

Supporting Information

for

Highly stereocontrolled synthesis of *trans*-enediynes via carbocupration of fluoroalkylated diynes

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Experimental, characterization details, and NMR spectra of synthesized compounds, 4e, 13a–e, and 14a–c

General methods

Infrared spectra (IR) were determined in a liquid film on a NaCl plate or KBr disk method with a JASCO FT/IR-4100 type A spectrometer. ^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 were 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 JEOL JMS-700MS spectrometer by electron impact (EI), chemical ionization (CI), and fast atom bombardment (FAB) methods.

All reactions were routinely monitored by ^{19}F NMR spectroscopy or TLC and carried out under an atmosphere of argon.

Materials

DMF, Et_3N and HMDS were freshly distilled from calcium hydride (CaH_2). All chemicals were of reagent grade and, if necessary, were purified in the usual manner prior to use. Thin-layer chromatography (TLC) was done with Merck silica gel 60 F₂₅₄ plates, and column chromatography was carried out using Wako gel C-200 as adsorbent.

Synthesis of 6-benzyloxy-1,1,1-trifluoro-6-methyl-2,4-heptadiyne (4e)

To a solution of HMDS (1.6 mL, 7.5 mmol) in THF (50 mL) was added 4.7 mL (7.5 mmol) of *n*-BuLi (1.6 M hexane solution) at $-78\text{ }^\circ\text{C}$ and the whole was stirred for 15 min. To this solution was added 6-benzyloxy-1,1,1,2-tetrafluoro-6-methyl-2-hepten-4-yne **3e** (1.43 g, 5.0 mmol) at $-78\text{ }^\circ\text{C}$ and the whole solution was stirred for 1 h. The reaction mixture was quenched with sat. NH_4Cl aq. and the whole was extracted with Et_2O three times. The combined organic layers were dried over anhydrous Na_2SO_4 , then filtered. The filtrate was evaporated to give the crude materials, which was purified by silica gel column chromatography, affording the 6-benzyloxy-1,1,1-trifluoro-6-methyl-2,4-heptadiyne **4e** as yellow liquid (1.26 g, 4.74 mmol, 95% yield).

^1H NMR (CDCl_3) δ = 1.59 (s, 6H), 4.62 (s, 2H), 7.28–7.36 (m, 5H); ^{13}C NMR (CDCl_3) δ = 2.07, 28.16, 53.34, 64.04 (q, J = 53.71 Hz), 66.06 (q, J = 3.31 Hz), 66.63, 67.18, 70.70 (q, J = 6.63 Hz), 70.92, 87.24, 113.79 (q, J = 257.36 Hz), 127.67, 127.71, 128.41, 138.034; ^{19}F NMR (CDCl_3) δ = -50.38 (s, 3F); IR (neat) : 3033, 2989, 2868, 2266, 2171, 1467, 1383, 1321, 1149, 1054 cm^{-1} ; HRMS (EI) calcd for $\text{C}_{15}\text{H}_{13}\text{F}_3\text{O}$ (M^+) 266.0918, found 266.0915.

Carbocupration of 6-benzyloxy-1,1,1-trifluoro-6-methyl-2,4-heptadiyne (4e)

To a solution of copper cyanide (54 mg, 0.6 mmol) in THF (2.0 mL) was added 0.75 mL (1.2 mmol) of *n*-BuLi (1.6 M hexane solution) at -45 °C and the whole was stirred for 10 min, then allowed to warm to -20 °C and stirred for 30 min. To this solution was added dropwise a solution of 6-benzyloxy-1,1,1-trifluoro-6-methyl-2,4-heptadiyne **4e** (133 mg, 0.5 mmol) in THF (0.5 mL). The reaction was stirred for 1 h at -45 °C, and was then quenched with NH₃ aq./MeOH (1 mL/5 mL) and extracted with Et₂O three times. The combined organic layers were dried over anhydrous Na₂SO₄ and concentrated *in vacuo*. The residue was chromatographed on silica gel to afford (*Z*)-6-benzyloxy-3-*n*-butyl-1,1,1-trifluoro-6-methyl-2-hepten-4-yne **5** (0.098 g, 0.30 mmol, 61% yield).

¹H NMR (CDCl₃) δ = 0.80–2.15 (m, 15H), 4.54 (s, 2H), 5.67 (q, *J* = 7.59 Hz, 1H), 7.17–7.28 (m, 5H); ¹³C NMR (CDCl₃) δ = 13.78, 21.84, 22.64, 28.53 (q, *J* = 1.61 Hz), 29.75, 30.34, 37.48, 66.78 (t, *J* = 2.06 Hz), 71.04, 79.67 (q, *J* = 1.66 Hz), 101.68 (q, *J* = 1.71 Hz), 122.07 (qd, *J* = 33.89, 2.46 Hz), 122.36 (q, *J* = 270.01 Hz), 127.42, 127.79, 134.21 (q, *J* = 5.79 Hz), 138.82; ¹⁹F NMR (CDCl₃) δ = -60.37 (d, *J* = 7.52 Hz, 3F); IR (neat) : 3066, 2934, 2864, 2224, 1642, 1497, 1455, 1379, 1123, 1056 cm⁻¹; HRMS (FAB) calcd for C₁₉H₂₃F₃NaO (M+Na) 347.1599, found 347.1601.

Preparation of (*E*)-6-benzyloxy-3-*n*-butyl-1,1,1-trifluoro-2-iodo-6-methyl-2-hepten-4-yne (13a)

To a solution of copper cyanide (54 mg, 0.6 mmol) in THF (2.0 mL) was added 0.75 mL (1.2 mmol) of *n*-BuLi (1.6 M hexane solution) at -45 °C and the whole was stirred for 10 min, then allowed to warm to -20 °C and stirred for 30 min. To this solution was added dropwise a solution of 6-benzyloxy-1,1,1-trifluoro-6-methyl-2,4-heptadiyne **4e** (133 mg, 0.5 mmol) in THF (0.5 mL). The reaction was stirred for 1 h at -45 °C. After stirring at that temperature for 1 h, iodine (609 mg, 2.4 mmol) was added to the reaction mixture. After 1 h, the reaction was quenched with NH₃ aq./MeOH (1 mL/5 mL), extracted with Et₂O three times. The combined organic layers were dried over anhydrous Na₂SO₄ and concentrated *in vacuo*. The residue was chromatographed on silica gel to afford (*E*)-6-benzyloxy-3-*n*-butyl-1,1,1-trifluoro-2-iodo-6-methyl-2-hepten-4-yne **13a** (0.121 g, 0.27 mmol, 54% yield).

¹H NMR (CDCl₃) δ = 0.92–2.51 (m, 15H), 4.61 (s, 2H), 7.26–7.36 (m, 5H); ¹³C NMR (CDCl₃) δ = 2.10, 13.87, 22.06, 28.38, 28.39, 29.37, 43.08, 66.84 (t, *J* = 2.05 Hz), 71.12, 79.22, 93.75 (q, *J* = 34.44 Hz), 102.69 (q, *J* = 2.21 Hz), 120.77 (q, *J* = 2.21 Hz), 127.45, 127.71, 128.31, 138.08 (q, *J* = 3.58 Hz), 138.69, 140.70; ¹⁹F NMR (CDCl₃) δ = -57.12 (s, 3F); IR (neat) : 3090, 3032, 2960, 2933, 2864, 2220, 1947, 1806, 1710, 1643, 1577, 1455, 1380, 1240, 1159, 1057 cm⁻¹; HRMS (FAB) calcd for C₁₉H₂₂F₃INaO (M+Na) 473.0565, found 473.0565.

(E)-6-Benzyloxy-1,1,1-trifluoro-2-iodo-3,6-dimethyl-2-hepten-4-yne (13b)

¹H NMR (CDCl₃) δ = 1.57 (s, 6H), 2.22 (s, 3H), 4.60 (s, 2H), 7.25–7.36 (m, 5H); ¹³C NMR (CDCl₃) δ = 28.36, 30.98 (q, *J* = 1.64 Hz), 66.80 (t, *J* = 2.06 Hz), 71.07, 80.11, 94.47 (q, *J* = 34.70 Hz), 102.18, 102.20, 120.77 (q, *J* = 273.02 Hz), 127.46, 127.72, 128.32, 128.99, 129.76, 133.67 (q, *J* = 3.58 Hz), 134.48, 138.72; ¹⁹F NMR (CDCl₃) δ = –57.50 (s, 3F); IR (neat): 3033, 2986, 1703, 1454, 1296, 1241, 1131, 1056 cm^{–1}; HRMS (FAB) calcd for C₁₆H₁₆F₃INaO (M+Na) 431.0096, found 431.0087.

(E)-6-Benzyloxy-3-cyclohexyl-1,1,1-trifluoro-2-iodo-6-methyl-2-hepten-4-yne (13c)

¹H NMR (CDCl₃) δ = 1.32–1.83 (m, 17H), 4.65 (s, 2H), 7.26–7.40 (m, 5H); ¹³C NMR (CDCl₃) δ = 2.11, 25.59, 25.72, 25.89, 31.42, 28.45, 28.47, 28.58, 30.21, 45.71, 50.86, 66.95 (t, *J* = 2.51 Hz), 71.11, 71.24, 92.72 (q, *J* = 34.17 Hz), 103.72 (q, *J* = 2.48 Hz), 120.83 (q, *J* = 273.59 Hz), 127.48, 127.75, 128.33, 142.25 (q, *J* = 3.31 Hz); ¹⁹F NMR (CDCl₃) δ = –56.45 (s, 3F); IR (neat): 3584, 3032, 2932, 2856, 1569, 1451, 1288, 1129, 1056 cm^{–1}; HRMS (FAB) calcd for C₂₁H₂₄F₃INaO (M+Na) 499.0722, found 499.0723.

(E)-6-Benzyloxy-1,1,1-trifluoro-2-iodo-6-methyl-3-phenyl-2-hepten-4-yne (13d)

¹H NMR (CDCl₃) δ = 1.56 (s, 6H), 4.56 (s, 2H), 7.26–7.43 (m, 10H); ¹³C NMR (CDCl₃) δ = 28.25, 28.53, 66.90 (t, *J* = 1.66 Hz), 71.19, 80.11, 94.91 (q, *J* = 34.44 Hz), 104.68 (q, *J* = 2.21 Hz), 121.11 (q, *J* = 273.29 Hz), 127.45, 127.82, 128.29, 128.53, 129.04, 130.00, 130.40, 138.01 (q, *J* = 3.85 Hz), 138.58, 138.70, 141.73; ¹⁹F NMR (CDCl₃) δ = –57.74 (s, 3F); IR (neat): 3584, 2985, 1563, 1454, 1299, 1248, 1132, 1053 cm^{–1}; HRMS (FAB) calcd for C₂₁H₁₈F₃INaO (M+Na) 493.0252, found 493.0251.

(E)-6-Benzyloxy-1,1,1-trifluoro-2-iodo-6-methyl-3-*p*-methoxyphenyl-2-hepten-4-yne (13e)

¹H NMR (CDCl₃) δ = 1.59 (s, 6H), 3.86 (s, 3H), 4.59 (s, 2H), 6.93–7.52 (m, 9H); ¹³C NMR (CDCl₃) δ = 28.26, 55.26, 55.28, 66.87 (t, *J* = 2.06 Hz), 71.19, 80.32, 93.67 (q, *J* = 34.17 Hz), 104.21 (q, *J* = 2.48 Hz), 113.67, 114.12, 121.18 (q, *J* = 273.59 Hz), 127.43, 127.69, 127.80, 128.27, 129.62, 133.42, 133.70, 137.59 (q, *J* = 3.58 Hz), 138.62, 158.64, 160.07; ¹⁹F NMR (CDCl₃) δ = –57.35 (s, 3F); IR (neat) : 3584, 3033, 2985, 2936, 2839, 2212, 1888, 1606, 1508, 1249, 1130, 1041 cm^{–1}; HRMS (FAB) calcd for C₂₂H₂₀F₃IO₂ (M+) 500.0460, found 500.0463.

Typical procedure for the preparation of 2-benzyloxy-2-methyl-6-trifluoromethyl-5-tetradecen-3,7-diyne (14a)

To a solution of (*E*)-6-benzyloxy-3-*n*-butyl-1,1,1-trifluoro-2-iodo-6-methyl-2-hepten-4-yne **13a** (0.450 g, 1.0 mmol), 1-octyne (0.220 g, 2.0 mmol), CuI (0.019 g, 0.10 mmol) in THF (10 mL) was

added Pd(PPh₃)₄ (0.116 mg, 0.10 mmol) followed by Et₃N (5.6 mL, 40 mmol) at room temperature. The whole was allowed to warm to 70 °C, then stirred 2 h, and then quenched with sat. NH₄Cl aq. The mixture was extracted with Et₂O three times, and the combined organic layers were dried over anhydrous Na₂SO₄, and then filtered. The filtrate was evaporated to give the corresponding crude materials, which were purified by silica-gel column chromatography, affording the (Z)-2-benzyloxy-6-*n*-butyl-2-methyl-6-trifluoromethyl-5-pentadecen-3,7-diyne **14a** (0.301 g, 0.70 mmol, 70% yield.)

