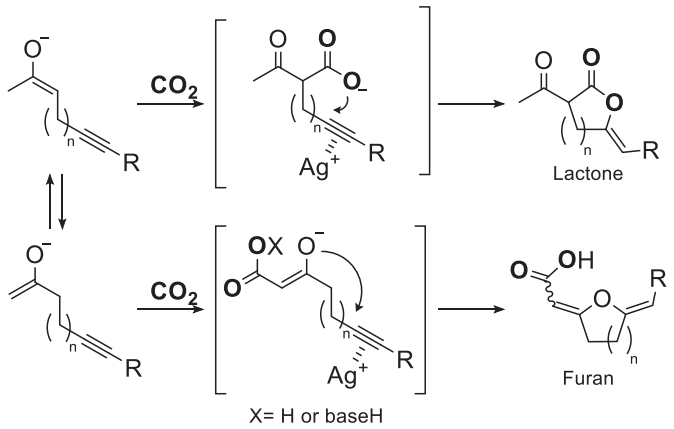
Contents

1. General

The ^{1}H and ^{13}C NMR spectra were recorded with a JEOL model AL-400, alpha-400 or ECX-400 spectrometer using $CDCl_3$ or *d*-DMSO as the solvent. The IR spectra were measured with a Thermo Electron Corporation model NICOLET 6700 FT-IR spectrometer. The melting points were measured with a Stanford Research Systems MPA100 or SHIMADZU DSC-60. The ESI high resolution mass spectra were obtained using a Waters LCT Premier XE mass spectrometer. Column chromatography was conducted on silica gel (Kanto 60 N). The dehydrated DMSO and DMF were purchased from Wako Pure Chemical Industries, Ltd., and used without further purification. All other solvents, such as CH_3CN and Toluene, etc., were distilled from CaH_2 before use. $^{i}Pr_2NEt$ were also distilled before use. DBU was purchased from Wako Pure Chemical Industries, Ltd., and used without further purification. MTBD was purchased from Aldrich, and used without further purification. Buffer (pH = 6) was prepared by KH_2PO_4 and Na_2HPO_4.

2. Supporting Results & Discussion

2-1. Scheme S1

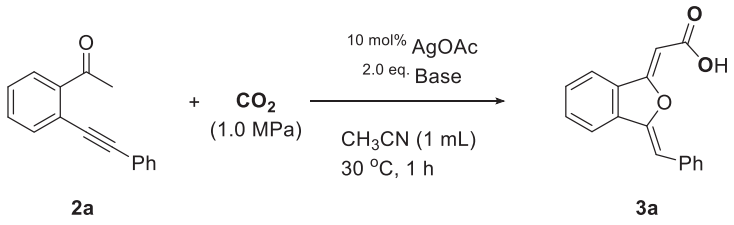


Scheme S1. C-C bond formation with CO_2 afford lactones and furans.

2-2. The screening of several bases.

Several bases were examined (Table S1). K_2CO_3 and *i*Pr_2NEt were not effective at all (Entries 1, 2). Using DBN, the reaction proceeded to afford the product **3a** in 41% yield (Entry 3). Guanidine bases such as TBD and MTBD were screened. TBD slightly promoted the reaction to give **3a** in 14% yield (Entry 5). The good yield was obtained using MTBD (Entry 6). When DBU was employed as base, the corresponding product **3a** was obtained in highest yield (Entry 4).

Table S1. The screening of several bases.



2a **3a**

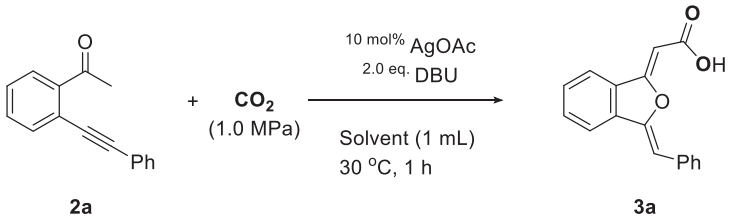
Entry[a)]	Base	Yield[b)] / %
1[c)]	K_2CO_3	ND
2	*i*Pr_2NEt	ND
3	DBN	41
4	DBU	99
5	TBD	14
6	MTBD	82

a) The reaction was carried out with 0.15 mmol of substrate. b) Yields were determined by ^{1}H NMR using trimethylphenylsilane as the internal standard. c) In DMF.

2-3. The screening of various solvents.

The reaction was conducted in various solvents (Table S2). Toluene and THF promoted the reaction to afford the product **3a** in 20% and 53% yields, respectively (Entries 1 and 2). Aprotic polar solvents such as DMF, DMSO, CH_3CN were effective for the reaction to produce **3a** in excellent yields (Entries 3-5).

Table S2. The screening of various solvents.



2a **3a**

Entry[a)]	Solvents	Yield[b)] / %
1	Toluene	20
2	THF	53
3	DMF	94
4	DMSO	91
5	CH_3CN	99

a) The reaction was carried out with 0.15 mmol of substrate. b) Yields were determined by ^{1}H NMR using trimethylphenylsilane as the internal standard.

2-4. The isomerization of products.

In the literature[1], in the case of 1-(alkoxycarbonyl)methylene-dihydroisobenzofuran, 7-H of *E* isomer was significantly shifted downfield because the COOR group was adjacent to 7-H. In the case of **4a**, a trace amount of strongly downfield proton peak derived from the *E* isomer was sometimes detected by ^{1}H NMR in $CDCl_3$, though the ration of *Z* and *E* isomers was >99:1. Moreover, after **4a** was kept in $CDCl_3$ for 48 h, the ration of *Z* and *E* isomers changed to 72:28 (the time course was shown in Figure S1). On the other hand, the product **4a**-(*Z*) could be stored under dry conditions without any isomerization. These observations assumed that the isomerization of **4a**-(*Z*) occurred in $CDCl_3$ like the previous study[2] which suggested that *Z*/*E* isomerization of a similar 1-(alkoxycarbonyl)methylenephthalan occurred under slightly acidic conditions.

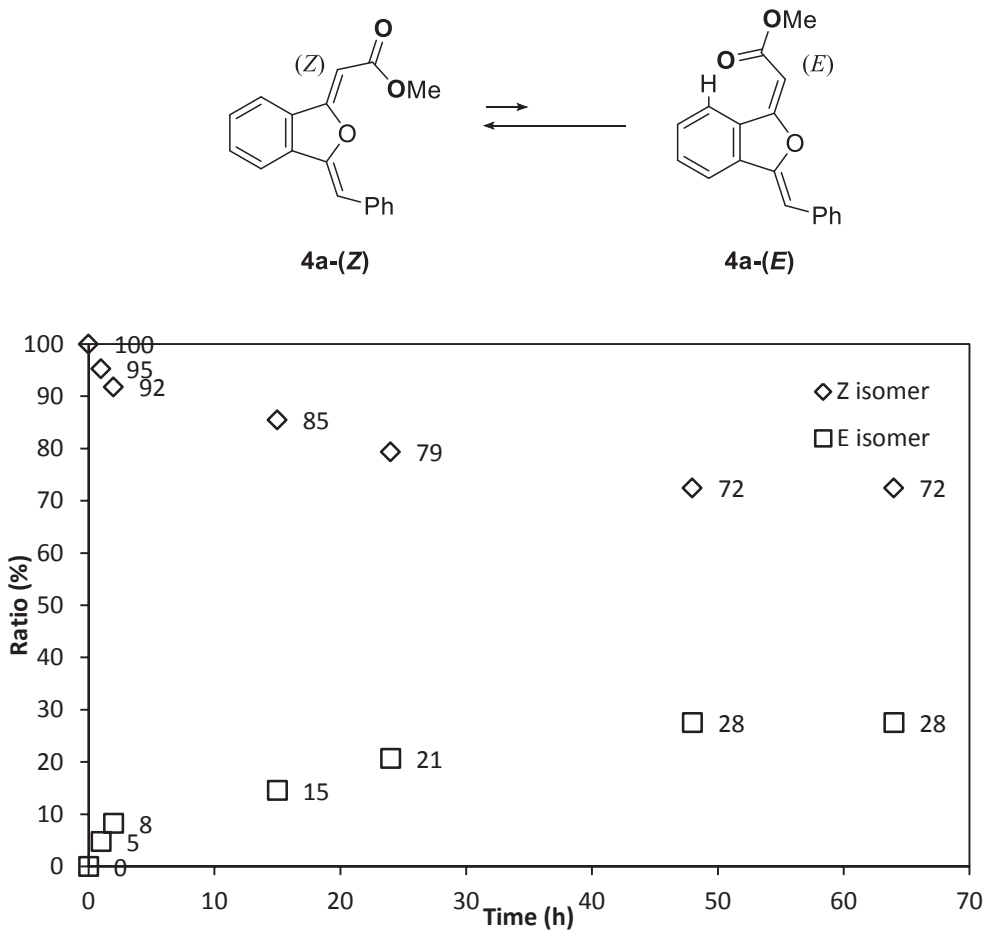


Figure S1. Time course about the **4a**-(*Z*) and **4a**-(*E*) isomerization.

The product **4k** was obtained as *Z*/*E* isomers (95/5) after purification using silica gel column chromatography. On the other hand, the crude carboxylic acid **3k**-(*Z*) was obtained as a solo isomer. In other words, the proton derived from **3k**-(*E*) isomer was not observed by ^{1}H NMR. It was supposed that the isomerization of alkyl-substituted alkyne **3k** or **4k** occurred in the esterification step or/and under slightly acidic conditions such as silica gel and $CDCl_3$ to afford **4k** as *Z*/*E* isomers.

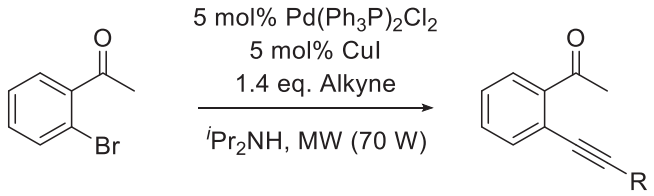
3. Methods

3-1. The synthesis of the starting materials.

o-Alkynylacetophenone **2a**, **2f**, **2k-2m** were synthesized by Sonogashira-coupling reaction between *o*-bromoacetophenone and alkyne. The substrate **2n** was prepared by Sonogashira-coupling reaction and desilylation. **2g-2j** were synthesized by

Sonogashira-coupling of **2n** and the corresponding aryl halide. The substrates **2b-2e** substituted on the phenyl ring and the α-substituted starting materials **2o-2q** were synthesized from the corresponding *o*-bromobenzaldehyde by Sonogashira-coupling reaction, Grignard reagents alkylation and oxidation.

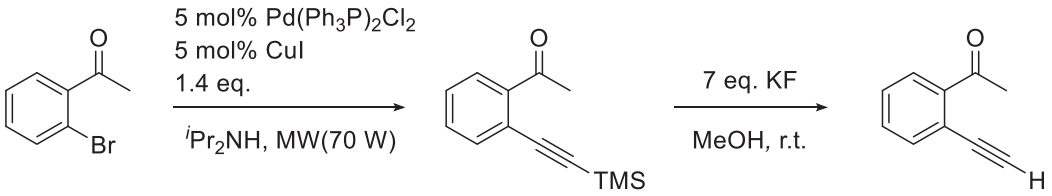
3-2. Procedure for the synthesis of the starting materials (2a, 2f, 2k-2m)



Compound **2a, 2f, 2k-2m** were synthesized by the modified procedure of the literature.[3]

The corresponding alkyne (1.4 eq.) was added to the solution of $Pd(PPh_3)_2Cl_2$ (5 mol%), CuI (5 mol%) and *o*-bromoacetophenone (1 eq., 5 mmol) in iPr_2NH (20 mL) using vial. The solution was heated at 80 °C with microwave (70 W, Biotage Initiator). After 4 h, the reactant was filtered through Celite, then solvent was removed under reduced pressure and the residue was purified by column chromatography (SiO_2, eluent: hexane/EtOAc) to afford the desired starting material **2a**, **2f**, **2k-2m**.

3-3. Procedure for the synthesis of the starting materials (2g-2j, 2n).

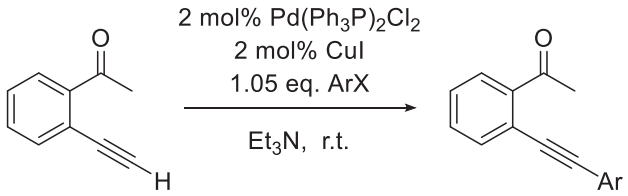


S1

Trimethylsilylacetylene (1.4 eq.) was added to the solution of $Pd(PPh_3)Cl_2$ (5 mol%), CuI (5 mol%) and *o*-bromoacetophenone (1 eq., 5 mmol) in iPr_2NH (20 mL) using vial. The solution was heated at 80 °C with microwave (70 W, Biotage Initiator). After 4 h, the reactant was filtered through Celite, then solvent was removed under reduced pressure and the residue was purified by column chromatography (SiO_2, eluent: hexane/EtOAc) to afford the corresponding *o*-alkynylacetophenone **S1**.

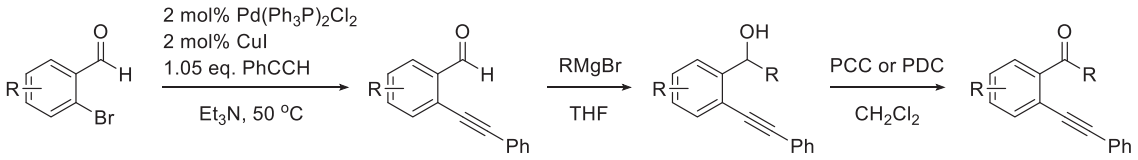
Desilylation was carried out according to the literature.[4] KF (7 eq.) was added to the solution of compound **S1** in MeOH (0.12 M). After stirring for 2 h, MeOH was

removed under reduced pressure, then Et_2O and water were added. The reaction mixture was extracted with Et_2O, washed with water. The conbined organic layer was dried with Na_2SO_4, filtererd and concentrated under reduced pressure. The residue was purified by column chromatography (SiO_2, eluent: hexane/EtOAc) to afford 2`-ethynylacetophenone **2n**.



2`-ethynylacetophenone **2n** (1.05 eq.) was added to the solution of $Pd(PPh_3)_2Cl_2$ (2 mol%), CuI (2 mol%) and the corresponding aryl halide (1 eq.) in Et_3N under N_2. The solution was stirred at room temperature overnight. The reactant was filtered through Celite, and solvent was removed under reduced pressure. The residue was purified by column chromatography (SiO_2, eluent: hexane/EtOAc) to afford the desired starting material **2g-2j**.

3-4. Procedure for the synthesis of the starting materials (2b-2e, 2o-2q).



S2 **S3** **S4** **S5**

The corresponding alkyne (1.05 eq.) was added to the solution of $Pd(PPh_3)_2Cl_2$ (2 mol%), CuI (2 mol%) and *o*-bromobenzaldehyde (1 eq., 10 mmol) in Et_3N (20 mL) under N_2. The reaction mixture was heated at 50 °C. After 4 h, the reaction mixture was filtered through Celite, then solvent was removed under reduced pressure and the residue was purified by column chromatography (SiO_2, eluent: hexane/EtOAc) to afford the corresponding *o*-alkynylbenzaldehyde **S3**.

Alkylation and oxidation steps were carried out according to the literature.[5]

To a solution of the corresponding 2-alkenylbenzaldehyde in dry THF (0.2 M) was added MeMgBr or *n*BuLi (1.5 eq.) at -78 °C under N_2. After stirring at -78 °C for 2 h, the reaction mixture was quenched with sat. NH_4Cl aq. then extracted with CH_2Cl_2. The combined organic layer was dried with $NaSO_4$ and concentrated under reduced pressure.

The residue was purified by column chromatography (SiO_2, eluent: hexane/EtOAc) to afford the corresponding alcohol **S4**.

To a solution of the corresponding **S4** in CH_2Cl_2 (0.1 M) was added PCC (2.5 eq.) at room temperature. The reaction mixture was stirred at room temperature for 2 h. After the reaction was completed, 1 g of Celite was added and stirred for 5-10 min. The reaction mixture was filtered through Celite and silica gel and concentrated under reduced pressure. The residue was purified by column chromatography (SiO_2, eluent: hexane/EtOAc) to afford the corresponding **2b-2e, 2o-2q**.

3-5. Procedure for the synthesis of 3a

The reaction was performed using a pressure test-tube equipped with a stirring bar in a 30 mL autoclave. To a mixture of AgOAc (2.5 mg, 0.015 mmol) and **2a** (33.0 mg, 0.15 mmol) in 1.0 mL CH_3CN in a pressure test-tube was added DBU (45 μL, 0.30 mmol) with a microsyringe. The pressure test-tube containing the reaction mixture was placed in the autoclave. The autoclave was purged with CO_2 (1.0 MPa) and the reaction mixture was stirred at 30 °C for 1 h. After the CO_2 was vented, the reaction was quenched with buffer (pH=6) and extracted with EtOAc, then solvent was removed to give **3a** (99%) (trimethylphenylsilane (5 μL, 0.029 mmol) was added, then the yield was determined by ^1H NMR spectrum). If necessary, the product was purified by recrystalization with CH_3CN to afford the corresponding dihydroisobenzofuran **3a** (66%) as a yellow solid.

3-6. Procedure for the synthesis of 4b

The reaction was performed using a pressure test-tube equipped with a stirring bar in a 30 mL autoclave. To a mixture of AgOAc (2.5 mg, 0.015 mmol) and **2b** (42.0 mg, 0.15 mmol) in 1.0 mL CH_3CN in a pressure test-tube was added DBU (45 μL, 0.30 mmol) with a microsyringe. The pressure test-tube containing the reaction mixture was placed in the autoclave. The autoclave was purged with CO_2 (1.0 MPa) and the reaction mixture was stirred at 30 °C for 6 h. After the CO_2 was released, MeI (37 μL, 0.60 mmol) was added to the reactant. After 3 h, the reaction mixture was purified by column chromatography (SiO_2, eluent: hexane/EtOAc 20/1 then 8/1) to produce the corresponding dihydroisobenzofuran **4b** (91%) as a yellow solid.

Reference

[1] a) A. Fürstner, H. Szillat, F. Stelzer *J. Am. Chem. Soc.* **2000,** *122,* 6785-6786; b) A.

Bacchi, M. Costa, N. D. Cà, M. Fabbricatore, A. Fazio, B. Gabriele, C. Nasi, G. Salerno, *Eur. J. Org. Chem.* **2004**, 574-585.

[2] S. Duan, K. Cress, K. Waynant, E. Ramos-Miranda, J. W. Herndon, *Tetrahedron* **2007**, *63*, 2959-2965

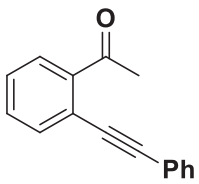
[3] X. Chen, J. Jin, N. Wang, P. Lu, Y. Wang, *Eur. J. Org. Chem.* **2012**, 824-830.

[4] C. P. Casey, N. A. Strotman, I. A. Guzei, *Beilstein J. Org. Chem.* **2005**, *1*, doi:10.1186/1860-5397-1-18.

[5] A. R. Jagdale, S. W. Youn, *Eur. J. Org. Chem.* **2011**, 3904-3910.

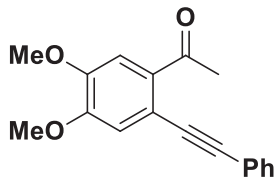
4. Material Data

1-(1-phenylethynyl)phenyl)ethan-1-one (2a):



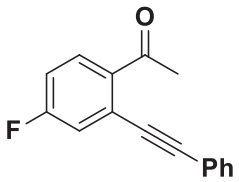
Pale yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.80 (s, 3H), 7.35-7.39 (m, 3H), 7.42 (dd, *J* = 1.3, 7.7 Hz, 1H), 7.48 (td, *J* = 1.4, 7.5 Hz, 1H), 7.54-7.57 (m, 2H), 7.64 (dd, *J* = 0.9, 7.7 Hz, 1H), 7.76 (dd, *J* = 1.2, 7.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 30.0, 88.4, 95.0, 121.7, 122.9, 128.3, 128.4, 128.67, 128.74, 131.3, 131.5, 133.9. 140.7, 200.4; IR (KBr): 3061, 2215, 1686, 1592, 1279, 757, 691; HRMS (ESI): $[M]^+$ calcd for $C_{16}H_{13}O$, 221.0966 ; found, m/z 221.0960.

1-(4, 5-dimethoxy-2-(phenylethynyl)phenyl)ethan-1-one (2b):



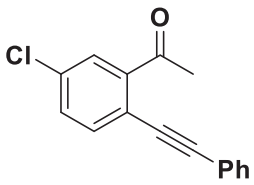
Colorless solid; m.p.: 119 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.86 (s, 3H), 3.96 (s, 3H), 3.98 (s, 3H), 7.07 (s, 1H), 7.34-7.41 (m, 3H), 7.43 (s, 1H), 7.51-7.58 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 30.4, 56.1, 56.2, 89. 1, 94.5, 111.3, 115.5, 116.1, 122.9, 128.5, 128.7, 131.2, 133.5, 149.1, 151.5, 198.4; IR (KBr): 2962, 2931, 1659, 1590, 1246, 1053, 756; HRMS (ESI): $[M]^+$ calcd for $C_{18}H_{17}O_3$, 281.1178 ; found, m/z 281.1176.

1-(4-fluoro-2-(1-phenylethynyl)phenyl)ethan-1-one (2c):



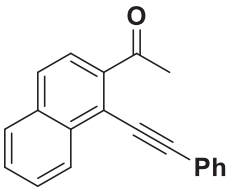
Pale yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.79 (s, 3H), 7.07-7.12 (m, 1H), 7.32 (dd, *J* = 2.6, 9.1 Hz, 1H), 7.36-7.41 (m, 3H), 7.53-7.58 (m, 2H), 7.83 (dd, *J* = 5.8, 8.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 29.9, 87.5 (d, *J* = 2.8 Hz), 96.2, 115.7 (d, *J* = 21.6 Hz), 120.4 (d, *J* = 23.5 Hz), 122.4, 124.5 (d, *J* = 10.3 Hz), 129.1, 131.5, 131.6, 131.6 (d, *J* = 9.4 Hz), 136.8 (d, *J* = 3.8 Hz), 164.0 (d, *J* = 254.6 Hz), 198.5; IR (KBr): 3068, 2214, 1685, 1572, 1239, 1100, 757, 690; HRMS (ESI): $[M]^+$ calcd for $C_{16}H_{12}FO$, 239.0872 ; found, m/z 239.0870.

1-(5-chloro-2-(1-phenylethynyl)phenyl)ethan-1-one (2d):



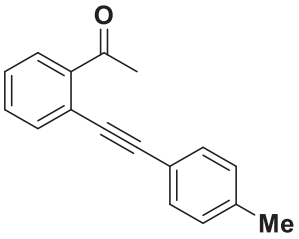
Pale yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.79 (s, 3H), 7.34-7.40 (m, 3H), 7.44 (dd, *J* = 2.4, 8.3 Hz, 1H), 7.50-7.58 (m, 3H), 7.73 (d, *J* = 2.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 29.9, 87.4, 96.0, 120.1, 122.5, 128.5, 128.8, 129.0, 131.4, 131.5, 134.5, 135.0, 141.8, 198.9; IR (KBr): 3064, 2215, 1686, 1493, 1469, 1356, 1281, 1254, 1102, 828, 690; HRMS (ESI): $[M]^+$ calcd for $C_{16}H_{12}ClO$, 255.0572 ; found, m/z 255.0574.

1-(1-(phenylethynyl)naphthalen-2-yl)ethan-1-one (2e):



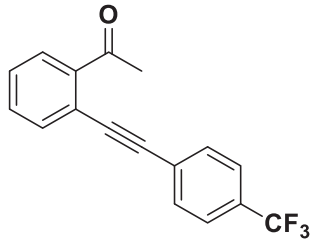
Pale yellow solid; m.p.: 46 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.92 (s, 3H), 7.38-7.45 (m, 3H), 7.57-7.70 (m, 4H), 7.79 (d, *J* = 8.8 Hz, 1H), 7.82-7.88 (m, 2H), 8.61 (d, *J* = 8.3 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 30.6, 86.3, 101.5, 119.9, 122.8, 124.5, 127.4, 127.6, 128.1, 128.2, 128.56, 128.61, 129.0, 131.5, 133.2, 134.2, 139.4, 201.4; IR (KBr): 3057, 2993, 2202, 1663, 1264, 1241, 752; HRMS (ESI): $[M]^+$ calcd for $C_{20}H_{15}O$, 271.1118 ; found, m/z 271.1122.

1-(2-(1-*p*-tolylethynyl)phenyl)ethan-1-one (2f):



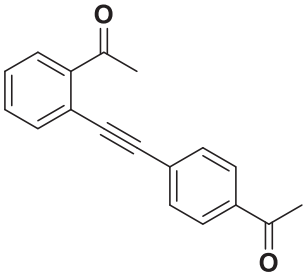
Oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.38 (s, 3H), 2.80 (s, 3H), 7.18 (d, *J* = 7.9 Hz, 2H), 7.36-7.41 (m, 1H), 7.42-7.50 (m, 3H), 7.62 (dd, *J* = 1.3, 7.9 Hz, 1H), 7.75 (dd, *J* = 1.5, 7.7 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 21.6, 30.1, 87.9, 95.4, 119.8, 121.9, 128.1, 128.7, 129.2, 131.3, 131.4, 133.8, 139.0, 140.7, 200.5; IR (KBr): 3027, 2873, 2214, 1686, 958, 817, 762; HRMS (ESI): $[M]^+$ calcd for $C_{17}H_{15}O$, 235.1123 ; found, m/z 235.1121.

1-(2-(1-(4-trifuluoromethylphenyl)ethynyl)phenyl)ethan-1-one (2g):



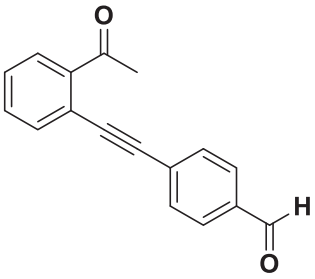
Oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.76 (s, 3H), 7.45 (td, *J* = 1.5, 7.6 Hz, 1H), 7.51 (td, *J* = 1.6, 7.6 Hz, 1H), 7.60-7.69 (m, 5H), 7.79 (dd, *J* = 1.5, 7.7 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 29.7, 90.7, 93.0, 120.9, 123.8 (d, *J* = 273.1 Hz), 125.4 (q, *J* = 4.1 Hz), 126.7 (d, *J* = 1.9 Hz), 128.8, 128.9, 130.3 (d, *J* = 32.6 Hz), 131.4, 131.8, 134.1, 140.7, 199.7; IR (KBr): 2220, 1684, 1320, 1123, 957, 843, 762, 598; HRMS (ESI): $[M]^+$ calcd for $C_{17}H_{12}F_3O$, 289.0835 ; found, m/z 289.0834.

1-(2-(1-(4-acetylphenyl)ethynyl)phenyl)ethan-1-one (2h):



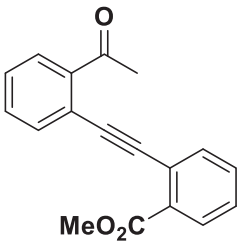
Pale yellow solid; m.p.: 62 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.63 (s, 3H), 2.78 (s, 3H), 7.45 (td, *J* = 1.5, 7.6 Hz, 1H), 7.51 (td, *J* = 1.6, 7.6 Hz, 1H), 7.62-7.68 (m, 3H), 7.77-7.80 (m, 1H), 7.93-7.98 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 26.6, 29.7, 91.6, 93.7, 121.0, 127.7, 128.3, 128.8, 128.9, 131.4, 131.7, 134.1, 136.5, 140.7, 197.2, 199.8; IR (KBr):2210, 1680, 1602, 1266, 826; HRMS (ESI): $[M]^+$ calcd for $C_{18}H_{15}O_2$, 263.1067 ; found, m/z 263.1066.

1-(2-(1-(4-formylphenyl)ethynyl)phenyl)ethan-1-one (2i):



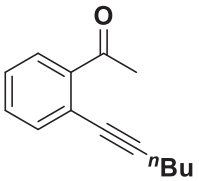
Pale yellow solid; m.p.: 53 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.76 (s, 3H), 7.45 (td, *J* = 1.3, 7.7 Hz, 1H), 7.51 (td, *J* = 1.5, 7.6 Hz, 1H), 7.64-7.68 (m, 1H), 7.69-7.71 (m, 2H), 7.79 (dd, *J* = 1.0, 7.8 Hz, 1H), 7.85-7.91 (m, 2H), 10.03 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 29.6, 92.3, 93.4, 120.8, 128.9, 128.9, 129.1, 129.5, 131.4, 132.0, 134.1, 135.6, 140.5, 191.3, 199.6; IR (KBr):3064, 2835, 2215, 1697, 1601, 1562, 1207; HRMS (ESI): $[M]^+$ calcd for $C_{17}H_{13}O_2$, 249.0919 ; found, m/z 249.0915.

1-(2-(1-(2-methoxycarbonylphenyl)ethynyl)phenyl)ethan-1-one (2j):



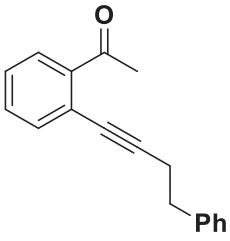
Pale yellow solid; m.p.: 72 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.81, (s, 3H), 3.96 (s, 3H), 7.39-7.45 (m, 2H), 7.50 (td, *J* = 1.4, 7.6 Hz, 2H), 7.53 (td, *J* = 1.5, 7.6 Hz, 2H), 7.76-7.79 (m, 1H), 7.99-8.02 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 30.0, 52.3, 93.2, 93.8, 121.8, 123.4, 128.4, 128.5, 128.7, 130.6, 131.4, 131.7, 131.8, 134.0, 134.2, 140.6, 166.4, 200.3; IR (KBr): 3068, 3002, 2958, 1725, 1670, 1251, 1080, 760; HRMS (ESI): $[M]^+$ calcd for $C_{18}H_{15}O_3$, 279.1016 ; found, m/z 279.1013.

1-(2-hexynylphenyl)ethan-1-one (2k):



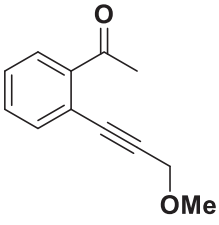
Oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.95 (t, *J* = 7.3 Hz, 3H), 1.44-1.53 (m, 2H), 1.58-1.65 (m, 2H), 2.47 (t, *J* = 7.1 Hz, 2H), 2.73 (s, 3H), 7.33 (td, *J* = 1.3, 7.6 Hz, 1H), 7.40 (td, *J* = 1.1, 7.6 Hz, 1H), 7.49 (dd, *J* = 1.0, 7.8 Hz, 1H), 7.66 (dd, *J* = 1.5, 7.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 13.6, 19.4, 22.1, 30.1, 30.5, 79.6, 96.9, 122.5, 127.5, 128.3, 131.1, 134.0, 141.0, 201.2; IR (KBr): 2960, 2932, 2230, 1684, 963, 763; HRMS (ESI): $[M]^+$ calcd for $C_{14}H_{17}O$, 201.1279 ; found, m/z 201.1268.

1-(2-(4-butynylphenly)phenyl)ethan-1-one (2l):



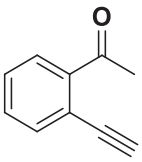
Oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.56 (s, 3H), 2.77 (t, *J* = 7.4 Hz, 2H), 2.94 (t, *J* = 7.4 Hz, 2H), 7.20-7.35 (m, 6H), 7.39 (td, *J* = 1.5, 7.5 Hz, 1H), 7.45 (dd, *J* = 1.5, 7.7 Hz, 1H), 7.65 (dq, *J* = 0.7, 7.7 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 21.8, 29.9, 34.7, 80.3, 95.7, 122.1, 126.4, 127.6, 128.3, 128.4, 128.5, 131.0, 134.0, 140.4, 141.0, 201.0; IR (KBr): 3028, 2927, 2227, 1688, 1279, 1244, 763, 700;HRMS (ESI): $[M]^+$ calcd for $C_{18}H_{17}O$, 249.1274 ; found, m/z 249.1275.

1-(2-(3-methoxyprop-1-yn-1-yl)phenyl)ethan-1-one (2m):



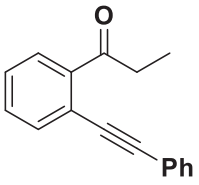
Oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.71 (s, 3H), 3.48 (s, 3H), 4.37 (s, 2H), 7.36-7.48 (m, 2H), 7.55 (d, *J* = 7.3 Hz, 1H), 7.71 (dd, *J* = 1.5, 7.3 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 29.7, 57.8, 60.4, 85.2, 90.7, 120.9, 128.4, 128.5, 131.2, 134.2, 140.8, 200.1; IR (KBr): 2933, 2823, 1690, 1357, 1246, 1090, 764; HRMS (ESI): $[M]^+$ calcd for $C_{12}H_{13}O_2$, 189.0911; found, m/z 189.0903.

1-(2-ethynylphenyl)ethan-1-one (2n):



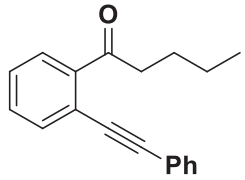
Oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.72 (s, 3H), 3.40 (s, 1H), 7.40-7.48 (m, 2H), 7.59-7.63 (m, 1H), 7.69-7.73 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 29.8, 82.4, 82.9, 120.3, 128.6, 128.8, 131.2, 134.7, 141.5, 200.1; IR (KBr): 3284, 3065, 2104, 1686, 1593, 1281, 764; HRMS (ESI): $[M]^+$ calcd for $C_{10}H_9O$, 145.0648 ; found, m/z 145.0646.

1-(1-phenylethynyl)phenyl)propanone (2o):



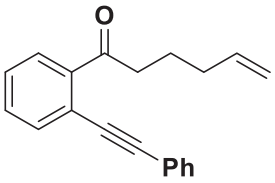
Pale yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.25 (t, *J* = 7.2 Hz, 3H), 3.18 (q, *J* = 7.3 Hz, 2H), 7.35-7.42 (m, 4H), 7.46 (tq, *J* = 1.6, 7.5 Hz, 2H), 7.51-7.56 (m, 1H), 7.62 (dd, *J* = 1.2, 7.7 Hz, 1H), 7.67 (d, *J* = 1.2, 7.7 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 8.5, 35.3, 88.2, 94.4, 121.1, 122.9, 128.1, 128.3, 128.4, 128.7, 130.8, 131.5, 133.7, 141.3, 204.1; IR (KBr): 2976, 2937, 2215, 1698, 950, 757, 732, 691; HRMS (ESI): $[M]^+$ calcd for $C_{17}H_{15}O$, 235.1118 ; found, m/z 235.1118.

1-(1-phenylethynyl)phenyl)pentanone (2p):



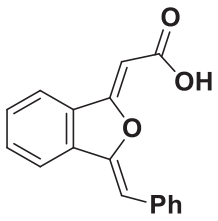
Pale yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.92 (t, *J* = 7.3 Hz, 3H), 1.35-1.45 (m, 2H), 1.69-1.78 (m, 2H), 3.16 (t, *J* = 7.6 Hz, 2H), 7.35-7.42 (m, 4H), 7.46 (td, *J* = 1.3, 7.4 Hz, 1H), 7.51-7.56 (m, 2H), 7.62 (dd, *J* = 1.0, 7.8 Hz, 1H), 7.65 (dd, *J* = 1.5, 7.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 13.9, 22.5, 26.6, 41.9, 88.2, 94.4, 121.1, 122.9, 128.1, 128.3, 128.4, 128.7, 130.8, 131.5, 133.7, 141.5, 203.9; IR (KBr): 3061, 2958, 2872, 2215, 1686, 1493, 1200, 758; HRMS (ESI): $[M]^+$ calcd for $C_{19}H_{19}O$, 263.1436 ; found, m/z 263.1434.

1-(1-phenylethynyl)phenyl)hex-5-en-1-one (2q):



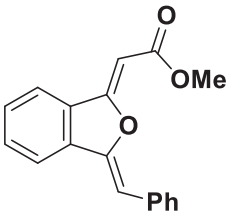
Pale yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.83-1.90 (m, 2H), 2.14 (q, *J* = 7.2 Hz, 2H), 3.16 (t, *J* = 7.6 Hz, 2H), 4.91-5.03 (m, 2H), 5.73-5.83 (m, 1H), 7.32-7.40 (m, 4H), 7.44 (td, *J* = 1.5, 7.6 Hz, 1H), 7.50-7.56 (m, 2H), 7.61 (dd, *J* = 1.5, 7.8 Hz, 1H), 7.65 (dd, *J* = 1.2, 7.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ =23.5, 33.2, 41.3, 88.2, 94.4, 115.2, 121.08, 122.8, 128.1, 128.2, 128.4, 128.6, 130.8, 131.5, 133.7, 137.9, 141.3, 203.3; IR (KBr):3063, 2933, 2215, 1686, 1474, 1197, 690; HRMS (ESI): $[M]^+$ calcd for $C_{20}H_{19}O$, 275.1430 ; found, m/z 275.1436.

(*Z*)-2-[(*Z*)-3-Benzylideneisobenzofuran-1(3*H*)-ylidene]acetic acid (3a):



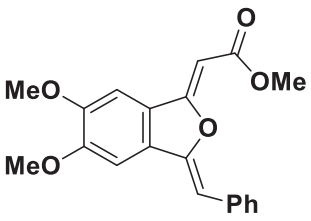
Yellow solid; m.p.: 192 °C; ^{1}H NMR (400 MHz, *d*-DMSO): δ = 5.92 (s, 1H), 6.72 (s, 1H), 7.28 (t, *J* = 7.4 Hz, 1H), 7.40 (t, *J* = 7.6 Hz, 2H), 7.52-7.59 (m, 1H), 7.63-7.69 (m, 1H), 8.00 (t, *J* = 6.1 Hz, 1H), 8.01-8.07 (m, 3H), 12.09 (s, 1H); ^{13}C NMR (100 MHz, *d*-DMSO): δ = 89.8, 103.1, 120.5, 122.1, 127.4, 128.7, 129.3, 129.9, 131.4, 132.0, 134.1, 134.6, 150.3, 161.1, 165.9; IR (KBr): 2584, 1698, 1629, 1462; HRMS (ESI): $[M]^{+}$ calcd for $C_{17}H_{13}O_3$, 265.0865 ; found, m/z 265.0863; NOE (δ =5.92) 4%, NOE (δ =6.72) 5 %, 7%.

Methyl (*Z*)-2-[(*Z*)-3-Benzylideneisobenzofuran-1(3*H*)-ylidene]acetate (4a):



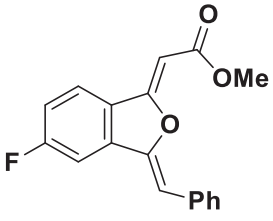
Pale yellow solid; m.p.: 96 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.90 (s, 3H), 5.69 (s, 1H), 6.33 (s, 1H), 7.29 (t, *J* = 7.5 Hz, 1H), 7.46 (t, *J* = 7.7 Hz, 3H), 7.56 (t, *J* = 7.3 Hz, 1H), 7.66 (d, *J* = 7.9 Hz, 1H), 7.70 (d, *J* = 7.6 Hz, 1H), 8.03 (d, *J* = 7.9 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 51.4, 88.4, 103.9, 119.9, 121.4, 127.6, 128.7, 129.37, 129.42, 131.6, 131.9, 134.0, 135.5, 150.7, 162.1, 165.9; IR (KBr): 3067, 1686, 1434, 1273, 1150, 1021, 758; HRMS (ESI): $[M]^{+}$ calcd for $C_{18}H_{15}O_3$, 279.1016 ; found, m/z 279.1016; NOE (δ = 5.69) 2.0%, NOE (δ = 6.33) 2.2%, 2.9%.

Methyl (*Z*)-2-[(*Z*)-3-benzylidene-5, 6-dimthoxy-isobenzofuran-1(3*H*)-ylidene]acetate (4b):



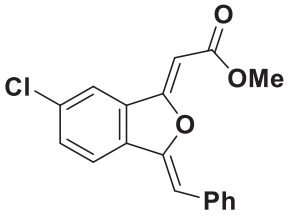
Pale yellow solid; m.p.: 194 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.88 (s, 3H), 3.96 (s, 3H), 4.01 (s, 3H), 5.52 (s, 1H), 6.18 (s, 1H), 6.99 (s, 1H), 7.05 (s, 1H), 7.27 (t, *J* = 7.3 Hz, 1H), 7.44 (t, *J* = 7.8 Hz, 2H), 8.00 (d, *J* = 7.3 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ =51.3, 56.2, 56.3, 86.8, 100.9, 102.0, 102.6, 124.8, 127.2, 128.7, 129.2, 129.3, 134.2, 150.8, 151.3, 153.2, 162.2, 166.0; IR (KBr): 3000, 2946, 1686, 1501, 1343, 1263, 1222, 1030, 826; HRMS (ESI): $[M]^{+}$ calcd for $C_{20}H_{19}O_5$, 339.1227 ; found, m/z 339.1230; NOE (δ =5.50) 3.7%, NOE (δ =6.15) 4.0 %, 5.2%.

Methyl (*Z*)-2-[(*Z*)-3-benzylidene-5-fluoro-isobenzofuran-1(3*H*)-ylidene]acetate (4c):



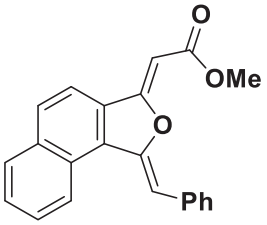
Pale yellow solid; m.p.: 162 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.87 (s, 3H), 5.59 (s, 1H), 6.24 (s, 1H), 7.12 (td, *J* = 2.6, 8.6 Hz, 1H), 7.25-7.32 (m, 2H), 7.44 (t, *J* = 7.7 Hz, 2H), 7.57 (dd, *J* = 4.5, 8.5 Hz, 1H), 7.57 (d, *J* = 7.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 51.4, 88.2, 104.9, 106.4 (d, *J* = 25.4 Hz), 117.7 (d, *J* = 24.4 Hz), 123.3 (d, *J* = 9.4 Hz), 127.8 (d, *J* = 1.9 Hz), 127.9, 128.7, 129.5, 133.6, 137.6 (d, *J* = 10.3 Hz), 149.7, 161.1, 163.7, 164.9 (d, *J* = 251.8 Hz); IR (KBr): 3067, 2944, 1679, 1480, 1267, 1200, 1026, 810; HRMS (ESI): $[M]^{+}$ calcd for $C_{18}H_{14}FO_3$, 297.0922 ; found, m/z 297.0918; NOE (δ = 5.59) 2.3%, NOE (δ = 6.19) 2.5 %, 3.5%.

Methyl (*Z*)-2-[(*Z*)-3-benzylidene-6-chloro-isobenzofuran-1(3*H*)-ylidene]acetate (4d):



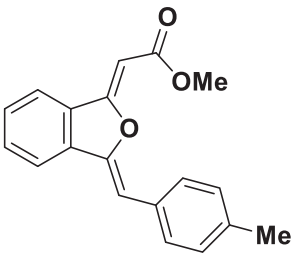
Pale yellow solid; m.p.: 172 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.87 (s, 3H), 5.62 (s, 1H), 6.25 (s, 1H), 7.24-7.32 (m, 1H), 7.40-7.50 (m, 3H), 7.53-7.62 (m, 2H), 7.98 (d, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 51.5, 89.2, 104.5, 121.0, 121.2, 127.8, 128.8, 129.5, 132.0, 133.3, 133.7, 133.8, 135.4, 149.8, 160.6, 165.5; IR (KBr): 3067, 2943, 1698, 1464, 1433, 1317, 1284, 1125, 1026, 815; HRMS (ESI): $[M]^+$ calcd for $C_{18}H_{14}ClO_3$, 313.0626; found, m/z 313.0626; NOE (δ = 5.62) 2.4%, NOE (δ = 6.25) 2.5 %, 3.7%.

Methyl (*Z*)-2-[(*Z*)-1-benzylidenenaththo[1.2-*c*]furan-3(1*H*)-ylidene]acetate (4e):



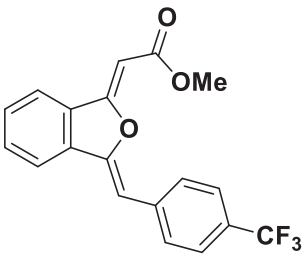
Yellow solid; m.p.: 164 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.89 (s, 3H), 5.65 (s, 1H), 6.80 (s, 1H), 7.32 (t, *J* = 7.3 Hz, 1H), 7.33-7.43 (m, 3H), 7.60 (t, *J* = 7.5 Hz, 1H), 7.67 (t, *J* = 7.5 Hz, 1H), 7.79 (d, *J* = 8.5 Hz, 1H), 7.91 (d, *J* = 7.9 Hz, 1H), 8.13 (d, *J* = 7.6 Hz, 2H), 8.39 (d, *J* = 8.3 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 51.4, 88.0, 109.2, 117.4, 123.7, 126.5, 127.6, 127.8, 128.4, 128.7, 129.7, 130.0, 130.9, 131.0, 131.3, 134.6, 135.3, 151.8, 161.8, 166.0; IR (KBr): 3079, 2947, 1686, 1431, 1264, 1094, 1038, 819; HRMS (ESI): $[M]^+$ calcd for $C_{22}H_{17}O_3$, 329.1173 ; found, m/z 329.1174; NOE (δ = 5.65) 3.6%, NOE (δ = 6.81) 4.7 %, 12.9%.

Methyl (*Z*)-2-[(*Z*)-3-(4-methylbenzylidene)isobenzofuran-1(3*H*)-ylidene]acetate (4f):



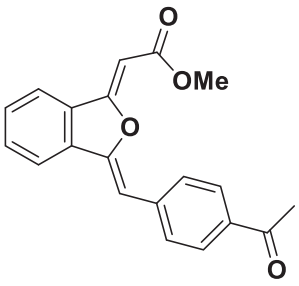
Pale yellow solid; m.p.: 119 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.38 (s, 3H), 3.89 (s, 3H), 5.67 (s, 1H), 6.31 (s, 1H), 7.27 (d, *J* = 7.4 Hz, 2H), 7.43 (td, *J* = 0.9, 7.5 Hz, 1H), 7.54 (td, *J* = 1.0, 7.5 Hz, 1H), 7.62-7.69 (m, 2H), 7.93 (d, *J* = 8.1 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 21.4, 51.4, 88.0, 104.0, 119.8, 121.3, 129.1, 129.4, 129.5, 131.2, 131.5, 131.7, 135.6, 137.6, 150.1, 162.1, 166.0; IR (KBr): 3075, 2948, 1697, 1636, 1265, 1035, 848, 754; HRMS (ESI): $[M]^+$ calcd for $C_{19}H_{17}O_3$, 293.1173 ; found, m/z 293.1174; NOE (δ =5.67) 2.3%, NOE (δ =6.31) 2.4, 3.4%.

Methyl (*Z*)-2-[(*Z*)-3-(4-trifluoromethylbenzylidene)isobenzofuran-1(3*H*)-ylidene]acetate (4g):



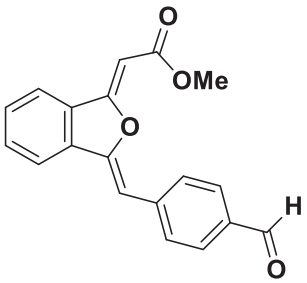
Pale yellow solid; m.p.: 135 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.89 (s, 3H), 5.73 (s, 1H), 6.33 (s, 1H), 7.50 (td, *J* = 0.9, 7.5 Hz, 1H), 7.58 (td, *J* = 0.9, 7.5 Hz, 1H), 7.66-7.74 (m, 4H), 8.12 (d, *J* = 8.1 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 51.5, 89.2, 102.1, 120.2, 121.4, 122.9, 125.6 (q, *J* = 3.4 Hz,), 128.8 (d, *J* = 32.6 Hz), 123.0, 124.3 (d, *J* = 271.5 Hz), 131.8, 132.2, 135.0, 137.6, 152.2, 161.7, 165.6; IR (KBr): 3066, 1682, 1474, 1331, 1267, 1162, 1070, 1026, 874, 760; HRMS (ESI): $[M]^+$ calcd for $C_{19}H_{14}F_3O_3$, 347.0890; found, m/z 347.0893; NOE (δ = 5.73) 2.3%, NOE (δ = 6.33) 2.9 %, 3.7%.

Methyl (*Z*)-2-[(*Z*)-3-(4-acetylbenzylidene)isobenzofuran-1(3*H*)-ylidene]acetate (4h):



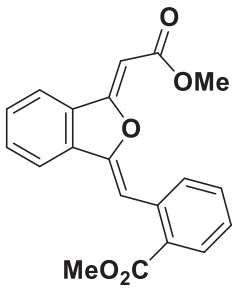
Pale yellow solid; m.p.: 194 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.63 (s, 3H), 3.90 (s, 3H), 5.73 (s, 1H), 6.33 (s, 1H), 7.49 (td, *J* = 1.0, 7.6 Hz, 1H), 7.57 (td, *J* = 1.0, 7.6 Hz, 1H), 7.65-7.67 (m, 1H), 7.70-7.72 (m, 1H), 8.01-8.05 (m, 2H), 8.08-8.12 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 26.6, 51.5, 89.2, 102.6, 120.2, 121.4, 128.8, 129.2, 130.0, 131.7, 132.1, 135.0, 135.3, 138.8, 152.4, 161.6, 165.6, 197.6; IR (KBr): 3085, 2953, 1710, 1668, 1645, 1276, 1154, 856; HRMS (ESI): $[M]^+$ calcd for $C_{20}H_{17}O_4$, 321.1121; found, m/z 321.1129; NOE (δ = 5.69) 8.4%, NOE (δ = 6.31) 10.8 %, 16.3%.

Methyl (*Z*)-2-[(*Z*)-3-(4-formylbenzylidene)isobenzofuran-1(3*H*)-ylidene]acetate (4i):



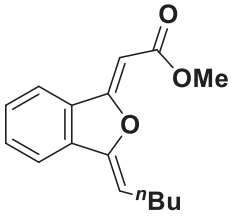
Pale yellow solid; m.p.: 144 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.90 (s, 3H), 5.74 (s, 1H), 6.33 (s, 1H), 7.50 (t, *J* = 7.3 Hz, 1H), 7.58 (t, *J* = 7.3 Hz, 1H), 7.67 (d, *J* = 7.8 Hz, 1H), 7.72 (d, *J* = 7.8 Hz, 1H), 7.94 (d, *J* = 8.3 Hz, 2H), 8.16 (d, *J* = 8.3 Hz, 2H), 10.00 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 51.5, 89.6, 102.4, 120.3, 121.4, 129.6, 130.1, 130.2, 131.8, 132.2, 134.6, 134.8, 140.3, 152.9, 161.5, 165.5, 191.7; IR (KBr):3025, 2794, 2717, 1721, 1691, 1594, 1151, 1041, 852, 765; HRMS (ESI): $[M]^+$ calcd for $C_{19}H_{15}O_4$, 307.0965; found, m/z 307.0966; NOE (δ = 5.69) 11.2%, NOE (δ = 6.29) 12.3 %, 19.7%.

Methyl (*Z*)-2-[(*Z*)-(2-methoxycarbonylbenzylidene)isobenzofuran-1(3*H*)-ylidene]acetate (4j):



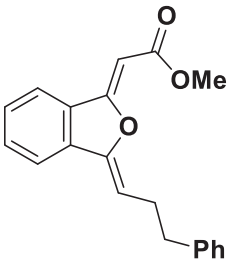
Pale yellow solid; m.p.: 139 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.85 (s, 3H), 3.93 (s, 3H), 5.68 (s, 1H), 7.31 (t, *J* = 7.6 Hz, 1H), 7.44 (t, *J* = 7.4 Hz, 1H), 7.51-7.57 (m, 2H), 7.60-7.70 (m, 2H), 7.77 (d, *J* = 7.6 Hz, 1H), 7.96 (d, *J* = 8.1 Hz, 1H), 8.76 (d, *J* = 8.1 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 51.3, 52.1, 88.6, 100.2, 120.5, 121.2, 126.8, 128.2, 129.6, 130.7, 131.0, 131.6, 131.8, 132.3, 134.7, 135.6, 151.8, 161.9, 165.7, 167.9; IR (KBr): 2047, 1724, 1684, 1487, 1425, 1265, 1243, 1107, 1034, 759; HRMS (ESI): $[M]^+$ calcd for $C_{20}H_{17}O_5$, 337.1071 ; found, m/z 337.1073; NOE (δ = 5.67) 2.6%.

Methyl (*Z*)-2-[(*Z*)-3-pentylideneisobenzofuran-1(3*H*)-ylidene]acetate (4k):



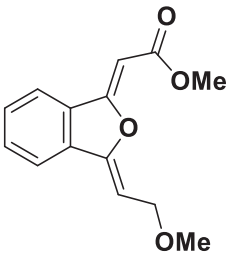
Pale yellow viscous oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.95 (t, *J* = 7.2 Hz, 3H), 1.37-1.48 (m, 2H), 1.49-1.58 (m, 2H), 2.56 (q, *J* = 7.4 Hz, 2H), 3.80 (s, 3H), 5.48 (t, *J* = 7.7 Hz, 1H), 5.56 (s, 1H), 7.37-7.42 (m, 1H), 7.47-7.52 (m, 1H), 7.56 (dd, *J* = 0.9, 7.9 Hz, 1H), 7.59 (dd, *J* = 1.0, 7.7 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 13.9, 22.4, 25.4, 31.6, 51.2, 86.6, 105.9, 119.7, 121.2, 128.9, 131.4, 132.5, 134.6, 151.5, 162.6, 166.2; IR (KBr): 2954, 2858, 1868, 1647, 1434, 1270, 1152, 1038, 767; HRMS (ESI): $[M]^+$ calcd for $C_{16}H_{19}O_3$, 259.1329 ; found, m/z 259.1326.

Methyl (*Z*)-2-[(*Z*)-3-(3-phenylpropylidene)isobenzofuran-1(3*H*)-ylidene]acetate (4l):



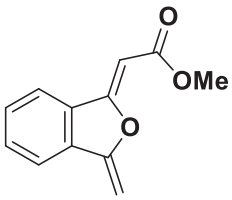
Pale yellow viscous oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.87-2.91 (m, 4H), 3.81 (s, 3H), 5.45-5.50 (m, 1H), 5.57 (s, 1H), 7.17-7.23 (m, 1H), 7.26-7.33 (m, 4H), 7.37-7.42 (m, 1H), 7.45-7.54 (m, 2H), 7.59 (dd, *J* = 0.7, 8.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 27.2, 35.5, 51.2, 86.9, 104.4, 119.8, 121.2, 126.0, 128.4, 128.6, 129.0, 131.4, 132.6, 134.5, 141.4, 151.9, 162.4, 166.1; IR (KBr): 3025, 2947, 1650, 1473, 1270, 1148, 1030; HRMS (ESI): $[M]^+$ calcd for $C_{20}H_{19}O_3$, 307.1329; found, m/z 307.1332.

Methyl (*Z*)-2-[(*Z*)-3-(2-methoxyethylidene)isobenzofuran-1(3*H*)-ylidene]acetate (4m):



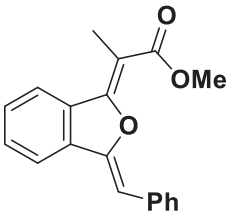
Colorless viscous oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.44 (s, 3H), 3.80 (s, 3H), 4.47 (d, *J* = 7.0 Hz, 2H), 5.60 (t, *J* = 7.2 Hz, 1H), 5.61 (s, 1H), 7.43-7.48 (m, 1H), 7.50-7.56 (m, 1H), 7.59-7.63 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 51.2, 58.3, 88.4, 87.9, 100.7, 120.3, 121.1, 129.8, 131.6, 132.9, 133.9, 153.0, 161.9, 165.8; IR (KBr): 2949, 1775, 1686, 1655, 1435, 1273, 1155, 769; HRMS (ESI): $[M]^+$ calcd for $C_{14}H_{15}O_4$, 247.0965 ; found, m/z 247.0968.

Methyl (*Z*)-2-[(*Z*)-3-methyleneisobenzofuran-1(3*H*)-ylidene]acetate (4n):



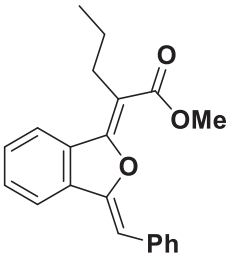
Viscous oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.80 (s, 3H), 5.06 (d, *J* = 3.1 Hz, 1H), 5.20 (d, *J* = 3.1 Hz, 1H), 5.60 (s, 1H), 7.45-7.50 (m, 1H), 7.52-7.57 (m, 1H), 7.59-7.67 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 51.3, 87.6, 87.7, 120.7, 121.1, 129.9, 131.6, 133.2, 134.0, 157.7, 162.0, 165.8; IR (KBr): 2947, 1686, 1638, 1467, 1433, 1152, 1052, 765; HRMS (ESI): $[M]^+$ calcd for $C_{12}H_{11}O_3$, 203.0703 ; found, m/z 203.0700.

Methyl (*Z*)-2-[(*Z*)-3-benzylideneisobenzofuran-1(3*H*)-ylidene]propionate (4o):



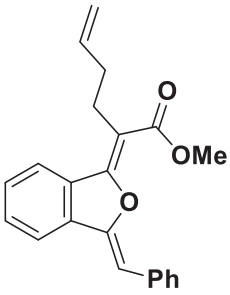
Pale yellow solid; m.p.: 118 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.35 (s, 3H), 3.98 (s, 3H), 6.21 (s, 1H), 7.25 (t, *J* = 7.3 Hz, 1H), 7.38-7.52 (m, 4H), 7.69 (d, *J* = 7.6 Hz, 1H), 7.86 (d, *J* = 7.6 Hz, 1H), 7.99 (d, *J* = 7.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 13.4, 52.0, 100.9, 101.4, 119.9, 125.3, 127.0, 128.5, 129.0, 129.2, 130.3, 132.5, 134.5, 136.4, 150.4, 157.5, 168.2; IR (KBr): 3050, 1672, 1619, 1128, 1108, 753; HRMS (ESI): $[M]^+$ calcd for $C_{19}H_{17}O_3$, 293.1173; found, m/z 293.1177; NOE (δ = 2.35) 4.6%, NOE (δ = 6.22) 2.5 %, 2.8%.

