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Supporting Information

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Synthesis of Biaryls Through Three-Component Assembly: Ambident Effect of *tert*-Butyldimethylsilyl Group for Regioselective Diels-Alder Reactions and Hiyama Couplings

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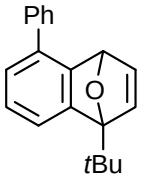
General Considerations

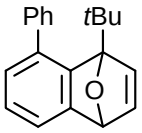
Reagents: Palladium catalyzed coupling reactions (Table 4) were carried out under an argon atmosphere in a reaction tube containing a stir-bar with a rubber septa. All other reactions including Diels-Alder reactions [Table 1, 2, 3, Eq (2 and 3)] were carried out under an argon or nitrogen atmosphere in a round bottom flask or a pear-shaped flask containing a stir-bar with an inlet adapter with a three-way stopcock. 1.6 M *n*BuLi in *n*-hexane was purchased from Kanto Chemical Co. $[(Allyl)PdCl]_2$ and $AsPh_3$ were obtained from Aldrich Chemical Co. and used without further purification. Anhydrous Cs_2CO_3 was purchased from Kishida Chemical Co. and stored in a bench-top desiccator filled with blue silica gel. Anhydrous toluene, tetrahydrofuran (THF), *N*,*N*-dimethylformamide (DMF) and 1,2-dimethoxyethane (DME) were purchased from Wako Pure Chemical Industries and used without further purification. All other reagents were purchased from Tokyo Chemical Industry Co., Aldrich Chemical Co., Kishida Chemical Co. or Nacalai Tesque and used without further purification. Benzyne precursors (**1c–i**)[1] 2-Phenylfuran **2d**[2,3] and 2-(trimethylsilyl)furan **2e**[4] were prepared according to the literature. Flash chromatography[5] was performed with Silica gel 60N, spherical neutral (40–50 μm) purchased from Kanto Chemical Co.

Analytical Methods. IR spectra were obtained on a JASCO WS/IR-8000. ^{1}H NMR and ^{13}C NMR spectra were recorded on a JEOL JMN-A500 or ECA-500 (^{1}H: 500 MHz, ^{13}C: 125 MHz) instrument with chemical shifts reported in ppm relative to the residual deuterated solvent or the internal standard tetramethylsilane. NOESY spectra were recorded using standard pulse sequences on a JEOL ECA-500. The mass spectra were measured on a JEOL MStation JMS-700 spectrometer. The GC-MS analysis was performed on Agilent Technologies 6890N (GC) combined with JEOL JMS-AMSUN (MS). Elemental analyses were performed by YANACO CHN CORDER MT-5 instrument. Yield refers to isolated yields of compounds greater than 95% purity as determined by ^{1}H NMR analysis. ^{1}H NMR and melting points (where applicable) of all known compounds were taken. All new products were further characterized by elemental analysis or HRMS.

General Procedure for Diels-Alder Reactions of 3-Substituted-Benzynes 3a–i and Furans 2a–f (Table 1 and 2). An oven-dried pear-shaped flask was charged with **1a–i** (1.0 equiv) and capped with an inlet adapter with a three-way stopcock and then evacuated and back-filled with nitrogen. The flask underwent an azeotropic dehydration using toluene (this process was repeated twice). Anhydrous toluene (0.2 M) and furan **2a–f** (3.0 equiv) were added via a syringe, followed by the slow addition of a 1.6 M *n*BuLi hexane solution (2.0 equiv) over 10 min at –78 °C. After 10–30 min, a saturated aqueous NH_4Cl solution was added to the reaction. The reaction mixture was extracted with EtOAc (this process was repeated three times). The combined organic layers were dried over $MgSO_4$ and concentrated under reduced pressure. The crude product was purified by flash column chromatography on silica gel to provide the adducts *anti*- and *syn*-**4** (We have expected that the major products of all Diels-Alder reactions between 3-TBDMS-benzynes **3d–i** and 2-alkylfurans **2a–c** would be *anti*-**4**, because the *anti*-**4d** determined by NOESY NMR study formed predominantly over *syn*-**4d**).



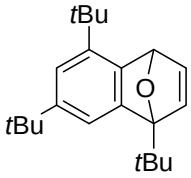
anti-**4a** ($CDCl_3$)
Table 1, entry 1



syn-**4a** ($CDCl_3$)
Table 1, entry 1

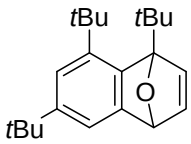
1-*tert*-Butyl-1,4-epoxy-5-phenyl-1,4-dihydronaphthalene *anti*-4a.

1-*tert*-Butyl-1,4-epoxy-8-phenyl-1,4-dihydronaphthalene *syn*-4a (Table 1, entry 1). Following general procedure, a mixture of **1a** (114 mg, 0.30 mmol), 2-*tert*-butylfuran **2a** (2.1×10^{-1} mL, 1.5 mmol), 1.6 M *n*BuLi in hexane (3.9×10^{-1} mL, 0.60 mmol) in toluene (1.5 mL) was stirred for 15 min at –78 °C. The crude product (*anti*-**4a**/*syn*-**4a** = 1.7 : 1) was purified by column chromatography (hexane/EtOAc = 50 : 1) to provide a mixture of titled compounds (*anti*- and *syn*-**4a**) with small amount of by-product as a colorless oil (59 mg, 70% total yield of *anti*-**4a** and *syn*-**4a**). The ratio and yield were determined by ^{1}H NMR and NOESY NMR data of the crude material because these isomers were inseparable by a silica gel column chromatography.



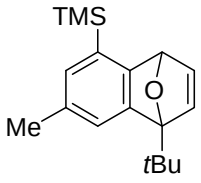
anti-**4b**

1,5,7-Tri-*tert*-butyl-1,4-epoxy-1,4-dihydronaphthalene *anti*-4b (Table 1, entry 2). Following general procedure, a mixture of **1b** (84 mg, 0.20 mmol), 2-*tert*-butylfuran **2a** (8.6×10^{-2} mL, 0.60 mmol), 1.6 M *n*BuLi in hexane (2.6×10^{-1} mL, 0.40 mmol) in toluene (0.1 mL) was stirred for 15 min at –78 °C. The crude product was purified by column chromatography (hexane/EtOAc = 50 : 1) to provide the titled compound *anti*-**4b** as a yellow solid (16 mg, 26%). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.34 (brs, 1H), 7.03 (dd, *J* = 1.5, 5.0 Hz, 1H), 6.98 (d, *J* = 5.0 Hz, 1H), 7.00 (brs, 1H), 6.06 (d, *J* = 1.5 Hz, 1H), 1.36 (s, 9H), 1.31 (s, 9H), 1.29 (s, 9H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 148.9, 146.9, 146.7, 143.9, 143.3, 142.0, 118.1, 117.3, 98.6, 81.9, 35.4, 34.8, 32.4, 31.6, 31.4, 26.7. IR ($CHCl_3$, cm^{-1}) 2960, 1477, 1364. HRMS (FAB, NBA) Calcd for $C_{22}H_{33}O$ $[M+H]^+$: 313.2531. Found: 313.2549.



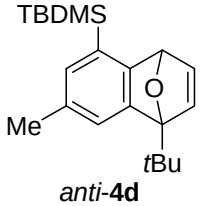
syn-**4b**

1,6,8-Tri-*tert*-butyl-1,4-epoxy-1,4-dihydronaphthalene *syn*-4b (Table 1, entry 2) was obtained via preparative TLC of the above-mentioned crude product as a yellow solid (17 mg, 27%). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.08 (d, *J* = 1.0 Hz, 1H), 7.05 (d, *J* = 1.0 Hz, 1H), 6.94 (d, *J* = 6.0 Hz, 1H), 6.91 (dd, *J* = 1.5, 6.0 Hz, 1H), 5.50 (d, *J* = 1.5 Hz, 1H), 1.40 (s, 9H), 1.39 (s, 9H), 1.28 (s, 9H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 151.5, 147.7, 147.0, 146.0, 144.5, 141.5, 120.4, 115.4, 105.8, 80.2, 36.4, 36.0, 34.4, 32.8, 31.3, 29.5. IR ($CHCl_3$, cm^{-1}) 2964, 1479, 1364. HRMS (FAB, NBA) Calcd for $C_{22}H_{33}O$ $[M+H]^+$: 313.2531. Found: 313.2524.



anti-**4c**

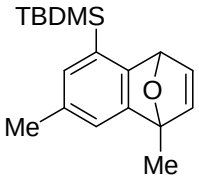
1-*tert*-Butyl-1,4-epoxy-7-methyl-5-trimethylsilyl-1,4-dihydronaphthalene *anti*-4c (Table 1, entry 3). Following general procedure, a mixture of **1c** (78 mg, 0.20 mmol), 2-*tert*-butylfuran **2a** (8.6×10^{-2} mL, 0.60 mmol), 1.6 M *n*BuLi in hexane (0.26 mL, 0.40 mmol) in toluene (1 mL) was stirred for 30 min at –78 °C. The crude product (*anti*-**4c**/*syn*-**4c** = 39 : 1) was purified by column chromatography (hexane/EtOAc = 50 : 1) to provide the titled compound *anti*-**4c** as a pale yellow oil (37 mg, 63%). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.23 (s, 1H), 6.98 (dd, *J* = 1.5, 5.5 Hz, 1H), 6.94 (d, *J* = 5.5 Hz, 1H), 6.84 (s, 1H), 5.73 (d, *J* = 1.5 Hz, 1H), 2.30 (s, 3H), 1.28 (s, 9H), 0.29 (s, 9H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 155.4, 148.2, 144.4, 142.9, 132.8, 130.8, 129.1, 123.8, 98.9, 81.5, 32.5, 26.6, 21.6, –0.3. IR ($CHCl_3$, cm^{-1}) 2958, 1463, 1366. HRMS (FAB, NBA) Calcd for $C_{14}H_{18}OSi$ $[M+H]^+$: 230.1127. Found: 230.1120.



anti-**4d**

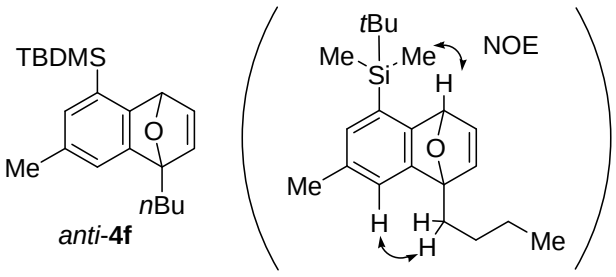
1-*tert*-Butyl-5-*tert*-butyldimethylsilyl-1,4-epoxy-7-methyl-1,4-dihydronaphthalene *anti*-4d (Table 1, entry 4 and Table 2, entry 4). Following general procedure, a mixture of **1d** (32 mg, 0.075 mmol), 2-*tert*-butylfuran **2a** (3.2×10^{-2} mL, 0.22 mmol), 1.6 M *n*BuLi in hexane (9.0×10^{-2} mL, 0.15 mmol) in toluene (0.4 mL) was stirred for 15 min at –78 °C. The crude product was purified by column chromatography (hexane/EtOAc = 50 : 1 then 40 : 1) to provide the titled compound *anti*-**4d** as a yellow solid (17 mg, 69%). Mp 84–87 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.23 (s, 1H), 6.98 (dd, *J* = 2.0, 5.5 Hz, 1H), 6.94 (d, *J* = 5.5 Hz, 1H), 6.82 (s, 1H), 5.72 (d, *J* = 2.0 Hz, 1H), 2.30 (s, 3H), 1.28 (s, 9H), 0.91 (s, 9H), 0.34 (s, 3H), 0.28 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 156.2, 148.2, 144.5, 142.7, 132.3, 130.5, 128.4, 123.7, 98.8, 82.2, 32.5, 26.6, 26.5, 21.6,

17.1, –4.4, –4.8. IR ($CHCl_3$, cm^{-1}) 2957, 1464. HRMS (FAB, NBA) Calcd for $C_{21}H_{32}OSi$ $[M]^+$: 328.2222. Found: 328.2226.



anti-**4e**

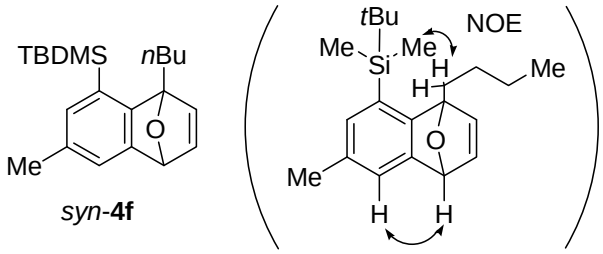
5-*tert*-Butyldimethylsilyl-1,4-epoxy-1,7-dimethyl-1,4-dihydronaphthalene *anti*-4e (Table 2, entry 1). Following general procedure, a mixture of **1d**[1] (99 mg, 0.23 mmol), 2-methylfuran **2b** (0.31 mL, 3.4 mmol), 1.6 M *n*BuLi in hexane (0.22 mL, 0.34 mmol) in toluene (1.2 mL) was stirred for 15 min at –78 °C. The crude product (*anti*-**4e**/*syn*-**4e** = 3.9 : 1) was purified by column chromatography (hexane/EtOAc = 25 : 1 then 20 : 1) to provide the titled compound *anti*-**4e** as a colorless solid (45 mg, 68% total yield of *anti*-**4e** and *syn*-**4e**). Mp 83–85 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 6.99–6.98 (m, 2H), 6.83 (s, 1H), 6.72 (d, J = 5.5 Hz, 1H), 5.69 (d, J = 2.0 Hz, 1H), 2.30 (s, 3H), 1.90 (s, 3H), 0.91 (s, 9H), 0.31 (s, 3H), 0.28 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 154.4, 150.2, 143.4, 144.4, 133.1, 103.7, 128.9, 120.8, 88.7, 82.7, 26.5, 21.4, 17.1, 15.2, –4.4, –5.0. IR ($CHCl_3$, cm^{-1}) 2928, 1462. HRMS (FAB, NBA) Calcd for $C_{18}H_{26}OSi$ $[M+H]^+$: 287.1831. Found 287.1829.



anti-**4f**

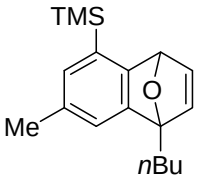
1-*n*-Butyl-5-*tert*-butyldimethylsilyl-1,4-epoxy-7-methyl-1,4-dihydronaphthalene *anti*-4f (Table 2, entry 2). Following general procedure, a mixture of **1d**[1] (3.0 g, 7.0 mmol), 2-*n*-butylfuran (**2c**) (3.0 mL, 21 mmol), 1.6 M *n*BuLi in hexane (8.7 mL, 14 mmol) in toluene (36 mL) was stirred for 15 min at –78 °C. The crude product (*anti*-**4f**/*syn*-**4f** = 5.9 : 1) was purified by column chromatography (hexane/EtOAc = 20 : 1) to provide the

titled compound *anti*-**4f** (1.2 g, 52%) as a pale yellow oil (71% total yield of *anti*-**4f** and *syn*-**4f**). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 6.99–6.98 (m, 2H), 6.82 (s, 1H), 6.74 (d, *J* = 5.5 Hz, 1H), 5.71 (d, *J* = 1.5 Hz, 1H), 2.36–2.30 (m, 1H), 2.31 (s, 3H), 2.24–2.18 (m, 1H), 1.66–1.55 (m, 2H), 1.52–1.44 (m, 2H), 1.00 (t, *J* = 6.0 Hz, 3H), 0.91 (s, 9H), 0.31 (s, 3H), 0.28 (s, 3H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ: 154.9, 149.8, 144.43, 144.36, 133.0, 130.7, 128.8, 121.1, 92.1, 82.6, 28.9, 26.8, 26.5, 23.2, 21.5, 17.1, 14.0, –4.43, –4.98. IR ($CHCl_3$, cm^{-1}) 2957, 1578, 1464. HRMS (FAB, NBA): Anal. Calcd for $C_{20}H_{31}OSi$ $[M+H]^+$: 315.2144. Found 315.2151.



syn-**4f**

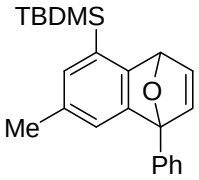
1-*n*-Butyl-8-*tert*-butyldimethylsilyl-1,4-epoxy-6-methyl-1,4-dihydronaphthalene *syn*-4f (Table 2, entry 2) was obtained via column chromatography of the above-mentioned crude product as a pale yellow oil. ^{1}H NMR ($CDCl_3$, 500 MHz) *δ*: 7.05 (s, 1H), 6.98 (dd, *J* = 1.5, 6.0 Hz, 1H), 6.96 (s, 1H), 6.80 (d, *J* = 6.0 Hz, 1H), 5.56 (d, *J* = 1.5 Hz, 1H), 2.61–2.55 (m, 1H), 2.28 (s, 3H), 2.22–2.16 (m, 1H), 1.65–1.60 (m, 1H), 1.50–1.44 (m, 3H), 0.98 (t, *J* = 7.0 Hz, 3H), 0.93 (s, 9H), 0.37 (s, 3H), 0.35 (s, 3H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ: 155.2, 150.7, 144.3, 144.2, 133.1, 132.5, 129.3, 121.8, 95.1, 81.0, 31.7, 27.6, 27.3, 23.1, 21.2, 17.7, 14.1, –0.9, –1.9. IR ($CHCl_3$, cm^{-1}) 2957, 1464. HRMS (FAB, NBA): Anal. Calcd for $C_{17}H_{23}OSi$ $[M–C_4H_9]^+$: 271.1518; Found 271.1507.



anti-**4g**

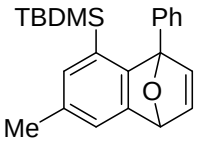
1-*n*-Butyl-1,4-epoxy-7-methyl-5-trimethylsilyl-1,4-dihydronaphthalene *anti*-4g (Table 2, entry 3). Following general procedure, a mixture of **1c** (117 mg, 0.30 mmol),

2-*n*-butylfuran **2c** (0.13 mL, 0.90 mmol), 1.6 M *n*BuLi in hexane (0.36 mL, 0.60 mmol) in toluene (1.5 mL) was stirred for 30 min at –78 °C. The crude product (*anti*-**4g**/*syn*-**4g** = 5.0 : 1) was purified by column chromatography (hexane/EtOAc = 40 : 1) to provide the titled compound *anti*-**4g** (46 mg, 54%) as a colorless solid (65% total yield of *anti*-**4g** and *syn*-**4g**). Mp 44–45 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 6.99–6.98 (m, 2H), 6.85 (s, 1H), 6.75 (d, *J* = 5.5 Hz, 1H), 5.73 (d, *J* = 2.0 Hz, 1H), 2.35–2.17 (m, 2H), 2.30 (s, 3H), 1.64–1.46 (m, 4H), 0.98 (t, *J* = 7.0 Hz, 3H), 0.30 (s, 9H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 154.1, 149.7, 144.6, 144.3, 133.4, 131.2, 129.3, 121.3, 92.2, 81.8, 28.9, 26.8, 23.2, 21.4, 14.0, –0.4. IR ($CHCl_3$, cm^{-1}) 2959, 1456. HRMS (FAB, NBA) Calcd for $C_{18}H_{26}OSi$ $[M]^+$: 286.1754. Found: 286.1753.



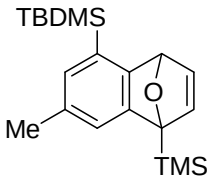
anti-**4h**

5-*tert*-Butyldimethylsilyl-1,4-epoxy-7-methyl-1-phenyl-1,4-dihydronaphthalene *anti*-4h (Table 2, entry 5). Following general procedure, a mixture of **1d**[1] (56 mg, 0.13 mmol), 2-phenylfuran **2d**[2,3] (56 mg, 0.39 mmol), 1.6 M *n*BuLi in hexane (0.16 mL, 0.26 mmol) in toluene (0.65 mL) was stirred for 20 min at –78 °C. The crude product (*anti*-**4h**/*syn*-**4h** = 1.2 : 1) was purified by column chromatography (hexane/EtOAc = 40 : 1 then 10 : 1) to provide the titled compound *anti*-**4h** (12 mg, 27%) as a colorless oil (53% total yield of *anti*-**4h** and *syn*-**4h**). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.62 (d, *J* = 7.5 Hz, 2H), 7.50 (t, *J* = 7.5 Hz, 2H), 7.42 (t, *J* = 7.5 Hz, 1H), 7.18 (d, *J* = 6.0 Hz, 1H), 7.14 (dd, *J* = 1.5, 6.0 Hz, 1H), 6.87 (s, 1H), 6.81 (s, 1H), 5.92 (d, *J* = 1.5 Hz, 1H), 2.25 (s, 3H), 0.95 (s, 9H), 0.36 (s, 3H), 0.34 (s, 3H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ: 153.6, 150.5, 144.4, 136.3, 133.2, 131.0, 129.1, 128.6, 128.1, 126.5, 122.0, 93.0, 83.1, 26.5, 21.6, 17.2, –4.4, –4.9. IR ($CHCl_3$, cm^{-1}) 1603, 1462, 1449. HRMS (FAB, NBA): Anal. Calcd for $C_{23}H_{29}OSi$ $[M+H]^+$: 349.1988. Found 349.1995.



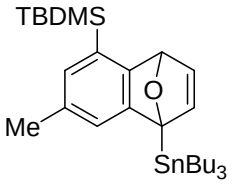
syn-**4h**

8-*tert*-Butyldimethylsilyl-1,4-epoxy-6-methyl-1-phenyl-1,4-dihydronaphthalene *syn*-4h (Table 2, entry 5) was obtained via column chromatography of the above-mentioned crude product as a colorless oil (12 mg, 26%). ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 7.59 (brs, 2H), 7.42–7.37 (m, 4H), 7.13 (dd, J = 2.0, 5.5 Hz, 1H), 7.10 (brs, 1H), 6.99 (brs, 1H), 5.68 (d, J = 2.0 Hz, 1H), 2.31 (s, 3H), 0.74 (s, 9H), –0.17 (s, 3H), –0.48 (s, 3H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ: 155.8, 150.4, 145.4, 141.5, 137.7, 133.3, 132.6, 131.2, 130.2, 129.0, 128.5, 121.6, 96.5, 81.6, 27.3, 21.2, 17.7, –3.5, –3.9. IR ($CHCl_3$, cm^{-1}) 1601, 1464, 1449. HRMS (FAB, NBA): Anal. Calcd for $C_{23}H_{28}OSi$ $[M]^+$: 348.1909; Found 348.1880.



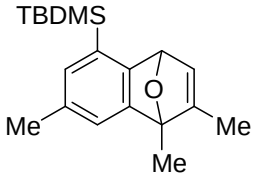
anti-**4i**

5-*tert*-Butyldimethylsilyl-1,4-epoxy-7-methyl-1-trimethylsilyl-1,4-dihydronaphthalene *anti*-4i (Table 2, entry 6). Following general procedure, a mixture of **1d** (33 mg, 7.6×10^{-2} mmol), 2-(trimethylsilyl)furan **2e**[4] (32 mg, 0.23 mmol), 1.6 M *n*BuLi in hexane (9.0×10^{-2} mL, 0.15 mmol) in toluene (0.4 mL) was stirred for 15 min at –78 °C. The crude product was purified by column chromatography (hexane/EtOAc = 40 : 1 then 35 : 1) to provide the titled compound *anti*-**4i** as a colorless solid (11 mg, 40%). Mp 94–96 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.00–6.99 (m, 2H), 6.93 (d, J = 5.5 Hz, 1H), 6.82 (s, 1H), 5.78 (d, J = 1.0 Hz, 1H), 2.29 (s, 3H), 0.90 (s, 9H), 0.32 (s, 3H), 0.30 (s, 9H), 0.28 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 154.5, 151.3, 145.6, 143.2, 132.5, 130.4, 129.0, 122.3, 85.4, 84.0, 26.5, 21.5, 17.1, –3.1, –4.4, –4.9. IR ($CHCl_3$, cm^{-1}) 2954, 2928, 1464. HRMS (FAB, NBA) Calcd for $C_{20}H_{33}OSi_2$ $[M+H]^+$: 345.2070. Found: 345.2063.



anti-**4j**

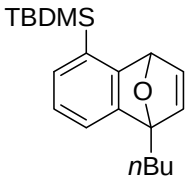
5-*tert*-Butyldimethylsilyl-1,4-epoxy-7-methyl-1-tributylstannyl-1,4-dihydronaphthalene *anti*-4j (Table 2, entry 7). Following general procedure, a mixture of **1d** (2.1×10^2 mg, 0.48 mmol), 2-tributylstannylfuran **2f** (5.2×10^2 mg, 0.46 mmol), 1.6 M *n*BuLi in hexane (0.58 mL, 0.97 mmol) in toluene (2.5. mL) was stirred for 15 min at –78 °C. The crude product was purified by column chromatography (hexane/EtOAc = 50 : 1 then 40 : 1) to provide the titled compound *anti*-**4j** as a yellow oil (151 mg, 56%). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 6.96 (brs, 1H×2), 6.87 (s, 1H), 6.79 (s, 1H), 5.73 (s, 1H), 1.66–1.55 (m, 6H), 1.34 (hex, J = 7.0 Hz, 6H), 1.19–1.05 (m, 6H), 0.894 (s, 9H), 0.888 (t, J = 7.0 Hz, 9H), 0.30 (s, 3H), 0.27 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 154.1, 153.9, 148.8, 142.0, 132.6, 130.3, 129.0, 122.3, 87.1, 83.3, 29.1, 27.4, 26.6, 21.5, 17.1, 13.6, 9.1, –4.4, –4.9. IR ($CHCl_3$, cm^{-1}) 1603. HRMS (FAB, NBA) Calcd for $C_{29}H_{51}OSi^{120}Sn$ $[M+H]^+$: 563.2736. Found: 563.2744.



anti-**4k**

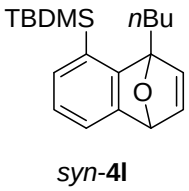
5-*tert*-Butyldimethylsilyl-1,4-epoxy-1,2-dimethyl-1,4-dihydronaphthalene *anti*-4k (Table 2, entry 8). Following general procedure, a mixture of **1d** (200 mg, 0.46 mmol), 2,3-dimethylfuran **2g** (0.15 mL, 1.4 mmol), 1.6 M *n*BuLi in hexane (0.56 mL, 0.92 mmol) in toluene (2.3 mL) was stirred for 15 min at –78 °C. The crude product was purified by column chromatography (hexane/EtOAc = 20 : 1) and preparative HPLC to provide the titled compound *anti*-**4k** as a colorless solid (83 mg, 60%). Mp 85–88 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.02 (s, 1H), 6.88 (s, 1H), 6.47 (s, 1H), 5.63 (s, 1H), 2.33 (s, 3H), 1.803 (s, 3H), 1.796 (s, 3H), 0.93 (s, 9H), 0.33 (s, 3H), 0.29 (s, 3H). ^{13}C NMR

(125 MHz, $CDCl_3$) δ: 155.1, 154.1, 150.2, 136.3, 132.6, 130.8, 127.8, 120.4, 89.7, 81.7, 26.5, 21.4, 17.1, 13.5, 12.8, –4.5, –5.0. IR ($CHCl_3$, cm^{-1}) 1603, 1462, 1435. HRMS (FAB, NBA) Calcd for $C_{19}H_{29}OSi$ $[M+H]^+$: 301.1988. Found: 301.1985.



anti-**4l**

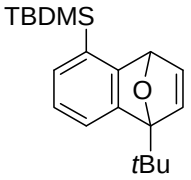
1-*n*-Butyl-5-*tert*-butyldimethylsilyl-1,4-epoxy-1,4-dihydronaphthalene *anti*-4l (Table 2, entry 9). Following general procedure, a mixture of **1e** (100 mg, 0.24 mmol), 2-*n*-butylfuran **2c** (0.10 mL, 0.71 mmol), 1.6 M *n*BuLi in hexane (0.29 mL, 0.47 mmol) in toluene (1.2 mL) was stirred for 60 min at –78 °C. The crude product (*anti*-**4l** /*syn*-**4l** = 7.0 : 1) was purified by column chromatography (hexane/EtOAc = 50 : 1 then 40 : 1) to provide the mixture of the isomers as a colorless oil (42 mg, 56% total isolated yield of *anti*-**4l** and *syn*-**4l**). The mixture was purified by preparative TLC (hexane/ether = 30 : 1) to afford the titled compound *anti*-**4l** as a colorless oil. ^1H NMR (500 MHz, $CDCl_3$) δ: 7.15 (d, *J* = 7.0 Hz, 1H), 7.05 (d, *J* = 7.0 Hz, 1H), 7.00 (dd, *J* = 2.0, 6.0 Hz, 1H), 6.96 (t, *J* = 7.0 Hz, 1H), 6.77 (d, *J* = 6.0 Hz, 1H), 5.75 (d, *J* = 2.0 Hz, 1H), 2.38–2.32 (m, 1H), 2.26–2.22 (m, 1H), 1.66–1.51 (m, 2H), 1.49 (hex, *J* = 7.5 Hz, 2H), 0.98 (t, *J* = 7.5 Hz, 3H), 0.91 (s, 9H), 0.32 (s, 3H), 0.30 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 157.6, 149.3, 144.6, 144.1, 130.8, 129.1, 123.6, 119.7, 92.2, 82.7, 28.9, 26.8, 26.5, 23.2, 17.2, 14.1, –4.5, –5.0. IR ($CHCl_3$, cm^{-1}) 2957, 2930, 1464. HRMS (FAB, NBA) Calcd for $C_{20}H_{31}OSi$ $[M+H]^+$: 315.2144. Found: 315.2121.



syn-**4l**

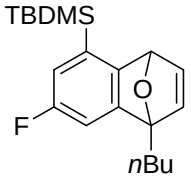
1-*n*-Butyl-8-*tert*-butyldimethylsilyl-1,4-epoxy-1,4-dihydronaphthalene *syn*-4l (Table 2, entry 9) was obtained via preparative TLC of the above-mentioned crude product as a colorless oil. ^1H NMR (500 MHz, $CDCl_3$) δ: 7.20 (d, *J* = 7.5 Hz, 1H), 7.16 (d, *J* = 7.5

Hz, 1H), 7.00 (d, *J* = 6.0 Hz, 1H), 6.91 (t, *J* = 7.5 Hz, 1H), 6.80 (d, *J* = 6.0 Hz, 1H), 5.60 (s, 1H), 2.62–2.56 (m, 1H), 2.21–2.26 (m, 1H), 1.66–1.55 (m, 2H), 1.49–1.43 (m, 2H), 0.97 (d, *J* = 7.0 Hz, 1H), 0.92 (s, 9H), 0.37 (s, 3H), 0.35 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 158.1, 150.1, 144.6, 144.1, 133.1, 129.6, 123.2, 120.4, 95.2, 81.0, 31.7, 27.6, 27.3, 23.1, 17.7, 14.1, –0.9, –1.9. IR ($CHCl_3$, cm^{-1}) 2957, 2930, 1464. HRMS (FAB, NBA) Calcd for $C_{20}H_{31}OSi$ $[M+H]^+$: 315.2144. Found: 315.2121.



anti-**4m**

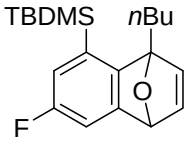
1-*tert*-Butyl-5-*tert*-butyldimethylsilyl-1,4-epoxy-1,4-dihydronaphthalene *anti*-4m (Table 2, entry 10). Following general procedure, a mixture of **1e** (105 mg, 0.25 mmol), 2-*tert*-butylfuran **2a** (0.11 mL, 0.75 mmol), 1.6 M *n*BuLi in hexane (0.30 mL, 0.50 mmol) in toluene (1.3 mL) was stirred for 20 min at –78 °C. The crude product was purified by column chromatography (hexane/EtOAc = 50 : 1 then 40 : 1) to provide the titled compound *anti*-**4m** as a yellow solid (50 mg, 63%). Mp 54–58 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.40 (d, *J* = 7.5 Hz, 1H), 7.05–6.91 (m, 4H), 5.76 (d, *J* = 2.0 Hz, 1H), 1.28 (s, 9H), 0.91 (s, 9H), 0.33 (s, 3H), 0.30 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 158.9, 147.7, 144.2, 142.9, 130.5, 128.7, 123.1, 122.1, 98.8, 82.3, 32.5, 26.6, 26.5, 17.2, –4.4, –4.8. IR ($CHCl_3$, cm^{-1}) 2957, 2930, 1464. HRMS (FAB, NBA) Calcd for $C_{20}H_{31}OSi$ $[M+H]^+$: 315.2144. Found: 315.2144.



anti-**4n**

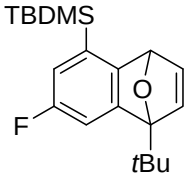
1-*n*-Butyl-5-*tert*-butyldimethylsilyl-1,4-epoxy-7-fluoro-1,4-dihydronaphthalene *anti*-4n (Table 2, entry 11). Following general procedure, a mixture of **1f** (131 mg, 0.30 mmol), 2-*n*-butylfuran **2c** (0.43 mL, 3.0 mmol), 1.6 M *n*BuLi in hexane (0.21 mL, 0.33

mmol) in toluene (1.5 mL) was stirred for 30 min at –78 °C. The crude product (*anti*-**4n**/*syn*-**4n** = 10 : 1) was purified by column chromatography (hexane/EtOAc = 25 : 1) to provide the titled compound *anti*-**4n** as a pale yellow oil (53 mg, 53% total isolated yield of *anti*-**4n** and *syn*-**4n**). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.00 (dd, *J* = 2.0, 5.5 Hz, 1H), 6.85 (dd, *J* = 2.5, 7.5 Hz, 1H), 6.75 (d, *J* = 5.5 Hz, 1H), 6.68 (dd, *J* = 2.5, 10.0 Hz, 1H), 5.71 (d, *J* = 2.0 Hz, 1H), 2.32–2.16 (m, 2H), 1.62–1.44 (m, 4H), 0.97 (t, *J* = 8.5 Hz, 3H), 0.90 (s, 9H), 0.31 (s, 3H), 0.28 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 189.9 (d, *J* = 245 Hz), 152.8 (d, *J* = 8 Hz), 144.6, 144.2, 130.9 (d, *J* = 4 Hz), 115.3 (d, *J* = 19 Hz), 108.9 (d, *J* = 25 Hz), 92.2, 82.4, 28.8, 26.7, 26.4, 23.1, 17.1, 14.0, –4.6, –5.1. IR ($CHCl_3$, cm^{-1}) 2957, 1456. HRMS (FAB, NBA) Calcd for $C_{20}H_{30}FOSi$ $[M+H]^+$: 333.2044. Found: 333.2050.



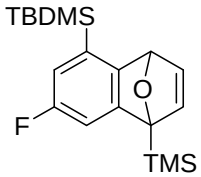
syn-**4n**

1-*n*-Butyl-8-*tert*-butyldimethylsilyl-1,4-epoxy-6-fluoro-1,4-dihydronaphthalene *syn*-4n (Table 2, entry 11) was obtained via column chromatography of the above-mentioned crude product as a colorless solid. Mp 55–56 °C. ^{1}H NMR (500 MHz, $CDCl_3$) *δ*: 6.98 (brd, *J* = 5.5 Hz, 1H), 6.92 (dd, *J* = 2.0, 7.0 Hz, 1H), 6.83–6.80 (m, 2H), 5.56 (d, *J* = 2.0 Hz, 1H), 2.59–2.21 (m, 1H), 2.18–2.16 (m, 1H), 1.64–1.60 (m, 1H), 1.45–1.43 (m, 3H), 0.98 (t, *J* = 7.5 Hz, 3H), 0.92 (s, 9H), 0.37 (s, 3H), 0.36 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 159.1 (d, *J* = 246 Hz), 153.6 (d, *J* = 8 Hz), 153.2, 144.6, 144.0, 131.6 (d, *J* = 4 Hz), 117.6 (d, *J* = 19 Hz), 109.3 (d, *J* = 25 Hz), 95.0, 80.8 (d, *J* = 2 Hz), 31.6, 27.5, 27.2, 23.1, 17.6, 14.1, –1.1, –2.0. IR (neat, cm^{-1}): 2959, 1460, 1242. HRMS (FAB, NBA) Calcd for $C_{20}H_{30}OFSi$ $[M+H]^+$: 333.2050. Found 333.2060.



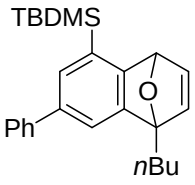
anti-**4o**

1-*tert*-Butyl-5-*tert*-butyldimethylsilyl-1,4-epoxy-7-fluoro-1,4-dihydronaphthalene *anti*-4o (Table 2, entry 12). Following general procedure, a mixture of **1f** (44 mg, 0.10 mmol), 2-*tert*-butylfuran **2a** (0.14 mL, 1.0 mmol), 1.6 M *n*BuLi in hexane (6.8×10^{-2} mL, 0.11 mmol) in toluene (0.5 mL) was stirred for 15 min at –78 °C. The crude product was purified by column chromatography (hexane/EtOAc = 50 : 1) to provide the titled compound *anti*-**4o** as a pale yellow oil (21 mg, 63%). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.09 (dd, *J* = 2.0, 9.0 Hz, 1H), 6.99 (dd, *J* = 2.0, 5.5 Hz, 1H), 6.94 (d, *J* = 5.5 Hz, 1H), 6.67 (dd, *J* = 2.0, 10.0 Hz, 1H), 5.72 (d, *J* = 2.0 Hz, 1H), 1.25 (s, 9H), 0.90 (s, 9H), 0.31 (s, 3H), 0.28 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 159.3 (d, *J* = 243 Hz), 153.9, 151.0 (d, *J* = 8 Hz), 144.7, 142.5, 130.3, 115.1 (d, *J* = 20 Hz), 111.3 (d, *J* = 25 Hz), 98.9, 82.0, 32.4, 26.48, 26.45, 17.2, –4.5, –4.9. IR ($CHCl_3$, cm^{-1}) 2957, 1508. HRMS (FAB, NBA) Calcd for $C_{19}H_{26}FOSi$ $[M–CH_3]^+$: 317.1737. Found: 317.1751.



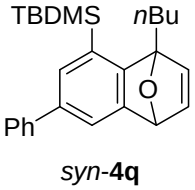
anti-**4p**

5-*tert*-butyldimethylsilyl-1,4-epoxy-7-fluoro-1-trimethylsilyl-1,4-dihydronaphthalene *anti*-4p (Table 2, entry 13). Following general procedure, a mixture of **1f** (131 mg, 0.30 mmol), 2-(trimethylsilyl)furan **2e**[4] (0.48 mL, 3.0 mmol), 1.6 M *n*BuLi in hexane (0.21 mL, 0.33 mmol) in toluene (1.5 mL) was stirred for 20 min at –78 °C. The crude product was purified by column chromatography (hexane/EtOAc = 50 : 1) to provide the titled compound *anti*-**4p** as a colorless solid (51 mg, 49%). Mp 72–73 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.02 (dd, *J* = 1.5, 5.5 Hz, 1H), 6.94 (d, *J* = 5.5 Hz, 1H), 6.87 (dd, *J* = 2.0, 7.5 Hz, 1H), 6.66 (dd, *J* = 2.0, 10.0 Hz, 1H), 5.77 (d, *J* = 1.5 Hz, 1H), 0.90 (s, 9H), 0.31 (s, 3H), 0.29 (s, 9H), 0.28 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ:159.4 (d, *J* = 244 Hz), 154.2 (d, *J* = 8 Hz), 152.4, 145.4, 143.5, 131.0 (d, *J* = 4 Hz), 114.9 (d, *J* = 19 Hz), 109.9 (d, *J* = 25 Hz), 85.6, 83.8, 26.5, 17.1, –3.2, –4.6, –5.0. IR ($CHCl_3$, cm^{-1}) 2928, 1454. Anal. Calcd for $C_{19}H_{29}FOSi_2$: C, 65.46; H, 8.38. Found: C, 65.34; H, 8.19.



anti-**4q**

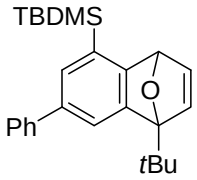
1-*n*-Butyl-5-*tert*-butyldimethylsilyl-1,4-epoxy-7-phenyl-1,4-dihydronaphthalene *anti*-4q (Table 2, entry 14). Following general procedure, a mixture of **1g** (148 mg, 0.30 mmol), 2-*n*-butylfuran **2c** (0.13 mL, 0.90 mmol), 1.6 M *n*BuLi in hexane (0.37 mL, 0.60 mmol) in toluene (1.5 mL) was stirred for 20 min at –78 °C. The crude product (*anti*-**4q**/*syn*-**4q** = 6.2 : 1) was purified by column chromatography (hexane/EtOAc = 40 : 1) to provide the titled compound *anti*-**4q** (62 mg, 53%) as a colorless oil (62% total isolated yield of *anti*-**4q** and *syn*-**4q**). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.54 (d, *J* = 7.5 Hz, 2H), 7.44 (t, *J* = 7.5 Hz, 2H), 7.36 (d, *J* = 1.5 Hz, 1H), 7.34 (t, *J* = 7.5 Hz, 1H), 7.24 (d, *J* = 1.5 Hz, 1H), 7.04 (dd, *J* = 1.5, 6.0 Hz, 1H), 6.81 (d, *J* = 6.0 Hz, 1H), 5.80 (d, *J* = 1.5 Hz, 1H), 2.43–2.37 (m, 1H), 2.31–2.25 (m, 1H), 1.69–1.58 (m, 2H), 1.53–1.46 (m, 2H), 0.99 (t, *J* = 7.5 Hz, 3H), 0.94 (s, 9H), 0.37 (s, 3H), 0.34 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 156.8, 150.3, 144.6, 144.1, 141.8, 137.0, 129.8, 129.3, 128.7, 127.4, 127.0, 119.4, 92.3, 82.6, 28.9, 26.8, 26.5, 23.2, 17.2, 14.1, –4.6, –4.9. IR (neat, cm^{-1}): 2953, 2930, 2857, 1454. HRMS (FAB, NBA) Calcd for $C_{26}H_{34}OSi$ $[M]^+$: 390.2379. Found 390.2375.



syn-**4q**

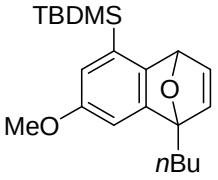
1-*n*-Butyl-8-*tert*-butyldimethylsilyl-1,4-epoxy-6-phenyl-1,4-dihydronaphthalene *syn*-4q (Table 2, entry 14) was obtained via column chromatography of the above-mentioned crude product as a colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) *δ*: 7.51 (d, *J* = 7.5 Hz, 2H), 7.42 (t, *J* = 7.5 Hz, 2H), 7.42 (s, 1H), 7.37 (d, *J* = 1.5 Hz, 1H), 7.33 (t, *J* = 7.5 Hz, 1H), 7.03 (d, *J* = 6.0 Hz, 1H), 6.83 (d, *J* = 6.0 Hz, 1H), 5.65 (d, *J* = 1.5 Hz, 1H), 2.64–2.58 (m, 1H), 2.25–2.19 (m, 1H), 1.67–1.62 (m, 1H), 1.50–1.45 (m, 3H), 0.98 (t, *J* = 7.5 Hz, 3H), 0.95 (s, 9H), 0.41 (s, 3H), 0.39 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 157.2, 151.2, 144.5, 144.1, 141.4, 136.3, 132.1, 129.9, 128.7, 127.2, 127.0, 119.6, 95.2, 81.1, 31.7, 27.6,

27.3, 23.1, 17.7, 14.1, –0.9, –1.9. IR (neat, cm^{-1}): 2957, 2930, 2857, 1462. HRMS (FAB, NBA) Calcd for $C_{26}H_{35}OSi$ $[M+H]^+$: 391.2457. Found 391.2431.



anti-**4r**

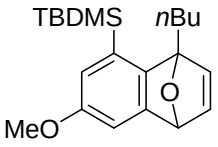
1-*tert*-Butyl-5-*tert*-butyldimethylsilyl-1,4-epoxy-7-phenyl-1,4-dihydronaphthalene *anti*-4r (Table 2, entry 12). Following general procedure, a mixture of **1g** (50 mg, 0.10 mmol), 2-*tert*-butylfuran **2a** (38 mg, 0.30 mmol), 1.6 M *n*BuLi in hexane (0.13 mL, 0.20 mmol) in toluene (0.5 mL) was stirred for 20 min at –78 °C. The crude product was purified by column chromatography (hexane/EtOAc = 50 : 1) to provide the titled compound *anti*-**4r** as a colorless oil (23 mg, 58%). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.59 (s, 1H), 7.52 (d, *J* = 7.5 Hz, 2H), 7.44 (t, *J* = 7.5 Hz, 1H), 7.34 (t, *J* = 7.5 Hz, 1H), 7.22 (d, *J* = 1.5 Hz, 1H), 7.03 (dd, *J* = 1.5, 6.0 Hz, 1H), 7.01 (d, *J* = 6.0 Hz, 1H), 5.80 (d, *J* = 1.5 Hz, 1H), 1.32 (s, 9H), 0.94 (s, 9H), 0.37 (s, 3H), 0.34 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 158.1, 148.7, 144.2, 142.9, 142.0, 136.5, 129.7, 128.9, 128.7, 127.4, 127.0, 121.7, 98.9, 82.2, 32.6, 26.7, 26.5, 17.2, –4.4, –4.8. IR (neat, cm^{-1}): 2955, 2930, 2857, 1462. HRMS (FAB, NBA) Calcd for $C_{26}H_{34}OSi$ $[M]^+$: 390.2379. Found 390.2378.



anti-**4s**

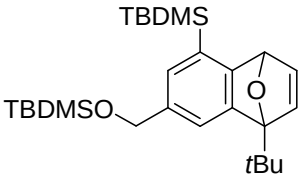
1-*n*-Butyl-5-*tert*-butyldimethylsilyl-1,4-epoxy-7-methoxyl-1,4-dihydronaphthalene *anti*-4s (Table 2, entry 16). Following general procedure, a mixture of **1h** (119 mg, 0.30 mmol), 2-*n*-butylfuran **2c** (0.13 mL, 0.90 mmol), 1.6 M *n*BuLi in hexane (0.37 mL, 0.60 mmol) in toluene (1.5 mL) was stirred for 45 min at –78 °C. The crude product (*anti*-**4s**/*syn*-**4s** = 6.3 : 1) was purified by column chromatography (hexane/EtOAc = 20 : 1) to provide the titled compound *anti*-**4s** (38 mg, 37%) as a brown oil (62% total isolated

yield of *anti*-**4s** and *syn*-**4s**). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.00 (dd, *J* = 1.5, 5.5 Hz, 1H), 6.78 (d, *J* = 2.5 Hz, 1H), 6.74 (d, *J* = 5.5 Hz, 1H), 6.49 (d, *J* = 2.5 Hz, 1H), 5.71 (d, *J* = 1.5 Hz, 1H), 3.79 (s, 3H), 2.34–2.28 (m, 1H), 2.23–2.17 (m, 1H), 1.66–1.43 (m, 4H), 0.97 (t, *J* = 7.0 Hz, 3H), 0.91 (s, 9H), 0.32 (s, 3H), 0.29 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 156.2, 151.8, 149.3, 144.6, 143.0, 129.8, 113.1, 108.7, 92.2, 82.4, 55.4, 28.9, 26.7, 26.5, 23.2, 17.1, 14.0, –4.5, –5.0. IR ($CHCl_3$, cm^{-1}) 2961, 2928, 1607, 1462, 1435. HRMS (FAB, NBA) Calcd for $C_{21}H_{32}O_2Si$ $[M]^+$: 345.2250. Found: 345.2276.



syn-**4s**

1-*n*-Butyl-8-*tert*-butyldimethylsilyl-1,4-epoxy-6-methoxyl-1,4-dihydronaphthalene *syn*-4s (Table 1, entry 16) was obtained via column chromatography of the above-mentioned crude product as a brown oil (6.0 mg, 6.0%). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 6.97 (dd, *J* = 2.5, 5.5 Hz, 1H), 6.85 (d, *J* = 2.5 Hz, 1H), 6.80 (d, *J* = 5.5 Hz, 1H), 6.63 (d, *J* = 2.5 Hz, 1H), 5.55 (d, *J* = 2.5 Hz, 1H), 3.77 (s, 3H), 2.58–2.52 (m, 1H), 2.21–2.15 (m, 1H), 1.49–1.42 (m, 2H), 1.31–1.25 (m, 2H), 0.97 (t, *J* = 7.5 Hz, 3H), 0.92 (s, 9H), 0.36 (s, 3H), 0.35 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 155.4, 152.7, 149.6, 144.7, 143.7, 130.5, 116.1, 108.7, 95.0, 81.1, 55.3, 31.7, 27.6, 27.2, 17.6, 14.1, –1.0, –2.0. IR ($CHCl_3$, cm^{-1}) 3007, 2957, 1620, 1471. HRMS (FAB, NBA) Calcd for $C_{21}H_{32}O_2Si$ $[M]^+$: 345.2250. Found: 345.2235.

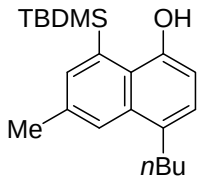


anti-**4t**

1-*tert*-Butyl-5-*tert*-butyldimethylsilyl-1,4-epoxy-7-(*tert*-butyldimethylsilyloxy)methyl-1,4-dihydronaphthalene *anti*-4t (Table 2, entry 17). Following general procedure, a mixture of **1i** (53 mg, 0.094 mmol), 1.6 M *n*BuLi in hexane (0.12 mL, 0.19 mmol), 2-*tert*-butylfuran **2a** (35 mg, 0.28 mmol) in toluene (0.5 mL) was stirred for 20 min at –78 °C.

The crude product was purified by column chromatography (hexane/EtOAc = 50 : 1) to provide the titled compound *anti*-**4t** as a colorless solid (28 mg, 64%). Mp 76–77 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.38 (s, 1H), 6.96–6.97 (m, 2H), 6.94 (d, *J* = 7.5 Hz, 1H), 5.73 (d, *J* = 1.5 Hz, 1H), 4.69 (s, 2H), 1.27 (s, 9H), 0.93 (s, 9H), 0.89 (s, 9H), 0.31 (s, 3H), 0.28 (s, 3H), 0.081 (s, 3H), 0.079 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 157.8, 148.1, 144.3, 142.8, 136.1, 128.2, 127.9, 120.6, 98.8, 82.2, 65.1, 32.5, 26.6, 26.5, 25.9, 18.3, 17.2, –4.4, –4.8, –5.2. IR (neat, cm^{-1}): 2955, 2930, 2857, 1361. HRMS (FAB, NBA) Calcd for $C_{27}H_{47}O_2Si_2$ $[M+H]^+$: 459.3115. Found 459.3117.

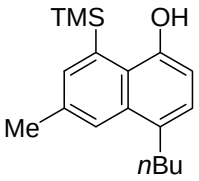
General Procedure for Regioselective Cleavage of 5-TBDMS-1,4-Dihydro-1,4-epoxynaphthalene *anti*-4 [Table 3 and Eq (2)]. A round bottom flask was charged with *anti*-**4** (1.0 equiv) and *p*TsOH·H_2O (0.5 equiv) then evacuated and back-filled with nitrogen. Anhydrous THF (0.15 M) was added via syringe and stirred at room temperature. After a few hours, a second portion of *p*TsOH·H_2O (0.5–2.0 equiv) was added into the reaction mixture and stirred for several hours (This portionwise addition was continued until *anti*-**4** was consumed as judged by TLC analysis). The reaction mixture was extracted with EtOAc (this process was repeated three times). The combined organic layers were dried over $MgSO_4$ and concentrated under reduced pressure. The crude product was purified by flash column chromatography on silica gel.



5a

4-*n*-Butyl-8-*tert*-butyldimethylsilyl-6-methyl-1-naphthol 5a (Table 3, entry 1)

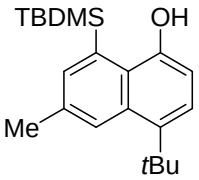
Following general procedure, a mixture of *anti*-**4f** (4.7×10^2 mg, 1.4 mmol) and *p*TsOH·H_2O (140 mg, 0.72 mmol) in anhydrous THF (10 mL) was stirred at room temperature for 12 h. *p*TsOH·H_2O (140 mg, 0.72 mmol) was added (6 h). The crude product was purified by flash column chromatography (hexane/EtOAc = 1 : 20) to provide the titled compound **5a** as a yellow oil (4.4×10^2 mg, 94%). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.81 (brs, 1H), 7.67 (d, *J* = 1.5 Hz, 1H), 7.11 (d, *J* = 7.5 Hz, 1H), 6.61 (d, *J* = 7.5 Hz, 1H), 5.13 (s, OH) , 2.99 (t, *J* = 7.5 Hz, 2H), 2.56 (s, 3H), 1.75–1.69 (m, 2H), 1.47 (hex, *J* = 7.5 Hz, 2H) , 1.00 (t, *J* = 7.5 Hz, 3H), 0.96 (s, 9H), 0.44 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 150.5, 137.6, 134.0, 133.6, 133.5, 130.9, 127.0, 125.3, 124.4, 107.5, 33.0, 32.8, 28.6, 22.8, 22.2, 18.6, 14.0, 0.2. IR ($CHCl_3$, cm^{-1}) 3412, 1568, 1462. HRMS (FAB, NBA) Calcd for $C_{20}H_{31}OSi$ $[M]^+$: 328.2222. Found: 328.2194.



5b

4-*n*-Butyl-6-methyl-8-trimethylsilyl-1-naphthol 5b (Table 3, entry 2)

Following general procedure, a mixture of *anti*-**4g** (50 mg, 0.17 mmol), *p*TsOH·H_2O (6.6 mg, 0.035 mmol) in anhydrous THF (5 mL) were stirred for 13 h. *p*TsOH·H_2O (6.6 mg, 0.035 mmol) was added (10 h). The crude product was purified by flash column chromatography (hexane/EtOAc = 50 : 1) to provide the titled compound **5b** as a yellow solid (29 mg, 59%). Mp 45–47 ^{o}C. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.84 (s, 1H), 7.65 (d, *J* = 1.5 Hz, 1H), 7.12 (d, *J* = 8.0 Hz, 1H), 6.63 (d, *J* = 8.0 Hz, 1H) , 5.22 (s, OH), 2.99 (t, *J* = 7.5 Hz, 2H), 2.55 (s, 3H), 1.71 (quint, *J* = 7.5 Hz, 2H), 1.46 (hex, *J* = 7.5 Hz, 2H), 0.99 (t, *J* = 7.5 Hz, 1H), 0.42 (s, 9H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 150.7, 136.00, 135.96, 134.4, 133.5, 131.2, 126.4, 125.4, 124.4, 107.8, 33.1, 32.8, 22.8, 22.1, 14.0, 2.3. IR ($CHCl_3$, cm^{-1}) 3510, 2959, 1667. HRMS (FAB, NBA) Calcd for $C_{18}H_{26}OSi$ $[M]^+$: 286.1753. Found: 286.1724.

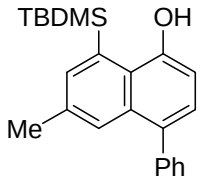


5c

4-*tert*-Butyl-8-*tert*-butyldimethylsilyl-6-methyl-1-naphthol 5c (Table 3, entry 3)

Following general procedure, a mixture of *anti*-**4d** (40 mg, 0.12 mmol), *p*TsOH·H_2O (23mg, 0.12 mmol) in anhydrous THF (1 mL) were stirred for 1 h. *p*TsOH·H_2O (12 mg, 0.060 mmol) was added (23 h). Then, 12 mg (0.060 mmol) was added (51 h). Then, 24 mg (0.12 mmol) was added (1 h). The crude product was purified by flash column chromatography (hexane/EtOAc = 7 : 1 then 5 : 1) to provide the titled compound **5c** as a brown oil (24 mg, 61%). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.02 (brs, 1H), 7.54 (s, 1H), 7.50 (s, OH), 7.25 (d, *J* = 10.5 Hz, 1H) , 6.46 (d, *J* = 10.5 Hz, 1H), 2.47 (s, 3H), 0.93 (s, 9H), 0.90 (s, 9H), 0.34 (s, 3H), 0.29 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 184.9,

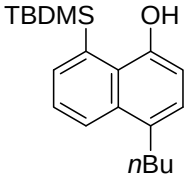
149.8, 142.0, 140.3, 139.9, 138.0, 136.0, 130.7, 127.5, 88.7, 41.3, 28.7, 25.9, 22.2, 18.8, –6.4, –2.4. IR ($CHCl_3$, cm^{-1}) 3520, 1672. HRMS (FAB, NBA) Calcd for $C_{19}H_{27}OSi$ $[M–C_2H_5]^+$: 299.1831. Found: 299.1822.



5d

8-*tert*-Butyldimehtylsilyl-6-methyl-4-phenyl-1-naphthol 5d (Table 3, entry 4)

Following general procedure, a mixture of *anti*-**4h** (30 mg, 0.086 mmol), *p*TsOH·H_2O (8.0 mg, 0.042 mmol) in anhydrous THF (1 mL) were stirred for 14 h. *p*TsOH·H_2O (16 mg, 0.084 mmol) was added (5 h). The crude product was purified by flash column chromatography (hexane/EtOAc = 20 : 1) to provide the titled compound **5d** as a colorless oil (26 mg, 87%). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.66 (brs, 1H), 7.62 (brs, 1H), 7.47–7.40 (m, 5H) , 7.19 (d, *J* = 7.5 Hz, 1H), 6.75 (d, *J* = 7.5 Hz, 1H), 5.38 (s, OH), 2.40 (s, 3H), 0.98 (s, 9H), 0.45 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 151.5, 141.6, 138.1, 134.5, 133.6, 133.2, 133.1, 130.4, 128.1, 126.70, 126.67, 126.63, 126.4, 107.1, 28.6, 21.9, 18.7, 0.2. IR ($CHCl_3$, cm^{-1}) 3588, 1599, 1574, 1502. HRMS (FAB, NBA) Calcd for $C_{23}H_{28}OSi$ $[M]^+$: 348.1909. Found: 348.1910.

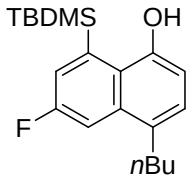


5e

4-*n*-Butyl-8-*tert*-butyldimethylsilyl-1-naphthol 5e (Table 3, entry 5)

Following general procedure, a mixture of *anti*-**4l** (30 mg, 0.096 mmol), *p*TsOH·H_2O (9.1 mg, 0.048 mmol) in anhydrous THF (1 mL) were stirred for 2.5 h. *p*TsOH·H_2O (9.1 mg, 0.048 mmol) was added (16 h). Then, 18 mg (0.096 mmol) was added (5 h). The crude product was purified by flash column chromatography (hexane/EtOAc = 20 : 1) to provide the titled compound **5e** as a colorless oil (26 mg, 87%). ^{1}H NMR (500 MHz,

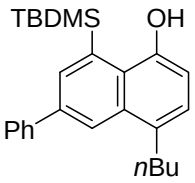
$CDCl_3$) δ: 8.03 (d, *J* = 8.0 Hz, 1H), 7.84 (d, *J* = 8.0 Hz, 1H), 7.48 (t, *J* = 8.0 Hz, 1H) , 7.14 (d, *J* = 7.5 Hz, 1H), 6.69 (d, *J* = 7.5 Hz, 1H), 5.18 (s, OH), 2.99 (t, *J* = 7.0 Hz, 2H), 1.71 (quint, *J* = 7.0 Hz, 2H), 1.45 (hex, *J* = 7.0 Hz, 2H), 0.97 (t, *J* = 7.0 Hz, 3H), 0.94 (s, 9H), 0.43 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 150.3, 135.4, 133.8, 133.2, 131.9, 128.9, 125.24, 125.20, 124.9, 108.5, 33.2, 32.9, 28.5, 22.9, 18.7, 14.0, 0.2. IR ($CHCl_3$, cm^{-1}) 3591, 1726, 1616, 1570. HRMS (FAB, NBA) Calcd for $C_{20}H_{30}OSi$ $[M]^+$: 314.2066. Found: 314.2053.



5f

4-*n*-Butyl-8-*tert*-butyldimethylsilyl-6-fluoro-1-naphthol 5f (Table 3, entry 6)

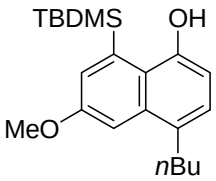
Following general procedure, a mixture of *anti*-**4n** (169 mg, 0.59 mmol), *p*TsOH·H_2O (56 mg, 0.30 mmol) in anhydrous THF (4 mL) were stirred for 12 h. *p*TsOH·H_2O (56 mg, 0.30 mmol) was added (4 h). Then, 112 mg (0.60 mmol) was added (32 h). The crude product was purified by flash column chromatography (hexane/EtOAc = 10 : 1) to provide the titled compound **5f** as a yellow oil (179 mg, 92%). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.62–7.58 (m, 2H), 7.16 (d, *J* = 7.5 Hz, 1H), 6.64 (d, *J* = 7.5 Hz, 1H), 5.55 (s, OH) , 2.92 (t, *J* = 7.5 Hz, 2H), 1.69 (quint, *J* = 7.5 Hz, 2H), 1.45 (hex, *J* = 7.5 Hz, 2H), 0.98 (t, *J* = 7.5 Hz, 3H), 0.95 (s, 9H), 0.44 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 160.0 (d, *J* = 246 Hz), 150.6, 138.6 (d, *J* = 5 Hz), 134.8 (d, *J* = 7 Hz), 131.2 (d, *J* = 6 Hz), 126.4, 125.9, 124.8 (d, *J* = 24 Hz), 108.5 (d, *J* = 20 Hz), 107.5, 32.9, 32.8, 28.5, 22.8, 18.6, 14.0, 0.1. IR ($CHCl_3$, cm^{-1}) 3590, 2930, 1574, 1470, 1327. HRMS (FAB, NBA) Calcd for $C_{20}H_{29}OFSi$ $[M]^+$: 332.1972. Found: 332.1968.



5g

4-*n*-Butyl-8-*tert*-butyldimethylsilyl-6-phenyl-1-naphthol 5g (Table 3, entry 7)

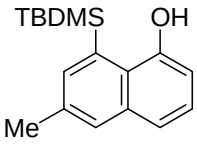
Following general procedure, a mixture of *anti*-**4r** (15 mg, 0.038 mmol), *p*TsOH·H_2O (3.7 mg, 0.019 mmol) in anhydrous THF (0.5 mL) were stirred for 12 h. *p*TsOH·H_2O (3.7 mg, 0.019 mmol) was added (12 h ×5). The crude product was purified by preparative TLC (hexane/EtOAc = 9 : 1) to provide the titled compound **5g** as a colorless oil (14.5 mg, 97%). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.20 (d, *J* = 1.5 Hz, 1H), 8.09 (d, *J* = 1.5 Hz, 1H), 7.72 (d, *J* = 7.5 Hz, 2H), 7.53 (t, *J* = 7.5 Hz, 2H), 7.41 (t, *J* = 7.5 Hz, 1H), 7.17 (d, *J* = 7.5 Hz, 1H), 6.70 (d, *J* = 7.5 Hz, 1H), 5.24 (s, OH), 3.05 (t, *J* = 7.5 Hz, 2H), 1.75 (quint, *J* = 7.5 Hz, 2H), 1.47 (hex, *J* = 7.5 Hz, 2H), 0.98 (t, *J* = 7.5 Hz, 3H) , 0.98 (s, 9H), 0.48 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 150.3, 142.0, 137.1, 135.1, 134.6, 133.5, 132.1, 128.9, 128.0, 127.6, 127.2, 125.7, 123.4, 108.6, 33.1, 32.9, 28.5, 22.8, 18.7, 14.1, 0.2. IR ($CHCl_3$, cm^{-1}) 3888, 1574, 1462. HRMS (FAB, NBA) Calcd for $C_{26}H_{34}OSi$ $[M]^+$: 390.2379. Found: 390.2370.



5h

4-*n*-Butyl-8-*tert*-butyldimethylsilyl-6-methoxy-1-naphthol 5h (Table 3, entry 8)

Following general procedure, a mixture of *anti*-**4s** (7.0 mg, 0.020 mmol), *p*TsOH·H_2O (1.9 mg, 0.010 mmol) in anhydrous THF (5 mL) were stirred for 12 h. *p*TsOH·H_2O (1.9 mg, 0.010 mmol) was added (12 h ×6). The crude product was purified by preparative TLC (hexane/EtOAc = 9 : 1) to provide the titled compound **5h** as a colorless oil (6.8 mg, 97%). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.48 (d, *J* = 2.0 Hz, 1H), 7.26 (d, *J* = 2.0 Hz, 1H), 7.10 (d, *J* = 7.5 Hz, 1H), 6.56 (d, *J* = 7.5 Hz, 1H), 5.12 (s, OH), 3.94 (s, 3H), 2.93 (t, *J* = 7.5 Hz, 2H), 1.71 (quint, *J* = 7.5 Hz, 2H), 1.45 (hex, *J* = 7.5 Hz, 2H), 0.98 (t, *J* = 7.5 Hz, 3H) , 0.93 (s, 9H), 0.41 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 156.3, 150.5, 136.3, 134.8, 130.5, 127.7, 125.9, 124.0, 106.5, 103.5, 55.0, 33.0, 32.5, 28.5, 22.8, 18.6, 14.0, 0.1. IR ($CHCl_3$, cm^{-1}) 3404, 1577, 1462. HRMS (FAB, NBA) Calcd for $C_{21}H_{32}O_2Si$ $[M]^+$: 344.2172. Found: 344.2176.

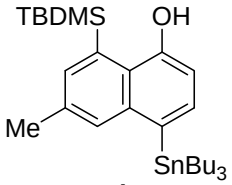


5i

8-*tert*-Butyldimethylsilyl-6-methyl-1-naphthol 5i [Eq (2)]

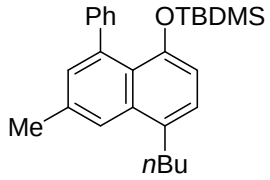
Following general procedure, a mixture of *anti*-**4i** (44 mg, 0.078 mmol), *p*TsOH·H_2O (10 mg, 0.053 mmol) in anhydrous THF (0.4 mL) were stirred for 2 h. *p*TsOH·H_2O (13 mg, 0.068 mmol) was added (1 h). The crude product was purified by flash column chromatography (hexane/EtOAc = 20 : 1) to provide the titled compound **5i** as a white solid (21 mg, 97%). Mp 94–97 ^{o}C. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.63 (s, 1H), 7.58 (s, 1H), 7.35 (d, *J* = 8.0 Hz, 1H), 7.25 (t, *J* = 8.0 Hz, 1H), 6.69 (d, *J* = 8.0 Hz, 1H), 5.22 (s, OH), 2.49 (s, 3H), 0.94 (s, 9H), 0.44 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 152.0, 138.2, 135.8, 134.4, 132.8, 128.5, 126.6, 125.3, 121.2, 108.4, 28.4, 21.7, 18.6, –0.11. IR ($CHCl_3$, cm^{-1}) 3584, 1568. HRMS (FAB, NBA) Calcd for $C_{16}H_{21}OSi$ $[M–CH_3]^+$: 257.1362. Found: 257.1322.

Procedure for Ru-Catalyzed Isomerization of 5-*tert*-Butyldimethylsilyl-1,4-dihydro-1-tributylstannyl-1,4-epoxynaphthalene *anti*-4j [Eq (3)].[6] A solution of *anti*-**4j** (304 mg, 0.53 mmol) and 1,2-dichloroethane (3 mL) in an oven-dried vial was added via a cannula to an oven-dried vial containing Cp*Ru(cod)Cl (30 mg, 0.079 mmol) (weighed out from a dry box) under argon. The reaction mixture was heated at 60 °C for 10 min and then allowed to cool to room temperature. The crude material was run through basic alumina (aluminium oxide 60; Merck) to obtain brown solid (304 mg). Into oven dried round bottom flask were added the solid product (44.1 mg, 0.079 mmol), activated silica gel (in the oven at 130 °C over night) and 1,2-dichloroethane (2.2 mL) and stirred for 10 min under argon at room temperature. The mixture was then filtered through a thin pad of Celite (eluting with EtOAc) and the eluent was concentrated under reduced pressure. The crude product was purified by flash column chromatography on silica gel (hexane/EtOAc = 30 : 1) to provide 8-*tert*-butyldimethylsilyl-6-methyl-4-tributylstannyl-1-naphthol **5j** (31.8 mg, 72%) as a yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.47 (brs, 1H ×2), 7.37 (d, *J* = 2.0 Hz, 1H), 7.18 (d, *J* = 2.0 Hz, 1H), 4.77 (s, 1H) , 2.47 (s, 3H), 1.62–1.51 (m, 2H), 1.35 (hex, *J* = 7.0 Hz, 2H), 1.26–1.11 (m, 2H), 0.95 (s, 9H), 0.89 (t, *J* = 7.0 Hz, 9H), 0.48 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 151.2, 145.8, 137.5, 137.1, 134.8, 134.4, 131.1, 131.9, 125.6, 111.8, 29.2, 27.4, 21.7, 18.0, 13.7, 10.5, –2.6. IR ($CHCl_3$, cm^{-1}) 3591, 1597. HRMS (FAB, NBA) Calcd for $C_{29}H_{50}OSi^{118}Sn$ $[M]^+$: 562.2653. Found:562.2697.



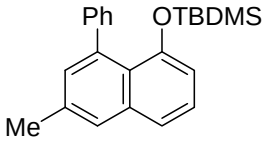
5j

General Procedure for Hiyama Coupling Reactions of 8-TBDMS-1-Naphthols 5 with Aryl Iodides (Table 4). An oven-dried reaction tube was charged with **5** (1.2 equiv), $AsPh_3$ (0.24 equiv) and Cs_2CO_3 (1.5 equiv). The reaction tube was capped with a rubber septum and then evacuated and back-filled with argon (this process was repeated twice). Anhydrous DME (0.1 M) was added via a syringe, through the septum, followed by the addition of an aryl iodide (1.0 equiv) and $[(allyl)PdCl]_2$ (0.06 equiv) (solid aryl halides were added with other reagents before the evacuation). The reaction mixture was heated to 60 °C until **5** was completely consumed as determined by a TLC analysis. At this point the reaction mixture was allowed to cool to room temperature. The reaction mixture was then filtered through a thin pad of Celite (eluting with EtOAc) and the eluent was concentrated under reduced pressure. The crude product was purified by flash column chromatography on silica gel.



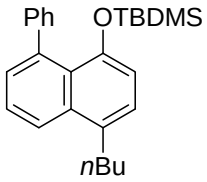
7a

***tert*-Butyldimethylsilyl 4-*n*-butyl-6-methyl-8-phenyl-1-naphthyl ether 7a (Table 4, entry 1).** Following general procedure, a mixture of **5e** (50 mg, 0.15 mmol), iodobenzene (14 μL, 0.13 mmol), Cs_2CO_3 (61 mg, 0.19 mmol), $[(allyl)PdCl]_2$ (2.8 mg, 7.7×10^{-3} mmol) and $AsPh_3$ (9.2 mg, 3.0×10^{-2} mmol) in DME (1.5 mL) was stirred for 2 h. The crude product was purified by column chromatography (hexane) to provide the titled compound **7a** as a pale yellow oil (39 mg, 77%). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.75 (brs, 1H), 7.34−7.25 (m, 5H), 7.14 (d, *J* = 7.5 Hz, 1H), 7.07 (d, *J* = 1.5 Hz, 1H), 6.72 (d, *J* = 7.5 Hz, 1H), 3.00 (t, *J* = 7.5 Hz, 2H), 2.51 (s, 3H), 1.76−1.71 (m, 2H), 1.53−1.45 (m, 2H), 0.99 (t, *J* = 7.5 Hz, 3H), 0.72 (s, 9H), −0.24 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 151.1, 145.4, 139.4, 134.5, 134.1, 131.9, 130.8, 129.2, 127.0, 126.0, 125.7, 123.3, 122.8, 113.2, 33.0, 32.9, 26.5, 22.9, 21.7, 19.1, 14.1, −4.3. IR ($CHCl_3$, cm^{-1}) 2957, 2930, 1612, 1581, 1462. HRMS (FAB, DTT/TG) Calcd for $C_{27}H_{37}OSi$ $[M+H]^+$: 405.2614. Found: 405.2609.



7b

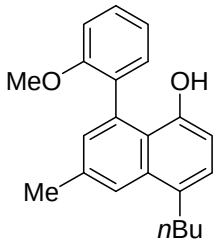
***tert*-Butyldimethylsilyl 6-methyl-8-phenyl-1-naphthyl ether 7b (Table 4, entry 2).** Following general procedure, a mixture of **5i** (12 mg, 0.045 mmol), iodobenzene (4.2 μL, 0.038 mmol), Cs_2CO_3 (18 mg, 0.056 mmol), $[(allyl)PdCl]_2$ (0.7 mg, 1.9×10^{-3} mmol) and $AsPh_3$ (2.4 mg, 7.5×10^{-3} mmol) in DME (0.4 mL) was stirred for 2.5 h. The crude product was purified by column chromatography (hexane) to provide the titled compound **7b** as a colorless solid (9.5 mg, 72%). Mp 83–86 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.57 (s, 1H), 7.42 (d, *J* = 7.5 Hz, 1H), 7.28–7.38 (m, 6H), 7.07 (s, 1H), 6.81 (d, *J* = 7.5 Hz, 1H), 2.48 (s, 3H), 0.74 (s, 9H), −0.23 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 152.7, 144.9, 138.8, 136.7, 134.6, 132.3, 129.3, 127.1, 126.8, 125.86, 125.81, 123.0, 120.9, 113.8, 26.4, 21.2, 19.1, –4.3. IR ($CHCl_3$, cm^{-1}) 1574. HRMS (FAB, NBA) Calcd for $C_{23}H_{29}OSi$ $[M+H]^+$: 349.1988. Found: 349.2024.



7c

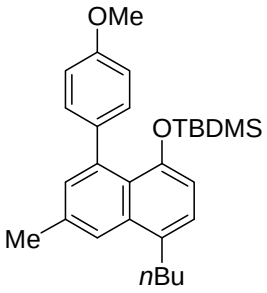
***tert*-Butyldimethylsilyl 4-*n*-butyl-8-phenyl-1-naphthyl ether 7c (Table 4, entry 3).** Following general procedure, a mixture of **5h** (19 mg, 0.061 mmol), iodobenzene (6.0 μL, 0.051 mmol), Cs_2CO_3 (40 mg, 0.12 mmol), $[(allyl)PdCl]_2$ (1.1 mg, 3.1×10^{-3} mmol) and $AsPh_3$ (3.7 mg, 1.2×10^{-2} mmol) in DME (0.6 mL) was stirred for 12 h. The crude product was purified by column chromatography (hexane) to provide the titled compound **7c** as a colorless oil (12 mg, 61%). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.00 (d, *J* = 8.0 Hz, 1H), 7.47 (t, *J* = 8.0 Hz, 1H), 7.36–7.27 (m, 5H), 7.23 (d, *J* = 8.0 Hz, 1H), 7.19 (d, *J* = 8.0 Hz, 1H), 6.79 (d, *J* = 8.0 Hz, 1H), 3.03 (t, *J* = 7.5 Hz, 2H), 1.74 (quint, *J* = 7.5 Hz, 2H), 1.47 (hex, *J* = 7.5 Hz, 2H), 0.99 (t, *J* = 7.5 Hz, 3H), 0.73 (s, 9H), −0.23 (s, 6H). ^{13}C

NMR (125 MHz, $CDCl_3$) δ: 151.1, 145.4, 139.5, 134.2, 131.6, 129.7, 129.2, 127.1, 125.9, 125.7, 125.0, 124.8, 123.6, 114.0, 33.10, 33.07, 26.5, 22.9, 19.1, 14.1, −4.3. IR ($CHCl_3$, cm^{-1}) 1582, 1458, 1410. HRMS (FAB, NBA) Calcd for $C_{26}H_{34}OSi$ $[M]^+$: 390.2379. Found: 390.2411.



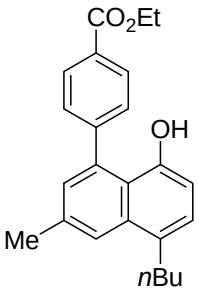
7d'

4-*n*-Butyl-6-methyl-8-(2-methoxyphenyl)-1-naphthol 7d' (Table 2, entry 4). Following general procedure, a mixture of **5a** (63 mg, 0.19 mmol), 2-iodoanisole (21 μL, 0.16 mmol), Cs_2CO_3 (78 mg, 0.24 mmol), $[(allyl)PdCl]_2$ (2.9 mg, 7.9×10^{-3} mmol) and $AsPh_3$ (9.8 mg, 3.2×10^{-2} mmol) in DME (2 mL) was stirred for 2 h. The crude product was purified by column chromatography (hexane) to provide *tert*-butyldimethylsilyl 4-*n*-butyl-6-methyl-8-(2-methoxyphenyl)-1-naphthyl ether **7d** with a small amount of impure. The mixture was stirred in 1.0 M tetrabutylammonium fluoride solution (0.20 mL, 0.20 mmol) in THF (1.0 mL) for 2 h. The crude product was purified by column chromatography (hexane/EtOAc = 10 : 1) to afford the titled compound **7d'** as a colorless oil (18.0 mg, 35%). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.81 (brs, 1H), 7.49–7.45 (m, 1H), 7.37 (dd, *J* = 2.0, 7.5 Hz, 1H), 7.18 (d, *J* = 7.5 Hz, 1H), 7.11–7.07 (m, 1H), 7.04 (d, *J* = 8.5 Hz, 1H), 7.02 (d, *J* = 2.0 Hz, 1H), 6.72 (d, *J* = 8.0 Hz, 1H), 5.51 (s, OH), 3.73 (s, 3H), 3.05–2.94 (m, 2H), 2.53 (s, 3H), 1.73 (quint, *J* = 7.0 Hz, 2H), 1.48 (hex, *J* = 7.0 Hz, 2H), 0.99 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 157.1, 151.8, 134.1, 133.6, 132.8, 131.3, 130.4, 130.3, 130.2, 129.9, 126.7, 123.7, 120.9, 120.7, 111.5, 109.8, 55.8, 33.00, 32.97, 22.9, 21.8, 14.1. IR ($CHCl_3$, cm^{-1}) 3499, 1609, 1462. HRMS (FAB, NBA) Calcd for $C_{22}H_{24}O_2$ $[M]^+$: 315.2144. Found: 315.2121.



7e

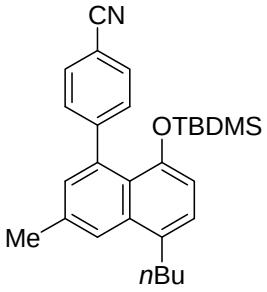
***tert*-Butyldimethylsilyl 4-*n*-butyl-8-(4-methoxyphenyl)-6-methyl-1-naphthyl ether 7e (Table 3, entry 5).** Following general procedure, a mixture of **5a** (50 mg, 0.15 mmol), 4-iodoanisole (30 mg, 0.13 mmol), Cs_2CO_3 (62 mg, 0.19 mmol), $[(allyl)PdCl]_2$ (2.8 mg, 7.7×10^{-3} mmol) and $AsPh_3$ (9.2 mg, 3.0×10^{-2} mmol) in DME (1.5 mL) was stirred for 8 h. The crude product was purified by column chromatography (hexane/EtOAc = 20 : 1) to provide the titled compound **7e** as a pale yellow oil (33 mg, 60%). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.73 (brs, 1H), 7.28 (d, *J* = 9.0 Hz, 2H), 7.14 (d, *J* = 8.0 Hz, 1H), 7.07 (d, *J* = 1.0 Hz, 1H), 6.88 (d, *J* = 9.0 Hz, 2H), 6.72 (d, *J* = 8.0 Hz, 1H), 3.85 (s, 3H), 3.00 (t, *J* = 8.0 Hz, 2H), 2.51 (s, 3H), 1.73 (quint, *J* = 8.0 Hz, 2H), 1.47 (hex, *J* = 8.0 Hz, 2H), 0.99 (t, *J* = 8.0 Hz, 3H), 0.73 (s, 9H), −0.21 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 158.0, 151.1, 139.0, 138.0, 134.6, 134.2, 131.8, 130.9, 130.1, 125.9, 123.4, 122.5, 113.3, 112.6, 55.5, 33.0, 32.9, 26.4, 22.9, 21.7, 19.0, 14.1, –4.2. IR ($CHCl_3$, cm^{-1}) 2957, 2930, 1609, 1582, 1516. HRMS (FAB, DTT/TG) Calcd for $C_{28}H_{39}O_2Si$ $[M+H]^+$: 435.2719. Found: 435.2717.



7f'

4-*n*-Butyl-8-(4-ethoxycarbonylphenyl)-6-methyl-1-naphthol 7f' (Table 3, entry 6). Following general procedure, a mixture of **5a** (50 mg, 0.15 mmol), ethyl 4-iodobenzoate (21 μL, 0.12 mmol), Cs_2CO_3 (62 mg, 0.19 mmol), $[(allyl)PdCl]_2$ (2.8 mg, 7.7×10^{-3} mmol)

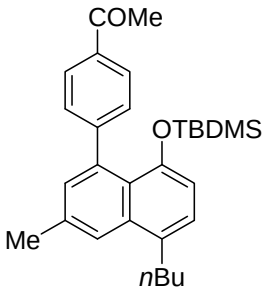
and $AsPh_3$ (9.2 mg, 3.0×10^{-2} mmol) in DME (1.5 mL) was stirred for 2 h. The crude product was purified by column chromatography (hexane/EtOAc = 20 : 1) to provide *tert*-butyldimethylsilyl 4-*n*-butyl-8-(4-ethoxycarbonylphenyl)-6-methyl-1-naphthyl ether **7f** with a small amount of 4-iodobenzoate. The mixture was stirred in 1.0 M tetrabutylammonium fluoride solution (0.30 mL, 0.30 mmol) in THF (1 mL) for 3 h. The crude product was purified by column chromatography (hexane/EtOAc = 1 : 10) to afford the titled compound **7f'** as a colorless solid (33 mg, 72%). Mp 116–121 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.15 (d, *J* = 8.0 Hz, 2H), 7.84 (brs, 1H), 7.55 (d, *J* = 8.0 Hz, 2H), 7.22 (d, *J* = 8.0 Hz, 1H), 7.05 (d, *J* = 1.5 Hz, 1H), 6.75 (d, *J* = 8.0 Hz, 1H), 4.89 (s, OH), 4.43 (q, *J* = 7.5 Hz, 2H), 3.01 (t, *J* = 7.5 Hz, 2H), 2.54 (s, 3H), 1.72 (quint, *J* = 7.5 Hz, 2H), 1.51–1.42 (m, 5H), 0.99 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 166.2, 150.9, 147.2, 136.1, 134.2, 133.9, 130.8, 130.3, 129.9, 129.6, 129.3, 127.0, 124.0, 119.8, 110.5, 61.2, 33.02, 32.95, 22.8, 21.7, 14.3, 14.0. IR ($CHCl_3$, cm^{-1}) 3543, 1714, 1607. HRMS (FAB, Gly) Calcd for $C_{24}H_{27}O_3$ $[M+H]^+$: 363.1960. Found: 363.1952.



7g

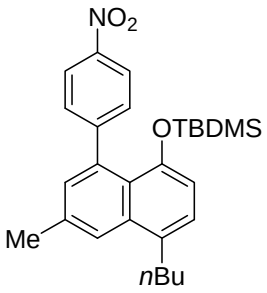
***tert*-Butyldimethylsilyl 4-*n*-butyl-8-(4-cyanophenyl)-6-methyl-1-naphthyl ether 7g (Table 3, entry 7).** Following general procedure, a mixture of **5a** (50 mg, 0.15 mmol), 4-iodobenzonitrile (29 mg, 0.13 mmol), Cs_2CO_3 (62 mg, 0.19 mmol), $[(allyl)PdCl]_2$ (2.8 mg, 7.7×10^{-3} mmol) and $AsPh_3$ (9.2 mg, 3.0×10^{-2} mmol) in DME (1.5 mL) was stirred for 2 h. The crude product was purified by column chromatography (hexane/EtOAc = 30 : 1) to provide the titled compound **7g** as a yellow solid (44 mg, 81%). Mp 99–101 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.83 (s, 1H), 7.63 (d, *J* = 8.5 Hz, 2H), 7.47 (d, *J* = 8.5 Hz, 2H), 7.19 (d, *J* = 8.0 Hz, 1H), 7.01 (s, 3H), 6.76 (d, *J* = 8.0 Hz, 1H), 3.02 (t, *J* = 7.5 Hz, 2H), 2.54 (s, 3H), 1.74 (quint, *J* = 7.5 Hz, 2H), 1.48 (hex, *J* = 7.5 Hz, 2H), 1.00 (t, *J* = 7.5

Hz, 3H), 0.73 (s, 9H), −0.21 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 150.6, 150.3, 137.2, 134.4, 134.2, 131.6, 131.2, 130.9, 129.9, 126.4, 123.9, 122.7, 119.5, 113.4, 109.2, 32.93, 32.89, 26.3, 22.8, 21.7, 19.0, 14.0, −4.3. IR ($CHCl_3$, cm^{-1}) 2957, 2930, 2226, 1605, 1582. Anal. Calcd for $C_{28}H_{35}NOSi$: C, 78.27; H, 8.21; N, 3.26. Found: C, 78.12; H, 8.49; N, 3.31.



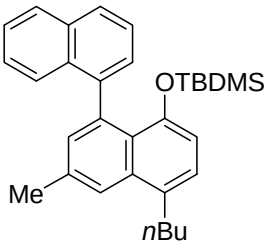
7h

***tert*-Butyldimethylsilyl 8-(4-acetylphenyl)-4-*n*-butyl-6-methyl-1-naphthyl ether 7h (Table 3, entry 8).** Following general procedure, a mixture of **5a** (50 mg, 0.15 mmol), 4-iodoacetophenone (31 μL, 0.13 mmol), Cs_2CO_3 (61 mg, 0.19 mmol), $[(allyl)PdCl]_2$ (2.8 mg, 7.7×10^{-3} mmol) and $AsPh_3$ (9.2 mg, 3.0×10^{-2} mmol) in DME (1.5 mL) was stirred for 2 h. The crude product was purified by column chromatography (hexane/EtOAc = 10 : 1) to provide the titled compound **7h** as a pale yellow oil (39 mg, 68%). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.95 (d, *J* = 8.0 Hz, 2H), 7.80 (brs, 1H), 7.46 (d, *J* = 8.0 Hz, 2H), 7.18 (d, *J* = 8.0 Hz, 1H), 7.07 (d, *J* = 1.5 Hz, 1H), 6.75 (d, *J* = 8.0 Hz, 1H), 3.01 (t, *J* = 7.5 Hz, 2H), 2.65 (s, 3H), 2.53 (s, 3H), 1.74 (quint, *J* = 7.5 Hz, 2H), 1.48 (hex, *J* = 7.5 Hz, 2H), 1.00 (t, *J* = 7.5 Hz, 3H), 0.71 (s, 9H), −0.25 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 198.1, 150.8, 150.6, 138.0, 134.6, 134.5, 134.2, 131.6, 131.0, 129.4, 127.3, 126.2, 123.5, 123.0, 113.3, 33.0, 32.9, 26.7, 26.3, 22.8, 21.7, 19.0, 14.1, –4.3. IR ($CHCl_3$, cm^{-1}) 2957, 2930, 1676, 1603. HRMS (FAB, DTT/TG) Calcd for $C_{29}H_{39}O_2Si$ $[M+H]^+$: 447.7219. Found: 447.2722.



7i

***tert*-Butyldimethylsilyl 4-*n*-butyl-6-methyl-8-(4-nitrophenyl)-1-naphthyl ether 7i (Table 3, entry 9).** Following general procedure, a mixture of **5a** (50 mg, 0.15 mmol), 4-iodonitrobenzene (32 mg, 0.13 mmol), Cs_2CO_3 (61 mg, 0.19 mmol), $[(allyl)PdCl]_2$ (2.8 mg, 7.7×10^{-3} mmol) and $AsPh_3$ (9.2 mg, 3.0×10^{-2} mmol) in DME (1.5 mL) was stirred for 7 h. The crude product was purified by column chromatography (hexane) to provide the titled compound **7i** as a yellow oil (29 mg, 55%). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.21 (d, *J* = 8.5 Hz, 2H), 7.83 (brs, 1H), 7.51 (d, *J* = 8.5 Hz, 2H), 7.19 (d, *J* = 7.0 Hz, 1H), 7.06 (d, *J* = 1.5 Hz, 1H), 6.77 (d, *J* = 7.0 Hz, 1H), 3.02 (t, *J* = 7.5 Hz, 2H), 2.54 (s, 1H), 1.74 (quint, *J* = 7.5 Hz, 2H), 1.48 (hex, *J* = 7.5 Hz, 2H), 1.00 (t, *J* = 7.5 Hz, 3H), 0.71 (s, 9H), −0.23 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 152.4, 150.5, 146.0, 136.8, 134.4, 134.2, 131.6, 131.3, 129.9, 126.4, 124.1, 122.7, 122.4, 113.5, 33.0, 32.9, 26.3, 22.8, 21.7, 19.0, 14.1, –4.3. IR ($CHCl_3$, cm^{-1}) 2957, 2930, 1595, 1582, 1516, 1344. HRMS (FAB, DTT/TG) Calcd for $C_{27}H_{36}NO_3Si$ $[M+H]^+$: 450.2464. Found: 450.2468.