¹H NMR (CDCl₃) δ = 0.88–2.53 (m, 28H), 4.62 (s, 2H), 7.29–7.38 (m, 5H); ¹³C NMR (CDCl₃) δ = 13.90 (d, *J* = 12.35 Hz), 19.77, 22.29 (d, *J* = 43.87 Hz), 28.26, 28.46 (d, *J* = 5.82 Hz), 29.81, 31.24, 35.86, 66.82 (t, *J* = 2.05 Hz), 71.23, 73.60 (q, *J* = 2.51 Hz), 80.99, 103.50, 105.54 (q, *J* = 1.61 Hz), 119.23 (q, *J* = 33.03 Hz), 121.53 (q, *J* = 274.43 Hz), 127.39, 127.76, 128.27, 128.42, 128.48, 128.67, 133.70 (d, *J* = 18.98 Hz), 136.15 (q, *J* = 2.48 Hz), 137.13 (d, *J* = 1.71 Hz), 137.23, 138.83; ¹⁹F NMR (CDCl₃) δ = –61.58 (s, 3F); IR (neat) : 3032, 2958, 2932, 2861, 2222, 1585, 1347, 1160, 1131, 1057 cm^{–1}; HRMS (FAB) calcd for C₂₇H₃₅F₃NaO (M+Na) 455.2538, found 455.2542.

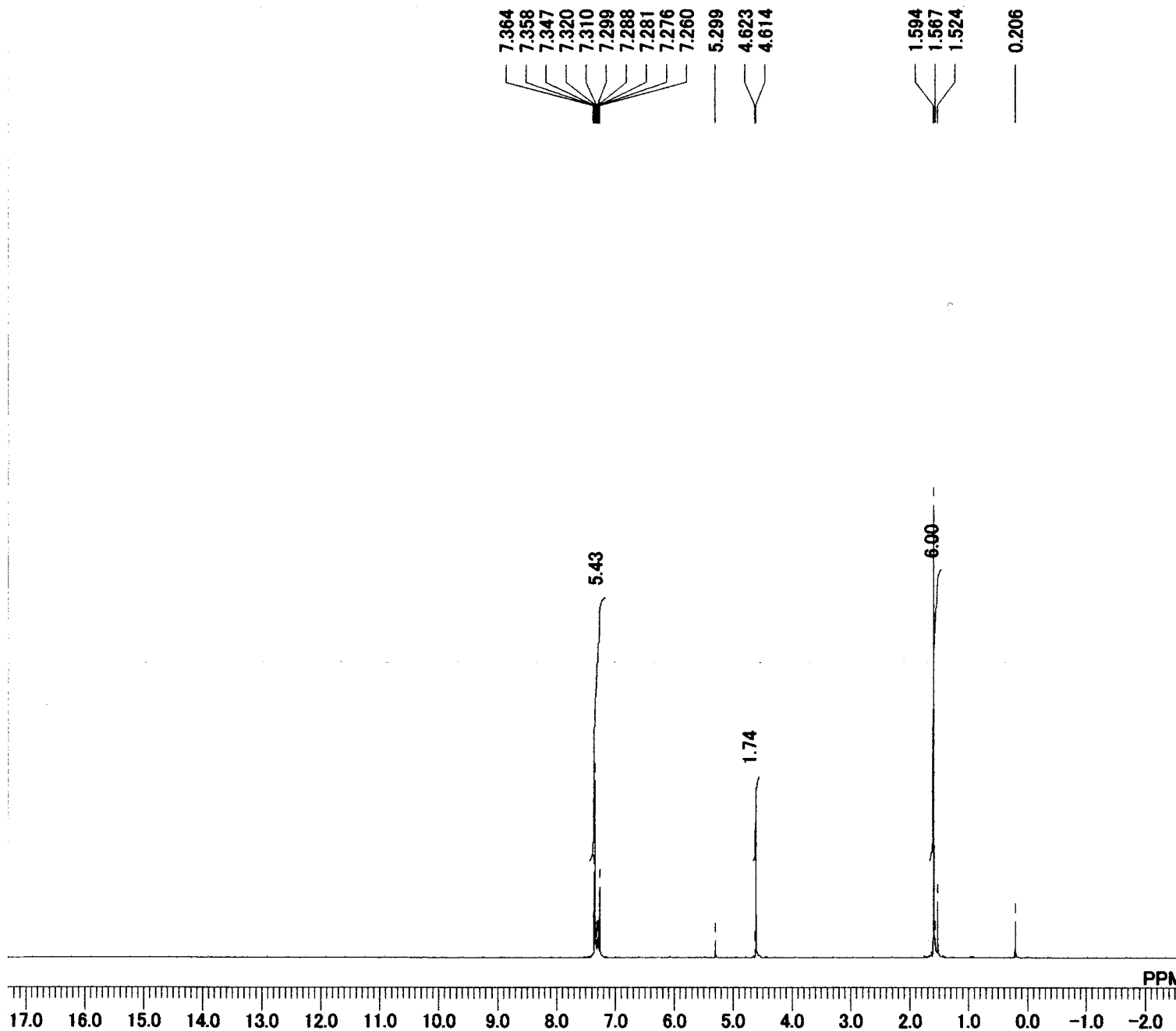
(Z)-7-Benzyloxy-4-*n*-butyl-7-methyl-1-phenyl-3-trifluoromethyl-3-octen-1,5-diyne (14b)

¹H NMR (CDCl₃) δ = 0.91–2.63 (m, 15H), 4.64 (s, 2H), 7.26–7.48 (m, 10H); ¹³C NMR (CDCl₃) δ = 13.89, 15.26, 22.11, 28.49, 29.90, 36.17, 65.85, 66.89 (t, *J* = 2.46 Hz), 71.28, 81.10, 82.07 (q, *J* = 2.48 Hz), 101.43, 106.81 (q, *J* = 1.91 Hz), 118.98 (q, *J* = 33.63 Hz), 121.43 (q, *J* = 274.43 Hz), 122.26, 127.45, 127.79, 128.32, 128.45, 129.09, 131.45, 137.60 (q, *J* = 2.48 Hz), 138.76; ¹⁹F NMR (CDCl₃) δ = –61.27 (s, 3F); IR (neat): 3033, 2959, 2932, 2862, 2214, 1576, 1491, 1355, 1235, 1133, 1058, 1000 cm^{–1}; HRMS (FAB) calcd for C₂₇H₂₇F₃NaO (M+Na) 447.1912, found 447.1909.

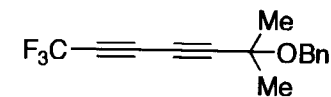
(Z)-7-Benzyloxy-4-*n*-butyl-7-methyl-3-trifluoromethyl-1-trimethylsilyl-3-octen-1,5-diyne (14c)

¹H NMR (CDCl₃) δ = 0.25 (s, 9H), 0.93–1.63 (m, 13H), 7.19 (t, *J* = 7.19 Hz, 2H), 4.63 (s, 2H), 7.26–7.38 (m, 5H); ¹³C NMR (CDCl₃) δ = –0.40, 13.80, 22.04, 28.44, 29.71, 36.09, 43.11, 66.87 (t, *J* = 2.06 Hz), 71.25, 80.89, 96.61 (q, *J* = 2.48 Hz), 107.15 (q, *J* = 1.67 Hz), 108.19, 118.97 (q, *J* = 33.60 Hz), 121.22 (q, *J* = 274.16 Hz), 127.45, 127.78, 128.31, 129.26, 138.74, 139.11 (q, *J* = 2.74 Hz); ¹⁹F NMR (CDCl₃) δ = –61.38 (s, 3F); IR (neat): 3033, 2960, 2933, 2863, 2144, 1578, 1455, 1339, 1253, 1134, 1057 cm^{–1}; HRMS (FAB) calcd for C₂₄H₃₁F₃NaOSi (M+Na) 443.1994, found 443.1992.

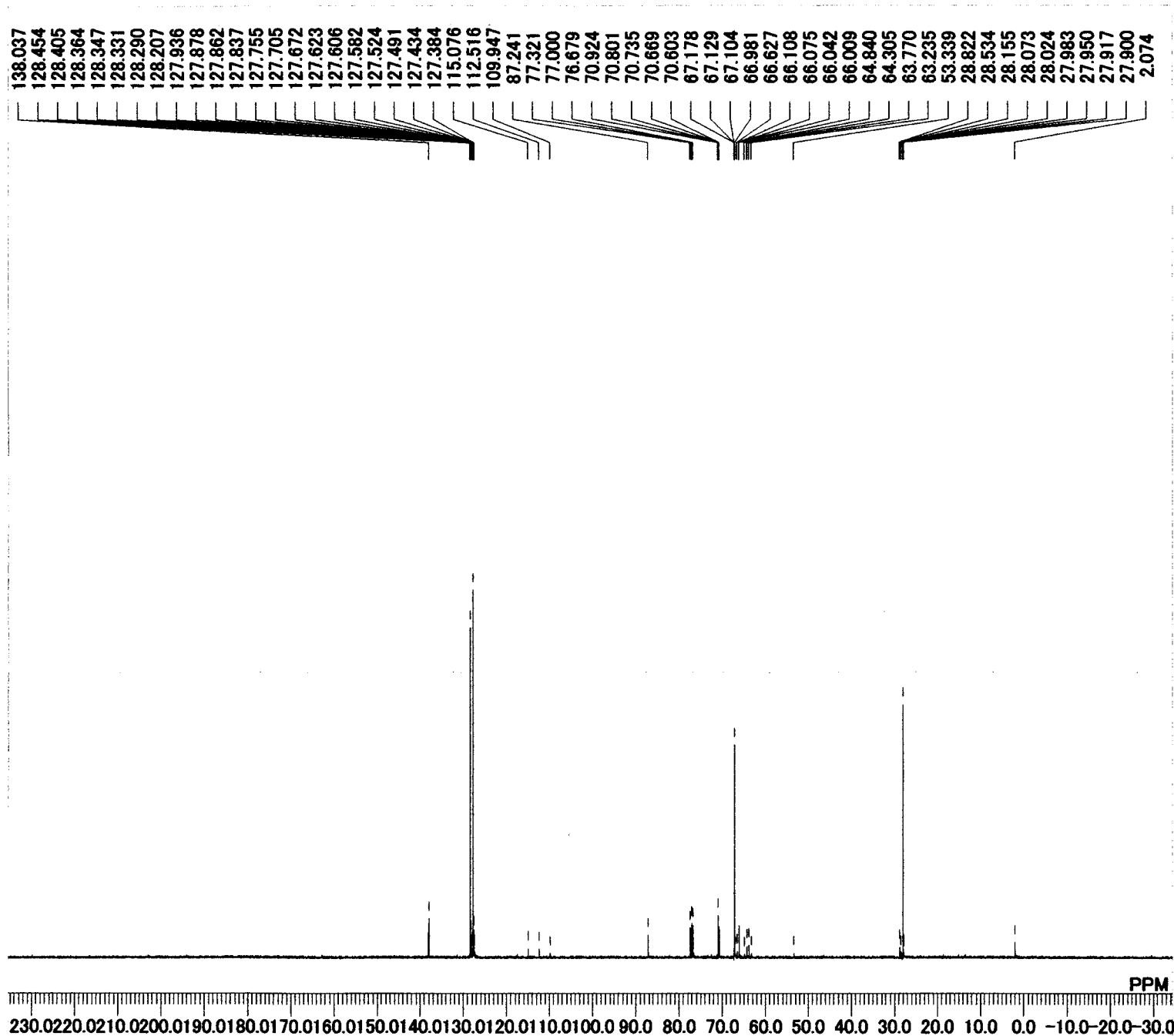
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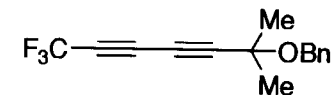
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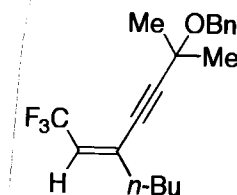
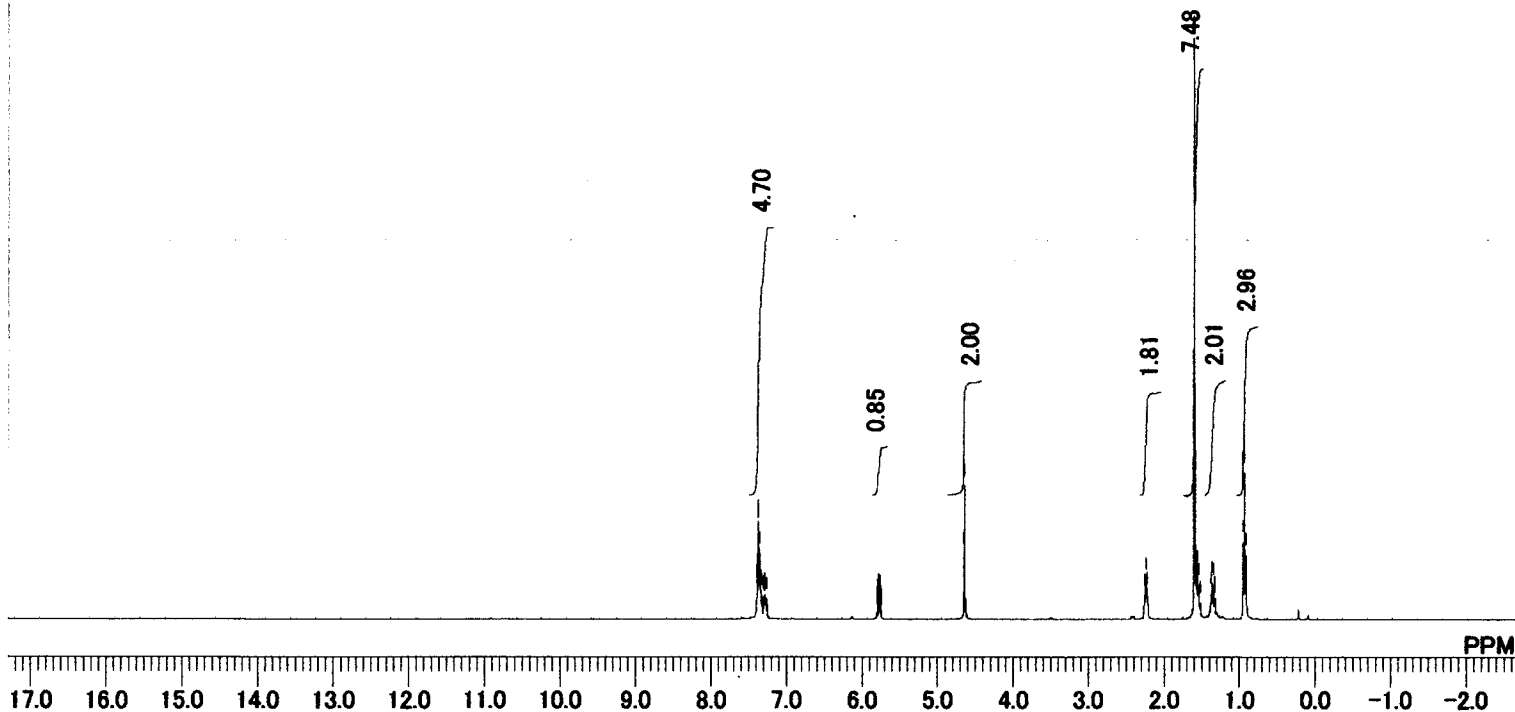