Methyl (*Z*)-2-[(*Z*)-3-benzylideneisobenzofuran-1(3*H*)-ylidene]pentanoate (4p):

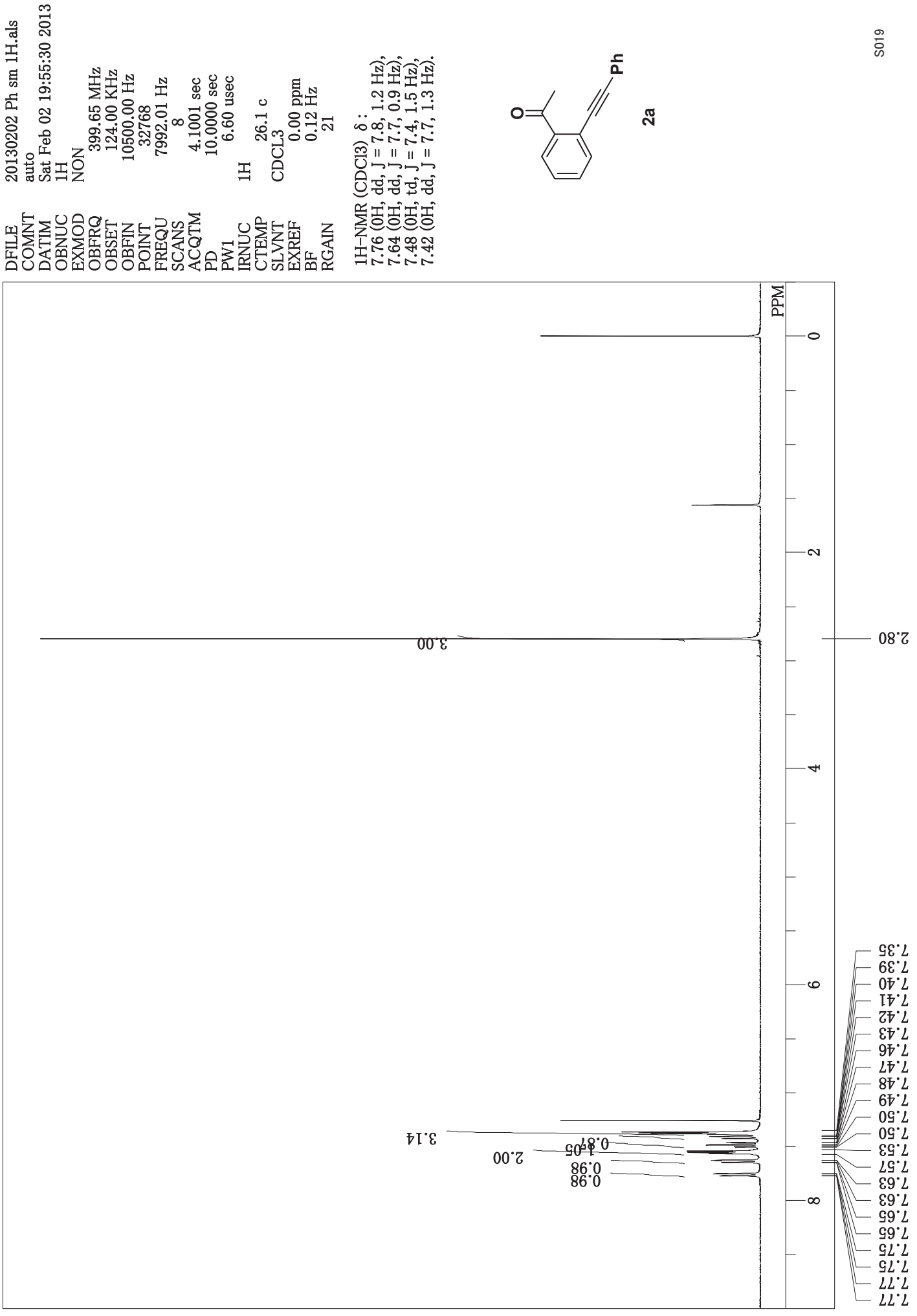


Pale yellow solid; m.p.: 93 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.07 (t, *J* = 7.3 Hz, 3H), 1.59-1.69 (m, 2H), 2.77 (t, *J* = 8.0 Hz, 2H), 3.98 (s, 3H), 6.23 (s, 1H), 7.22-7.29 (m, 1H), 7.37-7.54 (m, 4H), 7.70 (d, *J* = 7.2 Hz, 1H), 7.77 (d, *J* = 7.4 Hz, 1H), 7.99 (d, *J* = 7.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.0, 22.6, 29.0, 51.9, 101.3, 107.0, 120.0, 124.8, 127.0, 128.5, 129.0, 129.4, 130.4, 131.9, 134.5, 136.6, 150.2, 157.5, 168.2; IR (KBr): 2956, 1682, 1618, 1137, 1109, 757; HRMS (ESI): $[M]^+$ calcd for $C_{21}H_{21}O_3$, 321.1486; found, m/z 321.1488; NOE (δ = 2.78) 9.9%, NOE (δ = 6.23) 2.7 %, 3.3%.

Methyl (*Z*)-2-[(*Z*)-3-benzylideneisobenzofuran-1(3*H*)-ylidene]hex-5-enoate (4q):



Pale yellow solid; m.p.: 89 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.30-2.43 (m, 2H), 2.85-2.94 (m, 2H), 3.99 (s, 3H), 5.04-5.07 (m, 1H), 5.12-5.16 (m, 1H), 5.92-6.04 (m, 1H), 6.24 (s, 1H), 7.22-7.29 (m, 1H), 7.36-7.55 (m, 4H), 7.72 (d, *J* = 7.8 Hz, 1H), 7.79 (d, *J* = 7.8 Hz, 1H), 8.00 (d, *J* = 7.3 Hz, 2H),; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 26.4, 33.3, 52.0, 101.5, 106.1, 115.2, 120.0, 124.9, 127.1, 128.5, 129.0, 129.5, 130.5, 131.7, 134.4, 136.6, 137.6, 150.2, 157.8, 167.9; IR (KBr): 3075, 2943, 1675, 1618, 1321, 1108, 764; HRMS (ESI): $[M]^+$ calcd for $C_{22}H_{21}O_3$, 333.1485; found, m/z 333.1482; NOE (δ = 6.22) 13.0%, 16.7%.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COMNT auto
DATIM Sat Feb 02 19:55:30 2013
OBNUC 1H
EXMOD NON
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OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 7992.01 Hz
SCANS 8
ACQTM 4.1001 sec
PD 10.0000 sec
PW1 6.60 usec
IRNUC 1H
CTEMP 26.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 21
1H-NMR (CDCl3) δ :
7.76 (0H, dd, J = 7.8, 1.2 Hz),
7.64 (0H, dd, J = 7.7, 0.9 Hz),
7.48 (0H, td, J = 7.4, 1.5 Hz),
7.42 (0H, dd, J = 7.7, 1.3 Hz).
O
Ph
2a
S019
PPM
0
2
4
6
8
2.80
3.00
3.14
2.00
0.98
0.98
1.05
0.87
7.77
7.77
7.75
7.75
7.65
7.65
7.63
7.63
7.57
7.53
7.50
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7.49
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7.42
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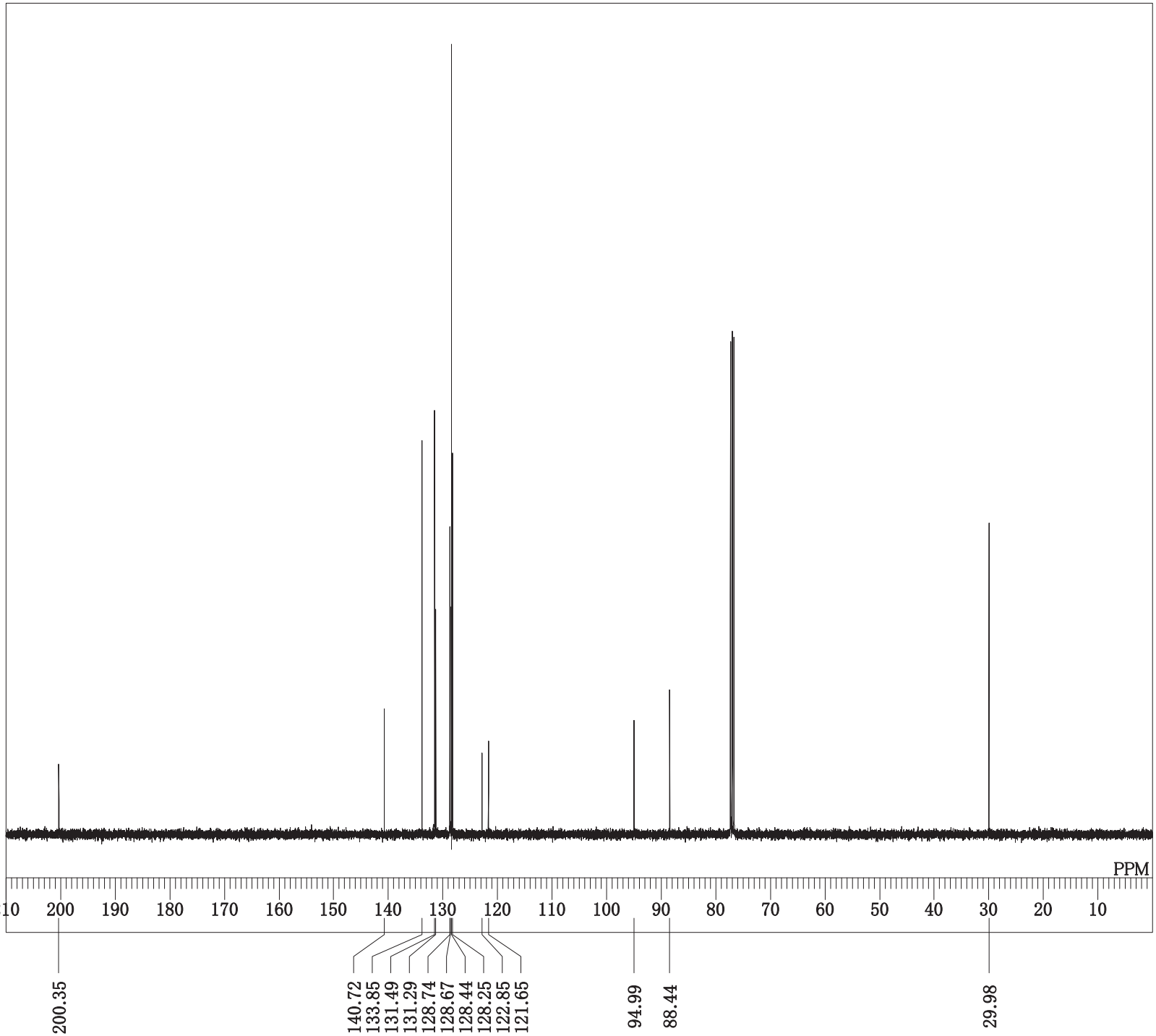
7.39
7.35



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OBSET	4.64 KHz
OBFIN	8.74 Hz
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FREQU	30788.18 Hz
SCANS	512
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	2.87 usec
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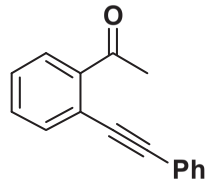
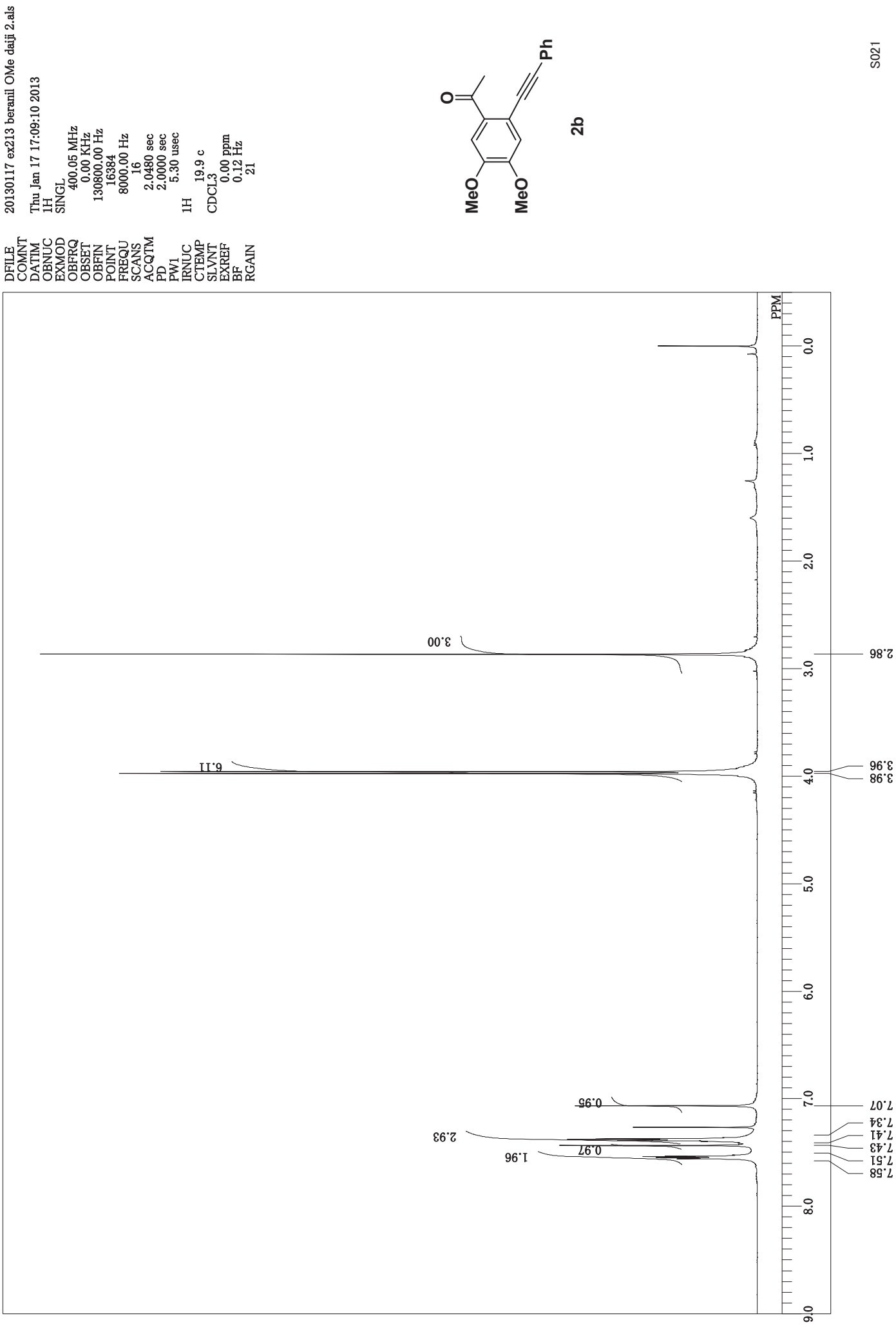
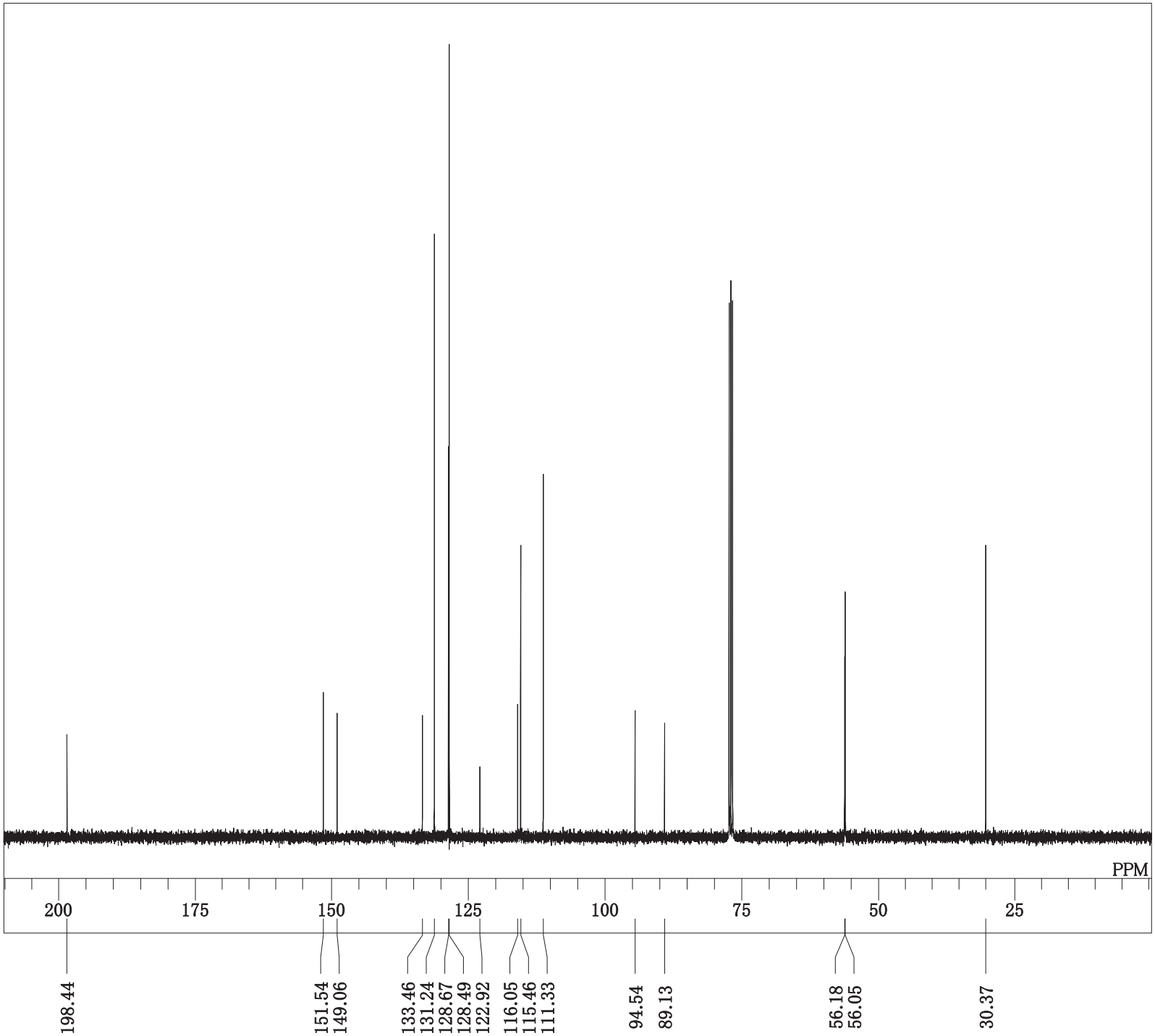
CTEMP	22.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.01 Hz
RGAIN	46



2a


20130117 ex213 beranil OMe daiji 2.als
Thu Jan 17 17:09:10 2013
1H
SINGL
400.05 MHz
0.00 KHz
130800.00 Hz
16384
8000.00 Hz
16
2.0480 sec
2.0000 sec
5.30 usec
1H
19.9 c
CDCL3
0.00 ppm
0.12 Hz
21
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
O
Ph
MeO
MeO
2b
S021
PPM
0.0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
3.00
6.11
0.96
2.93
0.97
1.96
2.86
3.96
3.98
7.07
7.34
7.41
7.43
7.51
7.58



DFILE	20130117 ex213 BCM beraniru -1.als
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OBNUC	13C
EXMOD	single_pulse_dec
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OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	32768
FREQU	30788.18 Hz
SCANS	1056
ACQTM	1.0643 sec
PD	2.0000 sec
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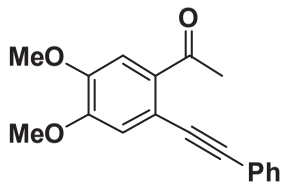
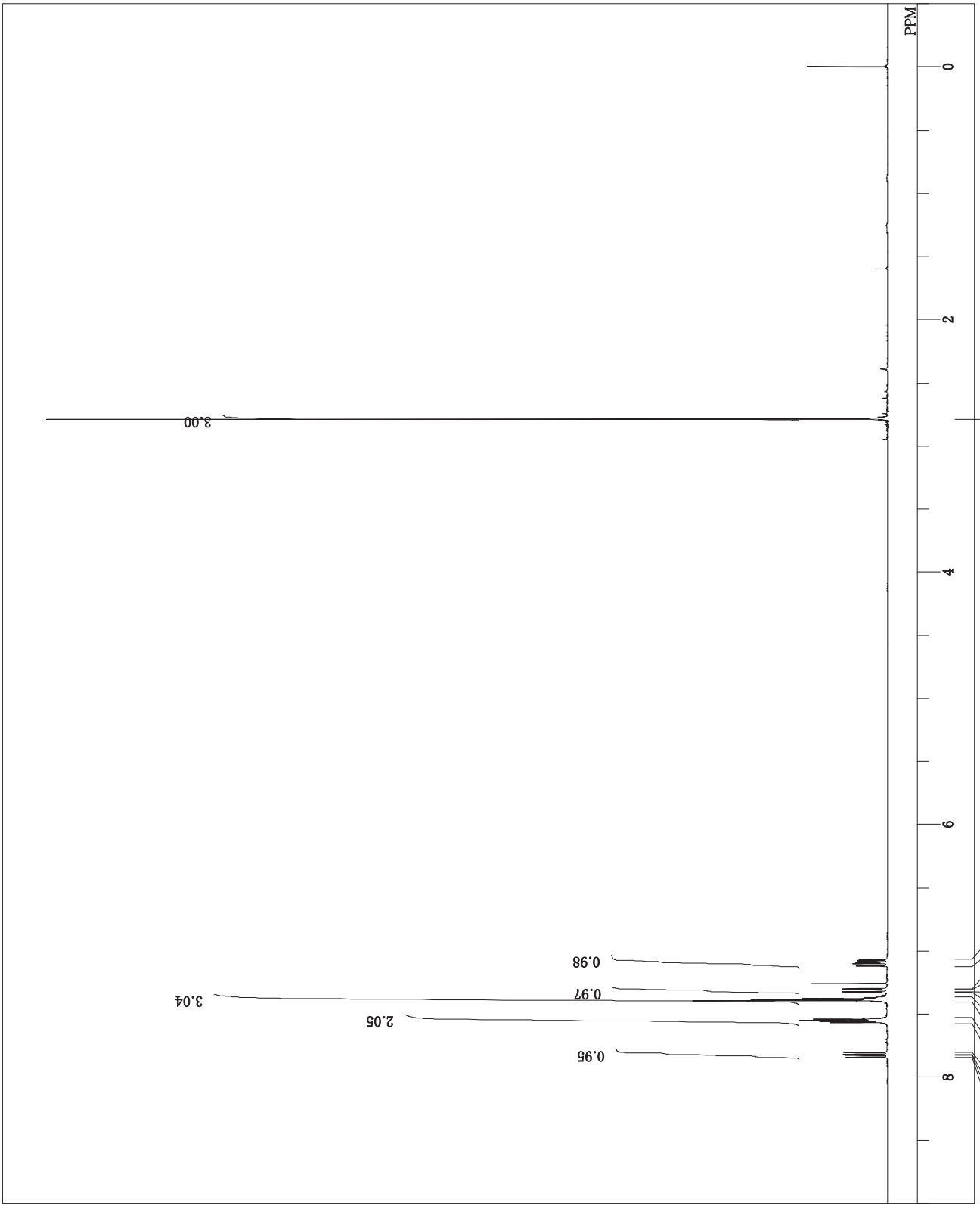
IRNUC	1H
CTEMP	19.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	44



2b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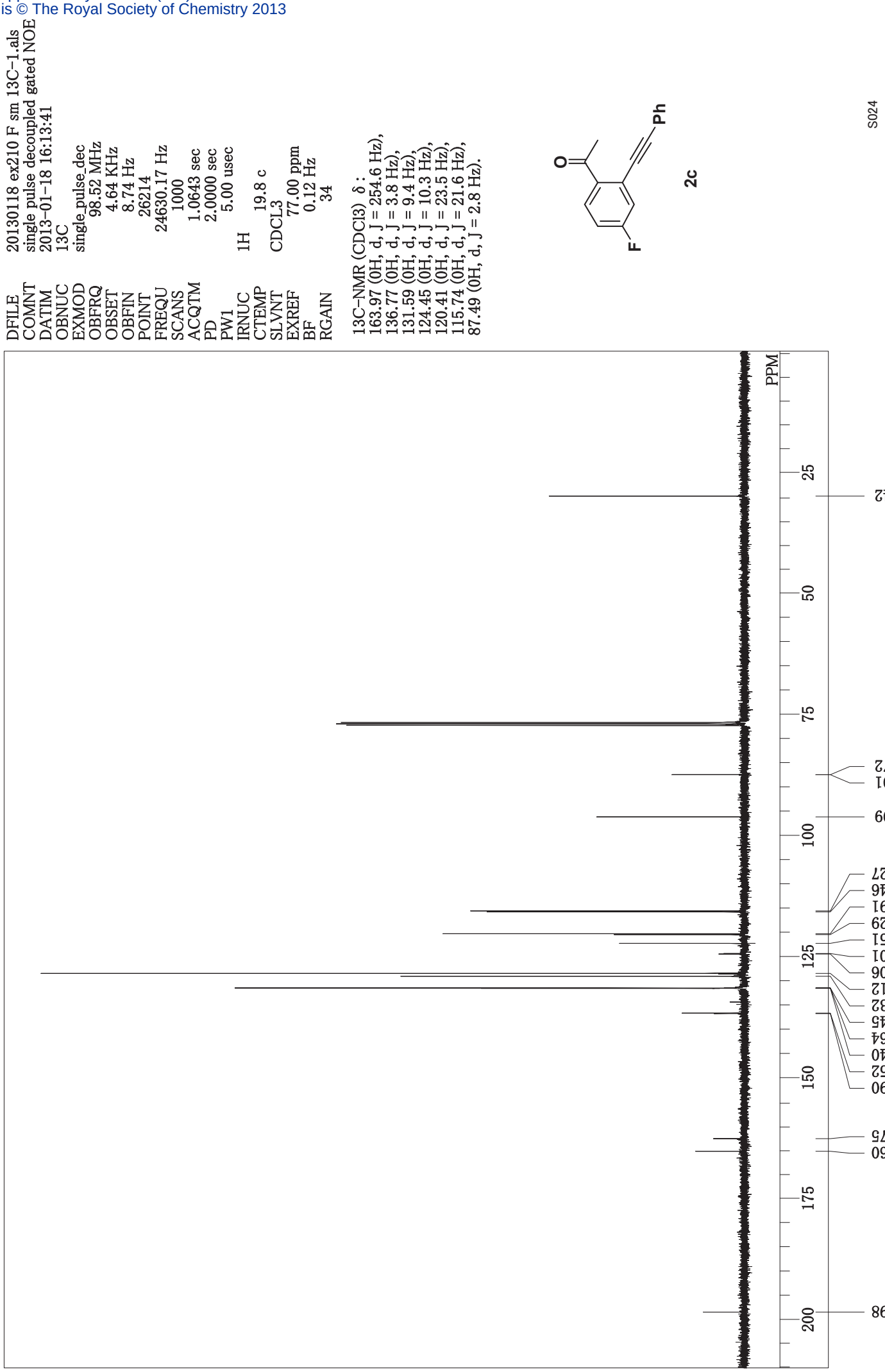
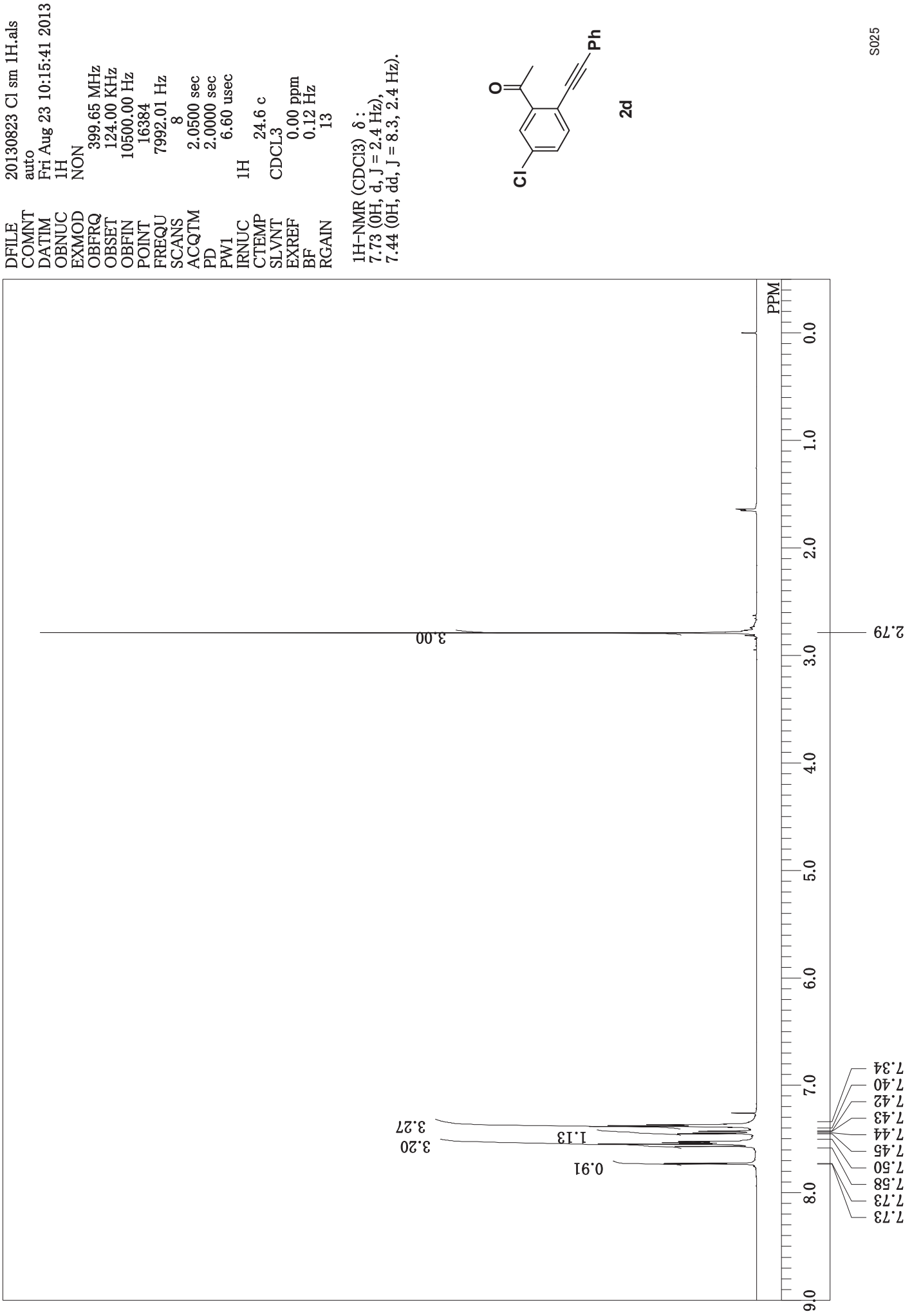
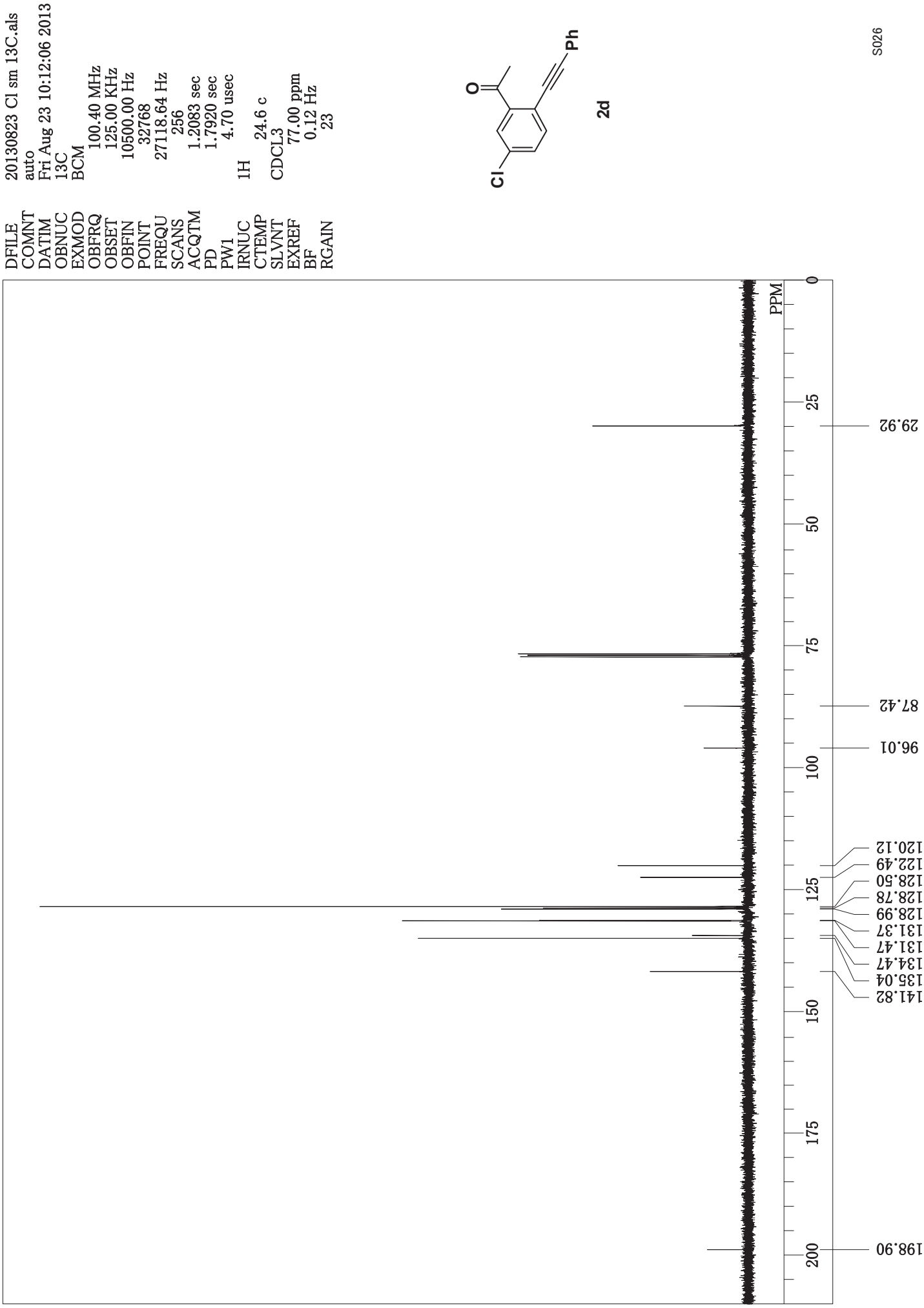
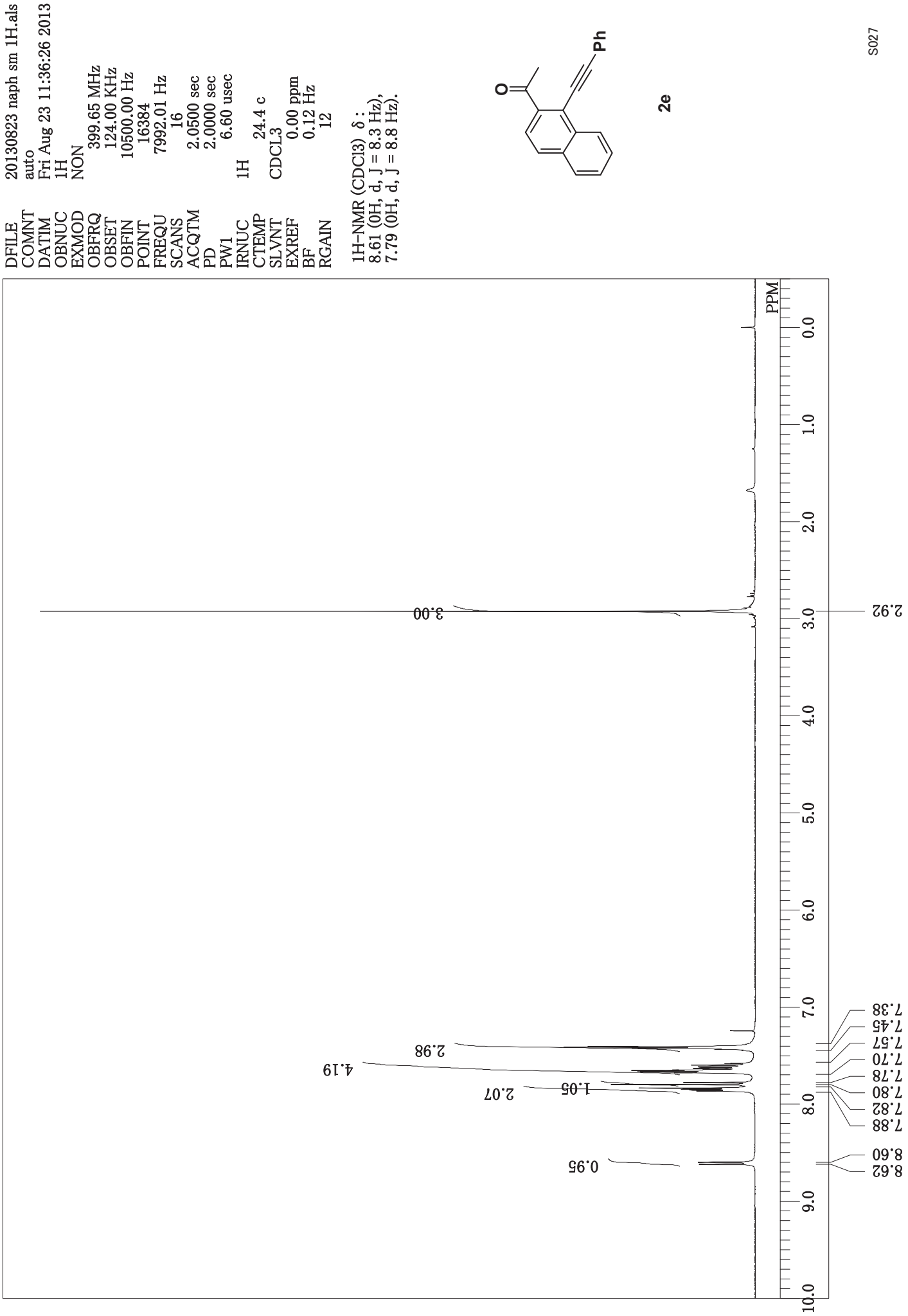
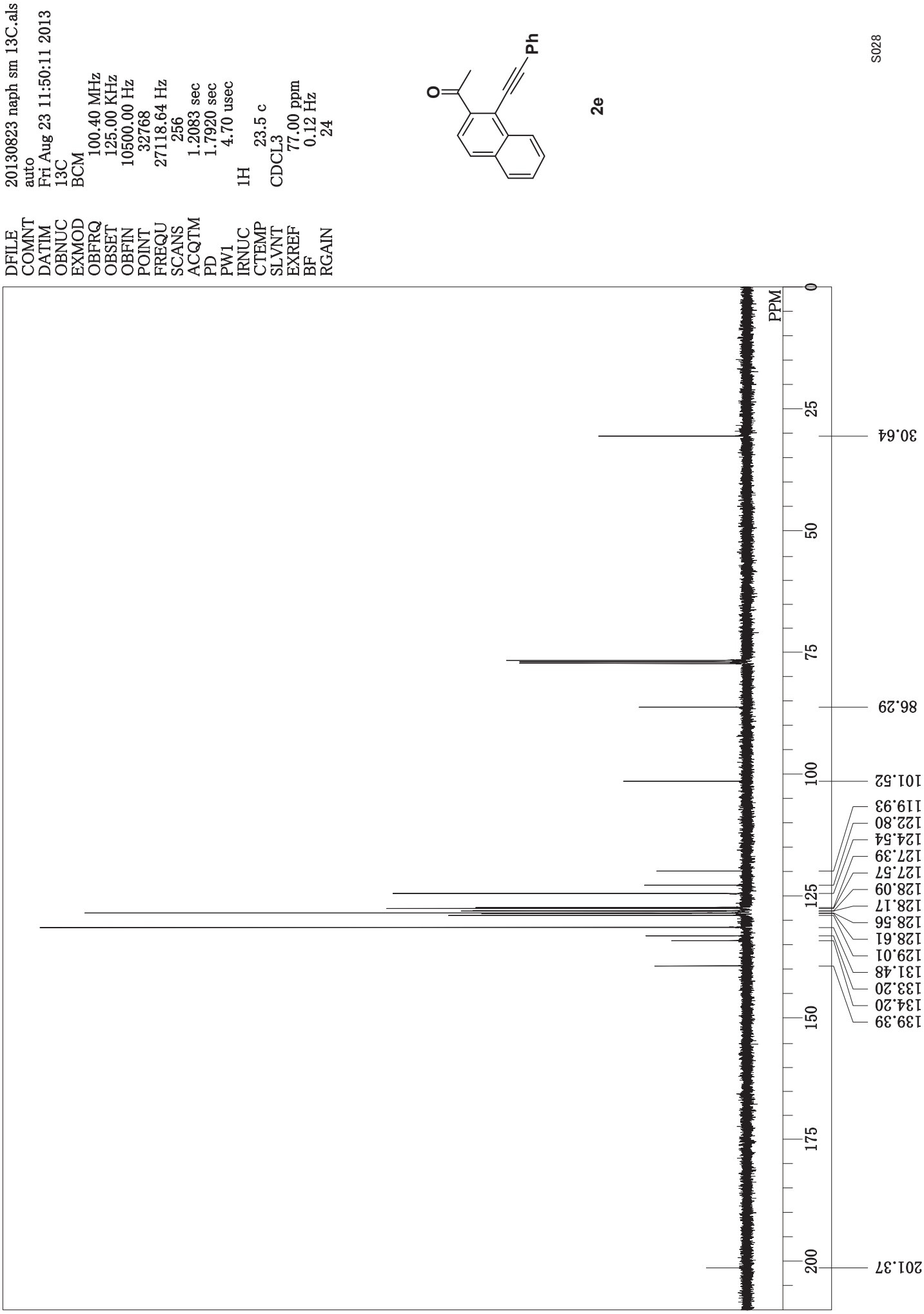
EXMOD single_pulse.ex2
OBFRQ 391.78 MHz
OBSET 8.51 KHz
OBFIN 3.34 Hz
POINT 26214
FREQU 5882.26 Hz
SCANS 16
ACQTM 4.4564 sec
PD 3.0000 sec
PW1 5.20 usec
IRNUC 1H
CTEMP 19.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 42
1H-NMR (CDCl3) δ :
7.83 (1H, dd, J = 8.8, 5.8 Hz),
7.32 (1H, dd, J = 9.1, 2.6 Hz).
O
Ph
F
2c
S023
PPM
0
2
4
6
8
3.00
2.79
0.98
0.97
3.04
2.05
0.95
7.84
7.83
7.82
7.81
7.58
7.53
7.41
7.36
7.33
7.32
7.31
7.30
7.12
7.07

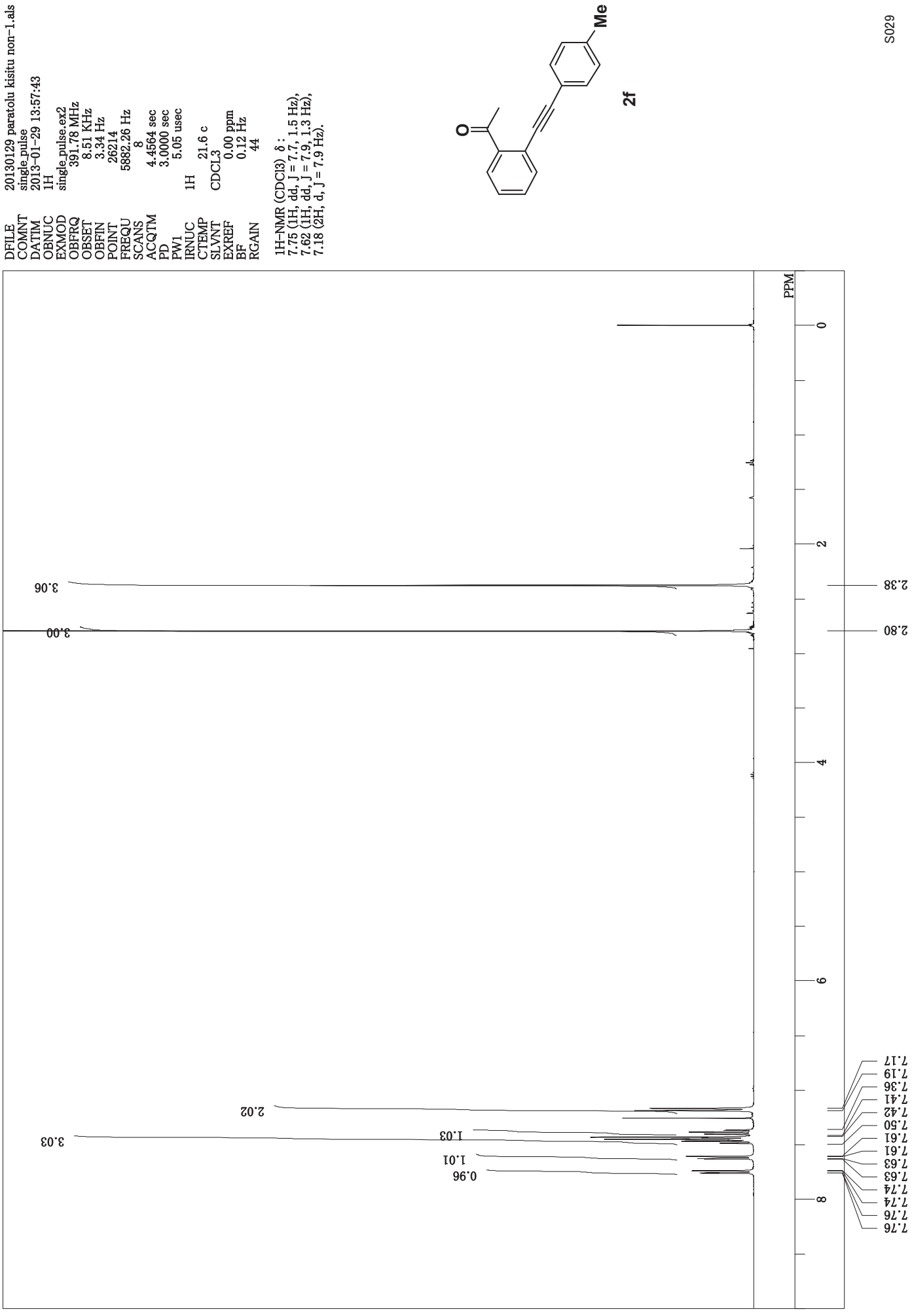

DFILE 20130118 ex210 F sm 13C-1.als
COMNT single pulse decoupled gated NOE
DATIM 2013-01-18 16:13:41
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.17 Hz
SCANS 1000
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 5.00 usec
IRNUC 1H
CTEMP 19.8 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 34
13C-NMR (CDCl3) δ :
163.97 (0H, d, J = 254.6 Hz),
136.77 (0H, d, J = 3.8 Hz),
131.59 (0H, d, J = 9.4 Hz),
124.45 (0H, d, J = 10.3 Hz),
120.41 (0H, d, J = 23.5 Hz),
115.74 (0H, d, J = 21.6 Hz),
87.49 (0H, d, J = 2.8 Hz).
O
Ph
F
2c
S024
PPM
25
50
75
100
125
150
175
200
29.942
87.472
87.501
96.199
115.627
115.846
120.291
120.529
122.351
124.401
124.506
128.512
129.132
131.545
131.564
131.640
136.752
136.790
162.675
165.260
198.498

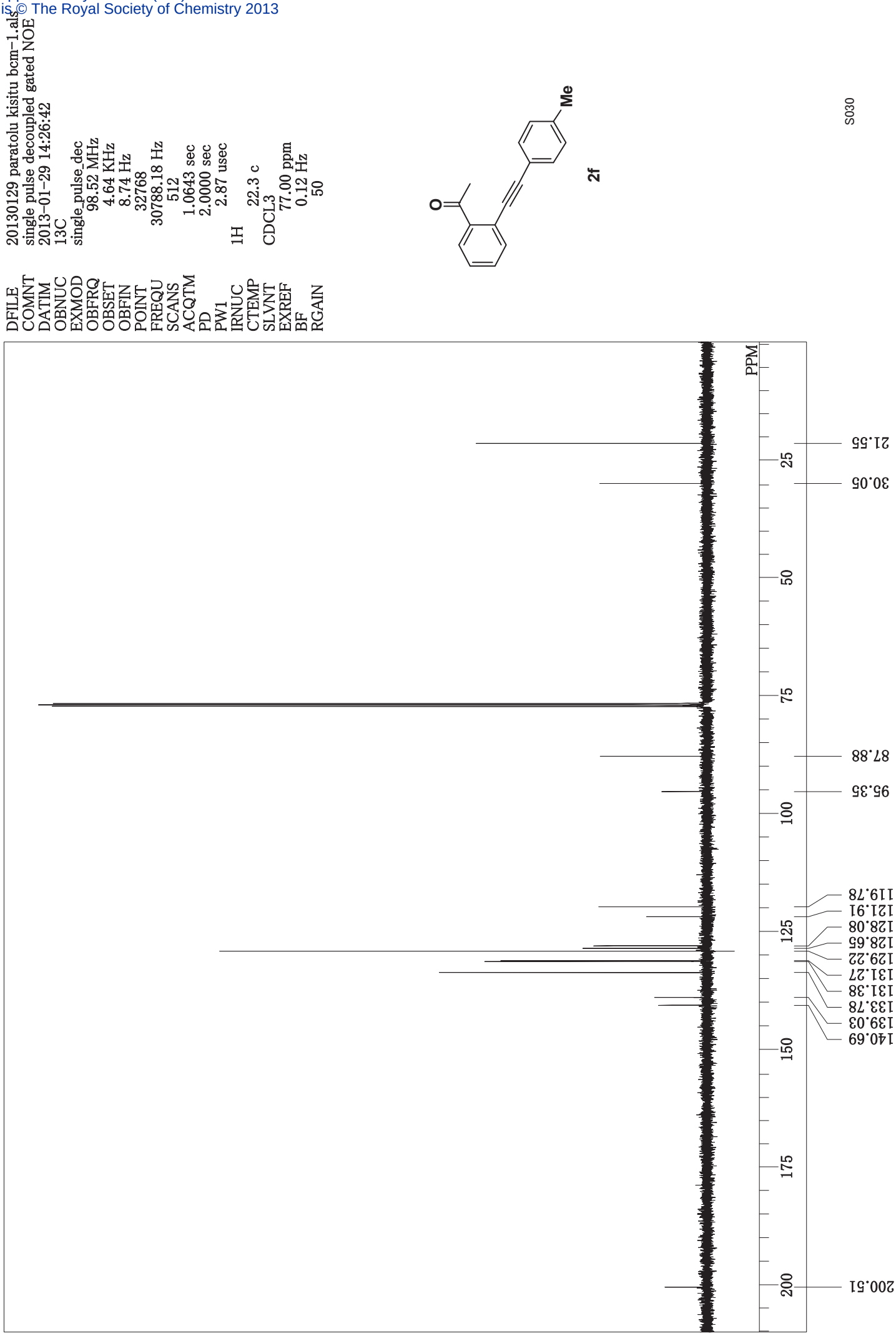

DFILE 20130823 Cl sm 1H.als
COMNT auto
DATIM Fri Aug 23 10:15:41 2013
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 8
ACQTM 2.0500 sec
PD 2.0000 sec
PW1 6.60 usec
IRNUC 1H
CTEMP 24.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 13
1H-NMR (CDCl3) δ :
7.73 (0H, d, J = 2.4 Hz),
7.44 (0H, dd, J = 8.3, 2.4 Hz).
O
Cl
Ph
2d
S025
PPM
0.0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
3.00
2.79
0.91
1.13
3.20
3.27
7.34
7.40
7.42
7.43
7.44
7.45
7.50
7.58
7.73
7.73

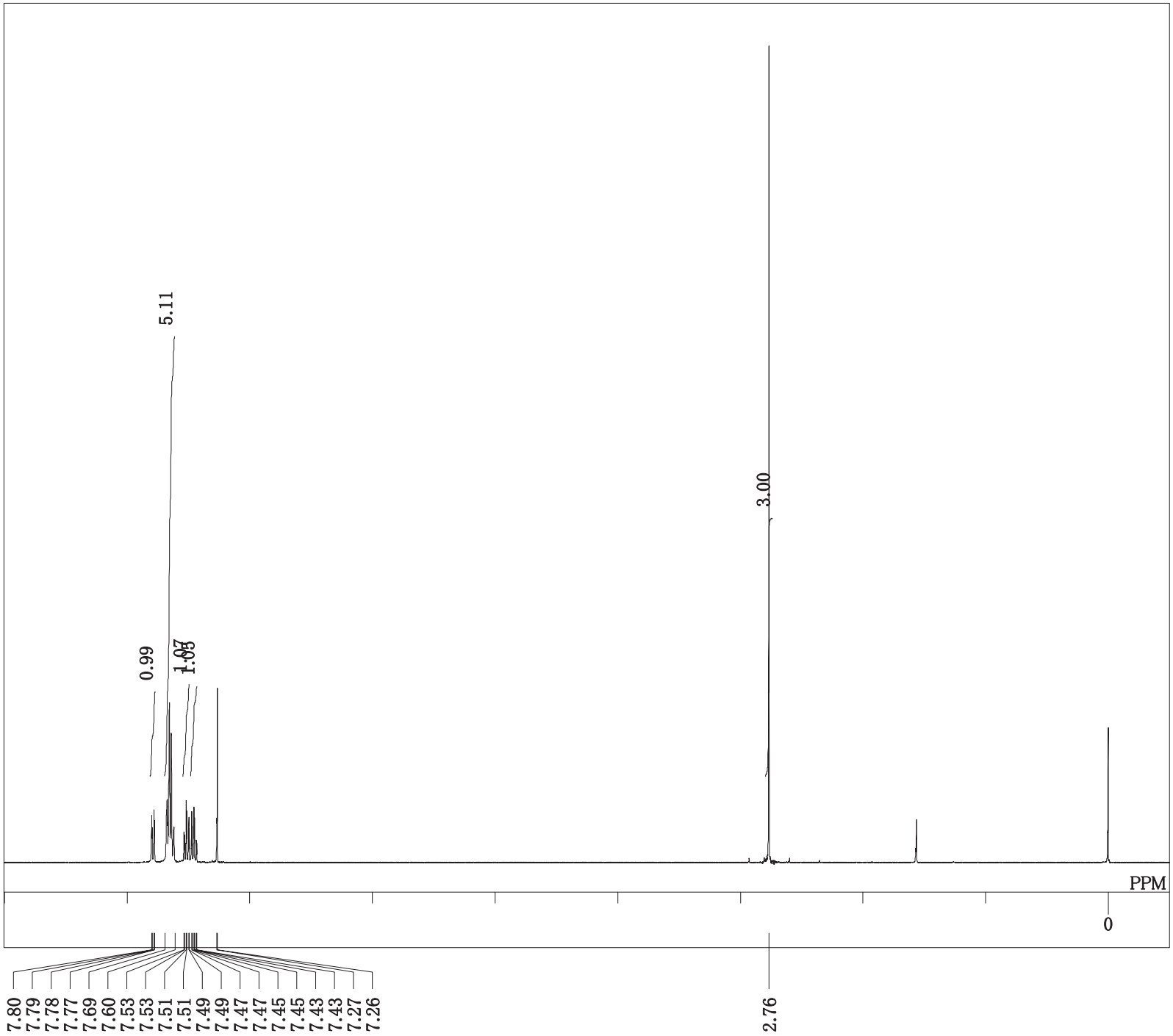

DFILE 20130823 Cl sm 13C.als
COMNT auto
DATIM Fri Aug 23 10:12:06 2013
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 256
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 4.70 usec
IRNUC 1H
CTEMP 24.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23
O
Ph
Cl
2d
S026
PPM
0
25
50
75
100
125
150
175
200
29.92
87.42
96.01
120.12
122.49
128.50
128.78
128.99
131.37
131.47
134.47
135.04
141.82
198.90


DFILE 20130823 naph sm 1H.als
COMNT auto
DATIM Fri Aug 23 11:36:26 2013
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 16
ACQTM 2.0500 sec
PD 2.0000 sec
PW1 6.60 usec
IRNUC 1H
CTEMP 24.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 12
1H-NMR (CDCl3) δ :
8.61 (0H, d, J = 8.3 Hz),
7.79 (0H, d, J = 8.8 Hz).
O
Ph
2e
S027
PPM
0.0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
10.0
3.00
2.98
4.19
2.07
1.05
0.95
2.92
7.38
7.45
7.57
7.70
7.78
7.80
7.82
7.88
8.60
8.62


20130823 naph sm 13C.als
auto
Fri Aug 23 11:50:11 2013
13C
BCM
100.40 MHz
125.00 KHz
10500.00 Hz
32768
27118.64 Hz
256
1.2083 sec
1.7920 sec
4.70 usec
1H
23.5 c
CDCL3
77.00 ppm
0.12 Hz
24
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
O
Ph
2e
S028
PPM
0
25
50
75
100
125
150
175
200
30.64
86.29
101.52
119.93
122.80
124.54
127.39
127.57
128.09
128.17
128.56
128.61
129.01
131.48
133.20
134.20
139.39
201.37

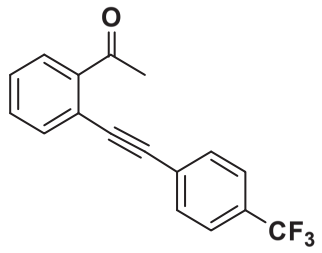




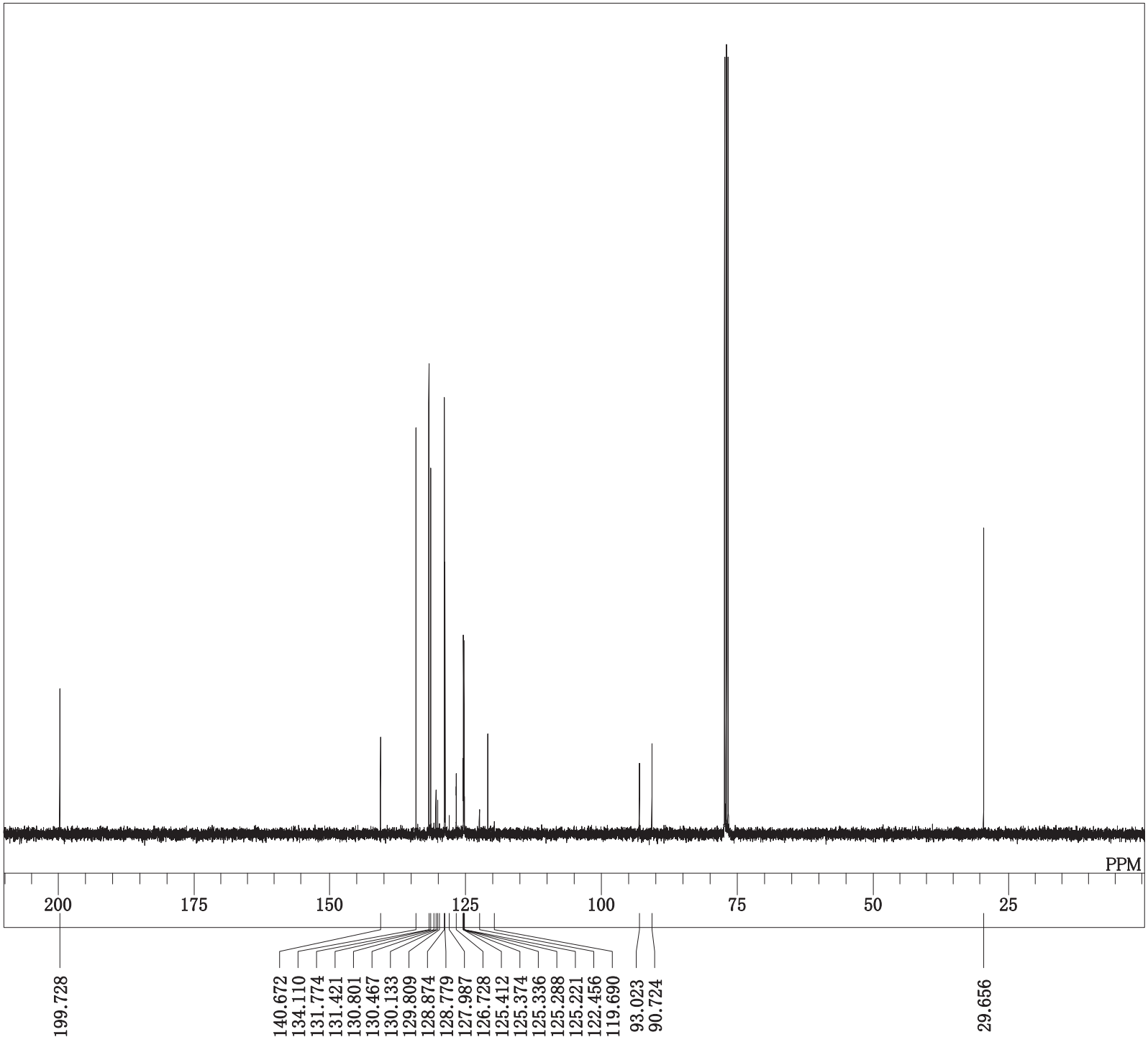


DFILE	Me Ph PhCF3-1.als
COMNT	single_pulse
DATIM	2013-04-17 10:08:29
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	26214
FREQU	5882.26 Hz
SCANS	8
ACQTM	4.4564 sec
PD	3.0000 sec
PW1	5.05 usec
IRNUC	1H
CTEMP	21.1 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.01 Hz
RGAIN	50

1H-NMR (CDCl3) δ :
7.79 (1H, dd, J = 7.7, 1.5 Hz),
7.51 (1H, td, J = 7.5, 1.6 Hz),
7.45 (1H, td, J = 7.6, 1.5 Hz).

