Supporting Information

Cationic Gold(I) Mediated Intramolecular Cyclization of 3-Alkyne-1,2-diols and 1-Amino-3-alkyn-2-ols: A Practical Route to Furans and Pyrroles

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Contents

Experimental Section

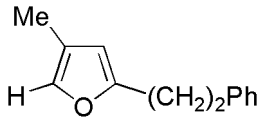
General. Melting points were determined on a Yazawa Micro Melting Point Apparatus and are uncorrected. Infrared (IR) absorption spectra were recorded in $CHCl_3$ solution on a SHIMADZU IRPrestige-21 spectrophotometer. ^1H and ^{13}C NMR spectra were measured on a JEOL JNM-ECA500 using $CDCl_3$ as a solvent. Mass spectra (MS) were measured on a JEOL JMS-SX102A or JEOL JMS-700 instrument. Kanto silica gel 60N was used for column chromatography. All reagents were of reagent grade unless otherwise stated. In general, reactions were carried out in anhydrous solvents.

General procedure for the preparation of furans

To a solution of the 3-alkyne-1,2-diols **1** (2.6 mmol) in toluene (6.5 mL, 0.4 M) were added $(Ph_3P)AuCl$ (1.3 mg, 0.0026 mmol) and $AgNTf_2$ (1.0 mg, 0.0026 mmol) in this order at room temperature. The reaction mixture was stirred at room temperature until the complete consumption of **1** and then quenched with saturated aqueous NH_4Cl. The organic materials were extracted with Et_2O, and the combined organic extracts were washed with brine, dried over $MgSO_4$, and evaporated in vacuo. The residue was purified by column chromatography (silica gel, usually hexanes/EtOAc or hexanes) to give the furans **2**.

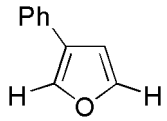
Spectroscopic data for furans

4-Methyl-2-(2-phenylethyl)furan (2a)[1]



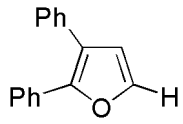
A colorless oil; ^1H NMR (500 MHz, $CDCl_3$) δ 1.98 (3H, s), 2.85–2.96 (4H, m), 5.85 (1H, s), 7.08 (1H, s), 7.16–7.31 (5H, m); ^{13}C NMR (125 MHz, $CDCl_3$) δ 9.8, 30.0, 34.3, 107.9, 120.4, 126.0, 128.33, 128.34, 137.4, 141.3, 155.4; IR ($CHCl_3$) ν 3020, 2930, 1615, 1600, 1550 cm^{-1}. HRMS (FAB) *m*/*z* calcd for $C_{13}H_{15}O$ $[M]^+$: 187.1123. Found: 187.1094.

3-Phenylfuran (2b)[2]



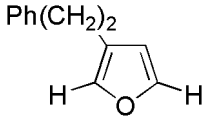
An off-white solid, mp 53–56 °C; ^1H NMR (500 MHz, $CDCl_3$) δ 6.70 (1H, s), 7.22–7.52 (5H, m), 7.73 (1H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ 108.8, 125.8, 126.4, 127.0, 128.8, 132.4, 138.4, 143.6; IR ($CHCl_3$) ν 3030, 1765, 1610 cm^{-1}.

2,3-Diphenylfuran (2c)[3]



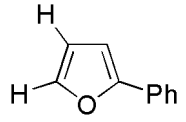
A colorless oil; ^1H NMR (500 MHz, $CDCl_3$) δ 6.56 (1H, d, *J*= 1.7 Hz), 7.22–7.45 (8H, m), 7.51 (1H, d, *J*= 1.7 Hz), 7.51–7.56 (2H, m); ^{13}C NMR (125 MHz, $CDCl_3$) δ 114.0, 122.2, 126.2, 127.1, 127.5, 128.4, 128.6, 128.7, 131.1, 134.3, 141.5, 148.5; IR ($CHCl_3$) ν 3065, 3015, 1605 cm^{-1}.

3-(2-Phenylethyl)furan (2d)[2]



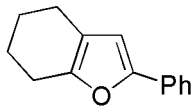
A colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.79 (2H, t, *J*= 7.7 Hz), 2.91 (2H, t, *J*= 7.7 Hz), 6.30 (1H, s), 7.20–7.36 (6H, m), 7.39 (1H, s); ^{13}C NMR (125 MHz, $CDCl_3$) δ 26.7, 36.4, 111.0, 124.4, 125.9, 128.3, 128.4, 138.9, 141.6, 142.6; IR ($CHCl_3$) ν 3030, 3015, 2930, 2860, 1605 cm^{-1}.

2-Phenylfuran (2e)[4]



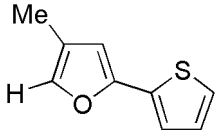
A colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.48 (1H, dd, *J*= 1.7, 3.4 Hz), 6.66 (1H, d, *J*= 3.4 Hz), 7.25–7.29 (1H, m), 7.37–7.42 (2H, m), 7.48 (1H, d, *J*= 1.7 Hz), 7.67–7.71 (2H, m); ^{13}C NMR (125 MHz, $CDCl_3$) δ 104.9, 111.6, 123.7, 127.3, 128.6, 130.8, 142.0, 153.9; IR ($CHCl_3$) ν 3075, 3015, 1610 cm^{-1}.