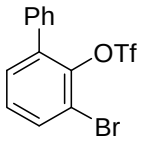


7j

***tert*-Butyldimethylsilyl 4-*n*-butyl-6-methyl-8-(1-naphthyl)-1-naphthyl ether 7j (Table 2, entry 10).** Following general procedure, a mixture of **5a** (50 mg, 0.15 mmol), 4-iodobenzonitrile (19 μL, 0.13 mmol), Cs_2CO_3 (62 mg, 0.19 mmol), $[(allyl)PdCl]_2$ (2.8 mg, 7.7×10^{-3} mmol) and $AsPh_3$ (9.2 mg, 3.0×10^{-2} mmol) in DME (1.5 mL) was stirred for 1.5

h. The crude product was purified by column chromatography (hexane/EtOAc = 40 : 1) to provide the titled compound **7j** as a colorless solid (28 mg, 48%). Mp 121–123 ^{o}C. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.87−7.81 (m, 3H), 7.49 (t, *J* = 7.5 Hz, 1H), 7.42−7.36 (m, 3H), 7.24 (dt, *J* = 1.0, 7.5 Hz, 1H), 7.18 (d, *J* = 7.5 Hz, 1H), 7.10 (d, *J* = 1.0 Hz, 1H), 6.65 (d, *J* = 7.5 Hz, 1H), 3.10−3.02 (m, 2H), 2.53 (s, 3H), 1.85−1.78 (m, 2H), 1.54 (hex, *J* = 7.5 Hz, 2H), 1.05 (t, *J* = 7.5 Hz, 3H), 0.55 (s, 9H), −0.38 (s, 3H), −0.63 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 151.2, 144.2, 173.3, 134.4, 134.1, 133.3, 133.2, 132.0, 130.4, 127.7, 126.9, 126.1, 125.9, 125.5, 125.3, 125.2, 125.1, 124.6, 123.1, 112.0, 33.0, 32.9, 26.3, 23.0, 21.7, 19.0, 14.1, −4.0, −4.9. IR ($CHCl_3$, cm^{-1}) 2957, 2930, 1578, 1462. Anal. Calcd for $C_{31}H_{38}OSi$: C, 81.88; H, 8.42. Found: C, 81.58; H, 8.59.

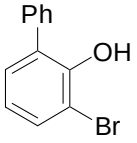
Procedure for the Synthesis of Benzyne Precursors 1a and b



1a

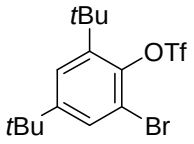
3-Bromobiphenyl-2-yl trifluoromethanesulfonate 1a (Table 1, entry 1).

Bromine (1.7 mL, 34 mmol) was added dropwise to a solution of dimethylamine (4.1 mL, 36 mmol) and NaOH (2.8 g, 69 mmol) in H_2O (15 mL) at –15 ° C. The mixture was stirred for 30 min at this temperature and extracted with toluene (200 mL). The organic layer was separated and dried with $MgSO_4$ to give a yellow solution of *N*-bromodimethylamine, which was added to a solution of 2-hydroxybiphenyl (5.0 g, 29 mmol) in toluene (30 mL) at –15 °C. The mixture was stirred for 4 h, slowly warm up to room temperature, and stirred overnight, then acidified with 6 M HCl (17 mL). The crude product was purified by flash column chromatography (hexane/CH_2Cl_2 = 2 : 1) to provide 2-bromo-6-phenylphenol (1.7 g, 23%).[7] ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.54 (dd, *J* = 1.5, 7.5 Hz, 2H), 7.49−7.37 (m, 4H), 7.25 (dd, *J* = 1.5, 7.5 Hz, 1H), 6.89 (t, *J* = 7.5 Hz, 1H), 5.68 (s, OH).



An oven-dried round bottom flask was charged with 3-bromo-2-hydroxybiphenyl[7] (1.7 g, 6.8 mmol). The flask was capped with an inlet adapter with a three-way stopcock and then evacuated and back-filled with nitrogen. Anhydrous CH_2Cl_2 (6.8 mL) and *N*,*N*-diisopropylethylamine (1.7 mL, 10 mmol) were added via syringe, followed by the slow addition of trifluoromethanesulfonc anhydride (1.7 mL, 10 mmol) over five minutes under 0 °C. After 30 min, the reaction mixture was slowly allowed to warm up to room temperature. After several hours stirring, saturated $NaHCO_3$ solution was added to the reaction and extracted with CH_2Cl_2 (this process was repeated three times). The organic layers were dried over magnesium sulfate and concentrated under reduced pressure. The crude product was purified by flash column chromatography (hexane/EtOAc = 20 :

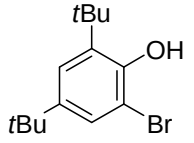
1) to provide titled compound **1a** as a colorless solid (2.6 g, 99%). Mp 71–72 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.67 (dd, *J* = 1.0, 7.8 Hz, 1H), 7.48−7.41 (m, 5H), 7.40 (dd, *J* = 1.0, 8.0 Hz, 1H), 7.28 (t, *J* = 8.0 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 144.4, 138.04, 138.03, 135.6, 133.3, 131.3, 129.3, 129.2, 128.7, 128.59, 128.57, 117.9 (q, *J* = 321 Hz), 117.4. IR ($CHCl_3$, cm^{-1}) 1420. Anal. Calcd for $C_{13}H_8BrF_3O_3S$: C, 40.96; H, 2.12. Found: C, 41.06; H, 2.32.



1b

2-Bromo-4,6-di-*tert*-butylphenyl trifluoromethanesulfonate 1b (Table 1, entry 2).

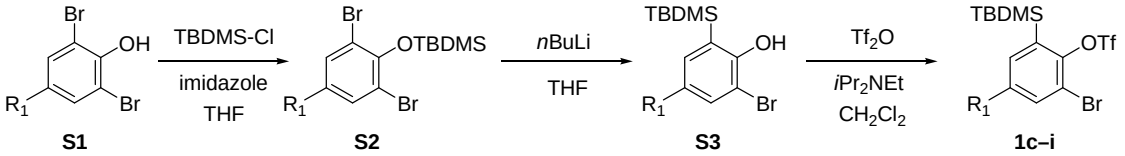
A solution of bromine (0.50 mL, 9.7 mmol) in CH_2Cl_2 (10 mL) was added dropwise to a solution of 2,4-Di-*tert*-butylphenol (2.0 g, 9.7 mmol) in CH_2Cl_2 (2 mL) over 10 min at 0 °C. The mixture was stirred for 1 h at this temperature. The organic layer was separated and washed six times with water, dried with $MgSO_4$. The crude product was purified by flash column chromatography (hexane) to provide 2-bromo-4,6-di-*tert*-butytlphenol (2.2 g, 80%).[8] ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 7.32 (d, *J* = 2.0 Hz, 1H), 7.24 (d, *J* = 2.0 Hz, 1H), 5.64 (s, 1H), 1.40 (s, 9H), 1.28 (s, 9H).



An oven-dried round bottom flask was charged with 2-bromo-4,6-di-*tert*-butylphenol (1.0 g, 3.5 mmol). The flask was capped with an inlet adapter with a three-way stopcock and then evacuated and back-filled with nitrogen. Anhydrous CH_2Cl_2 (17.5 mL) and *N*,*N*-diisopropylethylamine (1.2 mL, 7.0 mmol) were added via syringe, followed by the slow addition of trifluoromethanesulfonc anhydride (0.88 mL, 5.3 mmol) over five minutes under 0 °C. After 30 min, the reaction mixture was slowly allowed to warm up to room temperature. After several hours stirring, saturated $NaHCO_3$ solution was added to the reaction and extracted with CH_2Cl_2 (this process was repeated three times). The organic layers were dried over sodium sulfate and concentrated under reduced pressure. The

crude product was purified by flash column chromatography (hexane) to provide titled compound **1b** as a pale yellow oil (1.1 g, 75%). ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 7.51 (d, *J* = 2.5 Hz, 1H), 7.48 (d, *J* = 2.5 Hz, 1H), 1.47 (s, 9H), 1.31 (s, 9H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ: 151.9, 145.4, 141.1, 130.1, 126.3, 118.7 (q, *J* = 320 Hz), 117.0, 36.6, 34.8, 31.5, 31.1. IR (neat): 2968, 1416 cm^{-1}. HRMS (FAB) calcd for $C_{15}H_{21}{}^{79}BrF_3O_3S$: 417.0347 $[M+H]^+$; found 417.0327.

General procedure for the Synthesis of Silylated Benzyne Precursors 1c–i[1] (Table 1 and 2).



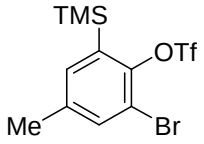
Scheme S1

General procedure I: An oven-dried round bottom flask was charged with 2,4-dibromophenol derivative **S1** (1.0 equiv) and imidazole (1.5 equiv). The flask was capped with a rubber septum and then evacuated and back-filled with nitrogen. Anhydrous THF was added via syringe, through the septum, followed by the addition of *tert*-butyldimethylsilyl chloride (1.5 equiv) at room temperature. After several hours, water was added to the reaction mixture and extracted with hexane (this process was repeated three times). The organic layers were dried over magnesium sulfate and concentrated under reduced pressure. The crude product was purified by flash column chromatography to provide *tert*-butyldimethylsilyl 2,4-dibromophenyl ether **S2**.

General procedure II: S2 was loaded into a round bottom flask. The flask was capped with an inlet adapter with a three-way stopcock and then evacuated and back-filled with nitrogen. Anhydrous THF was added via syringe, followed by the slow addition of 1.6 M *n*BuLi in hexane (1.0 equiv) over five minutes at –78 °C. After 30 min, mixture was allowed to warm up to room temperature. After several hours stirring, water was added to the reaction mixture and extracted with hexane (this process was repeated three times). The organic layers were dried over magnesium sulfate and concentrated under reduced pressure. The crude product was purified by flash column chromatography to provide 2-bromo-6-(*tert*-butyldimethylsilyl)phenol **S3**.

General procedure III: S3 was loaded into a round bottom flask. The flask was capped with an inlet adapter with a three-way stopcock and then evacuated and back-filled with nitrogen. Anhydrous CH_2Cl_2 and *N*,*N*-diisopropylethylamine (1.5 equiv) were added via syringe, followed by the slow addition of trifluoromethanesulfonic anhydride (1.5 equiv) over five minutes at 0 °C. After 30 min, the mixture was slowly allowed to warm up to room temperature and stirred for several hours. Saturated $NaHCO_3$ solution was added to the reaction mixture and extracted with CH_2Cl_2 (this

process was repeated three times). The organic layers were dried over magnesium sulfate and concentrated under reduced pressure. The crude product was purified by flash column chromatography to provide 2-bromo-6-(*tert*-butyldimethylsilyl)phenyl trifluoromethanesulfonate **1c–h**.

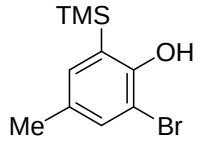


1c

2-Bromo-4-methyl-6-(trimethylsilyl)phenyl trifluoromethanesulfonate 1c[1] (Table 2, entry 3).

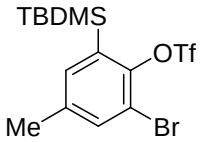
An oven-dried round bottom flask was charged with 2,6-dibromo-4-methylphenol **S1a** (1.3 g, 5.0 mmol). The flask was connected with condenser and septum then evacuated and back-filled with nitrogen. Anhydrous THF (2.5 mL) and hexamethyldisilazane (3.1 mL, 15 mmol) was added via syringe, through the septum. The reaction mixture was stirred for 22 h at 80 °C. After the reflux condenser was changed to inlet adapter with a three-way stopcock, residual solvent and reagent was removed by high vacuum pump to provide 2,6-dibromo-4-methylphenyl trimethylsilyl ether **2c** in the flask. The crude product was used without purification and/or determination of the structure.

Following general procedure II, a mixture of **S2c**, 1.6 M *n*BuLi hexane solution (4.7 mL, 5.0 mmol) in THF (10 mL) was stirred for 30 min at –78 °C and for 9 h at room temperature. The crude product was purified by column chromatography (hexane/EtOAc = 20 : 1) to provide 2-bromo-6-trimethylsilyl-4-methylphenol **S3c**[1] as a colorless oil (1.1 g, 83%). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.28 (d, *J* = 1.0 Hz, 1H), 7.07 (d, *J* = 1.0 Hz, 1H), 5.53 (s, OH), 2.26 (s, 3H), 0.30 (s, 9H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 153.9, 135.1, 133.1, 130.9, 126.6, 110.1, 20.2, –1.1. IR ($CHCl_3$, cm^{-1}) 3524, 2957, 1450. Anal. Calcd for $C_{10}H_{15}BrOSi$: C, 46.33; H, 5.83. Found: C, 46.55; H, 5.59.



S3c

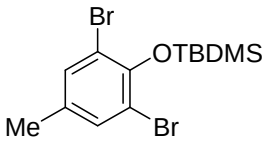
Following general procedure III, a mixture of **S3c** (5.0 g, 19 mmol), trifluoromethanesulfonic anhydride (4.9 mL, 29 mmol) and *N*,*N*-diisopropylethylamine (5.0 mL, 29 mmol) in THF (50 mL) was stirred for 1 h at 0 °C and for 13 h at room temperature. The crude product was purified by column chromatography (hexane/EtOAc = 10 : 1) to provide the titled compound **1c**[1] as a pale yellow oil (6.9 g, 91%). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.48 (s, 1H), 7.25 (s, 1H), 2.35 (s, 3H), 0.39 (s, 9H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 146.6, 139.3, 137.0, 136.2, 136.1, 118.6 (q, J = 318.7 Hz), 115.9, 20.5, 0.0. IR ($CHCl_3$, cm^{-1}) 1404. Anal. Calcd for $C_{11}H_{14}BrF_3O_3SSi$: C, 33.77; H, 3.61. Found: C, 33.97; H, 3.58.



1d

2-Bromo-6-*tert*-butyldimethylsilyl-4-methylphenyl trifluoromethanesulfonate 1c[1] (Table 1, entry 3 and Table 2, entries 1, 2, 4, 5).

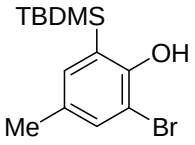
Following general procedure I, a mixture of 2,6-dibromo-4-methylphenol **S1c** (11 g, 40 mmol), *tert*-butyldimethylsilyl chloride (9.0 g, 60 mmol) in THF (50 mL) was stirred over night. The crude product was purified by column chromatography (hexane) to provide *tert*-butyldimethylsilyl 2,6-dibromo-4-methylphenyl ether **S2c**[1] as a colorless oil (15 g, 99%). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.28 (s, 2H), 2.24 (s, 3H), 1.05 (s, 9H), 0.34 (s, 6H).



S2d

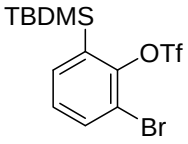
Following general procedure II, a mixture of **S2c** (15 g, 40 mmol), 1.6 M *n*BuLi hexane solution (25 mL, 40 mmol) in THF (50 mL) was stirred for 15 min at –78 °C and for 12 h at room temperature. The crude product was purified by column chromatography (hexane) to provide 2-bromo-6-*tert*-butyldimethylsilyl-4-methylphenol **S3c**[1] as a colorless oil (12 g, 97%). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.28 (s, 1H), 7.07 (s, 1H),

5.54 (s, OH), 2.26 (s, 3H), 0.90 (s, 9H), 0.31 (s, 6H).



S3d

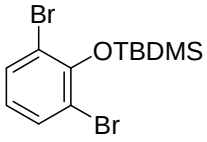
Following general procedure III, a mixture of **S3c** (8.0 g, 28 mmol), trifluoromethanesulfonic anhydride (9.3 mL, 55 mmol) and *N*,*N*-diisopropylethylamine (9.7 mL, 55 mmol) in CH_2Cl_2 (80 mL) was stirred for 30 min at 0 °C and for 17 h at room temperature. The crude product was purified by column chromatography (hexane) to provide the titled compound **1d**[1] as a colorless solid (8.4 g, 82%). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.49 (s, 1H), 7.28 (s, 1H), 2.35 (s, 3H), 0.90 (s, 9H), 0.40 (s, 6H).



1e

2-Bromo-6-(*tert*-butyldimethylsilyl)phenyl trifluoromethanesulfonate 1e (Table 2, entries 9 and 10).

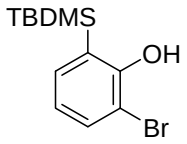
Following general procedure I, a mixture of 2,6-dibromophenol **S1e** (5.0 g, 20 mmol), *tert*-butyldimethylsilyl chloride (6.0 g, 40 mmol), imidazole (2.7 g, 40 mmol) in THF (66 mL) was stirred for 35 min. The crude product was purified by column chromatography (hexane) to provide *tert*-butyldimethylsilyl 2,6-dibromophenyl ether **S2e** (7.5 g, quant) as a colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.47 (d, J = 8.0 Hz, 2H), 6.70 (t, J = 8.0 Hz, 1H), 1.07 (s, 9H), 0.37 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 150.3, 132.8, 123.2, 116.0, 26.2, 19.0, –2.1. IR ($CHCl_3$, cm^{-1}) 2957, 2930, 1447. HRMS (FAB, NBA) Calcd for $C_{12}H_{19}{}^{79}Br^{81}BrOSi$ $[M+H]^+$: 366.9552. Found 366.9562.



S2e

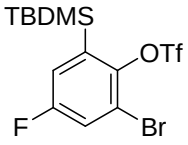
Following general procedure II, a mixture of **S2e** (7.0 g, 20 mmol), 1.6 M *n*BuLi hexane

solution (13 mL, 22 mmol) in THF (100 mL) was stirred for 15 min at –78 °C and for 70 min at room temperature. The crude product was purified by column chromatography (hexane) to provide 2-bromo-6-(*tert*-butyldimethylsilyl)phenol **S3e** as a colorless oil (5.3 g, 92%). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.46 (dd, *J* = 2.0, 8.0 Hz, 1H), 7.30 (dd, *J* = 2.0, 8.0 Hz, 1H), 6.79 (t, *J* = 8.0 Hz, 1H), 5.71 (s, OH), 0.91 (s, 9H), .0.32 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 156.2, 136.1, 133.0, 124.7, 121.3, 110.8, 26.9, 17.6, –4.8. IR ($CHCl_3$, cm^{-1}) 3516, 2955, 2928, 1584, 1425. Anal. Calcd for $C_{12}H_{19}BrOSi$: C, 50.17; H, 6.67. Found: C, 49.96; H, 6.70.



S3e

Following general procedure III, a mixture of **S3e** (5.3 g, 18 mmol), trifluoromethanesulfonic anhydride (5.4 mL, 32 mmol) and *N*,*N*-diisopropylethylamine (7.3 mL, 43 mmol) in THF (92 mL) was stirred for 30 min at 0 °C and for 17 h at room temperature. The crude product was purified by column chromatography (hexane/EtOAc = 50 : 1) to provide the titled compound **1e** as a pale yellow oil (4.7 g, 61%). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.68 (dd, *J* = 2.0, 7.5 Hz, 1H), 7.54 (dd, *J* = 2.0, 7.5 Hz, 1H), 7.22 (t, *J* = 7.5 Hz, 1H), 0.91 (s, 9H), 0.42 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 149.0, 136.7, 136.0, 135.6, 128.5, 118.1 (q, *J* = 321 Hz), 116.7, 27.1, 18.1, –3.6. IR ($CHCl_3$, cm^{-1}) 2959, 2932, 1406, 1390. Anal. Calcd for $C_{13}H_{18}BrF_3O_3SSi$: C, 37.24; H, 4.33. Found: C, 37.36; H, 4.33.

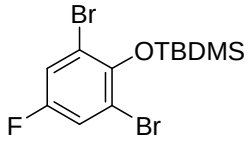


1f

2-Bromo-6-*tert*-butyldimethylsilyl-4-fluorophenyl trifluoromethanesulfonate 1f (Table 2, entries 11-13).

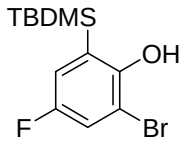
Following general procedure I, a mixture of 2,6-dibromo-4-fluorophenol **S1f** (4.9 g, 18 mmol), *tert*-butyldimethylsilyl chloride (4.1 g, 27 mmol), imidazole (4.1 g, 61 mmol) in

THF (70 mL) was stirred for 10 h. The crude product was purified by column chromatography (hexane) to provide *tert*-butyldimethylsilyl 2,6-dibromo-4-fluorophenyl ether **S2f** (6.5 g, 92%) as a colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.25 (d, *J* = 7.5 Hz, 2H), 1.05 (s, 9H), 0.34 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 156.0 (d, *J* = 247 Hz), 147.2, 119.7 (d, *J* = 25.0 Hz), 115.4 (d, *J* = 9.5 Hz), 26.2, 18.9, –2.1. IR ($CHCl_3$, cm^{-1}) 2932, 1460. Anal. Calcd for $C_{12}H_{17}Br_2FOSi$: C, 37.52; H, 4.46. Found: C, 37.45; H, 4.60.



S2f

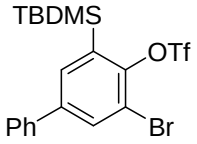
Following general procedure II, a mixture of **S2f** (6.5 g, 17 mmol), 1.6 M *n*BuLi hexane solution (10 mL, 17 mmol) in THF (40 mL) was stirred for 40 min at –78 °C and for 10 h at room temperature. The crude product was purified by column chromatography (hexane) to provide 2-bromo-6-*tert*-butyldimethylsilyl-4-fluorophenol **S3f** as a colorless oil (4.4 g, 85%). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.20 (dd, *J* = 3.0, 7.5 Hz, 1H), 7.01 (dd, *J* = 3.0, 7.5 Hz, 1H), 5.53 (s, OH), 0.91 (s, 9H), 0.31 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 155.8 (d, *J* = 243 Hz), 152.5, 125.9 (d, *J* = 4 Hz), 121.9 (d, *J* = 20 Hz), 119.4 (d, *J* = 26 Hz), 109.7 (d, *J* = 8 Hz), 26.9, 17.6, –5.0. IR ($CHCl_3$, cm^{-1}) 3524, 2928, 1447, 1400. Anal. Calcd for $C_{12}H_{18}FOSi$: C, 47.22; H, 5.94. Found: C, 47.55; H, 5.95.



S3f

Following general procedure III, a mixture of **S3f** (4.4 g, 14.2 mmol), trifluoromethanesulfonic anhydride (4.8 mL, 29 mmol) and *N*,*N*-diisopropylethylamine (5.3 mL, 30 mmol) in CH_2Cl_2 (40 mL) was stirred for 30 min at 0 °C and for 9 h at room temperature. The crude product was purified by column chromatography (hexane) to provide the titled compound **1f** as a pale yellow oil (3.2 g, 51%). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.42 (dd, *J* = 3.0, 7.5 Hz, 1H), 7.23 (dd, *J* = 3.0, 7.5 Hz, 1H), 0.91 (s, 9H), 0.42 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 160.0 (d, *J* = 254 Hz), 145.0 (d, *J* = 3 Hz),

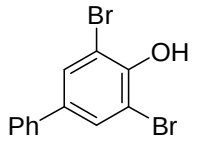
137.8 (d, J = 5 Hz), 122.8 (d, J = 26 Hz), 122.7 (d, J = 21 Hz), 118.6 (q, J = 320 Hz), 117.3 (d, J = 4 Hz), 27.1, 18.1, –3.7. IR ($CHCl_3$, cm^{-1}) 2959, 2932, 1570, 1410. Anal. Calcd for $C_{13}H_{17}BrF_4O_3SSi$: C, 35.70; H, 3.92. Found: C, 35.77; H, 3.95.



1g

2-Bromo-6-*tert*-butyldimethylsilyl-4-phenylphenyl trifluoromethanesulfonate (1g) (Table 2, entries 14 and 15).

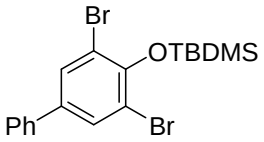
An oven-dried round bottom flask was charged with 4-phenylphenol (2.1 g, 12 mmol). The flask was capped with a rubber septum and then evacuated and back-filled with nitrogen. Anhydrous CH_2Cl_2 (50 mL) was added via syringe, through the septum, followed by the addition of *N*,*N*-diisopropylamine (0.18 mL, 0.12 mmol) at room temperature. *N*-Bromosuccinimide (4.2 g, 24 mmol) solution in CH_2Cl_2 (150 mL) was added dropwise and stirred at room temperature. After 6 hours, conc. H_2SO_4 and water was added to the reaction and extracted with CH_2Cl_2 (this process was repeated three times). The organic layers were dried over sodium sulfate and concentrated under reduced pressure. The crude product was recrystalyzed from hexane to provide 2,6-dibromo-4-phenylphenol (**S1g**)[9] as a yellow solid (1.1 g). Mother liquid was purified by column chromatography (hexane/EtOAc = 10 : 1) to provide desired product (0.62 g) (1.7 g, 44% total isolated yield). Mp 78–80 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.67 (s, 2H), 7.49 (d, J = 7.5 Hz, 2H), 7.43 (t, J = 7.5 Hz, 2H), 7.36 (t, J = 7.5 Hz, 1H), 5.89 (s, OH). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 148.6, 138.1, 136.2, 130.5, 129.0, 127.8, 126.7, 110.2. IR ($CHCl_3$, cm^{-1}) 3509, 1469. HRMS (FAB, NBA) Calcd for $C_{12}H_8Br_2O$ $[M]^+$: 325.8942. Found: 325.8936.



S1g

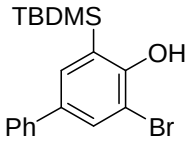
Following general procedure I, a mixture of **S1g** (1.7 g, 5.0 mmol), *tert*-

butyldimethylsilyl chloride (1.3 g, 7.5 mmol) in THF (25 mL) was stirred for 3 h. The crude product was purified by column chromatography (hexane/EtOAc = 10 : 1) to provide *tert*-butyldimethylsilyl 2,6-dibromo-4-phenylphenyl ether **S2g** as a colorless solid (2.2 g, 99%). Mp 99–100 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.72 (s, 2H), 7.51 (d, *J* = 7.5 Hz, 2H), 7.43 (t, *J* = 7.5 Hz, 2H), 7.35 (t, *J* = 7.5 Hz, 1H), 1.10 (s, 9H), 0.41 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 149.5, 138.1, 136.6, 131.2, 128.9, 127.7, 126.7, 116.1, 26.2, 19.0, –2.1. IR ($CHCl_3$, cm^{-1}) 2959, 2930, 1460. Anal. Calcd for $C_{18}H_{22}Br_2OSi$: C, 48.88; H, 5.01. Found: C, 48.65; H, 4.77.



S2g

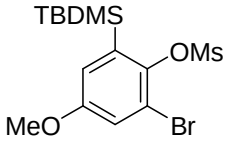
Following general procedure II, a mixture of **S2g** (2.1 g, 5.0 mmol), 1.6 M *n*BuLi hexane solution (3.0 mL, 5.0 mmol) in THF (25 mL) was stirred for 30 min at –78 °C and for 24 h at room temperature. The crude product was purified by column chromatography (hexane/EtOAc = 40 : 1) to provide 2-bromo-6-*tert*-butyldimethylsilyl-4-phenylphenol (**S3g**) as a colorless solid (1.6 g, 90%). Mp 66–67 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.70 (d, *J* = 2.0 Hz, 1H), 7.52–7.50 (m, 3H), 7.44 (t, *J* = 7.3 Hz, 2H), 7.34 (t, *J* = 7.3 Hz, 1H), 5.76 (s, OH), 0.95 (s, 9H), 0.37 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 155.6, 139.8, 134.7, 131.5, 128.8, 127.7, 126.82, 126.80, 124.9, 111.2, 26.9, 17.6, –4.8. IR ($CHCl_3$, cm^{-1}) 3516, 2955, 2928, 1443. Anal. Calcd for $C_{18}H_{23}BrOSi$: C, 59.50; H, 6.38. Found: C, 59.27; H, 6.09.



S3g

Following general procedure III, a mixture of **S3g** (1.6 g, 4.5 mmol), trifluoromethanesulfonic anhydride (1.1 mL, 6.8 mmol) and *N*,*N*-diisopropylethylamine (1.8 mL, 6.8 mmol) in CH_2Cl_2 (23 mL) was stirred for 30 min at 0 °C and for 24 h at room temperature. The crude product was purified by column chromatography (hexane/EtOAc = 40 : 1) to provide the titled compound **1g** as a brown solid (2.1 g, 97%).

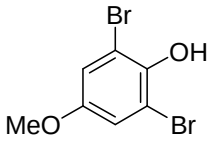
Mp 77–78 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.87 (d, J = 2.5 Hz, 1H), 7.68 (d, J = 2.5 Hz, 1H), 7.53–7.45 (m, 4H), 7.42–7.40 (m, 1H), 0.95 (s, 9H), 0.45 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 148.3, 141.7, 138.3, 135.6, 135.2, 134.4, 129.1, 128.4, 127.2, 118.7 (q, J = 322 Hz), 116.9, 27.2, 18.2, –3.5. IR ($CHCl_3$, cm^{-1}) 2957, 2930, 1404. Anal. Calcd for $C_{19}H_{22}Br\,F_3O_2SSi$: C, 49.10; H, 4.36. Found: C, 46.06; H, 4.48.



1h

2-Bromo-6-*tert*-butyldimethylsilyl-4-methoxyphenyl methanesulfonate 1h (Table 2, entry 16).

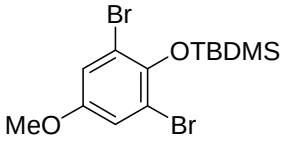
An oven-dried round bottom flask was charged with 4-methoxyphenol (0.77 g, 6.1 mmol). The flask was capped with a rubber septum and then evacuated and back-filled with nitrogen. Anhydrous CH_2Cl_2 (22 mL)/MeOH (8.7 mL) was added via syringe, through the septum, followed by the addition of benzyltrimethylammonium tribromide (5.0 g, 13 mmol) at 0 °C and stirred at room temperature. After 3 days, sat. $Na_2S_2O_3$ solution was added to the reaction mixture and extracted with CH_2Cl_2 (this process was repeated three times). The organic layers were dried over magnesium sulfate and concentrated under reduced pressure. The crude product was purified by column chromatography (hexane/EtOAc = 10 : 1) to provide 2,6-dibromo-4-methoxyphenol **S1h**[10] as a colorless solid (1.3 g, 79%). Mp 89–90 °C (Lit[10] 83–84 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.03, (s, 2H), 5.49 (s, OH), 3.75 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 153.6, 143.7, 117.7, 109.6, 56.1. IR ($CHCl_3$, cm^{-1}) 3518, 1479. Anal. Calcd for $C_7H_6Br_2O_2$: C, 29.91; H, 2.09. Found: C, 29.82; H, 2.15.



S1h

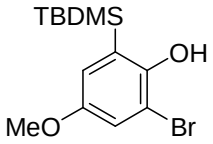
Following general procedure I, a mixture of 2,6-dibromo-4-methoxyphenol **S1h** (1.2 g, 4.0 mmol), *tert*-butyldimethylsilyl chloride (1.0 g, 6.0 mmol), imidazole (4.3×10^2 mg, 6.0

mmol) in THF (20 mL) was stirred for 3 h. The crude product was purified by column chromatography (hexane/EtOAc = 10 : 1) to provide *tert*-butyldimethylsilyl 2,6-dibromo-4-methoxyphenyl ether **S2h** (1.4 g, 89%) as a colorless solid. Mp 36–37 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.04 (s, 2H), 3.74 (s, 3H), 1.05 (s, 9H), 0.33 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 154.0, 144.3, 118.3, 115.6, 55.9, 26.2, 18.9, –2.2. IR ($CHCl_3$, cm^{-1}) 1470, 1435. Anal. Calcd for $C_{13}H_{20}Br_2O_2Si$: C, 39.36; H, 4.96. Found: C, 39.41; H, 5.09.



S2h

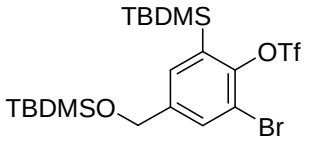
Following general procedure II, a mixture of **S2h** (1.4 g, 3.6 mmol), 1.6 M *n*BuLi hexane solution (2.2 mL, 3.6 mmol) in THF (18 mL) was stirred for 30 min at –78 °C and for 2 h at room temperature. The crude product was purified by column chromatography (hexane/EtOAc = 30 : 1) to provide 2-bromo-6-*tert*-butyldimethylsilyl-4-methoxyphenol **S3h** as a colorless oil (1.0 g, 90%). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.01 (d, J = 3.0 Hz, 1H), 6.88 (d, J = 3.0 Hz, 1H), 5.38 (s, OH), 3.75 (s, 3H), 0.91 (s, 9H), 0.31 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 152.9, 150.4, 125.3, 122.3, 117.1, 110.1, 55.9, 26.9, 17.6, –4.9. IR ($CHCl_3$, cm^{-1}) 3528, 2955, 2930, 1562, 1458. HRMS (FAB, NBA) Calcd for $C_{13}H_{21}BrO_2Si_2$ $[M]^+$: 316.0494. Found 316.0508.



S3h

Following general procedure III, a mixture of **S3h** (189 mg, 0.60 mmol), methanesulfonyl chloride (70 μL, 0.90 mmol) and triethylamine (0.25 mL, 1.8 mmol) in CH_2Cl_2 (3 mL) was stirred for 30 min at 0 °C and for 3 h at room temperature. The crude product was purified by column chromatography (hexane/EtOAc = 10 : 1) to provide the titled compound **1h** as a colorless solid (193 mg, 81%). Mp 99–100 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.13 (d, J = 3.0 Hz, 1H), 7.01 (d, J = 3.0 Hz, 1H), 3.80 (s, 3H), 3.43 (s, 3H), 0.90 (s, 9H), 0.41 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 157.2, 145.7, 138.6,

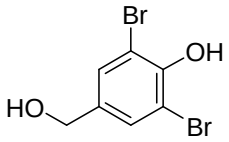
122.3, 119.3, 117.3, 55.7, 40.9, 27.2, 18.0, –3.6. IR ($CHCl_3$, cm^{-1}) 1475, 1371. Anal. Calcd for $C_{14}H_{23}BrO_4SSi$: C, 42.68; H, 5.72. Found: C, 42.53; H, 5.86.



1i

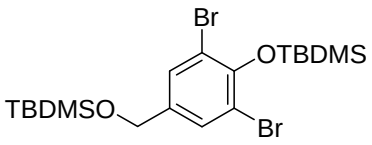
2-Bromo-6-*tert*-butyldimethylsilyl-4-(*tert*-butyldimethylsilyloxymethyl)phenyl trifluoromethanesulfonate 1i (Table 2, entry 17).

An oven-dried round bottom flask was charged with 3,5-dibromo-4-hydroxybenzaldehyde (5.0 g, 18 mmol) and sodium borohydride (3.0 g, 79 mmol). The flask was capped with a rubber septum and then evacuated and back-filled with nitrogen. Anhydrous ethanol was added via syringe, through the septum, and stirred at room temperature. After several hours, water was added to the reaction mixture and extracted with EtOAc (this process was repeated three times). The organic layers were dried over magnesium sulfate and concentrated under reduced pressure to provide 3,5-dibromo-4-hydroxybenzylalcohol **S1i**[11] as a colorless solid (5.1 g, quant).



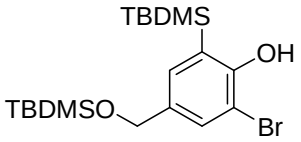
S1i

Following general procedure I, a mixture of **S1i**[11] (1.0 g, 3.6 mmol), *tert*-butyldimethylsilyl chloride (1.8 g, 12 mmol) in THF (10 mL) was stirred for 12 h. The crude product was purified by column chromatography (hexane) to provide *tert*-butyldimethylsilyl 2,6-dibromo-4-(*tert*-butyldimethylsilyloxymethyl)phenyl ether **S2i** as a colorless oil (1.5 g, 83 %). ^1H NMR (500 MHz, $CDCl_3$) δ: 7.41 (s, 2H), 4.60 (s, 2H), 1.05 (s, 9H), 0.93 (s, 9H), 0.35 (s, 6H), 0.10 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 149.0, 136.7, 130.3, 115.6, 63.3, 26.2, 25.9, 18.9, 18.4, –2.1, –5.3. IR (neat, cm^{-1}): 2954, 2930, 2859, 1462. HRMS (FAB, NBA) Calcd for $C_{19}H_{34}{}^{79}Br^{81}BrO_2Si_2$ $[M]^+$: 510.0445. Found 510.0422.



S2i

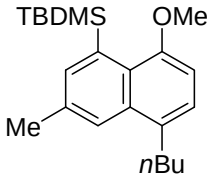
Following general procedure II, a mixture of **S2i** (1.1 g, 2.2 mmol), 1.6 M *n*BuLi hexane solution (1.5 mL, 2.4 mmol) in THF (10 mL) was stirred for 30 min at –78 °C. The crude product was purified by column chromatography (hexane) to provide 2-bromo-6-*tert*-butyldimethylsilyl-4-(*tert*-butyldimethylsilylmethyl)phenol **S3i** as a colorless solid (0.95 g, quant.). Mp 29–30 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.42 (s, 1H), 7.22 (s, 1H), 5.64 (s, OH), 4.63 (s, 2H), 0.93 (s, 9H), 0.89 (s, 9H), 0.30 (s, 6H), 0.09 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 155.1, 134.2, 133.8, 130.8, 124.2, 110.6, 64.1, 26.9, 25.9, 18.3, 17.6, –4.8, –5.2. IR (neat, cm^{-1}): 3518, 2953, 2927, 2857, 1452. HRMS (FAB, NBA) Calcd for $C_{19}H_{35}{}^{79}BrO_2Si_2$ $[M]^+$: 430.1359. Found 430.1336.



S3i

Following general procedure III, a mixture of **S3i** (0.80 g, 1.9 mmol), trifluoromethanesulfonic anhydride (0.47 mL, 2.8 mmol) and *N*,*N*-diisopropylethylamine (0.96 mL, 5.6 mmol) in THF (10 mL) was stirred for 30 min at 0 °C and for 12 h at room temperature. The crude product was purified by column chromatography (hexane/EtOAc = 50 : 1) to provide the titled compound **1i** (0.96 g, 92%) as a yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.59 (d, J = 2.5 Hz, 1H), 7.50 (d, J = 2.5 Hz, 1H), 4.71 (s, 2H), 0.95 (s, 9H), 0.90 (s, 9H), 0.40 (s, 6H), 0.11 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 147.7, 142.0, 135.0, 133.7, 133.1, 118.7 (q, J = 322 Hz), 116.3, 63.3, 27.1, 25.8, 18.3, 18.1, –3.6, –5.4. IR (neat, cm^{-1}): 2955, 2932, 2859, 1396. Anal. Calcd for $C_{20}H_{34}BrF_3O_4SSi_2$: C, 42.62; H, 6.08. Found: C, 42.66; H, 6.06.

Procedure for the Synthesis of Methyl Ether of 5a (ref. 13)



4-*n*-butyl-8-*tert*-butyldimethylsilyl-1-methoxy-6-methylnaphthalene

An oven-dried pear-shaped flask was charged with **5a** (127 mg, 0.39 mmol). The flask was capped with an inlet adapter with a three-way stopcock and then evacuated and back-filled with nitrogen. Anhydrous DMF (1.3 mL) were added via syringe, followed by the addition of 1.0 M *t*-BuOLi in THF (0.46 mL, 0.46 mmol) and MeI (24 μL, 0.39 mmol) under 0 °C. After 20 min, the mixture was slowly allowed to warm up to room temperature and stirred. After 2 hours stirring, 30% ammonia solution was added to the reaction and extracted with EtOAc (this process was repeated three times). The organic layers were dried over magnesium sulfate and concentrated under reduced pressure. The crude product was purified by flash column chromatography (hexane) to provide titled compound as a colorless solid (92 mg, 70%). Mp 75–77 ^{o}C. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.79 (s, 1H), 7.67 (d, J = 2.0 Hz, 1H), 7.19 (d, J = 8.0 Hz, 1H), 6.67 (d, J = 8.0 Hz, 1H), 3.88 (s, 3H), 2.98 (t, J = 8.0 Hz, 2H), 2.53 (s, 3H), 1.71 (quint, J = 8.0 Hz, 2H), 1.47 (hex, J = 8.0 Hz, 2H), 0.99 (t, J = 8.0 Hz, 3H), 0.92 (s, 9H), 0.39 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 154.3, 137.6, 133.9, 133.3, 133.1, 130.6, 128.1, 125.3, 124.4, 102.4, 54.0, 33.0, 32.8, 28.3, 22.9, 22.1, 18.9, 14.1, –0.1. IR ($CHCl_3$, cm^{-1}) 2957, 2930, 1574, 1464. Anal. Calcd for $C_{22}H_{34}OSi$: C, 77.13; H, 10.00. Found: C, 77.02; H, 10.02.

Experimental References:

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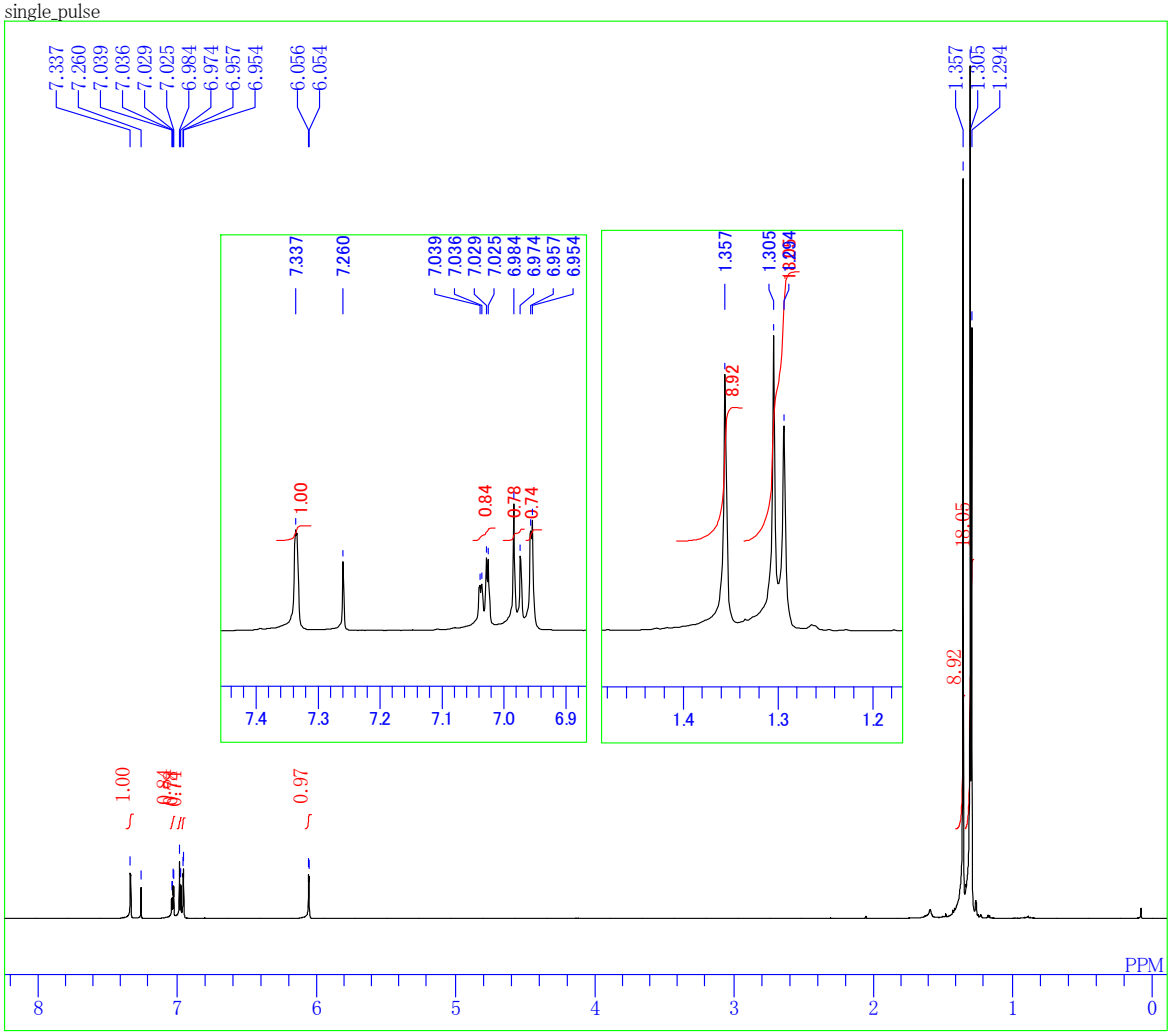
single_pulse



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EXMOD	single_pulse.ex2
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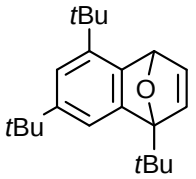
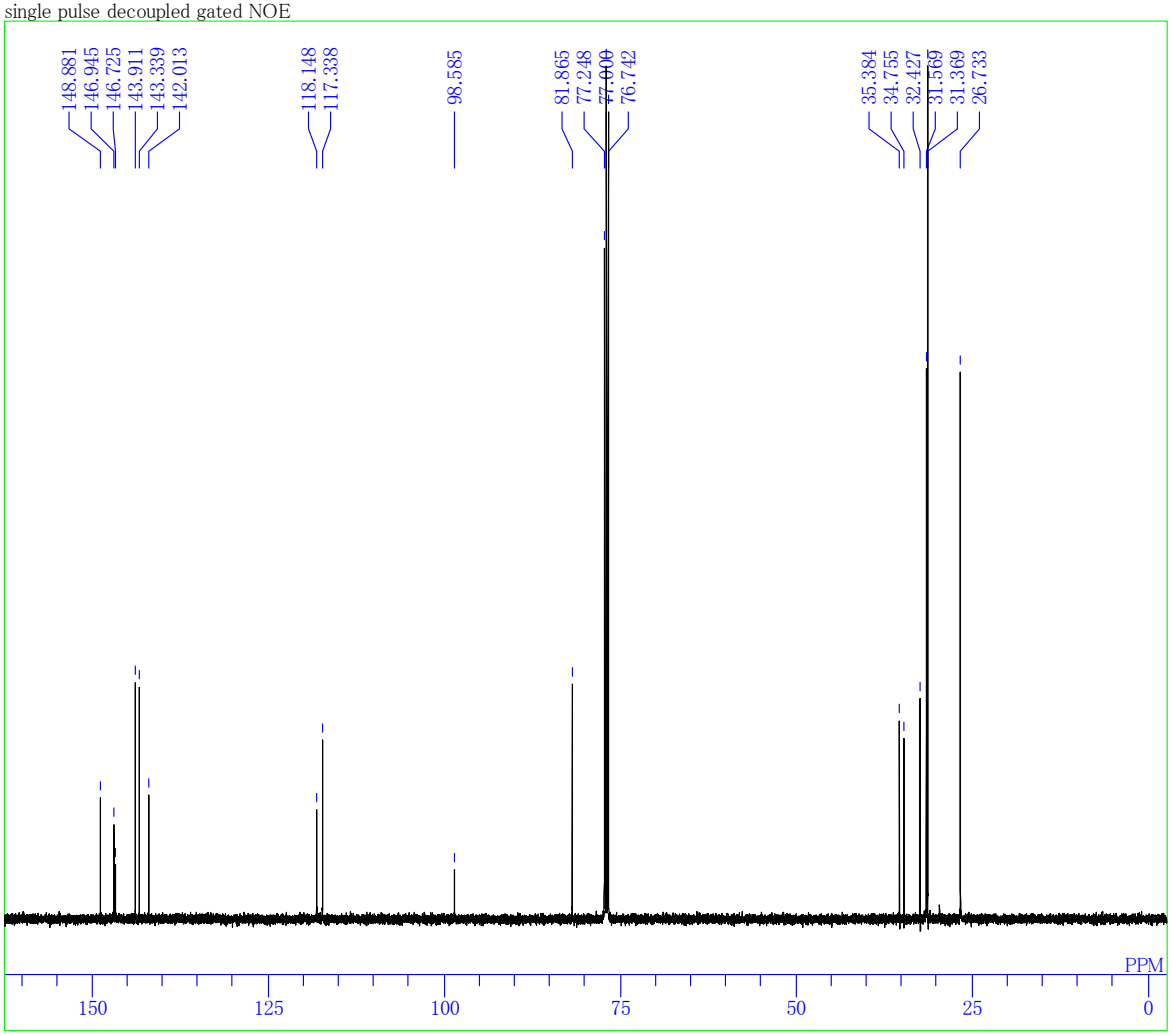
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	20480
FREQU	11730.66 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	6.00 usec
IRNUC	1H
CTEMP	18.0 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	44



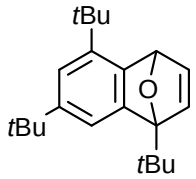
anti-**4b** ($CDCl_3$)

Table 1, entry 2

single pulse decoupled gated NOE



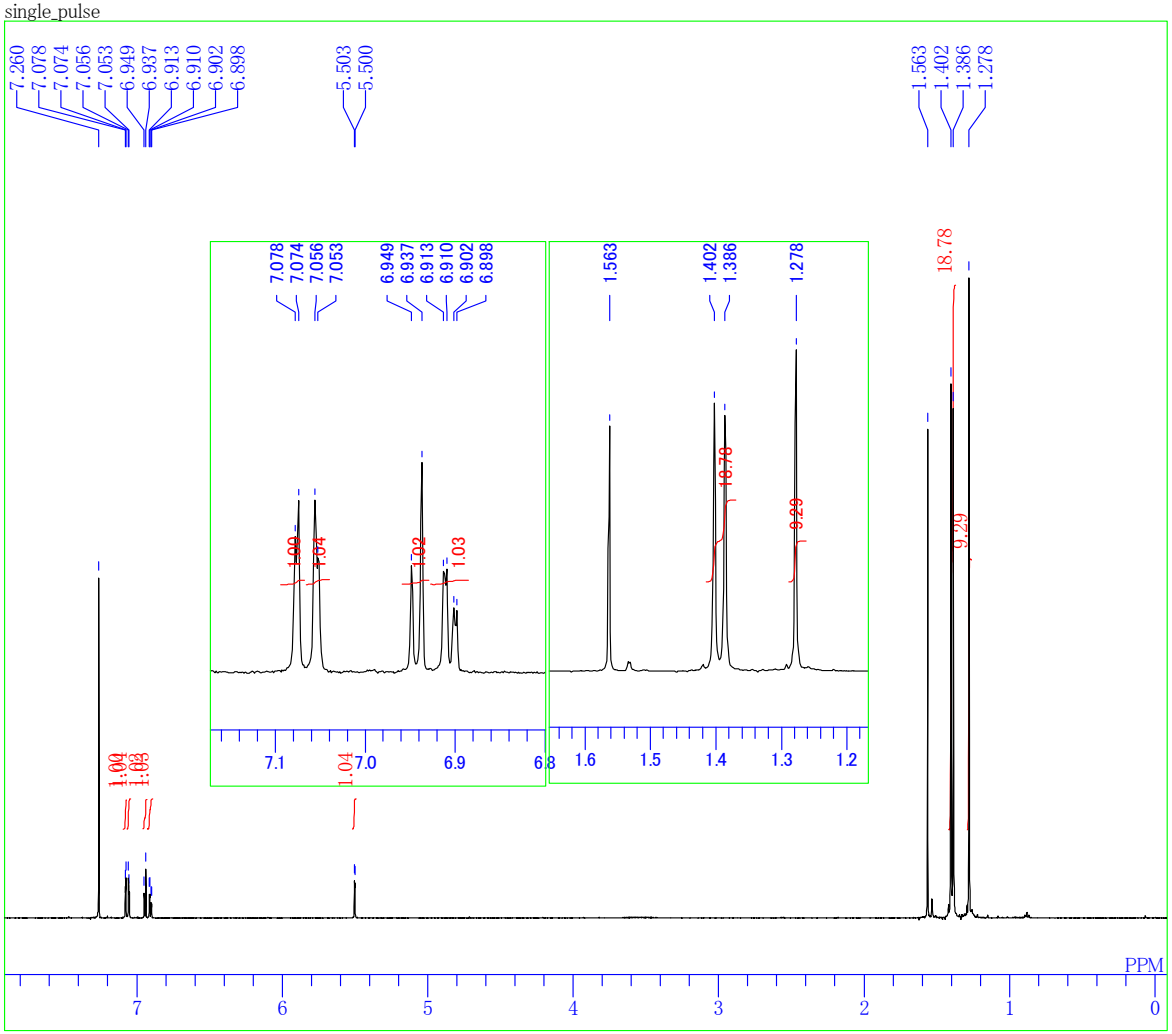
DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single pulse decoupled gated NOE
DATIM	11-03-2008 20:05:37
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32768
FREQU	39308.18 Hz
SCANS	1721
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	18.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60



anti-**4b** ($CDCl_3$)

Table 1, entry 2

single_pulse



DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single_pulse
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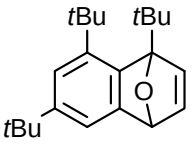
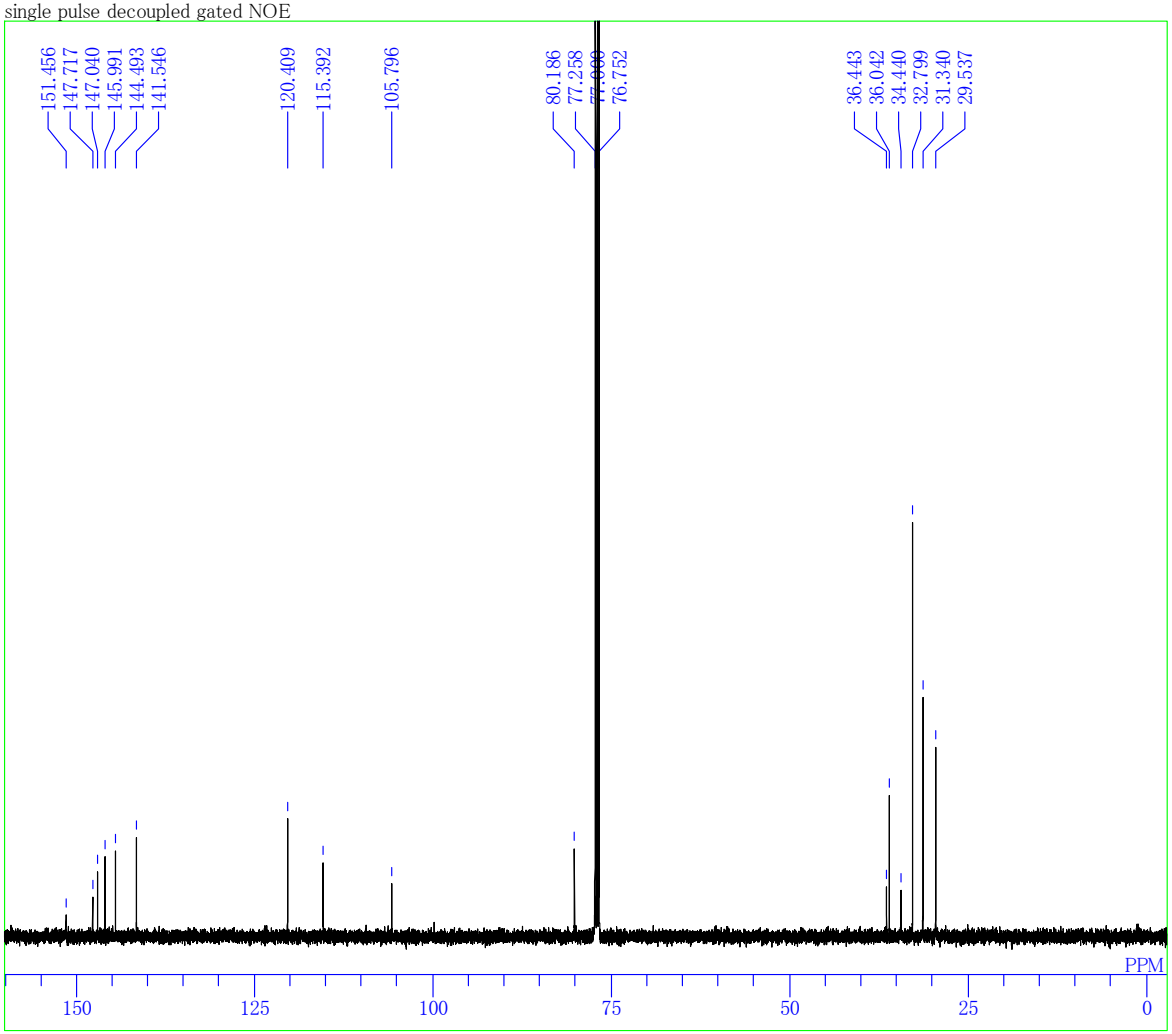
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16384
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	6.00 usec
IRNUC	1H
CTEMP	17.2 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	56



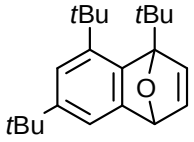
syn-**4b** ($CDCl_3$)

Table 1, entry 2

single pulse decoupled gated NOE



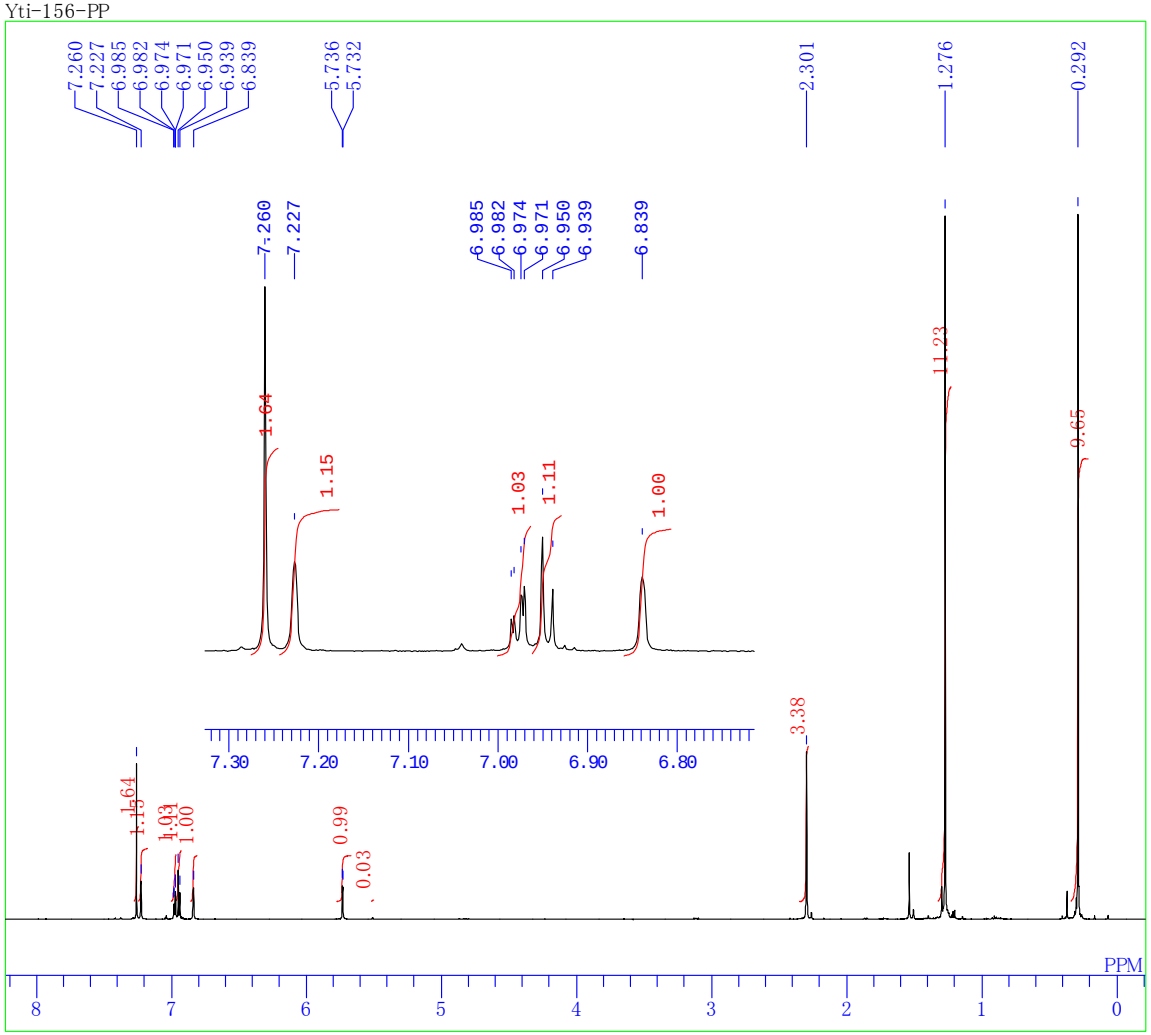
DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single pulse decoupled gated NOE
DATIM	08-03-2008 18:05:25
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32768
FREQU	39308.18 Hz
SCANS	2247
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	17.7 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60



syn-**4b** ($CDCl_3$)

Table 1, entry 2

Yti-156-PP



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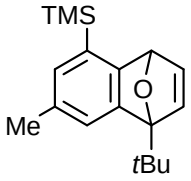
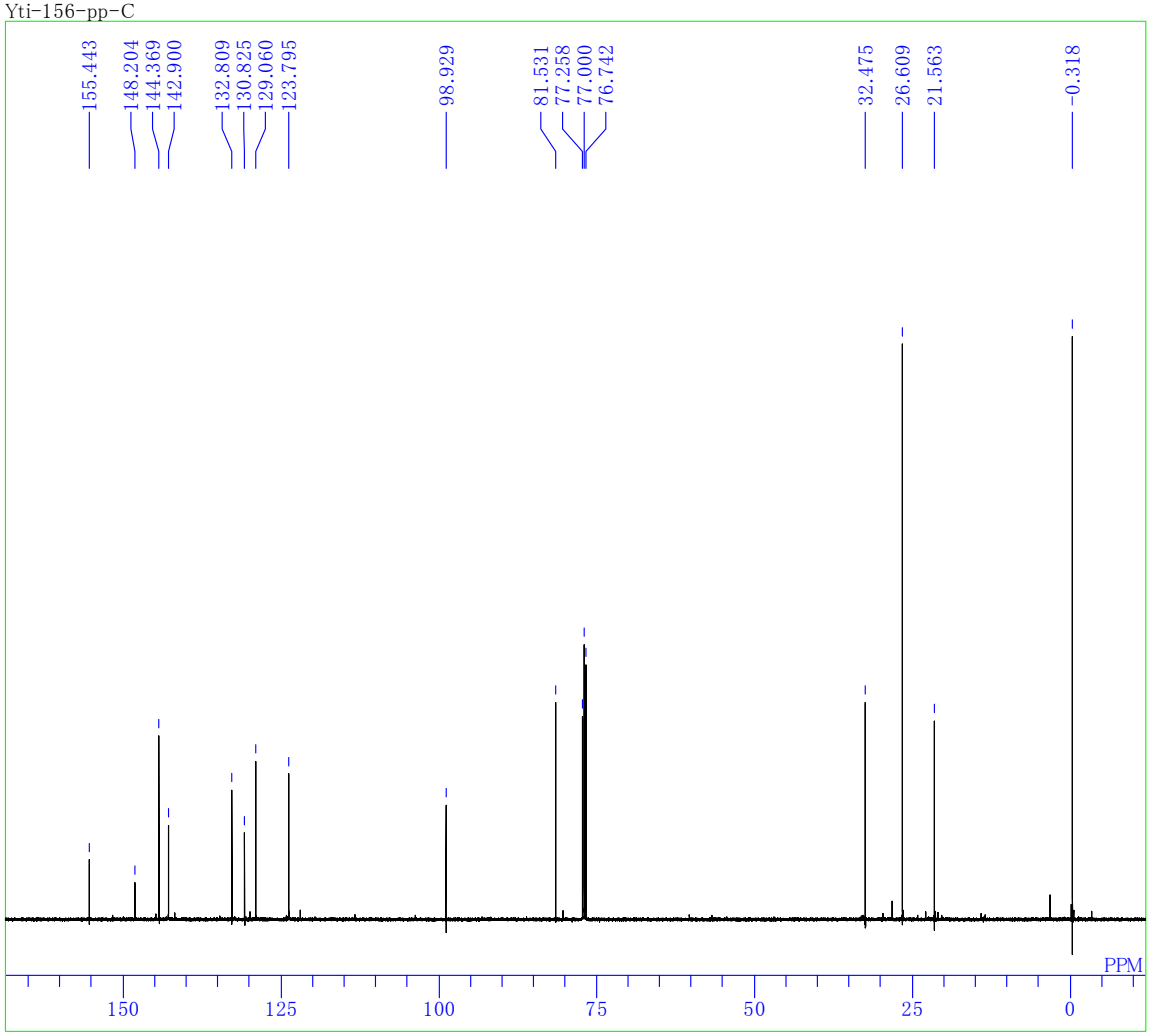
COMNT	Yti-156-PP
DATIM	Wed Mar 12 23:18:09 2008
OBNUC	1H
EXMOD	SINGL
OBFRQ	499.10 MHz
OBSET	0.00 KHz
OBFIN	126750.00 Hz
POINT	16384
FREQU	9980.04 Hz
SCANS	8
ACQTM	1.6417 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	-204.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	24



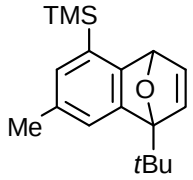
anti-**4c** ($CDCl_3$)

Table 1, entry 3

Yti-156-pp-C

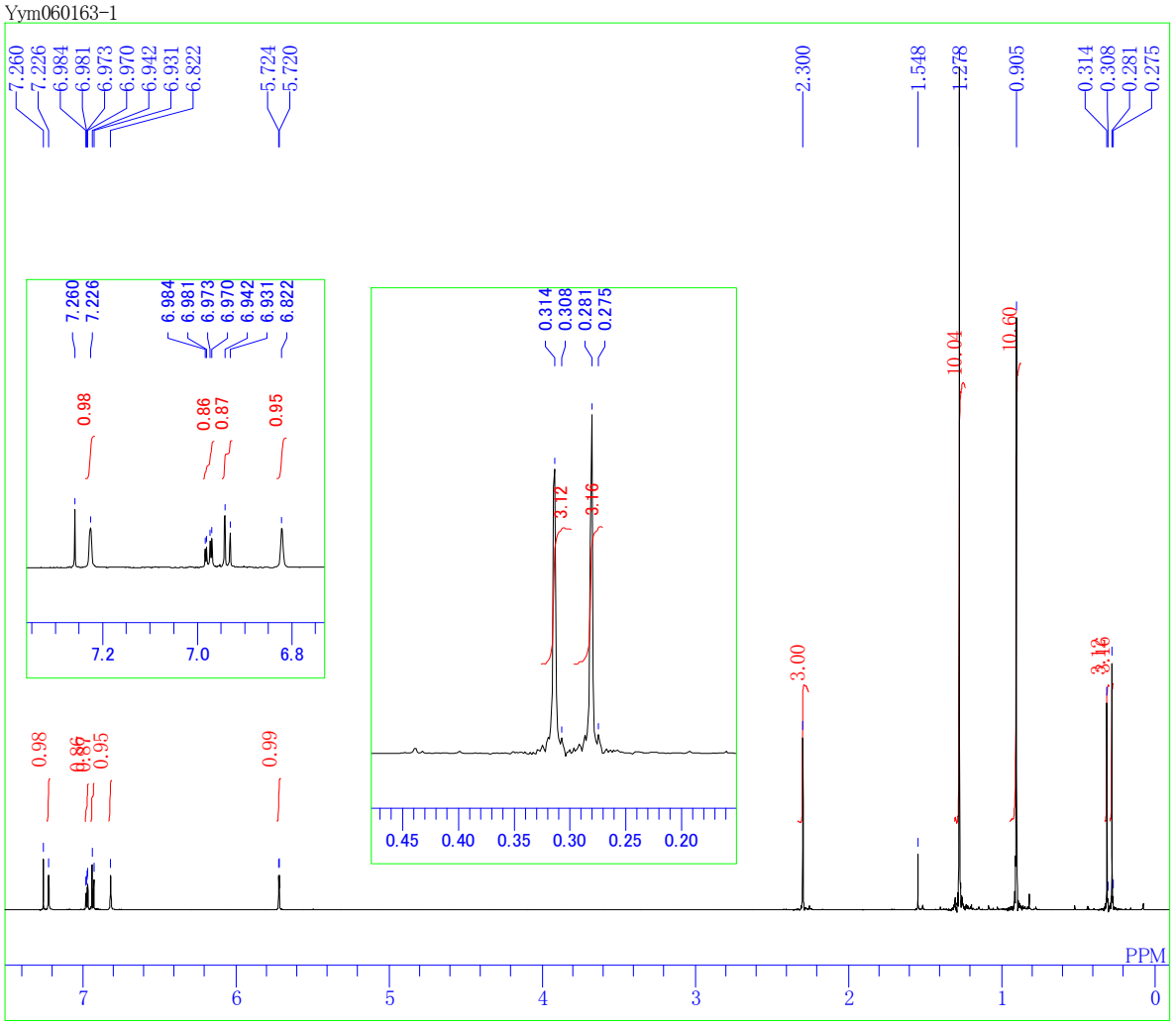


DFILE	C:¥Documents and Settings¥Owner
COMNT	Yti-156-pp-C
DATIM	04-06-2008 23:25:37
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32768
FREQU	39308.18 Hz
SCANS	1306
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	20.7 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60



anti-**4c** ($CDCl_3$)

Table 1, entry 3



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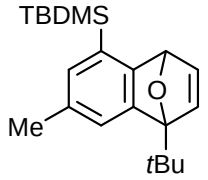
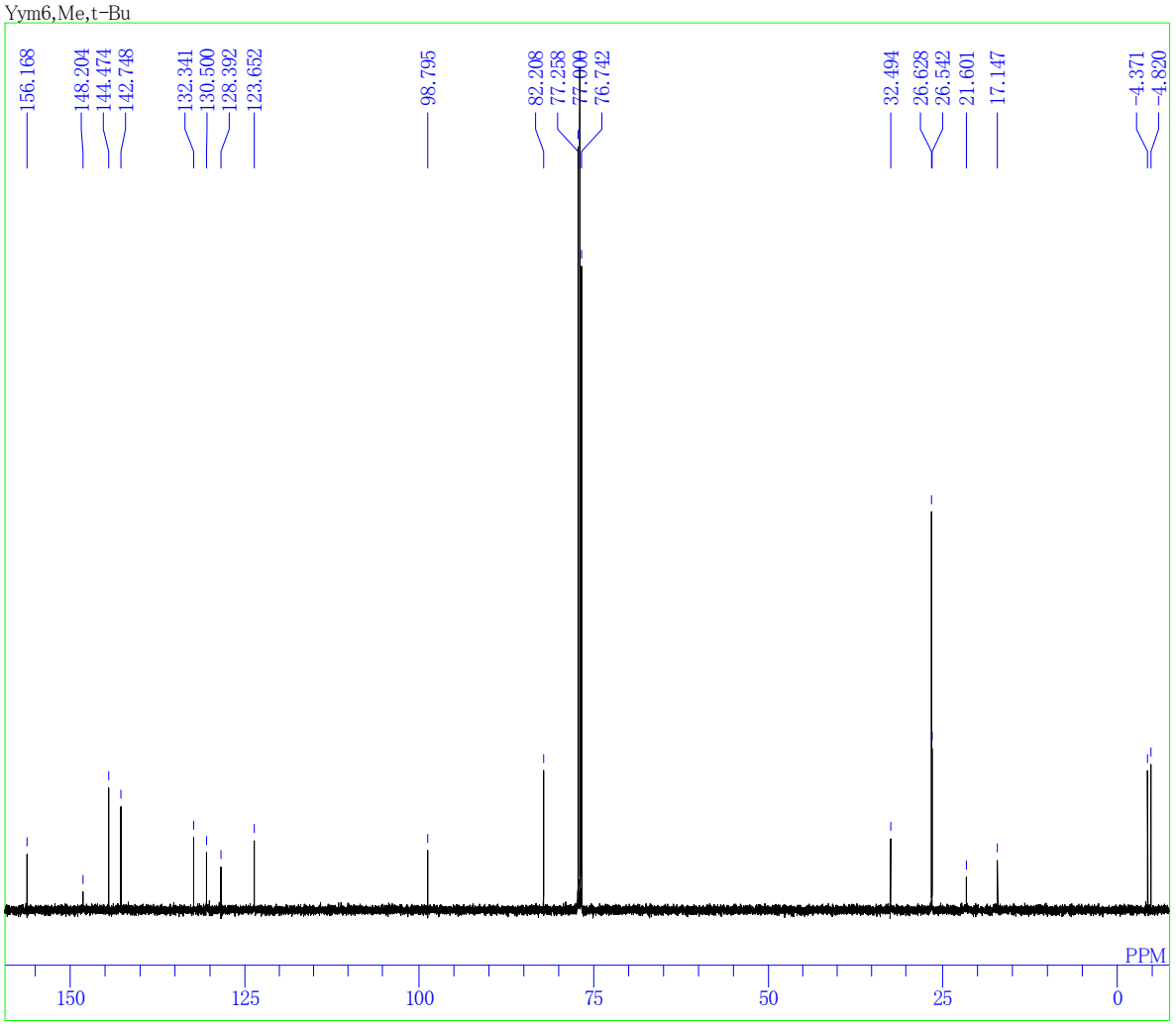
OBSET	0.00 KHz
OBFIN	125250.00 Hz
POINT	16384
FREQU	9980.04 Hz
SCANS	8
ACQTM	1.6417 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	-204.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	20



anti-**4d** ($CDCl_3$)

Table 1, entry 4

Table 2, entry 4



DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	Yym6,Me,t-Bu
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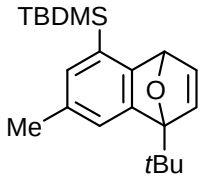
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.06 Hz
SCANS	1508
ACQTM	0.8336 sec
PD	1.5000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	18.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

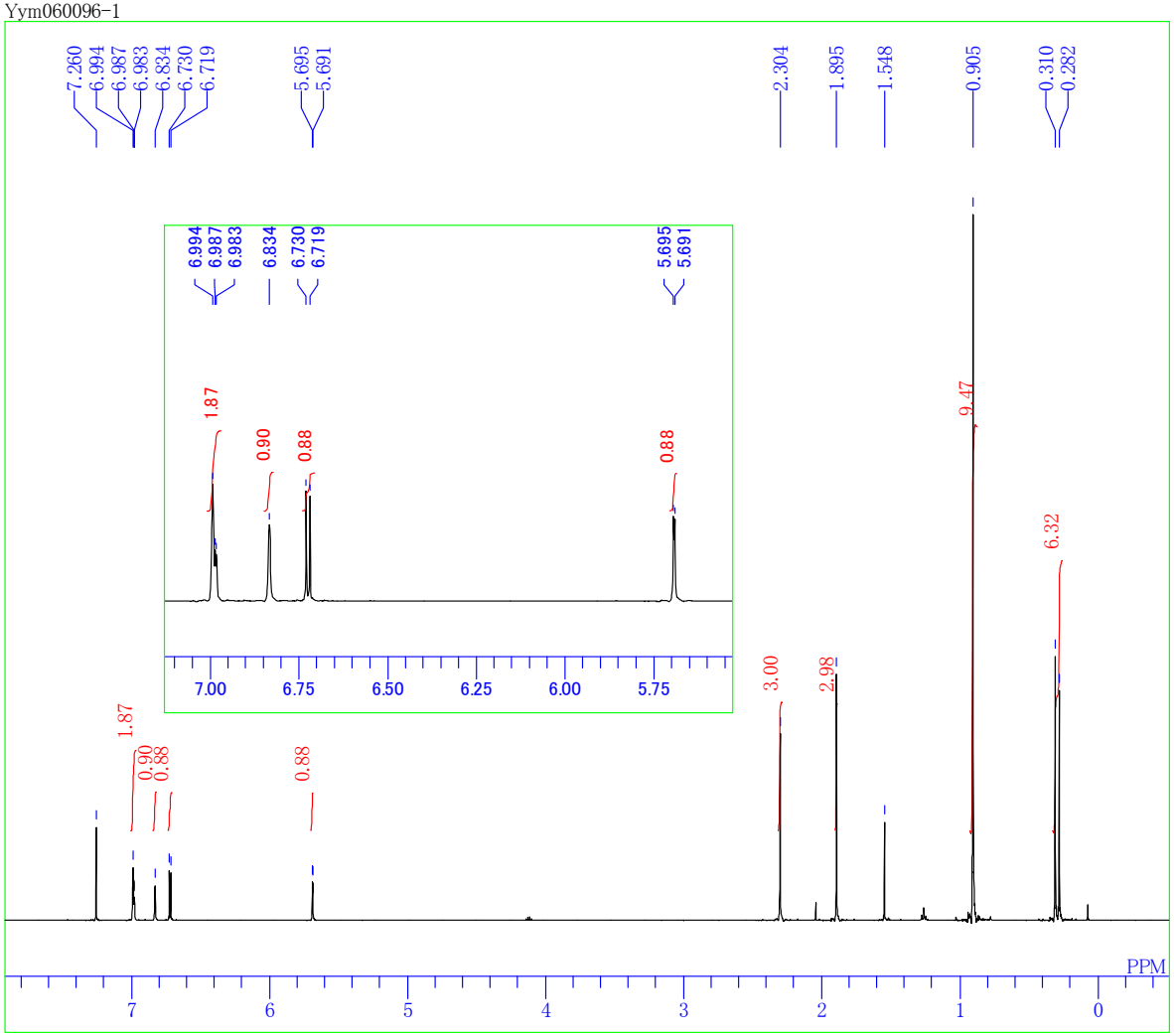


anti-**4d** ($CDCl_3$)

Table 1, entry 4

Table 2, entry 4

Yym060096-1



DFILE	C:¥Documents and Settings¥Owner¥M
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DATIM	Mon May 14 17:07:07 2007
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EXMOD	SINGL
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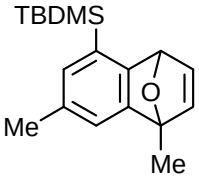
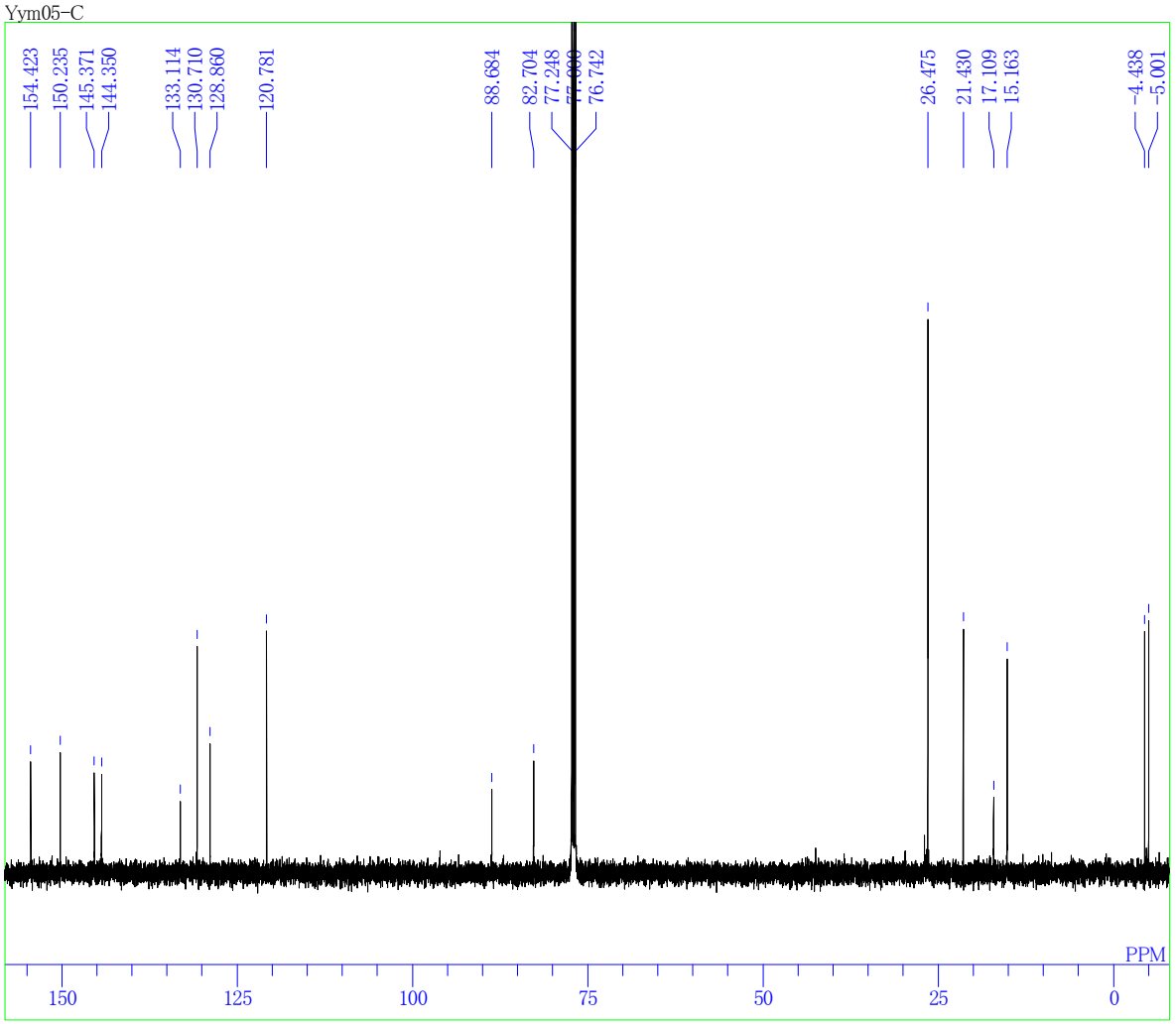
OBSET	0.00 KHz
OBFIN	125250.00 Hz
POINT	16384
FREQU	9980.04 Hz
SCANS	8
ACQTM	1.6417 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	-204.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	22



anti-**4e** ($CDCl_3$)
Table 2, entry 1



DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	Yym05-C
DATIM	31-01-2008 12:34:15
OBNUC	13C
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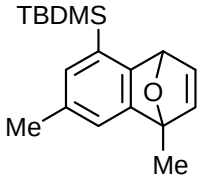
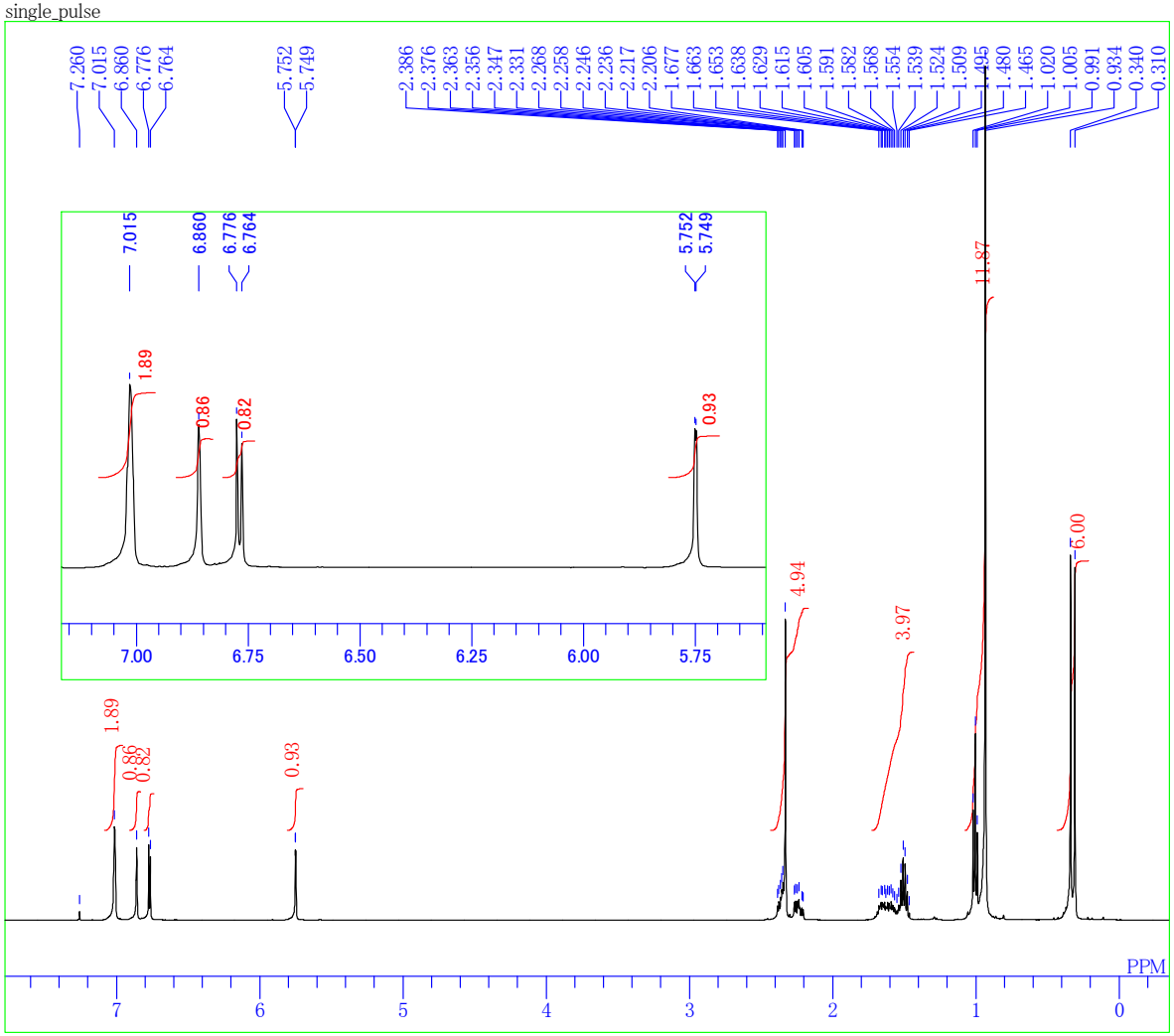
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.06 Hz
SCANS	2602
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	17.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60



anti-**4e** ($CDCl_3$)

Table 2, entry 1

single_pulse



DFILE	C:¥Documents and Settings¥Owner¥デ
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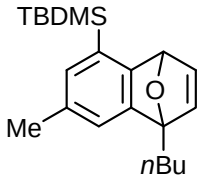
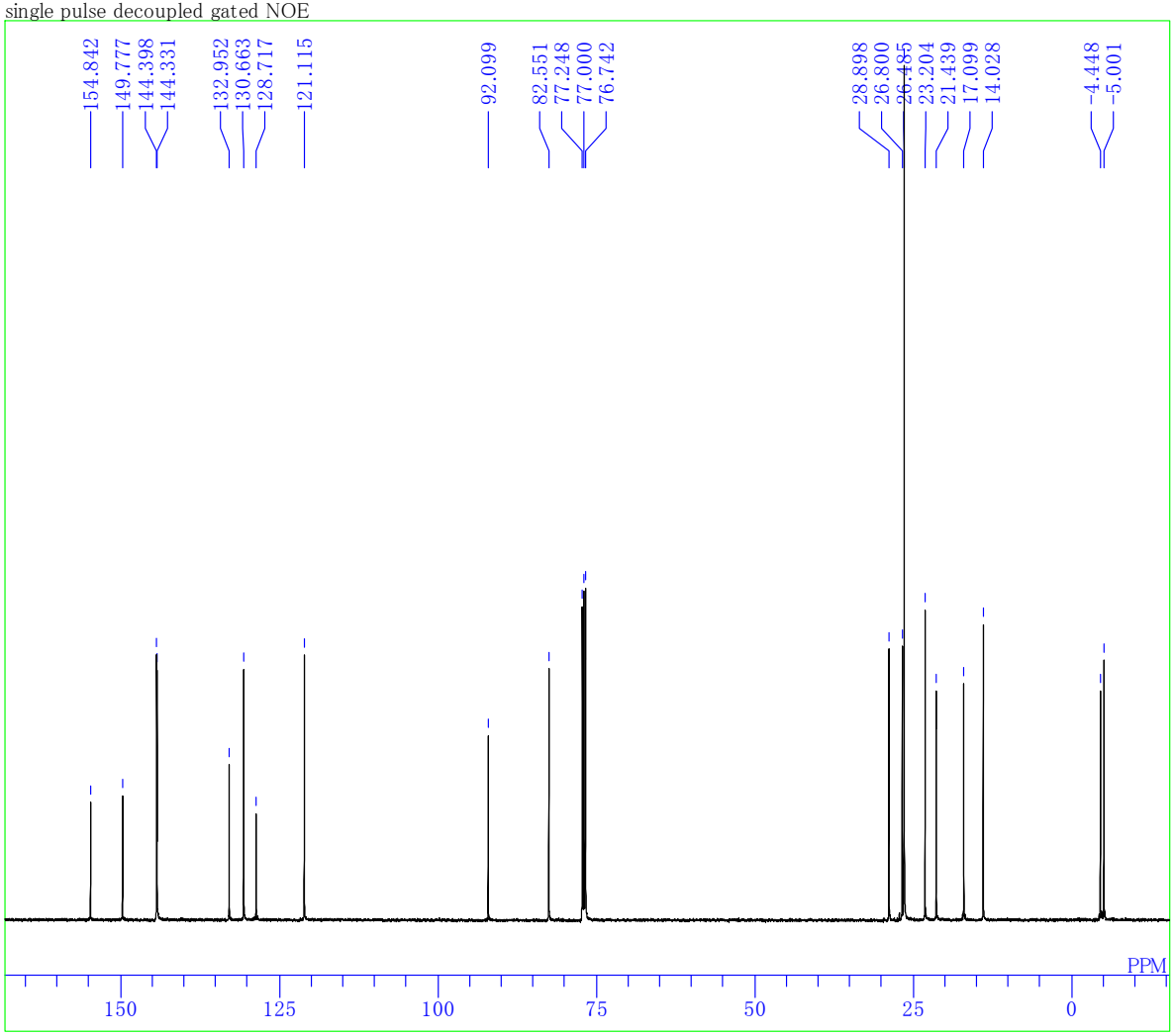
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13106
FREQU	7507.39 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	6.00 usec
IRNUC	1H
CTEMP	17.1 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	30