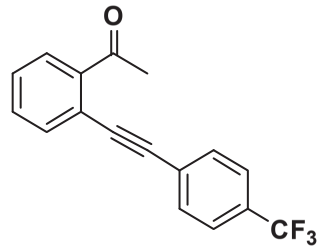


2g

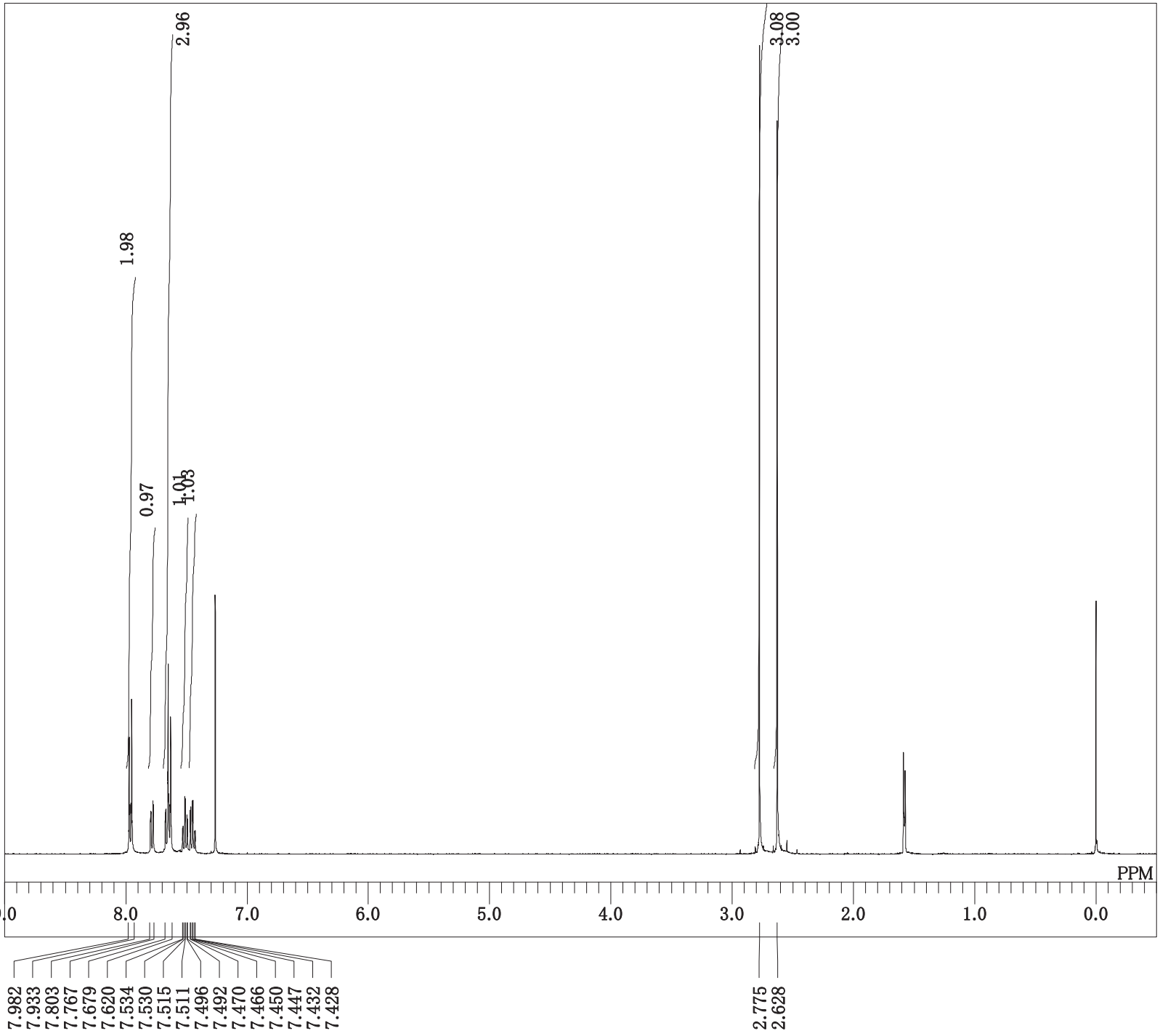


DFILE	20130417 Me Ph PhCF3 13C-2.als
COMNT	single pulse decoupled gated NOE
DATIM	2013-04-17 11:56:55
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.17 Hz
SCANS	1000
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	2.87 usec
IRNUC	1H
CTEMP	21.5 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	34

13C-NMR (CDCl3) δ :
130.30 (0H, q, J = 32.6 Hz),
125.35 (0H, q, J = 4.1 Hz),
123.85 (0H, q, J = 273.1 Hz).

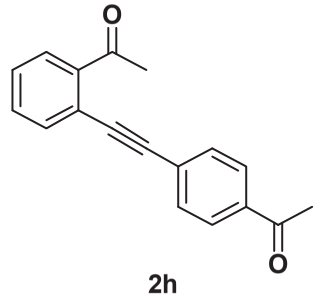


2g

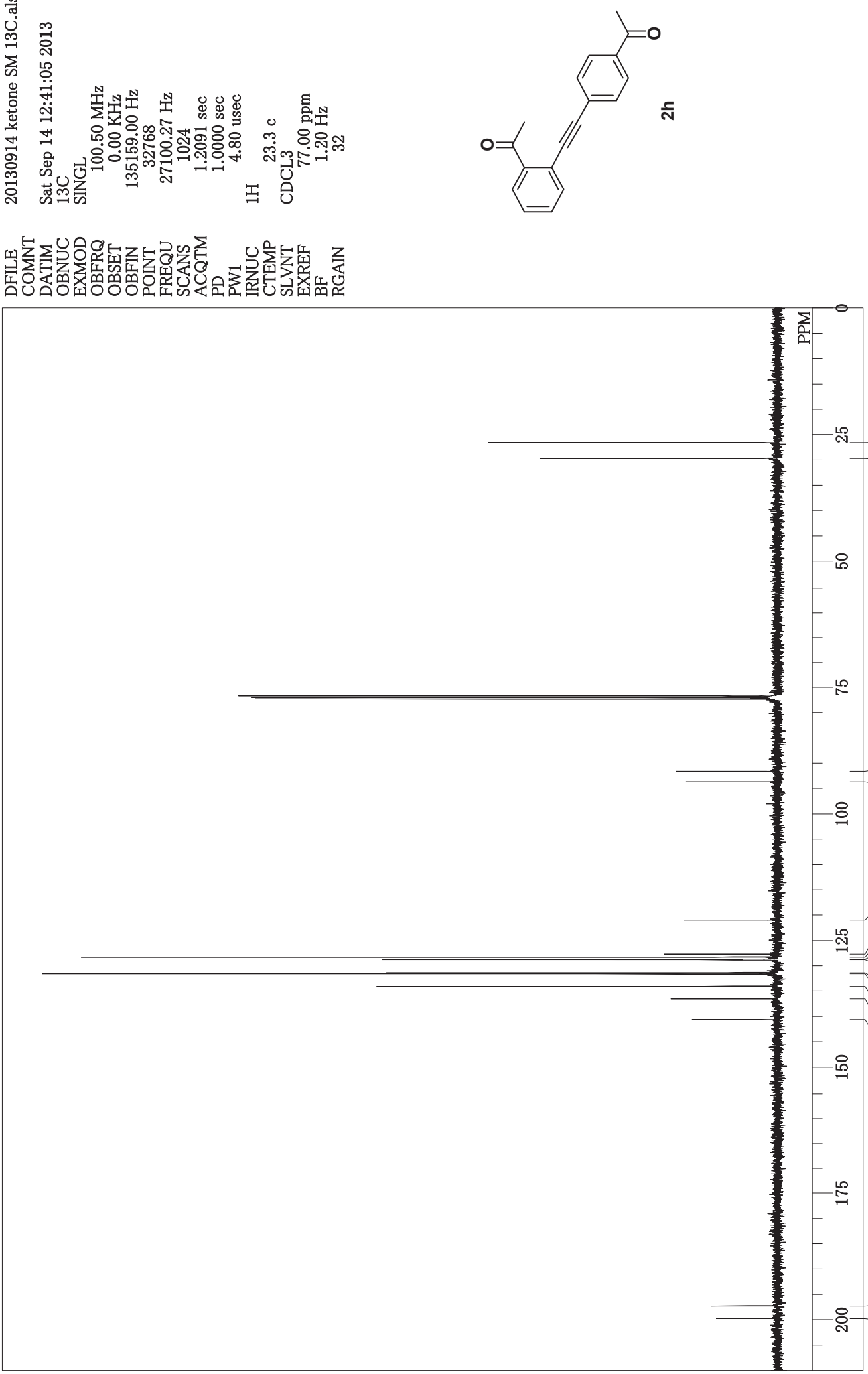


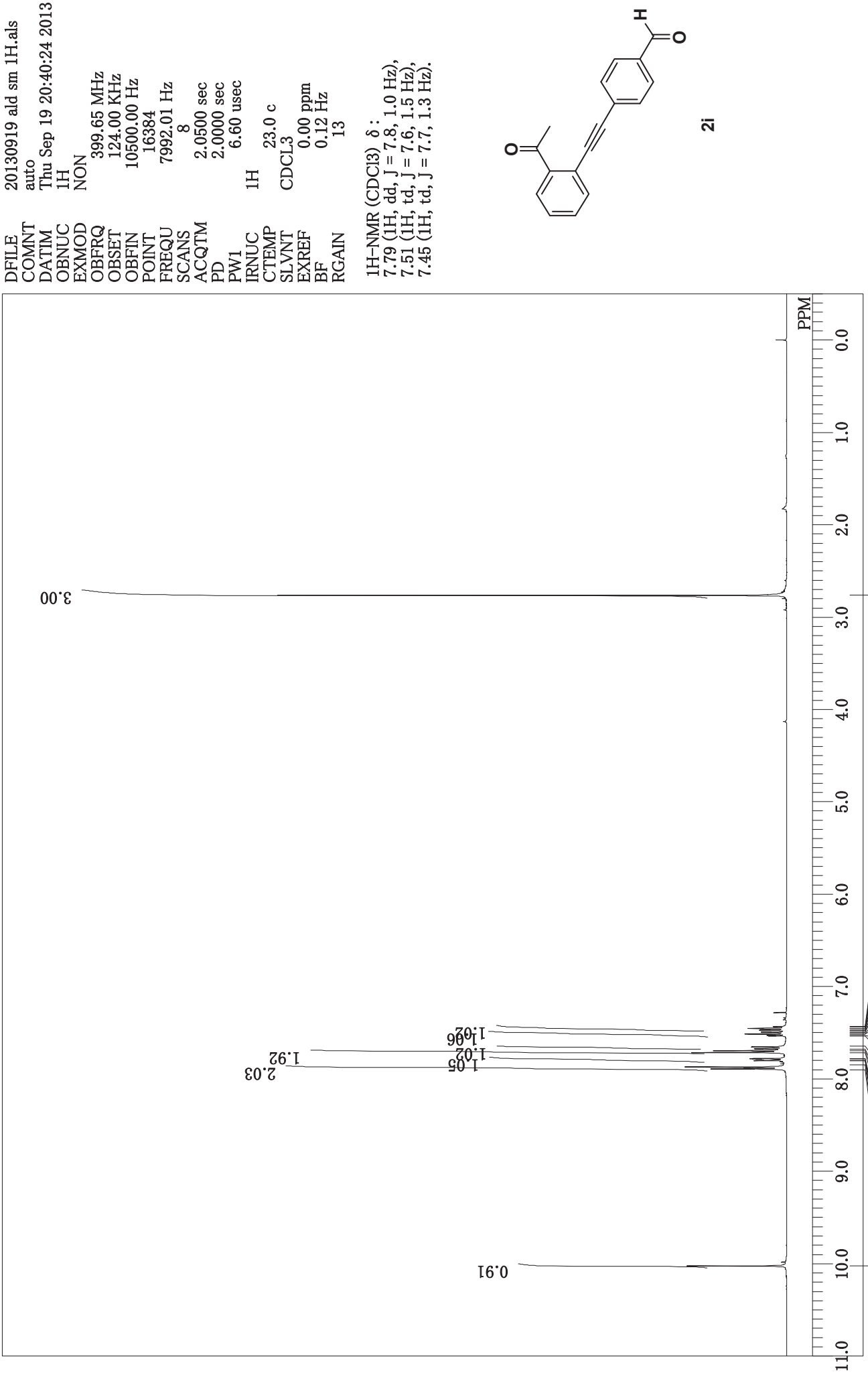
DFILE	20130917 ex1577 ketone sm 1H.als
COMNT	
DATIM	Tue Sep 17 09:51:55 2013
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.00 Hz
POINT	32768
FREQU	7992.01 Hz
SCANS	16
ACQTM	4.1001 sec
PD	10.0000 sec
PW1	6.60 usec
IRNUC	1H
CTEMP	22.1 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	20

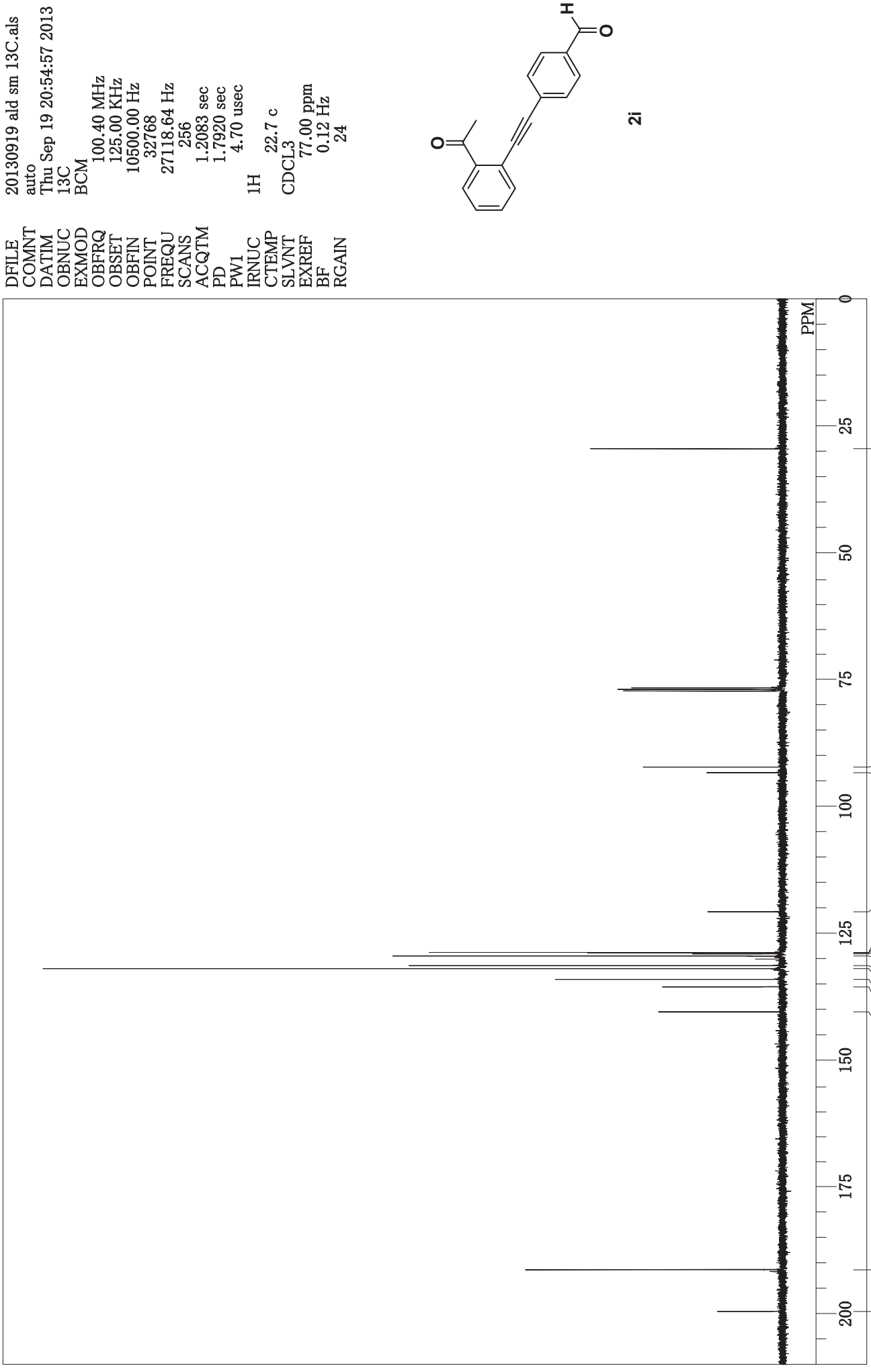
1H-NMR (CDCl3) δ :
7.51 (1H, td, J = 7.6, 1.6 Hz),
7.45 (1H, td, J = 7.6, 1.5 Hz).

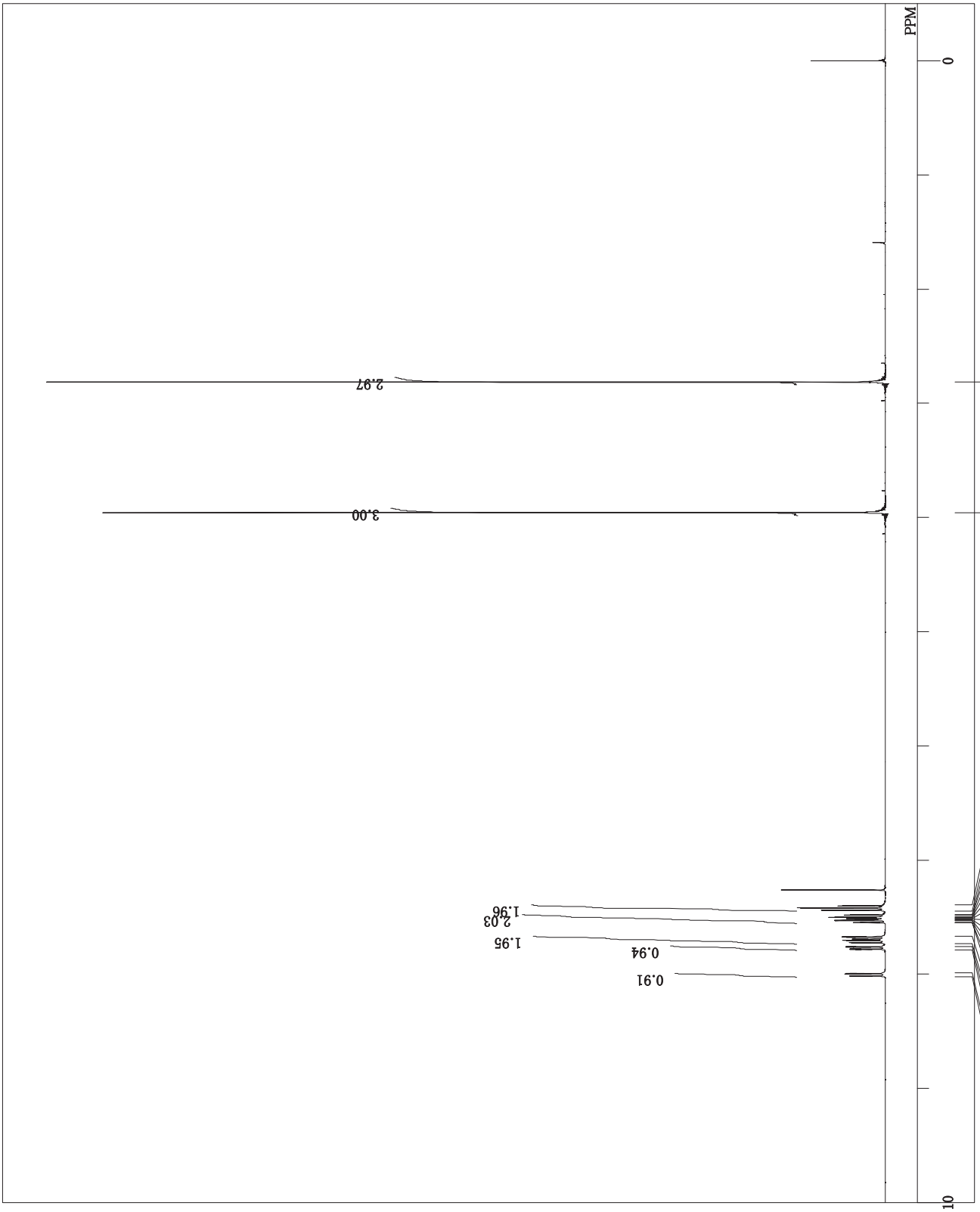


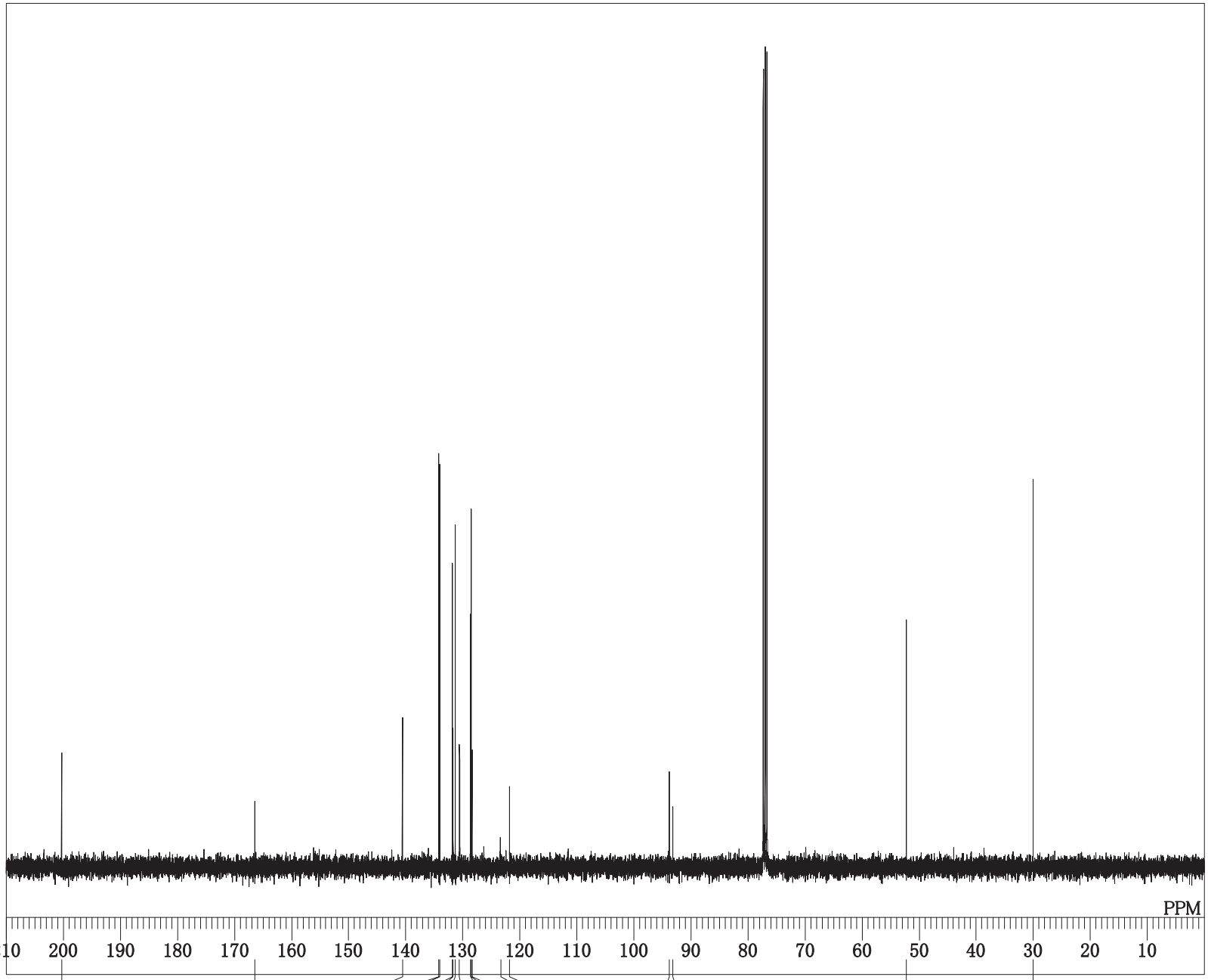
2h


20130914 ketone SM 13C.als
Sat Sep 14 12:41:05 2013
13C
SINGL
100.50 MHz
0.00 KHz
135159.00 Hz
32768
27100.27 Hz
1024
1.2091 sec
1.0000 sec
4.80 usec
1H
23.3 c
CDCL3
77.00 ppm
1.20 Hz
32
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
O
O
2h
S034
PPM
0
25
50
75
100
125
150
175
200
26.626
29.700
91.605
93.685
121.021
127.744
128.303
128.763
128.854
131.401
131.656
134.097
136.514
140.664
197.244
199.783

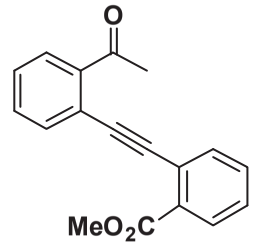

DFILE 20130919 ald sm 1H.als
COMNT auto
DATIM Thu Sep 19 20:40:24 2013
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 8
ACQTM 2.0500 sec
PD 2.0000 sec
PW1 6.60 usec
IRNUC 1H
CTEMP 23.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 13
1H-NMR (CDCl3) δ :
7.79 (1H, dd, J = 7.8, 1.0 Hz),
7.51 (1H, td, J = 7.6, 1.5 Hz),
7.45 (1H, td, J = 7.7, 1.3 Hz).
O
H
O
2i
S035
PPM
0.0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
10.0
11.0
3.00
2.03
1.92
1.05
1.02
1.06
1.02
0.91
2.764
10.025
7.905
7.848
7.802
7.799
7.782
7.780
7.714
7.693
7.677
7.644
7.534
7.531
7.516
7.512
7.497
7.493
7.475
7.471
7.455
7.451
7.436
7.433


DFILE 20130919 ald sm 13C.als
COMNT auto
DATIM Thu Sep 19 20:54:57 2013
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 256
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 4.70 usec
IRNUC 1H
CTEMP 22.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 24
O
H
O
2i
S036
PPM
0
25
50
75
100
125
150
175
200
29.571
92.288
93.383
120.790
128.850
128.882
129.088
129.549
131.410
132.019
134.118
135.592
140.532
191.336
199.552

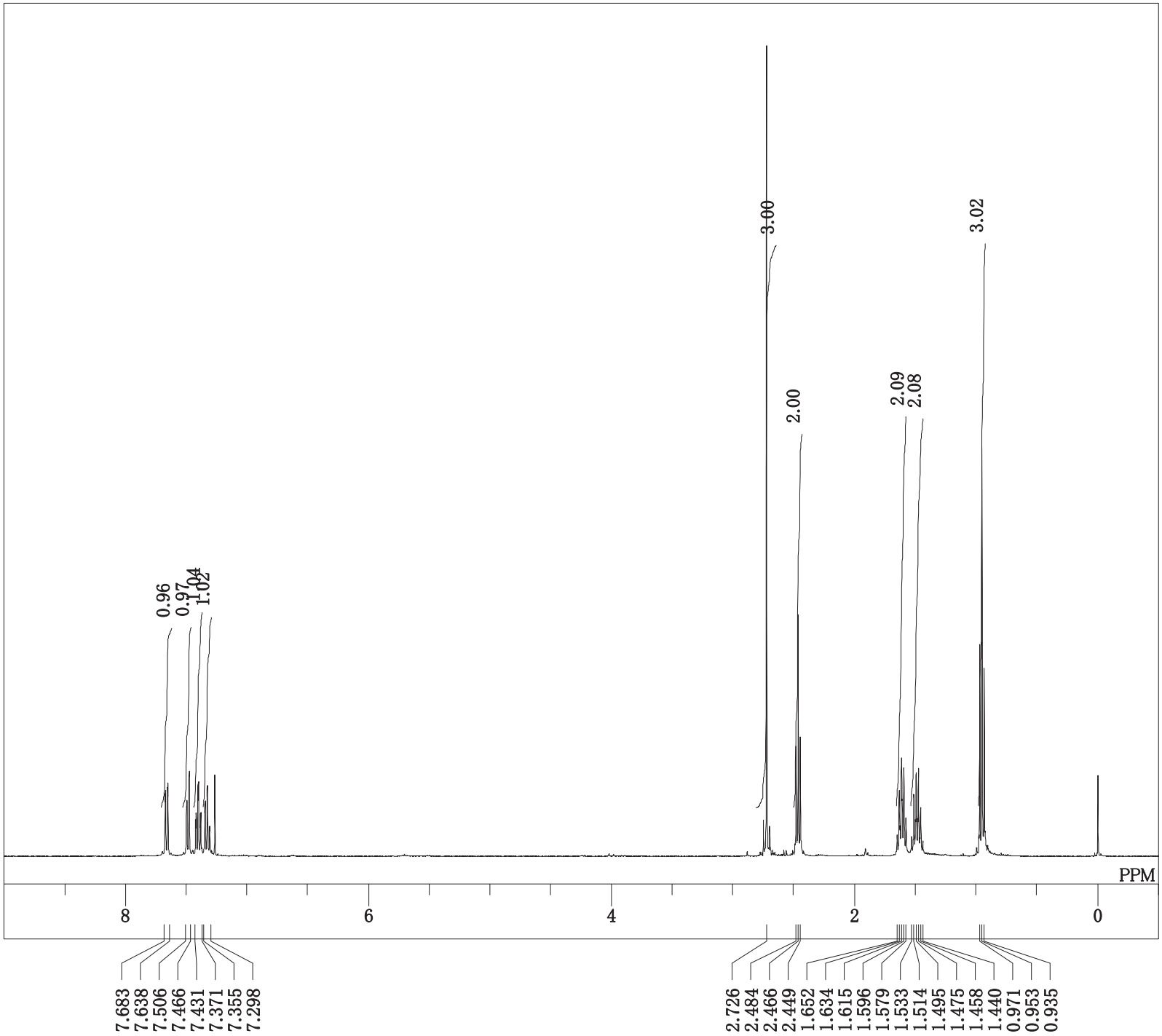

DFILE Me Ph oCO2Me sm 1H-1.als
COMNT single_pulse
DATIM 2013-04-24 09:19:21
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 391.78 MHz
OBSET 8.51 KHz
OBFIN 3.34 Hz
POINT 26214
FREQU 5882.26 Hz
SCANS 8
ACQTM 4.4564 sec
PD 3.0000 sec
PW1 5.05 usec
IRNUC 1H
CTEMP 20.3 c
SLVNT CDCl3
EXREF 0.00 ppm
BF 0.01 Hz
RGAIN 46
1H-NMR (CDCl3) δ :
7.53 (2H, td, J = 7.6, 1.5 Hz),
7.50 (2H, td, J = 7.6, 1.4 Hz).
O
MeO2C
2j
S037
PPM
0
10
2.97
3.00
2.814
3.957
0.91
0.94
1.95
2.03
1.96
7.391
7.449
7.479
7.483
7.499
7.502
7.507
7.511
7.518
7.522
7.526
7.530
7.546
7.549
7.667
7.731
7.756
7.786
7.990
8.021



DFILE	Me Ph oCO2Me sm 13C-1.als
COMNT	single pulse decoupled gated NOE
DATIM	2013-04-24 09:47:39
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.17 Hz
SCANS	512
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	2.87 usec
IRNUC	1H
CTEMP	20.7 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.01 Hz
RGAIN	50

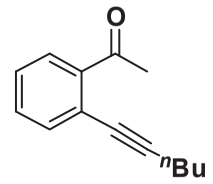


2j

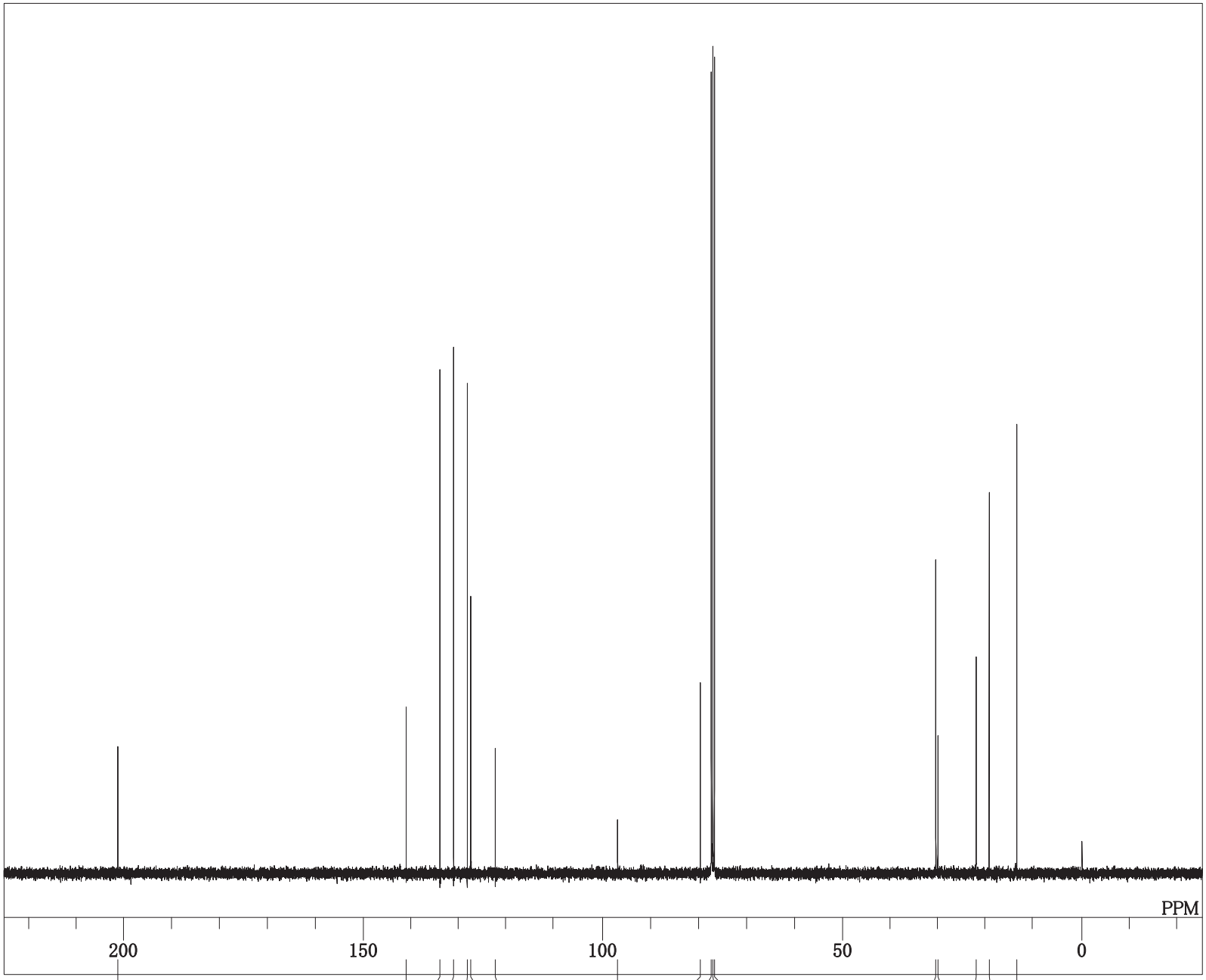


DFILE 20130123 mattan nBu NON.als
COMNT
DATIM Wed Jan 23 17:40:16 2013
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 16
ACQTM 2.0500 sec
PD 2.0000 sec
PW1 6.60 usec
IRNUC 1H
CTEMP 22.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 16

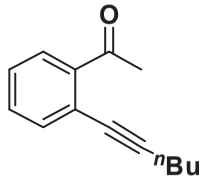
1H-NMR (CDCl3) δ :
7.66 (1H, dd, J = 7.8, 1.5 Hz),
7.49 (1H, dd, J = 7.8, 1.0 Hz),
7.40 (1H, td, J = 7.6, 1.1 Hz),
7.33 (1H, td, J = 7.6, 1.3 Hz),
2.47 (2H, t, J = 7.1 Hz),
1.65–1.58 (2H, m),
1.53–1.44 (2H, m),
0.95 (3H, t, J = 7.3 Hz).



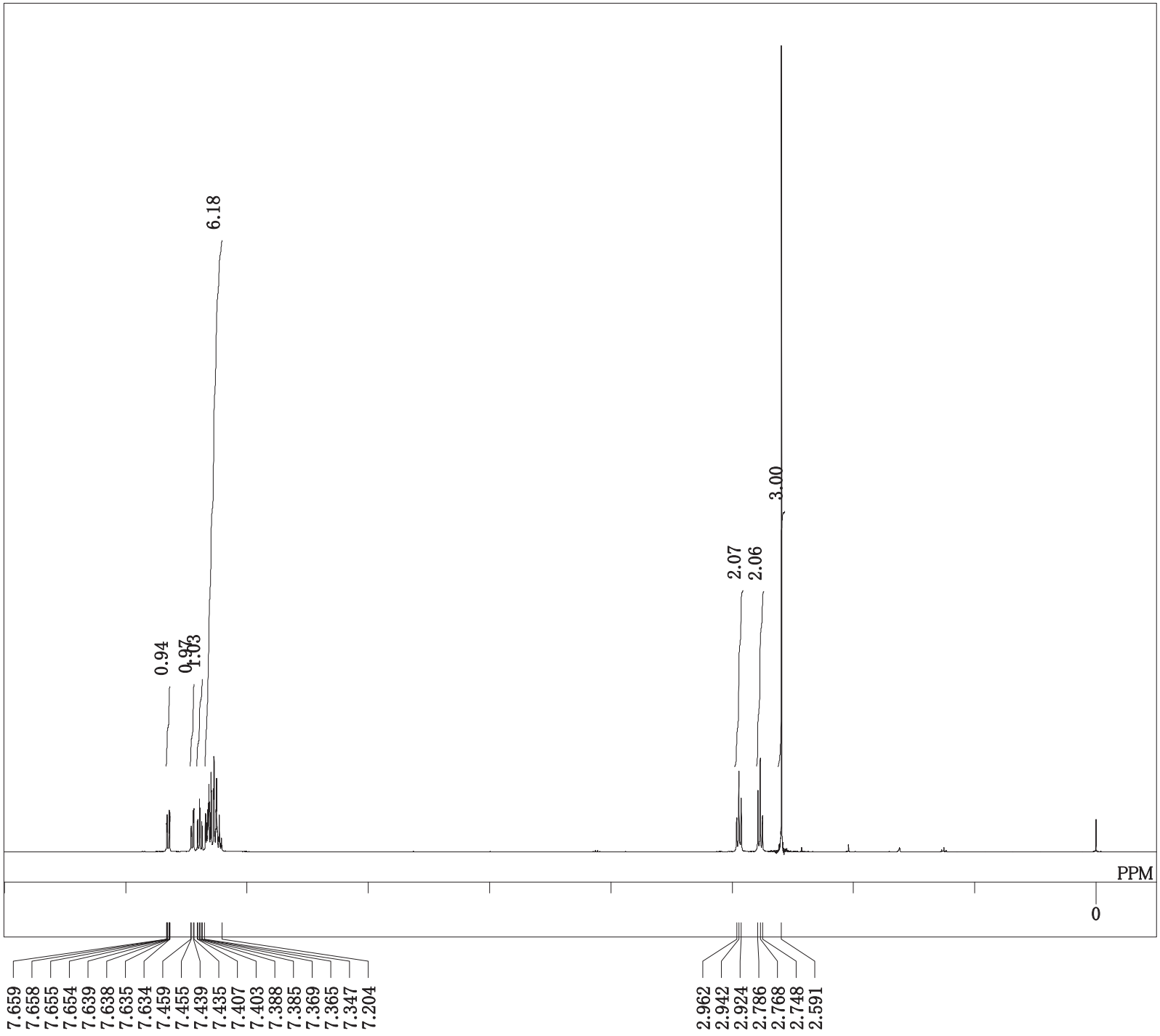
2k



DFILE	20130123 mattan nBu BCM-1.als
COMNT	single pulse decoupled gated NOE
DATIM	2013-01-23 18:12:36
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.17 Hz
SCANS	1024
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	2.87 usec
IRNUC	1H
CTEMP	19.5 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	36

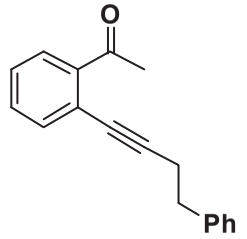


2k

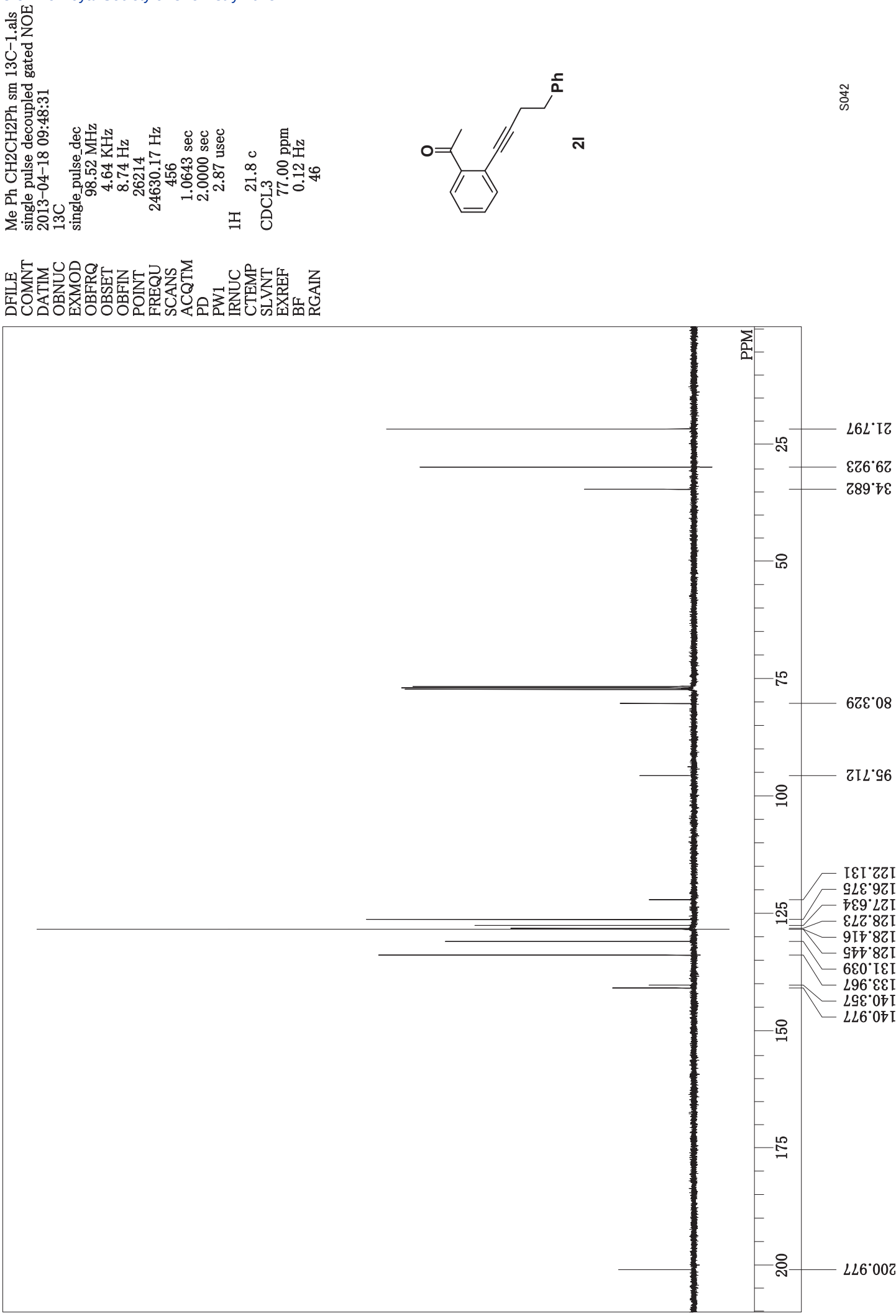


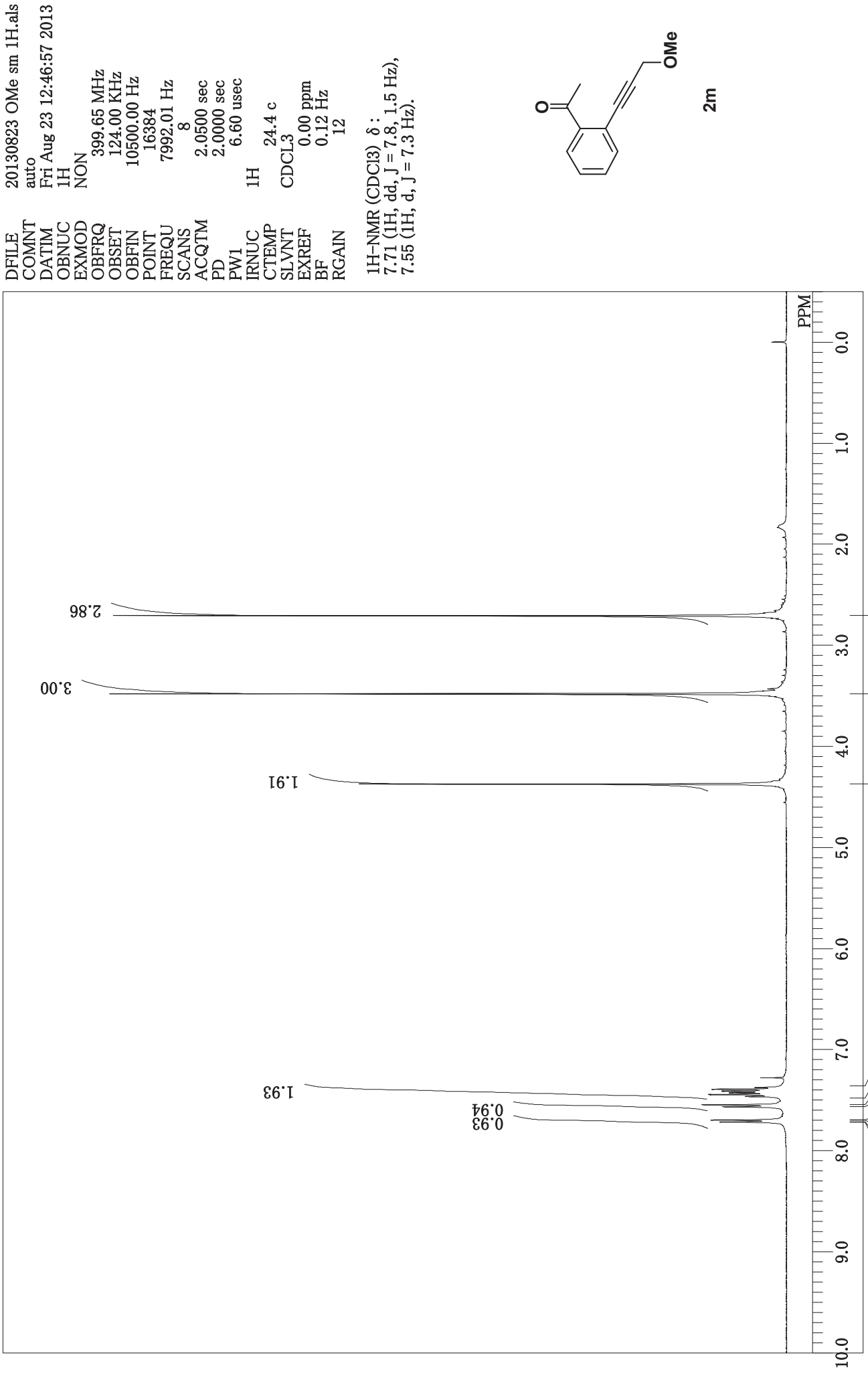
DFILE	Me Ph CH2CH2Ph sm 1H-1.als
COMNT	single_pulse
DATIM	2013-04-18 09:22:28
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	26214
FREQU	5882.26 Hz
SCANS	8
ACQTM	4.4564 sec
PD	3.0000 sec
PW1	5.05 usec
IRNUC	1H
CTEMP	21.3 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.01 Hz
RGAIN	36

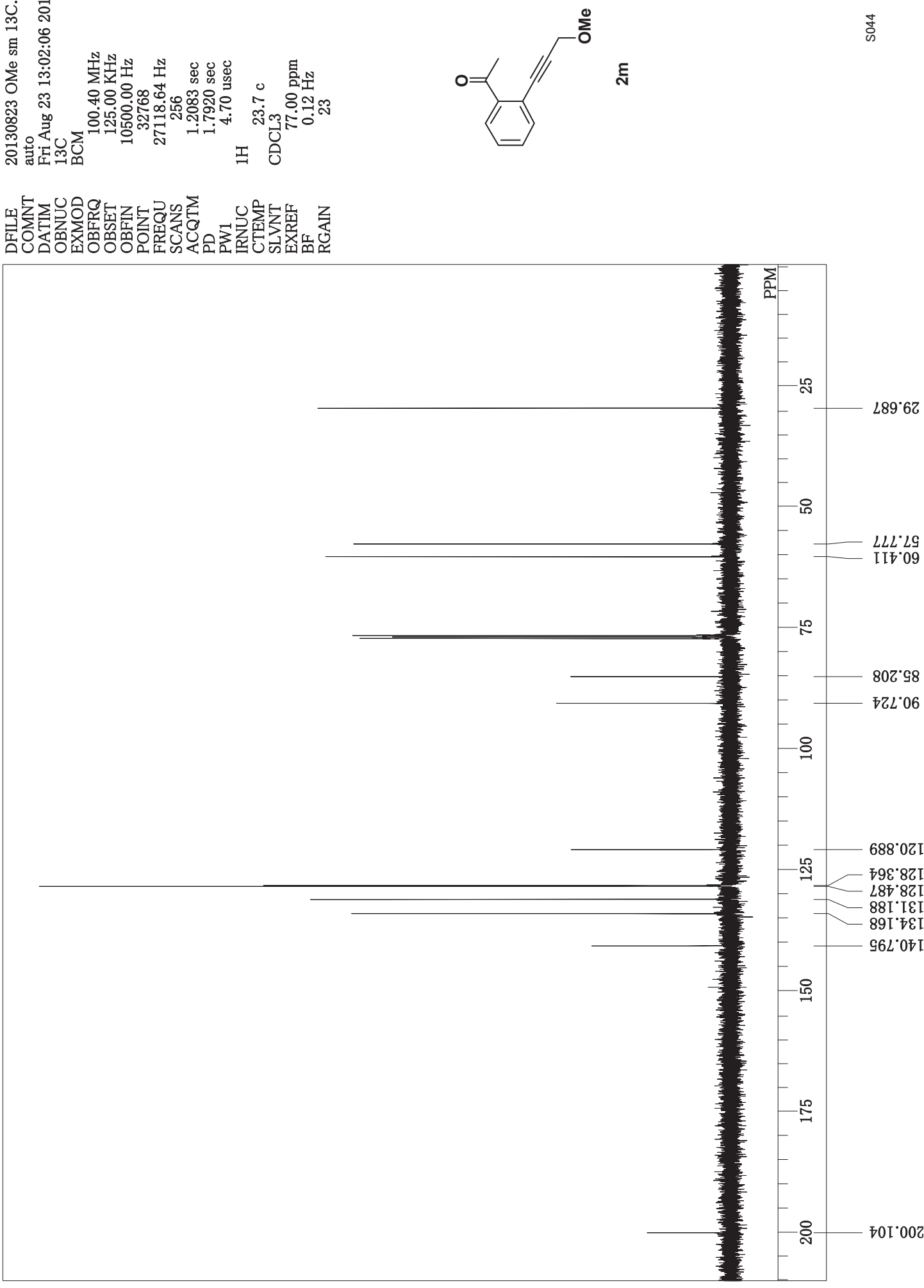
1H-NMR (CDCl3) δ :
7.65 (1H, dq, J = 7.7, 0.7 Hz),
7.45 (1H, dd, J = 7.7, 1.5 Hz),
7.39 (1H, td, J = 7.5, 1.5 Hz),
2.94 (2H, t, J = 7.4 Hz),
2.77 (2H, t, J = 7.4 Hz).

