	1093
CSPED	13 Hz
FILDC	
FILDF	

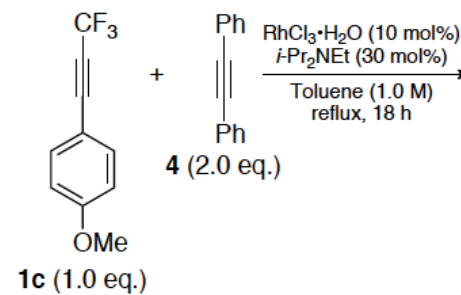


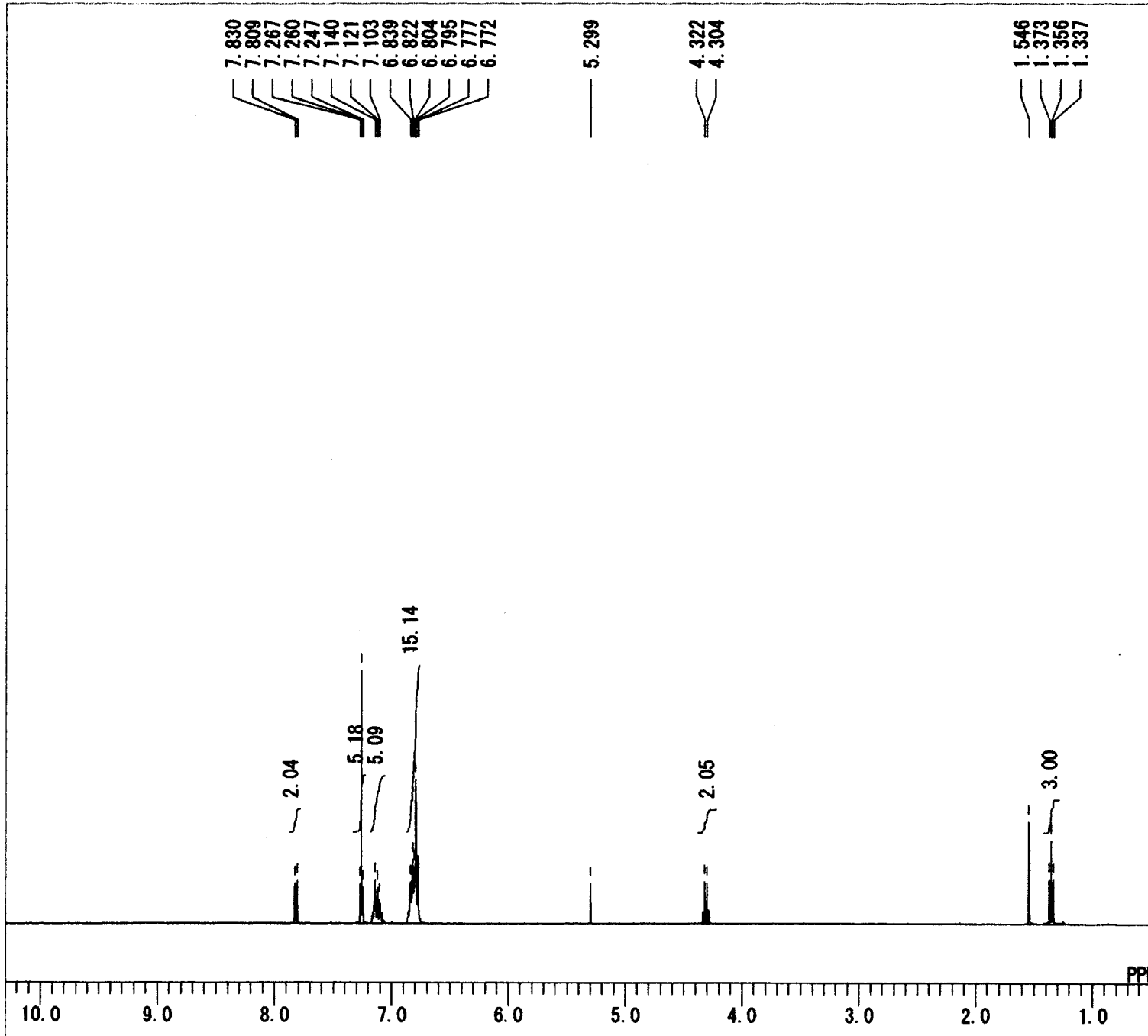
After *one* silica gel column chromatography



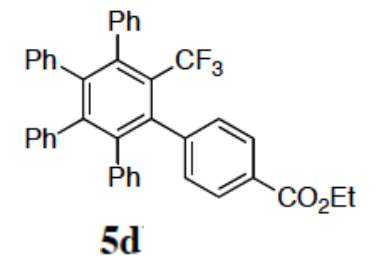
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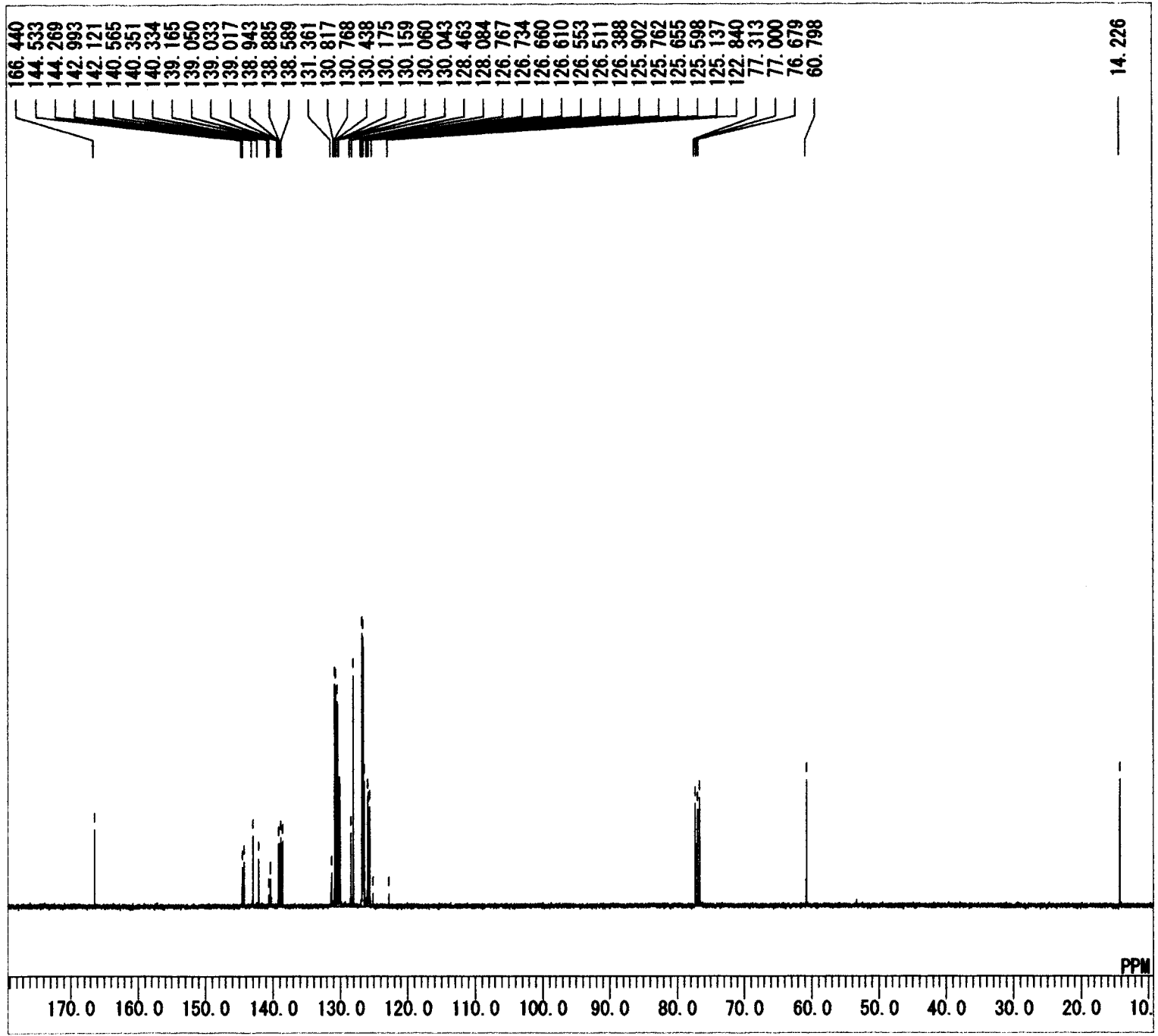
NAME      OBNUC      19F
OFR       376.05 MHz
OBSET     139.60 KHz
OBFIN     36.10 Hz
PW1       6.00 usec
DEADT     10.00 usec
PREDL     0.20000 msec
IWT       1.0000 msec
POINT     32768
SPO       32768
TIMES     16
DUMMY     1
FREQU     80000.00 Hz
FLT       40000 Hz
DELAY     5.00 usec
ACQTM     0.4096 sec
PD        4.9500 sec
ADBIT     16
RGAIN     14
BF        0.10 Hz
T1        0.00
T2        0.00
T3        90.00
T4        100.00
EXMOD     NON
EXPCM     NON:Single.coupled:PW1_ACQTM
IRNUC     1H
IFR       399.65 MHz
IRSET     124.00 KHz
IRFIN     10500.00 Hz
IRRPW     45 usec
IRATN     511
DFILE     bypro-CF3-p-MeO-diPh(19F).al:
SF        TH5ATFG2
LKSET     61.60 KHz
LKFIN     79.0 Hz
LKLEV     180
LGAIN     23
LKPHS     250
LKSIG     1080
CSPED     13 Hz
FILDC
FILDF
    
```





MENUF 1H
 OBNUC 1H
 OFR 399.65 MHz
 OBSET 135.40 KHz
 OBFIN 24.90 Hz
 PW1 5.20 usec
 DEADT 72.40 usec
 PREDL 0.20000 msec
 IWT 1.0000 msec
 POINT 16384
 SPO 16384
 TIMES 8
 DUMMY 1
 FREQU 7992.01 Hz
 FLT 4000 Hz
 DELAY 50.00 usec
 ACQTM 2.0501 sec
 PD 4.9500 sec
 ADBIT 16
 RGAIN 21
 BF 0.10 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD NON
 EXPCM NON:Single.coupled:PW1_ACQTM.
 IRNUC 1H
 IFR 399.65 MHz
 IRSET 136.90 KHz
 IRFIN 97.50 Hz
 IRRPW 45 usec
 IRATN 511
 DFILE CF3-PhCO2Et+2diphenylacetyler
 SF TH5ATFG2
 LKSET 61.60 KHz
 LKFIN 79.0 Hz
 LKLEV 180
 LGAIN 20
 LKPHS 222
 LKSIG 544
 CSPED 12 Hz
 FILDC
 FILDF

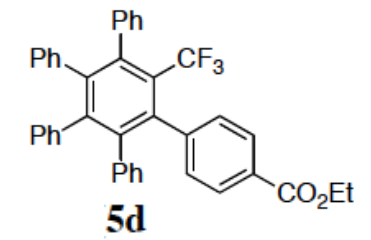


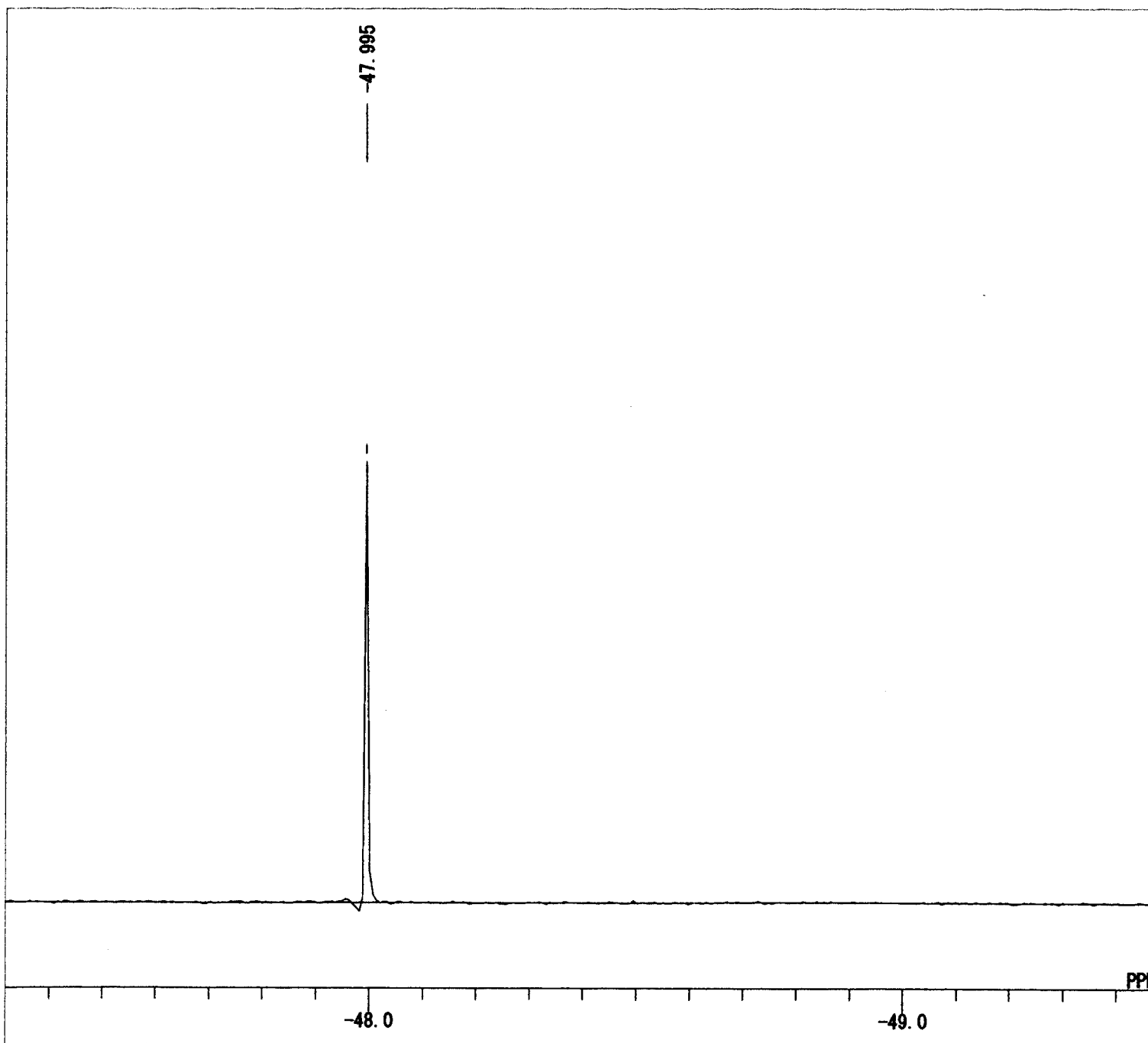


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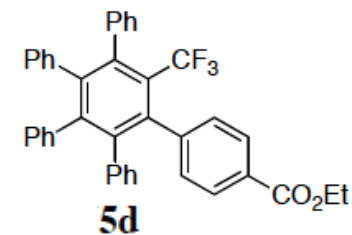
MENUF 13C
OBNUC 13C
OFR 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
PW1 6.00 usec
DEADT 19.10 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 32768
SPO 32768
TIMES 256
DUMMY 1
FREQU 27118.64 Hz
FLT 13550 Hz
DELAY 14.80 usec
ACQTM 1.2083 sec
PD 1.7920 sec
ADBIT 16
RGAIN 26
BF 0.60 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD BCM
EXPCM Bilevel. complete. decoupling:
IRNUC 1H
IFR 399.65 MHz
IRSET 124.00 KHz
IRFIN 10500.00 Hz
IRRPW 45 usec
IRATN 511
DFILE CF3-PhCO2Et+2diphenylacetyler
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 24
LKPHS 222
LKSIG 1130
CSPED 11 Hz
FILDC
FILDF

```

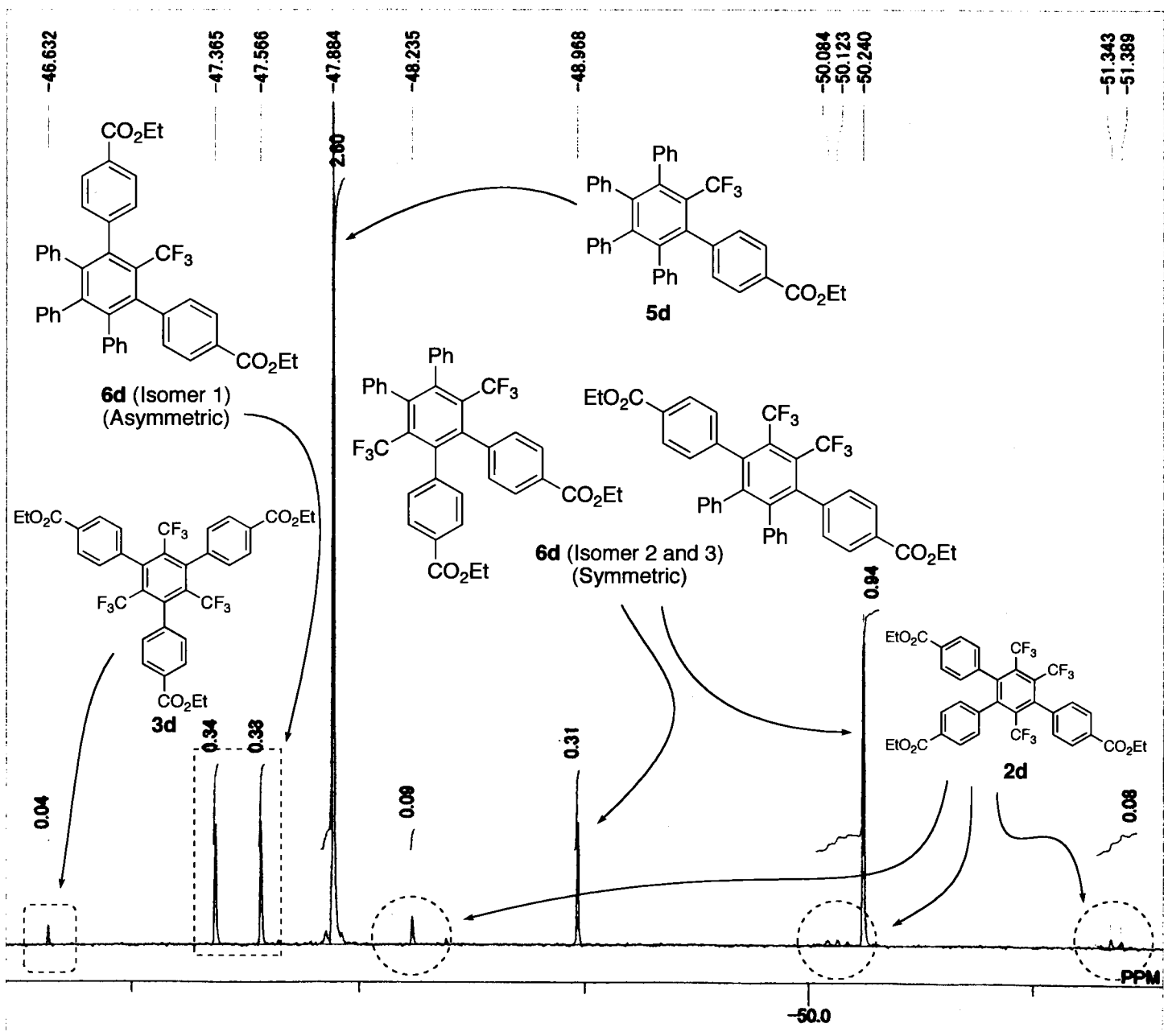




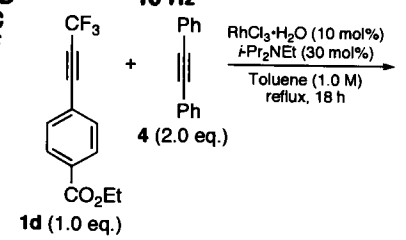
MENUF 19F
 OBNUC 19F
 OFR 376.05 MHz
 OBSET 139.60 KHz
 OBFIN 36.10 Hz
 PW1 6.00 usec
 DEADT 10.00 usec
 PREDL 0.20000 msec
 IWT 1.0000 msec
 POINT 32768
 SPO 32768
 TIMES 16
 DUMMY 1
 FREQU 80000.00 Hz
 FLT 40000 Hz
 DELAY 5.00 usec
 ACQTM 0.4096 sec
 PD 4.9500 sec
 ADBIT 16
 RGAIN 14
 BF 0.10 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD NON
 EXPCM NON:Single.coupled:PW1_ACQTM
 IRNUC 1H
 IFR 399.65 MHz
 IRSET 124.00 KHz
 IRFIN 10500.00 Hz
 IRRPW 45 usec
 IRATN 511
 DFILE CF3-PhCO2Et+2diphenylacetyl
 SF TH5ATFG2
 LKSET 61.60 KHz
 LKFIN 79.0 Hz
 LKLEV 180
 LGAIN 20
 LKPHS 222
 LKSIG 551
 CSPED 13 Hz
 FILDC
 FILDF



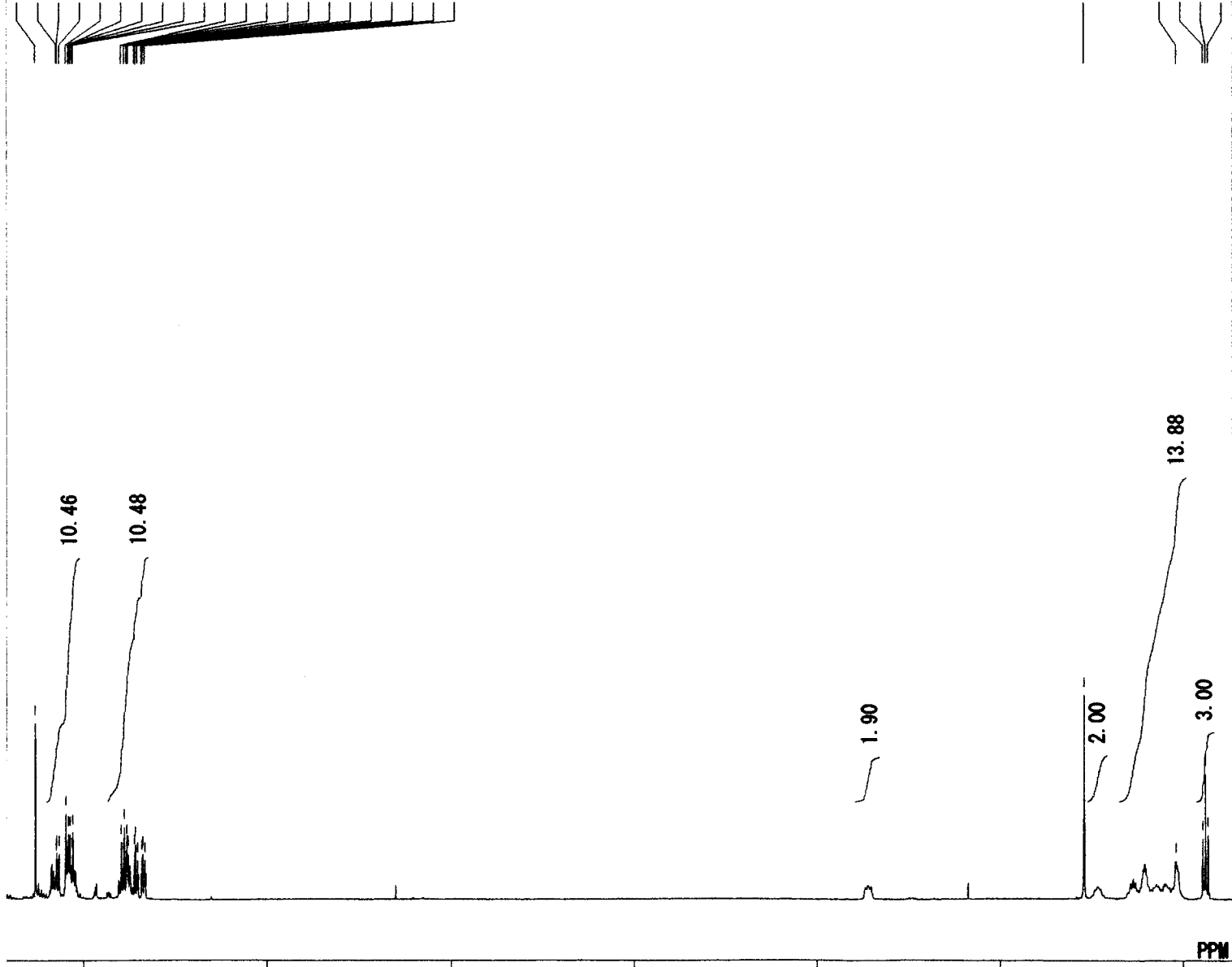
¹⁹F NMR Analysis of the reaction mixture (Total yield for all CF₃-containing compounds : 76%)



Parameter	Value
MENUF	19F
OBNUC	19F
OFR	376.05 MHz
OBSET	139.60 KHz
OBFIN	38.10 Hz
PW1	6.00 usec
DEADT	10.00 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	16
DUMMY	1
FREQU	80000.00 Hz
FLT	40000 Hz
DELAY	5.00 usec
ACQTM	0.4096 sec
PD	4.9500 sec
ADBIT	16
RGAIN	14
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM_PD:
IRNUC	1H
IFR	399.65 MHz
IRSET	124.00 KHz
IRFIN	10500.00 Hz
IRFPW	45 usec
IRATN	511
DFILE	DEFAULT.ALS
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	23
LKPHS	222
LKSIG	926
CSPED	13 Hz
FILDC	
FILDF	



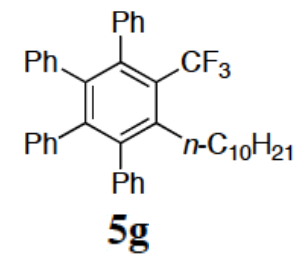
7.260
7.147
7.145
7.133
7.094
7.082
7.080
7.072
7.061
7.057
6.792
6.778
6.776
6.764
6.760
6.757
6.720
6.717
6.704
6.681
6.677
6.664



PPM

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MENUF
OBNUC 1H
OFR 500.13 MHz
OBSET 3.08 KHz
OBF IN 8.51 Hz
PW1 10.00 usec
DEADT 6.00 usec
PREDL 1.00000 msec
IWT 1.0000 msec
POINT 32768
SPO 32768
TIMES 8
DUMMY 2
FREQU 10330.58 Hz
FLT 2000 Hz
DELAY 0.00 usec
ACQTM 0.0000 sec
PD 0.0000 sec
ADBIT 16
RGAIN 322
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD zg30
EXPCM
IRNUC
IFR 0.00 MHz
IRSET 0.00 KHz
IRFIN 0.00 Hz
IRRPW 0 usec
IRATN 0
DFILE CF3-C10H21-diPh(1H).als
SF
LKSET 0.00 KHz
LKFIN 0.0 Hz
LKLEV 0
LGA IN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
  
```

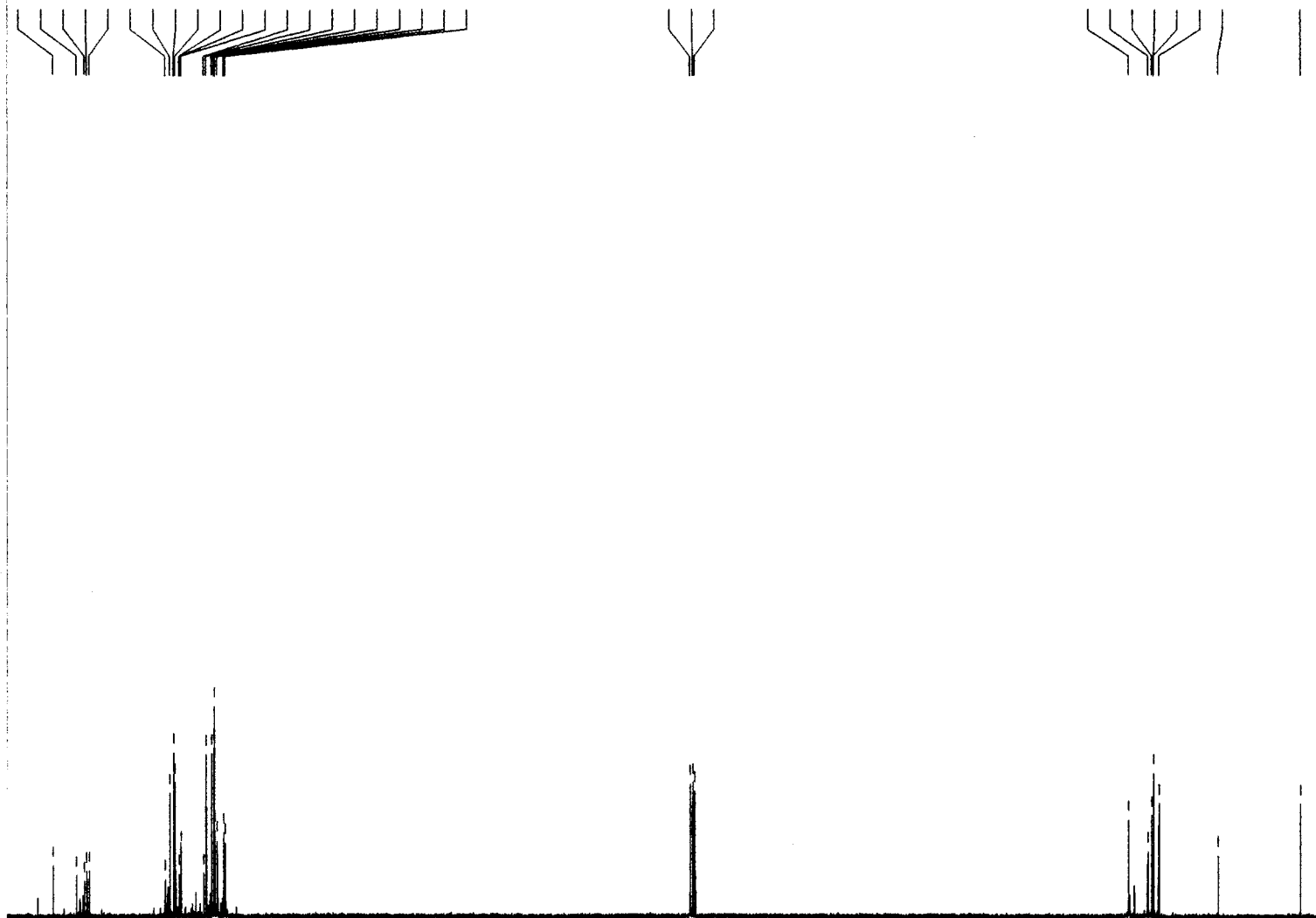


143.060
140.677
139.824
139.548
139.314
131.502
131.007
130.555
130.424
130.016
129.877
129.863
127.509
127.283
126.693
126.474
126.408
126.387
126.131
125.446
125.315

77.255
77.000
76.745

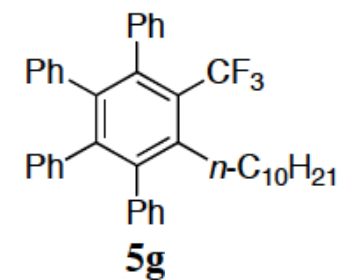
31.891
29.924
29.494
29.311
29.275
28.765
22.673

14.103

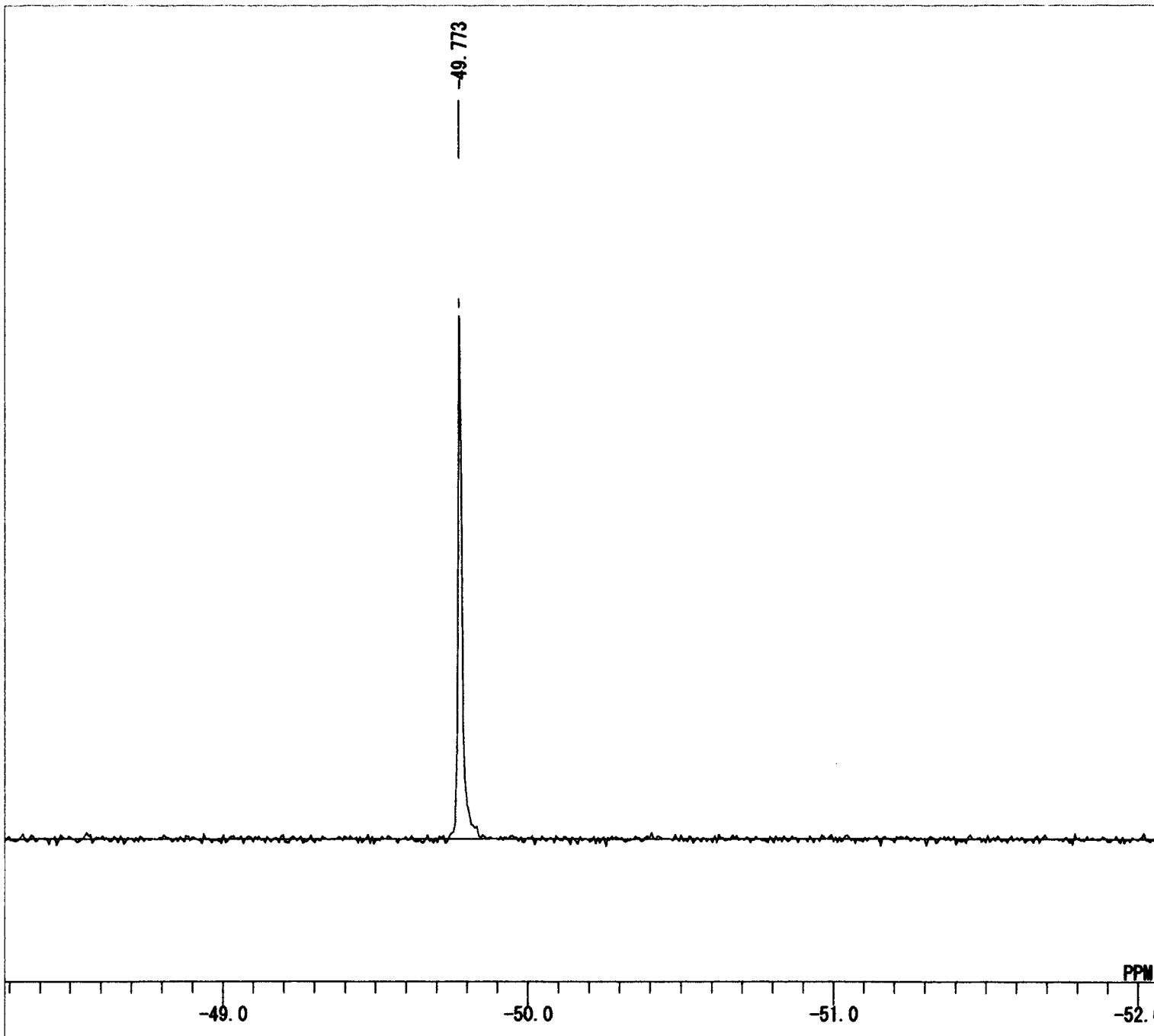


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MENUF
OBNUC 13C
OFR 125.77 MHz
OBSET 0.36 KHz
OBF IN 4.30 Hz
PW1 10.00 usec
DEADT 6.00 usec
PREDL 1.0000 msec
IWT 1.0000 msec
POINT 32768
SPO 32768
TIMES 8
DUMMY 2
FREQU 30030.03 Hz
FLT 2000 Hz
DELAY 0.00 usec
ACQTM 0.0000 sec
PD 0.0000 sec
ADBIT 16
RGAIN 3649
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD zpg30
EXPCM
IRNUC
IFR 0.00 MHz
IRSET 0.00 KHz
IRF IN 0.00 Hz
IRRPW 0 usec
IRATN 0
DFILE CF3-C10H21-diPh(13C).als
SF
LKSET 0.00 KHz
LKF IN 0.0 Hz
LKLEV 0
LGA IN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
  
```

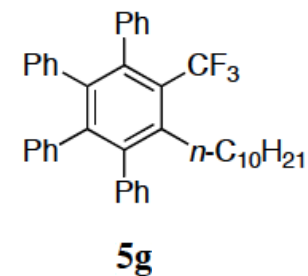


140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 PPM

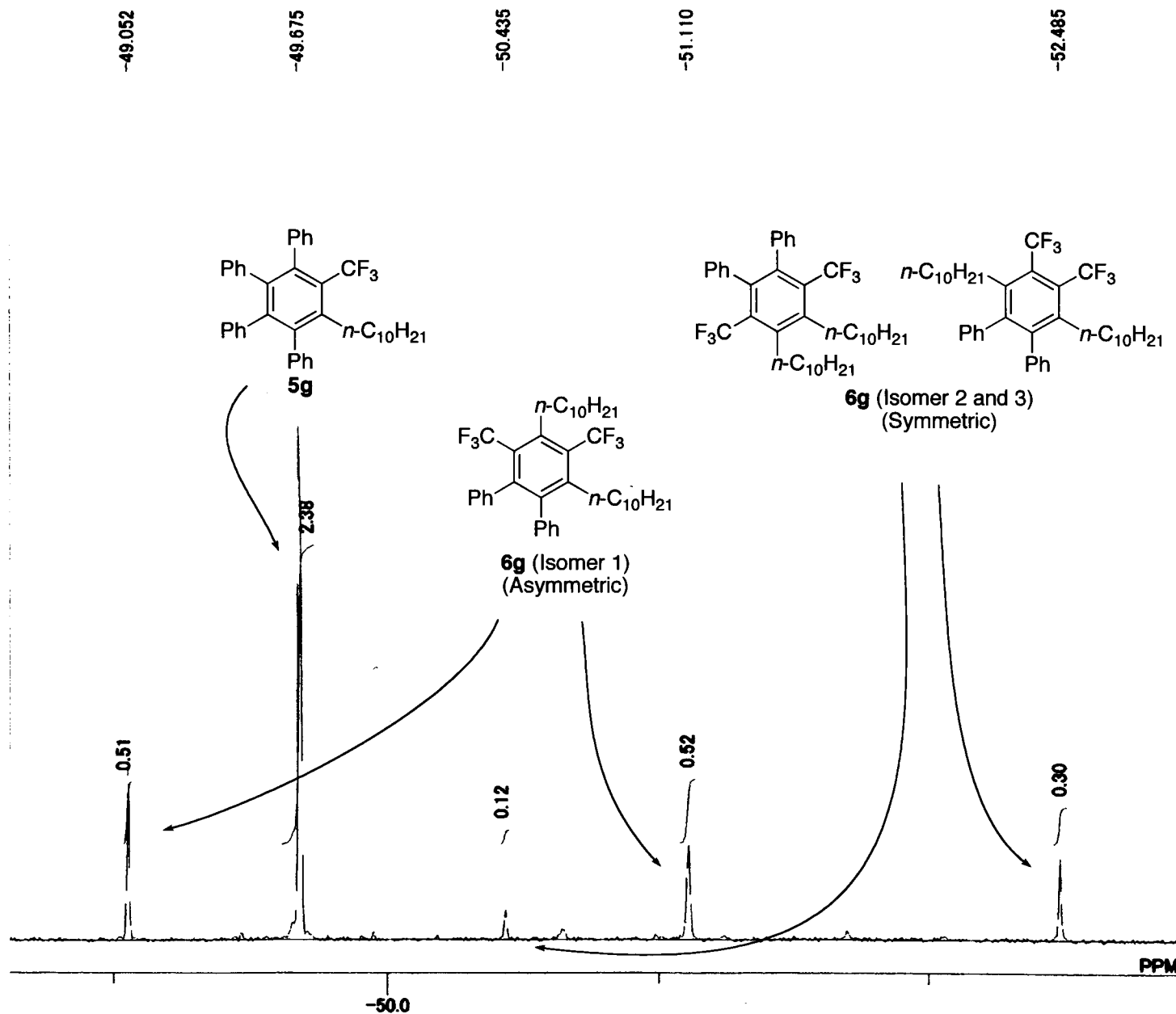


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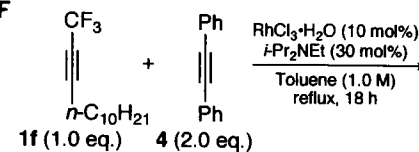
MENUF 19F
OBNUC 19F
OFR      376.05 MHz
OBSET    139.60 KHz
OBFIN    36.10 Hz
PW1      6.00 usec
DEADT    10.00 usec
PREDL    0.20000 msec
IWT      1.0000 msec
POINT    32768
SPO      32768
TIMES    16
DUMMY    1
FREQU    80000.00 Hz
FLT      40000 Hz
DELAY    5.00 usec
ACQTM    0.4096 sec
PD       4.9500 sec
ADBIT    16
RGAIN    15
BF       0.00 Hz
T1       0.00
T2       0.00
T3       90.00
T4       100.00
EXMOD    NON
EXPCM    NON:Single.coupled:PW1_ACQTM
IRNUC    1H
IFR      399.65 MHz
IRSET    124.00 KHz
IRFIN    10500.00 Hz
IRRPW    45 usec
IRATN    511
DFILE    CF3-C10H21+2diphenylacetylene
SF       TH5ATFG2
LKSET    61.60 KHz
LKFIN    79.0 Hz
LKLEV    180
LGAIN    22
LKPHS    212
LKSIG    671
CSPED    11 Hz
FILDC
FILDF
  
```



¹⁹F NMR Analysis of the reaction mixture (Total yield for all CF₃-containing compounds : 73%)



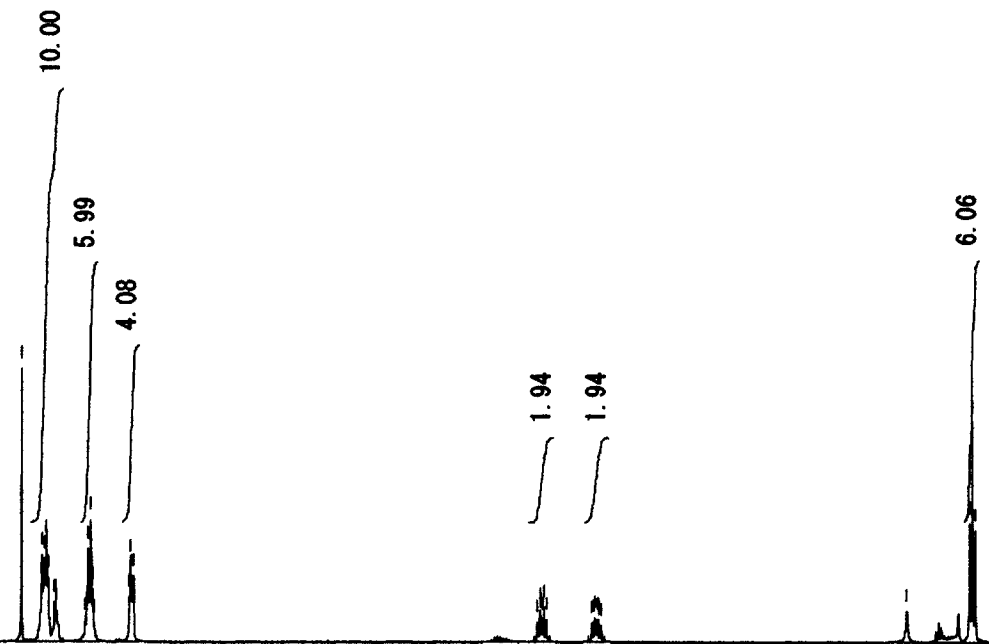
MENUF	19F
OBNUC	19F
OFR	376.05 MHz
OBSET	139.60 KHz
OBFIN	36.10 Hz
PW1	6.00 usec
DEADT	10.00 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	16
DUMMY	1
FREQU	80000.00 Hz
FLT	40000 Hz
DELAY	5.00 usec
ACQTM	0.4098 sec
PD	4.9500 sec
ADBIT	16
RGAIN	15
BF	0.00 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM_PD:
IRNUC	1H
IFR	399.65 MHz
IRSET	124.00 KHz
IRFIN	10500.00 Hz
IRRPW	45 usec
IRATN	511
DFILE	DEFAULT.ALS
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	22
LKPHS	212
LKSIG	668
CSPED	11 Hz
FILDC	
FILDF	



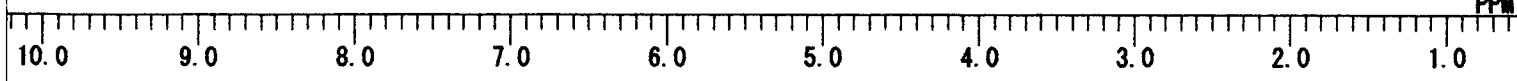
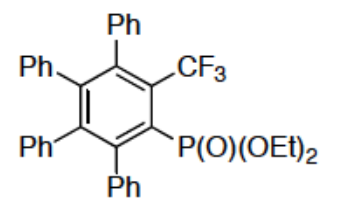
7.260
7.131
7.126
7.115
7.110
7.104
7.098
7.098
7.048
7.042
7.033
6.850
6.835
6.822
6.817
6.810
6.804
6.795
6.569
6.565
6.560
6.545
6.540

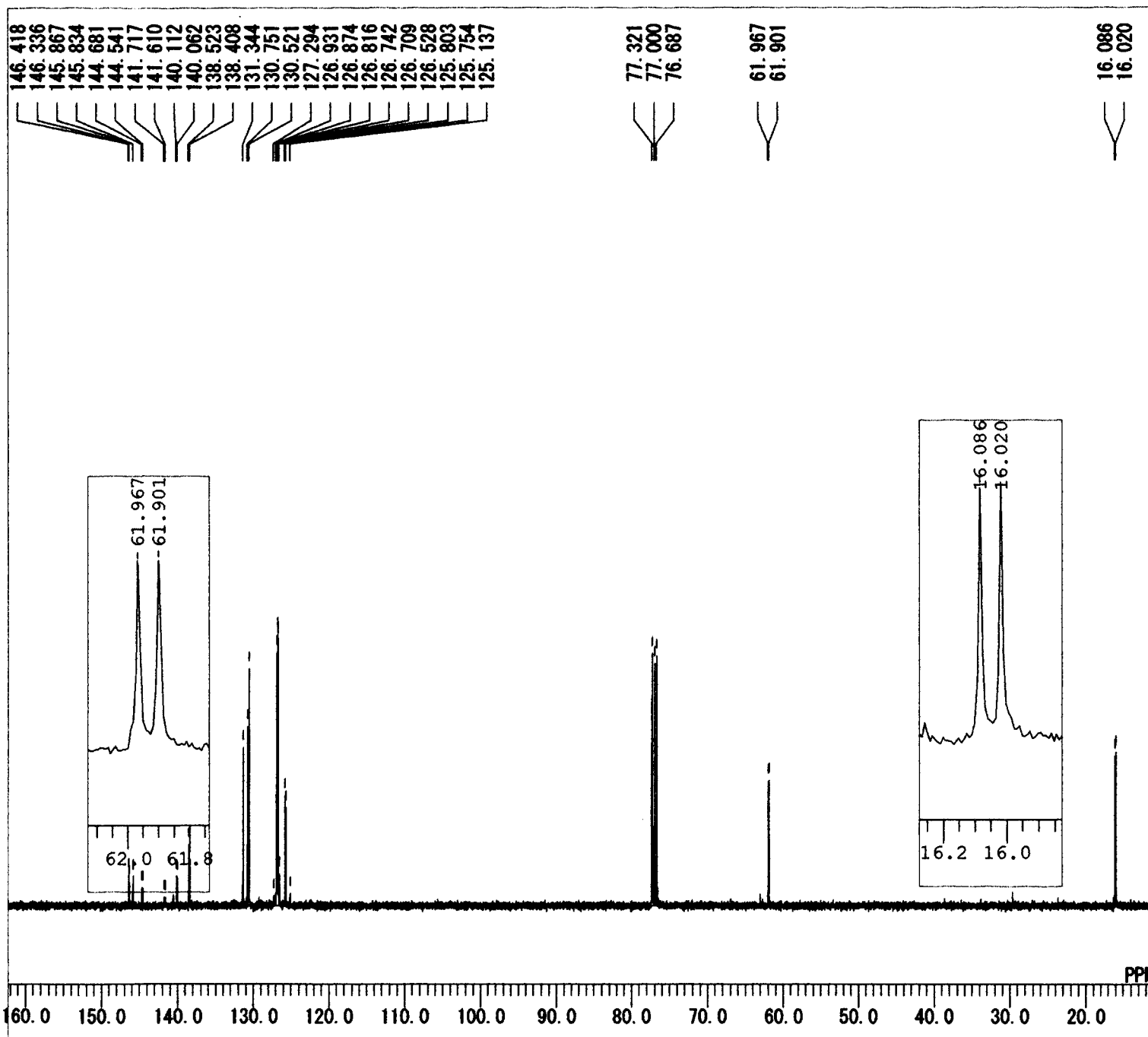
3.955
3.936
3.929
3.918
3.911
3.894
3.607
3.590
3.585
3.568
3.564
3.559
3.542

1.584
1.182
1.165
1.147



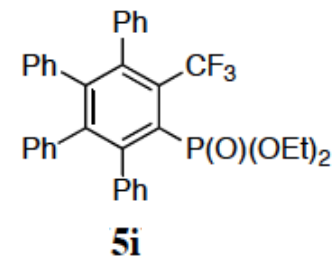
MENUF 1H
OBNUC 1H
OFR 399.65 MHz
OBSET 135.40 KHz
OBFIN 24.90 Hz
PW1 5.80 usec
DEADT 72.10 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7992.01 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 2.0501 sec
PD 4.9500 sec
ADBIT 16
RGAIN 18
BF 0.00 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM.
IRNUC 1H
IFR 399.65 MHz
IRSET 136.90 KHz
IRFIN 97.50 Hz
IRRPW 45 usec
IRATN 511
DFILE CF3-P(0)(OEt)2+2diphenylacet;
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 22
LKPHS 231
LKSIG 739
CSPED 12 Hz
FILDC
FILDF

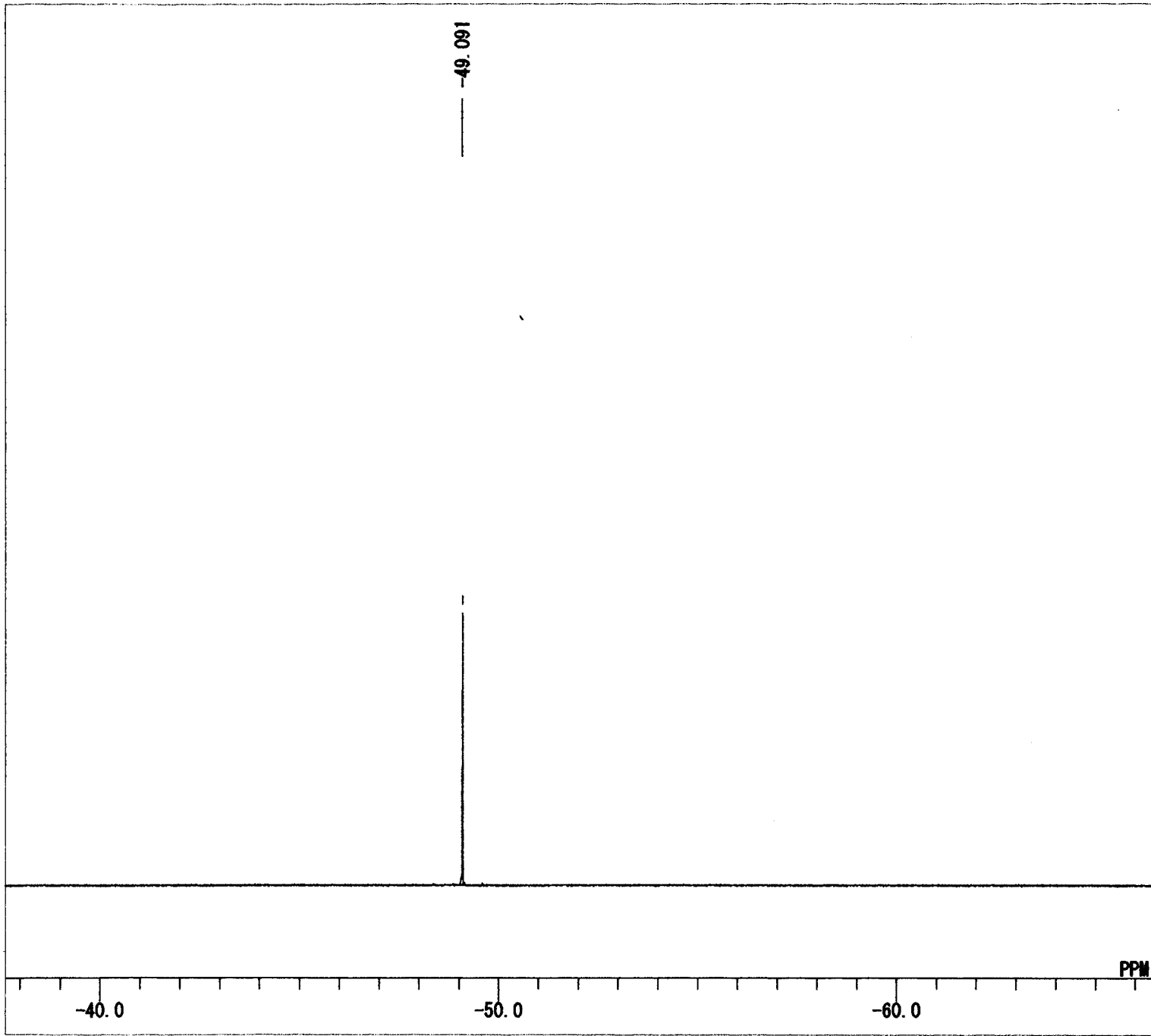