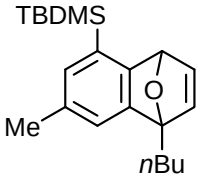


anti-**4f** ($CDCl_3$)
Table 2, entry 2

single pulse decoupled gated NOE



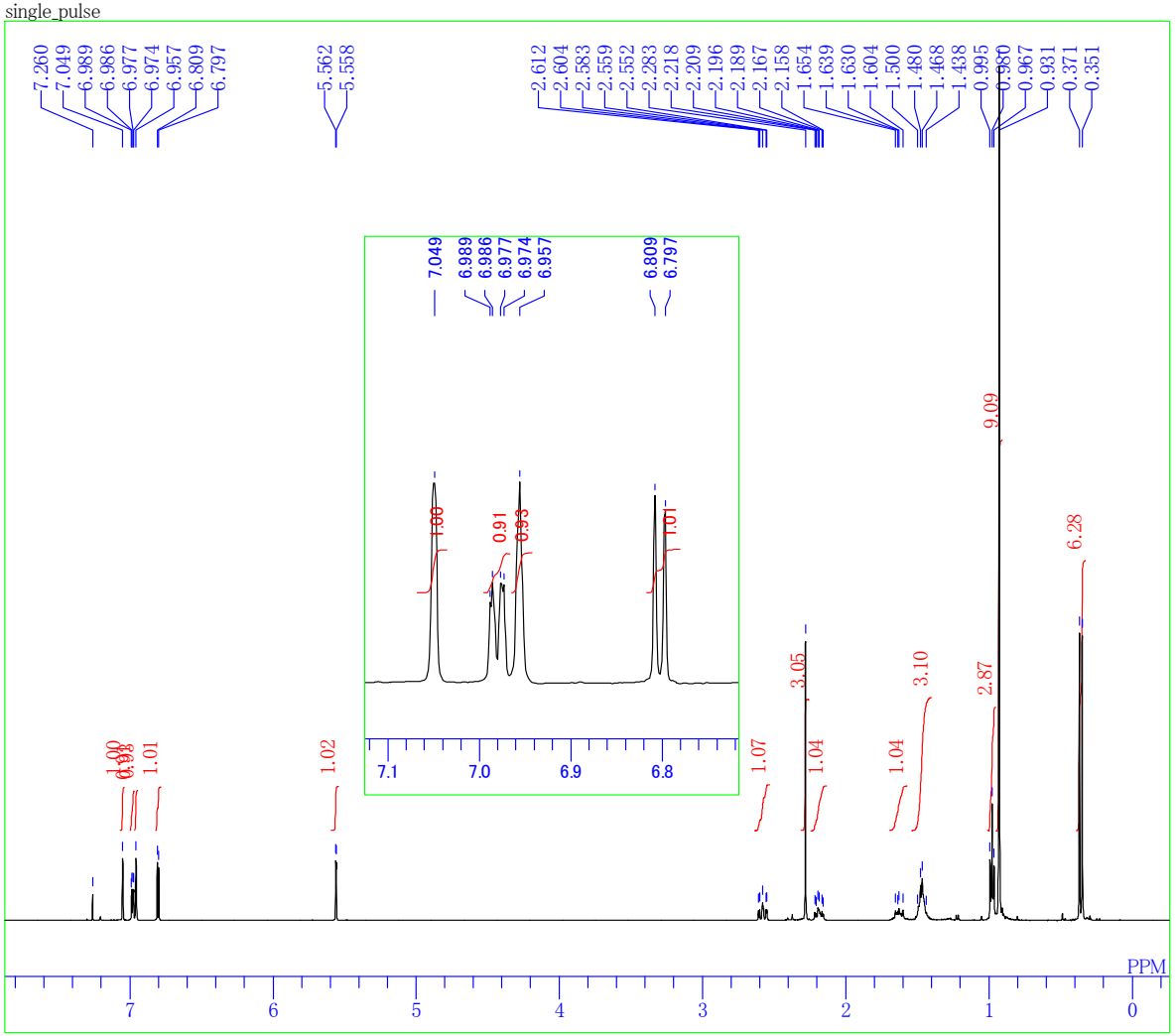
DFILE	C:¥Documents and Settings¥Owner
COMNT	single pulse decoupled gated NOE
DATIM	21-02-2008 20:16:14
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32768
FREQU	39308.18 Hz
SCANS	1999
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	17.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60



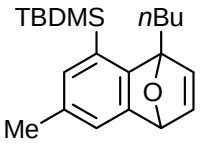
anti-**4f** ($CDCl_3$)

Table 2, entry 2

single_pulse



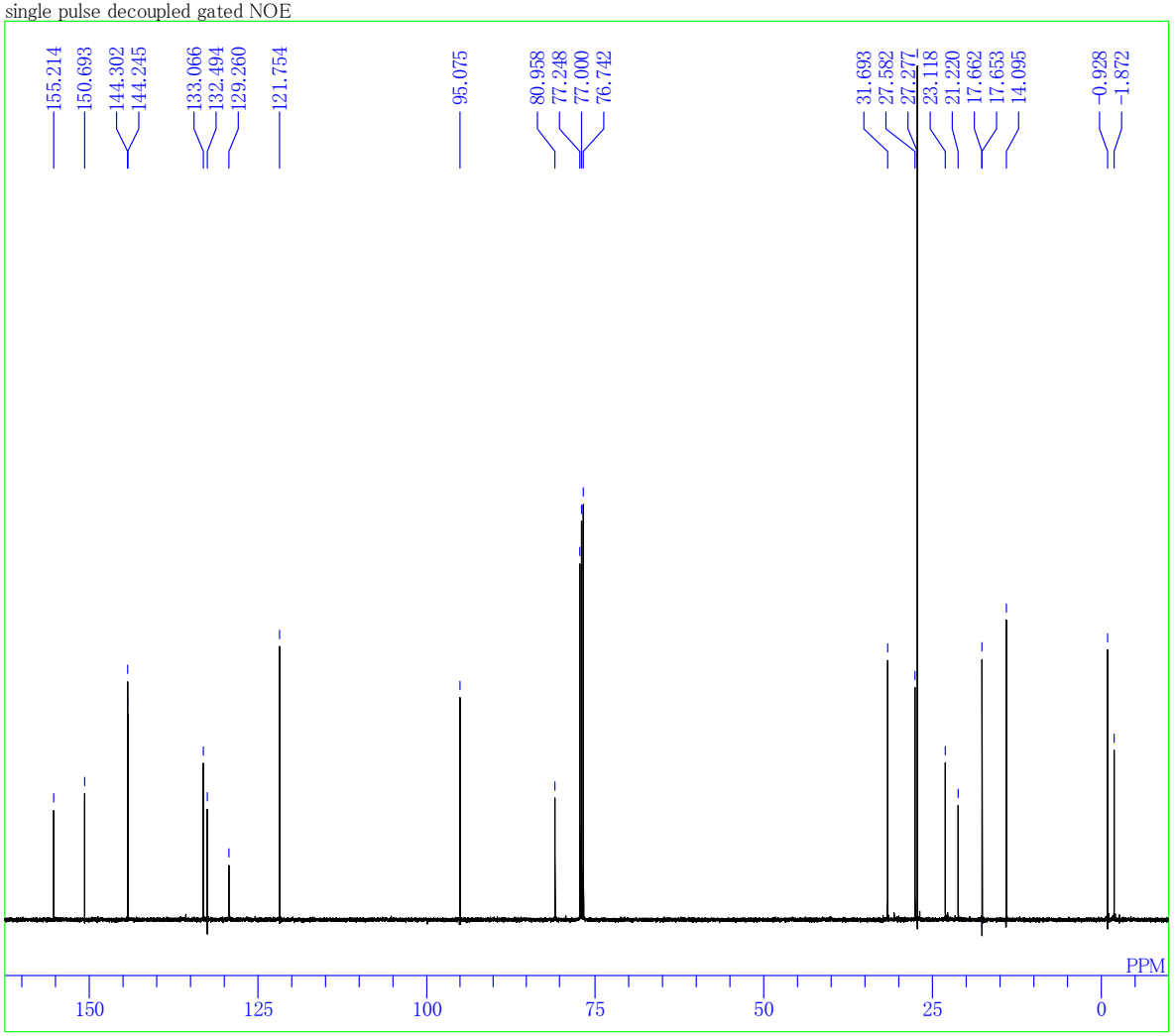
DFILE	C:¥Documents and Settings¥Owner¥ﾃﾞ
COMNT	single_pulse
DATIM	20-02-2008 21:37:17
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16384
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	6.00 usec
IRNUC	1H
CTEMP	17.4 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	36



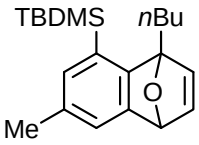
syn-**4f** ($CDCl_3$)

Table 2, entry 2

single pulse decoupled gated NOE

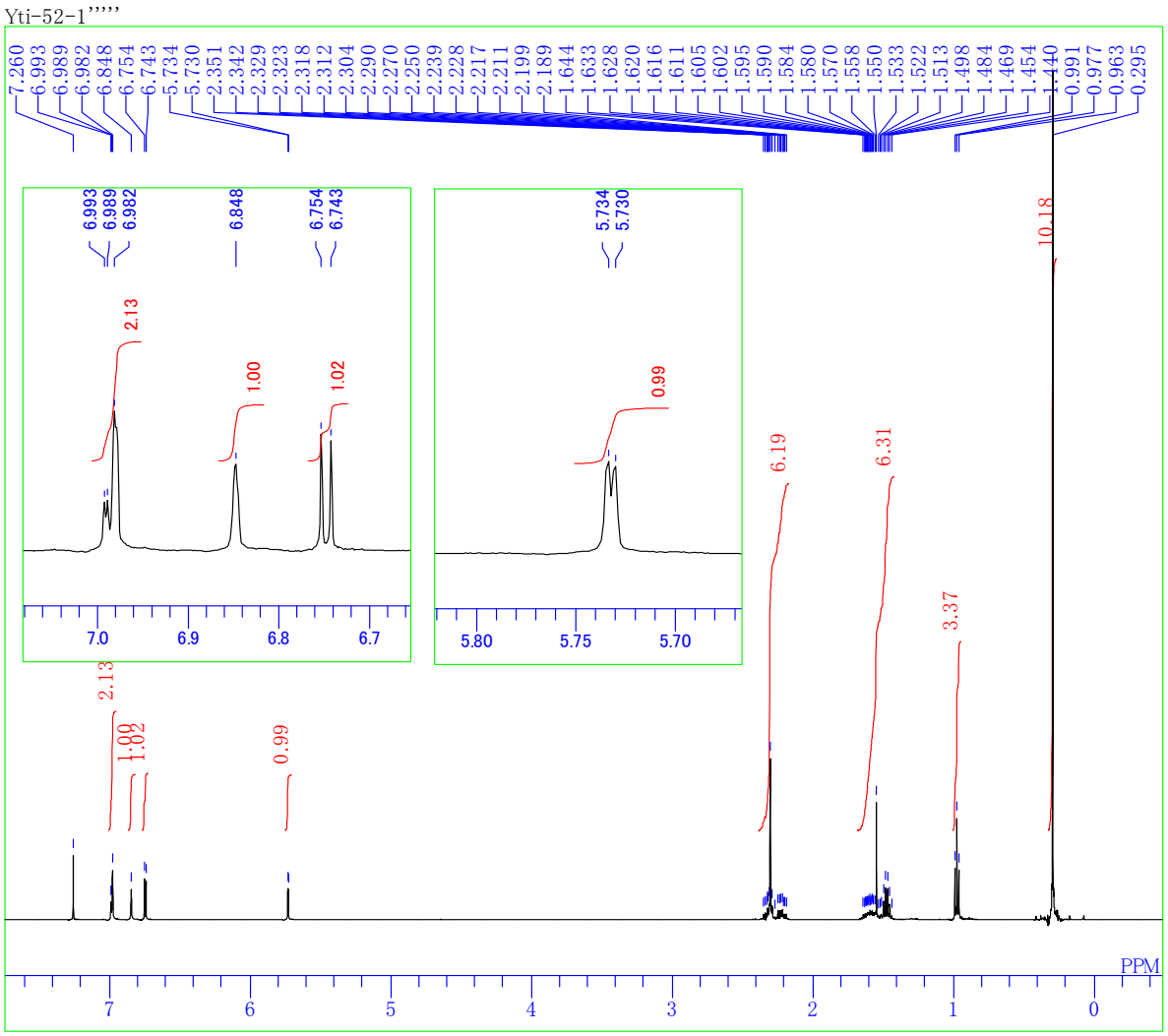


DFILE	C:¥Documents and Settings¥Owner¥デ
COMNT	single pulse decoupled gated NOE
DATIM	20-02-2008 21:34:31
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32768
FREQU	39308.18 Hz
SCANS	1571
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	17.7 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

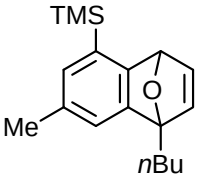


syn-**4f** ($CDCl_3$)

Table 2, entry 2



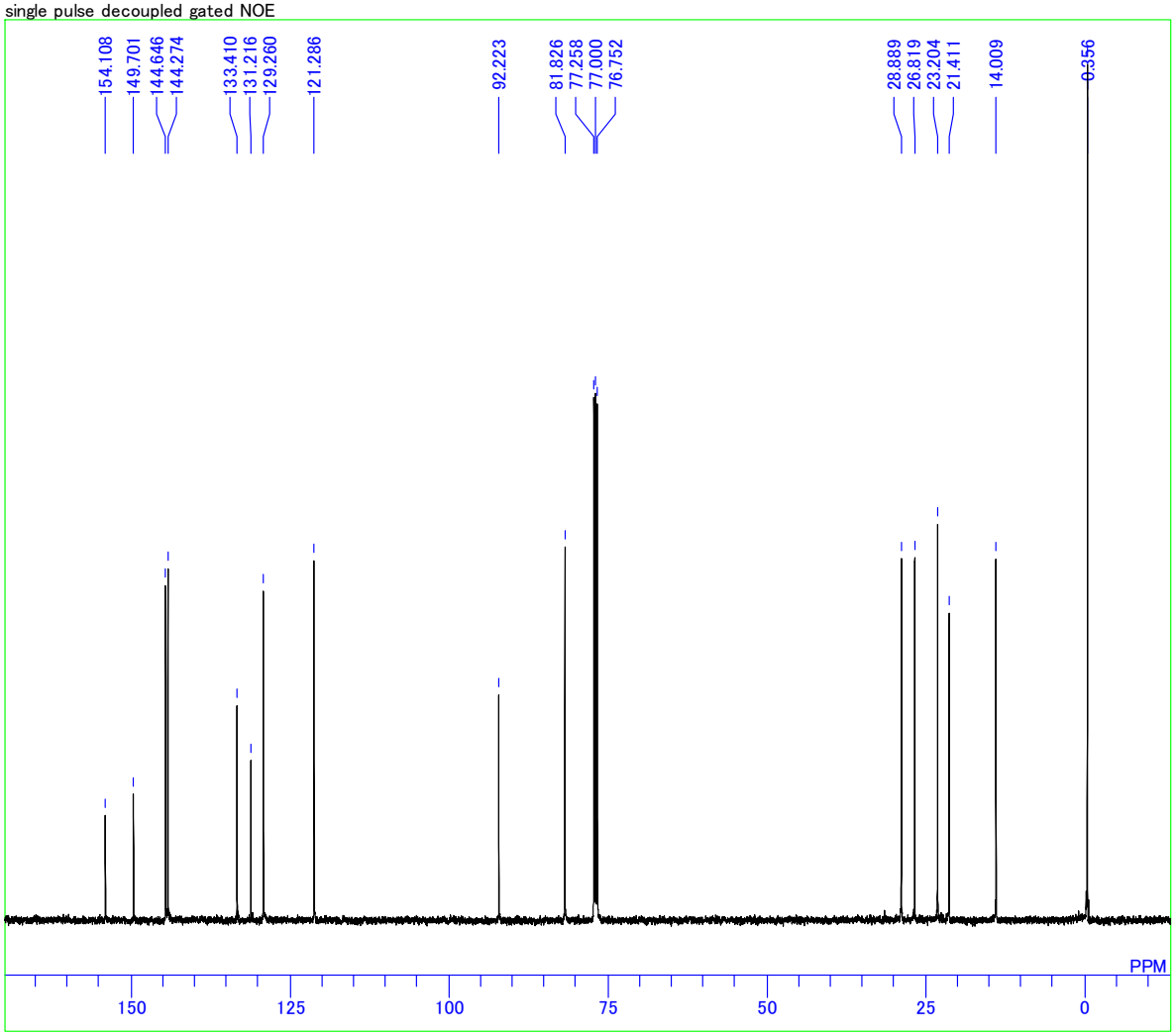
DFILE C:¥Documents and Settings¥Owner
COMNT Yti-52-1’’’’’
DATIM Mon Jun 25 10:01:05 2007
OBNUC 1H
EXMOD SINGL
OBFRQ 499.10 MHz
OBSET 0.00 KHz
OBFIN 125250.00 Hz
POINT 16384
FREQU 9980.04 Hz
SCANS 8
ACQTM 1.6417 sec
PD 2.0000 sec
PW1 5.50 usec
IRNUC 1H
CTEMP -204.8 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 21



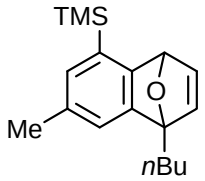
anti-**4g** ($CDCl_3$)

Table 2, entry 3

single pulse decoupled gated NOE



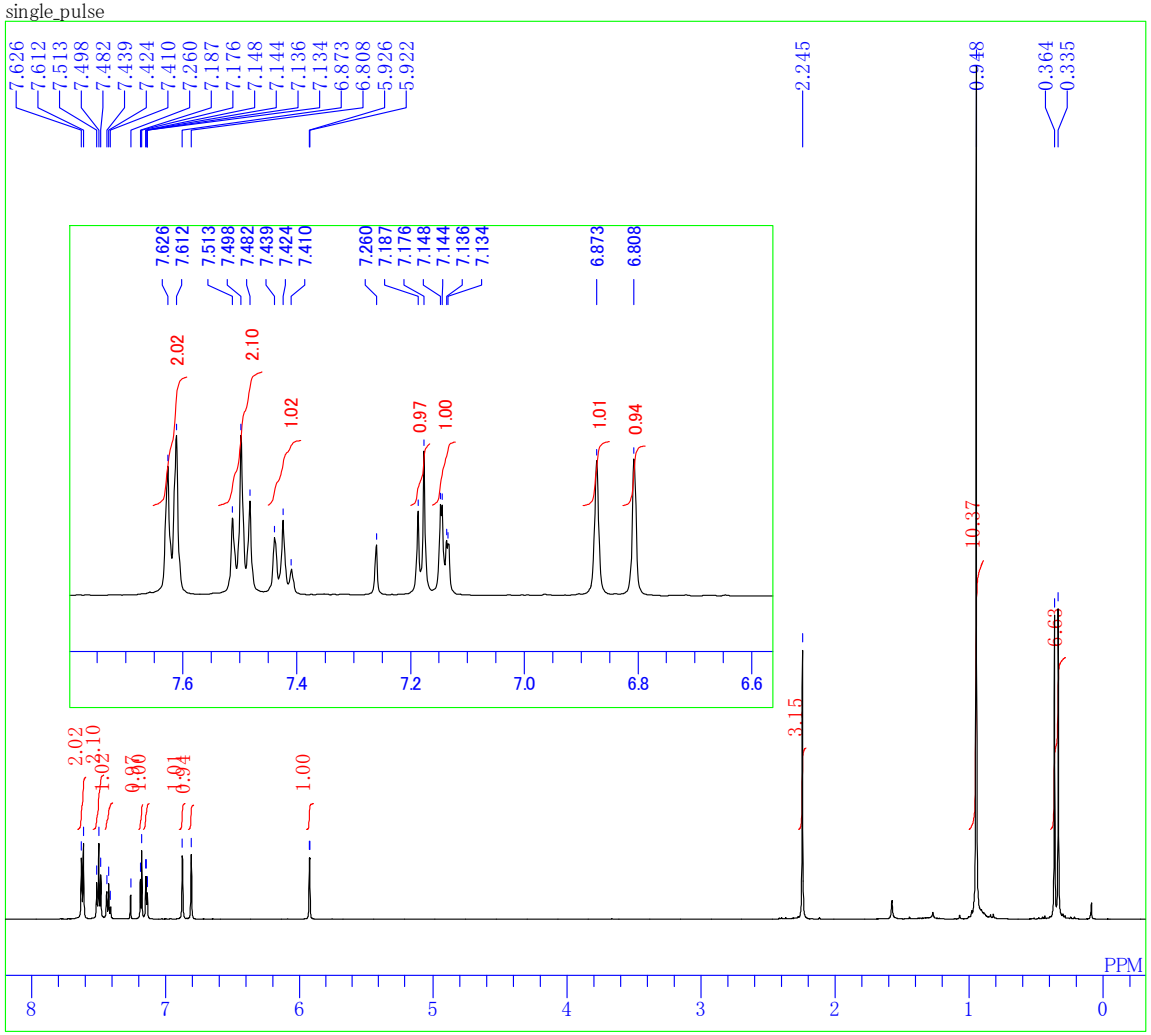
DFILE	C:¥Documents and Settings¥Owner¥My D
COMNT	single pulse decoupled gated NOE
DATIM	26-06-2007 21:49:45
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	40961
FREQU	49135.97 Hz
SCANS	850
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	21.3 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60



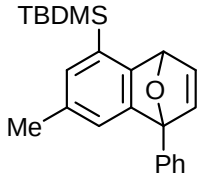
anti-**4g** ($CDCl_3$)

Table 2, entry 3

single_pulse

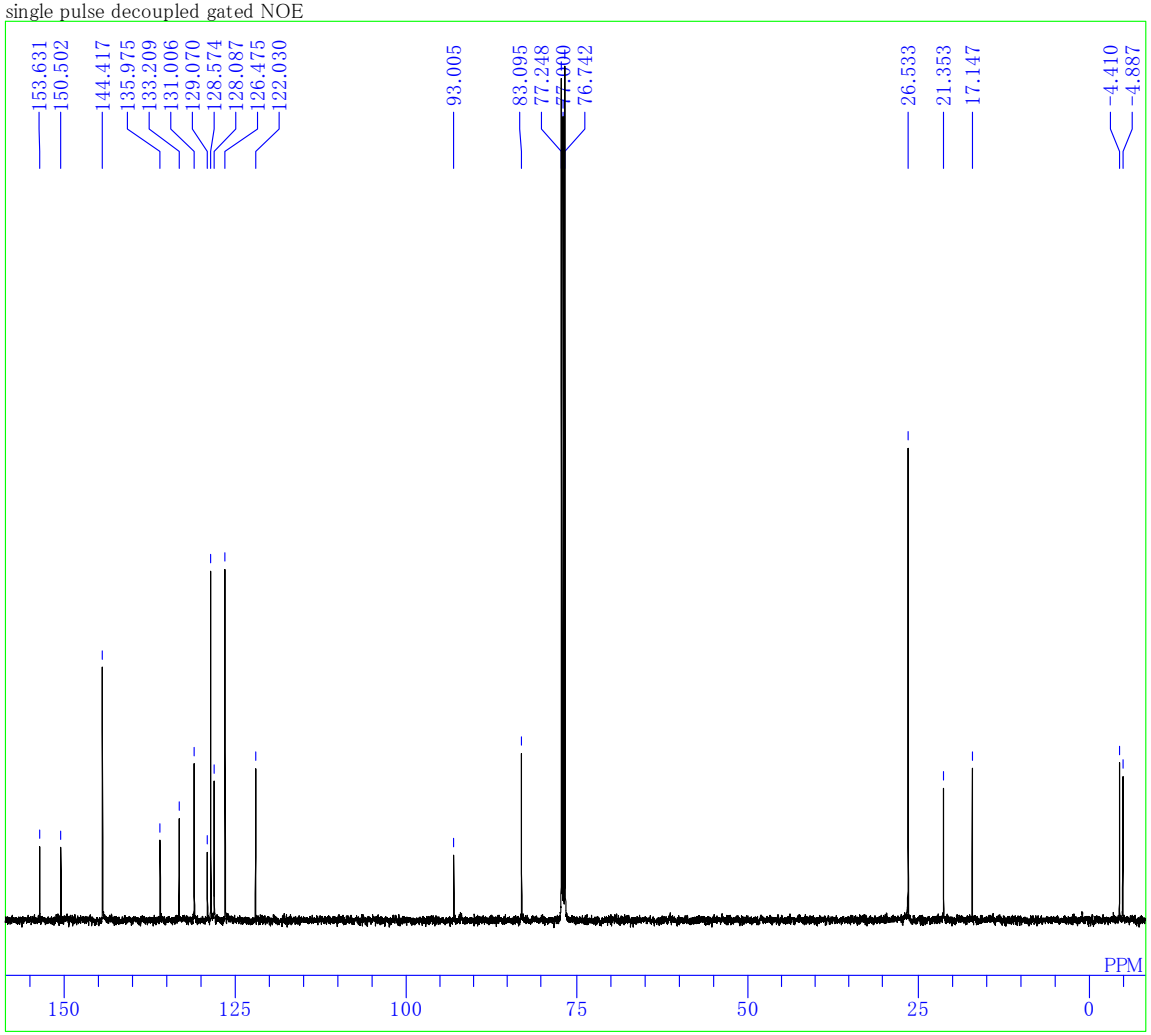


DFILE	C:¥Documents and Settings¥Owner
COMNT	single_pulse
DATIM	28-05-2008 13:59:02
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.39 Hz
SCANS	8
ACQTM	1.7459 sec
PD	1.5000 sec
PW1	5.75 usec
IRNUC	1H
CTEMP	21.3 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	1.20 Hz
RGAIN	44

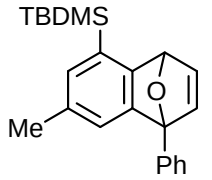


anti-**4h** ($CDCl_3$)
Table 2, entry 5

single pulse decoupled gated NOE

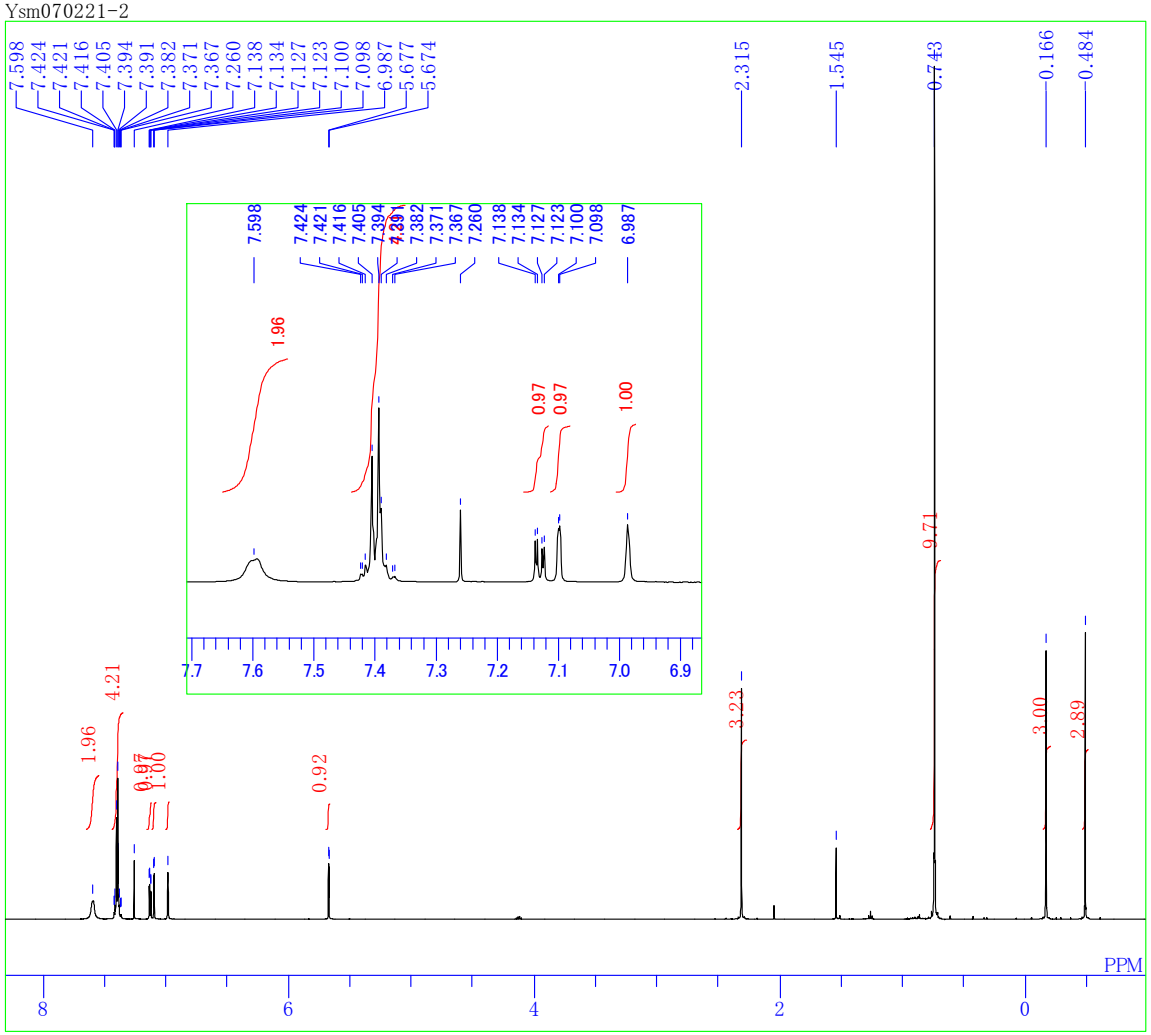


DFILE	C:¥Documents and Settings¥Owner
COMNT	single pulse decoupled gated NOE
DATIM	28-05-2008 14:36:14
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32768
FREQU	39308.18 Hz
SCANS	713
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	21.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60

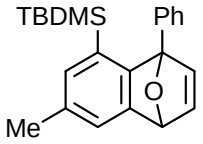


anti-**4h** ($CDCl_3$)
Table 2, entry 5

Ysm070221-2

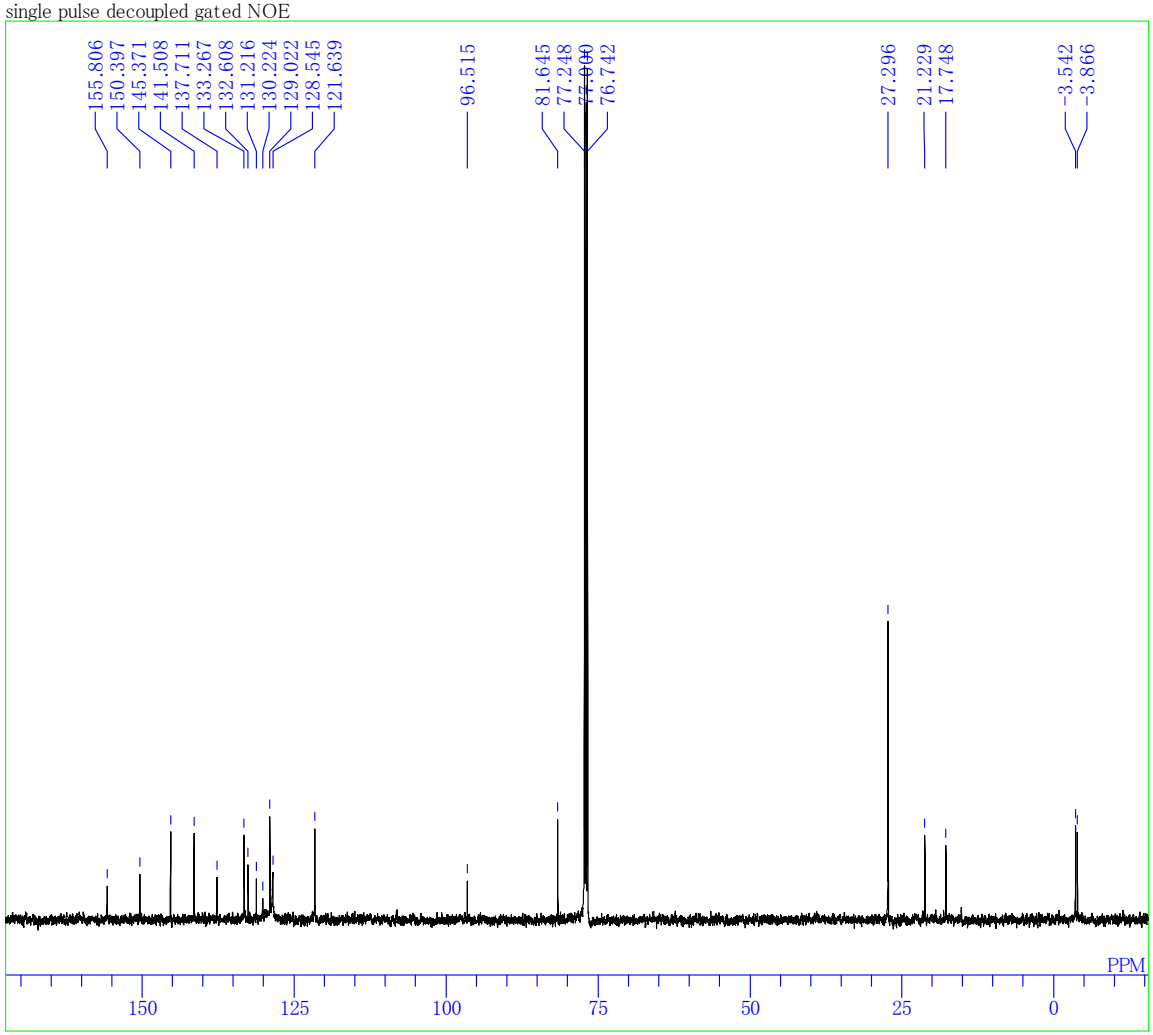


DFILE	C:¥Documents and Settings¥Owner
COMNT	Ysm070221-2
DATIM	Tue May 13 19:48:26 2008
OBNUC	1H
EXMOD	SINGL
OBFRQ	499.10 MHz
OBSET	0.00 KHz
OBFIN	126750.00 Hz
POINT	16384
FREQU	9980.04 Hz
SCANS	8
ACQTM	1.6417 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	-204.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	21

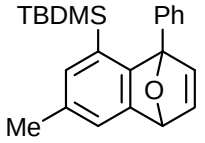


syn-**4h** ($CDCl_3$)
Table 2, entry 5

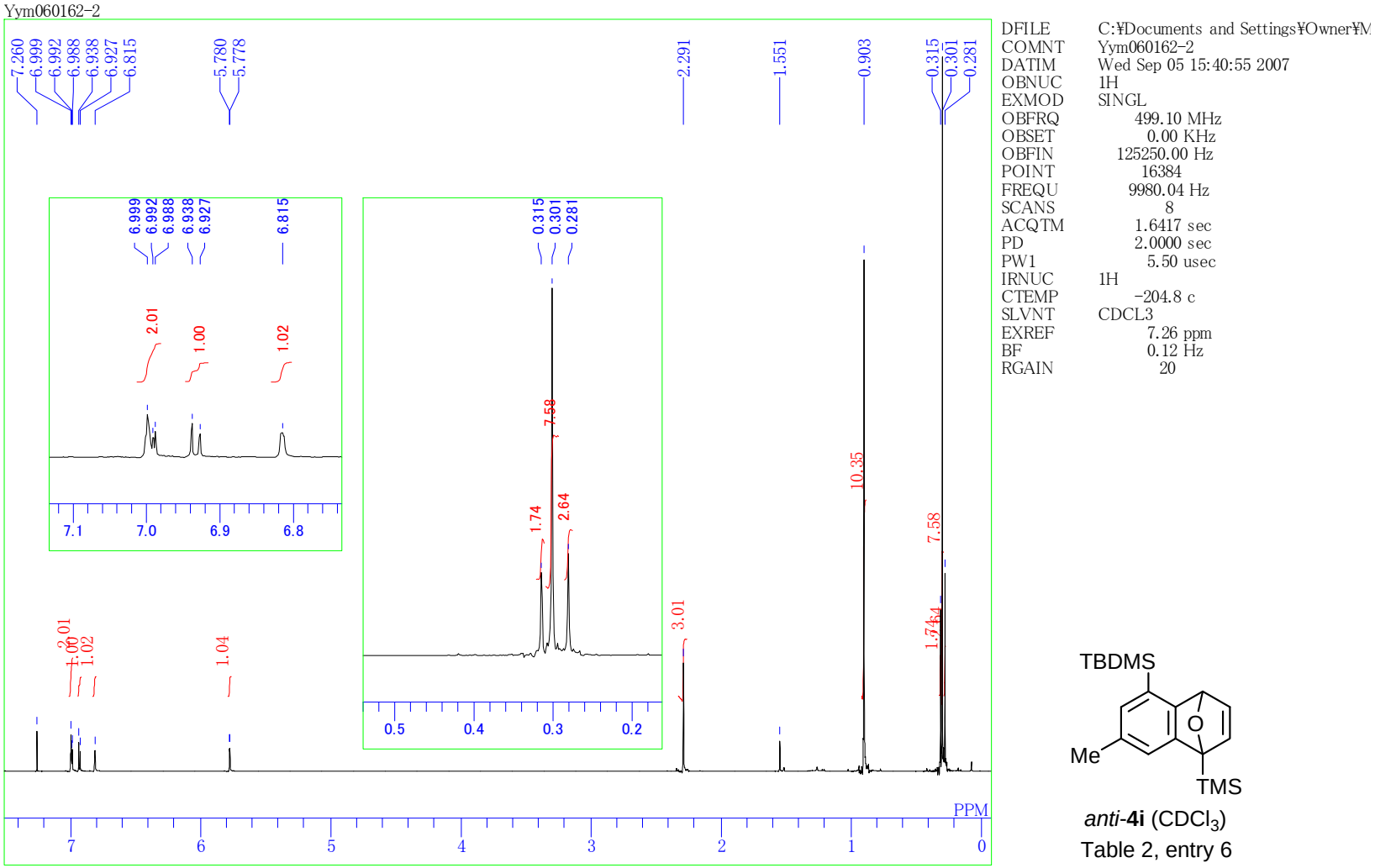
single pulse decoupled gated NOE



DFILE	C:¥Documents and Settings¥Owner¥
COMNT	single pulse decoupled gated NOE
DATIM	13-06-2008 13:46:26
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32768
FREQU	39308.18 Hz
SCANS	538
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	19.7 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	3.00 Hz
RGAIN	60



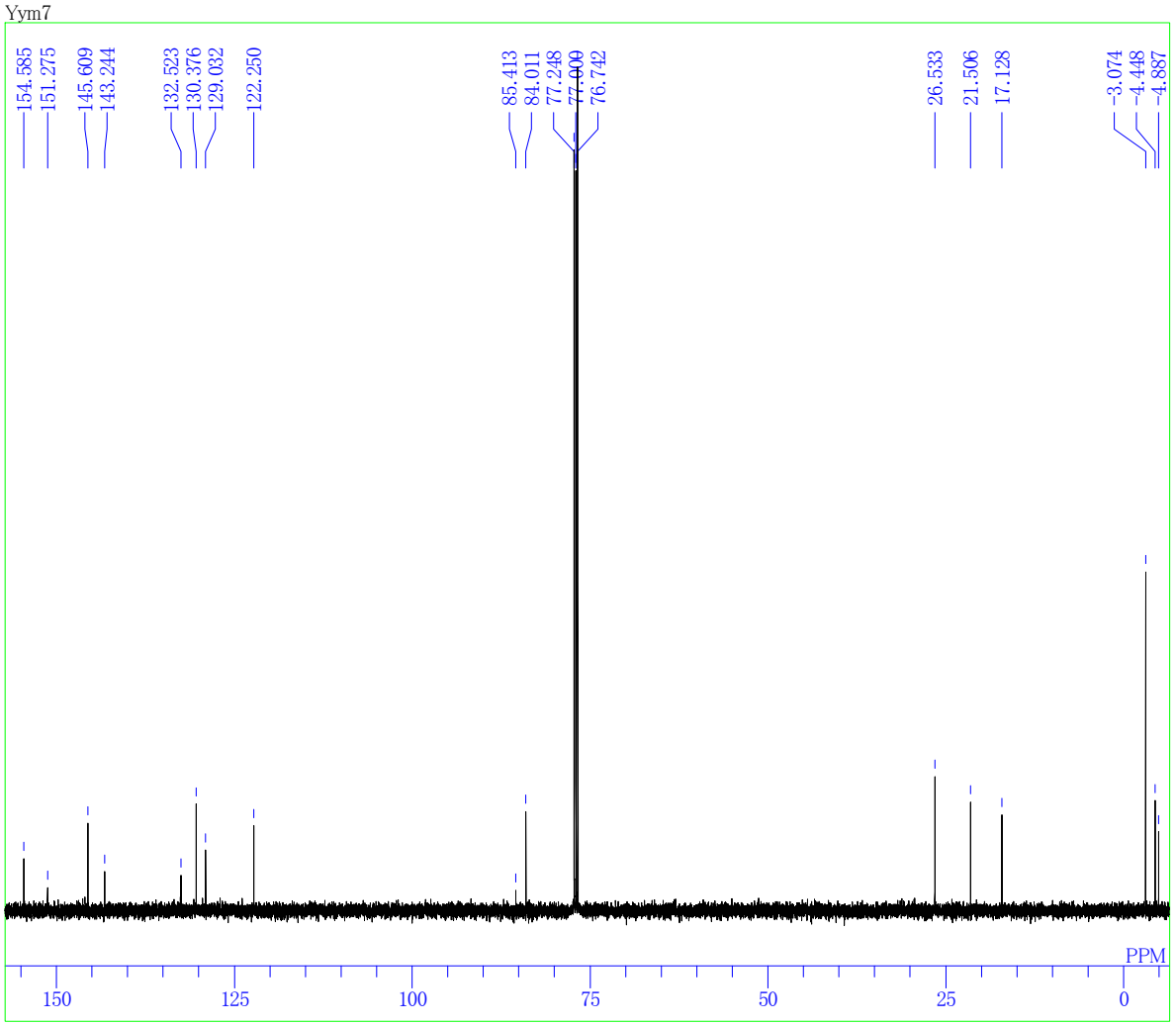
syn-**4h** ($CDCl_3$)
Table 2, entry 5



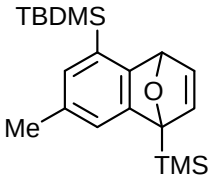
DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	Yym060162-2
DATIM	Wed Sep 05 15:40:55 2007
OBNUC	1H
EXMOD	SINGL
OBFRQ	499.10 MHz
OBSET	0.00 KHz
OBFIN	125250.00 Hz
POINT	16384
FREQU	9980.04 Hz
SCANS	8
ACQTM	1.6417 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	-204.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	20

anti-**4i** ($CDCl_3$)

Table 2, entry 6

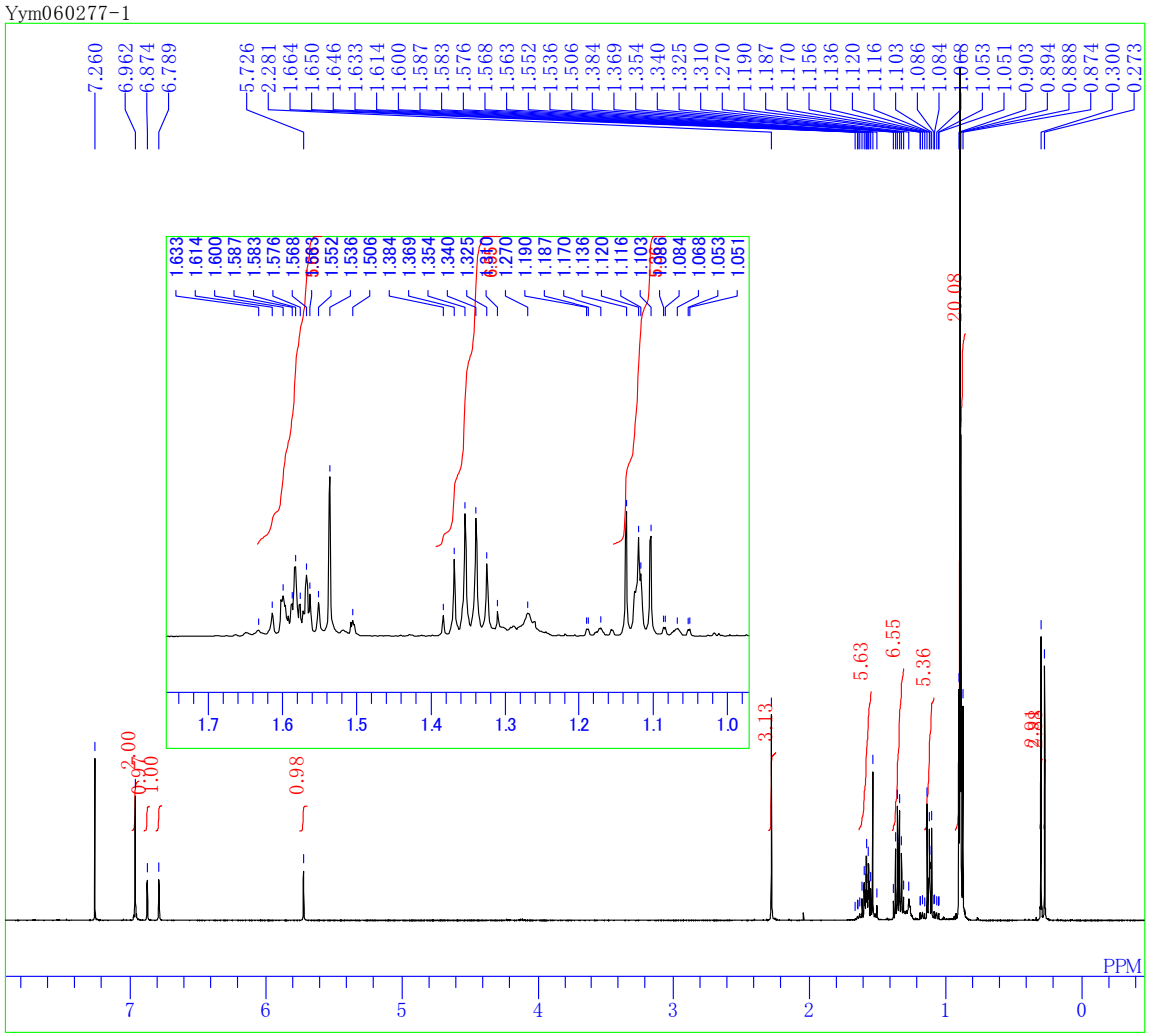


DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	Yym7
DATIM	28-12-2007 11:41:35
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.06 Hz
SCANS	367
ACQTM	0.8336 sec
PD	1.5000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	17.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

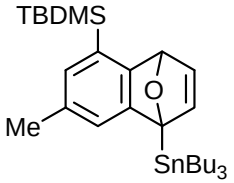


anti-**4i** ($CDCl_3$)

Table 2, entry 6

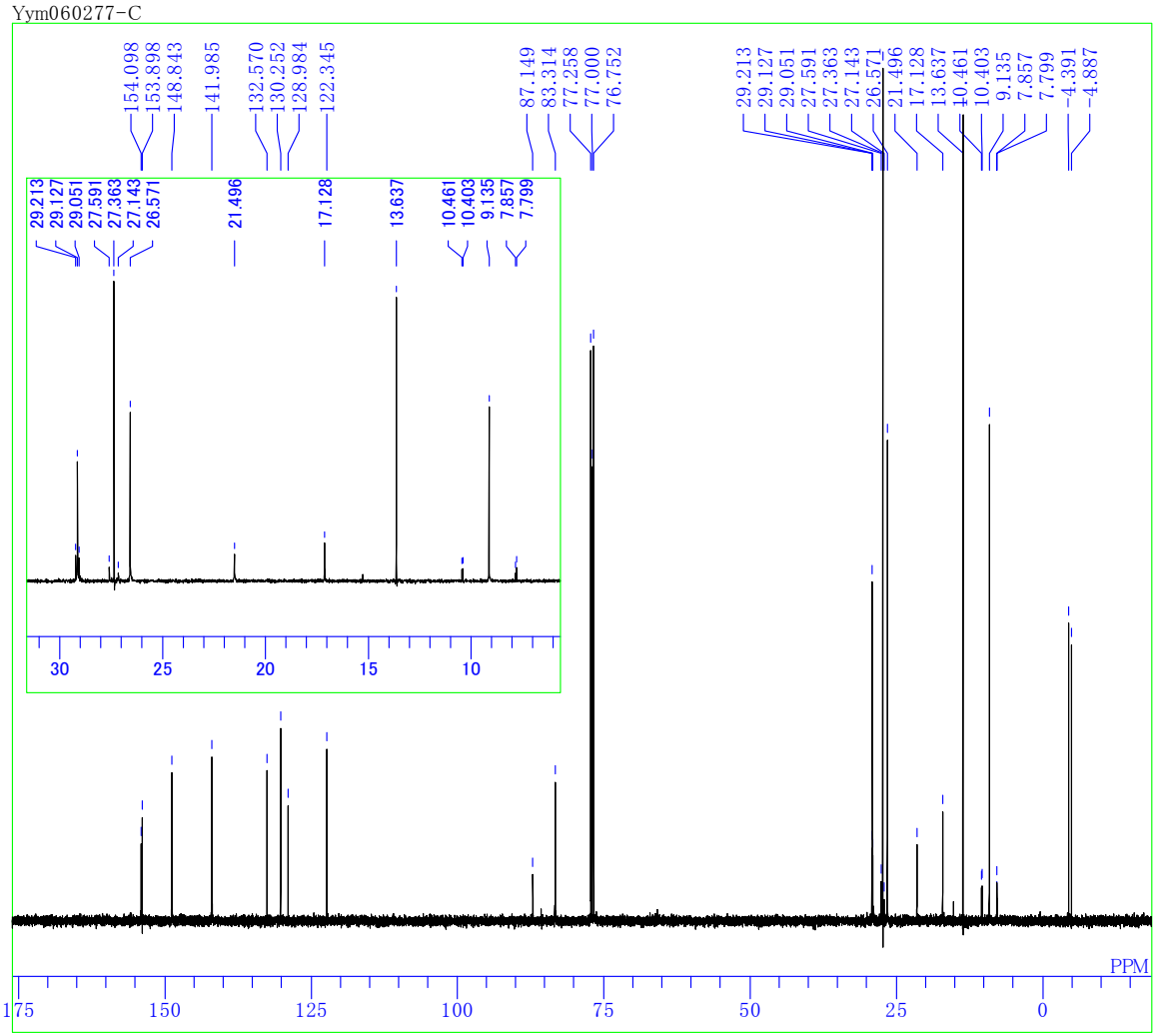


DFILE	C:¥Documents and Settings¥Owner
COMNT	Yym060277-1
DATIM	Tue May 27 23:25:08 2008
OBNUC	1H
EXMOD	SINGL
OBFRQ	499.10 MHz
OBSET	0.00 KHz
OBFIN	126750.00 Hz
POINT	16384
FREQU	9980.04 Hz
SCANS	8
ACQTM	1.6417 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	-204.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	23

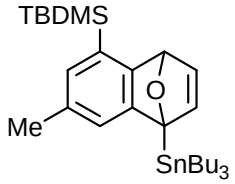


anti-**4j** ($CDCl_3$)

Table 2, entry 7



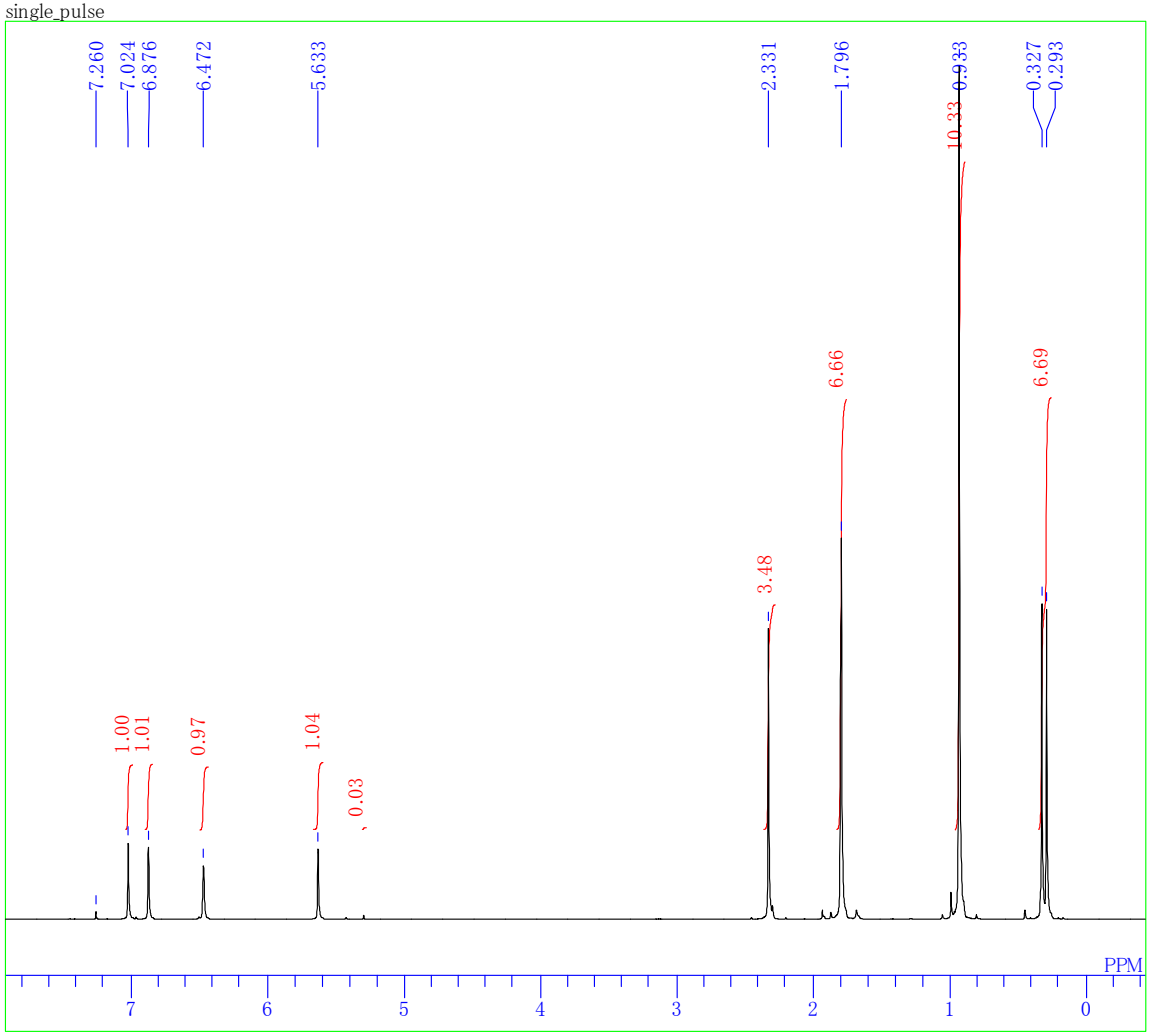
DFILE	C:¥Documents and Settings¥Owner
COMNT	Yym060277-C
DATIM	30-05-2008 14:07:56
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.06 Hz
SCANS	431
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	21.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60



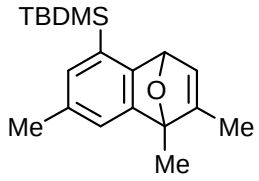
anti-**4j** ($CDCl_3$)

Table 2, entry 7

single_pulse

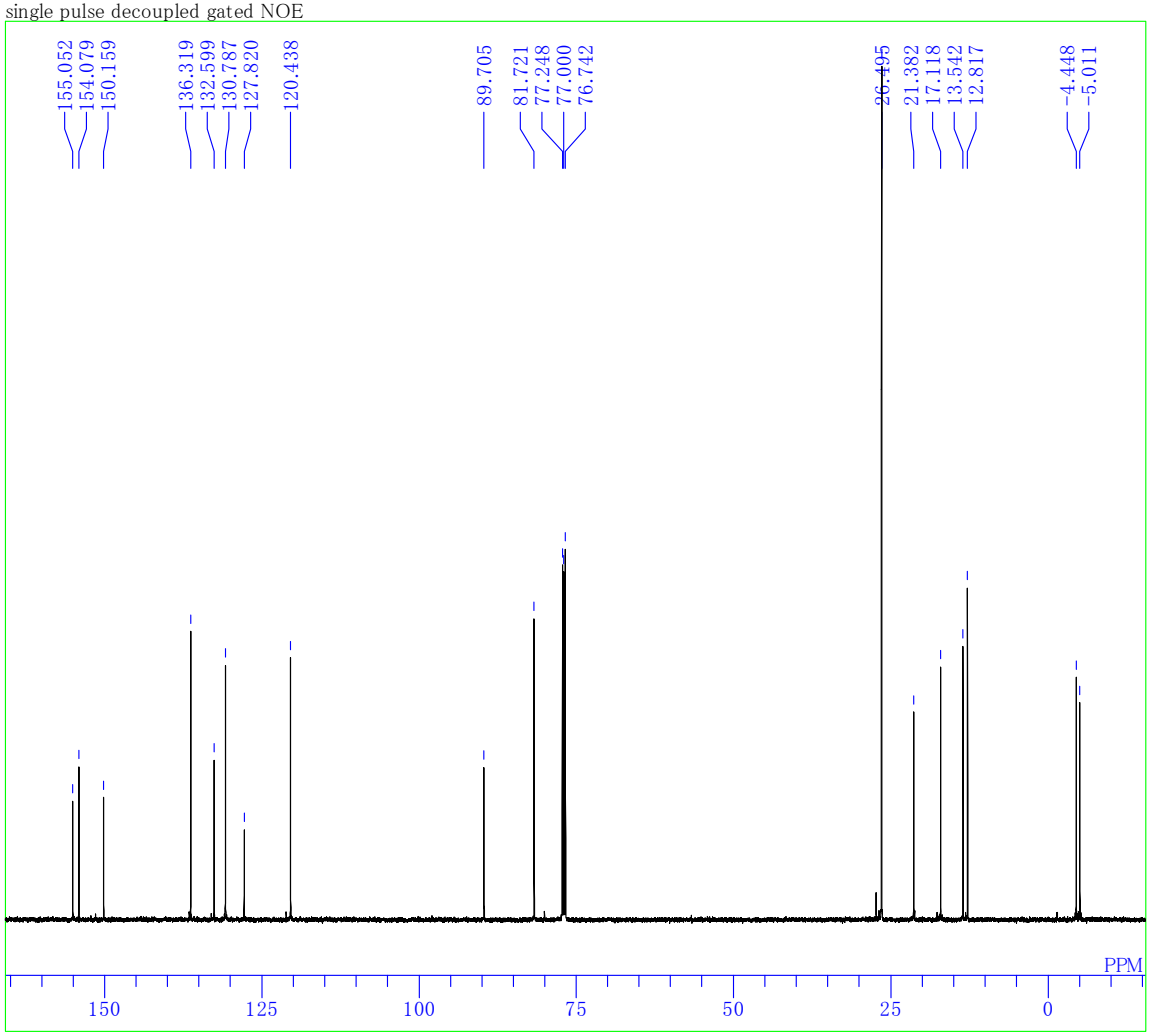


DFILE	C:¥Documents and Settings¥Owner
COMNT	single_pulse
DATIM	04-06-2008 10:54:06
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.39 Hz
SCANS	8
ACQTM	1.7459 sec
PD	1.5000 sec
PW1	5.75 usec
IRNUC	1H
CTEMP	20.4 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	1.20 Hz
RGAIN	34

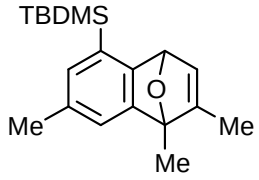


anti-**4k** ($CDCl_3$)
Table 2, entry 8

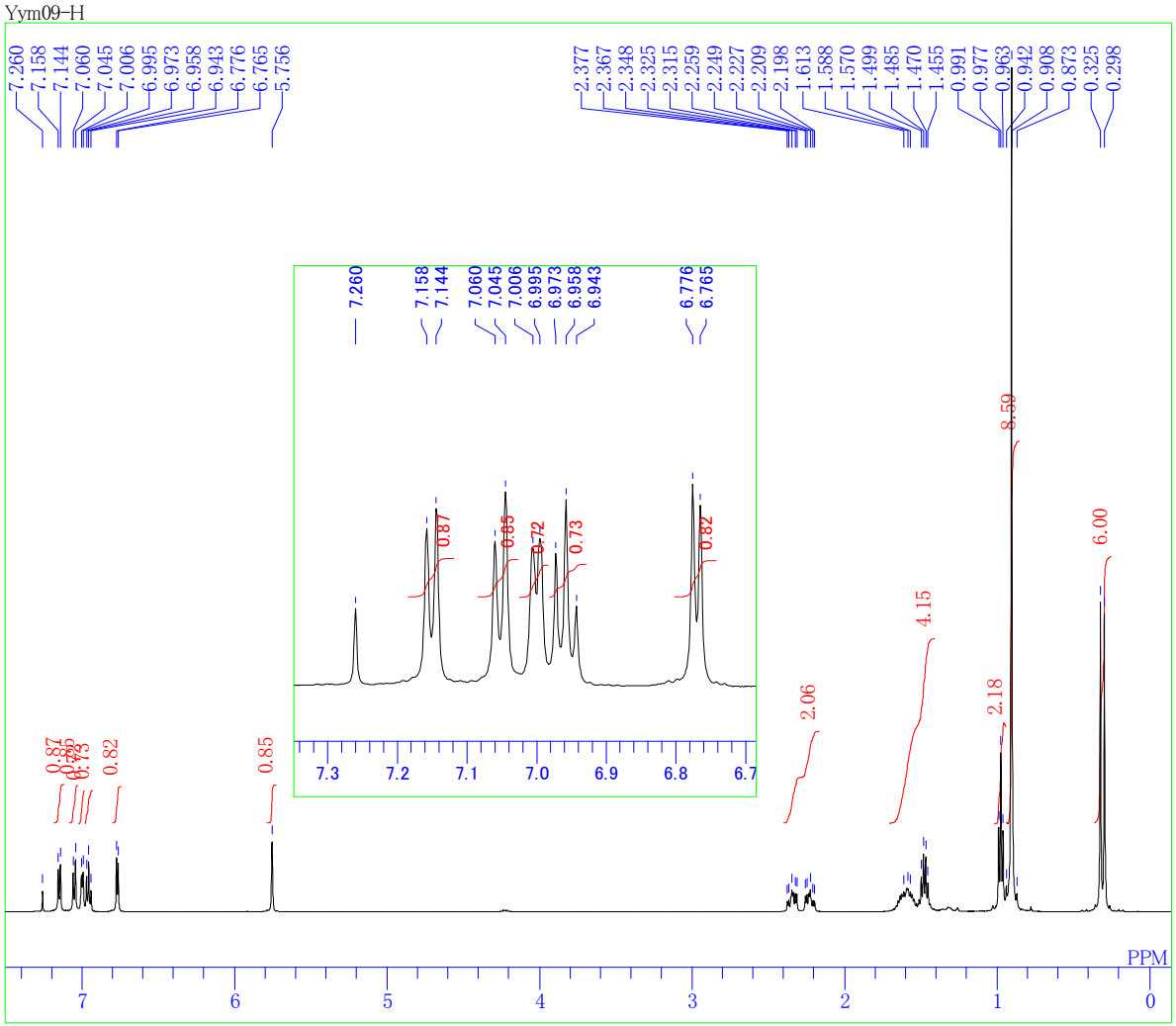
single pulse decoupled gated NOE



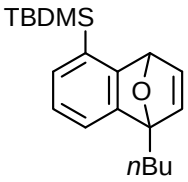
DFILE	C:¥Documents and Settings¥Owner
COMNT	single pulse decoupled gated NOE
DATIM	04-06-2008 11:33:06
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.06 Hz
SCANS	761
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	20.7 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60



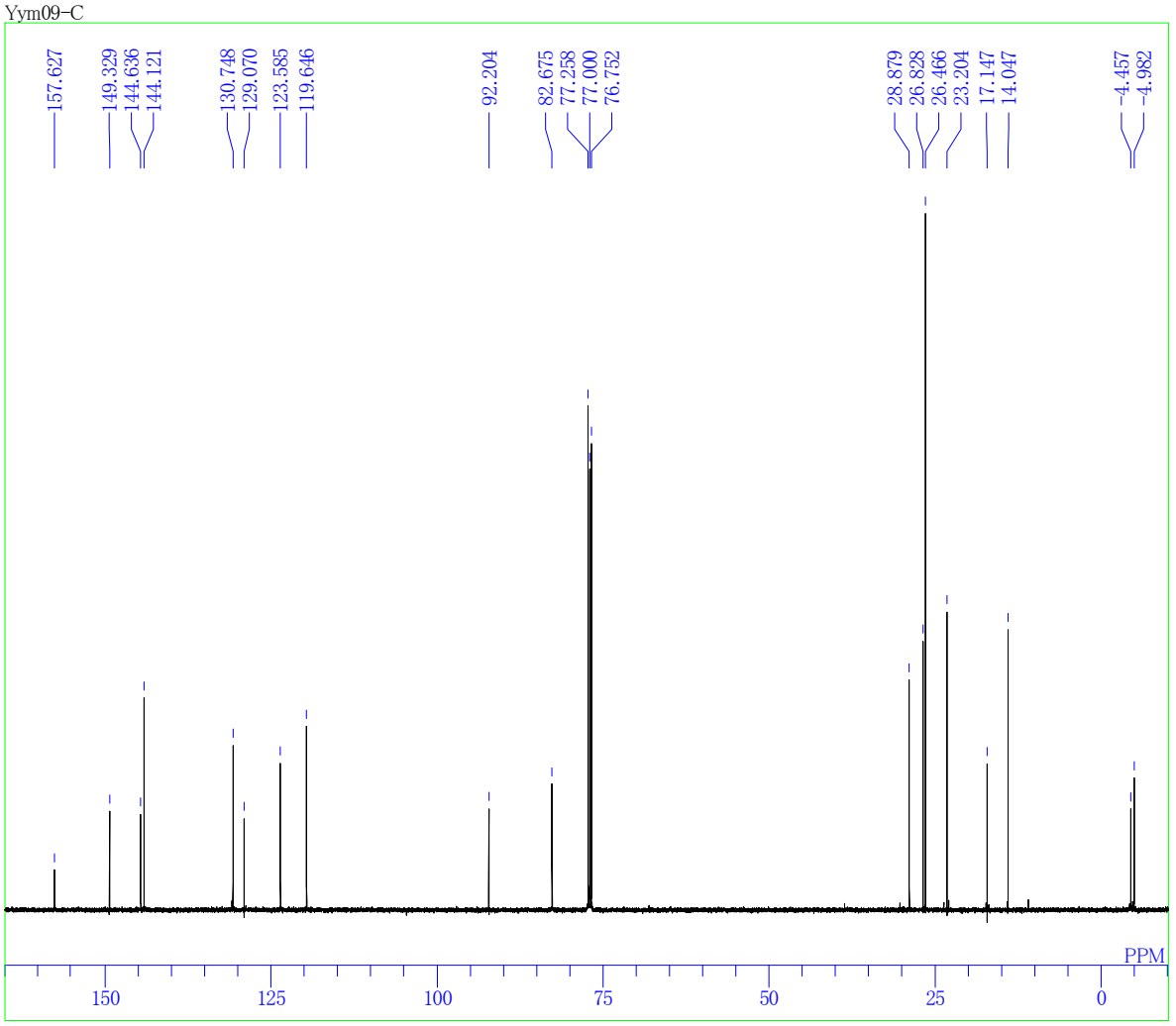
anti-**4k** ($CDCl_3$)
Table 2, entry 8



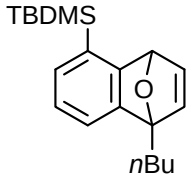
DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	Yym09-H
DATIM	01-02-2008 11:00:46
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16384
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	1.5000 sec
PW1	6.00 usec
IRNUC	1H
CTEMP	16.7 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	40



anti-**4l** ($CDCl_3$)
Table 2, entry 9

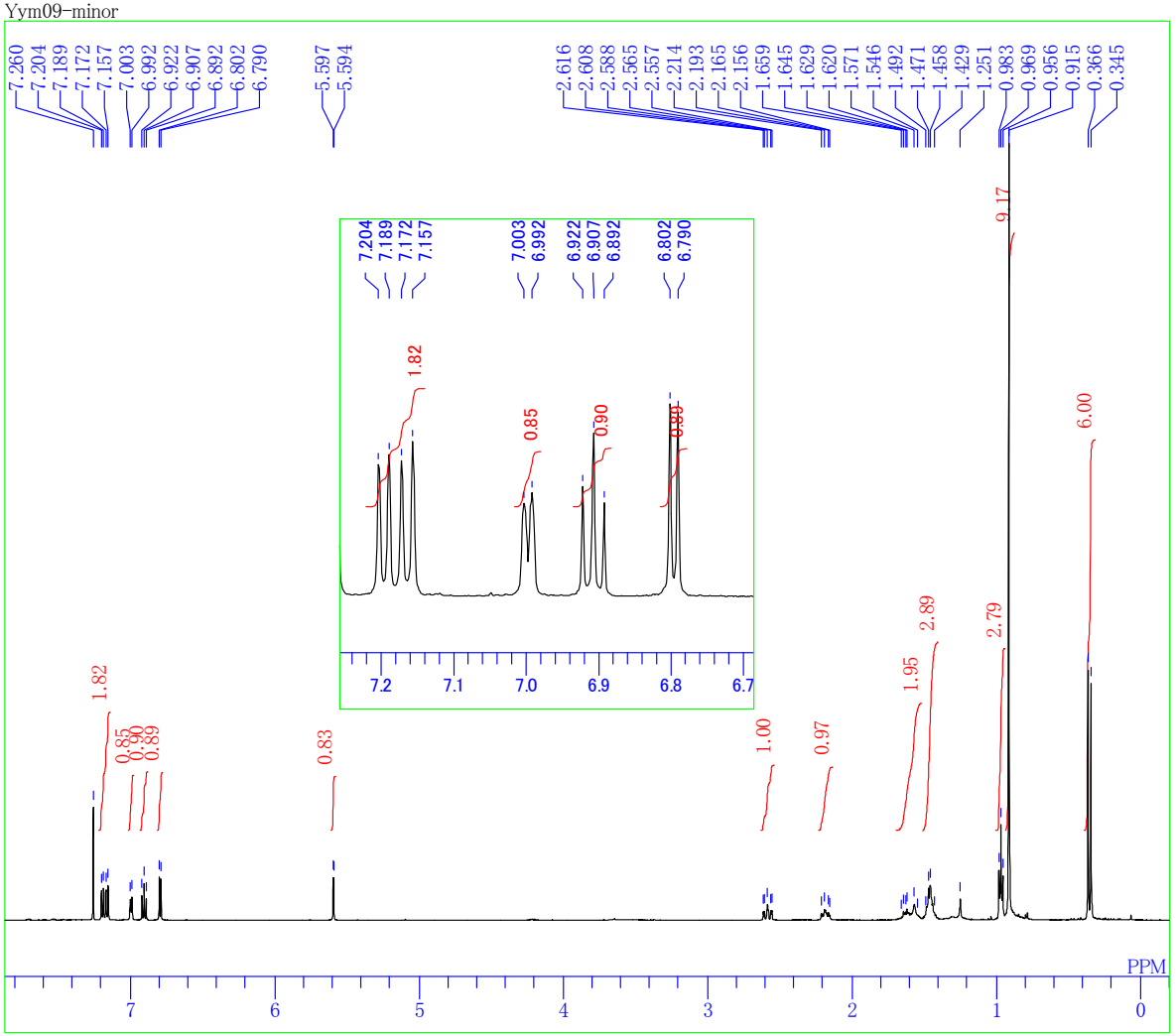


DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	Yym09-C
DATIM	01-02-2008 12:53:48
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32768
FREQU	39308.18 Hz
SCANS	2328
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	17.5 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

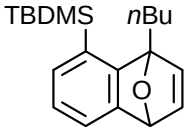


anti-**4l** ($CDCl_3$)

Table 2, entry 9



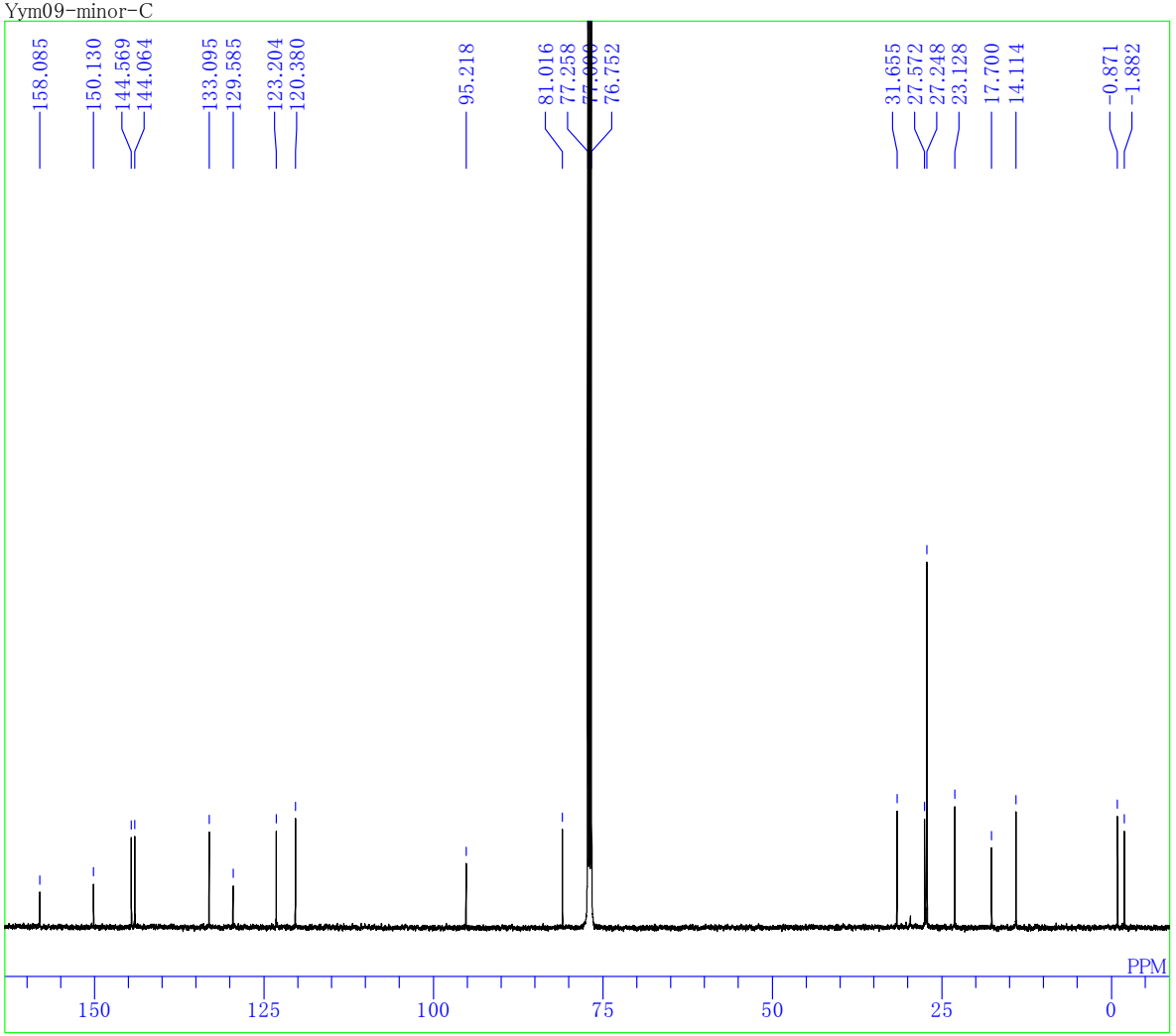
DFILE	C:¥Documents and Settings¥Owner¥デ
COMNT	Yym09-minor
DATIM	18-02-2008 20:36:21
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16384
FREQU	9384.38 Hz
SCANS	32
ACQTM	1.7459 sec
PD	1.5000 sec
PW1	6.00 usec
IRNUC	1H
CTEMP	16.4 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	50



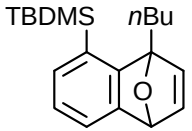
syn-**4l** ($CDCl_3$)

Table 2, entry 9

Yym09-minor-C

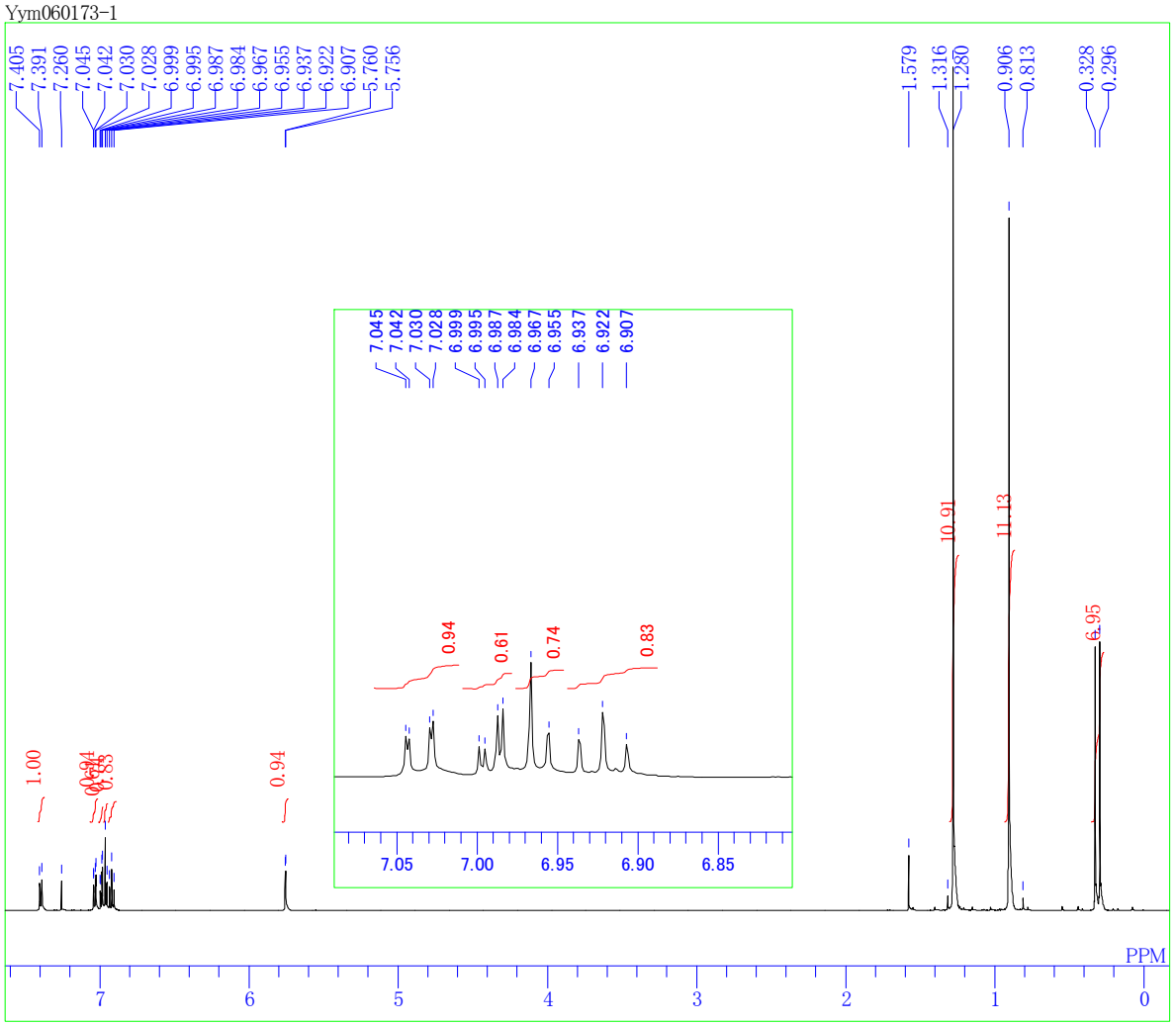


DFILE	C:¥Documents and Settings¥Owner
COMNT	Yym09-minor-C
DATIM	19-02-2008 08:37:50
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	40961
FREQU	49135.97 Hz
SCANS	15050
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	17.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

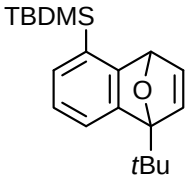


syn-**4l** ($CDCl_3$)

Table 2, entry 9

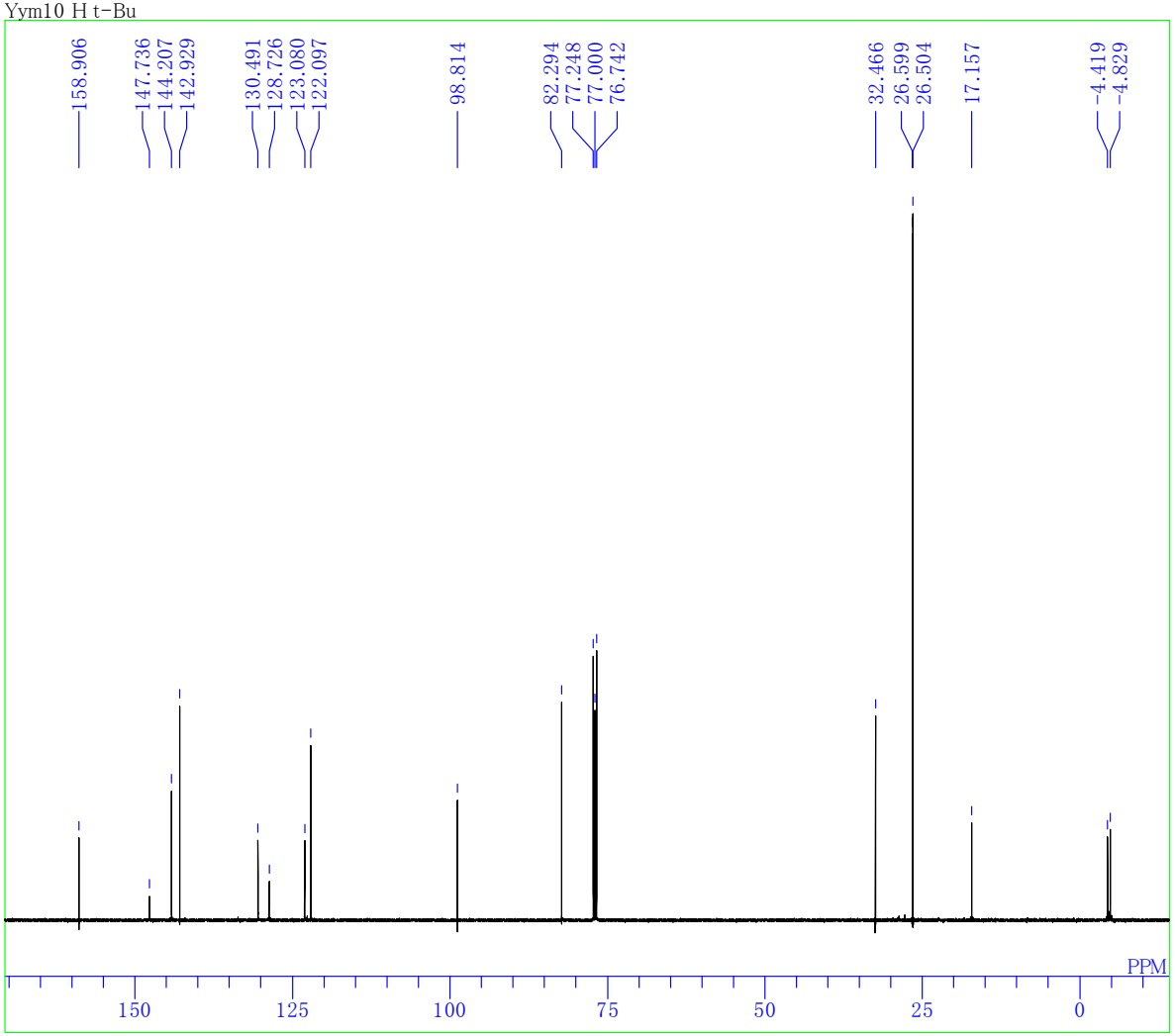


DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	Yym060173-1
DATIM	02-10-2007 15:27:38
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.39 Hz
SCANS	16
ACQTM	1.7459 sec
PD	1.5000 sec
PW1	6.00 usec
IRNUC	1H
CTEMP	20.2 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.00 Hz
RGAIN	44

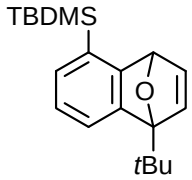


anti-**4m** ($CDCl_3$)
Table 2, entry 10

Yym10 H t-Bu

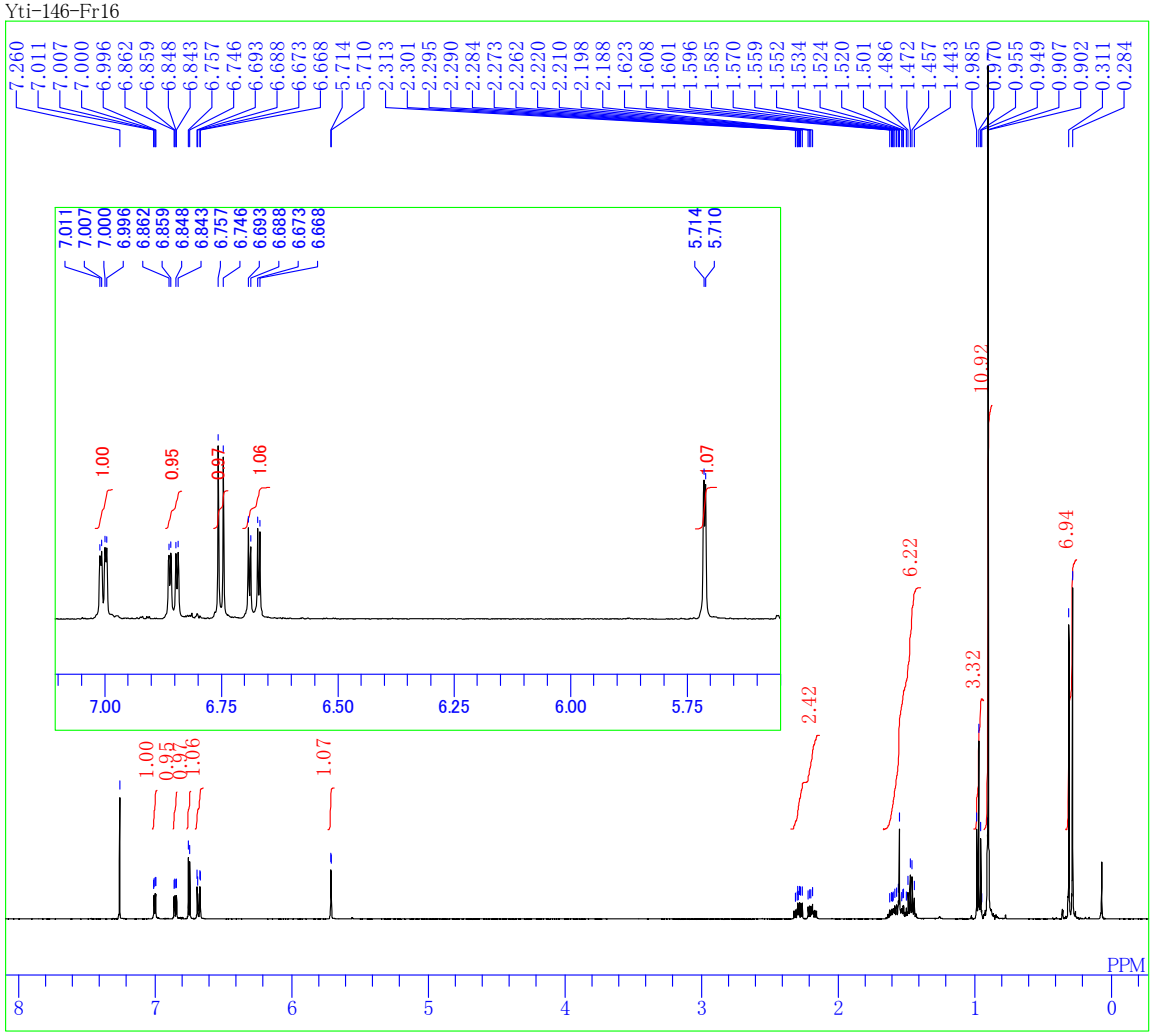


DFILE	C:¥Documents and Settings¥Owner
COMNT	Yym10 H t-Bu
DATIM	19-01-2008 22:00:44
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.06 Hz
SCANS	1575
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	17.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