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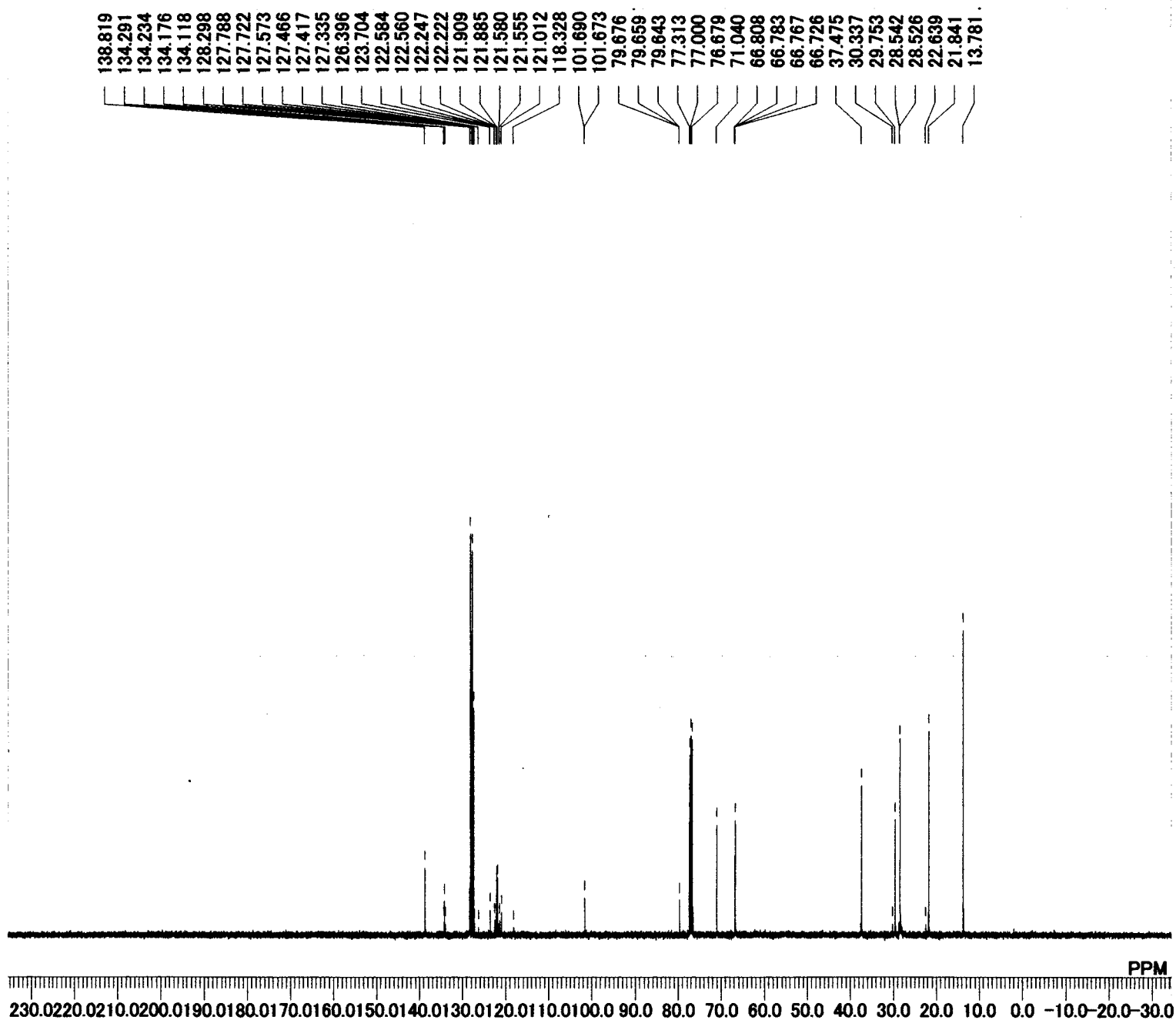
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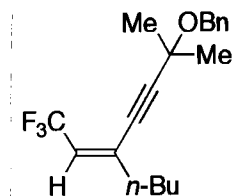
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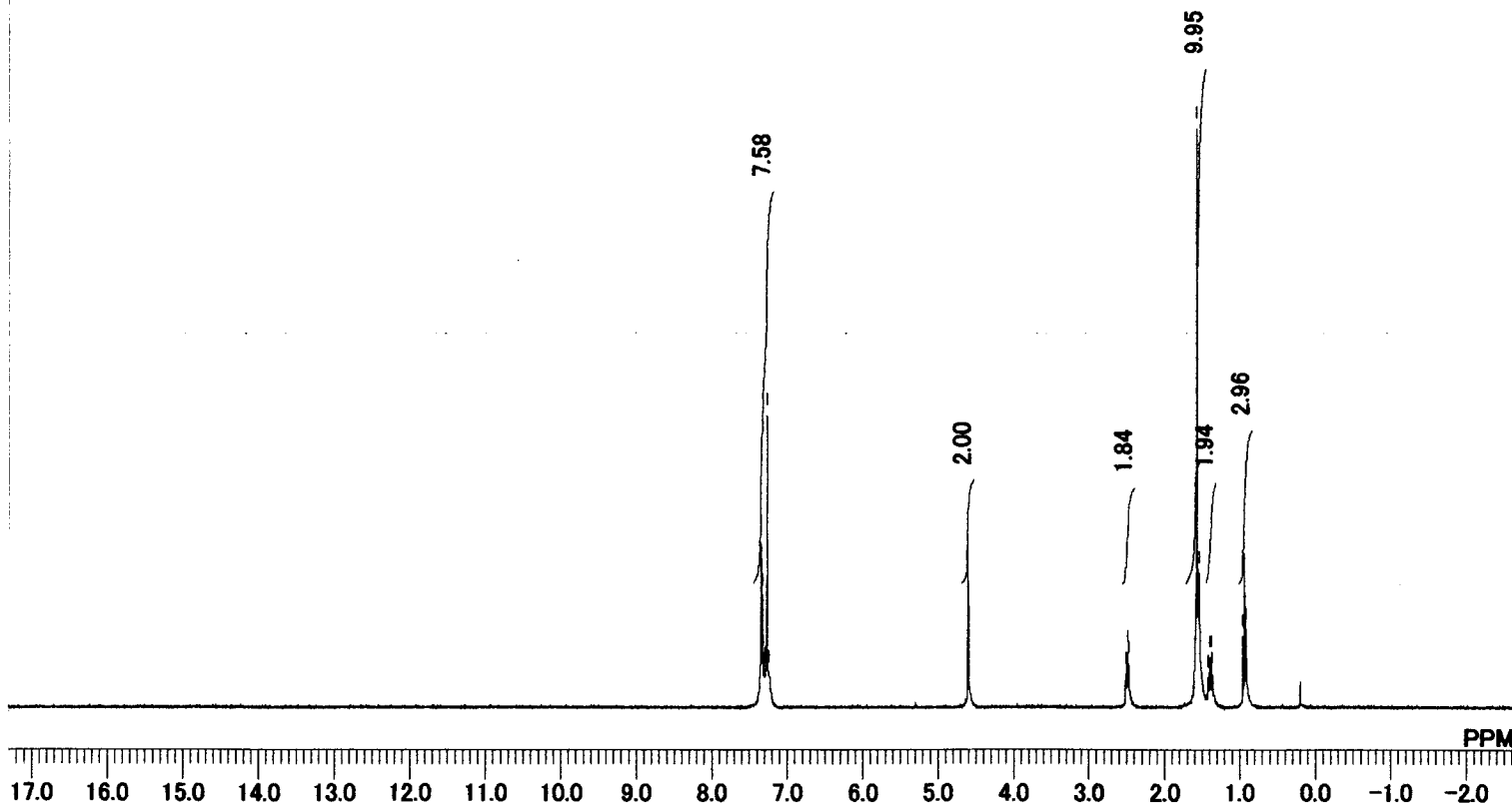
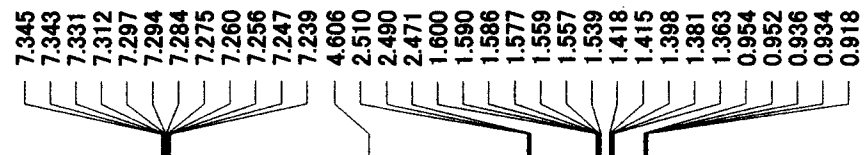
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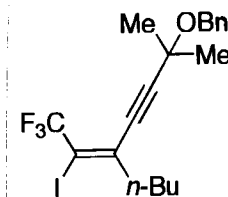
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LKSET	61.60 KHz
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FILDC	
FILDF	



auto



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T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM_PI
IRNUC	1H
IFR	399.65 MHz
IRSET	136.90 KHz
IRFIN	97.50 Hz
IRRPW	45 usec
IRATN	511
DFILE	Rxn376 カラム H.als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	25
LKPHS	193
LKSIG	683
CSPED	12 Hz
FILDC	
FILDF	



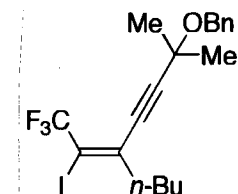
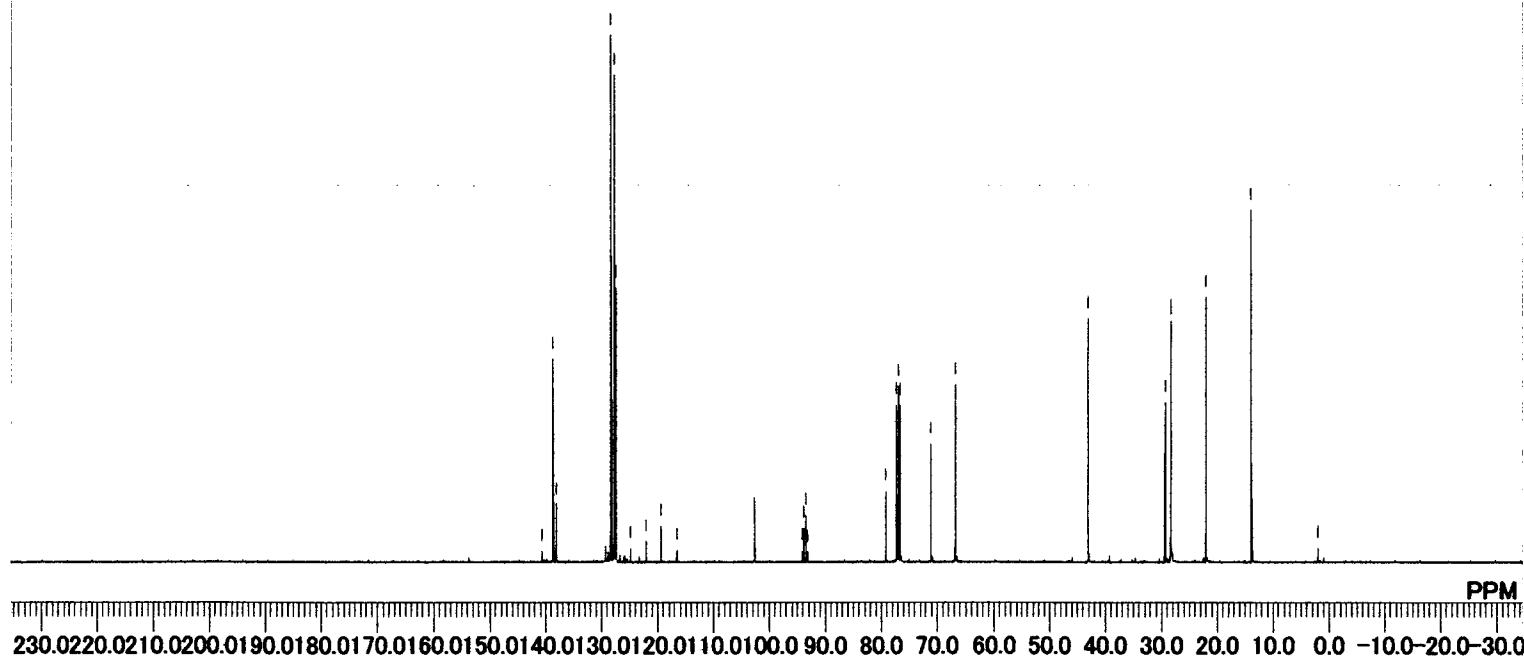
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138.029
128.306
127.705
127.450
124.848
122.123
119.407
116.682
102.719
102.702
102.678
102.653
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93.581
93.235
79.223
77.321
77.000
76.679
71.122
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66.841
66.816