```

MENUF 13C
OBNUC 13C
OFR 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
PW1 6.00 usec
DEADT 19.10 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 32768
SPO 32768
TIMES 1024
DUMMY 1
FREQU 27118.64 Hz
FLT 13550 Hz
DELAY 14.80 usec
ACQTM 1.2083 sec
PD 1.7920 sec
ADBIT 16
RGAIN 26
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD BCM
EXPCM Bilevel, complete decoupling:
IRNUC 1H
IFR 399.65 MHz
IRSET 124.00 KHz
IRFIN 10500.00 Hz
IRRPW 45 usec
IRATN 511
DFILE CF3-P(O)(OEt)2+2diphenylacet
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 25
LKPHS 231
LKSIG 1153
CSPED 13 Hz
FILDC
FILDF
  
```

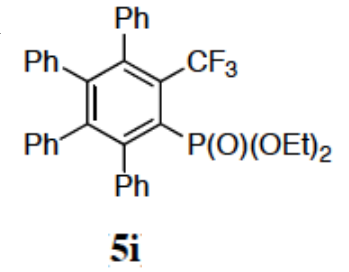


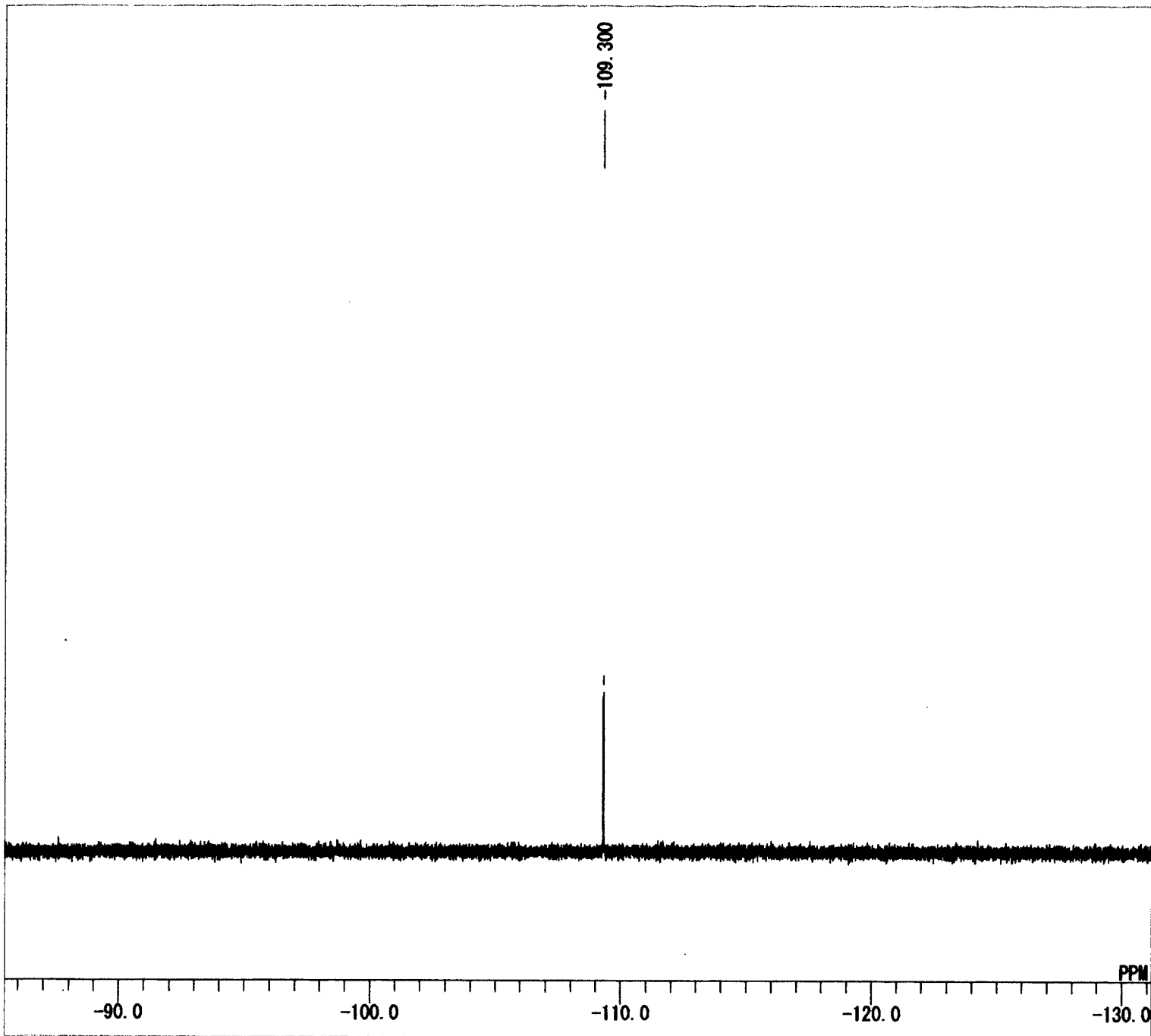


```

MNUF 19F
OBNUC 19F
OFR 376.05 MHz
OBSET 139.60 KHz
OBFIN 36.10 Hz
PW1 6.00 usec
DEADT 10.00 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 32768
SPO 32768
TIMES 16
DUMMY 1
FREQ 80000.00 Hz
FLT 40000 Hz
DELAY 5.00 usec
ACQTM 0.4096 sec
PD 4.9500 sec
ADBIT 16
RGAIN 14
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM
IRNUC 1H
IFR 399.65 MHz
IRSET 124.00 KHz
IRFIN 10500.00 Hz
IRRPW 45 usec
IRATN 511
DFILE CF3-P(0)(OEt)2+2diphenylacet;
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGA IN 24
LKPHS 231
LKSIG 1510
CSPED 14 Hz
FILDC
FILDF

```

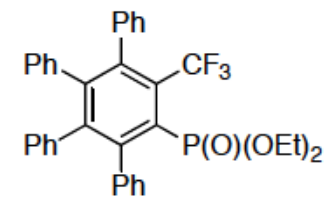




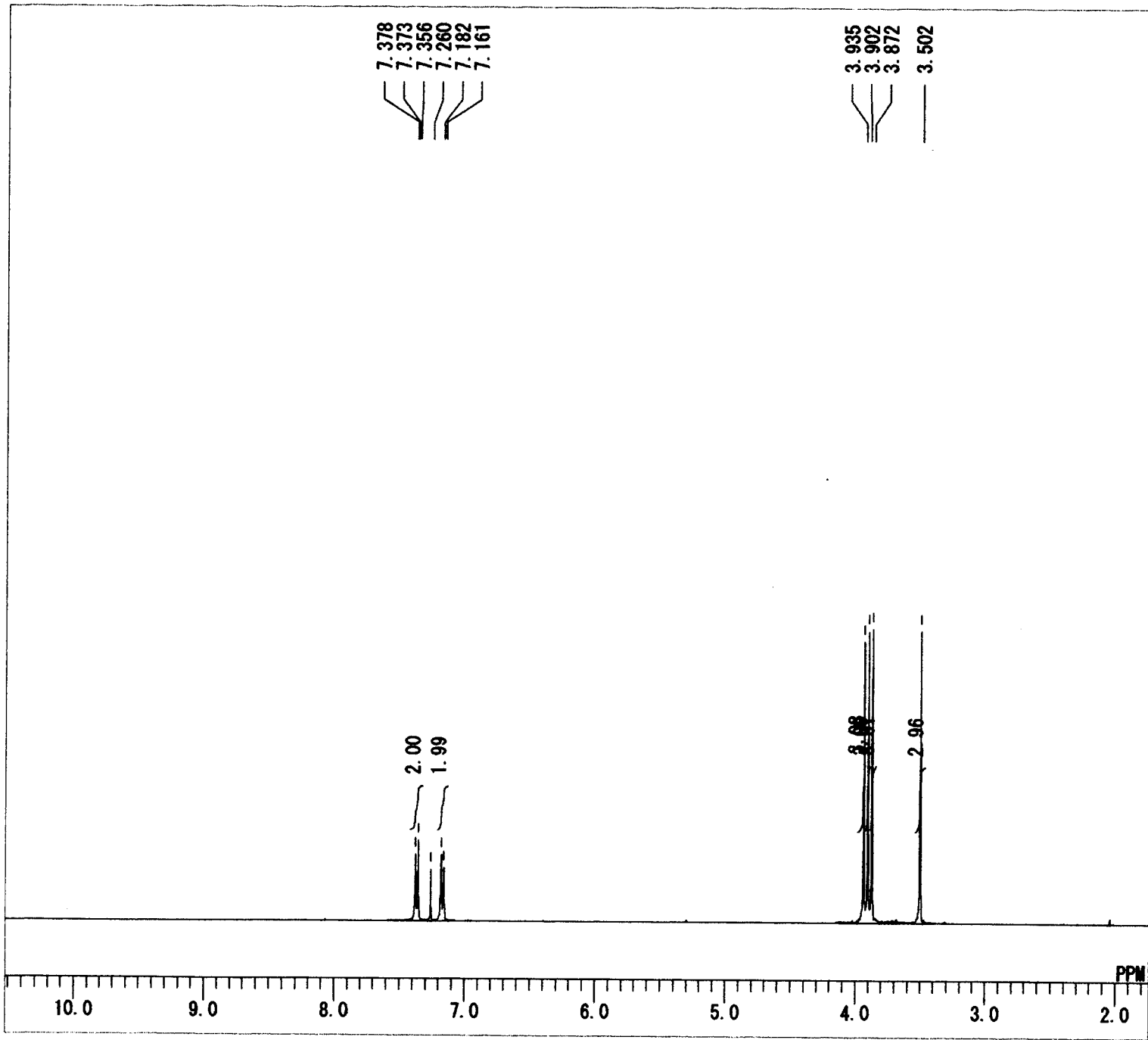
```

MENUF 31P
OBNUC 31P
OFR      161.70 MHz
OBSET    144.10 KHz
OBFIN    70.00 Hz
PW1      5.70 usec
DEADT    15.70 usec
PREDL    0.20000 msec
IWT      1.0000 msec
POINT    65536
SPO      65536
TIMES    32
DUMMY    1
FREQU    32258.06 Hz
FLT      16150 Hz
DELAY    12.40 usec
ACQTM    2.0316 sec
PD        1.7920 sec
ADBIT    16
RGAIN    25
BF        0.00 Hz
T1        0.00
T2        0.00
T3        90.00
T4       100.00
EXMOD    BCM
EXPCM    Bilevel. complete. decoupling:
IRNUC    1H
IFR      399.65 MHz
IRSET    124.00 KHz
IRFIN    10500.00 Hz
IRRPW    45 usec
IRATN    511
DFILE    CF3-P(0)(OEt)2+2diphenylacet;
SF        TH5ATFG2
LKSET    61.60 KHz
LKFIN    79.0 Hz
LKLEV    180
LGAIN    22
LKPHS    231
LKSIG    723
CSPED    12 Hz
FILDC
FILDF

```



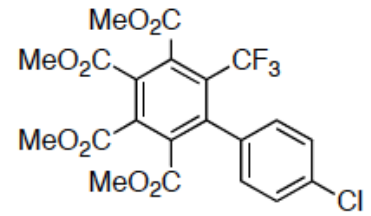
Si



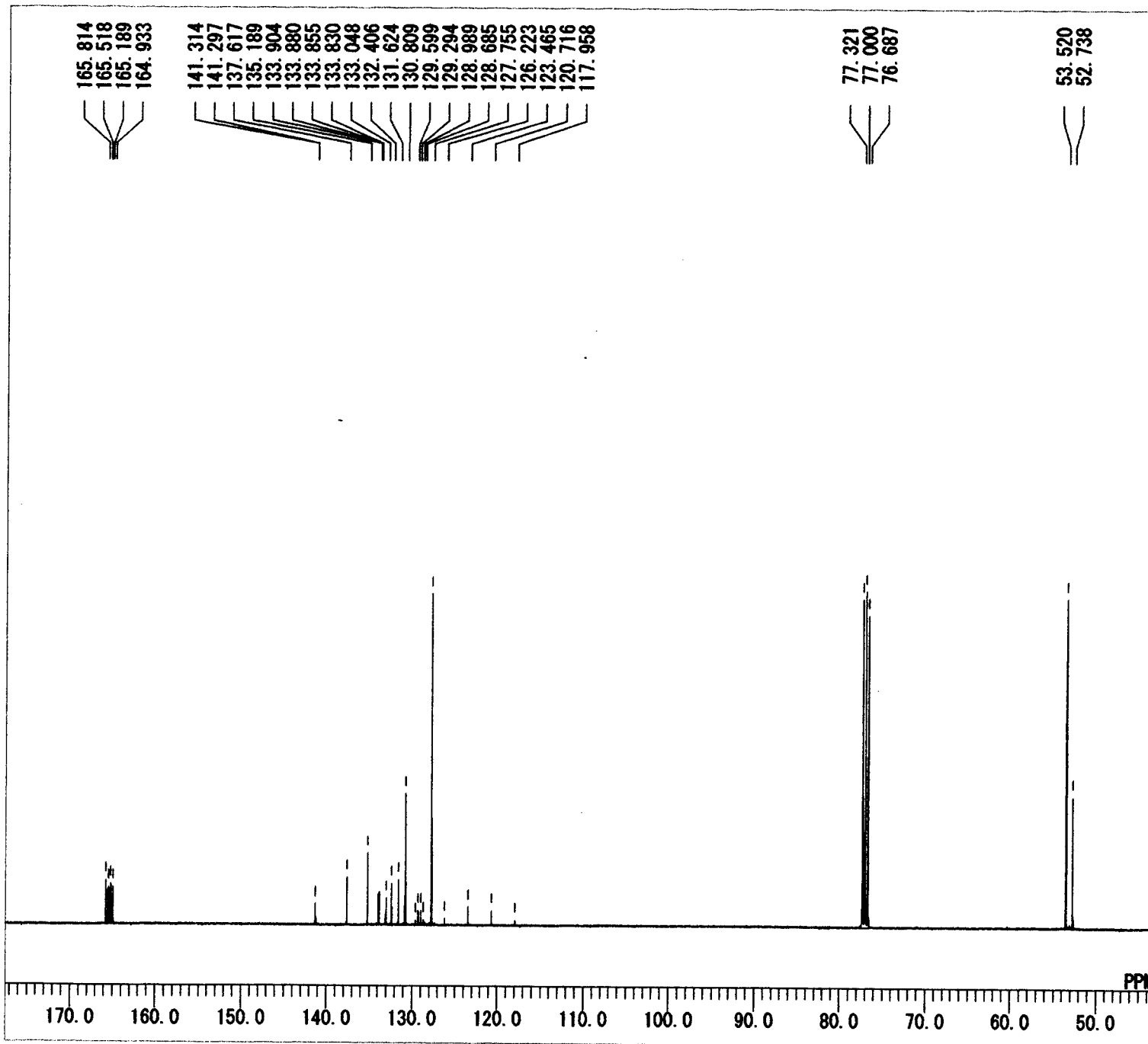
```

MENUF 1H
OBNUC 1H
OFR 399.65 MHz
OBSET 135.40 KHz
OBFIN 24.90 Hz
PW1 5.80 usec
DEADT 72.10 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQ 7992.01 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 2.0501 sec
PD 4.9500 sec
ADBIT 16
RGAIN 17
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM
IRNUC 1H
IFR 399.65 MHz
IRSET 136.90 KHz
IRFIN 97.50 Hz
IRRPW 45 usec
IRATN 511
DFILE CF3-PhCl+2diCO2Me-alkyne(1H).
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 25
LKPHS 231
LKSIG 1515
CSPED 10 Hz
FILDC
FILDF

```

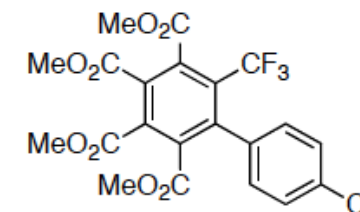


5j

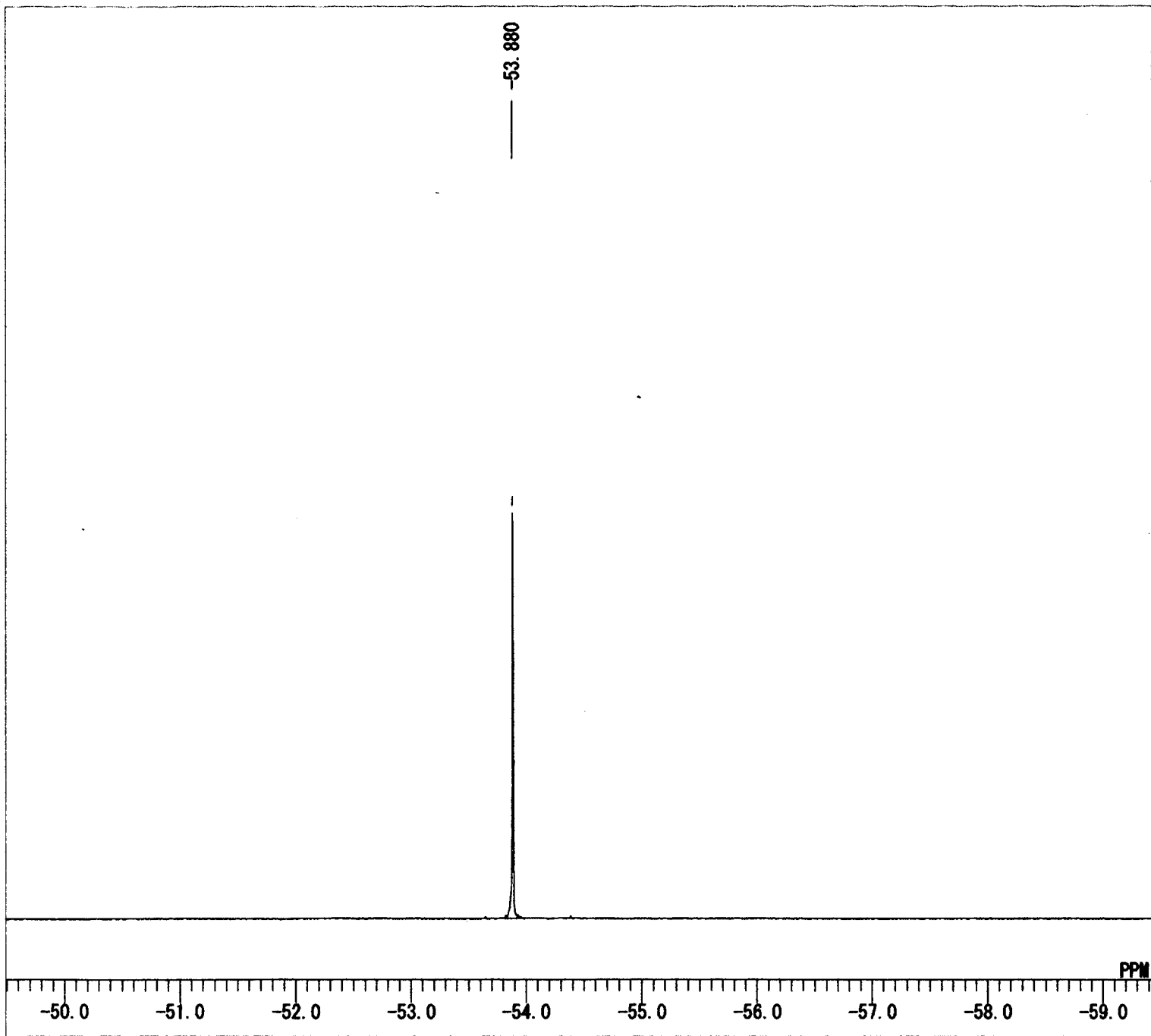


```

MENUF 13C
OBNUC 13C
OFR 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
PW1 6.00 usec
DEADT 19.10 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 32768
SPO 32768
TIMES 10000000
DUMMY 1
FREQ 27118.64 Hz
FLT 13550 Hz
DELAY 14.80 usec
ACQTM 1.2083 sec
PD 1.7920 sec
ADBIT 16
RGAIN 25
BF 0.60 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD BCM
EXPCM Bilevel. complete. decoupling:
IRNUC 1H
IFR 399.65 MHz
IRSET 124.00 KHz
IRFIN 10500.00 Hz
IRRPW 45 usec
IRATN 511
DFILE CF3-PhCl+2diCO2Me-alkyne (13C)
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGA1N 25
LKPHS 231
LKSIG 1768
CSPED 14 Hz
FILDC
FILDF
  
```

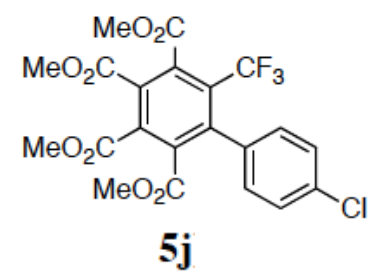


5j

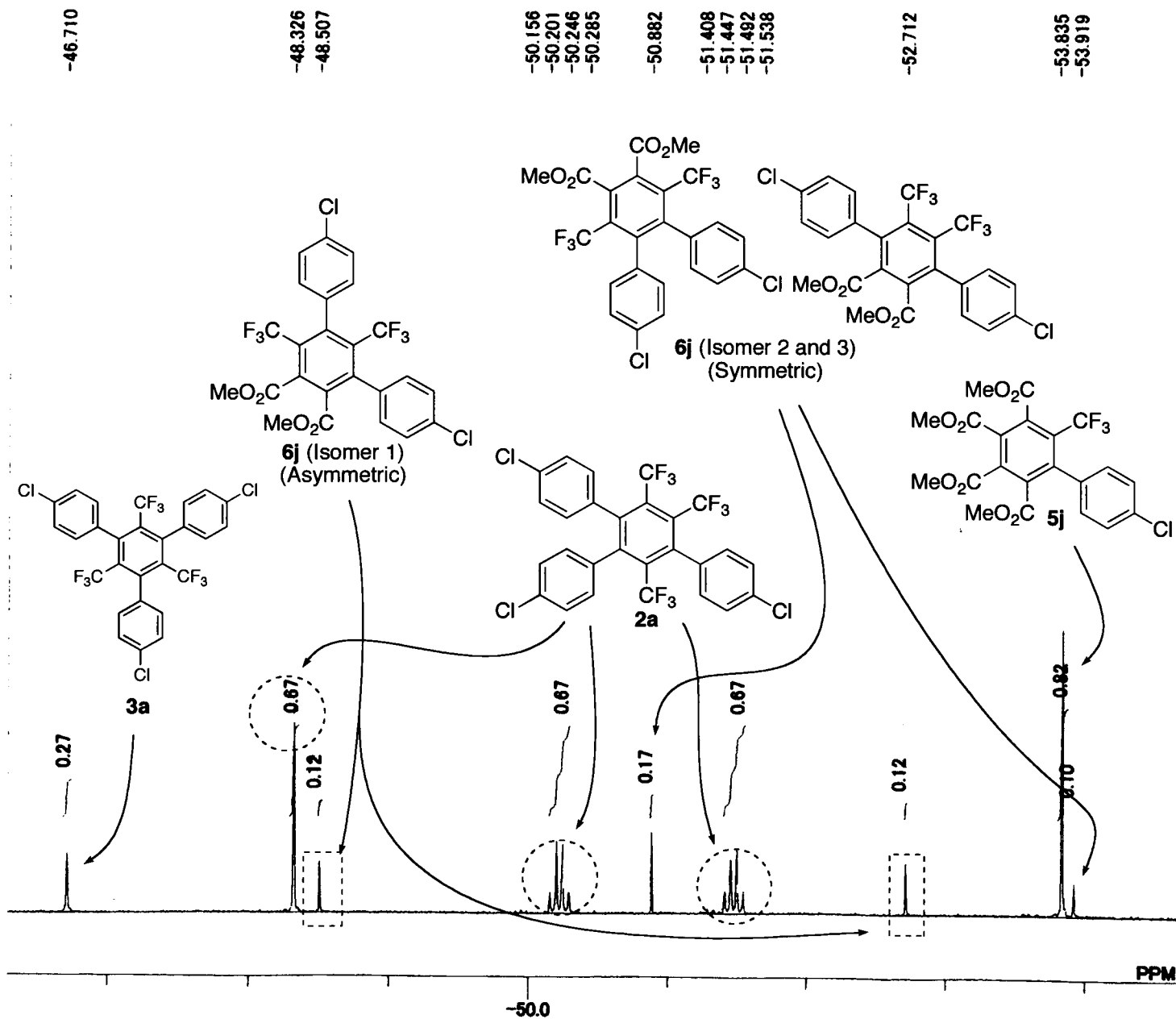


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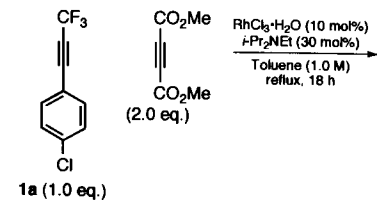
MENUF 19F
OBNUC 19F
OFR      376.05 MHz
OBSET    139.60 KHz
OBFIN    36.10 Hz
PW1      6.00 usec
DEADT    10.00 usec
PREDL    0.20000 msec
IWT      1.0000 msec
POINT    32768
SPO      32768
TIMES    16
DUMMY    1
FREQU    80000.00 Hz
FLT      40000 Hz
DELAY    5.00 usec
ACQTM    0.4096 sec
PD       4.9500 sec
ADBIT    16
RGAIN    14
BF        0.10 Hz
T1        0.00
T2        0.00
T3        90.00
T4       100.00
EXMOD    NON
EXPCM    NON:Single.coupled:PW1_ACQTM.
IRNUC    1H
IFR      399.65 MHz
IRSET    124.00 KHz
IRFIN    10500.00 Hz
IRRPW    45 usec
IRATN    511
DFILE    CF3-PhCl+2diC02Me-alkyne(19F)
SF       TH5ATFG2
LKSET    61.60 KHz
LKFIN    79.0 Hz
LKLEV    180
LGAIN    25
LKPHS    231
LKSIG    1506
CSPED    14 Hz
FILDC
FILDF
  
```

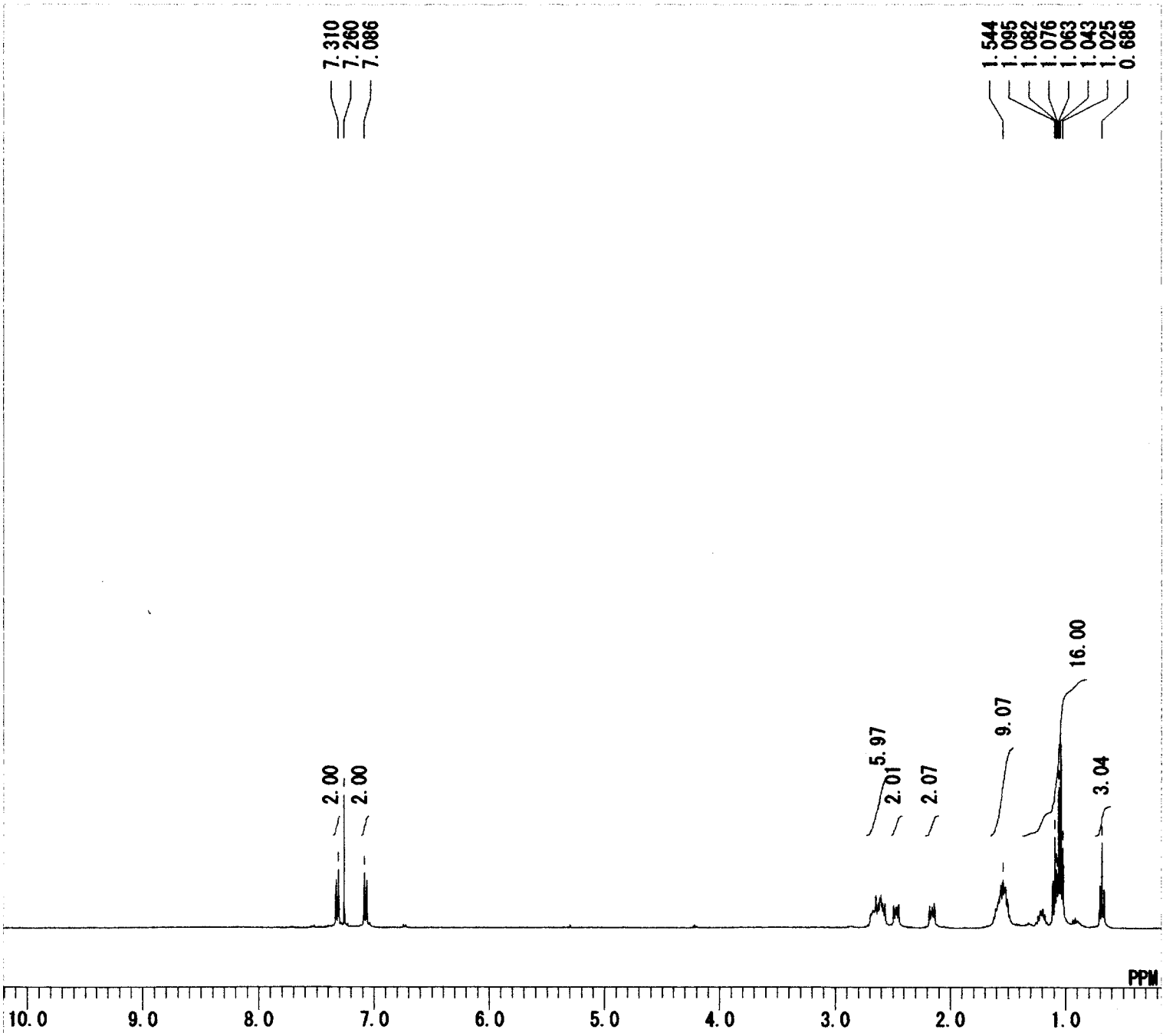


¹⁹F NMR Analysis of the reaction mixture (Total yield for all CF₃-containing compounds : 86%)



MENUF 19F
 OBNUC 19F
 OFR 376.05 MHz
 OBSET 139.60 KHz
 OBFIN 36.10 Hz
 PW1 6.00 usec
 DEADT 10.00 usec
 PREDL 0.2000 msec
 IWT 1.0000 msec
 POINT 32768
 SPO 32768
 TIMES 16
 DUMMY 1
 FREQU 80000.00 Hz
 FLT 40000 Hz
 DELAY 5.00 usec
 ACQTM 0.4096 sec
 PD 4.9500 sec
 ADBIT 16
 RGAIN 14
 BF 0.10 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD NON
 EXPCM NON:Single.coupled:PW1_ACQTM_PD:
 IRNUC 1H
 IFR 399.65 MHz
 IRSET 124.00 KHz
 IRFIN 10500.00 Hz
 IRRPW 45 usec
 IRATN 511
 DFILE DEFAULT.ALS
 SF TH5ATFG2
 LKSET 61.60 KHz
 LKFIN 79.0 Hz
 LKLEV 180
 LGAIN 25
 LKPHS 231
 LKSIG 1717
 CSPED 10 Hz
 FILDC
 FILDF

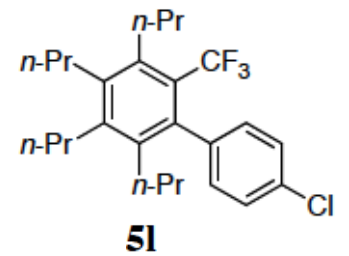


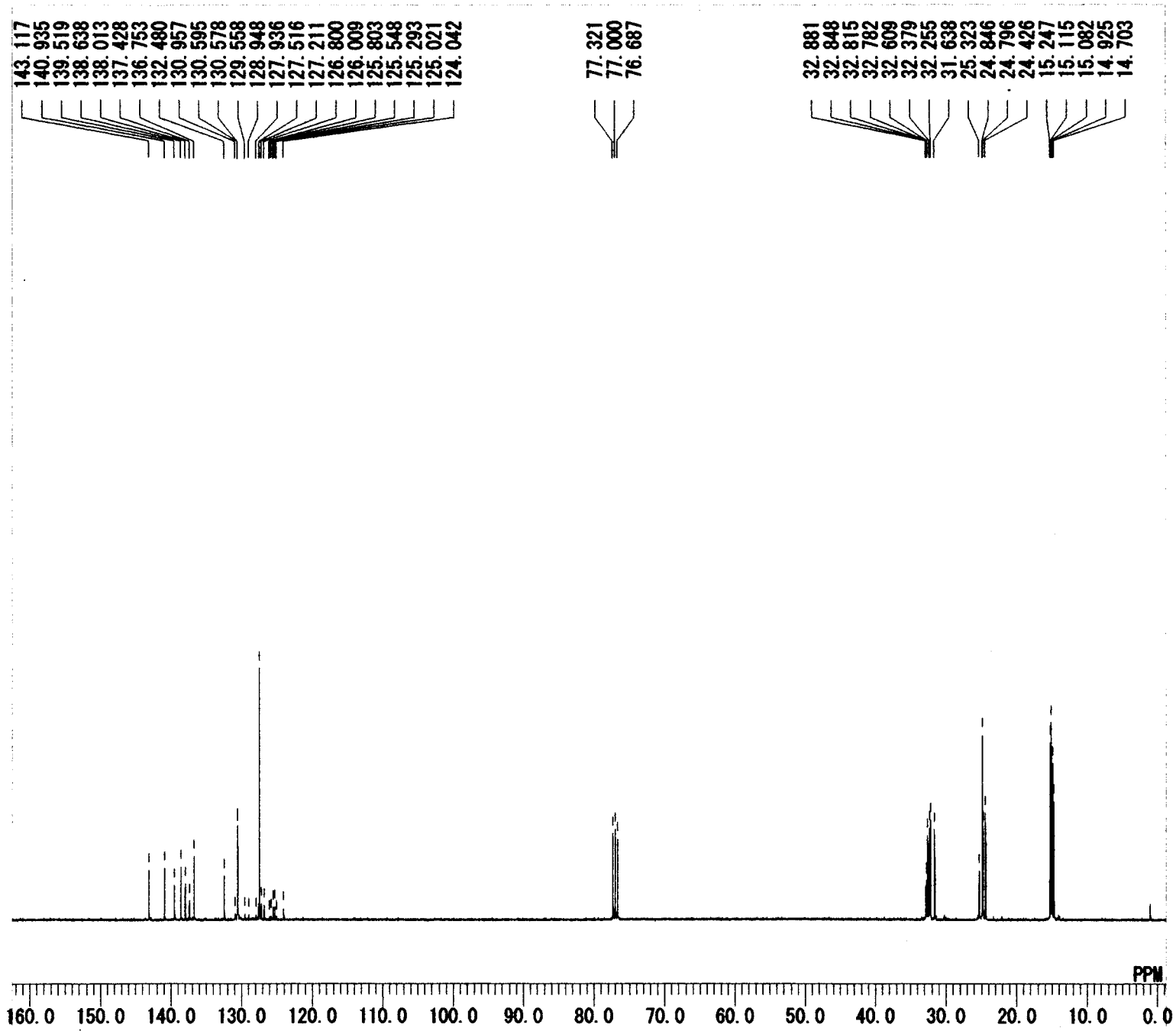


```

MENUF 1H
OBNUC 1H
OFR 399.65 MHz
OBSET 135.40 KHz
OBFIN 24.90 Hz
PW1 5.80 usec
DEADT 72.10 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7992.01 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 2.0501 sec
PD 4.9500 sec
ADBIT 16
RGAIN 17
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM
IRNUC 1H
IFR 399.65 MHz
IRSET 136.90 KHz
IRFIN 97.50 Hz
IRRPW 45 usec
IRATN 511
DFILE product-alkyne-p-Cl-di(n-Pr)
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 22
LKPHS 250
LKSIG 730
CSPED 11 Hz
FILDC
FILDF

```

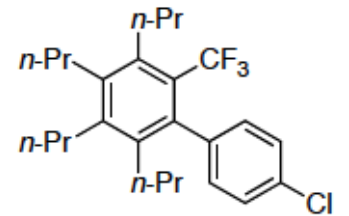




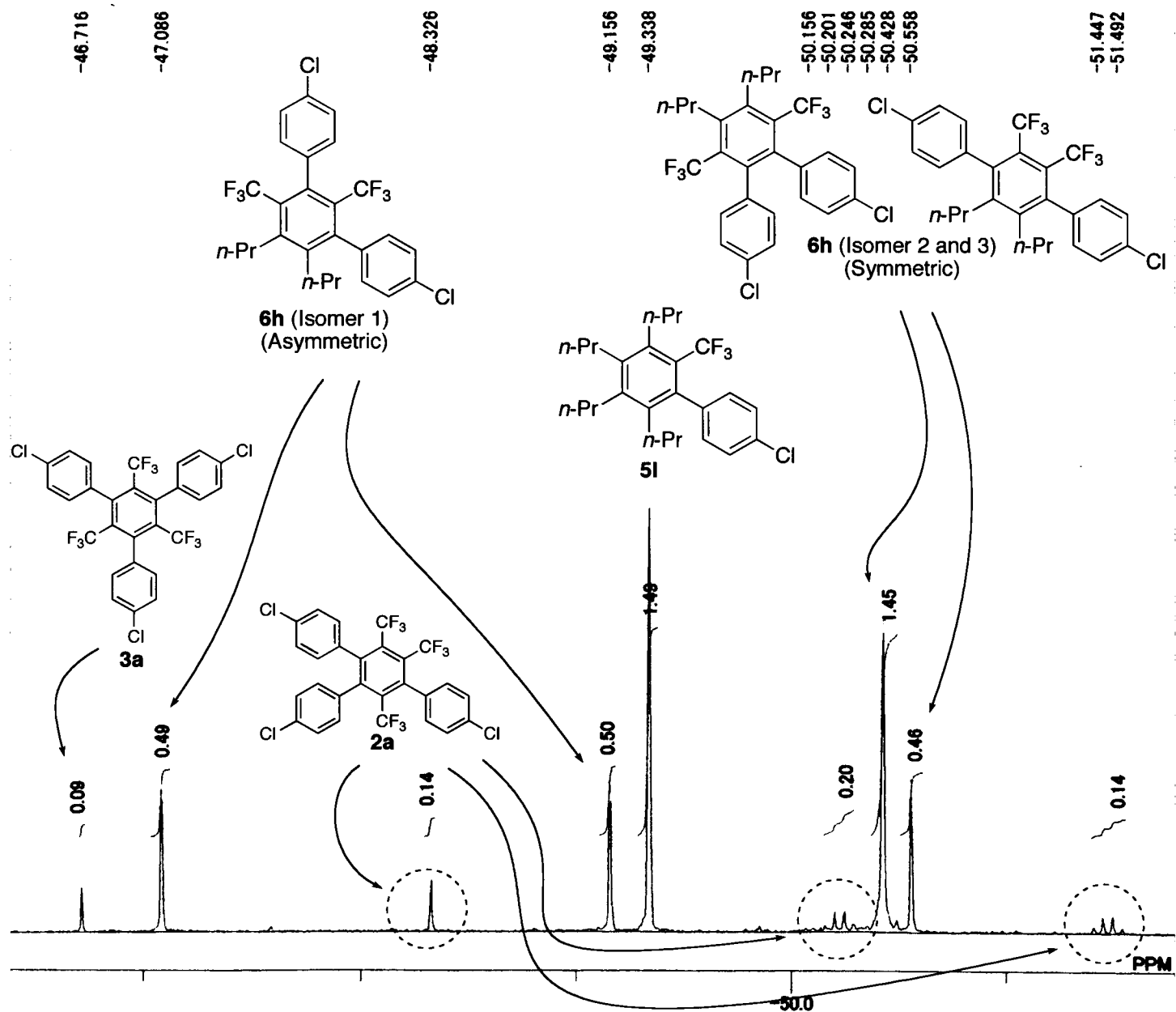
```

MENUF 13C
OBNUC 13C
OFR 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
PW1 6.00 usec
DEADT 19.10 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 32768
SPO 32768
TIMES 1024
DUMMY 1
FREQU 27118.64 Hz
FLT 13550 Hz
DELAY 14.80 usec
ACQTM 1.2083 sec
PD 1.7920 sec
ADBIT 16
RGAIN 24
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD BCM
EXPCM Bilevel. complete. decoupling:
IRNUC 1H
IFR 399.65 MHz
IRSET 124.00 KHz
IRFIN 10500.00 Hz
IRRPW 45 usec
IRATN 511
DFILE product-alkyne-p-Cl-di(n-Pr)
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 25
LKPHS 250
LKSIG 1111
CSPED 13 Hz
FILDC
FILDF

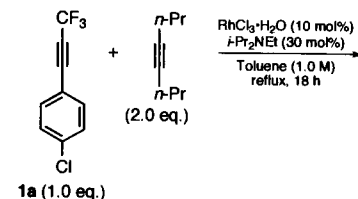
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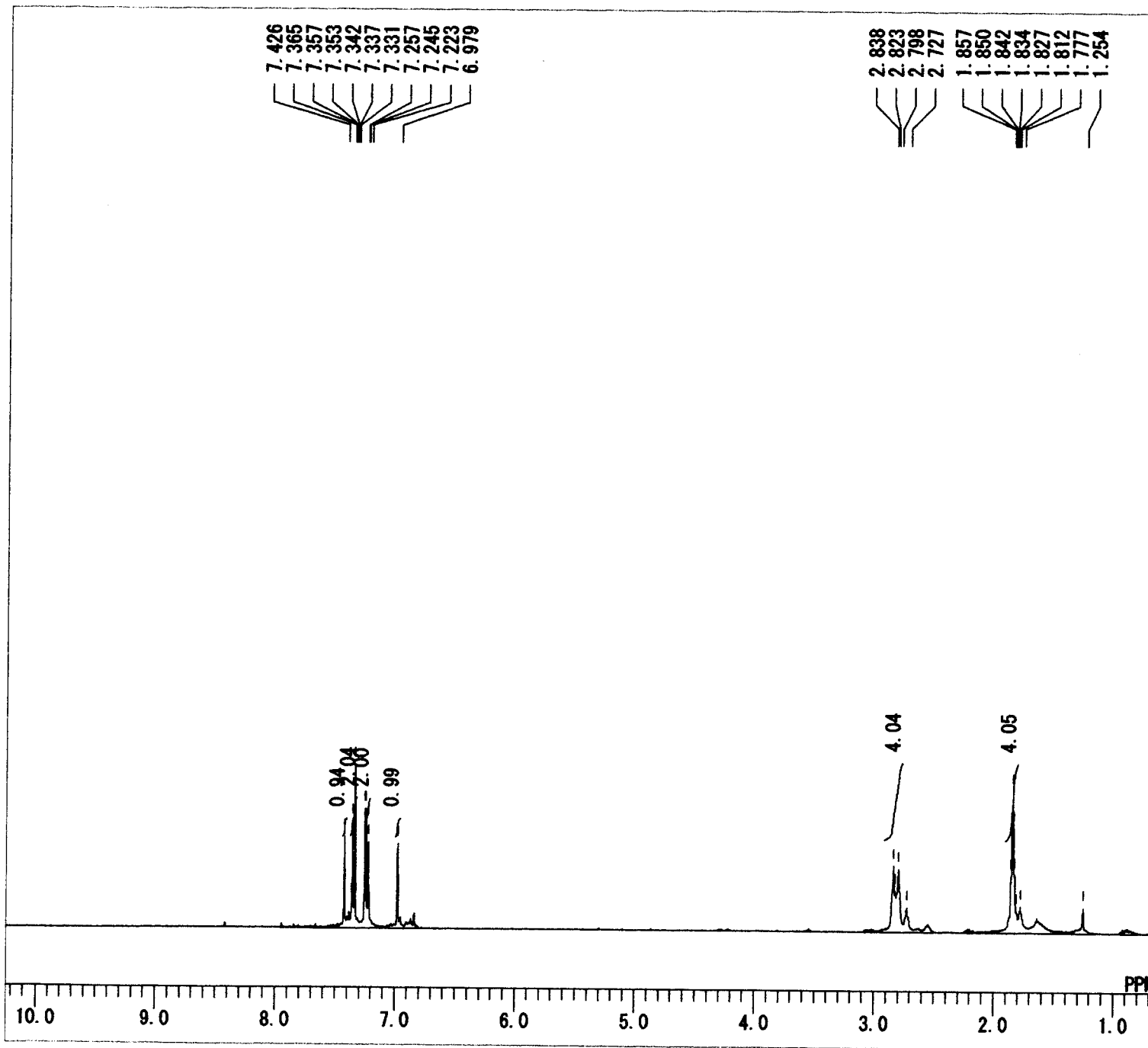


¹⁹F NMR Analysis of the reaction mixture (Total yield for all CF₃-containing compounds : 100%)



MENUF	19F
OBNUC	19F
OFR	376.05 MHz
OBSET	139.60 KHz
OBFIN	36.10 Hz
PW1	6.00 usec
DEADT	10.00 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	16
DUMMY	1
FREQU	80000.00 Hz
FLT	40000 Hz
DELAY	5.00 usec
ACQTM	0.4096 sec
PD	4.9500 sec
ADBIT	16
RGAIN	14
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM_PD:
IRNUC	1H
IFR	399.65 MHz
IRSET	124.00 KHz
IRFIN	10500.00 Hz
IRRPW	45 usec
IRATN	511
DFILE	_DEFAULT.ALS
SF	TH5ATFG2
LKSET	81.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	21
LKPHS	231
LKSIG	570
CSPED	12 Hz
FILDC	
FILDF	

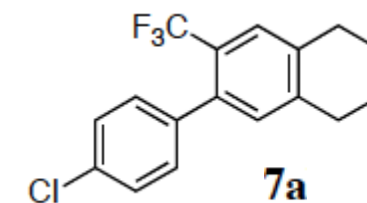


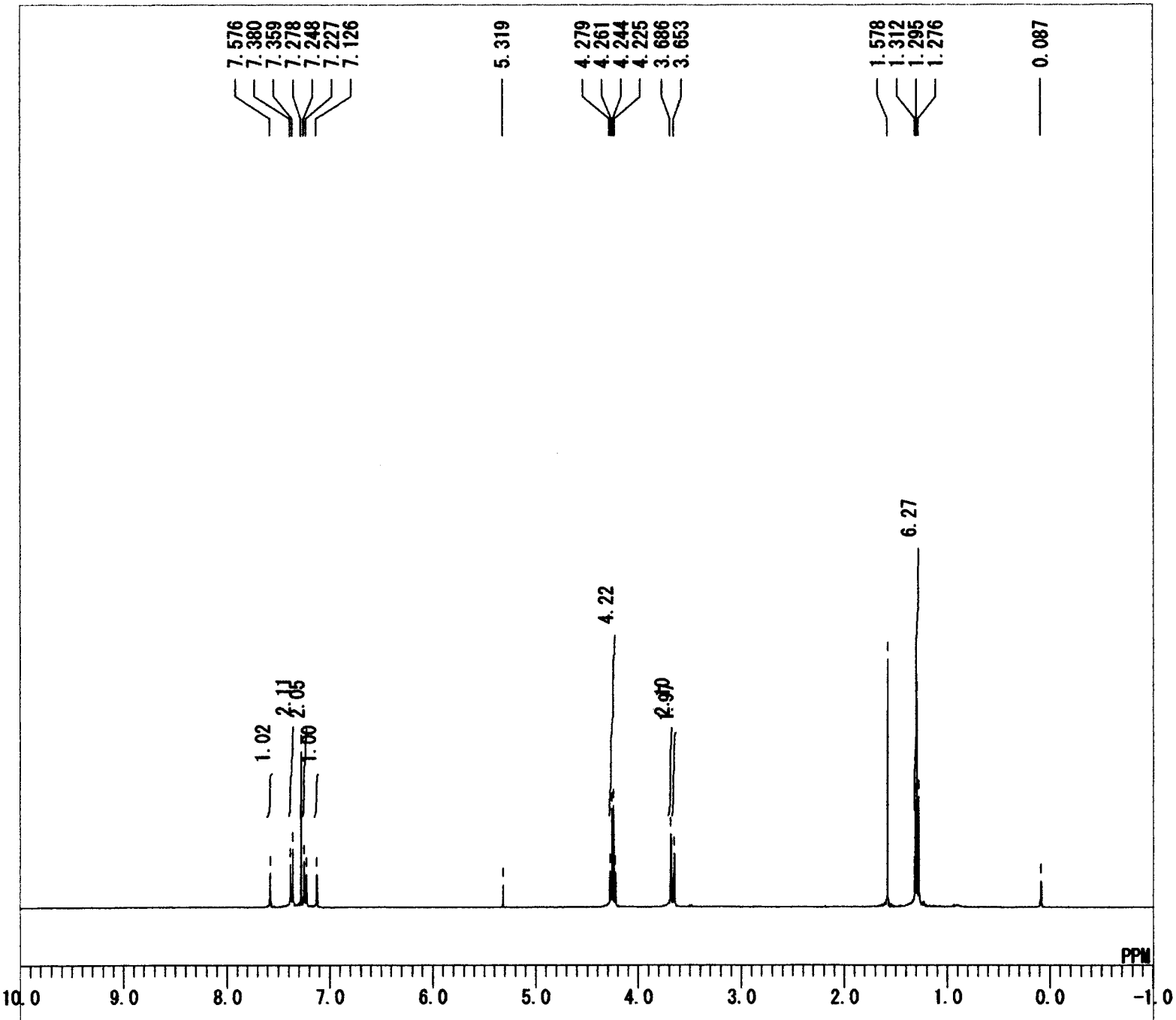