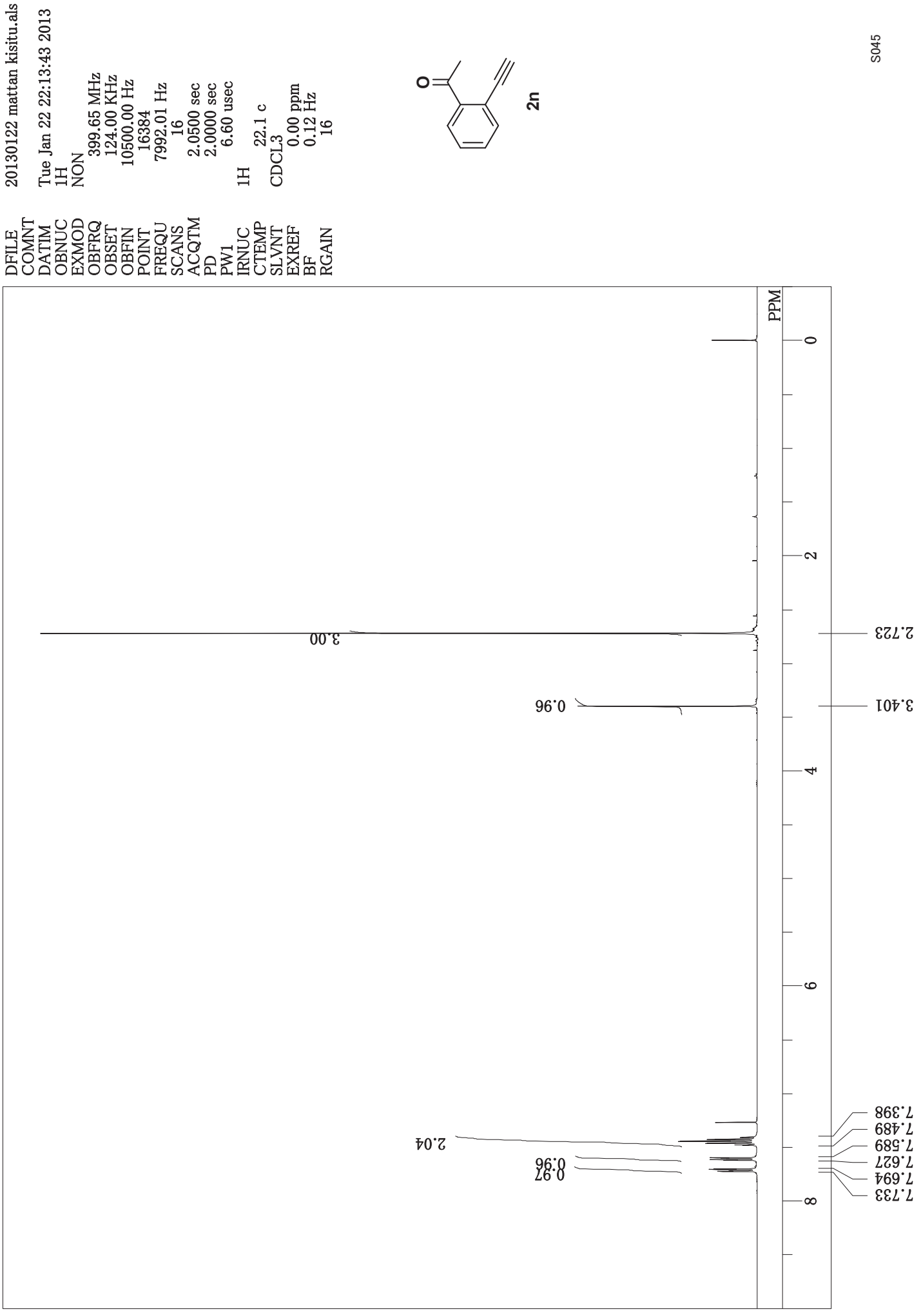


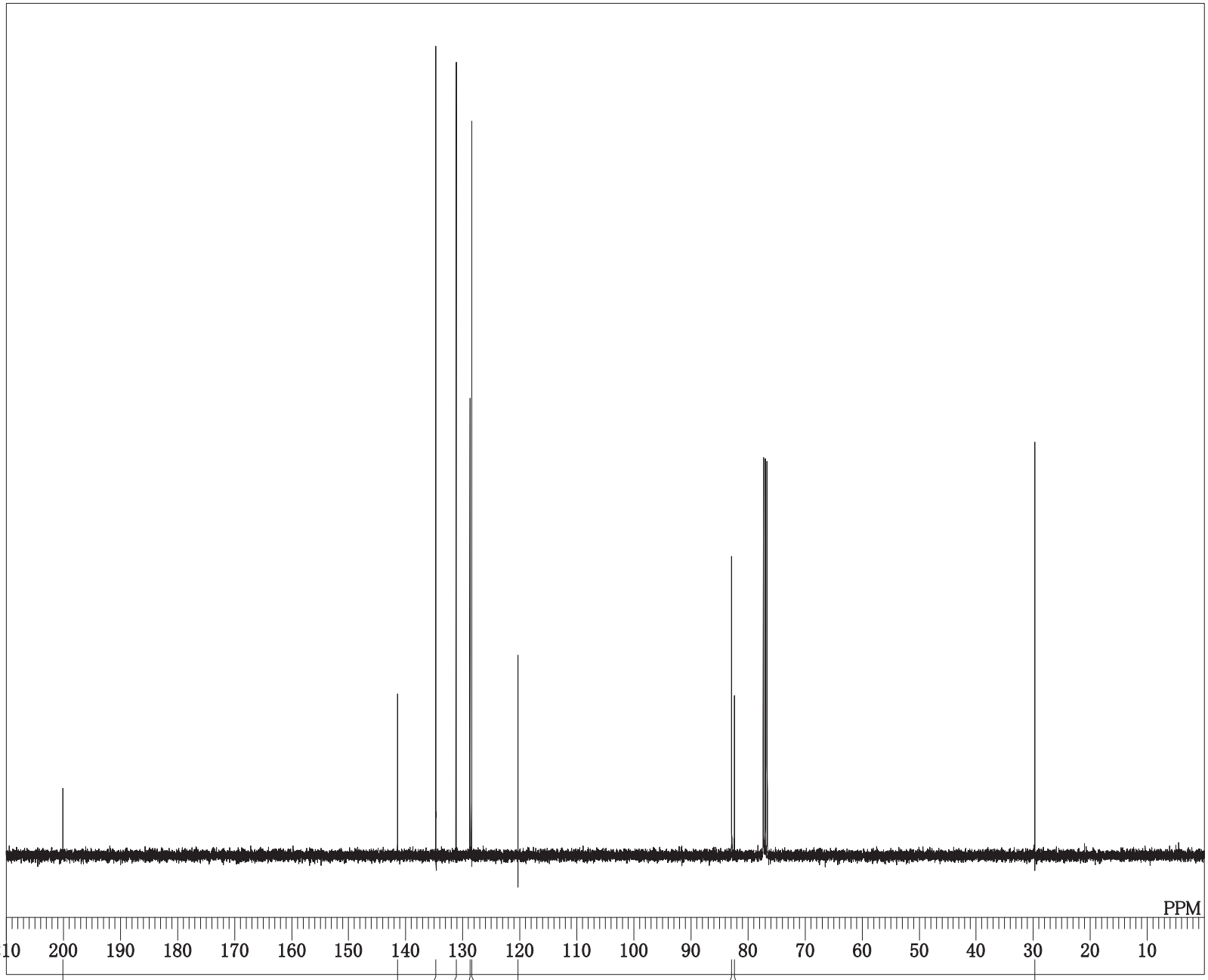
2l



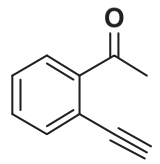

DFILE 20130823 OMe sm 1H.als
COMNT auto
DATIM Fri Aug 23 12:46:57 2013
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 8
ACQTM 2.0500 sec
PD 2.0000 sec
PW1 6.60 usec
IRNUC 1H
CTEMP 24.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 12
1H-NMR (CDCl3) δ :
7.71 (1H, dd, J = 7.8, 1.5 Hz),
7.55 (1H, d, J = 7.3 Hz).
O
OMe
2m
S043
PPM
0.0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
10.0
2.86
3.00
1.91
1.93
0.93
0.94
2.706
3.479
4.372
7.359
7.476
7.545
7.564
7.694
7.698
7.714
7.717



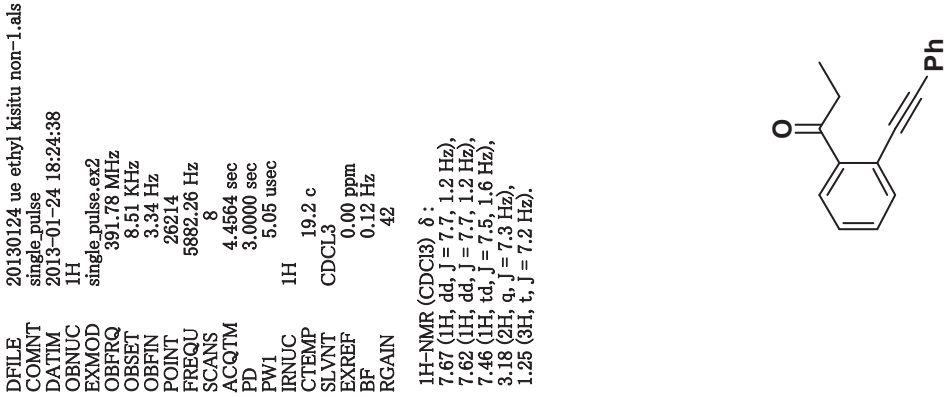

20130122 mattan kisitu.als
Tue Jan 22 22:13:43 2013
1H
NON
399.65 MHz
124.00 KHz
10500.00 Hz
16384
7992.01 Hz
16
2.0500 sec
2.0000 sec
6.60 usec
1H
22.1 c
CDCL3
0.00 ppm
0.12 Hz
16
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
O
2n
S045
PPM
0
2
4
6
8
3.00
0.96
2.04
0.97
0.96
2.723
3.401
7.398
7.489
7.589
7.627
7.694
7.733



DFILE	20130201 terminal sm 13C-1.als
COMNT	single pulse decoupled gated NOE
DATIM	2013-02-01 20:01:29
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.17 Hz
SCANS	512
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	2.87 usec
IRNUC	1H
CTEMP	21.8 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.01 Hz
RGAIN	46



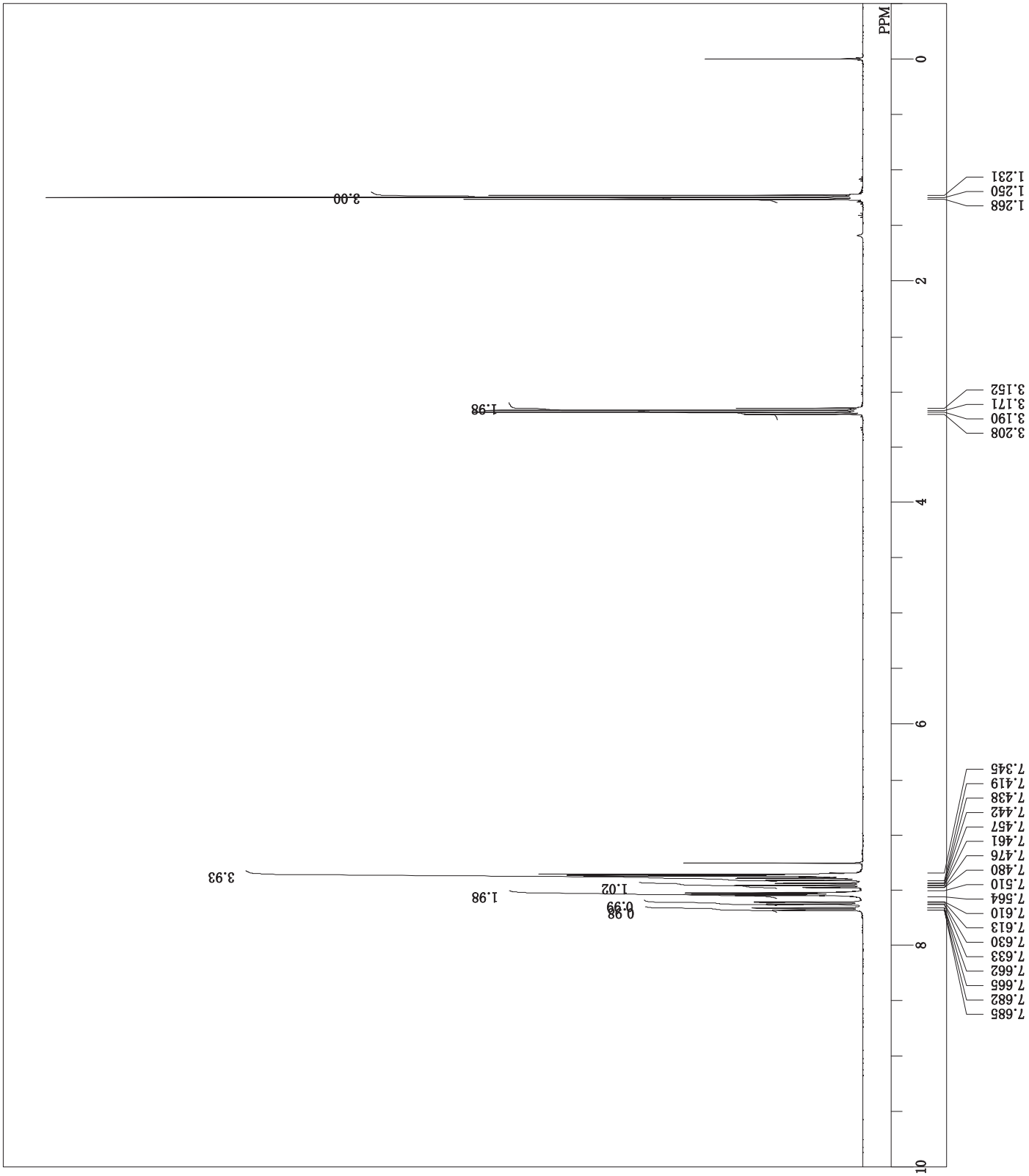
2n

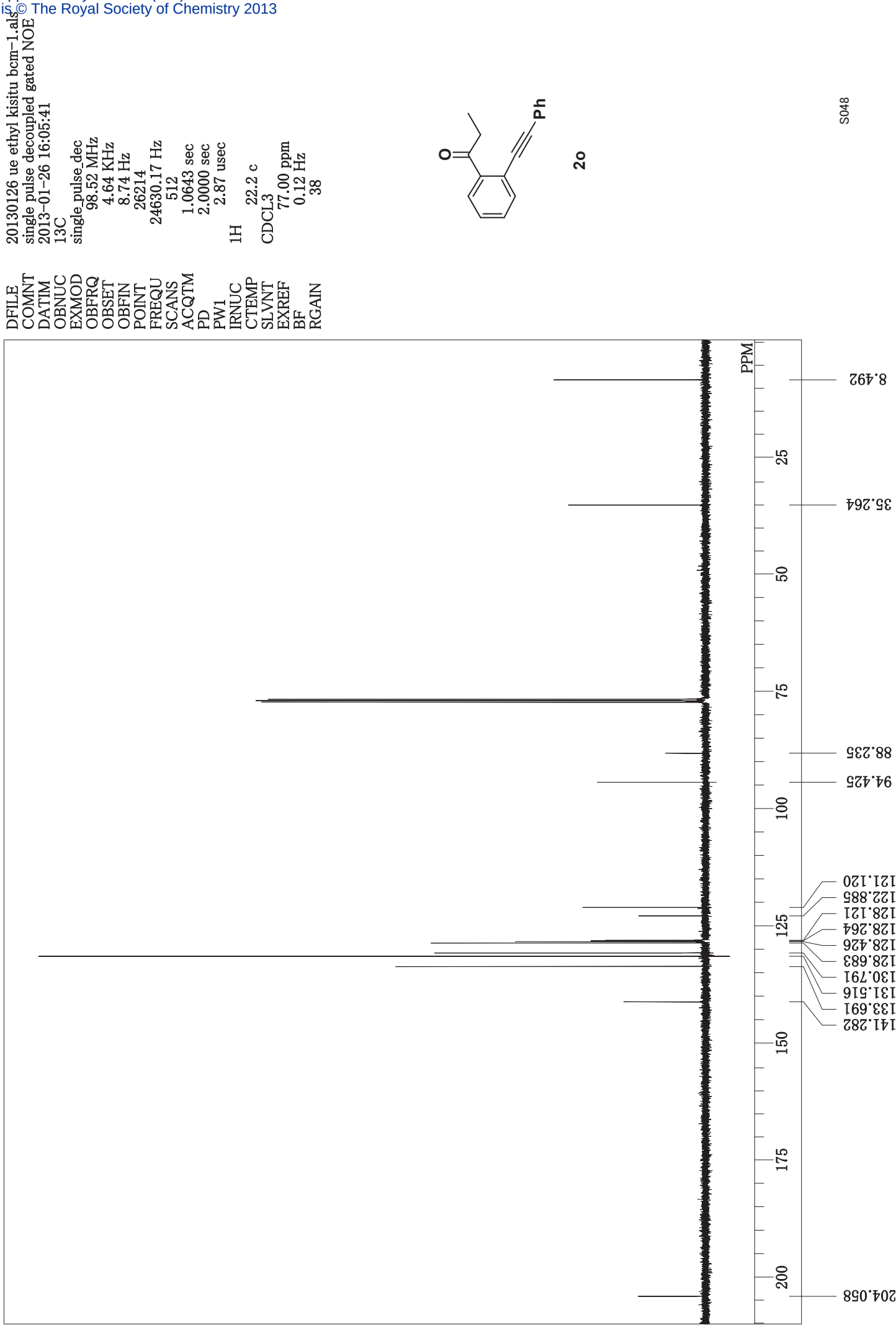


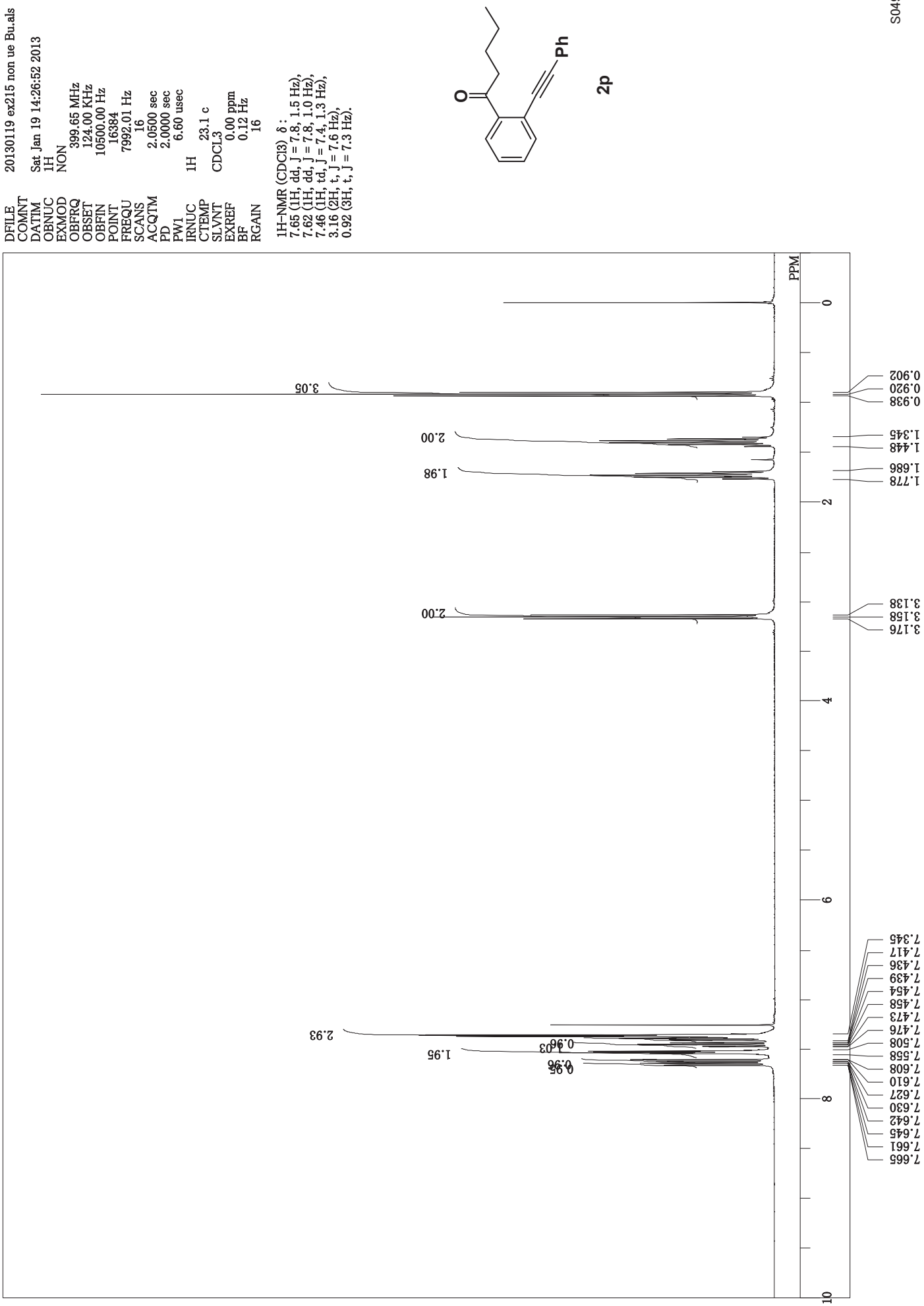
2o

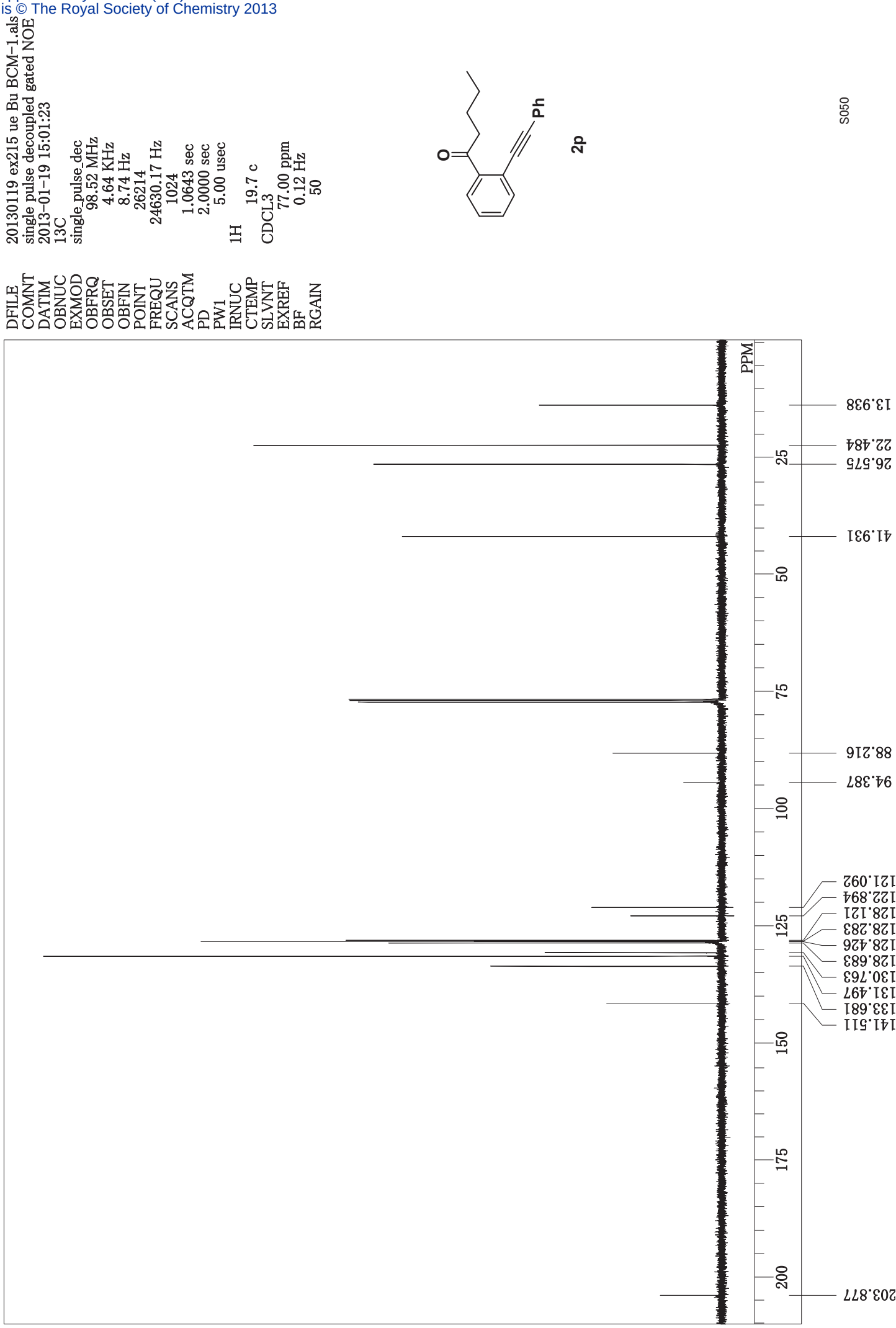
DFILE 20130124 ue ethyl kisitu non-1.als
COMNT single_pulse
DATIM 2013-01-24 18:24:38
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 391.78 MHz
OBSET 8.51 KHz
OBFIN 3.34 Hz
POINT 26214
FREQU 5882.26 Hz
SCANS 8
ACQTM 4.4564 sec
PD 3.0000 sec
PW1 5.05 usec
IRNUC 1H
CTEMP 19.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 42

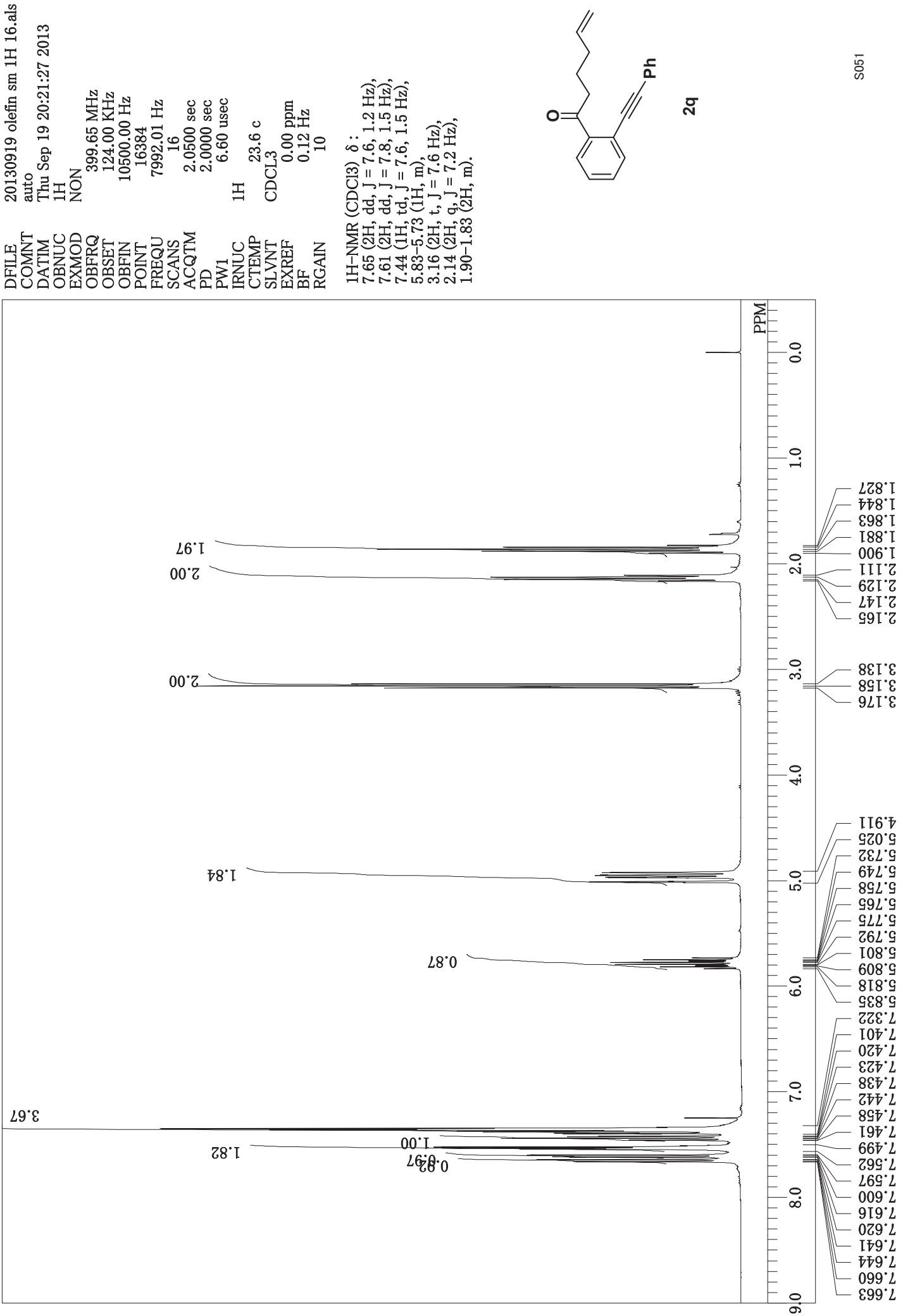
1H-NMR (CDCl3) δ :
7.67 (1H, dd, J = 7.7, 1.2 Hz),
7.62 (1H, dd, J = 7.7, 1.2 Hz),
7.46 (1H, td, J = 7.5, 1.6 Hz),
3.18 (2H, q, J = 7.3 Hz),
1.25 (3H, t, J = 7.2 Hz).

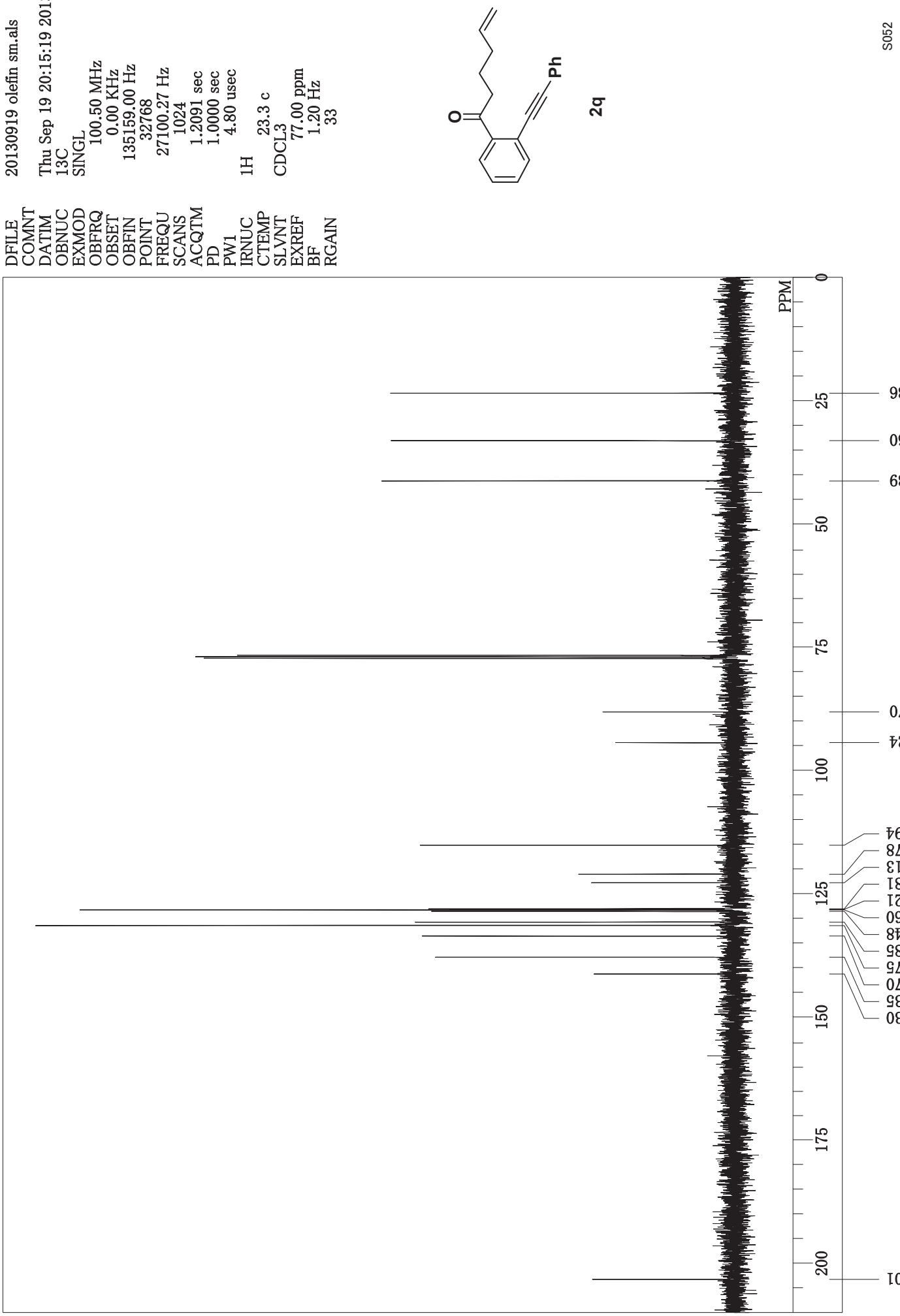


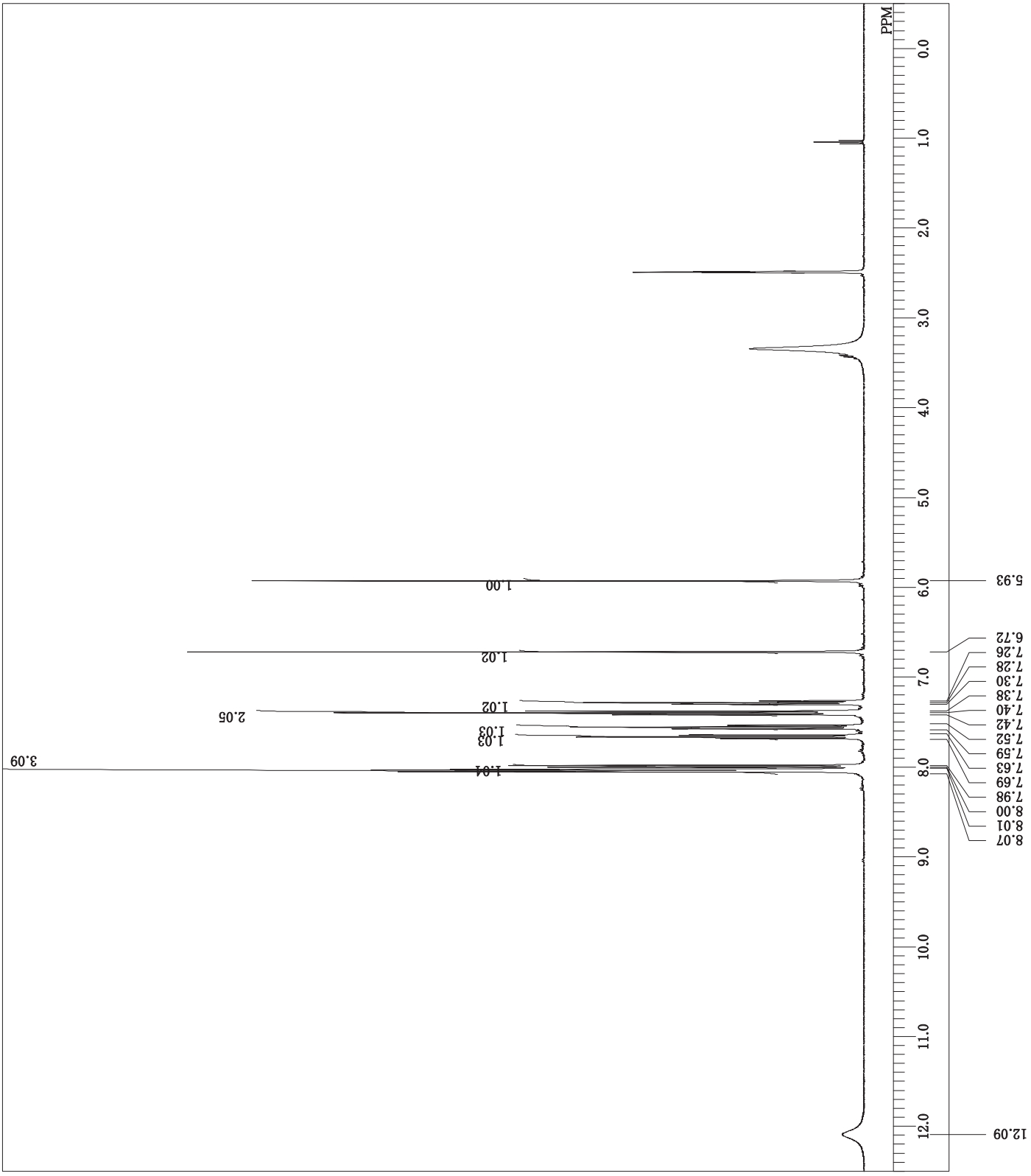

20130126 ue ethyl kisitu bcm-1.als
single pulse decoupled gated NOE
2013-01-26 16:05:41
13C
single_pulse_dec
98.52 MHz
4.64 KHz
8.74 Hz
26214
24630.17 Hz
512
1.0643 sec
2.0000 sec
2.87 usec
1H
22.2 c
CDCL3
77.00 ppm
0.12 Hz
38
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
O
Ph
2o
S048
PPM
8.492
35.264
88.235
94.425
121.120
122.885
128.121
128.264
128.426
128.683
130.791
131.516
133.691
141.282
204.058

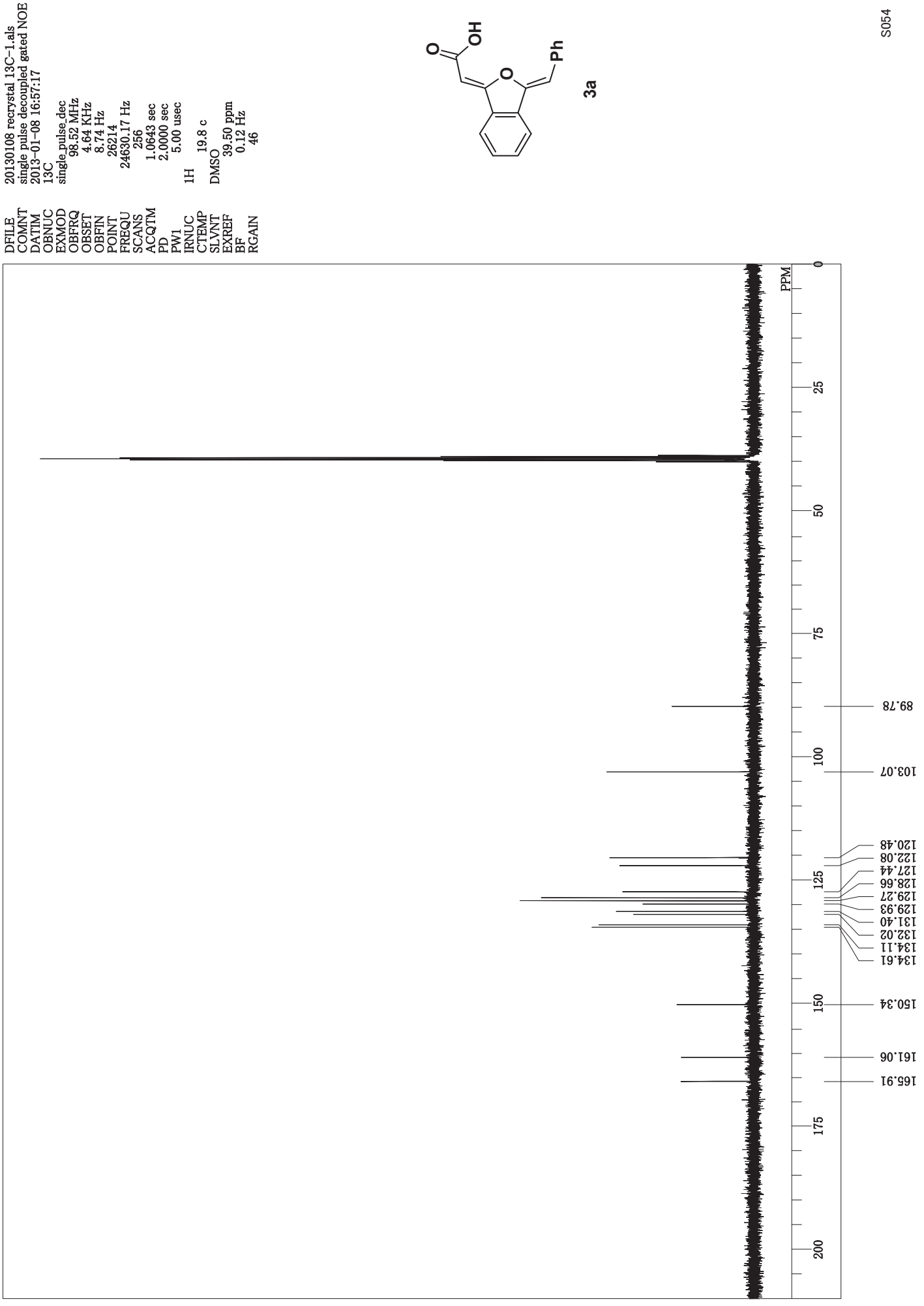

DFILE 20130119 ex215 non ue Bu.als
COMNT
DATIM Sat Jan 19 14:26:52 2013
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 16
ACQTM 2.0500 sec
PD 2.0000 sec
PW1 6.60 usec
IRNUC 1H
CTEMP 23.1 c
SLVNT CDCl3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 16
1H-NMR (CDCl3) δ :
7.65 (1H, dd, J = 7.8, 1.5 Hz),
7.62 (1H, dd, J = 7.8, 1.0 Hz),
7.46 (1H, td, J = 7.4, 1.3 Hz),
3.16 (2H, t, J = 7.6 Hz),
0.92 (3H, t, J = 7.3 Hz).
O
Ph
2p
S049
PPM
0
2
4
6
8
10
3.05
2.00
1.98
2.00
2.93
1.95
0.95
0.96
1.03
0.902
0.920
0.938
1.345
1.448
1.686
1.778
3.138
3.158
3.176
7.345
7.417
7.436
7.439
7.454
7.458
7.473
7.476
7.508
7.558
7.608
7.610
7.627
7.630
7.642
7.645
7.661
7.665

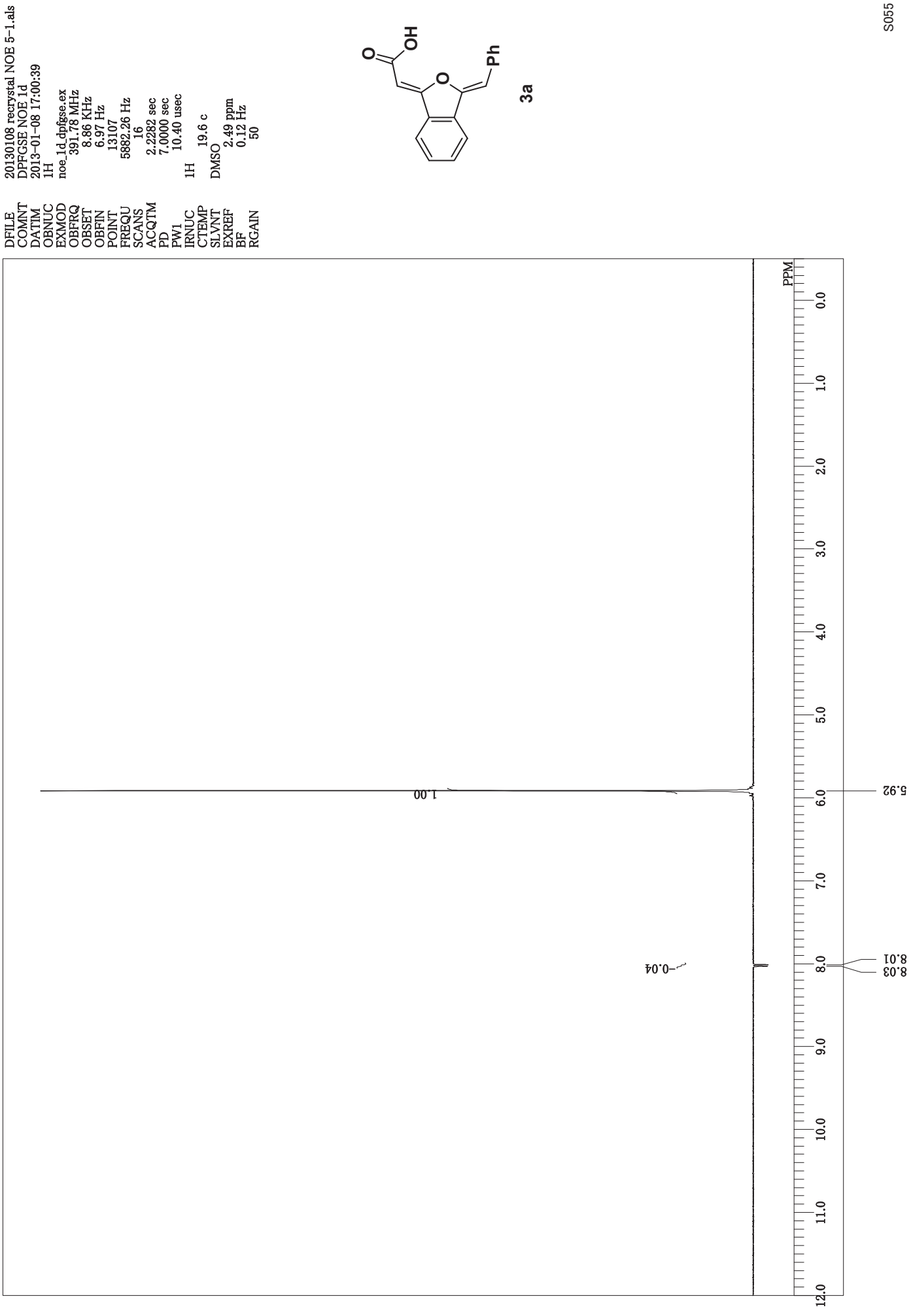

20130119 ex215 ue Bu BCM-1.als
single pulse decoupled gated NOE
2013-01-19 15:01:23
13C
single_pulse_dec
98.52 MHz
4.64 KHz
8.74 Hz
26214
24630.17 Hz
1024
1.0643 sec
2.0000 sec
5.00 usec
1H
19.7 c
CDCL3
77.00 ppm
0.12 Hz
50
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
O
Ph
2p
S050
PPM
13.938
22.484
26.575
41.931
88.216
94.387
121.092
122.894
128.121
128.283
128.426
128.683
130.763
131.497
133.681
141.511
203.877

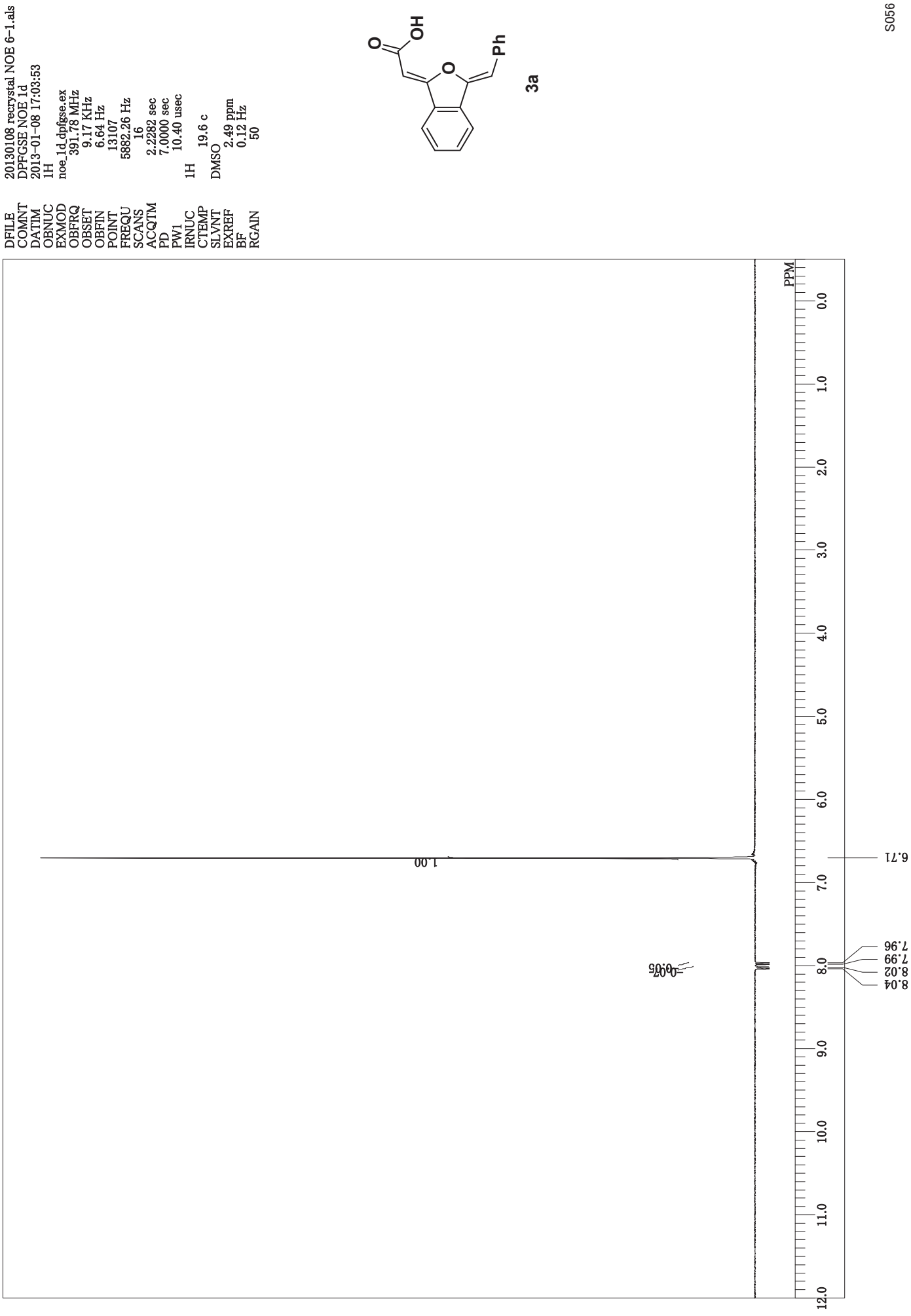

DFILE 20130919 olefin sm 1H 16.als
COMNT auto
DATIM Thu Sep 19 20:21:27 2013
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 16
ACQTM 2.0500 sec
PD 2.0000 sec
PW1 6.60 usec
IRNUC 1H
CTEMP 23.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 10
1H-NMR (CDCl3) δ :
7.65 (2H, dd, J = 7.6, 1.2 Hz),
7.61 (2H, dd, J = 7.8, 1.5 Hz),
7.44 (1H, td, J = 7.6, 1.5 Hz),
5.83–5.73 (1H, m),
3.16 (2H, t, J = 7.6 Hz),
2.14 (2H, q, J = 7.2 Hz),
1.90–1.83 (2H, m).
O
Ph
2q
S051
PPM
0.0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
3.67
1.82
0.97
1.00
0.82
1.84
0.87
2.00
2.00
1.97
7.663
7.660
7.644
7.641
7.620
7.616
7.600
7.597
7.562
7.499
7.461
7.458
7.442
7.438
7.423
7.420
7.401
7.322
5.835
5.818
5.809
5.801
5.792
5.775
5.765
5.758
5.749
5.732
5.025
4.911
3.176
3.158
3.138
2.165
2.147
2.129
2.111
1.900
1.881
1.863
1.844
1.827

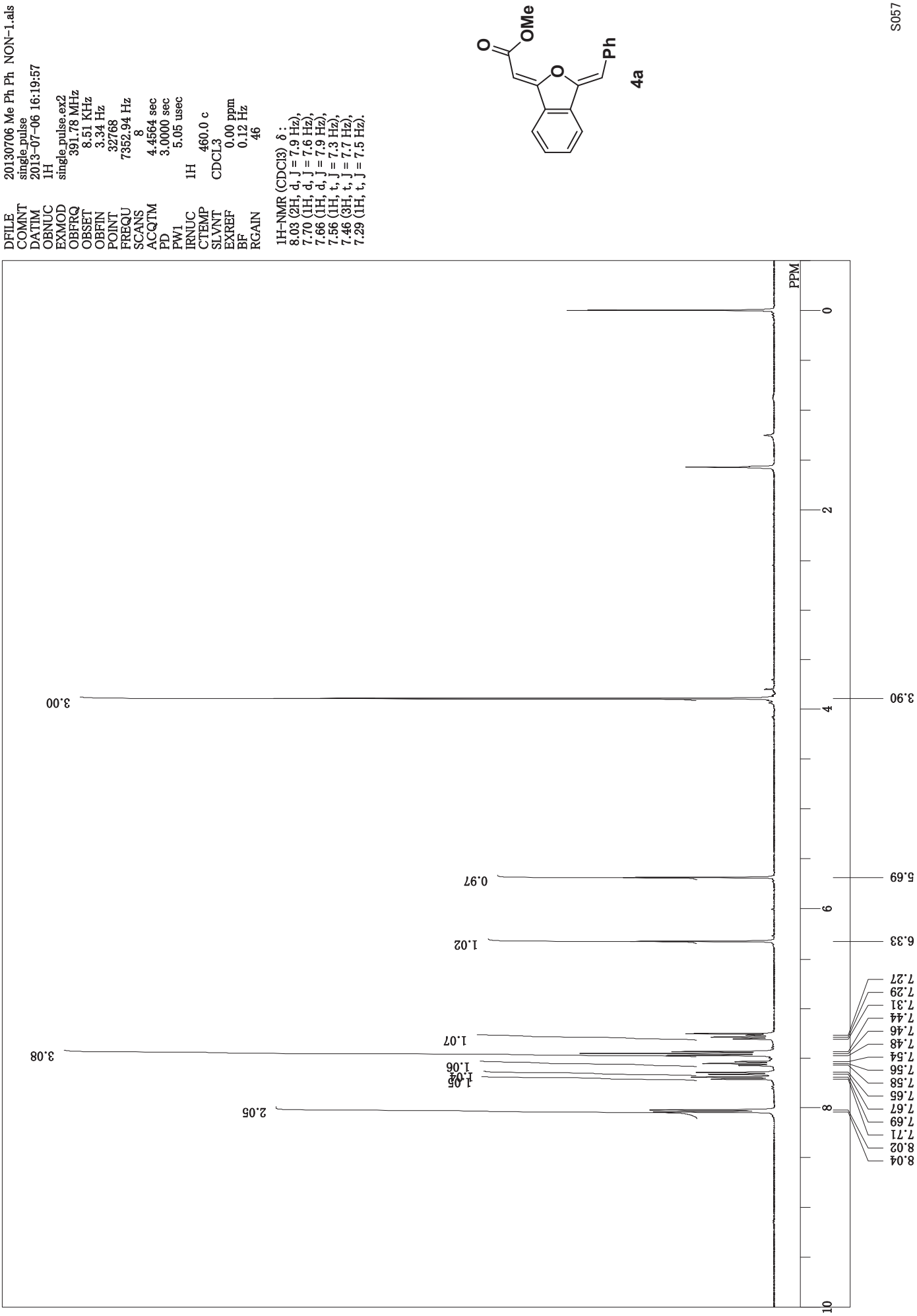


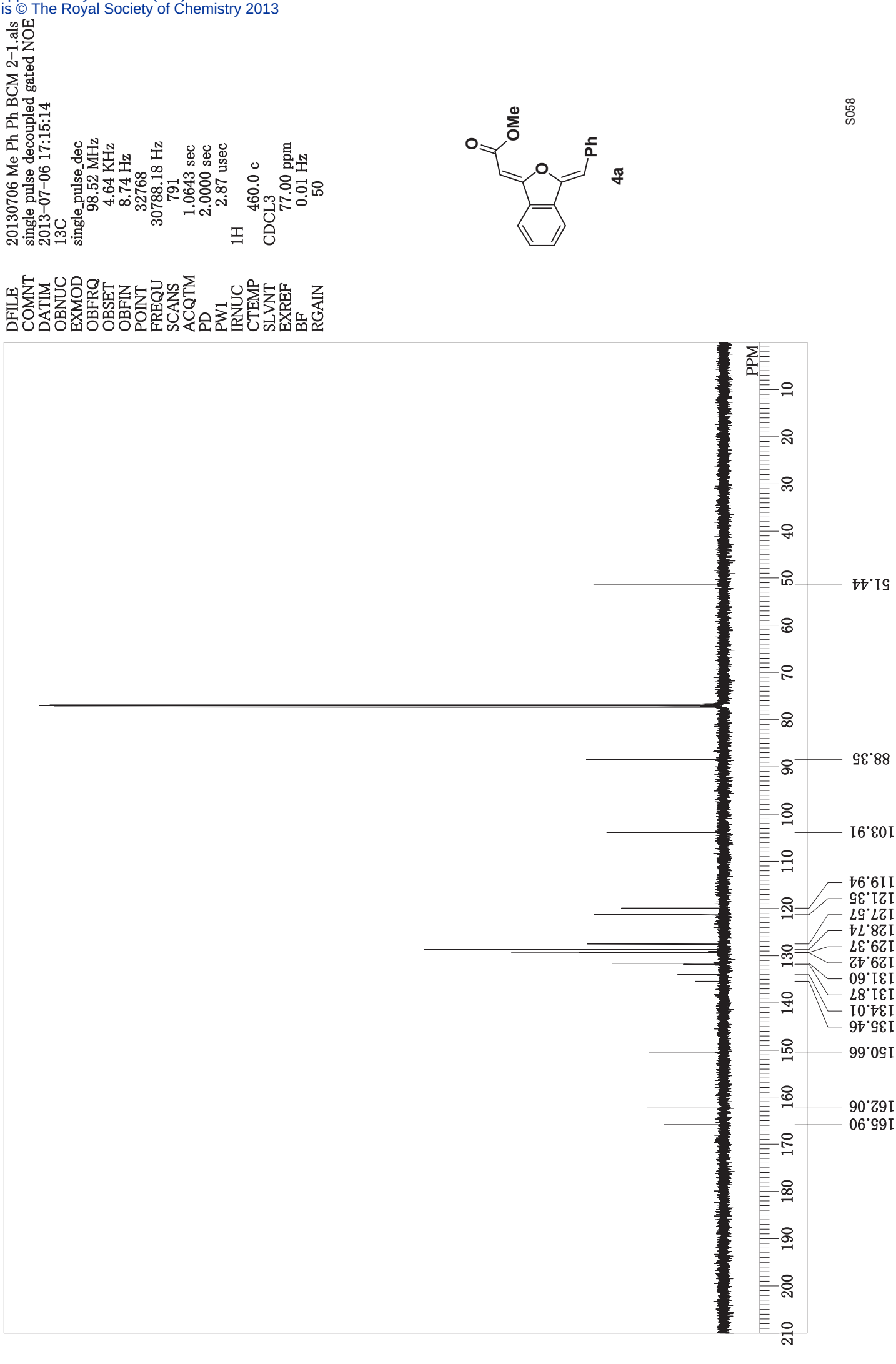


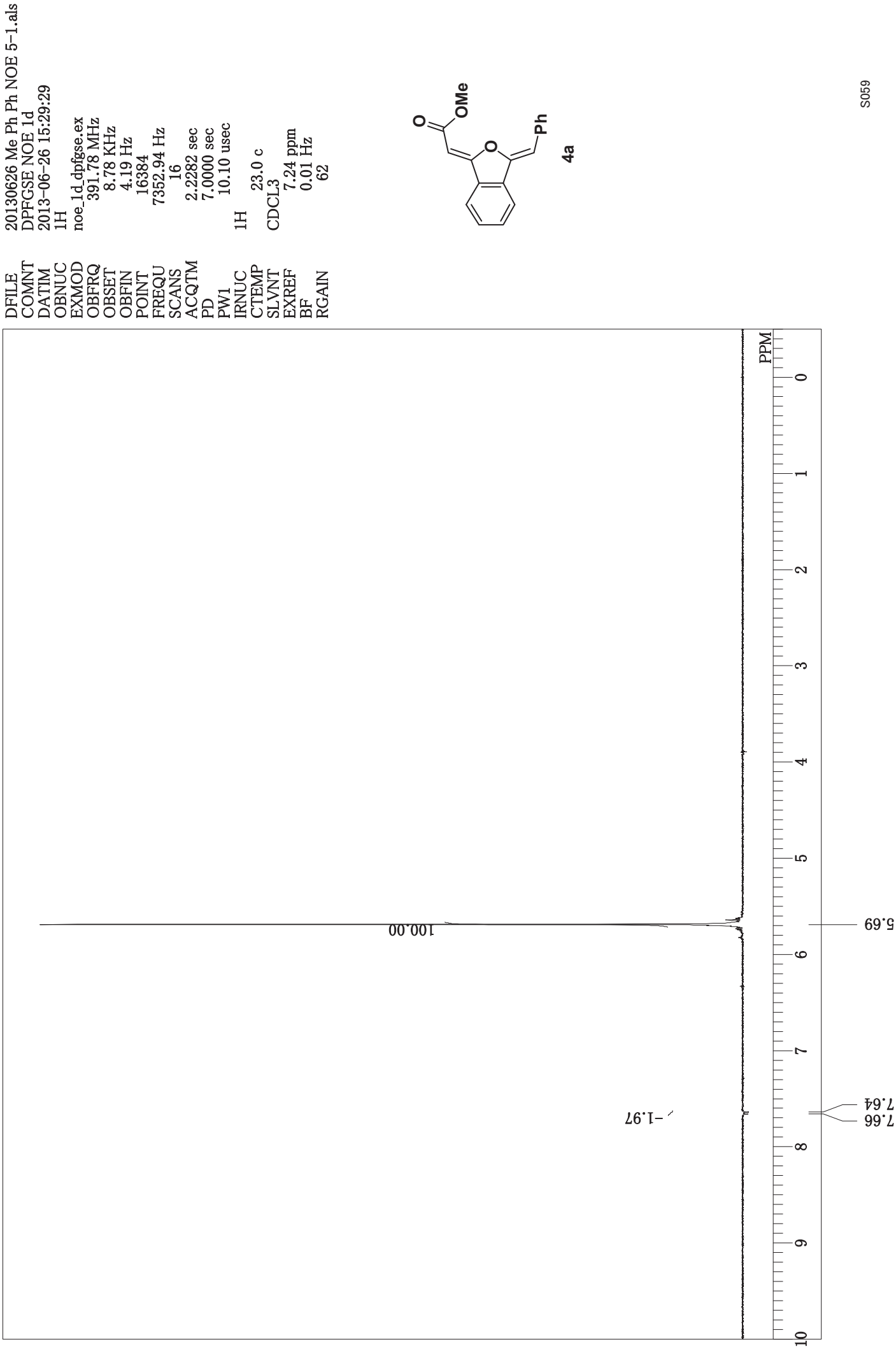

DFILE 20130108 recrystal 13C-1.als
COMNT single pulse decoupled gated NOE
DATIM 2013-01-08 16:57:17
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.17 Hz
SCANS 256
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 5.00 usec
IRNUC 1H
CTEMP 19.8 c
SLVNT DMSO
EXREF 39.50 ppm
BF 0.12 Hz
RGAIN 46
O
OH
O
Ph
3a
S054
PPM
0
25
50
75
100
125
150
175
200
89.78
103.07
120.48
122.08
127.44
128.66
129.27
129.93
131.40
132.02
134.11
134.61
150.34
161.06
165.91