2-Phenyl-4,5,6,7-tetrahydrobenzofuran (2f)[5]



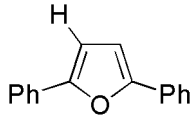
A colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.72–1.80 (2H, m), 1.84–1.91 (2H, m), 2.47 (2H, t, *J*= 5.5 Hz), 2.67 (2H, t, *J*= 6.0 Hz), 6.48 (1H, s), 7.21 (1H, t, *J*= 7.4 Hz), 7.32–7.39 (2H, m), 7.62 (2H, d, *J*= 8.0 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 22.1, 23.05, 23.10, 23.3, 106.0, 119.0, 123.2, 126.5, 128.5, 131.4, 150.8, 151.5; IR ($CHCl_3$) ν 3065, 3010, 2935, 2850, 1605 cm^{-1}.

4-Methyl-2-(2-thienyl)furan (2g)



A colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.05 (3H, s), 6.38 (1H, s), 7.02 (1H, dd, *J*= 3.7, 5.0 Hz), 7.16–7.22 (3H, m); ^{13}C NMR (125 MHz, $CDCl_3$) δ 9.8, 107.7, 122.0, 122.2, 123.9, 127.6, 134.0, 138.3, 149.3; IR ($CHCl_3$) ν 3010, 2925, 1605 cm^{-1}. ***Anal.*** Calcd for C_9H_8OS: C, 65.82; H, 4.91. Found: C, 65.53; H, 5.14.

2,5-Diphenylfuran (2h)[6]



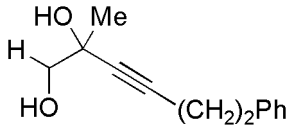
A white solid, mp 83–85 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.75 (2H, s), 7.28 (2H, t, *J*= 7.4 Hz), 7.42 (4H, t, *J*= 7.4 Hz), 7.76 (4H, d, *J*= 7.4 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 107.2, 123.7, 127.3, 128.7, 130.7, 153.3; IR ($CHCl_3$) ν 3065, 3015, 1610, 1590 cm^{-1}. ***Anal.*** Calcd for $C_{16}H_{12}O$: C, 87.25; H, 5.49. Found: C, 87.12; H, 5.48.

General procedure for the preparation of 3-alkyne-1,2-diols

To a solution of the alkynes (2.2 equiv) in THF (0.4 M) was added dropwise *n*-BuLi (1.6 M in hexane, 2.2 equiv) at 0 °C. After the mixture was stirred for 30 min at the same temperature, the appropriate α-hydroxycarbonyl compound (1.0 equiv) was added. The reaction mixture was allowed to get room temperature over 30 min. Upon the completed consumption of the carbonyl compound, the reaction was quenched with saturated aqueous NH_4Cl. The organic materials were extracted with EtOAc, and the combined organic extracts were washed with brine, dried over $MgSO_4$, and evaporated in vacuo. The residue was purified by column chromatography (silica gel, hexanes/EtOAc) to give the 3-alkyne-1,2-diols **1**.

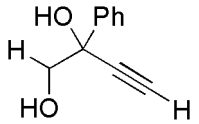
Spectroscopic data for 3-alkyne-1,2-diols

2-Methyl-6-phenyl-3-hexyne-1,2-diol (1a)



80% yield. A white solid, mp 52–55 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.39 (3H, s), 1.87–1.93 (1H, m), 2.51 (2H, t, *J*= 7.4 Hz), 2.58 (1H, s), 2.81 (2H, t, *J*= 7.4 Hz), 3.35–3.42 (1H, m), 3.52 (1H, dd, *J*= 4.6, 10.9 Hz), 7.19–7.34 (5H, m); ^{13}C NMR (125 MHz, $CDCl_3$) δ 20.8, 25.3, 34.7, 68.6, 70.9, 82.6, 84.3, 126.4, 128.4, 128.5, 140.4; IR ($CHCl_3$) ν 3585, 3015, 2935, 2870, 2240 cm^{-1}. *Anal*. Calcd for $C_{13}H_{16}O_2$: C, 76.44; H, 7.90. Found: C, 76.75; H, 8.00. HRMS (FAB) *m*/*z* calcd for $C_{13}H_{16}O_2Na$ $[M+Na]^+$: 227.10480. Found: 227.10426.

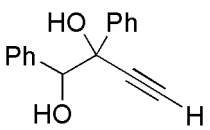
2-Phenyl-3-butyne-1,2-diol (1b)



52% yield: Prepared from 2-hydroxyacetophenone and ethynylmagnesium bromide.