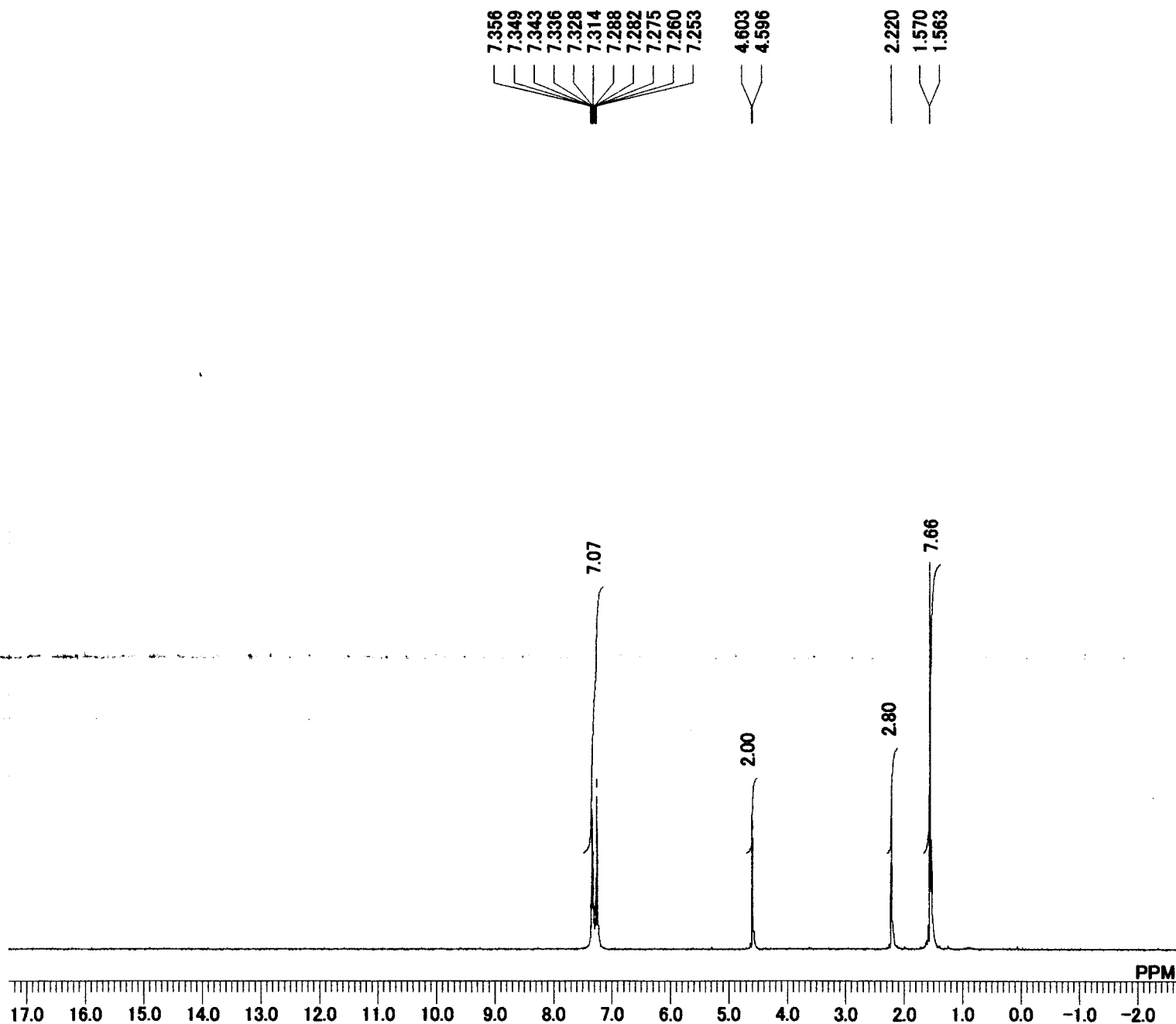
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2.099

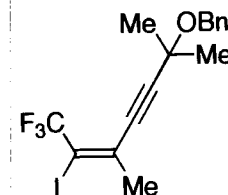
MENUF 13C
OBNUC 13C
OFR 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
PW1 6.20 usec
DEADT 19.00 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 32768
SPO 32768
TIMES 20000
DUMMY 1
FREQU 27118.64 Hz
FLT 13550 Hz
DELAY 14.80 usec
ACQTM 1.2083 sec
PD 1.7920 sec
ADBIT 16
RGAIN 25
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD BCM
EXPCM Bilevel.complete.decoupling:Set_IRRF
IRNUC 1H
IFR 399.65 MHz
IRSET 124.00 KHz
IRFIN 10500.00 Hz
IRRPW 45 usec
IRATN 511
DFILE Rxn469 Cu→I C.als
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 24
LKPHS 240
LKSIG 985
CSPED 10 Hz
FILDC
FILDF



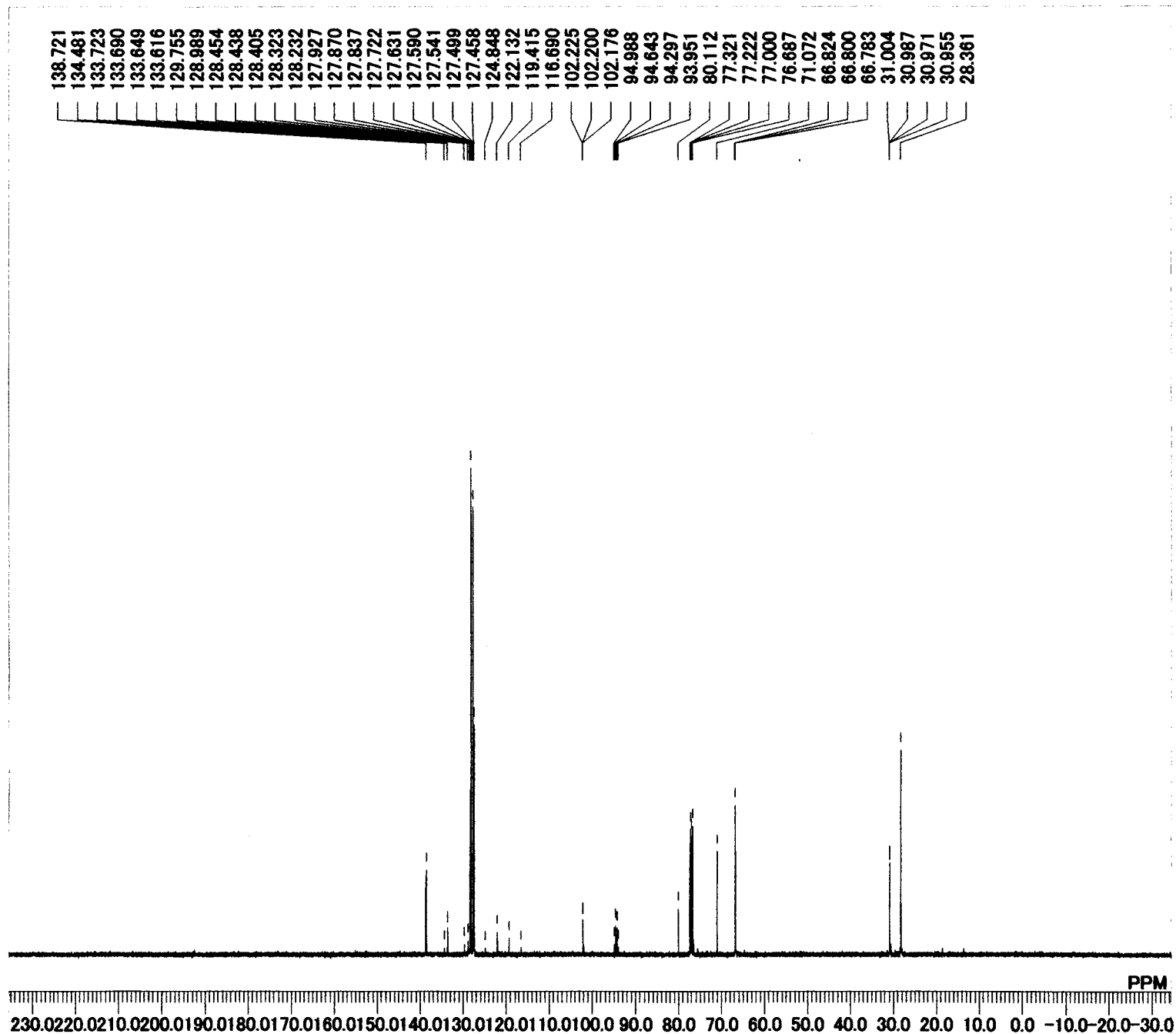
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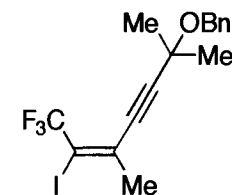
MENUF 1H
OBNUC 1H
OFR 399.65 MHz
OBSET 135.40 KHz
OBFIN 24.90 Hz
PW1 5.80 usec
DEADT 72.10 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7992.01 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 2.0501 sec
PD 4.9500 sec
ADBIT 16
RGAIN 21
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM_PI
IRNUC 1H
IFR 399.65 MHz
IRSET 136.90 KHz
IRFIN 97.50 Hz
IRRPW 45 usec
IRATN 511
DFILE 悪.als
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 25
LKPHS 193
LKSIG 679
CSPED 11 Hz
FILDC
FILDF