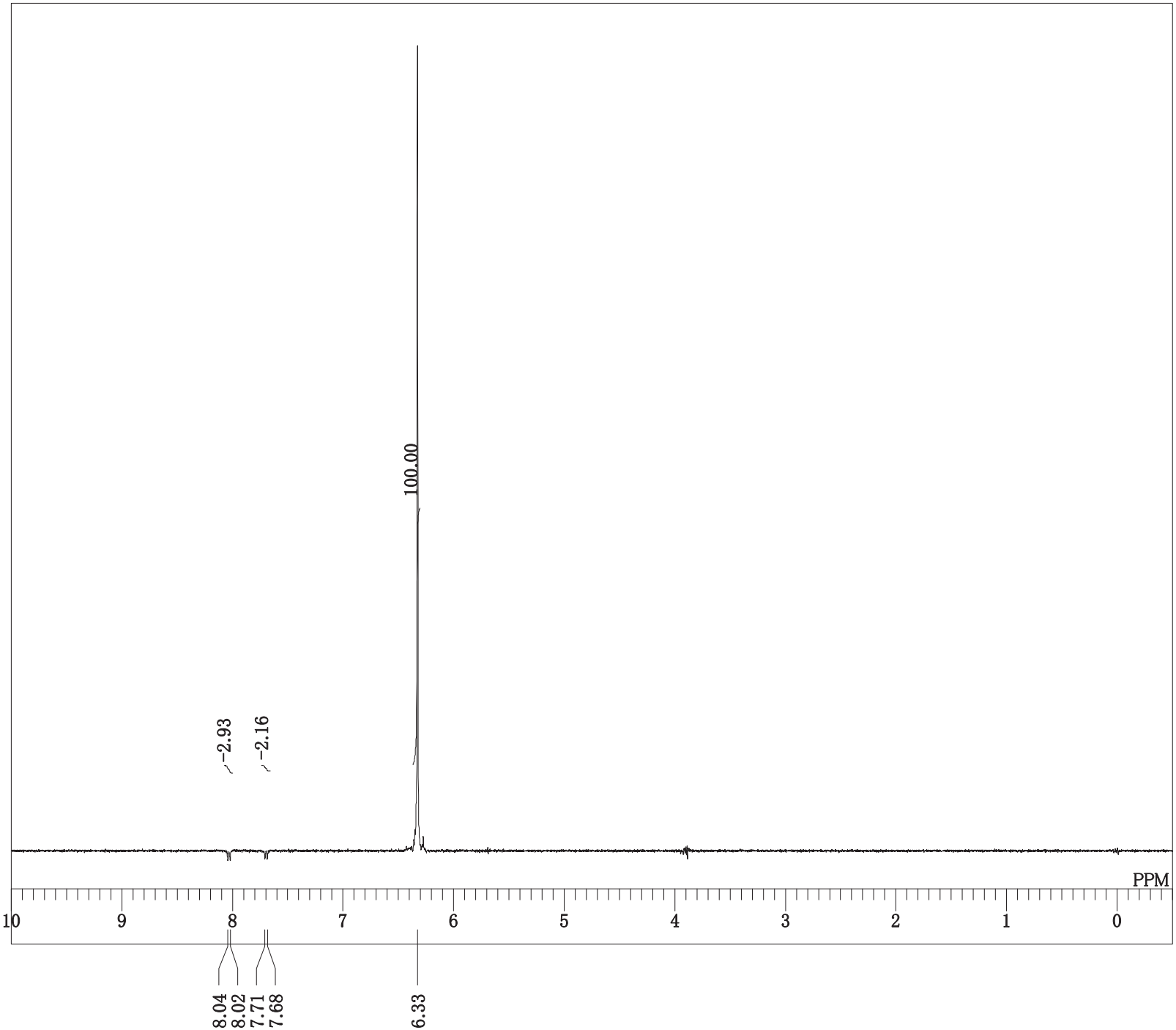




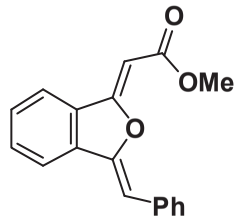

DFILE 20130706 Me Ph Ph NON-1.als
COMNT single_pulse
DATIM 2013-07-06 16:19:57
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 391.78 MHz
OBSET 8.51 KHz
OBFIN 3.34 Hz
POINT 32768
FREQU 7352.94 Hz
SCANS 8
ACQTM 4.4564 sec
PD 3.0000 sec
PW1 5.05 usec
IRNUC 1H
CTEMP 460.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 46
1H-NMR (CDCl3) δ :
8.03 (2H, d, J = 7.9 Hz),
7.70 (1H, d, J = 7.6 Hz),
7.66 (1H, d, J = 7.9 Hz),
7.56 (1H, t, J = 7.3 Hz),
7.46 (3H, t, J = 7.7 Hz),
7.29 (1H, t, J = 7.5 Hz).
O
OMe
O
Ph
4a
S057
PPM
0
2
4
6
8
10
3.00
0.97
1.02
1.07
3.08
1.05
1.04
1.06
2.05
3.90
5.69
6.33
7.27
7.29
7.31
7.44
7.46
7.48
7.54
7.56
7.58
7.65
7.67
7.69
7.71
8.02
8.04


20130706 Me Ph Ph BCM 2-1.als
single pulse decoupled gated NOE
2013-07-06 17:15:14
13C
single_pulse_dec
98.52 MHz
4.64 KHz
8.74 Hz
32768
30788.18 Hz
791
1.0643 sec
2.0000 sec
2.87 usec
1H
460.0 c
CDCL3
77.00 ppm
0.01 Hz
50
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
O
OMe
O
Ph
4a
PPM
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210
51.44
88.35
103.91
119.94
121.35
127.57
128.74
129.37
129.42
131.60
131.87
134.01
135.46
150.66
162.06
165.90

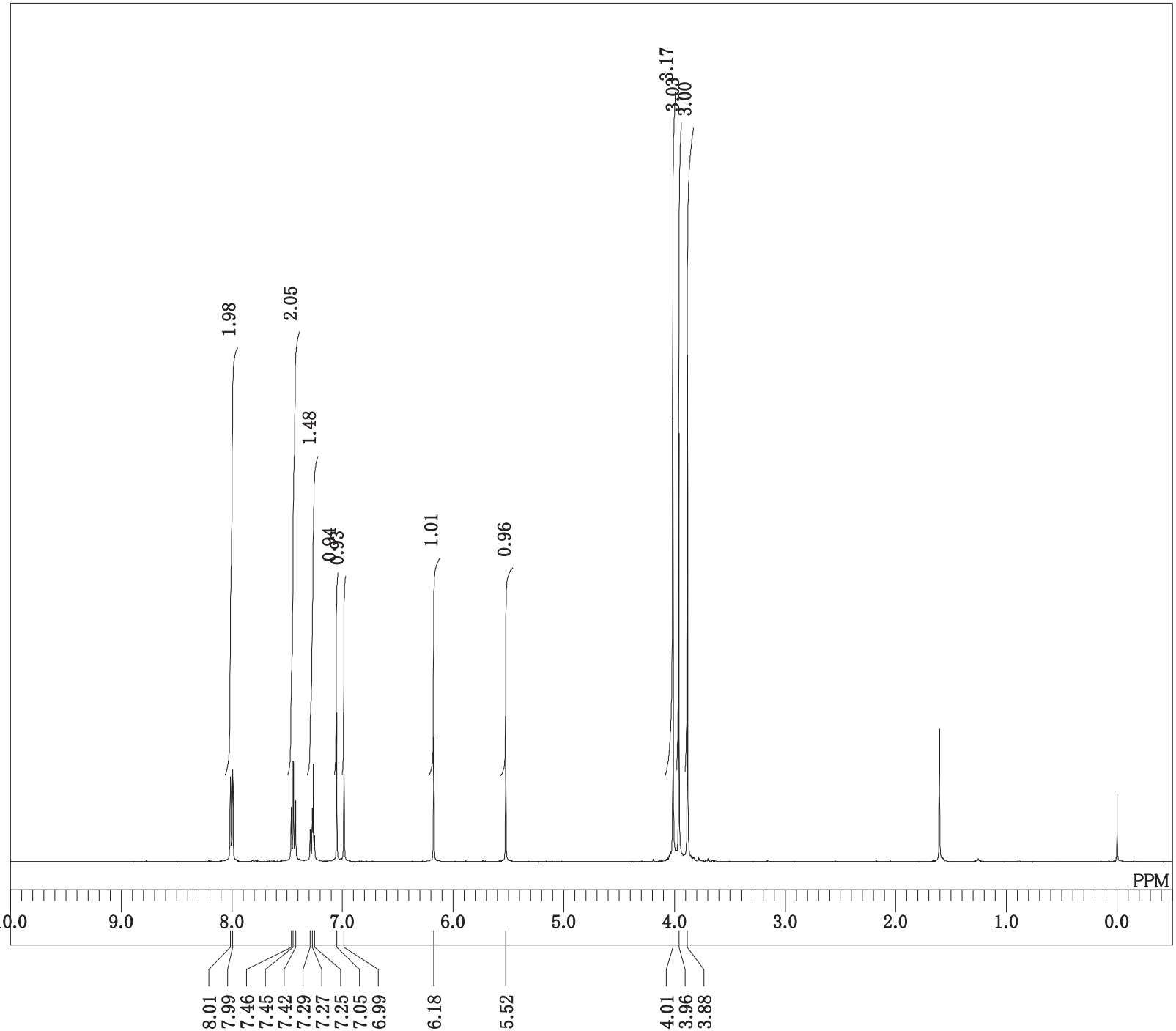

20130626 Me Ph Ph NOE 5-1.als
DPFGSE NOE 1d
2013-06-26 15:29:29
1H
noe_1d_dpfgse.ex
391.78 MHz
8.78 KHz
4.19 Hz
16384
7352.94 Hz
16
2.2282 sec
7.0000 sec
10.10 usec
1H
23.0 c
CDCL3
7.24 ppm
0.01 Hz
62
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
O
OMe
O
Ph
4a
S059
PPM
0
1
2
3
4
5
6
7
8
9
10
100.00
1.97
5.69
7.66
7.64



DFILE	20130626 Me Ph Ph NOE 6-1.als
COMNT	DPFGSE NOE 1d
DATIM	2013-06-26 15:36:11
OBNUC	1H
EXMOD	noe_1d_dpfgse.ex
OBFRQ	391.78 MHz
OBSET	9.03 KHz
OBFIN	6.18 Hz
POINT	16384
FREQU	7352.94 Hz
SCANS	16
ACQTM	2.2282 sec
PD	7.0000 sec
PW1	10.10 usec
IRNUC	1H
CTEMP	22.8 c
SLVNT	CDCL3
EXREF	7.24 ppm
BF	0.01 Hz
RGAIN	62

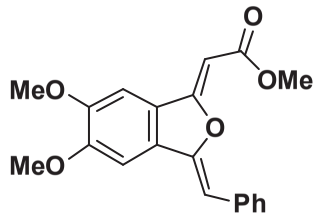


4a

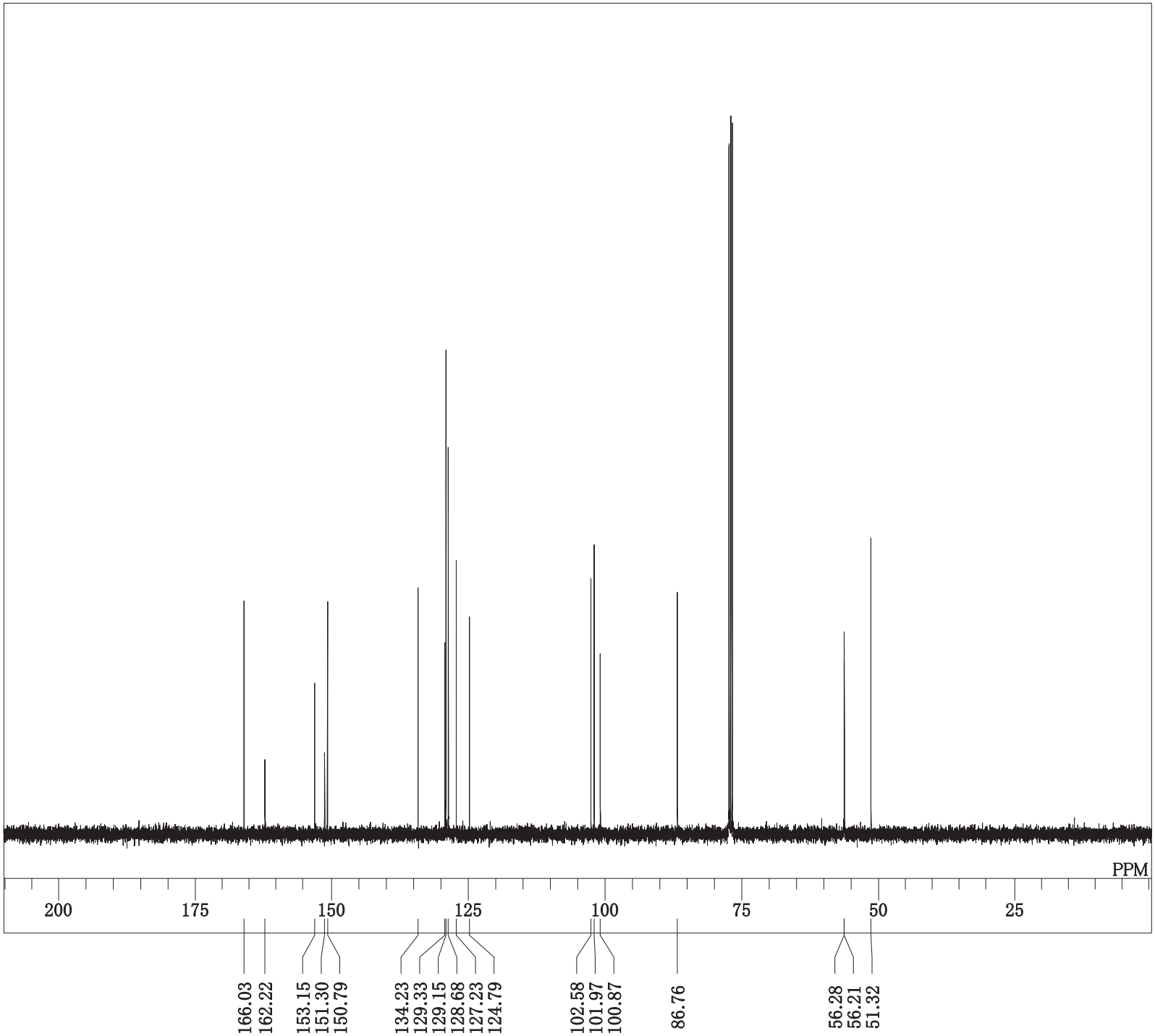


DFILE	20130617 ex1509E2 bera OMe 1H.als
COMNT	auto
DATIM	Mon Jun 17 20:53:14 2013
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.00 Hz
POINT	16384
FREQU	7992.01 Hz
SCANS	16
ACQTM	2.0500 sec
PD	2.0000 sec
PW1	6.60 usec
IRNUC	1H
CTEMP	19.5 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	17

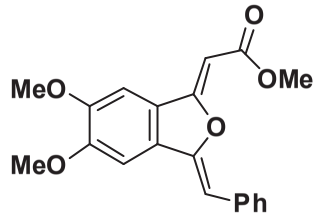
1H-NMR (CDCl3) δ :
8.00 (2H, d, J = 7.3 Hz),
7.44 (2H, t, J = 7.8 Hz),
7.27 (1H, t, J = 7.3 Hz),
6.18 (1H, s),
5.52 (1H, s).



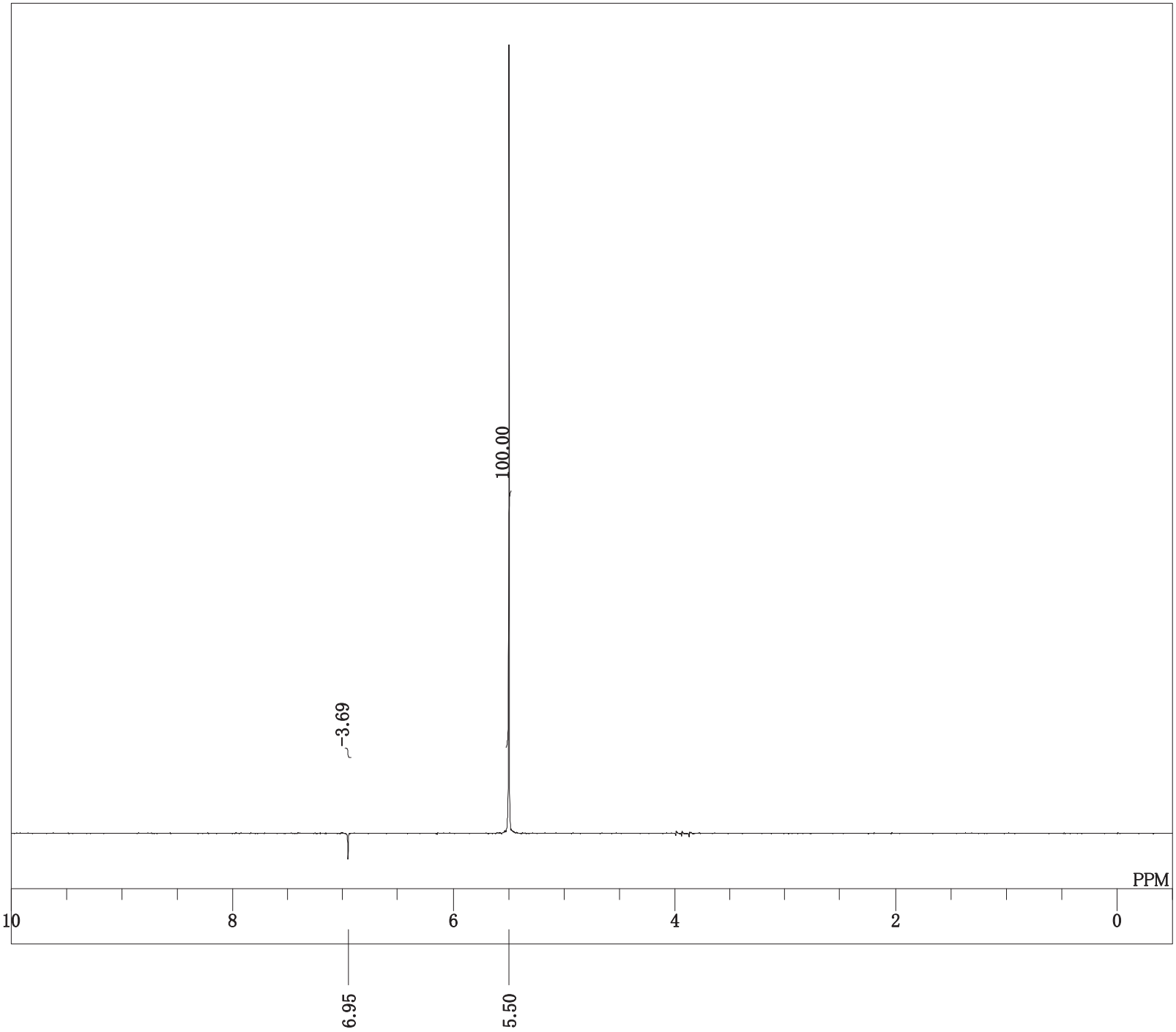
4b



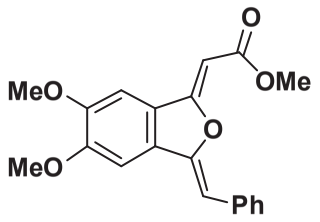
DFILE	20130617 ex1509E2 bera OMe pro 13C-
COMNT	single pulse decoupled gated NOE
DATIM	2013-06-17 16:13:36
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	32768
FREQU	30788.18 Hz
SCANS	512
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	2.87 usec
IRNUC	1H
CTEMP	22.3 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	36



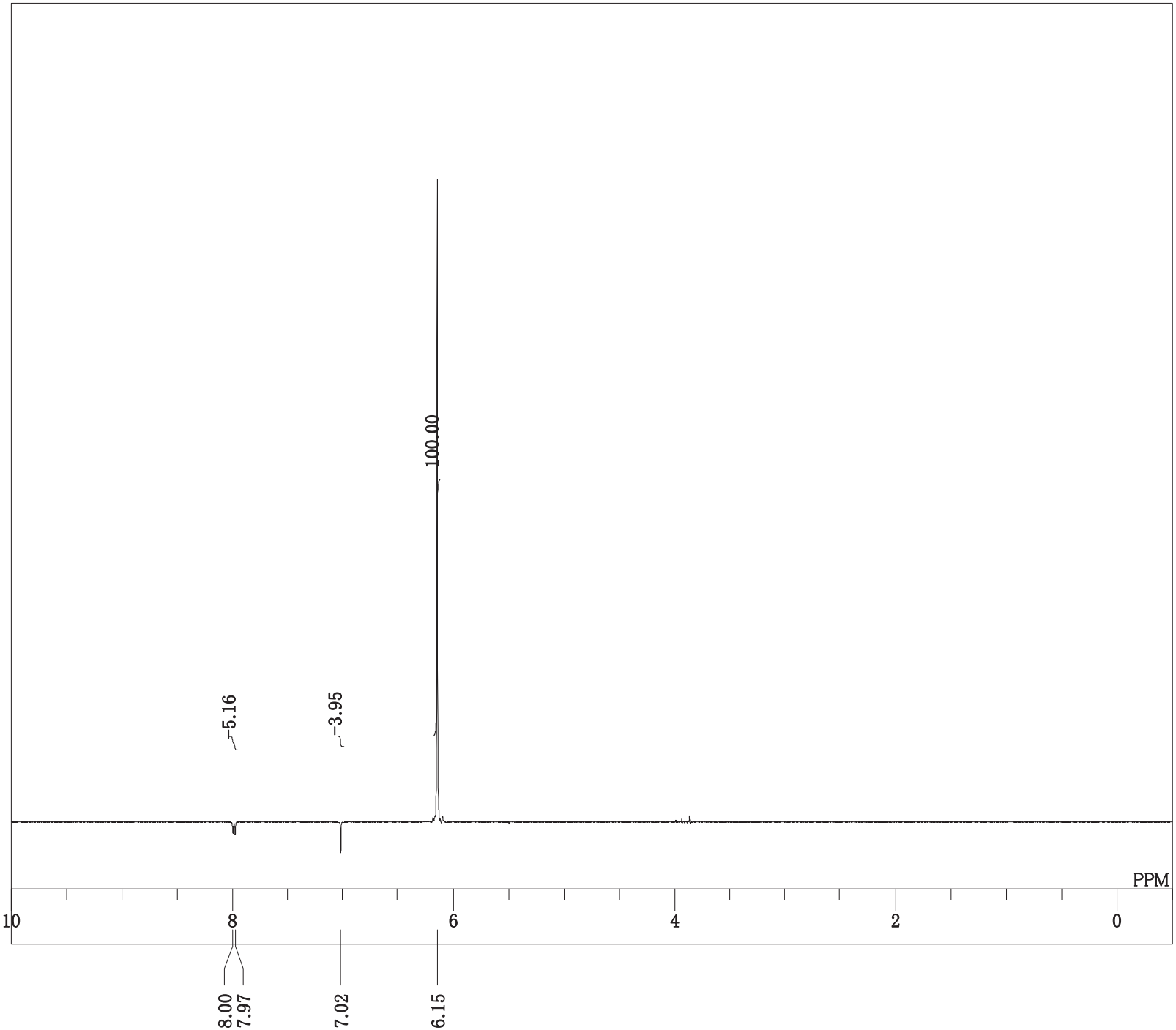
4b



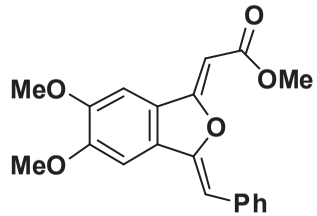
DFILE	20130617 ex1509E2 bera OMe pro NOE
COMNT	DPFGSE NOE 1d
DATIM	2013-06-17 15:36:58
OBNUC	1H
EXMOD	noe_1d_dpfgse.ex
OBFRQ	391.78 MHz
OBSET	8.70 KHz
OBFIN	7.90 Hz
POINT	16384
FREQU	7352.94 Hz
SCANS	16
ACQTM	2.2282 sec
PD	7.0000 sec
PW1	10.10 usec
IRNUC	1H
CTEMP	22.3 c
SLVNT	CDCL3
EXREF	7.24 ppm
BF	0.12 Hz
RGAIN	60



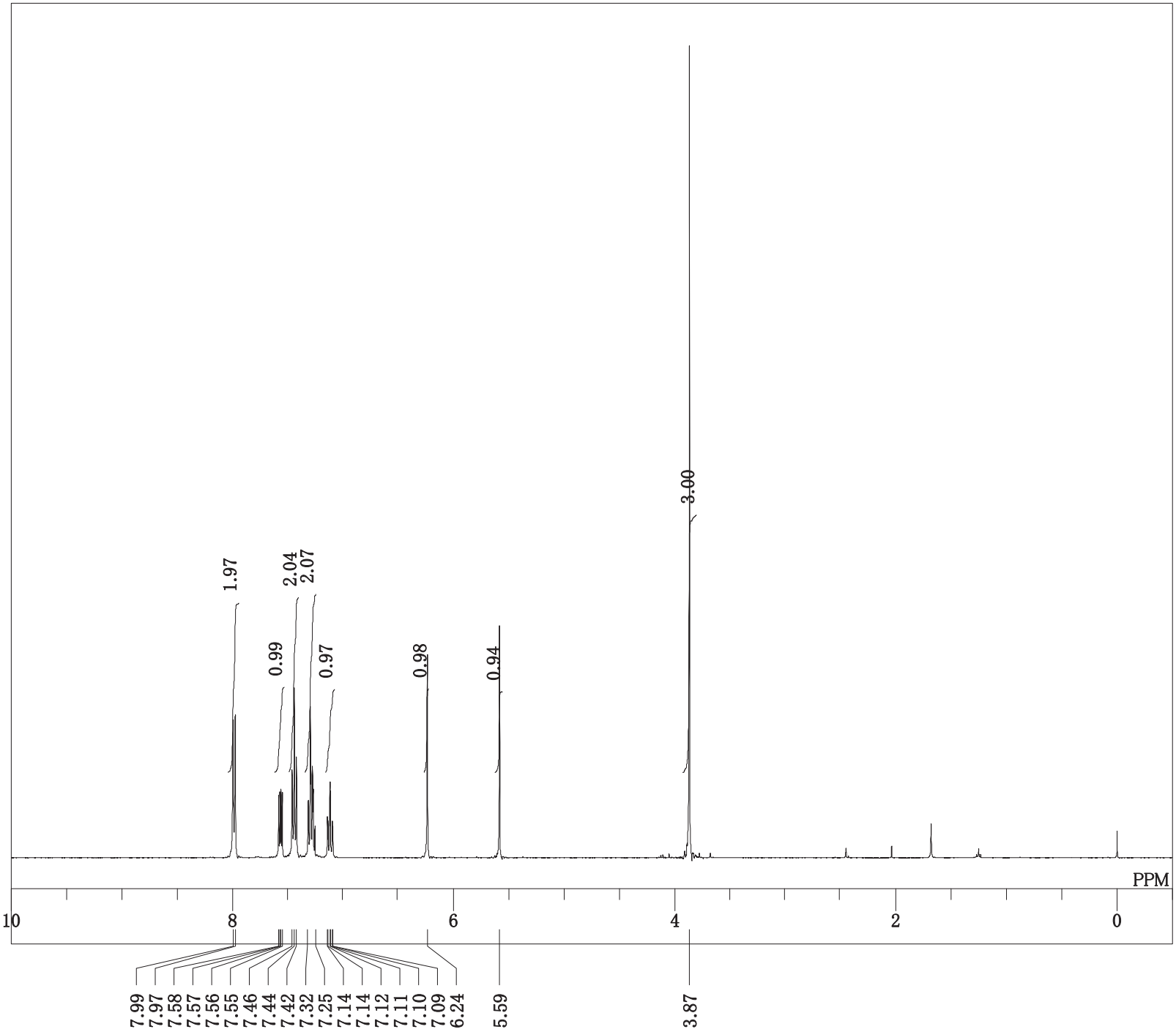
4b



DFILE	20130617 ex1509E2 bera OMe pro NOE
COMNT	DPFGSE NOE 1d
DATIM	2013-06-17 15:43:42
OBNUC	1H
EXMOD	noe_1d_dpfgse.ex
OBFRQ	391.78 MHz
OBSET	8.96 KHz
OBFIN	1.90 Hz
POINT	16384
FREQU	7352.94 Hz
SCANS	16
ACQTM	2.2282 sec
PD	7.0000 sec
PW1	10.10 usec
IRNUC	1H
CTEMP	22.3 c
SLVNT	CDCL3
EXREF	7.24 ppm
BF	0.12 Hz
RGAIN	60

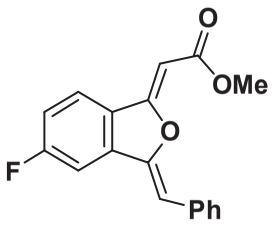


4b

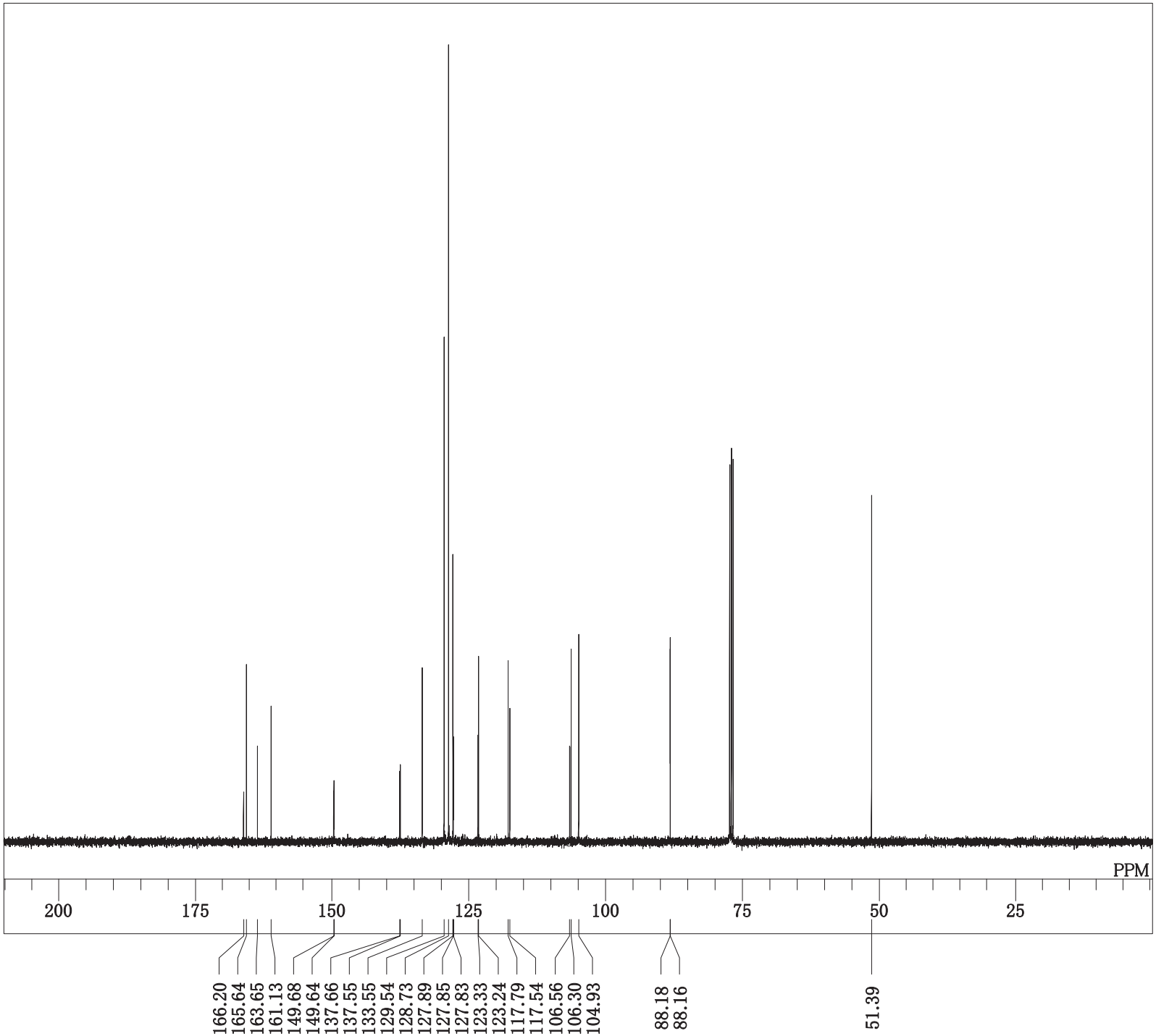


DFILE	20130615 ex1509E1 F pro 1H-1.as
COMNT	single_pulse
DATIM	2013-06-15 09:07:02
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	32768
FREQU	7352.94 Hz
SCANS	8
ACQTM	4.4564 sec
PD	3.0000 sec
PW1	5.05 usec
IRNUC	1H
CTEMP	22.0 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	38

1H-NMR (CDCl3) δ :
7.98 (2H, d, J = 7.6 Hz),
7.57 (1H, dd, J = 8.5, 4.5 Hz),
7.44 (2H, t, J = 7.7 Hz),
7.12 (1H, td, J = 8.6, 2.1 Hz).

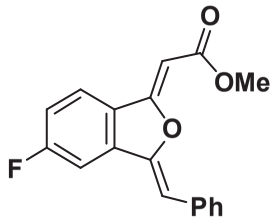


4c

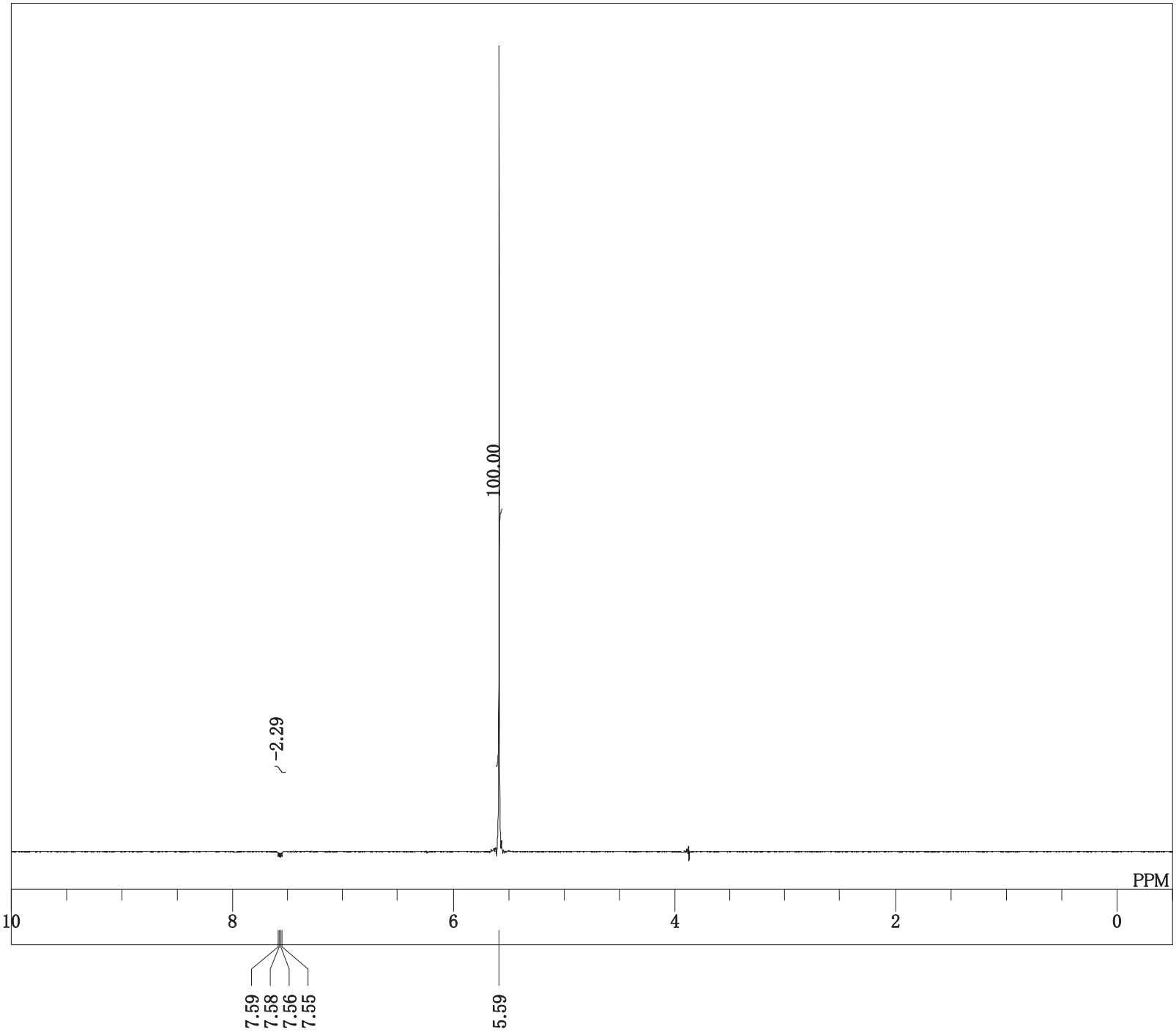


DFILE 20130615 ex1509E1 F pro 13C-1.ais
COMNT single pulse decoupled gated NOE
DATIM 2013-06-15 09:46:34
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 32768
FREQU 30788.18 Hz
SCANS 512
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 2.87 usec
IRNUC 1H
CTEMP 22.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 36

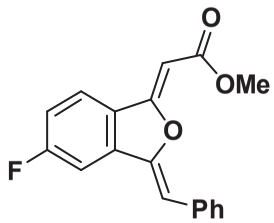
13C-NMR (CDCl3) δ :
164.93 (0H, d, J = 251.8 Hz),
149.66 (0H, d, J = 4.7 Hz),
137.61 (0H, d, J = 10.3 Hz),
127.84 (0H, d, J = 1.9 Hz),
123.29 (0H, d, J = 9.4 Hz),
117.67 (0H, d, J = 24.4 Hz),
106.43 (0H, d, J = 25.4 Hz),
88.17 (0H, d, J = 1.9 Hz).



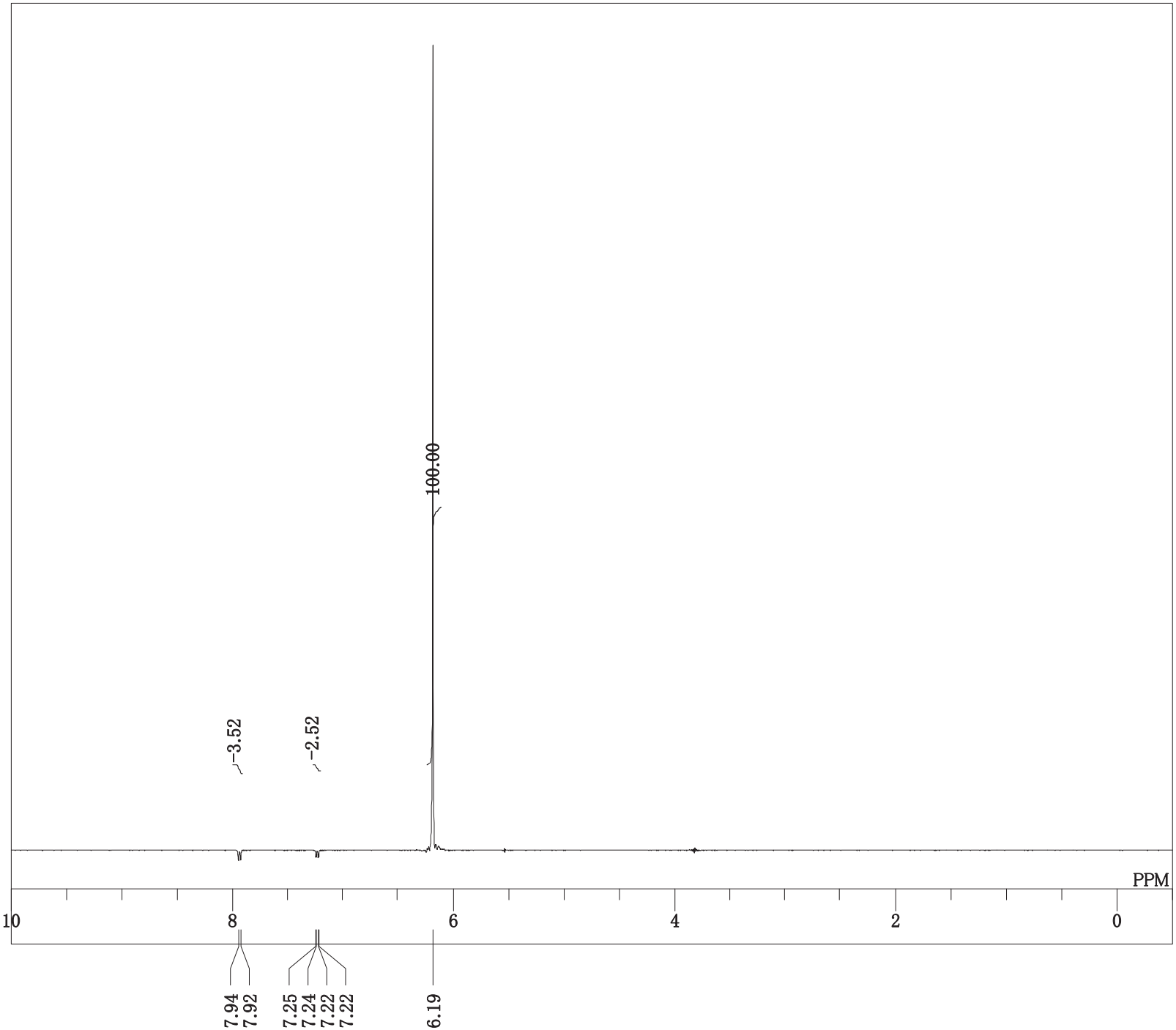
4c



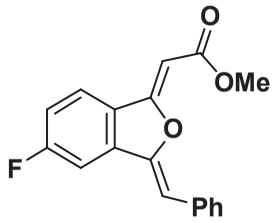
DFILE	20130615 ex1509E1 F pro NOE 5-1 1d
COMNT	DPFGSE NOE 1d
DATIM	2013-06-15 09:12:58
OBNUC	1H
EXMOD	noe_1d_dpfgse.ex
OBFRQ	391.78 MHz
OBSET	8.74 KHz
OBFIN	2.89 Hz
POINT	16384
FREQU	7352.94 Hz
SCANS	16
ACQTM	2.2282 sec
PD	7.0000 sec
PW1	10.10 usec
IRNUC	1H
CTEMP	21.9 c
SLVNT	CDCL3
EXREF	7.24 ppm
BF	0.12 Hz
RGAIN	56



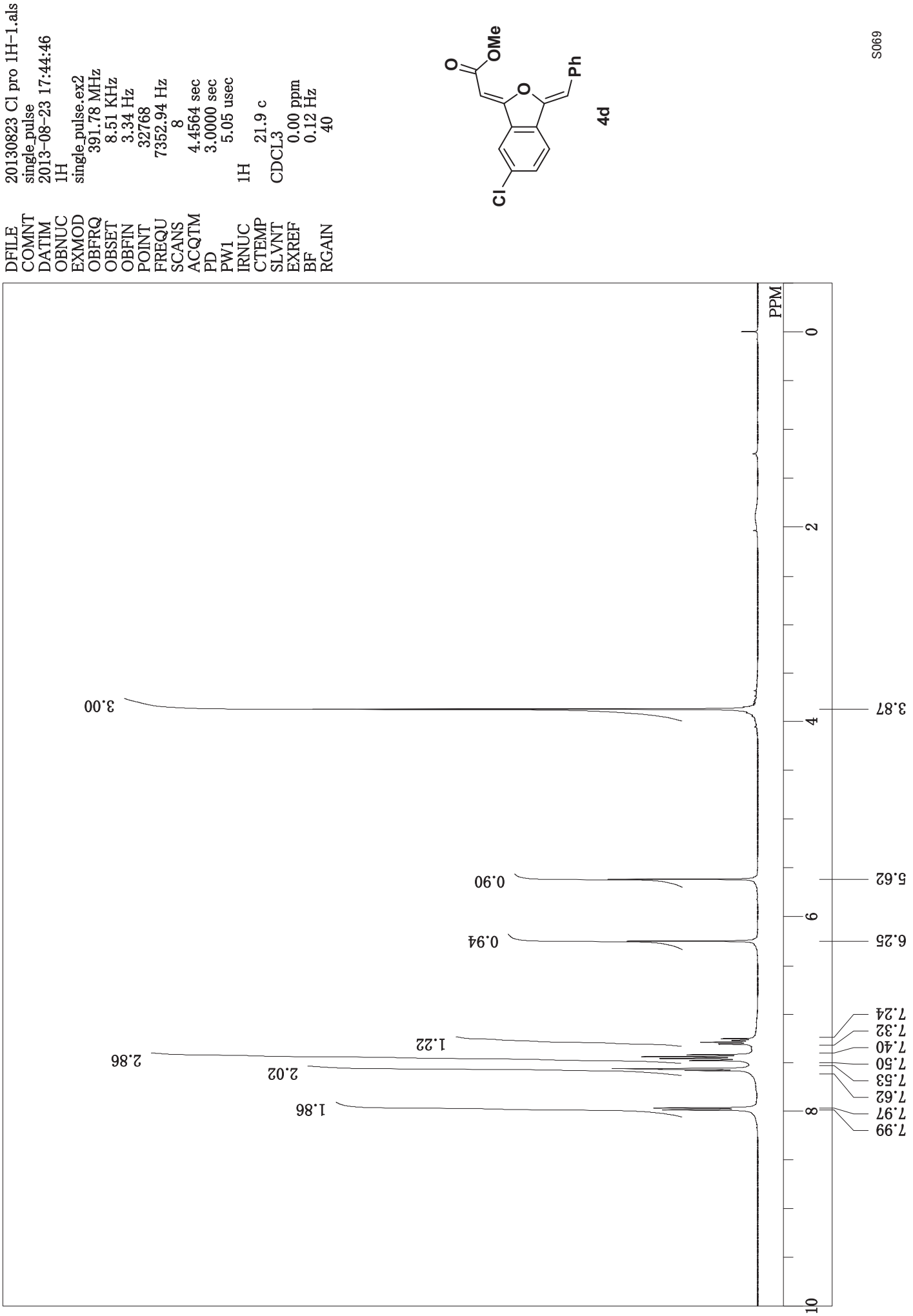
4c

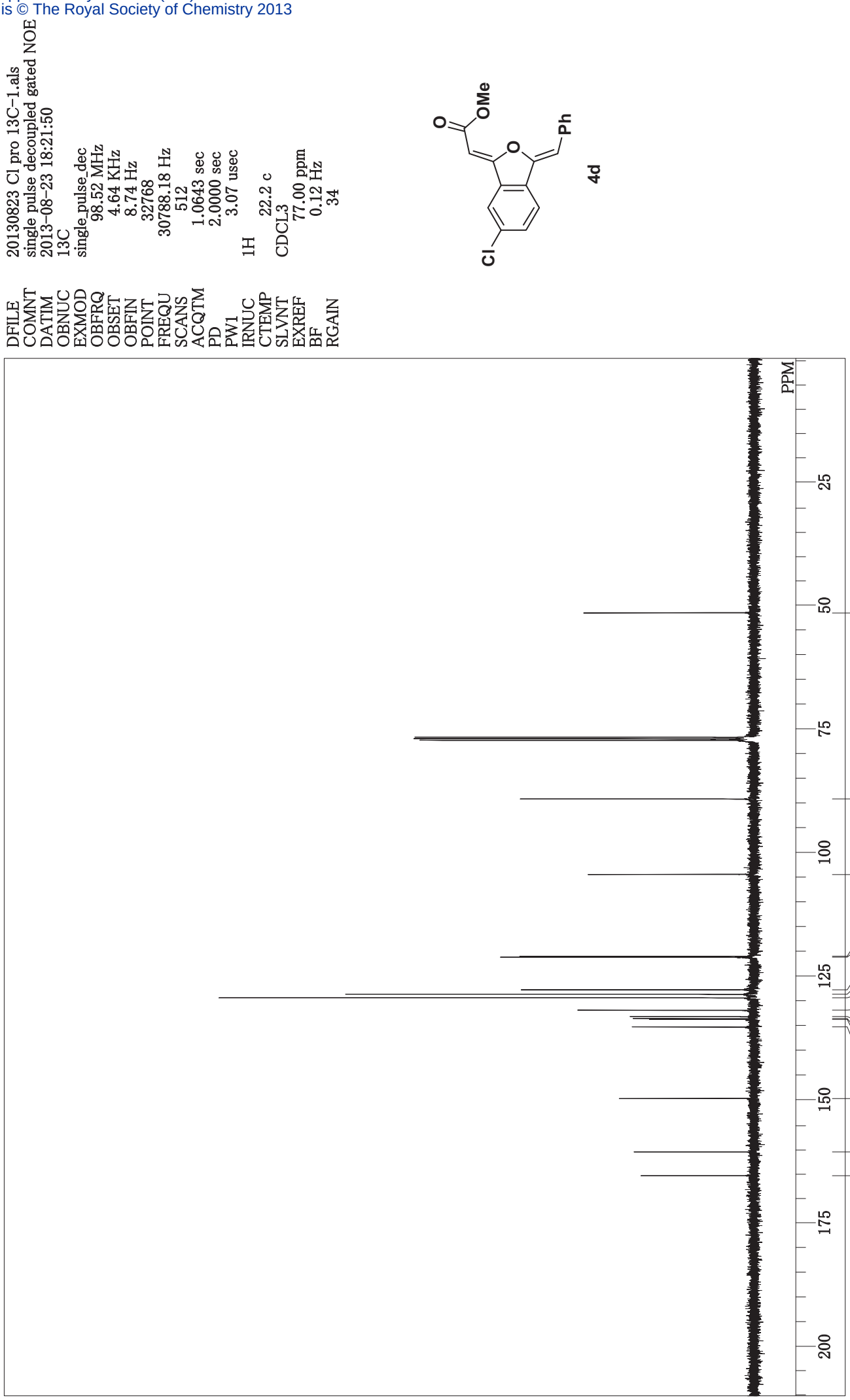


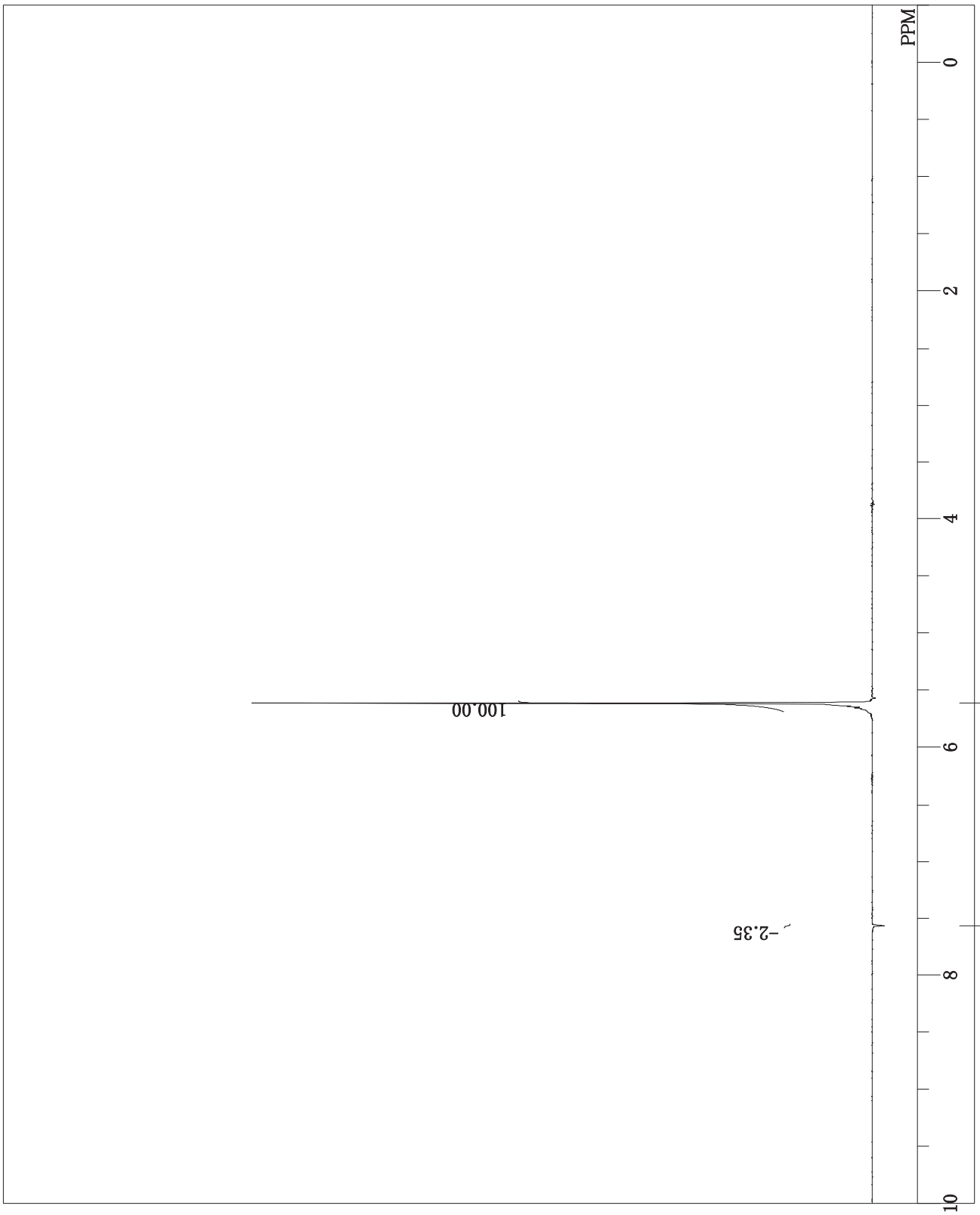
DFILE	20130615 ex1509E1 F pro NOE 6-1 1s
COMNT	DPFGSE NOE 1d
DATIM	2013-06-15 09:17:46
OBNUC	1H
EXMOD	noe_1d_dpfgse.ex
OBFRQ	391.78 MHz
OBSET	8.99 KHz
OBFIN	8.49 Hz
POINT	16384
FREQU	7352.94 Hz
SCANS	16
ACQTM	2.2282 sec
PD	7.0000 sec
PW1	10.10 usec
IRNUC	1H
CTEMP	22.1 c
SLVNT	CDCL3
EXREF	7.24 ppm
BF	0.12 Hz
RGAIN	56