```

MENUF 1H
OBNUC 1H
OFR 399.65 MHz
OBSET 135.40 KHz
OBFIN 24.90 Hz
PW1 5.80 usec
DEADT 72.10 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7992.01 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 2.0501 sec
PD 4.9500 sec
ADBIT 16
RGAIN 17
BF 0.00 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM
IRNUC 1H
IFR 399.65 MHz
IRSET 136.90 KHz
IRFIN 97.50 Hz
IRRPW 45 usec
IRATN 511
DFILE CF3-PhCl+1,7-octadiyne (1H).a
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 24
LKPHS 250
LKSIG 1153
CSPED 11 Hz
FILDC
FILDF

```

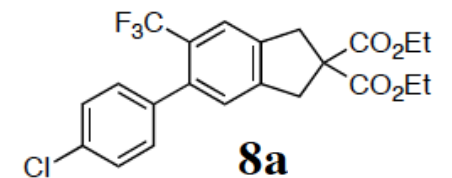


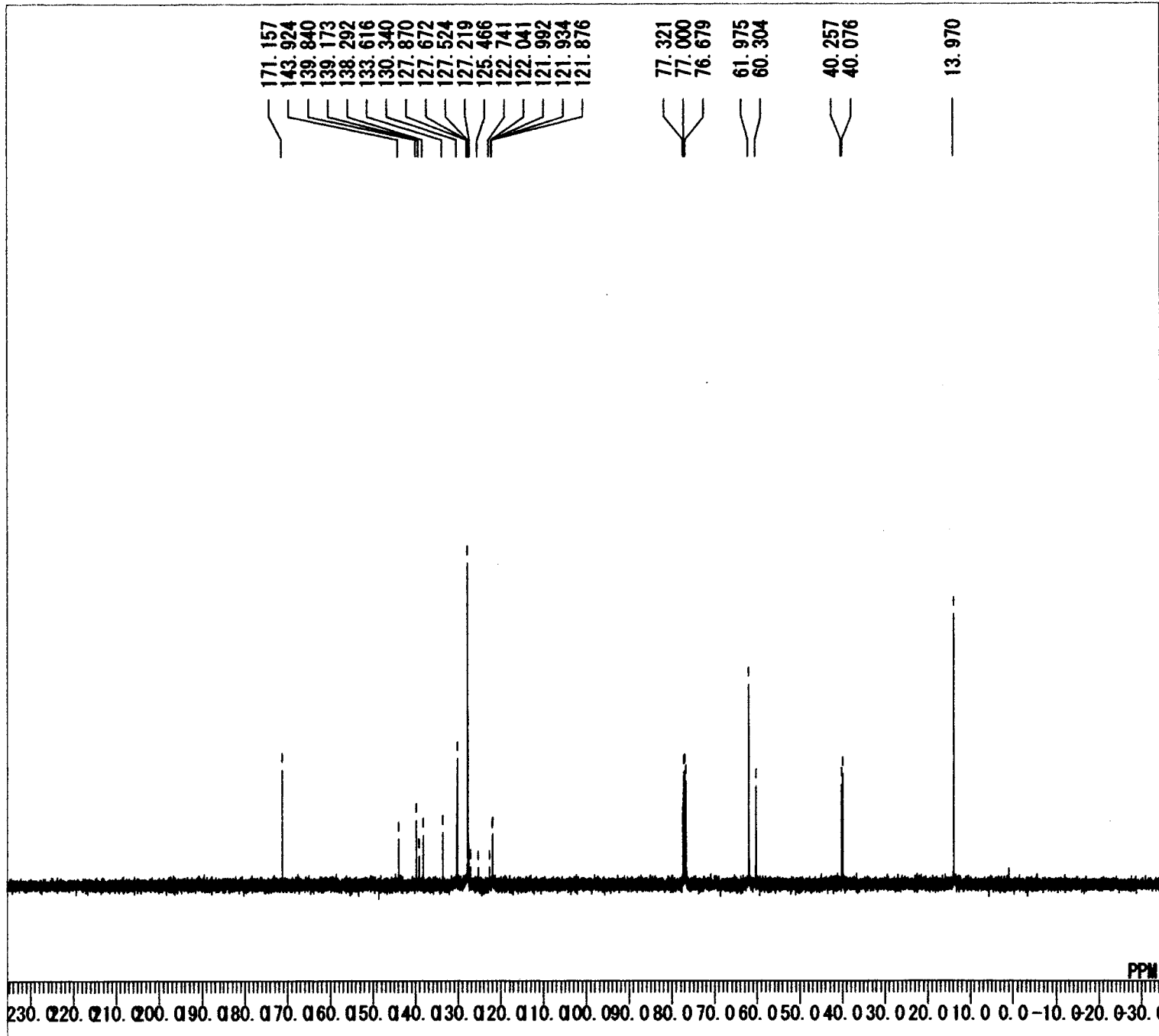


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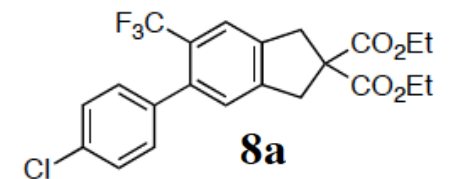
MENUF 1H
OBNUC 1H
OFR 399.65 MHz
OBSET 135.40 KHz
OBFIN 24.90 Hz
PW1 5.20 usec
DEADT 72.40 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7992.01 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 2.0501 sec
PD 4.9500 sec
ADBIT 16
RGAIN 20
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON: Single. coupled: PW1_ACQTM
IRNUC 1H
IFR 399.65 MHz
IRSET 136.90 KHz
IRFIN 97.50 Hz
IRRPW 45 usec
IRATN 511
DFILE product-p-Cl(1H).als
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 26
LKPHS 219
LKSIG 2104
CSPED 11 Hz
FILDC
FILDF

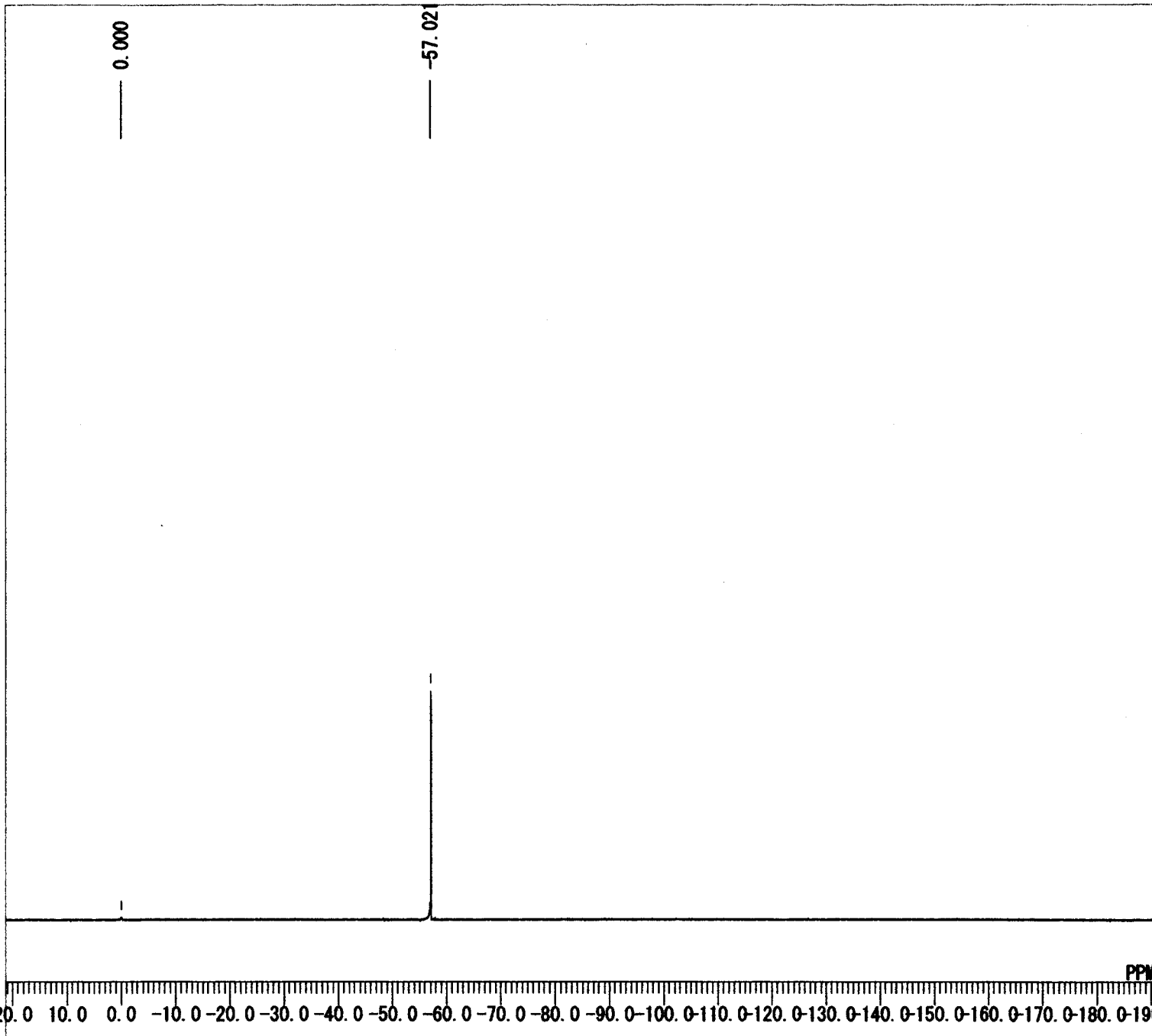
```





MENUF 13C
 OBNUC 13C
 OFR 100.40 MHz
 OBSSET 125.00 KHz
 OBF IN 10500.00 Hz
 PW1 6.00 usec
 DEADT 19.10 usec
 PREDL 0.20000 msec
 IWT 1.0000 msec
 POINT 32768
 SPO 32768
 TIMES 256
 DUMMY 1
 FREQU 27118.64 Hz
 FLT 13550 Hz
 DELAY 14.80 usec
 ACQTM 1.2083 sec
 PD 1.7920 sec
 ADBIT 16
 RGA IN 25
 BF 0.10 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD BCM
 EXPCM Bilevel. complete. decoupling:
 IRNUC 1H
 IFR 399.65 MHz
 IRSET 124.00 KHz
 IRF IN 10500.00 Hz
 IRRPW 45 usec
 IRATN 511
 DFILE product-p-CI (13C).als
 SF TH5ATFG2
 LKSET 61.60 KHz
 LKF IN 79.0 Hz
 LKLEV 180
 LGAIN 25
 LKPHS 219
 LKSIG 291
 CSPED 11 Hz
 FILDC
 FILDf