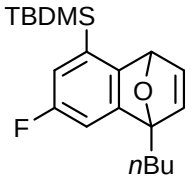


anti-**4m** ($CDCl_3$)
Table 2, entry 10

Yti-146-Fr16



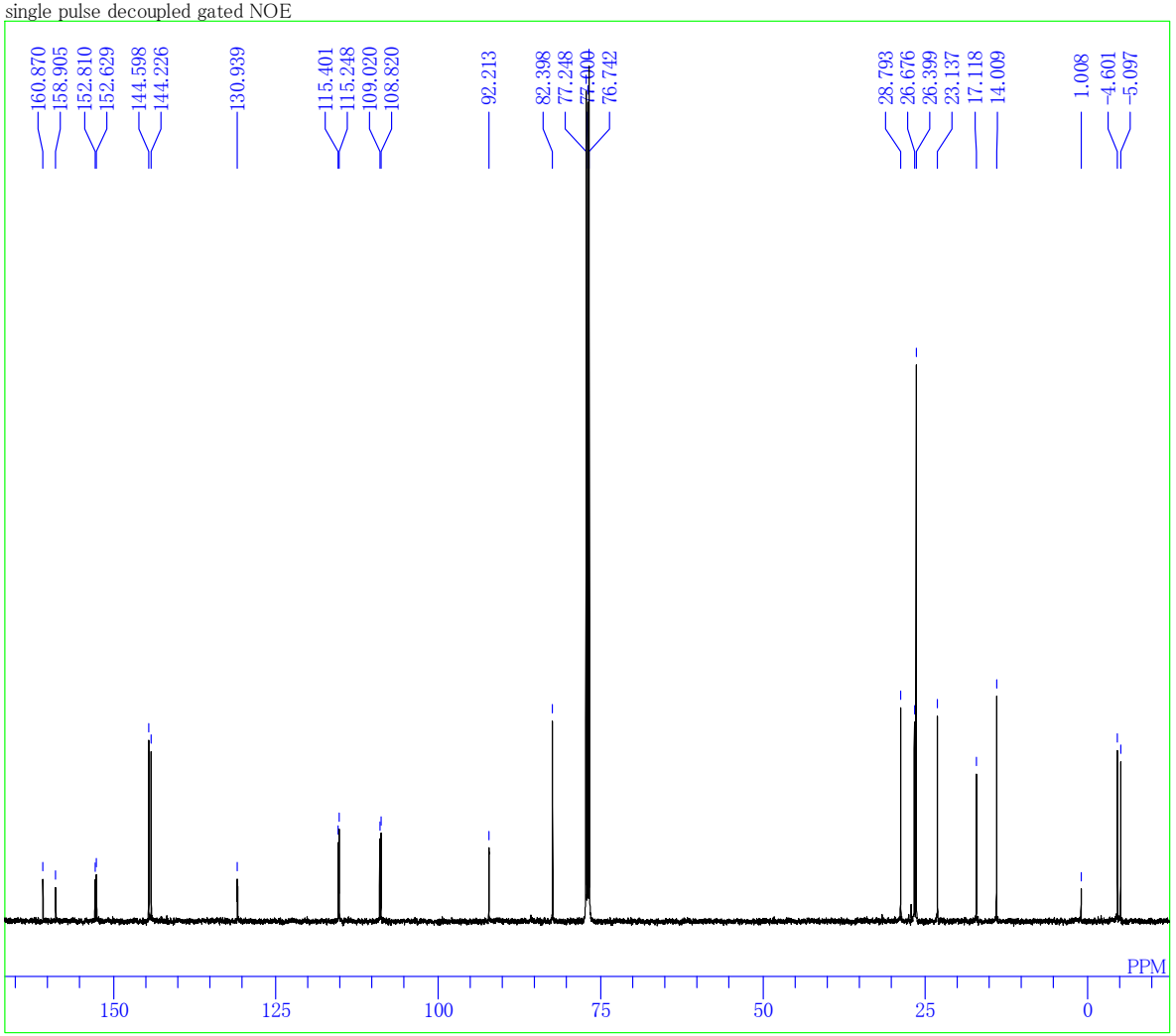
DFILE	F:¥NMRdata¥backupNMR2.9.2008¥Yt
COMNT	Yti-146-Fr16
DATIM	Sat Jan 26 11:36:40 2008
OBNUC	1H
EXMOD	SINGL
OBFRQ	499.10 MHz
OBSET	0.00 KHz
OBFIN	125250.00 Hz
POINT	16384
FREQU	9980.04 Hz
SCANS	8
ACQTM	1.6417 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	-204.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	22



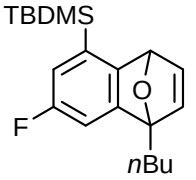
anti-**4n** ($CDCl_3$)

Table 2, entry 11

single pulse decoupled gated NOE

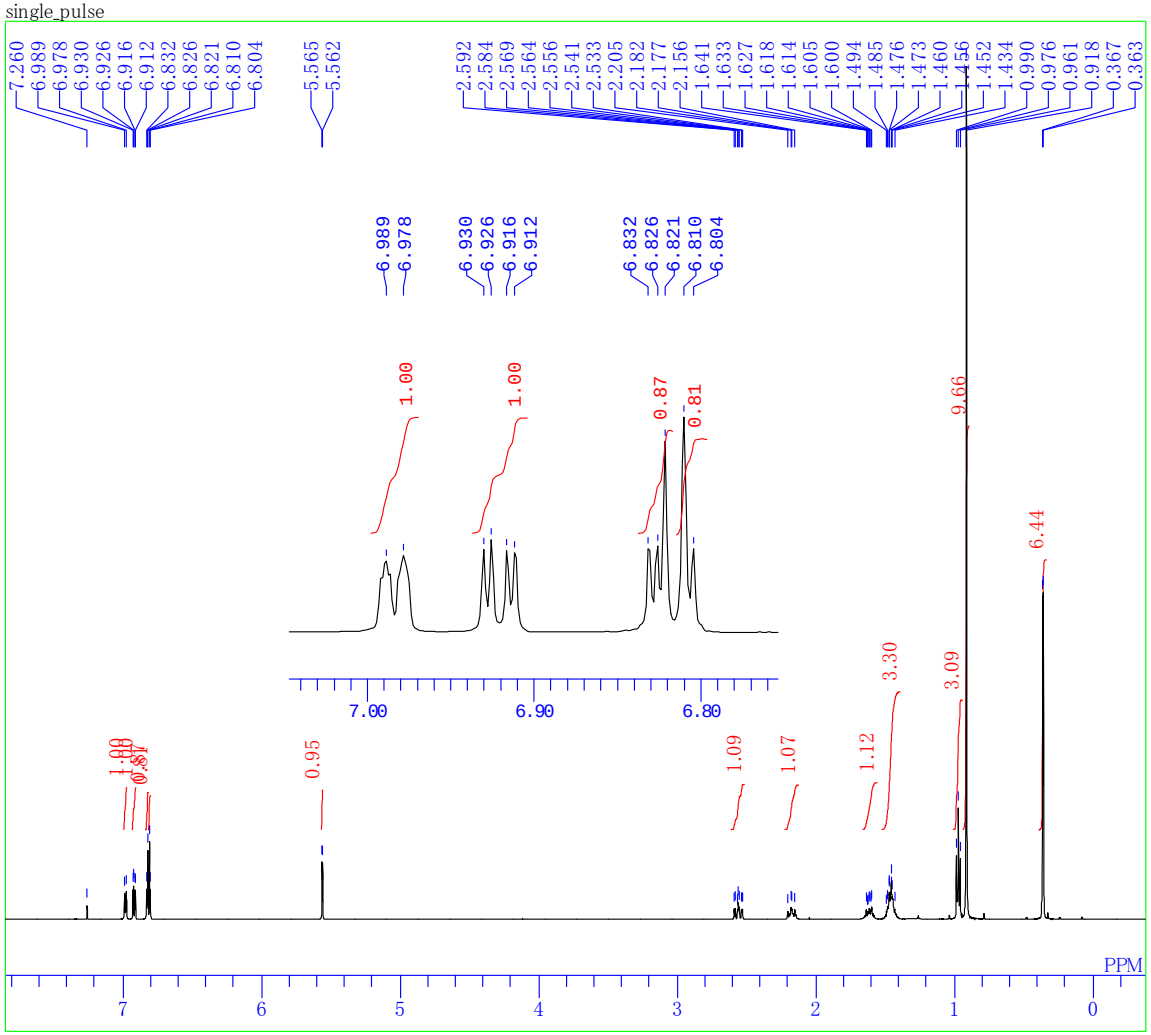


DFILE	F:¥backupNMR2.9.2008¥Yti¥ECA¥Yti
COMNT	single pulse decoupled gated NOE
DATIM	26-01-2008 16:37:59
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	40961
FREQU	49135.97 Hz
SCANS	1825
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	17.0 c
SLVNT	CDCL3
EXREF	224.89 ppm
BF	0.12 Hz
RGAIN	60

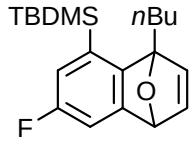


anti-**4n** ($CDCl_3$)

Table 2, entry 11



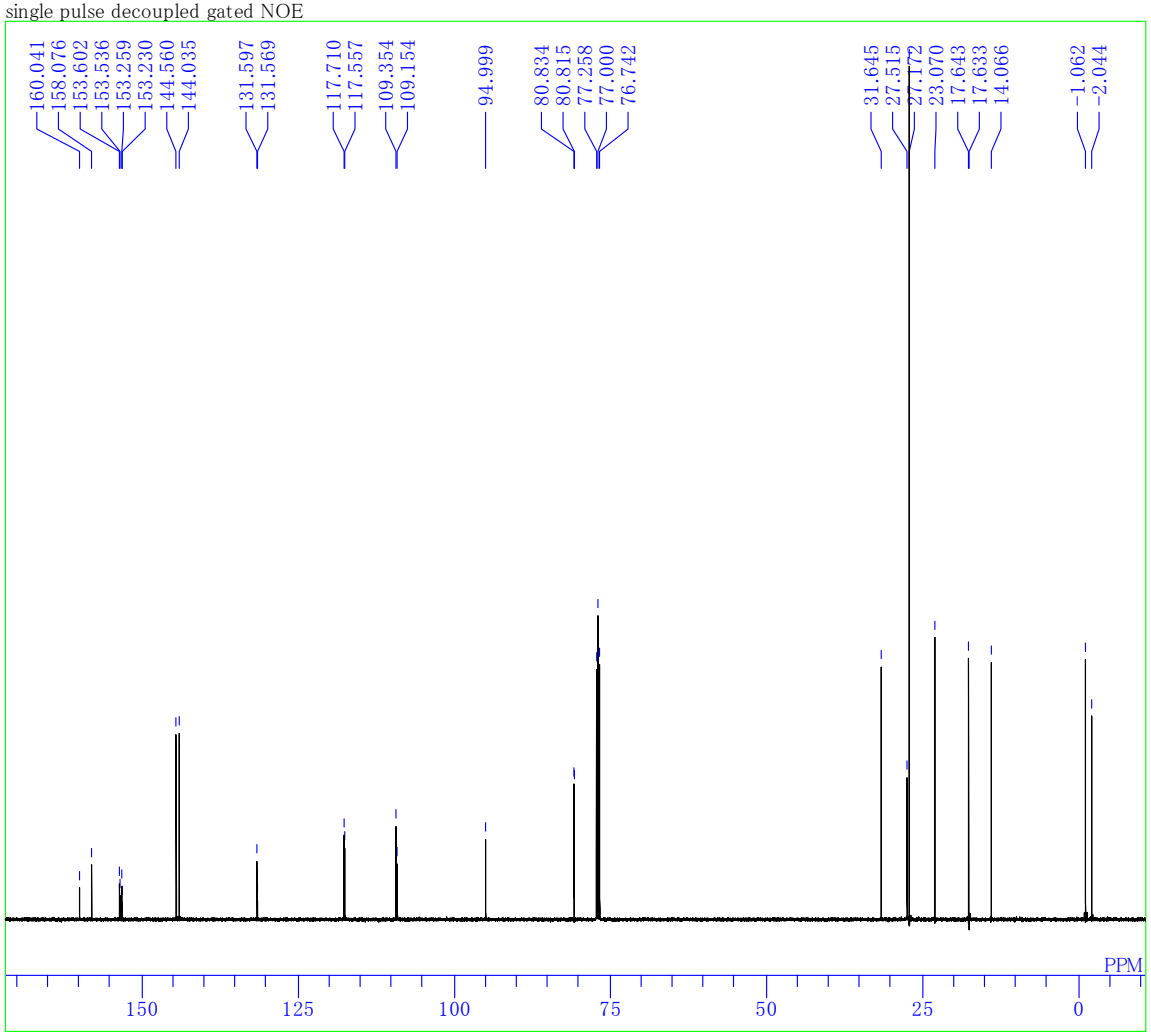
DFILE	C:¥Documents and Settings¥Owner
COMNT	single_pulse
DATIM	09-05-2008 09:19:09
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.39 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	5.75 usec
IRNUC	1H
CTEMP	20.6 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	36



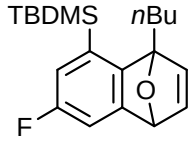
syn-**4n** ($CDCl_3$)

Table 2, entry 11

single pulse decoupled gated NOE



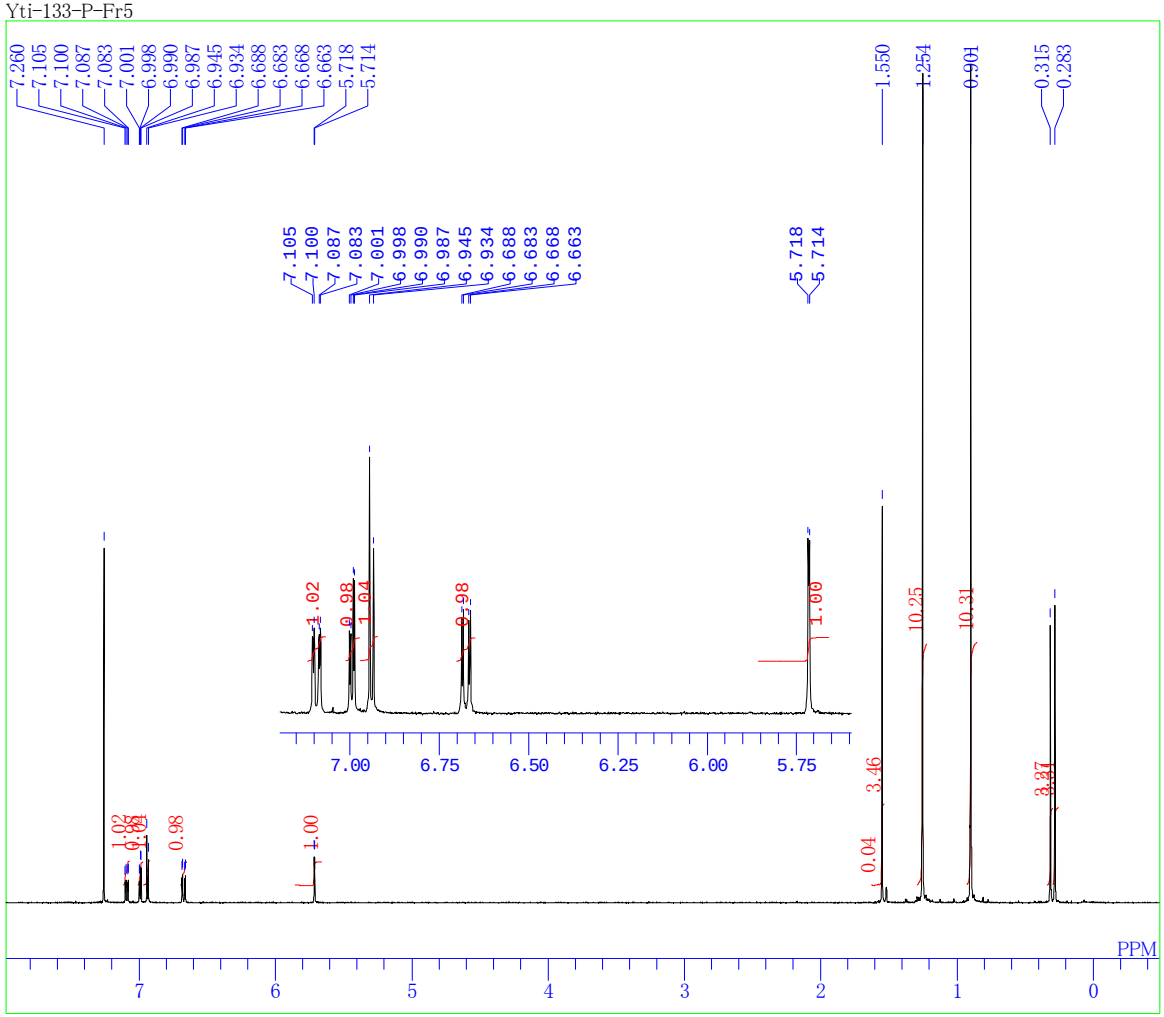
DFILE	C:¥Documents and Settings¥Owner
COMNT	single pulse decoupled gated NOE
DATIM	09-05-2008 10:31:10
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.06 Hz
SCANS	1469
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	21.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60



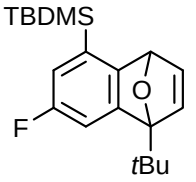
syn-**4n** ($CDCl_3$)

Table 2, entry 11

Yti-133-P-Fr5



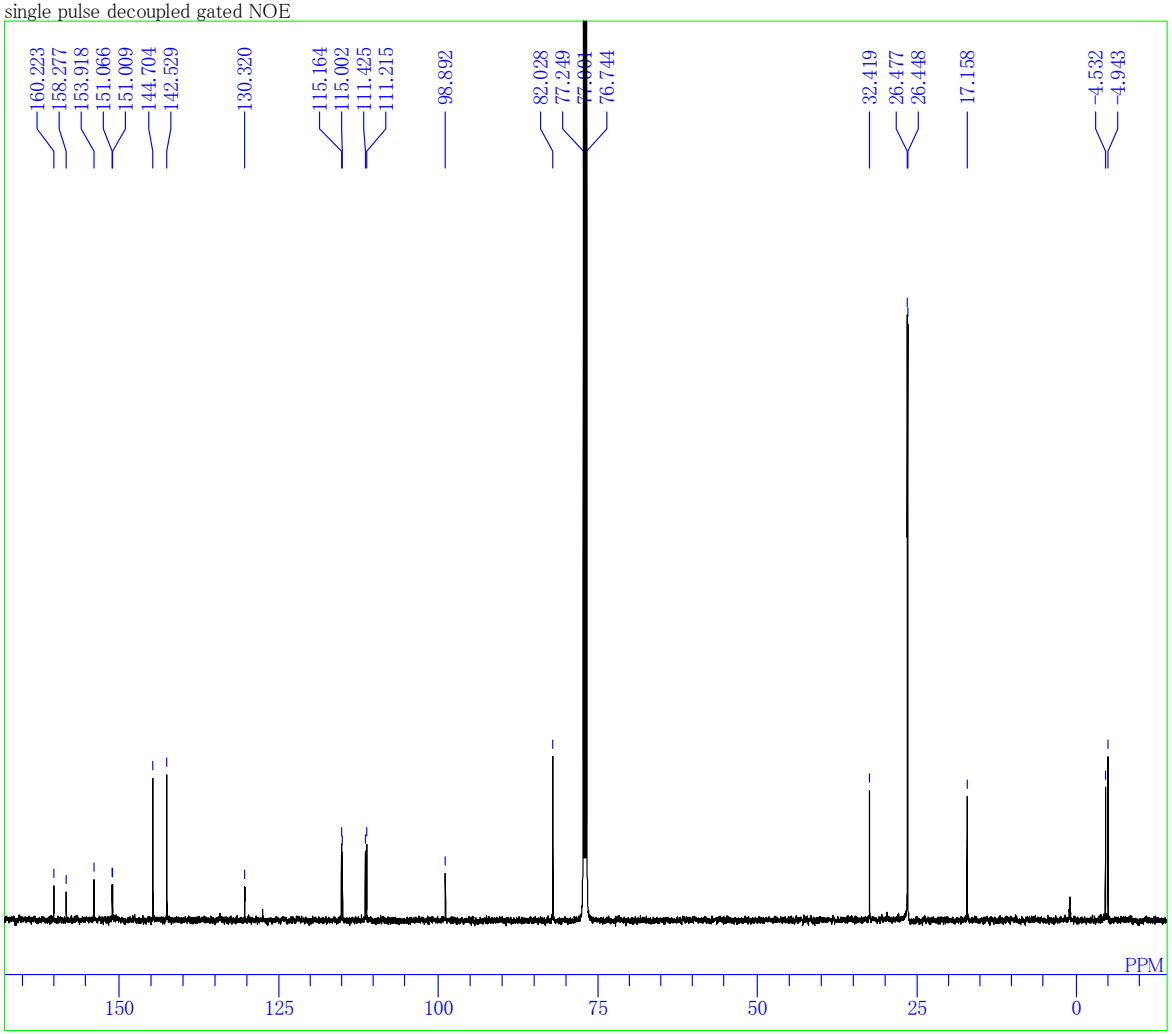
DFILE	C:¥Documents and Settings¥Owner¥デ
COMNT	Yti-133-P-Fr5
DATIM	Wed Jan 30 01:43:33 2008
OBNUC	1H
EXMOD	SINGL
OBFRQ	499.10 MHz
OBSET	0.00 KHz
OBFIN	125250.00 Hz
POINT	16384
FREQU	9980.04 Hz
SCANS	8
ACQTM	1.6417 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	-204.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	25



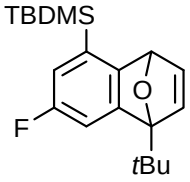
anti-**4o** ($CDCl_3$)

Table 2, entry 12

single pulse decoupled gated NOE



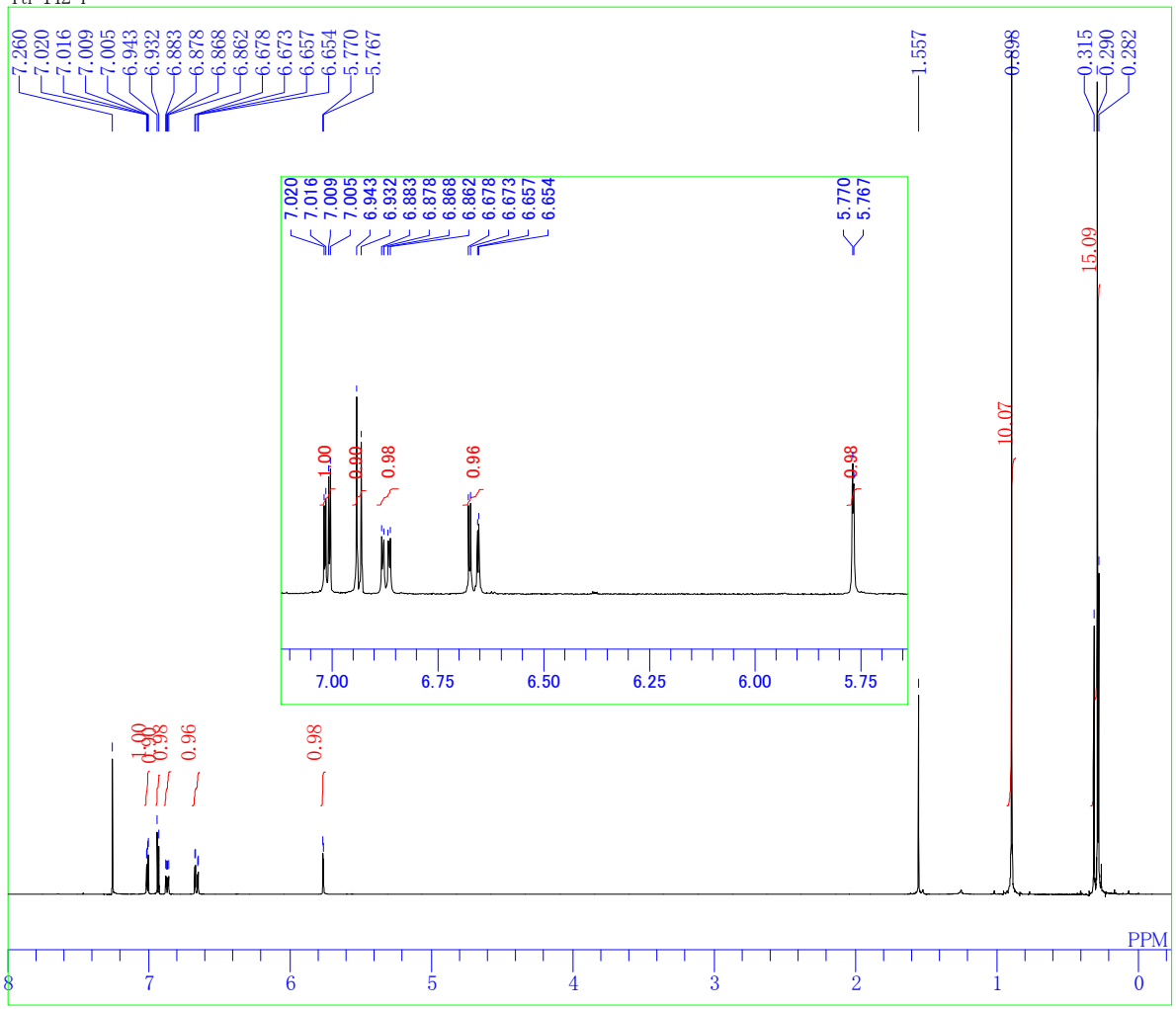
DFILE	F:¥backupNMR2.9.2008¥Yti¥ECA¥Yti
COMNT	single pulse decoupled gated NOE
DATIM	01-02-2008 08:43:29
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	40961
FREQU	49135.97 Hz
SCANS	10005
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	17.0 c
SLVNT	CDCL3
EXREF	224.90 ppm
BF	0.12 Hz
RGAIN	60



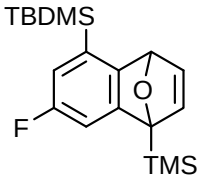
anti-**4o** ($CDCl_3$)

Table 2, entry 12

Yti-142-P'



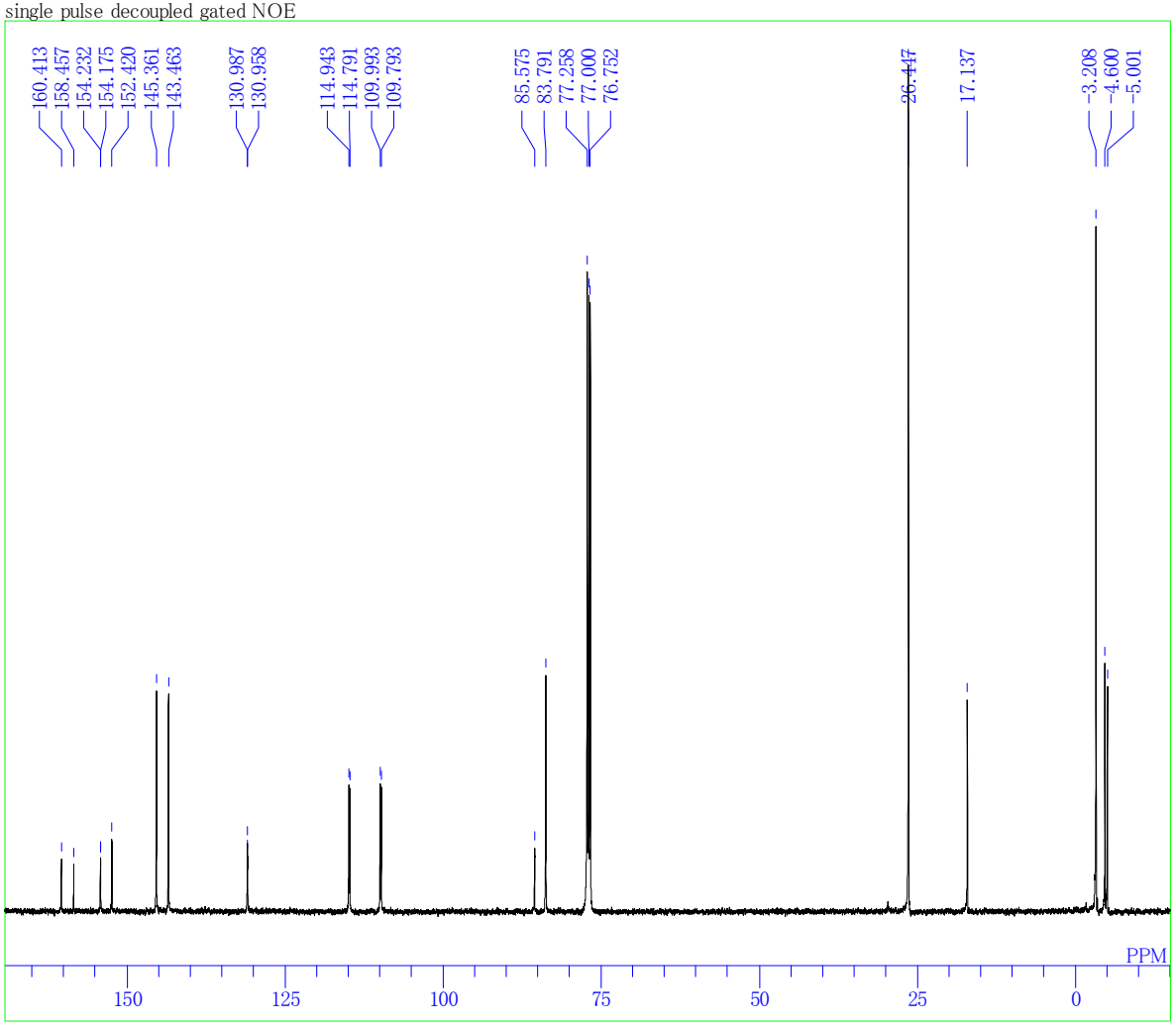
DFILE	F:¥backupNMR2.9.2008¥Yti¥未保存ラ
COMNT	Yti-142-P'
DATIM	Thu Jan 10 22:41:46 2008
OBNUC	1H
EXMOD	SINGL
OBFRQ	499.10 MHz
OBSET	0.00 KHz
OBFIN	125250.00 Hz
POINT	16384
FREQU	9980.04 Hz
SCANS	8
ACQTM	1.6417 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	-204.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.00 Hz
RGAIN	22



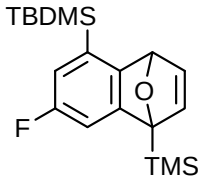
anti-**4p** ($CDCl_3$)

Table 2, entry 13

single pulse decoupled gated NOE

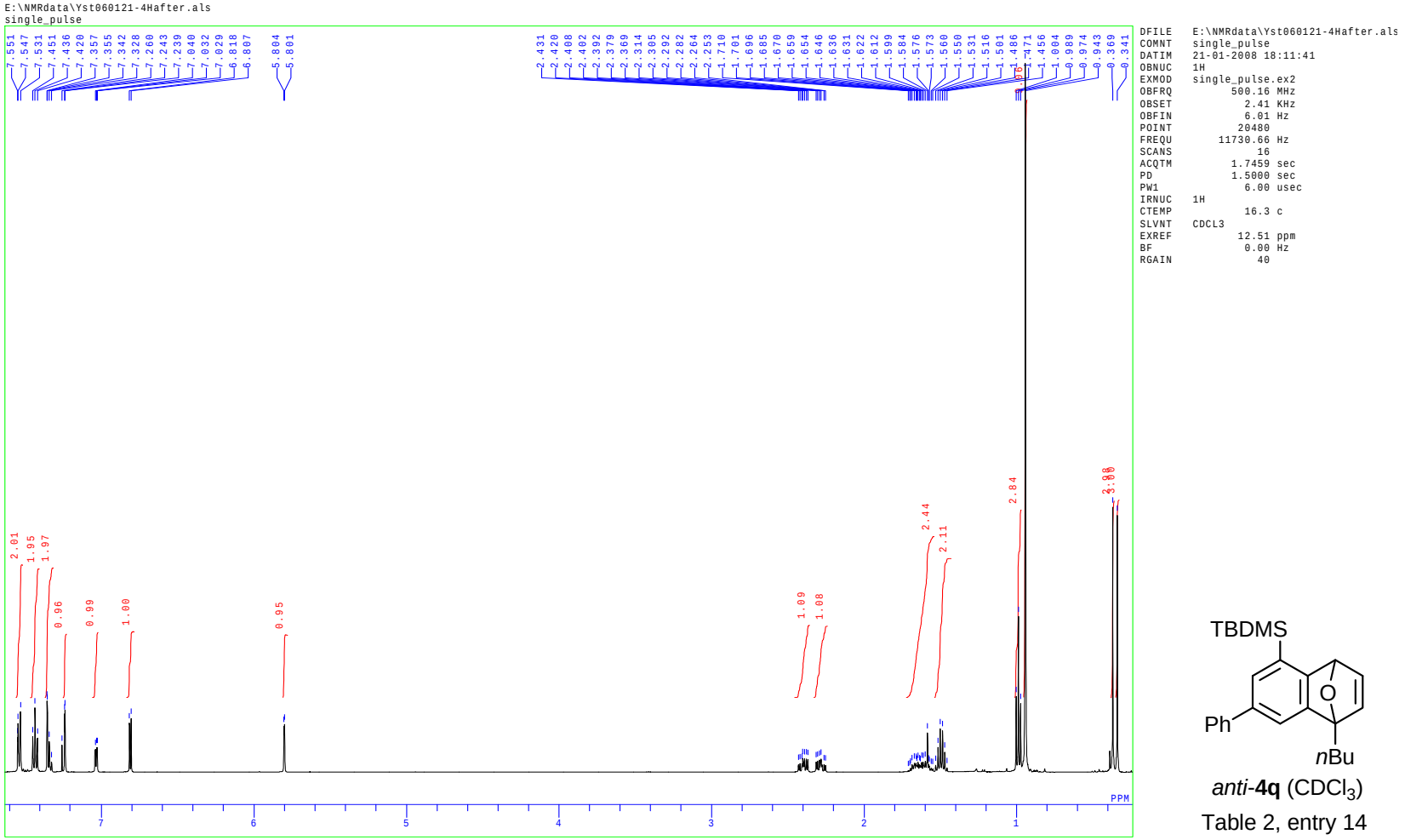


DFILE	F:¥backupNMR2.9.2008¥Yti¥ECA¥Yti
COMNT	single pulse decoupled gated NOE
DATIM	25-01-2008 21:43:04
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	40961
FREQU	49135.97 Hz
SCANS	2176
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	16.8 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60



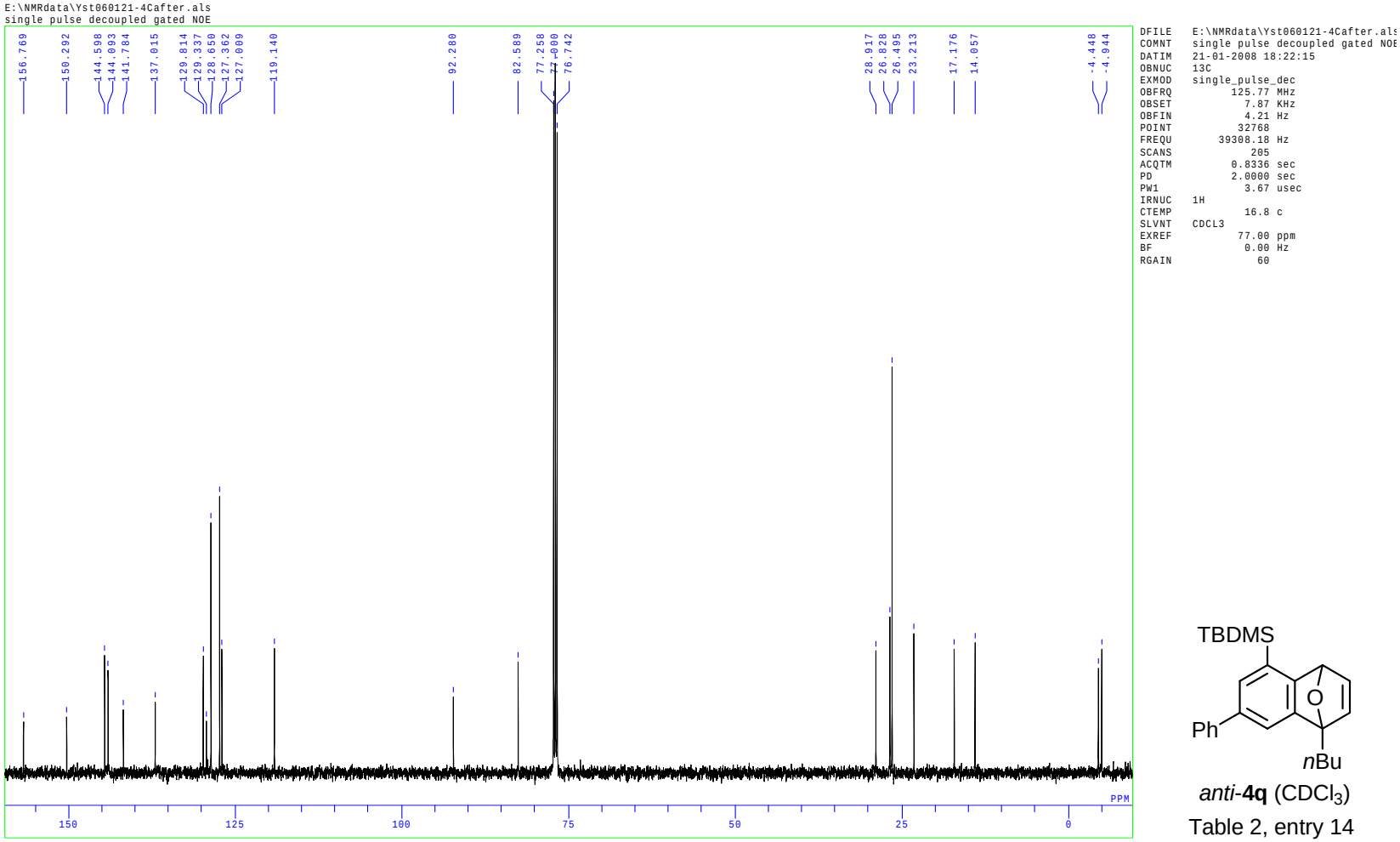
anti-**4p** ($CDCl_3$)

Table 2, entry 13



anti-**4q** ($CDCl_3$)

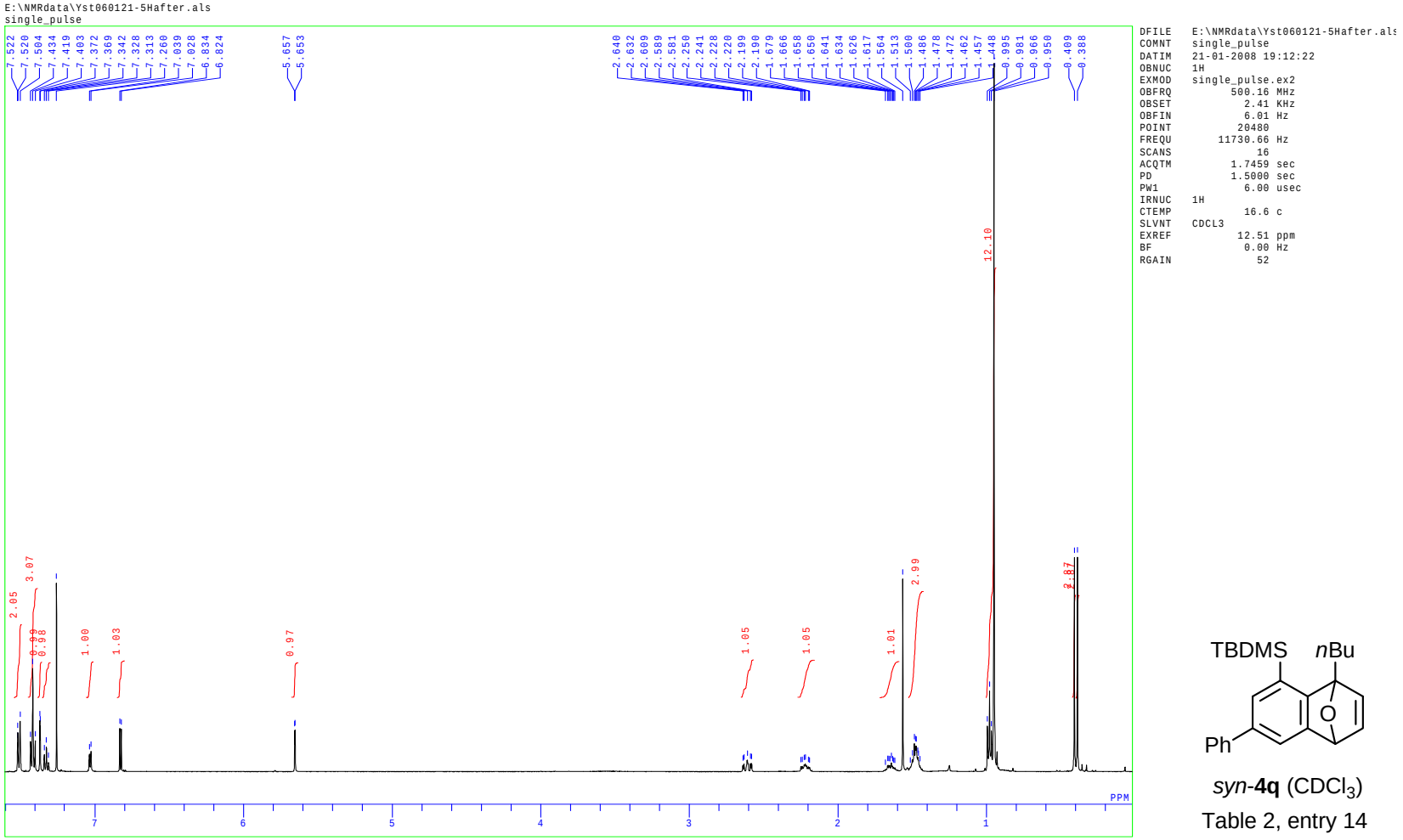
Table 2, entry 14



DFILE E:\NMRdata\Yst060121-4Cafter.als
COMNT single pulse decoupled gated NOE
DATIM 21-01-2008 18:22:15
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32768
FREQU 39308.18 Hz
SCANS 205
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 16.8 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.00 Hz
RGAIN 60

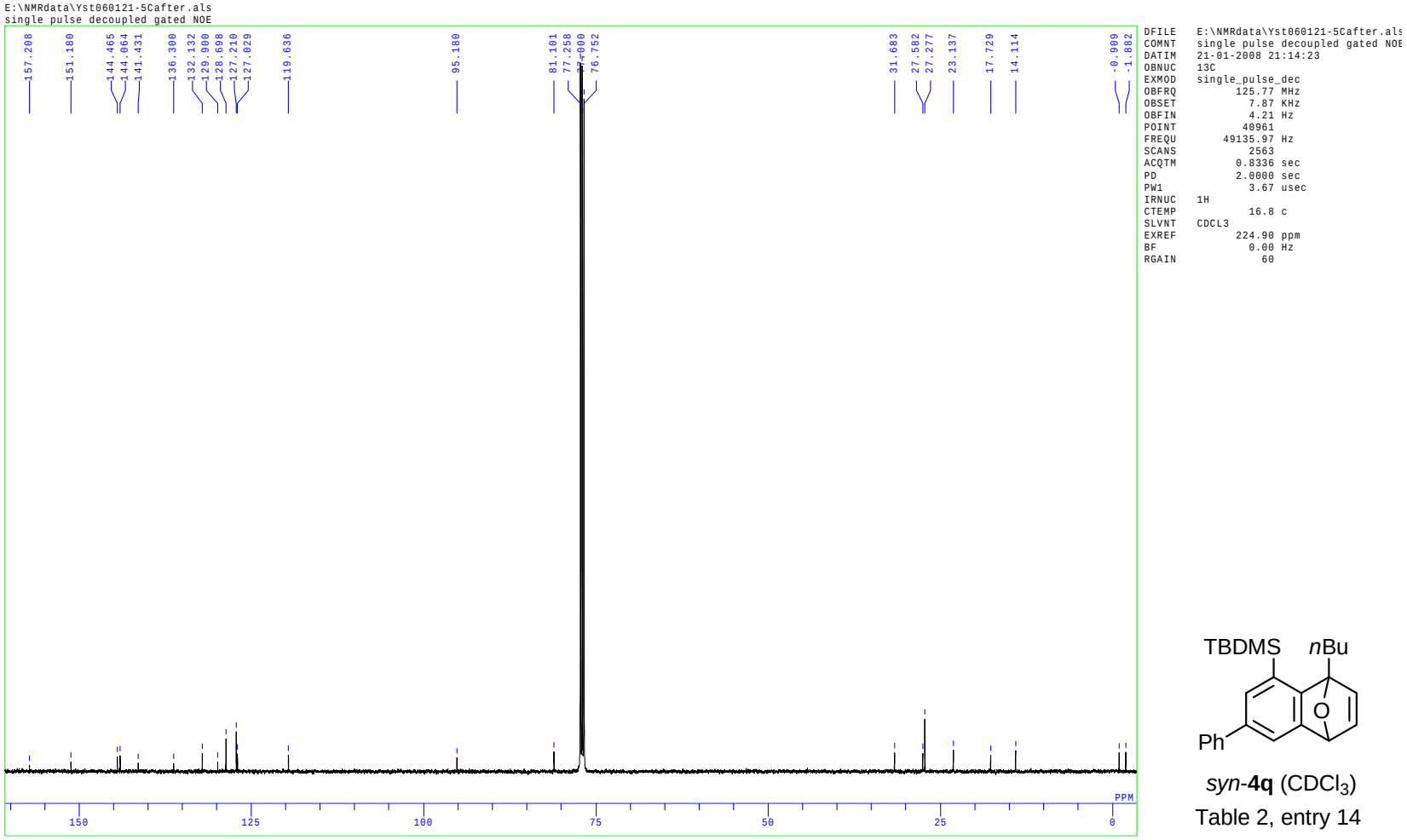
anti-**4q** ($CDCl_3$)

Table 2, entry 14



DFILE E:\NMRdata\Yst060121-5Hafter.als
COMNT single_pulse
DATIM 21-01-2008 19:12:22
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 20480
FREQU 11730.66 Hz
SCANS 16
ACQTM 1.7459 sec
PD 1.5000 sec
PW1 6.00 usec
IRNUC 1H
CTEMP 16.6 c
SLVNT CDCL3
EXREF 12.51 ppm
BF 0.00 Hz
RGAIN 52

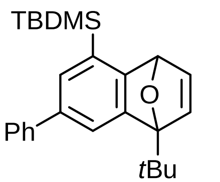
syn-**4q** ($CDCl_3$)
Table 2, entry 14




```
DFILE   E:\NMRdata\Yst060121-5Cafter.als
COMNT   single pulse decoupled gated NOE
DATIM   21-01-2008 21:14:23
OBNUC   13C
EXMOD   single_pulse_dec
OBFRQ         125.77 MHz
OBSET           7.87 KHz
OBFIN           4.21 Hz
POINT          40961
FREQU       49135.97 Hz
SCANS           2563
ACQTM         0.8336 sec
PD            2.0000 sec
PW1             3.67 usec
IRNUC   1H
CTEMP           16.8 c
SLVNT   CDCL3
EXREF         224.90 ppm
BF              0.00 Hz
RGAIN             60
```

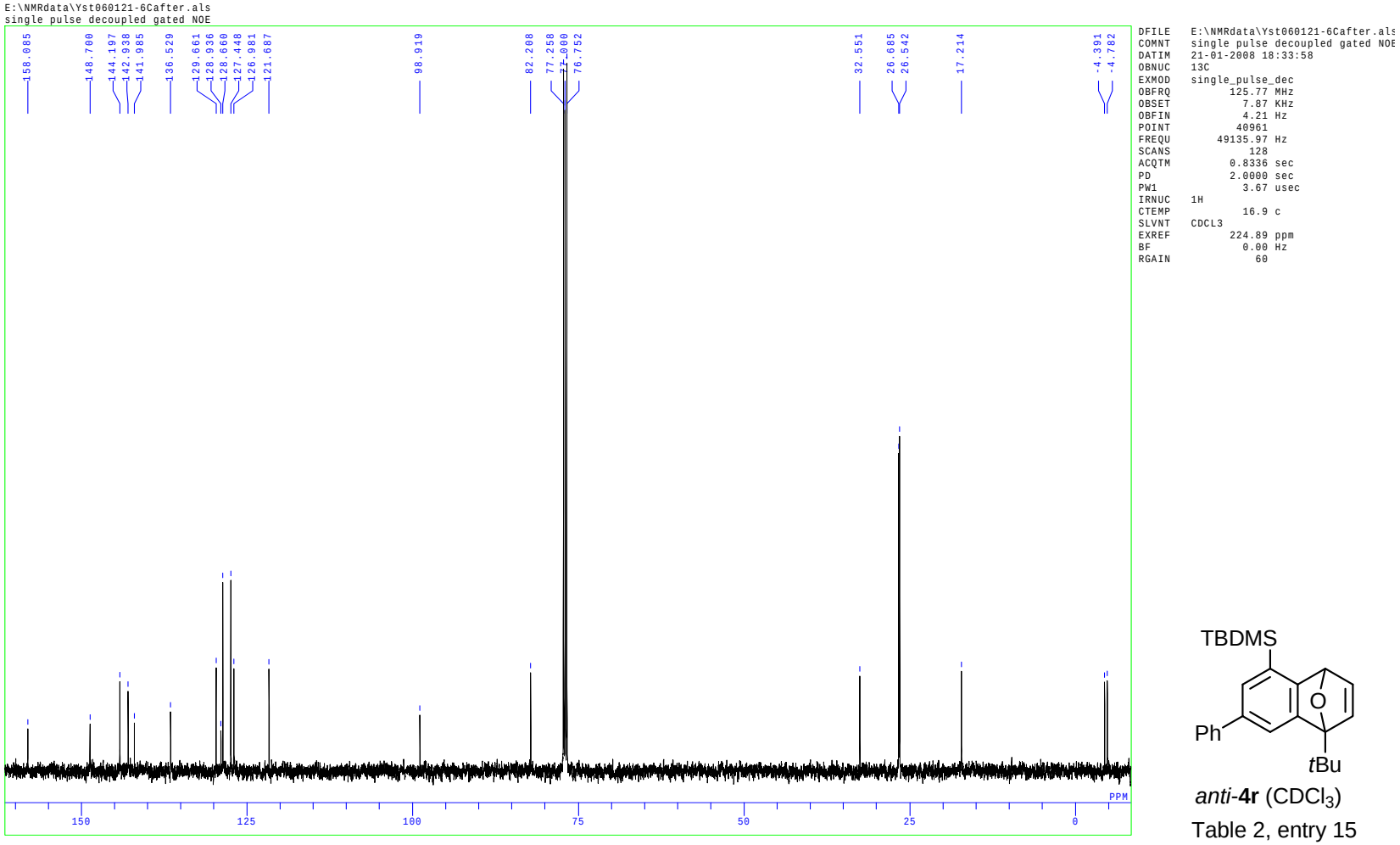

syn-**4q** ($CDCl_3$)

Table 2, entry 14



anti-**4r** ($CDCl_3$)

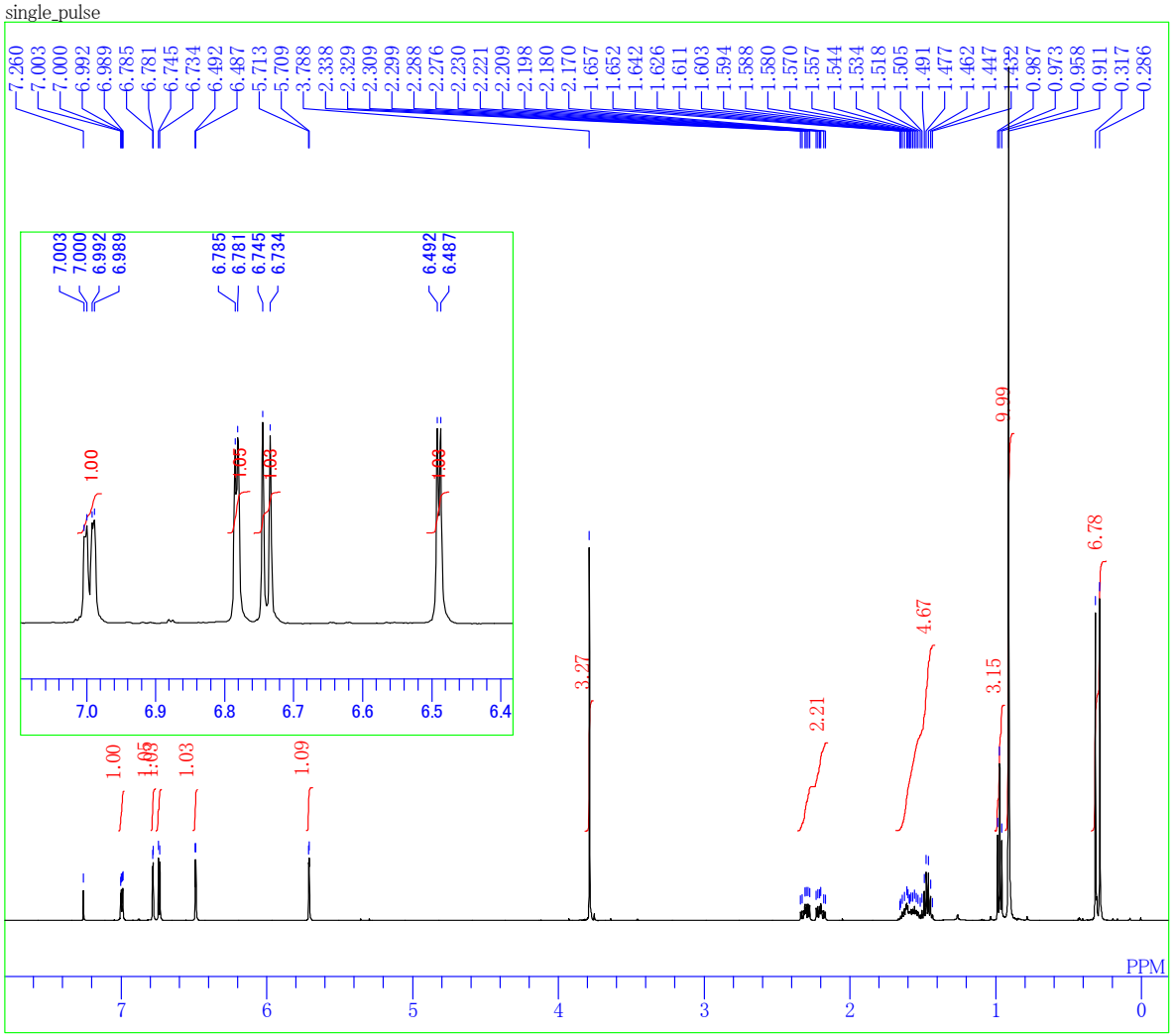
Table 2, entry 15



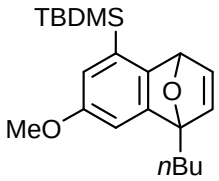
DFILE E:\NMRdata\Yst060121-6Cafter.als
COMNT single pulse decoupled gated NOE
DATIM 21-01-2008 18:33:58
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 40961
FREQU 49135.97 Hz
SCANS 128
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 16.9 c
SLVNT CDCL3
EXREF 224.89 ppm
BF 0.00 Hz
RGAIN 60

anti-**4r** ($CDCl_3$)

Table 2, entry 15



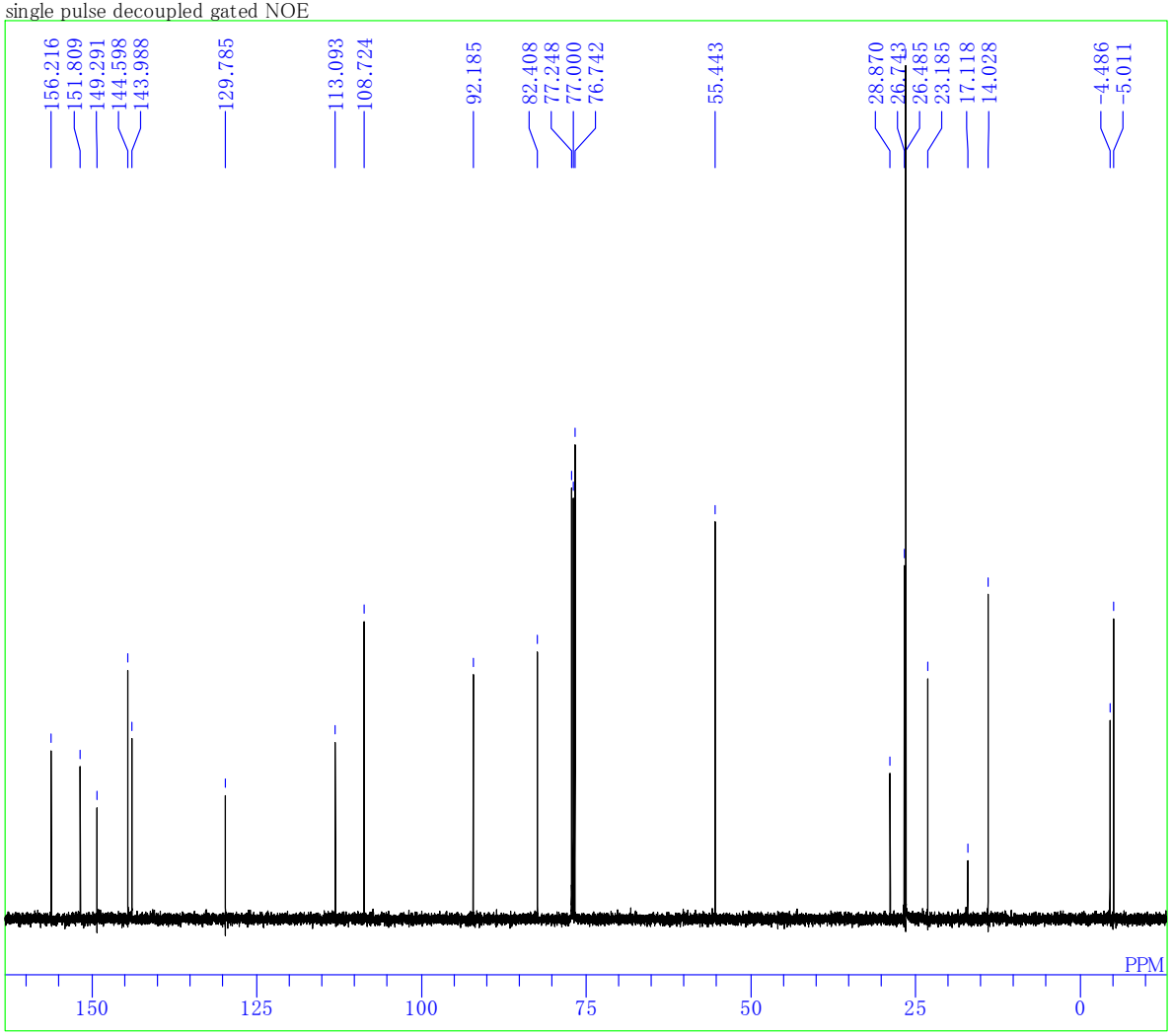
DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single_pulse
DATIM	19-12-2007 15:17:54
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16384
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	6.00 usec
IRNUC	1H
CTEMP	17.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	38



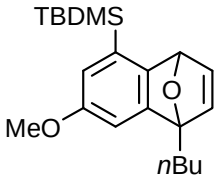
anti-**4s** ($CDCl_3$)

Table 2, entry 16

single pulse decoupled gated NOE

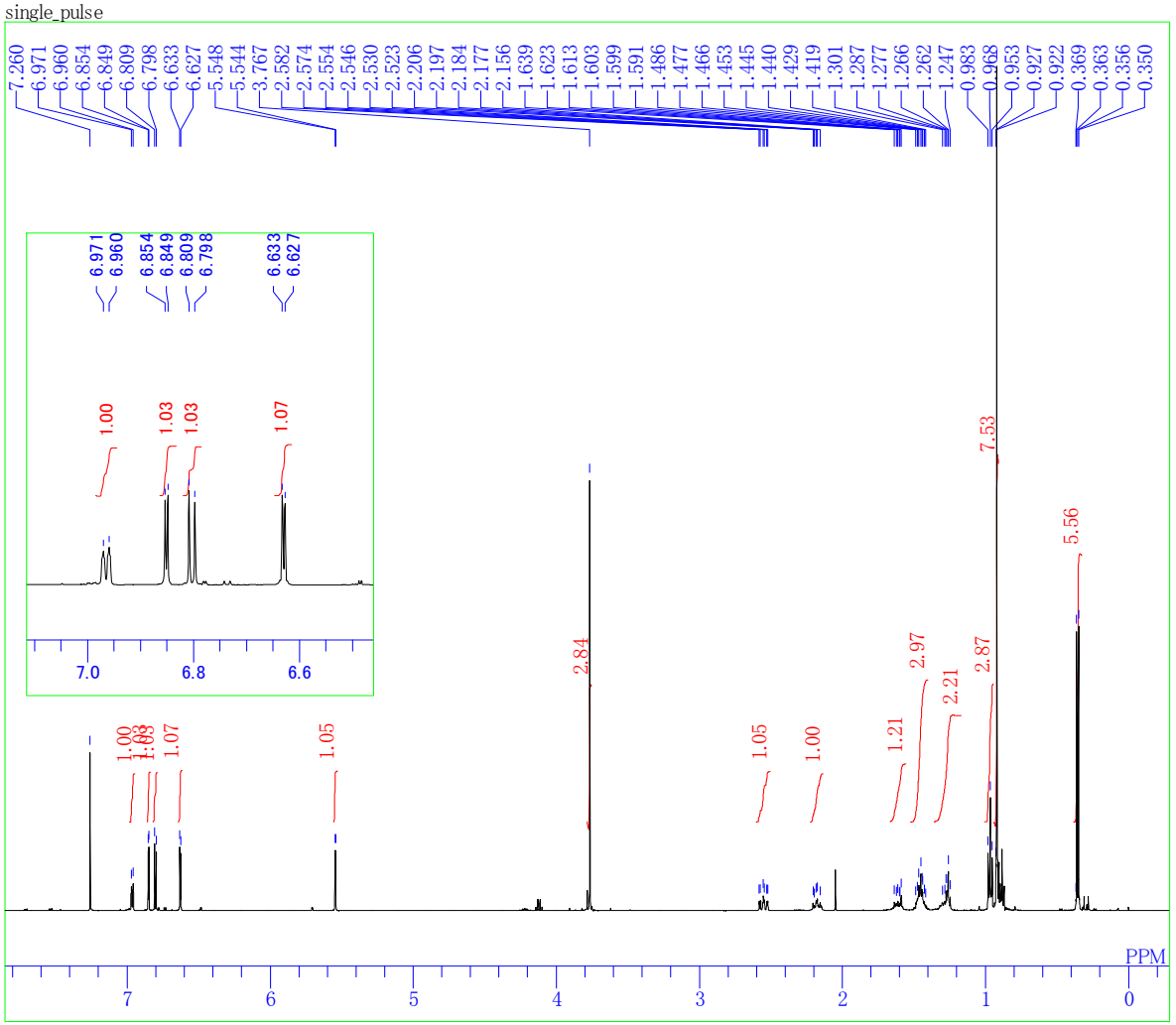


DFILE	C:¥Documents and Settings¥Owner
COMNT	single pulse decoupled gated NOE
DATIM	19-12-2007 15:37:37
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.06 Hz
SCANS	361
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	18.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.00 Hz
RGAIN	60

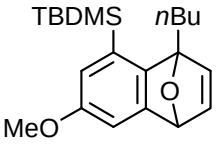


anti-**4s** ($CDCl_3$)

Table 2, entry 16



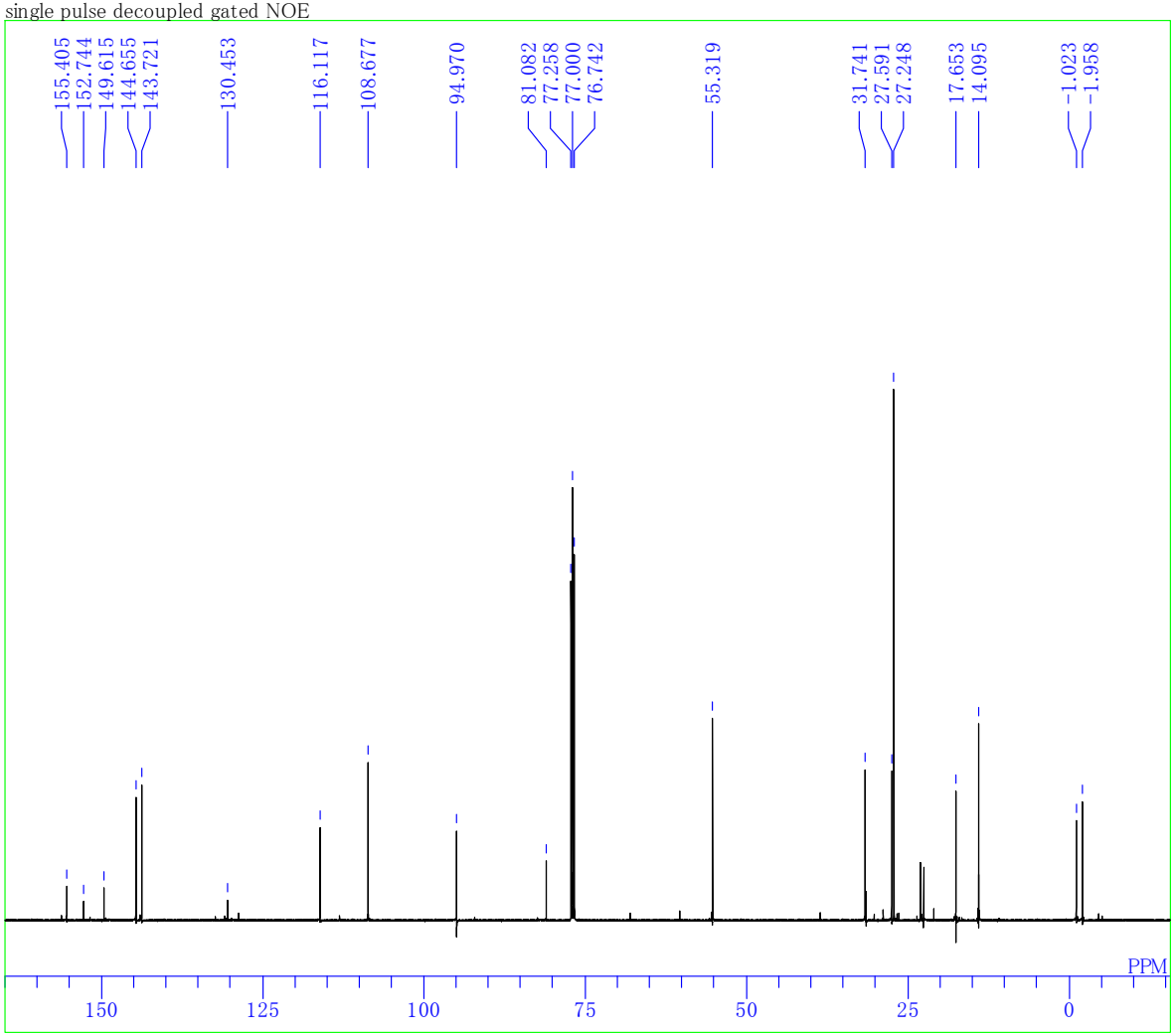
DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single_pulse
DATIM	25-12-2007 20:10:33
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16384
FREQU	7503.00 Hz
SCANS	8
ACQTM	2.1837 sec
PD	5.0000 sec
PW1	6.00 usec
IRNUC	1H
CTEMP	17.1 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.00 Hz
RGAIN	46



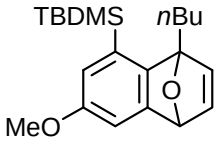
syn-**4s** ($CDCl_3$)

Table 2, entry 16

single pulse decoupled gated NOE

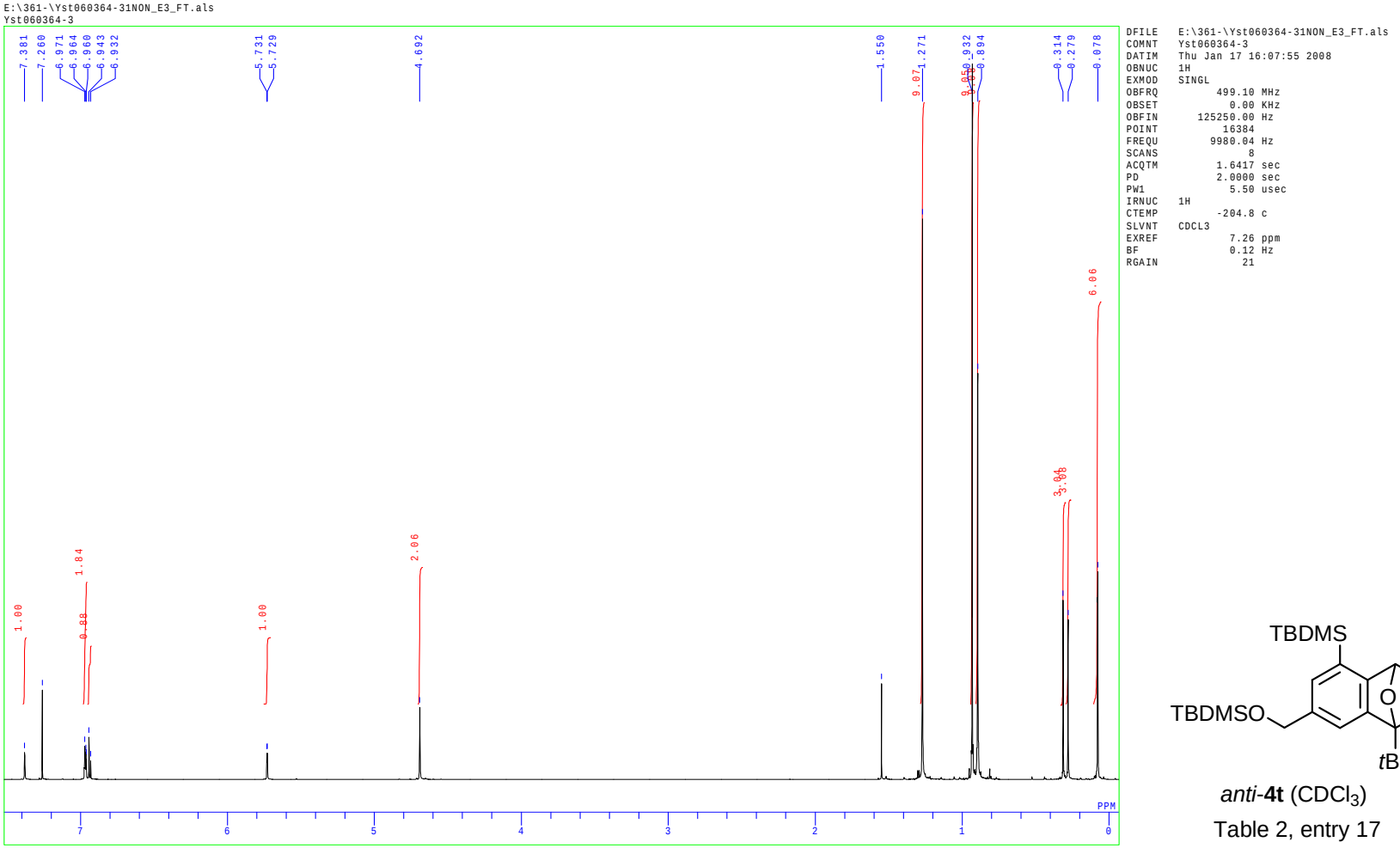


DFILE	C:¥Documents and Settings¥Owner
COMNT	single pulse decoupled gated NOE
DATIM	26-12-2007 08:39:48
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.06 Hz
SCANS	15805
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	17.5 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.00 Hz
RGAIN	60



syn-**4s** ($CDCl_3$)

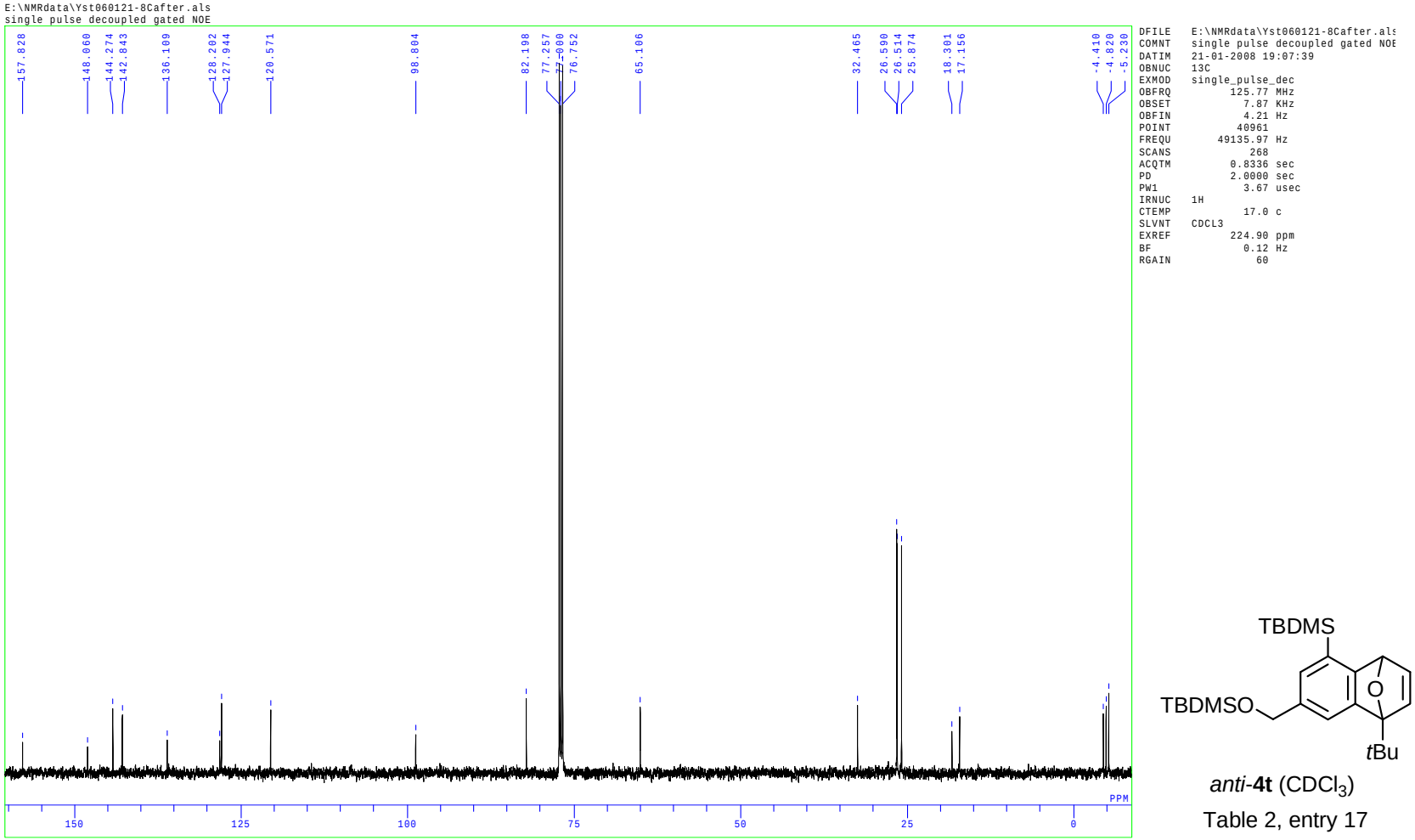
Table 2, entry 16




```
DFILE   E:\361-\Yst060364-31NON_E3_FT.als
COMNT   Yst060364-3
DATIM   Thu Jan 17 16:07:55 2008
OBNUC   1H
EXMOD   SINGL
OBFRQ         499.10 MHz
OBSET           0.00 KHz
OBFIN      125250.00 Hz
POINT          16384
FREQU        9980.04 Hz
SCANS             8
ACQTM          1.6417 sec
PD             2.0000 sec
PW1               5.50 usec
IRNUC   1H
CTEMP         -204.8 c
SLVNT   CDCL3
EXREF             7.26 ppm
BF                0.12 Hz
RGAIN               21
```


anti-**4t** ($CDCl_3$)

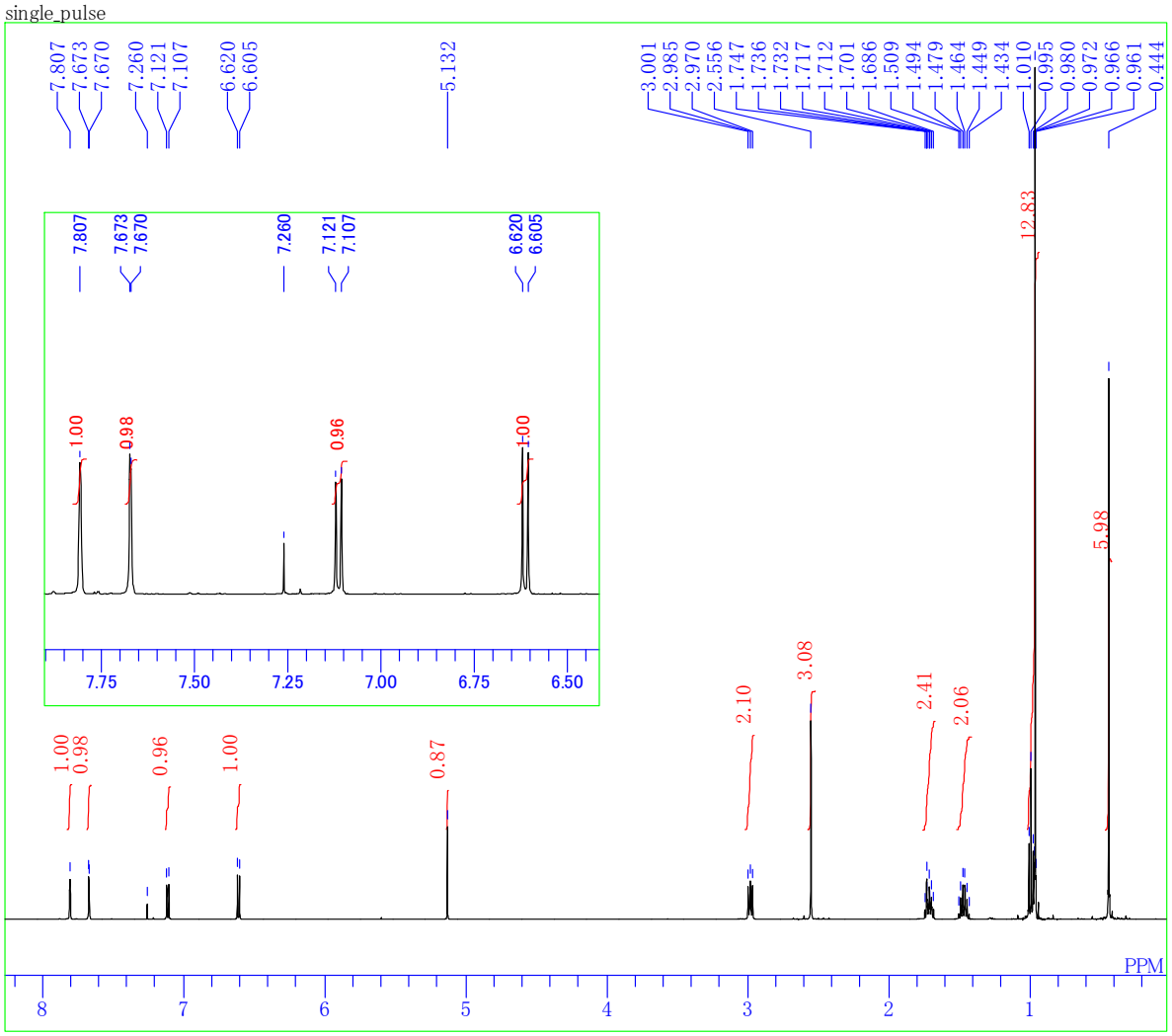
Table 2, entry 17



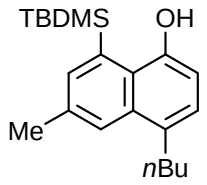

```
DFILE   E:\NMRdata\Yst060121-8Cafter.als
COMNT   single pulse decoupled gated NOE
DATIM   21-01-2008 19:07:39
OBNUC   13C
EXMOD   single_pulse_dec
OBFRQ         125.77 MHz
OBSET           7.87 KHz
OBFIN           4.21 Hz
POINT          40961
FREQU       49135.97 Hz
SCANS            268
ACQTM          0.8336 sec
PD             2.0000 sec
PW1              3.67 usec
IRNUC   1H
CTEMP           17.0 c
SLVNT   CDCL3
EXREF         224.90 ppm
BF              0.12 Hz
RGAIN             60
```


anti-**4t** ($CDCl_3$)

Table 2, entry 17



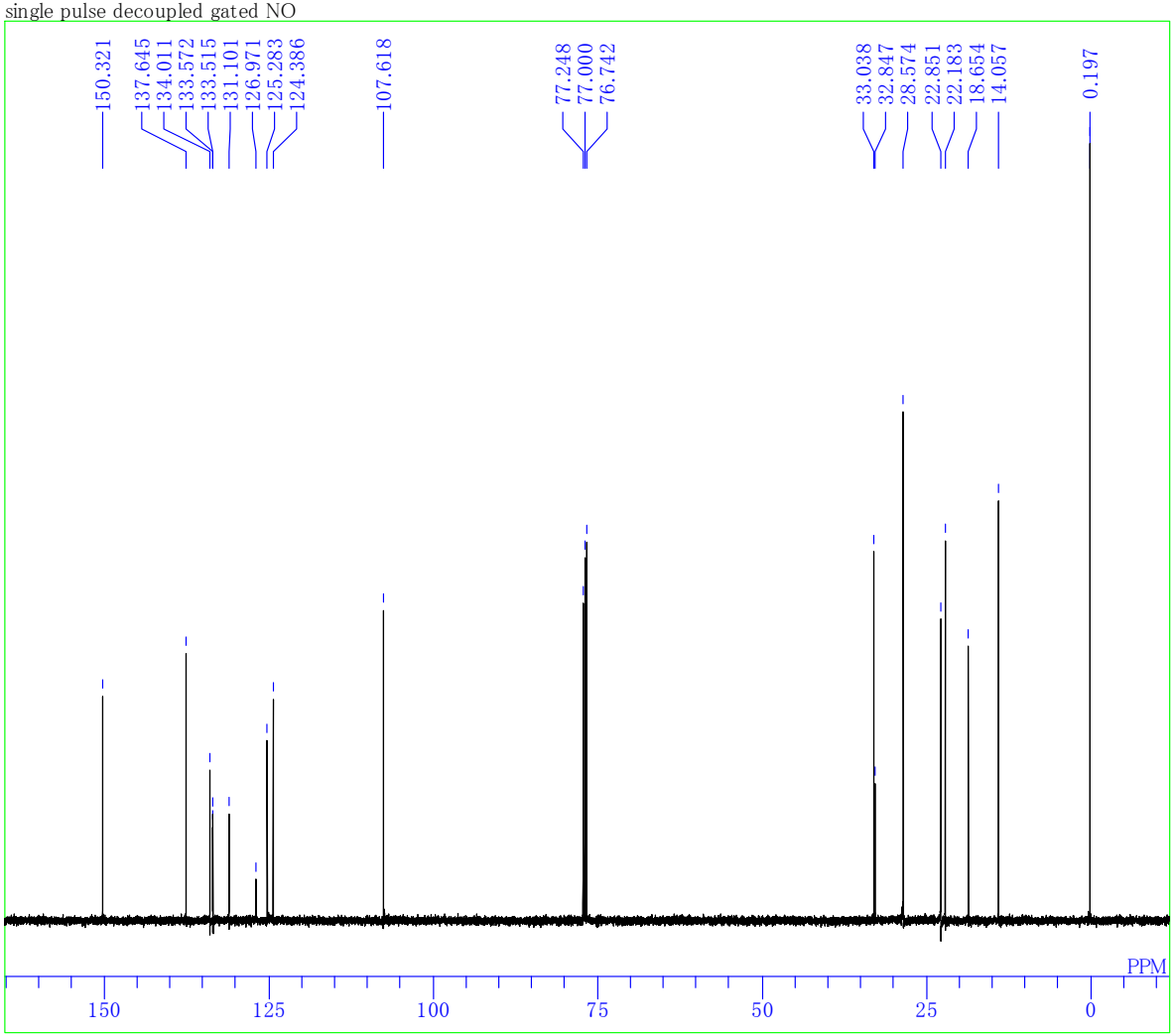
DFILE	C:¥Documents and Settings¥Owner
COMNT	single_pulse
DATIM	25-12-2007 15:30:18
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.39 Hz
SCANS	8
ACQTM	1.7459 sec
PD	1.5000 sec
PW1	6.00 usec
IRNUC	1H
CTEMP	17.2 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.00 Hz
RGAIN	36



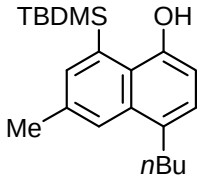
5a ($CDCl_3$)

Table 3, entry 1

single pulse decoupled gated NO

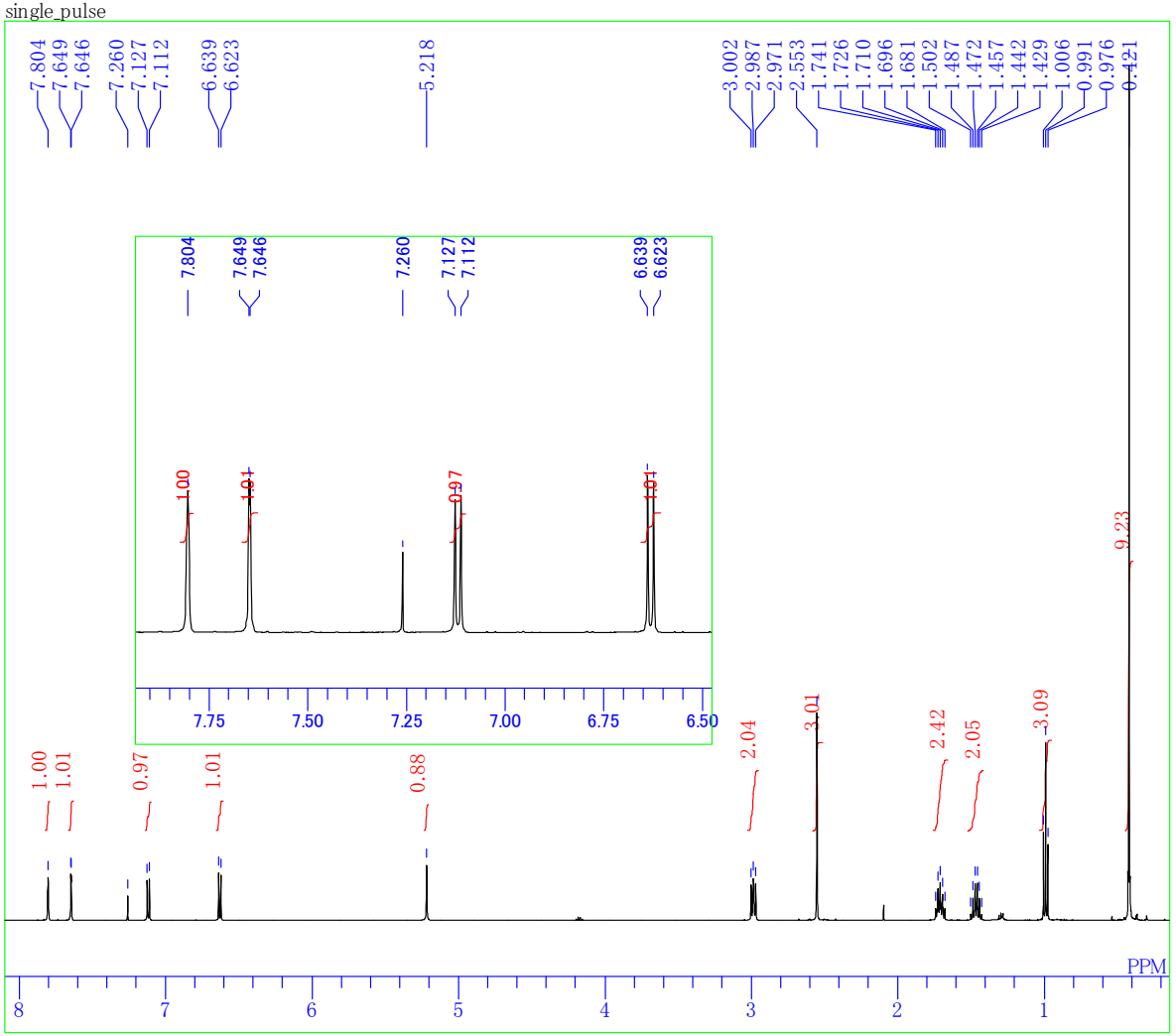


DFILE	C:¥Documents and Settings¥Owner
COMNT	single pulse decoupled gated NO
DATIM	25-12-2007 15:59:19
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.06 Hz
SCANS	533
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	17.8 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.00 Hz
RGAIN	60