A white solid, mp 90–92 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.16–2.21 (1H, m), 2.72 (1H, s), 3.11 (1H, s), 3.67 (1H, dd, *J*= 8.3, 11.2 Hz), 3.78 (1H, dd, *J*= 6.0, 11.2 Hz), 7.32–7.43 (3H, m), 7.64 (2H, d, *J*= 7.4 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 72.0, 73.6, 74.8, 84.2, 125.7, 128.4, 139.9; IR ($CHCl_3$) ν 3585, 3300, 3015, 2930 cm^{-1}. HRMS (EI) *m*/*z* calcd for $C_{10}H_{10}O_2$ $[M]^+$: 162.0681. Found: 162.0678. *Anal.* Calcd for $C_{10}H_{10}O_2$: C, 74.06; H, 6.21. Found: C, 73.99; H, 6.04.

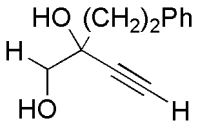
1,2-Diphenyl-3-butyne-1,2-diol (1c)



84% yield: Prepared from benzoin and ethynylmagnesium bromide.

A white solid, mp 109–112 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.73 (1H, s), 2.75 (1H, d, *J*= 3.5 Hz), 2.97 (1H, s), 4.88 (1H, d, *J*= 3.0 Hz), 7.12–7.29 (8H, m), 7.40–7.44 (2H, m); ^{13}C NMR (125 MHz, $CDCl_3$) δ 75.8, 76.1, 80.8, 84.6, 126.5, 127.4, 127.8, 128.0, 128.2, 137.1, 139.5; IR ($CHCl_3$) ν 3575, 3300, 3030, 3015, 2895 cm^{-1}. HRMS (EI) *m*/*z* calcd for $C_{16}H_{14}O_2$ $[M]^+$: 238.09938. Found: 238.10007. *Anal.* Calcd for $C_{16}H_{14}O_2$: C, 80.65; H, 5.92. Found: C, 80.61; H, 6.09.

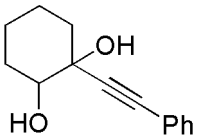
2-(2-Phenylethyl)-3-butyne-1,2-diol (1d)



83% yield: Prepared from 1-hydroxy-4-phenyl-2-butanone[7] and ethynylmagnesium bromide.

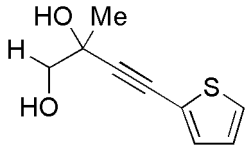
A white solid, mp 78–81 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.91–2.06 (3H, m), 2.57 (1H, s), 2.66 (1H, s), 2.85–2.96 (2H, m), 3.56 (1H, dd, *J*= 8.6, 11.0 Hz), 3.72 (1H, dd, *J*= 5.2, 11.0 Hz), 7.18–7.32 (5H, m); ^{13}C NMR (125 MHz, $CDCl_3$) δ 30.3, 39.3, 69.8, 71.6, 74.1, 84.2, 126.0, 128.4, 128.5, 141.5; IR ($CHCl_3$) ν 3585, 3300, 3015, 2930, 1605 cm^{-1}. HRMS (FAB) *m*/*z* calcd for $C_{12}H_{14}O_2Na$ $[M+Na]^+$: 213.0854. Found: 213.0902.

1-(2-Phenylethynyl)cyclohexane-1,2-diol (1f)



52% yield: A white solid, mp 90–92 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.23 (1H, m), 1.52–1.78 (5H, m), 1.94–2.01 (1H, m), 2.10–2.19 (2H, m), 3.21 (1H, s), 3.46–3.53 (1H, m), 7.28–7.35 (3H, m), 7.43–7.48 (2H, m); ^{13}C NMR (125 MHz, $CDCl_3$) δ 23.3, 24.2, 32.2, 37.7, 74.4, 77.2, 87.4, 88.5, 122.3, 128.3, 128.5, 131.8; IR ($CHCl_3$) ν 3565, 3015, 2940, 2860, 2220 cm^{-1}. HRMS (EI) *m*/*z* calcd for $C_{14}H_{16}O_2$ $[M]^+$: 216.1151. Found: 216.1148.

2-Methyl-4-(2-thienyl)-3-butyne-1,2-diol (1g)

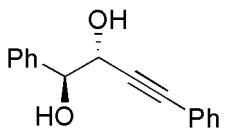


56% yield: Prepared from hydroxyacetone and (2-thienylethynyl)lithium, generated in 2-(2,2-dibromoethenyl)thiophene[8] and *n*-BuLi.

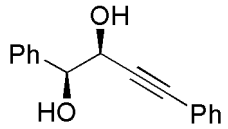
A white solid, mp 79–81 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.55 (3H, s), 2.08 (1H, dd, *J*= 5.2, 8.6 Hz), 2.68 (1H, s), 3.57 (1H, dd, *J*= 8.6, 11.0 Hz), 3.76 (1H, dd, *J*= 5.2, 11.0 Hz), 6.97 (1H, dd, *J*= 3.4, 5.2 Hz), 7.21 (1H, d, *J*= 3.4 Hz), 7.26 (1H, d, *J*= 5.2 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 25.2, 69.2, 70.6, 77.8, 94.1, 122.0, 127.0, 127.5, 132.5; IR ($CHCl_3$) ν 3590, 3015, 2935, 2220 cm^{-1}. *Anal.* Calcd for $C_9H_{10}O_2S$: C, 59.32; H, 5.53. Found: C, 59.24; H, 5.54.