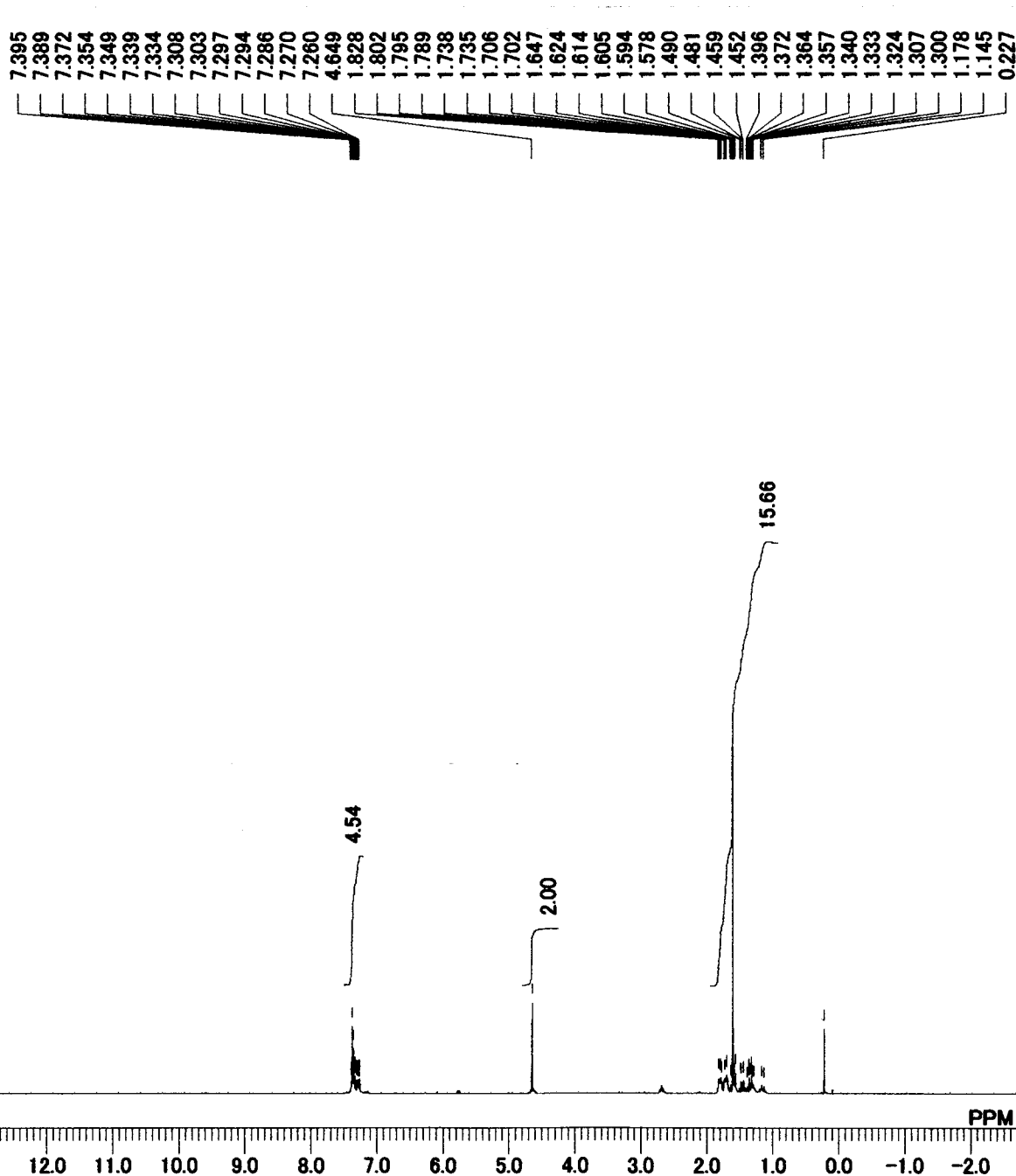
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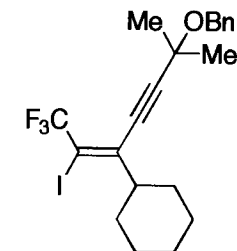
MENUF	13C
OBNUC	13C
OFR	100.40 MHz
OBSET	125.00 KHz
OBFIN	10500.00 Hz
PW1	6.20 usec
DEADT	19.00 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	2000
DUMMY	1
FREQU	27118.64 Hz
FLT	13550 Hz
DELAY	14.80 usec
ACQTM	1.2083 sec
PD	1.7920 sec
ADBIT	16
RGAIN	25
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	BCM
EXPCM	Bilevel.complete.decoupling.Set_IRRF
IRNUC	1H
IFR	399.65 MHz
IRSET	124.00 KHz
IRFIN	10500.00 Hz
IRRPW	45 usec
IRATN	511
DFILE	Rxn377 carbocup Me C.als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	23
LKPHS	240
LKSIG	794
CSPED	11 Hz
FILDC	
FILDF	



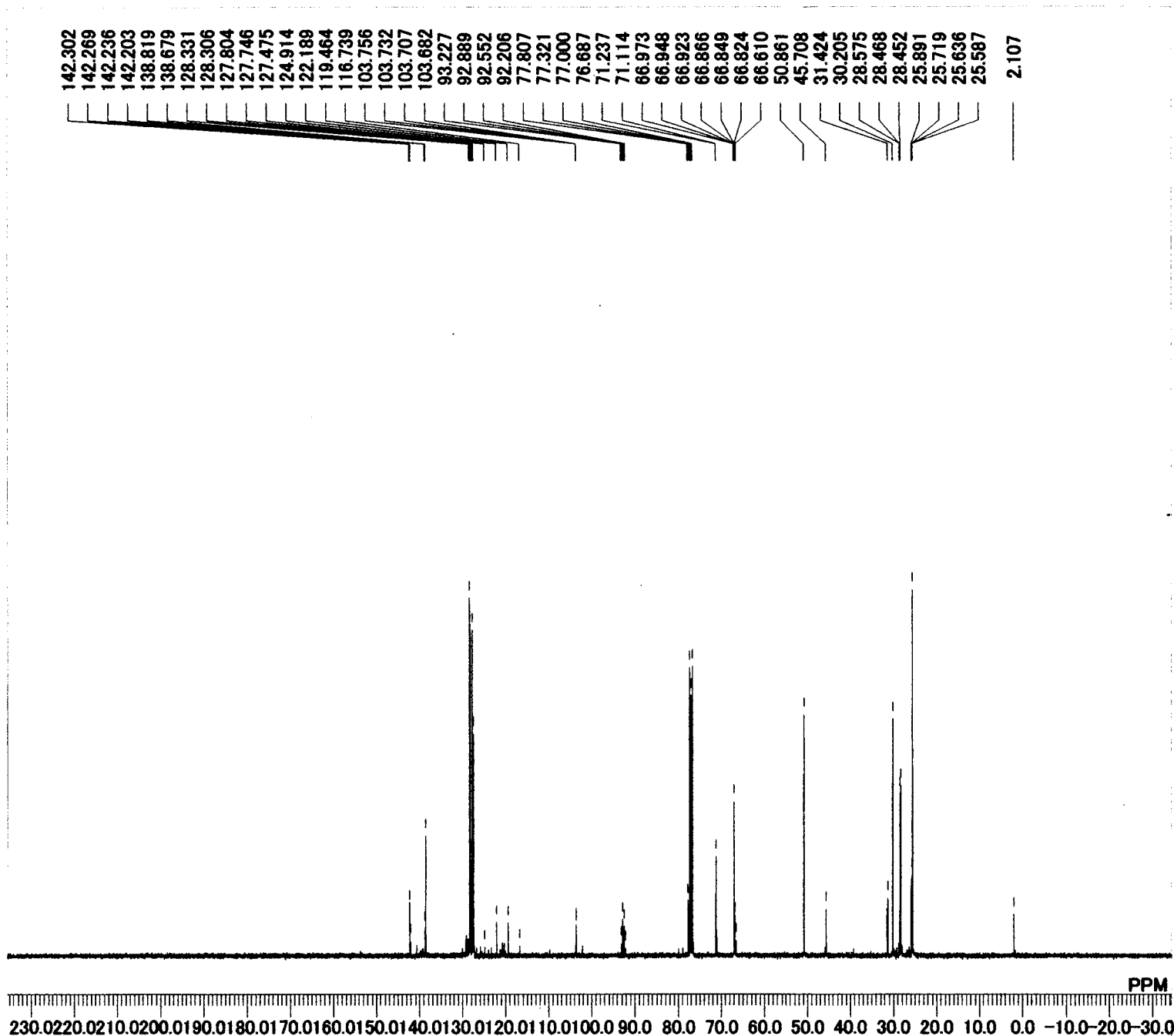
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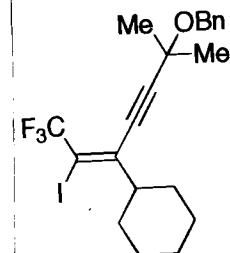
MENUF	1H
OBNUC	1H
OFR	399.65 MHz
OBSET	135.40 KHz
OBFIN	24.90 Hz
PW1	5.50 usec
DEADT	72.20 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7992.01 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	2.0501 sec
PD	4.9500 sec
ADBIT	16
RGAIN	11
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM_PI
IRNUC	1H
IFR	399.65 MHz
IRSET	136.90 KHz
IRFIN	97.50 Hz
IRRPW	45 usec
IRATN	511
DFILE	Rxn481 c-Hex カラム H.als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	25
LKPHS	240
LKSIG	1749
CSPED	14 Hz
FILDC	
FILDF	



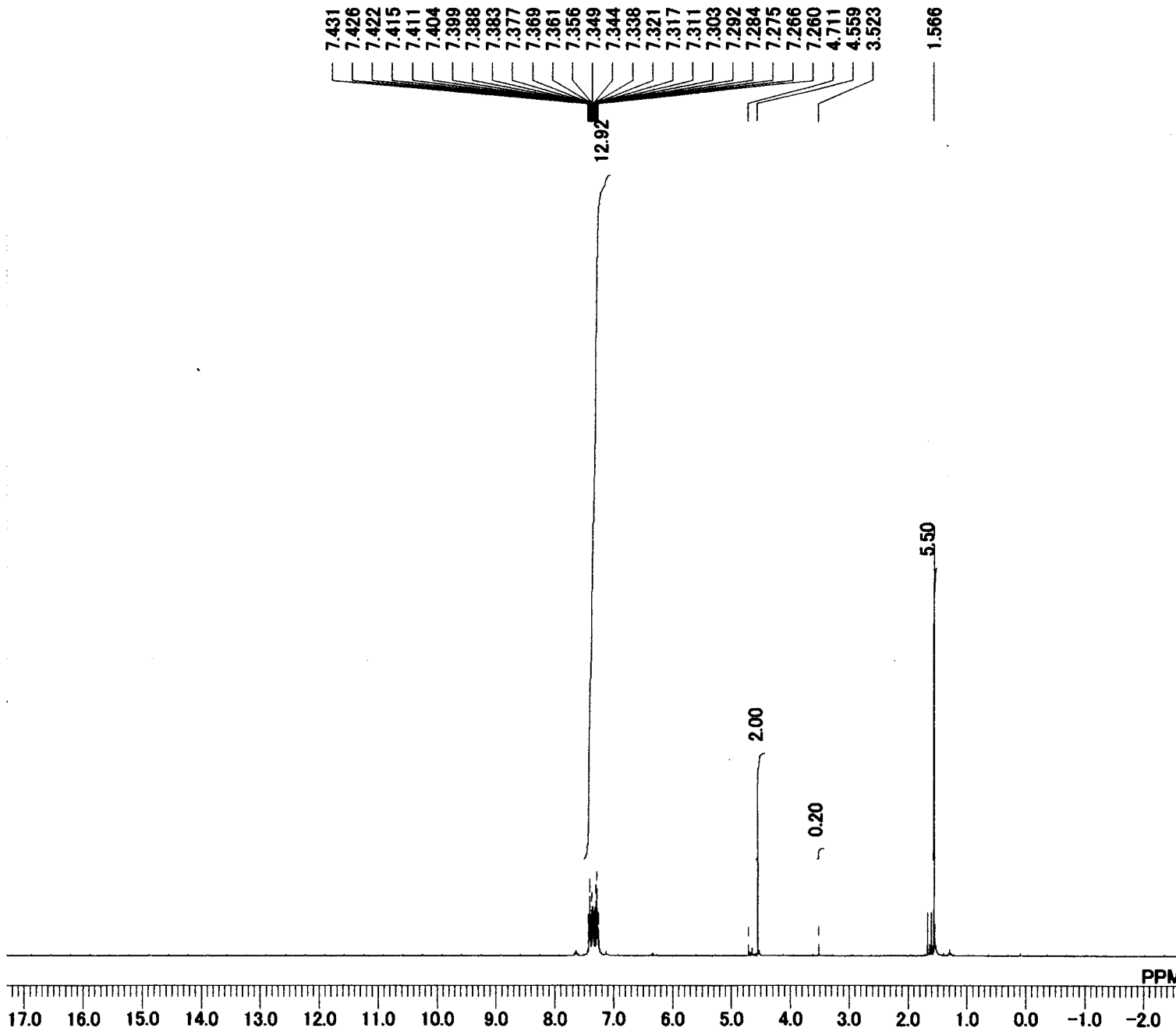
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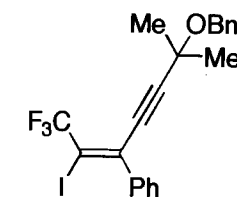
MENUF	13C
OBNUC	13C
OFR	100.40 MHz
OBSET	125.00 KHz
OBFIN	10500.00 Hz
PW1	6.20 usec
DEADT	19.00 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	10000
DUMMY	1
FREQU	27118.64 Hz
FLT	13550 Hz
DELAY	14.80 usec
ACQTM	1.2083 sec
PD	1.7920 sec
ADBIT	16
RGAIN	25
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	BCM
EXPCM	Bilevel.complete.decoupling:Set_IRRF
IRNUC	1H
IFR	399.65 MHz
IRSET	124.00 KHz
IRFIN	10500.00 Hz
IRRPW	45 usec
IRATN	511
DFILE	Rxn481 c-Hex カラム C.als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	24
LKPHS	240
LKSIG	1197
CSPED	13 Hz
FILDC	
FILDF	