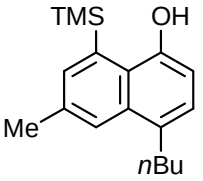


5a ($CDCl_3$)

Table 3, entry 1



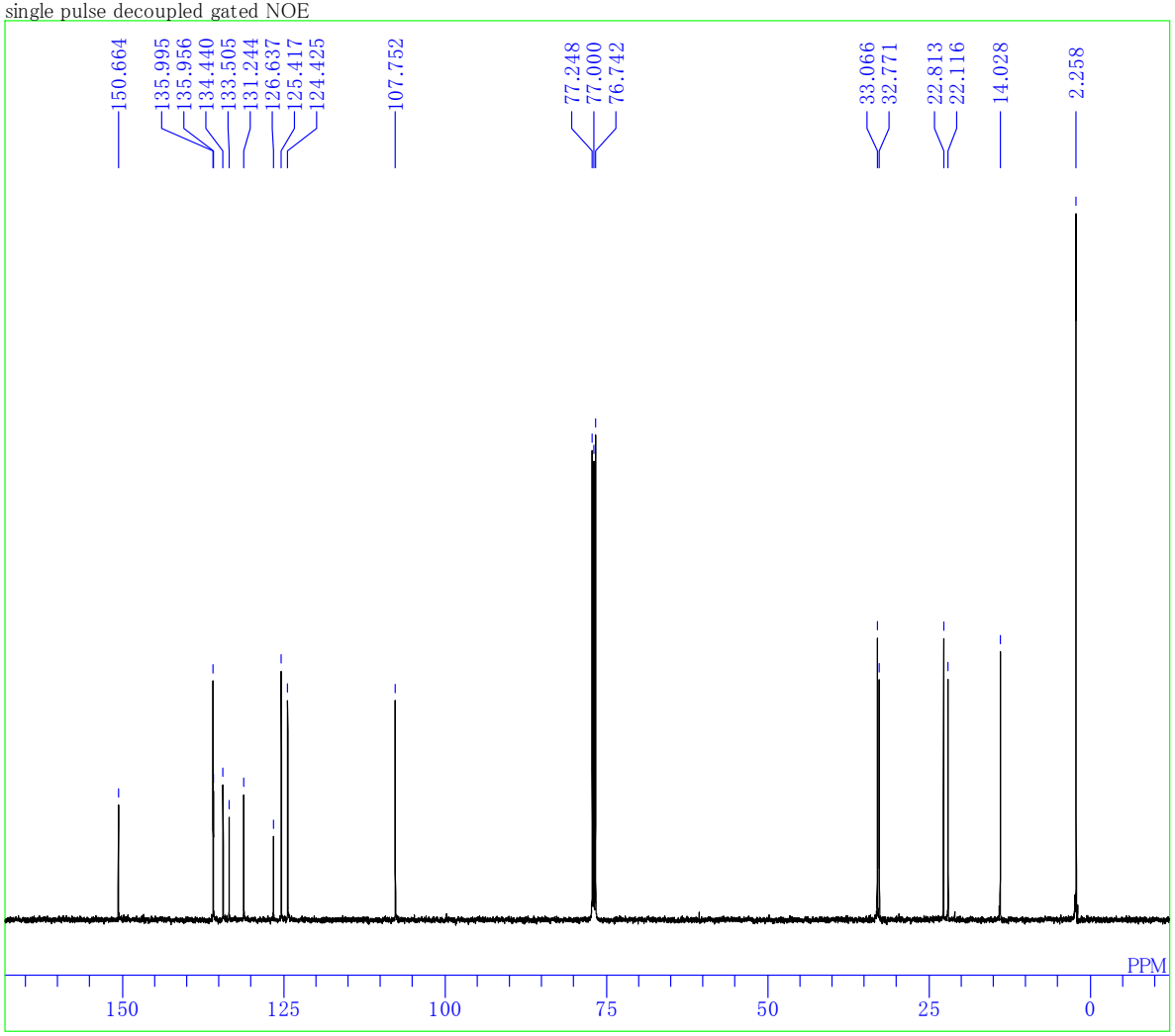
DFILE C:¥Documents and Settings¥Owner
COMNT single_pulse
DATIM 26-06-2007 00:31:11
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.00 usec
IRNUC 1H
CTEMP 393.0 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 38



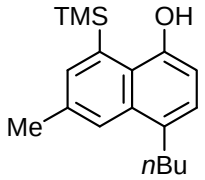
5b ($CDCl_3$)

Table 3, entry 2

single pulse decoupled gated NOE

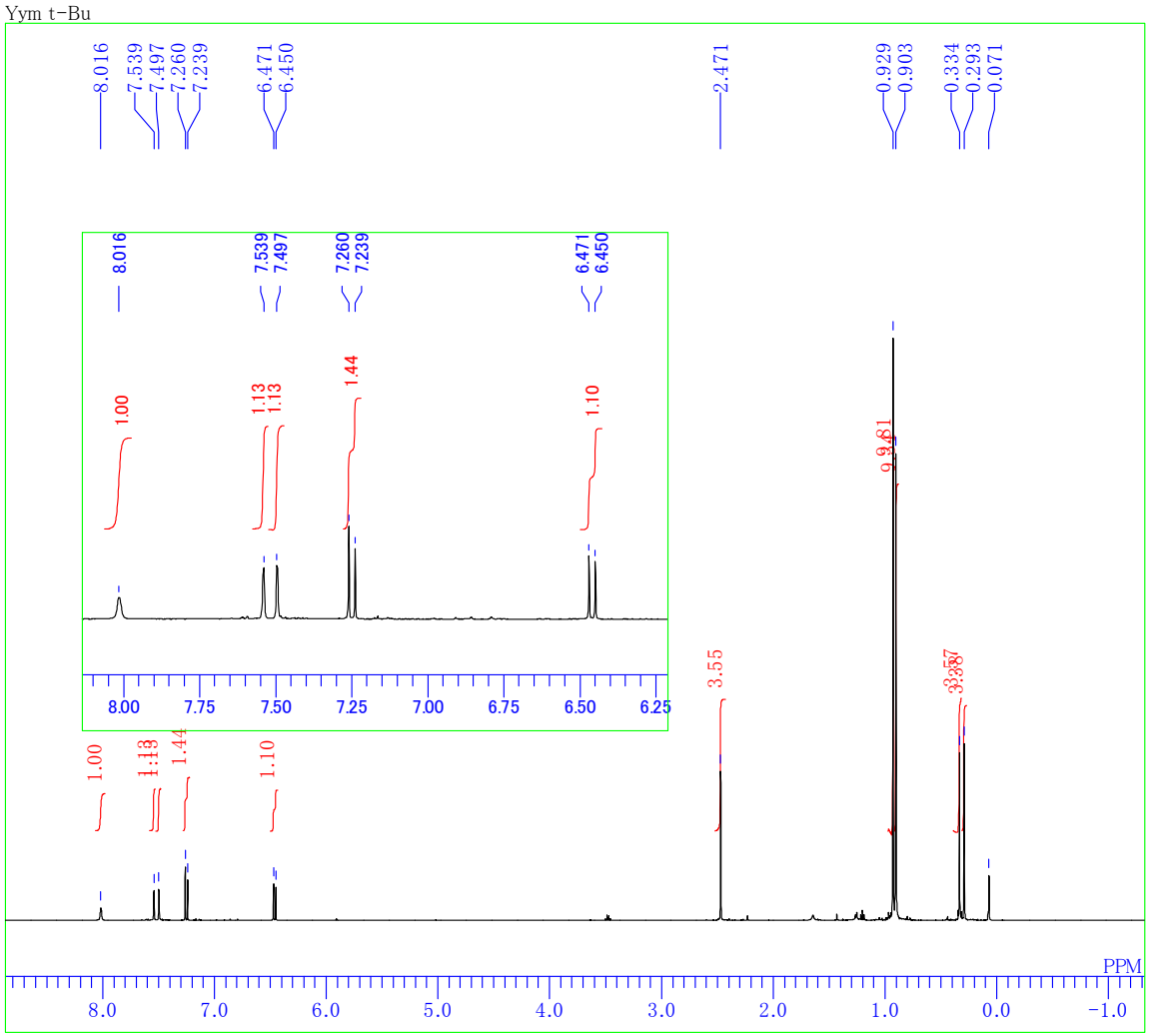


DFILE	C:¥Documents and Settings¥Owner
COMNT	single pulse decoupled gated NOE
DATIM	26-06-2007 01:14:33
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32768
FREQU	39308.18 Hz
SCANS	845
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	393.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

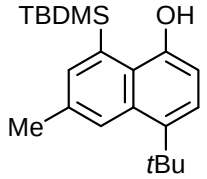


5b ($CDCl_3$)

Table 3, entry 2

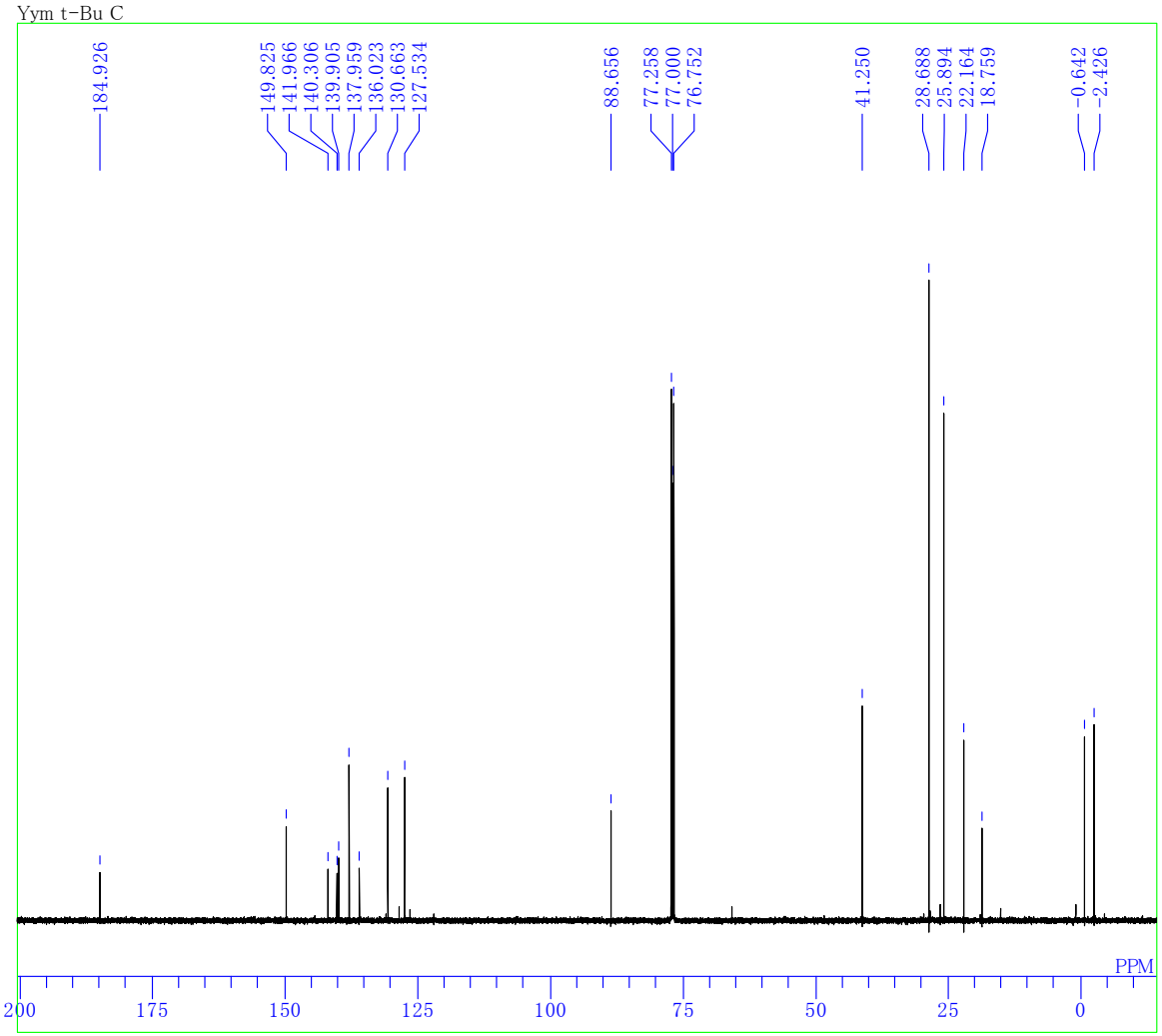


DFILE	C:¥Documents and Settings¥Owner
COMNT	Yym t-Bu
DATIM	12-05-2008 20:52:51
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.39 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	5.75 usec
IRNUC	1H
CTEMP	19.0 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	40

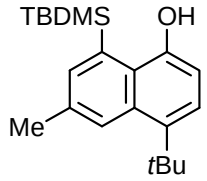


5c ($CDCl_3$)

Table 3, entry 3



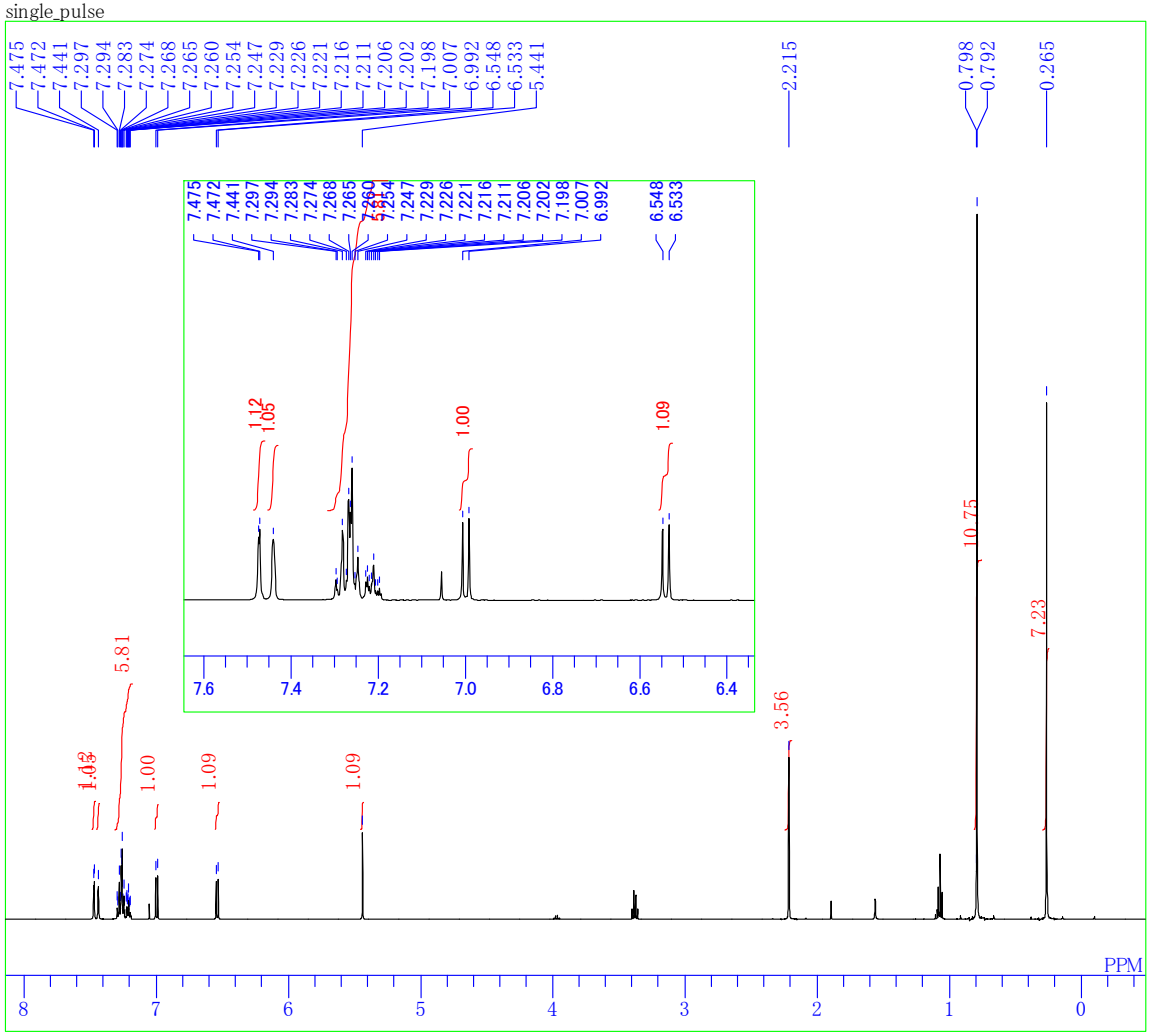
DFILE	C:¥Documents and Settings¥Owner
COMNT	Yym t-Bu C
DATIM	12-05-2008 22:42:21
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.06 Hz
SCANS	2260
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	19.3 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60



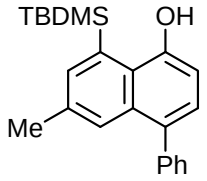
5c ($CDCl_3$)

Table 3, entry 3

single_pulse



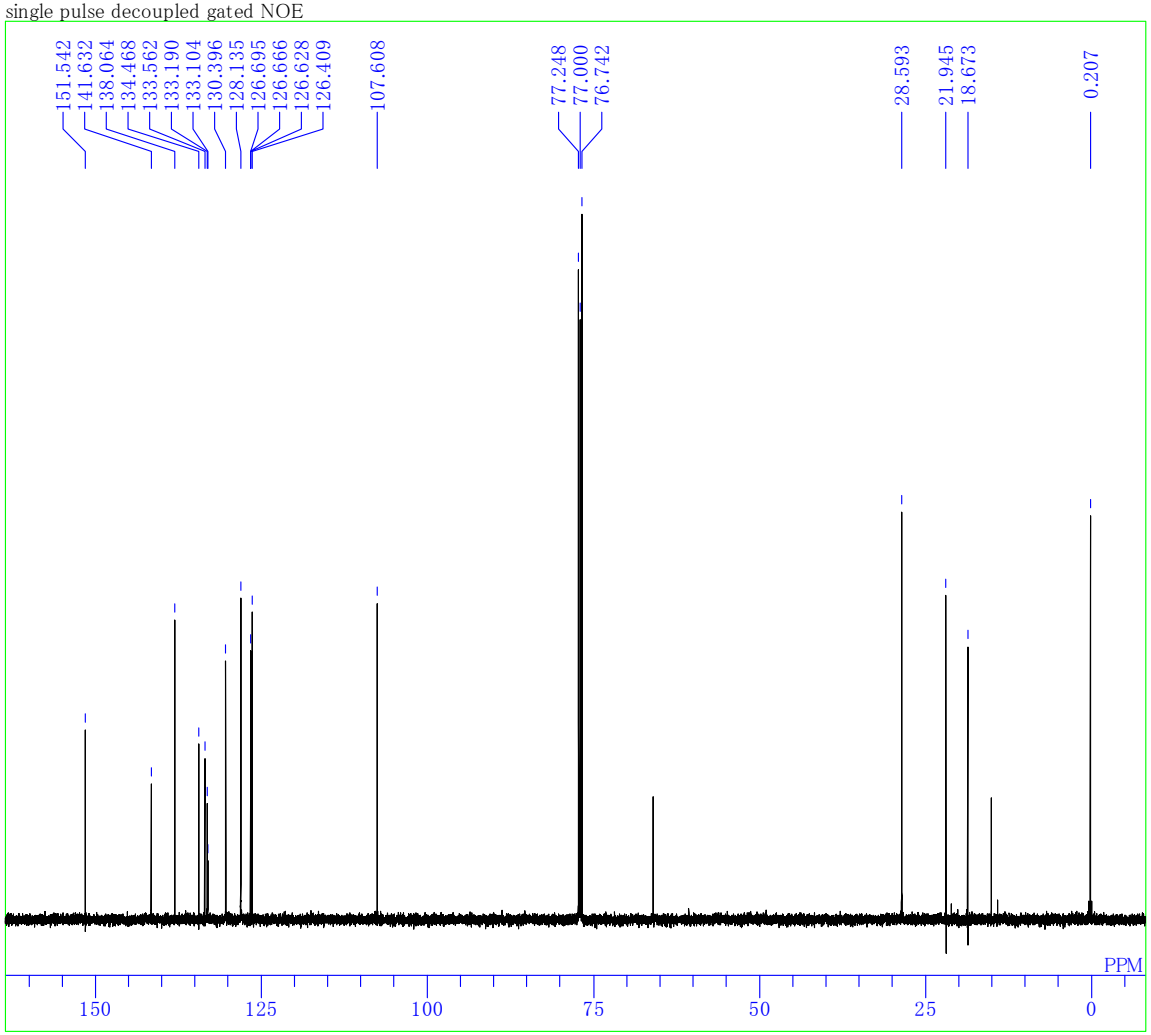
DFILE	C:¥Documents and Settings¥Owner
COMNT	single_pulse
DATIM	16-05-2008 16:50:56
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.39 Hz
SCANS	8
ACQTM	1.7459 sec
PD	1.5000 sec
PW1	5.75 usec
IRNUC	1H
CTEMP	19.9 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	40



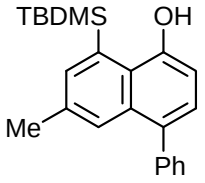
5g ($CDCl_3$)

Table 3, entry 4

single pulse decoupled gated NOE

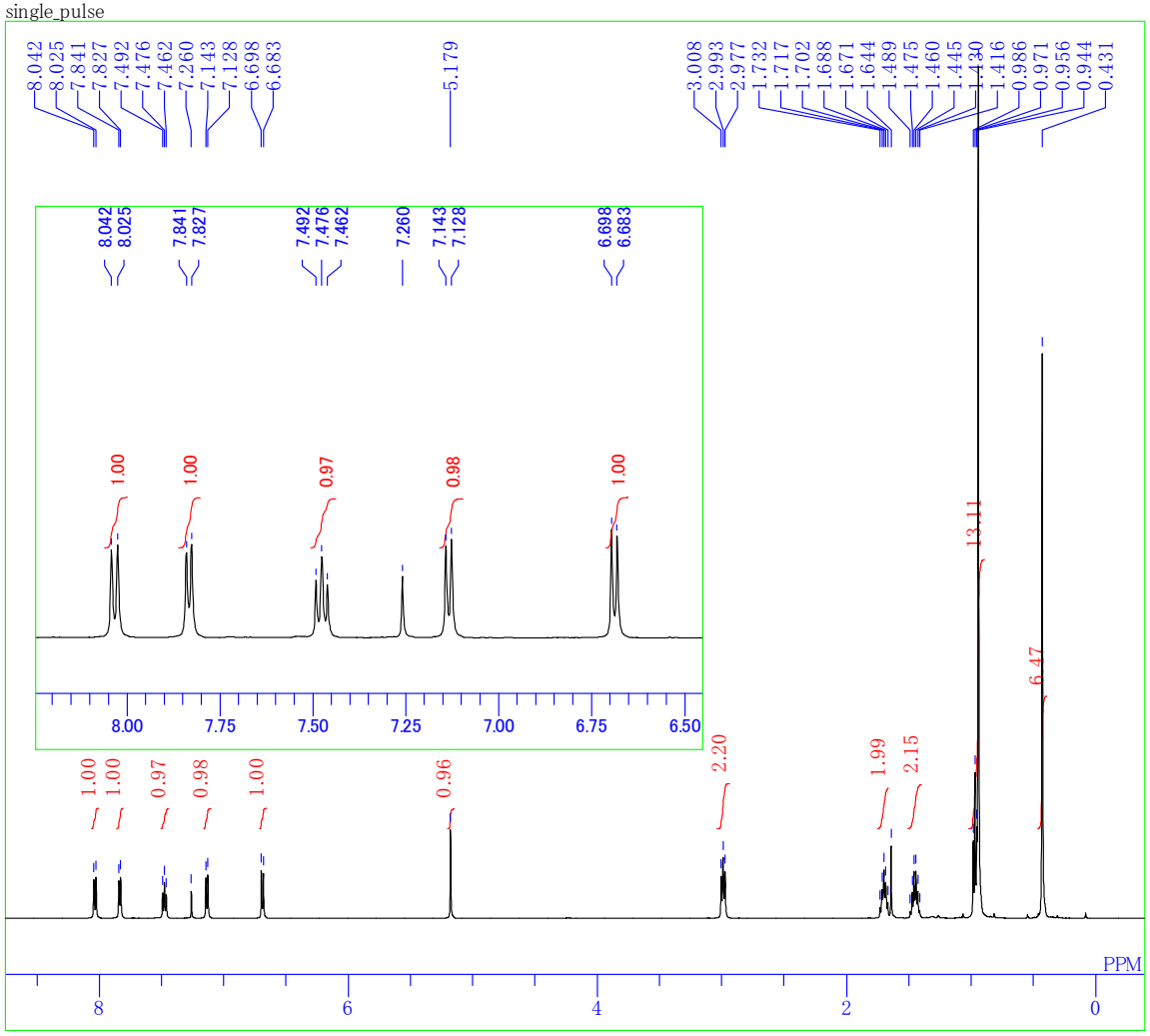


DFILE	C:¥Documents and Settings¥Owner
COMNT	single pulse decoupled gated NOE
DATIM	16-05-2008 17:21:58
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.06 Hz
SCANS	567
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	20.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

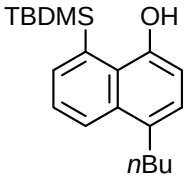


5g ($CDCl_3$)

Table 3, entry 4

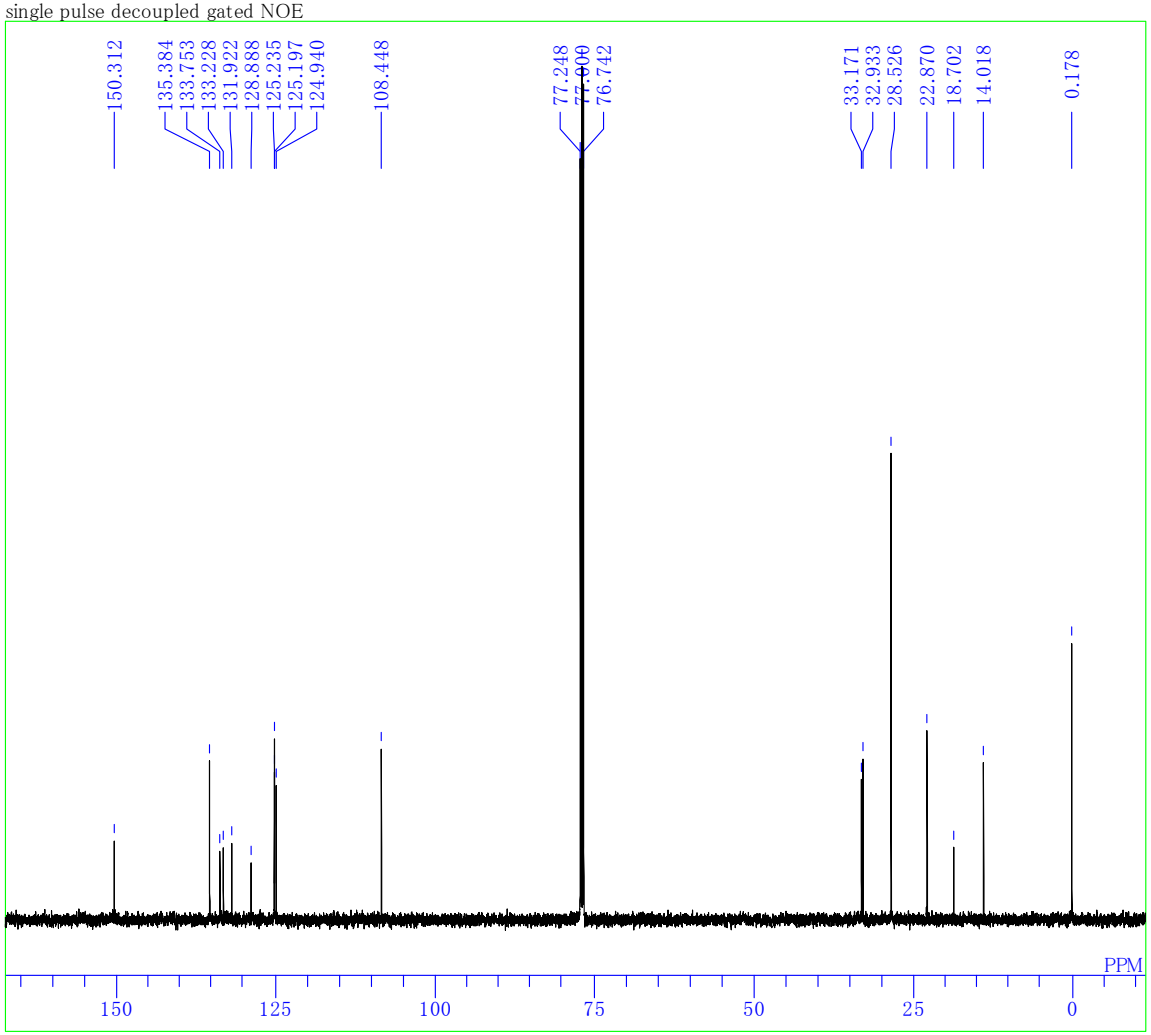


DFILE	C:¥Documents and Settings¥Owner
COMNT	single_pulse
DATIM	30-05-2008 17:55:33
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.39 Hz
SCANS	8
ACQTM	1.7459 sec
PD	1.5000 sec
PW1	5.75 usec
IRNUC	1H
CTEMP	20.5 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	1.20 Hz
RGAIN	44

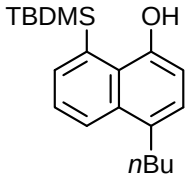


5e ($CDCl_3$)
Table 3, entry 5

single pulse decoupled gated NOE

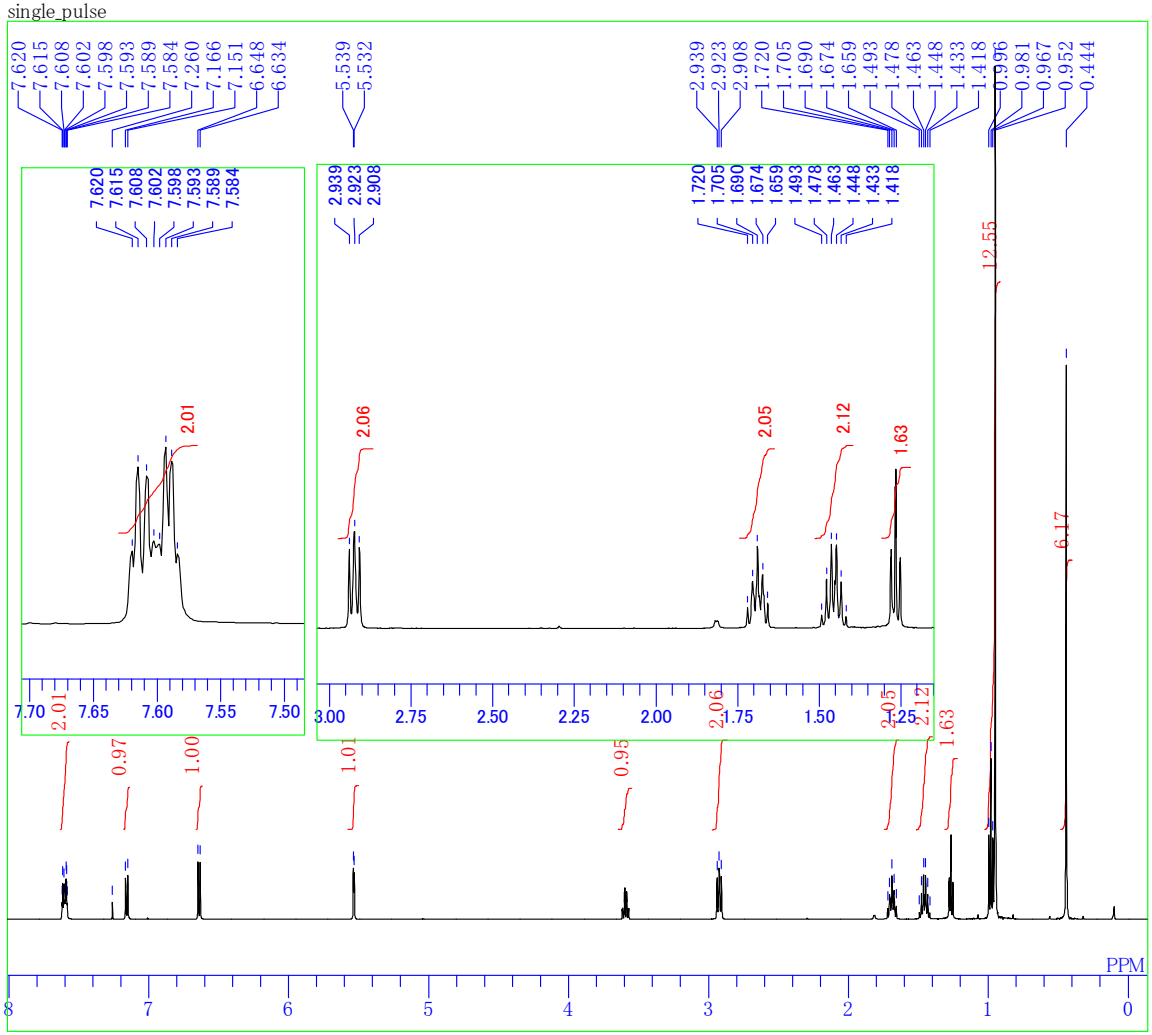


DFILE	C:¥Documents and Settings¥Owner
COMNT	single pulse decoupled gated NOE
DATIM	30-05-2008 18:20:30
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.06 Hz
SCANS	448
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	21.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60

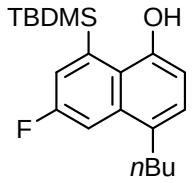


5e ($CDCl_3$)

Table 3, entry 5



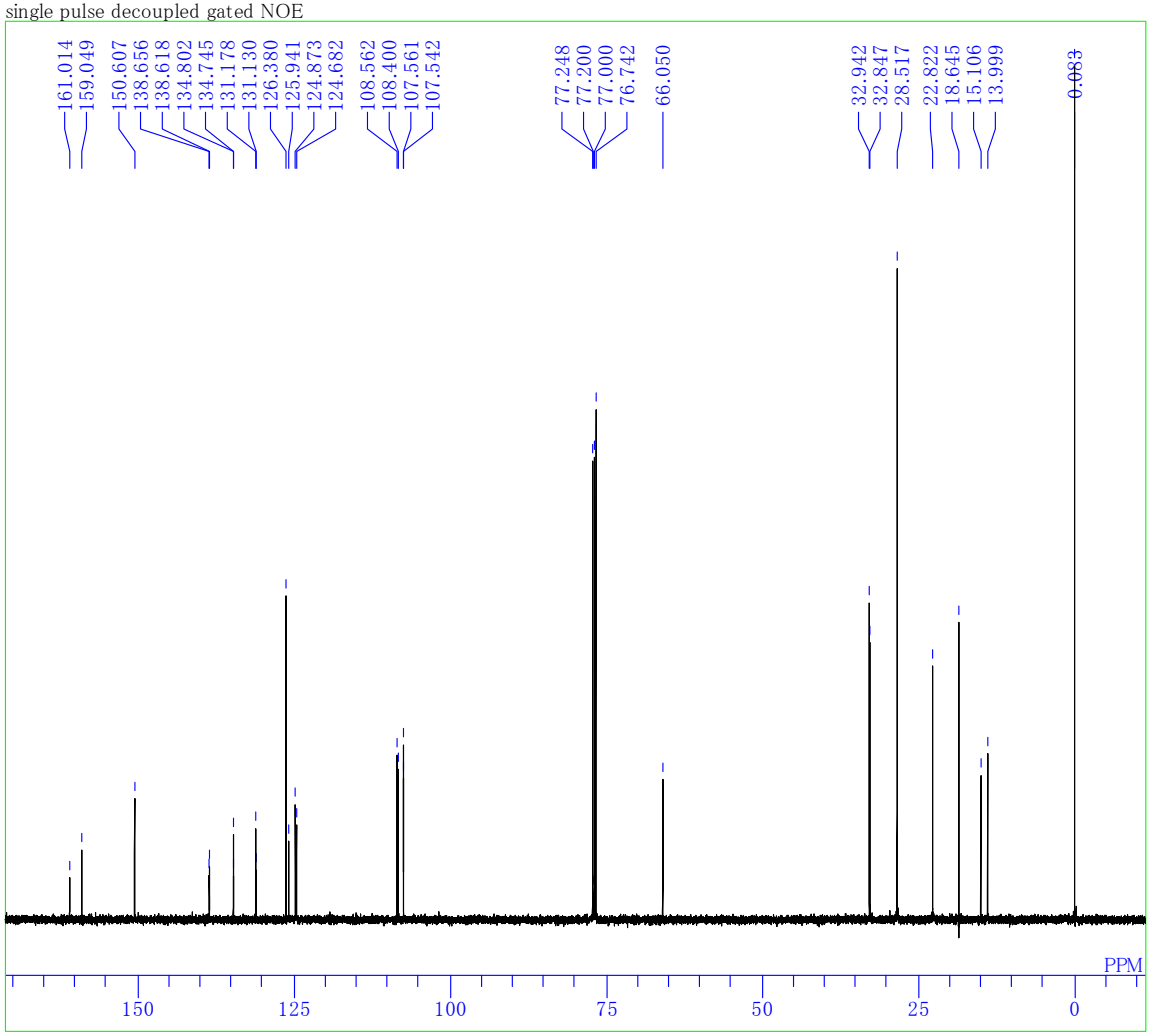
DFILE C:¥Documents and Settings¥Owner
COMNT single_pulse
DATIM 13-05-2008 09:36:37
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 9384.38 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.75 usec
IRNUC 1H
CTEMP 19.1 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 36



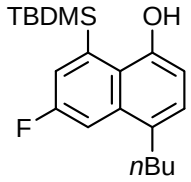
5f ($CDCl_3$)

Table 3, entry 6

single pulse decoupled gated NOE



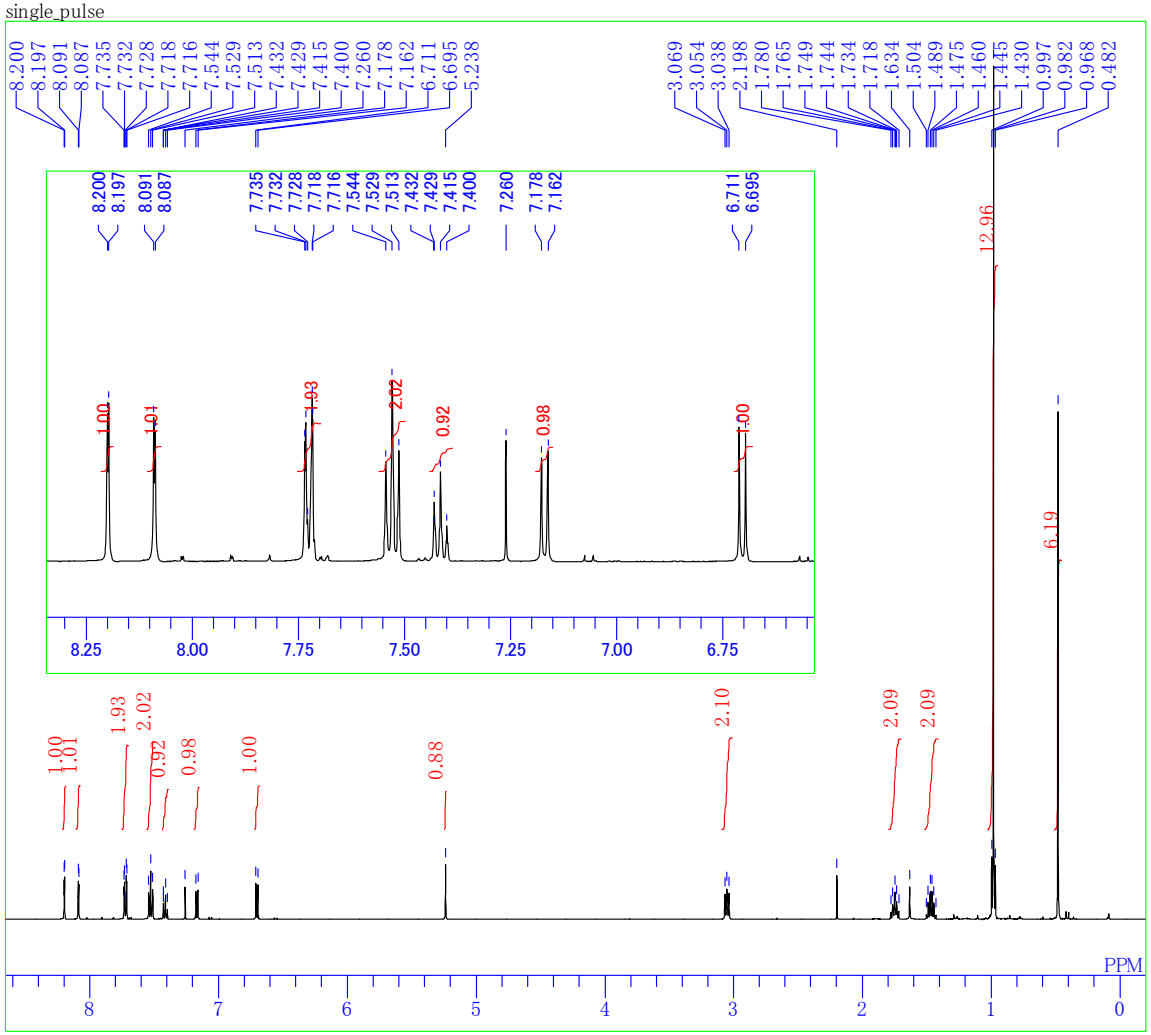
DFILE	C:¥Documents and Settings¥Owner
COMNT	single pulse decoupled gated NOE
DATIM	13-05-2008 10:16:40
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.06 Hz
SCANS	798
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	19.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60



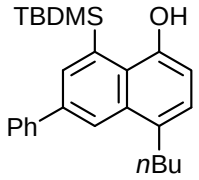
5f ($CDCl_3$)

Table 3, entry 6

single_pulse



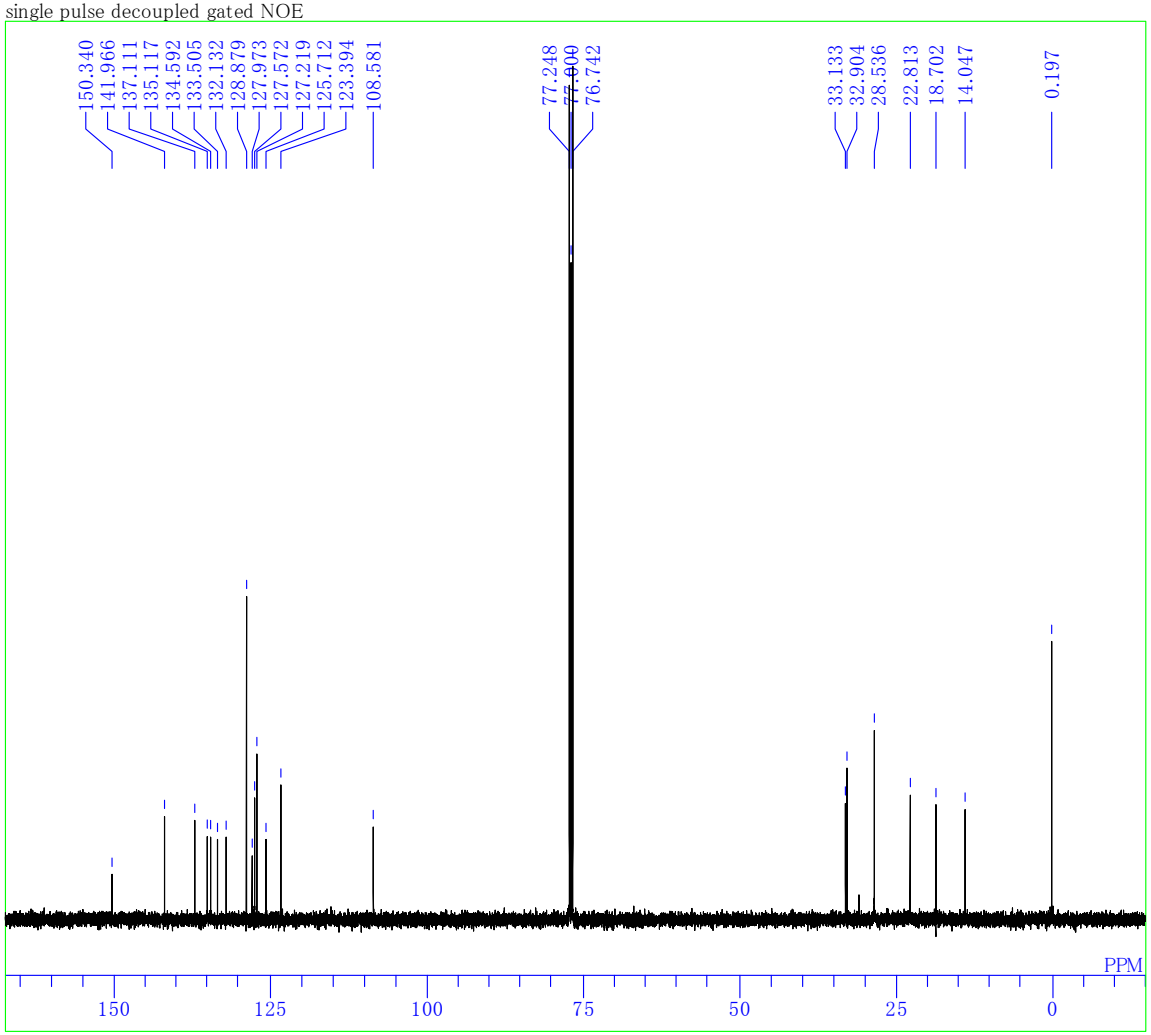
DFILE	C:¥Documents and Settings¥Owner
COMNT	single_pulse
DATIM	13-05-2008 19:00:08
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16384
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	1.5000 sec
PW1	5.75 usec
IRNUC	1H
CTEMP	19.4 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	44



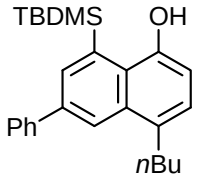
5g ($CDCl_3$)

Table 3, entry 7

single pulse decoupled gated NOE

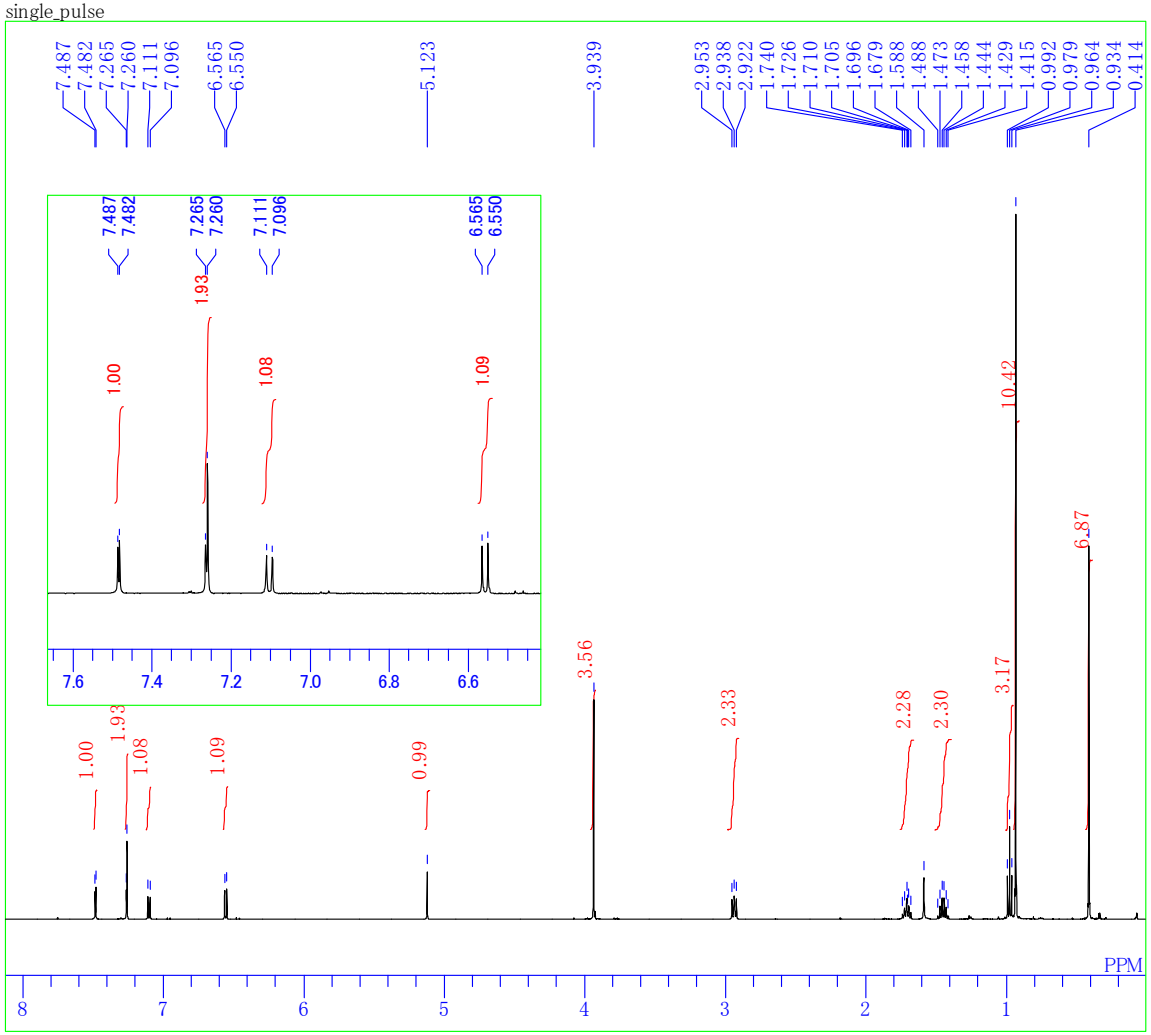


DFILE	C:¥Documents and Settings¥Owner
COMNT	single pulse decoupled gated NOE
DATIM	13-05-2008 19:22:34
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32768
FREQU	39308.18 Hz
SCANS	460
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	19.7 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50

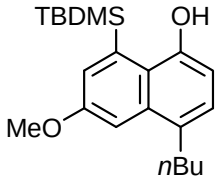


5g ($CDCl_3$)

Table 3, entry 7



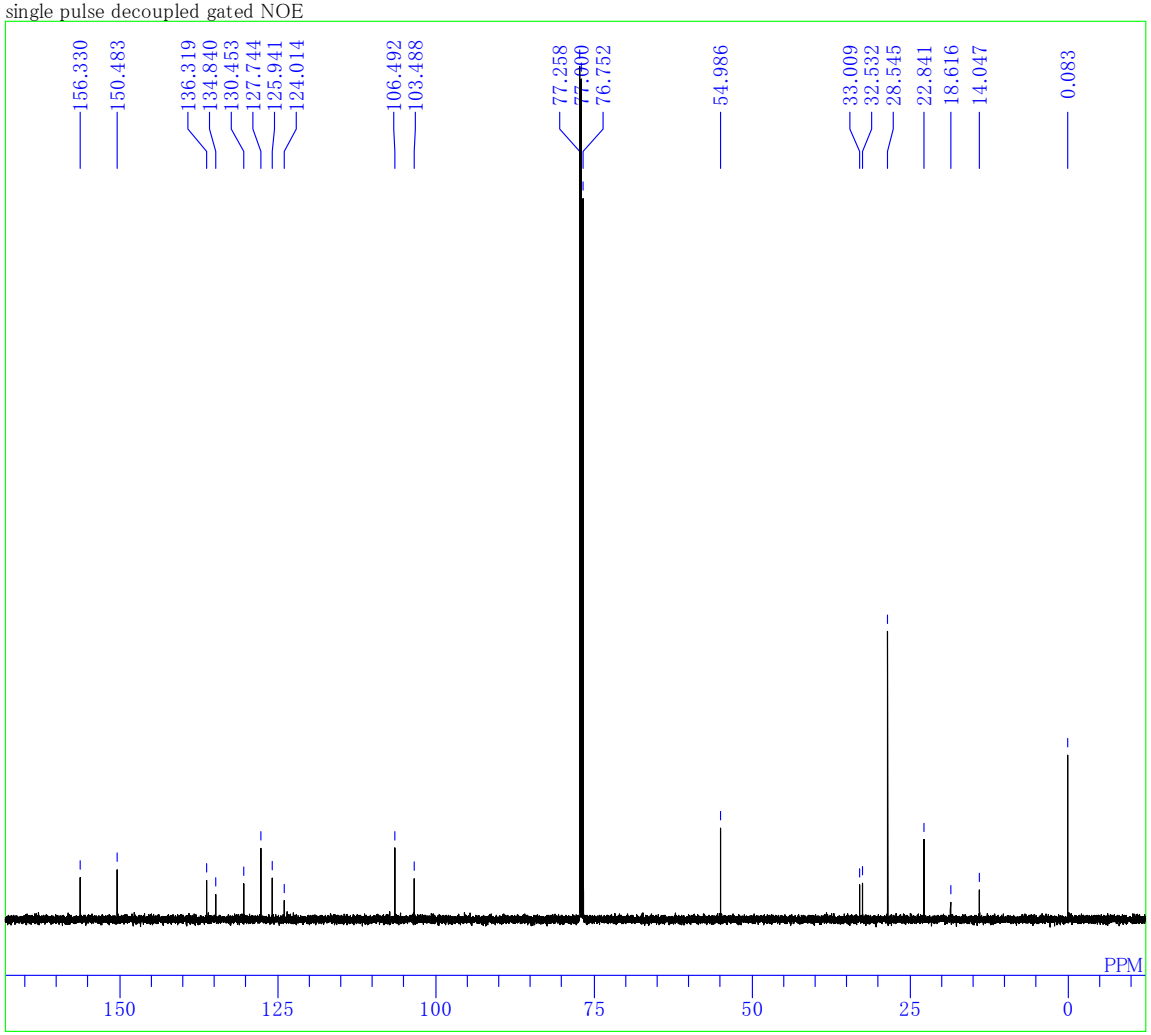
DFILE	C:¥Documents and Settings¥Owner
COMNT	single_pulse
DATIM	13-05-2008 19:26:54
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13106
FREQU	7507.39 Hz
SCANS	8
ACQTM	1.7459 sec
PD	1.5000 sec
PW1	5.75 usec
IRNUC	1H
CTEMP	19.3 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	50



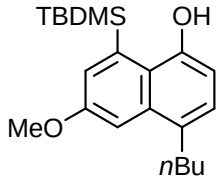
5h ($CDCl_3$)

Table 3, entry 8

single pulse decoupled gated NOE

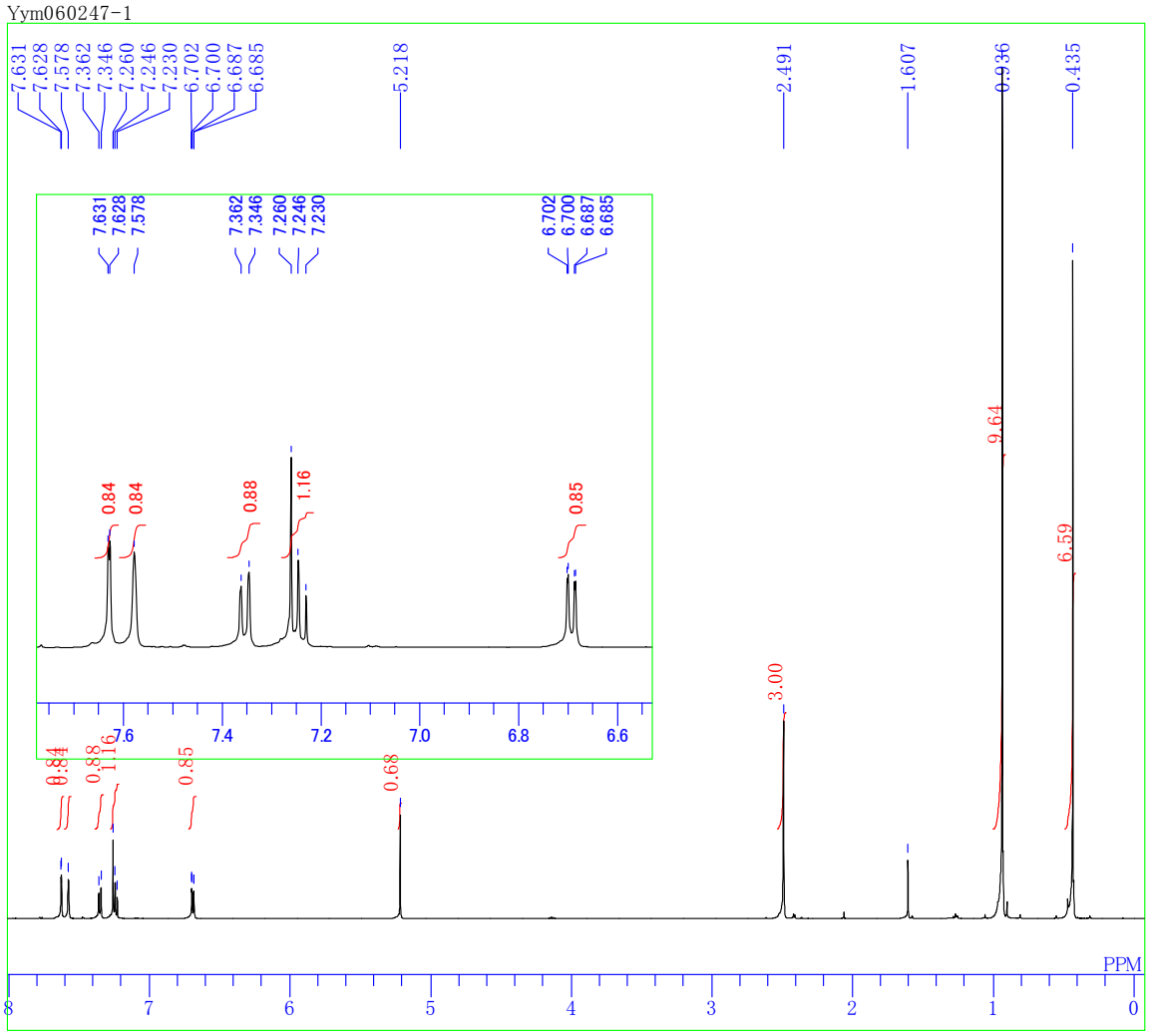


DFILE	C:¥Documents and Settings¥Owner
COMNT	single pulse decoupled gated NOE
DATIM	13-05-2008 20:41:32
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32768
FREQU	39308.18 Hz
SCANS	1572
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	19.7 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50

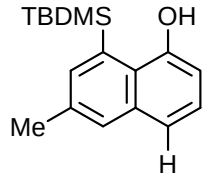


5h ($CDCl_3$)

Table 3, entry 8

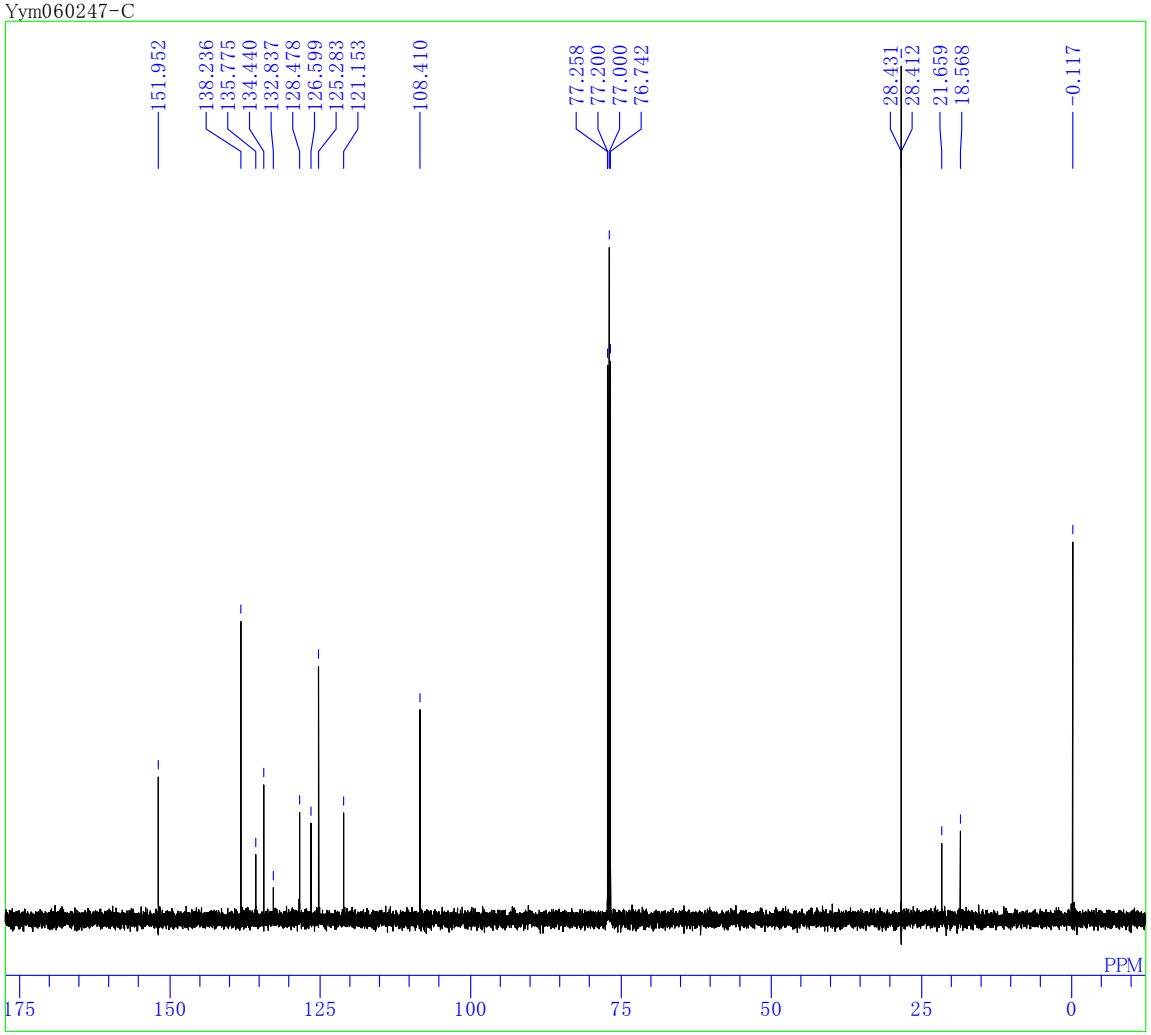


DFILE	C:¥Documents and Settings¥Owner
COMNT	Yym060247-1
DATIM	22-04-2008 16:28:35
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16384
FREQU	9384.38 Hz
SCANS	32
ACQTM	1.7459 sec
PD	1.5000 sec
PW1	5.75 usec
IRNUC	1H
CTEMP	20.2 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	50

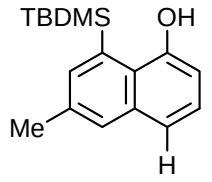


5i, Eq (2) ($CDCl_3$)

Yym060247-C

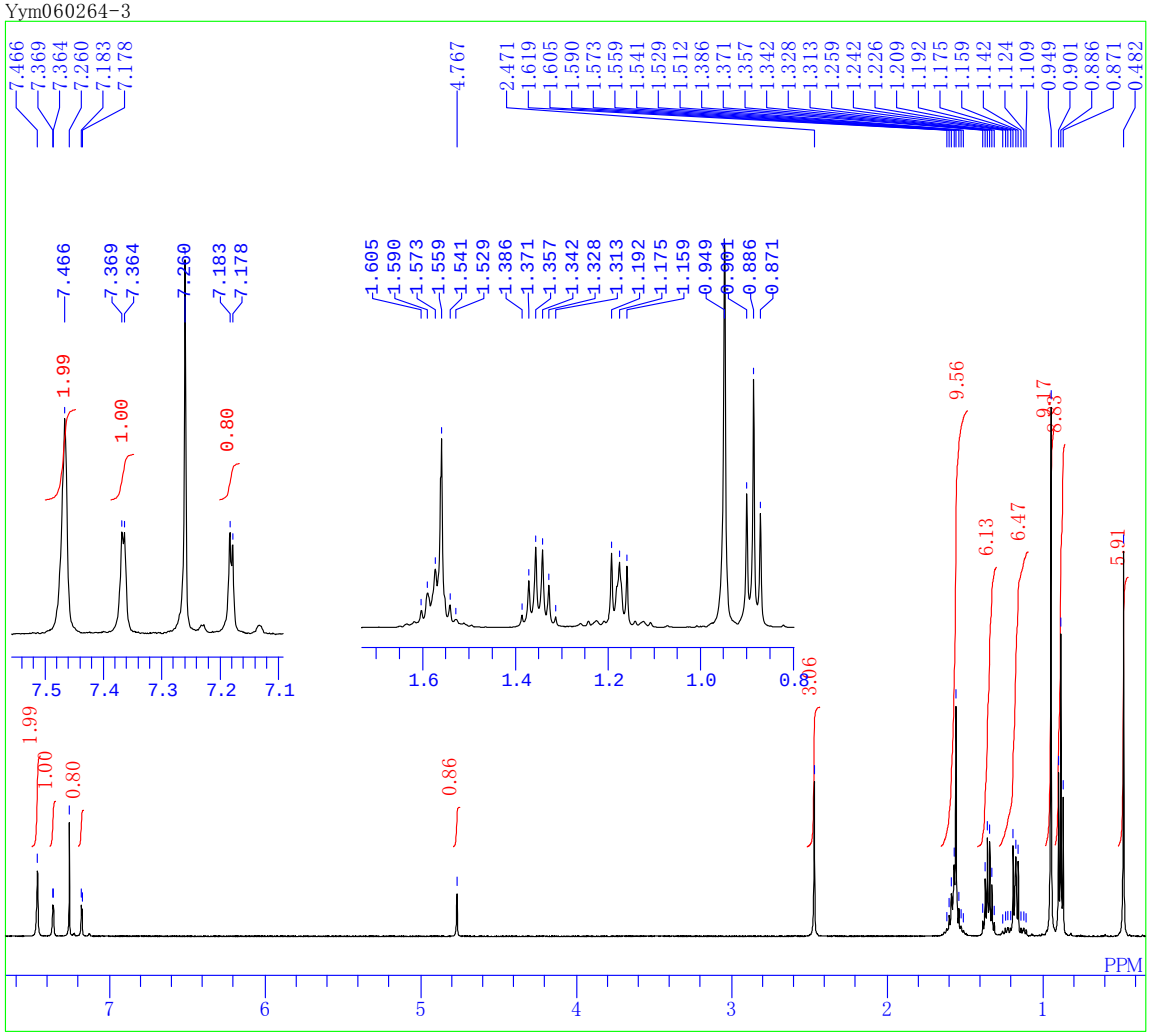


DFILE	C:¥Documents and Settings¥Owner
COMNT	Yym060247-C
DATIM	29-05-2008 18:48:11
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.06 Hz
SCANS	198
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	20.9 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

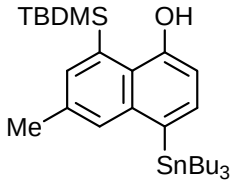


5i, Eq (2) ($CDCl_3$)

Yym060264-3

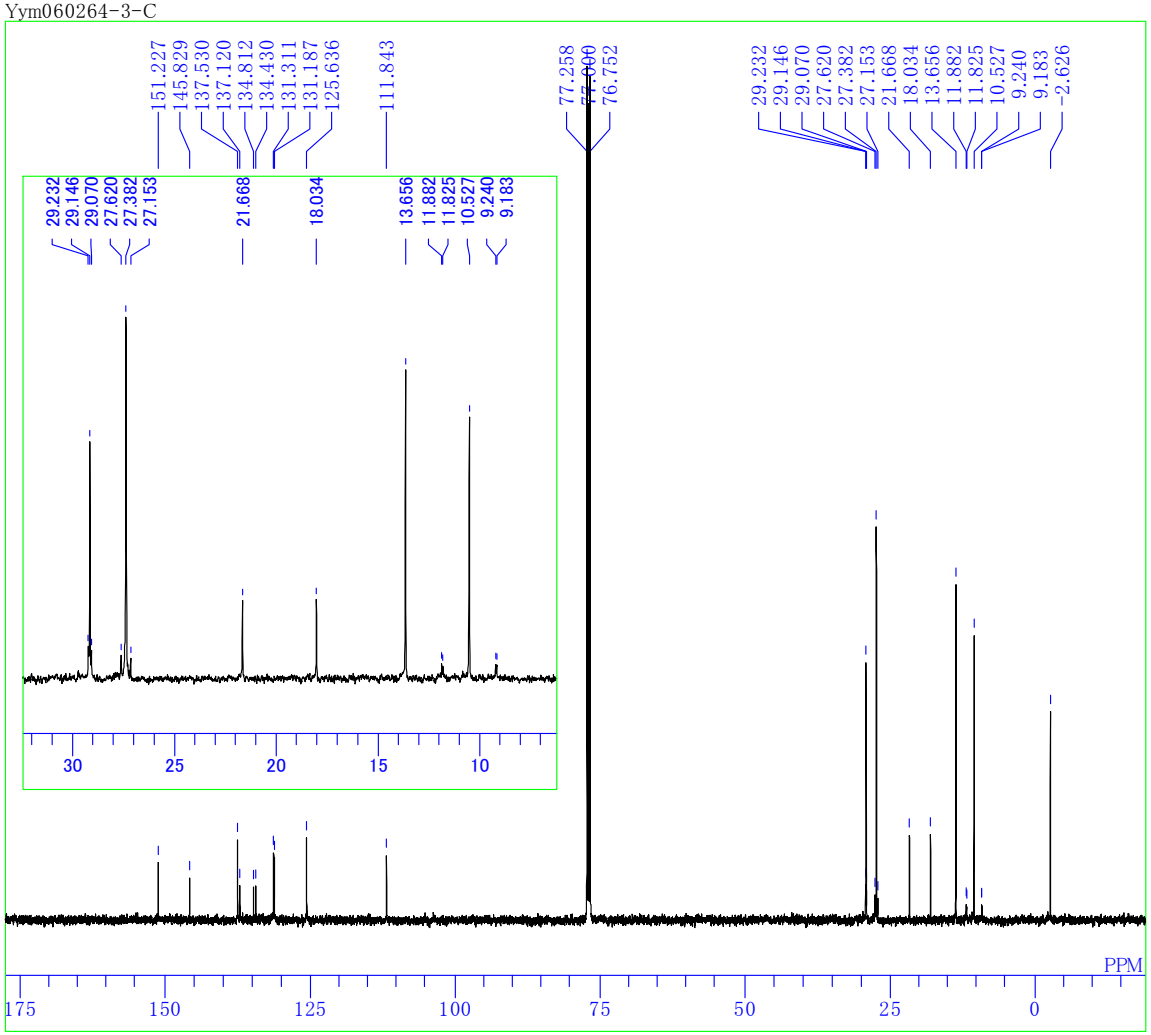


DFILE	C:¥Documents and Settings¥Owner
COMNT	Yym060264-3
DATIM	Wed May 21 16:15:42 2008
OBNUC	1H
EXMOD	SINGL
OBFRQ	499.10 MHz
OBSET	0.00 KHz
OBFIN	126750.00 Hz
POINT	16384
FREQU	9980.04 Hz
SCANS	8
ACQTM	1.6417 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	-204.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	23

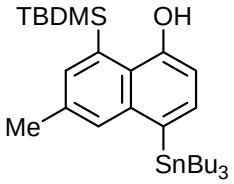


5j, Eq (3) ($CDCl_3$)

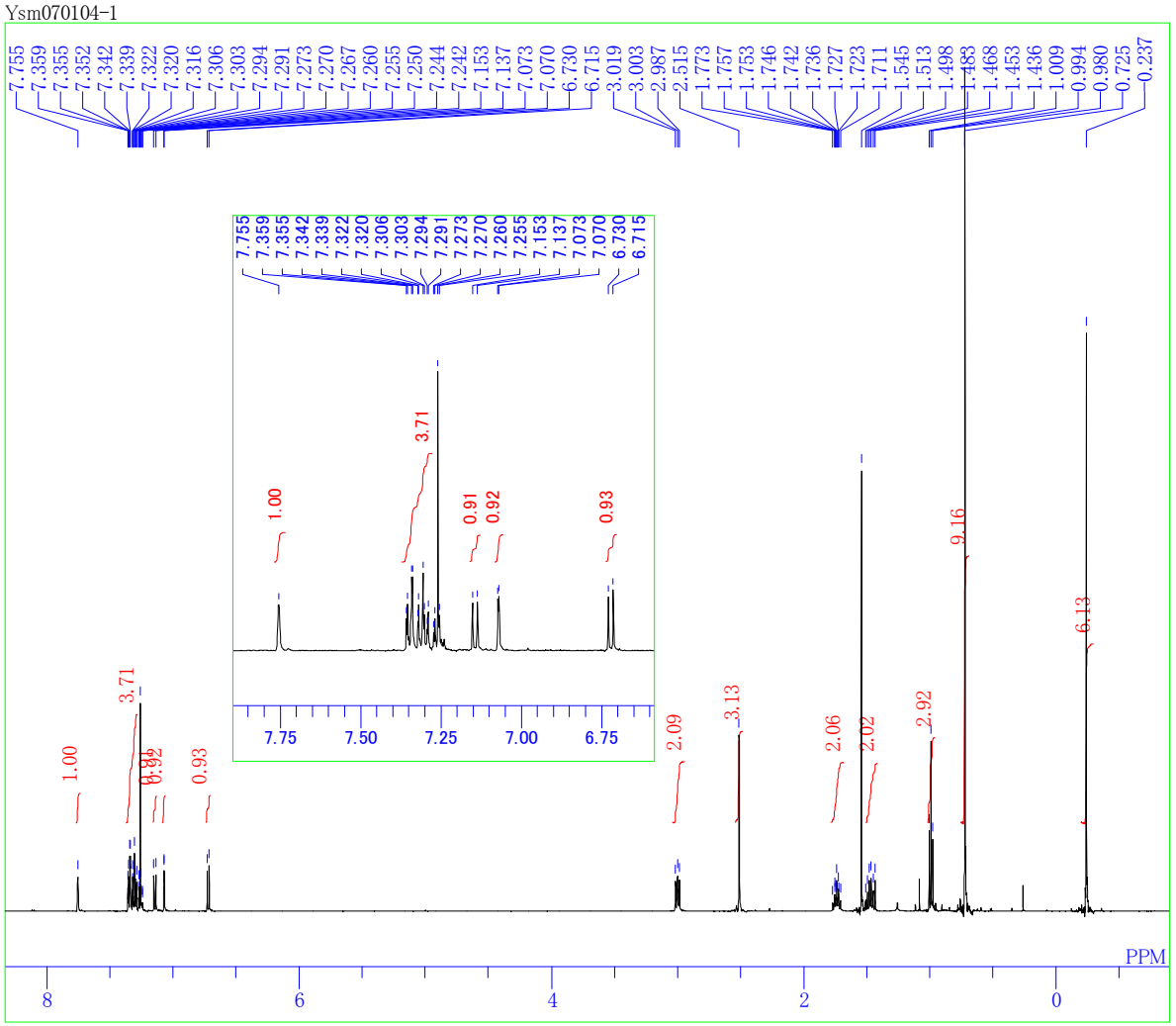
Yym060264-3-C



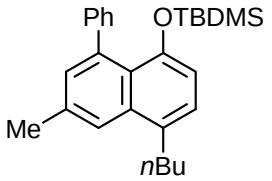
DFILE	C:¥Documents and Settings¥Owner
COMNT	Yym060264-3-C
DATIM	21-05-2008 17:43:36
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32768
FREQU	39308.18 Hz
SCANS	712
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	20.9 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60



5j, Eq (3) ($CDCl_3$)



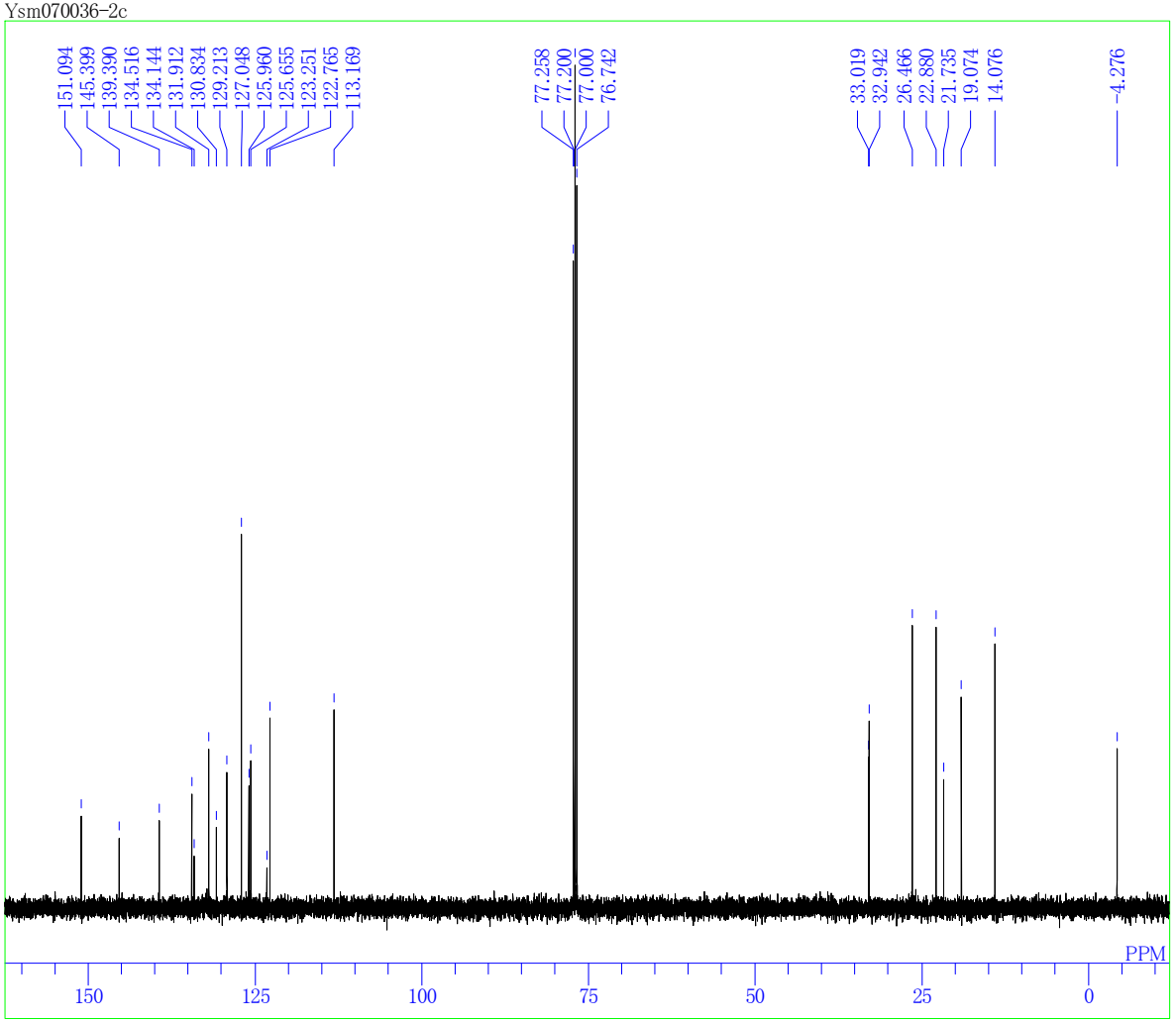
DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	Ysm070104-1
DATIM	Sat Aug 04 15:51:07 2007
OBNUC	1H
EXMOD	SINGL
OBFRQ	499.10 MHz
OBSET	0.00 KHz
OBFIN	125250.00 Hz
POINT	16384
FREQU	9980.04 Hz
SCANS	8
ACQTM	1.6417 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	-204.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	24



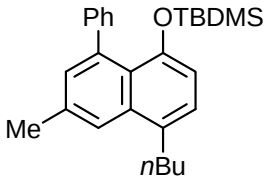
7a ($CDCl_3$)

Table 4, entry 1

Ysm070036-2c

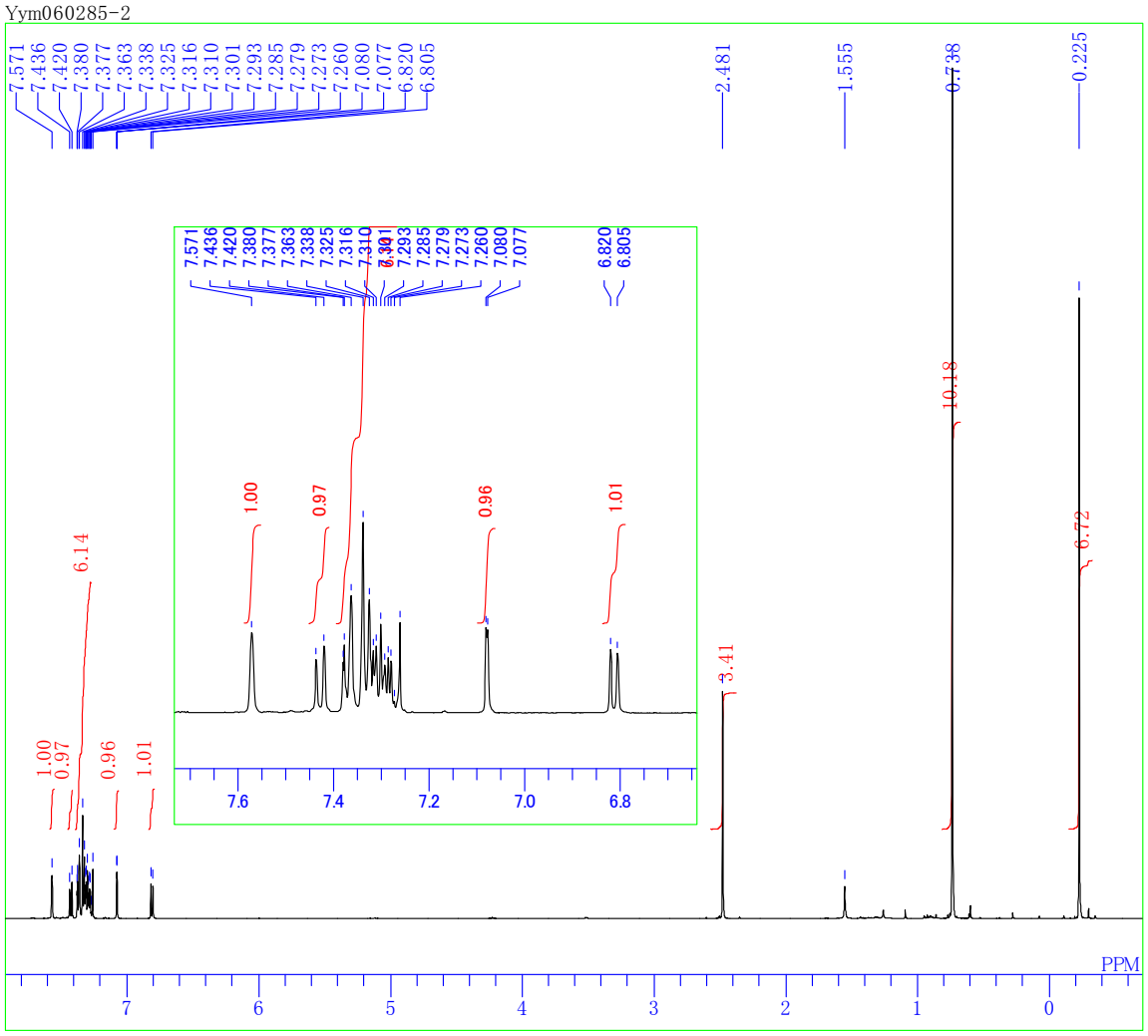


DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	Ysm070036-2c
DATIM	16-06-2007 18:08:17
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.06 Hz
SCANS	182
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	393.3 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

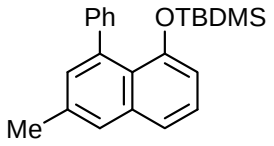


7a ($CDCl_3$)

Table 4, entry 1

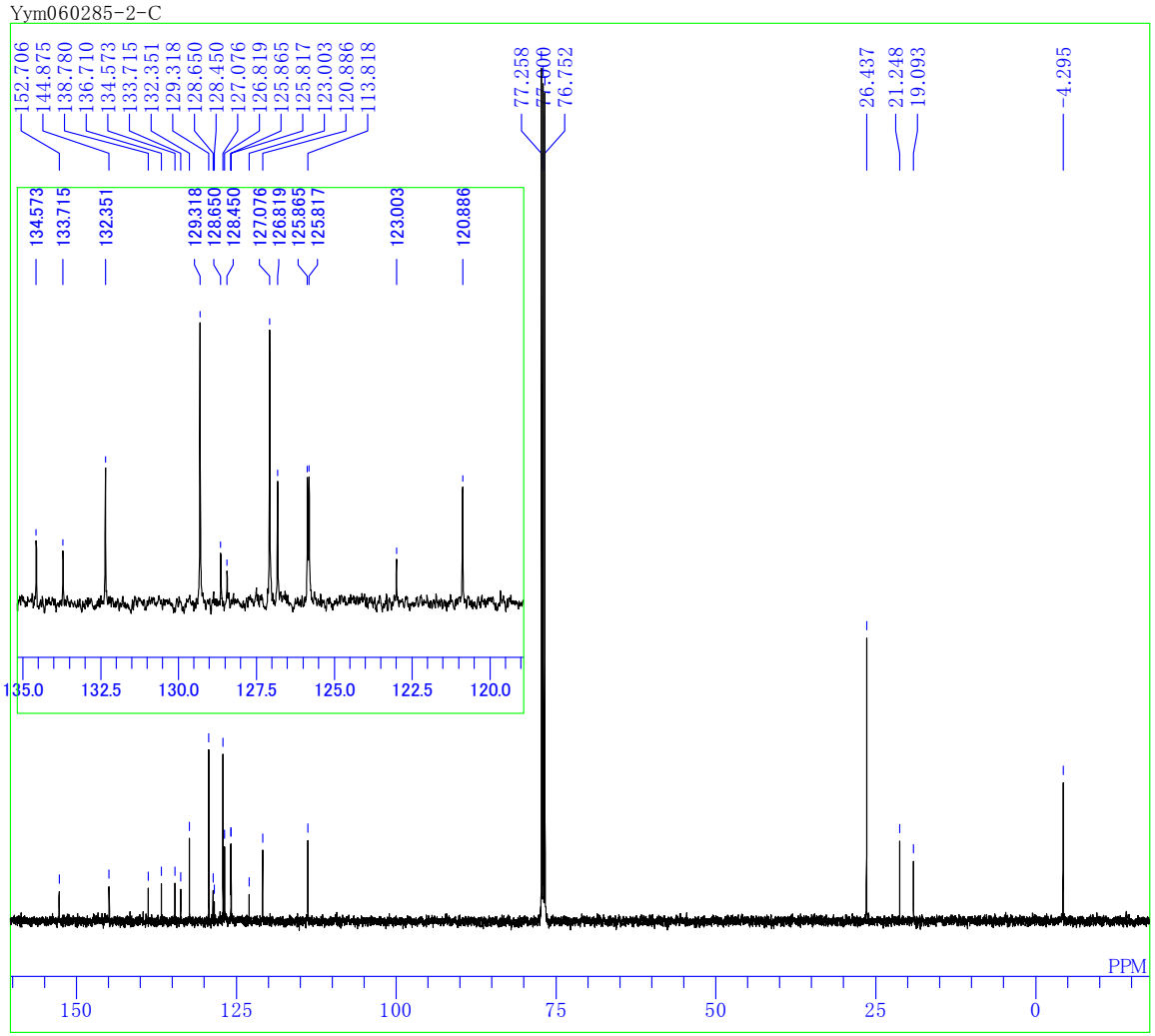


DFILE	C:¥Documents and Settings¥Owner
COMNT	Yym060285-2
DATIM	04-06-2008 11:59:24
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.39 Hz
SCANS	16
ACQTM	1.7459 sec
PD	1.5000 sec
PW1	5.75 usec
IRNUC	1H
CTEMP	20.2 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	48