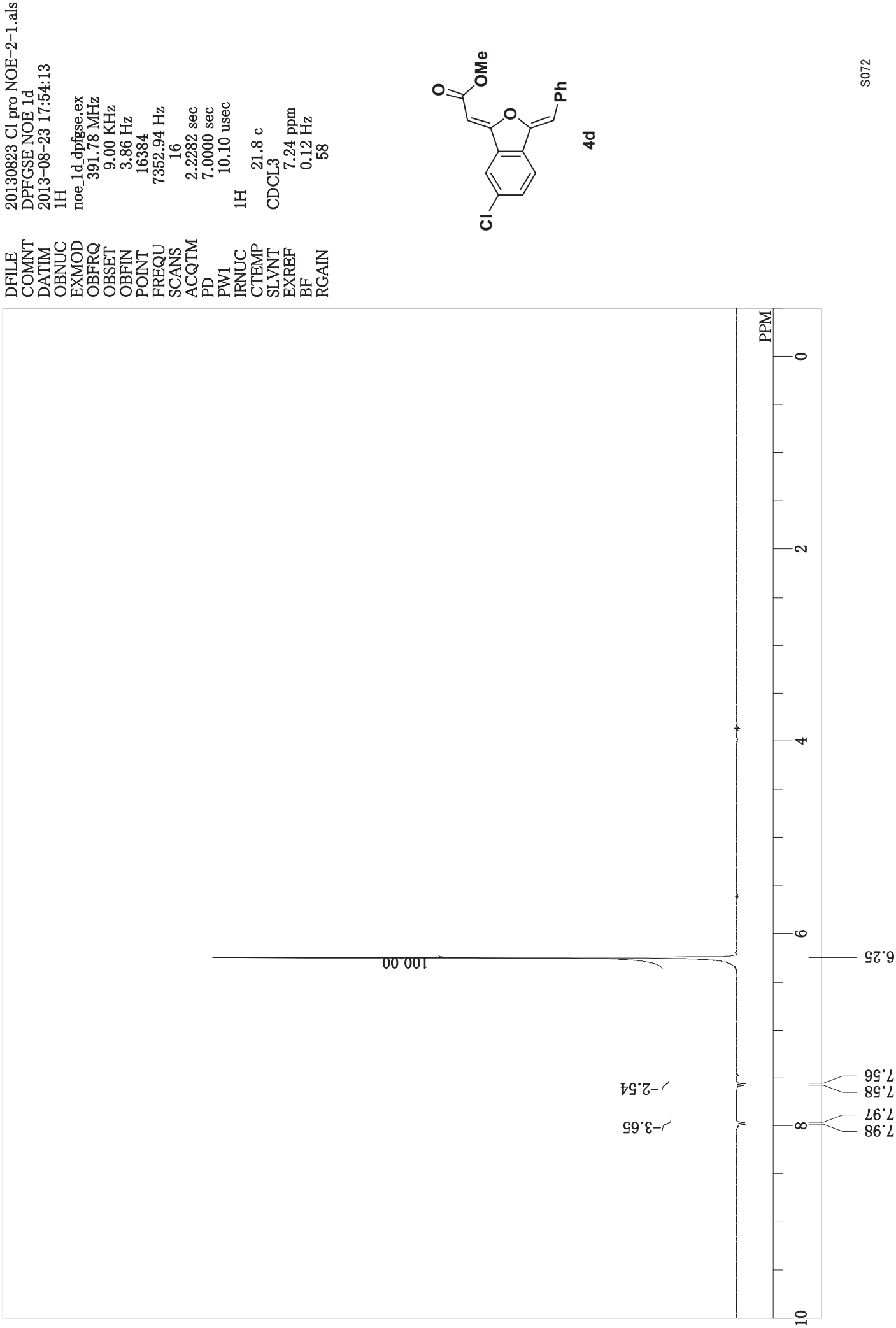


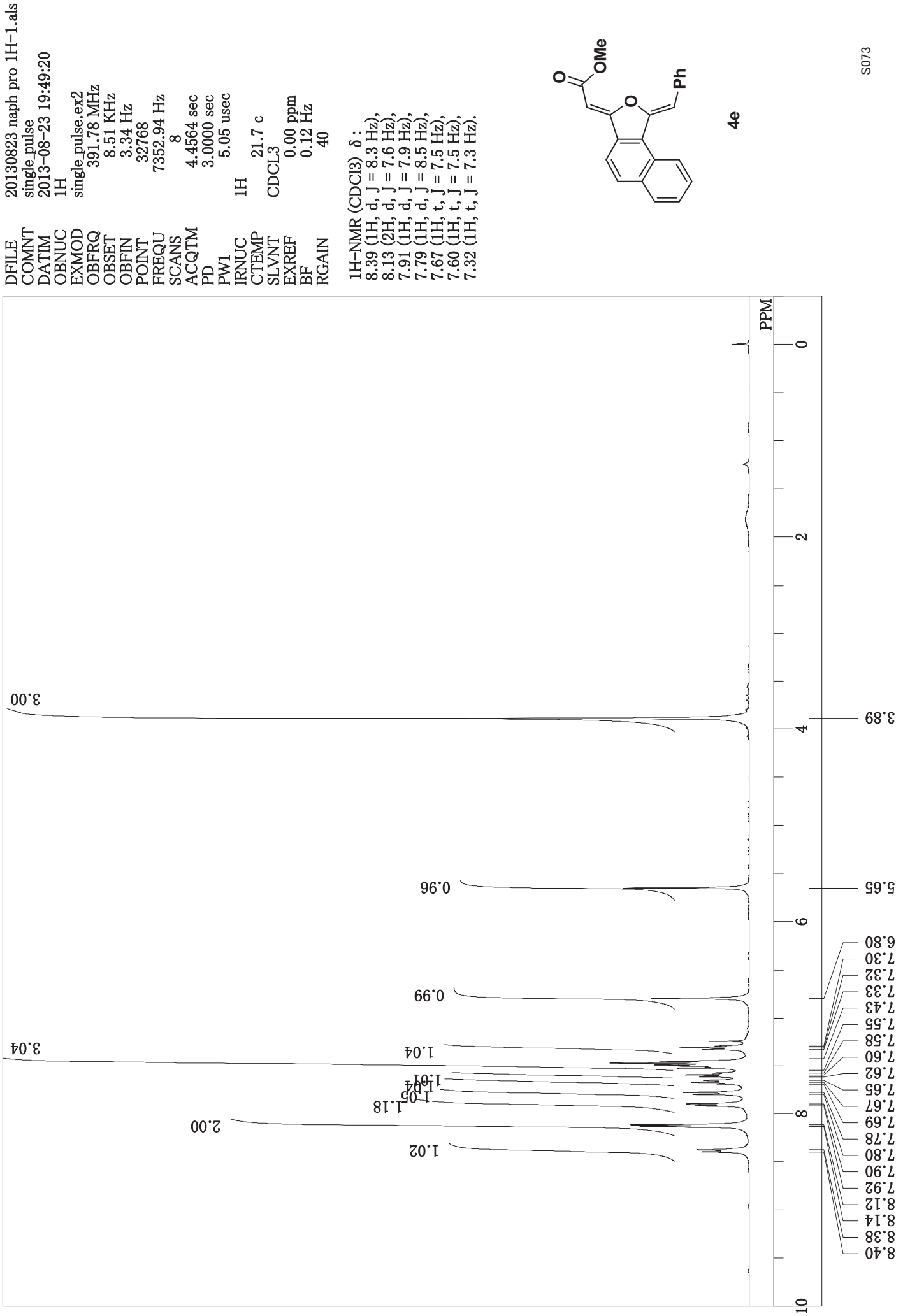
4c

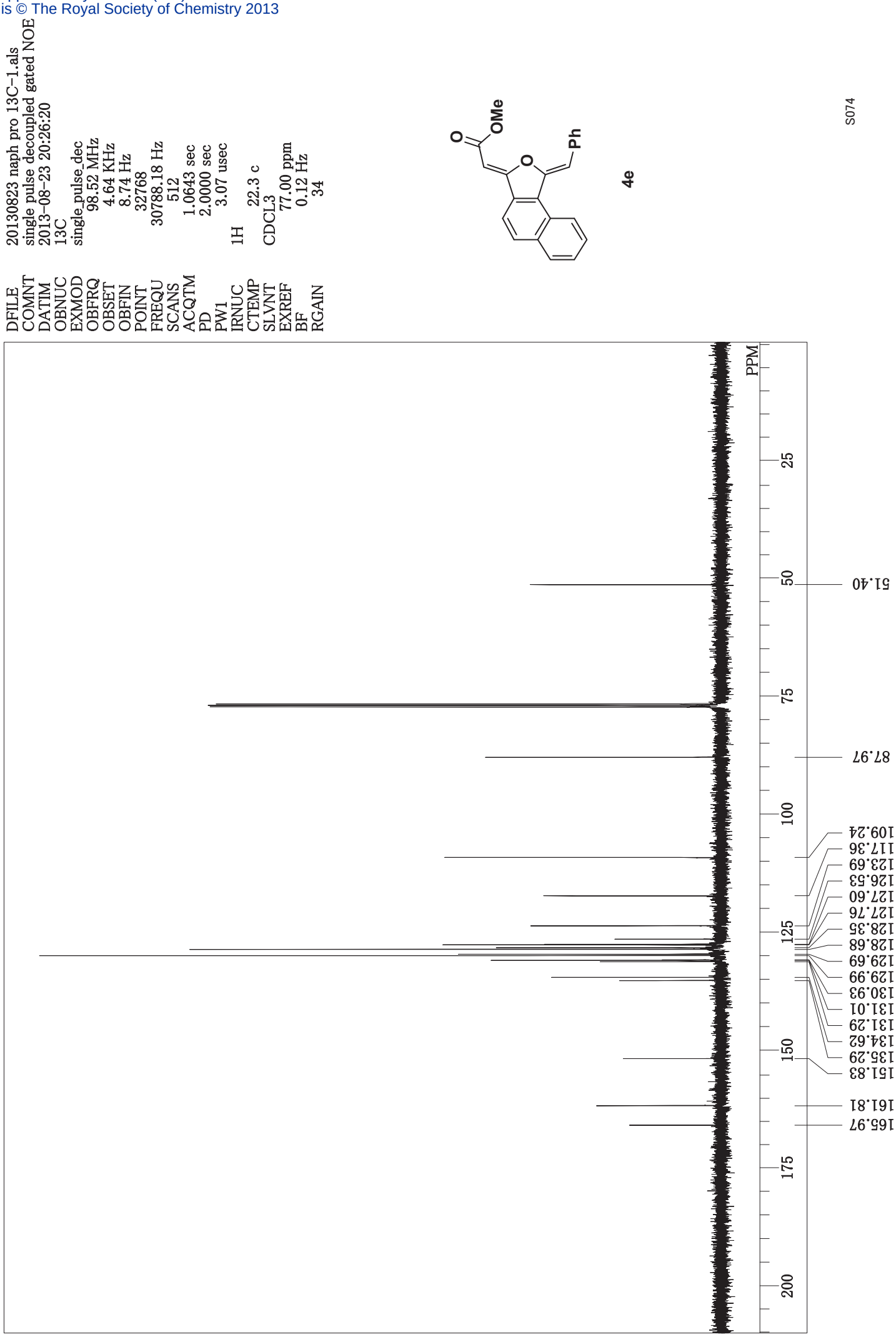

DFILE 20130823 Cl pro 1H-1.als
COMNT single_pulse
DATIM 2013-08-23 17:44:46
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 391.78 MHz
OBSET 8.51 KHz
OBFIN 3.34 Hz
POINT 32768
FREQU 7352.94 Hz
SCANS 8
ACQTM 4.4564 sec
PD 3.0000 sec
PW1 5.05 usec
IRNUC 1H
CTEMP 21.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 40
O
OMe
O
Ph
Cl
4d
S069
PPM
0
2
4
6
8
10
3.87
5.62
6.25
7.24
7.32
7.40
7.50
7.53
7.62
7.97
7.99
3.00
0.90
0.94
1.22
2.86
2.02
1.86

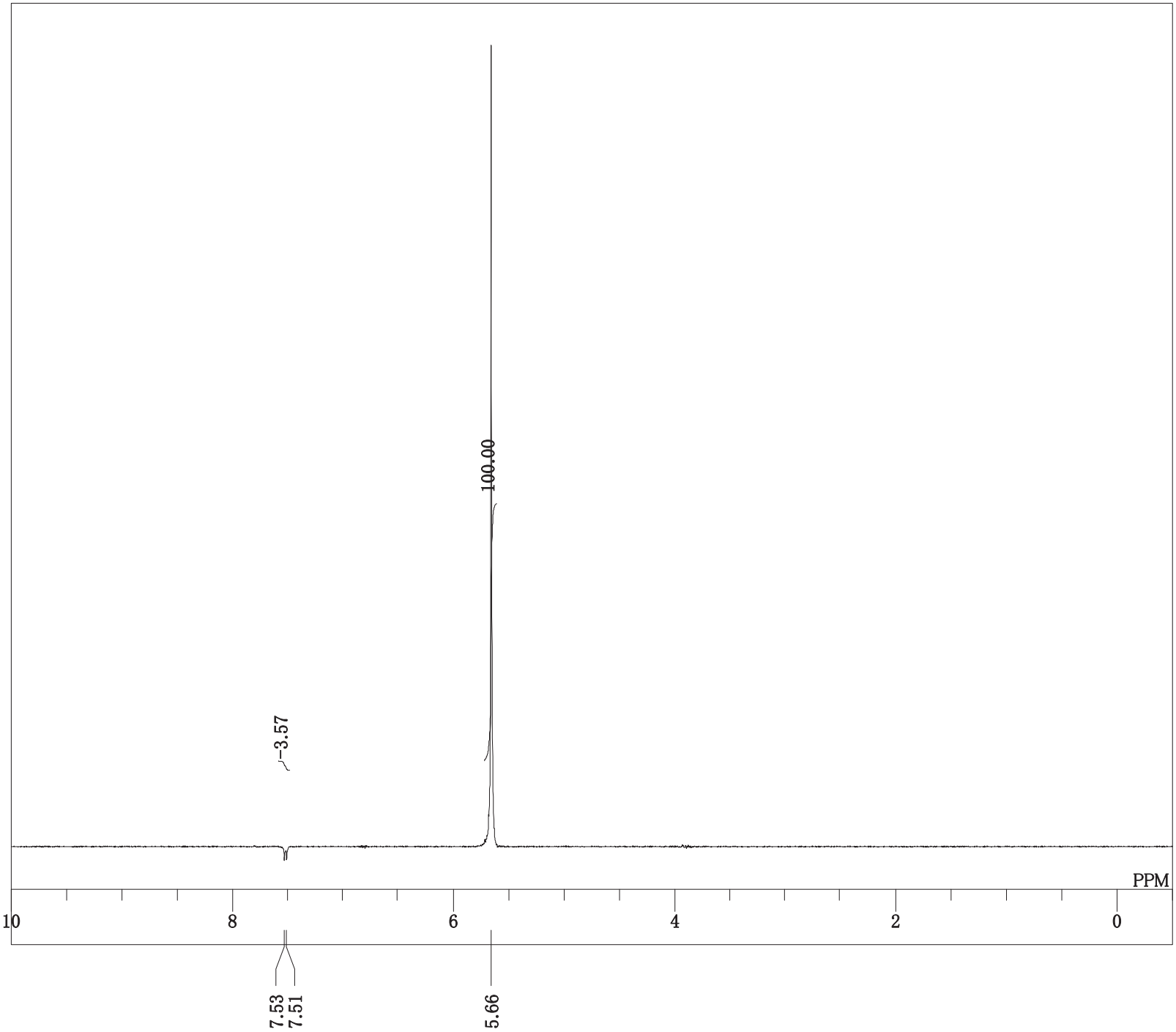



20130823 Cl pro NOE-1-1.als
DPFGSE NOE 1d
2013-08-23 17:49:37
1H
noe_1d_dpfgse.ex
391.78 MHz
8.75 KHz
5.23 Hz
16384
7352.94 Hz
16
2.2282 sec
7.0000 sec
10.10 usec
1H
21.9 c
CDCL3
7.24 ppm
0.12 Hz
58
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
O
OMe
O
Ph
Cl
4d
S071
PPM
0
2
4
6
8
10
100.00
2.35
5.62
7.57

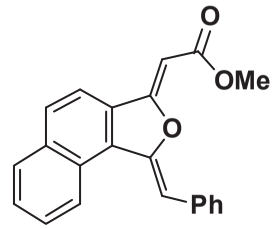

20130823 Cl pro NOE-2-1.als
DPFGSE NOE 1d
2013-08-23 17:54:13
1H
noe_1d_dpfgse.ex
391.78 MHz
9.00 KHz
3.86 Hz
16384
7352.94 Hz
16
2.2282 sec
7.0000 sec
10.10 usec
1H
21.8 c
CDCL3
7.24 ppm
0.12 Hz
58
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
O
OMe
O
Ph
Cl
4d
S072
100.00
3.65
2.54
7.98
7.97
7.58
7.56
6.25
PPM
10
8
6
4
2
0


DFILE 20130823 naph pro 1H-1.als
COMNT single_pulse
DATIM 2013-08-23 19:49:20
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 391.78 MHz
OBSET 8.51 KHz
OBFIN 3.34 Hz
POINT 32768
FREQU 7352.94 Hz
SCANS 8
ACQTM 4.4564 sec
PD 3.0000 sec
PW1 5.05 usec
IRNUC 1H
CTEMP 21.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 40
1H-NMR (CDCl3) δ :
8.39 (1H, d, J = 8.3 Hz),
8.13 (2H, d, J = 7.6 Hz),
7.91 (1H, d, J = 7.9 Hz),
7.79 (1H, d, J = 8.5 Hz),
7.67 (1H, t, J = 7.5 Hz),
7.60 (1H, t, J = 7.5 Hz),
7.32 (1H, t, J = 7.3 Hz).
O
OMe
O
Ph
4e
S073
PPM
0
2
4
6
8
10
3.00
0.96
0.99
3.04
1.04
1.01
1.04
1.05
1.18
2.00
1.02
3.89
5.65
6.80
7.30
7.32
7.33
7.43
7.55
7.58
7.60
7.62
7.65
7.67
7.69
7.78
7.80
7.90
7.92
8.12
8.14
8.38
8.40

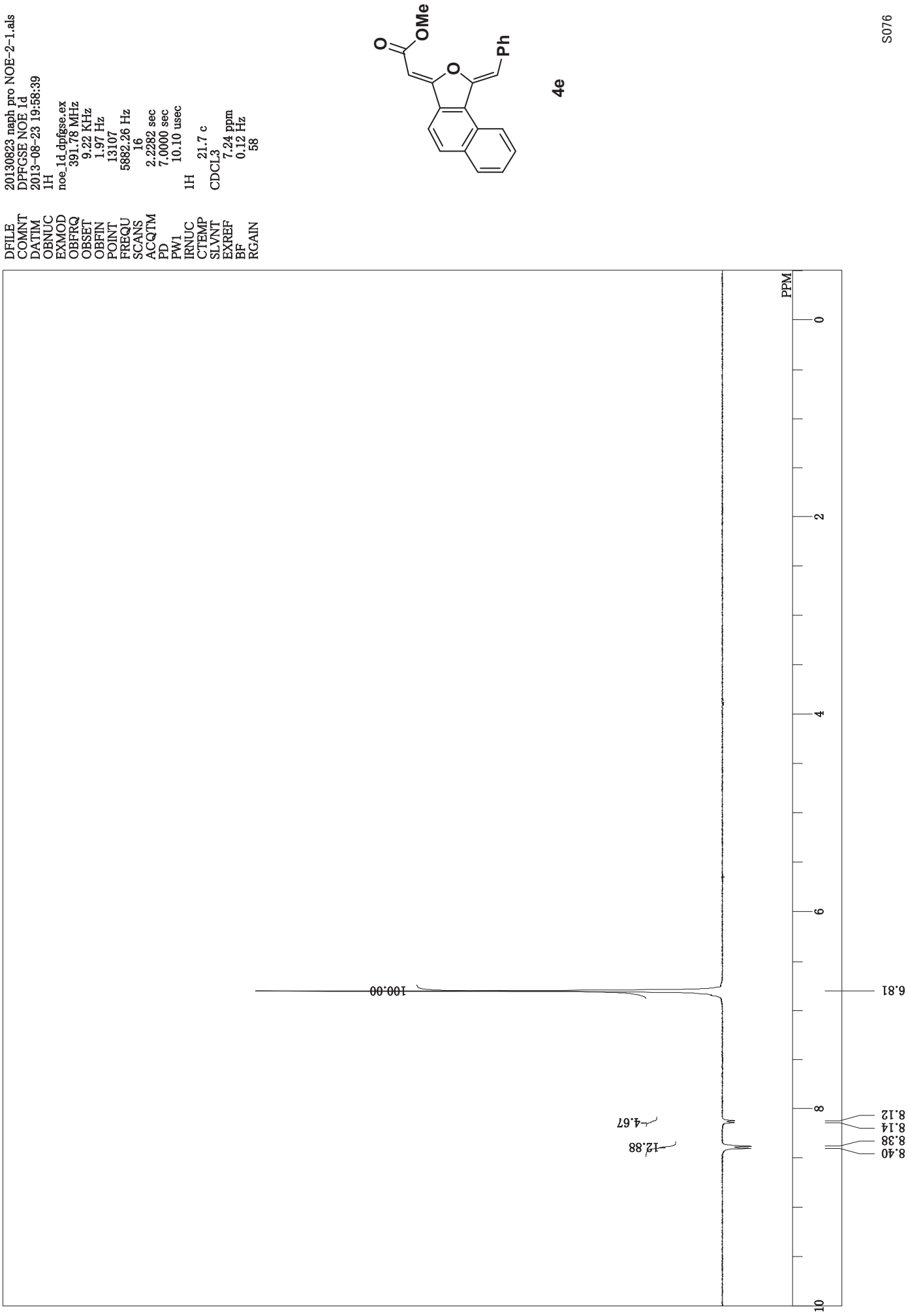

20130823 naph pro 13C-1.als
single pulse decoupled gated NOE
2013-08-23 20:26:20
13C
single_pulse_dec
98.52 MHz
4.64 KHz
8.74 Hz
32768
30788.18 Hz
512
1.0643 sec
2.0000 sec
3.07 usec
1H
22.3 c
CDCL3
77.00 ppm
0.12 Hz
34
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
O
OMe
O
Ph
4e
S074
PPM
25
50
75
100
125
150
175
200
51.40
87.97
109.24
117.36
123.69
126.53
127.60
127.76
128.35
128.68
129.69
129.99
130.93
131.01
131.29
134.62
135.29
151.83
161.81
165.97

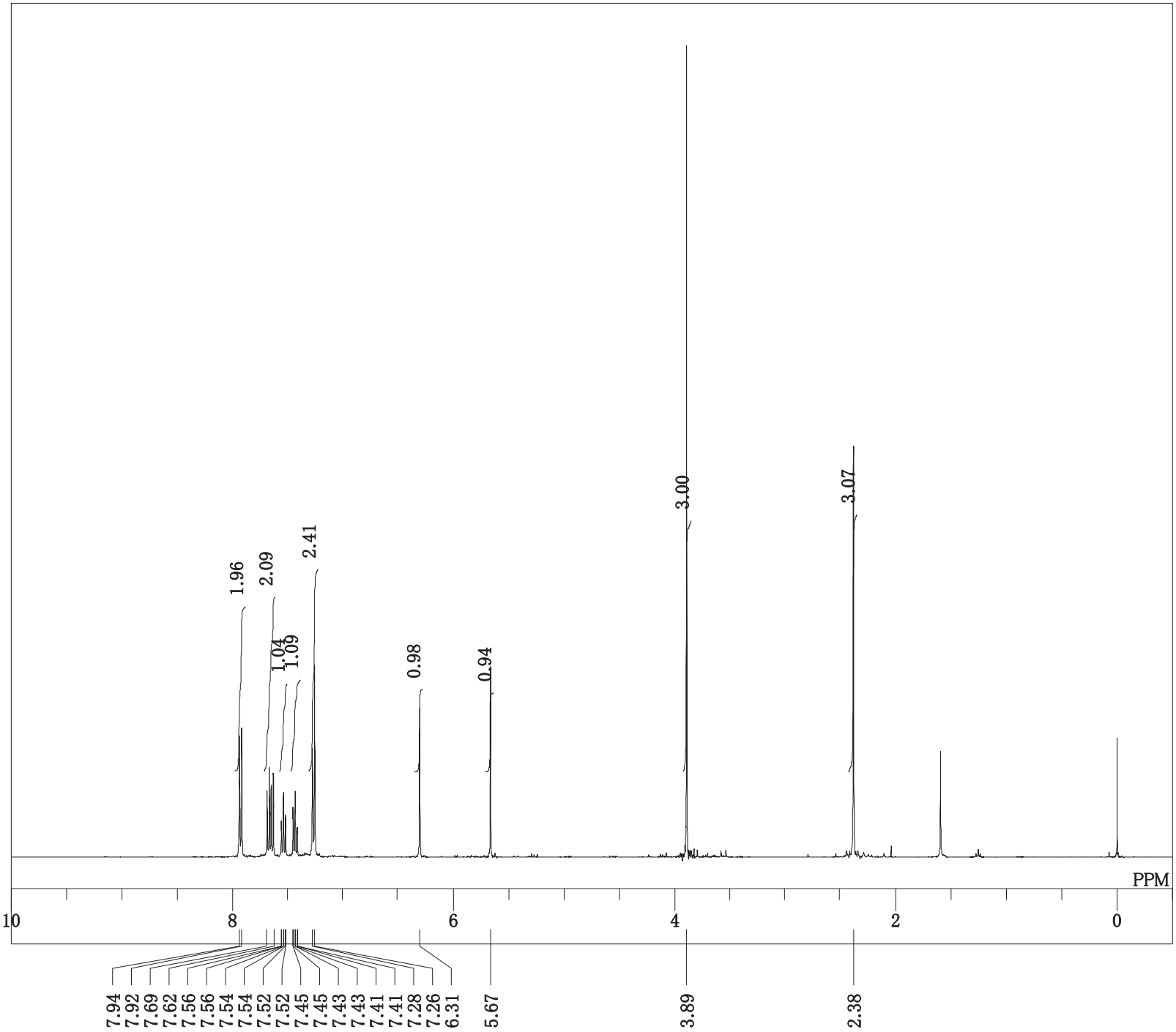


DFILE	20130823 naph pro NOE-1-1.als
COMNT	DPFGSE NOE 1d
DATIM	2013-08-23 19:54:03
OBNUC	1H
EXMOD	noe_1d_dpfgse.ex
OBFRQ	391.78 MHz
OBSET	8.77 KHz
OBFIN	2.08 Hz
POINT	16384
FREQU	7352.94 Hz
SCANS	16
ACQTM	2.2282 sec
PD	7.0000 sec
PW1	10.10 usec
IRNUC	1H
CTEMP	21.8 c
SLVNT	CDCL3
EXREF	7.24 ppm
BF	0.12 Hz
RGAIN	58



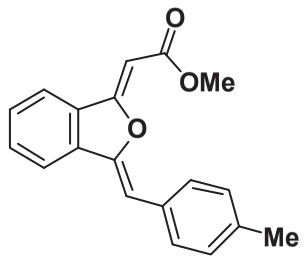
4e



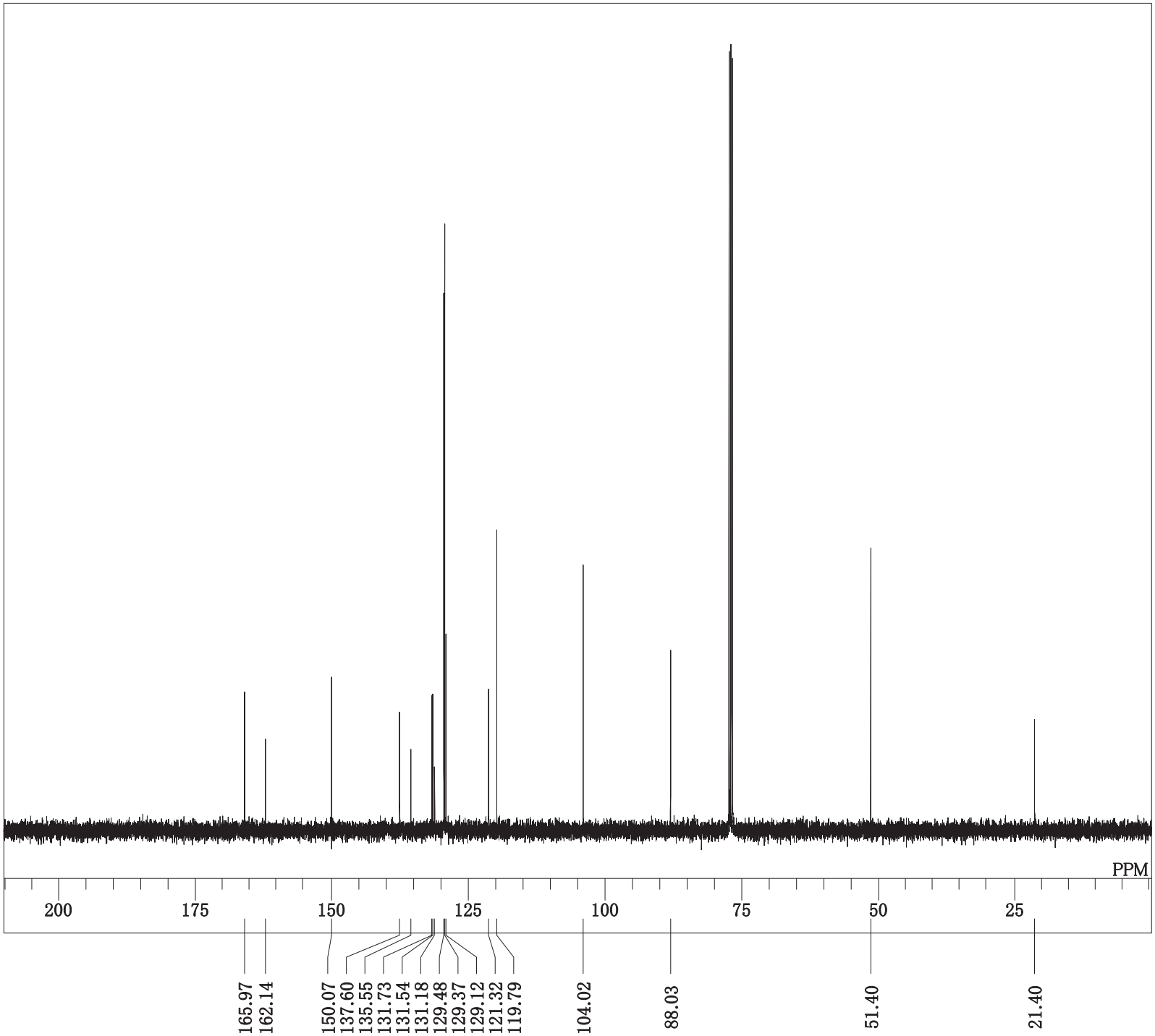


DFILE	20130601 ex1489E1 p-Tol pro 1H-1.fid
COMNT	single_pulse
DATIM	2013-06-01 09:22:57
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	32768
FREQU	7352.94 Hz
SCANS	8
ACQTM	4.4564 sec
PD	3.0000 sec
PW1	5.05 usec
IRNUC	1H
CTEMP	22.2 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	44

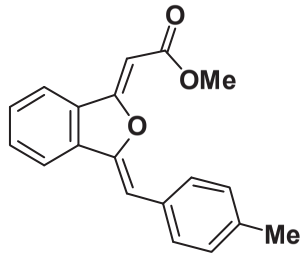
1H-NMR (CDCl3) δ :
7.93 (2H, d, J = 8.1 Hz),
7.54 (1H, td, J = 7.5, 1.0 Hz),
7.43 (1H, td, J = 7.5, 0.9 Hz),
7.27 (2H, d, J = 7.4 Hz).



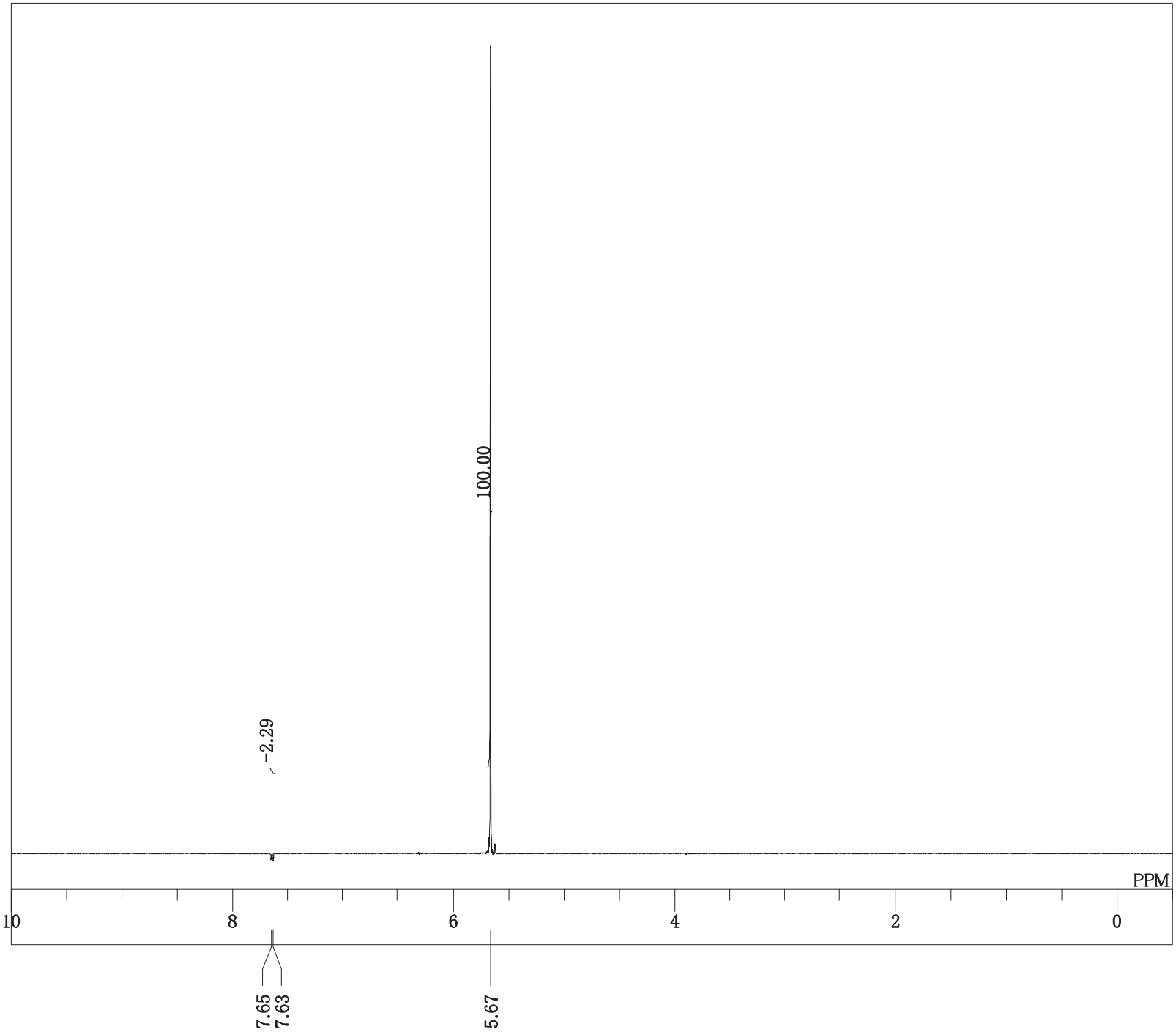
4f



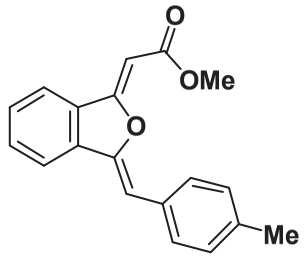
DFILE	20130601 ex1489E1 p-Tol pro 13C 1.fal
COMNT	single pulse decoupled gated NOE
DATIM	2013-06-01 10:17:34
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	32768
FREQU	30788.18 Hz
SCANS	512
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	2.87 usec
IRNUC	1H
CTEMP	22.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	34



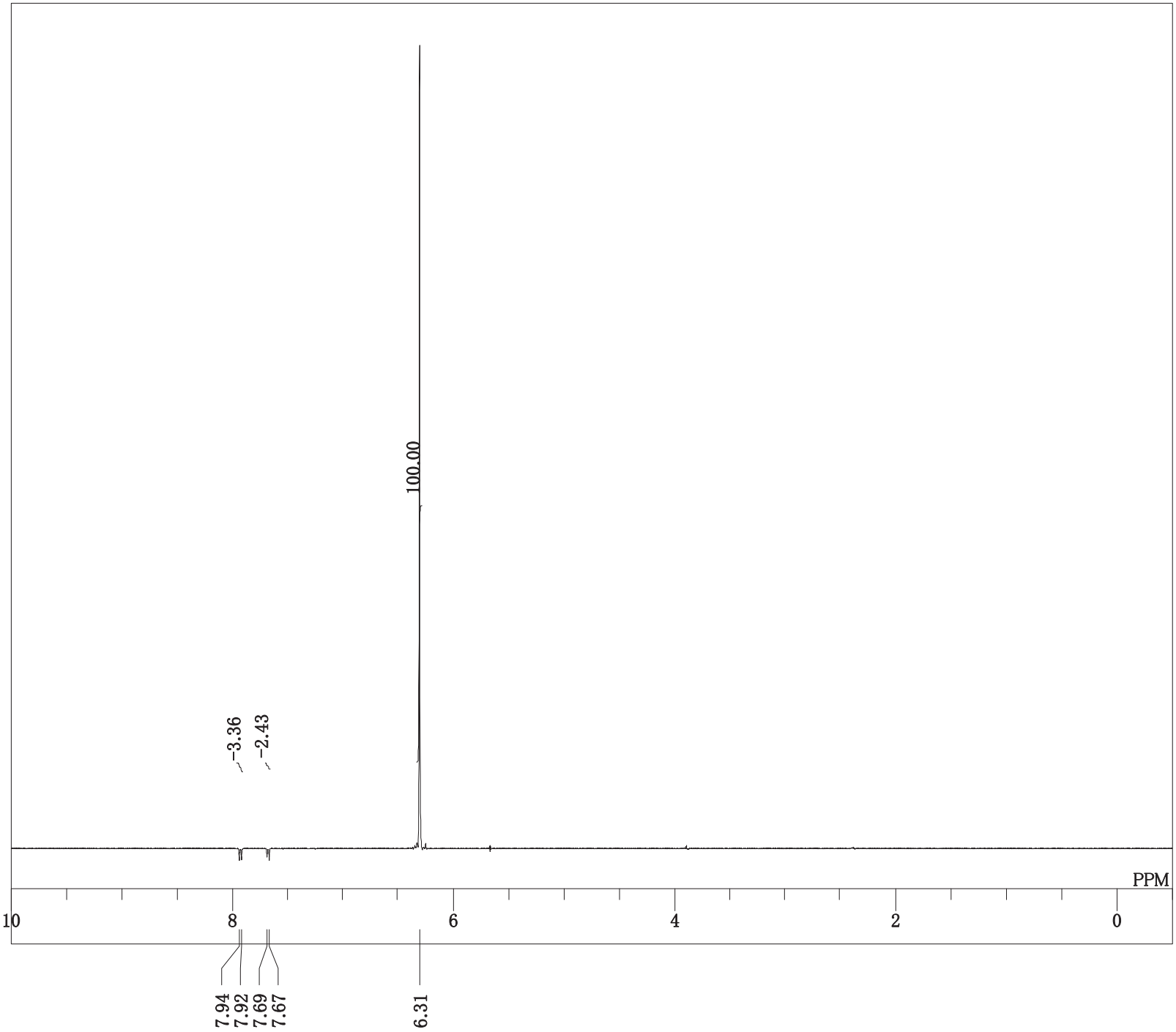
4f



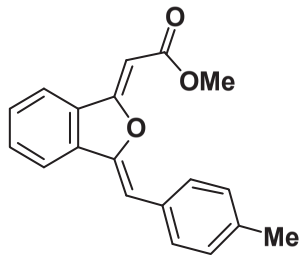
DFILE	20130601 ex1489E1 p-Tol pro NOE 1-2
COMNT	DPFGSE NOE 1d
DATIM	2013-06-01 09:47:56
OBNUC	1H
EXMOD	noe_1d_dpfgse.ex
OBFRQ	391.78 MHz
OBSET	8.77 KHz
OBFIN	3.65 Hz
POINT	13107
FREQU	5882.26 Hz
SCANS	32
ACQTM	2.2282 sec
PD	7.0000 sec
PW1	10.10 usec
IRNUC	1H
CTEMP	22.2 c
SLVNT	CDCL3
EXREF	7.24 ppm
BF	0.12 Hz
RGAIN	64



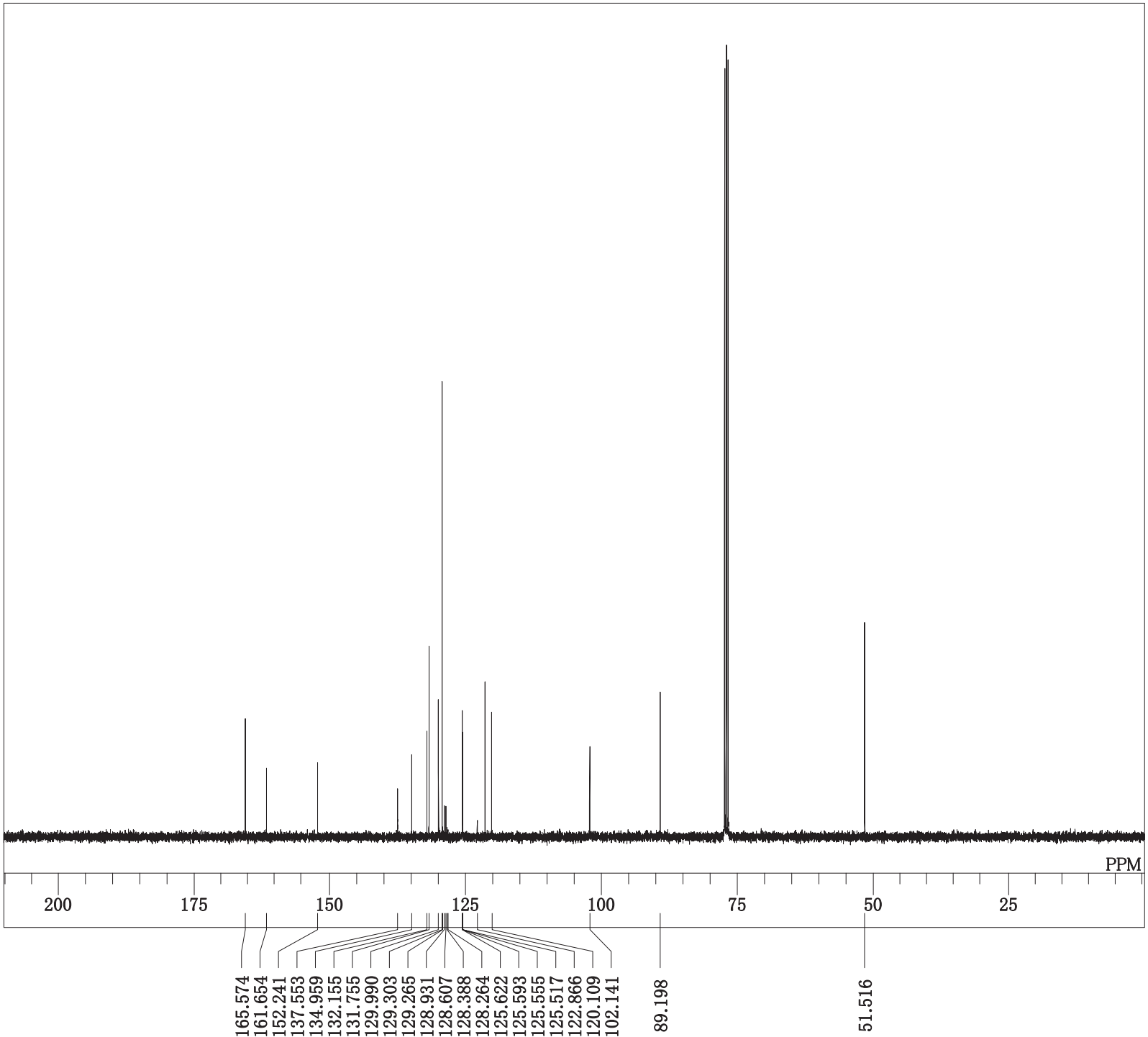
4f



DFILE	20130601 ex1489E1 p-Tol pro NOE 6-1
COMNT	DPFGSE NOE 1d
DATIM	2013-06-01 09:39:45
OBNUC	1H
EXMOD	noe_1d_dpfgse.ex
OBFRQ	391.78 MHz
OBSET	9.02 KHz
OBFIN	4.74 Hz
POINT	16384
FREQU	7352.94 Hz
SCANS	32
ACQTM	2.2282 sec
PD	7.0000 sec
PW1	10.10 usec
IRNUC	1H
CTEMP	22.4 c
SLVNT	CDCL3
EXREF	7.24 ppm
BF	0.12 Hz
RGAIN	64

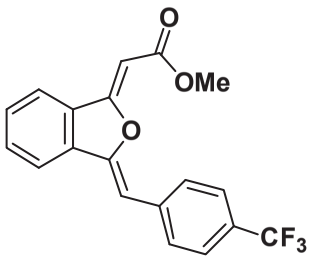


4f

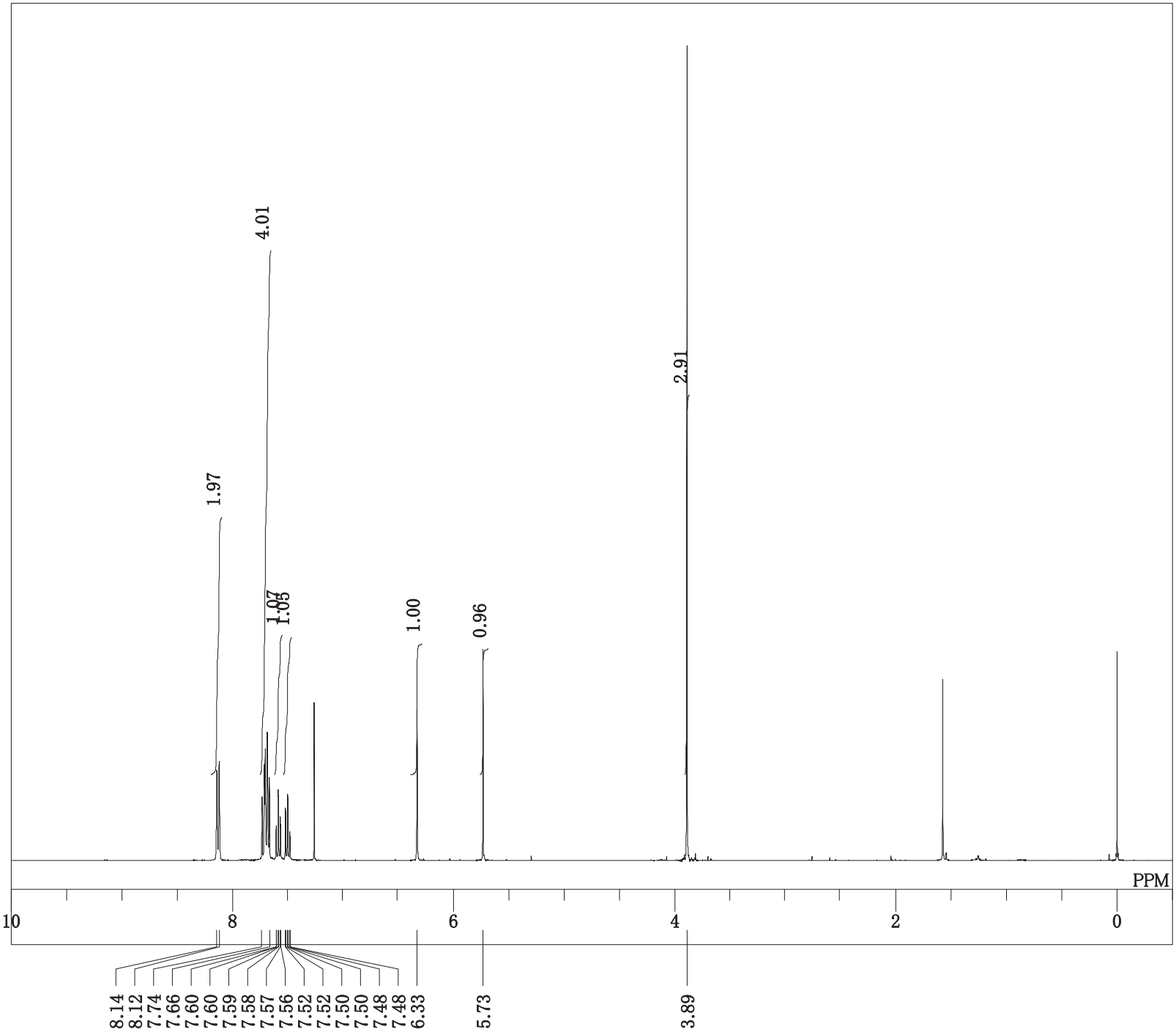


DFILE 20130530 ex1488E1 CF3 Pro 13C-1.fid
COMNT single pulse decoupled gated NOE
DATIM 2013-05-30 17:42:22
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 98.52 MHz
OBSET 4.64 KHz
OBFIN 8.74 Hz
POINT 26214
FREQU 24630.17 Hz
SCANS 1024
ACQTM 1.0643 sec
PD 2.0000 sec
PW1 2.87 usec
IRNUC 1H
CTEMP 23.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 42

13C-NMR (CDCl3) δ :
128.77 (0H, q, J = 32.6 Hz),
125.57 (0H, q, J = 3.4 Hz).

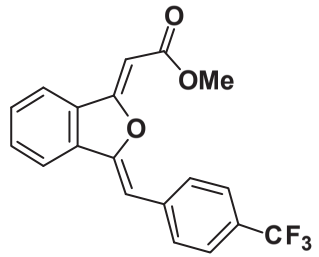


4g

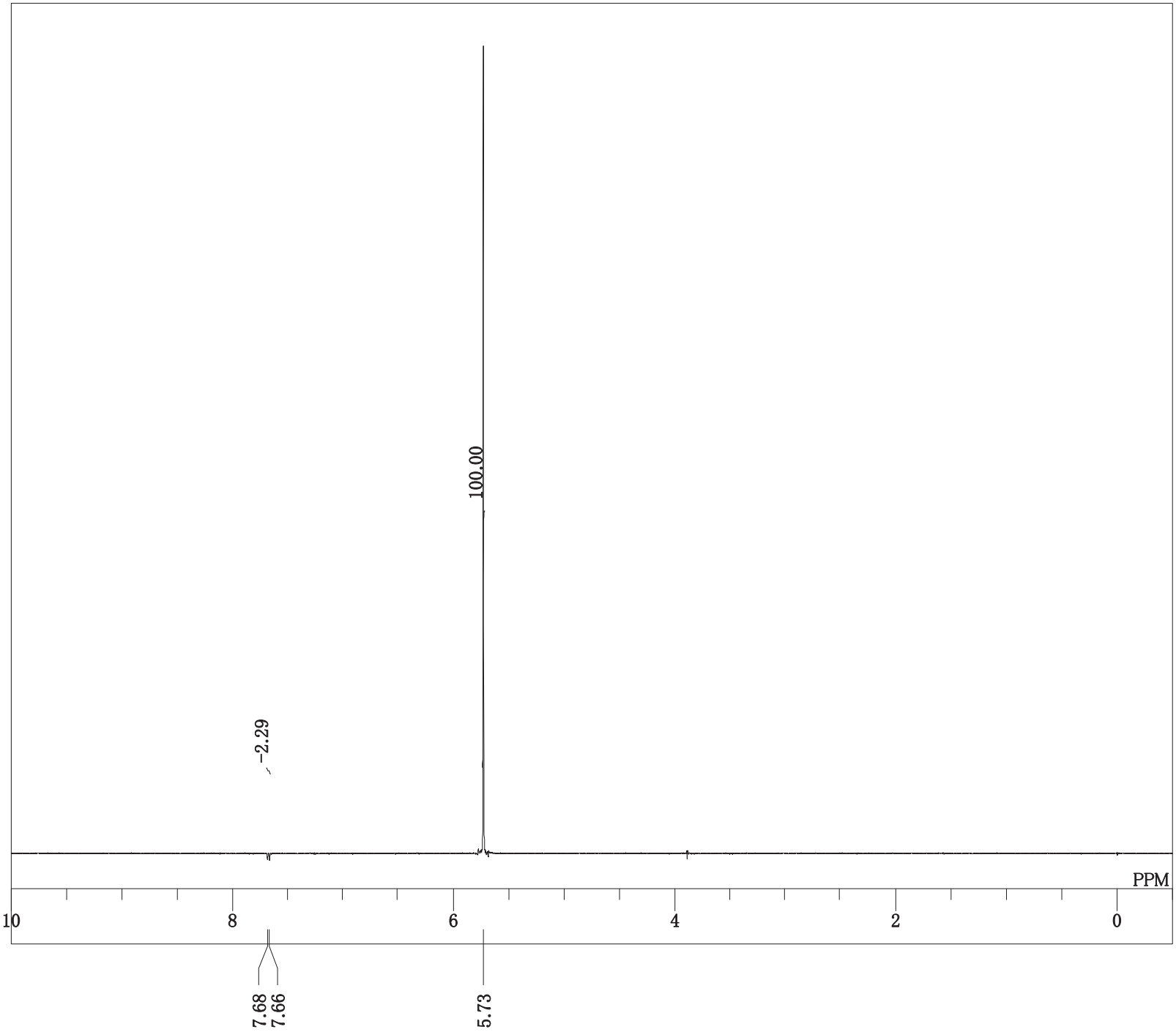


DFILE	20130530 ex1488E1 CF3 Pro 1H-1.fid
COMNT	single_pulse
DATIM	2013-05-30 16:27:57
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	26214
FREQU	5882.26 Hz
SCANS	8
ACQTM	4.4564 sec
PD	3.0000 sec
PW1	5.05 usec
IRNUC	1H
CTEMP	23.2 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	46

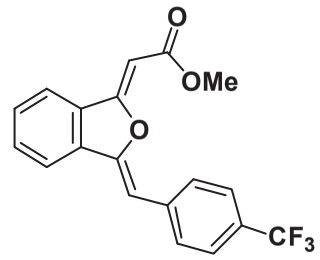
1H-NMR (CDCl3) δ :
8.13 (0H, d, J = 8.1 Hz),
7.58 (0H, td, J = 7.5, 0.9 Hz),
7.50 (0H, td, J = 7.5, 0.9 Hz).