1,4-Diphenyl-3-butyne-1,2-diol (1h)

***anti*-1h** 46% yield, ***syn*-1h** 17% yield: Prepared from 2-(*tert*-butyl-dimethylsilanyloxy)-2-phenyl-acetaldehyde[9] and lithium phenylacetylide, followed by TBAF desilylation.



***anti*-1h**: A white solid, mp 90–92 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.39 (1H, d, *J*= 6.9 Hz), 2.64 (1H, d, *J*= 5.2 Hz), 4.77 (1H, dd, *J*= 4.6, 6.9 Hz), 4.92–4.95 (1H, m), 7.27–7.42 (8H, m), 7.49 (2H, d, *J*= 6.9 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 67.9, 76.4, 86.0, 87.4, 122.0, 126.7, 128.13, 128.16, 128.21, 128.6, 131.7, 138.9; IR ($CHCl_3$) ν 3605, 3575, 3065, 3015, 2900, 2230 cm^{-1}. *Anal.* Calcd for $C_{16}H_{14}O_2$: C, 80.65; H, 5.92. Found: C, 80.61; H, 5.87.

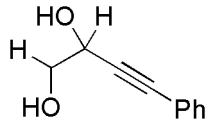


***syn*-1h**: A white solid, mp 72–73 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.55 (1H, br s), 2.89 (1H, br s), 4.61 (1H, dd, *J*= 5.7, 7.0 Hz), 4.82 (1H, dd, *J*= 2.5, 7.0 Hz), 7.26–7.51 (10H, m); ^{13}C NMR (125 MHz, $CDCl_3$) δ 68.0, 77.4, 86.4, 87.1, 122.0, 127.0, 128.3, 128.4, 128.7, 131.6, 139.0; IR ($CHCl_3$) ν 3605, 3565, 3085, 3015, 2900, 2230 cm^{-1}. *Anal.* Calcd for $C_{16}H_{14}O_2$: C, 80.65; H, 5.92. Found: C, 80.34; H, 5.79.

4-Phenyl-3-butyne-1,2-diol (1e)

To a solution of 1-(*tert*-butyl-dimethylsilanyloxy)-4-phenyl-3-butyn-2-one[10] (2.67 g, 9.73 mmol) in CH_3CN (70 mL) were added dropwise pyridine (7.0 mL) and HF (7.0 mL). After stirring for 2.5 h, the reaction was quenched with saturated aqueous NH_4Cl. The organic materials were extracted with EtOAc, and the combined organic extracts were washed with brine, dried over $MgSO_4$, and evaporated in vacuo. The residue was purified by column chromatography (silica gel, hexanes/EtOAc= 4:1) to give the hydroxyl alkynyl ketone (1.25 g, 80%).

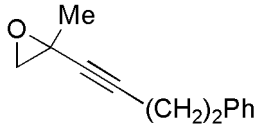
At 0 °C, $NaBH_4$ (443 mg, 11.7 mmol) was added in portions to a solution of the obtained hydroxyl alkynyl ketone (1.25 g, 7.80 mmol) in MeOH (100 mL). The reaction mixture was stirred for 2.0 h at room temperature, quenched with saturated aqueous NH_4Cl, and evaporated in vacuo. After the dilution with EtOAc, and the resulting solution was washed with brine, dried over $MgSO_4$, and evaporated in vacuo. The residue was purified by column chromatography (silica gel, hexanes/EtOAc= 1:1) to give **1e** (1.10 g, 87%).



A white solid, mp 72–73 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.12 (1H, dd, *J*= 5.0, 8.3 Hz), 2.38 (1H, d, *J*= 5.7 Hz), 3.76–3.88 (2H, m), 4.67–4.73 (1H, m), 7.29–7.36 (3H, m), 7.42–7.47 (2H, m); ^{13}C NMR (125 MHz, $CDCl_3$) δ 63.7, 66.6, 86.3, 86.4, 122.0, 128.3, 128.8, 131.8; IR ($CHCl_3$) ν 3600, 3015, 2930, 2225, 1490 cm^{-1}. *Anal.* Calcd for $C_{10}H_{10}O_2$: C, 74.06; H, 6.21. Found: C, 73.78; H, 6.01.

2-(4-Phenyl-1-butyn-1-yl)-2-methyloxirane (5)

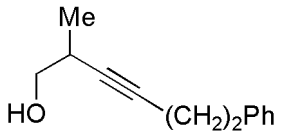
To a solution of 4-phenyl-1-butyne (1.74 g, 13.4 mmol) in THF (26.5 mL) was added dropwise *n*-BuLi (1.59 M in hexane; 9.25 mL, 14.7 mmol) at -78 °C. After the mixture was stirred for 30 min at -78 °C, chloroacetone (1.06 mL, 13.3 mmol) was added. The reaction was allowed to get room temperature over 30 min and kept stirring overnight. After quenching with saturated aqueous NH_4Cl, the organic materials were extracted with Et_2O. The combined organic extracts were washed with brine, dried over $MgSO_4$, and evaporated in vacuo. The residue was purified by column chromatography (silica gel, hexanes/EtOAc= 10:1) to give **5** (1.53 g, 62%).



A colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.51 (3H, s), 2.47 (2H, t, *J*= 7.5 Hz), 2.72 (1H, d, *J*= 5.5 Hz), 2.82 (2H, t, *J*= 7.5 Hz), 2.95 (1H, d, *J*= 5.5 Hz), 7.18–7.32 (5H, m); ^{13}C NMR (125 MHz, $CDCl_3$) δ 20.9, 23.2, 34.8, 47.5, 55.5, 80.2, 82.2, 126.3, 128.3, 128.4, 140.4; IR ($CHCl_3$) ν 3015, 2935, 2240 cm^{-1}. *Anal.* Calcd for $C_{13}H_{14}O$: C, 83.83; H, 7.58. Found: C, 83.82; H, 7.66.

2-Methyl-6-phenyl-3-hexyn-1-ol (6)

DIBAL in hexane (1.02 M; 6.1 mL, 6.22 mmol) was evaporated to dryness in vacuo. Under argon atmosphere, 6.0 mL THF was added to the residue. A solution of **5** (1.06 g, 5.69 mol) in THF (2.5 mL) was added dropwise to the mixture at -30 °C. After the mixture was stirred for 40 min at -30 °C, the reaction mixture was quenched with saturated aqueous NH_4Cl, and extracted with EtOAc. The combined organic extracts were washed with brine, dried over $MgSO_4$, and evaporated in vacuo. The residue was purified by column chromatography (silica gel, hexanes/EtOAc= 5:1) to give **6** (792 mg, 74%).



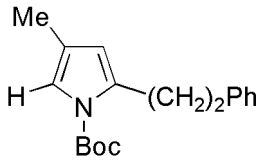
A colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.11 (3H, d, *J*= 6.9 Hz), 1.59–1.63 (1H, m), 2.48 (2H, dt, *J*= 2.3, 7.4 Hz), 2.57–2.65 (1H, m), 2.81 (2H, t, *J*= 7.4 Hz), 3.35–3.53 (2H, m), 7.20–7.33 (5H, m); ^{13}C NMR (125 MHz, $CDCl_3$) δ 17.1, 20.9, 29.5, 35.2, 67.0, 81.7, 82.4, 126.3, 128.3, 128.4, 140.7; IR ($CHCl_3$) ν 3565, 3015, 2935, 2875, 1602 cm^{-1}. HRMS (FAB) *m*/*z* calcd for $C_{13}H_{17}O$ $[M+H]^+$: 189.1279. Found: 189.1280.

General procedure for the preparation of pyrroles

$(Ph_3P)AuCl$ (1.9 mg, 0.0039 mmol) and AgOTf (1.0 mg, 0.0039 mmol) were added in this order to a solution of the 1-amino-3-alkyn-2-ols **3** (3.9 mmol) in toluene (10 mL, 0.4 M) at room temperature. The reaction mixture was stirred at room temperature until the complete consumption of **3** and then quenched with saturated aqueous NH_4Cl. The organic materials were extracted with Et_2O, and the combined organic extracts were washed with brine, dried over $MgSO_4$, and evaporated in vacuo. The residue was purified by column chromatography (silica gel, usually hexanes/EtOAc or hexanes) to give the pyrroles **4**.

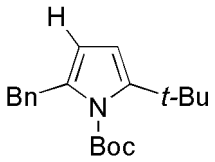
Spectroscopic data for pyrroles

***N*-Boc-4-methyl-2-(2-phenylethyl)pyrrole (4a)**



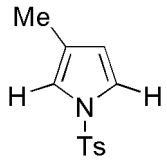
A colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.58 (9H, s), 2.01 (3H, s), 2.92 (2H, t, *J*= 8.0 Hz), 3.13 (2H, t, *J*= 8.0 Hz), 5.85 (1H, s), 6.95 (1H, s), 7.17–7.31 (5H, m); ^{13}C NMR (125 MHz, $CDCl_3$) δ 11.7, 28.0, 30.6, 35.3, 82.8, 113.4, 117.9, 120.3, 125.8, 128.2, 128.4, 135.7, 141.9, 149.3; IR ($CHCl_3$) ν 3010, 2980, 2930, 1730 cm^{-1}. HRMS (FAB) *m*/*z* calcd for $C_{18}H_{23}NO_2$ $[M]^+$: 285.1714. Found: 285.1706.

***N*-Boc-2-benzyl-5-*tert*-butylpyrrole (4b)**



A white solid, mp 64–67 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.39 (9H, s), 1.42 (9H, s), 4.06 (2H, s), 5.62 (1H, d, J= 3.5 Hz), 5.92 (1H, d, J= 3.5 Hz), 7.11–7.31 (5H, m); ^{13}C NMR (125 MHz, $CDCl_3$) δ 27.4, 30.6, 33.0, 35.0, 84.0, 107.1, 109.7, 126.1, 128.3, 128.6, 132.6, 139.6, 144.1, 151.8; IR ($CHCl_3$) ν 3020, 2970, 1740 cm^{-1}. *Anal.* Calcd for $C_{20}H_{27}NO_2$: C, 76.64; H, 8.68; N, 4.47. Found: C, 76.31; H, 8.78; N, 4.42.