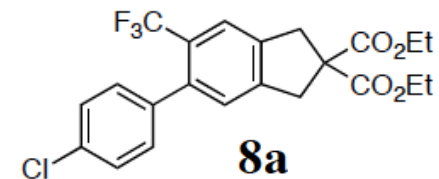


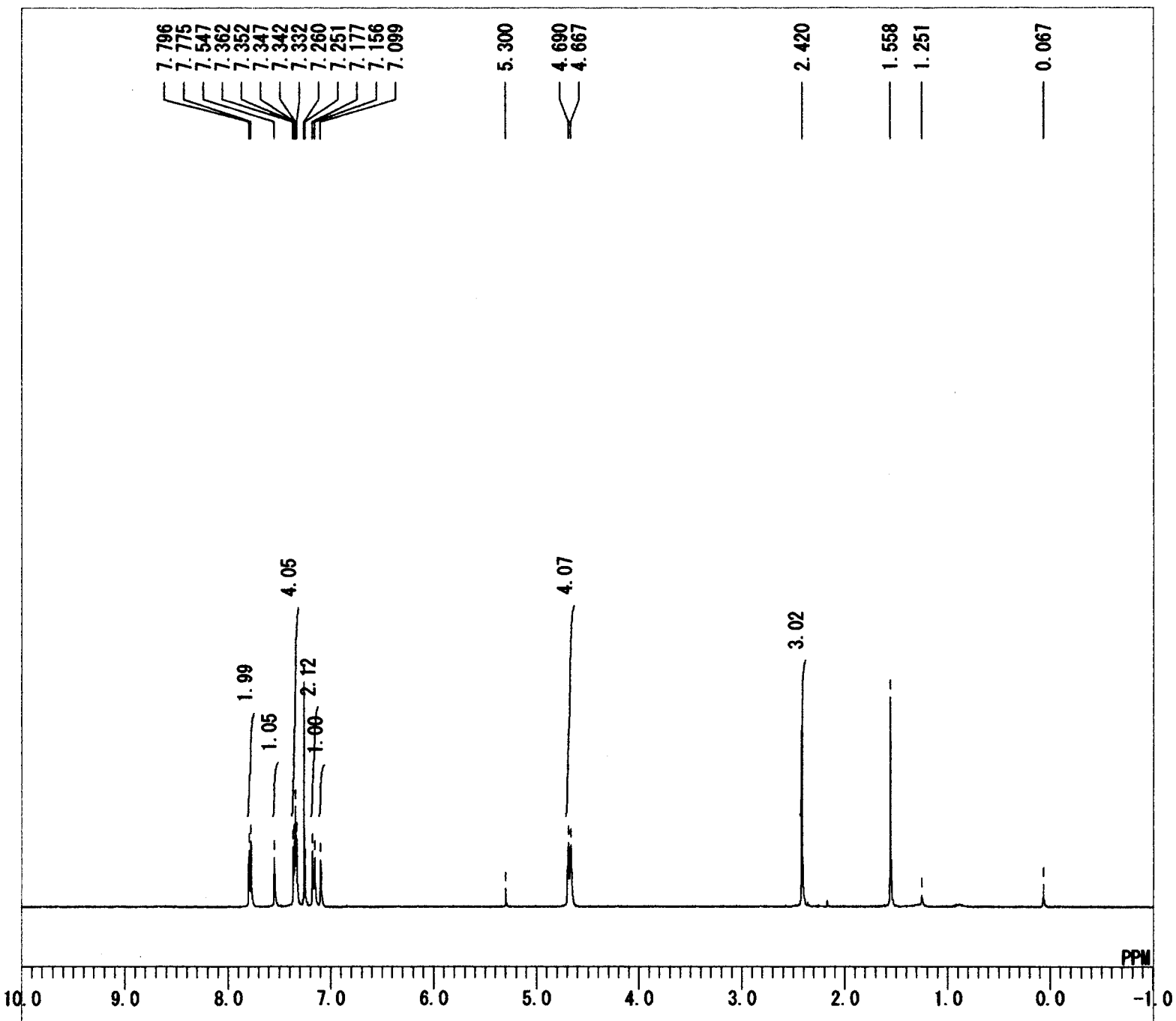


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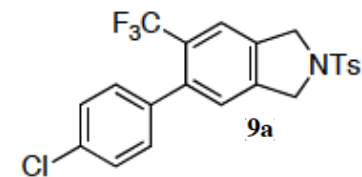
MENUF 19F
OBNUC 19F
OFR 376.05 MHz
OBSET 139.60 KHz
OBFIN 36.10 Hz
PW1 6.00 usec
DEADT 10.00 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 32768
SPO 32768
TIMES 16
DUMMY 1
FREQU 80000.00 Hz
FLT 40000 Hz
DELAY 5.00 usec
ACQTM 0.4096 sec
PD 4.9500 sec
ADBIT 16
RGAIN 14
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM
IRNUC 1H
IFR 399.65 MHz
IRSET 124.00 KHz
IRFIN 10500.00 Hz
IRRPW 45 usec
IRATN 511
DFILE product-p-Cl(19F).als
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 27
LKPHS 219
LKSIG 396
CSPED 11 Hz
FILDC
FILDF

```



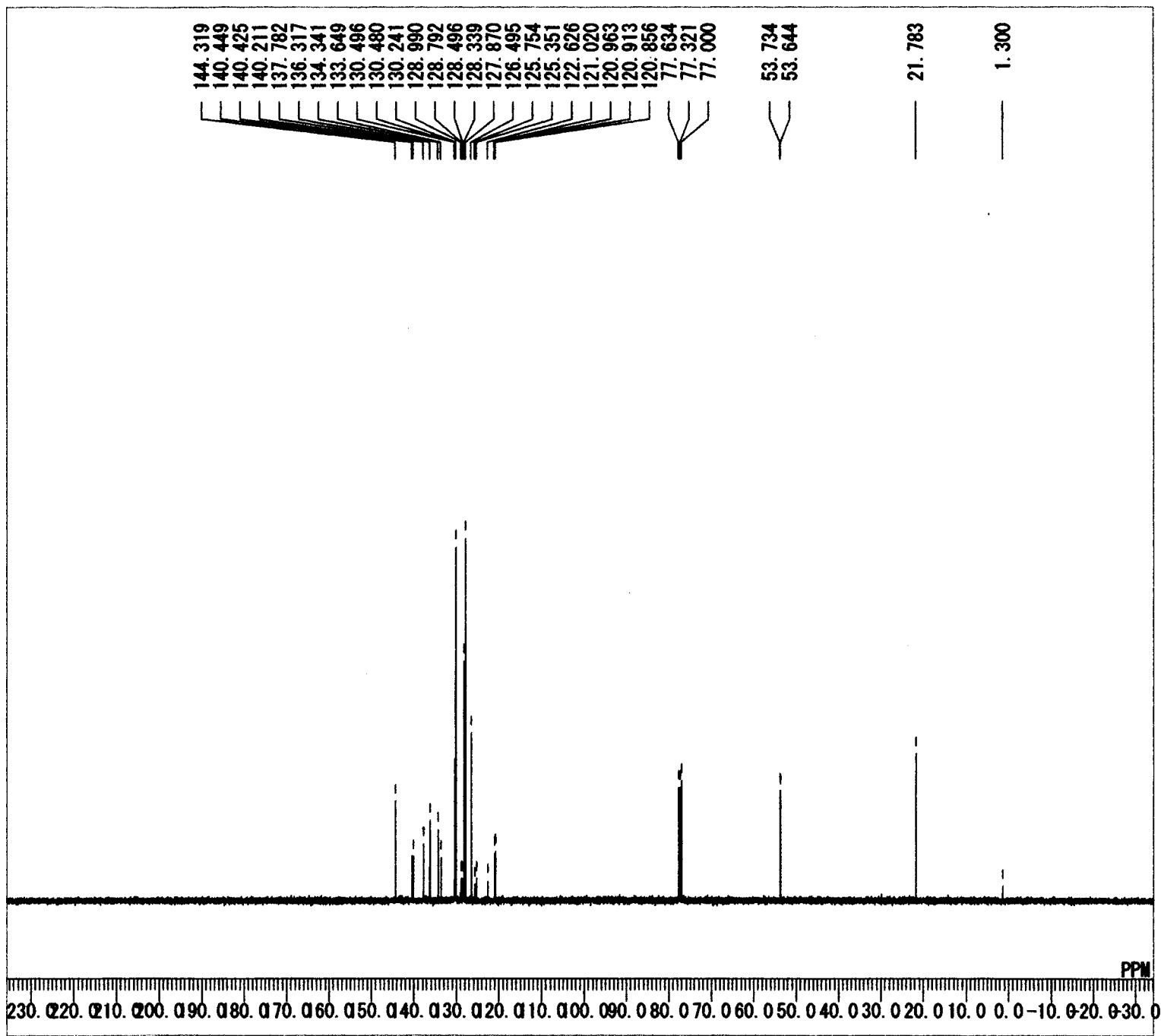


MENUF 1H
 OBNUC 1H
 OFR 399.65 MHz
 OBSSET 135.40 KHz
 OBFIN 24.90 Hz
 PW1 5.20 usec
 DEADT 72.40 usec
 PREDL 0.20000 msec
 IWT 1.0000 msec
 POINT 16384
 SPO 16384
 TIMES 8
 DUMMY 1
 FREQU 7992.01 Hz
 FLT 4000 Hz
 DELAY 50.00 usec
 ACQTM 2.0501 sec
 PD 4.9500 sec
 ADBIT 16
 RGAIN 22
 BF 0.10 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD NON
 EXPCM NON:Single.coupled:PW1_ACQTM
 IRNUC 1H
 IFR 399.65 MHz
 IRSET 136.90 KHz
 IRFIN 97.50 Hz
 IRRPW 45 usec
 IRATN 511
 DF FILE product-p-cl-N(1H).als
 SF TH5ATFG2
 LKSET 61.60 KHz
 LKFIN 79.0 Hz
 LKLEV 180
 LGAIN 22
 LKPHS 219
 LKSIG 600
 CSPED 12 Hz
 FILDC
 FILDF

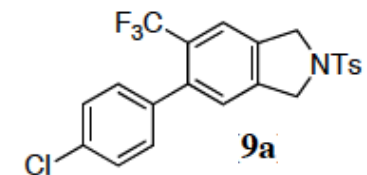


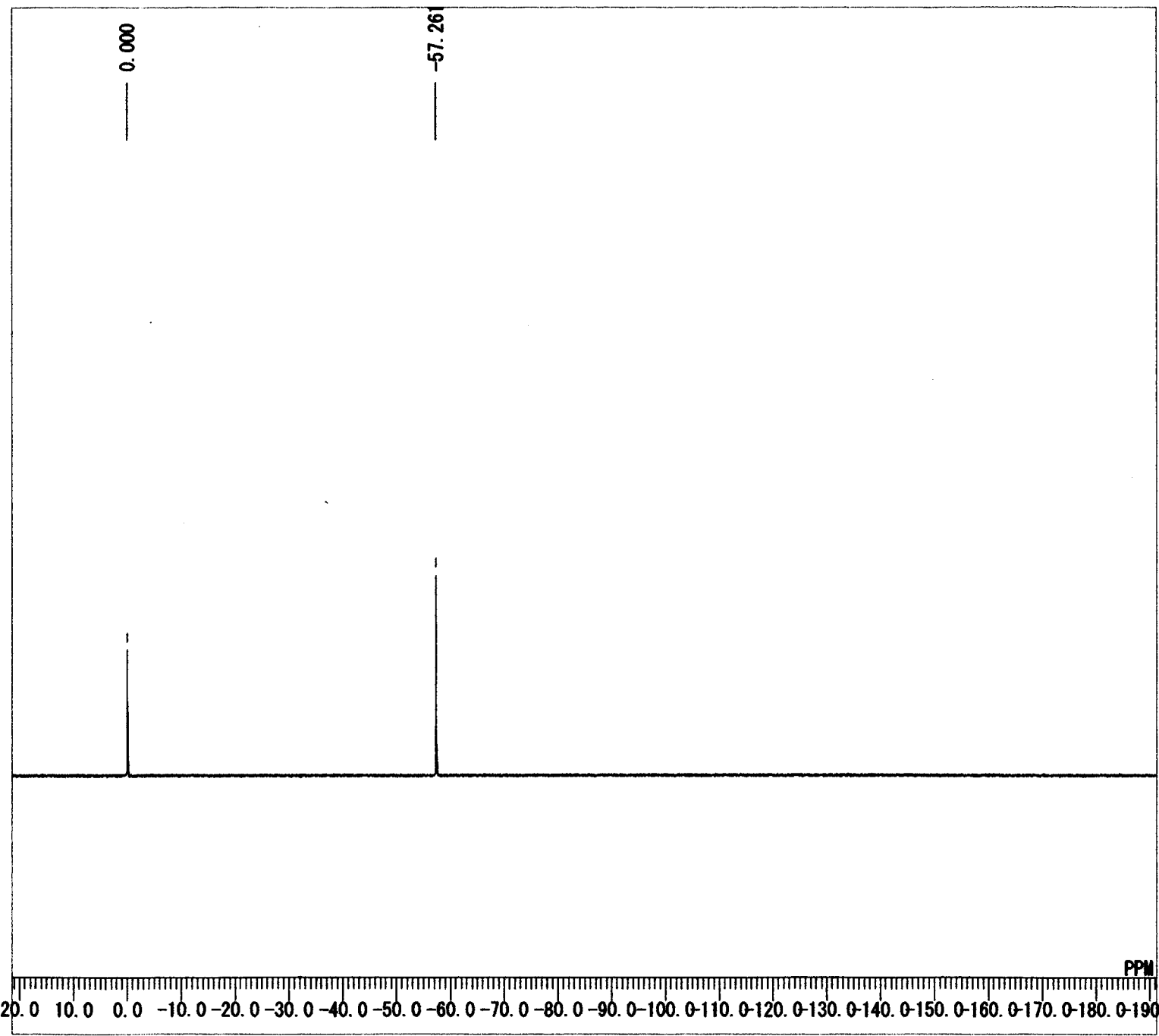
144.319
140.449
140.425
140.211
137.782
136.317
134.341
133.649
130.496
130.480
130.241
128.990
128.792
128.496
128.339
127.870
126.495
125.754
125.351
122.626
121.020
120.963
120.913
120.856
77.634
77.321
77.000

53.734
53.644
21.783
1.300



MENUF 13C
OBNUC 13C
OFR 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
PW1 6.00 usec
DEADT 19.10 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 32768
SPO 32768
TIMES 256
DUMMY 1
FREQU 27118.64 Hz
FLT 13550 Hz
DELAY 14.80 usec
ACQTM 1.2083 sec
PD 1.7920 sec
ADBIT 16
RGAIN 25
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD BCM
EXPCM Bi level. complete. decoupling:
IRNUC 1H
IFR 399.65 MHz
IRSET 124.00 KHz
IRFIN 10500.00 Hz
IRRPW 45 usec
IRATN 511
DFILE product-p-Cl-N(13C).als
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 24
LKPHS 221
LKSIG 960
CSPED 14 Hz
FILDC
FILDF

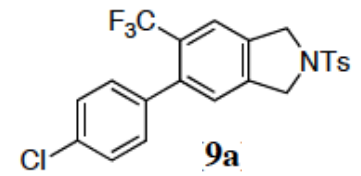




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MENUF 19F
OBNUC 19F
OFR 376.05 MHz
OBSET 139.60 KHz
OBFIN 36.10 Hz
PW1 6.00 usec
DEADT 10.00 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 32768
SPO 32768
TIMES 16
DUMMY 1
FREQU 80000.00 Hz
FLT 40000 Hz
DELAY 5.00 usec
ACQTM 0.4096 sec
PD 4.9500 sec
ADBIT 16
RGAIN 13
BF 5.00 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM
IRNUC 1H
IFR 399.65 MHz
IRSET 124.00 KHz
IRFIN 10500.00 Hz
IRRPW 45 usec
IRATN 511
DFILE product-p-Cl-N(19F).als
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 24
LKPHS 219
LKSIG 658
CSPED 11 Hz
FILDC
FILDF

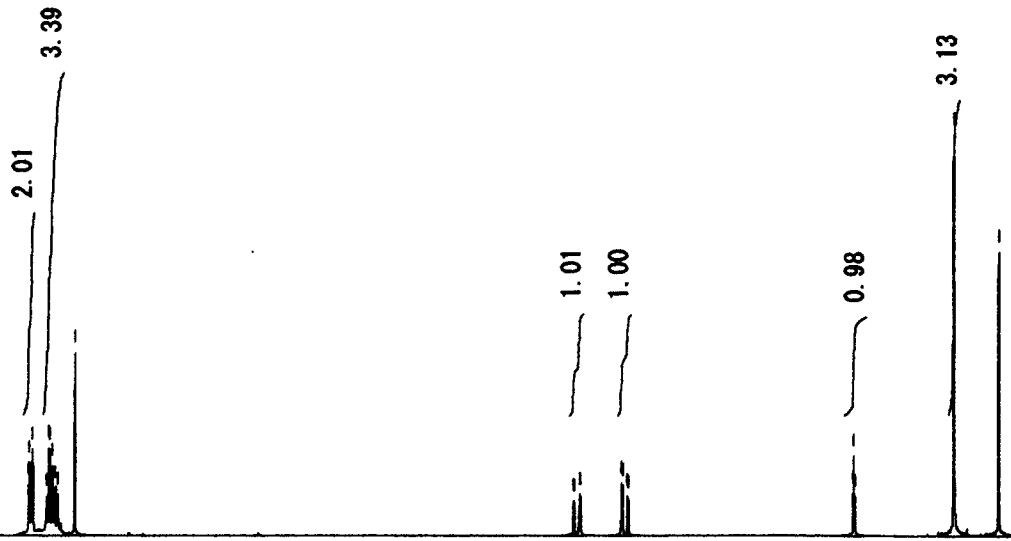
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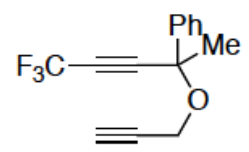
7.546
7.542
7.528
7.524
7.436
7.420
7.417
7.414
7.399
7.383
7.380
7.366
7.260

4.180
4.174
4.143
4.138
3.885
3.879
3.848
3.842

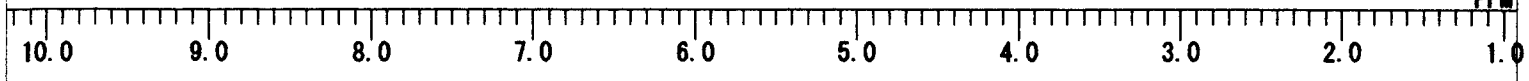
2.455
2.449
2.443
1.832
1.556

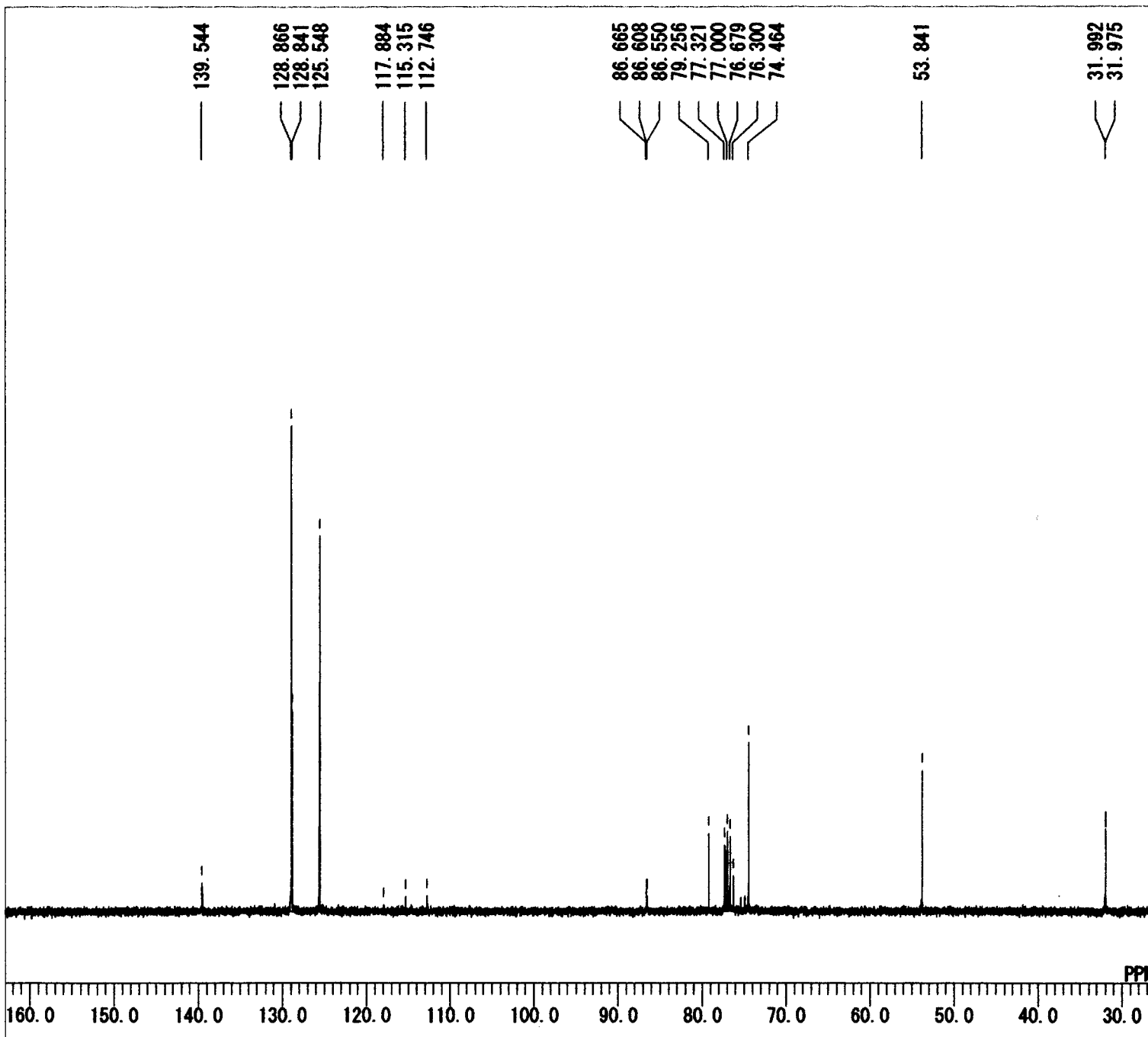


MENUF 1H
OBNUC 1H
OFR 399.65 MHz
OBSET 135.40 KHz
OBF IN 24.90 Hz
PW1 5.80 usec
DEADT 72.10 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7992.01 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 2.0501 sec
PD 4.9500 sec
ADBIT 16
RGAIN 19
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM
IRNUC 1H
IFR 399.65 MHz
IRSET 136.90 KHz
IRFIN 97.50 Hz
IRRPW 45 usec
IRATN 511
DFILE CF3-Diyne(Ph, Me)-H(1H).als
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 24
LKPHS 249
LKSIG 1241
CSPED 12 Hz
FILDC
FILDF



10A

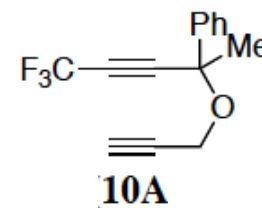


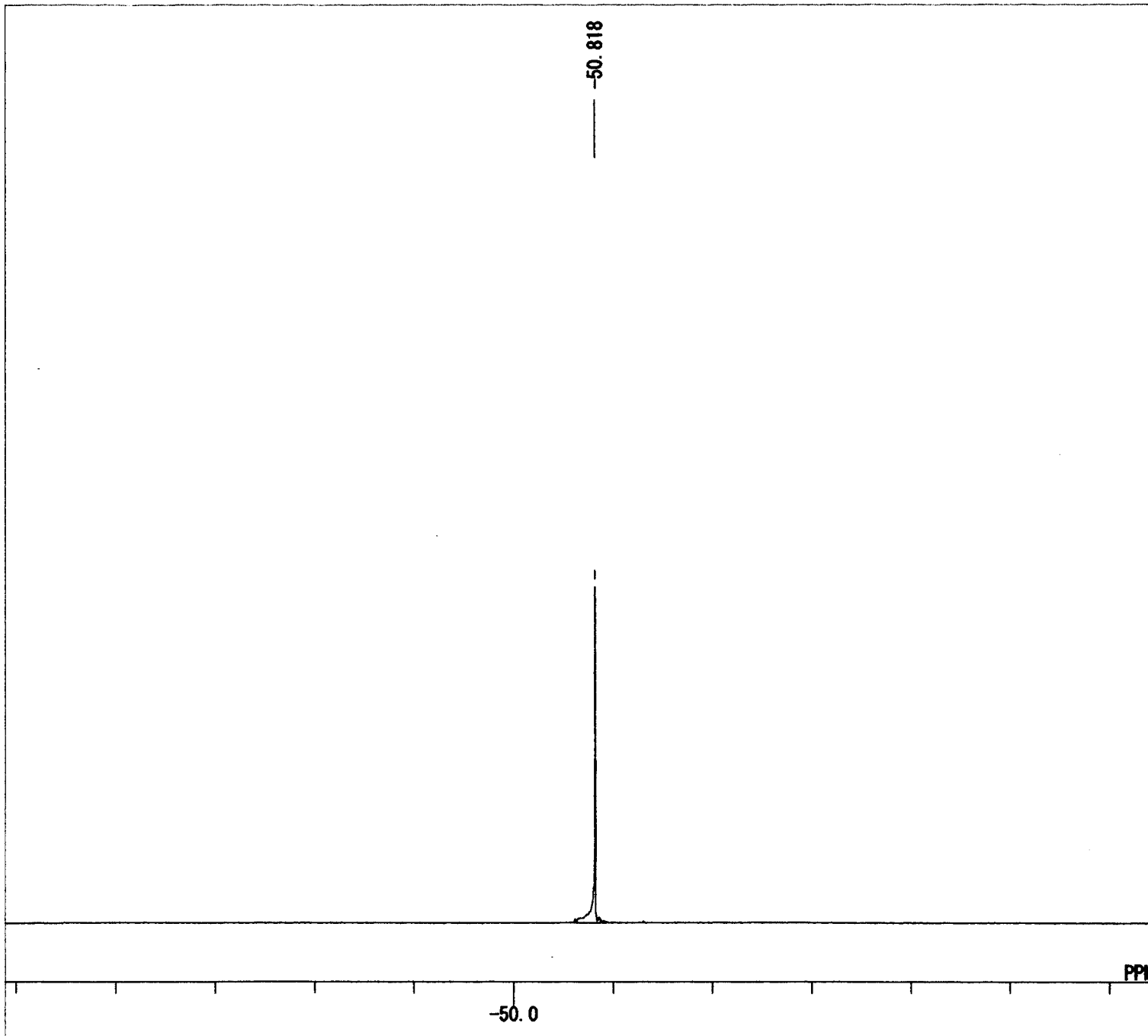


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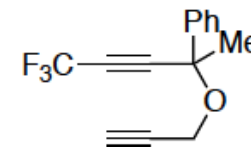
MENUF 13C
OBNUC 13C
OFR 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
PW1 6.00 usec
DEADT 19.10 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 32768
SPO 32768
TIMES 256
DUMMY 1
FREGU 27118.64 Hz
FLT 13550 Hz
DELAY 14.80 usec
ACQTM 1.2083 sec
PD 1.7920 sec
ADBIT 16
RGAIN 25
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD BCM
EXPCM Bi level. complete. decoupling:
IRNUC 1H
IFR 399.65 MHz
IRSET 124.00 KHz
IRFIN 10500.00 Hz
IRRPW 45 usec
IRATN 511
DFILE CF3-Diyne (Ph, Me)-H(13C). als
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 24
LKPHS 249
LKSIG 710
CSPED 10 Hz
FILDC
FILDF

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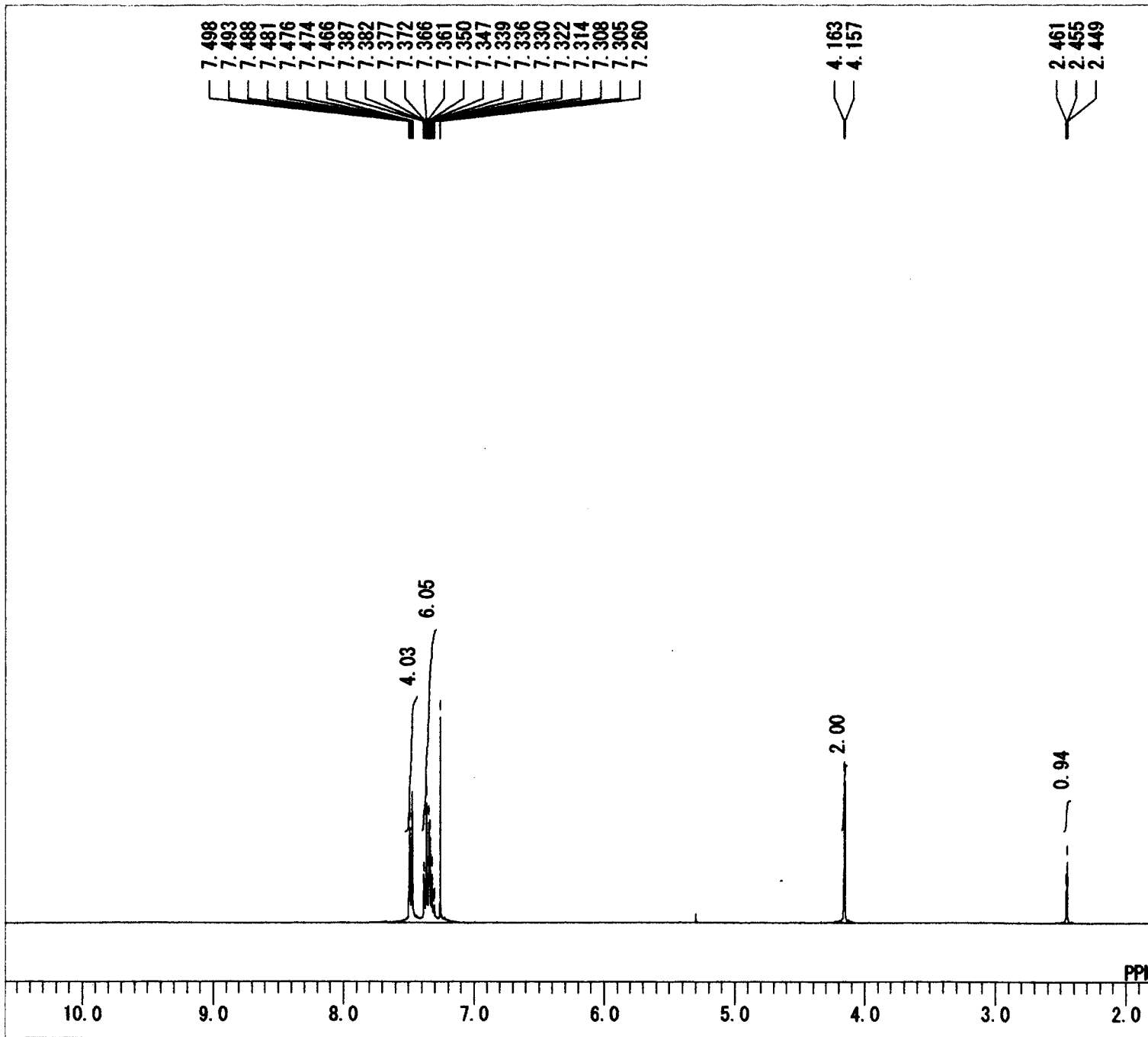




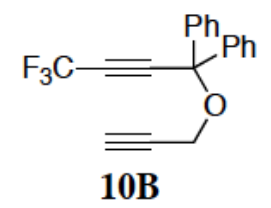
MENUF	19F
OBNUC	19F
OFR	376.05 MHz
OBSET	139.60 KHz
OBFIN	36.10 Hz
PW1	6.00 usec
DEADT	10.00 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	16
DUMMY	1
FREQU	80000.00 Hz
FLT	40000 Hz
DELAY	5.00 usec
ACQTM	0.4096 sec
PD	4.9500 sec
ADBIT	16
RGAIN	14
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM.
IRNUC	1H
IFR	399.65 MHz
IRSET	124.00 KHz
IRFIN	10500.00 Hz
IRRPW	45 usec
IRATN	511
DFILE	CF3-Diyne(Ph, Me)-H(19F).als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	23
LKPHS	249
LKSIG	647
CSPED	11 Hz
FILDC	
FILDF	

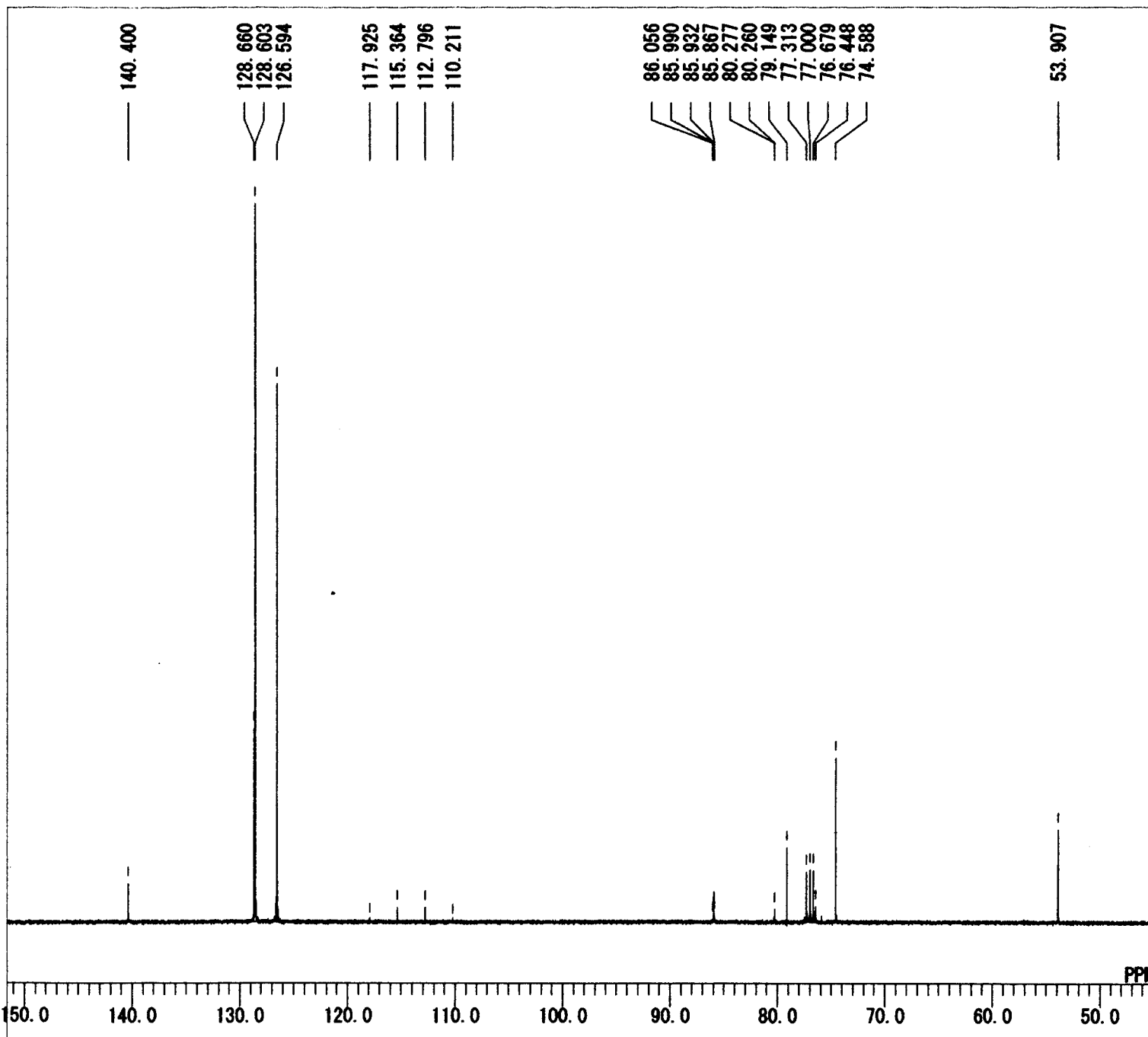


10A

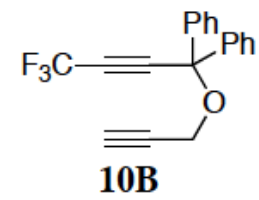


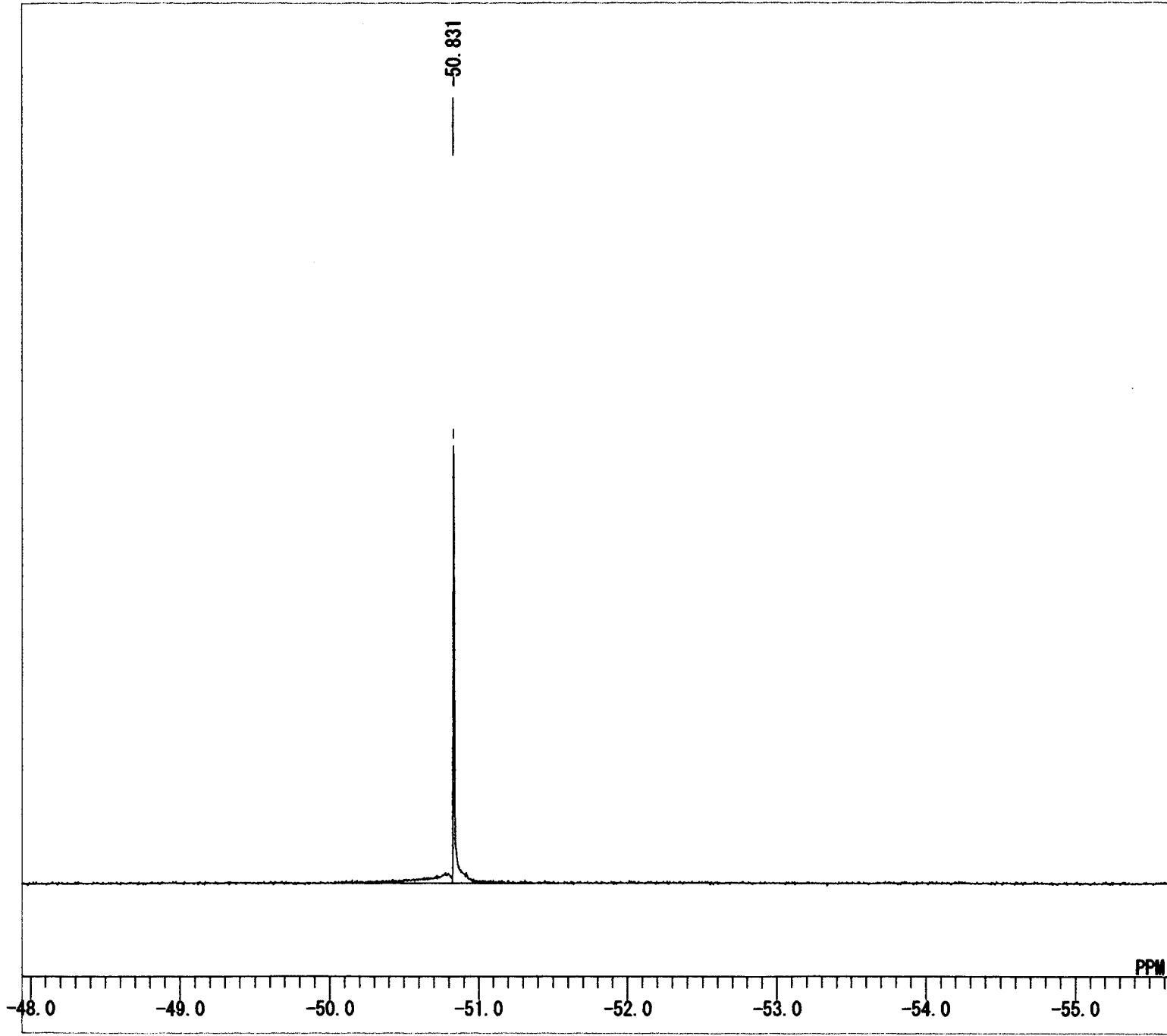
MENUF 1H
 OBNUC 1H
 OFR 399.65 MHz
 OBSET 135.40 KHz
 OBF IN 24.90 Hz
 PW1 5.80 usec
 DEADT 72.10 usec
 PREDL 0.20000 msec
 IWT 1.0000 msec
 POINT 16384
 SPO 16384
 TIMES 8
 DUMMY 1
 FREQU 7992.01 Hz
 FLT 4000 Hz
 DELAY 50.00 usec
 ACQTM 2.0501 sec
 PD 4.9500 sec
 ADBIT 16
 RGAIN 21
 BF 0.10 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD NON
 EXPCM NON:Single.coupled:PW1_ACQTM
 IRNUC 1H
 IFR 399.65 MHz
 IRSET 136.90 KHz
 IRFIN 97.50 Hz
 IRRPW 45 usec
 IRATN 511
 DF1LE CF3-Diyne (Ph, Ph)-H(1H). als
 SF TH5ATFG2
 LKSET 61.60 KHz
 LKFIN 79.0 Hz
 LKLEV 180
 LGAIN 23
 LKPHS 249
 LKSIG 874
 CSPED 14 Hz
 FILDC
 FILDF





MENUF 13C
 OBNUC 13C
 OFR 100.40 MHz
 OBSET 125.00 KHz
 OBF IN 10500.00 Hz
 PW1 6.00 usec
 DEADT 19.10 usec
 PREDL 0.20000 msec
 IWT 1.0000 msec
 POINT 32768
 SPO 32768
 TIMES 256
 DUMMY 1
 FREQU 27118.64 Hz
 FLT 13550 Hz
 DELAY 14.80 usec
 ACQTM 1.2083 sec
 PD 1.7920 sec
 ADBIT 16
 RGA IN 24
 BF 0.10 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD BCM
 EXPCM Bilevel. complete. decoupling:
 IRNUC 1H
 IFR 399.65 MHz
 IRSET 124.00 KHz
 IRFIN 10500.00 Hz
 IRRPW 45 usec
 IRATN 511
 DFILE CF3-Diyne (Ph, Ph)-H(13C). als
 SF TH5ATFG2
 LKSET 61.60 KHz
 LKFIN 79.0 Hz
 LKLEV 180
 LGAIN 23
 LKPHS 249
 LKSIG 695
 CSPED 13 Hz
 FILDC
 FILDF

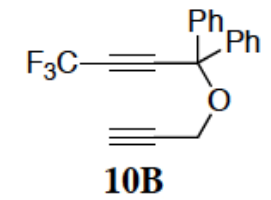


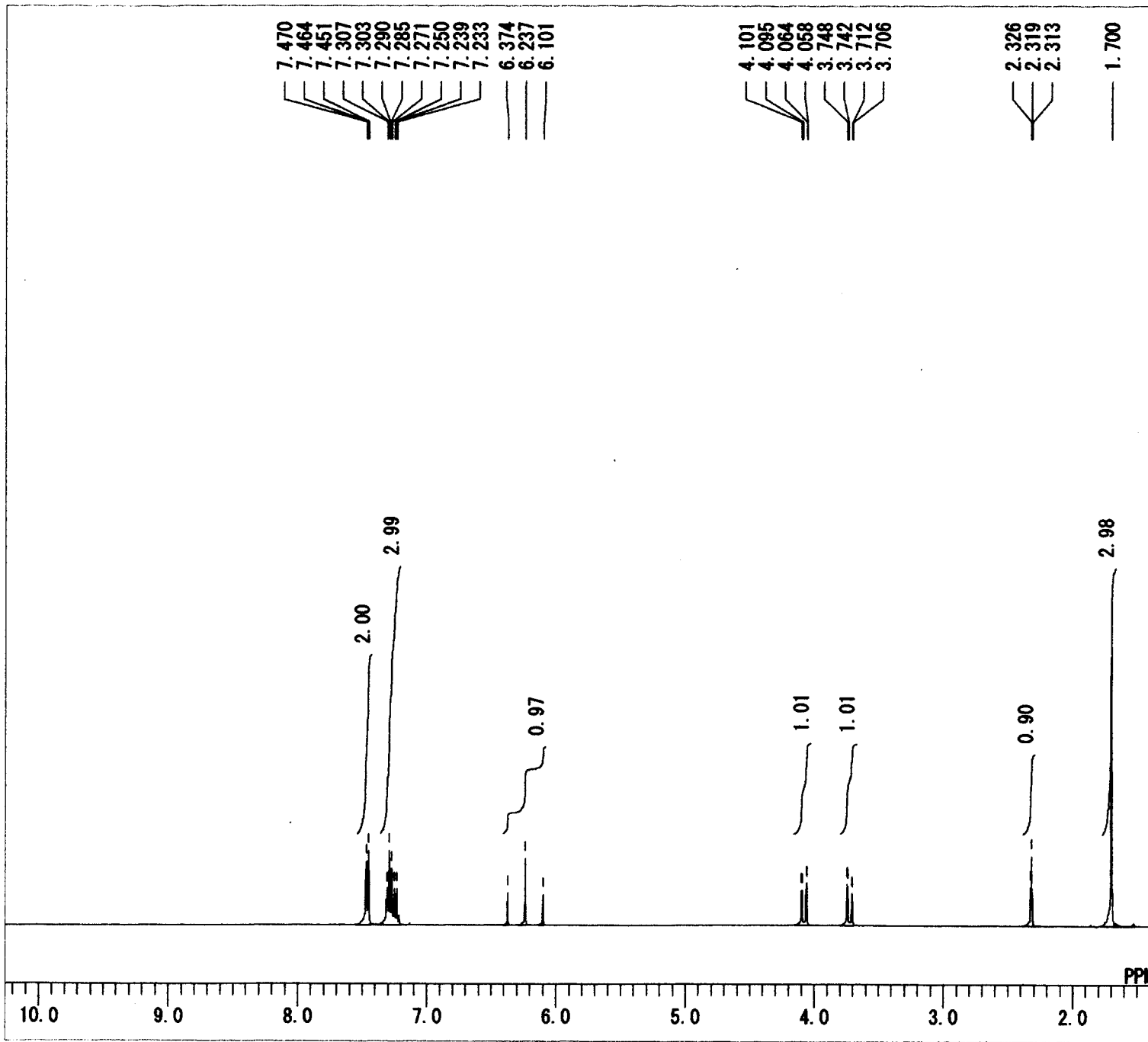


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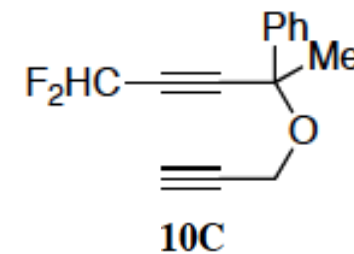
MENUF 19F
OBNUC 19F
OFR      376.05 MHz
OBSET    139.60 KHz
OBFIN    36.10 Hz
PW1      6.00 usec
DEADT    10.00 usec
PREDL    0.20000 msec
IWT      1.0000 msec
POINT    32768
SPO      32768
TIMES    16
DUMMY    1
FREQU    80000.00 Hz
FLT      40000 Hz
DELAY    5.00 usec
ACQTM    0.4096 sec
PD       4.9500 sec
ADBIT    16
RGAIN    14
BF       0.10 Hz
T1       0.00
T2       0.00
T3       90.00
T4       100.00
EXMOD    NON
EXPCM    NON:Single.coupled:PW1_ACQTM
IRNUC    1H
IFR      399.65 MHz
IRSET    124.00 KHz
IRFIN    10500.00 Hz
IRRPW    45 usec
IRATN    511
DFILE    CF3-Diyne (Ph, Ph)-H(19F).als
SF       TH5ATFG2
LKSET    61.60 KHz
LKFIN    79.0 Hz
LKLEV    180
LGAIN    24
LKPHS    249
LKSIG    559
CSPED    13 Hz
FILDC
FILDF

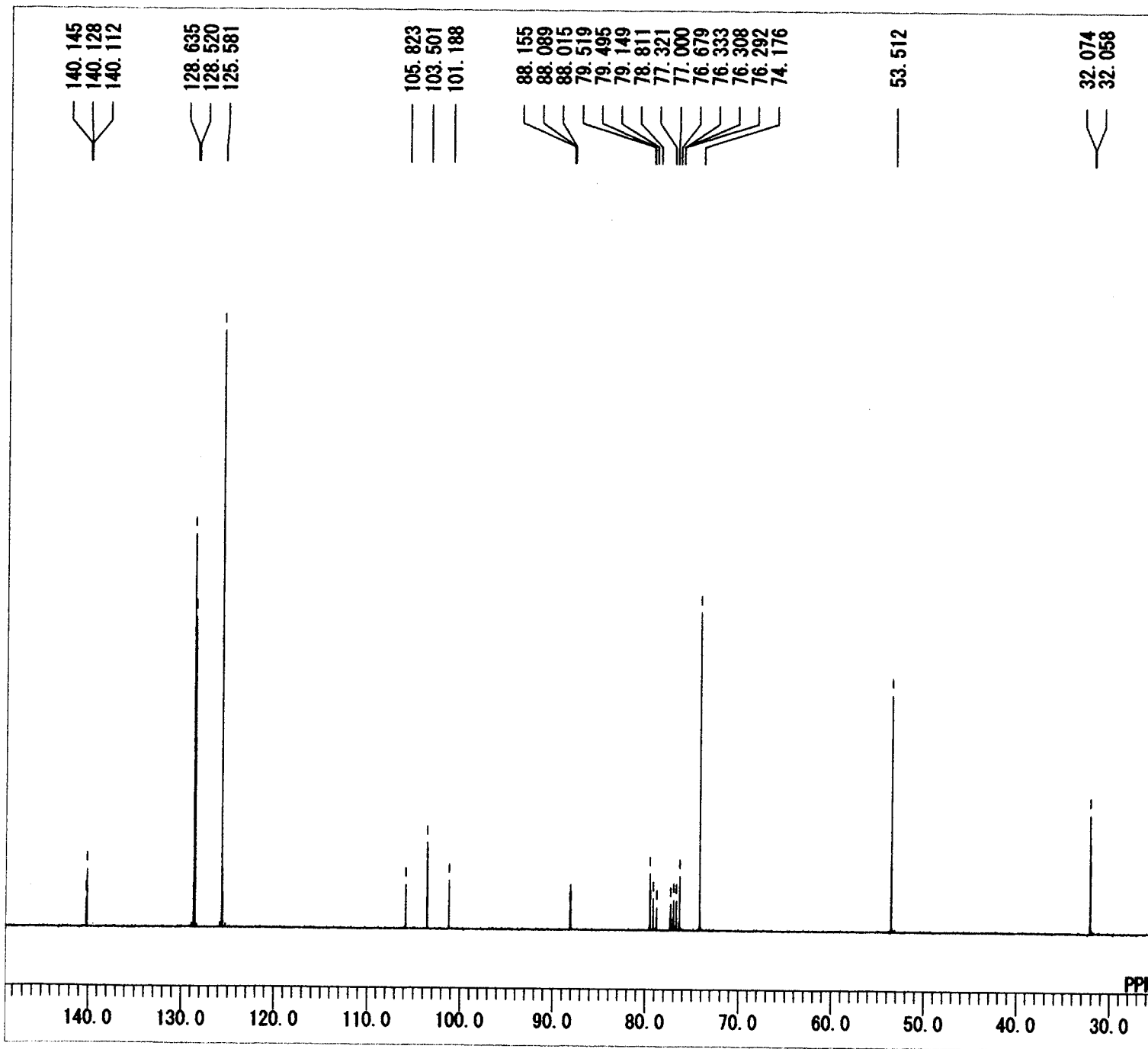
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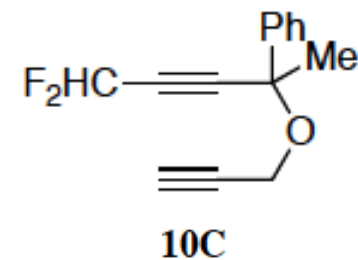


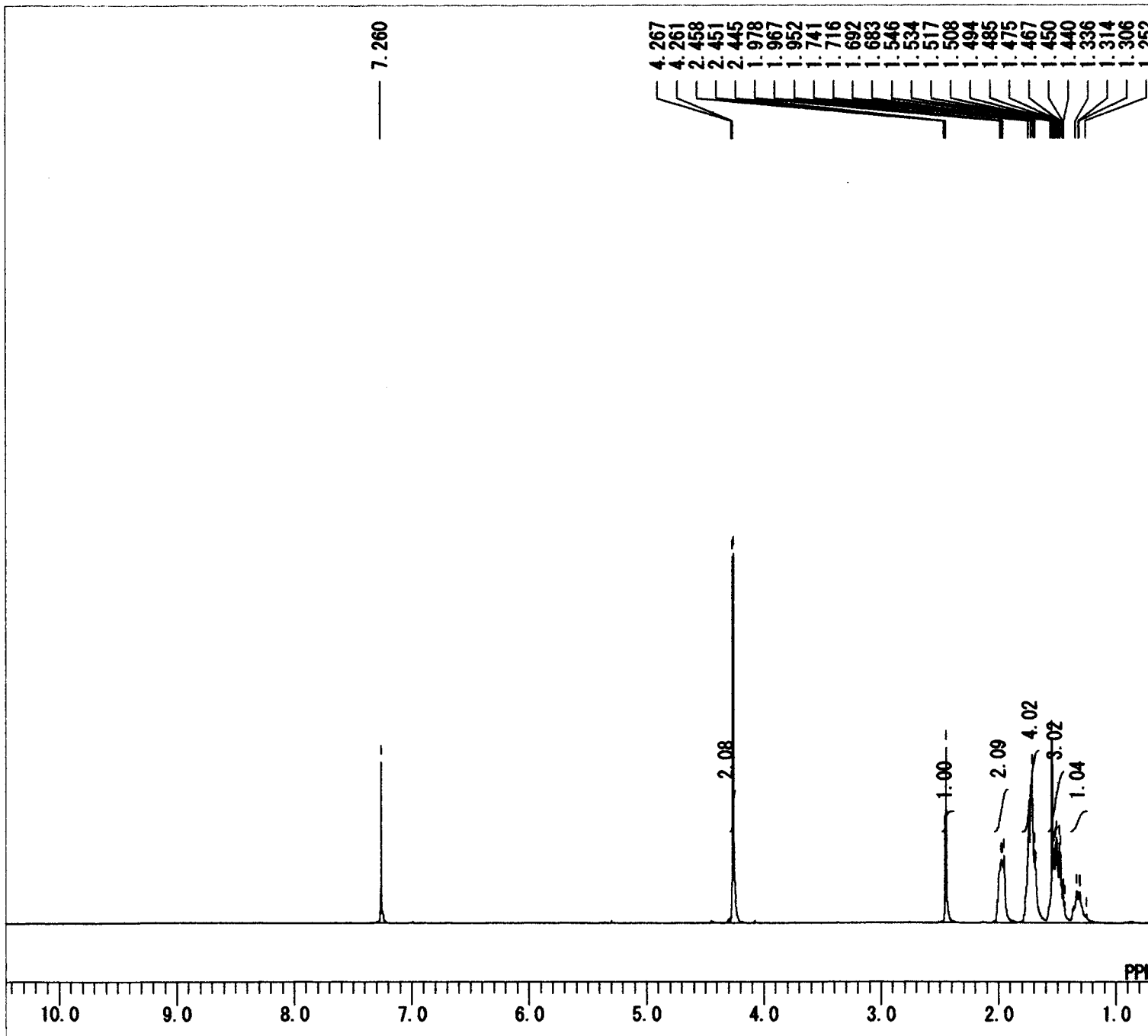
MENUF 1H
 OBNUC 1H
 OFR 399.65 MHz
 OBSET 135.40 KHz
 OBFIN 24.90 Hz
 PW1 5.80 usec
 DEADT 72.10 usec
 PREDL 0.20000 msec
 IWT 1.0000 msec
 POINT 16384
 SPO 16384
 TIMES 8
 DUMMY 1
 FREQU 7992.01 Hz
 FLT 4000 Hz
 DELAY 50.00 usec
 ACQTM 2.0501 sec
 PD 4.9500 sec
 ADBIT 16
 RGAIN 8
 BF 0.10 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD NON
 EXPCM NON:Single.coupled:PW1_ACQTM
 IRNUC 1H
 IFR 399.65 MHz
 IRSET 136.90 KHz
 IRFIN 97.50 Hz
 IRRPW 45 usec
 IRATN 511
 DF1LE CHF2-Diyne (Ph, Me)-H(1H). als
 SF TH5ATFG2
 LKSET 61.60 KHz
 LKFIN 79.0 Hz
 LKLEV 180
 LGAIN 25
 LKPHS 250
 LKSIG 999
 CSPED 13 Hz
 FILDC
 FILDF



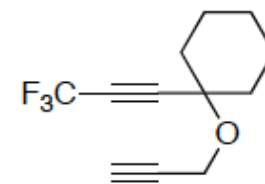


MENUF 13C
 OBNUC 13C
 OFR 100.40 MHz
 OBSSET 125.00 KHz
 OBFIN 10500.00 Hz
 PW1 6.00 usec
 DEADT 19.10 usec
 PREDL 0.20000 msec
 IWT 1.0000 msec
 POINT 32768
 SPO 32768
 TIMES 512
 DUMMY 1
 FREQU 27118.64 Hz
 FLT 13550 Hz
 DELAY 14.80 usec
 ACQTM 1.2083 sec
 PD 1.7920 sec
 ADBIT 16
 RGAIN 23
 BF 0.10 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD BCM
 EXPCM Bilevel. complete. decoupling: (S
 IRNUC 1H
 IFR 399.65 MHz
 IRSET 124.00 KHz
 IRFIN 10500.00 Hz
 IRRPW 45 usec
 IRATN 511
 DFILE CHF2-Diyne (Ph, Me)-H(13C). als
 SF TH5ATFG2
 LKSET 61.60 KHz
 LKFIN 79.0 Hz
 LKLEV 180
 LGAIN 25
 LKPHS 250
 LKSIG 1008
 CSPED 13 Hz
 FILDC
 FILDF

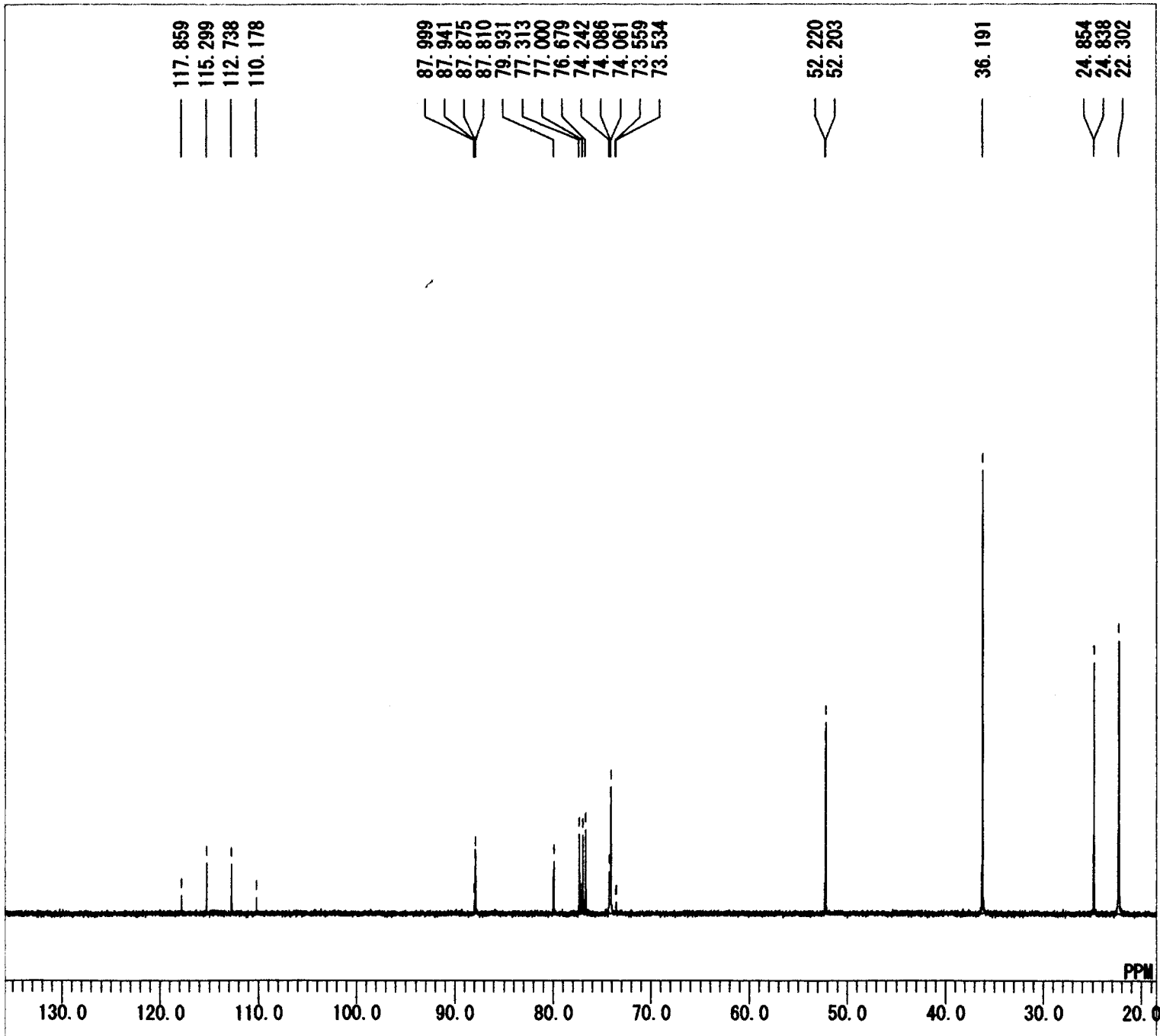




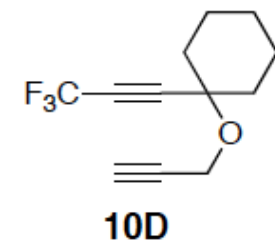
MENUF	1H
OBNUC	1H
OFR	399.65 MHz
OBSET	135.40 KHz
OBFIN	24.90 Hz
PW1	5.80 usec
DEADT	72.10 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7992.01 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	2.0501 sec
PD	4.9500 sec
ADBIT	16
RGAIN	17
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM
IRNUC	1H
IFR	399.65 MHz
IRSET	136.90 KHz
IRFIN	97.50 Hz
IRRPW	45 usec
IRATN	511
DFILE	CF3-Diyne(c-Hex)-H(1H).als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	23
LKPHS	250
LKSIG	850
CSPED	11 Hz
FILDC	
FILDF	

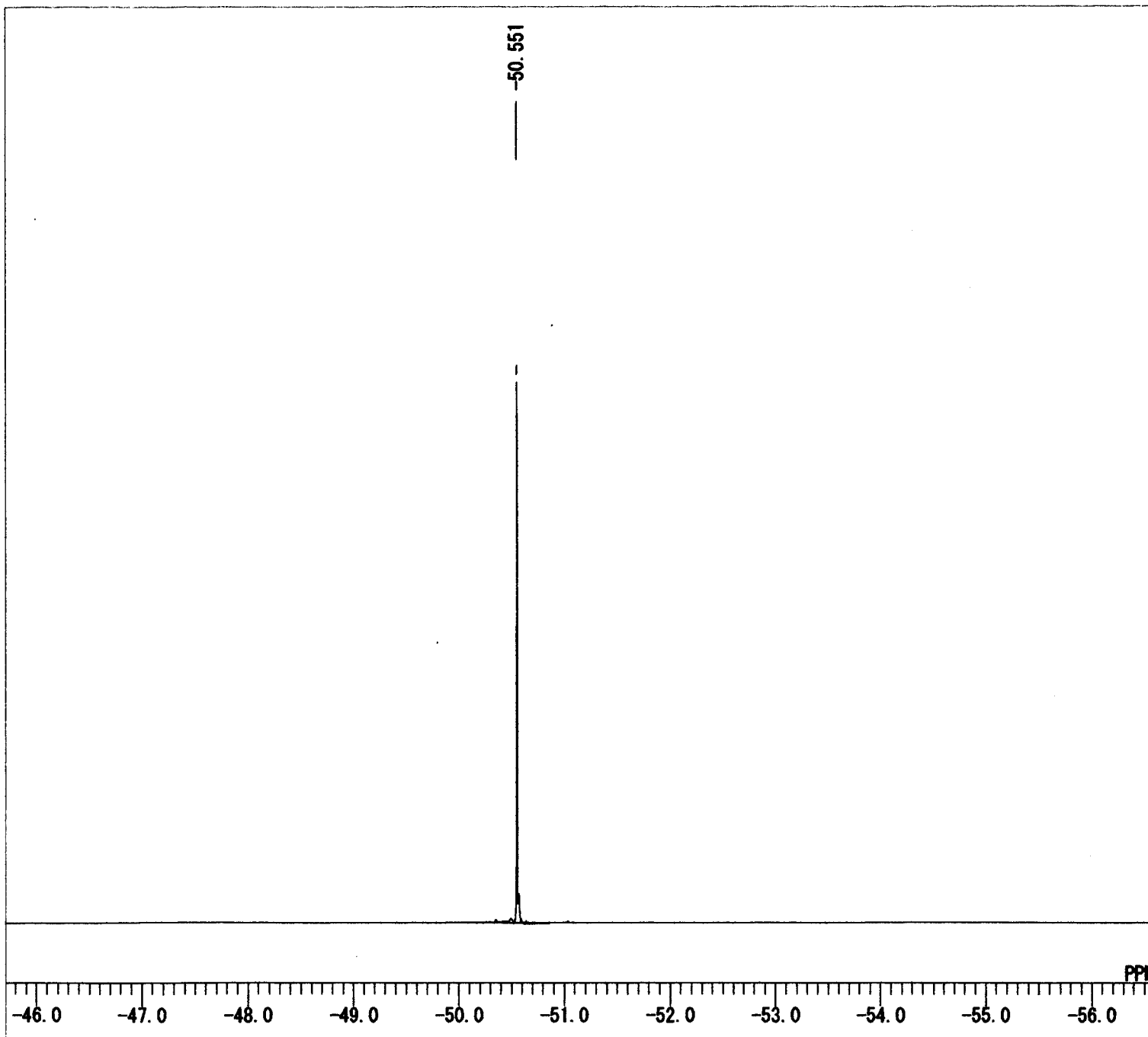


10D



MENUF 13C
 OBNUC 13C
 OFR 100.40 MHz
 OBSET 125.00 KHz
 OBF IN 10500.00 Hz
 PW1 6.00 usec
 DEADT 19.10 usec
 PREDL 0.20000 msec
 IWT 1.0000 msec
 POINT 32768
 SPO 32768
 TIMES 256
 DUMMY 1
 FREQU 27118.64 Hz
 FLT 13550 Hz
 DELAY 14.80 usec
 ACQTM 1.2083 sec
 PD 1.7920 sec
 ADBIT 16
 RGAIN 24
 BF 0.10 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD BCM
 EXPCM Bilevel. complete. decoupling:
 IRNUC 1H
 IFR 399.65 MHz
 IRSET 124.00 KHz
 IRF IN 10500.00 Hz
 IRRPW 45 usec
 IRATN 511
 DFILE CF3-Diyne (c-Hex)-H(13C). als
 SF TH5ATFG2
 LKSET 61.60 KHz
 LKF IN 79.0 Hz
 LKLEV 180
 LGAIN 24
 LKPHS 250
 LKSIG 811
 CSPED 7 Hz
 FILDC
 FILDF

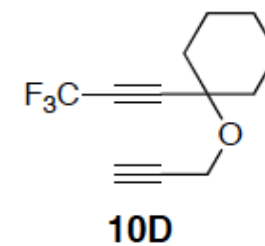


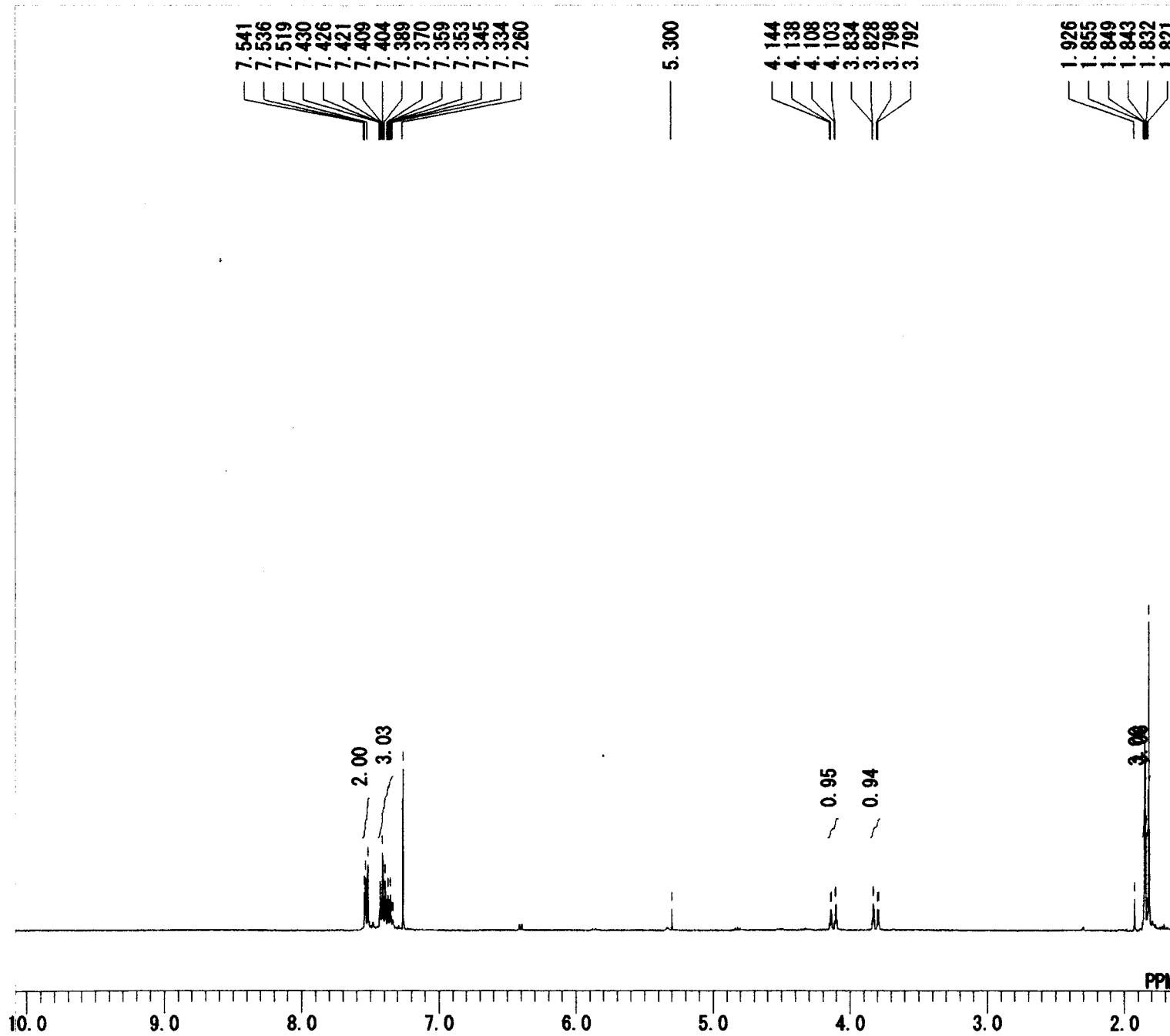


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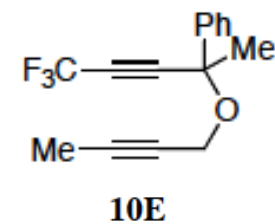
MENUF 19F
OBNUC 19F
OFR      376.05 MHz
OBSET    139.60 KHz
OBFIN    36.10 Hz
PW1      6.00 usec
DEADT    10.00 usec
PREDL    0.20000 msec
IWT      1.0000 msec
POINT    32768
SPO      32768
TIMES    16
DUMMY    1
FREGU    80000.00 Hz
FLT      40000 Hz
DELAY    5.00 usec
ACQTM    0.4096 sec
PD       4.9500 sec
ADBIT    16
RGAIN    14
BF       0.10 Hz
T1       0.00
T2       0.00
T3       90.00
T4       100.00
EXMOD    NON
EXPCM    NON:Single.coupled:PW1_ACQTM
IRNUC    1H
IFR      399.65 MHz
IRSET    124.00 KHz
IRFIN    10500.00 Hz
IRRPW    45 usec
IRATN    511
DFILE    CF3-Diyne(c-Hex)-H(19F).als
SF       TH5ATFG2
LKSET    61.60 KHz
LKFIN    79.0 Hz
LKLEV    180
LGAIN    23
LKPHS    250
LKSIG    838
CSPED    11 Hz
FILDC
FILDF

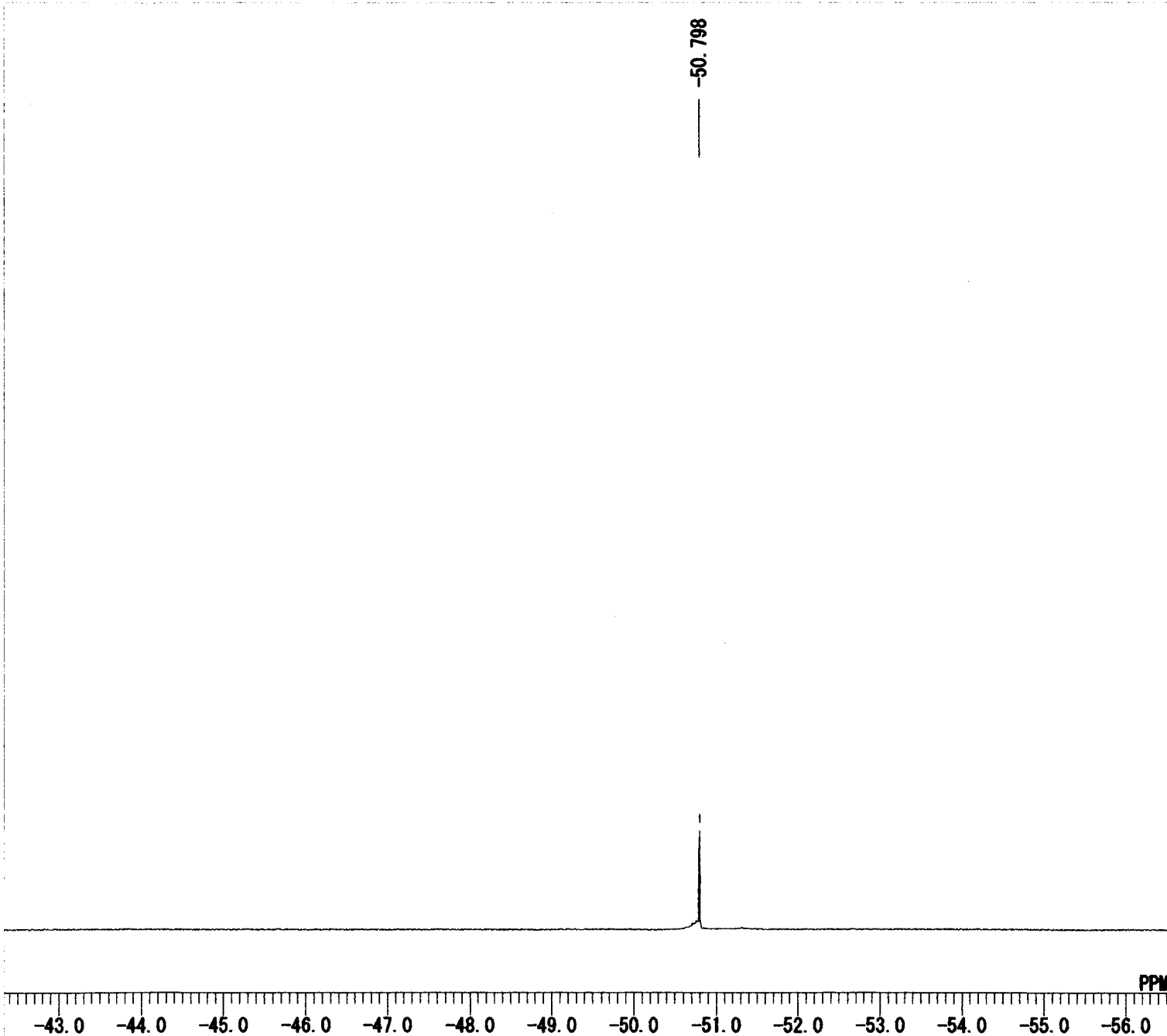
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MENUF	1H
OBNUC	1H
OFR	399.65 MHz
OBSET	135.40 KHz
OBFIN	24.90 Hz
PW1	5.80 usec
DEADT	72.10 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7992.01 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	2.0501 sec
PD	4.9500 sec
ADBIT	16
RGAIN	19
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single. coupled:PW1_ACQTM
IRNUC	1H
IFR	399.65 MHz
IRSET	136.90 KHz
IRFIN	97.50 Hz
IRRPW	45 usec
IRATN	511
DFILE	diyne-PhMe-Me(1H)混.als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	26
LKPHS	250
LKSIG	2681
CSPED	12 Hz
FILDC	
FILDF	

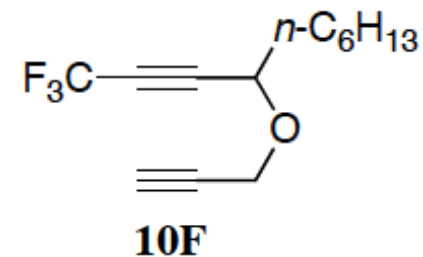


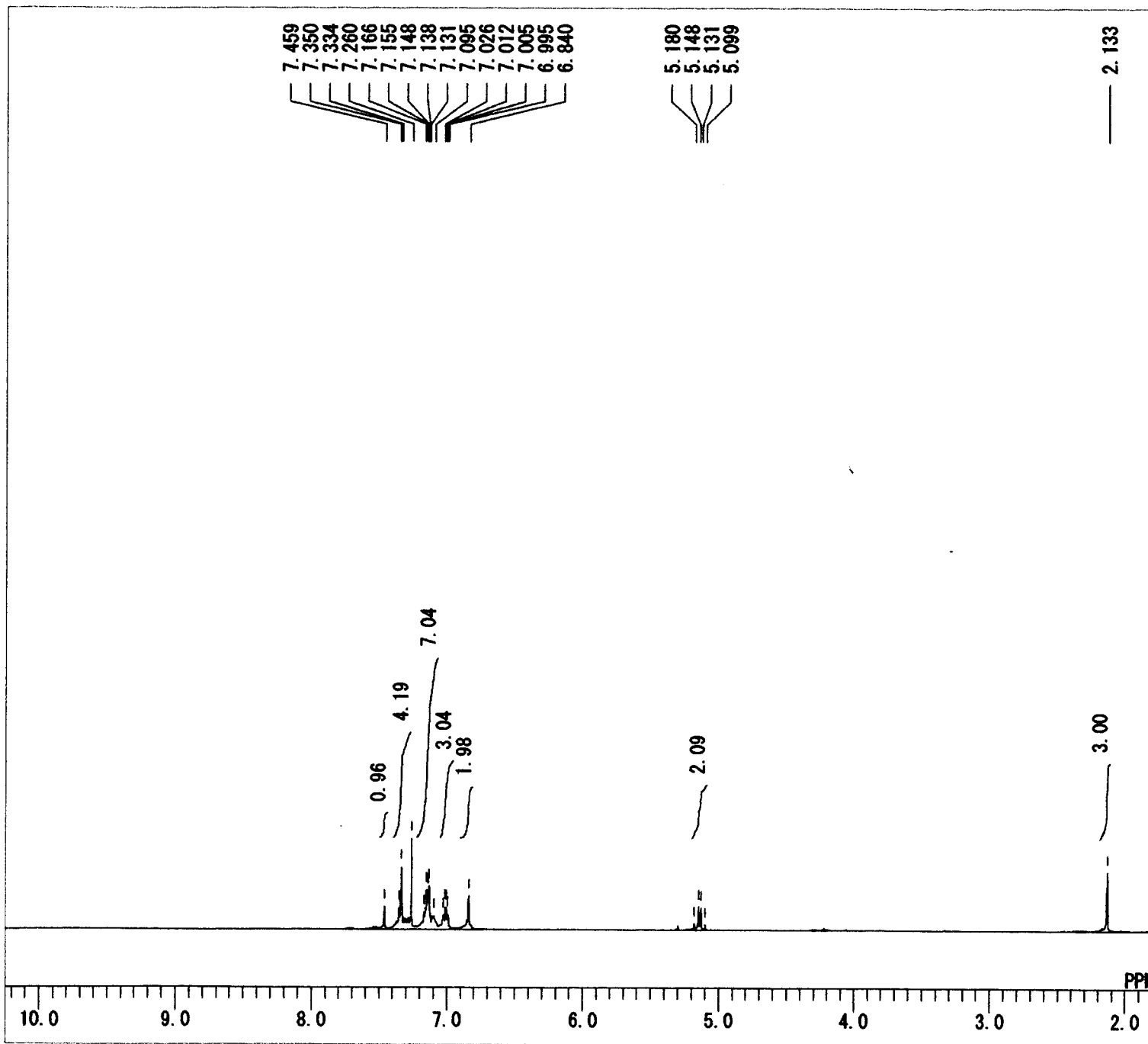


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MENUF 19F
OENUC 19F
OFR      376.05 MHz
OBSET    139.60 KHz
OBFIN    36.10 Hz
PW1       6.00 usec
DEADT    10.00 usec
PREDL    0.20000 msec
IWT      1.0000 msec
POINT    32768
SPO      32768
TIMES    16
DUMMY    1
FREQU    80000.00 Hz
FLT      40000 Hz
DELAY    5.00 usec
ACQTM    0.4096 sec
PD       4.9500 sec
ADBIT    16
RGAIN    14
BF        0.10 Hz
T1        0.00
T2        0.00
T3        90.00
T4       100.00
EXMOD    NON
EXPCM    NON:Single.coupled:PW1_ACQTM
IRNUC    1H
IFR      399.65 MHz
IRSET    124.00 KHz
IRFIN    10500.00 Hz
IRRPW    45 usec
IRATN    511
DFILE    CF3-diyne-n-C6H13(19F).als
SF       TH5ATFG20
LKSET    61.60 KHz
LKFIN    79.0 Hz
LKLEV    180
LGAIN    23
LKPHS    250
LKSIG    626
CSPED    11 Hz
FILDC
FILDF

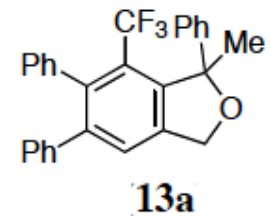
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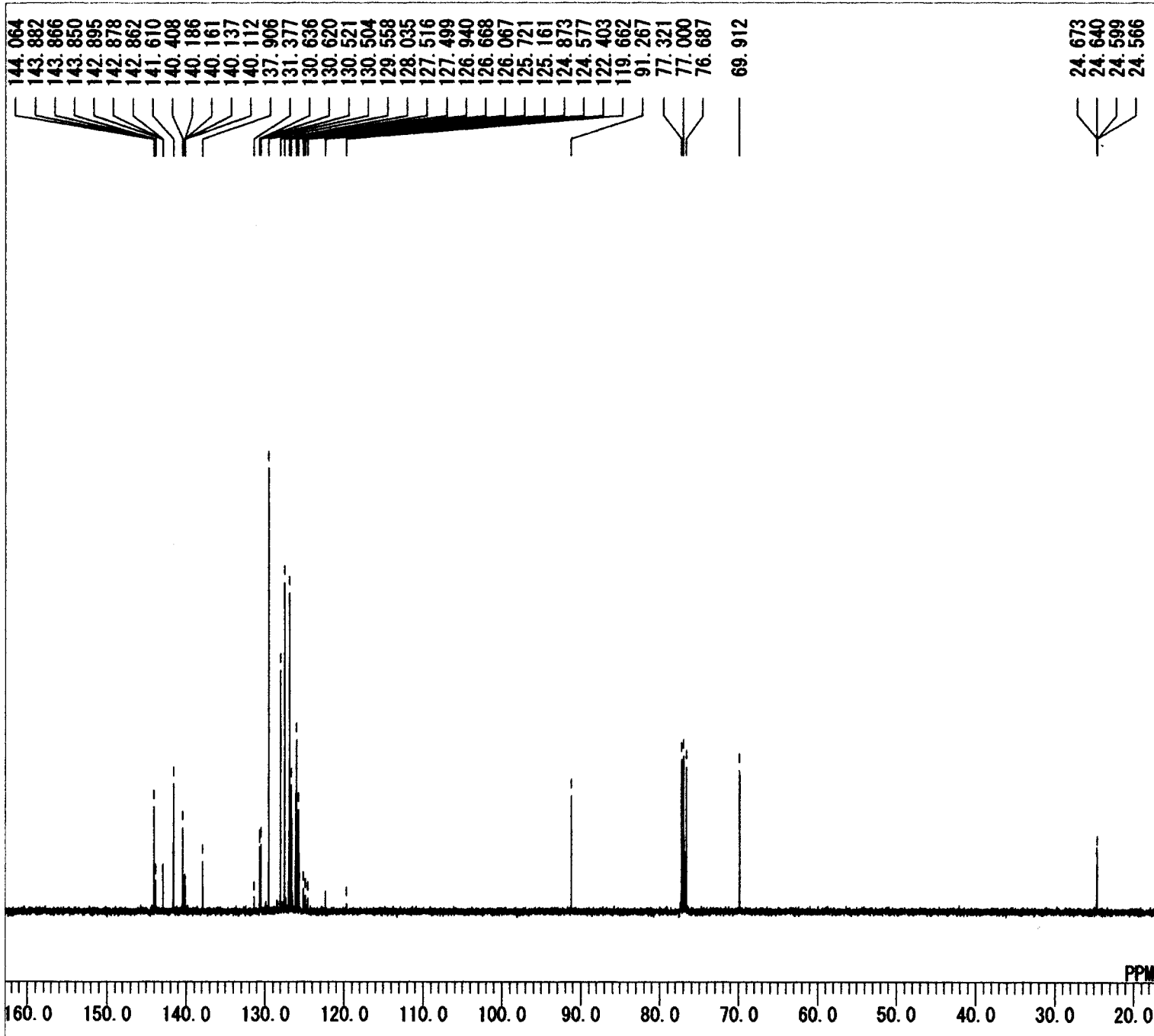




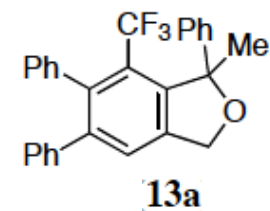
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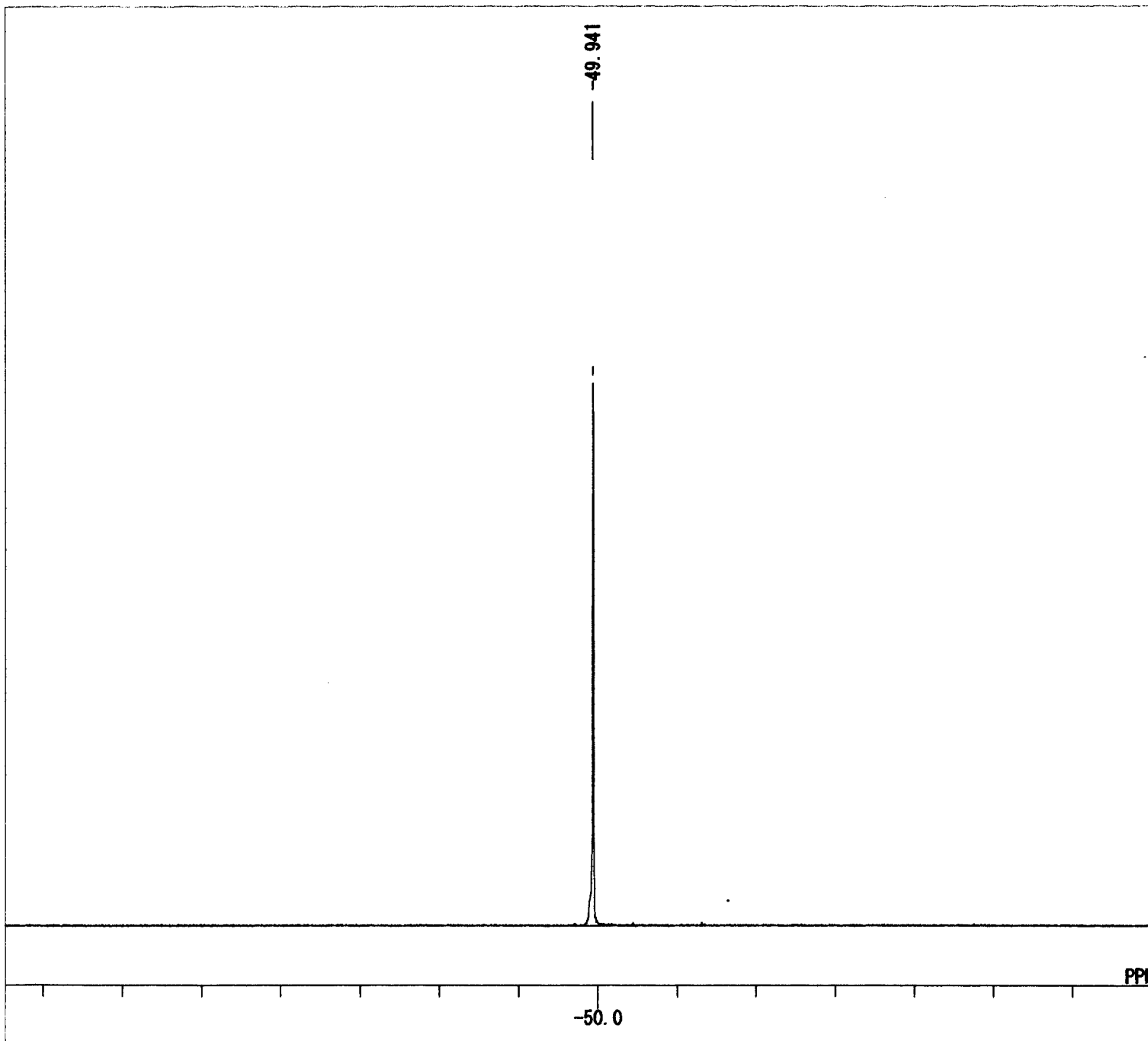
MNUF 1H
OBNUC 1H
OFR 399.65 MHz
OBSET 135.40 KHz
OBF1N 24.90 Hz
PW1 5.80 usec
DEADT 72.10 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7992.01 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 2.0501 sec
PD 4.9500 sec
ADBIT 16
RGAIN 18
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM
IRNUC 1H
IFR 399.65 MHz
IRSET 136.90 KHz
IRFIN 97.50 Hz
IRRPW 45 usec
IRATN 511
DFILE CF3-Diyne(PhMe)-H+diphenylac
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 24
LKPHS 249
LKSIG 1157
CSPED 13 Hz
FILDC
FILDF
  
```





MENUF 13C
 OBNUC 13C
 OFR 100.40 MHz
 OBSET 125.00 KHz
 OBFIN 10500.00 Hz
 PW1 6.00 usec
 DEADT 19.10 usec
 PREDL 0.20000 msec
 IWT 1.0000 msec
 POINT 32768
 SPO 32768
 TIMES 256
 DUMMY 1
 FREQU 27118.64 Hz
 FLT 13550 Hz
 DELAY 14.80 usec
 ACQTM 1.2083 sec
 PD 1.7920 sec
 ADBIT 16
 RGAIN 25
 BF 0.10 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD BCM
 EXPCM Bi level. complete. decoupling:
 IRNUC 1H
 IFR 399.65 MHz
 IRSET 124.00 KHz
 IRFIN 10500.00 Hz
 IRRPW 45 usec
 IRATN 511
 DFILE CF3-Diyne (PhMe)-H+diphenylac
 SF TH5ATFG2
 LKSET 61.60 KHz
 LKFIN 79.0 Hz
 LKLEV 180
 LGAIN 25
 LKPHS 250
 LKSIG 1541
 CSPED 10 Hz
 FILDC
 FILDF