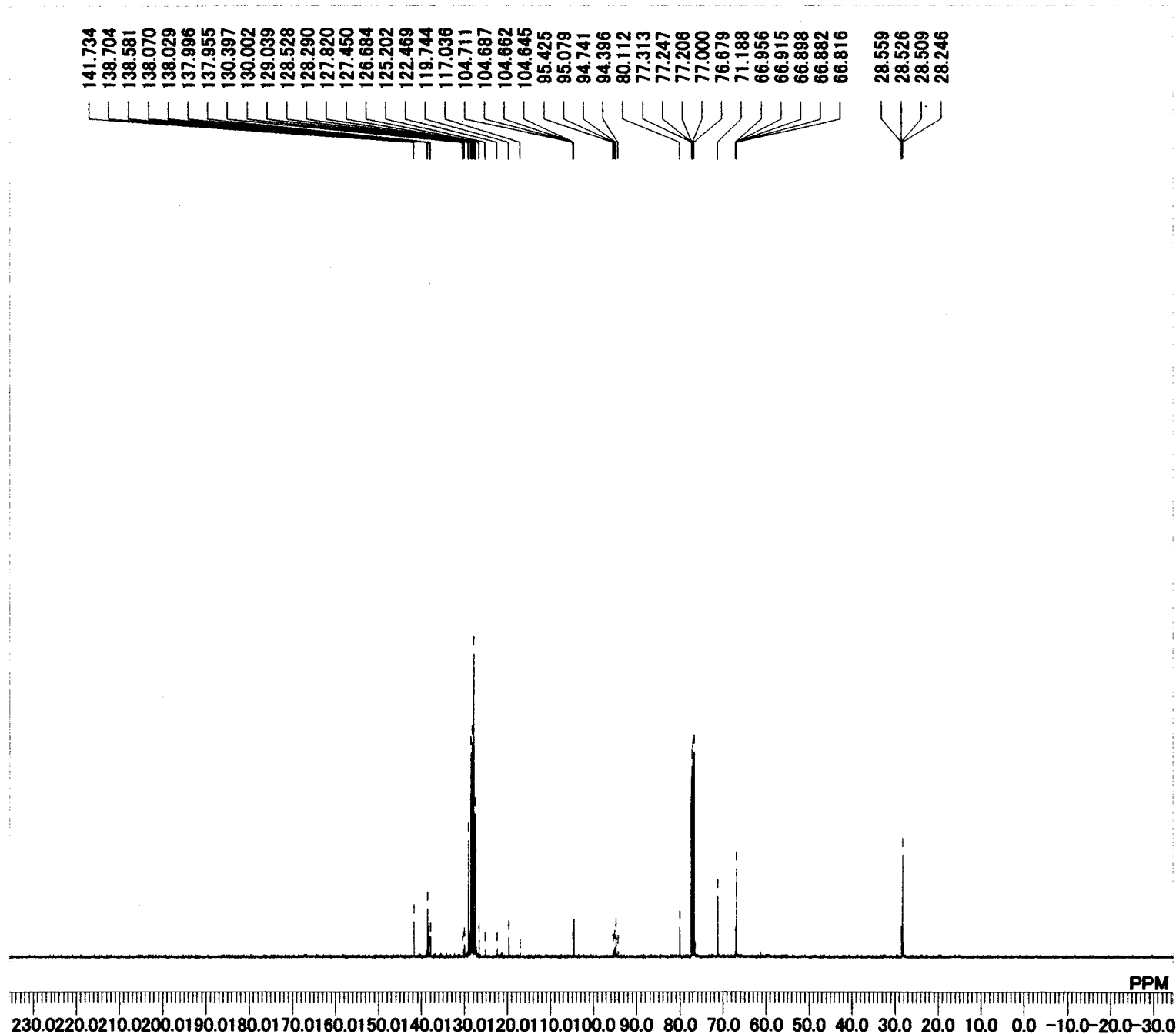
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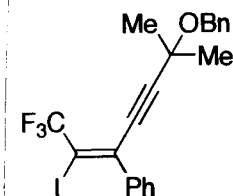
MENUF	1H
OBNUC	1H
OFR	399.65 MHz
OBSET	135.40 KHz
OBFIN	24.90 Hz
PW1	5.50 usec
DEADT	72.20 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7992.01 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	2.0501 sec
PD	4.9500 sec
ADBIT	16
RGAIN	13
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM_PI
IRNUC	1H
IFR	399.65 MHz
IRSET	136.90 KHz
IRFIN	97.50 Hz
IRRPW	45 usec
IRATN	511
DFILE	Rxn480 カラム H.als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	24
LKPHS	240
LKSIG	1274
CSPED	14 Hz
FILDC	
FILDF	



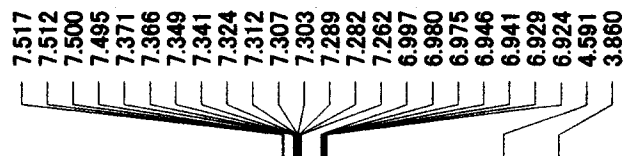
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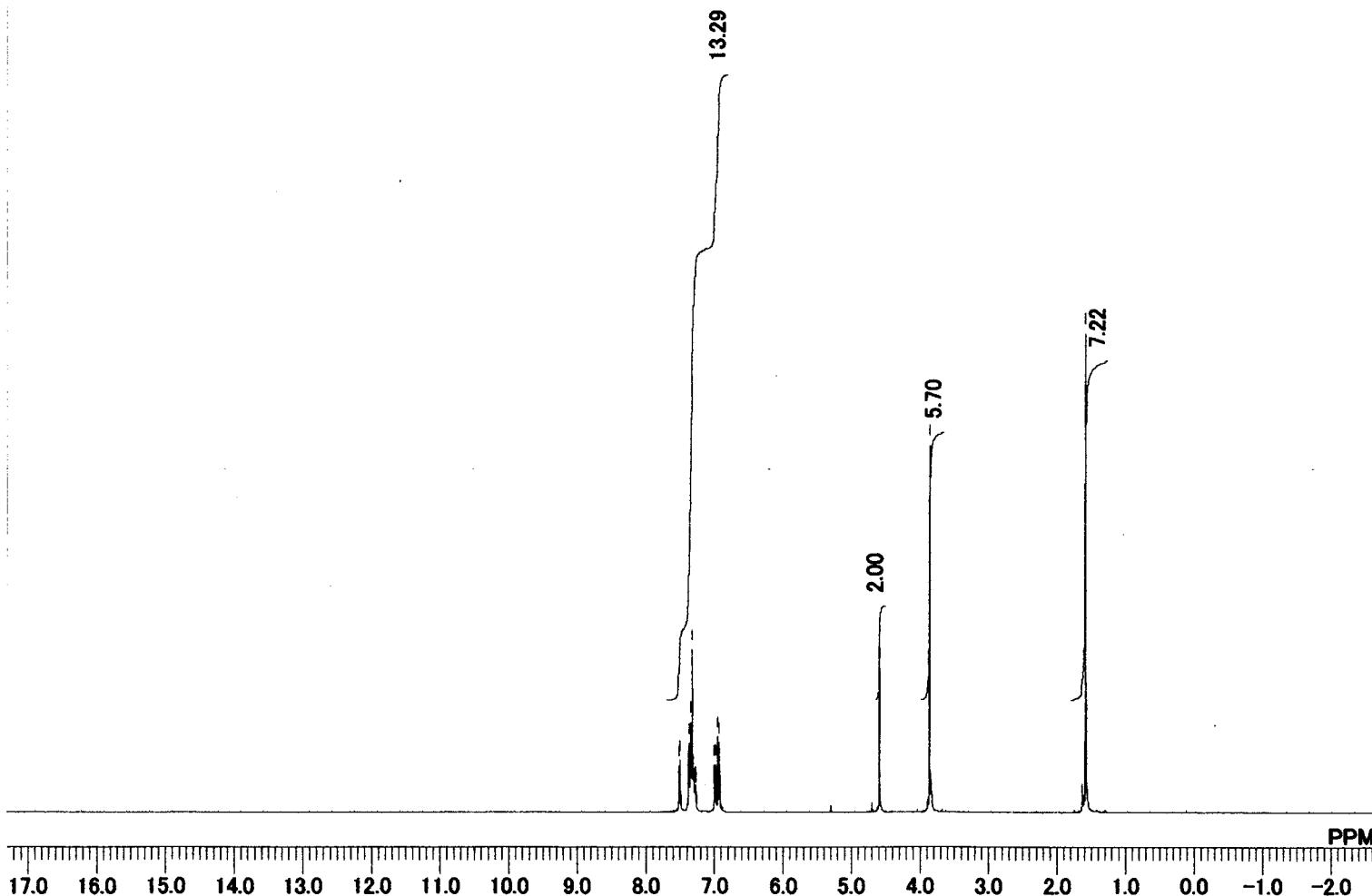
MENUF	13C
OBNUC	13C
OFR	100.40 MHz
OBSET	125.00 KHz
OBFIN	10500.00 Hz
PW1	6.20 usec
DEADT	19.00 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	20000
DUMMY	1
FREQU	27118.64 Hz
FLT	13550 Hz
DELAY	14.80 usec
ACQTM	1.2083 sec
PD	1.7920 sec
ADBIT	16
RGAIN	25
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	BCM
EXPCM	Bilevel.complete.decoupling:Set_IRRF
IRNUC	1H
IFR	399.65 MHz
IRSET	124.00 KHz
IRFIN	10500.00 Hz
IRRPW	45 usec
IRATN	511
DFILE	Rxn.480 13C.als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	24
LKPHS	240
LKSIG	1243
CSPED	14 Hz
FILDC	
FILDF	



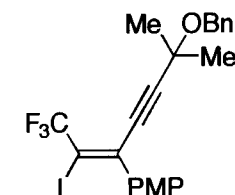
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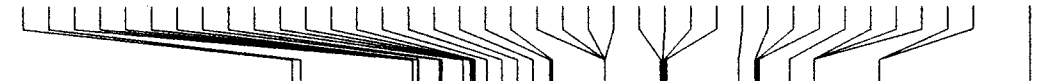


MENUF	1H
OBNUC	1H
OFR	399.65 MHz
OBSET	135.40 KHz
OBFIN	24.90 Hz
PW1	5.50 usec
DEADT	72.20 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7992.01 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	2.0501 sec
PD	4.9500 sec
ADBIT	16
RGAIN	12
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM_PI
IRNUC	1H
IFR	399.65 MHz
IRSET	136.90 KHz
IRFIN	97.50 Hz
IRRPW	45 usec
IRATN	511
DFILE	Rxn483 PMP カラム H.als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	25
LKPHS	240
LKSIG	1200
CSPED	12 Hz
FILDC	
FILDF	

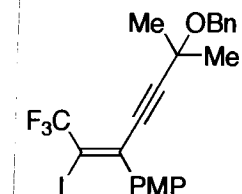


auto

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127.796
127.689
127.425
125.268
122.543
119.818
117.093
114.121
113.668
104.242
104.217
104.193
104.168
94.182
93.844
93.507
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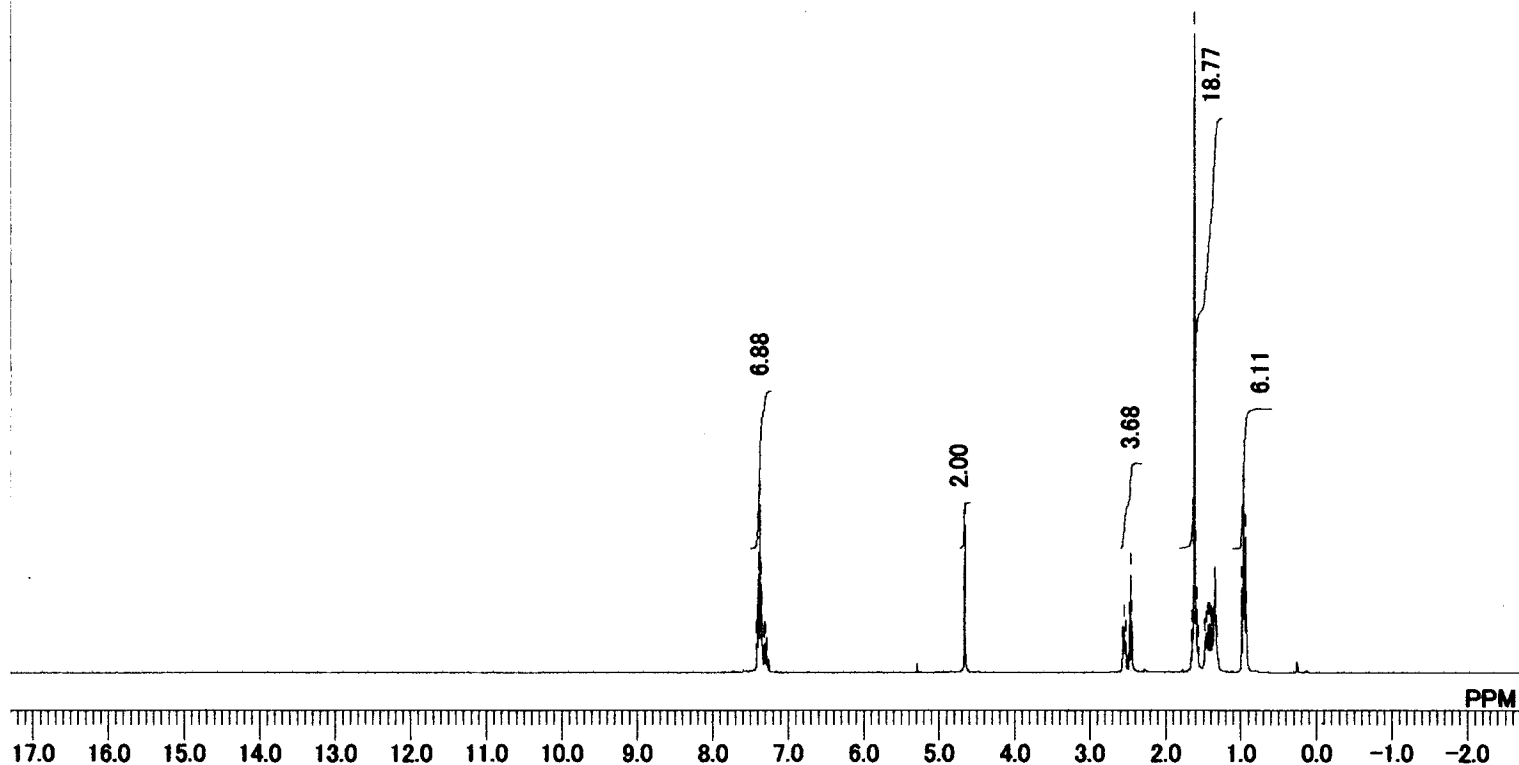
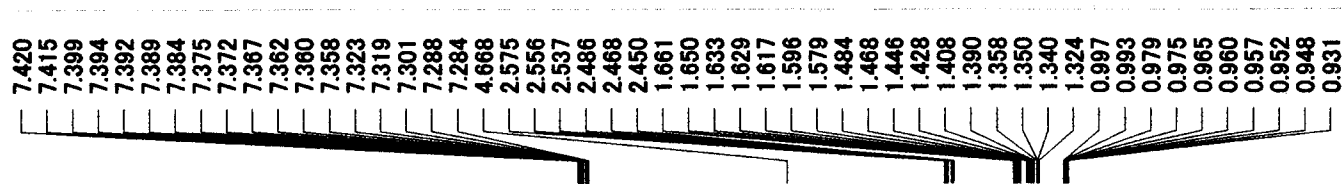