***N*-Ts-3-methylpyrrole (4c)**

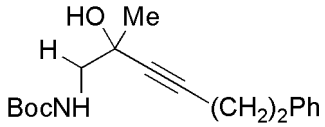


A white solid, mp 67–68 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.01 (3H, s), 2.39 (3H, s), 6.10–6.12 (1H, m), 6.87 (1H, s), 7.04–7.06 (1H, m), 7.27 (2H, d, *J*= 8.5 Hz), 7.72 (2H, d, *J*= 8.5 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 11.8, 21.6, 115.8, 117.8, 120.8, 124.5, 126.7, 129.9, 136.3, 144.7; IR ($CHCl_3$) ν 3140, 3030, 2930, 1595 cm^{-1}. *Anal.* Calcd for $C_{12}H_{13}NO_2S$: C, 61.25; H, 5.57; N, 5.95. Found: C, 61.49; H, 5.73; N, 5.74.

Spectroscopic data for 1-amino-3-alkyn-2-ols

***N*-Boc-1-amino-2-methyl-6-phenyl-3-hexyn-2-ol (3a)**

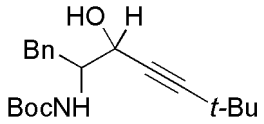
To a solution of 4-phenyl-1-butyne (1.80 g, 13.8 mmol) in THF (11.2 mL) was added dropwise ethylmagnesium bromide (1.0 M in THF; 13.8 mL, 13.8 mmol) at 0 °C. The mixture was refluxed for 10 min and then cooled to room temperature. After this mixture was diluted with CH_2Cl_2 (25.0 mL), *N*-Boc-1-amino-2-propanone[11] (1.09 g, 6.29 mmol) in CH_2Cl_2 (5.0 mL) at -20 °C was slowly added via a canula. The mixture was stirred at room temperature for 2 h, quenched with saturated aqueous NH_4Cl, and extracted with EtOAc. The combined organic extracts were washed with brine, dried over $MgSO_4$, and evaporated in vacuo. The residue was purified by column chromatography (silica gel, hexanes/EtOAc= 3:1) to give **3a** (1.80 g, 94%).



A colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.40 (3H, s), 1.46 (9H, s), 2.49 (2H, t, *J*= 7.4 Hz), 2.81 (2H, t, *J*= 7.4 Hz), 2.88 (1H, br s), 3.17 (1H, dd, *J*= 5.7, 13.7 Hz), 3.29 (1H, dd, *J*= 6.9, 13.7 Hz), 4.86 (1H, br s), 7.19–7.33 (5H, m); ^{13}C NMR (125 MHz, $CDCl_3$) δ 20.8, 27.2, 28.3, 34.8, 51.4, 68.2, 79.8, 83.0, 83.8, 126.4, 128.3, 128.5, 140.5, 156.8; IR ($CHCl_3$) ν 3600, 3455, 3015, 2980, 2930, 2235, 1710cm^{-1}. ***Anal.*** Calcd for $C_{18}H_{25}NO_3$: C, 71.26; H, 8.31; N, 4.62. Found: C, 70.91; H, 8.19; N, 4.57. HRMS (FAB) *m*/*z* calcd for $C_{18}H_{26}NO_3$ $[M+H]^+$: 304.1879. Found: 304.1927.

***N*-Boc-2-amino-6,6-dimethyl-1-phenyl-4-heptyn-3-ol (3b)**

50% yield: Prepared from *N*-Boc-phenylalaninal and 3,3-dimethyl-1butyne, according to the method for the synthesis of **3a**.

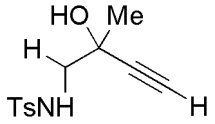


(mixture of *syn* and *anti*) A colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.22 and 1.27 (9H, s), 1.40 (9H, s), 2.40–3.15 (3H, m), 3.80–4.10 (1H, m), 4.25–4.45 (1H, m), 4.50–4.85 (1H, m), 7.15–7.33 (5H, m); IR ($CHCl_3$) ν 3610, 3440, 3015, 2970, 2235, 1705 cm^{-1}. ***Anal.*** Calcd for $C_{20}H_{29}NO_3$: C, 72.47; H, 8.82; N, 4.23. Found: C, 72.12; H, 8.61; N, 4.27. HRMS (FAB) *m*/*z* calcd for $C_{20}H_{30}NO_3$ $[M+H]^+$: 332.2271. Found: 332.2203.

***N*-Ts-1-amino-2-methyl-3-butyn-2-ol (3c)**

To a solution of ethynyltrimethylsilane (300 mg, 3.05 mmol) in THF (11 mL) was added dropwise *n*-BuLi (1.65 M in hexane; 1.80 mL, 3.05 mmol) at -78 °C. After the mixture was stirred for 1.5 h at the same temperature, *N*-Ts-1-amino-2-propanone (284 mg, 1.30 mmol) was added. The reaction mixture was allowed to get room temperature, and stirred for 3.5 h. The reaction mixture was quenched with saturated aqueous NH_4Cl, and extracted with EtOAc. The combined organic extracts were washed with brine, dried over $MgSO_4$, and evaporated in vacuo. The residue was purified by column chromatography (silica gel, hexanes/EtOAc= 2:1).