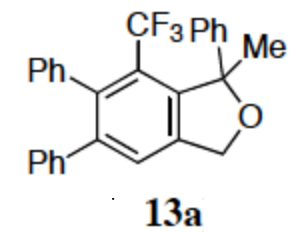


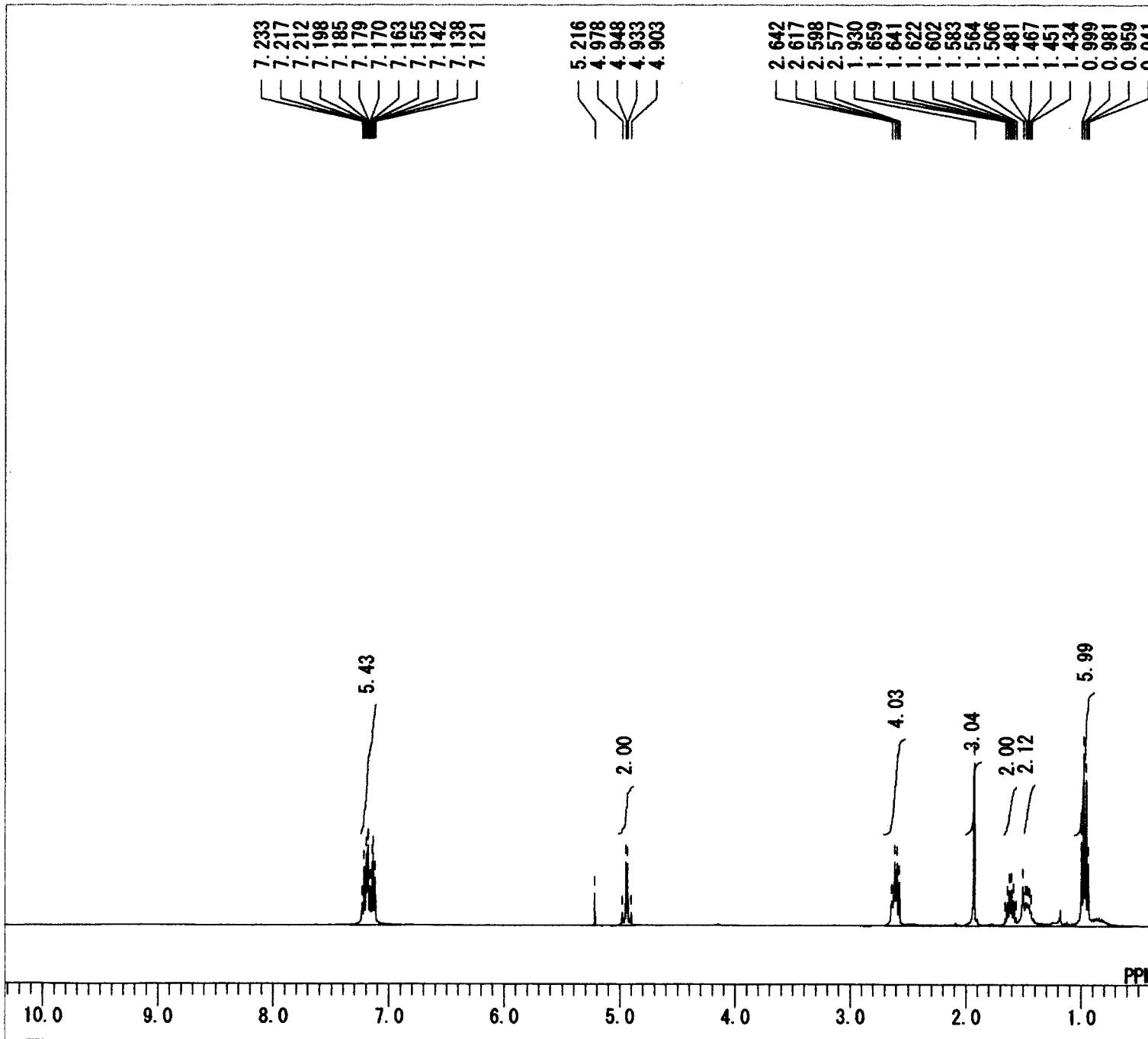


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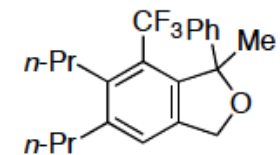
MENUF 19F
OBNUC 19F
OFR 376.05 MHz
OBSET 139.60 KHz
OBFIN 36.10 Hz
PW1 6.00 usec
DEADT 10.00 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 32768
SPO 32768
TIMES 16
DUMMY 1
FREQU 80000.00 Hz
FLT 40000 Hz
DELAY 5.00 usec
ACQTM 0.4096 sec
PD 4.9500 sec
ADBIT 16
RGAIN 14
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM
IRNUC 1H
IFR 399.65 MHz
IRSET 124.00 KHz
IRFIN 10500.00 Hz
IRRPW 45 usec
IRATN 511
DFILE CF3-Diyne(PhMe)-H+diphenylac
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 24
LKPHS 249
LKSIG 1161
GSPED 13 Hz
FILDC
FILDF

```

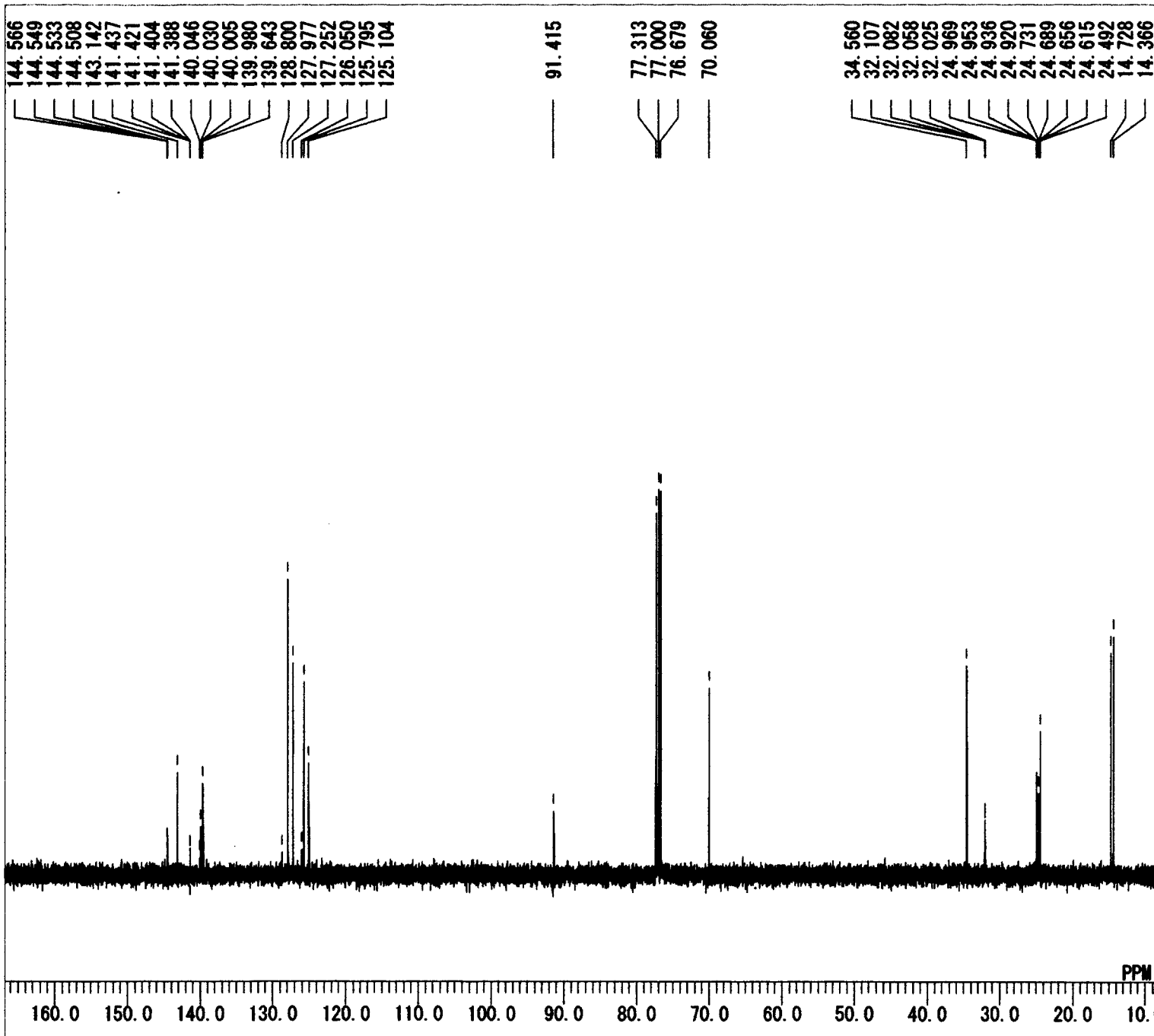




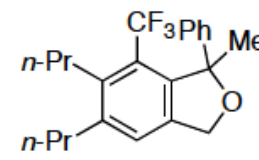
MENUF 1H
 OBNUC 1H
 OFR 399.65 MHz
 OBSET 135.40 KHz
 OBFIN 24.90 Hz
 PW1 5.80 usec
 DEADT 72.10 usec
 PREDL 0.20000 msec
 IWT 1.0000 msec
 POINT 16384
 SPO 16384
 TIMES 8
 DUMMY 1
 FREQU 7992.01 Hz
 FLT 4000 Hz
 DELAY 50.00 usec
 ACQTM 2.0501 sec
 PD 4.9500 sec
 ADBIT 16
 RGAIN 13
 BF 0.10 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD NON
 EXPCM NON:Single.coupled:PW1_ACQTM
 IRNUC 1H
 IFR 399.65 MHz
 IRSET 136.90 KHz
 IRFIN 97.50 Hz
 IRRPW 45 usec
 IRATN 511
 DFILE CF3-Diyne(PhMe)-H+4-Octyne-(
 SF TH5ATFG2
 LKSET 61.60 KHz
 LKFIN 79.0 Hz
 LKLEV 180
 LGAIN 25
 LKPHS 250
 LKSIG 1596
 CSPED 10 Hz
 FILDC
 FILDF



13b

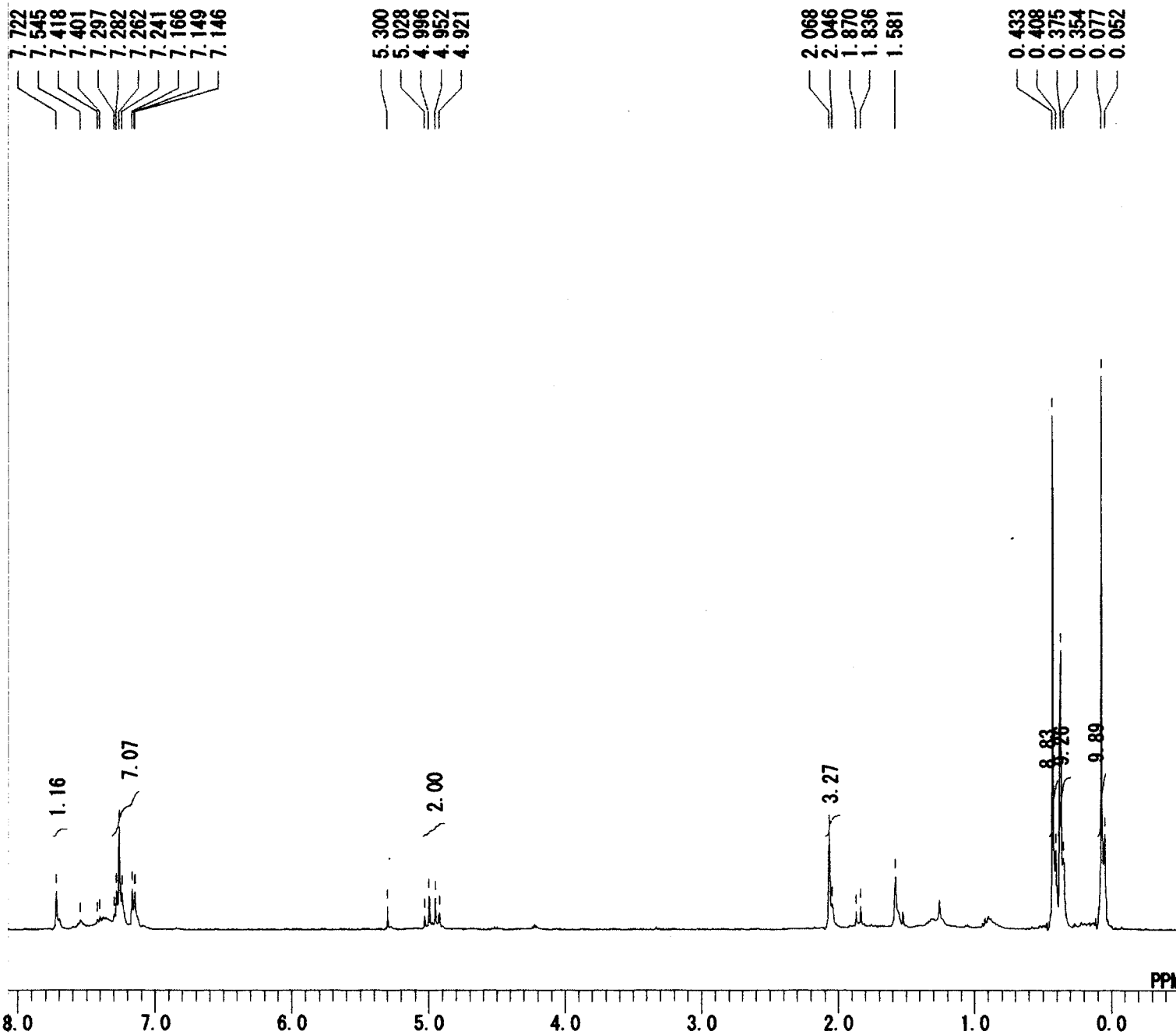


MENUF 13C
OBRUC 13C
OFR 100.40 MHz
OBSET 125.00 KHz
OBF IN 10500.00 Hz
PW1 6.00 usec
DEADT 19.10 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 32768
SPO 32768
TIMES 512
DUMMY 1
FREQU 27118.64 Hz
FLT 13550 Hz
DELAY 14.80 usec
ACQTM 1.2083 sec
PD 1.7920 sec
ADBIT 16
RGAIN 25
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD BCM
EXPCM Bilevel. complete. decoupling:
IRNUC 1H
IFR 399.65 MHz
IRSET 124.00 KHz
IRFIN 10500.00 Hz
IRRPW 45 usec
IRATN 511
DFILE CF3-Diyne(PhMe)-H+4-Octyne-(
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 25
LKPHS 250
LKSIG 1580
CSPED 13 Hz
FILDC
FILDF

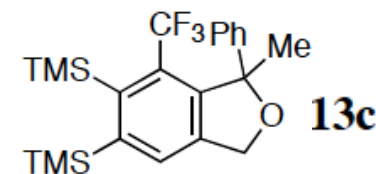


13b

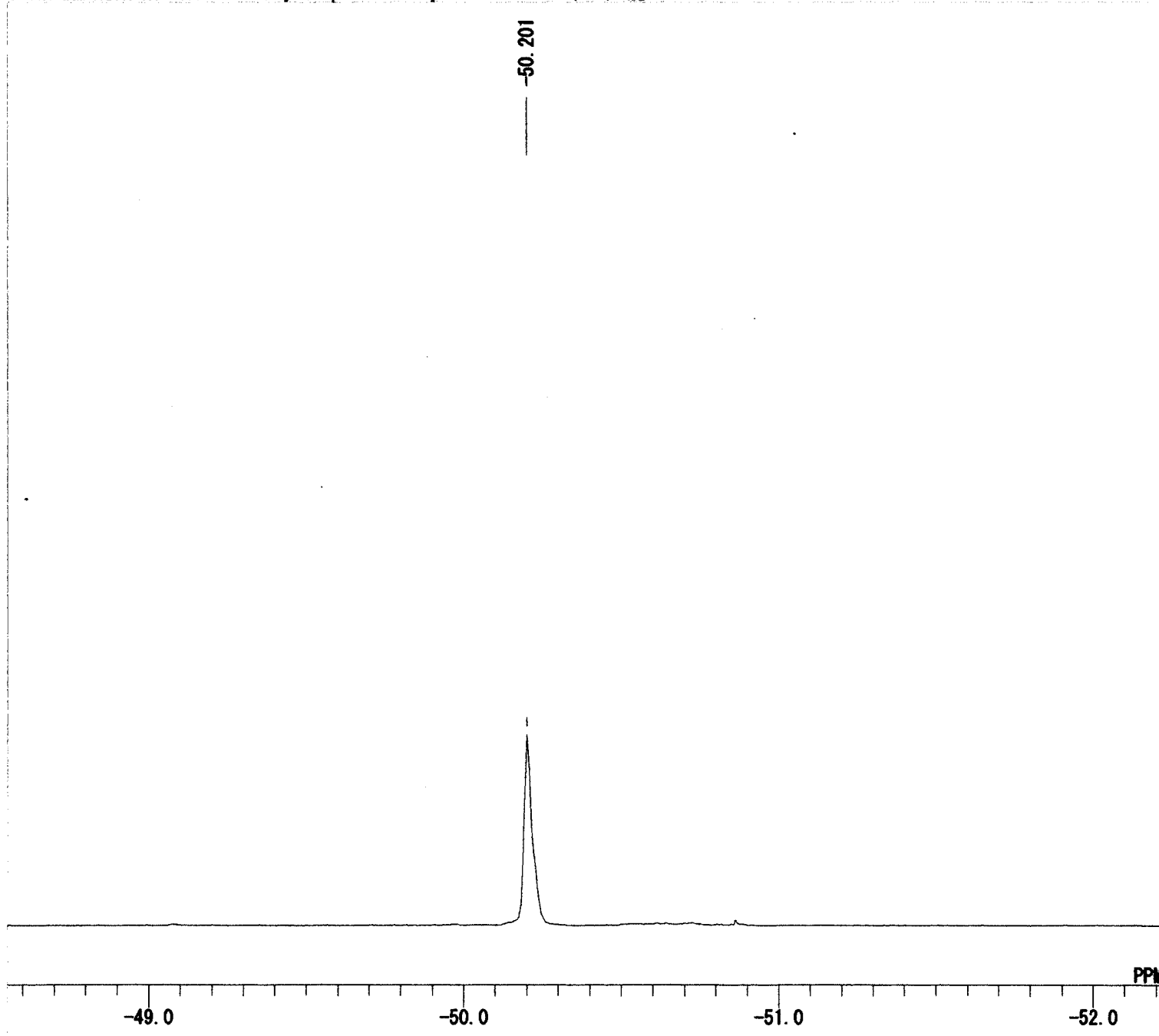
C:\WINNMR\DATA\Fluorine\Moriyasu\product-diyne-PhMe-diTMS(1H)-混.als



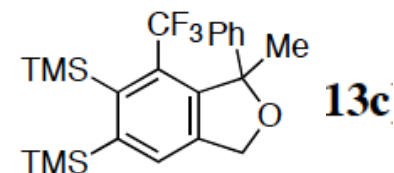
MENUF 1H
 OBNUC 1H
 OFR 399.65 MHz
 OBSET 135.40 KHz
 OBF IN 24.90 Hz
 PW1 5.80 usec
 DEADT 72.10 usec
 PREDL 0.20000 msec
 IWT 1.0000 msec
 POINT 16384
 SPO 16384
 TIMES 8
 DUMMY 1
 FREQU 7992.01 Hz
 FLT 4000 Hz
 DELAY 50.00 usec
 ACQTM 2.0501 sec
 PD 4.9500 sec
 ADBIT 16
 RGAIN 15
 BF 0.10 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD NON
 EXPCM NON:Single.coupled:PW1_ACQTM
 IRNUC 1H
 IFR 399.65 MHz
 IRSET 136.90 KHz
 IRF IN 97.50 Hz
 IRRPW 45 usec
 IRATN 511
 DF FILE product-diyne-PhMe-diTMS(1H)-
 SF TH5ATFG2
 LKSET 61.60 KHz
 LKF IN 79.0 Hz
 LKLEV 180
 LGAIN 27
 LKPHS 250
 LKSIG 1717
 CSPED 13 Hz
 FILDC
 FILDF

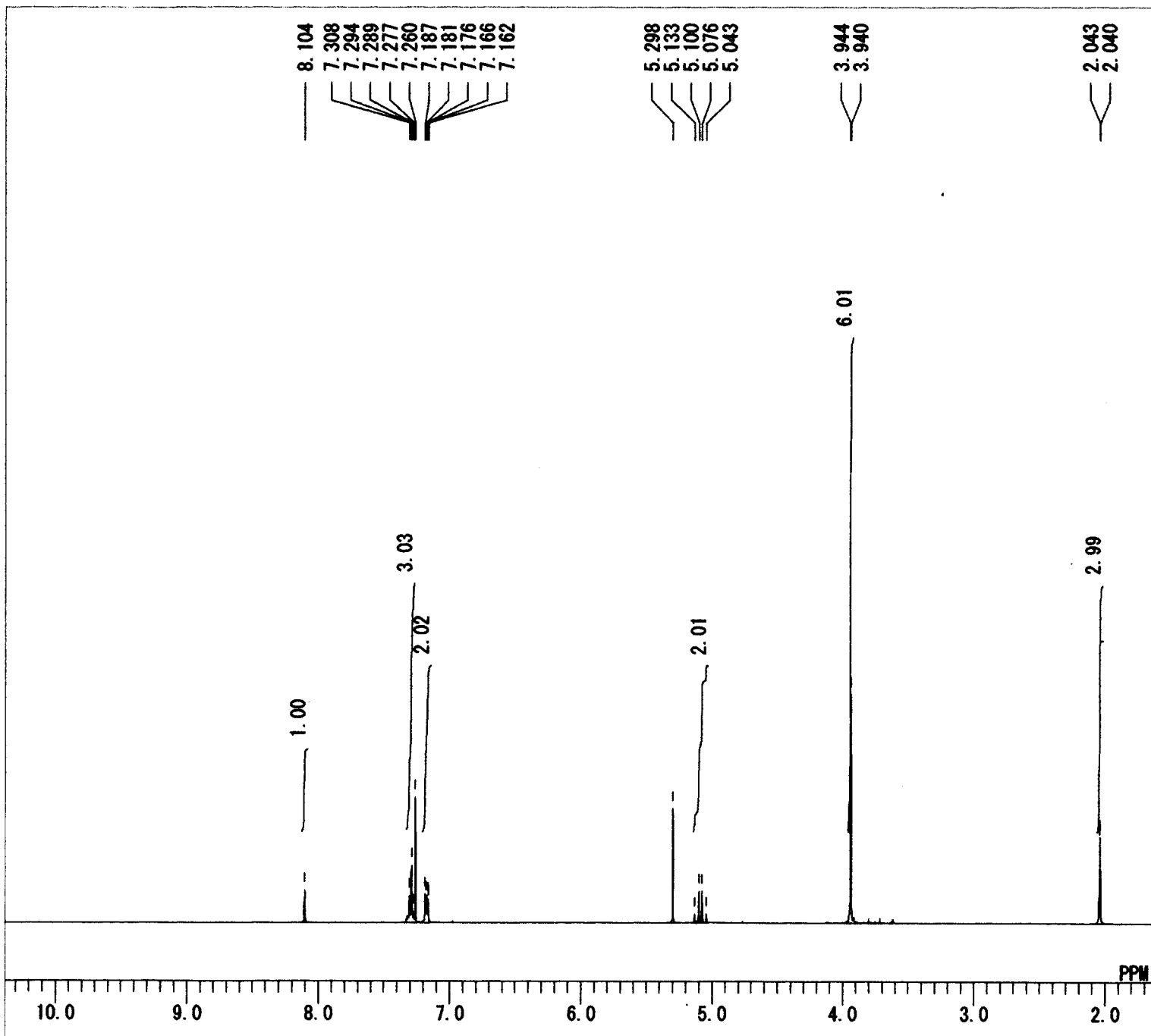


C:\WINNMR\DATA\Fluorine\Moriyasu\product-diyne-PhMe-diTMS (19F)-混. als

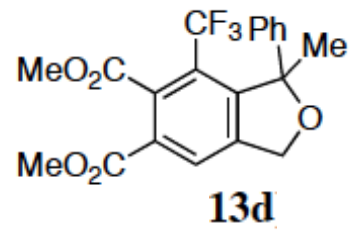


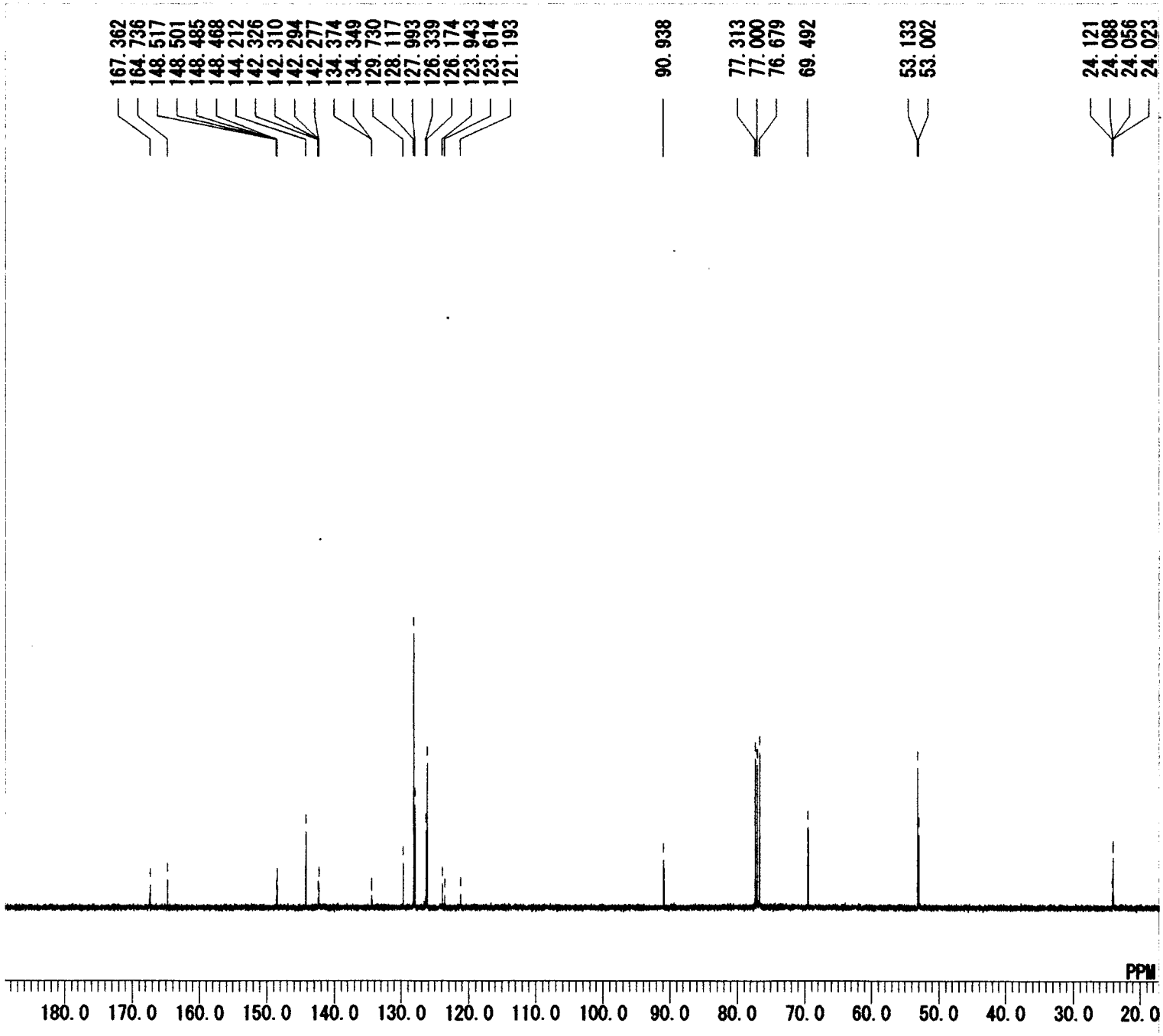
MENUF 19F
OBNUC 19F
OFR 376.05 MHz
OBSET 139.60 KHz
OBFIN 36.10 Hz
PW1 6.00 usec
DEADT 10.00 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 32768
SPO 32768
TIMES 16
DUMMY 1
FREQU 80000.00 Hz
FLT 40000 Hz
DELAY 5.00 usec
ACQTM 0.4096 sec
PD 4.9500 sec
ADBIT 16
RGAIN 14
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM.
IRNUC 1H
IFR 399.65 MHz
IRSET 124.00 KHz
IRFIN 10500.00 Hz
IRRPW 45 usec
IRATN 511
DFILE product-diyne-PhMe-diTMS (19F)
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 27
LKPHS 250
LKSIG 1636
CSPED 12 Hz
FILDC
FILDF



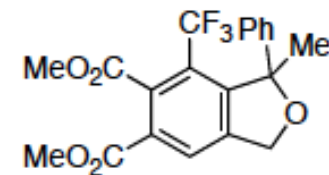


MENUF	1H
OBNUC	1H
OFR	399.65 MHz
OBSET	135.40 KHz
OBFIN	24.90 Hz
PW1	5.80 usec
DEADT	72.10 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7992.01 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	2.0501 sec
PD	4.9500 sec
ADBIT	16
RGAIN	19
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM.
IRNUC	1H
IFR	399.65 MHz
IRSET	136.90 KHz
IRFIN	97.50 Hz
IRRPW	45 usec
IRATN	511
DFILE	CF3-Diyne (PhMe)-H+Di (CO2Me) A
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGA1N	23
LKPHS	250
LKSIG	1090
CSPED	12 Hz
FILDG	
FILDF	

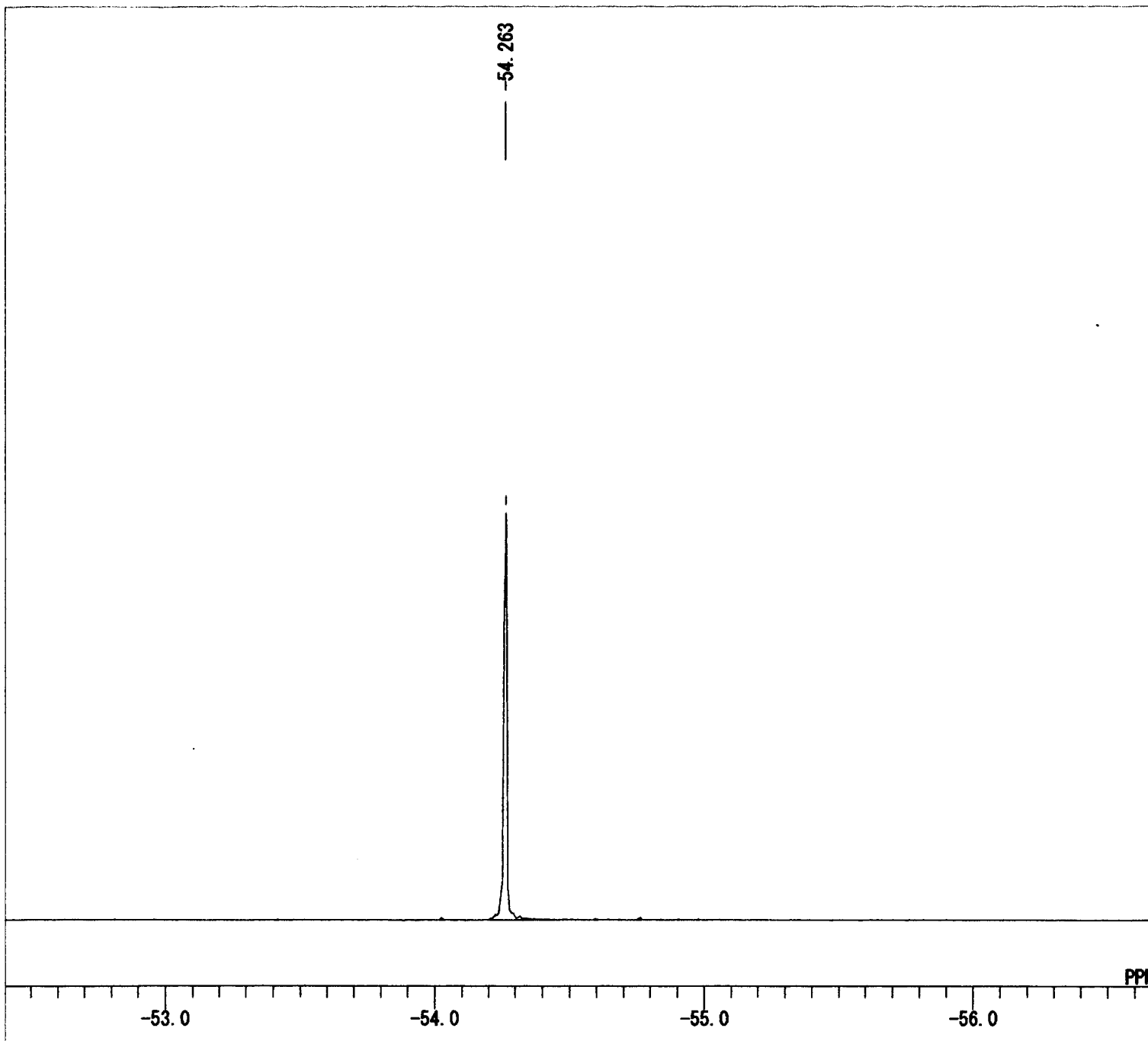




MENUF 13C
 OBNUC 13C
 OFR 100.40 MHz
 OBSSET 125.00 KHz
 OBF IN 10500.00 Hz
 PW1 6.00 usec
 DEADT 19.10 usec
 PREDL 0.20000 msec
 IWT 1.0000 msec
 POINT 32768
 SPO 32768
 TIMES 512
 DUMMY 1
 FREQU 27118.64 Hz
 FLT 13550 Hz
 DELAY 14.80 usec
 ACQTM 1.2083 sec
 PD 1.7920 sec
 ADBIT 16
 RGAIN 25
 BF 0.10 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD BCM
 EXPCM Bilevel. complete. decoupling:
 IRNUC 1H
 IFR 399.65 MHz
 IRSET 124.00 KHz
 IRF IN 10500.00 Hz
 IRRPW 45 usec
 IRATN 511
 DF FILE product-diyne-PhMe-di (CO2Me)-
 SF TH5ATFG2
 LKSET 61.60 KHz
 LKF IN 79.0 Hz
 LKLEV 180
 LGAIN 23
 LKPHS 250
 LKSIG 809
 CSPED 13 Hz
 FILDC
 FILDF



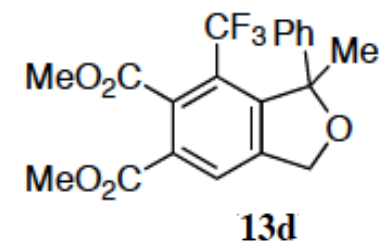
13d

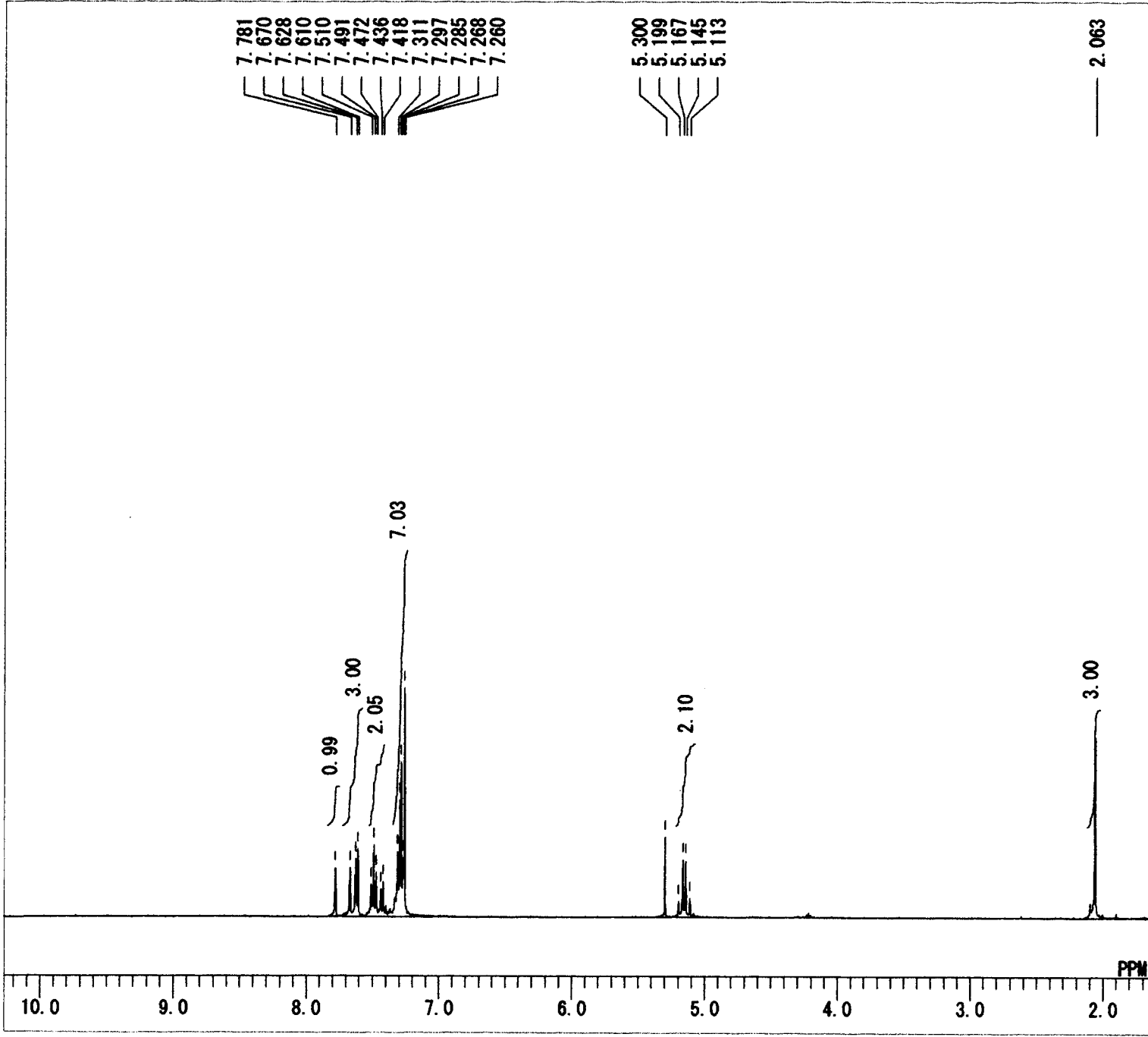


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MENUF 19F
OBNUC 19F
OFR      376.05 MHz
OBSET    139.60 KHz
OBF IN   36.10 Hz
PW1      6.00 usec
DEADT    10.00 usec
PREDL    0.20000 msec
IWT      1.0000 msec
POINT    32768
SPO      32768
TIMES    16
DUMMY    1
FREQU    80000.00 Hz
FLT      40000 Hz
DELAY    5.00 usec
ACQTM    0.4096 sec
PD       4.9500 sec
ADBIT    16
RGAIN    14
BF        0.10 Hz
T1        0.00
T2        0.00
T3        90.00
T4       100.00
EXMOD    NON
EXPCM    NON:Single.coupled:PW1_ACQTM
IRNUC    1H
IFR      399.65 MHz
IRSET    124.00 KHz
IRF IN   10500.00 Hz
IRRPW    45 usec
IRATN    511
DFILE    CF3-Diyne(PhMe)-H+Di(CO2Me)A
SF       TH5ATFG2
LKSET    61.60 KHz
LKFIN    79.0 Hz
LKLEV    180
LGA IN   23
LKPHS    250
LKSIG    1090
CSPED    9 Hz
FILDC
FILDF

```

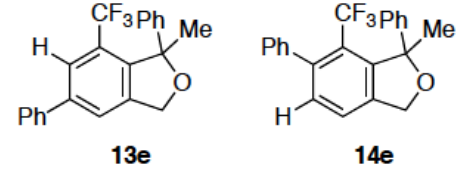


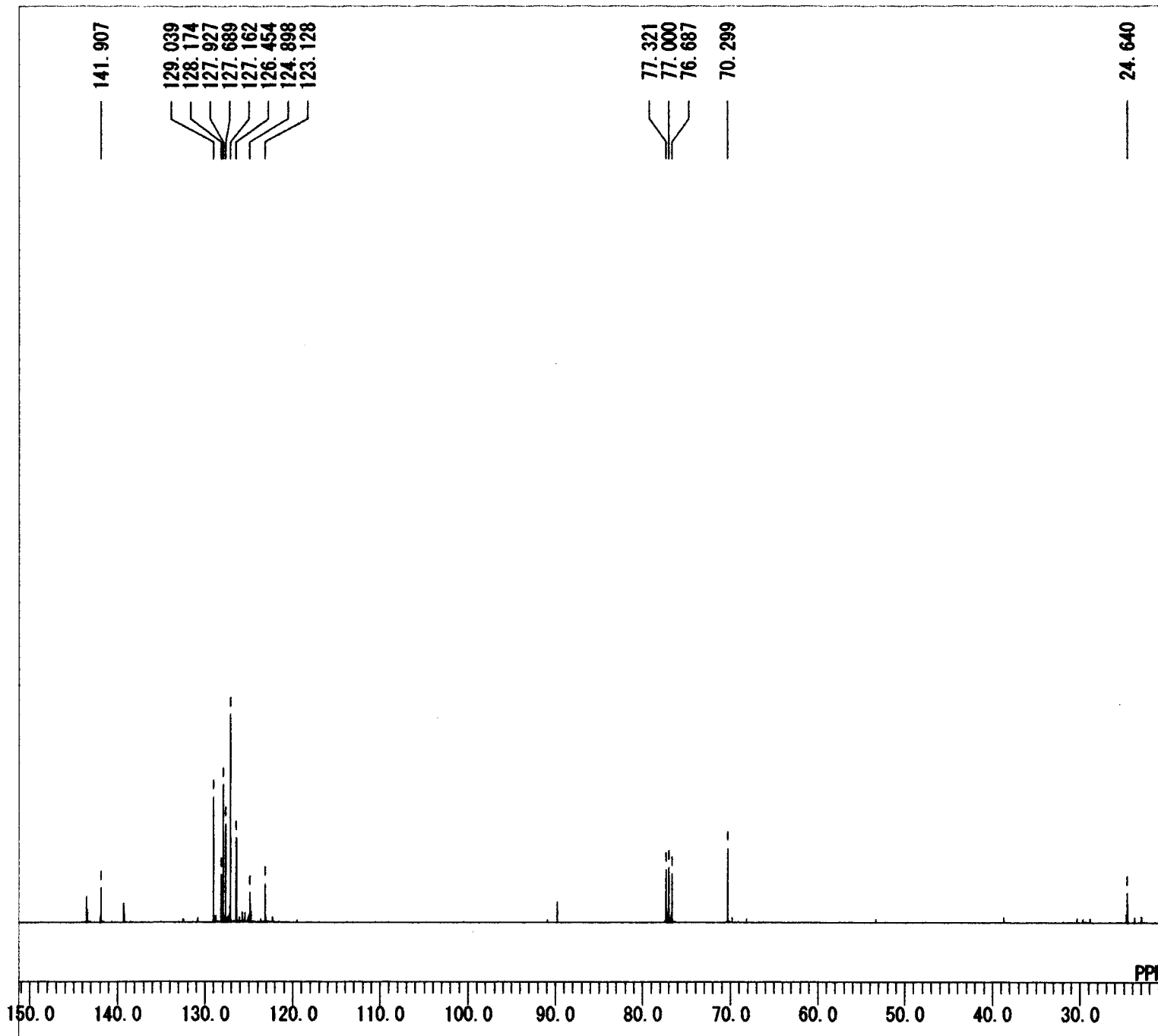


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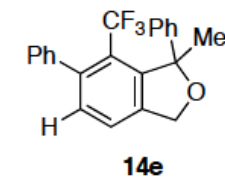
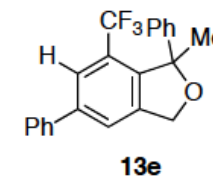
MENUF 1H
OBNUC 1H
OFR 399.65 MHz
OBSET 135.40 KHz
OBFIN 24.90 Hz
PW1 5.80 usec
DEADT 72.10 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREGU 7992.01 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 2.0501 sec
PD 4.9500 sec
ADBIT 16
RGAIN 19
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM
IRNUC 1H
IFR 399.65 MHz
IRSET 136.90 KHz
IRFIN 97.50 Hz
IRRPW 45 usec
IRATN 511
DFILE CF3-Diyne (PhMe)-H+Phenylacet.
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 24
LKPHS 250
LKSIG 1303
CSPED 11 Hz
FILDC
FILDF

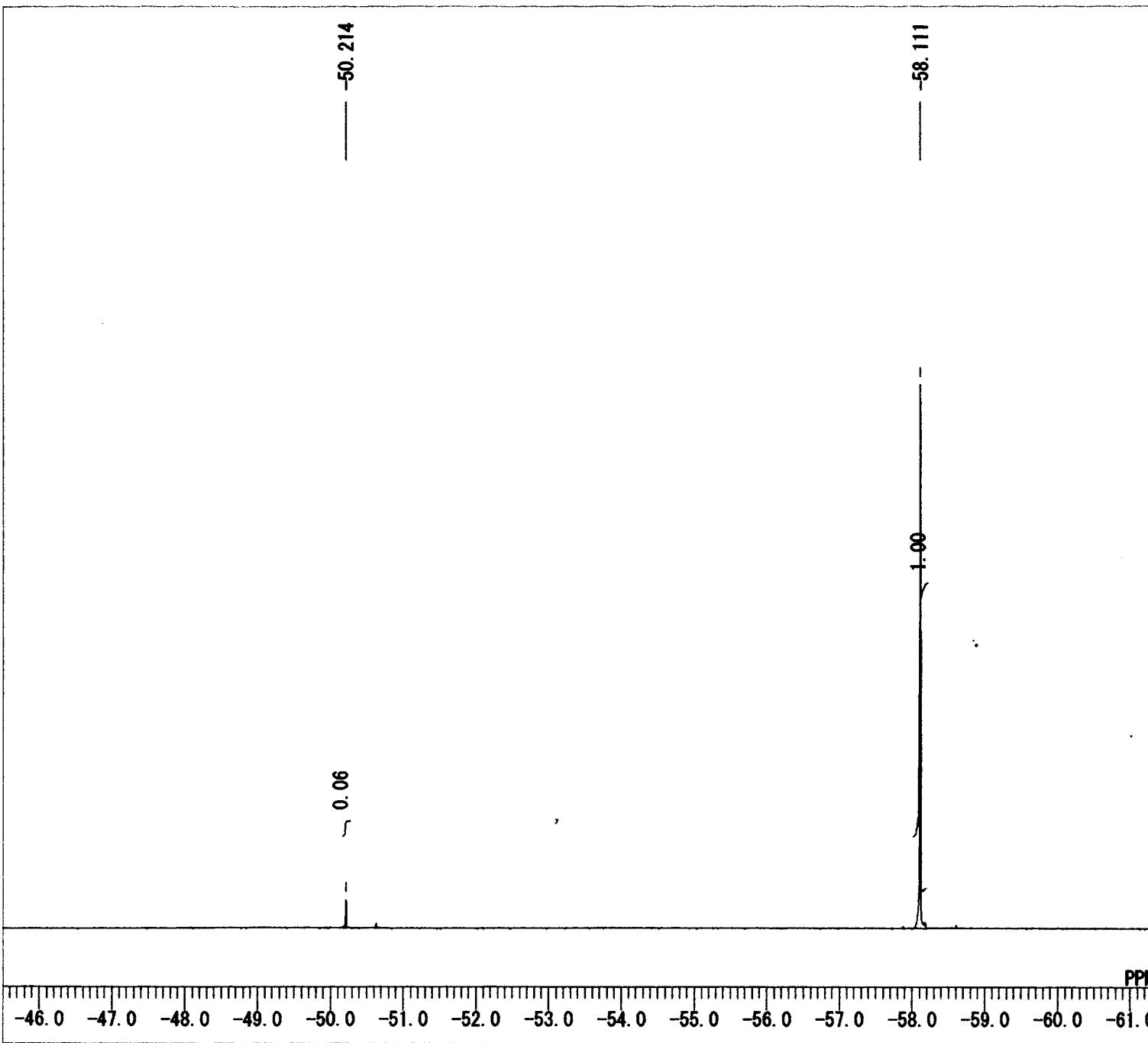
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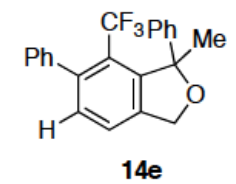
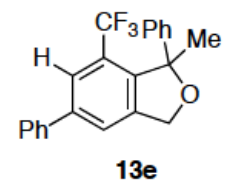
MENUF 13C
 OBNUC 13C
 OFR 100.40 MHz
 OBSET 125.00 KHz
 OBFIN 10500.00 Hz
 PW1 6.00 usec
 DEADT 19.10 usec
 PREDL 0.20000 msec
 IWT 1.0000 msec
 POINT 32768
 SPO 32768
 TIMES 100000000
 DUMMY 1
 FREQU 27118.64 Hz
 FLT 13550 Hz
 DELAY 14.80 usec
 ACQTM 1.2083 sec
 PD 1.7920 sec
 ADBIT 16
 RGA IN 25
 BF 0.10 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD BCM
 EXPCM Bilevel. complete. decoupling: {
 IRNUC 1H
 IFR 399.65 MHz
 IRSET 124.00 KHz
 IRFIN 10500.00 Hz
 IRRPW 45 usec
 IRATN 511
 DFILE CF3-Diyne (PhMe)-H+Phenylacet;
 SF TH5ATFG2
 LKSET 61.60 KHz
 LKFIN 79.0 Hz
 LKLEV 180
 LGAIN 25
 LKPHS 250
 LKSIG 1539
 CSPED 12 Hz
 FILDC
 FILDf

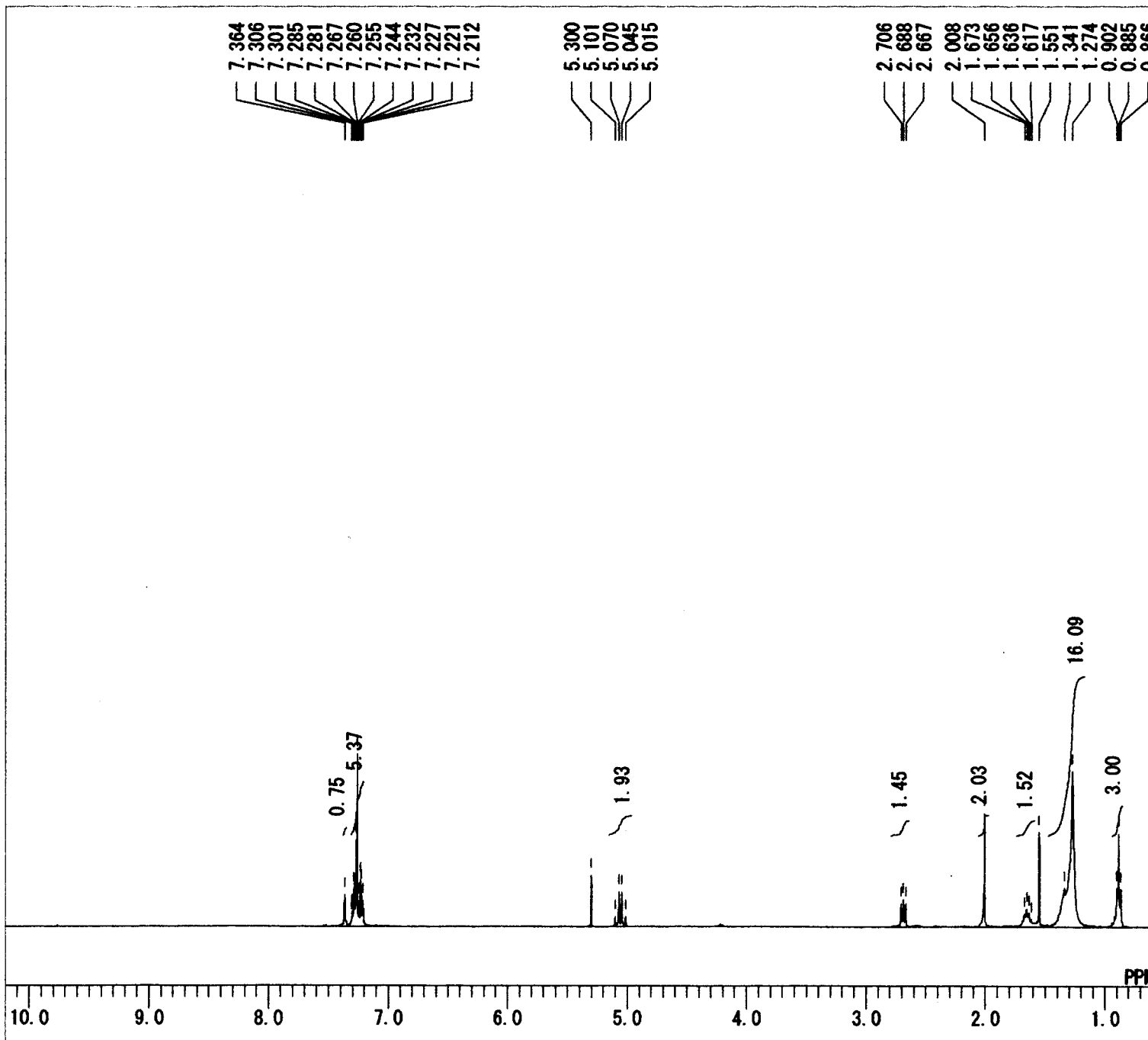




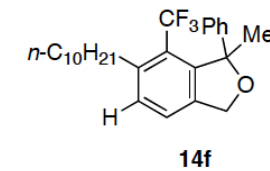
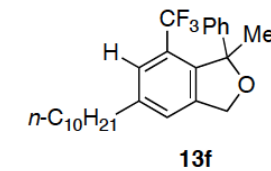
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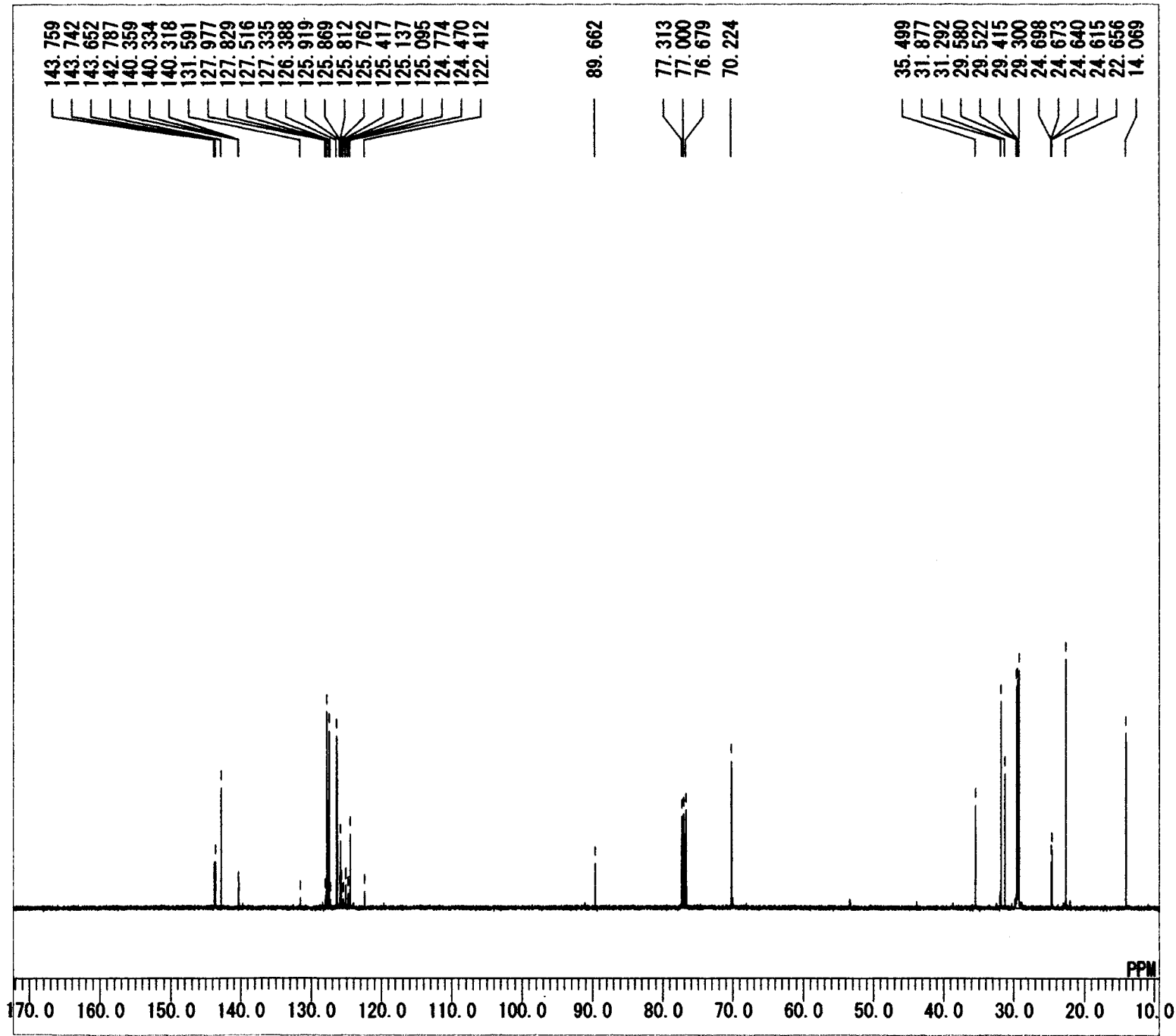
MENUF 19F
OBNUC 19F
OFR 376.05 MHz
OBSET 139.60 KHz
OBFIN 36.10 Hz
PW1 6.00 usec
DEADT 10.00 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 32768
SPO 32768
TIMES 16
DUMMY 1
FREQU 80000.00 Hz
FLT 40000 Hz
DELAY 5.00 usec
ACQTM 0.4096 sec
PD 4.9500 sec
ADBIT 16
RGAIN 14
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM.
IRNUC 1H
IFR 399.65 MHz
IRSET 124.00 KHz
IRFIN 10500.00 Hz
IRRPW 45 usec
IRATN 511
DFILE CF3-Diyne (PhMe)-H+Phenylacet
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 24
LKPHS 250
LKSIG 1304
CSPED 12 Hz
FILDC
FILDF
  
```





MENUF 1H
 OBNUC 1H
 OFR 399.65 MHz
 OBSET 135.40 KHz
 OBFIN 24.90 Hz
 PW1 5.80 usec
 DEADT 72.10 usec
 PREDL 0.20000 msec
 IWT 1.0000 msec
 POINT 16384
 SPO 16384
 TIMES 8
 DUMMY 1
 FREQU 7992.01 Hz
 FLT 4000 Hz
 DELAY 50.00 usec
 ACQTM 2.0501 sec
 PD 4.9500 sec
 ADBIT 16
 RGAIN 17
 BF 0.10 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD NON
 EXPCM NON:Single.coupled:PW1_ACQTM
 IRNUC 1H
 IFR 399.65 MHz
 IRSET 136.90 KHz
 IRFIN 97.50 Hz
 IRRPW 45 usec
 IRATN 511
 DFIL CF3-Diyne (PhMe)-H+1-Decyne-(
 SF TH5ATFG2
 LKSET 61.60 KHz
 LKFIN 79.0 Hz
 LKLEV 180
 LGAIN 26
 LKPHS 250
 LKSIG 2254
 CSPED 14 Hz
 FILDC
 FILDF

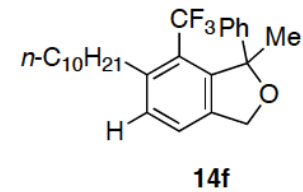
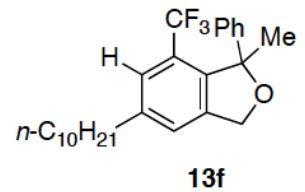


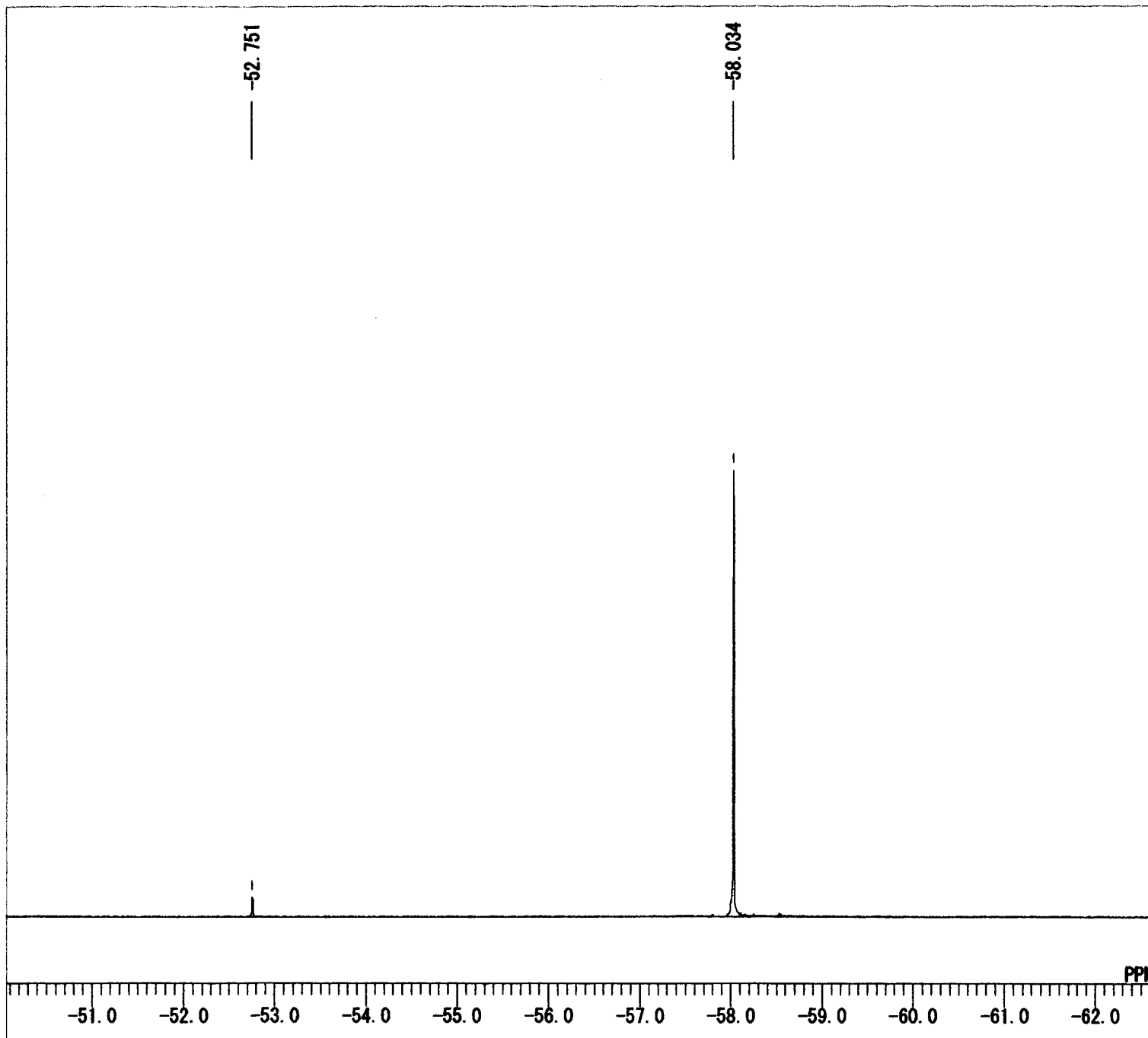