MENUF	13C
OBNUC	13C
OFR	100.40 MHz
OBSET	125.00 KHz
OBFIN	10500.00 Hz
PW1	6.20 usec
DEADT	19.00 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	10000
DUMMY	1
FREQU	27118.64 Hz
FLT	13550 Hz
DELAY	14.80 usec
ACQTM	1.2083 sec
PD	1.7920 sec
ADBIT	16
RGAIN	25
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	BCM
EXPCM	Bilevel.complete.decoupling: Set_IRRF
IRNUC	1H
IFR	399.65 MHz
IRSET	124.00 KHz
IRFIN	10500.00 Hz
IRRPW	45 usec
IRATN	511
DFILE	Rxn483 PMP カラム C.als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	24
LKPHS	240
LKSIG	997
CSPED	12 Hz
FILDC	
FILDF	

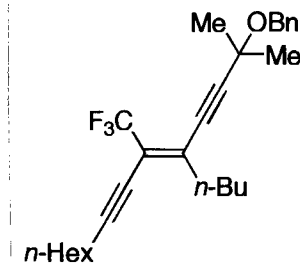


230.0220.0210.0200.0190.0180.0170.0160.0150.0140.0130.0120.0110.0100.090.080.070.060.050.040.030.020.010.00-10.0-20.0-30.0

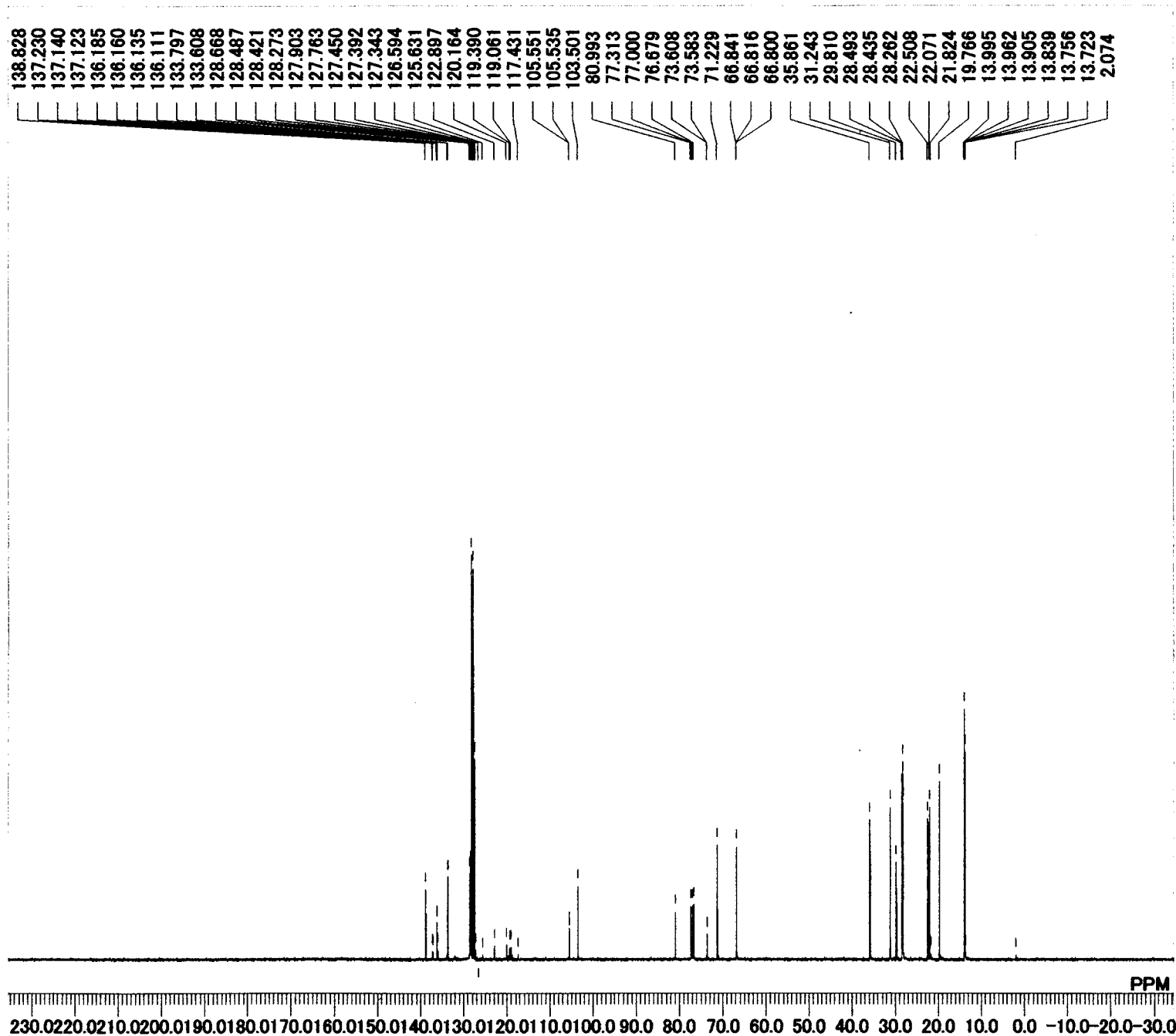
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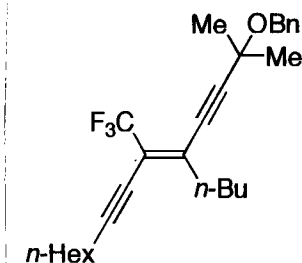
MENUF	1H
OBNUC	1H
OFR	399.65 MHz
OBSET	135.40 KHz
OBFIN	24.90 Hz
PW1	5.50 usec
DEADT	72.20 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7992.01 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	2.0501 sec
PD	4.9500 sec
ADBIT	16
RGAIN	8
BF	0.00 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM_PI
IRNUC	1H
IFR	399.65 MHz
IRSET	136.90 KHz
IRFIN	97.50 Hz
IRRPW	45 usec
IRATN	511
DFILE	Rxn463 sonogashira H.als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	26
LKPHS	240
LKSIG	1692
CSPED	13 Hz
FILDC	
FILDF	



auto

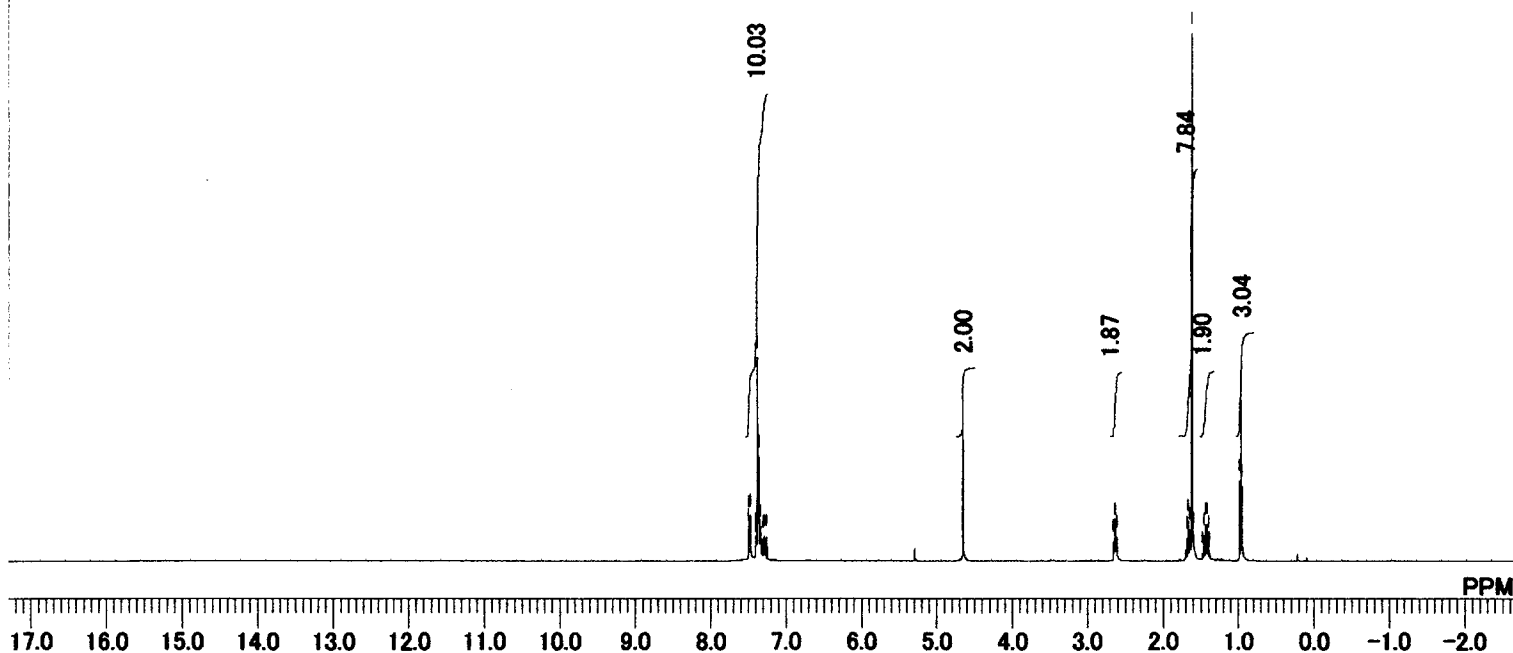


MENUF	13C
OBNUC	13C
OFR	100.40 MHz
OBSET	125.00 KHz
OBFIN	10500.00 Hz
PW1	6.20 usec
DEADT	19.00 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	2000
DUMMY	1
FREQU	27118.64 Hz
FLT	13550 Hz
DELAY	14.80 usec
ACQTM	1.2083 sec
PD	1.7920 sec
ADBIT	16
RGAIN	24
BF	0.00 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	BCM
EXPCM	Bilevel.complete.decoupling:Set_IRRF
IRNUC	1H
IFR	399.65 MHz
IRSET	124.00 KHz
IRFIN	10500.00 Hz
IRRPW	45 usec
IRATN	511
DFILE	Rxn463 sonogashira C.als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	24
LKPHS	240
LKSIG	967
CSPED	13 Hz
FILDC	
FILDF	

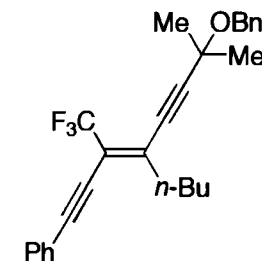


auto

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7.369
7.364
7.343
7.309
7.292
7.260
4.661
2.660
2.656
2.641
2.638
2.622
2.619
1.691
1.673
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0.954