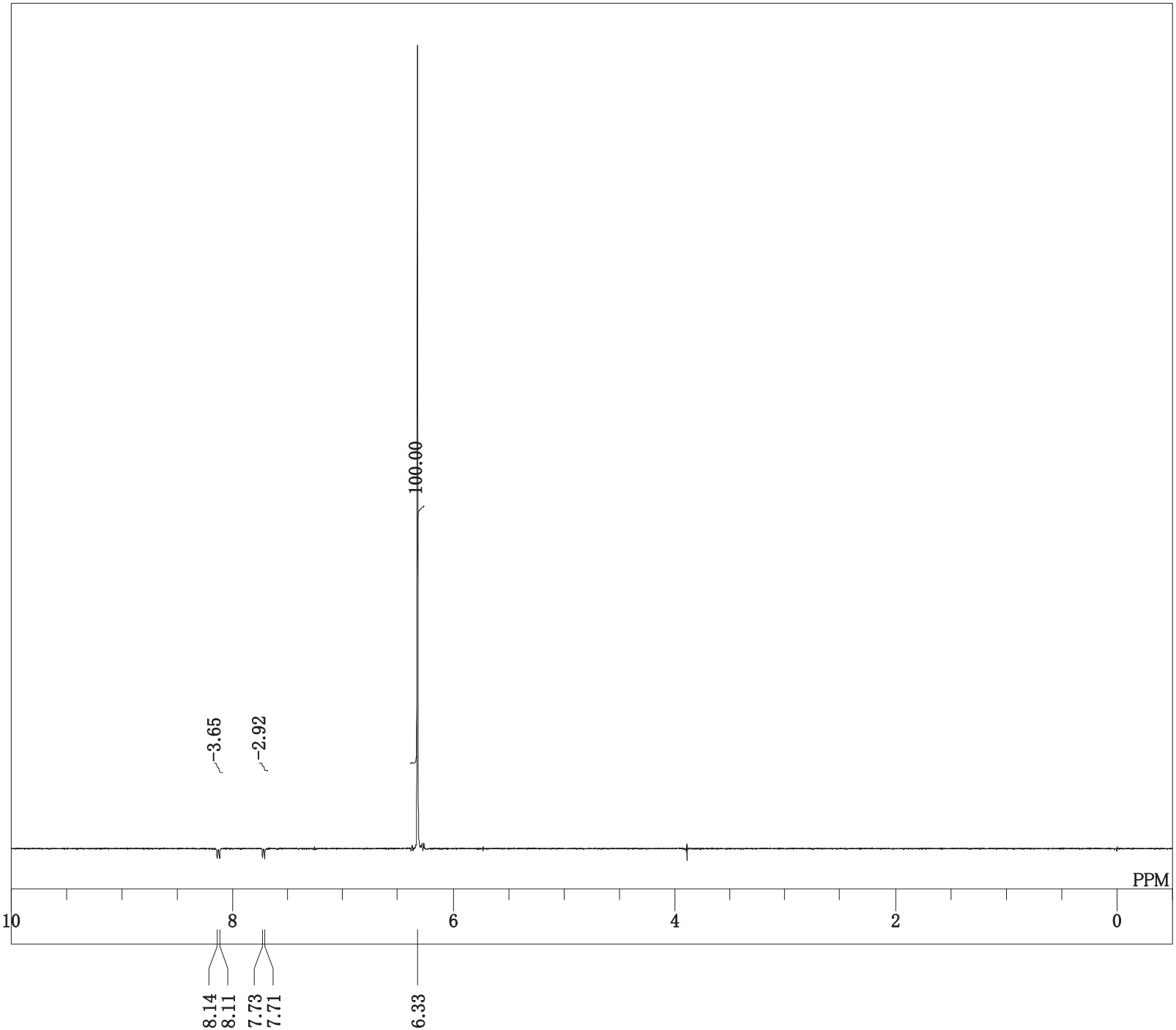
4g



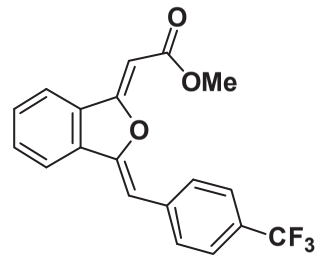
DFILE	20130530 ex1488E1 CF3 Pro NOE low-
COMNT	DPFGSE NOE 1d
DATIM	2013-05-30 16:33:53
OBNUC	1H
EXMOD	noe_1d_dpfgse.ex
OBFRQ	391.78 MHz
OBSET	8.79 KHz
OBFIN	9.00 Hz
POINT	13107
FREQU	5882.26 Hz
SCANS	16
ACQTM	2.2282 sec
PD	7.0000 sec
PW1	10.10 usec
IRNUC	1H
CTEMP	23.3 c
SLVNT	CDCL3
EXREF	7.24 ppm
BF	0.12 Hz
RGAIN	64



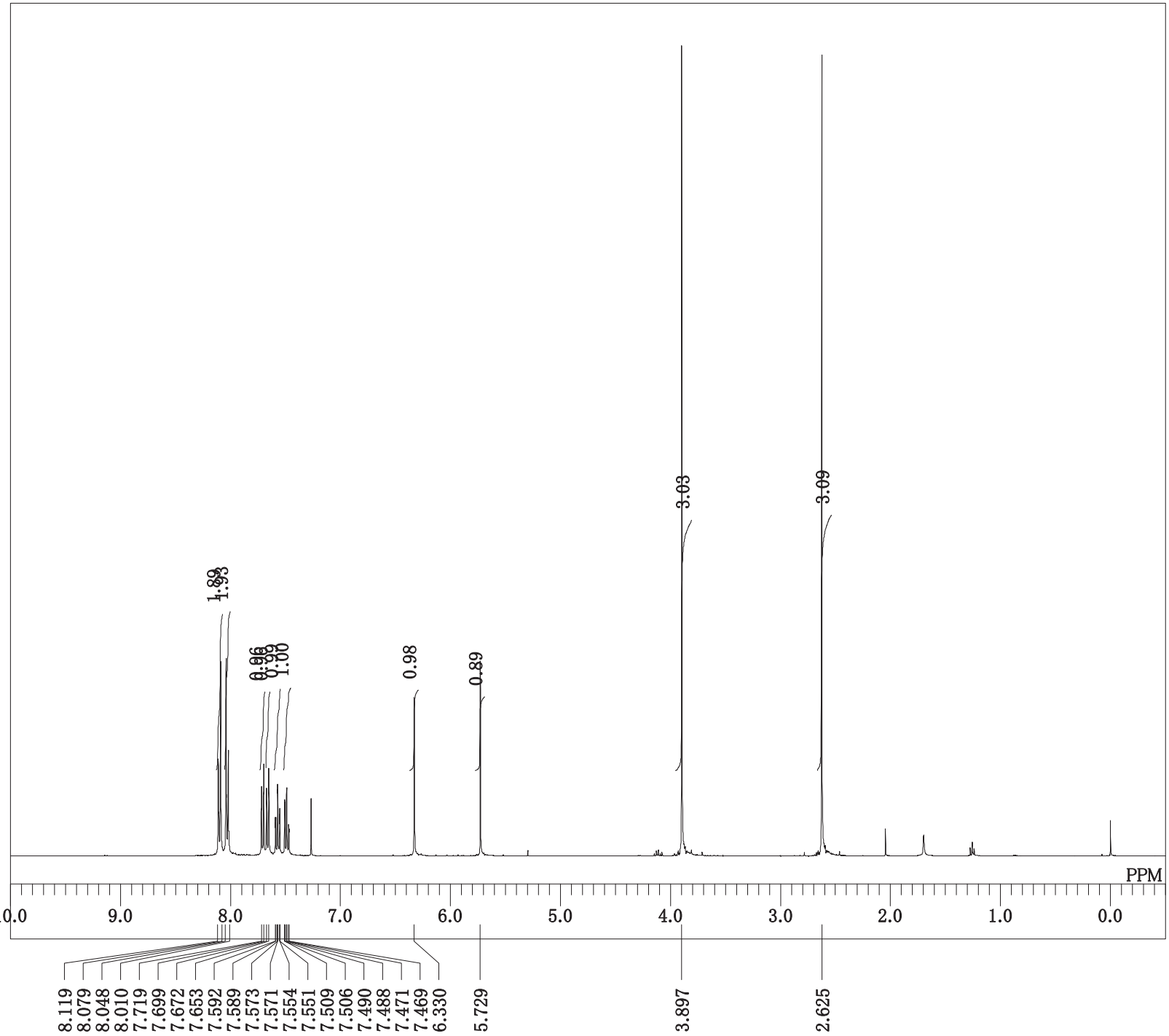
4g



DFILE	20130530 ex1488E1 CF3 Pro NOE high
COMNT	DPFGSE NOE 1d
DATIM	2013-05-30 16:39:30
OBNUC	1H
EXMOD	noe_1d_dpfgse.ex
OBFRQ	391.78 MHz
OBSET	9.03 KHz
OBFIN	2.58 Hz
POINT	13107
FREQU	5882.26 Hz
SCANS	16
ACQTM	2.2282 sec
PD	7.0000 sec
PW1	10.10 usec
IRNUC	1H
CTEMP	23.3 c
SLVNT	CDCL3
EXREF	7.24 ppm
BF	0.12 Hz
RGAIN	66

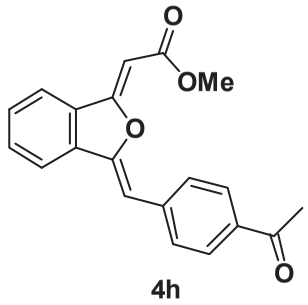


4g

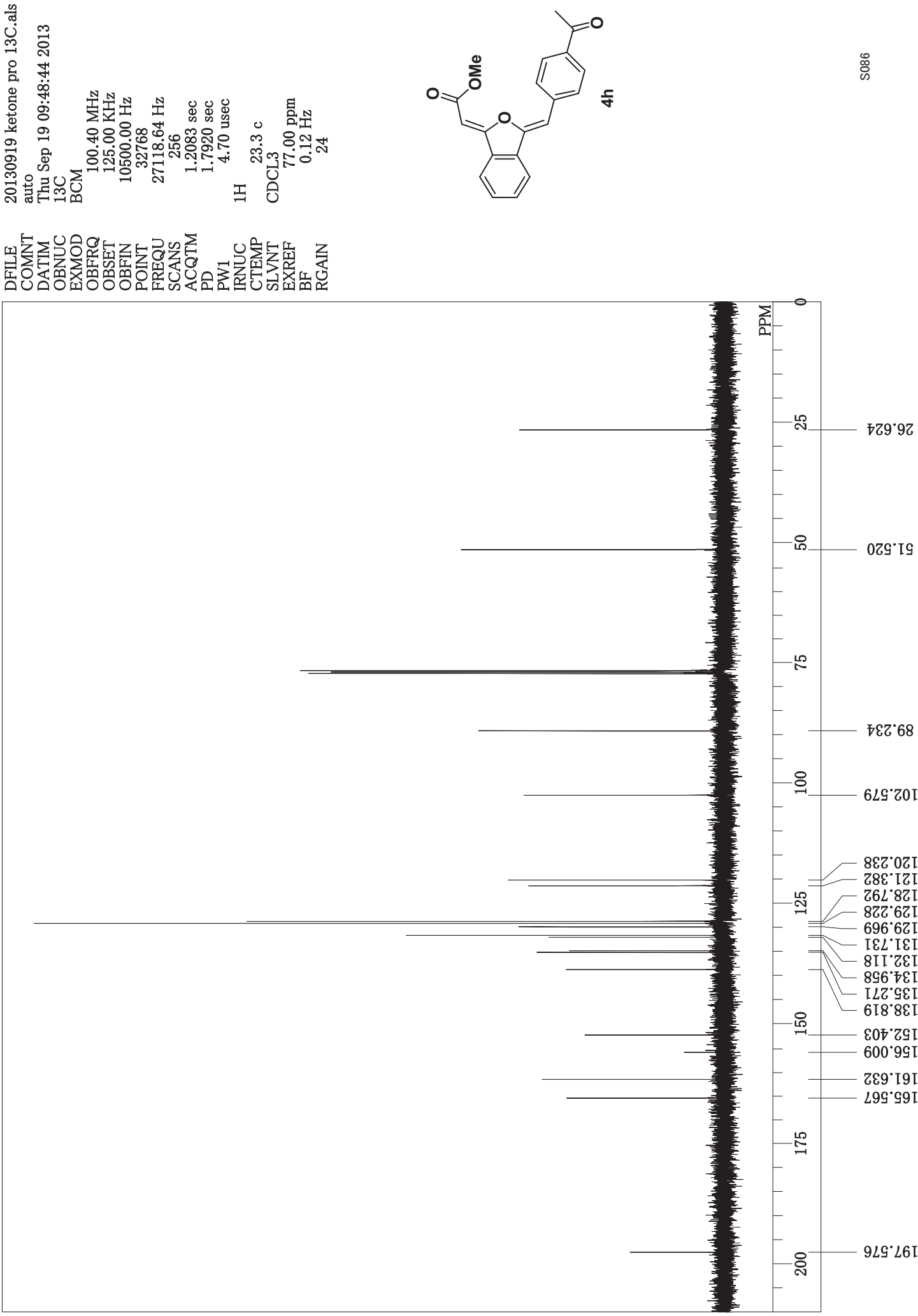


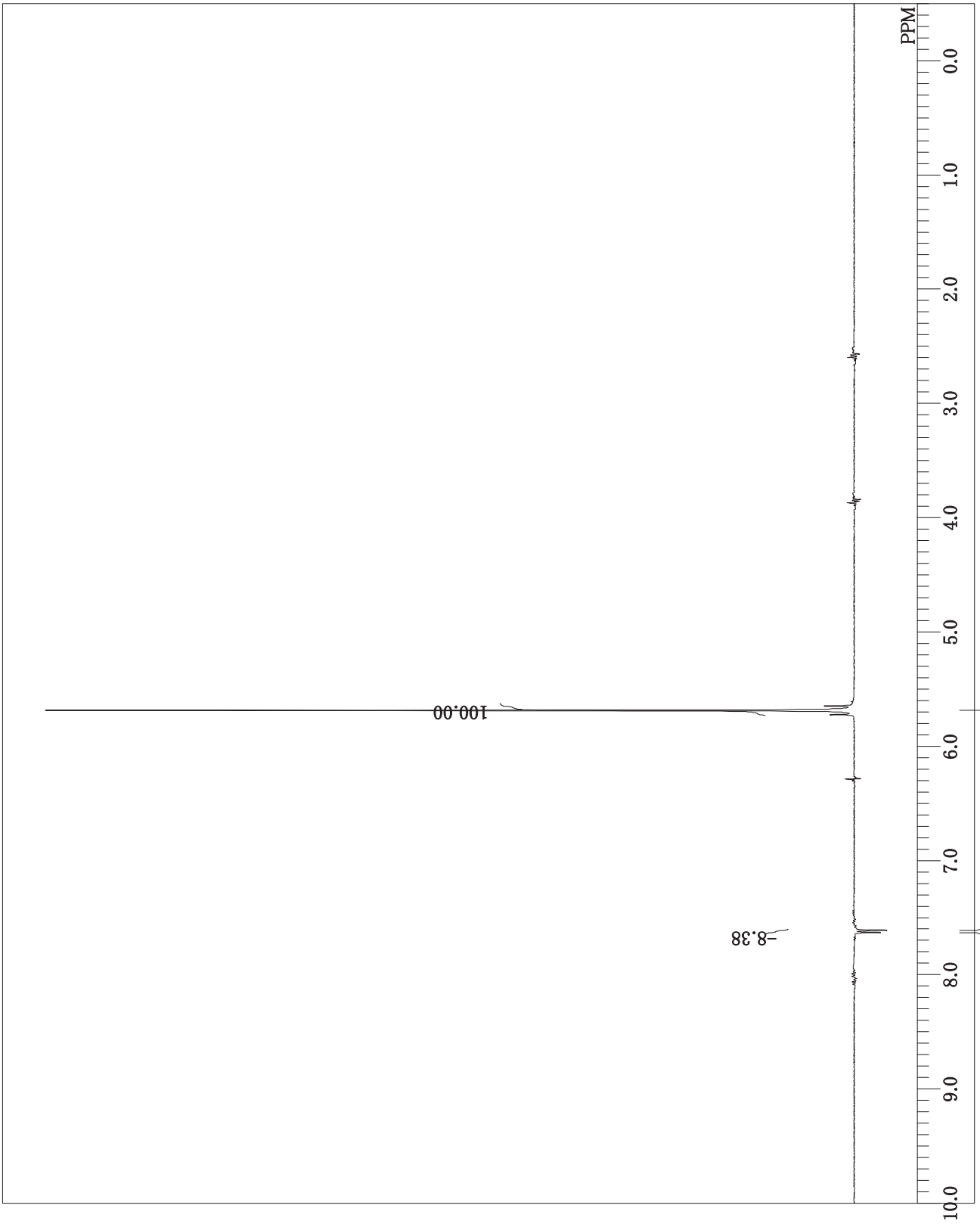
DFILE	20130919 ketone pro 1H.als
COMNT	auto
DATIM	Thu Sep 19 09:34:37 2013
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.00 Hz
POINT	16384
FREQU	7992.01 Hz
SCANS	8
ACQTM	2.0500 sec
PD	2.0000 sec
PW1	6.60 usec
IRNUC	1H
CTEMP	24.5 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	16

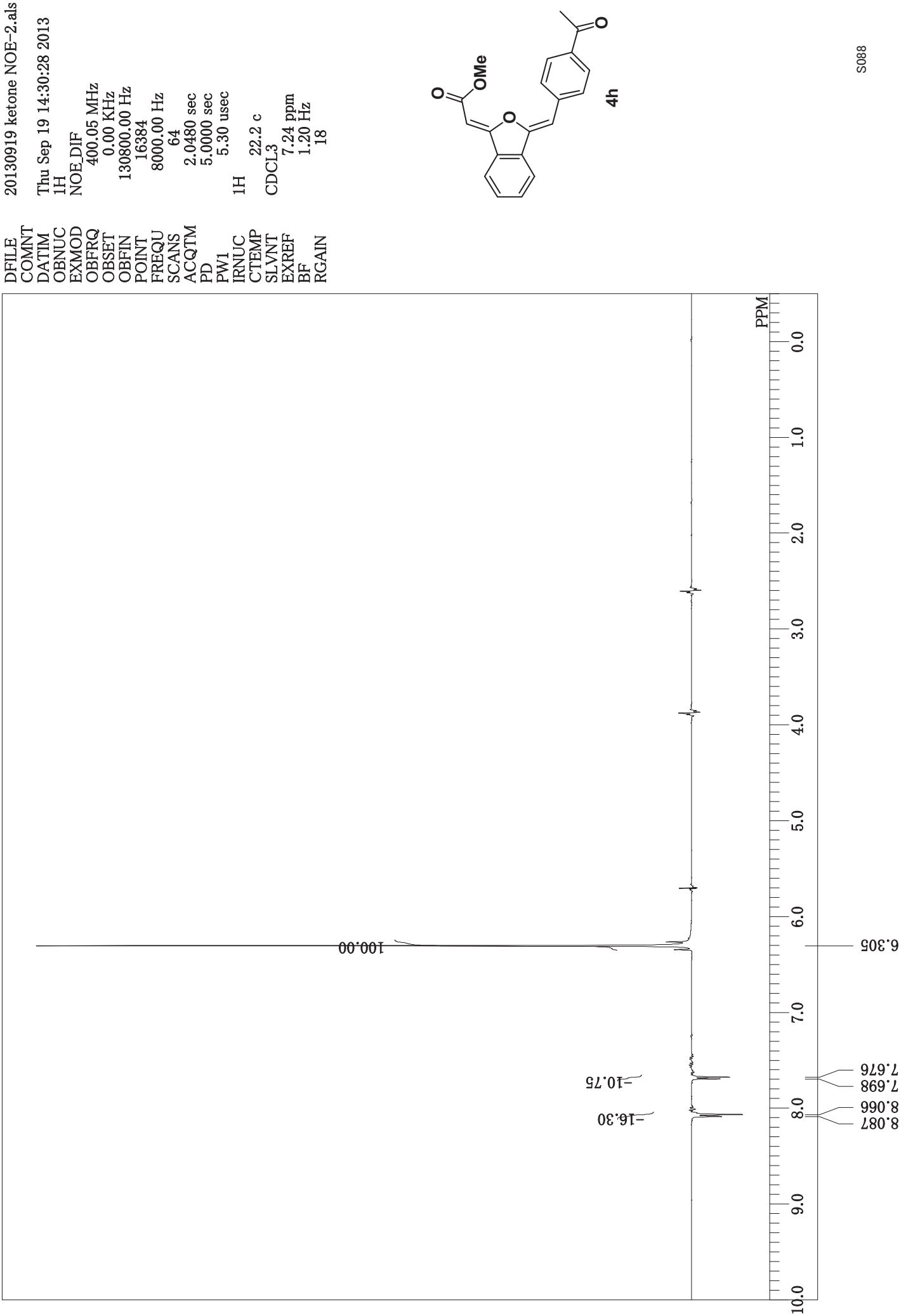
1H-NMR (CDCl3) δ :
7.57 (1H, td, J = 7.6, 1.0 Hz),
7.49 (1H, td, J = 7.6, 1.0 Hz).

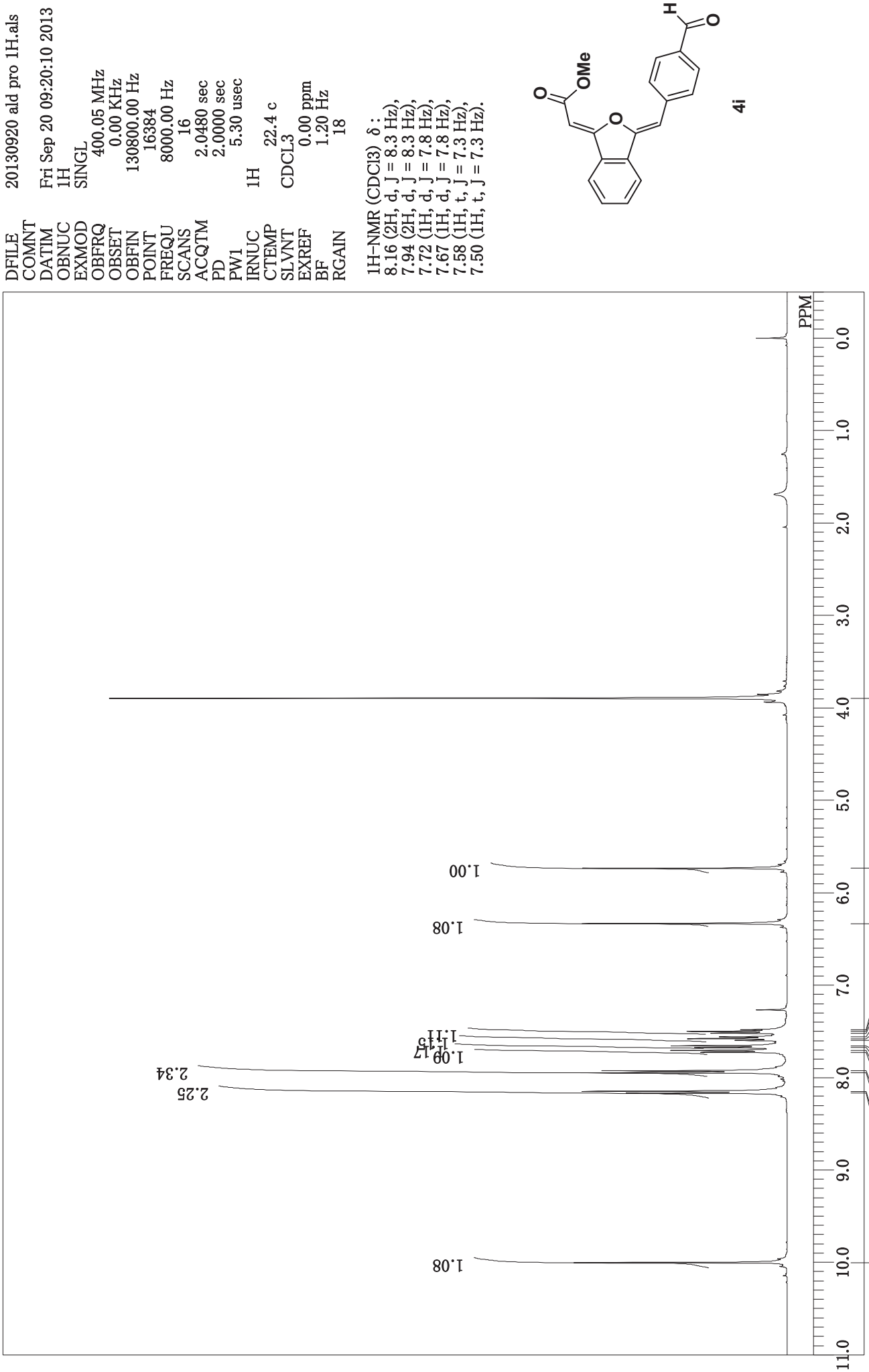


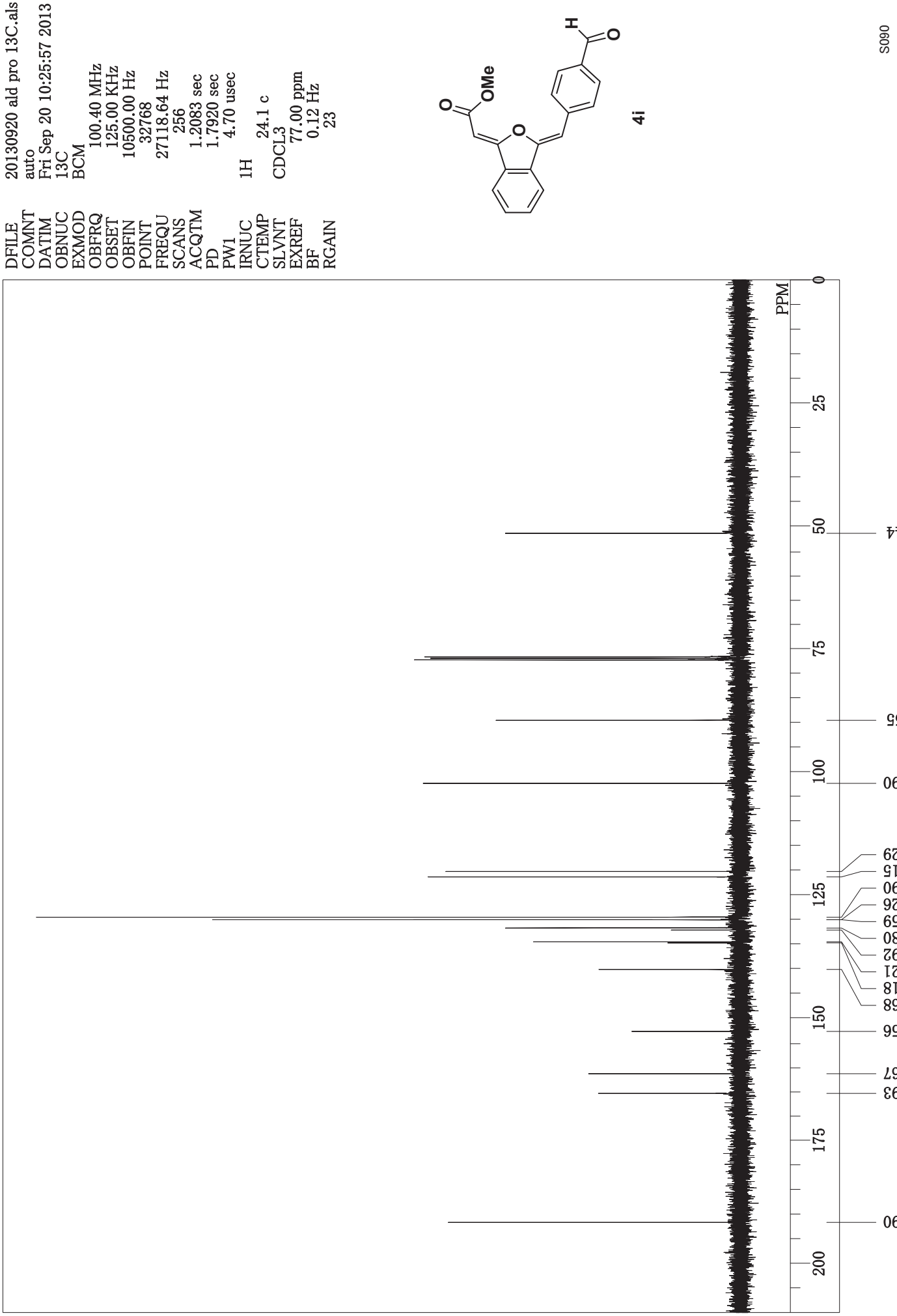
4h

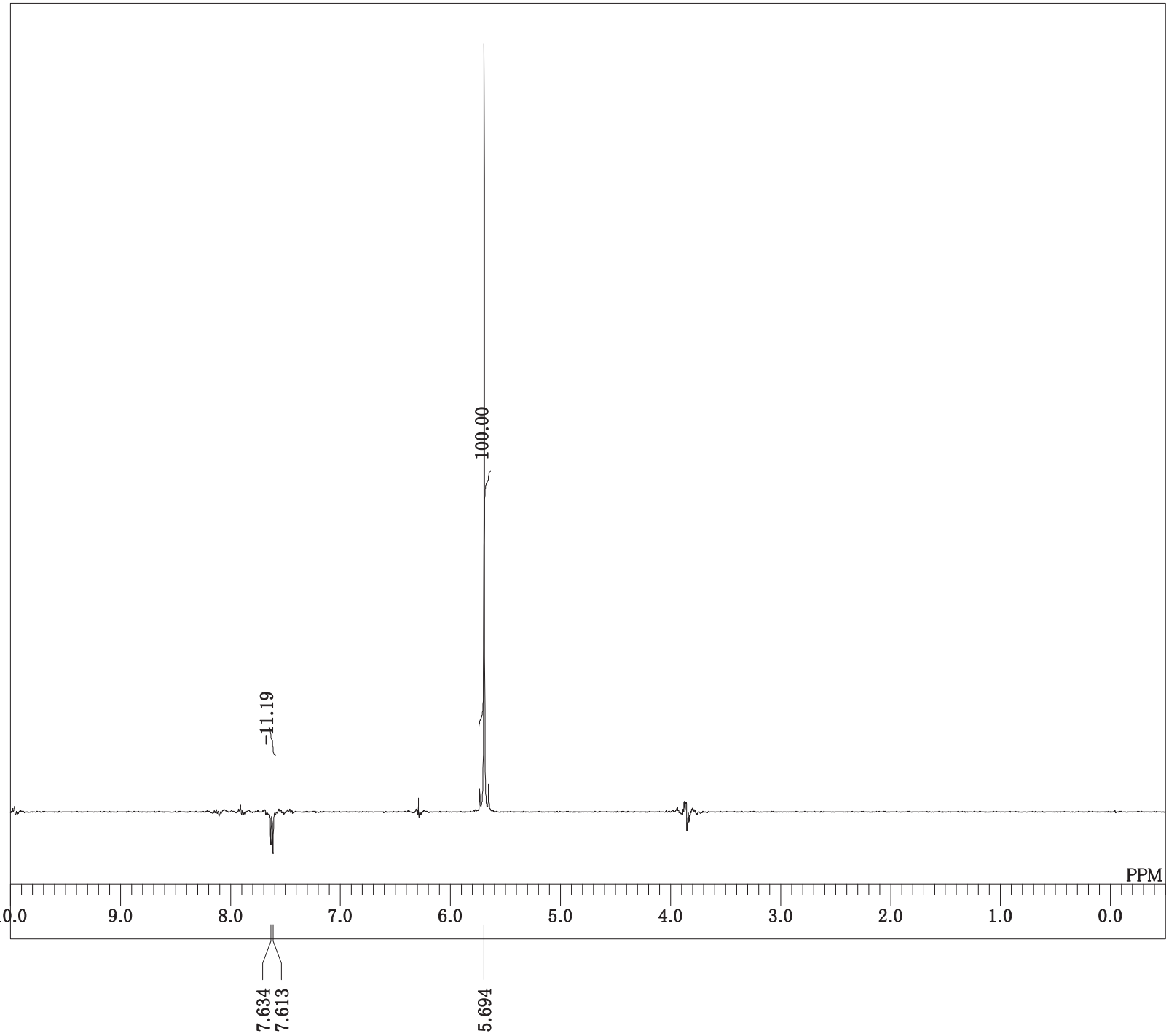

20130919 ketone pro 13C.als
auto
Thu Sep 19 09:48:44 2013
13C
BCM
100.40 MHz
125.00 KHz
10500.00 Hz
32768
27118.64 Hz
256
1.2083 sec
1.7920 sec
4.70 usec
1H
23.3 c
CDCL3
77.00 ppm
0.12 Hz
24
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
O
OMe
O
O
4h
S086
PPM
0
25
50
75
100
125
150
175
200
26.624
51.520
89.234
102.579
120.238
121.382
128.792
129.228
129.969
131.731
132.118
134.958
135.271
138.819
152.403
156.009
161.632
165.567
197.576


20130919 ketone NOE-1.als
Thu Sep 19 15:07:14 2013
1H
NOE_DIF
400.05 MHz
0.00 KHz
130800.00 Hz
16384
8000.00 Hz
64
2.0480 sec
5.0000 sec
5.30 usec
1H
22.2 c
CDCL3
7.24 ppm
1.20 Hz
14
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
O
OMe
O
O
4h
PPM
0.0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
10.0
100.00
8.38
5.688
7.632
7.611

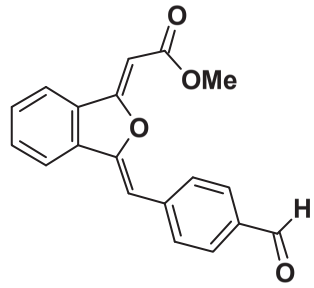

20130919 ketone NOE-2.als
Thu Sep 19 14:30:28 2013
1H
NOE_DIF
400.05 MHz
0.00 KHz
130800.00 Hz
16384
8000.00 Hz
64
2.0480 sec
5.0000 sec
5.30 usec
1H
22.2 c
CDCL3
7.24 ppm
1.20 Hz
18
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
O
OMe
O
O
4h
PPM
0.0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
10.0
100.00
16.30
10.75
6.305
7.676
7.698
8.066
8.087


DFILE 20130920 ald pro 1H.als
COMNT
DATIM Fri Sep 20 09:20:10 2013
OBNUC 1H
EXMOD SINGL
OBFRQ 400.05 MHz
OBSET 0.00 KHz
OBFIN 130800.00 Hz
POINT 16384
FREQU 8000.00 Hz
SCANS 16
ACQTM 2.0480 sec
PD 2.0000 sec
PW1 5.30 usec
IRNUC 1H
CTEMP 22.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.20 Hz
RGAIN 18
1H-NMR (CDCl3) δ :
8.16 (2H, d, J = 8.3 Hz),
7.94 (2H, d, J = 8.3 Hz),
7.72 (1H, d, J = 7.8 Hz),
7.67 (1H, d, J = 7.8 Hz),
7.58 (1H, t, J = 7.3 Hz),
7.50 (1H, t, J = 7.3 Hz).
O
OMe
O
O
H
4i
S089
PPM
0.0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
10.0
11.0
3.896
5.736
6.334
7.482
7.500
7.519
7.560
7.580
7.597
7.656
7.676
7.707
7.726
7.927
7.948
8.148
8.169
10.003
1.00
1.08
1.09
1.17
1.13
1.11
2.34
2.25
1.08

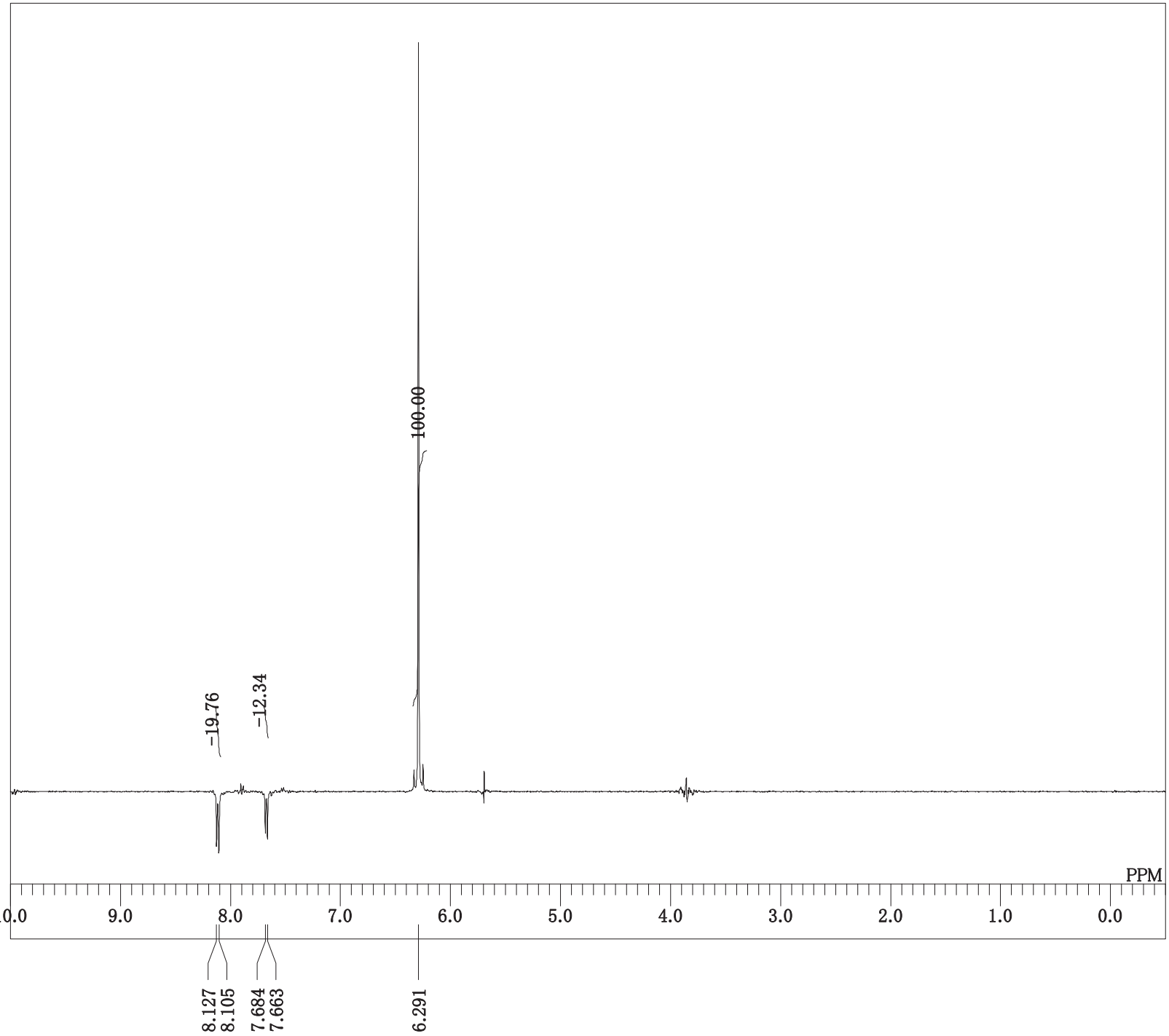

DFILE 20130920 ald pro 13C.als
COMNT auto
DATIM Fri Sep 20 10:25:57 2013
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 256
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 4.70 usec
IRNUC 1H
CTEMP 24.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 23
OMe
O
O
O
H
4i
S090
PPM
0
25
50
75
100
125
150
175
200
51.544
89.555
102.390
120.329
121.415
129.590
130.126
130.159
131.780
132.192
134.621
134.818
140.268
152.856
161.467
165.493
191.690



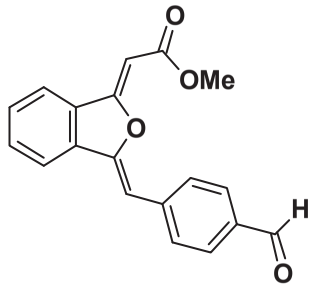
DFILE	20130920 ald pro NOE-1 PD7.als
COMNT	
DATIM	Fri Sep 20 11:41:54 2013
OBNUC	1H
EXMOD	NOE_DIF
OBFRQ	400.05 MHz
OBSET	0.00 KHz
OBFIN	130800.00 Hz
POINT	16384
FREQU	8000.00 Hz
SCANS	8
ACQTM	2.0480 sec
PD	7.0000 sec
PW1	5.30 usec
IRNUC	1H
CTEMP	22.0 c
SLVNT	CDCL3
EXREF	7.24 ppm
BF	1.20 Hz
RGAIN	14



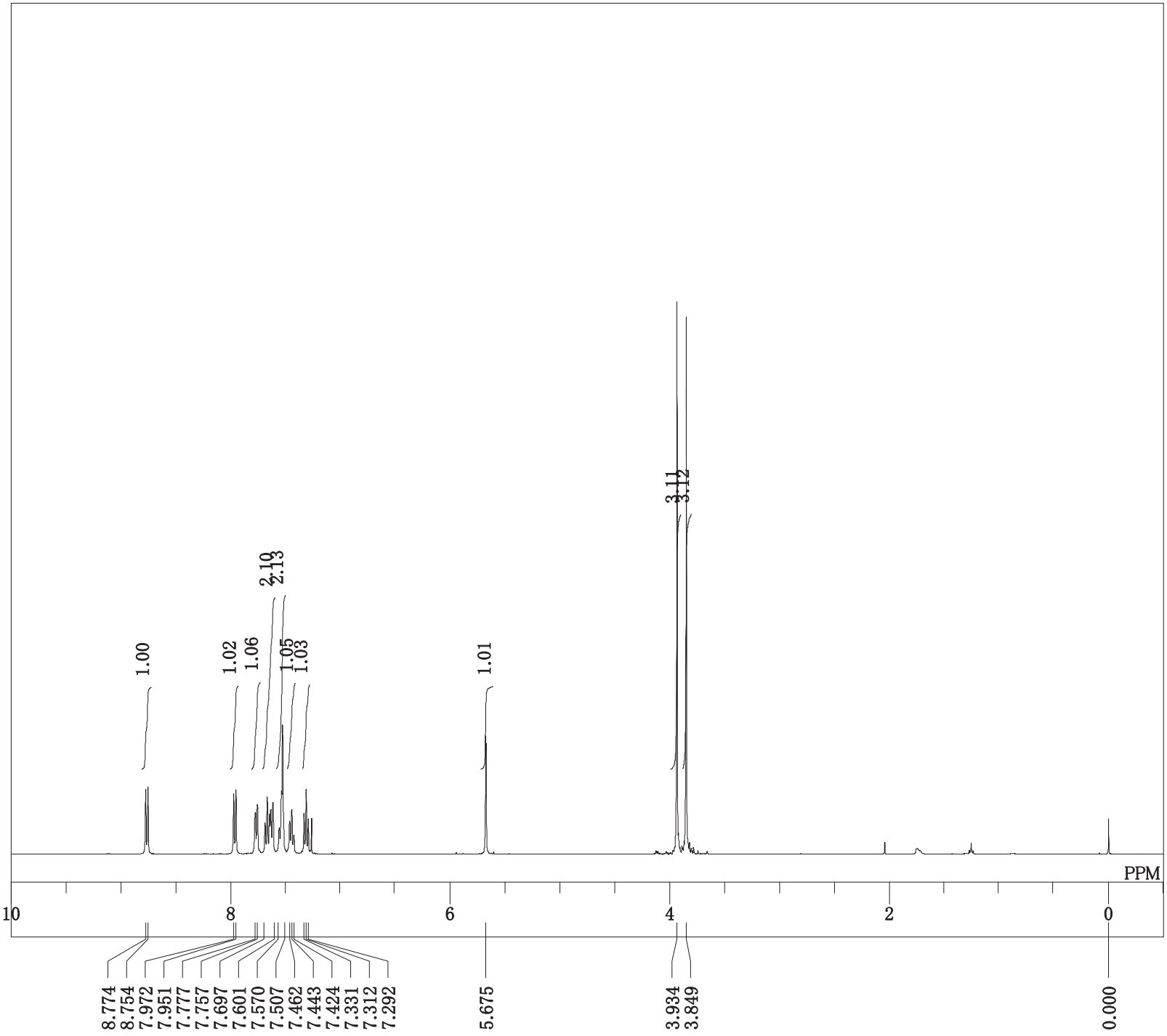
4i



DFILE	20130920 ald pro NOE-2 PD7.als
COMNT	
DATIM	Fri Sep 20 12:02:59 2013
OBNUC	1H
EXMOD	NOE_DIF
OBFRQ	400.05 MHz
OBSET	0.00 KHz
OBFIN	130800.00 Hz
POINT	16384
FREQU	8000.00 Hz
SCANS	8
ACQTM	2.0480 sec
PD	7.0000 sec
PW1	5.30 usec
IRNUC	1H
CTEMP	21.9 c
SLVNT	CDCL3
EXREF	7.24 ppm
BF	1.20 Hz
RGAIN	14

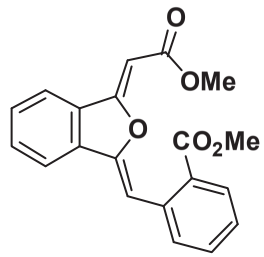


4i

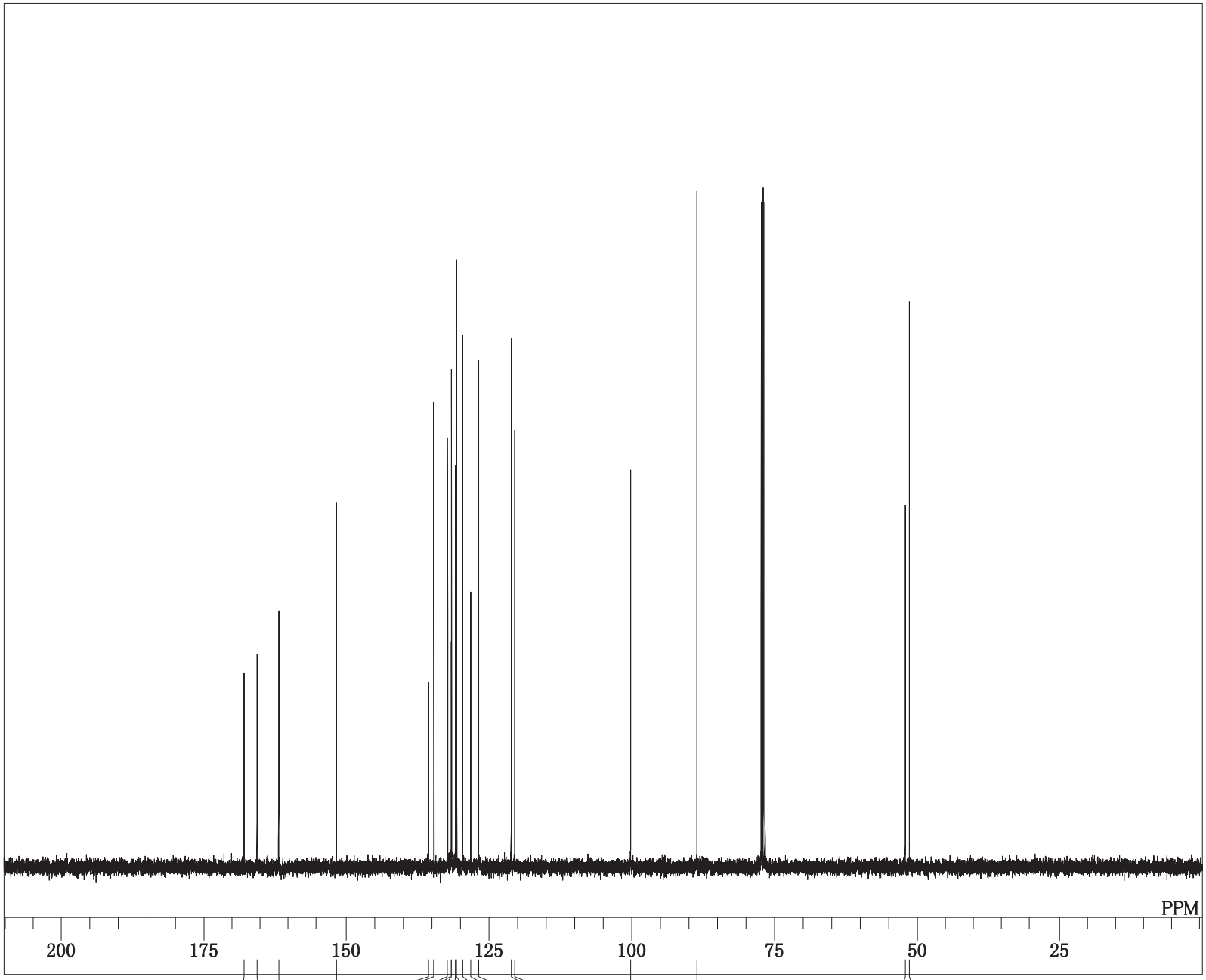


DFILE	20130603 ex1488E3 o-COOMe pro 1H-
COMNT	single_pulse
DATIM	2013-06-03 20:13:04
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	26214
FREQU	5882.26 Hz
SCANS	8
ACQTM	4.4564 sec
PD	3.0000 sec
PW1	5.05 usec
IRNUC	1H
CTEMP	22.0 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	38

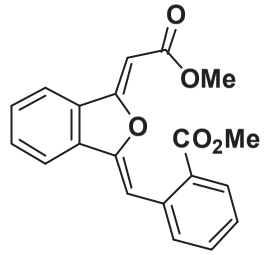
1H-NMR (CDCl3) δ :
8.76 (1H, d, J = 8.1 Hz),
7.96 (1H, d, J = 8.1 Hz),
7.77 (1H, d, J = 7.6 Hz),
7.44 (1H, t, J = 7.4 Hz),
7.31 (1H, t, J = 7.6 Hz).



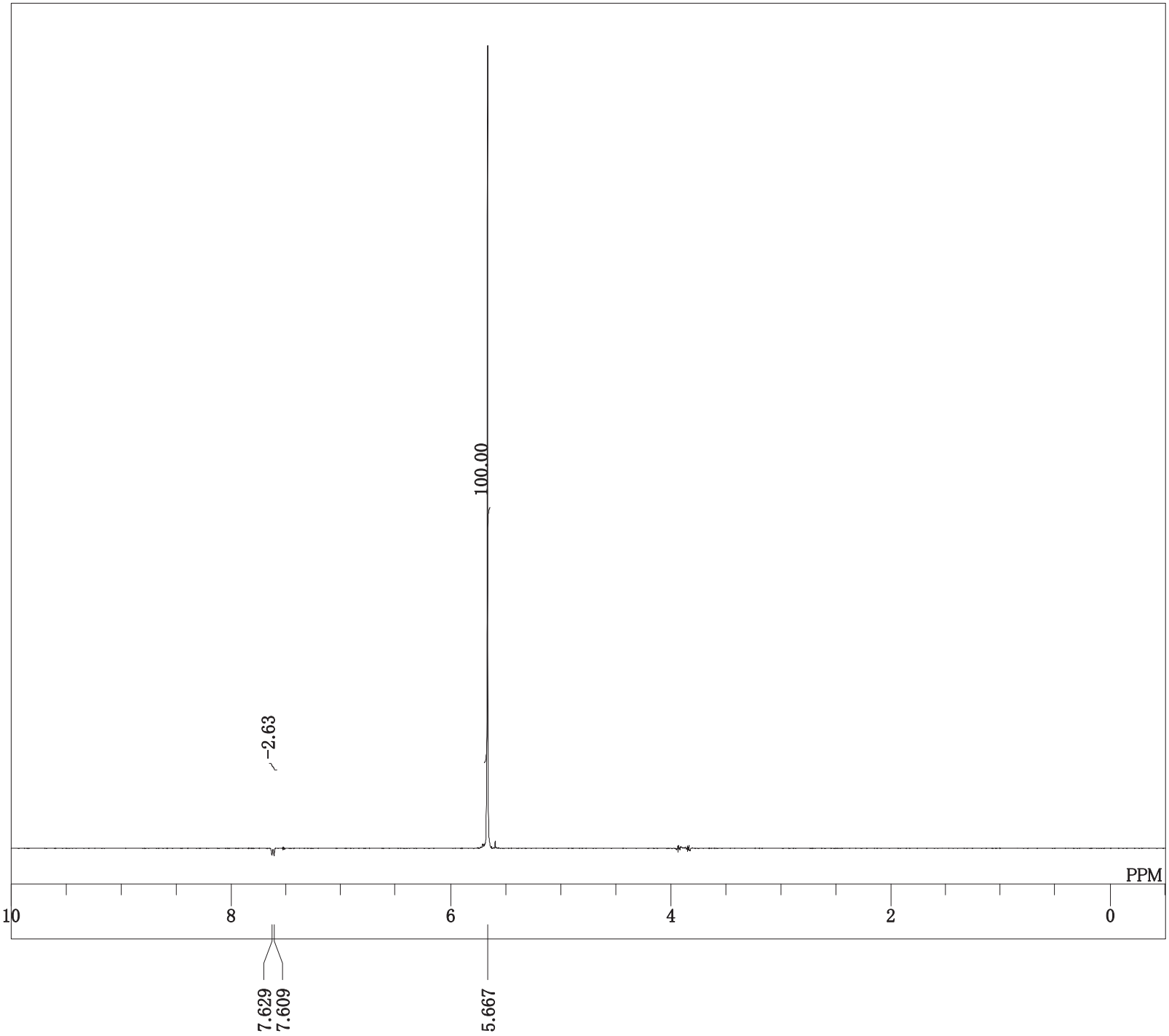
4j



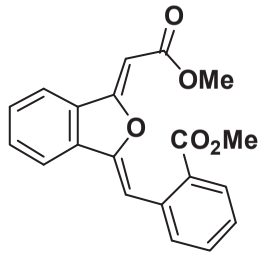
DFILE	20130603 ex1488E3 o-COOMe pro 3C
COMNT	single pulse decoupled gated NOE
DATIM	2013-06-03 20:49:22
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.17 Hz
SCANS	442
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	2.87 usec
IRNUC	1H
CTEMP	22.7 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	46



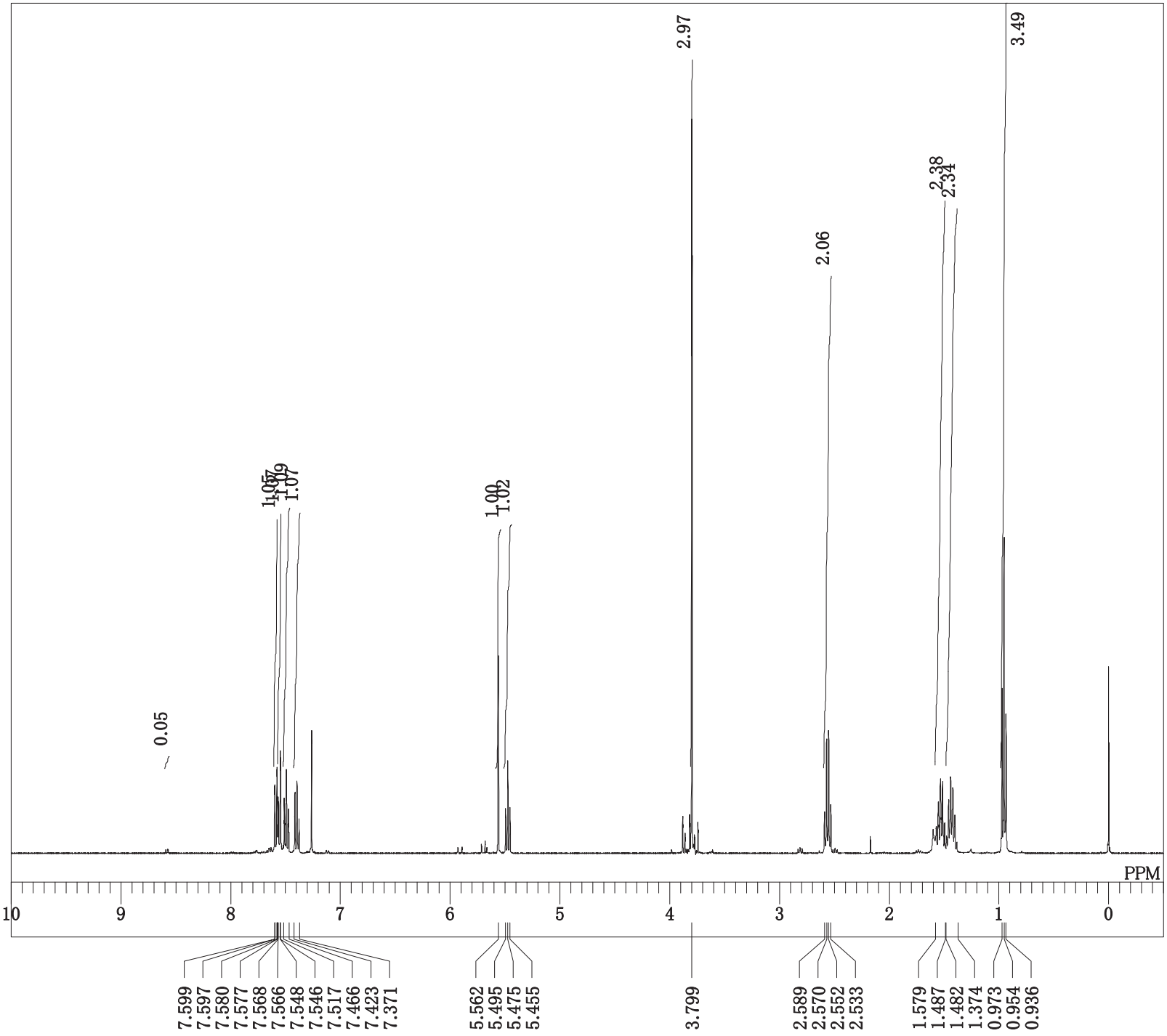
4j



DFILE	20130603 ex1488E3 o-COOMe pro NOE
COMNT	DPFGSE NOE 1d
DATIM	2013-06-03 20:18:54
OBNUC	1H
EXMOD	noe_1d_dpfgse.ex
OBFRQ	391.78 MHz
OBSET	8.77 KHz
OBFIN	4.55 Hz
POINT	13107
FREQU	5882.26 Hz
SCANS	16
ACQTM	2.2282 sec
PD	7.0000 sec
PW1	10.10 usec
IRNUC	1H
CTEMP	22.2 c
SLVNT	CDCL3
EXREF	7.24 ppm
BF	0.12 Hz
RGAIN	56

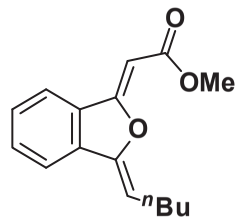


4j



DFILE	20130603 ex1488E2 nBu pro 1H-1.als
COMNT	single_pulse
DATIM	2013-06-03 11:06:03
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	26214
FREQU	5882.26 Hz
SCANS	8
ACQTM	4.4564 sec
PD	3.0000 sec
PW1	5.05 usec
IRNUC	1H
CTEMP	21.9 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	48

1H-NMR (CDCl3) δ :
7.59 (2H, dd, J = 7.7, 1.0 Hz),
7.56 (2H, dd, J = 7.9, 0.9 Hz),
5.48 (1H, t, J = 7.7 Hz),
2.56 (2H, q, J = 7.4 Hz),
0.95 (4H, t, J = 7.2 Hz).



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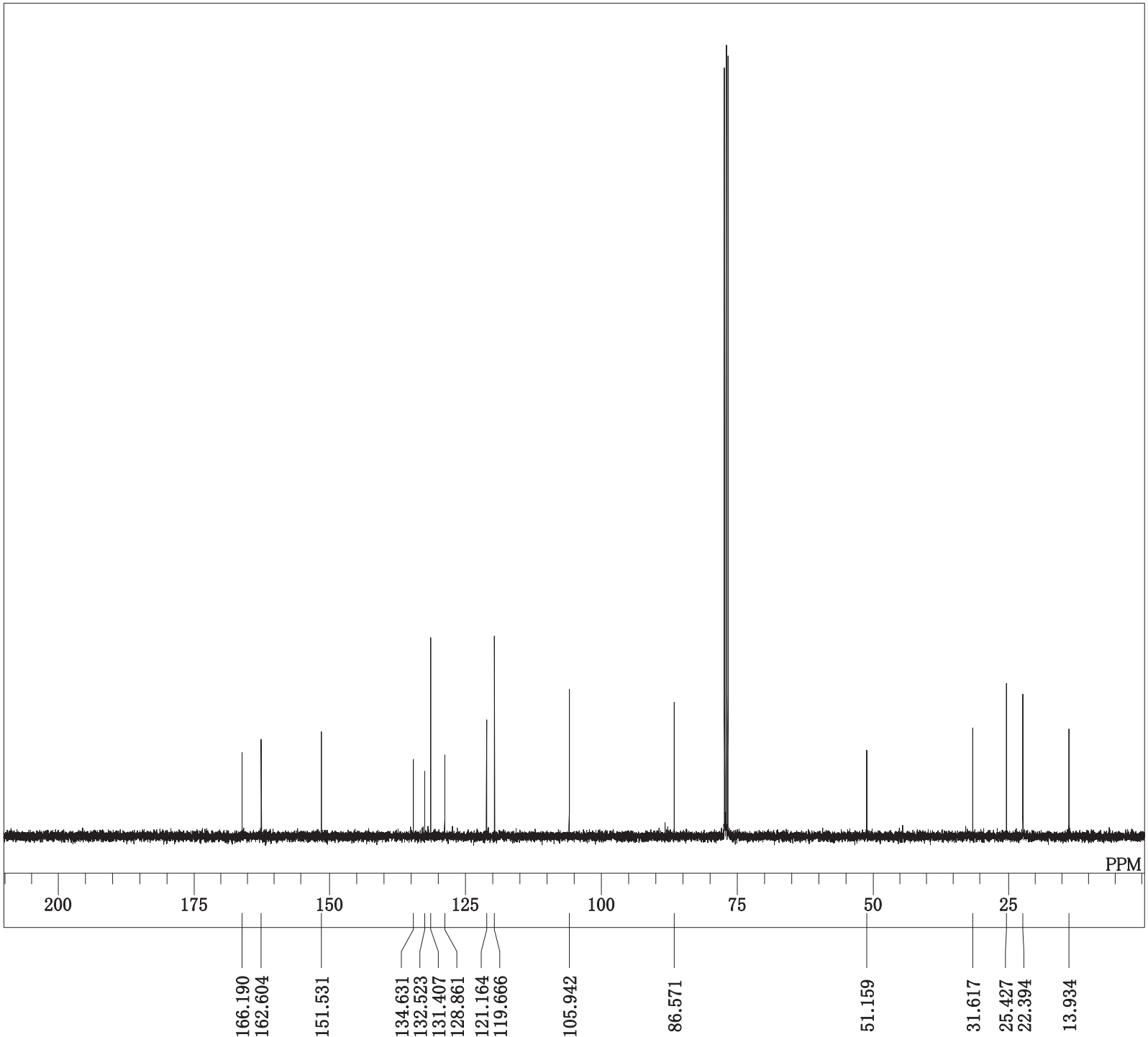
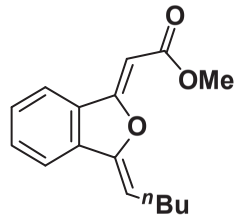
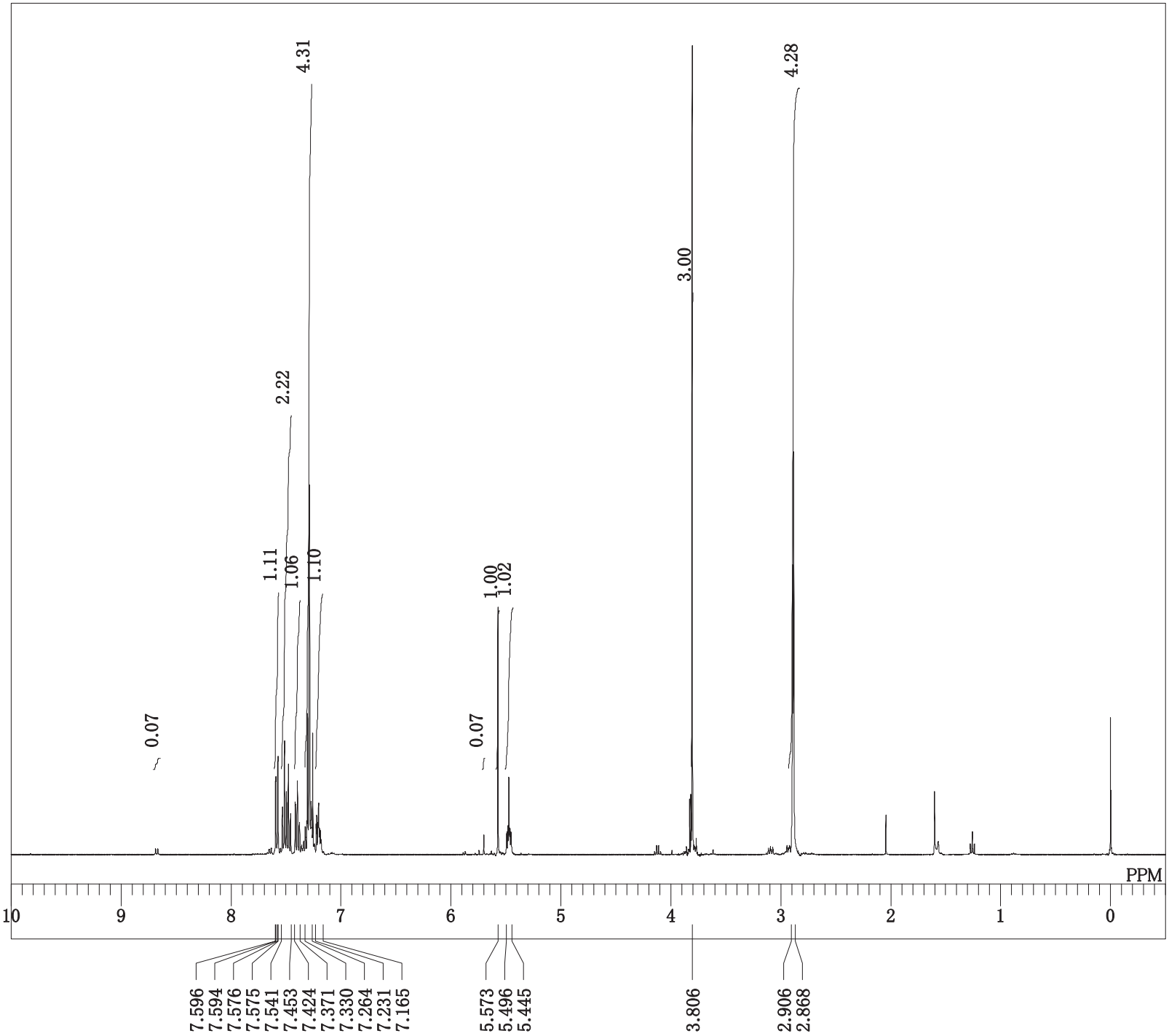


DFILE	20130603 ex1488E2 alkyne nBu pro 13C
COMNT	single pulse decoupled gated NOE
DATIM	2013-06-03 12:01:29
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.17 Hz
SCANS	1024
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	2.87 usec
IRNUC	1H
CTEMP	22.3 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	36

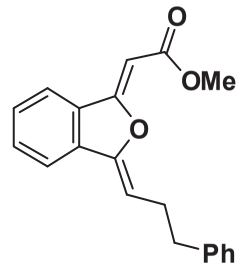


4k



DFILE	20130603 ex1489E3 CH2CH2Ph proton
COMNT	single_pulse
DATIM	2013-06-03 09:49:49
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	26214
FREQU	5882.26 Hz
SCANS	8
ACQTM	4.4564 sec
PD	3.0000 sec
PW1	5.05 usec
IRNUC	1H
CTEMP	22.0 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	44

1H-NMR (CDCl3) δ :
7.59 (1H, dd, J = 7.6, 0.7 Hz).