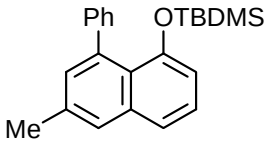


7b ($CDCl_3$)

Table 4, entry 2

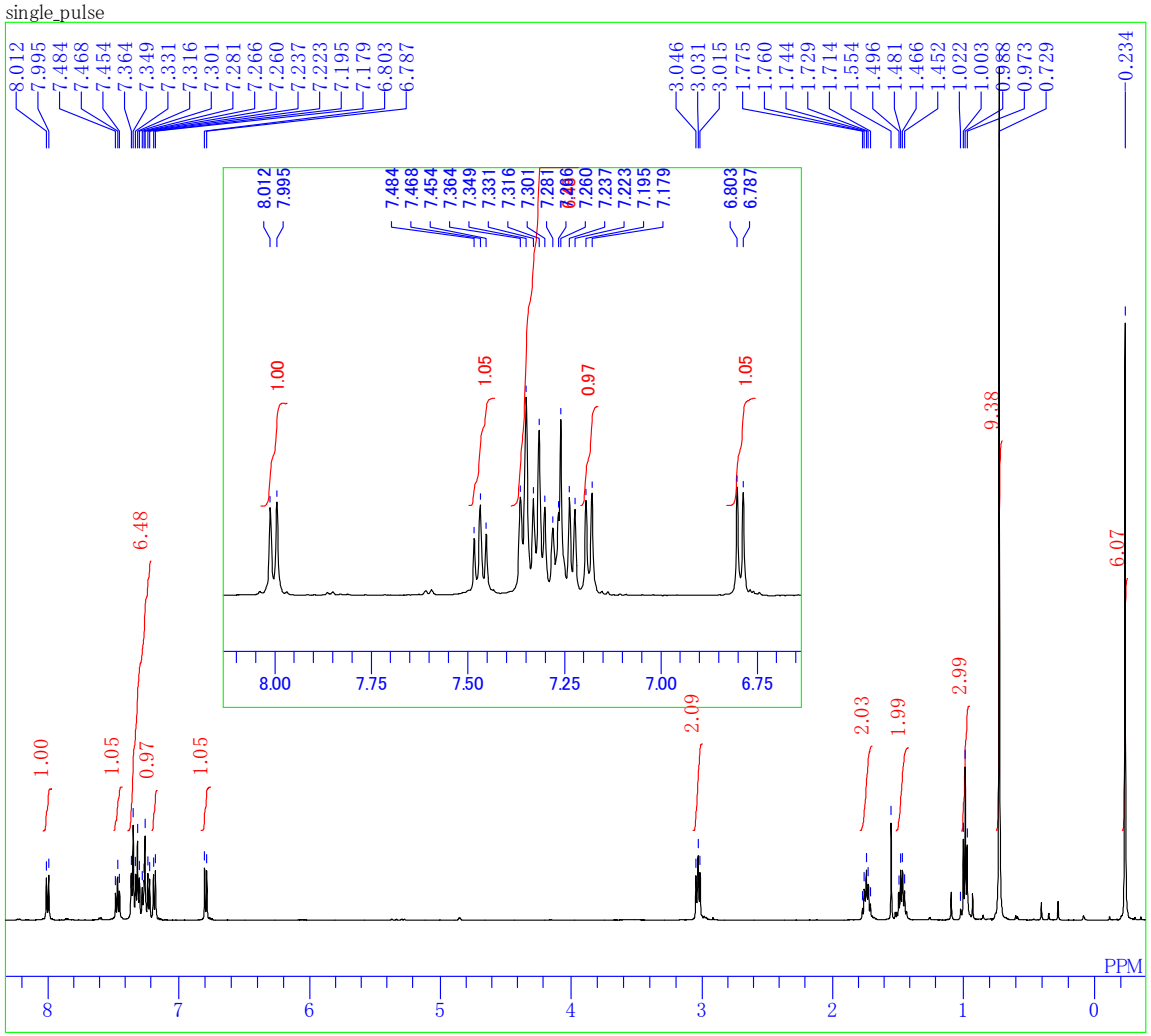


DFILE	C:¥Documents and Settings¥Owner
COMNT	Yym060285-2-C
DATIM	04-06-2008 12:30:57
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32768
FREQU	39308.18 Hz
SCANS	591
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	20.7 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

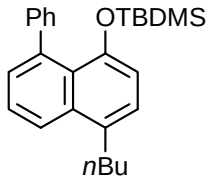


7b ($CDCl_3$)

Table 4, entry 2



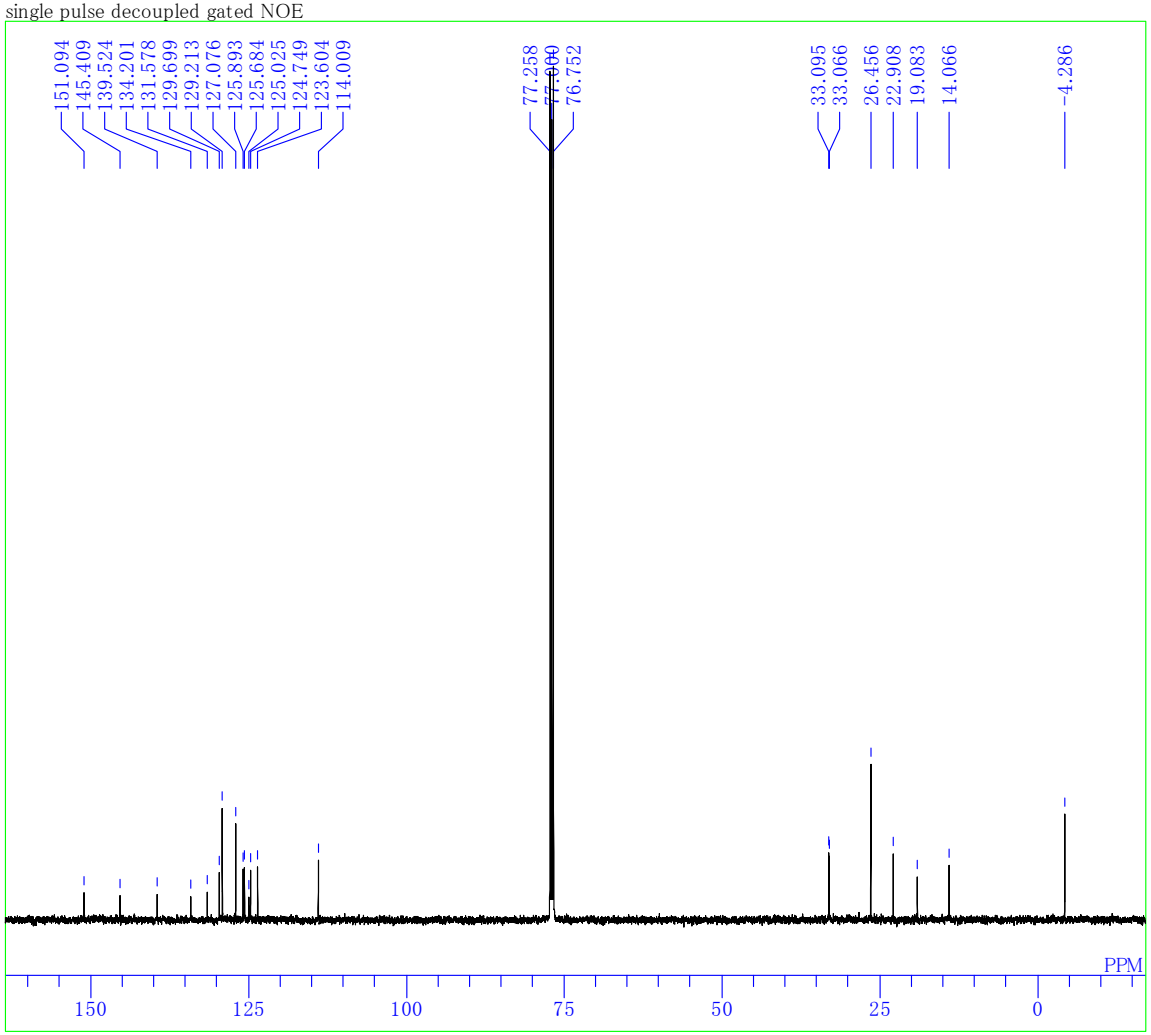
DFILE	C:¥Documents and Settings¥Owner
COMNT	single_pulse
DATIM	13-05-2008 17:19:10
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.39 Hz
SCANS	8
ACQTM	1.7459 sec
PD	1.5000 sec
PW1	5.75 usec
IRNUC	1H
CTEMP	19.4 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	1.20 Hz
RGAIN	48



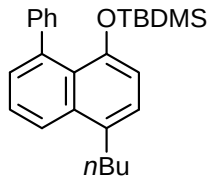
7c ($CDCl_3$)

Table 4, entry 3

single pulse decoupled gated NOE



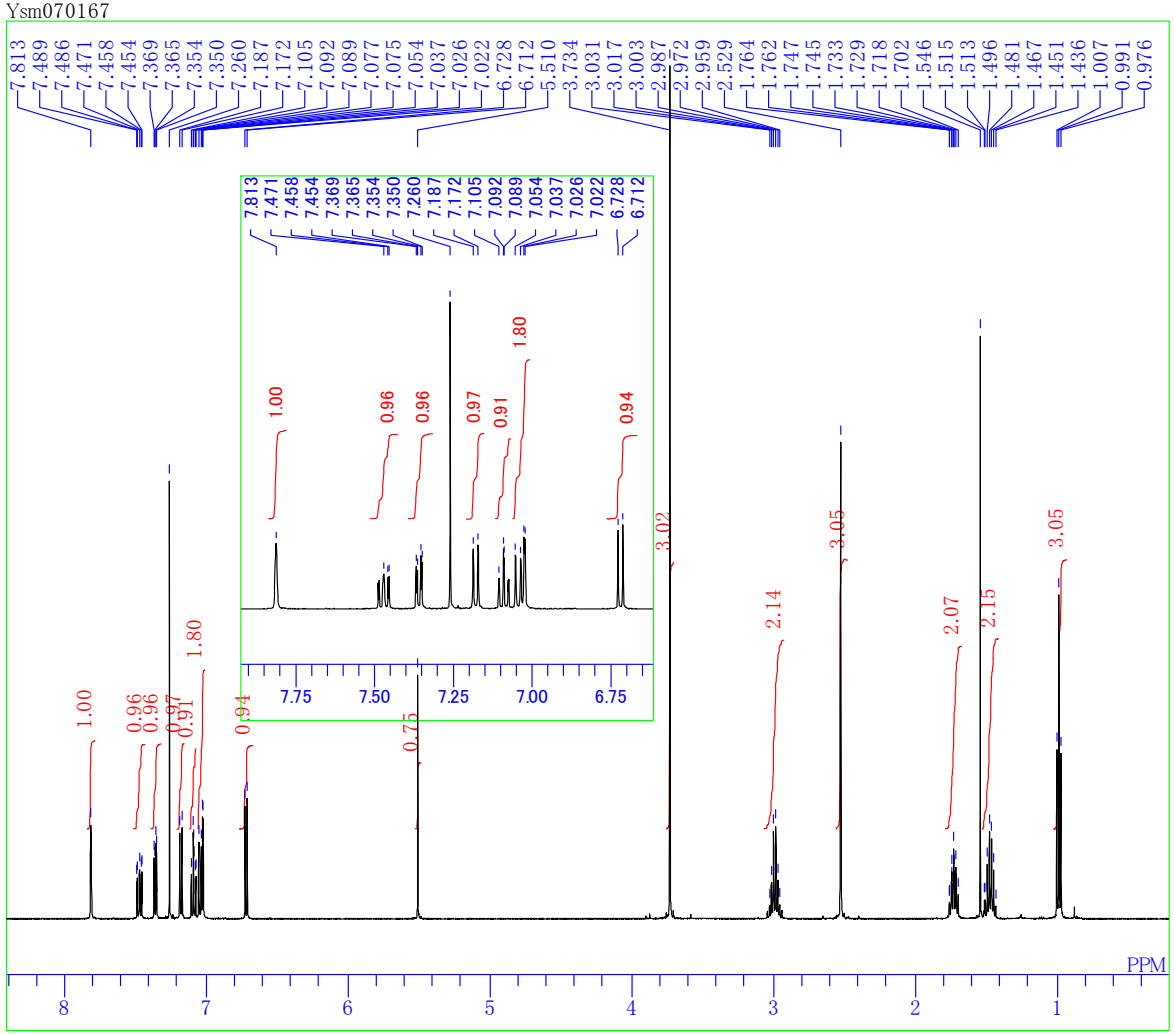
DFILE	C:¥Documents and Settings¥Owner
COMNT	single pulse decoupled gated NOE
DATIM	13-05-2008 18:07:00
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32768
FREQU	39308.18 Hz
SCANS	948
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	19.8 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60



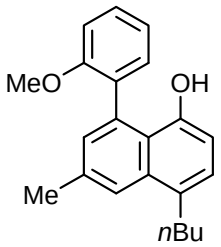
7c ($CDCl_3$)

Table 4, entry 3

Ysm070167



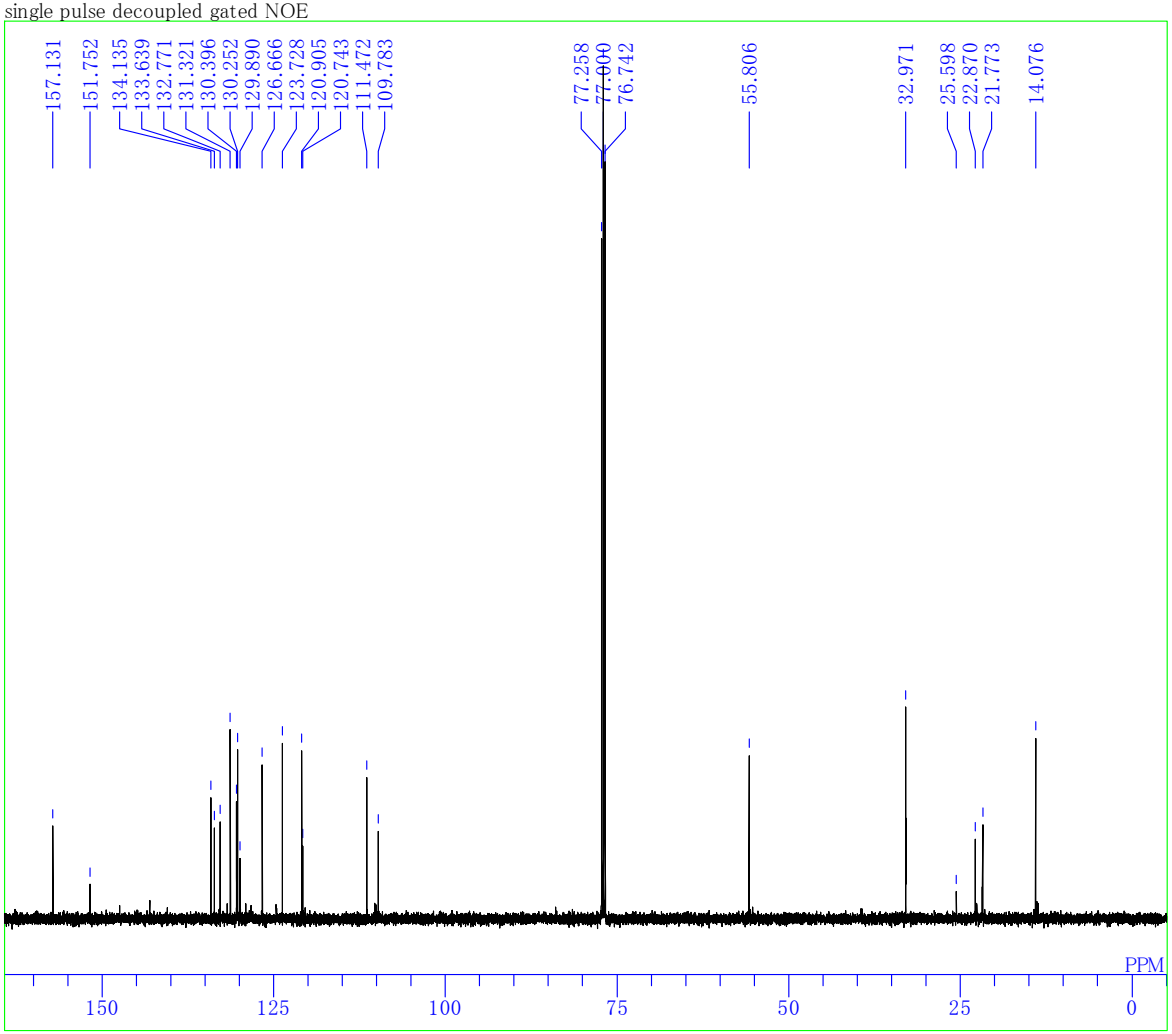
DFILE	C:¥Documents and Settings¥Owner
COMNT	Ysm070167
DATIM	Wed Jan 30 18:59:46 2008
OBNUC	1H
EXMOD	SINGL
OBFRQ	499.10 MHz
OBSET	0.00 KHz
OBFIN	125250.00 Hz
POINT	16384
FREQU	9980.04 Hz
SCANS	8
ACQTM	1.6417 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	-204.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	25



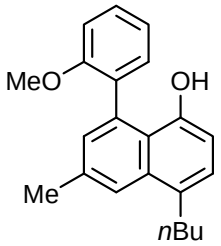
7d' ($CDCl_3$)

Table 4, entry 4

single pulse decoupled gated NOE

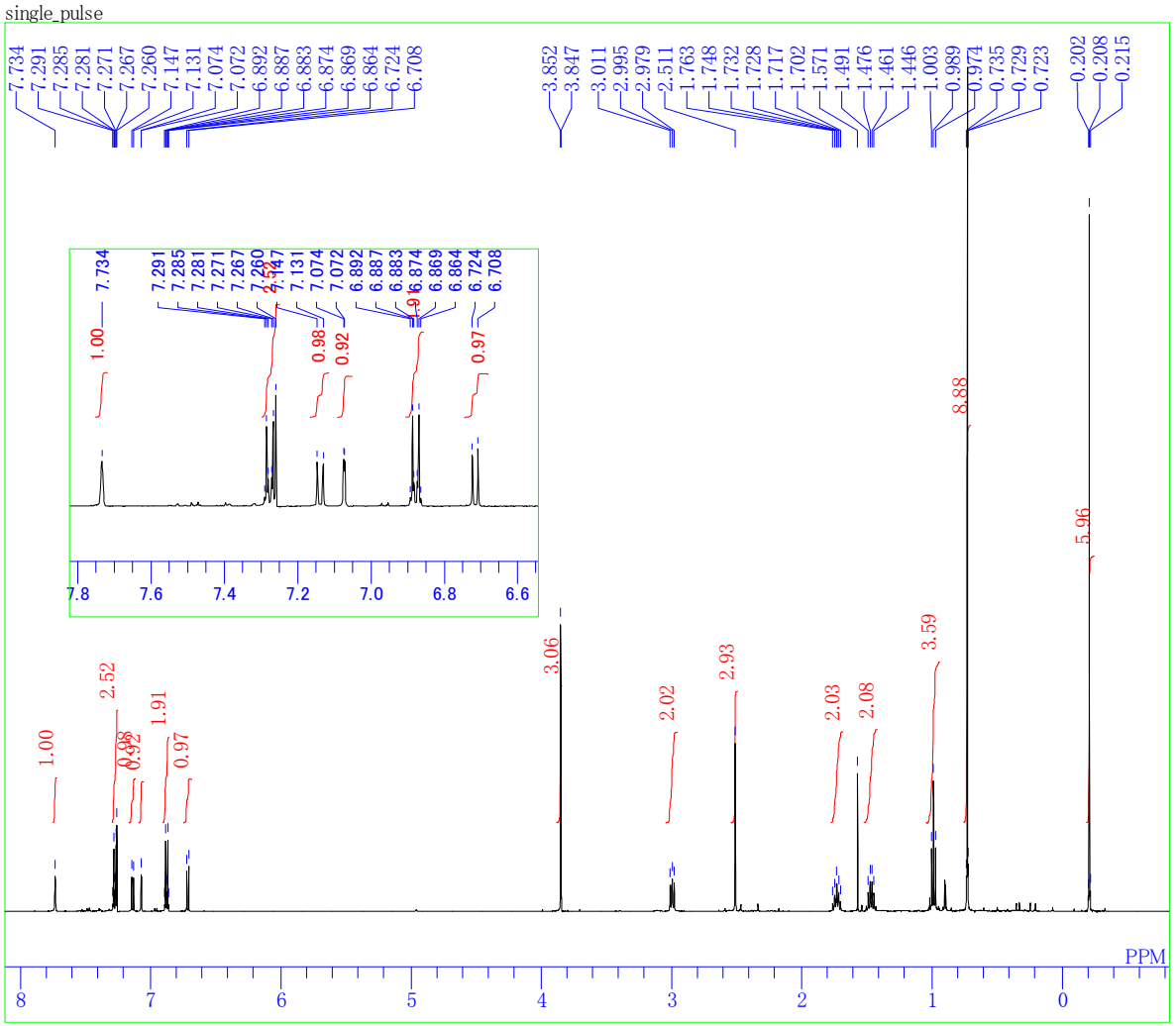


DFILE	C:¥Documents and Settings¥Owner
COMNT	single pulse decoupled gated NOE
DATIM	31-01-2008 14:35:47
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.06 Hz
SCANS	823
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	17.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

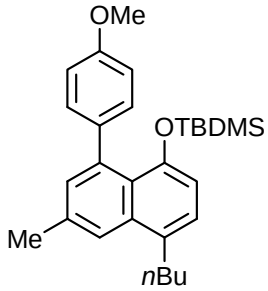


7d' ($CDCl_3$)

Table 4, entry 4



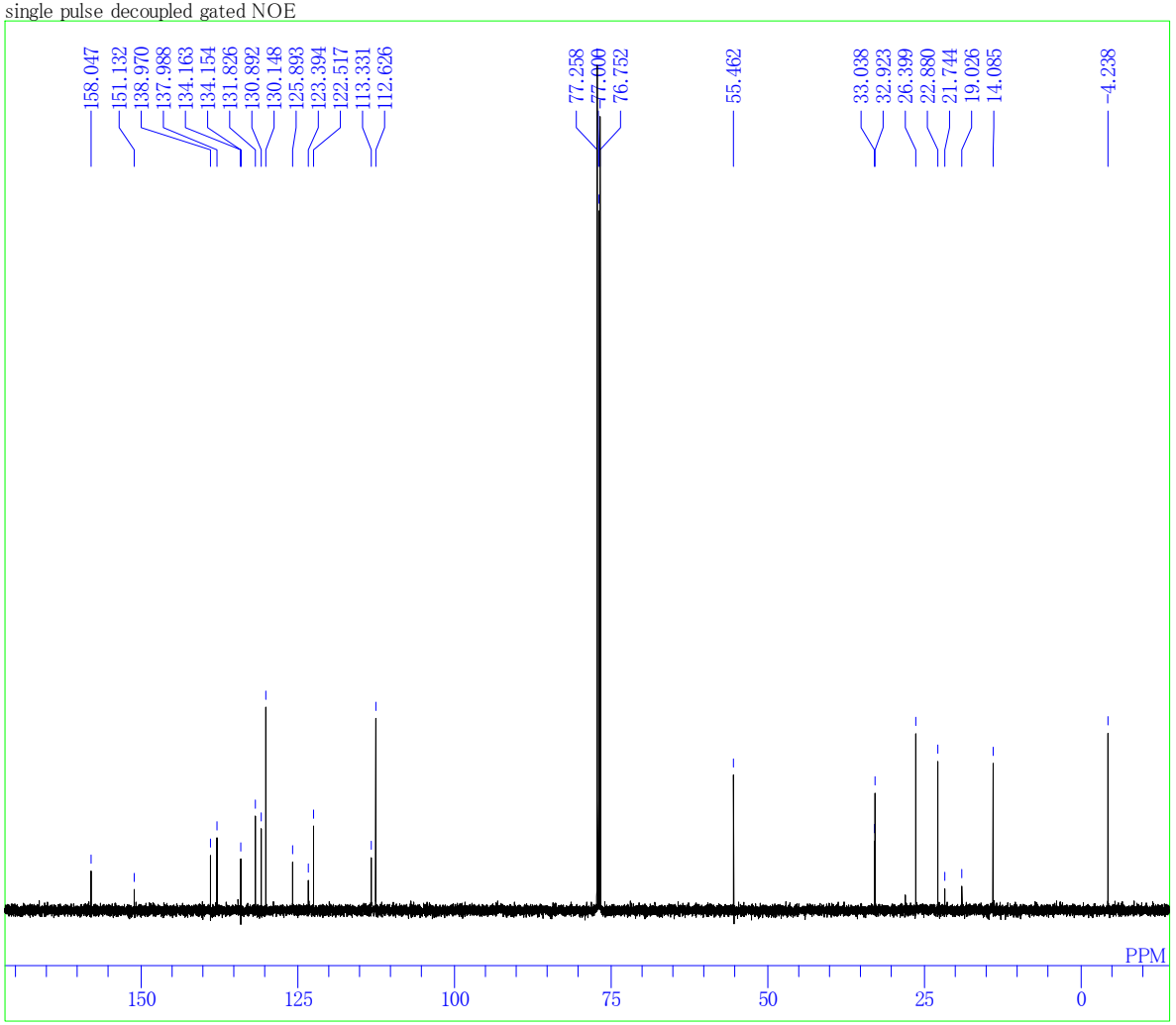
DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single_pulse
DATIM	11-12-2007 14:56:51
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16384
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	1.5000 sec
PW1	6.00 usec
IRNUC	1H
CTEMP	17.4 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.00 Hz
RGAIN	48



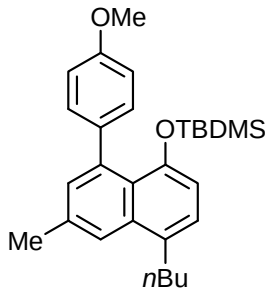
7e ($CDCl_3$)

Table 4, entry 5

single pulse decoupled gated NOE



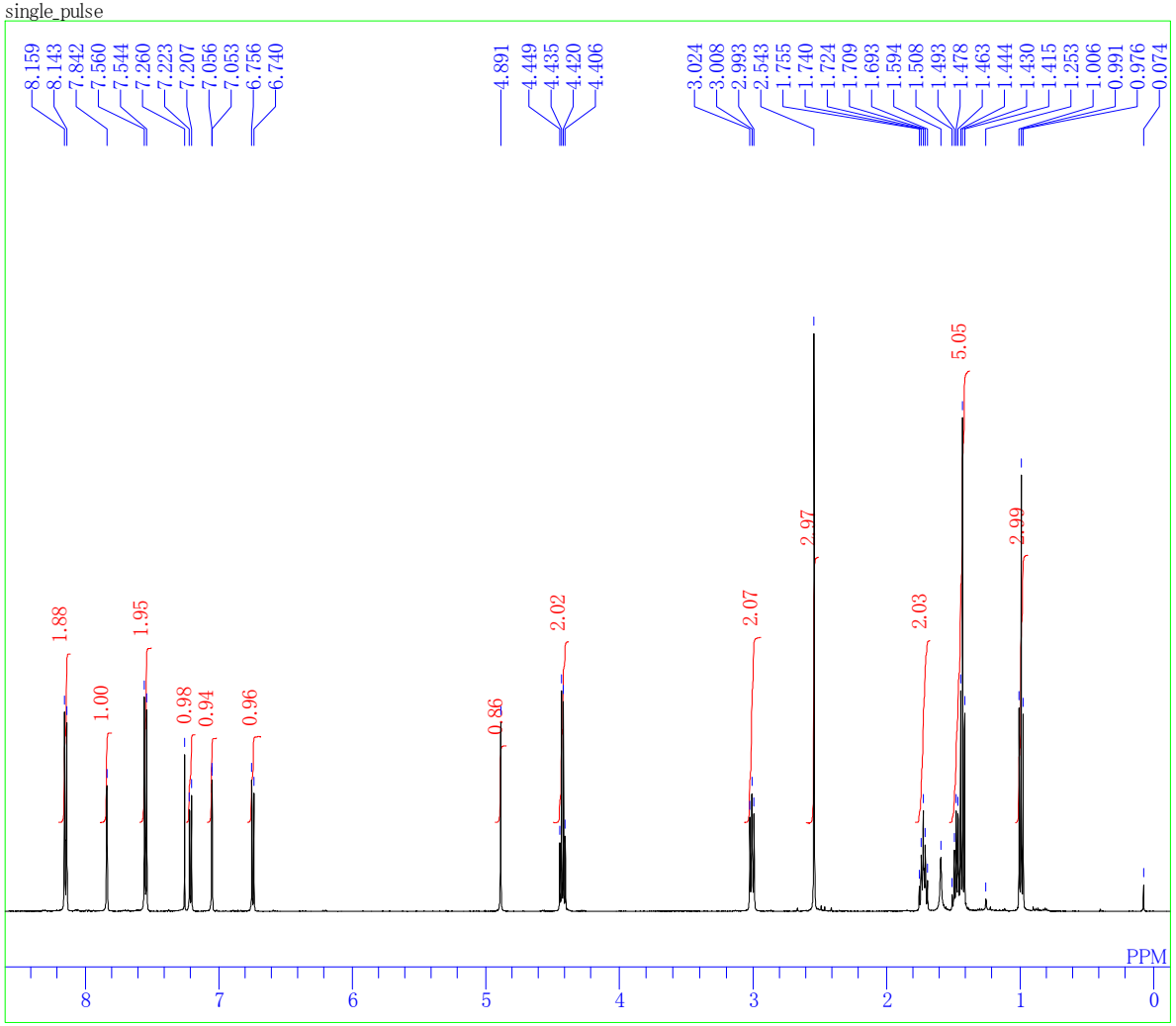
DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single pulse decoupled gated NOE
DATIM	11-12-2007 15:47:36
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32768
FREQU	39308.18 Hz
SCANS	1000
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	17.9 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.00 Hz
RGAIN	60



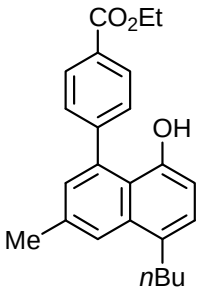
7e ($CDCl_3$)

Table 4, entry 5

single_pulse



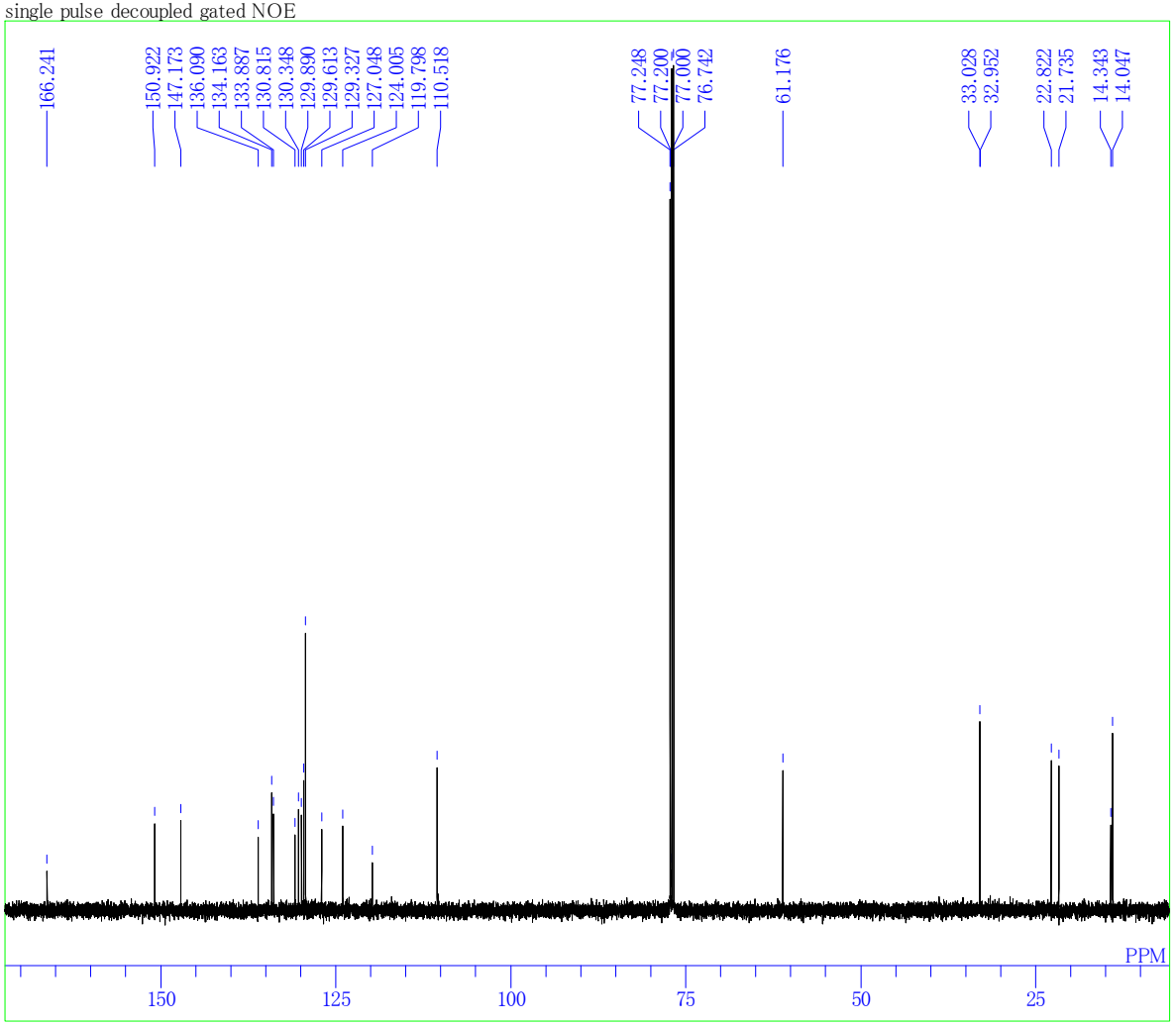
DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single_pulse
DATIM	15-12-2007 12:48:32
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16384
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	1.5000 sec
PW1	6.00 usec
IRNUC	1H
CTEMP	17.7 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.00 Hz
RGAIN	48



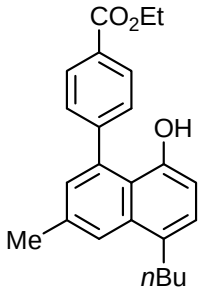
7f' ($CDCl_3$)

Table 4, entry 6

single pulse decoupled gated NOE

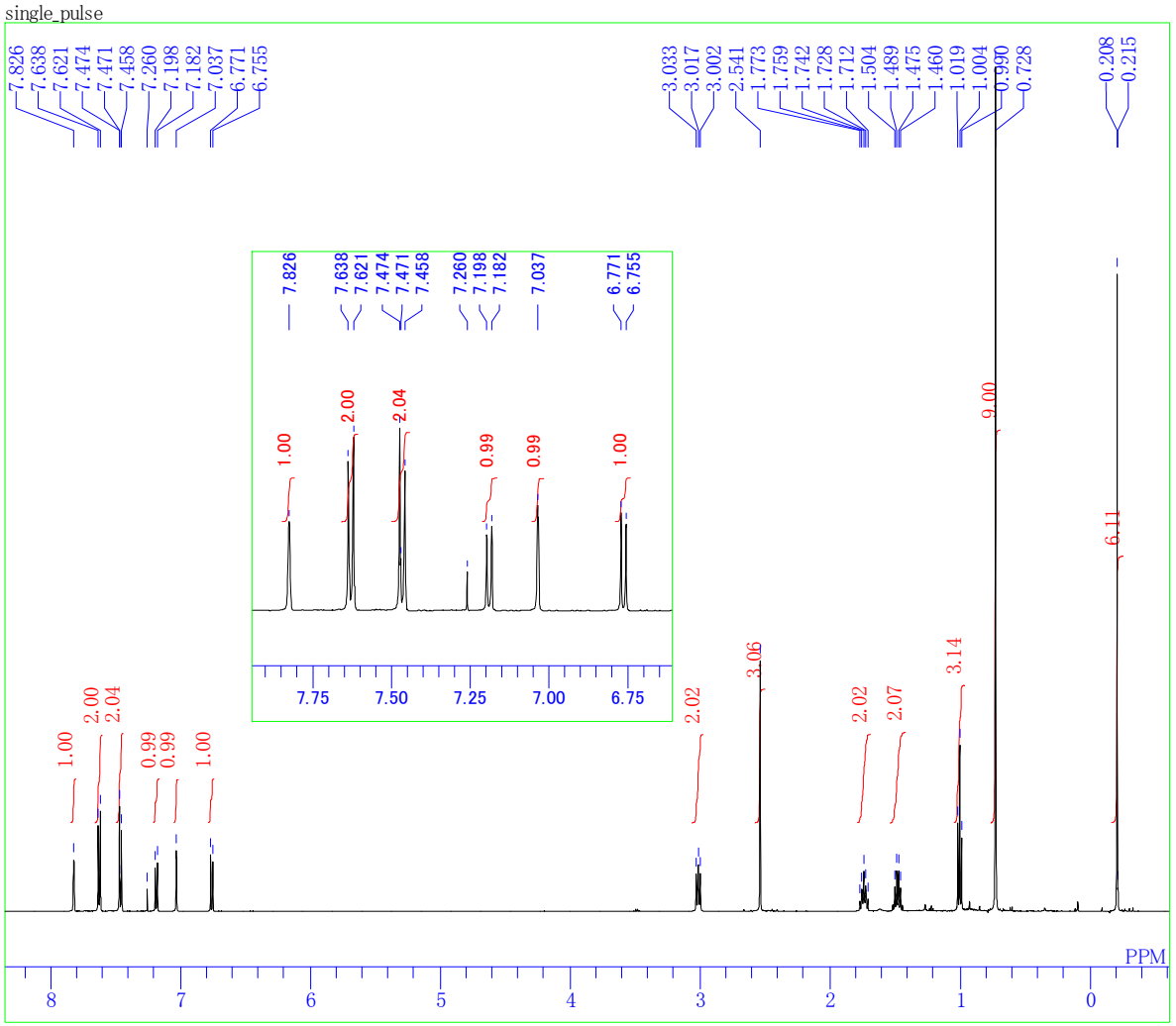


DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single pulse decoupled gated NOE
DATIM	15-12-2007 13:19:20
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32768
FREQU	39308.18 Hz
SCANS	569
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	18.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.00 Hz
RGAIN	60

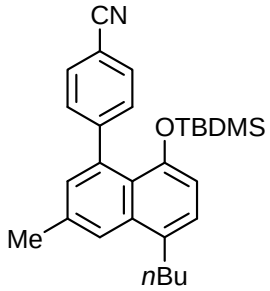


7f' ($CDCl_3$)

Table 4, entry 6



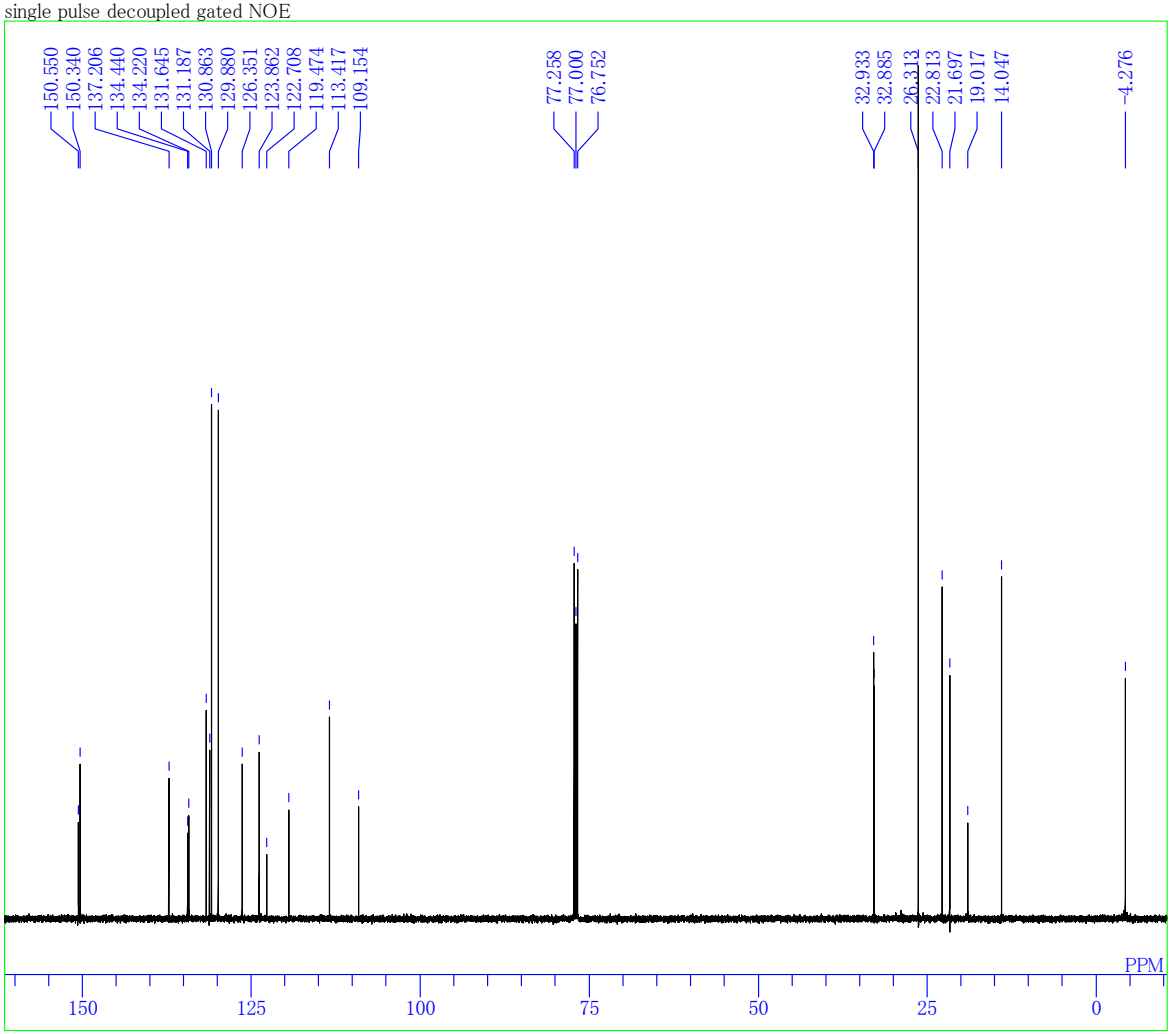
DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single_pulse
DATIM	05-12-2007 13:35:16
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16384
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	6.00 usec
IRNUC	1H
CTEMP	17.6 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.00 Hz
RGAIN	36



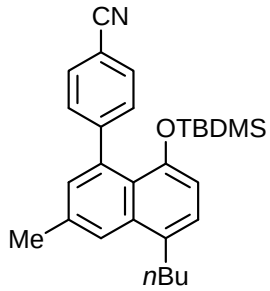
7g ($CDCl_3$)

Table 4, entry 7

single pulse decoupled gated NOE

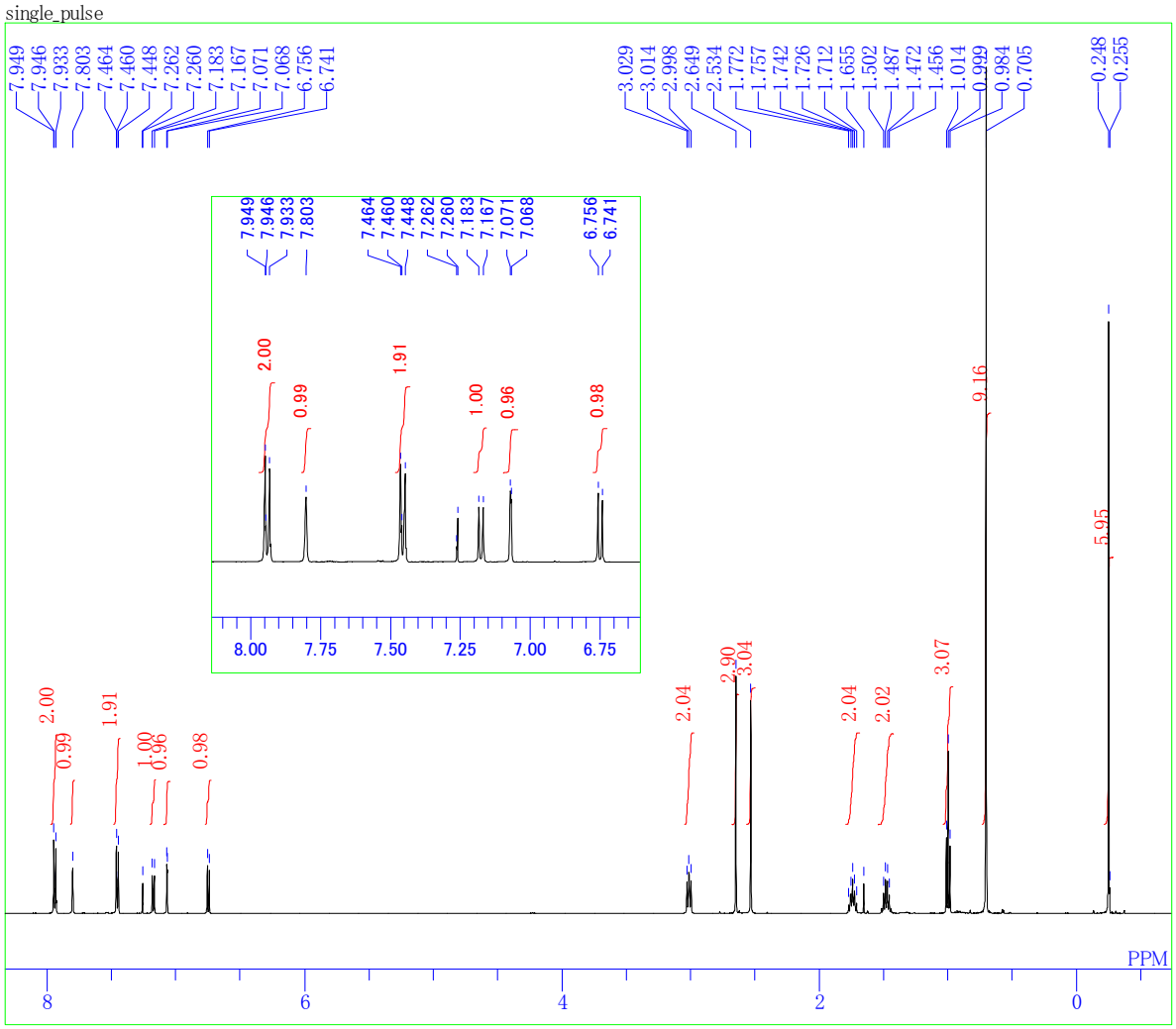


DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single pulse decoupled gated NOE
DATIM	05-12-2007 14:04:33
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32768
FREQU	39308.18 Hz
SCANS	522
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	18.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

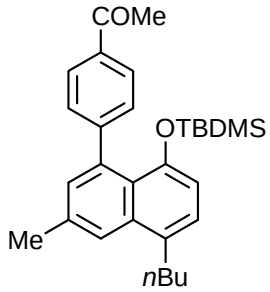


7g ($CDCl_3$)

Table 4, entry 7



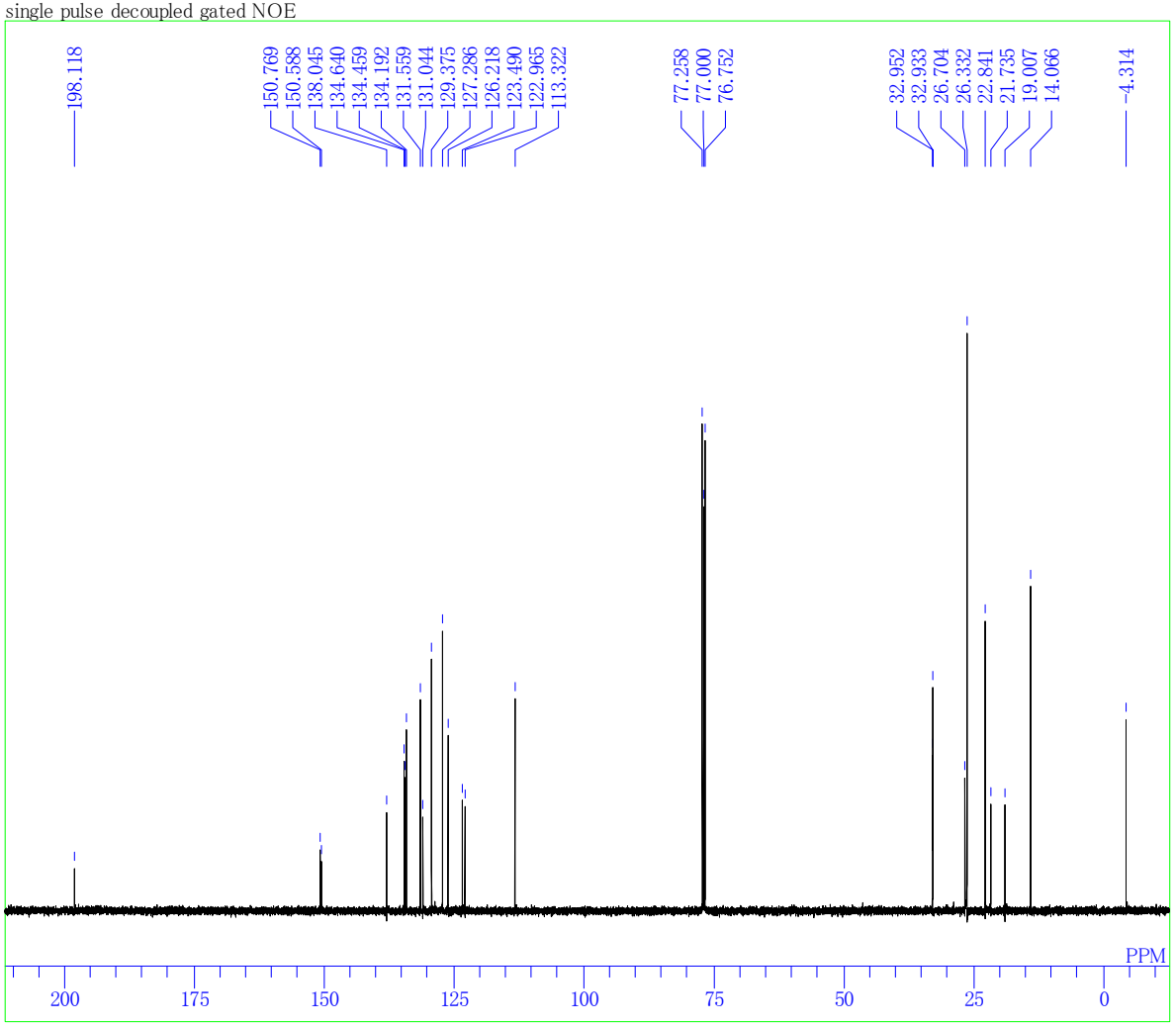
DFILE C:¥Documents and Settings¥Owner¥M
COMNT single_pulse
DATIM 17-12-2007 18:01:31
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 9384.38 Hz
SCANS 8
ACQTM 1.7459 sec
PD 1.5000 sec
PW1 6.00 usec
IRNUC 1H
CTEMP 17.5 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.00 Hz
RGAIN 38



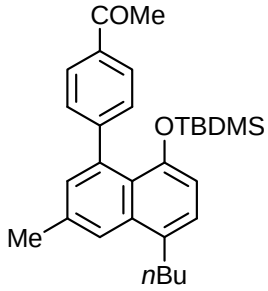
7h ($CDCl_3$)

Table 4, entry 8

single pulse decoupled gated NOE

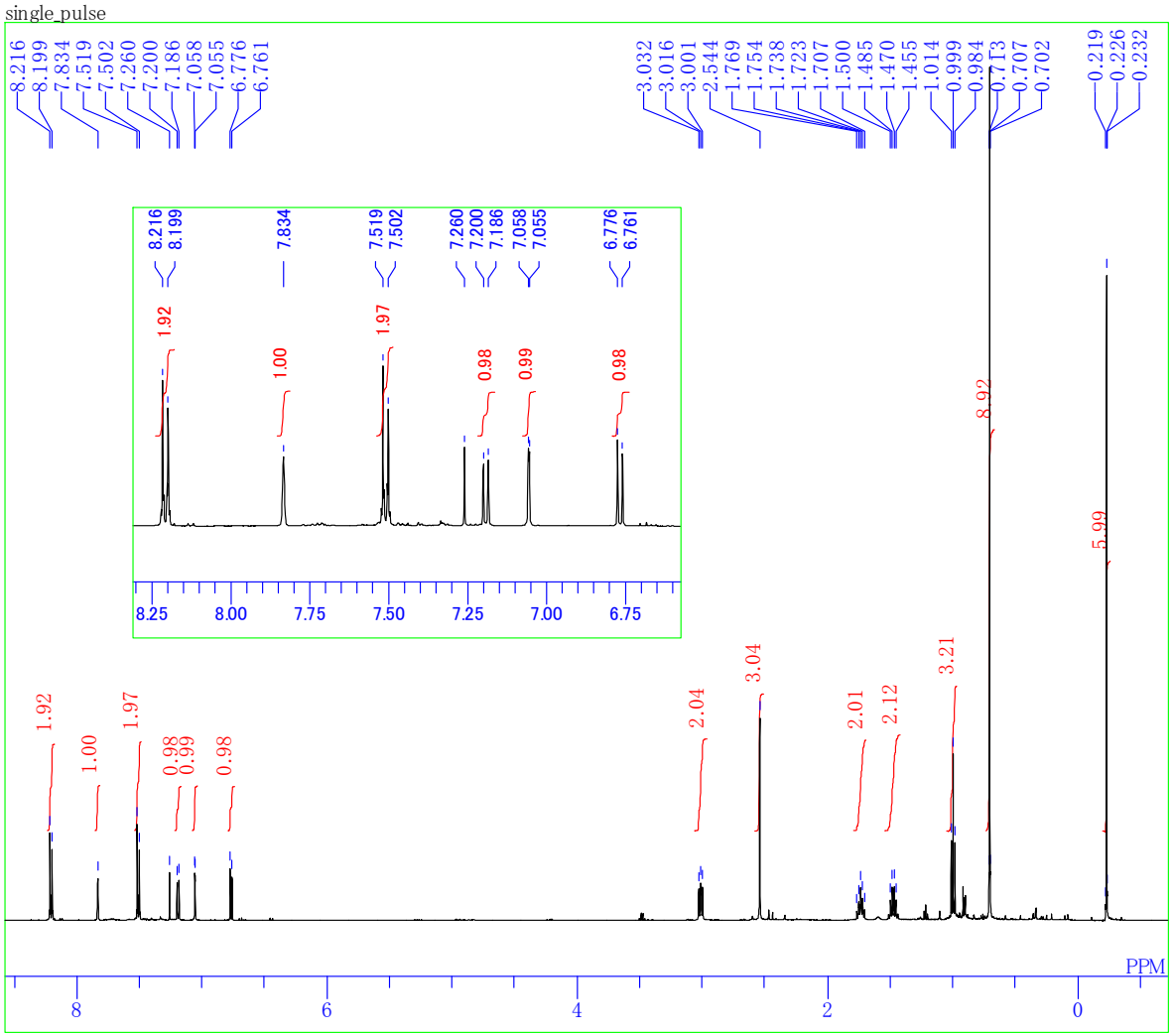


DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single pulse decoupled gated NOE
DATIM	17-12-2007 18:42:37
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32768
FREQU	39308.18 Hz
SCANS	795
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	18.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.00 Hz
RGAIN	60

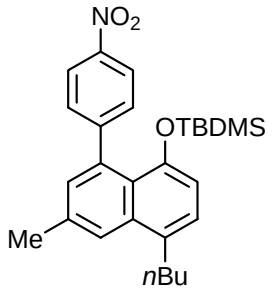


7h ($CDCl_3$)

Table 4, entry 8



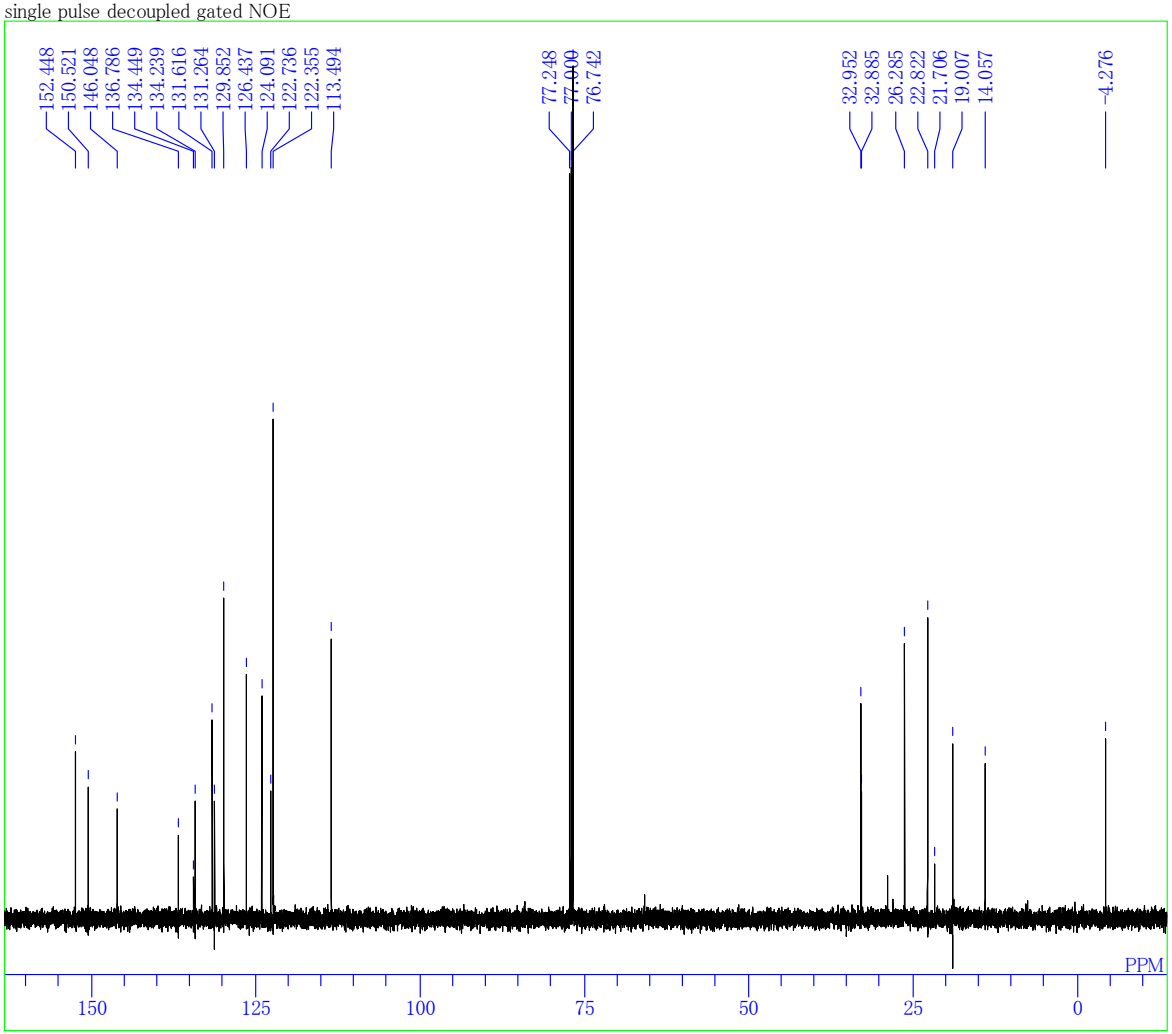
DFILE	C:¥Documents and Settings¥Owner
COMNT	single_pulse
DATIM	05-12-2007 14:14:24
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.39 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	6.00 usec
IRNUC	1H
CTEMP	17.6 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.00 Hz
RGAIN	40



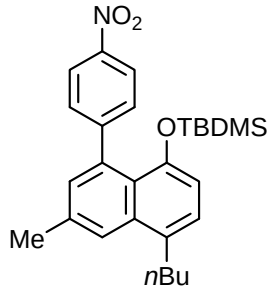
7i ($CDCl_3$)

Table 4, entry 9

single pulse decoupled gated NOE

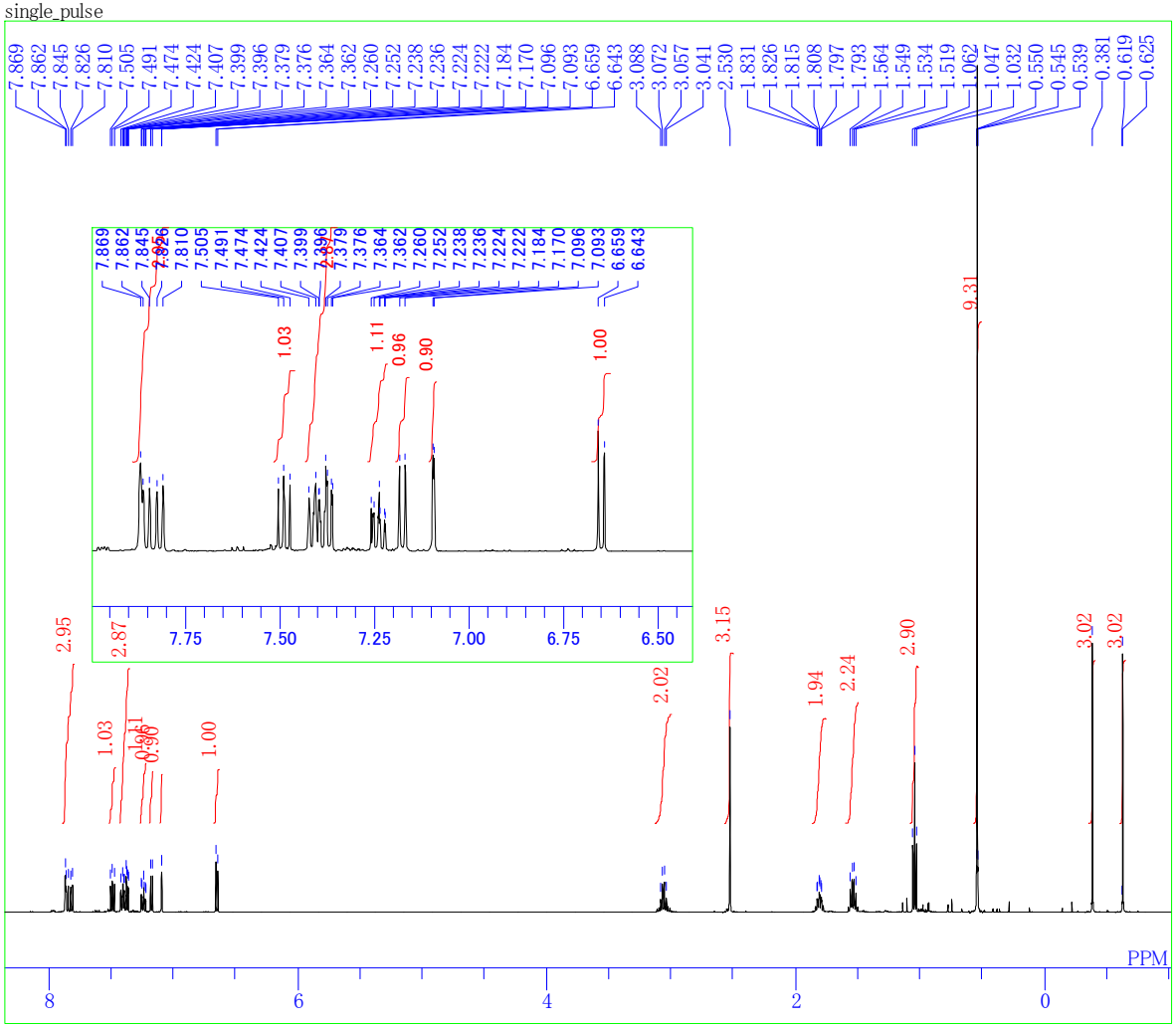


DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single pulse decoupled gated NOE
DATIM	05-12-2007 14:33:40
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32768
FREQU	39308.18 Hz
SCANS	335
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	18.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.00 Hz
RGAIN	60

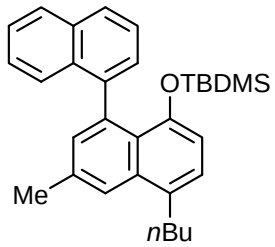


7i ($CDCl_3$)

Table 4, entry 9



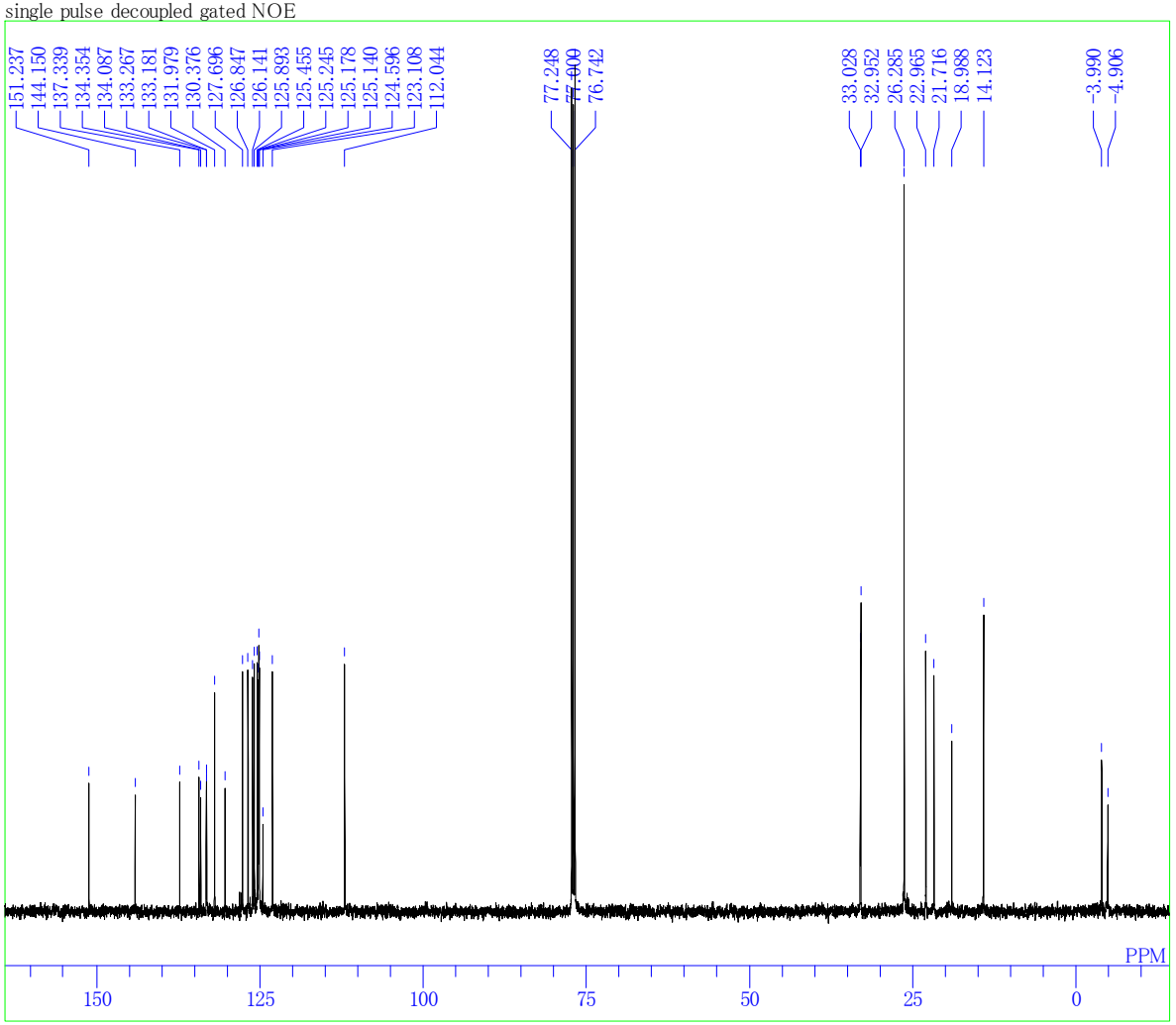
DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single_pulse
DATIM	05-12-2007 13:07:35
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16384
FREQU	9384.38 Hz
SCANS	16
ACQTM	1.7459 sec
PD	1.5000 sec
PW1	6.00 usec
IRNUC	1H
CTEMP	17.5 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.00 Hz
RGAIN	38



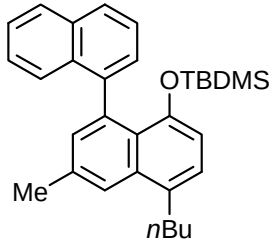
7j ($CDCl_3$)

Table 4, entry 10

single pulse decoupled gated NOE

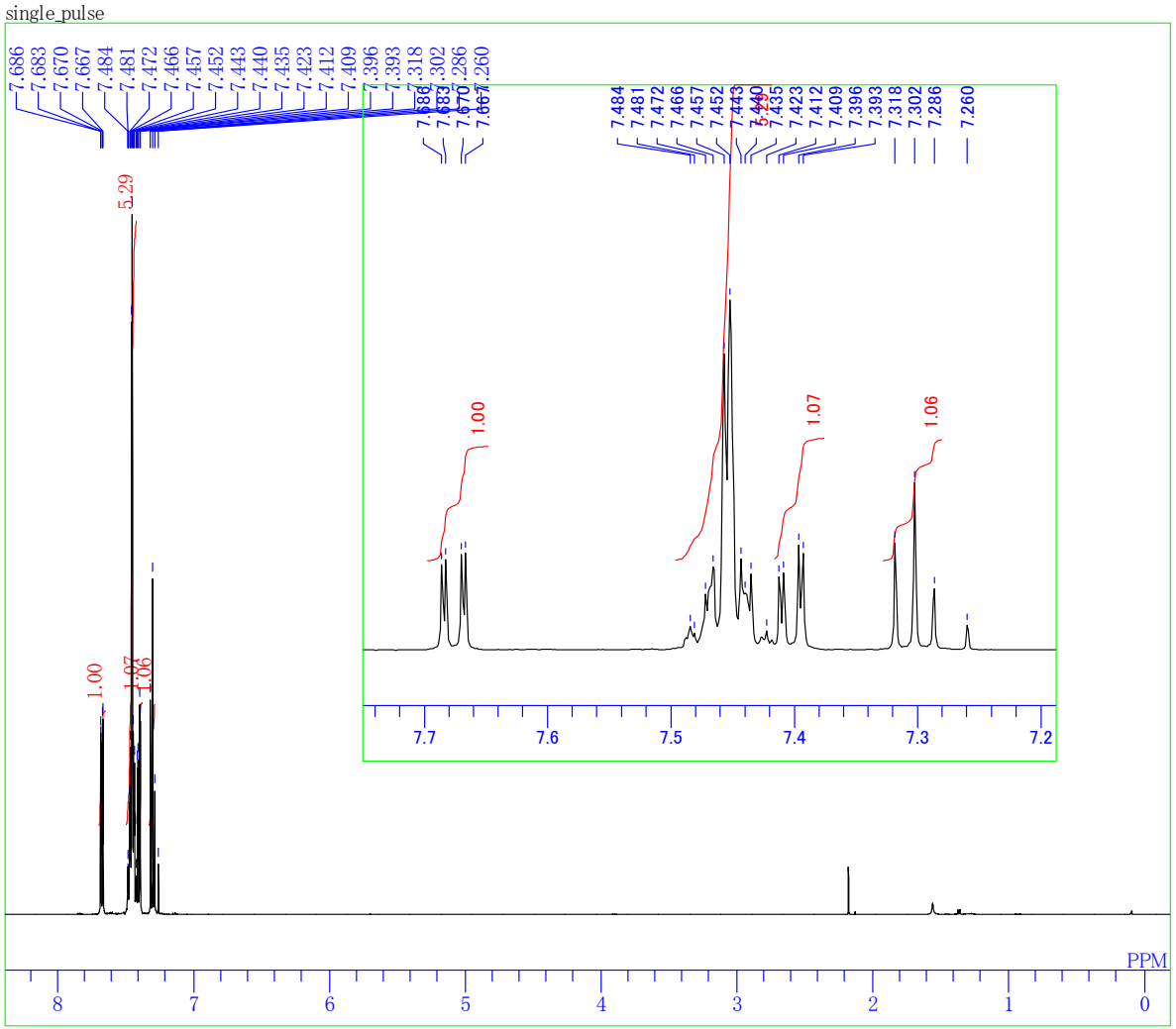


DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single pulse decoupled gated NOE
DATIM	05-12-2007 13:27:59
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	40961
FREQU	49135.97 Hz
SCANS	336
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	18.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.00 Hz
RGAIN	60

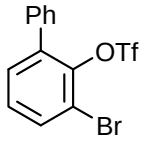


7j ($CDCl_3$)

Table 4, entry 10



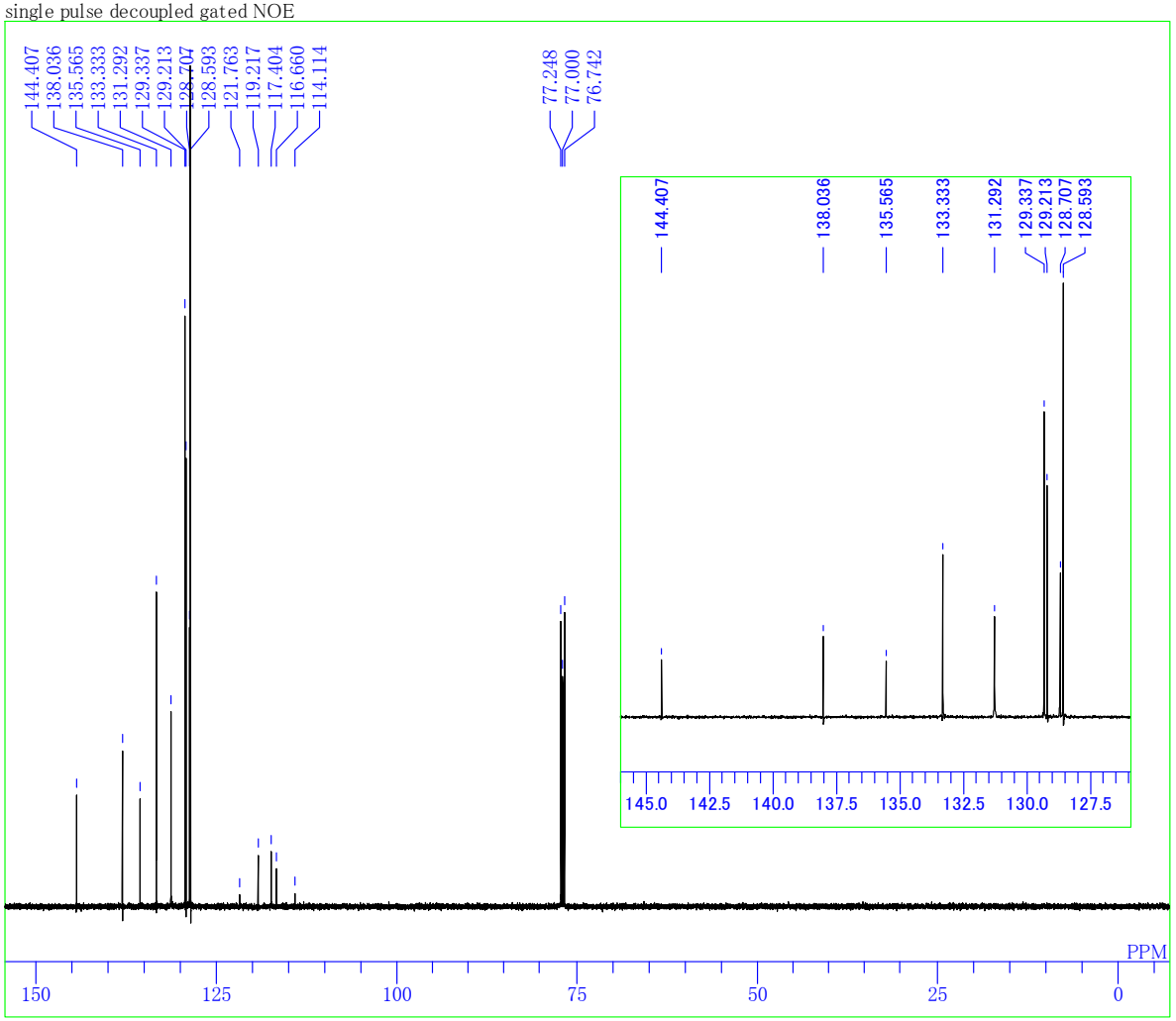
DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single_pulse
DATIM	19-12-2007 18:53:33
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.39 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	6.00 usec
IRNUC	1H
CTEMP	17.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	42



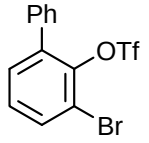
1a ($CDCl_3$)

Table 1, entry 1

single pulse decoupled gated NOE



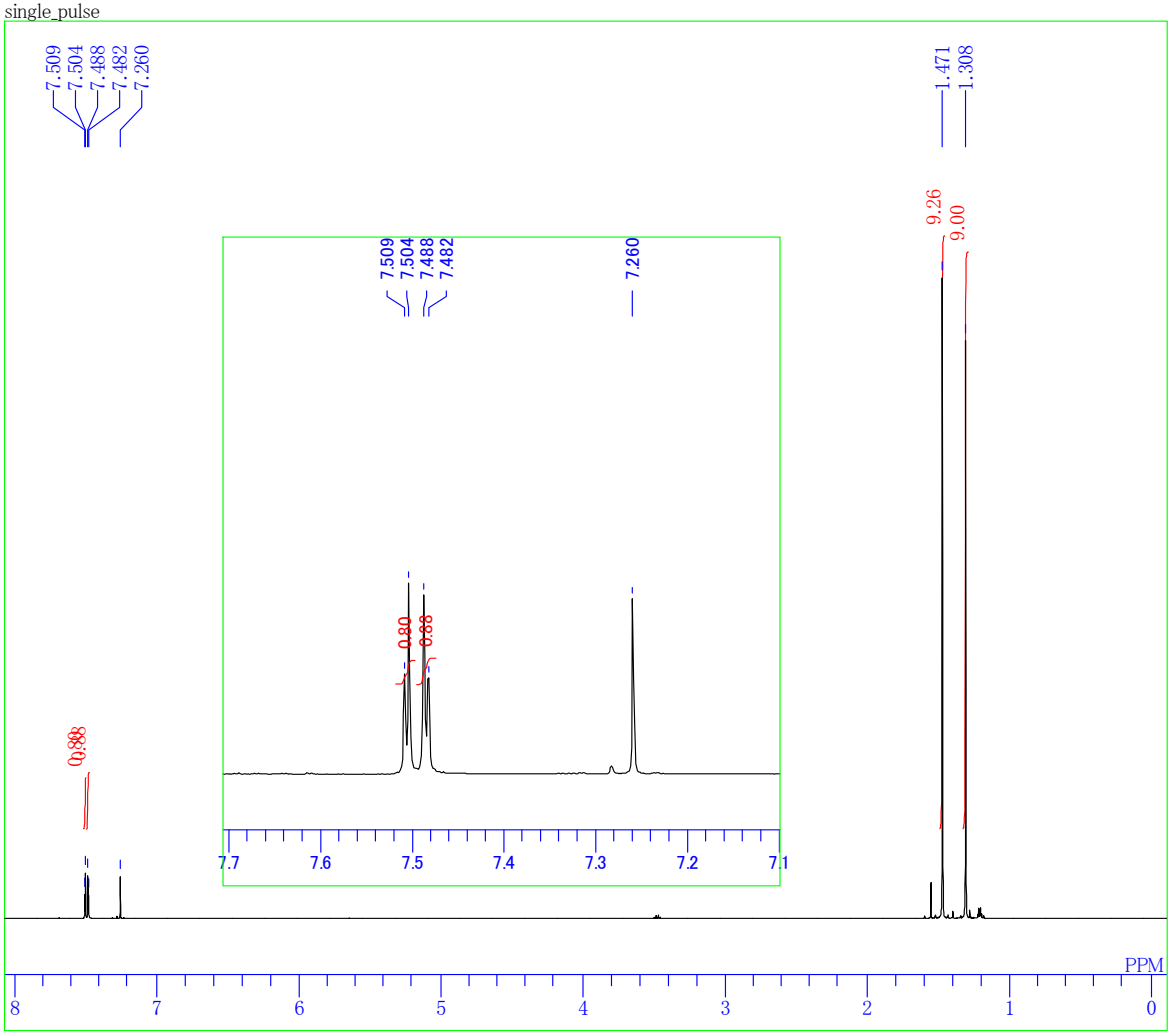
DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single pulse decoupled gated NOE
DATIM	19-12-2007 19:19:19
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32768
FREQU	39308.18 Hz
SCANS	489
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	18.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60



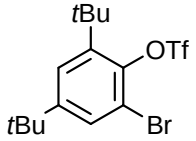
1a ($CDCl_3$)

Table 1, entry 1

single_pulse



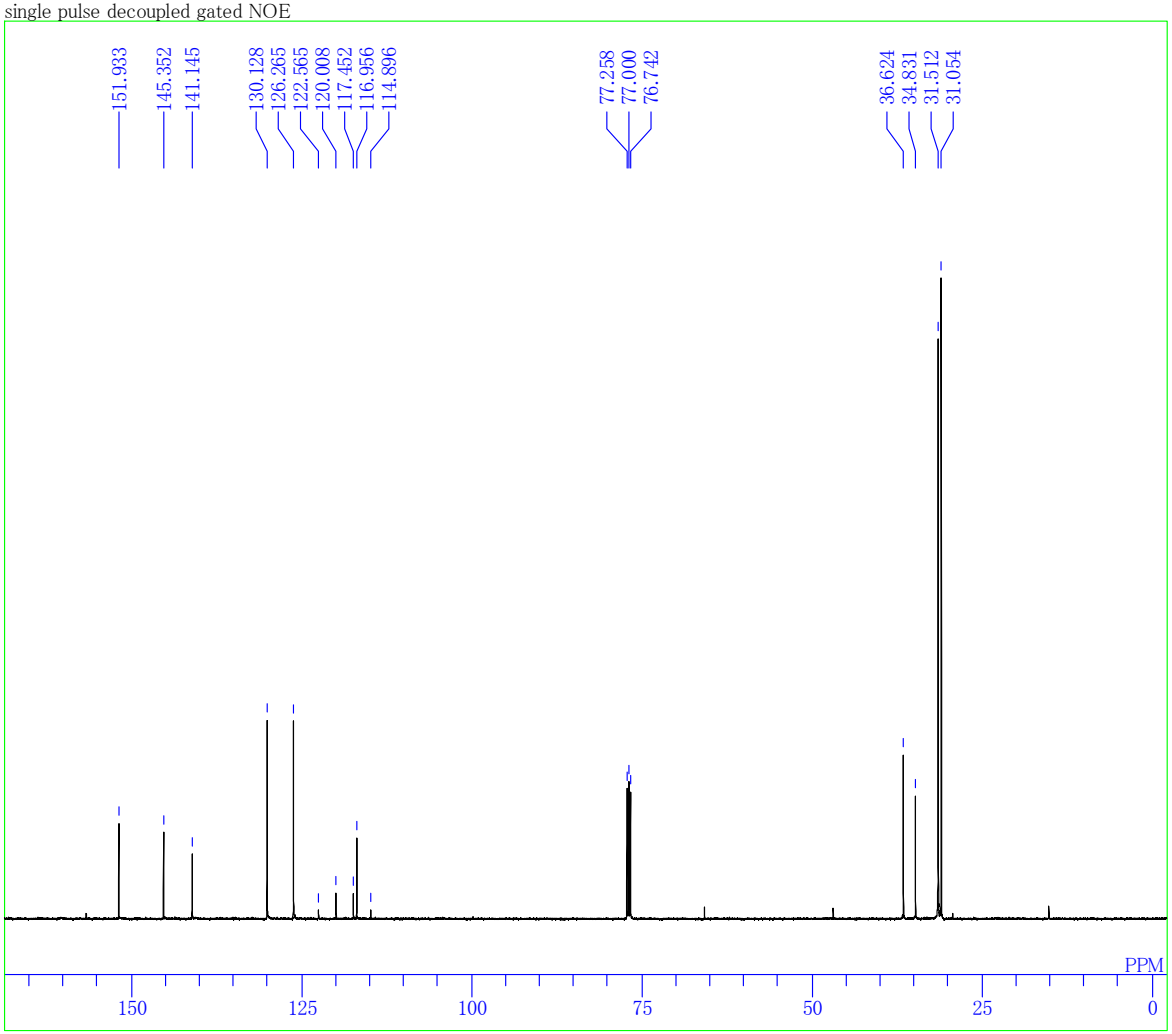
DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single_pulse
DATIM	14-04-2008 12:58:49
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	20480
FREQU	11730.66 Hz
SCANS	8
ACQTM	1.7459 sec
PD	1.5000 sec
PW1	6.00 usec
IRNUC	1H
CTEMP	19.5 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	50



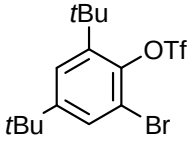
1b ($CDCl_3$)

Table 1, entry 2

single pulse decoupled gated NOE

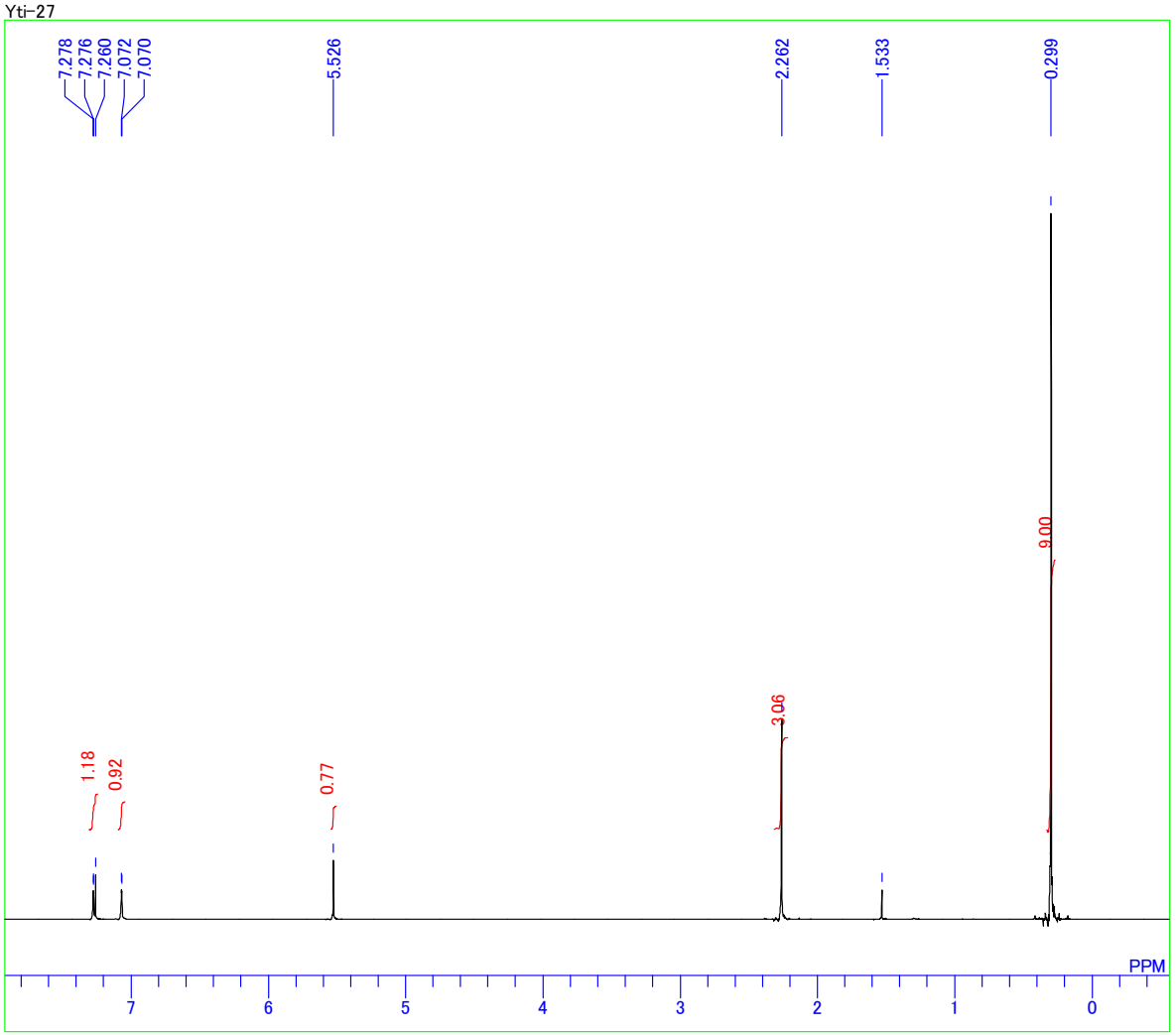


DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single pulse decoupled gated NOE
DATIM	14-04-2008 12:54:49
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	40961
FREQU	49135.97 Hz
SCANS	464
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	20.0 c
SLVNT	CDCL3
EXREF	224.91 ppm
BF	0.12 Hz
RGAIN	60