To a solution of the obtained compound (340 mg) in MeOH (5.5 mL) was added K_2CO_3 (219 mg, 1.6 mmol). The mixture was stirred at room temperature for 3 h, and evaporated in vacuo. To the residue were added EtOAc and saturated aqueous NH_4Cl. The organic layers were washed with brine, dried over $MgSO_4$, and evaporated in vacuo. The residue was purified by column chromatography (silica gel, hexanes/EtOAc= 3:2) to give **3c** (258 mg, 98%).



A colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.47 (3H, s), 2.42 (3H, s), 2.46 (1H, br s), 2.95 (1H, br s), 3.03–3.12 (2H, m), 5.15–5.25 (1H, m), 7.31 (2H, d, *J*= 8.0 Hz), 7.76 (2H, d, *J*= 8.0 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 21.5, 26.6, 53.2, 66.8, 73.1, 84.9, 127.1, 129.8, 136.6, 143.8; IR ($CHCl_3$) ν 3600, 3385, 3305, 3030, 2990, 2255, 1600 cm^{-1}.

Procedure for the larger-scale preparation of furan (Scheme 2)

A solution of $(Ph_3P)AuCl$ (31.5 mg, 0.064 mmol) and $AgNTf_2$ (24.7 mg, 0.064 mmol) in toluene (35 mL) was stirred at room temperature for 5 min. After a solution of **1a** (26.0 g, 127 mmol) in toluene (125 mL) was added, the reaction mixture was stirred at room temperature for 2 h and then quenched with saturated aqueous NH_4Cl. The organic materials were extracted with Et_2O, and the combined organic extracts were washed with brine, dried over $MgSO_4$, and evaporated in vacuo. The residue was purified by column chromatography (silica gel, hexanes) to give **2a** (23.2 g, 98%).

References

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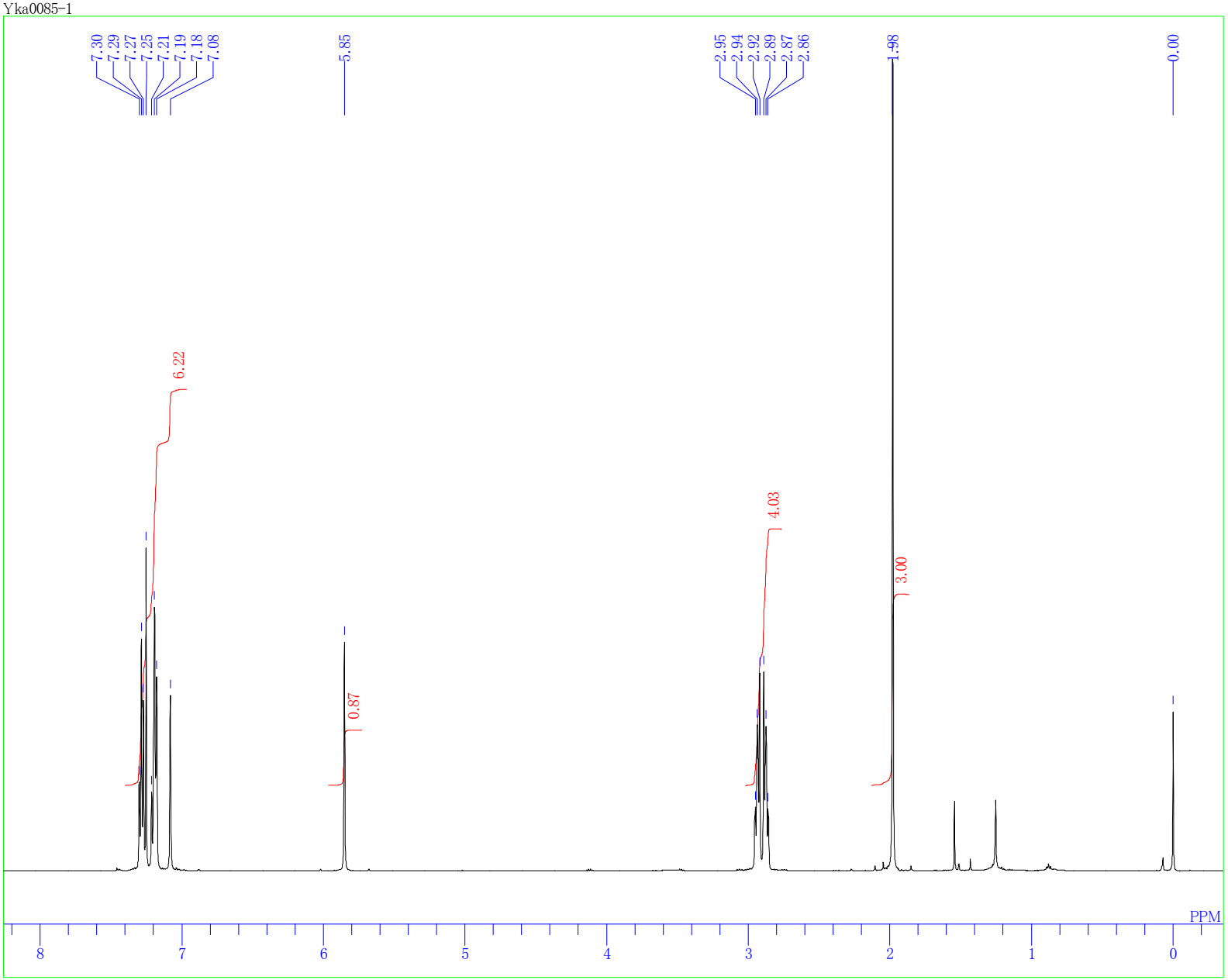
F:¥furan¥NMR (furan)¥4-Methyl-2-(2-phenylethyl)furan (H).als
Yka0085-1