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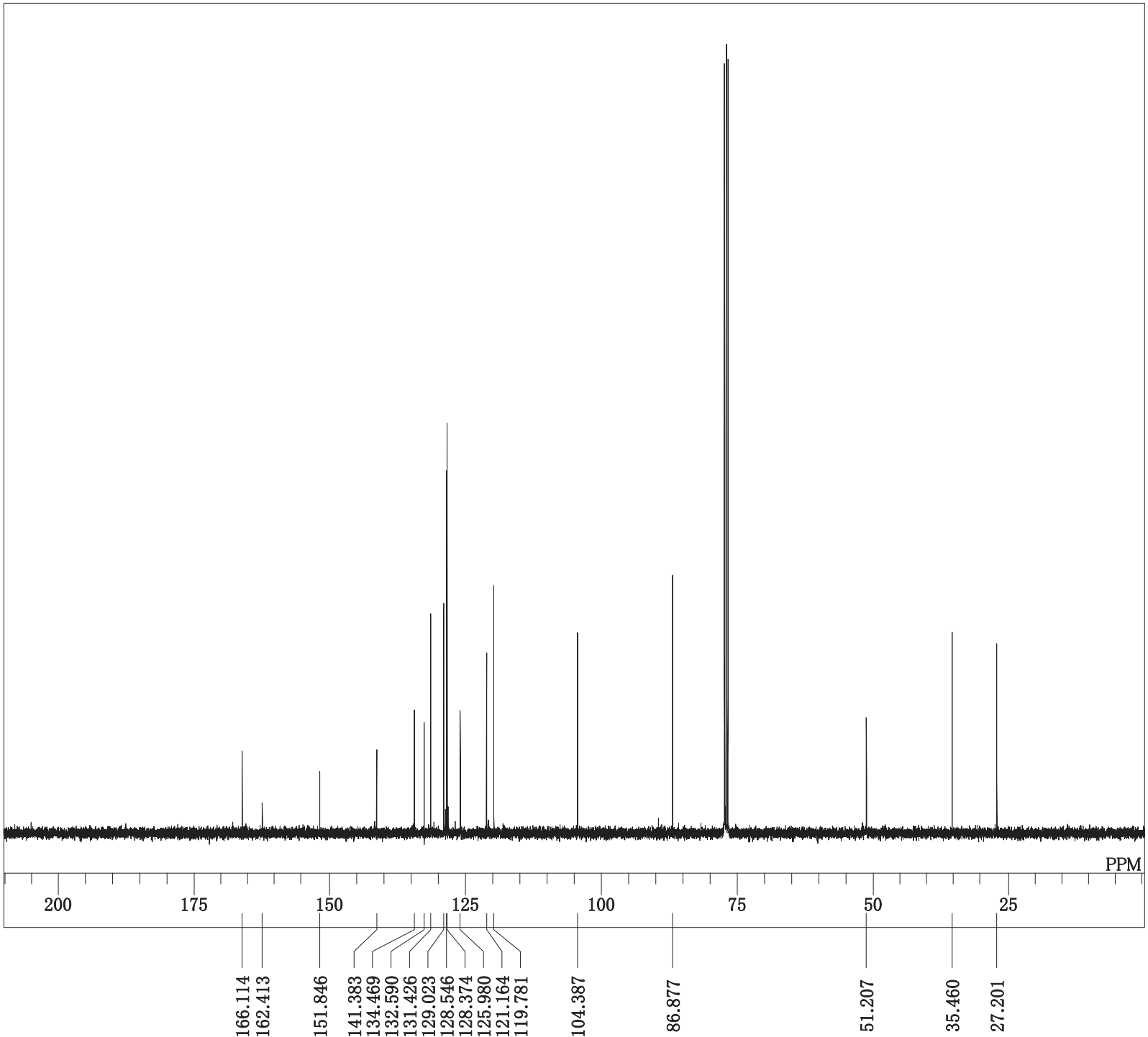
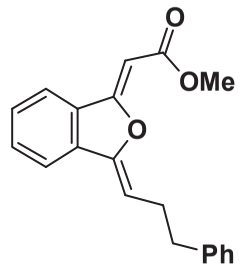


DFILE	20130603 ex1489E3 CH2CH2Ph pro 13
COMNT	single pulse decoupled gated NOE
DATIM	2013-06-03 10:45:39
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.17 Hz
SCANS	1024
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	2.87 usec
IRNUC	1H
CTEMP	22.2 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	36



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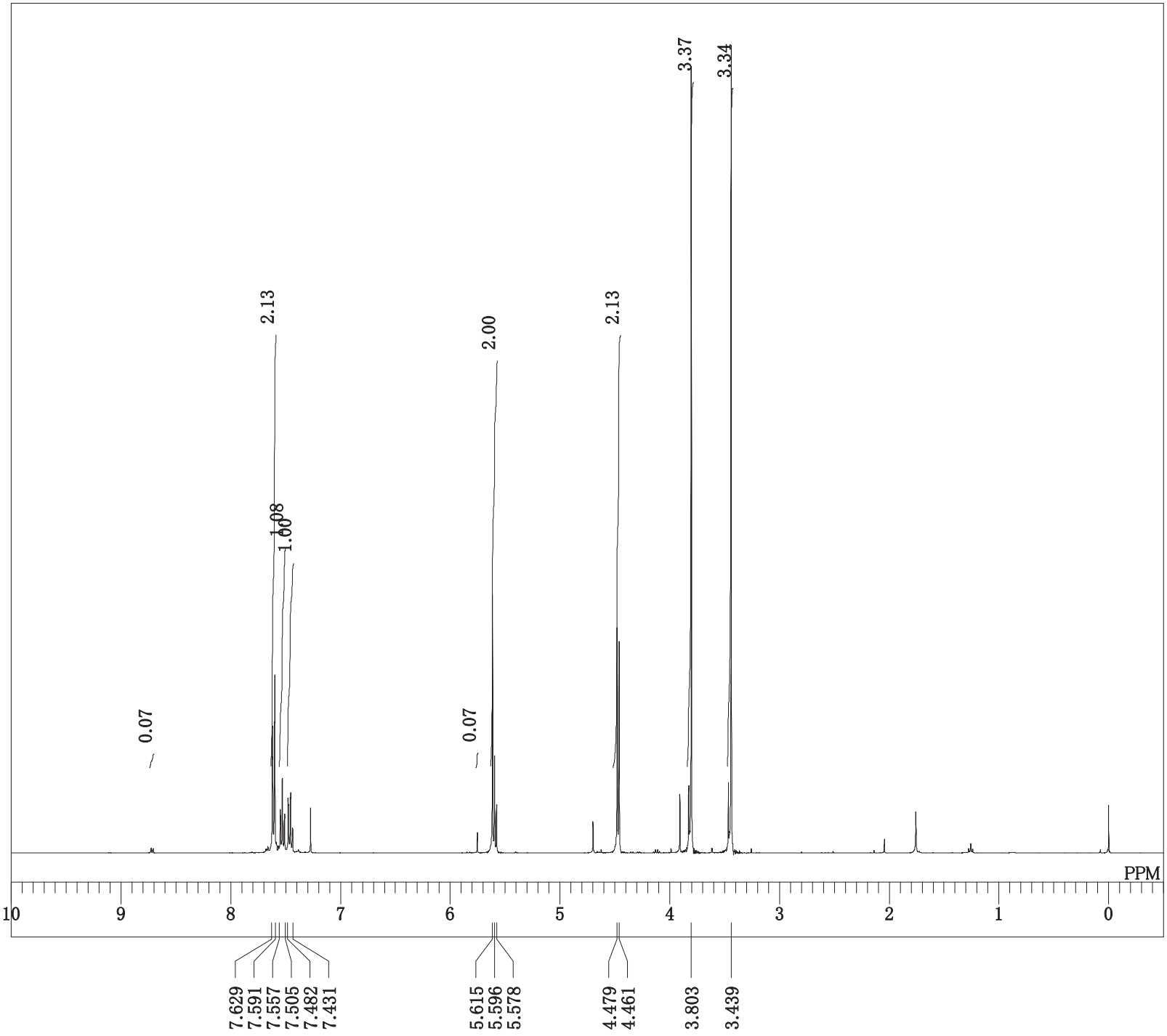
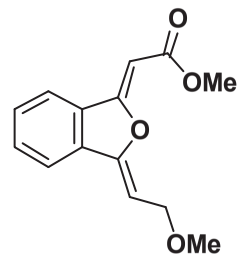


DFILE	20130604 ex1489E2 OMe pro 1H-NMR
COMNT	single_pulse
DATIM	2013-06-04 20:43:53
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	32768
FREQU	7352.94 Hz
SCANS	8
ACQTM	4.4564 sec
PD	3.0000 sec
PW1	5.05 usec
IRNUC	1H
CTEMP	22.5 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	40

1H-NMR (CDCl3) δ :
5.60 (2H, t, J = 7.2 Hz),
4.47 (2H, d, J = 7.0 Hz).



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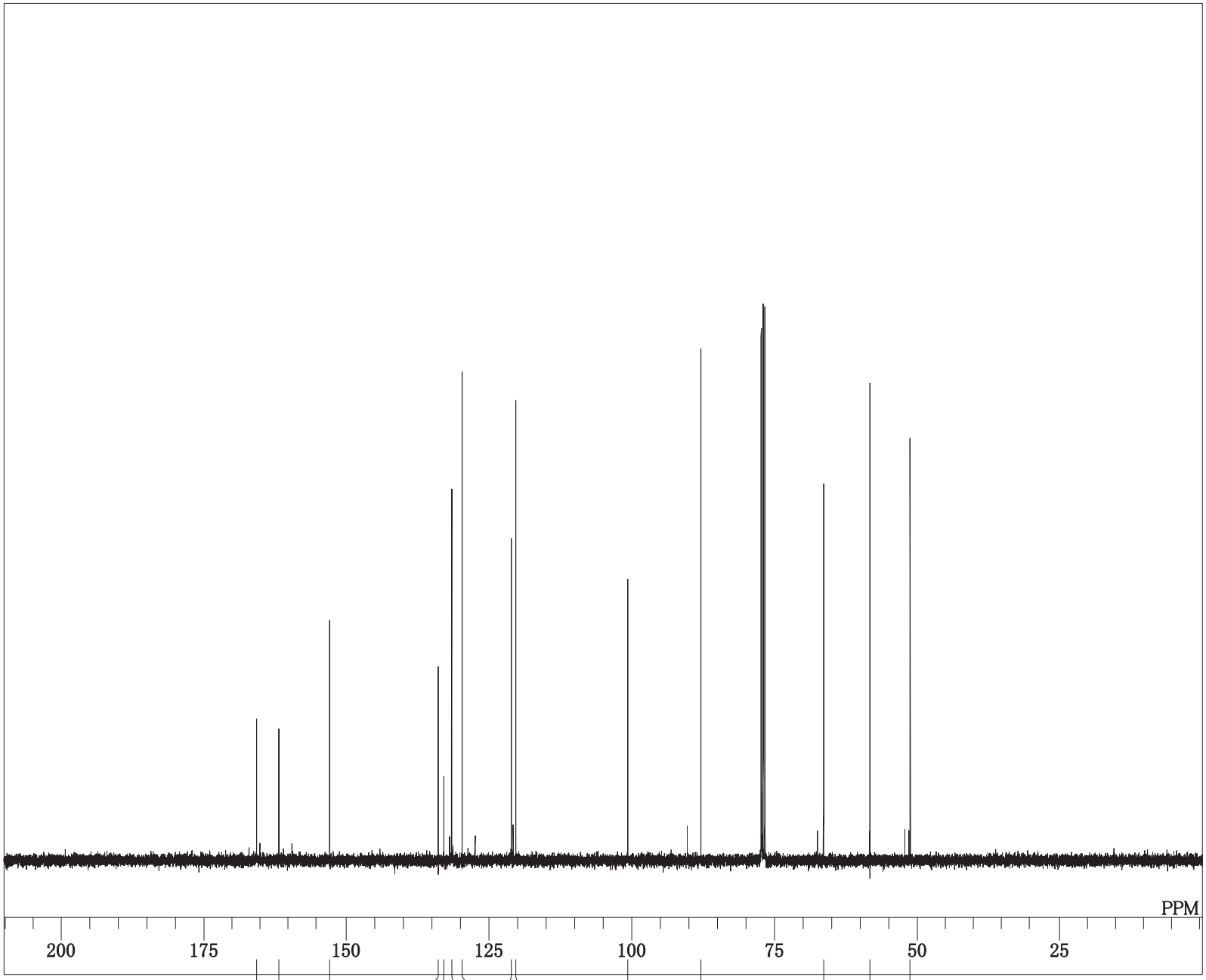
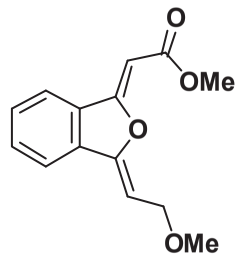
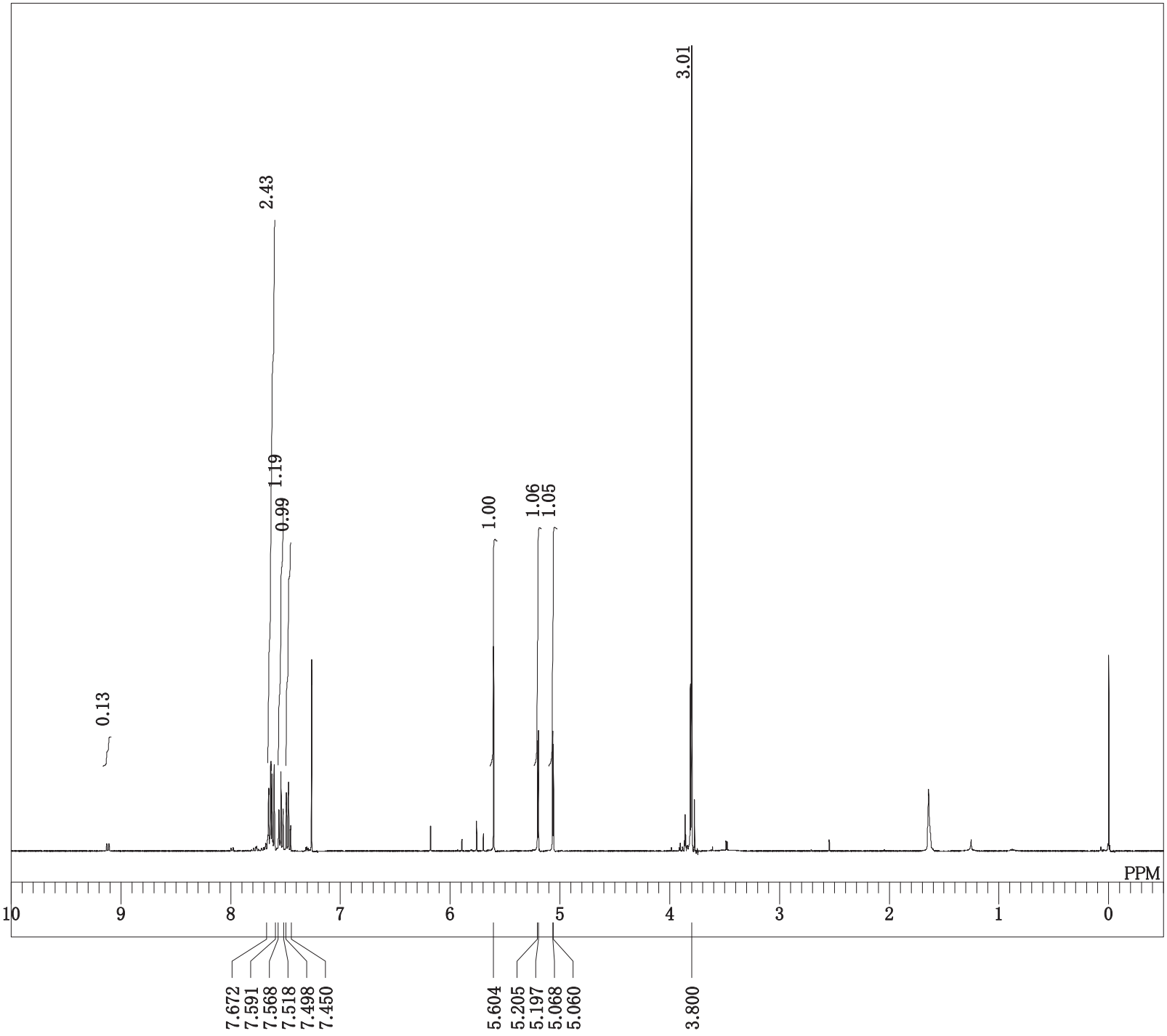


DFILE	20130604 ex1489E2 OMe pro 13C 1als
COMNT	single pulse decoupled gated NOE
DATIM	2013-06-04 21:15:24
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.17 Hz
SCANS	512
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	2.87 usec
IRNUC	1H
CTEMP	22.8 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	44

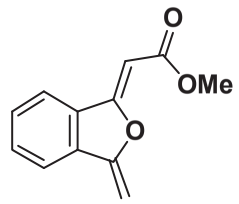


4m

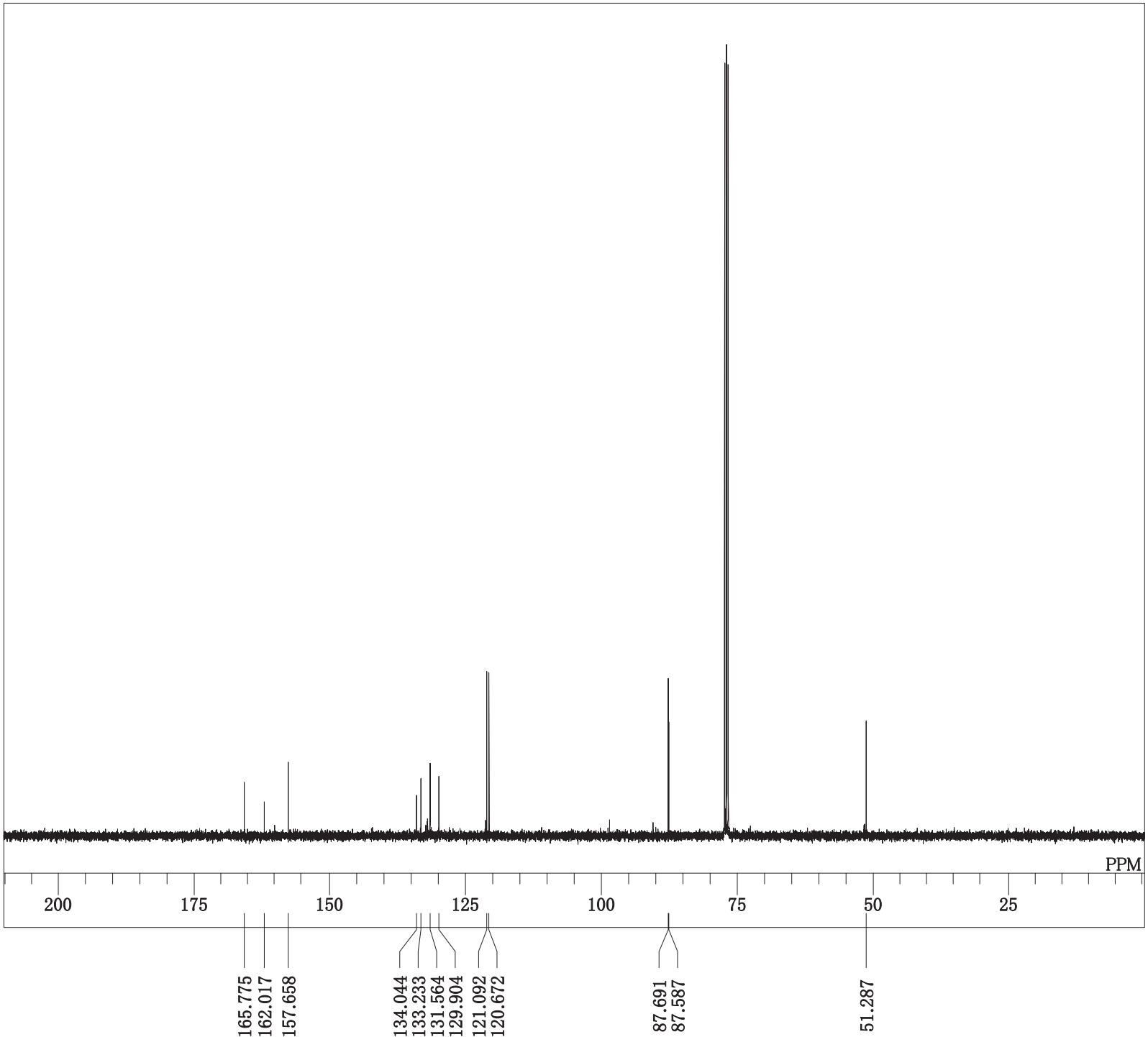


DFILE	20130605 ex1501 mattan pro1 1H-1.als
COMNT	single_pulse
DATIM	2013-06-05 20:22:31
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	26214
FREQU	5882.26 Hz
SCANS	8
ACQTM	4.4564 sec
PD	3.0000 sec
PW1	5.05 usec
IRNUC	1H
CTEMP	23.2 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	48

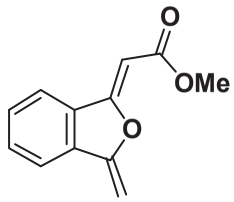
1H-NMR (CDCl3) δ :
5.20 (1H, d, J = 3.1 Hz),
5.06 (1H, d, J = 3.1 Hz).



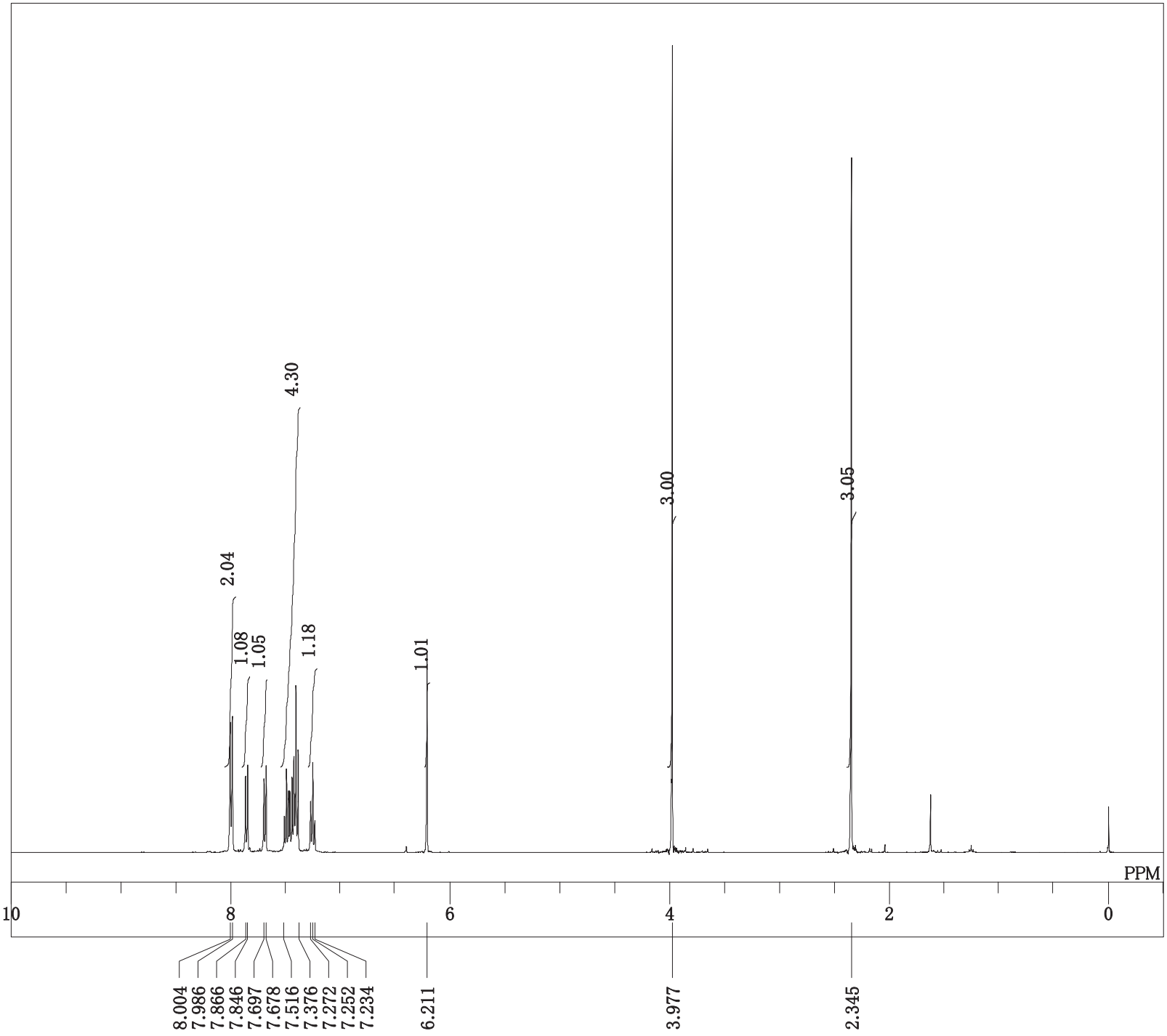
4n



DFILE	20130605 ex1501 mattann pro1 13C 1.a
COMNT	single pulse decoupled gated NOE
DATIM	2013-06-05 21:18:26
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.17 Hz
SCANS	1024
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	2.87 usec
IRNUC	1H
CTEMP	23.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	36

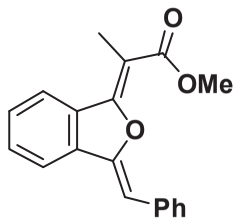


4n

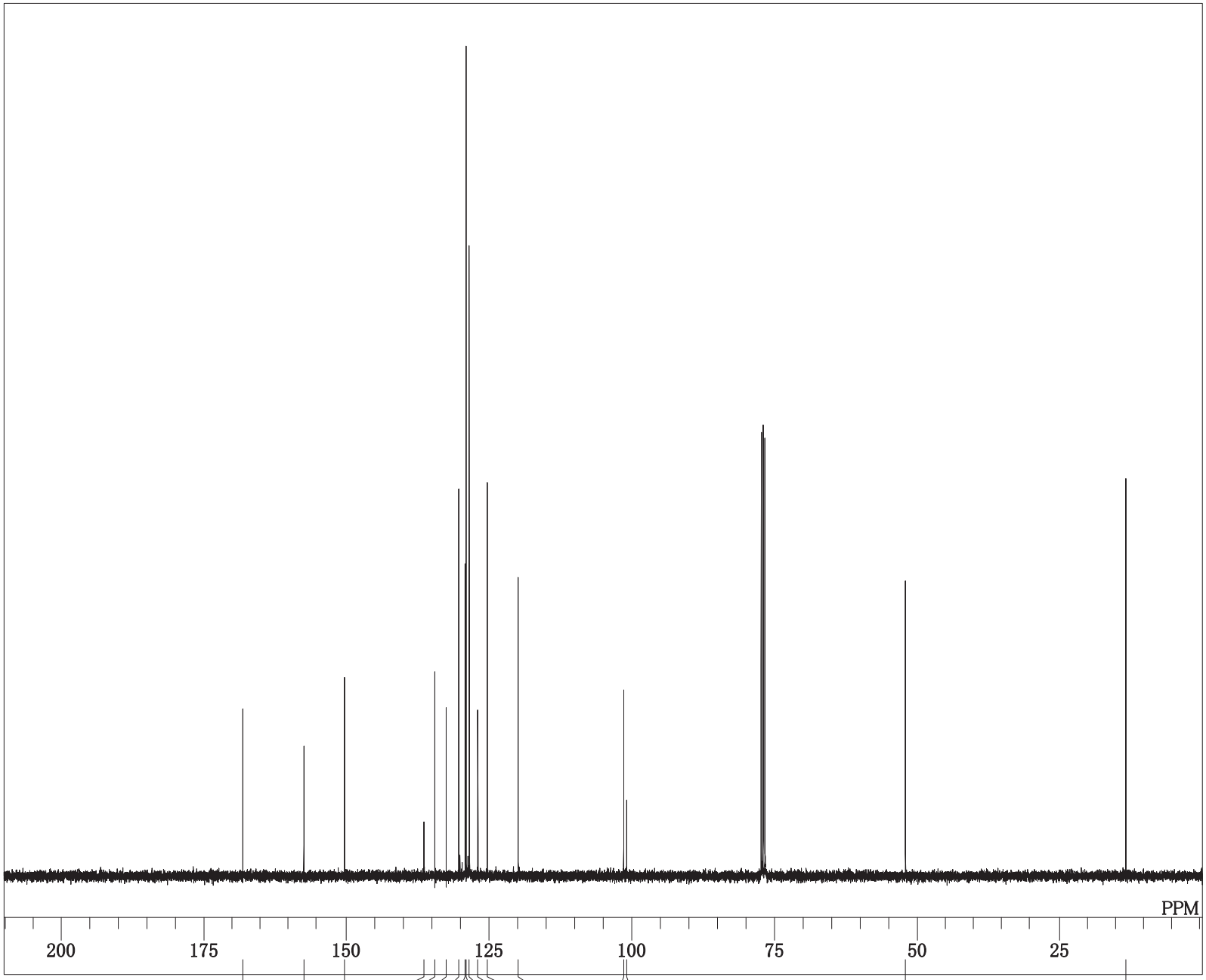


DFILE	20130613 ex1510 Et Ph Ph pro 1H 1.als
COMNT	single_pulse
DATIM	2013-06-13 16:15:15
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	26214
FREQU	5882.26 Hz
SCANS	8
ACQTM	4.4564 sec
PD	3.0000 sec
PW1	5.05 usec
IRNUC	1H
CTEMP	22.9 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	40

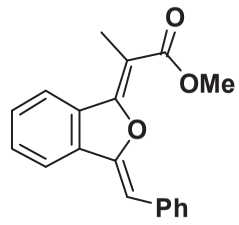
1H-NMR (CDCl3) δ :
7.99 (2H, d, J = 7.2 Hz),
7.86 (1H, d, J = 7.6 Hz),
7.69 (1H, d, J = 7.6 Hz),
7.25 (1H, t, J = 7.3 Hz).



4o



DFILE	20130613 ex1510 Et Ph Ph pro 13C-1.a
COMNT	single pulse decoupled gated NOE
DATIM	2013-06-13 16:55:45
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.17 Hz
SCANS	512
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	2.87 usec
IRNUC	1H
CTEMP	24.5 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	40



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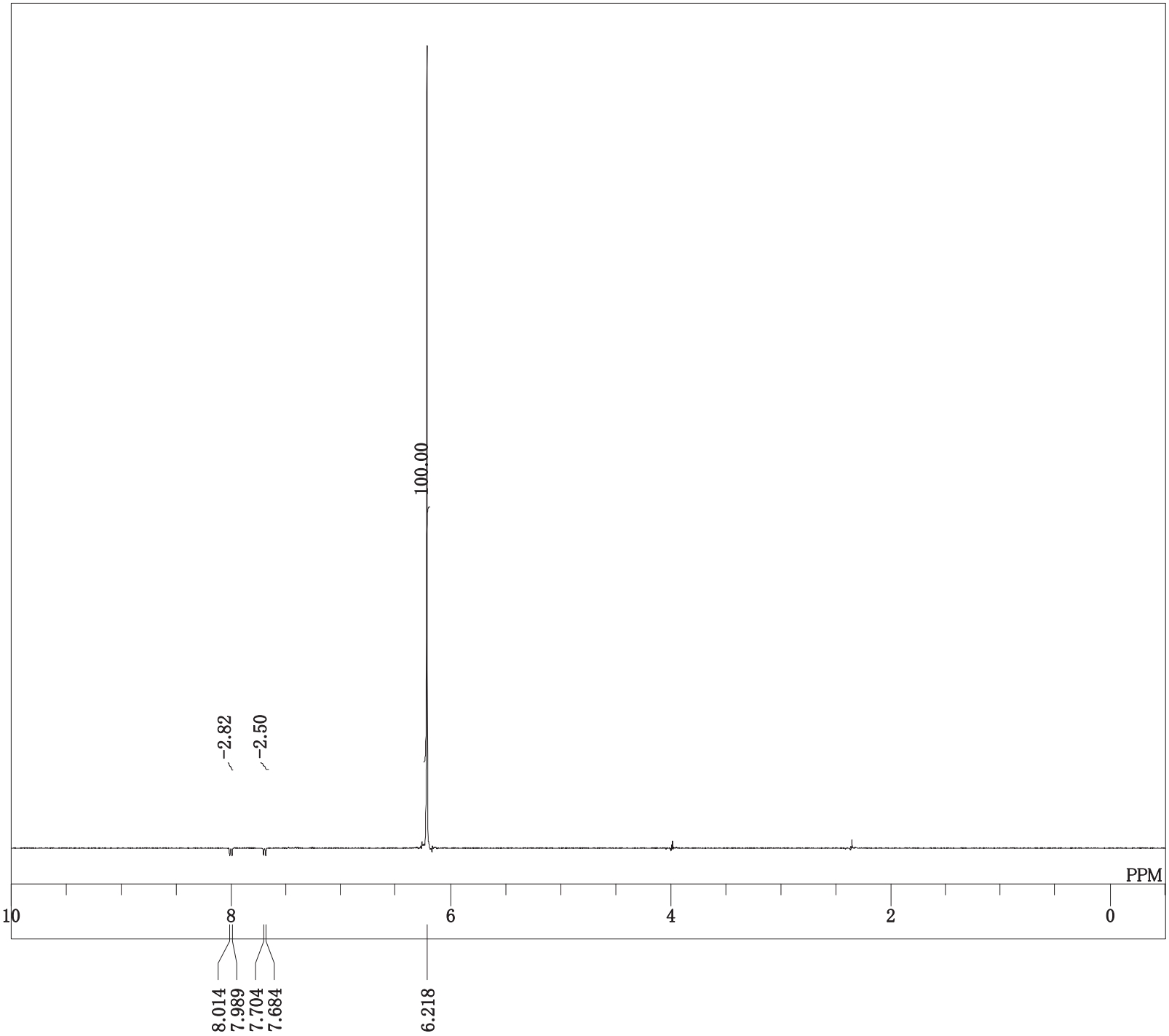
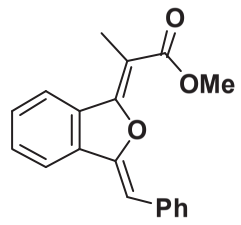


DFILE	20130613 ex1510 Et Ph Ph pro NOE 1.a
COMNT	DPFGSE NOE 1d
DATIM	2013-06-13 16:22:19
OBNUC	1H
EXMOD	noe_1d_dpfgse.ex
OBFRQ	391.78 MHz
OBSET	8.98 KHz
OBFIN	9.95 Hz
POINT	13107
FREQU	5882.26 Hz
SCANS	16
ACQTM	2.2282 sec
PD	7.0000 sec
PW1	10.10 usec
IRNUC	1H
CTEMP	23.5 c
SLVNT	CDCL3
EXREF	7.24 ppm
BF	0.12 Hz
RGAIN	58



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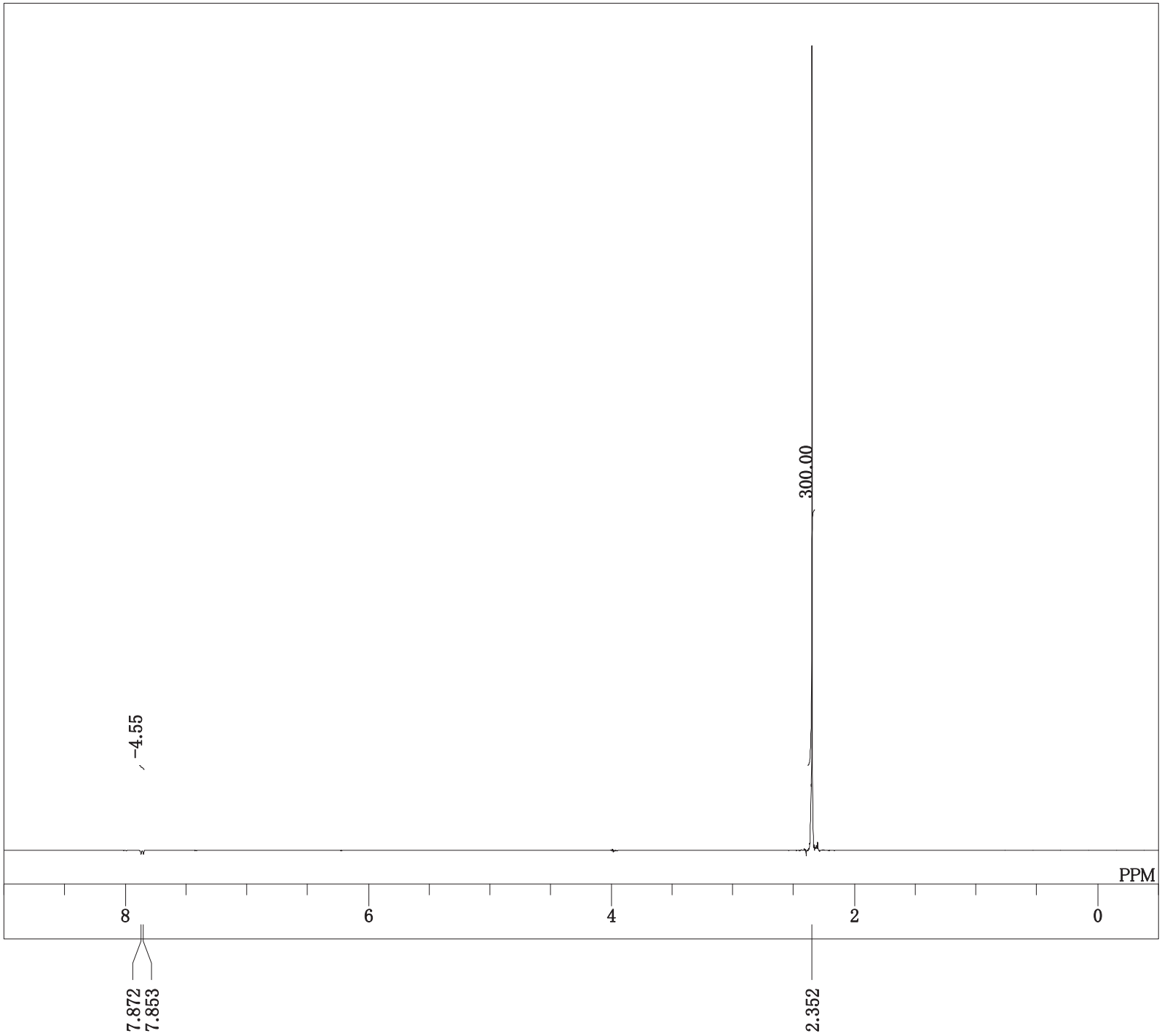
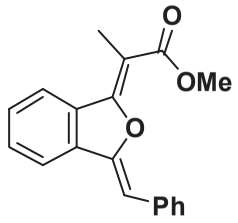
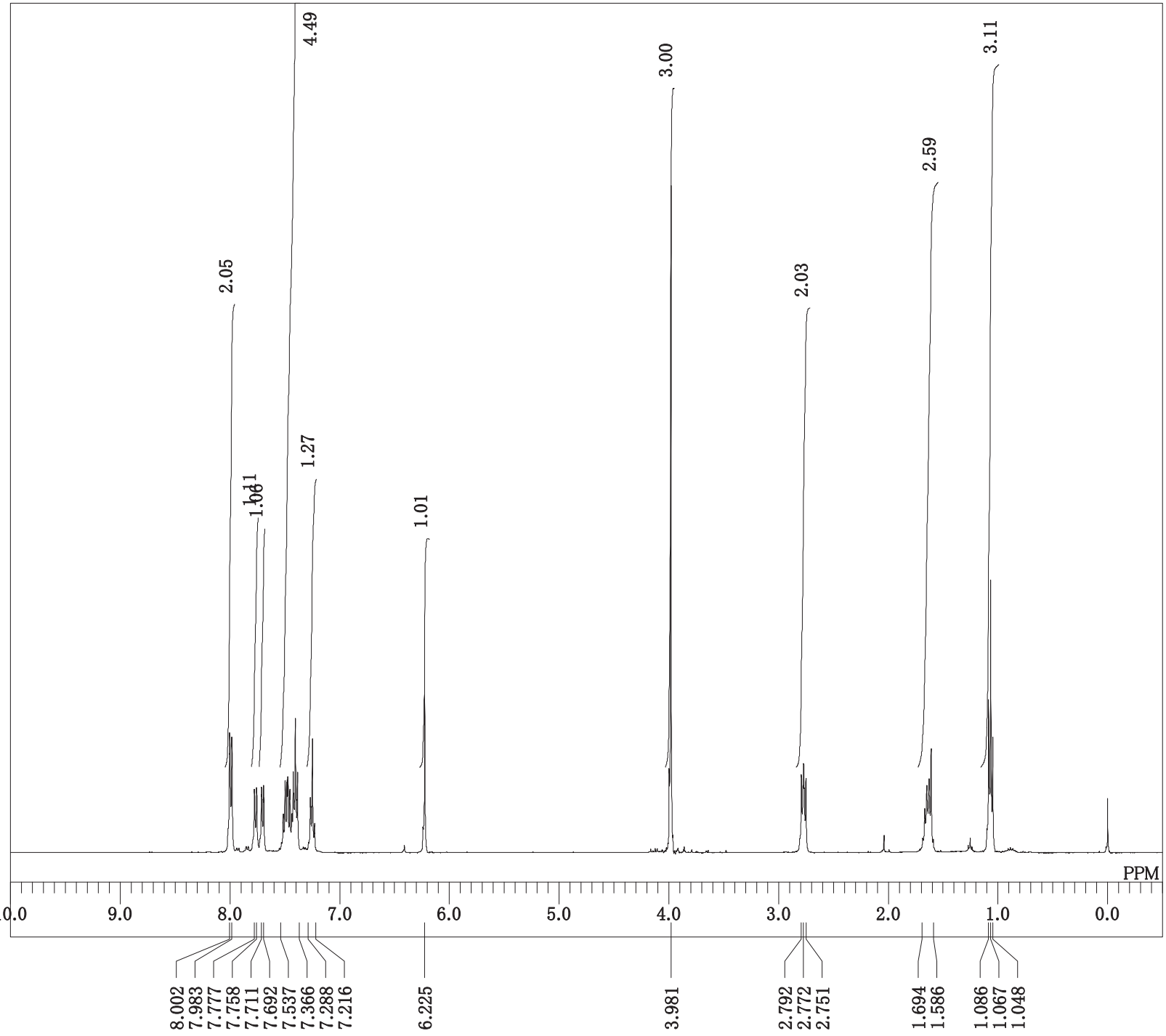


DFILE	20130613 ex1510 Et Ph Ph pro NOE-1
COMNT	DPFGSE NOE 1d
DATIM	2013-06-13 16:27:04
OBNUC	1H
EXMOD	noe_1d_dpfgse.ex
OBFRQ	391.78 MHz
OBSET	7.47 KHz
OBFIN	5.30 Hz
POINT	13107
FREQU	5882.26 Hz
SCANS	16
ACQTM	2.2282 sec
PD	7.0000 sec
PW1	10.10 usec
IRNUC	1H
CTEMP	23.8 c
SLVNT	CDCL3
EXREF	7.24 ppm
BF	0.12 Hz
RGAIN	54

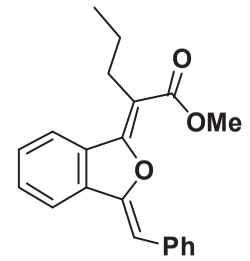


4o



DFILE	20130611 ex1506E2 Bu Ph Ph pro 1H-5
COMNT	single_pulse
DATIM	2013-06-11 20:37:23
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	26224
FREQU	5882.26 Hz
SCANS	8
ACQTM	4.4564 sec
PD	3.0000 sec
PW1	5.05 usec
IRNUC	1H
CTEMP	22.5 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	40

1H-NMR (CDCl3) δ :
7.99 (0H, d, J = 7.4 Hz),
7.77 (0H, d, J = 7.4 Hz),
7.70 (0H, d, J = 7.2 Hz),
2.77 (0H, t, J = 8.0 Hz),
1.07 (0H, t, J = 7.3 Hz).



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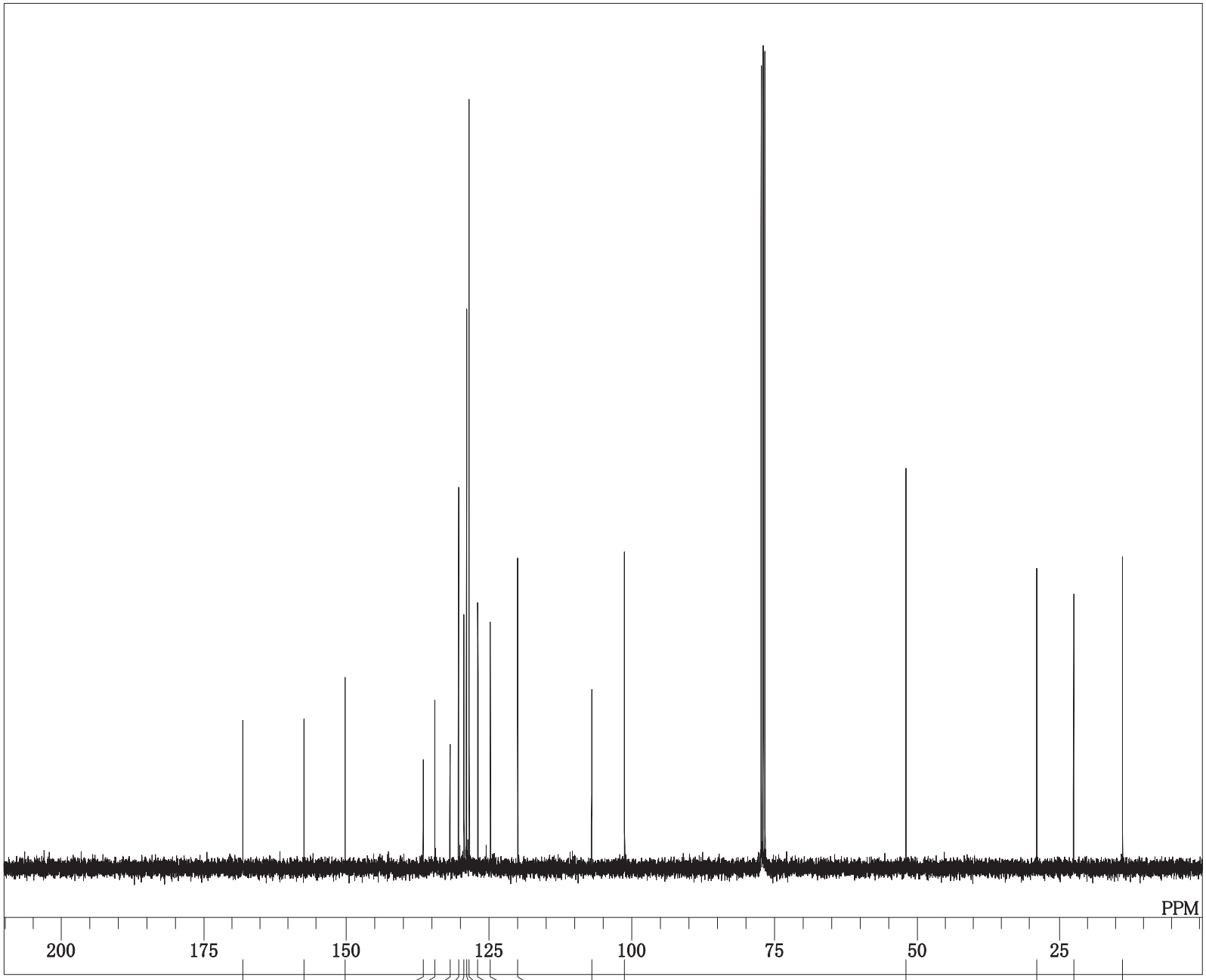
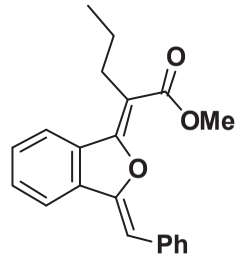
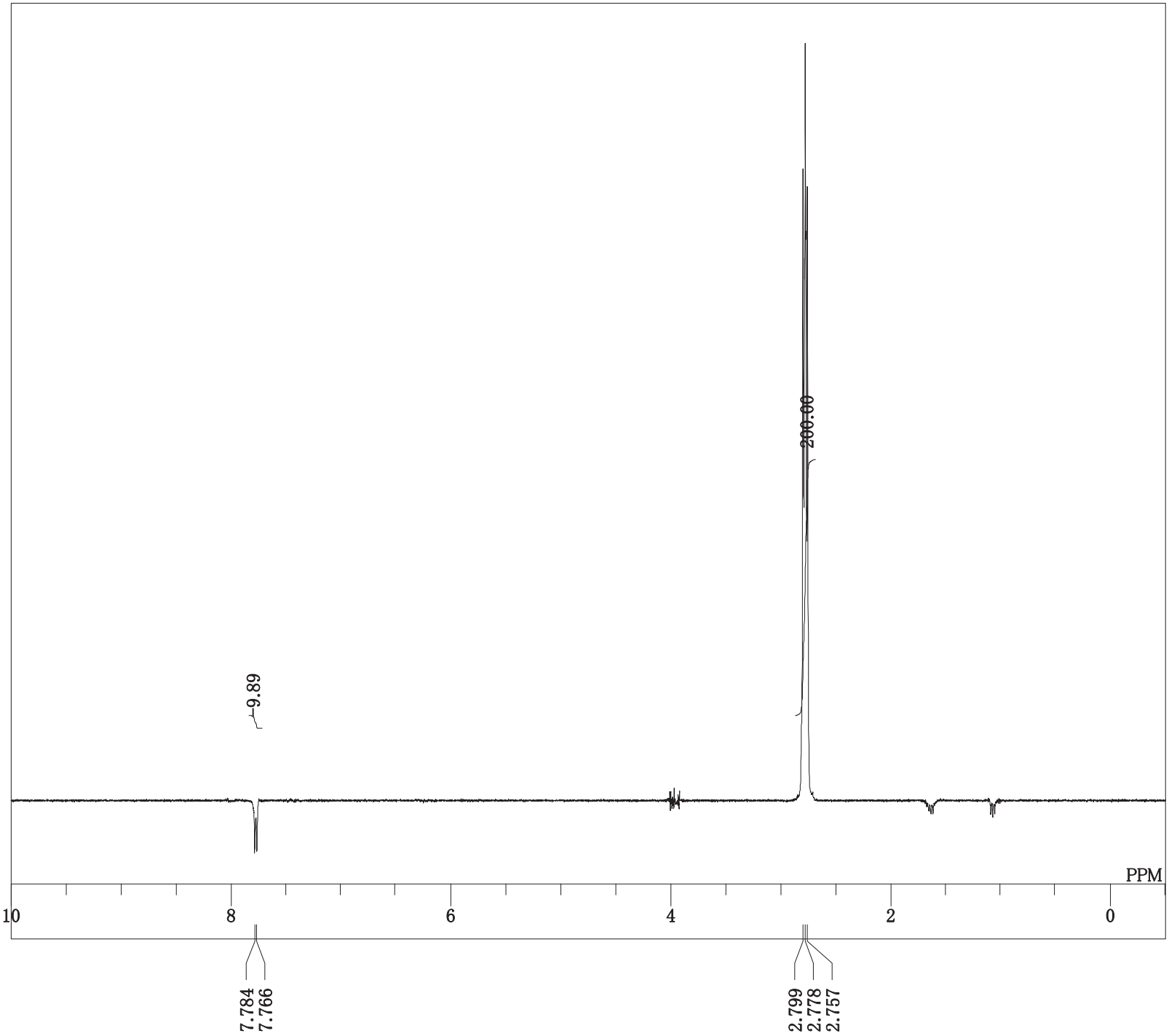


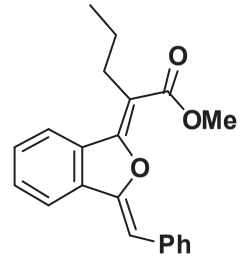
DFILE	20130611 ex1506E2 Bu Ph Ph pro 13C-
COMNT	single pulse decoupled gated NOE
DATIM	2013-06-11 21:20:02
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	26214
FREQU	24630.17 Hz
SCANS	512
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	2.87 usec
IRNUC	1H
CTEMP	22.7 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	46



4p



DFILE	20130611 ex1506E2 Bu Ph Ph pro NOE
COMNT	DPFGSE NOE 1d
DATIM	2013-06-11 20:50:55
OBNUC	1H
EXMOD	noe_1d_dpfgse.ex
OBFRQ	391.78 MHz
OBSET	7.64 KHz
OBFIN	2.01 Hz
POINT	13107
FREQU	5882.26 Hz
SCANS	16
ACQTM	2.2282 sec
PD	7.0000 sec
PW1	10.10 usec
IRNUC	1H
CTEMP	22.7 c
SLVNT	CDCL3
EXREF	7.24 ppm
BF	0.12 Hz
RGAIN	58



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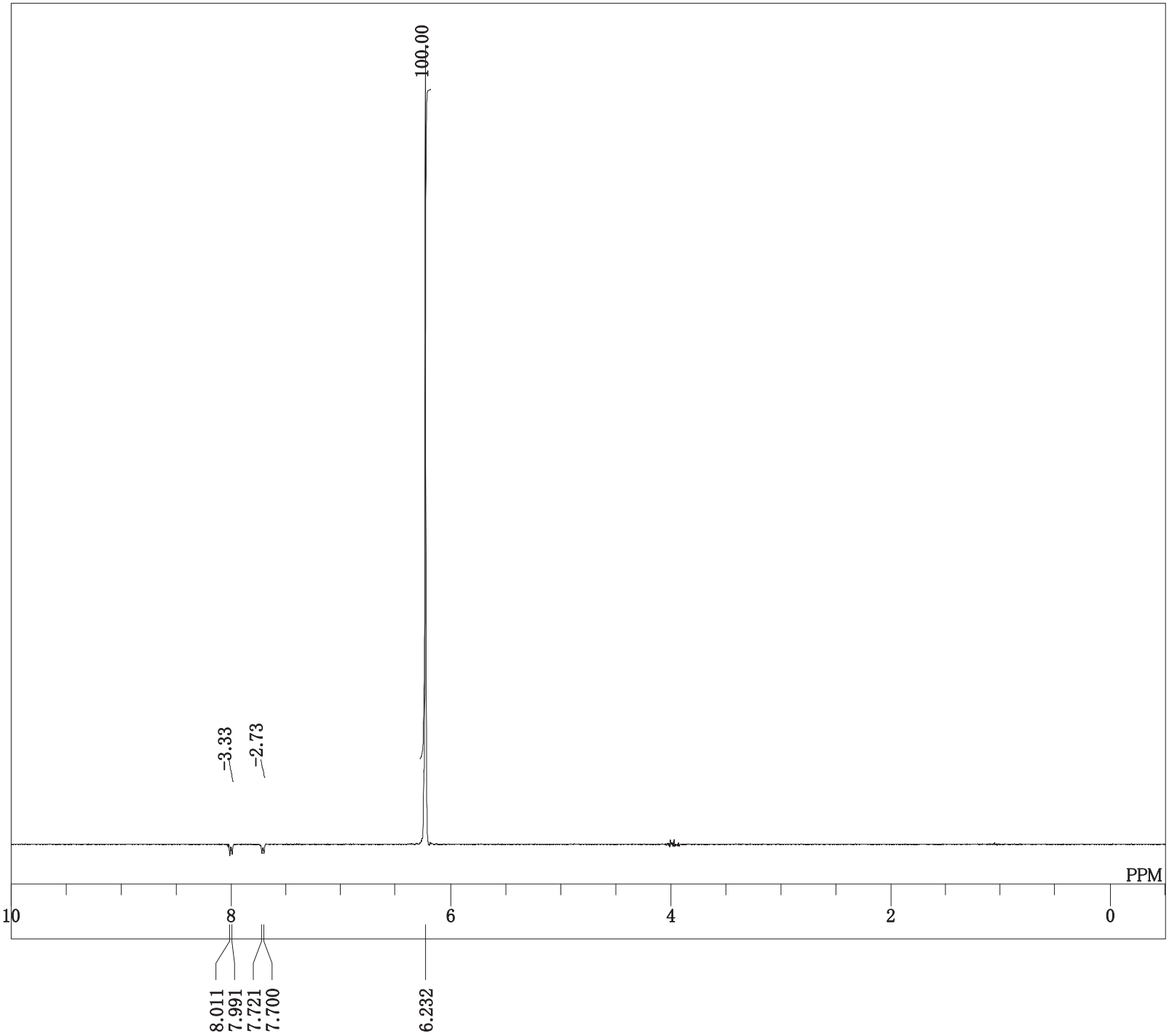
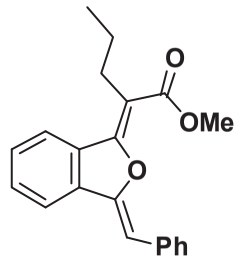


DFILE	20130611 ex1506E2 Bu Ph Ph pro NOE
COMNT	DPFGSE NOE 1d
DATIM	2013-06-11 20:44:37
OBNUC	1H
EXMOD	noe_1d_dpfgse.ex
OBFRQ	391.78 MHz
OBSET	8.99 KHz
OBFIN	5.55 Hz
POINT	13107
FREQU	5882.26 Hz
SCANS	16
ACQTM	2.2282 sec
PD	7.0000 sec
PW1	10.10 usec
IRNUC	1H
CTEMP	22.6 c
SLVNT	CDCL3
EXREF	7.24 ppm
BF	0.12 Hz
RGAIN	58



4p