```

MENUF 13C
OBNUC 13C
OFR 100.40 MHz
OBSET 125.00 KHz
OBF IN 10500.00 Hz
PW1 6.00 usec
DEADT 19.10 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 32768
SPO 32768
TIMES 512
DUMMY 1
FREQU 27118.64 Hz
FLT 13550 Hz
DELAY 14.80 usec
ACQTM 1.2083 sec
PD 1.7920 sec
ADBIT 16
RGAIN 25
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD BCM
EXPCM Bi-level, complete, decoupling:
IRNUC 1H
IFR 399.65 MHz
IRSET 124.00 KHz
IRF IN 10500.00 Hz
IRRPW 45 usec
IRATN 511
DFILE CF3-Diyne (PhMe)-H+1-Decyne-(
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGA IN 23
LKPHS 250
LKSIG 761
CSPED 14 Hz
FILDC
FILDF

```

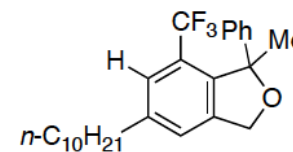




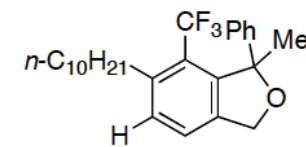
```

MENUF 19F
OBNUC 19F
OFR 376.05 MHz
OBSET 139.60 KHz
OBF IN 36.10 Hz
PW1 6.00 usec
DEADT 10.00 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 32768
SPO 32768
TIMES 16
DUMMY 1
FREQU 80000.00 Hz
FLT 40000 Hz
DELAY 5.00 usec
ACQTM 0.4096 sec
PD 4.9500 sec
ADBIT 16
RGAIN 14
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM.
IRNUC 1H
IFR 399.65 MHz
IRSET 124.00 KHz
IRFIN 10500.00 Hz
IRRPW 45 usec
IRATN 511
DFILE CF3-Diyne (PhMe)-H+1-Decyne-(
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 26
LKPHS 250
LKSIG 2257
CSPED 13 Hz
FILDC
FILDF

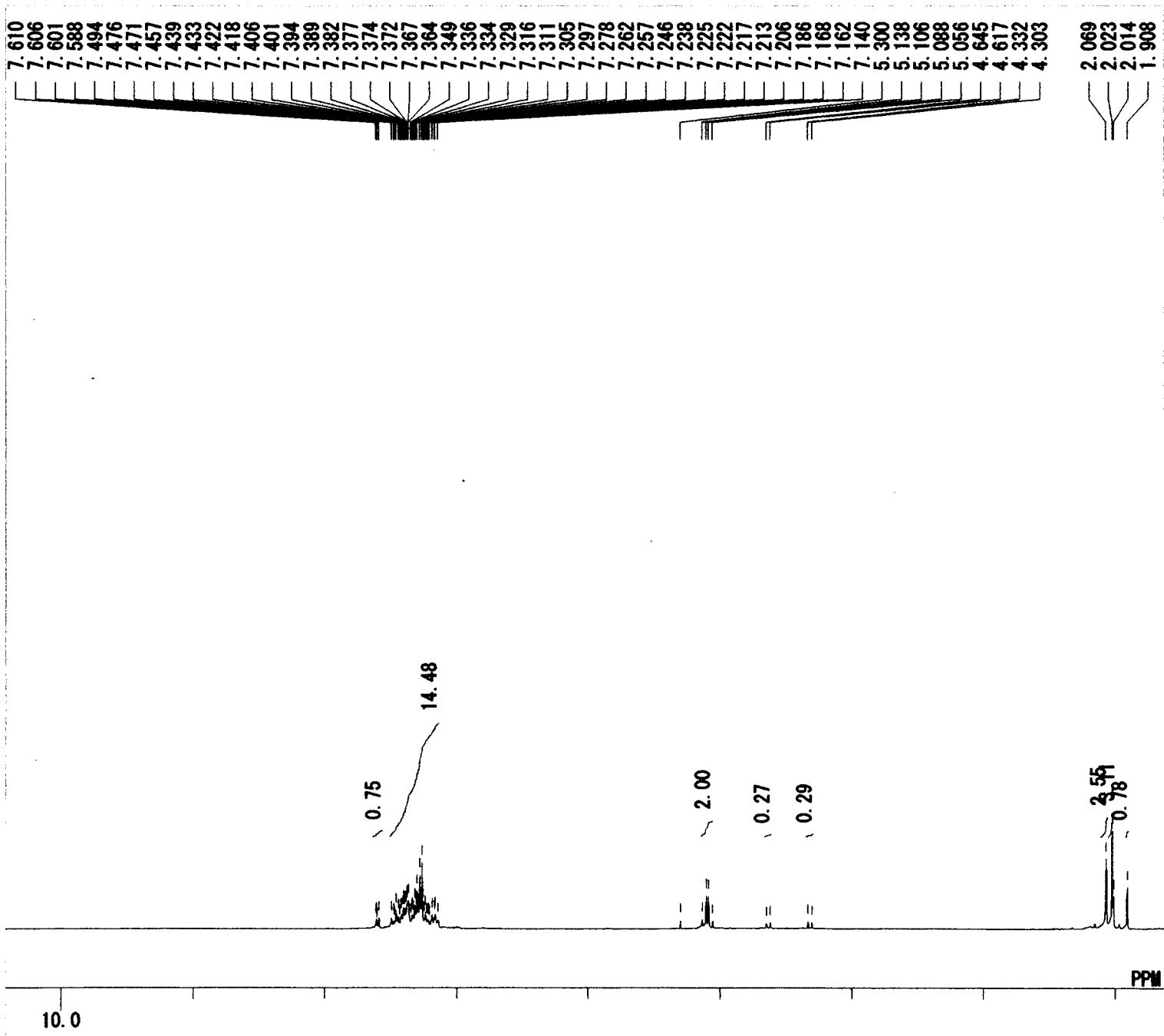
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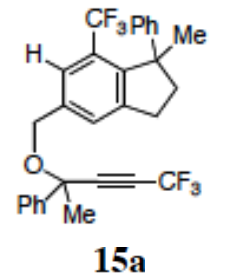
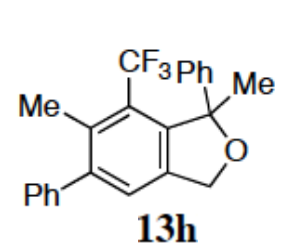
13f

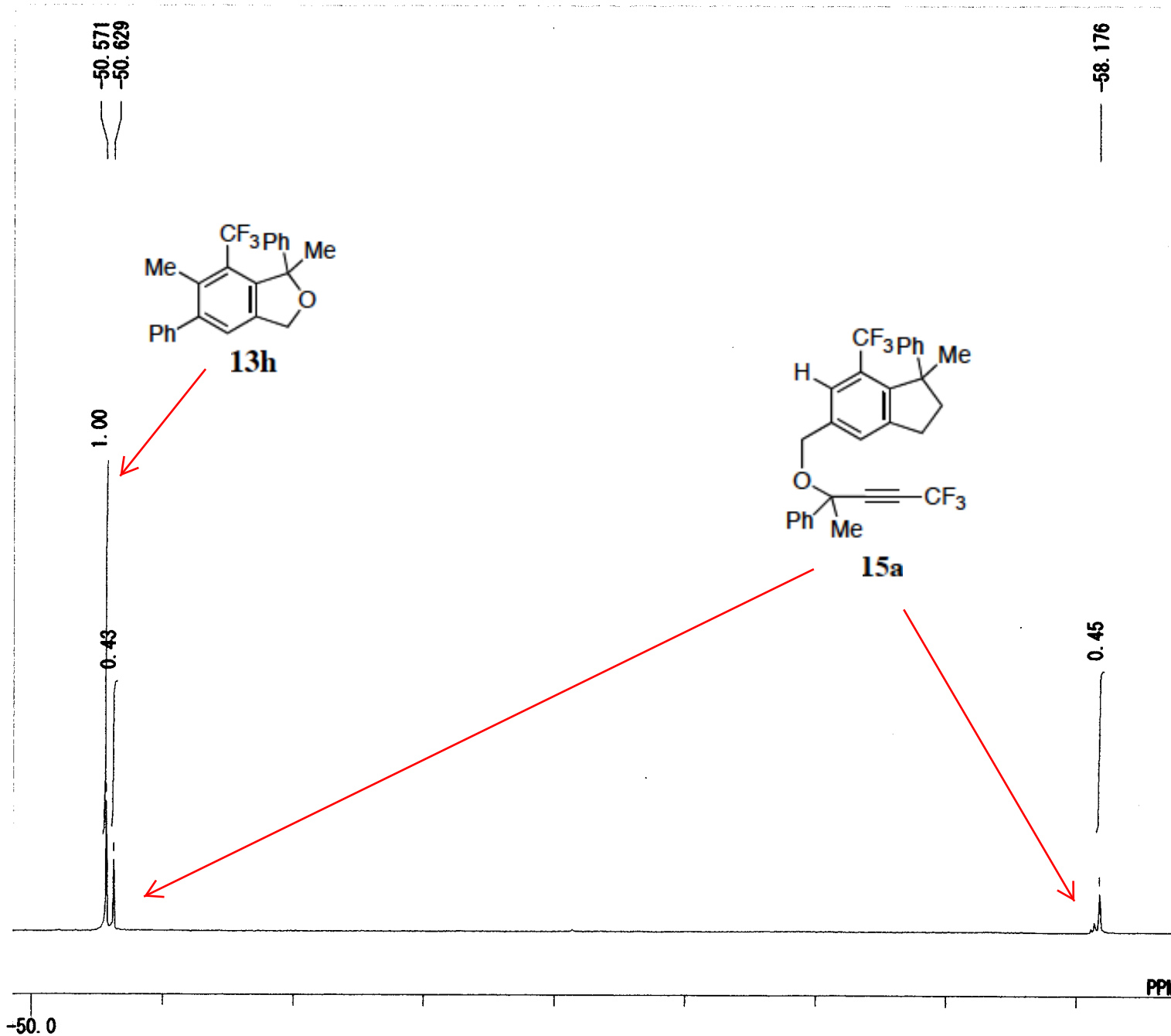


14f

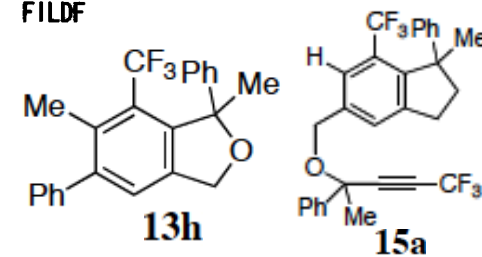


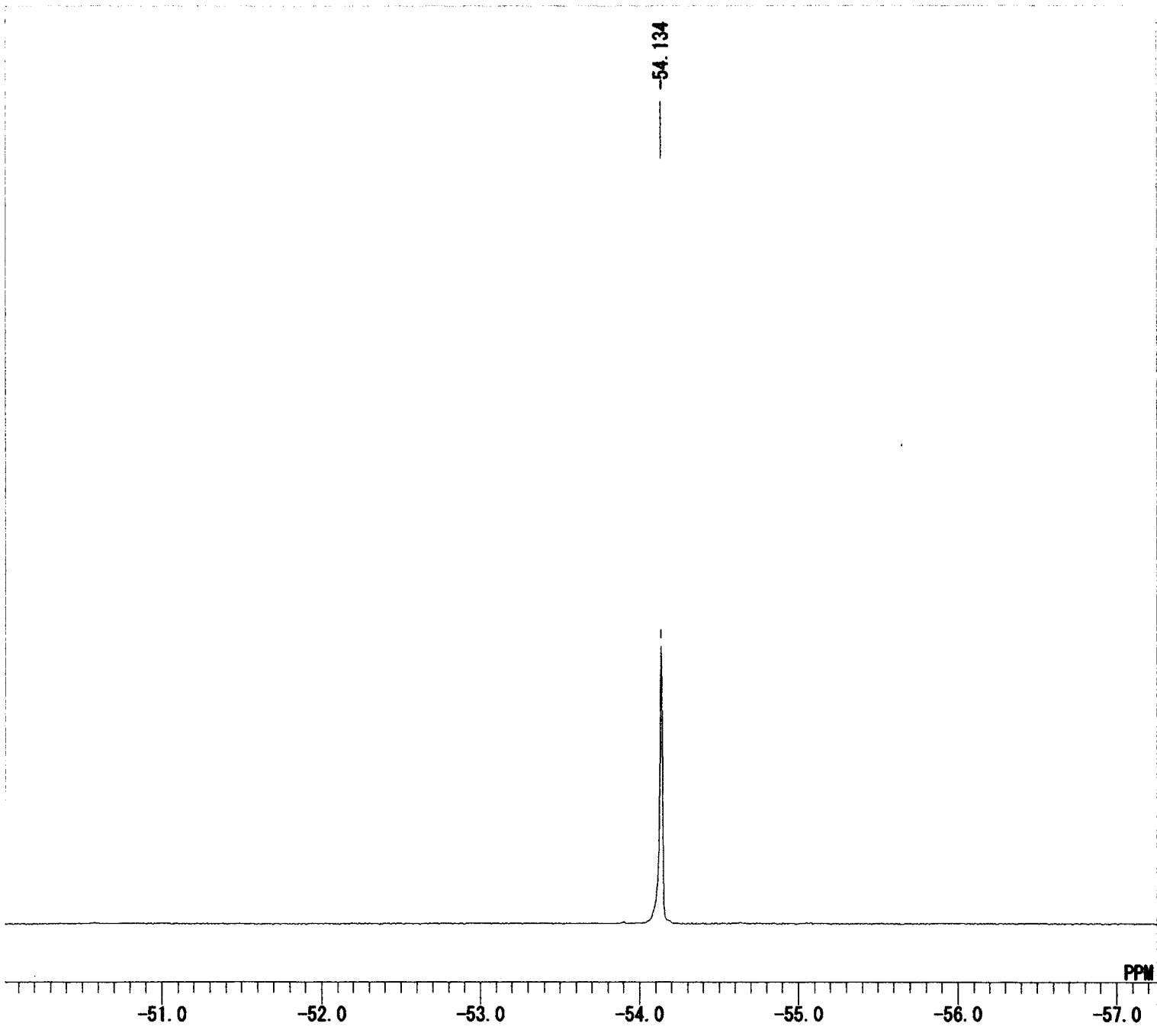
MENUF	1H
OBNUC	1H
OFR	399.65 MHz
OBSET	135.40 KHz
OBFIN	24.90 Hz
PW1	5.80 usec
DEADT	72.10 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7992.01 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	2.0501 sec
PD	4.9500 sec
ADBIT	16
RGAIN	17
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM
IRNUC	1H
IFR	399.65 MHz
IRSET	136.90 KHz
IRFIN	97.50 Hz
IRRPW	45 usec
IRATN	511
DFILE	product-diyne-PhMe-alkyne-Ph
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	22
LKPHS	250
LKSIG	610
CSPED	11 Hz
FILDC	
FILDF	





MENUF 19F
 OBNUC 19F
 OFR 376.05 MHz
 OBSET 139.60 KHz
 OBFIN 36.10 Hz
 PW1 6.00 usec
 DEADT 10.00 usec
 PREDL 0.20000 msec
 IWT 1.0000 msec
 POINT 32768
 SPO 32768
 TIMES 16
 DUMMY 1
 FREQU 80000.00 Hz
 FLT 40000 Hz
 DELAY 5.00 usec
 ACQTM 0.4096 sec
 PD 4.9500 sec
 ADBIT 16
 RGAIN 14
 BF 0.10 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD NON
 EXPCM NON:Single.coupled:PW1_ACQTM.
 IRNUC 1H
 IFR 399.65 MHz
 IRSET 124.00 KHz
 IRFIN 10500.00 Hz
 IRRPW 45 usec
 IRATN 511
 DFILE product-diyne-PhMe-alkyne-Phl
 SF TH5ATFG2
 LKSET 61.60 KHz
 LKFIN 79.0 Hz
 LKLEV 180
 LGAIN 22
 LKPHS 250
 LKSIG 604
 CSPED 11 Hz
 FILDC
 FILDF

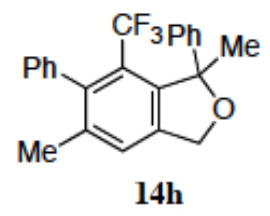




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MENUM 19F
OBNUC 19F
OFR 376.05 MHz
OBSET 139.60 KHz
OBF IN 36.10 Hz
PW1 6.00 usec
DEADT 10.00 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 32768
SPO 32768
TIMES 16
DUMMY 1
FREQU 80000.00 Hz
FLT 40000 Hz
DELAY 5.00 usec
ACQTM 0.4096 sec
PD 4.9500 sec
ADBIT 16
RGAIN 14
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM
IRNUC 1H
IFR 399.65 MHz
IRSET 124.00 KHz
IRF IN 10500.00 Hz
IRRPW 45 usec
IRATN 511
DFILE product-diyne-PhMe-alkyne-Ph
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 22
LKPHS 250
LKSIG 583
CSPED 13 Hz
FILDC
FILDF

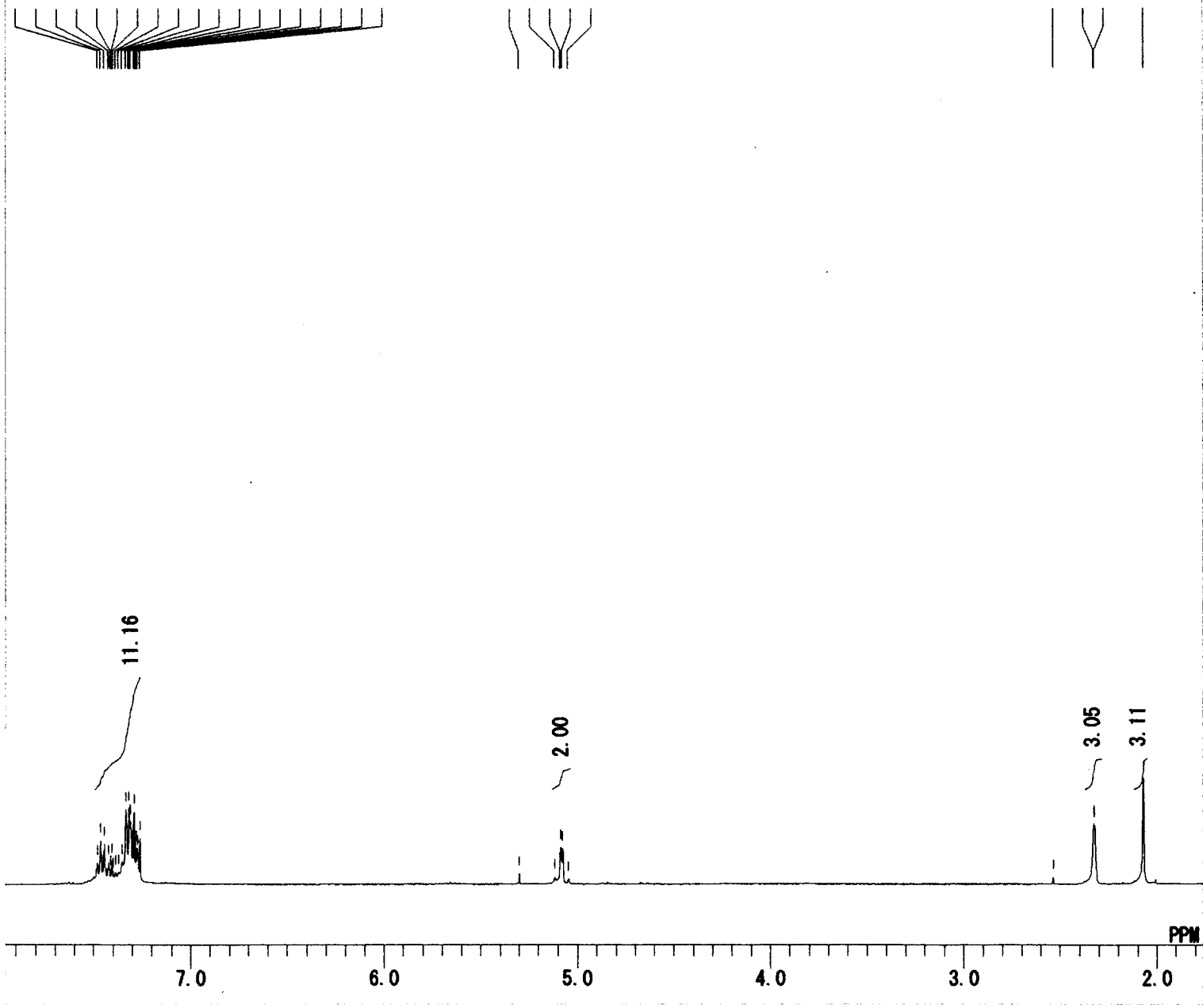
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7.481
7.465
7.445
7.422
7.411
7.405
7.398
7.387
7.371
7.353
7.333
7.317
7.312
7.309
7.292
7.283
7.277
7.272
7.260

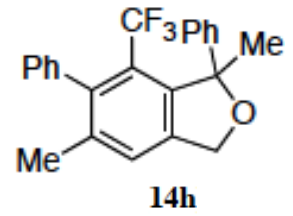
5.299
5.116
5.086
5.076
5.045

2.537
2.328
2.323
2.072

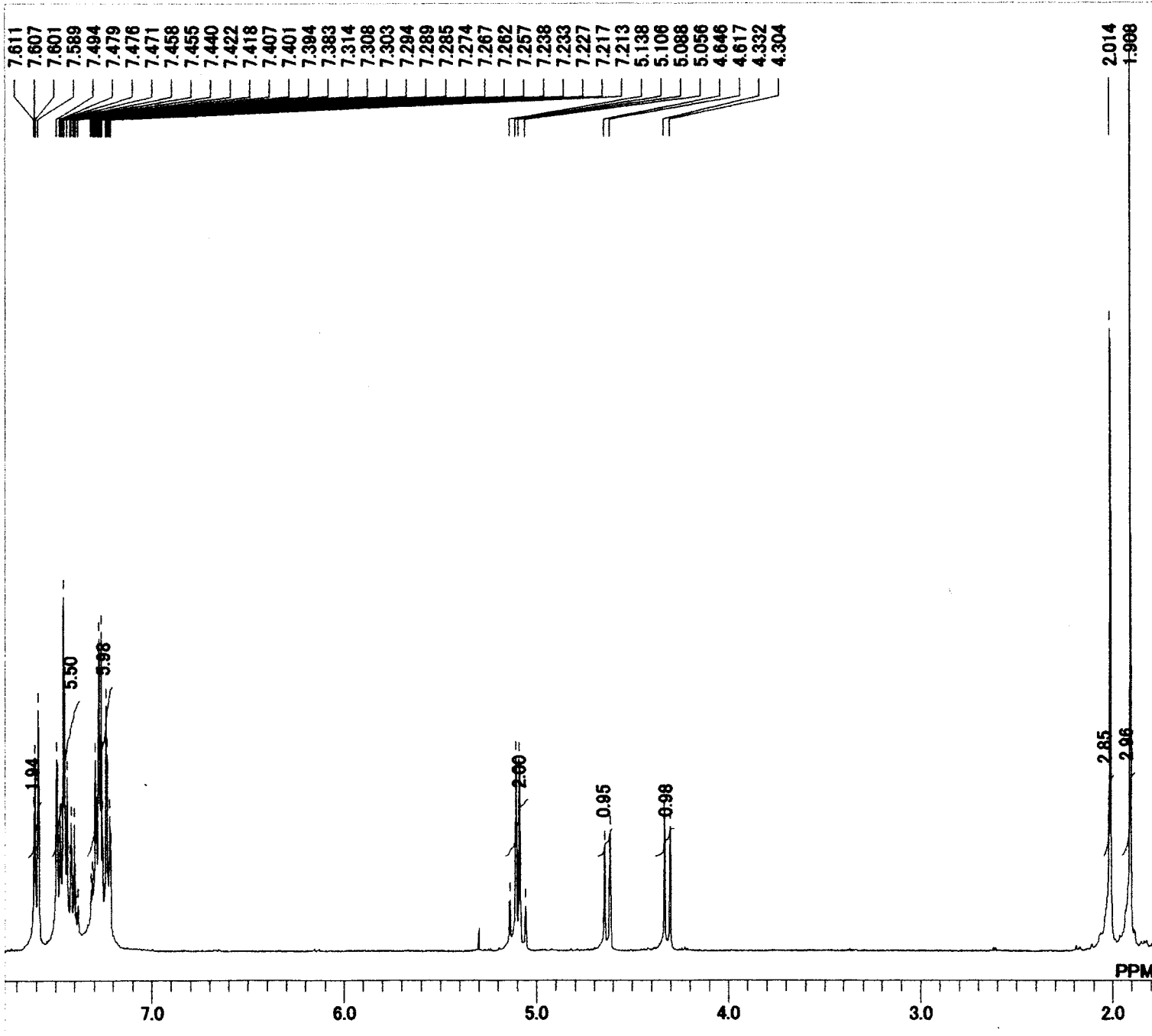


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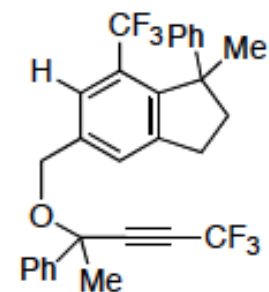
MENUF 1H
OBNUC 1H
OFR 399.65 MHz
OBSET 135.40 KHz
OBF IN 24.90 Hz
PW1 5.80 usec
DEADT 72.10 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7992.01 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 2.0501 sec
PD 4.9500 sec
ADBIT 16
RGAIN 16
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM
IRNUC 1H
IFR 399.65 MHz
IRSET 136.90 KHz
IRFIN 97.50 Hz
IRRPW 45 usec
IRATN 511
DFILE product-diyne-PhMe-alkyne-Ph
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 22
LKPHS 250
LKSIG 585
CSPED 13 Hz
FILDC
FILDF
  
```



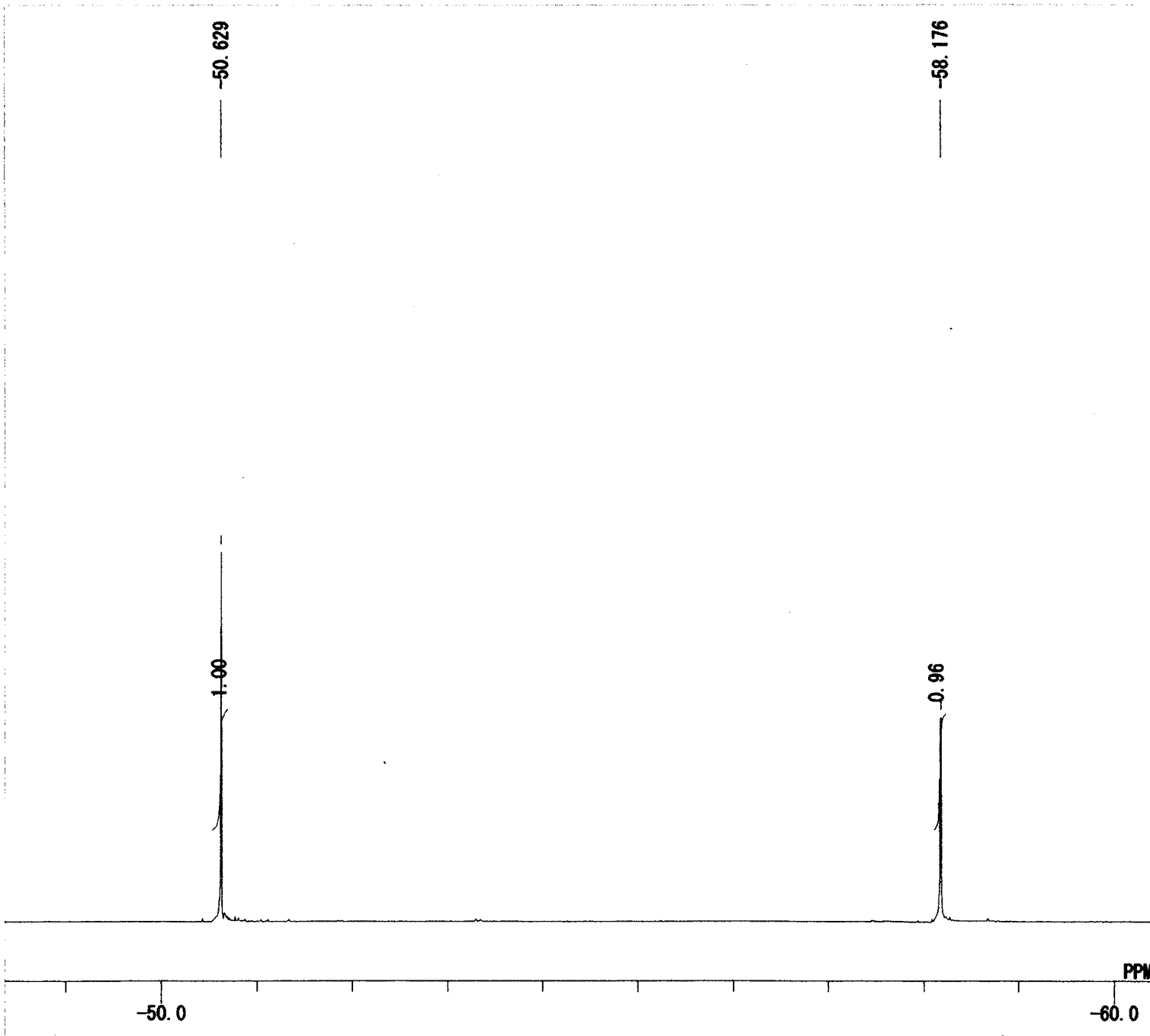
product-diyne-PhMe-MeCN(1H)



MENUF 1H
 OBNUC 1H
 OFR 399.65 MHz
 OBSSET 135.40 KHz
 OBFIN 24.90 Hz
 PW1 5.80 usec
 DEADT 72.10 usec
 PREDL 0.20000 msec
 IWT 1.0000 msec
 POINT 16384
 SPO 16384
 TIMES 8
 DUMMY 1
 FREQU 7992.01 Hz
 FLT 4000 Hz
 DELAY 50.00 usec
 ACQTM 2.0501 sec
 PD 4.9500 sec
 ADBIT 16
 RGAIN 16
 BF 0.10 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD NON
 EXPCM NON:Single.coupled:PW1_ACQTM_PE
 IRNUC 1H
 IFR 399.65 MHz
 IRSET 136.90 KHz
 IRFIN 97.50 Hz
 IRRPW 45 usec
 IRATN 511
 DFILE _DEFAULT.ALS
 SF TH5ATFG2
 LKSET 61.60 KHz
 LKFIN 79.0 Hz
 LKLEV 180
 LGAIN 22
 LKPHS 250
 LKSIG 648
 CSPED 11 Hz
 FILDC
 FILDF



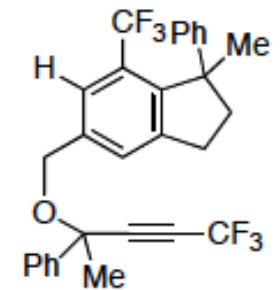
15a



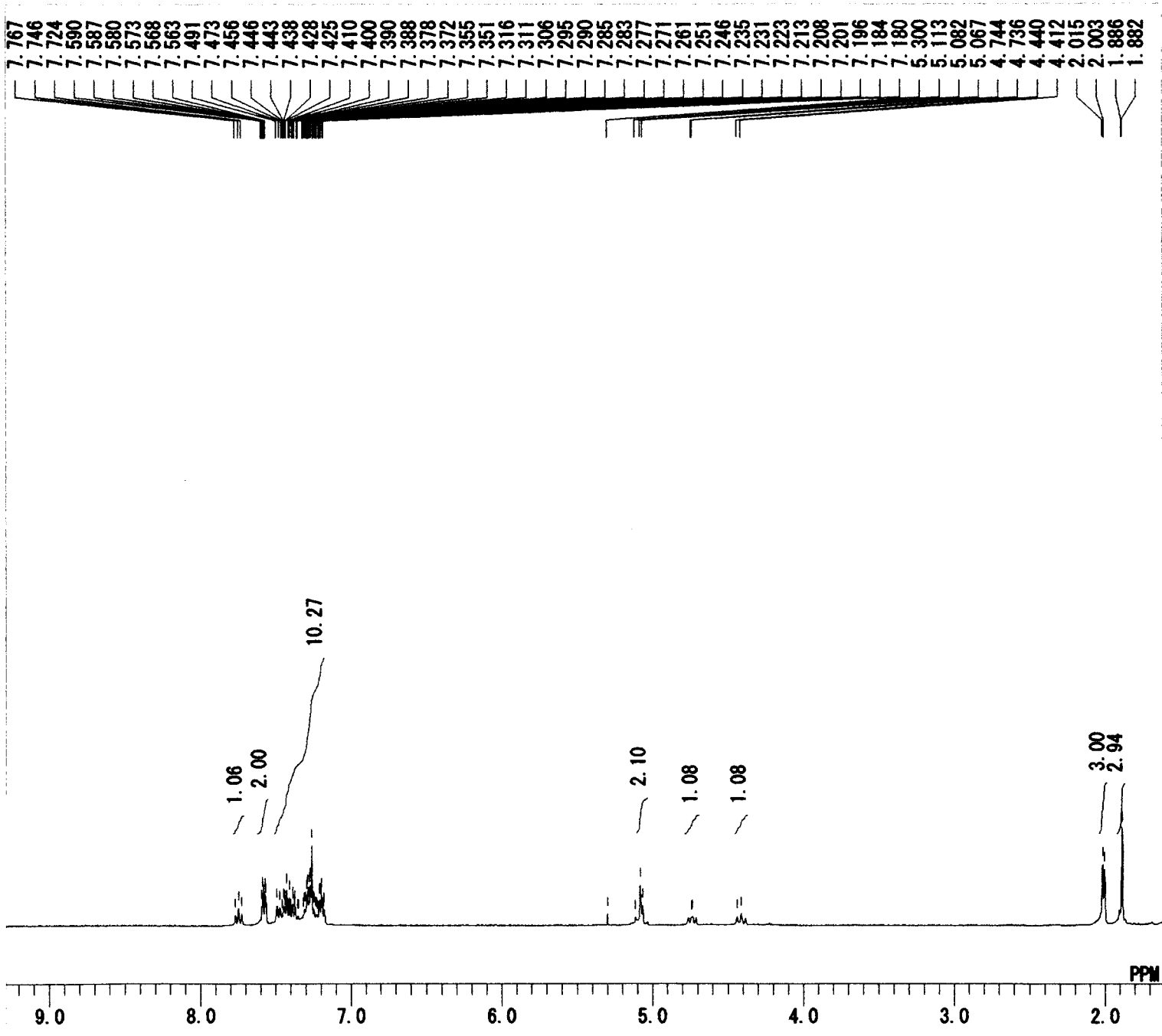
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MENUF 19F
OBNUC 19F
OFR 376.05 MHz
OBSET 139.60 KHz
OBFIN 36.10 Hz
PW1 6.00 usec
DEADT 10.00 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 32768
SPO 32768
TIMES 16
DUMMY 1
FREQU 80000.00 Hz
FLT 40000 Hz
DELAY 5.00 usec
ACQTM 0.4096 sec
PD 4.9500 sec
ADBIT 16
RGAIN 14
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single. coupled:PW1_ACQTM
IRNUC 1H
IFR 399.65 MHz
IRSET 124.00 KHz
IRFIN 10500.00 Hz
IRRPW 45 usec
IRATN 511
DFILE product-diyne-PhMe-MeCN(19F).
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 22
LKPHS 250
LKSIG 646
CSPED 11 Hz
FILDC
FILDF

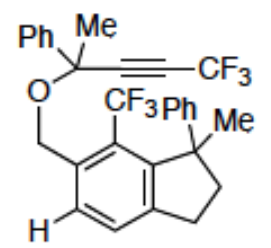
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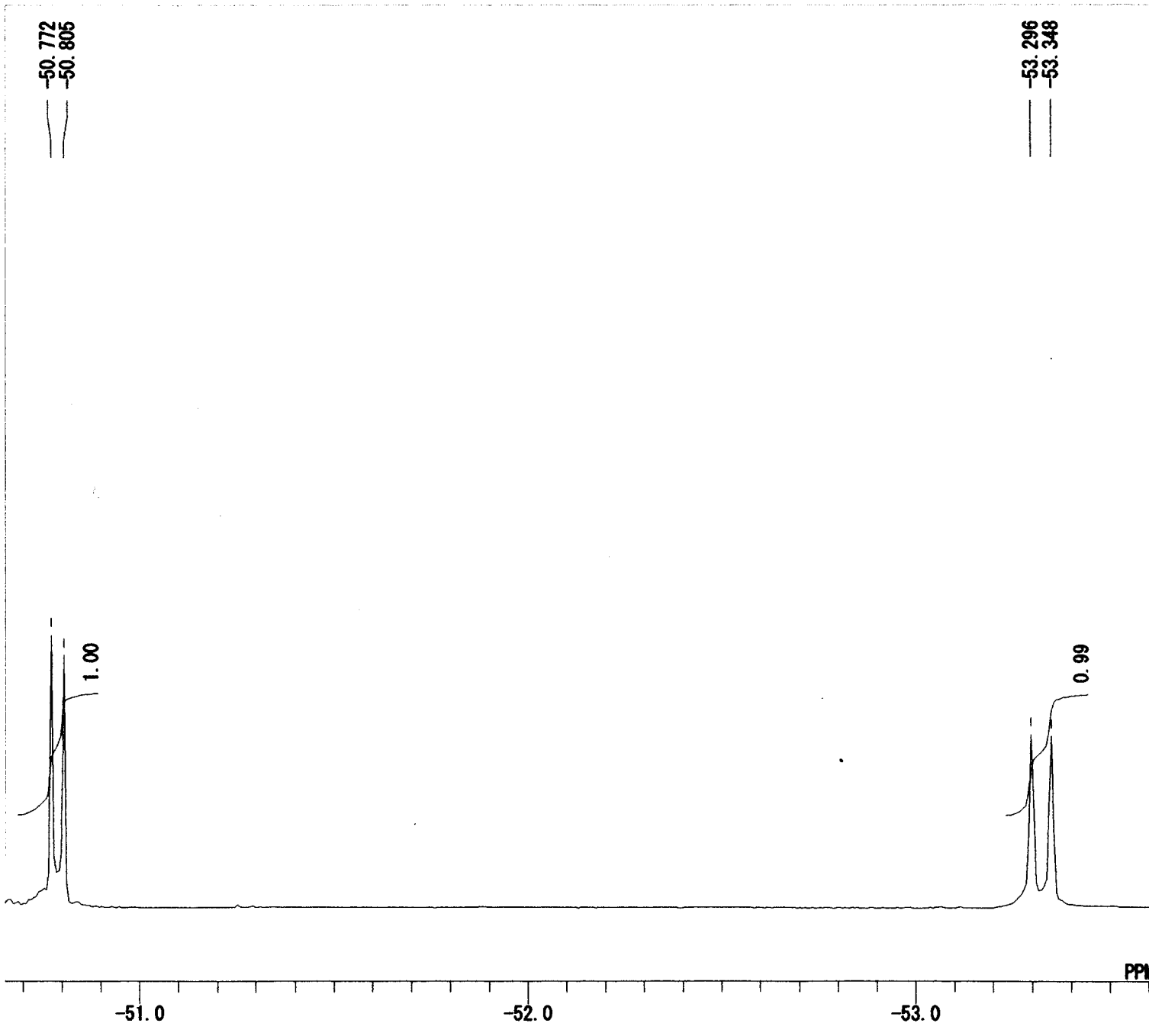
15a



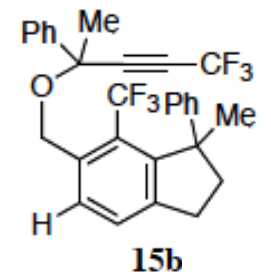
MENUF	1H
OBNUC	1H
OFR	399.65 MHz
OBSET	135.40 KHz
OBFIN	24.90 Hz
PW1	5.80 usec
DEADT	72.10 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7992.01 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	2.0501 sec
PD	4.9500 sec
ADBIT	16
RGAIN	17
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM
IRNUC	1H
IFR	399.65 MHz
IRSET	136.90 KHz
IRFIN	97.50 Hz
IRRPW	45 usec
IRATN	511
DFILE	byproduct-diyne-PhMe-MeCN (1H)
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	23
LKPHS	250
LKSIG	838
CSPED	11 Hz
FILDC	
FILDF	

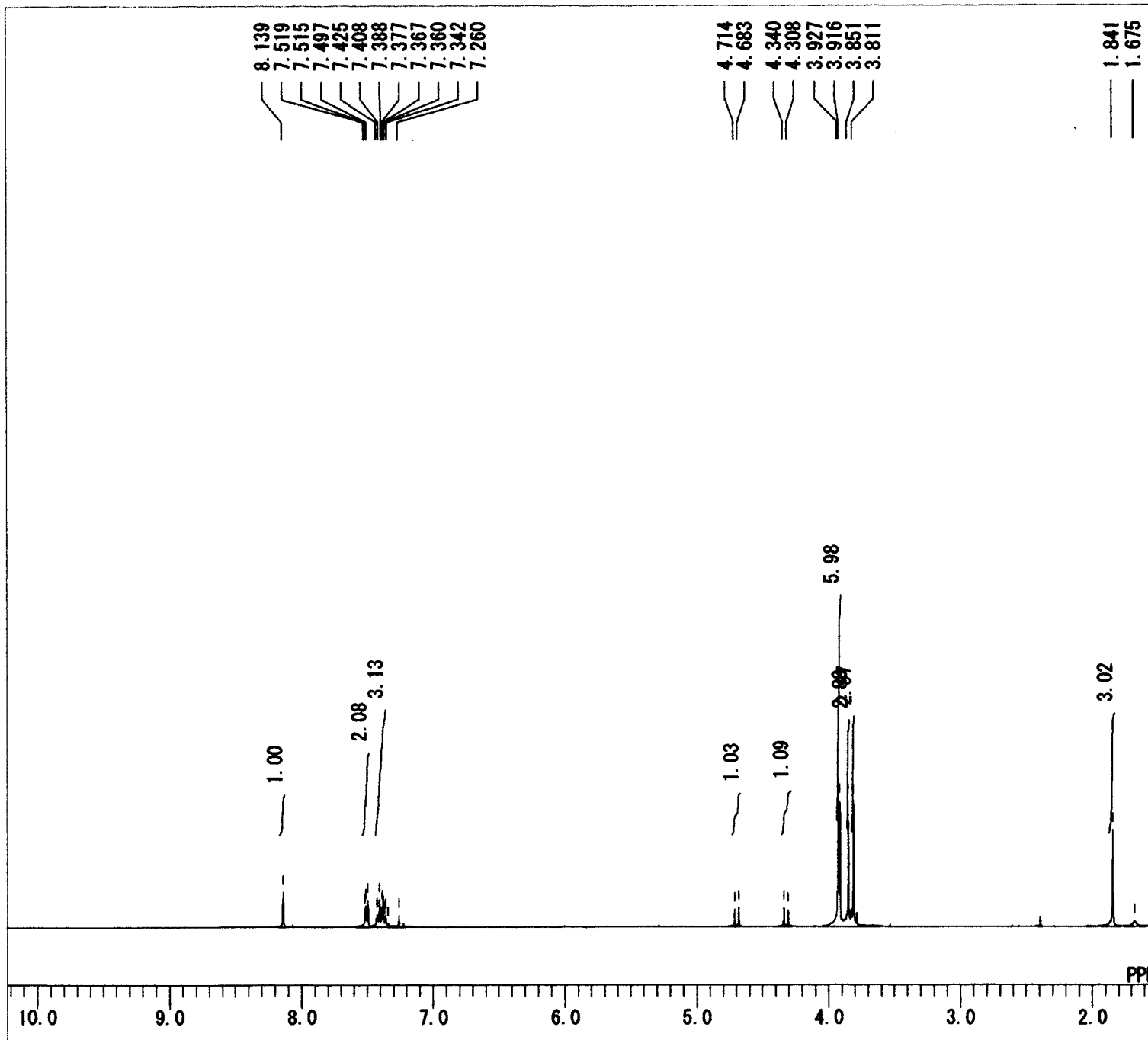


15b

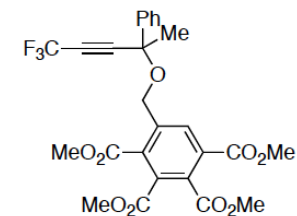


MENUF	19F
OBNUC	19F
OFR	376.05 MHz
OBSET	139.60 KHz
OBFIN	36.10 Hz
PW1	6.00 usec
DEADT	10.00 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	16
DUMMY	1
FREQU	80000.00 Hz
FLT	40000 Hz
DELAY	5.00 usec
ACQTM	0.4096 sec
PD	4.9500 sec
ADBIT	16
RGAIN	14
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON: Single. coupled: PW1_ACQTM
IRNUC	1H
IFR	399.65 MHz
IRSET	124.00 KHz
IRFIN	10500.00 Hz
IRRPW	45 usec
IRATN	511
DFILE	byproduct-diyne-PhMe-MeCN (19f
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGA1N	23
LKPHS	250
LKSIG	834
CSPED	11 Hz
FILDG	
FILDF	

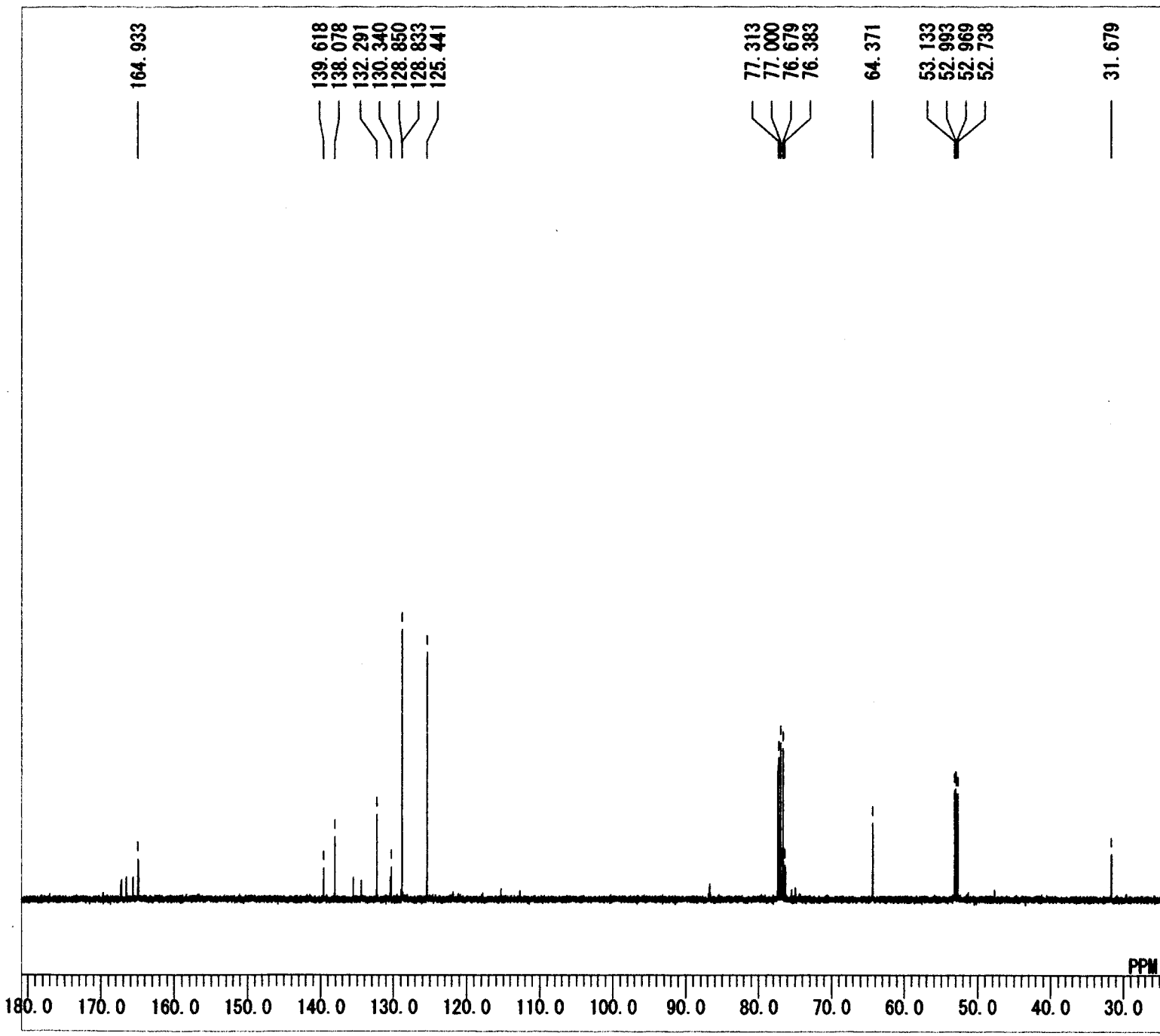




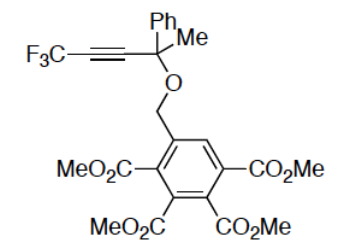
MENUF 1H
 OBNUC 1H
 OFR 399.65 MHz
 OBSET 135.40 KHz
 OBFIN 24.90 Hz
 PW1 5.80 usec
 DEADT 72.10 usec
 PREDL 0.20000 msec
 IWT 1.0000 msec
 POINT 16384
 SPO 16384
 TIMES 8
 DUMMY 1
 FREQU 7992.01 Hz
 FLT 4000 Hz
 DELAY 50.00 usec
 ACQTM 2.0501 sec
 PD 4.9500 sec
 ADBIT 16
 RGA IN 13
 BF 0.10 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD NON
 EXPCM NON:Single.coupled:PW1_ACQTM.
 IRNUC 1H
 IFR 399.65 MHz
 IRSET 136.90 KHz
 IRFIN 97.50 Hz
 IRRPW 45 usec
 IRATN 511
 DFILE CF3-Diyne (PhMe)-H+2Di (CO2Me) /
 SF TH5ATFG2
 LKSET 61.60 KHz
 LKFIN 79.0 Hz
 LKLEV 180
 LGAIN 24
 LKPHS 250
 LKSIG 935
 CSPED 14 Hz
 FILDC
 FILDF



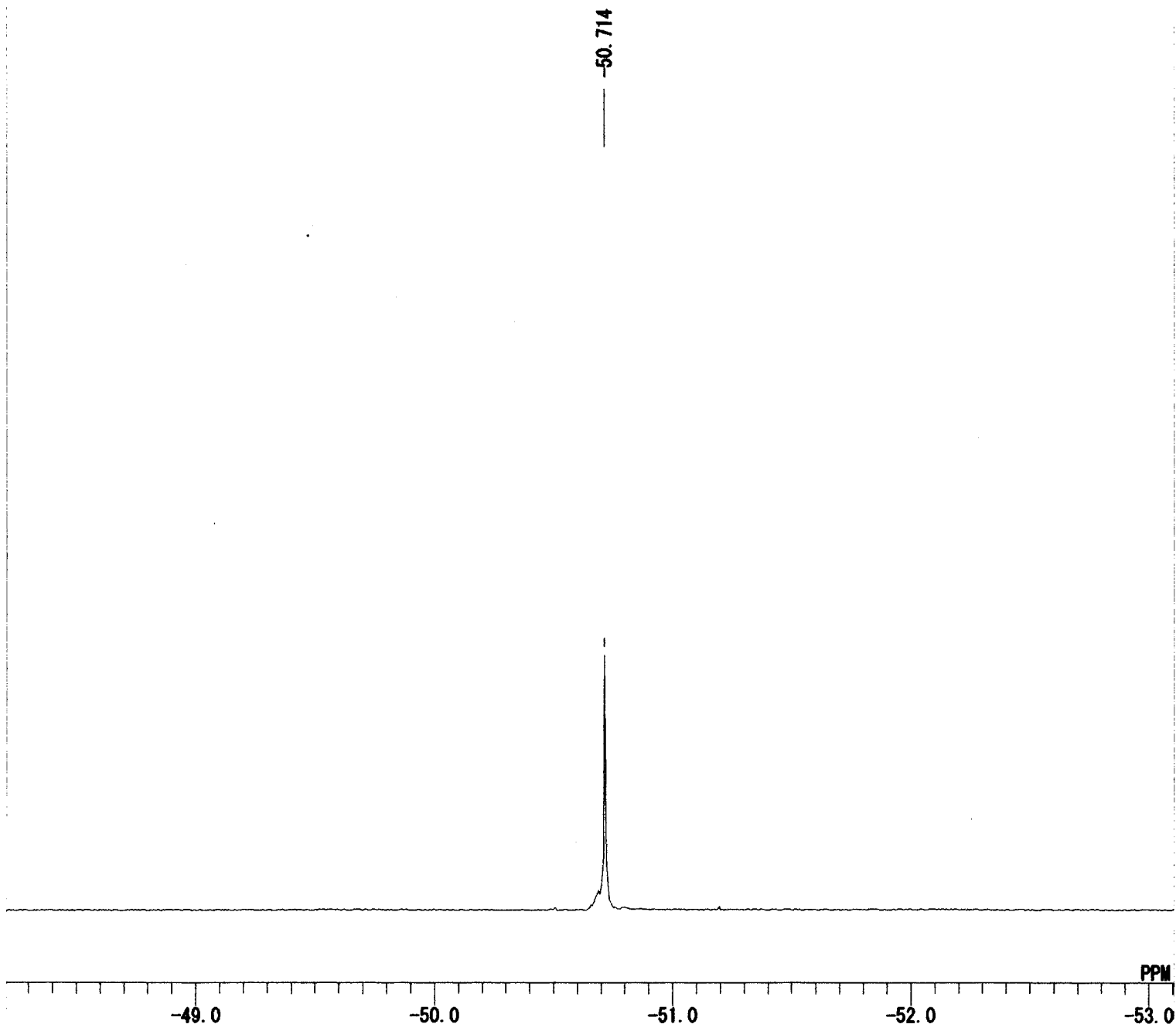
16



MENUF	13C
OBNUC	13C
OFR	100.40 MHz
OBSET	125.00 KHz
OBFIN	10500.00 Hz
PW1	6.00 usec
DEADT	19.10 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	512
DUMMY	1
FREQU	27118.64 Hz
FLT	13550 Hz
DELAY	14.80 usec
ACQTM	1.2083 sec
PD	1.7920 sec
ADBIT	16
RGAIN	25
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	BCM
EXPCM	Bi level. complete. decoupling: 3
IRNUC	1H
IFR	399.65 MHz
IRSET	124.00 KHz
IRFIN	10500.00 Hz
IRRPW	45 usec
IRATN	511
DFILE	CF3-Diyne(PhMe)-H+2Di(CO2Me)/
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	24
LKPHS	250
LKSIG	958
CSPED	14 Hz
FILDC	
FILDF	

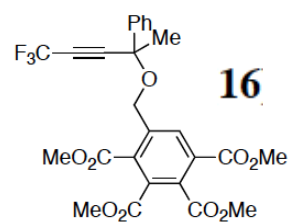


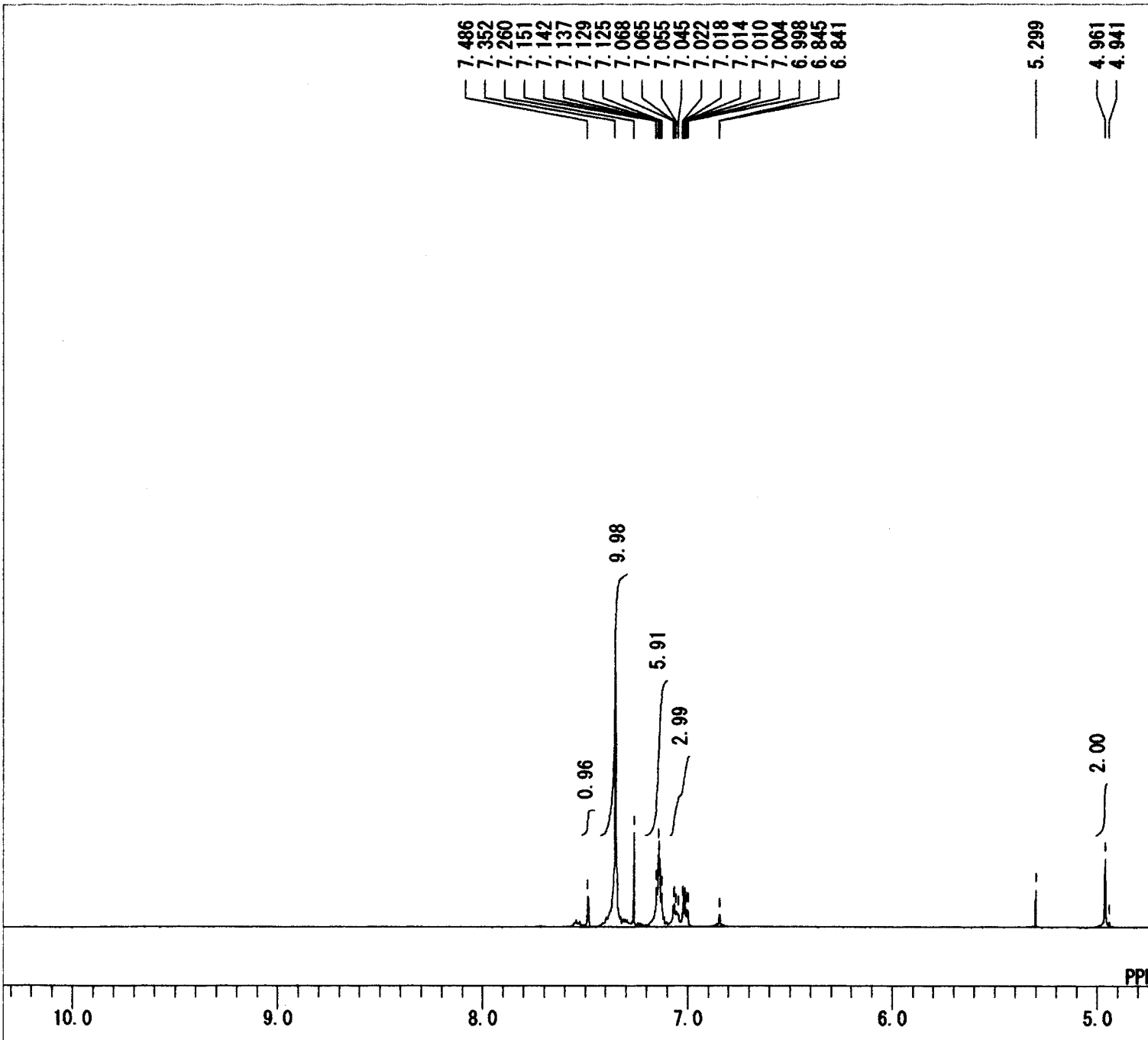
C:\WINNMR\DATA\Fluorine\Moriyasu\product-diyne-PhMe-di(CO2Me)-high-polor(19F).als