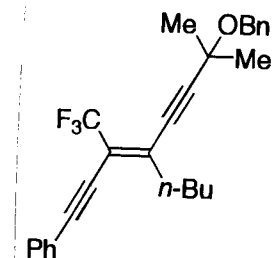
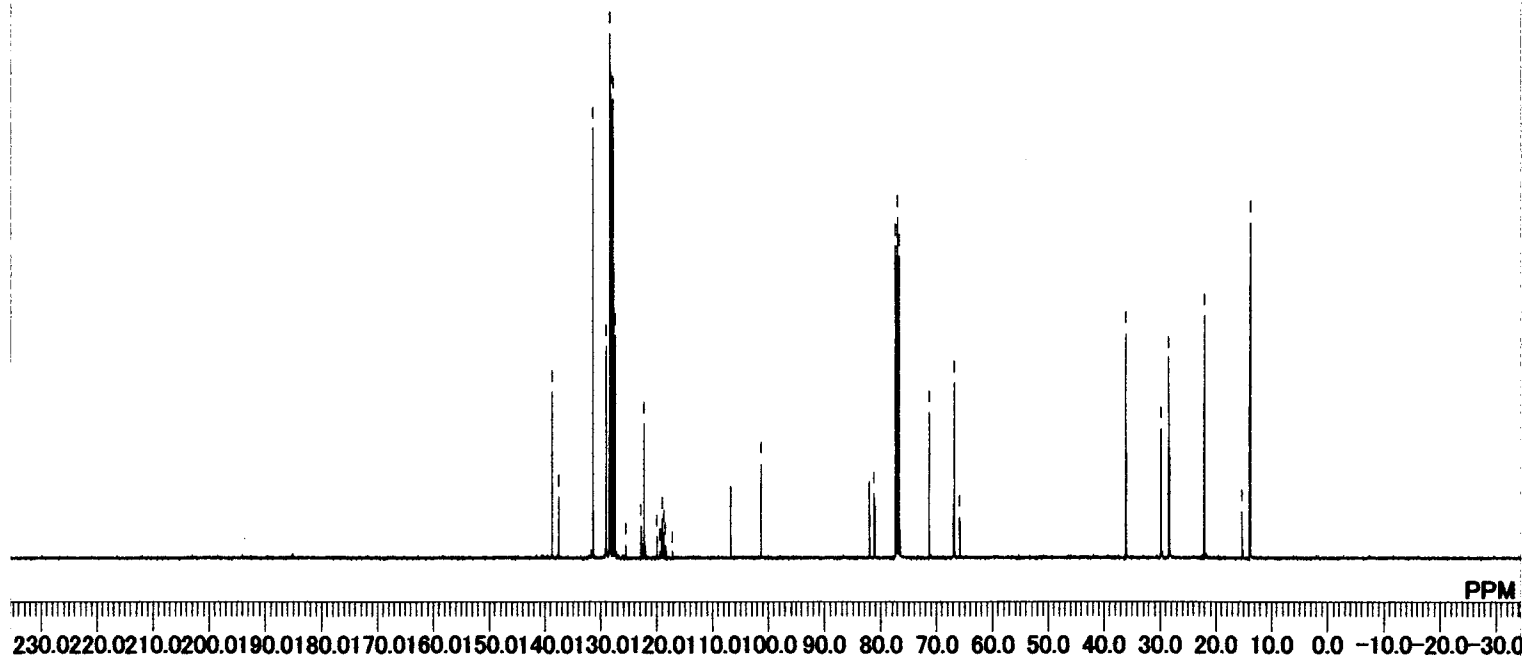
MENUF 1H
OBNUC 1H
OFR 399.65 MHz
OBSET 135.40 KHz
OBFIN 24.90 Hz
PW1 5.50 usec
DEADT 72.20 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7992.01 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 2.0501 sec
PD 4.9500 sec
ADBIT 16
RGAIN 13
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD NON
EXPCM NON:Single.coupled:PW1_ACQTM_PI
IRNUC 1H
IFR 399.65 MHz
IRSET 136.90 KHz
IRFIN 97.50 Hz
IRRPW 45 usec
IRATN 511
DFILE Rxn471 sonogashira H.als
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 23
LKPHS 240
LKSIG 884
CSPED 14 Hz
FILDG
FILDF



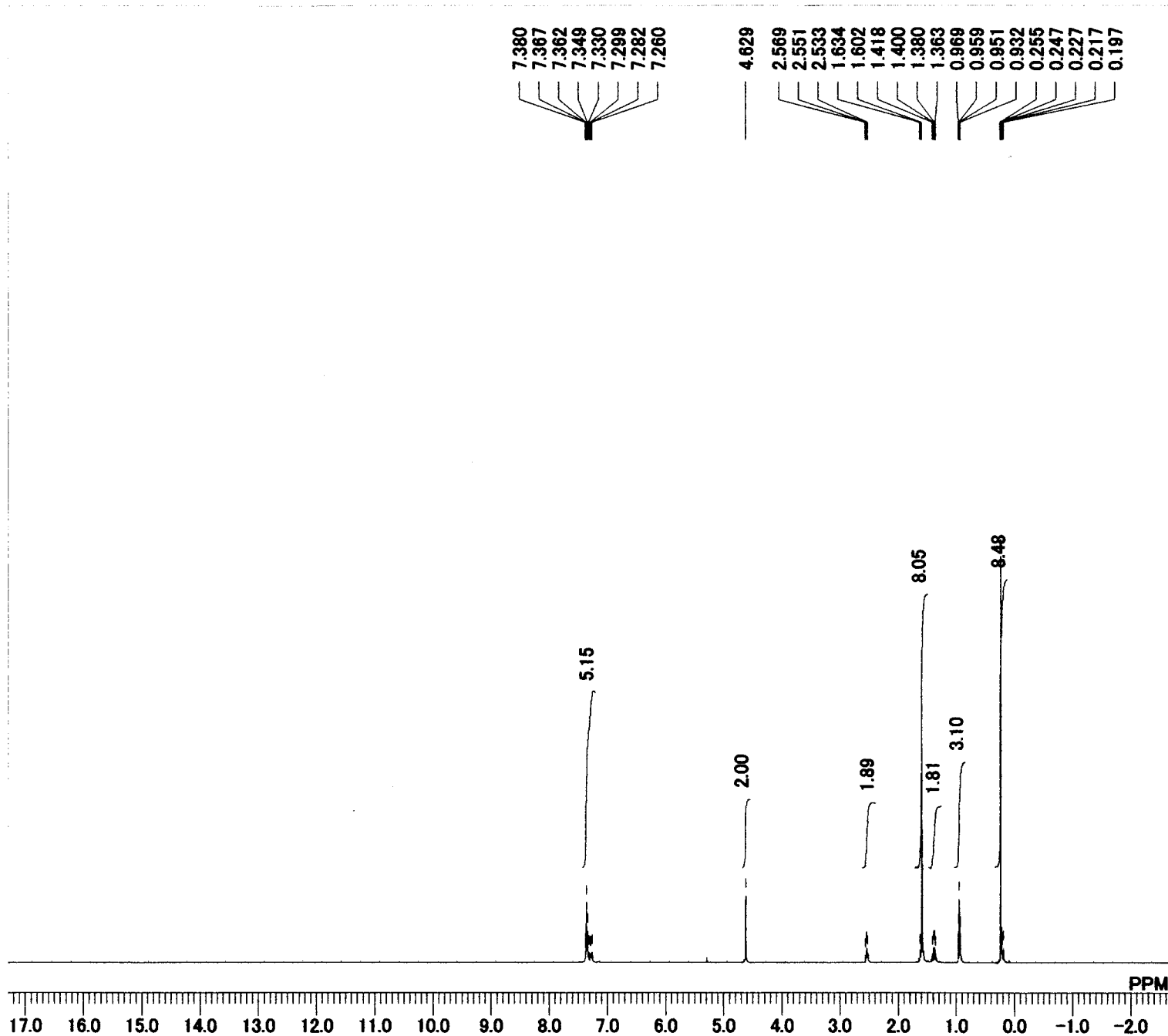
auto

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101.426
82.104
82.080
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77.321
77.000
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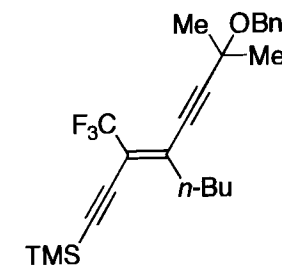
MENUF 13C
OBNUC 13C
OFR 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
PW1 6.20 usec
DEADT 19.00 usec
PREDL 0.20000 msec
IWT 1.0000 msec
POINT 32768
SPO 32768
TIMES 20000
DUMMY 1
FREQU 27118.64 Hz
FLT 13550 Hz
DELAY 14.80 usec
ACQTM 1.2083 sec
PD 1.7920 sec
ADBIT 16
RGAIN 25
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD BCM
EXPCM Bilevel.complete.decoupling:Set_IRRF
IRNUC 1H
IFR 399.65 MHz
IRSET 124.00 KHz
IRFIN 10500.00 Hz
IRRPW 45 usec
IRATN 511
DFILE Rxn471 sonogashira C.als
SF TH5ATFG2
LKSET 61.60 KHz
LKFIN 79.0 Hz
LKLEV 180
LGAIN 23
LKPHS 240
LKSIG 866
CSPED 11 Hz
FILDG
FILDG



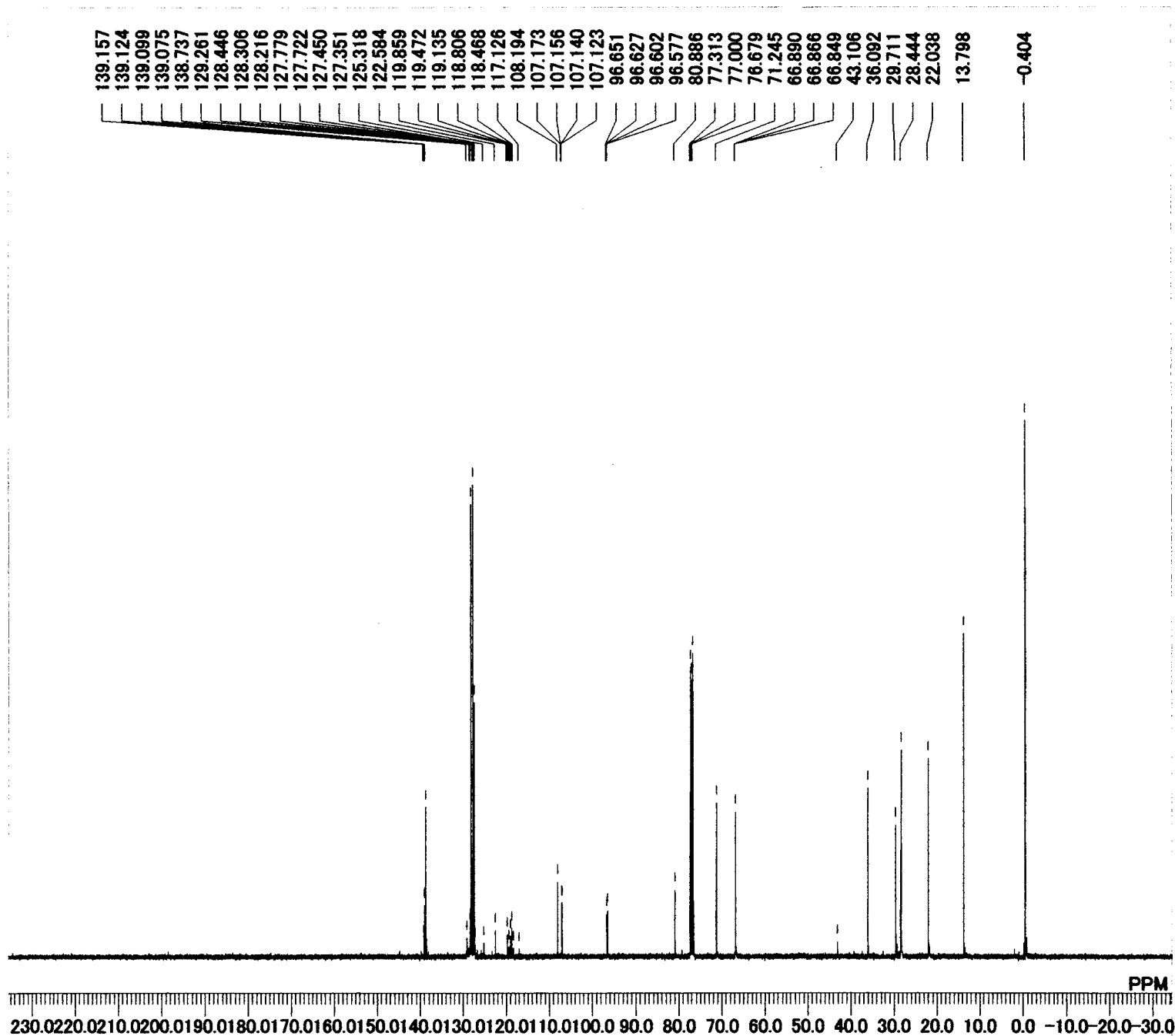
auto



MENUF	1H
OBNUC	1H
OFR	399.65 MHz
OBSET	135.40 KHz
OBFIN	24.90 Hz
PW1	5.50 usec
DEADT	72.20 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	11
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM_PT
IRNUC	1H
IFR	399.65 MHz
IRSET	136.90 KHz
IRFIN	97.50 Hz
IRRPW	45 usec
IRATN	511
DFILE	Rxn472 カラム H.als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	24
LKPHS	240
LKSIG	1186
CSPED	12 Hz
FILDG	
FILDF	



auto



MENUF	13C
OBNUC	13C
OFR	100.40 MHz
OBSET	125.00 KHz
OBFIN	10500.00 Hz
PW1	6.20 usec
DEADT	19.00 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	3000
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.Set_IRRF
IRNUC	1H
IFR	399.65 MHz
IRSET	124.00 KHz
IRFIN	10500.00 Hz
IRRPW	45 usec
IRATN	511
DFILE	Rxn472 カラム C.als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	24
LKPHS	240
LKSIG	1147
CSPED	13 Hz
FILDC	
FILDF	