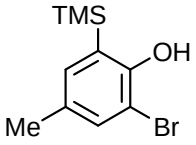


1b ($CDCl_3$)

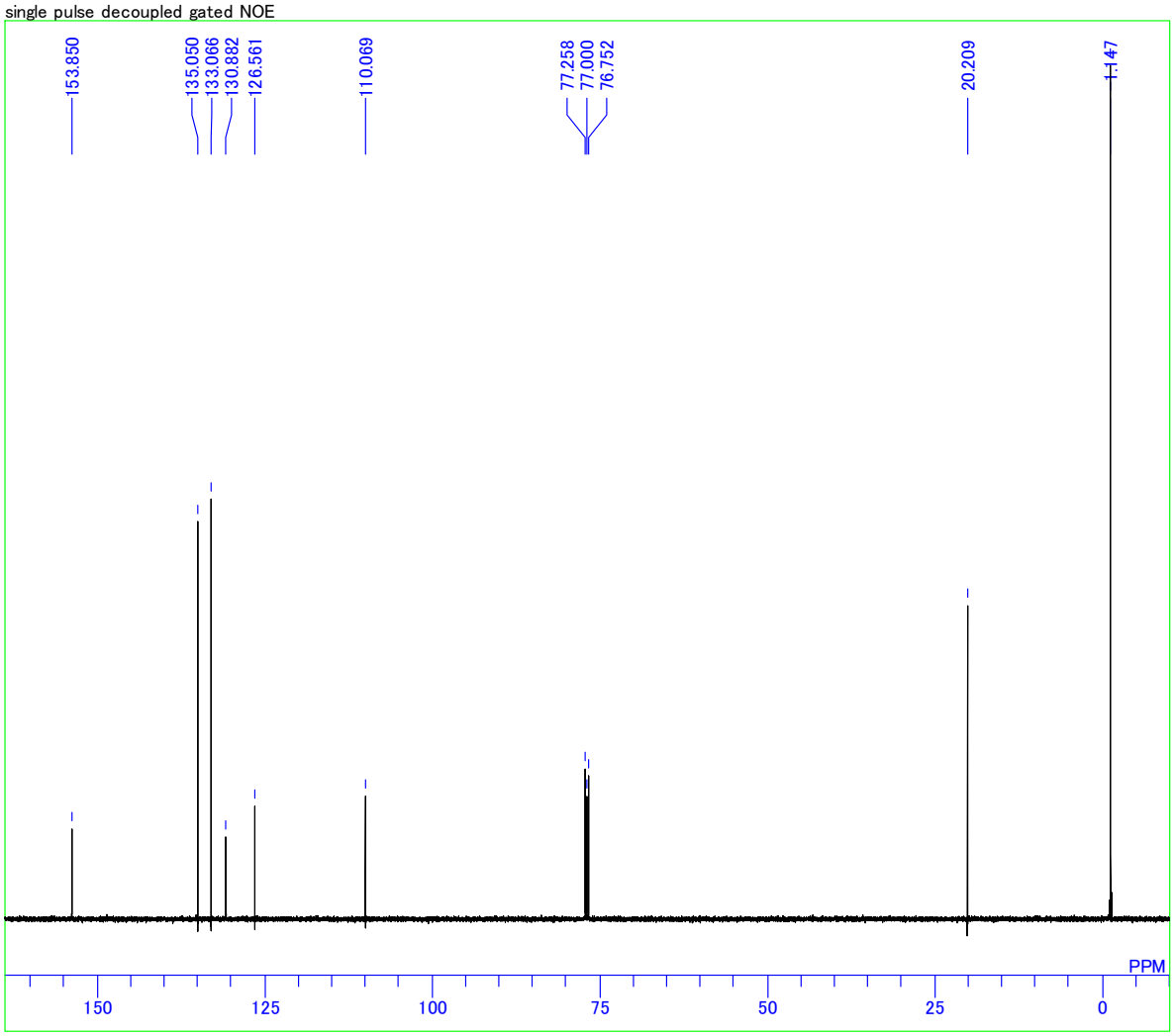
Table 1, entry 2



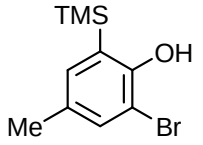
DFILE C:¥Documents and Settings¥Owner¥My D
COMNT Yti-27
DATIM Fri Jun 01 11:51:02 2007
OBNUC 1H
EXMOD SINGL
OBFRQ 499.10 MHz
OBSET 0.00 KHz
OBFIN 125250.00 Hz
POINT 16384
FREQU 9980.04 Hz
SCANS 8
ACQTM 1.6417 sec
PD 2.0000 sec
PW1 5.50 usec
IRNUC 1H
CTEMP -204.8 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 21



S3c ($CDCl_3$)
Starting Material of **1c**

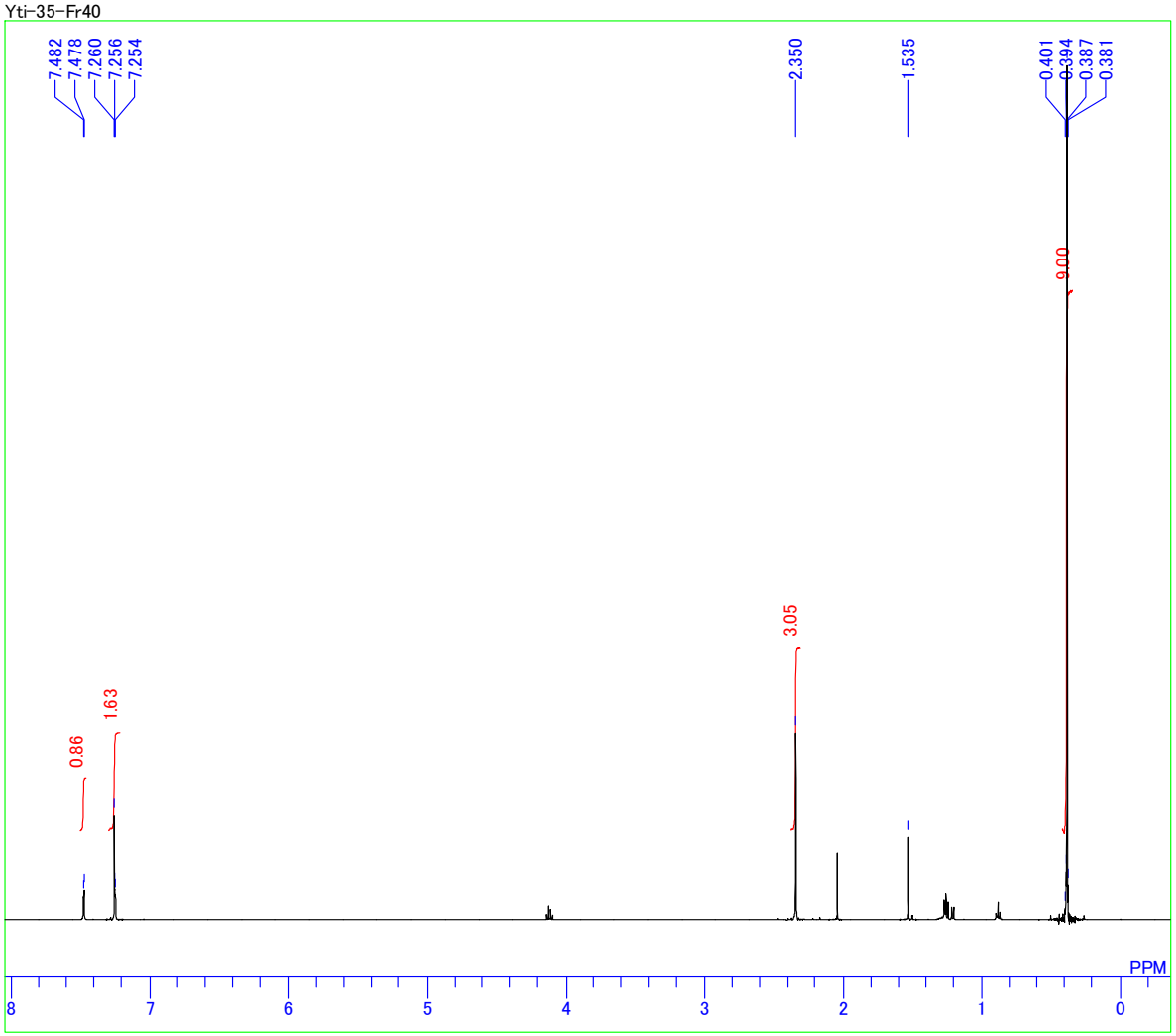


DFILE	C:¥Documents and Settings¥Owner¥My D
COMNT	single pulse decoupled gated NOE
DATIM	19-12-2007 19:32:25
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32768
FREQU	39308.18 Hz
SCANS	166
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	18.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

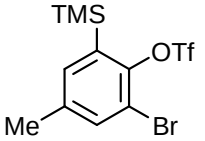


S3c ($CDCl_3$)

Starting Material of **1c**



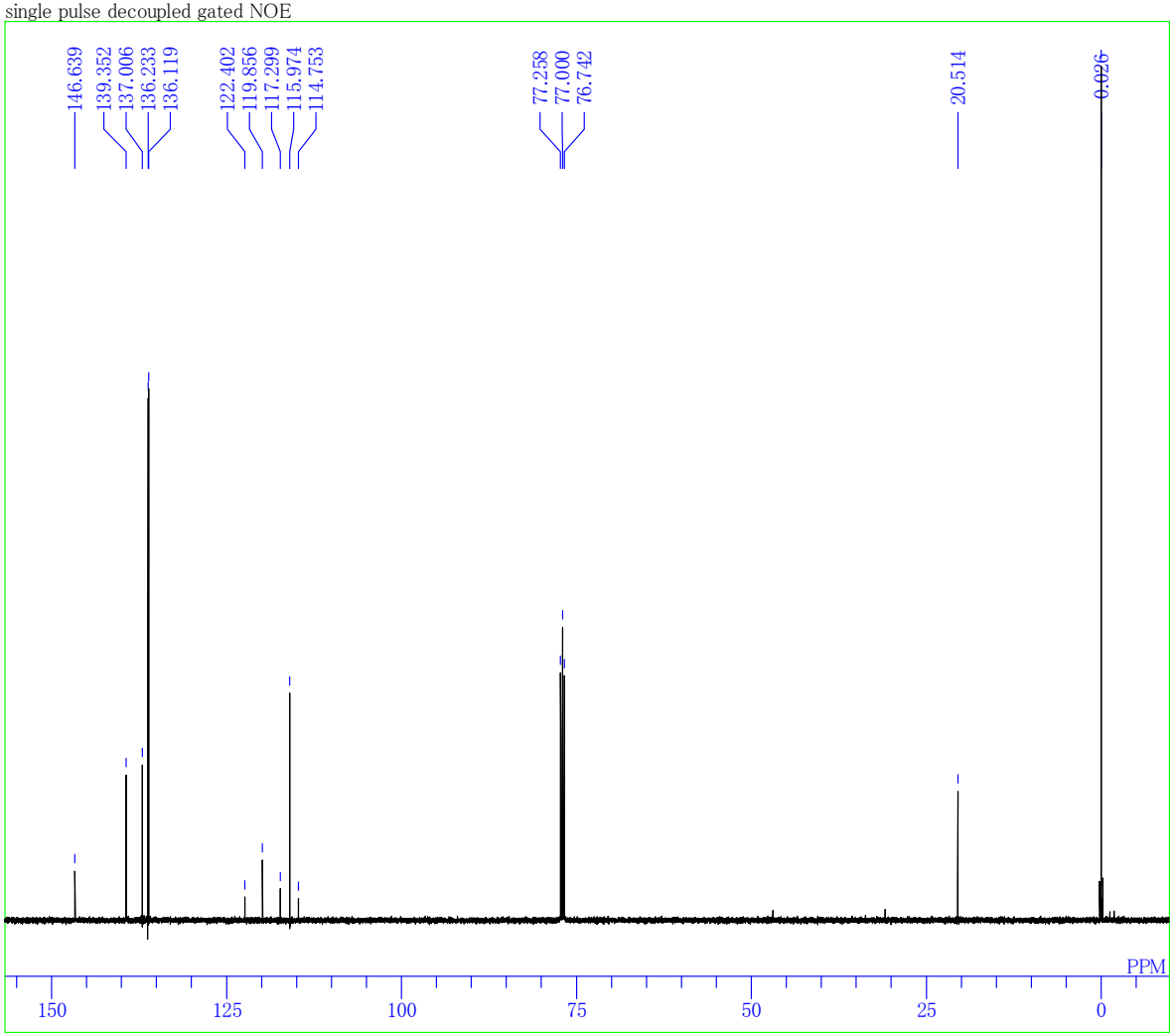
DFILE	C:¥Documents and Settings¥Owner¥My D
COMNT	Yti-35-Fr40
DATIM	Tue Jun 05 21:02:36 2007
OBNUC	1H
EXMOD	SINGL
OBFRQ	499.10 MHz
OBSET	0.00 KHz
OBFIN	125250.00 Hz
POINT	16384
FREQU	9980.04 Hz
SCANS	8
ACQTM	1.6417 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	-204.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	23



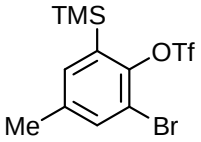
1c ($CDCl_3$)

Table 2, Entry 3

single pulse decoupled gated NOE

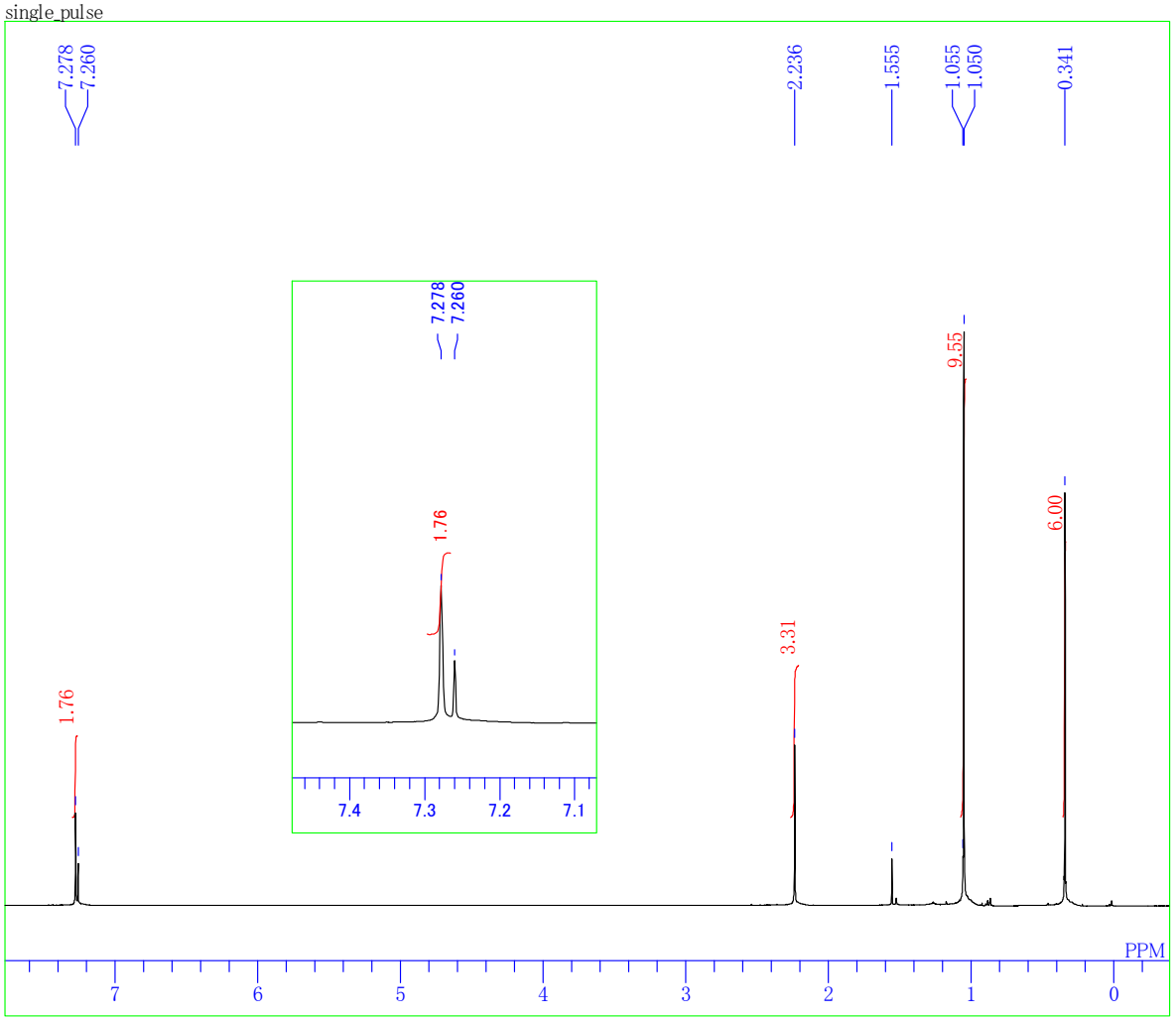


DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single pulse decoupled gated NOE
DATIM	20-12-2007 11:42:14
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32768
FREQU	39308.18 Hz
SCANS	446
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	18.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

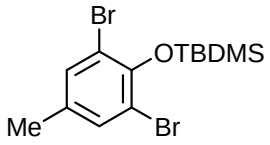


1c ($CDCl_3$)

Table 2, Entry 3

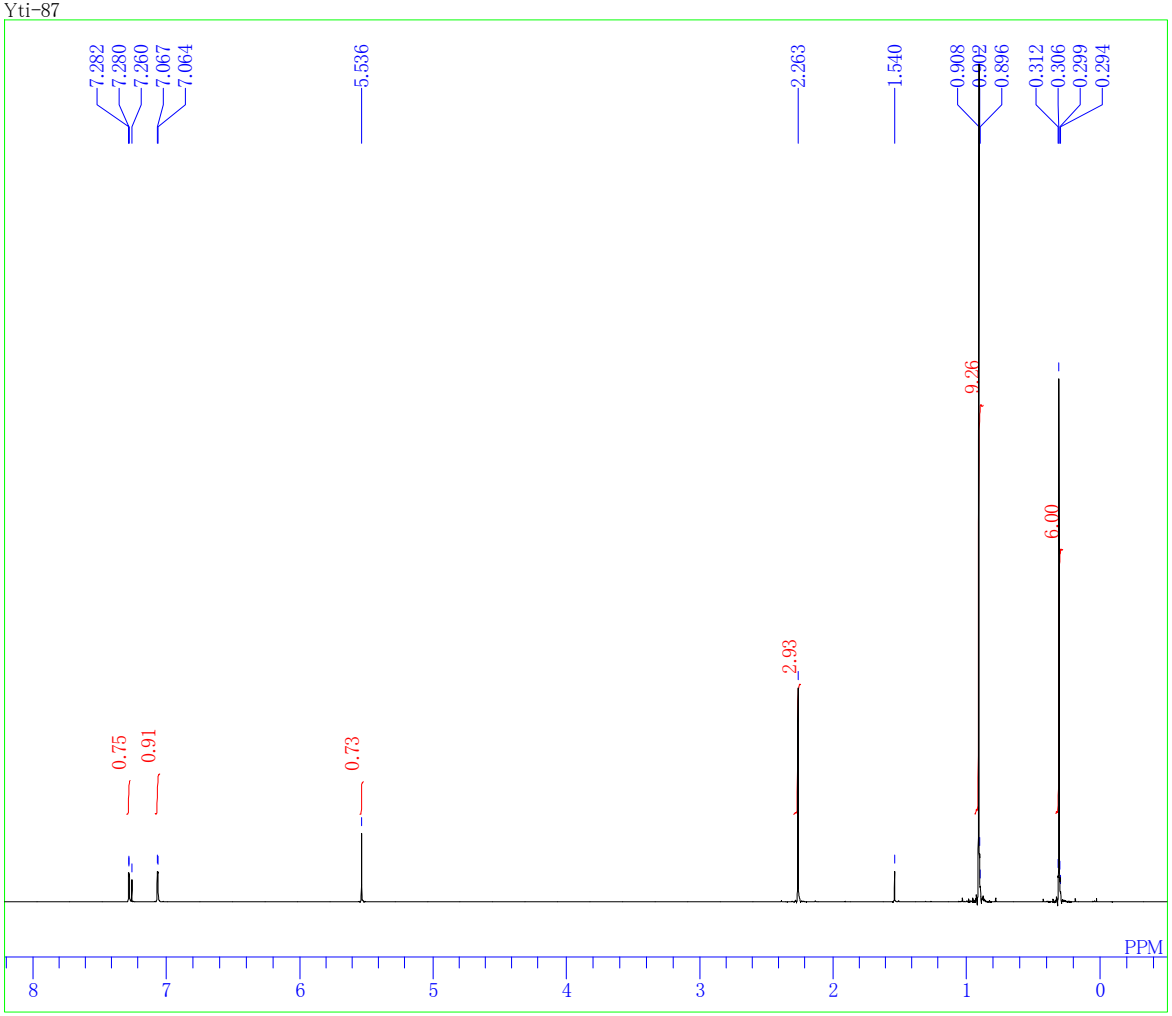


DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single_pulse
DATIM	17-07-2007 21:27:00
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13106
FREQU	7507.39 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	6.00 usec
IRNUC	1H
CTEMP	21.0 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	50

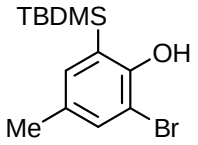


S2d ($CDCl_3$)
Starting Material of **1d**

Yti-87

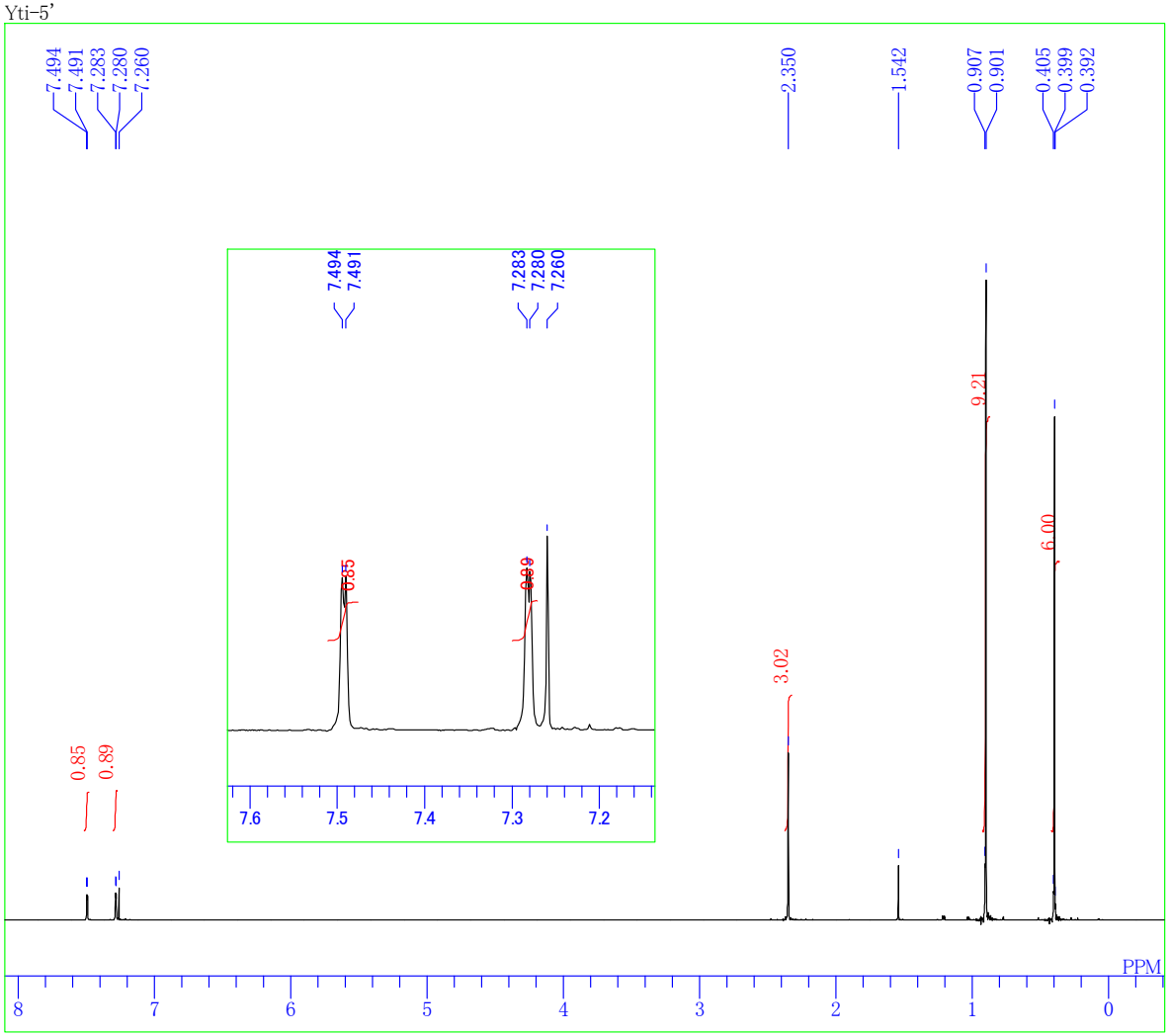


DATIM	Thu Jul 19 18:05:57 2007
EXMOD	SINGL
OBNUC	1H
OFR	499.10 MHz
OBSET	0.00 KHz
OBFIN	125250.00 Hz
OBATN	511
PW1	5.50 usec
PW2	26.00 usec
POINT	16384
SPO	16384
SCANS	8
FREQU	9980.04 Hz
PD	2.0000 sec
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	125250.00 Hz
IRATN	511
EXREF	7.26 ppm
TMSP	6326

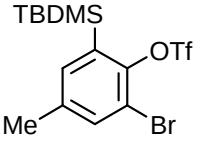


S3d ($CDCl_3$)

Starting Material of **1d**



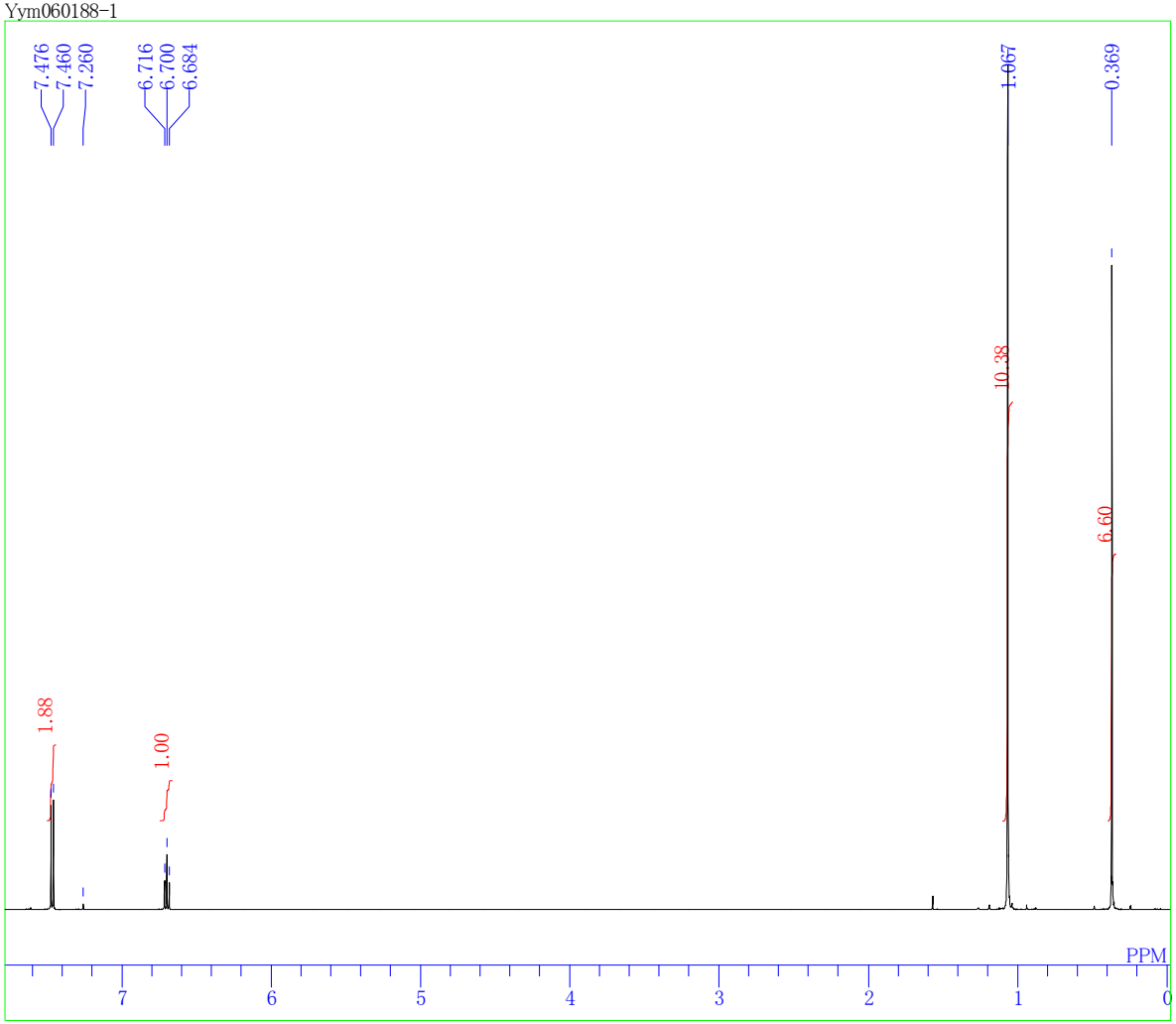
DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	Yti-5'
DATIM	Fri Apr 27 10:43:41 2007
OBNUC	1H
EXMOD	SINGL
OBFRQ	499.10 MHz
OBSET	0.00 KHz
OBFIN	125250.00 Hz
POINT	16384
FREQU	9980.04 Hz
SCANS	8
ACQTM	1.6417 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	-204.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	20



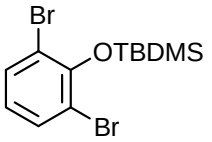
1d ($CDCl_3$)

Table 1, entry 4
Table 2, entries 1, 2, 4–8

Yym060188-1



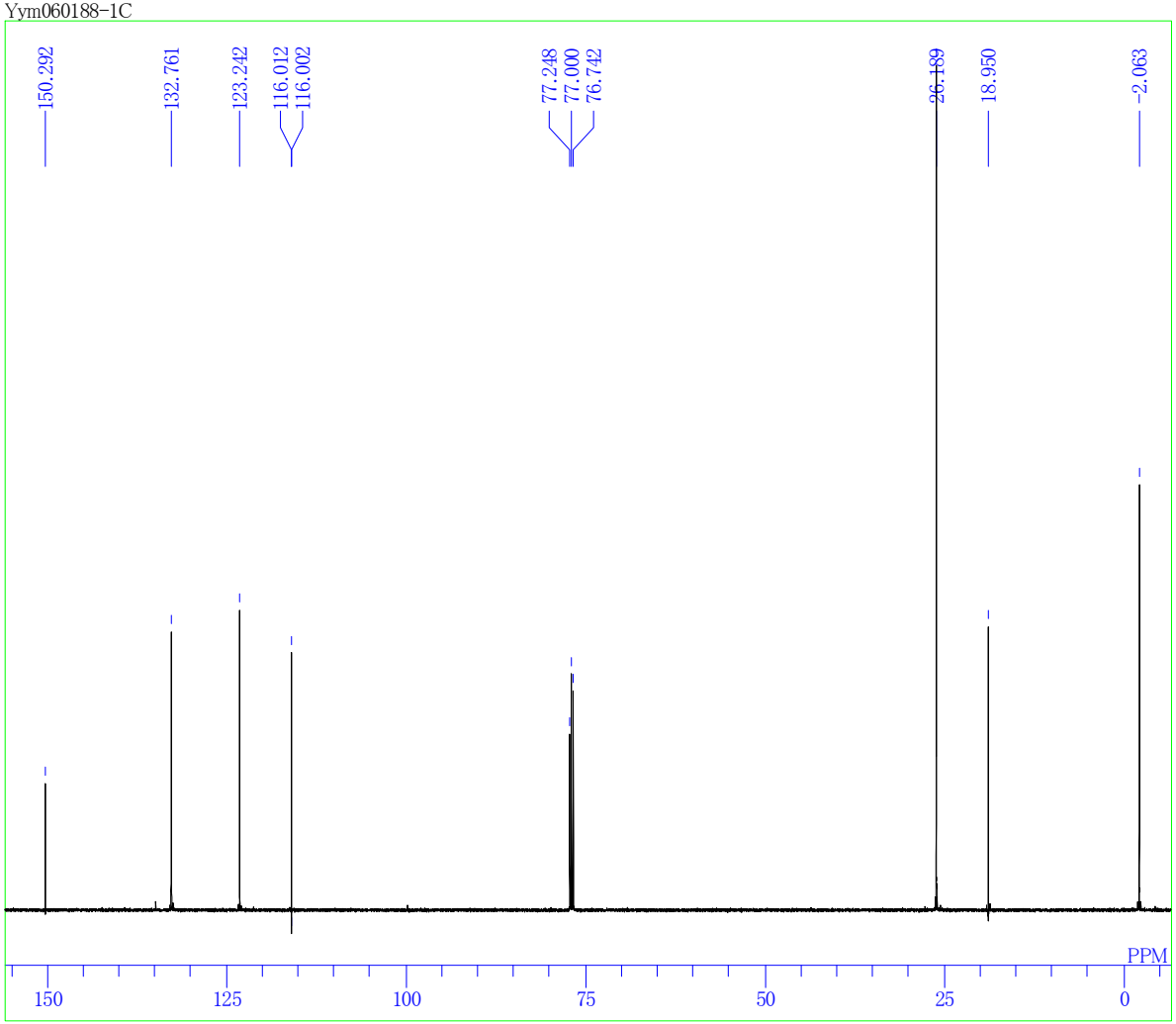
DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	Yym060188-1
DATIM	22-12-2007 23:50:32
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.39 Hz
SCANS	8
ACQTM	1.7459 sec
PD	1.5000 sec
PW1	6.00 usec
IRNUC	1H
CTEMP	17.1 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	36



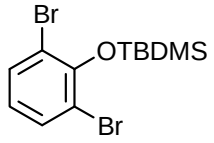
S2e ($CDCl_3$)

Starting Material of **1e**

Ym060188-1C

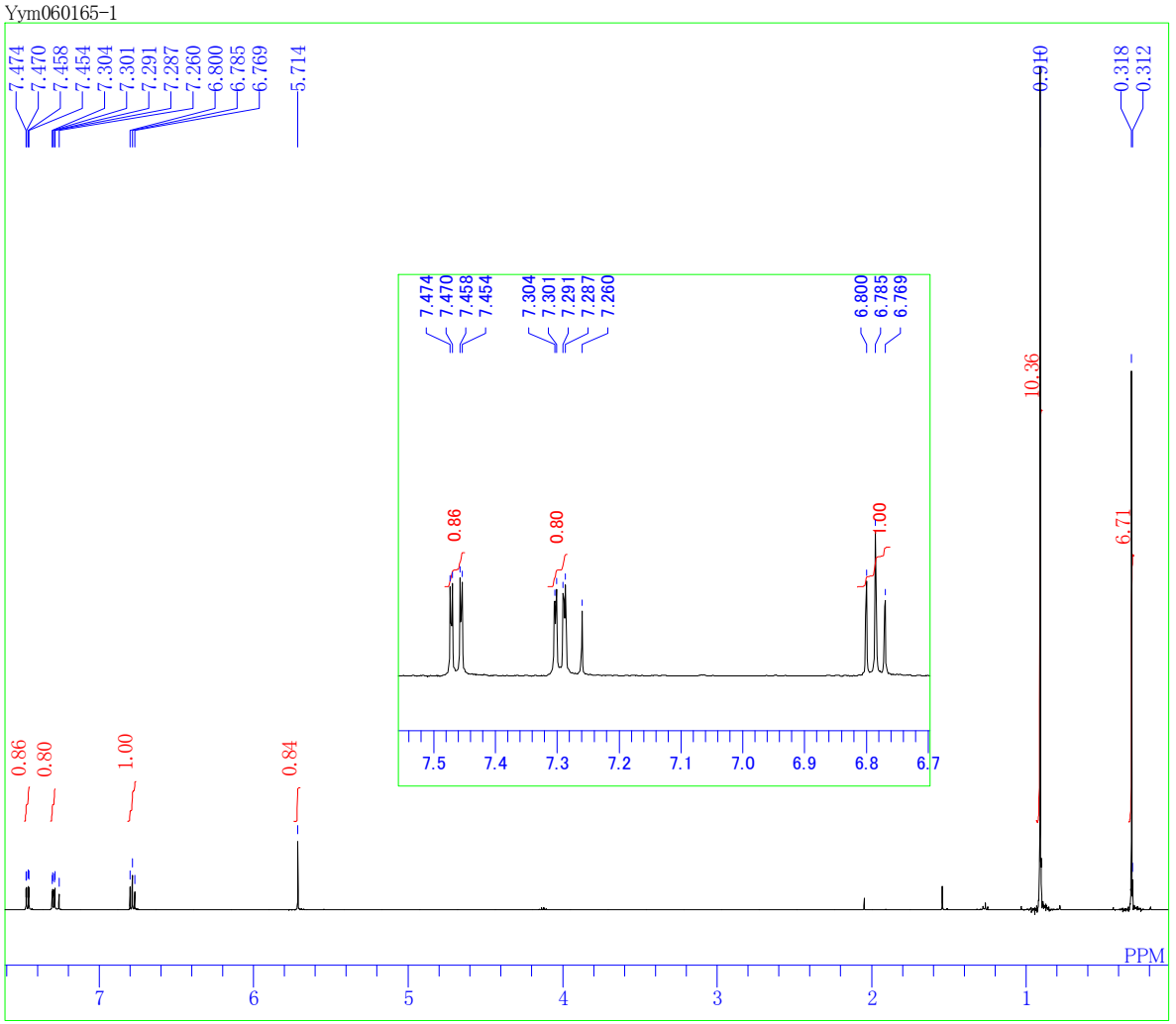


DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	Ym060188-1C
DATIM	23-12-2007 00:57:33
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.06 Hz
SCANS	1637
ACQTM	0.8336 sec
PD	1.5000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	17.5 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

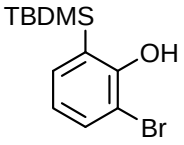


S2e ($CDCl_3$)

Starting Material of **1e**



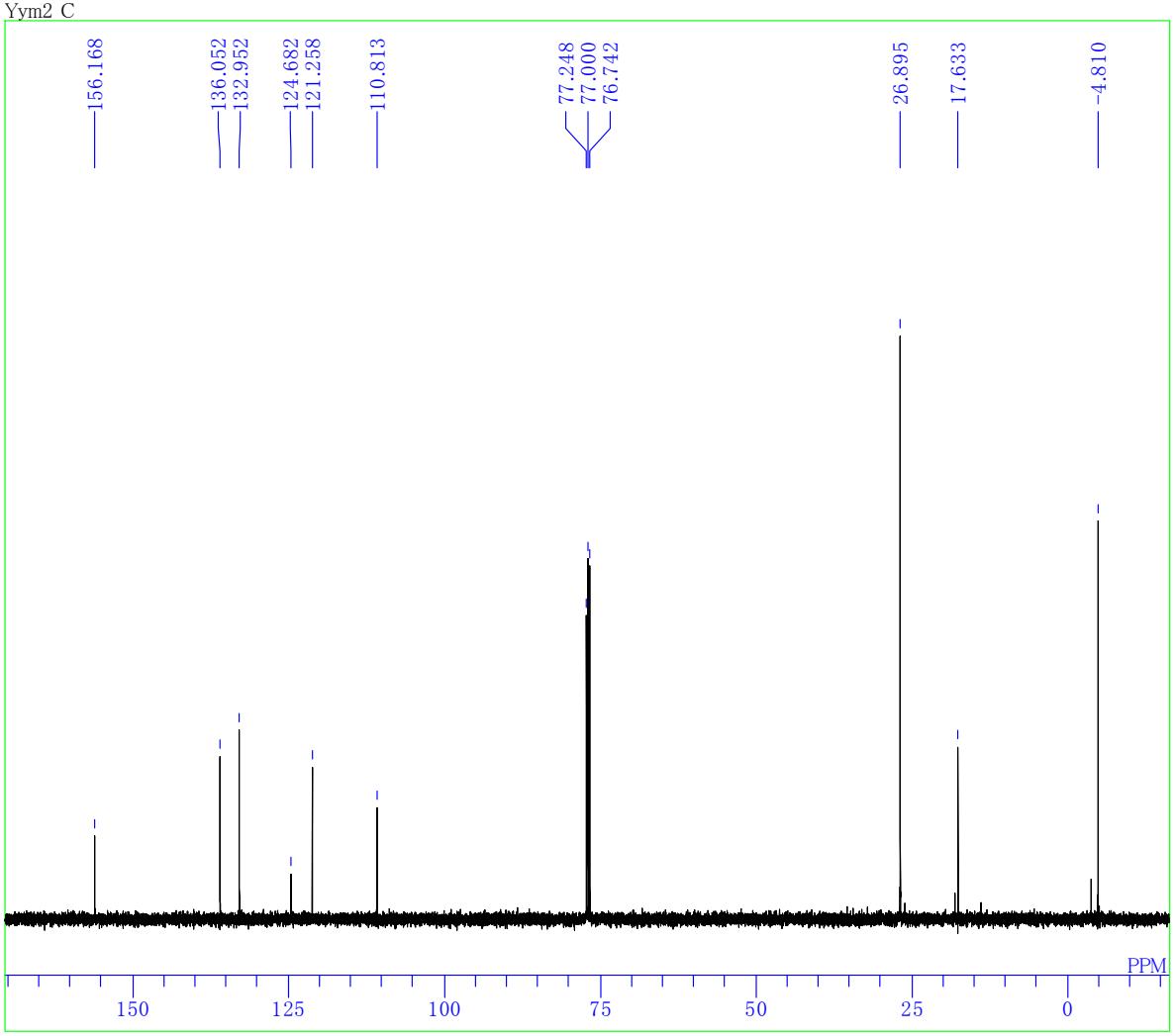
DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	Yym060165-1
DATIM	Fri Sep 07 14:47:57 2007
OBNUC	1H
EXMOD	SINGL
OBFRQ	499.10 MHz
OBSET	0.00 KHz
OBFIN	125250.00 Hz
POINT	16384
FREQU	9980.04 Hz
SCANS	8
ACQTM	1.6417 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	-204.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	18



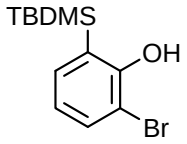
S3e ($CDCl_3$)

Starting Material of **1e**

Yym2 C

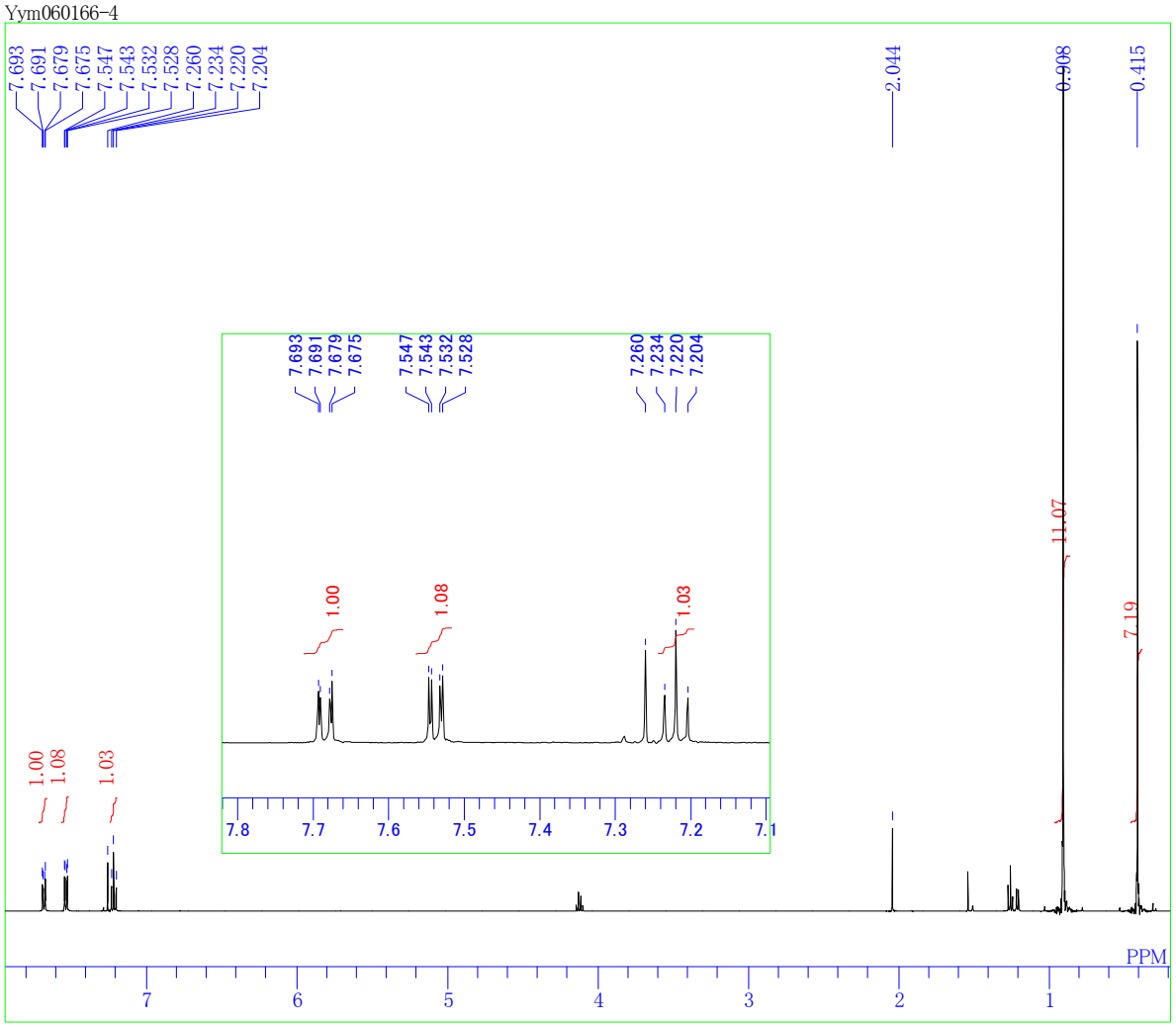


DFILE	C:¥Documents and Settings¥Owner
COMNT	Yym2 C
DATIM	29-12-2007 19:36:17
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.06 Hz
SCANS	840
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	18.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

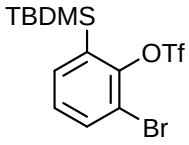


S3e ($CDCl_3$)

Starting Material of **1e**



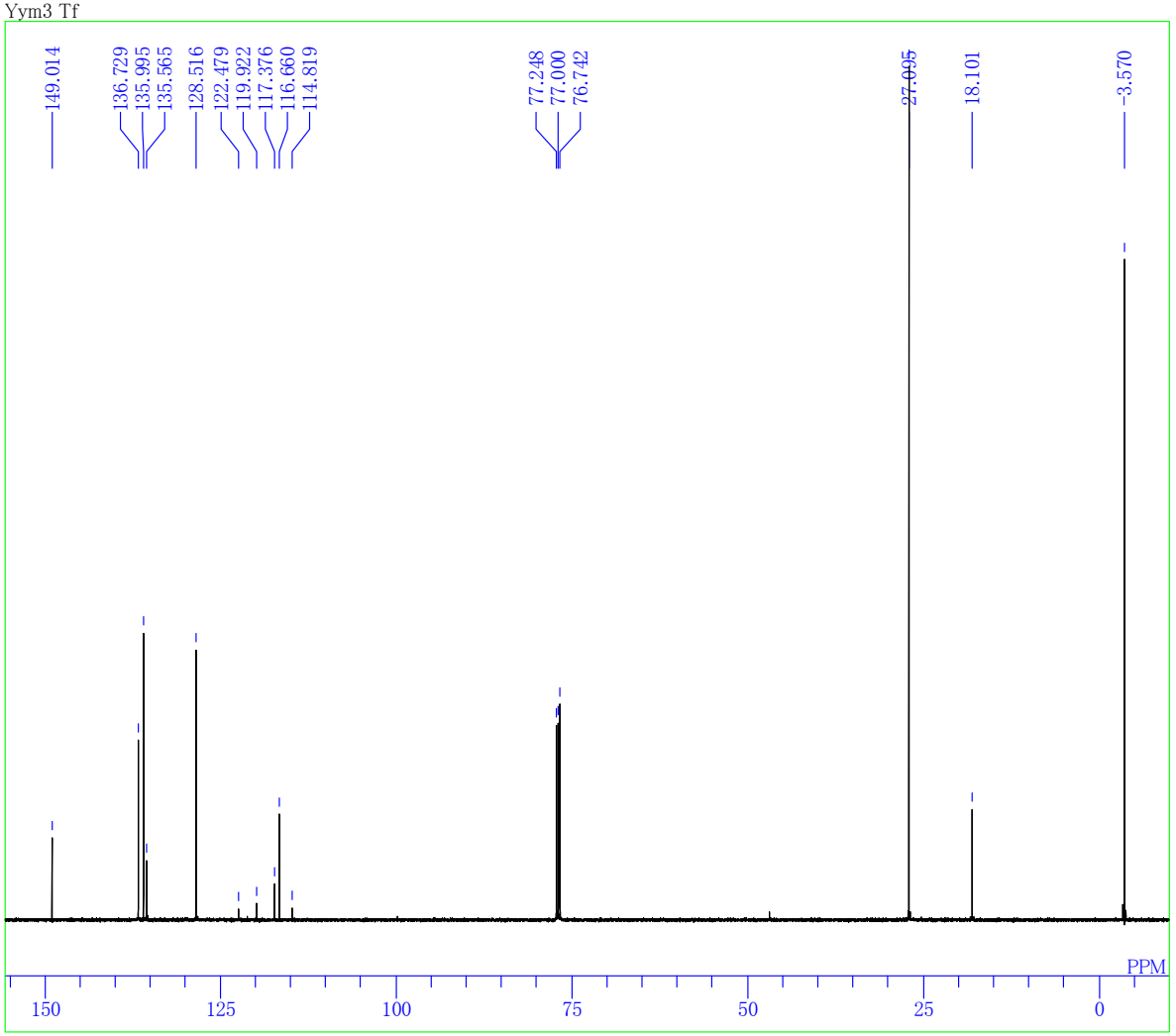
DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	Ym060166-4
DATIM	Sun Sep 09 17:35:23 2007
OBNUC	1H
EXMOD	SINGL
OBFRQ	499.10 MHz
OBSET	0.00 KHz
OBFIN	125250.00 Hz
POINT	16384
FREQU	9980.04 Hz
SCANS	8
ACQTM	1.6417 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	-204.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	20



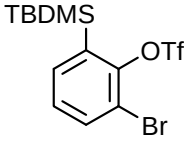
1e ($CDCl_3$)

Table 2, entries 9 and 10

Yym3 Tf

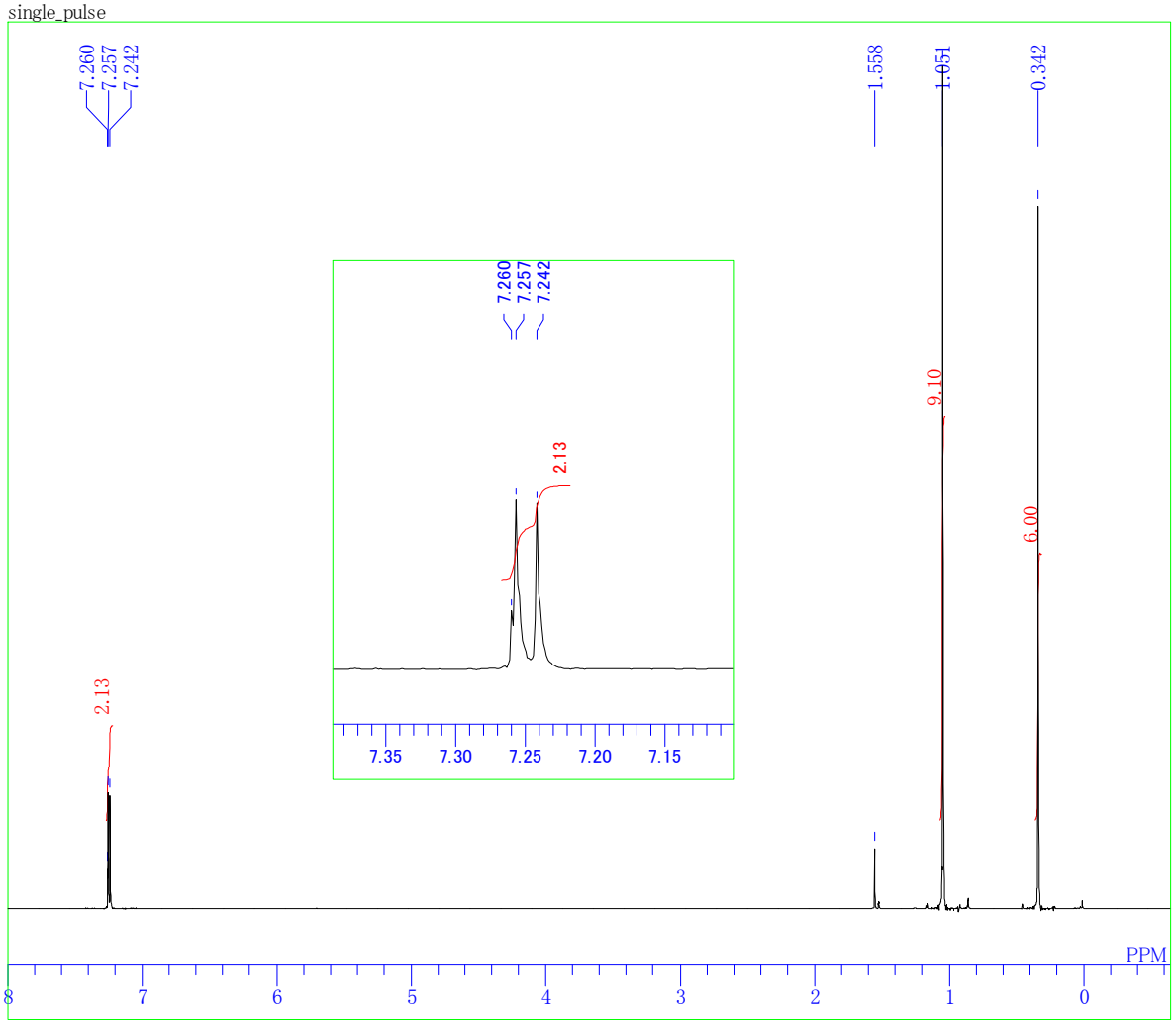


DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	Yym3 Tf
DATIM	22-12-2007 22:15:21
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.06 Hz
SCANS	1459
ACQTM	0.8336 sec
PD	1.5000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	17.7 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

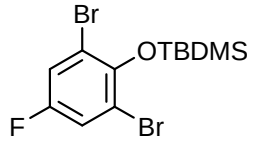


1e ($CDCl_3$)

Table 2, entries 9 and 10



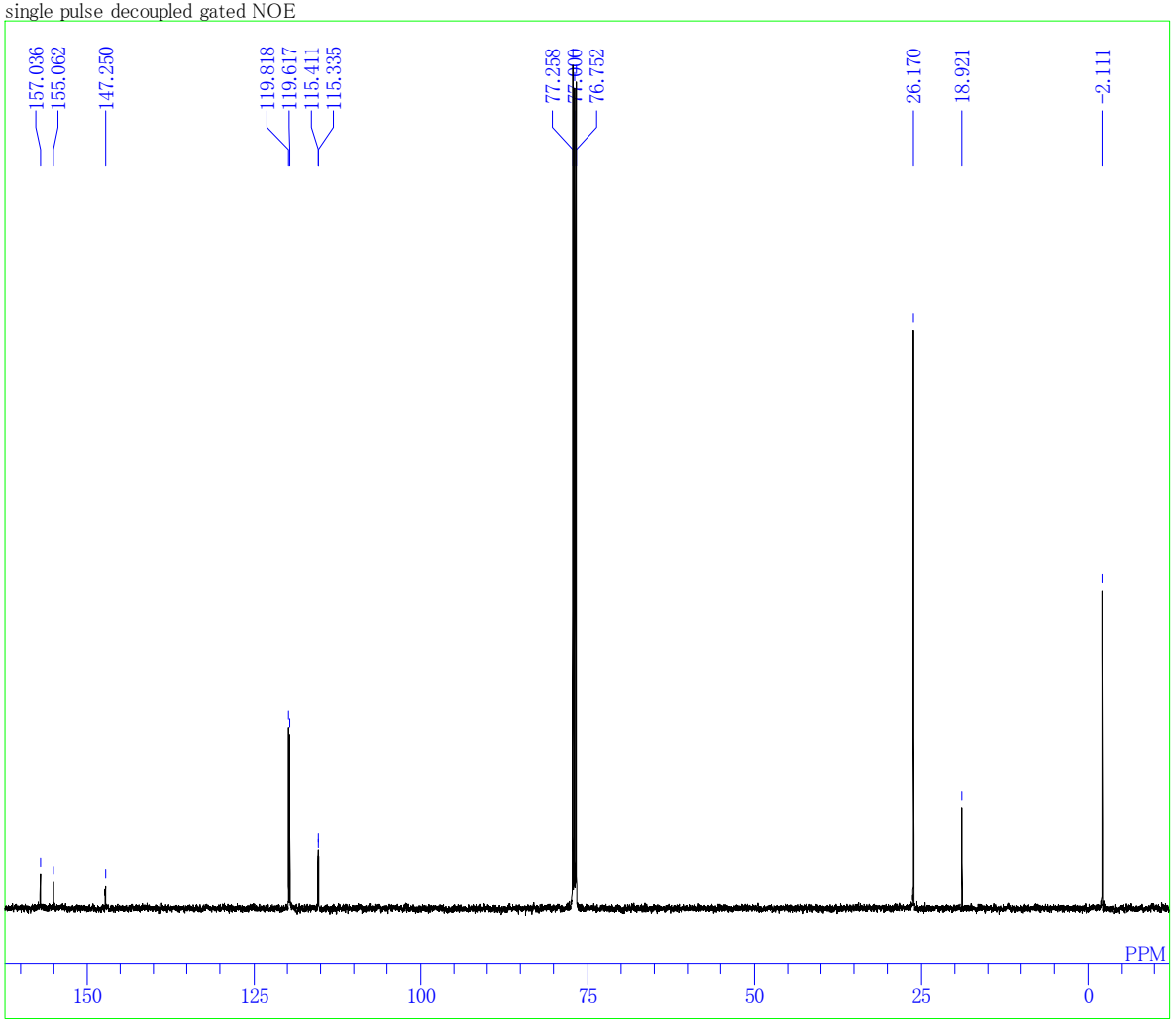
DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single_pulse
DATIM	06-09-2007 20:58:59
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	20480
FREQU	11730.66 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	6.00 usec
IRNUC	1H
CTEMP	20.6 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	48



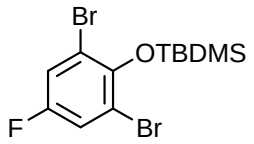
S2f ($CDCl_3$)

Starting Material of **1f**

single pulse decoupled gated NOE



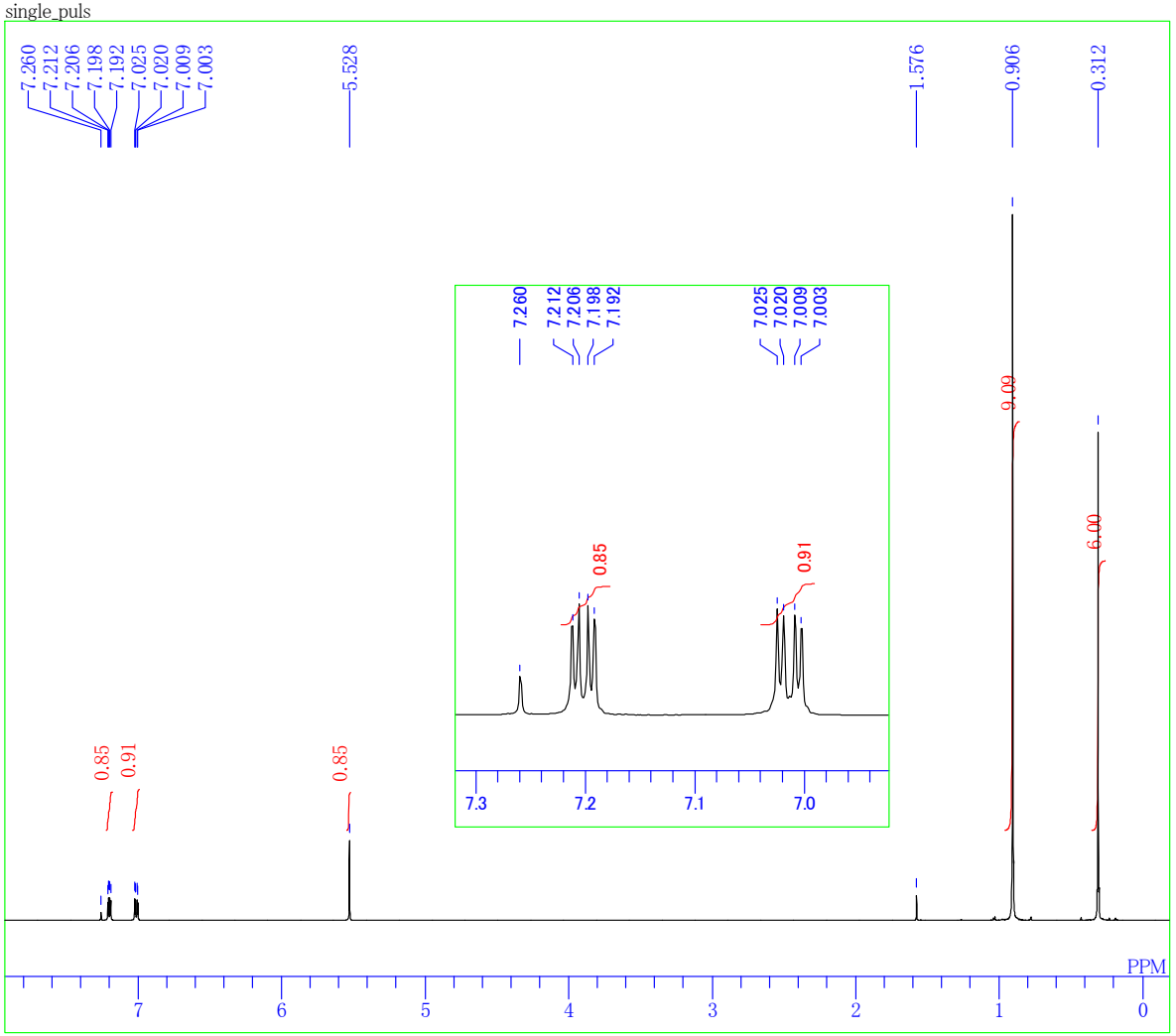
DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single pulse decoupled gated NOE
DATIM	06-09-2007 22:03:02
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	40961
FREQU	49135.97 Hz
SCANS	1265
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	20.9 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60



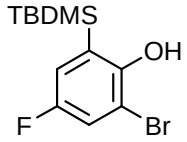
S2f ($CDCl_3$)

Starting Material of **1f**

single_puls



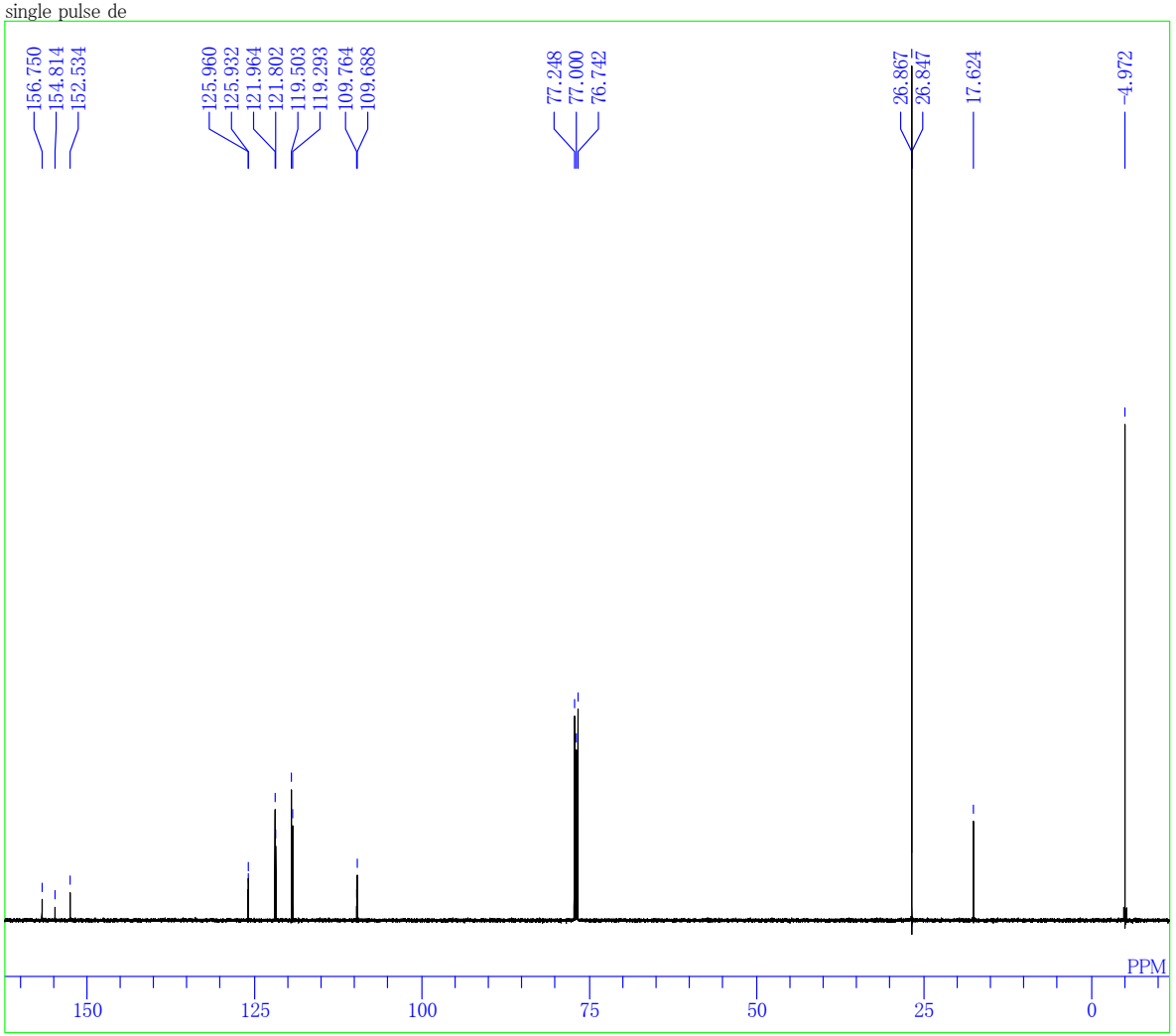
DFILE	C:¥Documents and Settings¥Owner¥デ
COMNT	single_puls
DATIM	07-09-2007 22:46:31
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16384
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	6.00 usec
IRNUC	1H
CTEMP	21.5 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	38



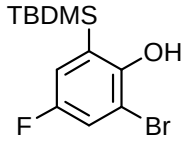
S3f ($CDCl_3$)

Starting Material of **1f**

single pulse de

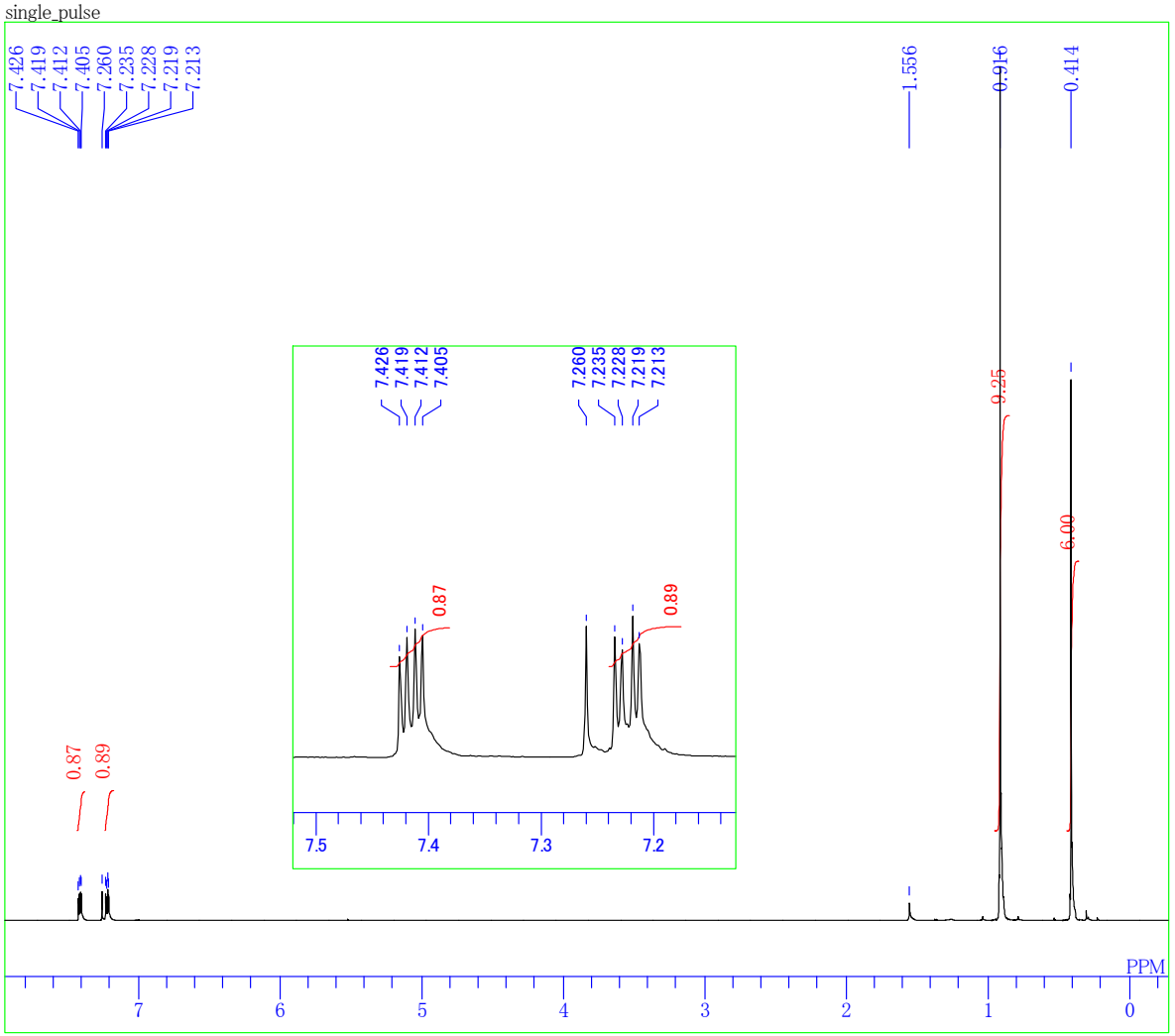


DFILE	C:¥Documents and Settings¥Owner¥デ
COMNT	single pulse de
DATIM	07-09-2007 22:42:59
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32768
FREQU	39308.18 Hz
SCANS	954
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	21.8 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

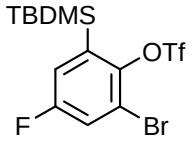


S3f ($CDCl_3$)

Starting Material of **1f**



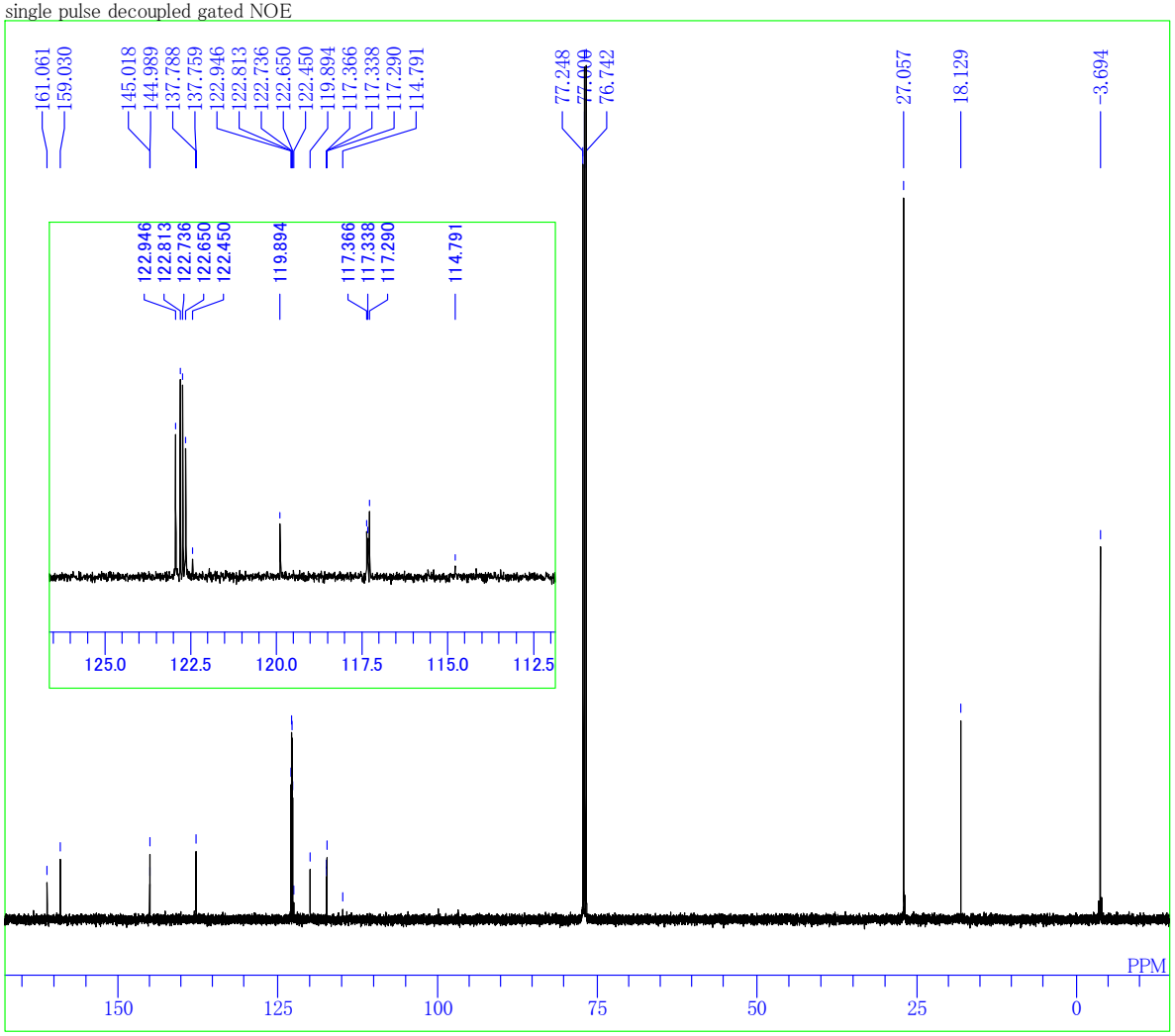
DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single_pulse
DATIM	11-09-2007 05:52:16
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	20480
FREQU	11730.66 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	6.00 usec
IRNUC	1H
CTEMP	21.4 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	46



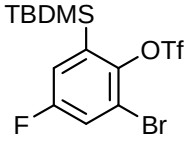
1f ($CDCl_3$)

Table 2, Entries 11–13

single pulse decoupled gated NOE



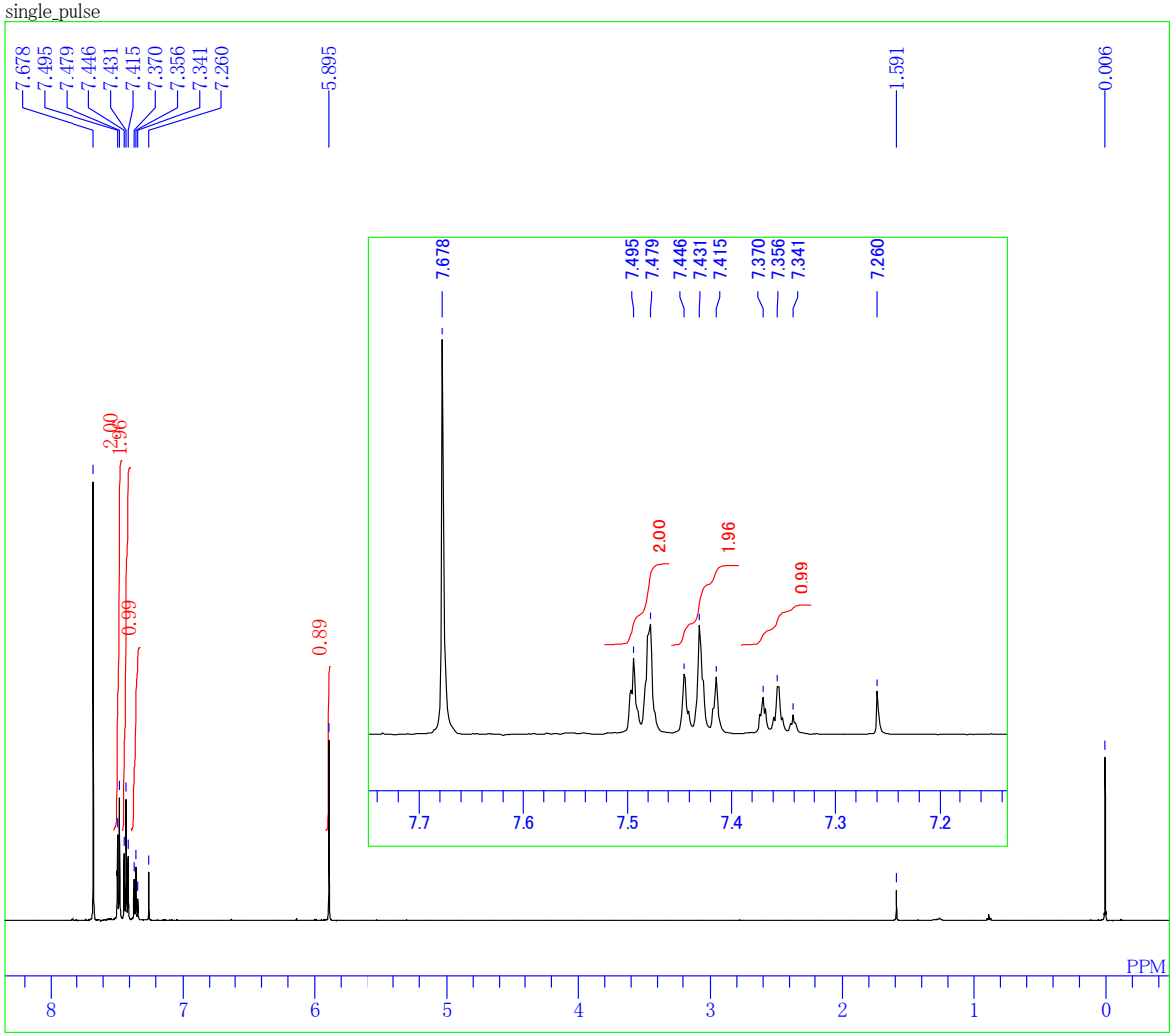
DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single pulse decoupled gated NOE
DATIM	11-09-2007 05:48:48
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32768
FREQU	39308.18 Hz
SCANS	2190
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	21.8 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60



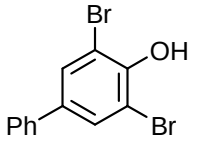
1f ($CDCl_3$)

Table 2, Entries 11–13

single_pulse



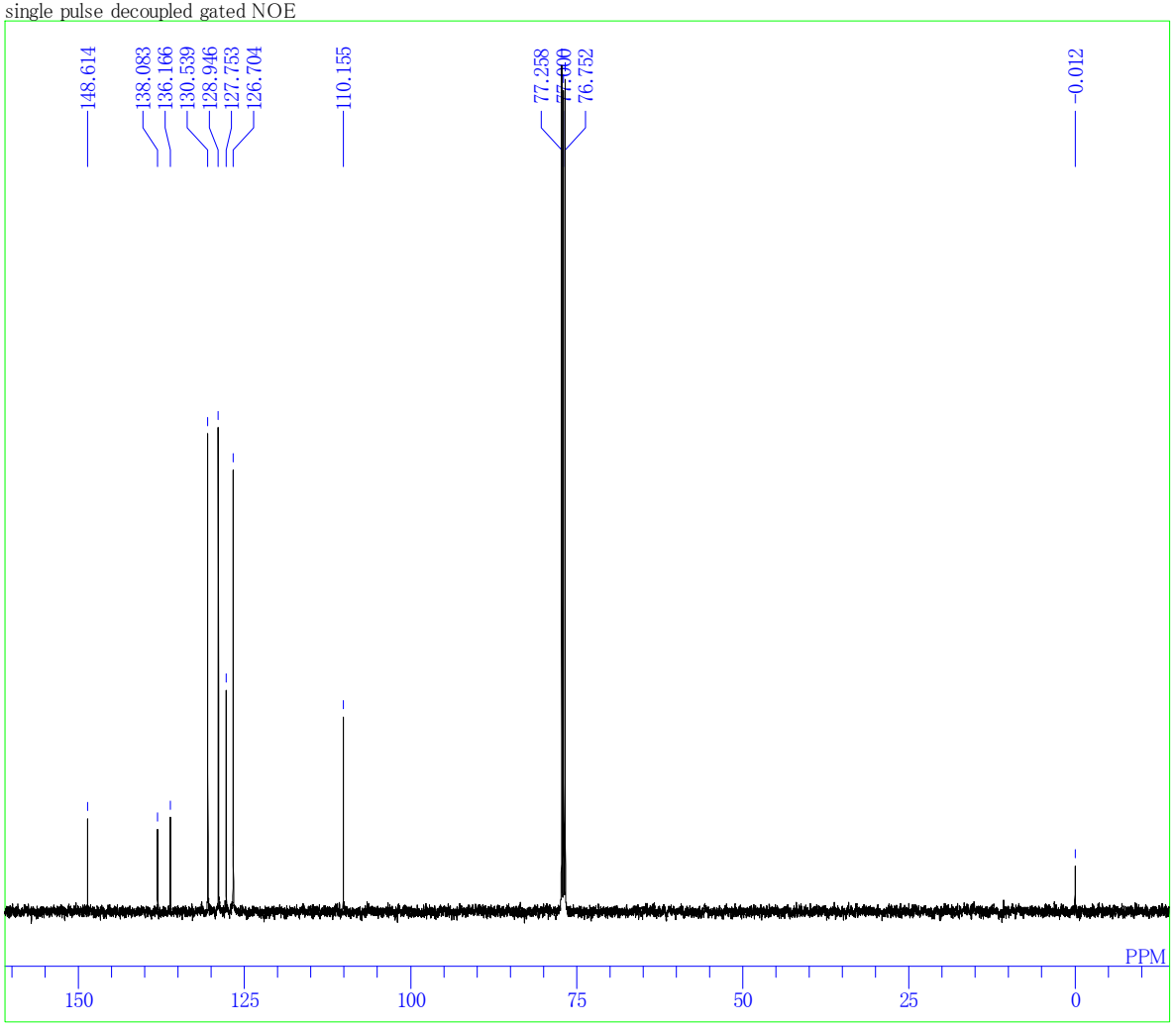
DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single_pulse
DATIM	19-01-2008 09:57:20
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16384
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	6.00 usec
IRNUC	1H
CTEMP	17.0 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	48



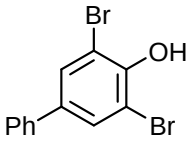
S1g ($CDCl_3$)

Starting Material of **1g**

single pulse decoupled gated NOE



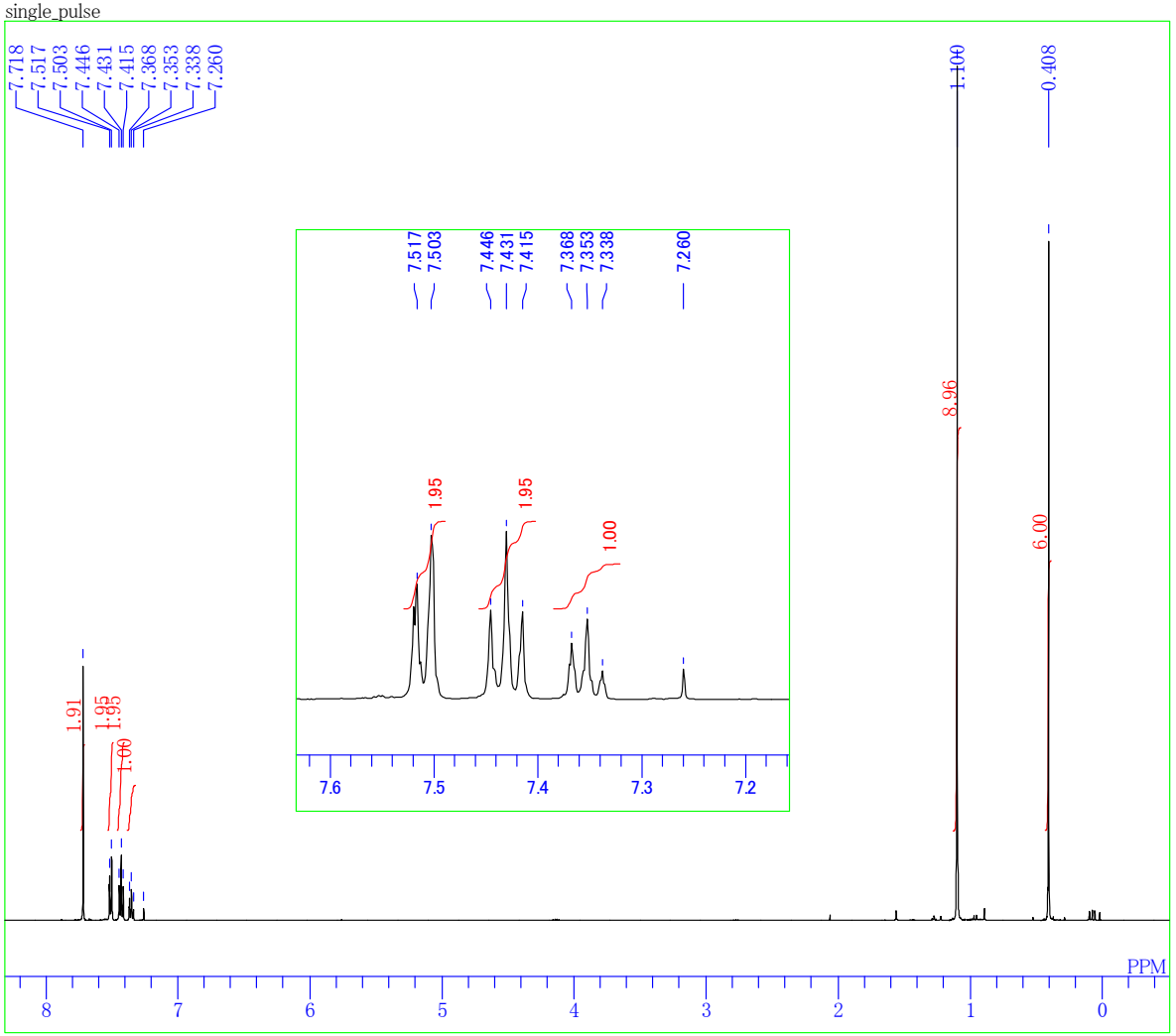
DFILE	F:¥Ymt070109-1 13C.3
COMNT	single pulse decoupled gated NOE
DATIM	19-01-2008 10:15:03
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	40961
FREQU	49135.97 Hz
SCANS	321
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	17.5 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60