```

MENUF 19F
OBNUC 19F
OFR      376.05 MHz
OBSET    139.60 KHz
OBFIN    36.10 Hz
PW1      6.00 usec
DEADT    10.00 usec
PREDL    0.20000 msec
IWT      1.0000 msec
POINT    32768
SPO      32768
TIMES    16
DUMMY    1
FREQU    80000.00 Hz
FLT      40000 Hz
DELAY    5.00 usec
ACQTM    0.4096 sec
PD       4.9500 sec
ADBIT    16
RGAIN    14
BF       0.10 Hz
T1       0.00
T2       0.00
T3       90.00
T4       100.00
EXMOD    NON
EXPCM    NON:Single.coupled:PW1_ACQTM.
IRNUC    1H
IFR      399.65 MHz
IRSET    124.00 KHz
IRFIN    10500.00 Hz
IRRPW    45 usec
IRATN    511
DFILE    product-diyne-PhMe-di(CO2Me)-
SF        TH5ATFG2
LKSET    61.60 KHz
LKFIN    79.0 Hz
LKLEV    180
LGAIN    25
LKPHS    250
LKSIG    1701
CSPED    12 Hz
FILDC
FILDF
    
```

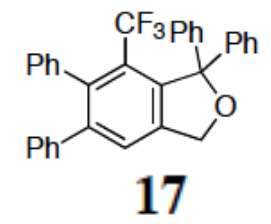


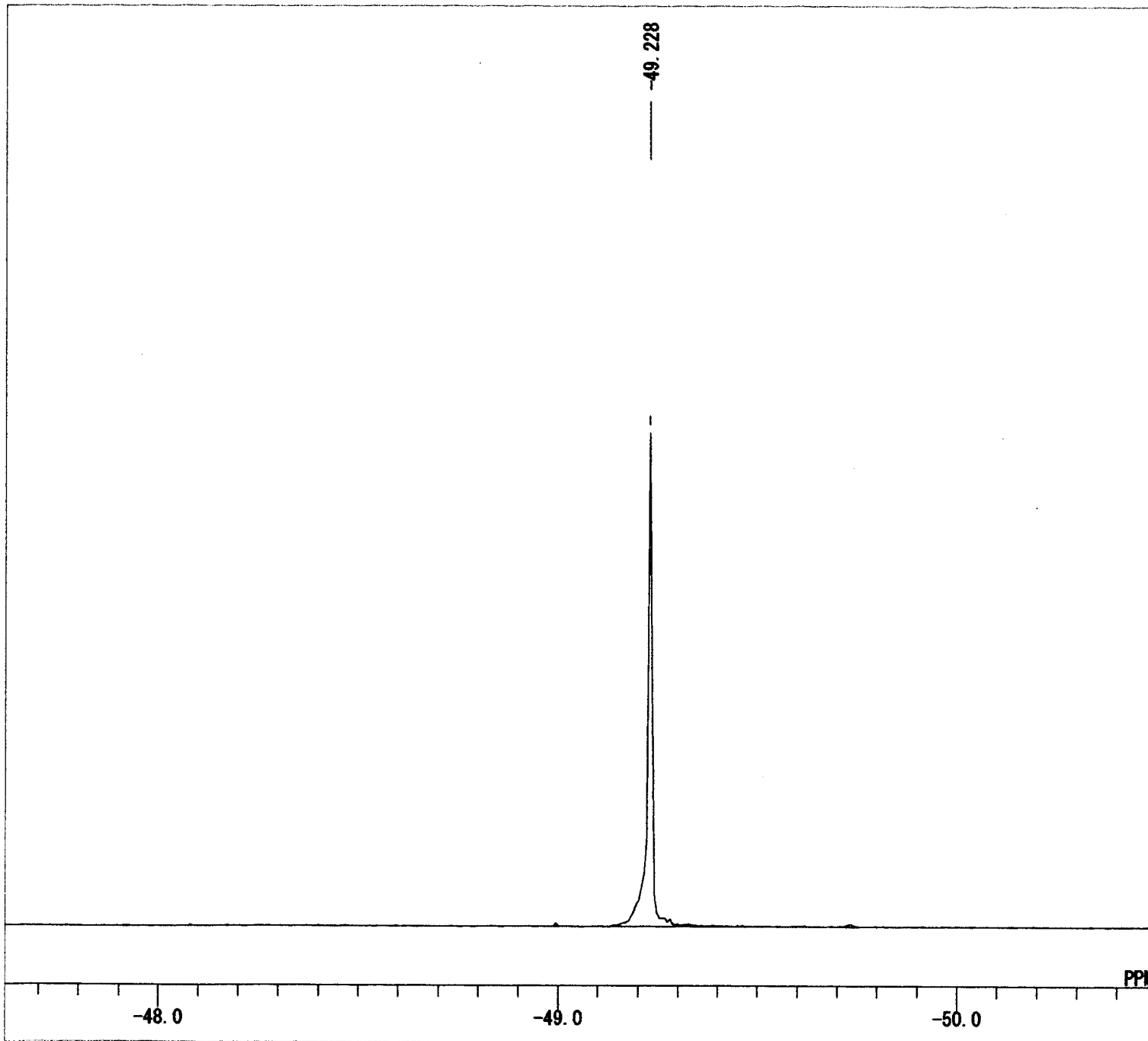


```

MENUF 1H
OBNUC 1H
OFR 399.65 MHz
OBSET 135.40 KHz
OBFIN 24.90 Hz
PW1 5.80 usec
DEADT 72.10 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7992.01 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 2.0501 sec
PD 4.9500 sec
ADBIT 16
RGAIN 19
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM
IRNUC 1H
IFR 399.65 MHz
IRSET 136.90 KHz
IRFIN 97.50 Hz
IRRPW 45 usec
IRATN 511
DFILE CF3-Diyne (PhPh)-H+diphenylacetone
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 24
LKPHS 250
LKSIG 1270
CSPED 13 Hz
FILDC
FILDF

```

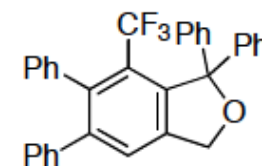




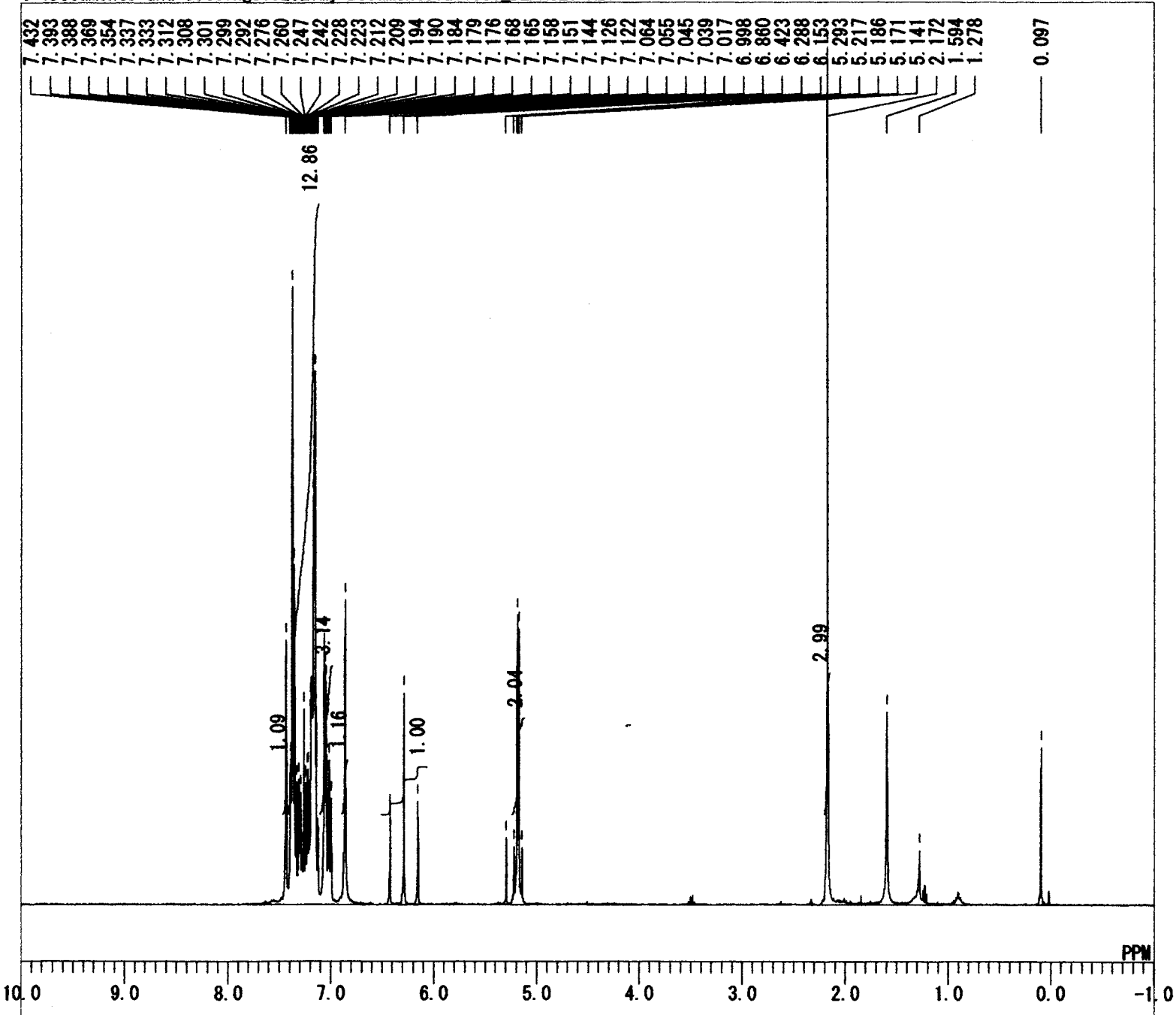
```

MENUF 19F
OBNUC 19F
OFR      376.05 MHz
OBSET    139.60 KHz
OBFIN    36.10 Hz
PW1      6.00 usec
DEADT    10.00 usec
PREDL    0.20000 msec
IWT      1.0000 msec
POINT    32768
SPO      32768
TIMES    16
DUMMY    1
FREQU    80000.00 Hz
FLT      40000 Hz
DELAY    5.00 usec
ACQTM    0.4096 sec
PD       4.9500 sec
ADBIT    16
RGAIN    14
BF       0.10 Hz
T1       0.00
T2       0.00
T3       90.00
T4       100.00
EXMOD    NON
EXPCM    NON:Single.coupled:PW1_ACQTM
IRNUC    1H
IFR      399.65 MHz
IRSET    124.00 KHz
IRFIN    10500.00 Hz
IRRPW    45 usec
IRATN    511
DFILE    CF3-Diyne (PhPh)-H+diphenyl lact
SF       TH5ATFG2
LKSET    61.60 KHz
LKFIN    79.0 Hz
LKLEV    180
LGAIN    24
LKPHS    250
LKSIG    1267
CSPED    13 Hz
FILDC
FILDF

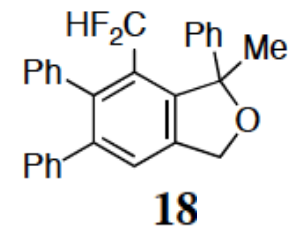
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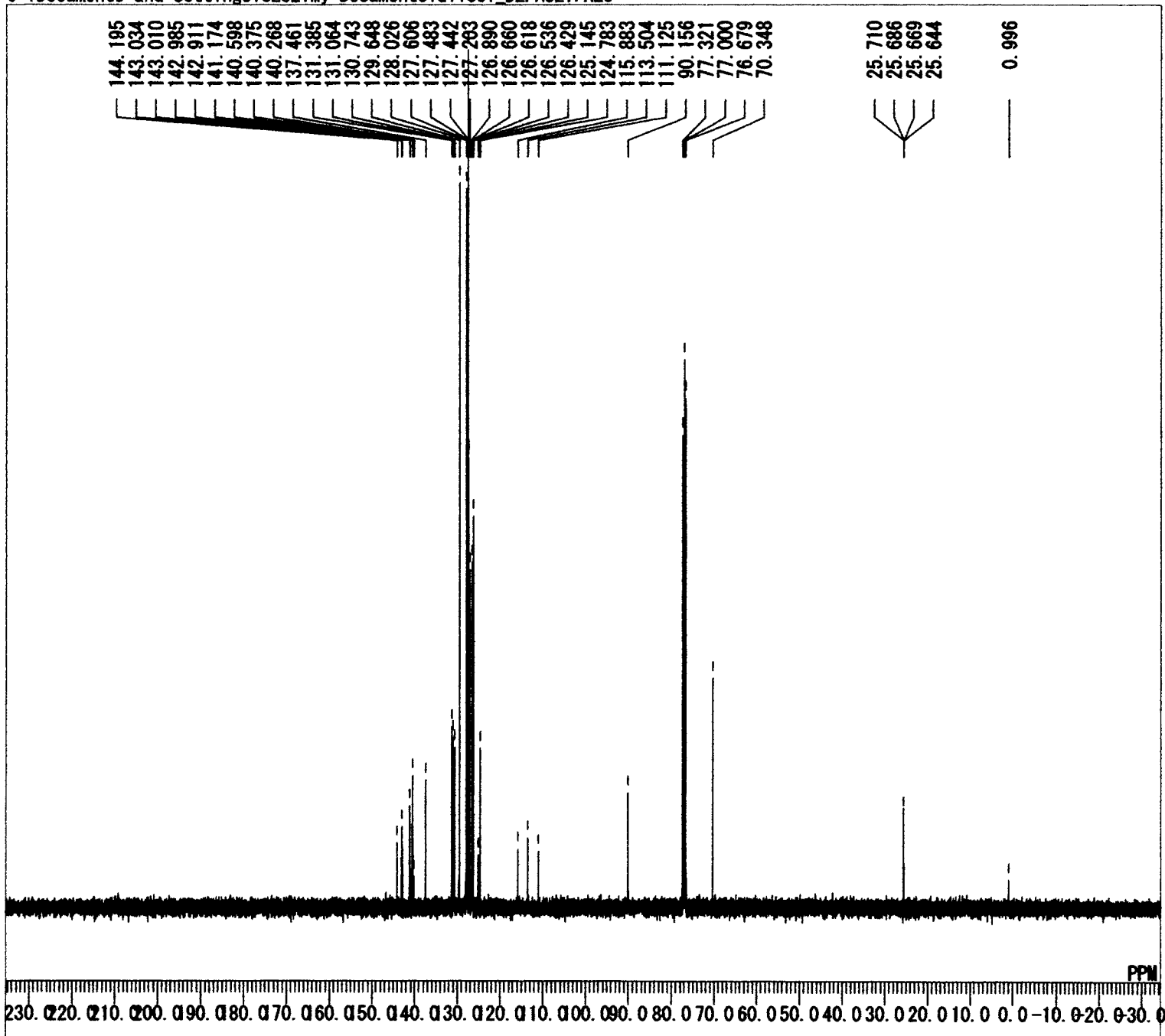


17

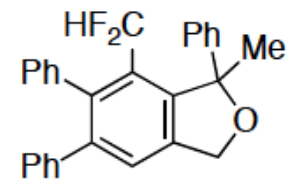


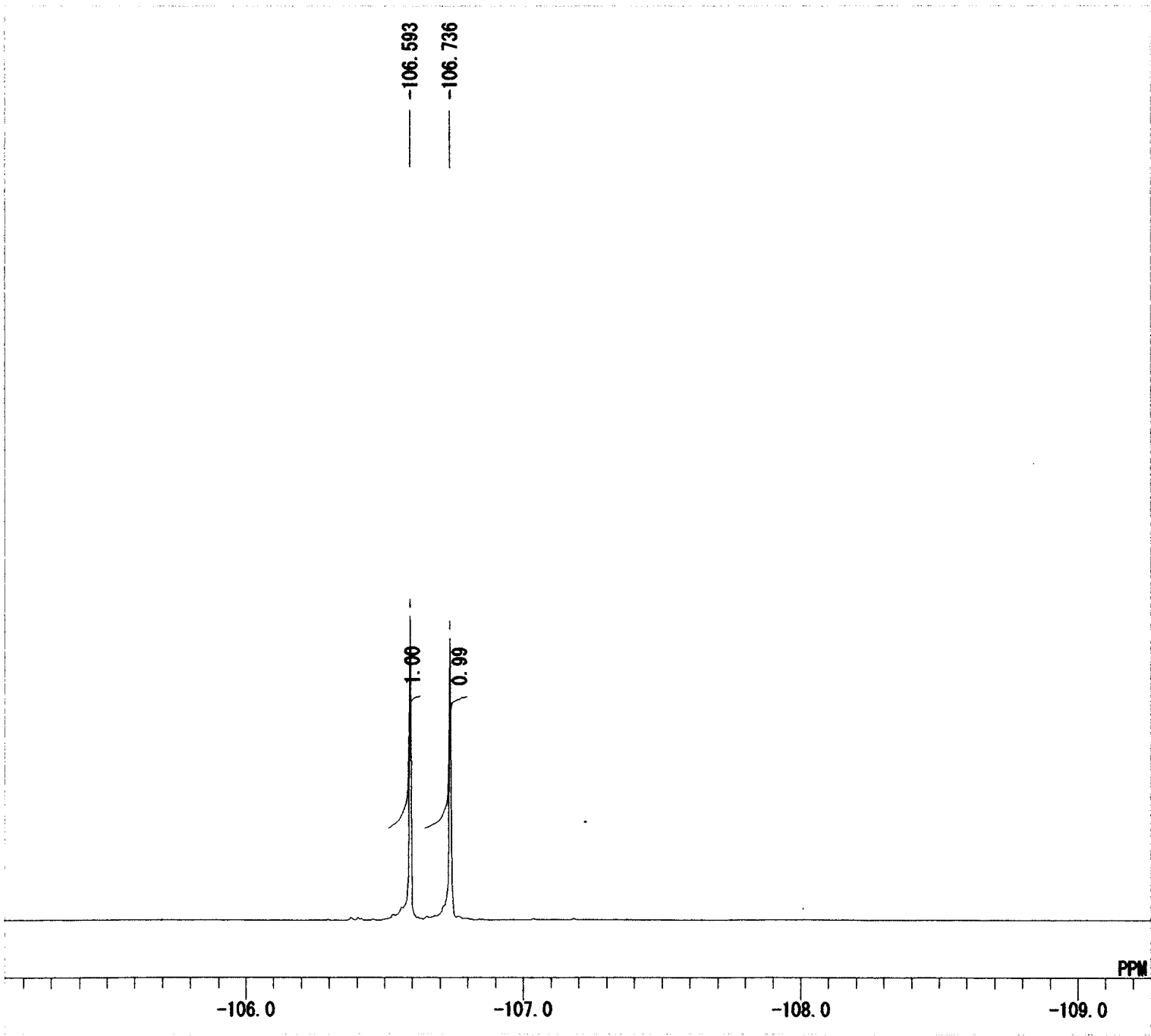
MENUF 1H
 OBNUC 1H
 OFR 399.65 MHz
 OBSET 135.40 KHz
 OBFIN 24.90 Hz
 PW1 5.80 usec
 DEADT 72.10 usec
 PREDL 0.20000 msec
 IWT 1.0000 msec
 POINT 16384
 SPO 16384
 TIMES 8
 DUMMY 1
 FREQU 7992.01 Hz
 FLT 4000 Hz
 DELAY 50.00 usec
 ACQTM 2.0501 sec
 PD 4.9500 sec
 ADBIT 16
 RGAIN 13
 BF 0.10 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD NON
 EXPCM NON:Single.coupled:PW1_ACQTM.
 IRNUC 1H
 IFR 399.65 MHz
 IRSET 136.90 KHz
 IRFIN 97.50 Hz
 IRRPW 45 usec
 IRATN 511
 DF ILE DEFAULT.ALS
 SF TH5ATFG2
 LKSET 61.60 KHz
 LKFIN 79.0 Hz
 LKLEV 180
 LGAIN 22
 LKPHS 250
 LKSIG 670
 CSPED 11 Hz
 FILDC
 FILDF





MENUF 13C
 OBNUC 13C
 OFR 100.40 MHz
 OBSET 125.00 KHz
 OBFIN 10500.00 Hz
 PW1 6.00 usec
 DEADT 19.10 usec
 PREDL 0.2000 msec
 IWT 1.0000 msec
 POINT 32768
 SPO 32768
 TIMES 512
 DUMMY 1
 FREQU 27118.64 Hz
 FLT 13550 Hz
 DELAY 14.80 usec
 ACQTM 1.2083 sec
 PD 1.7920 sec
 ADBIT 16
 RGAIN 25
 BF 0.10 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD BCM
 EXPCM Bilevel. complete. decoupling:
 IRNUC 1H
 IFR 399.65 MHz
 IRSET 124.00 KHz
 IRFIN 10500.00 Hz
 IRRPW 45 usec
 IRATN 511
 DF FILE DEFAULT.ALS
 SF TH5ATFG2
 LKSET 61.60 KHz
 LKFIN 79.0 Hz
 LKLEV 180
 LGAIN 22
 LKPHS 250
 LKSIG 658
 CSPED 11 Hz
 FILDC
 FILDF

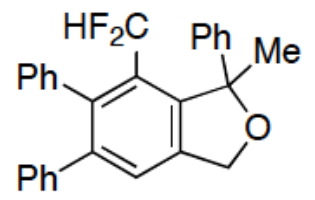




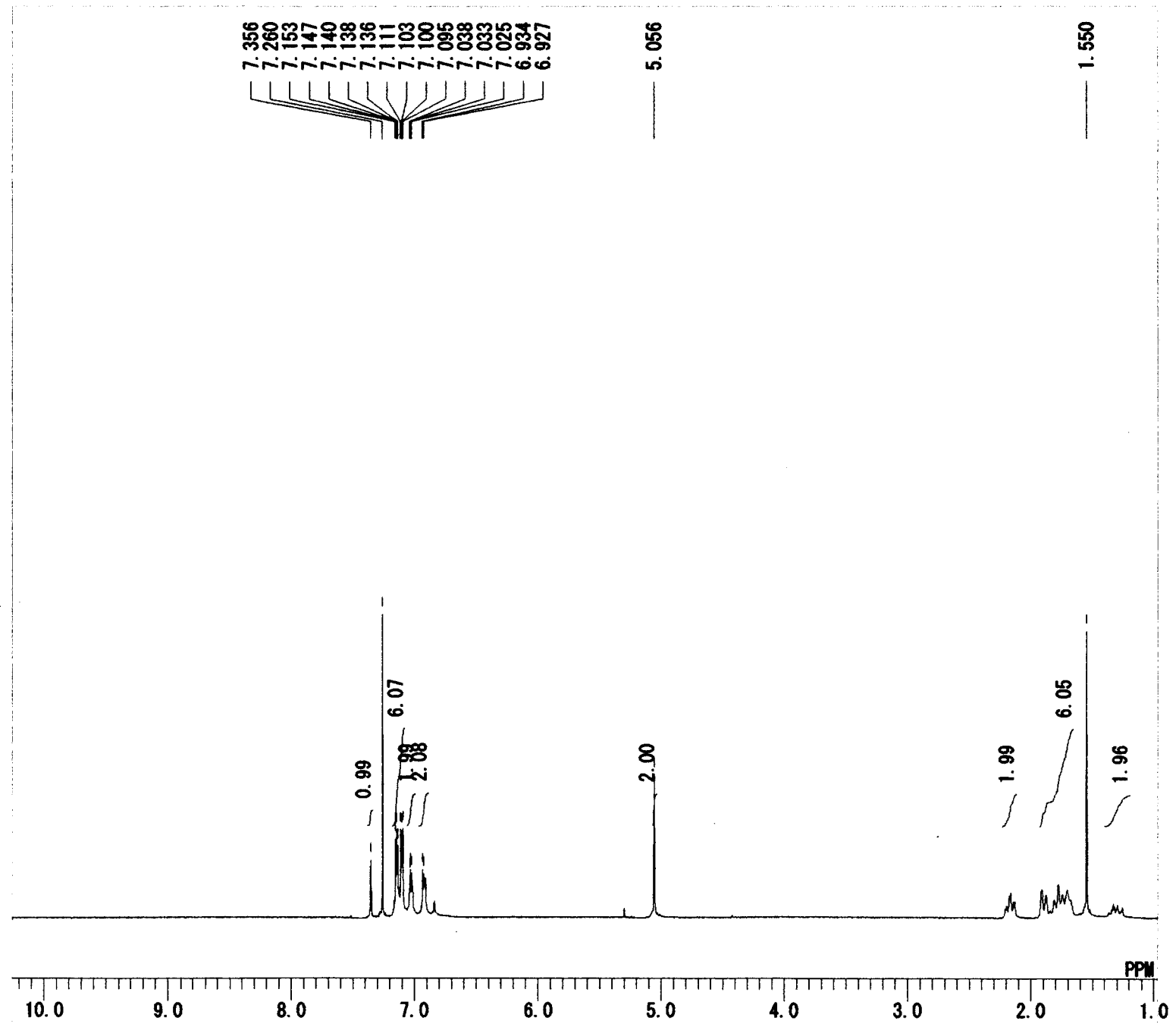
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MENUF 19F
OBNUC 19F
OFR 376.05 MHz
OBSET 139.60 KHz
OBFIN 36.10 Hz
PW1 6.00 usec
DEADT 10.00 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 32768
SPO 32768
TIMES 16
DUMMY 1
FREQU 80000.00 Hz
FLT 40000 Hz
DELAY 5.00 usec
ACQTM 0.4096 sec
PD 4.9500 sec
ADBIT 16
RGAIN 14
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM
IRNUC 1H
IFR 399.65 MHz
IRSET 124.00 KHz
IRFIN 10500.00 Hz
IRRPW 45 usec
IRATN 511
DFILE product-diyne-HCF2-PhMe-diPh
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 24
LKPHS 250
LKSIG 1125
CSPED 10 Hz
FILDC
FILDF

```



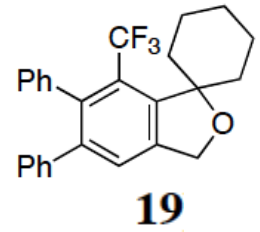
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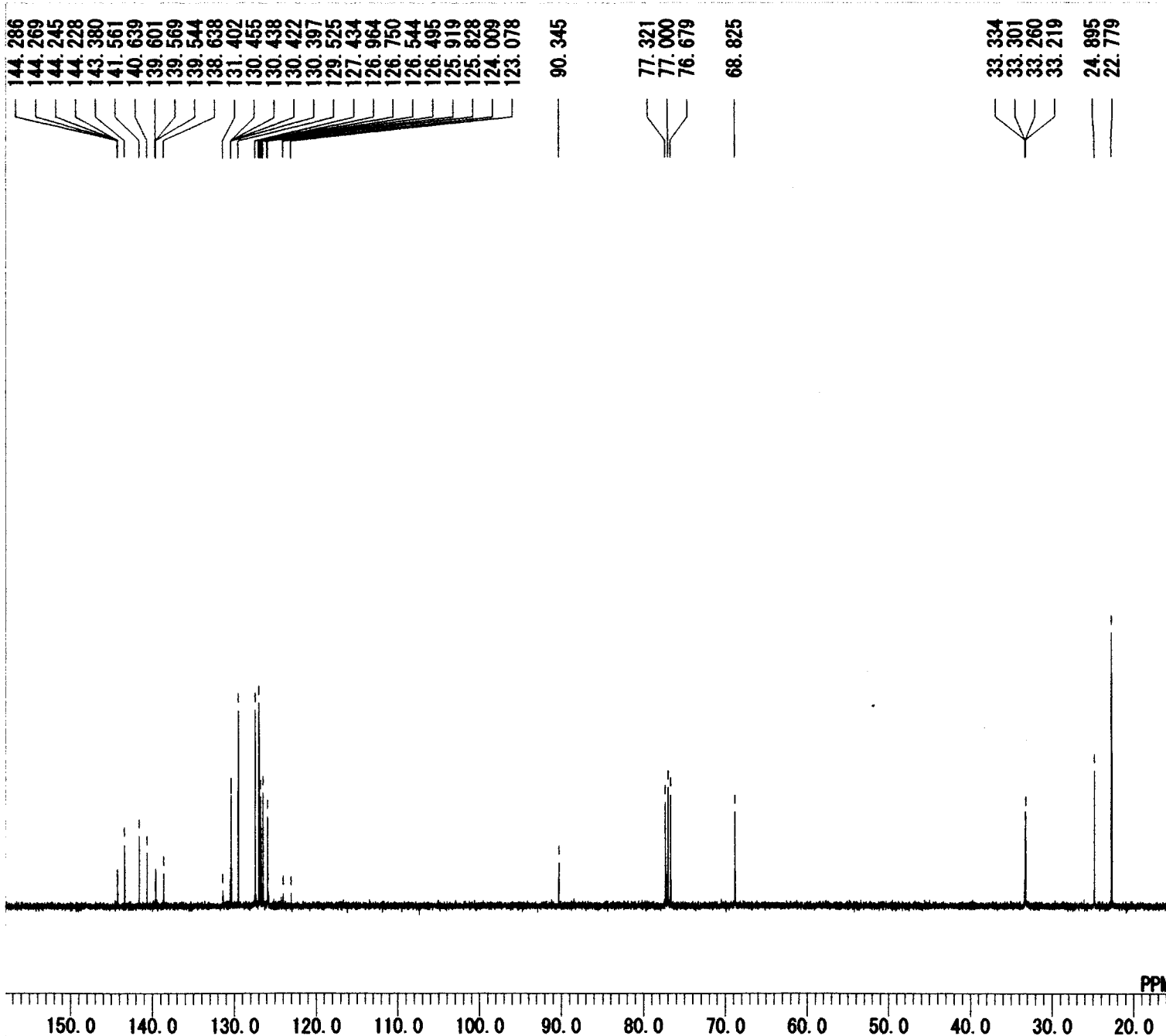


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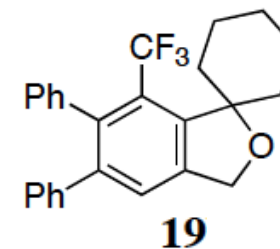
MENUF 1H
OBNUC 1H
OFR 399.65 MHz
OBSET 135.40 KHz
OBFIN 24.90 Hz
PW1 5.80 usec
DEADT 72.10 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7992.01 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 2.0501 sec
PD 4.9500 sec
ADBIT 16
RGAIN 19
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM
IRNUC 1H
IFR 399.65 MHz
IRSET 136.90 KHz
IRFIN 97.50 Hz
IRRPW 45 usec
IRATN 511
DFILE product-diyne-c-Hex-diPh(1H).
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGA1N 24
LKPHS 250
LKSIG 1271
CSPED 10 Hz
FILDC
FILDF

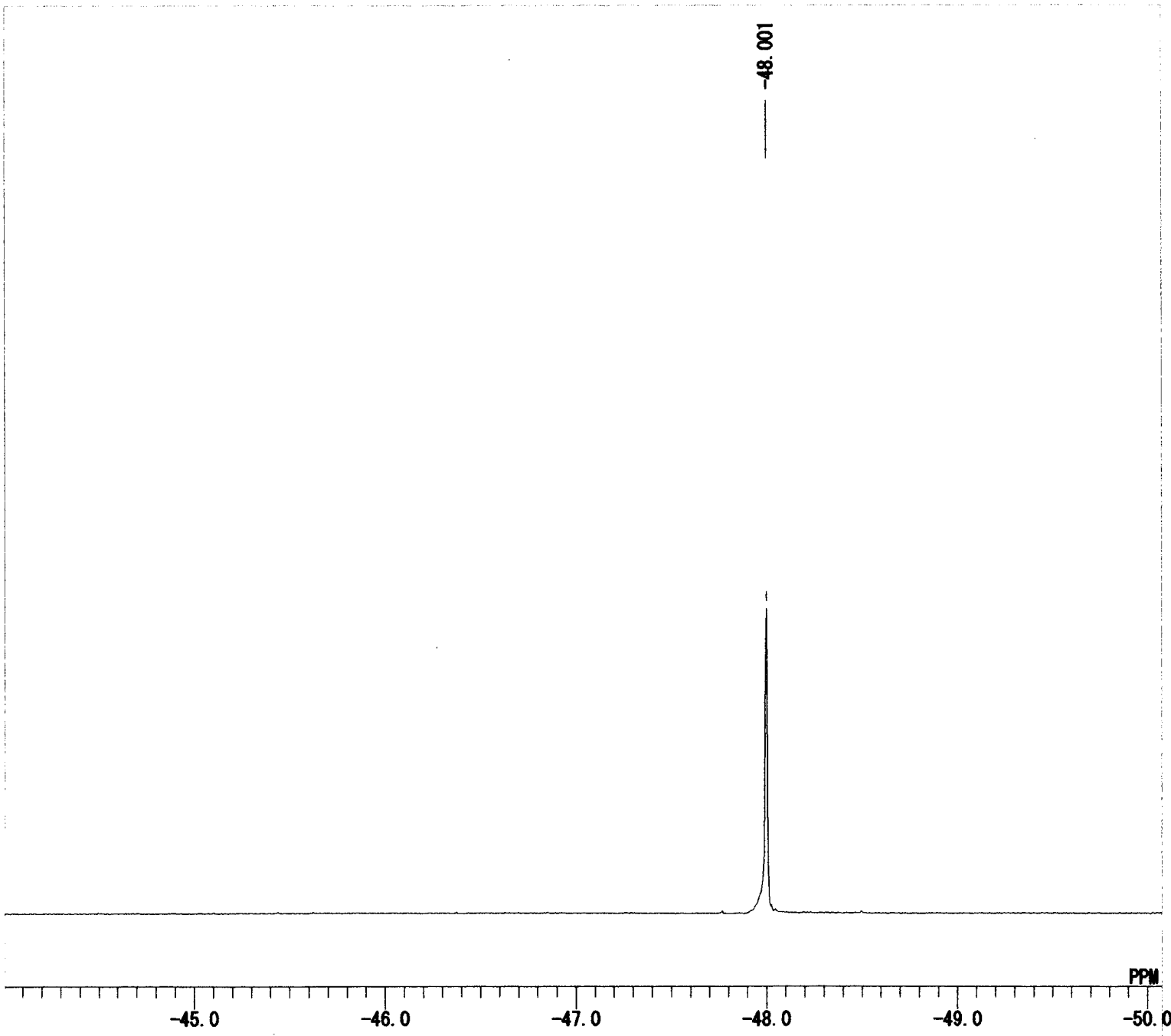
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MENUF 13C
 OBNUC 13C
 OFR 100.40 MHz
 OBSET 125.00 KHz
 OBFIN 10500.00 Hz
 PW1 6.00 usec
 DEADT 19.10 usec
 PREDL 0.20000 msec
 IWT 1.0000 msec
 POINT 32768
 SPO 32768
 TIMES 256
 DUMMY 1
 FREQU 27118.64 Hz
 FLT 13550 Hz
 DELAY 14.80 usec
 ACQTM 1.2083 sec
 PD 1.7920 sec
 ADBIT 16
 RGAIN 25
 BF 0.10 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD BCM
 EXPCM Bilevel. complete. decoupling: :
 IRNUC 1H
 IFR 399.65 MHz
 IRSET 124.00 KHz
 IRFIN 10500.00 Hz
 IRRPW 45 usec
 IRATN 511
 DF FILE product-diyne-c-Hex-diPh (13C)
 SF TH5ATFG2
 LKSET 61.60 KHz
 LKFIN 79.0 Hz
 LKLEV 180
 LGAIN 26
 LKPHS 250
 LKSIG 1471
 CSPED 15 Hz
 FILDC
 FILDF

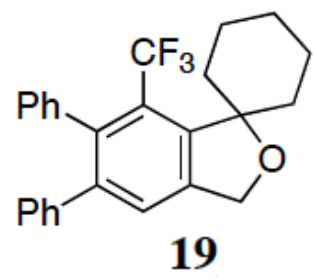


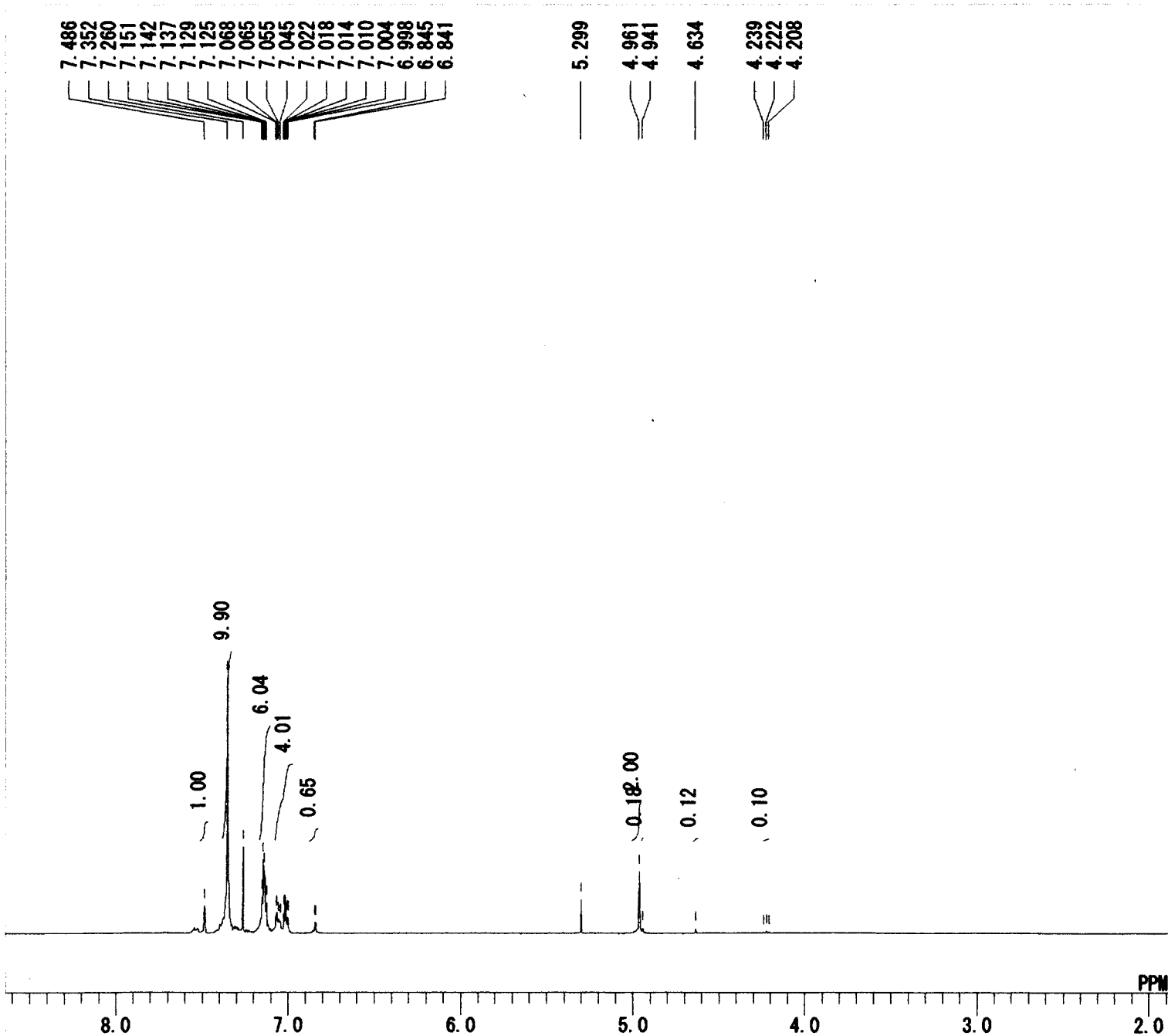


```

MENUF 19F
OBNUC 19F
OFR      376.05 MHz
OBSET    139.60 KHz
OBFIN    36.10 Hz
PW1      6.00 usec
DEADT    10.00 usec
PREDL    0.20000 msec
IWT      1.0000 msec
POINT    32768
SPO      32768
TIMES    16
DUMMY    1
FREQU    80000.00 Hz
FLT      40000 Hz
DELAY    5.00 usec
ACQTM    0.4096 sec
PD       4.9500 sec
ADBIT    16
RGAIN    14
BF       0.10 Hz
T1       0.00
T2       0.00
T3       90.00
T4       100.00
EXMOD    NON
EXPCM    NON:Single.coupled:PW1_ACQTM
IRNUC    1H
IFR      399.65 MHz
IRSET    124.00 KHz
IRFIN    10500.00 Hz
IRRPW    45 usec
IRATN    511
DFILE    product-diyne-c-Hex-diPh(19F)
SF       TH5ATFG2
LKSET    61.60 KHz
LKFIN    79.0 Hz
LKLEV    180
LGAIN    24
LKPHS    250
LKSIG    1269
CSPED    10 Hz
FILDC
FILDF

```

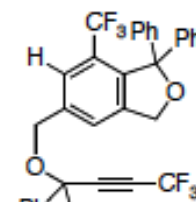
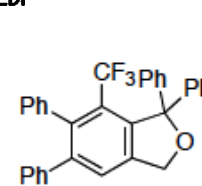


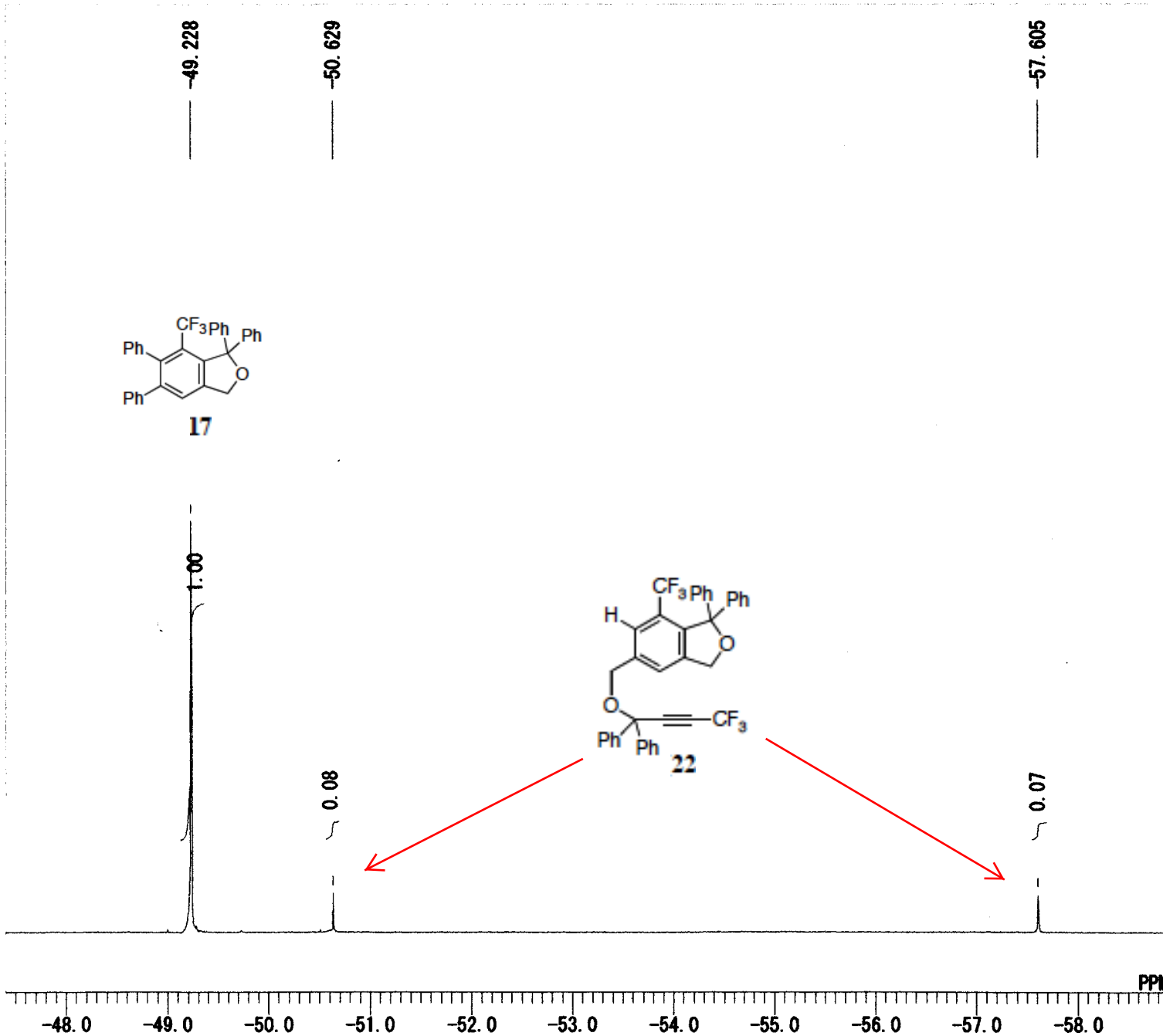
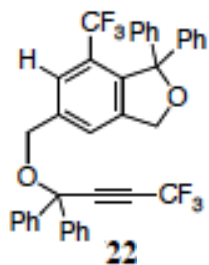


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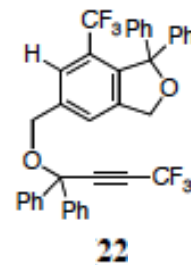
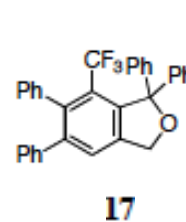
MENUF 1H
OBNUC 1H
OFR 399.65 MHz
OBSET 135.40 KHz
OBF IN 24.90 Hz
PW1 5.80 usec
DEADT 72.10 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7992.01 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 2.0501 sec
PD 4.9500 sec
ADBIT 16
RGAIN 19
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM
IRNUC 1H
IFR 399.65 MHz
IRSET 136.90 KHz
IRF IN 97.50 Hz
IRRPW 45 usec
IRATN 511
DFILE product-diyne-PhPh-diPh-high-
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 24
LKPHS 250
LKSIG 1270
CSPED 13 Hz
FILDC
FILDF