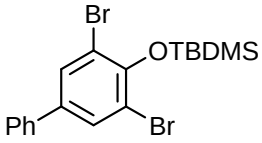
S1g ($CDCl_3$)

Starting Material of **1g**

single_pulse



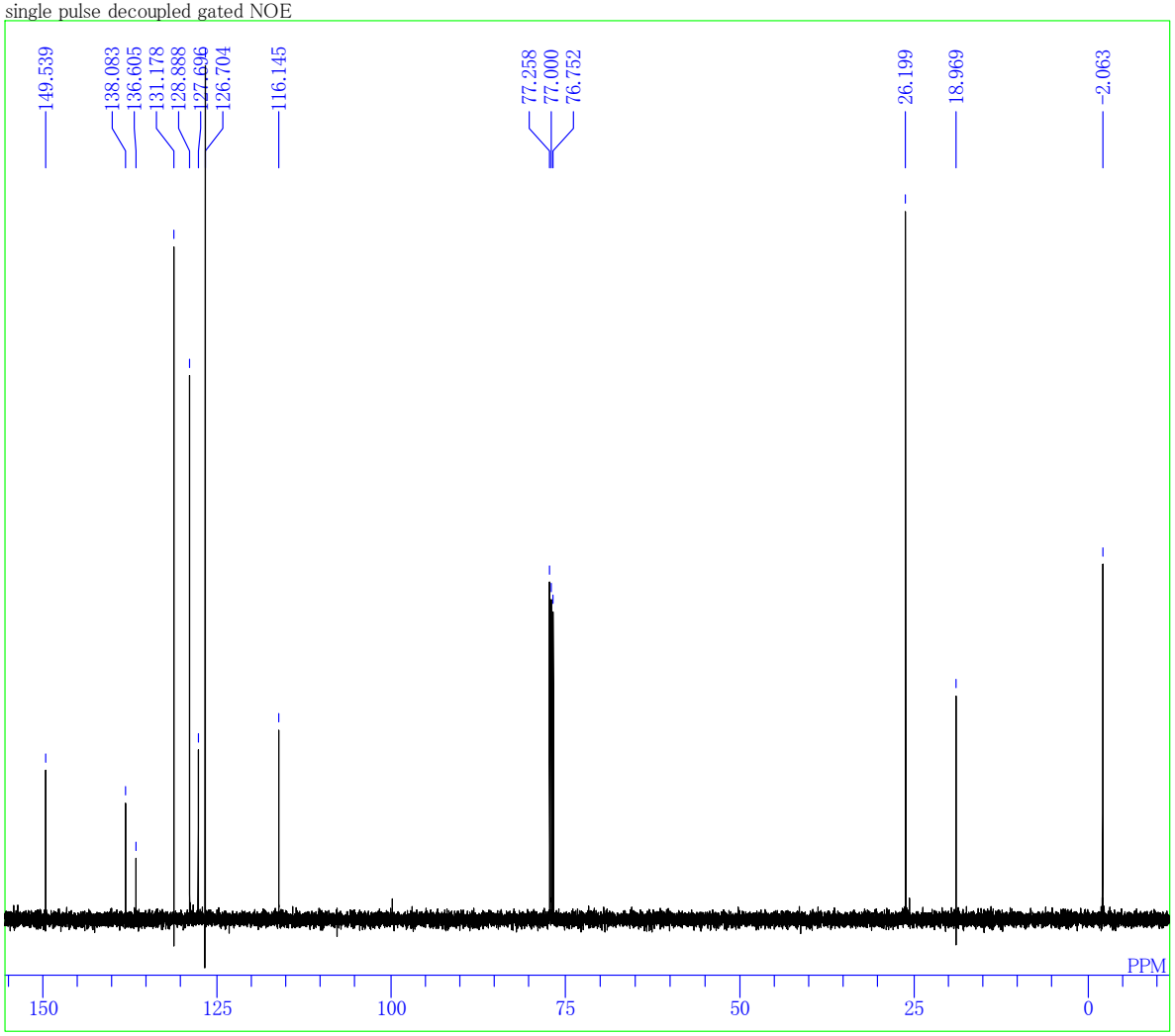
DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single_pulse
DATIM	18-12-2007 12:42:00
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.39 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	6.00 usec
IRNUC	1H
CTEMP	17.3 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	36



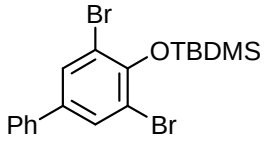
S2g ($CDCl_3$)

Starting Material of **1g**

single pulse decoupled gated NOE

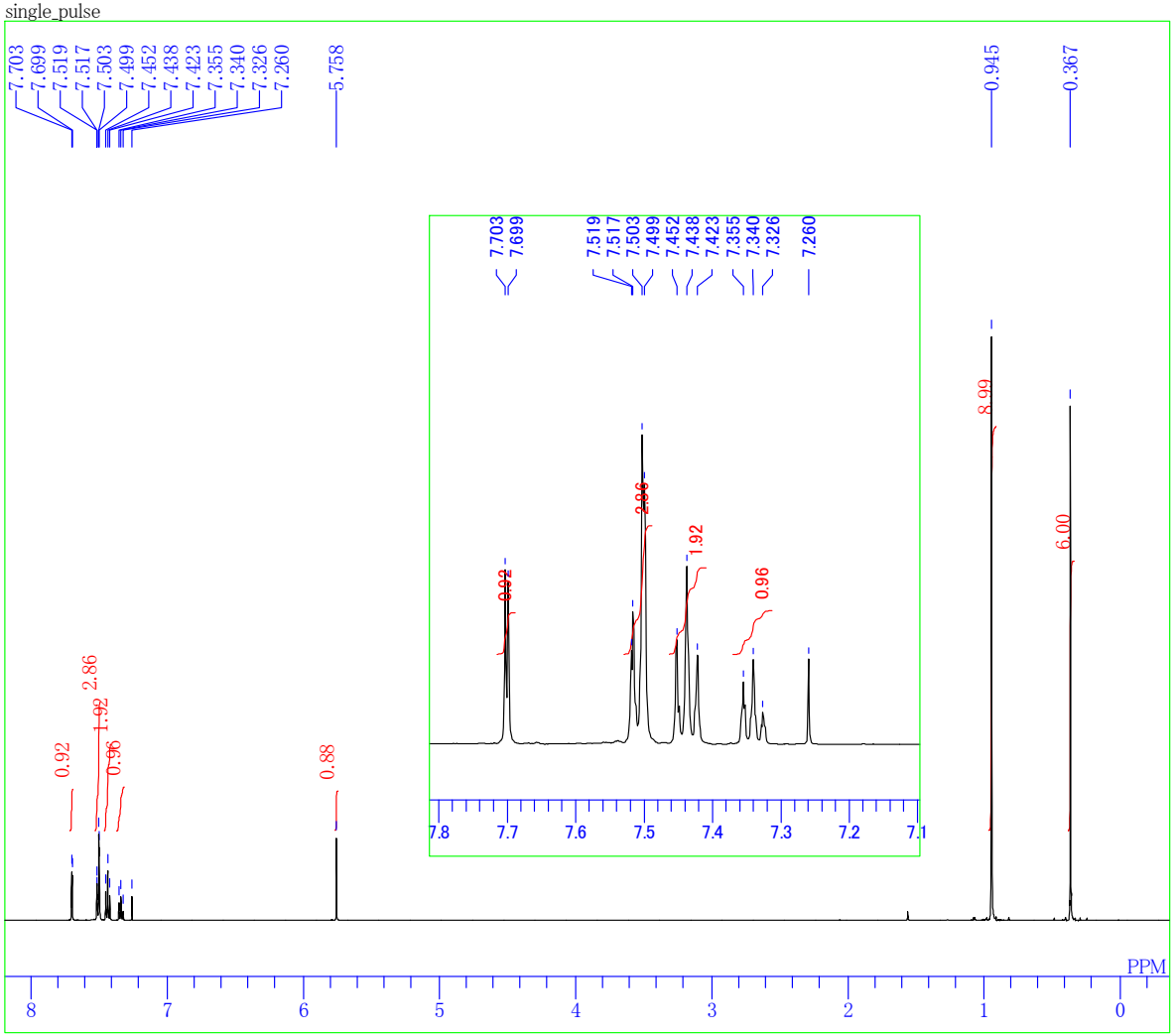


DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single pulse decoupled gated NOE
DATIM	18-12-2007 12:48:20
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.06 Hz
SCANS	63
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	17.8 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

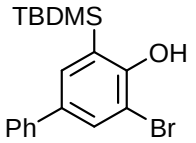


S2g ($CDCl_3$)

Starting Material of **1g**



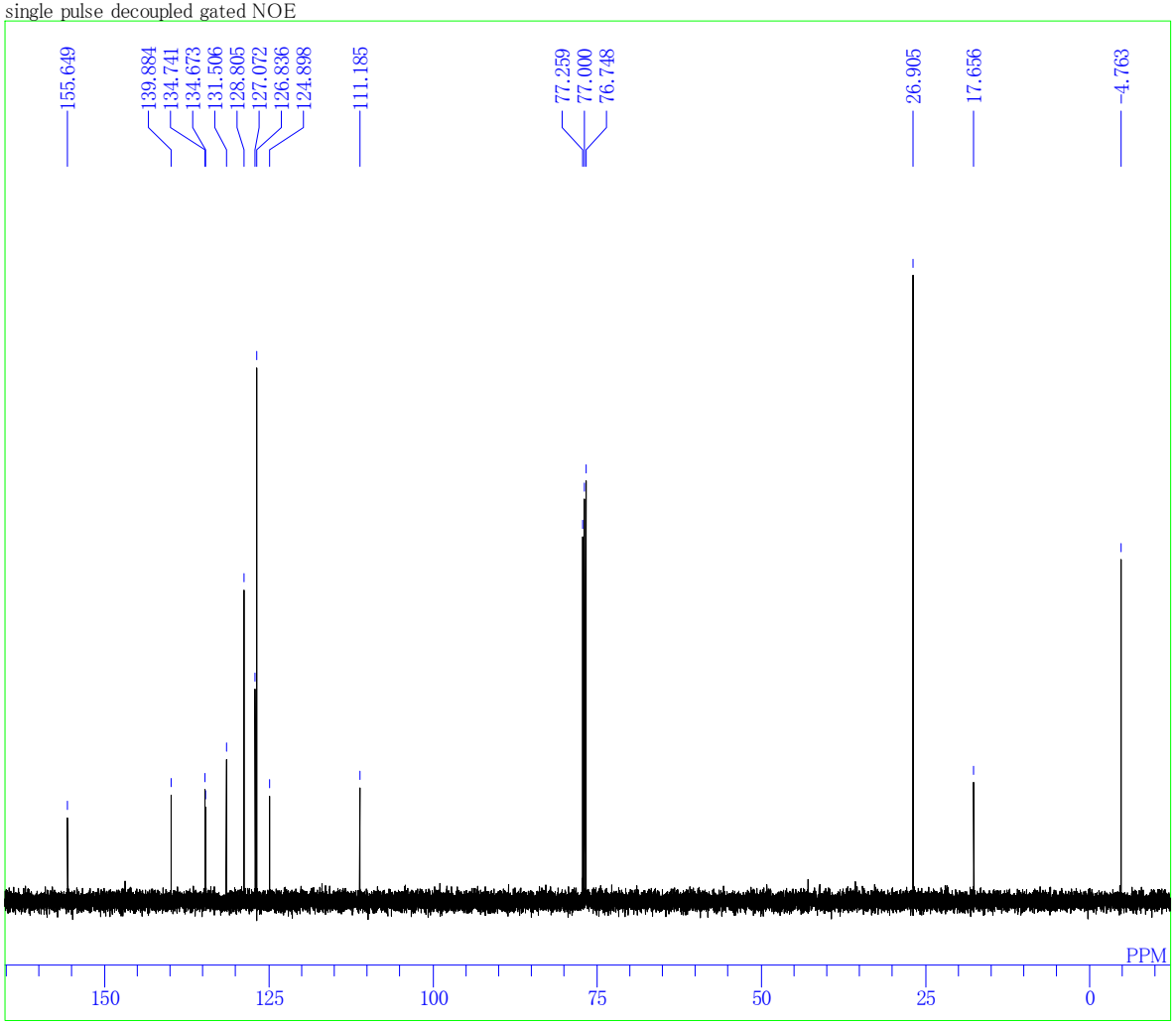
DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single_pulse
DATIM	25-12-2007 19:58:59
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.39 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	6.00 usec
IRNUC	1H
CTEMP	17.2 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	42



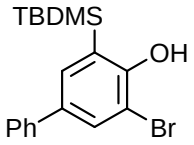
S3g ($CDCl_3$)

Starting Material of **1g**

single pulse decoupled gated NOE

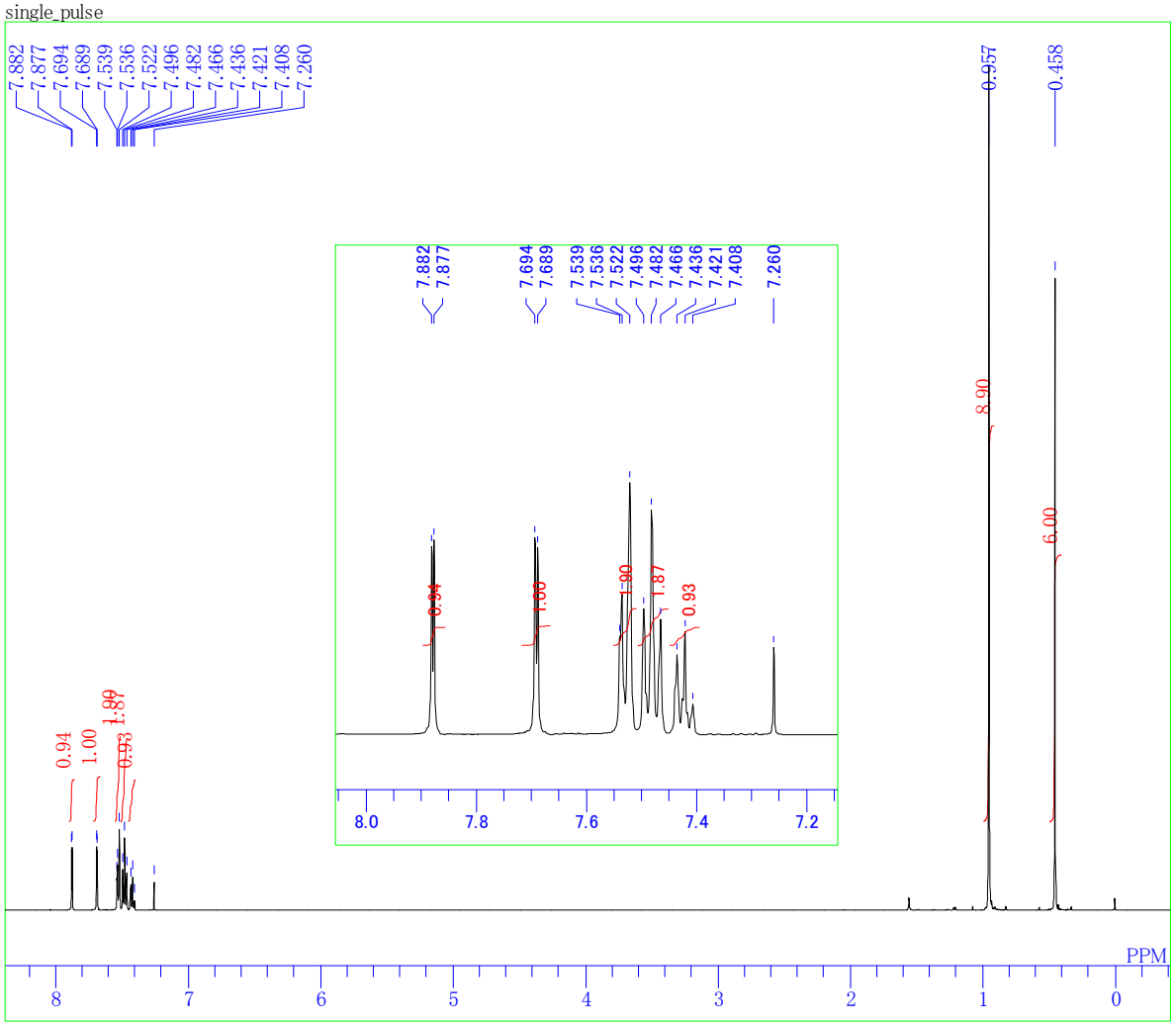


DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single pulse decoupled gated NOE
DATIM	25-12-2007 20:04:06
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32768
FREQU	31446.54 Hz
SCANS	51
ACQTM	1.0420 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	17.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	76

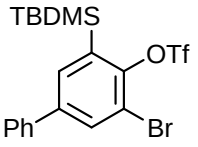


S3g ($CDCl_3$)

Starting Material of **1g**



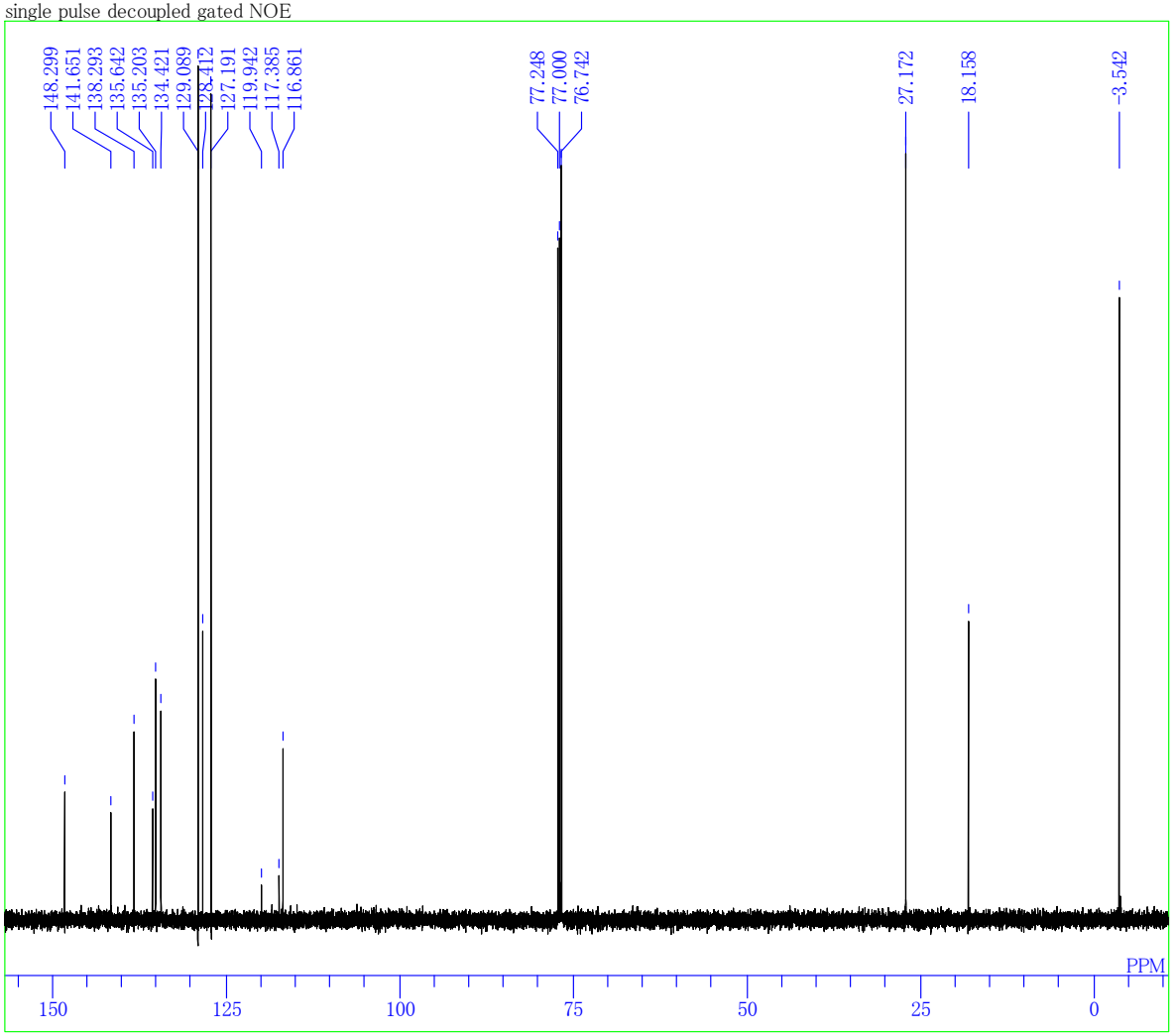
DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single_pulse
DATIM	18-12-2007 13:07:40
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16384
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	6.00 usec
IRNUC	1H
CTEMP	17.4 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	42



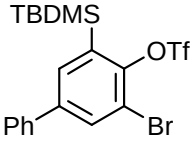
1g ($CDCl_3$)

Table 2, entries 14 and 15

single pulse decoupled gated NOE



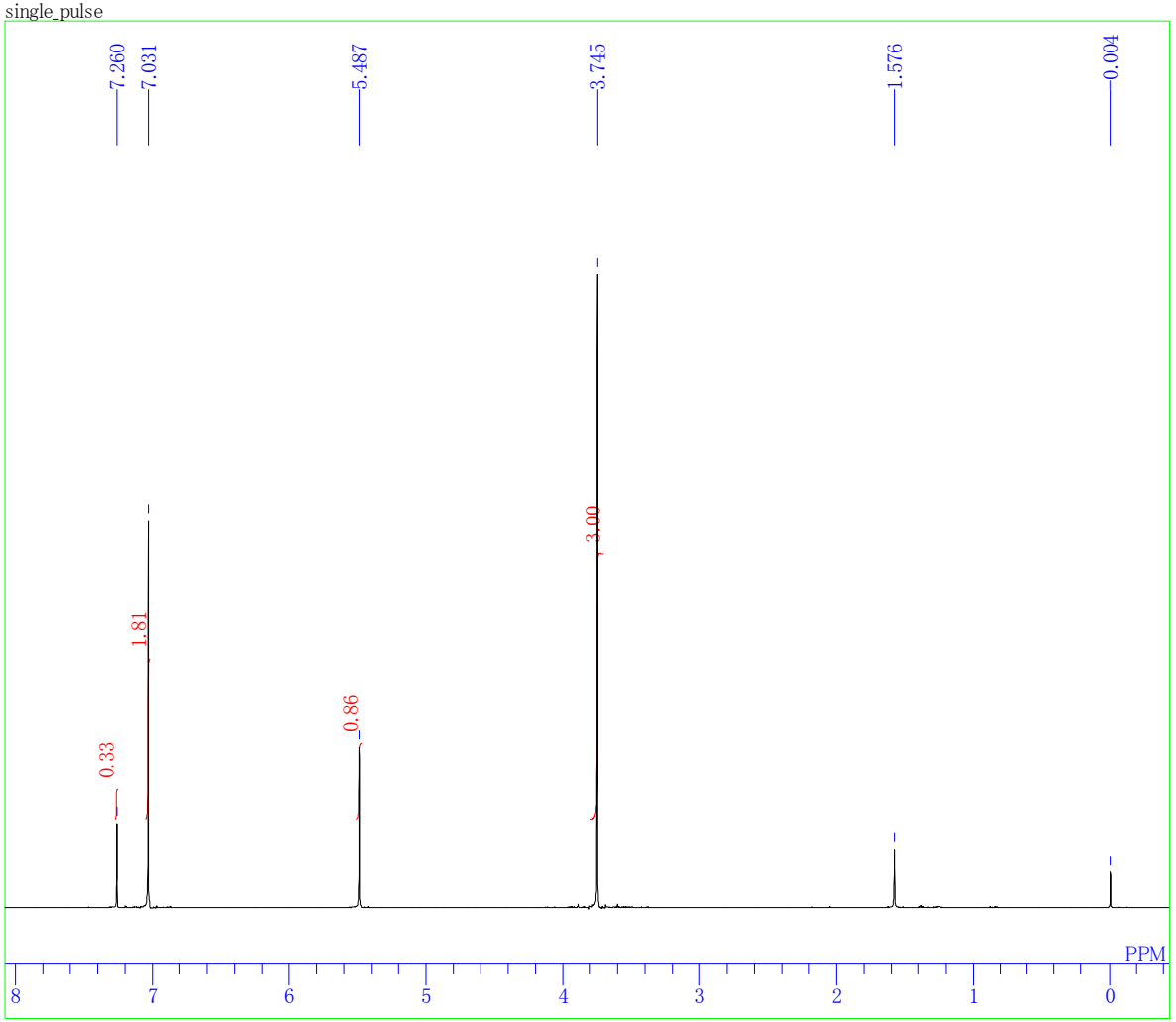
DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single pulse decoupled gated NOE
DATIM	18-12-2007 13:23:09
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.06 Hz
SCANS	263
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	17.9 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60



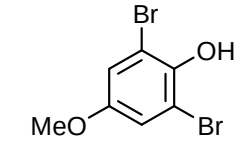
1g ($CDCl_3$)

Table 2, entries 14 and 15

single_pulse



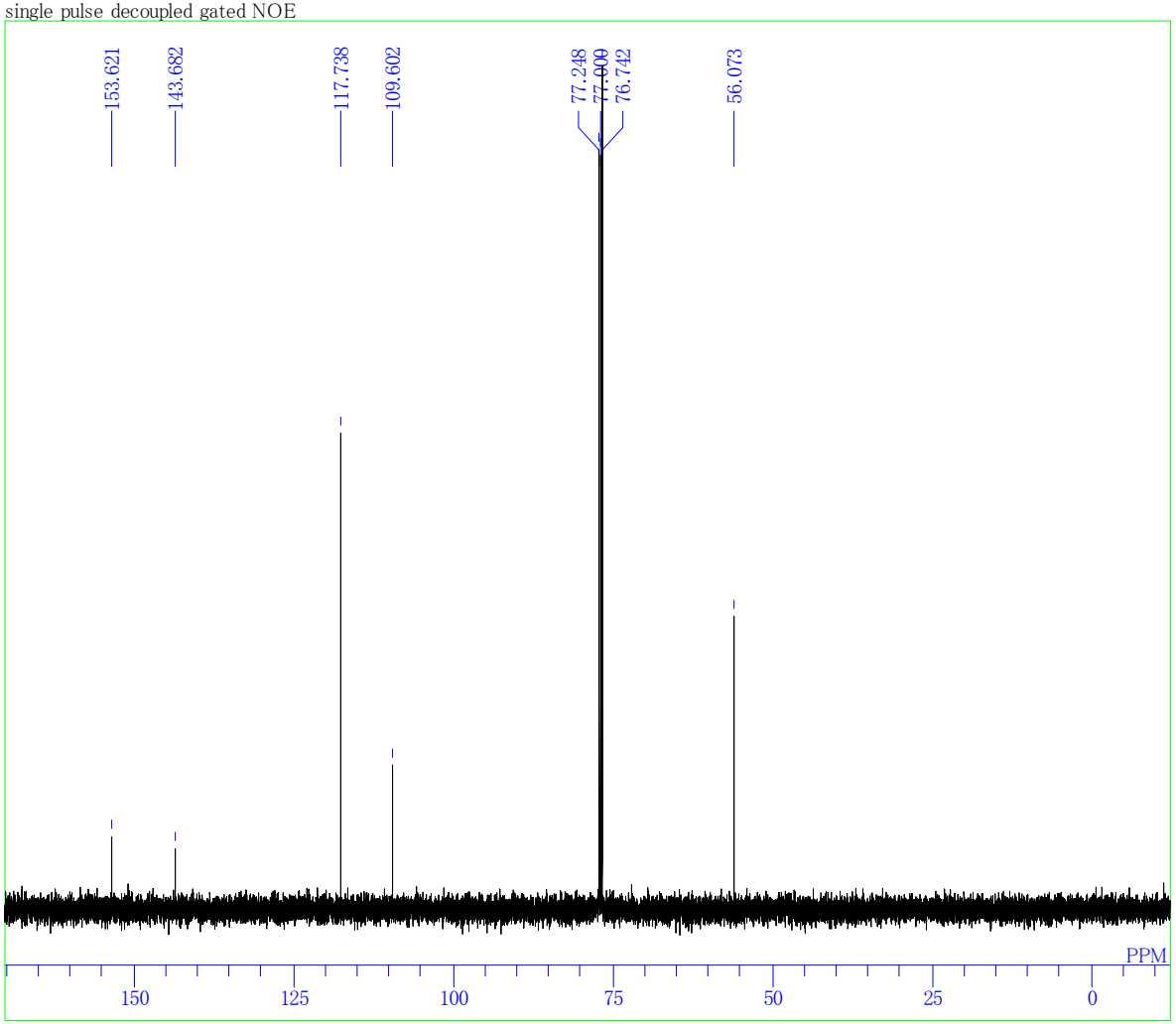
DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single_pulse
DATIM	18-12-2007 13:28:05
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16384
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	6.00 usec
IRNUC	1H
CTEMP	17.5 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	50



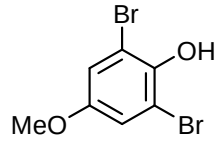
S1h ($CDCl_3$)

Starting Material of **1h**

single pulse decoupled gated NOE



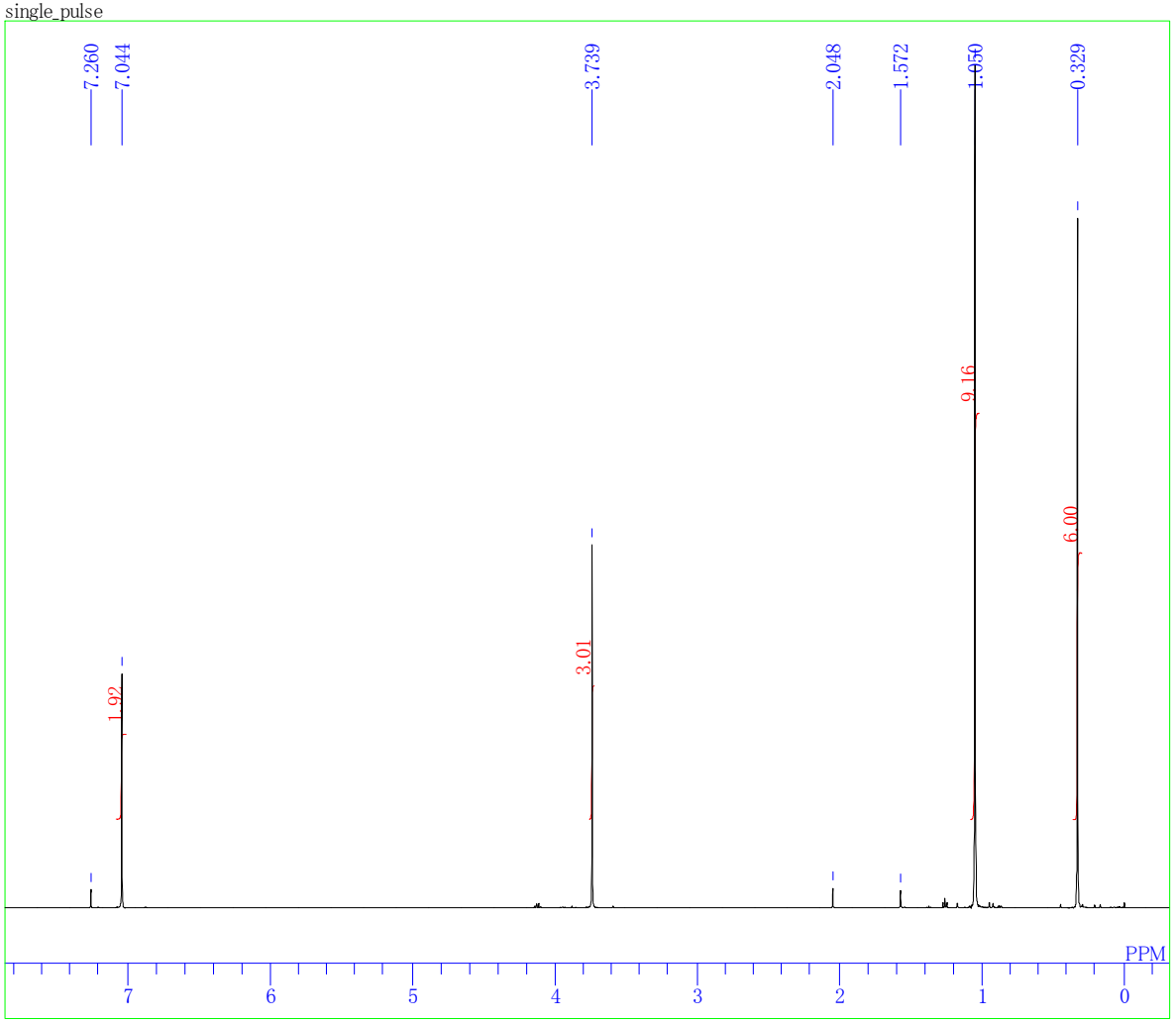
DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single pulse decoupled gated NOE
DATIM	18-12-2007 13:37:47
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32768
FREQU	39308.18 Hz
SCANS	125
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	18.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60



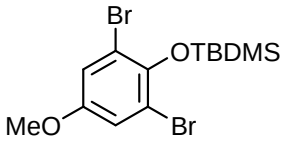
S1h ($CDCl_3$)

Starting Material of **1h**

single_pulse



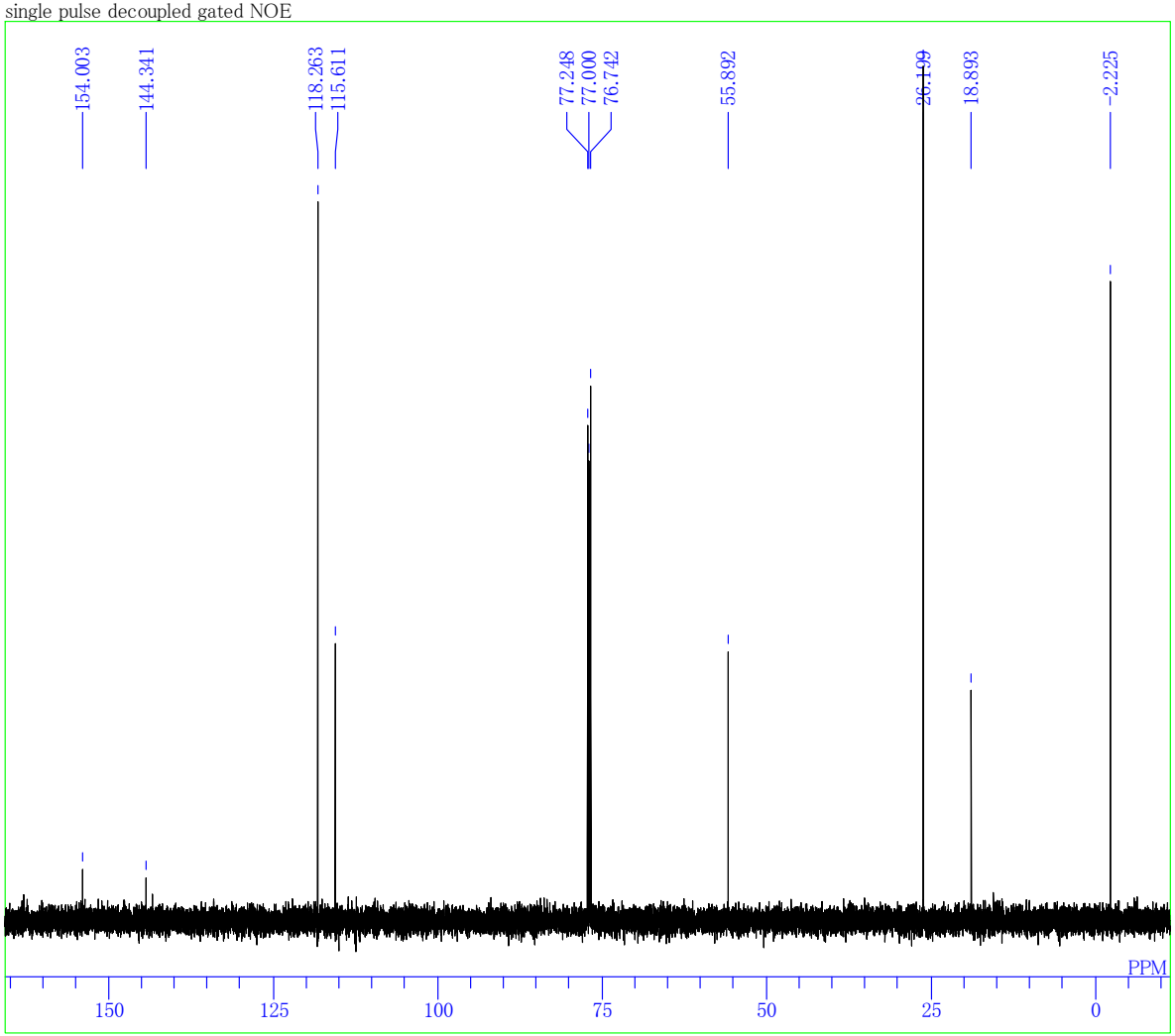
DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single_pulse
DATIM	18-12-2007 13:43:53
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16384
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	6.00 usec
IRNUC	1H
CTEMP	17.5 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	40



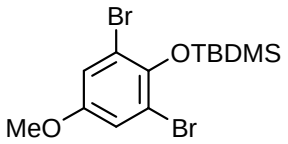
S2h ($CDCl_3$)

Starting Material of **1h**

single pulse decoupled gated NOE



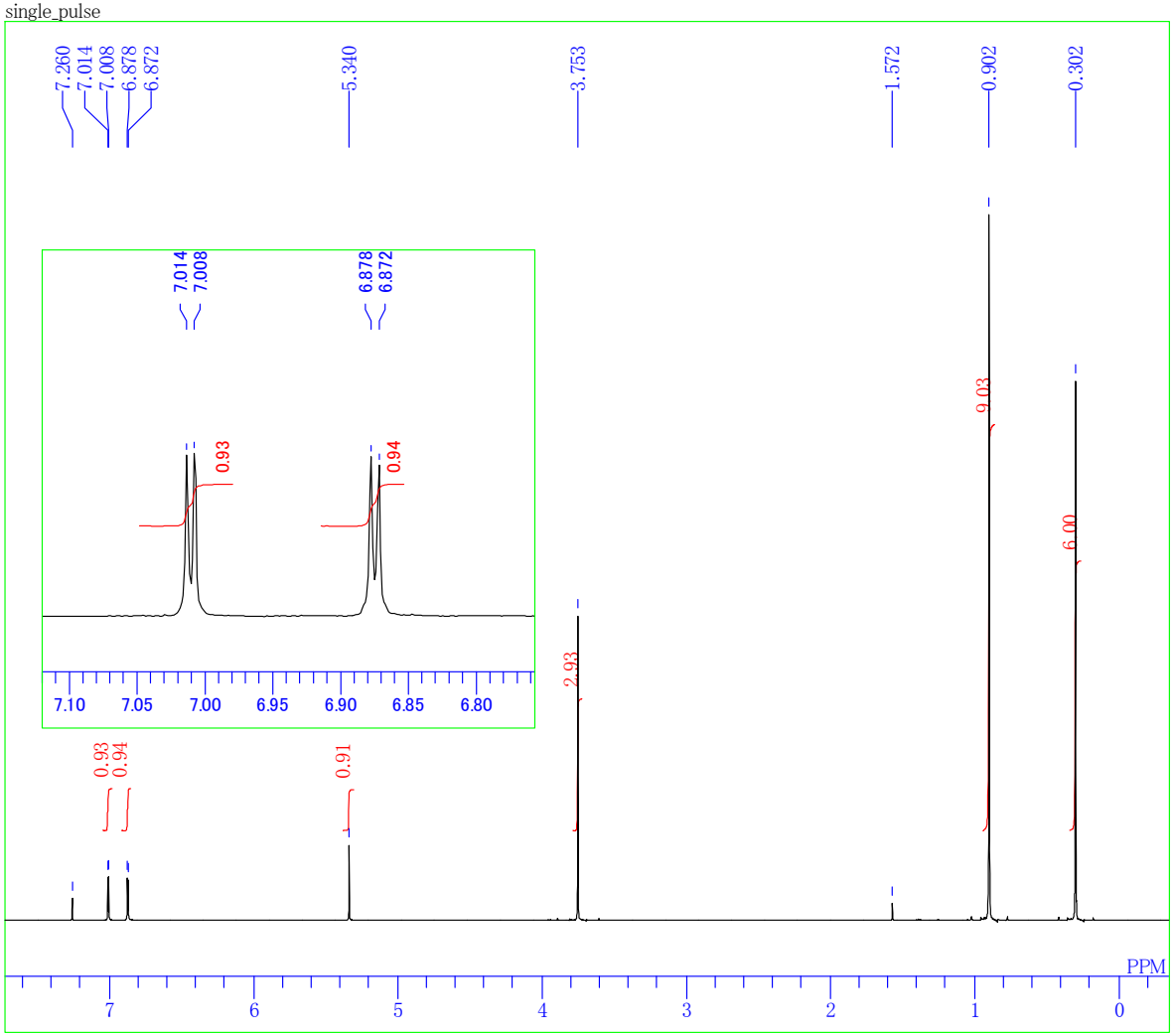
DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single pulse decoupled gated NOE
DATIM	18-12-2007 13:48:60
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.06 Hz
SCANS	40
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	17.9 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60



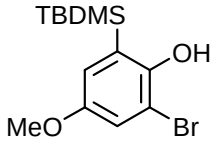
S2h ($CDCl_3$)

Starting Material of **1h**

single_pulse



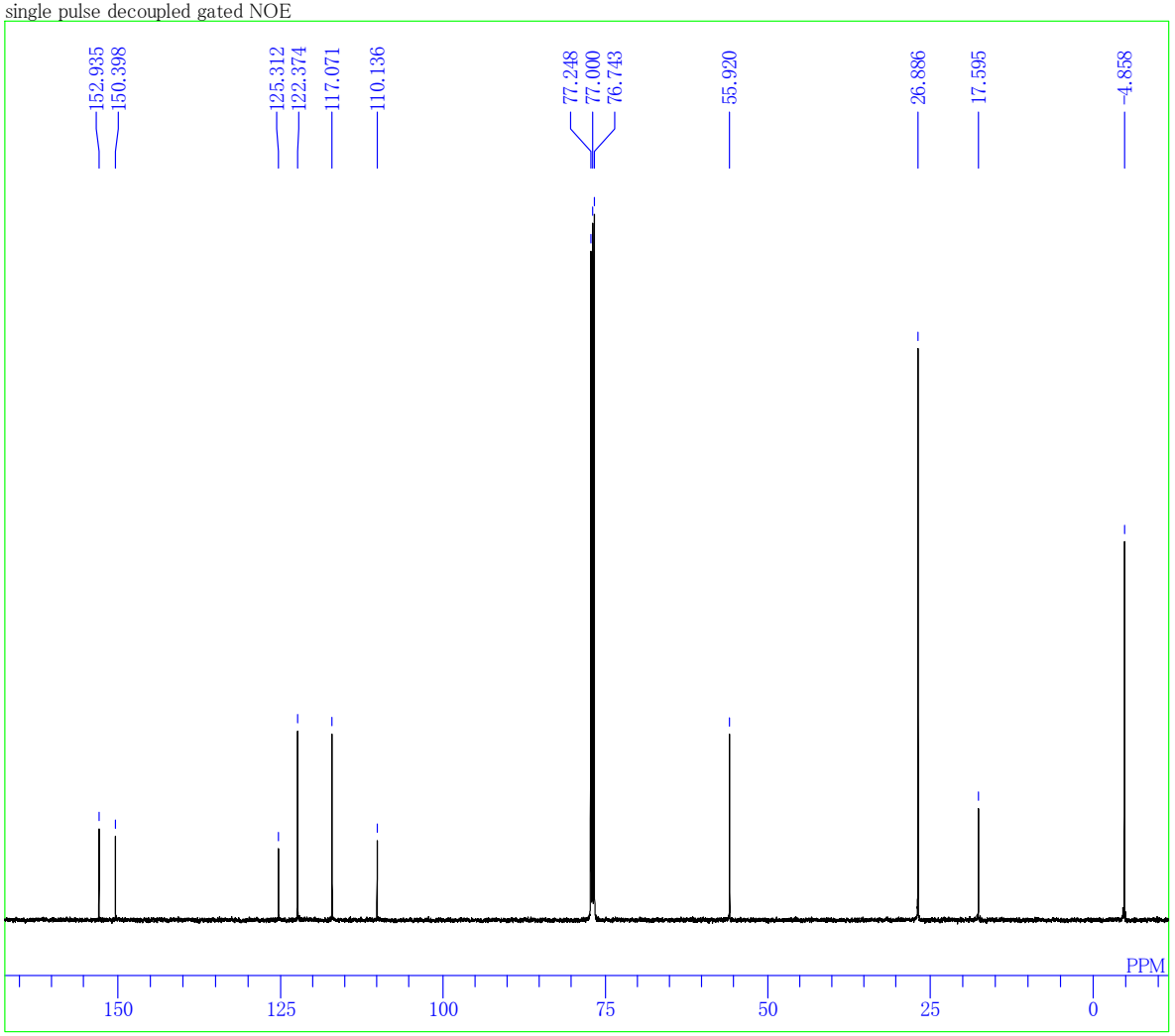
DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single_pulse
DATIM	18-02-2008 13:50:10
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	20480
FREQU	11730.66 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	6.00 usec
IRNUC	1H
CTEMP	16.6 c
SLVNT	CDCL3
EXREF	12.51 ppm
BF	0.12 Hz
RGAIN	42



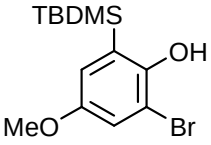
S3h ($CDCl_3$)

Starting Material of **1h**

single pulse decoupled gated NOE



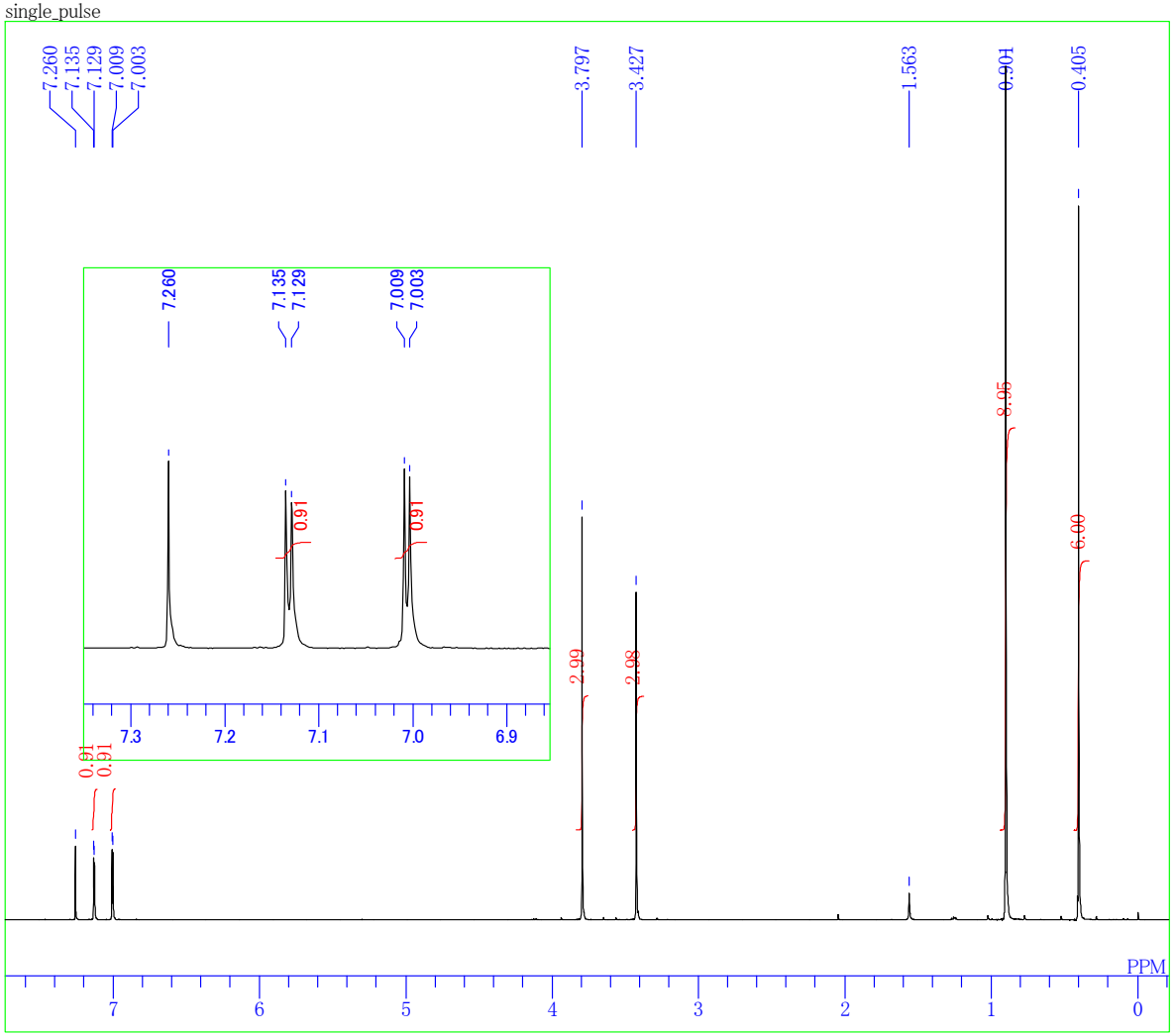
DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single pulse decoupled gated NOE
DATIM	18-02-2008 15:22:35
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	40961
FREQU	49135.97 Hz
SCANS	1883
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	17.1 c
SLVNT	CDCL3
EXREF	224.89 ppm
BF	0.12 Hz
RGAIN	60



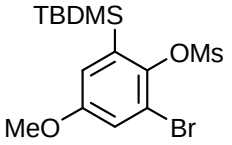
S3h ($CDCl_3$)

Starting Material of **1h**

single_pulse



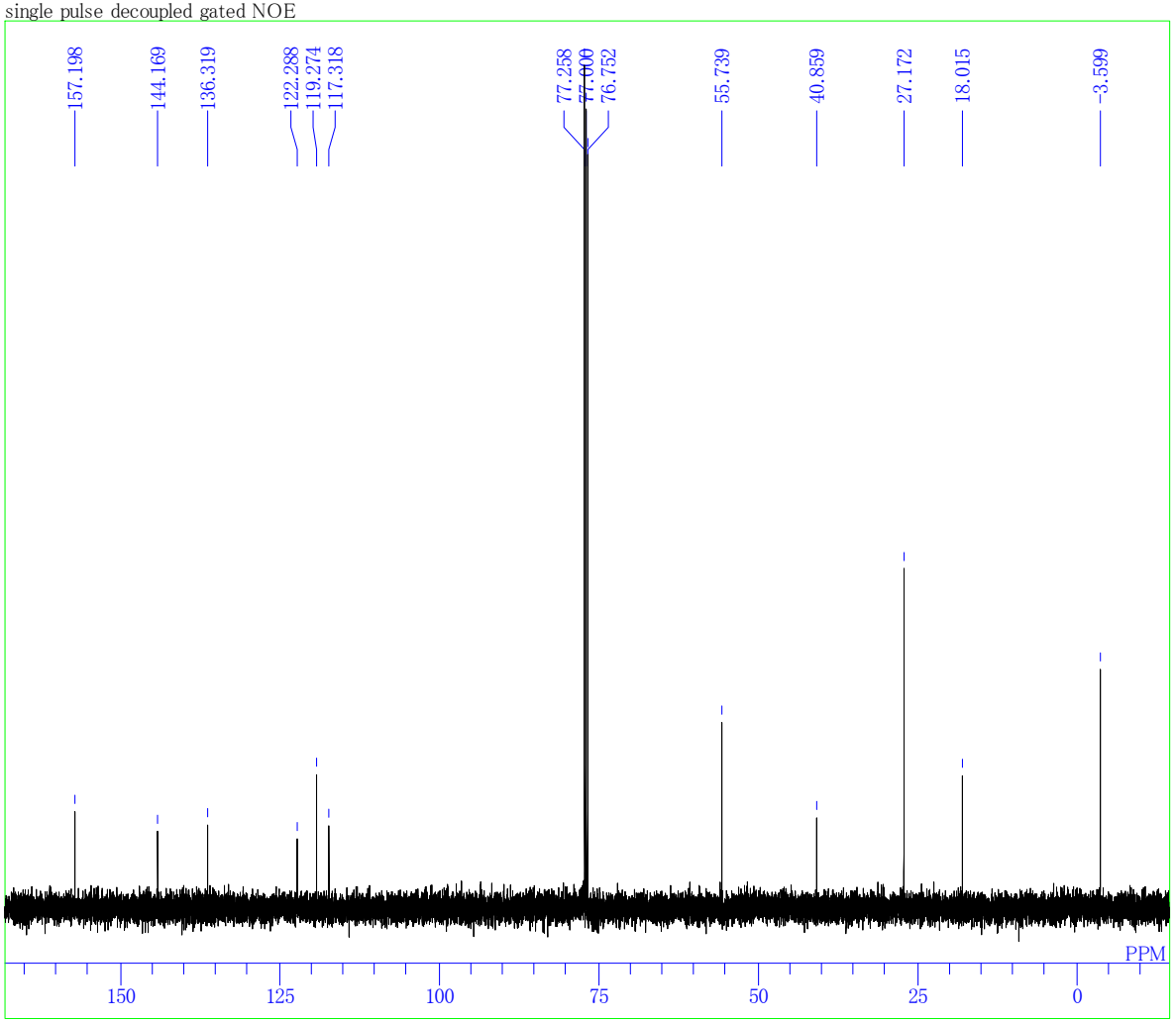
DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single_pulse
DATIM	19-12-2007 14:58:05
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.39 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	6.00 usec
IRNUC	1H
CTEMP	17.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	48



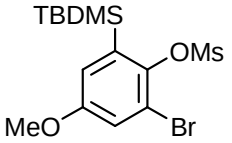
1h ($CDCl_3$)

Table 2, entry 16

single pulse decoupled gated NOE

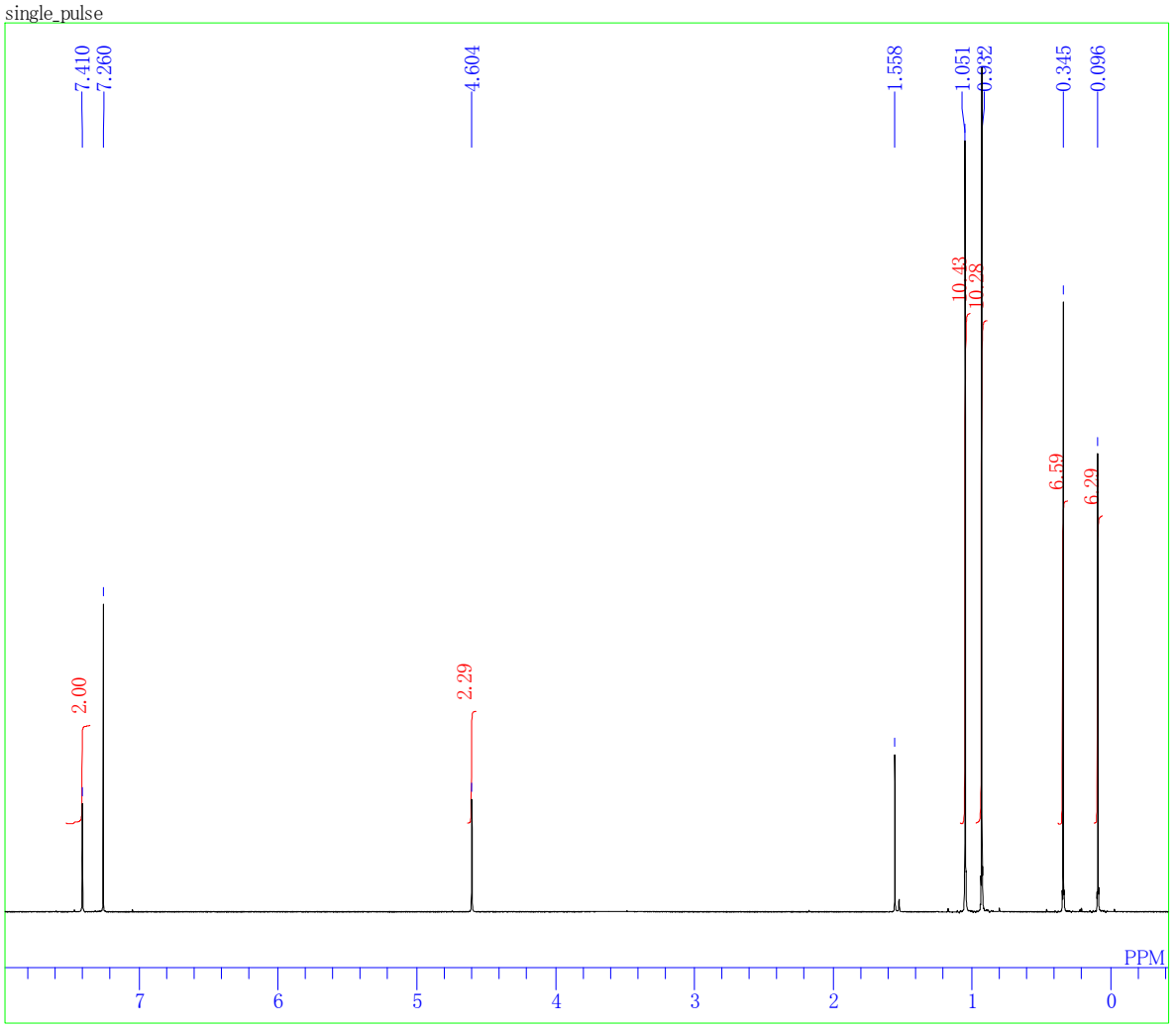


DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single pulse decoupled gated NOE
DATIM	19-12-2007 15:06:42
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32768
FREQU	39308.18 Hz
SCANS	109
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	18.3 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

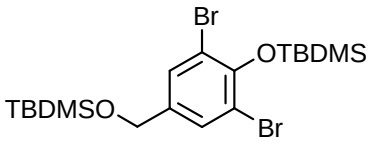


1h ($CDCl_3$)

Table 2, entry 16



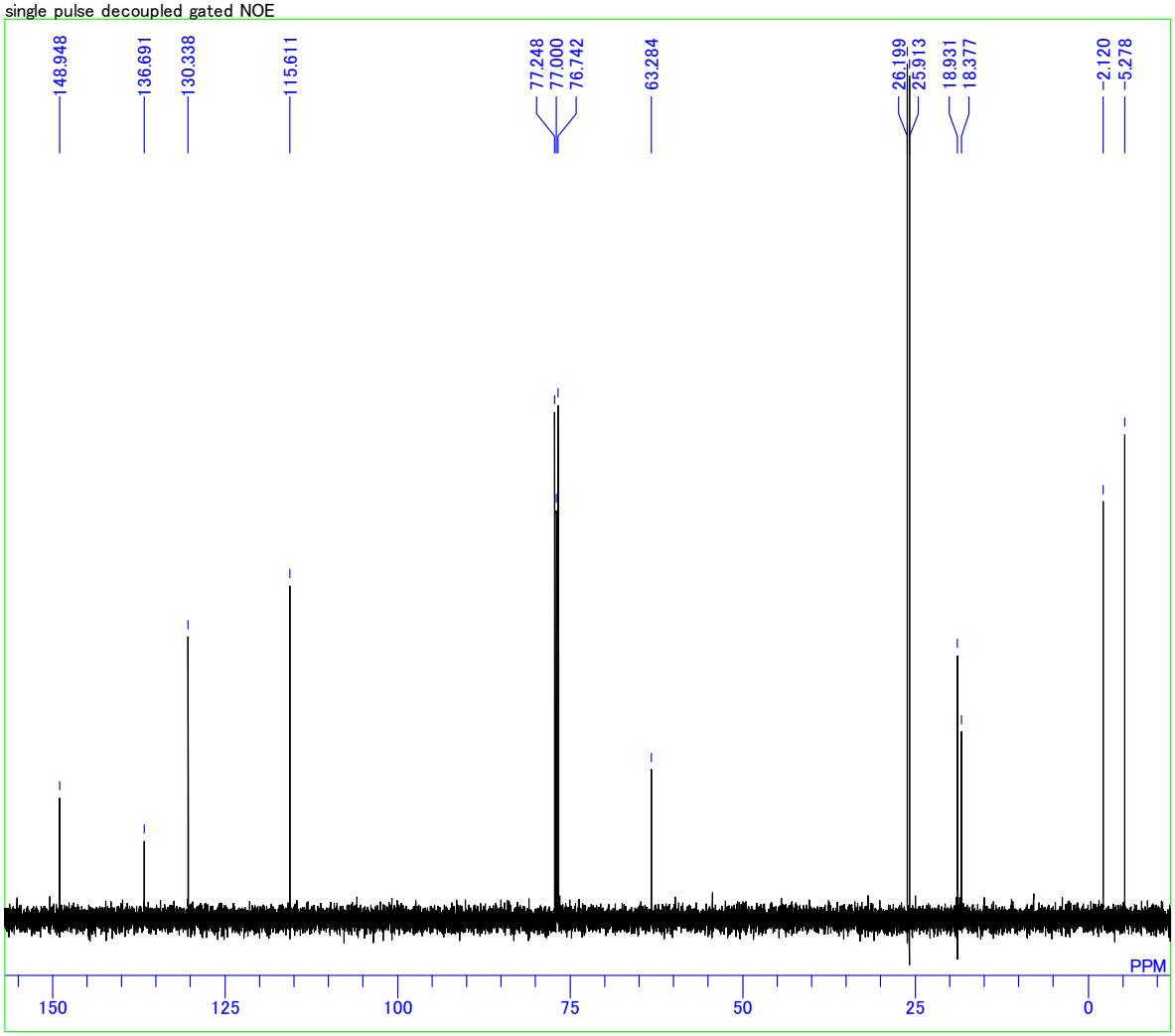
DFILE	F:¥TBSOCH2PhBr2OTBDMS_1H
COMNT	single_pulse
DATIM	10-01-2008 09:49:18
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16384
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	1.5000 sec
PW1	6.00 usec
IRNUC	1H
CTEMP	16.7 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	54



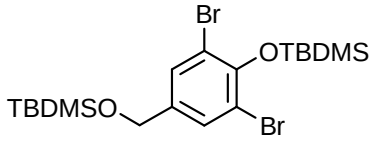
S2i ($CDCl_3$)

Starting Material of **1i**

single pulse decoupled gated NOE

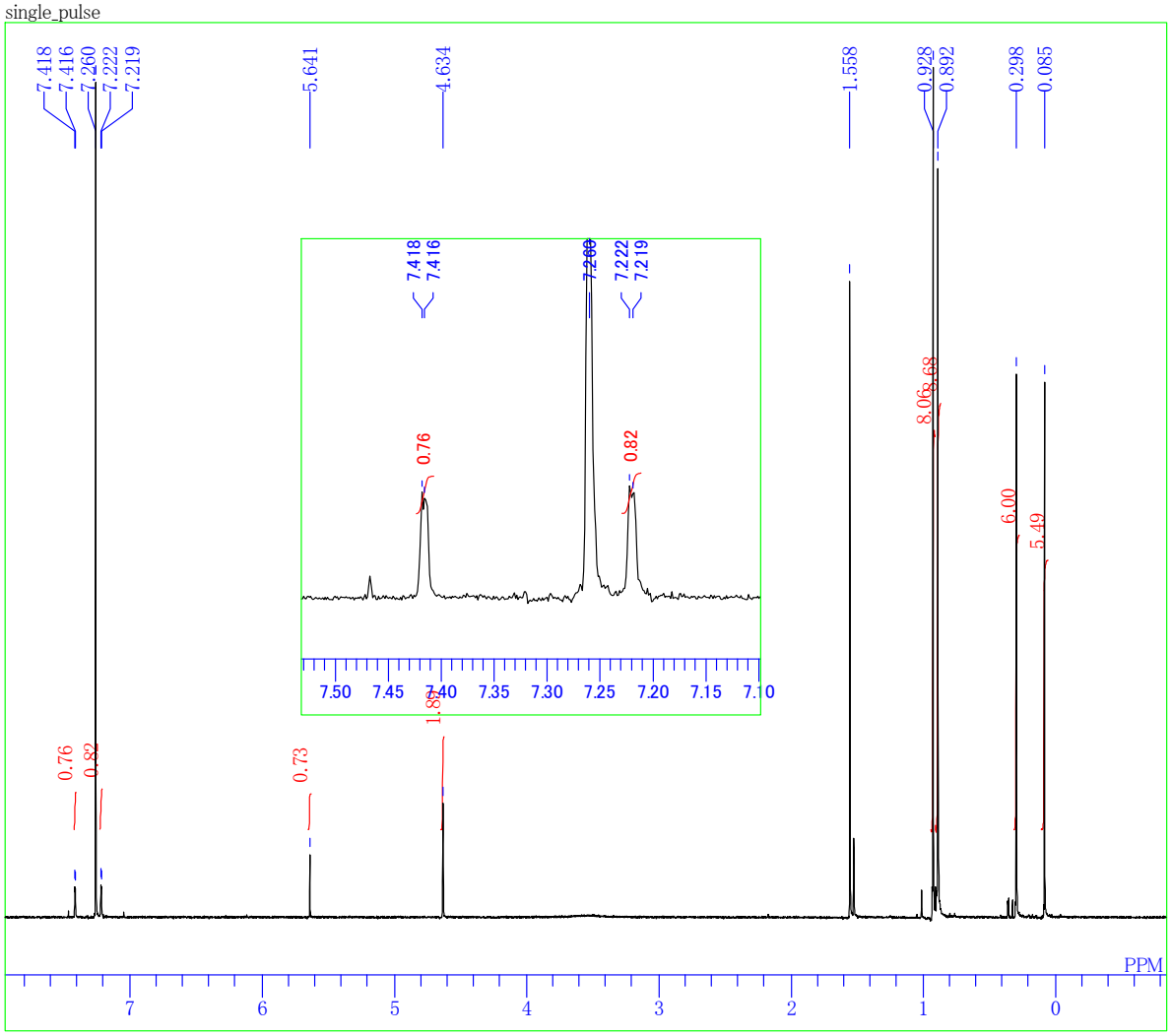


DFILE	C:¥Documents and Settings¥Owner¥My D
COMNT	single pulse decoupled gated NOE
DATIM	10-01-2008 13:18:42
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32768
FREQU	39308.18 Hz
SCANS	1412
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	17.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

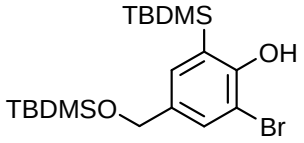


S2i ($CDCl_3$)

Starting Material of **1i**

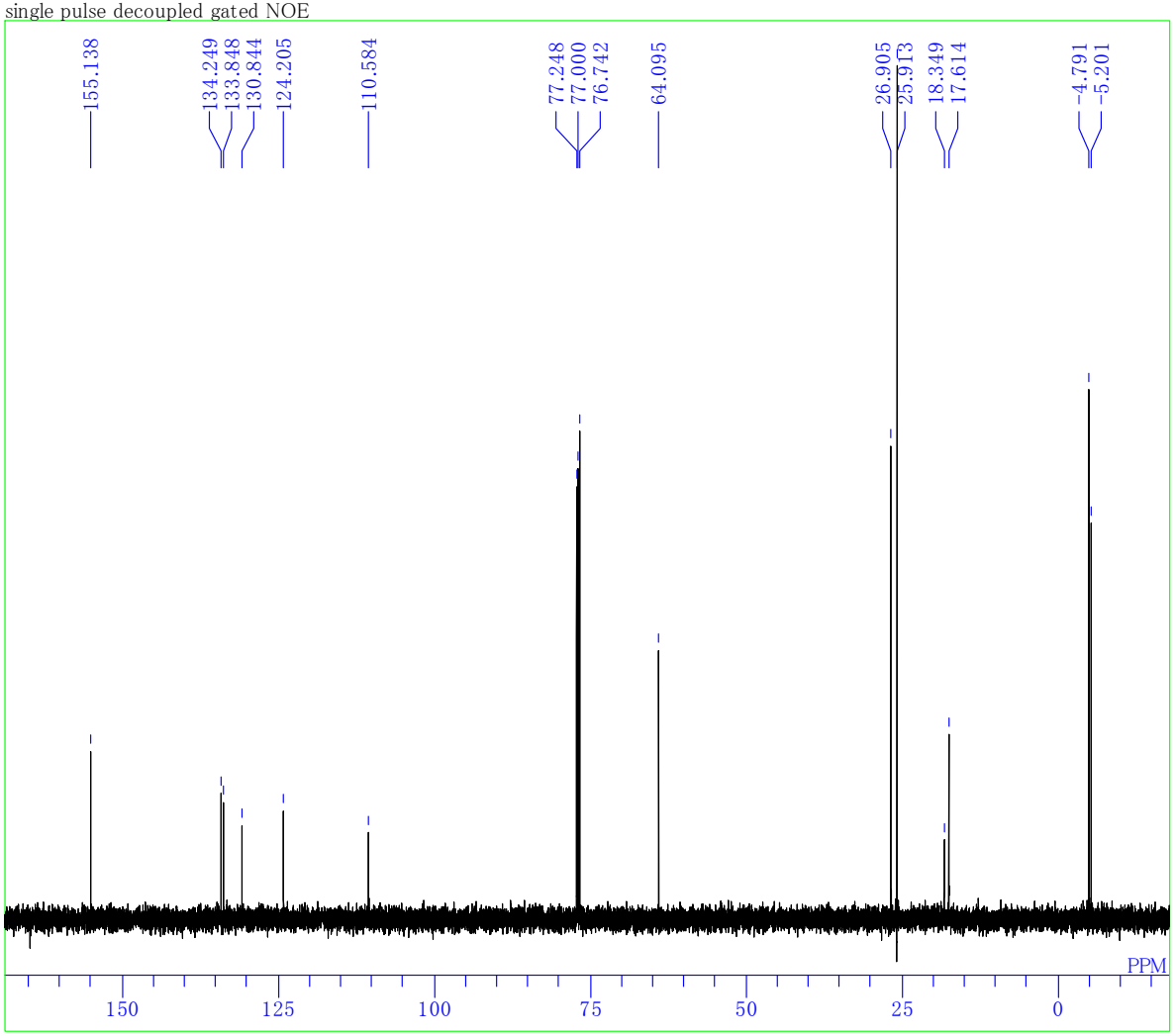


DFILE	C:¥Documents and Settings¥Owner¥M
COMNT	single_pulse
DATIM	10-01-2008 09:53:56
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16384
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	1.5000 sec
PW1	6.00 usec
IRNUC	1H
CTEMP	16.7 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	56

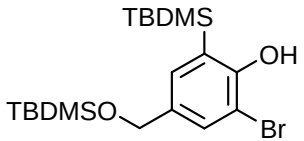


S3i ($CDCl_3$)
Starting Material of **1i**

single pulse decoupled gated NOE

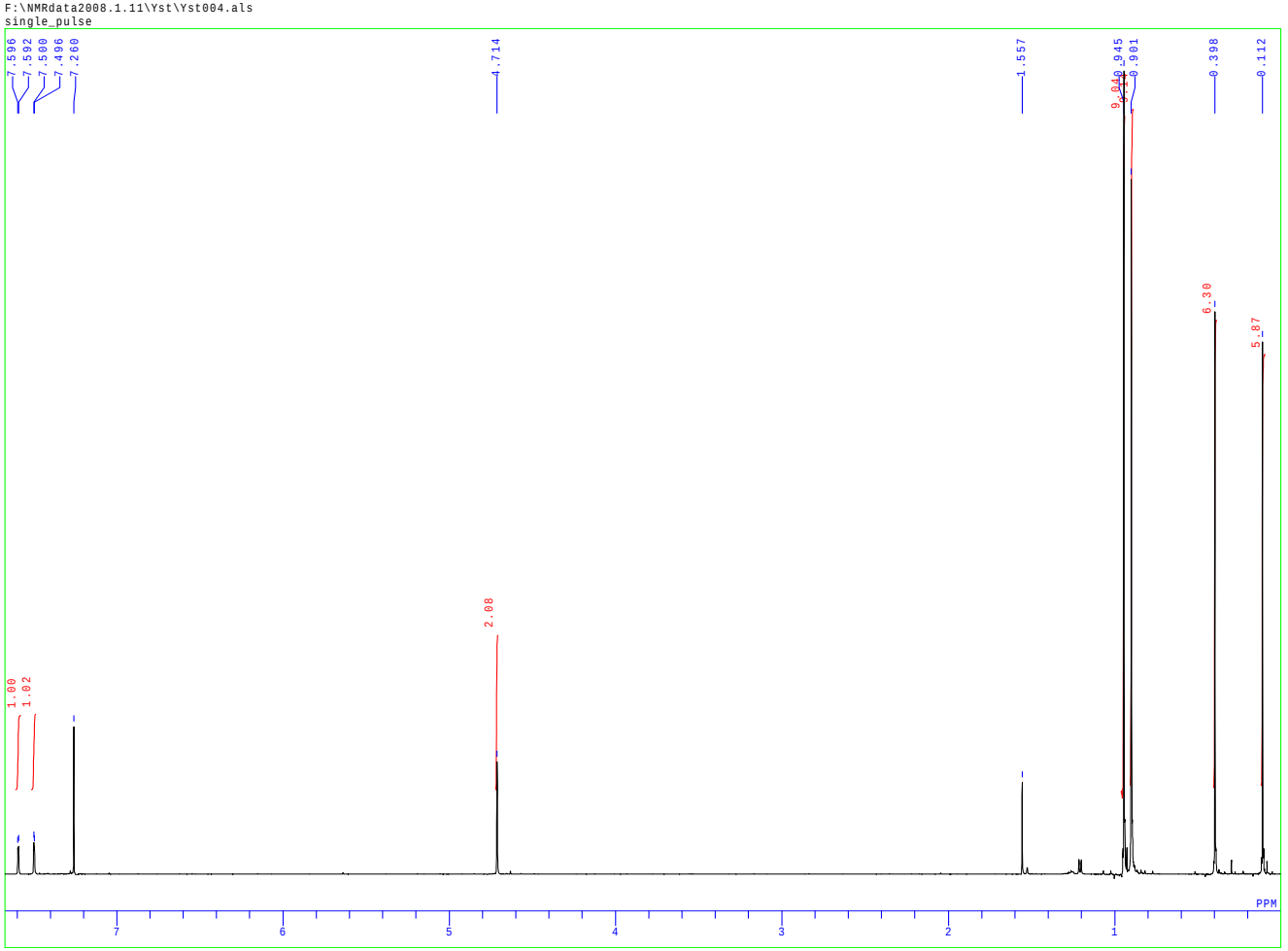


DFILE	C:¥Documents and Settings¥Owner
COMNT	single pulse decoupled gated NOE
DATIM	10-01-2008 14:33:15
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32768
FREQU	39308.18 Hz
SCANS	1459
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	17.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60



S3i ($CDCl_3$)

Starting Material of **1i**




```
DFILE   F:\NMRdata2008.1.11\Yst\Yst004.a
COMNT   single_pulse
DATIM   10-01-2008 16:21:03
OBNUC   1H
EXMOD   single_pulse.ex2
OBFRQ         500.16 MHz
OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          13106
FREQU        7507.39 Hz
SCANS               8
ACQTM         1.7459 sec
PD            1.5000 sec
PW1             6.00 usec
IRNUC   1H
CTEMP           17.1 c
SLVNT   CDCL3
EXREF           7.26 ppm
BF              0.00 Hz
RGAIN             50
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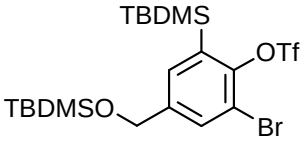
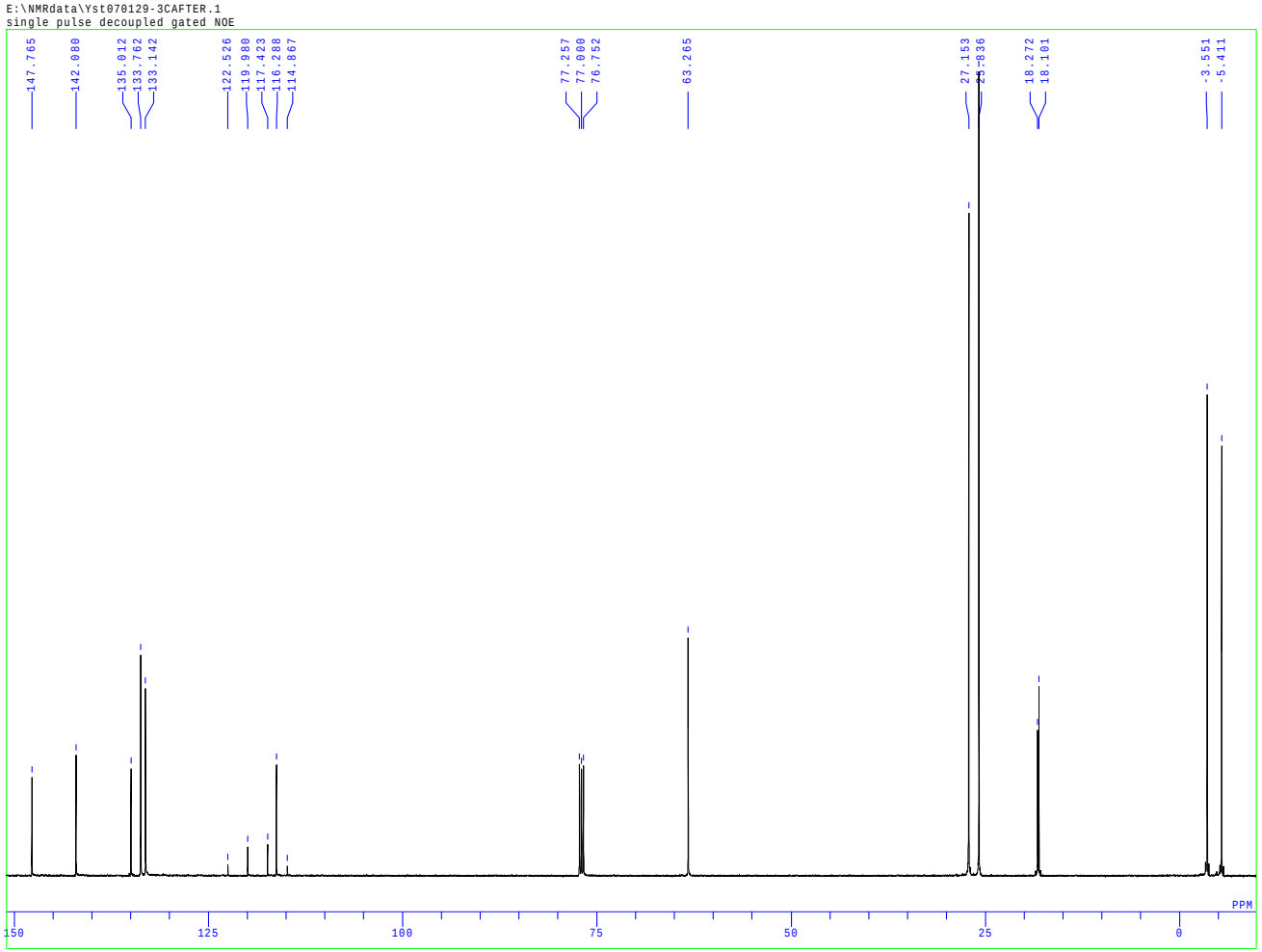
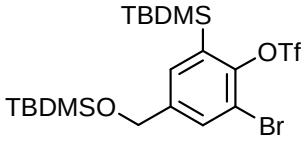


1i ($CDCl_3$)

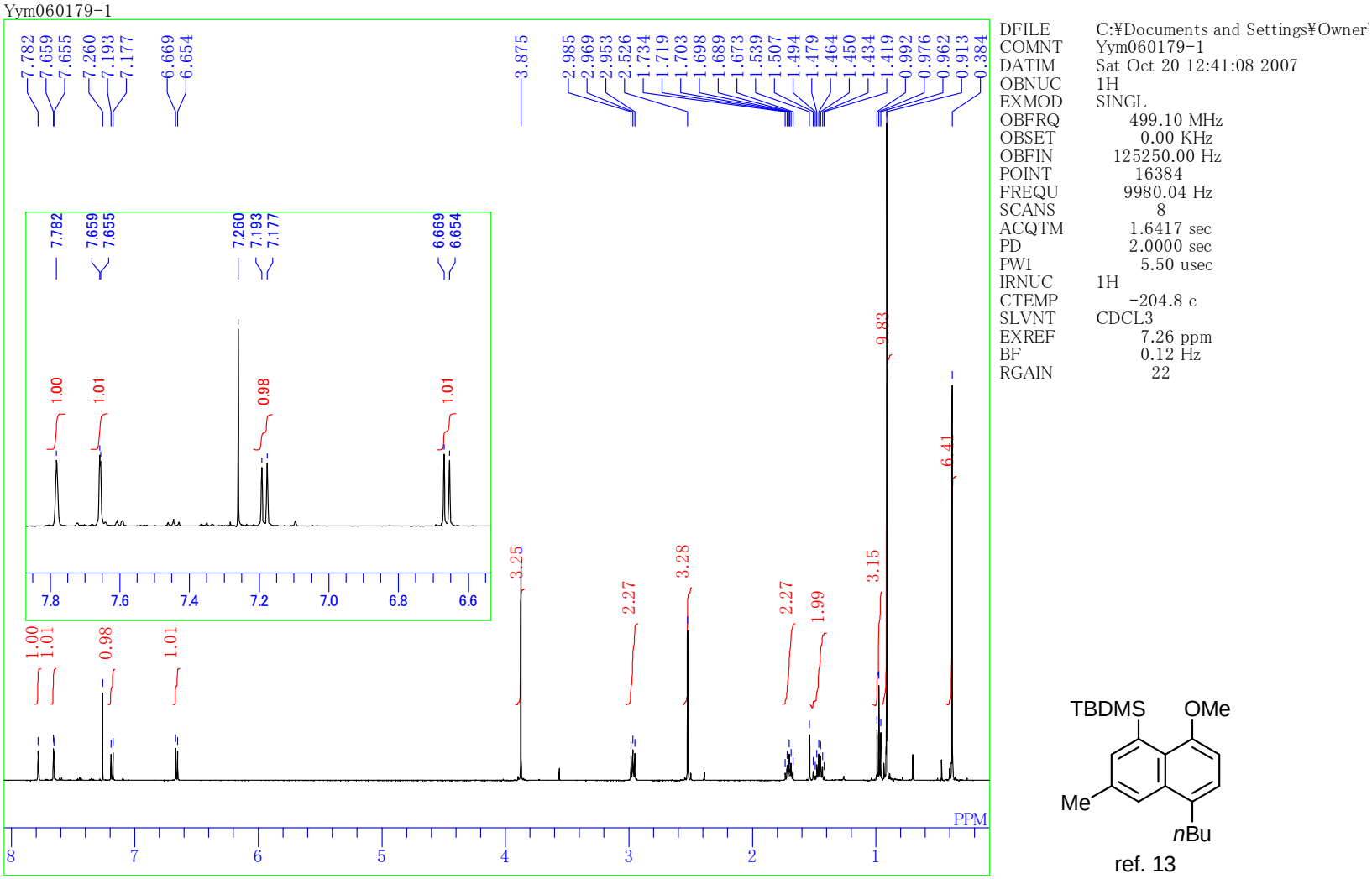
Table 2, entry 17



DFILE E:\NMRdata\Yst070129-3CAFTER.1
COMNT single pulse decoupled gated NOE
DATIM 29-01-2008 20:47:59
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 40961
FREQU 49135.97 Hz
SCANS 673
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 17.1 c
SLVNT CDCL3
EXREF 224.92 ppm
BF 0.00 Hz
RGAIN 60



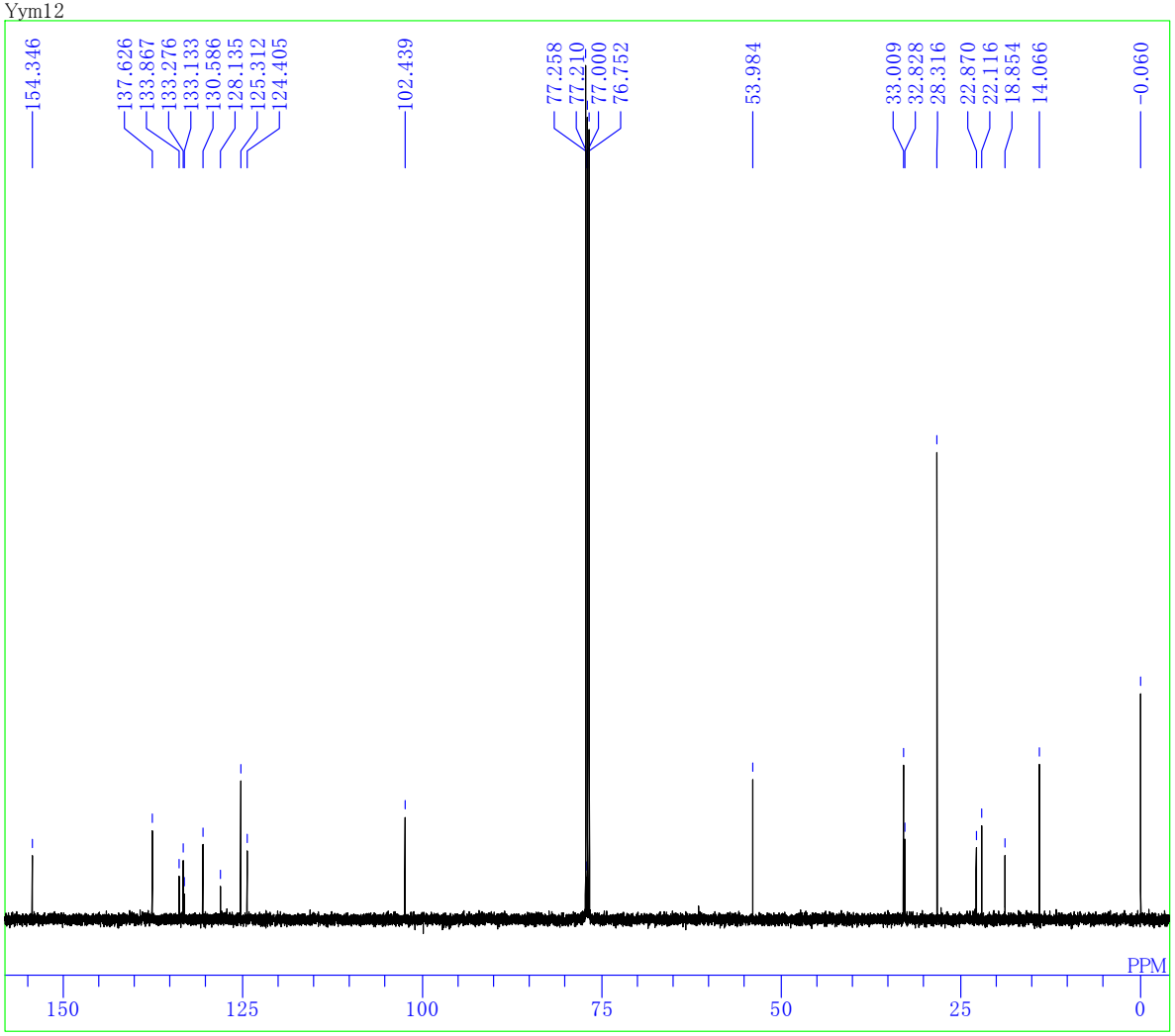
1i ($CDCl_3$)
Table 2, entry 17



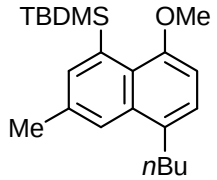
DFILE	C:¥Documents and Settings¥Owner
COMNT	Yym060179-1
DATIM	Sat Oct 20 12:41:08 2007
OBNUC	1H
EXMOD	SINGL
OBFRQ	499.10 MHz
OBSET	0.00 KHz
OBFIN	125250.00 Hz
POINT	16384
FREQU	9980.04 Hz
SCANS	8
ACQTM	1.6417 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	-204.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	22

ref. 13

Yym12



DFILE	C:¥Documents and Settings¥Owner
COMNT	Yym12
DATIM	22-12-2007 23:10:20
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.06 Hz
SCANS	1274
ACQTM	0.8336 sec
PD	1.5000 sec
PW1	3.67 usec
IRNUC	1H
CTEMP	17.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60



ref. 13