```

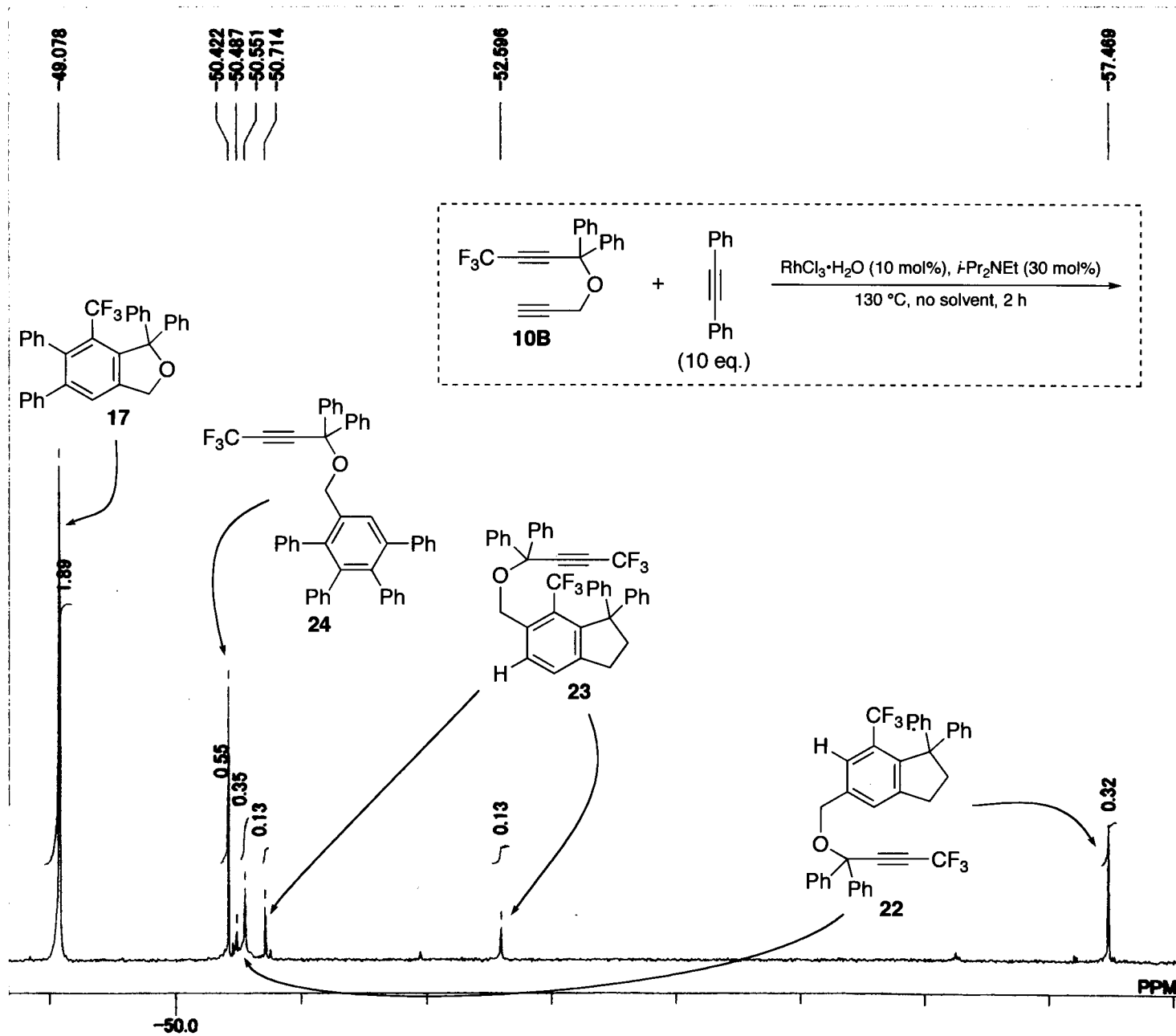




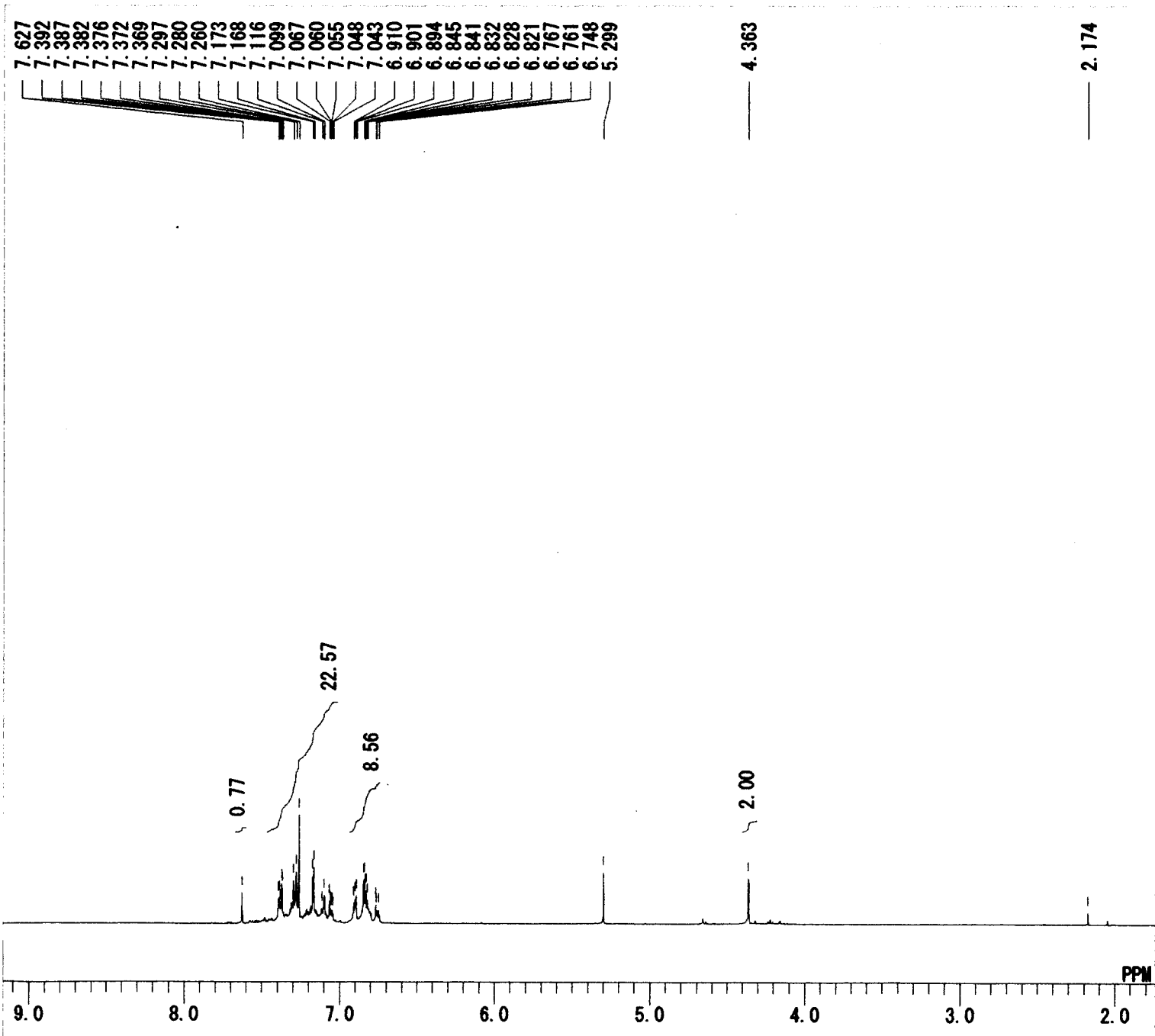
MENUF	19F
OBNUC	19F
QFR	376.05 MHz
OBSET	139.60 KHz
OBFIN	36.10 Hz
PW1	6.00 usec
DEADT	10.00 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	16
DUMMY	1
FREQU	80000.00 Hz
FLT	40000 Hz
DELAY	5.00 usec
ACQTM	0.4096 sec
PD	4.9500 sec
ADBIT	16
RGAIN	14
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM
IRNUC	1H
IFR	399.65 MHz
IRSET	124.00 KHz
IRFIN	10500.00 Hz
IRRPW	45 usec
IRATN	511
DFILE	product-diyne-PhPh-diPh-high-
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGA IN	24
LKPHS	250
LKSIG	1267
CSPED	13 Hz
FILDC	
FILDF	



¹⁹F NMR Analysis of the reaction mixture (Total yield for all CF₃-containing compounds : 100%)



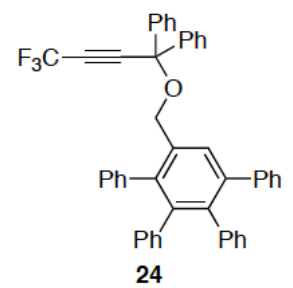
MENUF	19F
OBNUC	19F
OFR	376.05 MHz
OBSET	139.60 KHz
OBFIN	36.10 Hz
PW1	6.00 usec
DEADT	10.00 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	16
DUMMY	1
FREQU	80000.00 Hz
FLT	40000 Hz
DELAY	5.00 usec
ACQTM	0.4096 sec
PD	4.9500 sec
ADBIT	16
RGAIN	14
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM_PC
IRNUC	1H
IFR	399.65 MHz
IRSET	124.00 KHz
IRFIN	10500.00 Hz
IRRPW	45 usec
IRATN	511
DFILE	_DEFAULT.ALS
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	23
LKPHS	250
LKSIG	689
CSPED	12 Hz
FILDC	
FILDF	



```

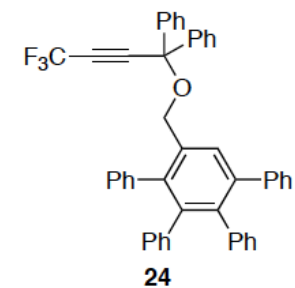
MENUF 1H
IRNUC 1H
OFR 399.65 MHz
OBSET 135.40 KHz
OBFIN 24.90 Hz
PW1 5.80 usec
DEADT 72.10 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7992.01 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 2.0501 sec
PD 4.9500 sec
ADBIT 16
RGAIN 18
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM
IRNUC 1H
IFR 399.65 MHz
IRSET 136.90 KHz
IRFIN 97.50 Hz
IRRPW 45 usec
IRATN 511
DFILE product-diyne-PhPh-diPh-low-
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 22
LKPHS 250
LKSIG 703
CSPED 11 Hz
FILDC
FILDF

```



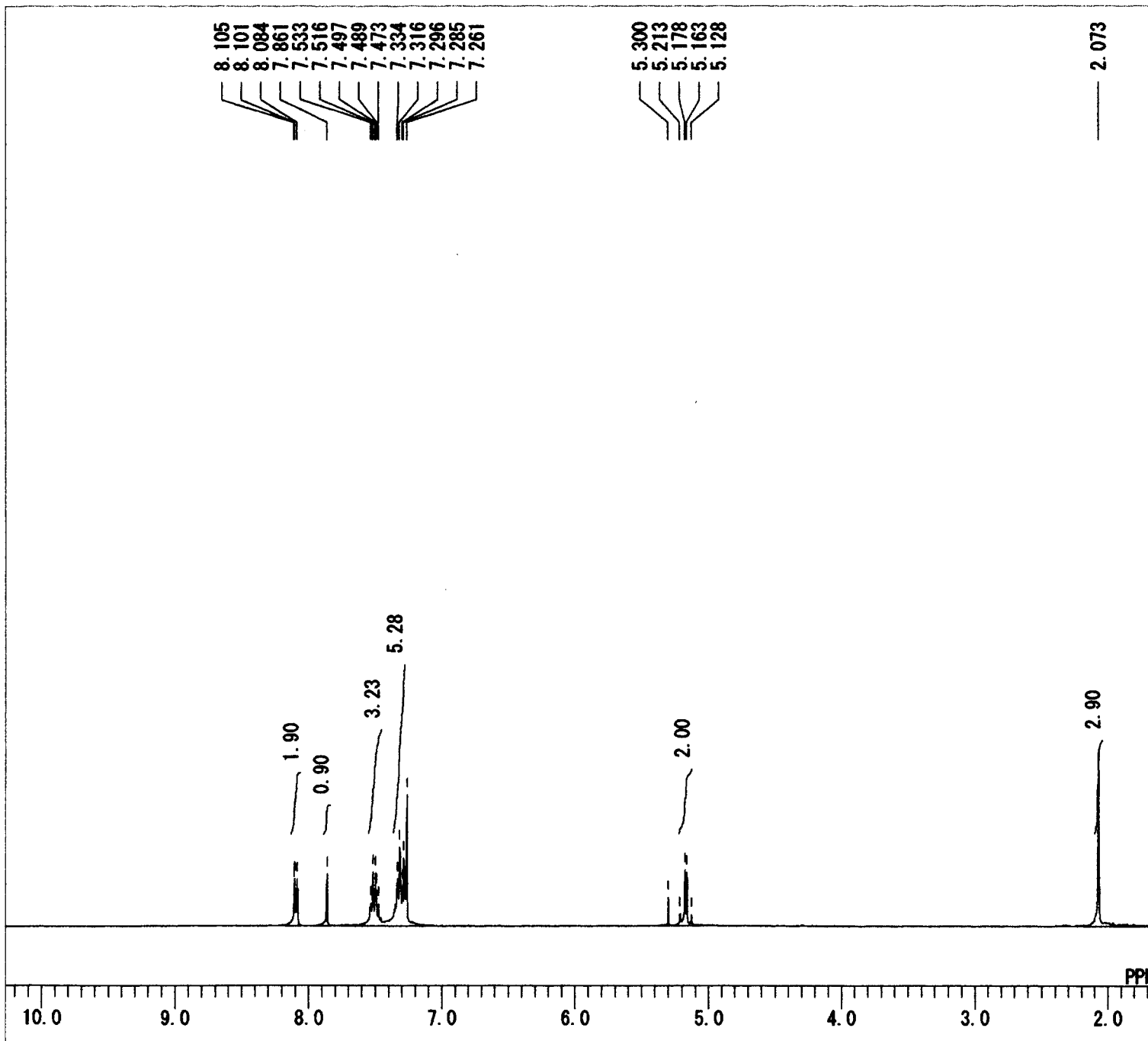
-50.506

MENUF 19F
OBNUC 19F
OFR 376.05 MHz
OBSET 139.60 KHz
OBF IN 36.10 Hz
PW1 6.00 usec
DEADT 10.00 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 32768
SPO 32768
TIMES 16
DUMMY 1
FREQU 80000.00 Hz
FLT 40000 Hz
DELAY 5.00 usec
ACQTM 0.4096 sec
PD 4.9500 sec
ADBIT 16
RGAIN 14
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM
IRNUC 1H
IFR 399.65 MHz
IRSET 124.00 KHz
IRFIN 10500.00 Hz
IRRPW 45 usec
IRATN 511
DFILE product-diyne-PhPh-diPh-low-
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 22
LKPHS 250
LKSIG 700
CSPED 10 Hz
FILDC
FILDF

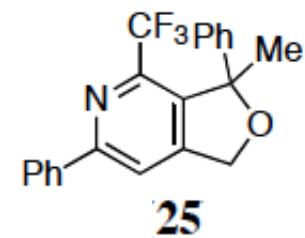


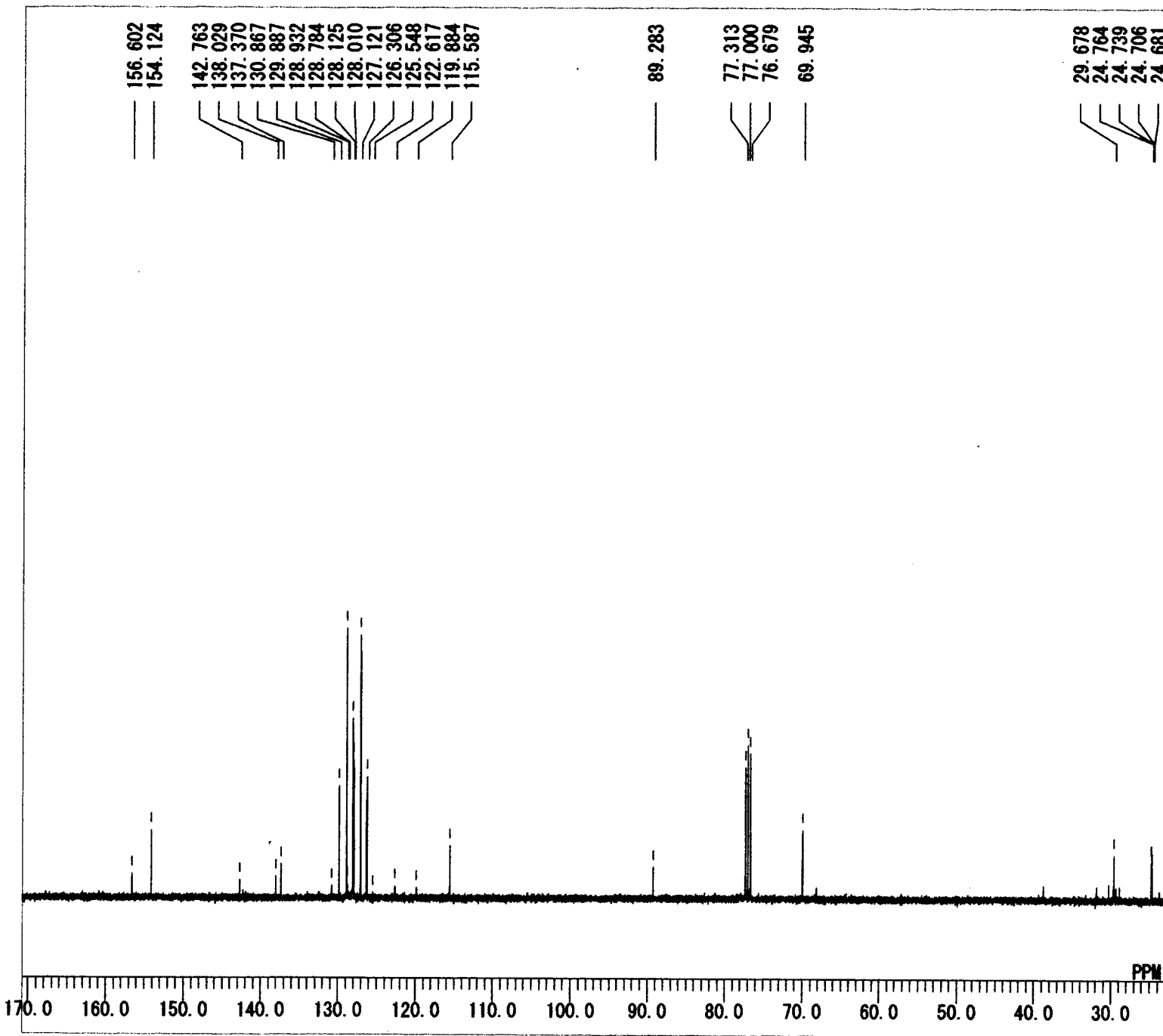
PPM

-47.0 -48.0 -49.0 -50.0 -51.0 -52.0 -53.0 -54.0 -55.0

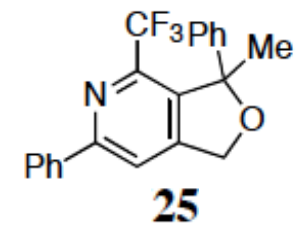


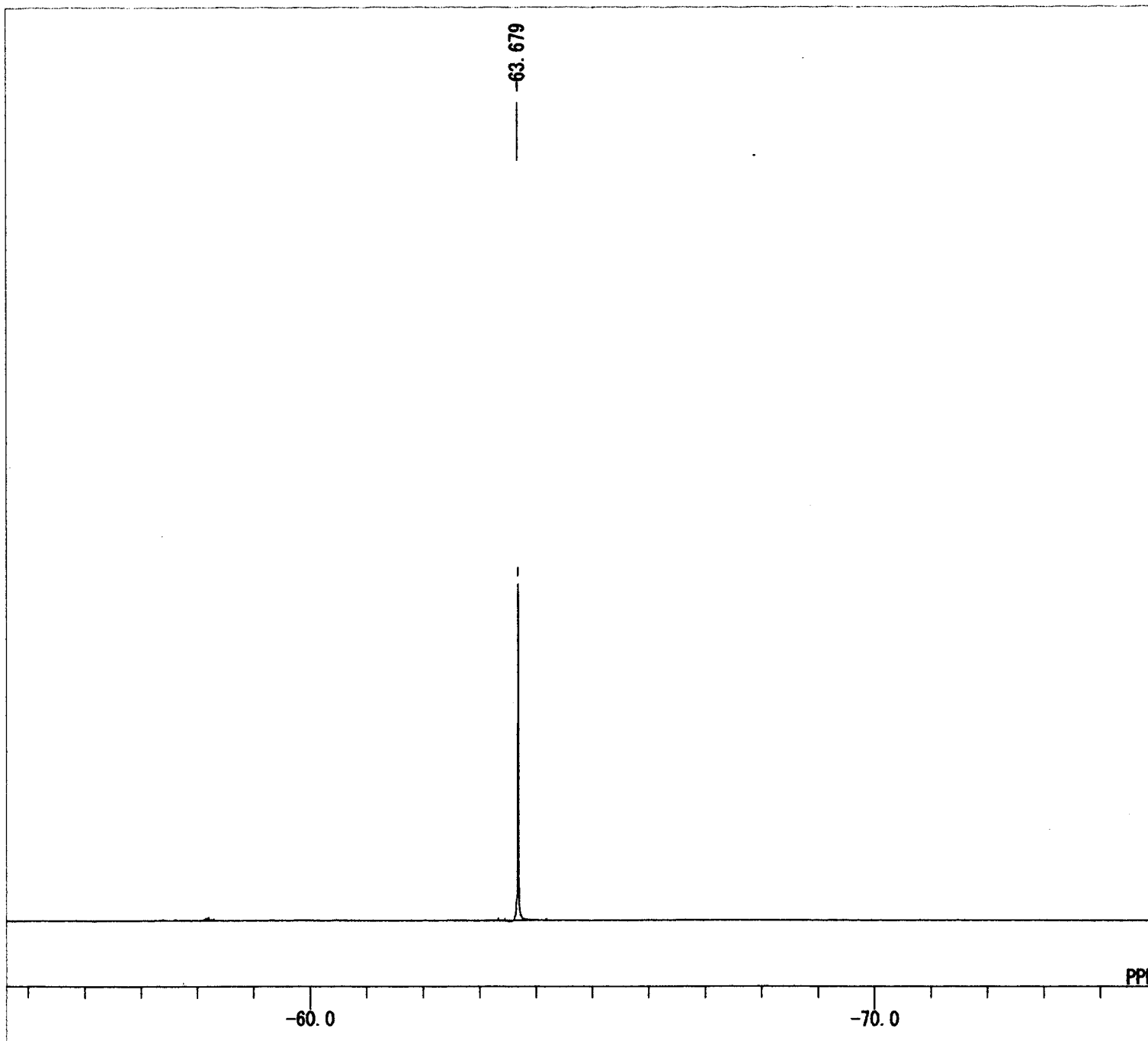
MENUF 1H
 OBNUC 1H
 OFR 399.65 MHz
 OBSET 135.40 KHz
 OBF IN 24.90 Hz
 PW1 5.80 usec
 DEADT 72.10 usec
 PREDL 0.20000 msec
 IWT 1.0000 msec
 POINT 16384
 SPO 16384
 TIMES 8
 DUMMY 1
 FREQU 7992.01 Hz
 FLT 4000 Hz
 DELAY 50.00 usec
 ACQTM 2.0501 sec
 PD 4.9500 sec
 ADBIT 16
 RGAIN 19
 BF 0.10 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD NON
 EXPCM NON:Single.coupled:PW1_ACQTM
 IRNUC 1H
 IFR 399.65 MHz
 IRSET 136.90 KHz
 IRF IN 97.50 Hz
 IRRPW 45 usec
 IRATN 511
 DFILE CF3-Diyne(PhMe)-H+PhCN-(1H).a
 SF TH5ATFG2
 LKSET 61.60 KHz
 LKF IN 79.0 Hz
 LKLEV 180
 LGAIN 22
 LKPHS 250
 LKSIG 581
 CSPED 10 Hz
 FILDC
 FILDF





MENUF 13C
 OBNUC 13C
 OFR 100.40 MHz
 OBSET 125.00 KHz
 OFBIN 10500.00 Hz
 PW1 6.00 usec
 DEADT 19.10 usec
 PREDL 0.20000 msec
 IWT 1.0000 msec
 POINT 32768
 SPO 32768
 TIMES 512
 DUMMY 1
 FREQU 27118.64 Hz
 FLT 13550 Hz
 DELAY 14.80 usec
 ACQTM 1.2083 sec
 PD 1.7920 sec
 ADBIT 16
 RGAIN 25
 BF 0.10 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD BCM
 EXPCM Bilevel. complete. decoupling:
 IRNUC 1H
 IFR 399.65 MHz
 IRSET 124.00 KHz
 IRFIN 10500.00 Hz
 IRRPW 45 usec
 IRATN 511
 DFILE CF3-Diyne (PhMe)-H-PhCN- (13C).
 SF TH5ATFG2
 LKSET 61.60 KHz
 LKFIN 79.0 Hz
 LKLEV 180
 LGAIN 27
 LKPHS 250
 LKSIG 2809
 GSPED 12 Hz
 FILDC
 FILDF

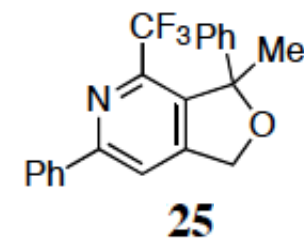




```

MENUF 19F
OBNUC 19F
OFR      376.05 MHz
OBSET    139.60 KHz
OBFIN    36.10 Hz
PW1      6.00 usec
DEADT    10.00 usec
PREDL    0.20000 msec
IWT      1.0000 msec
POINT    32768
SPO      32768
TIMES    16
DUMMY    1
FREQU    80000.00 Hz
FLT      40000 Hz
DELAY    5.00 usec
ACQTM    0.4096 sec
PD       4.9500 sec
ADBIT    16
RGAIN    14
BF       0.10 Hz
T1       0.00
T2       0.00
T3       90.00
T4       100.00
EXMOD    NON
EXPCM    NON:Single.coupled:PW1_ACQTM.
IRNUC    1H
IFR      399.65 MHz
IRSET    124.00 KHz
IRFIN    10500.00 Hz
IRRPW    45 usec
IRATN    511
DFILE    CF3-Diyne (PhMe)-H+PhCN-(19F).
SF       TH5ATFG2
LKSET    61.60 KHz
LKFIN    79.0 Hz
LKLEV    180
LGAIN    22
LKPHS    250
LKSIG    582
CSPED    10 Hz
FILDC
FILDF

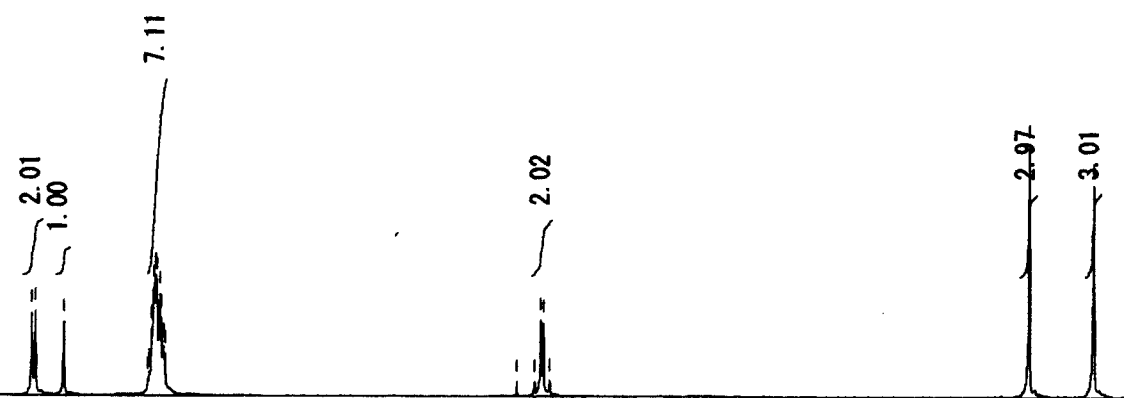
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8.010
7.990
7.832
7.358
7.354
7.338
7.328
7.319
7.314
7.310
7.300
7.294
7.289
7.279
7.273
7.263

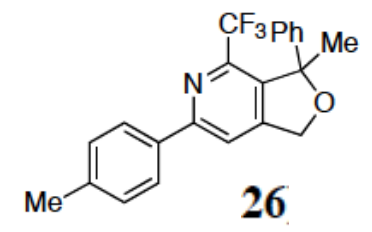
5.300
5.201
5.166
5.152
5.117

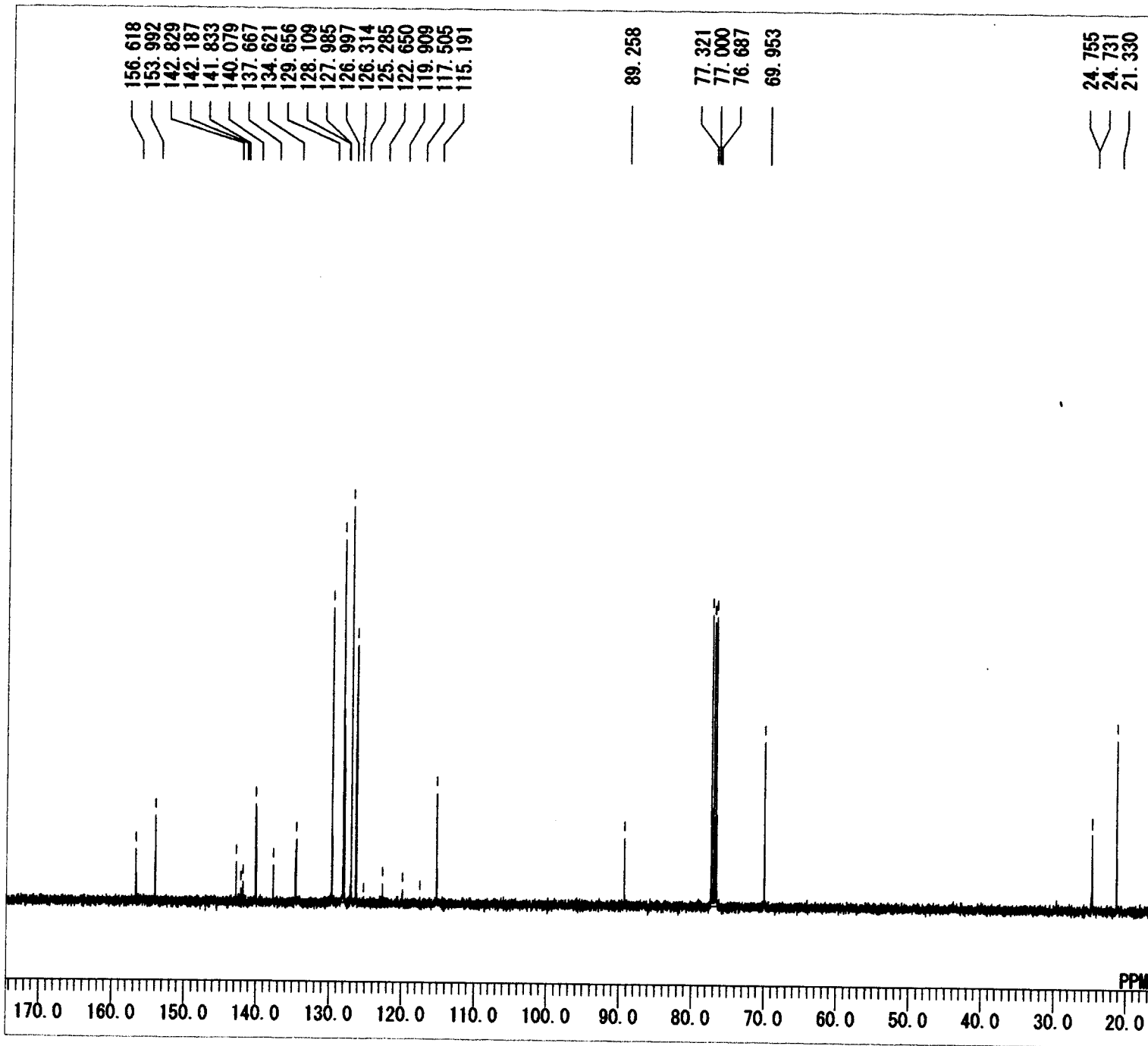
2.436
2.074



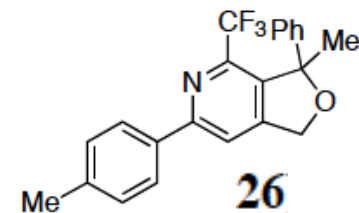
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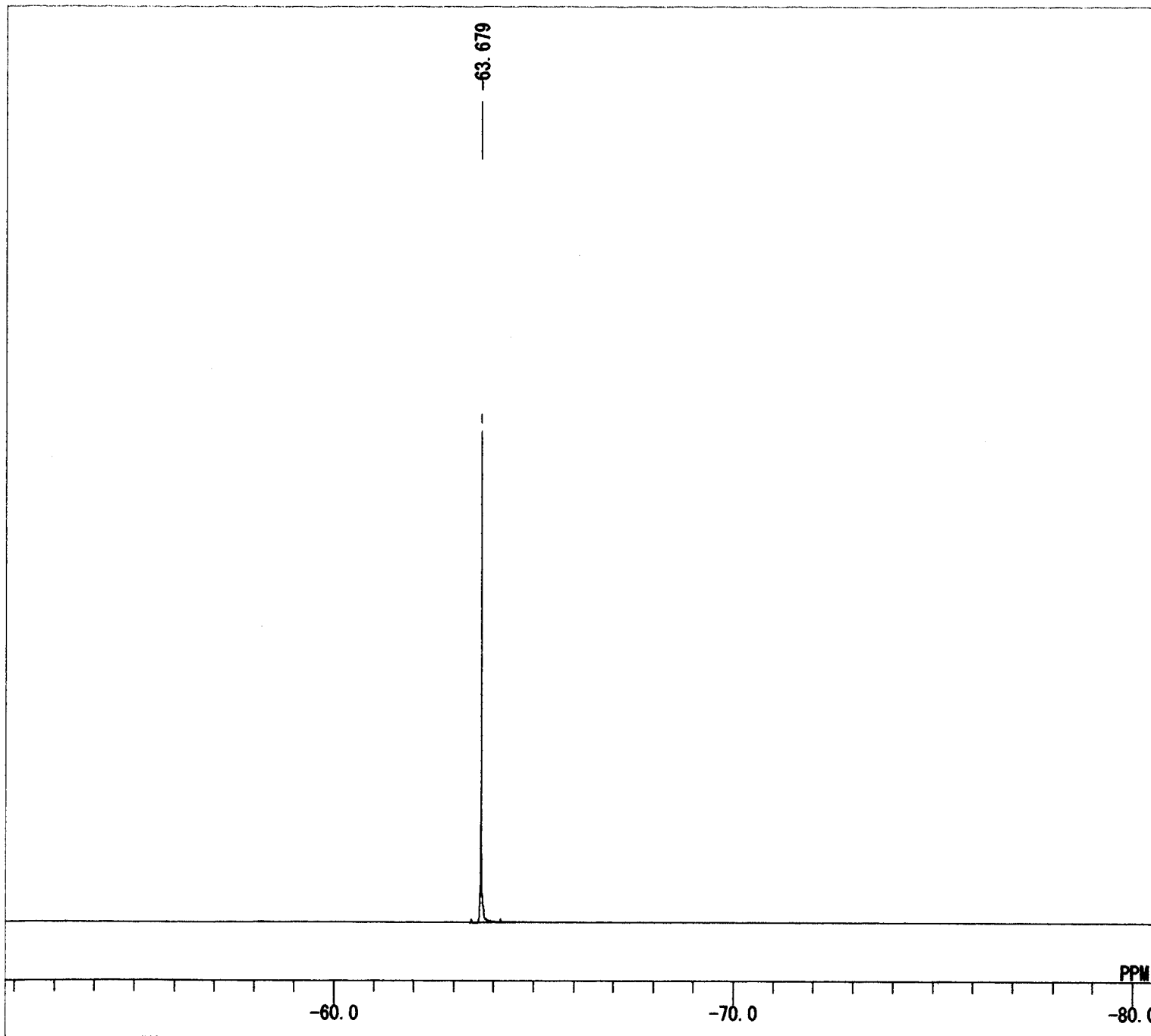
MENUF 1H
OBNUC 1H
OFR 399.65 MHz
OBSET 135.40 KHz
OBFIN 24.90 Hz
PW1 5.80 usec
DEADT 72.10 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7992.01 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 2.0501 sec
PD 4.9500 sec
ADBIT 16
RGAIN 14
BF 0.60 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM.
IRNUC 1H
IFR 399.65 MHz
IRSET 136.90 KHz
IRFIN 97.50 Hz
IRRPW 45 usec
IRATN 511
DFILE CF3-Diyne(PhMe)-H+ToICN-(1H).
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 22
LKPHS 250
LKSIG 633
CSPED 13 Hz
FILDC
FILDF
  
```





MENUF 13C
 OBNUC 13C
 OFR 100.40 MHz
 OBSET 125.00 KHz
 OBF IN 10500.00 Hz
 PW1 6.00 usec
 DEADT 19.10 usec
 PREDL 0.20000 msec
 IWT 1.0000 msec
 POINT 32768
 SPO 32768
 TIMES 512
 DUMMY 1
 FREQU 27118.64 Hz
 FLT 13550 Hz
 DELAY 14.80 usec
 ACQTM 1.2083 sec
 PD 1.7920 sec
 ADBIT 16
 RGAIN 25
 BF 0.60 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD BCM
 EXPCM Bilevel. complete. decoupling:
 IRNUC 1H
 IFR 399.65 MHz
 IRSET 124.00 KHz
 IRFIN 10500.00 Hz
 IRRPW 45 usec
 IRATN 511
 DFILE CF3-Diyne (PhMe)-H+ToICN-(13C)
 SF TH5ATFG2
 LKSET 61.60 KHz
 LKFIN 79.0 Hz
 LKLEV 180
 LGAIN 22
 LKPHS 250
 LKSIG 635
 CSPED 14 Hz
 FILDC
 FILDF

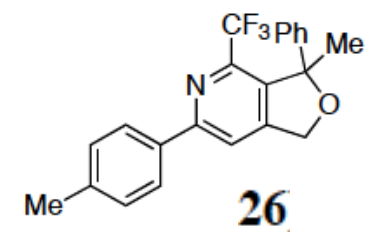


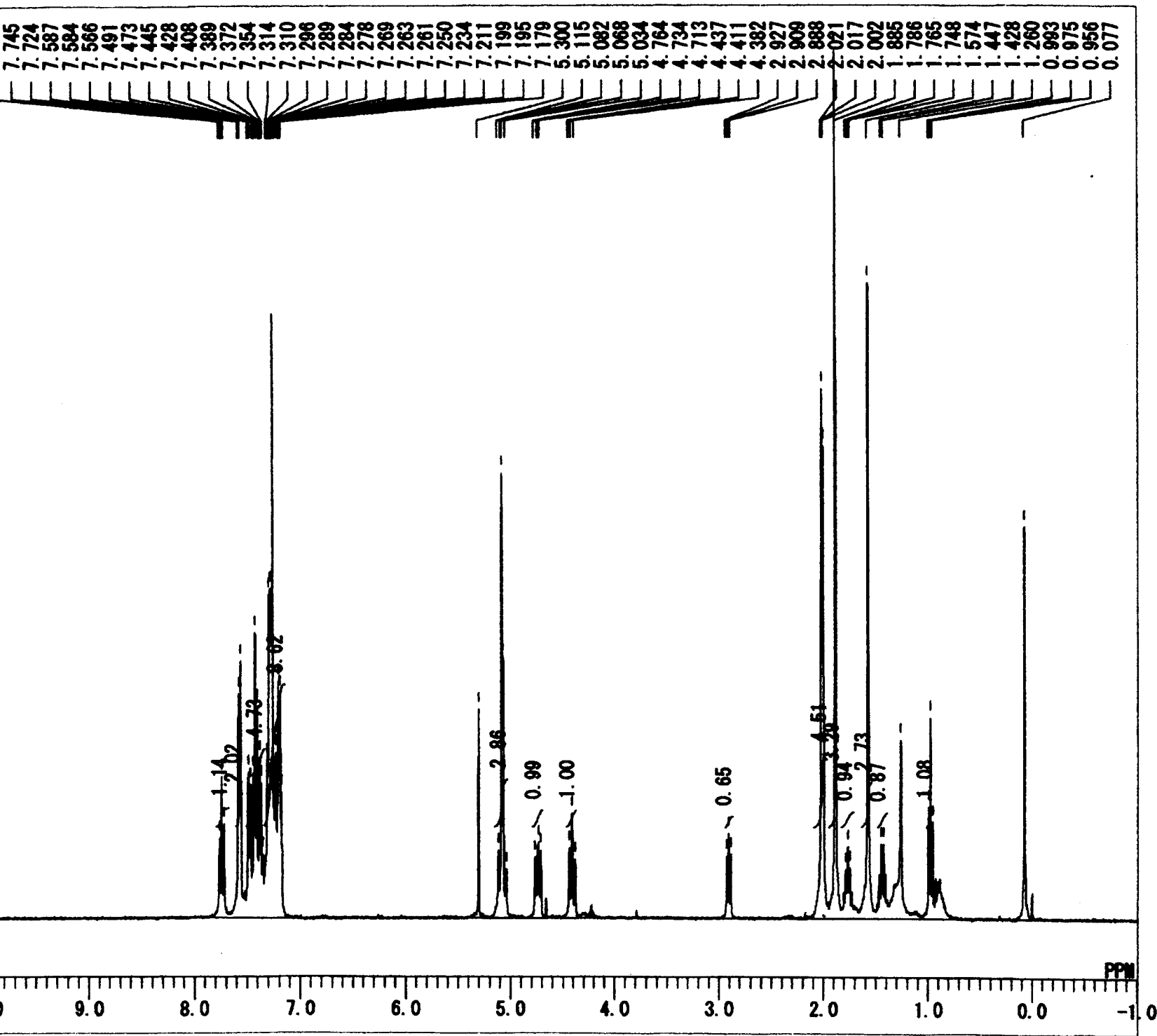


```

MNUF 19F
OBNUC 19F
OFR 376.05 MHz
OBSET 139.60 KHz
OBFIN 36.10 Hz
PW1 6.00 usec
DEADT 10.00 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 32768
SPO 32768
TIMES 16
DUMMY 1
FREQU 80000.00 Hz
FLT 40000 Hz
DELAY 5.00 usec
ACQTM 0.4096 sec
PD 4.9500 sec
ADBIT 16
RGAIN 14
BF 0.60 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM
IRNUC 1H
IFR 399.65 MHz
IRSET 124.00 KHz
IRFIN 10500.00 Hz
IRRPW 45 usec
IRATN 511
DFILE CF3-Diyne (PhMe)-H+ToICN-(19F)
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 22
LKPHS 250
LKSIG 634
CSPED 14 Hz
FILDC
FILDF

```

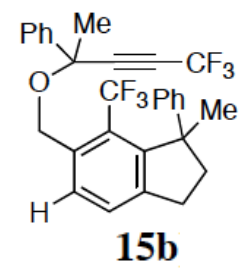
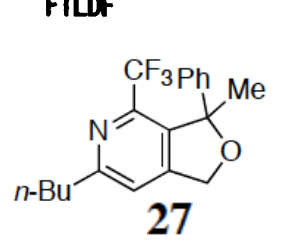


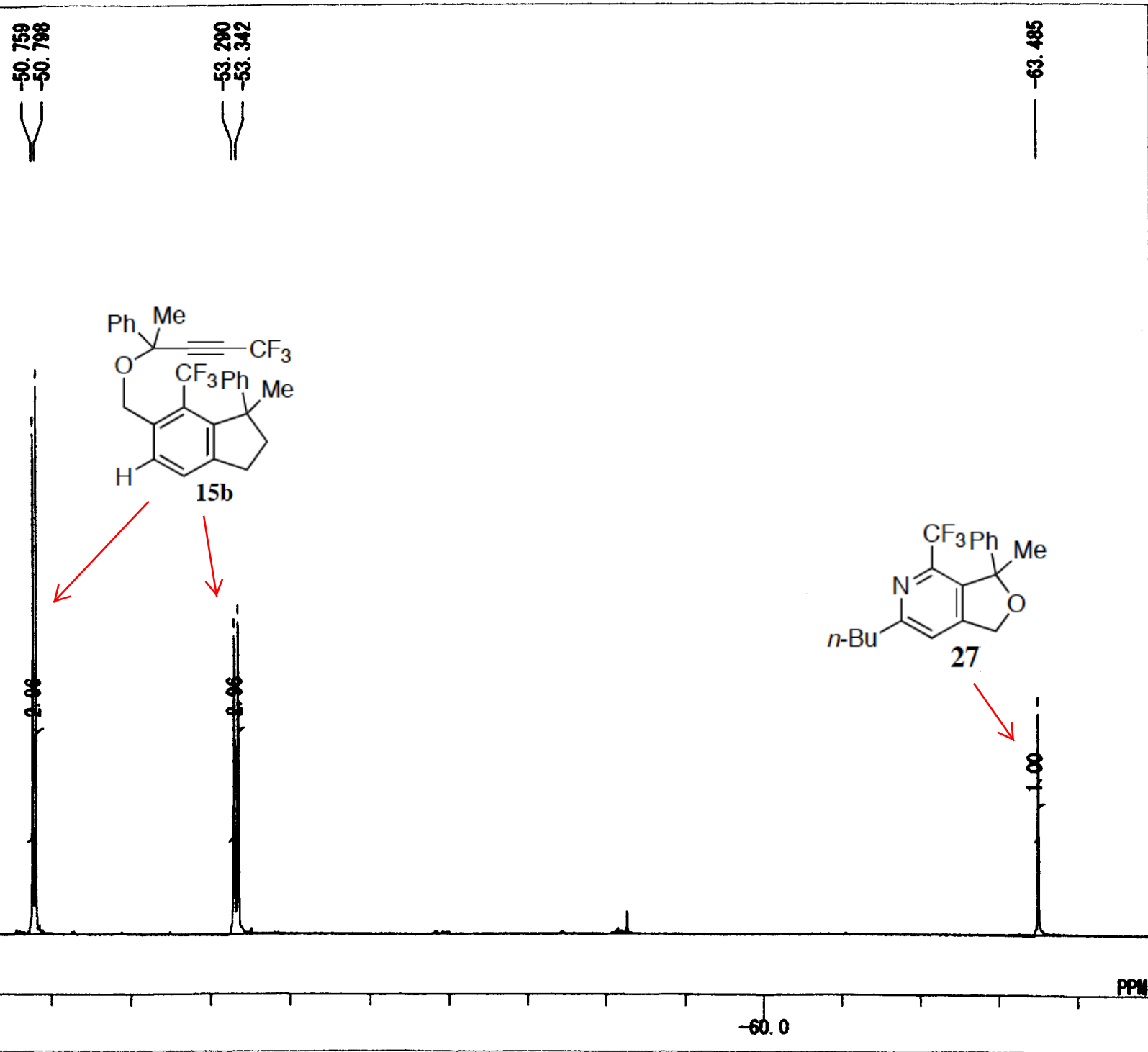


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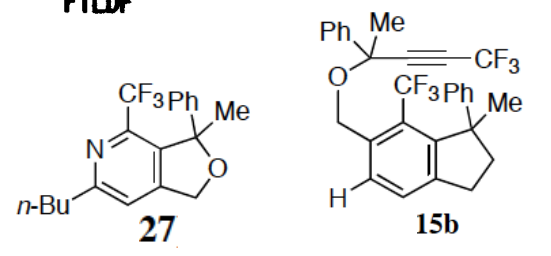
MENUF 1H
OBNUC 1H
OFR 399.65 MHz
OBSET 135.40 KHz
OBFIN 24.90 Hz
PW1 5.80 usec
DEADT 72.10 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7992.01 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 2.0501 sec
PD 4.9500 sec
ADBIT 16
RGAIN 17
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON: Single. coupled: PW1_ACQ
IRNUC 1H
IFR 399.65 MHz
IRSET 136.90 KHz
IRFIN 97.50 Hz
IRRPW 45 usec
IRATN 511
DFILE _DEFAULT.ALS
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 25
LKPHS 250
LKSIG 1382
CSPED 14 Hz
FILDC
FILDF

```

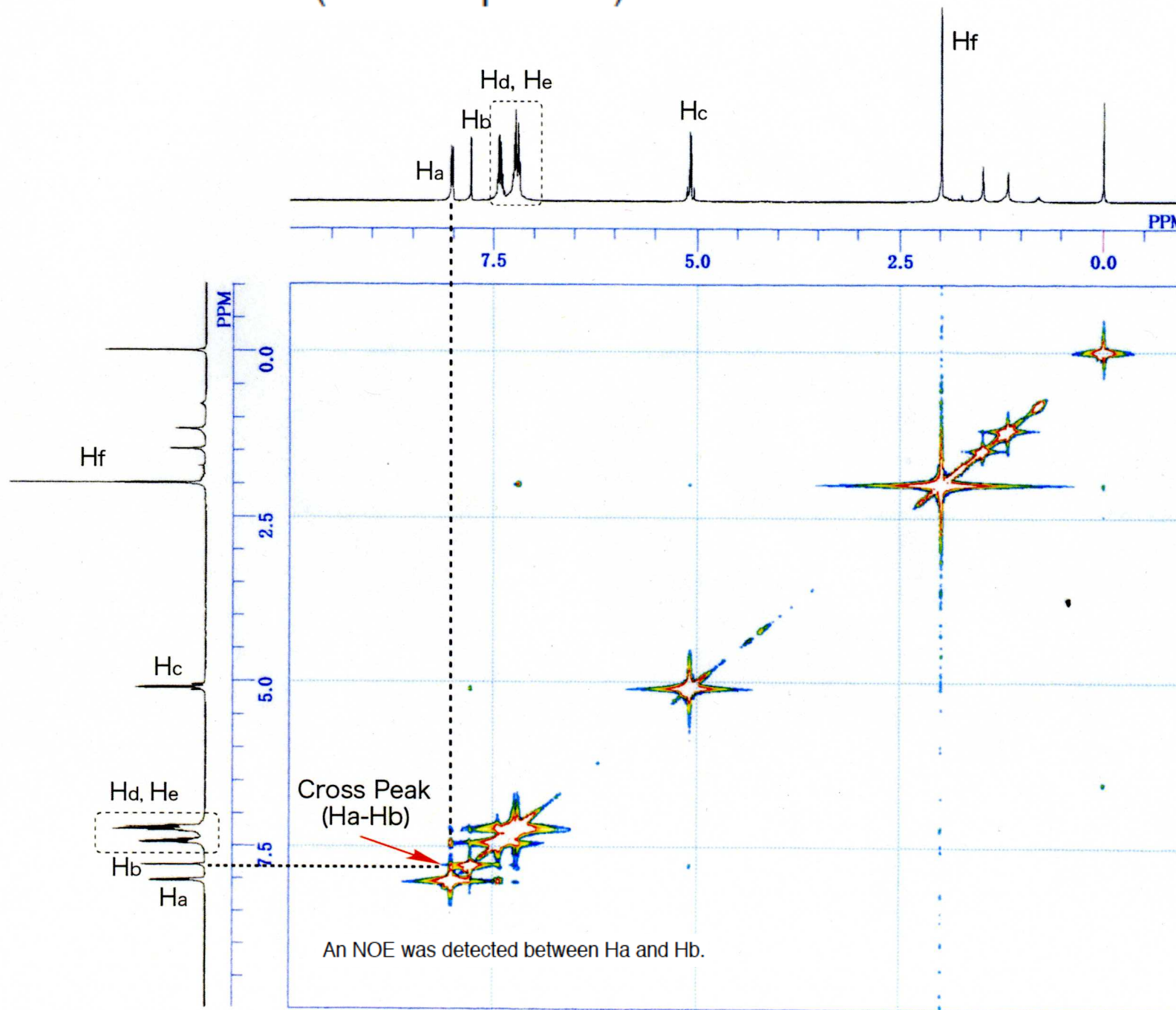




MENUF	19F
OBNUC	19F
OFR	376.05 MHz
OBSET	139.60 KHz
OBFIN	36.10 Hz
PW1	6.00 usec
DEADT	10.00 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	16
DUMMY	1
FREQU	80000.00 Hz
FLT	40000 Hz
DELAY	5.00 usec
ACQTM	0.4096 sec
PD	4.9500 sec
ADBIT	16
RGAIN	14
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACO
IRNUC	1H
IFR	399.65 MHz
IRSET	124.00 KHz
IRFIN	10500.00 Hz
IRRPW	45 usec
IRATN	511
DFILE	DEFAULT.ALS
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	25
LKPHS	250
LKSIG	1401
CSPED	14 Hz
FILDC	
FILDF	



Determination of the structure of the compound (NOESY spectrum)



```

DFILE      _DEFAULT.ALS
COMNT      product-diyne-PhMe-Ph
DATIM      Wed Sep 09 07:17:07 20
EXMOD      VNOENH
OBNUC      1H
OBFRQ      399.65 MHz
OBSET      134.30 KHz
OBFIN      36.60 Hz
POINT      2048
FREQU      4398.02 Hz
CLPNT      256
TODAT      256
CLFRQ      4405.29 Hz
SCANS      64
ACQTM      0.1164 sec
PD         1.5000 sec
PW1        11.60 usec
PW2        10.00 usec
PW3        10.00 usec
PI1        0.2270 msec
PI2        0.2270 msec
PI3        500.0000 msec
IRNUC      1H
CTEMP      22.0 c
SLVNT      CDCL3
EXREF      0.00 ppm
CLEXR      0.00
RGAIN      17
OBATN      511
LOOP1      1000
    
```