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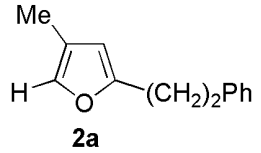
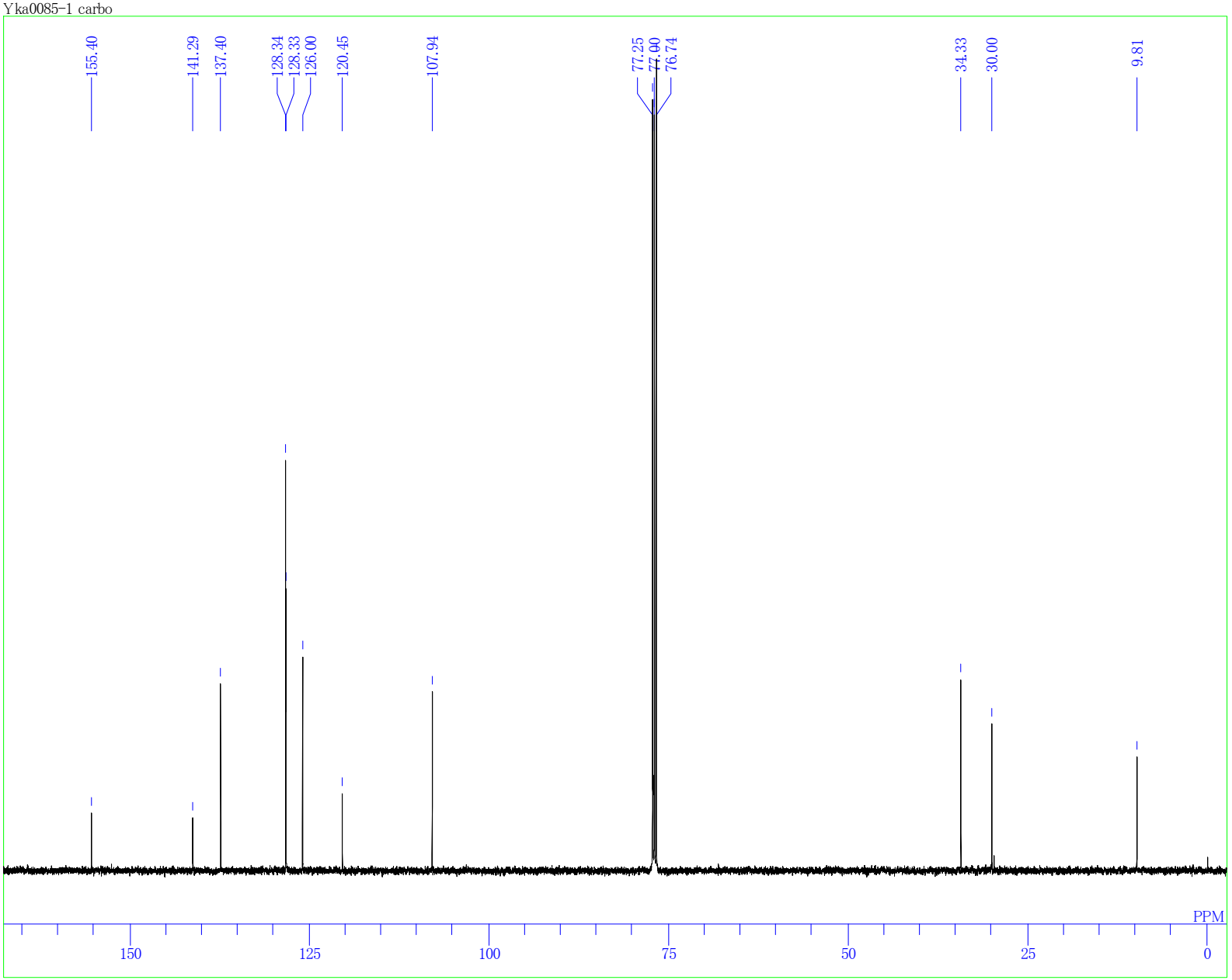


2a

F:¥furan¥NMR (furan)¥4-Methyl-2-(2-phenylethyl)furan (C).als
Yka0085-1 carbo



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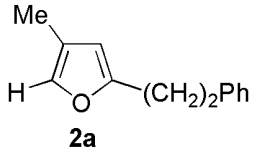
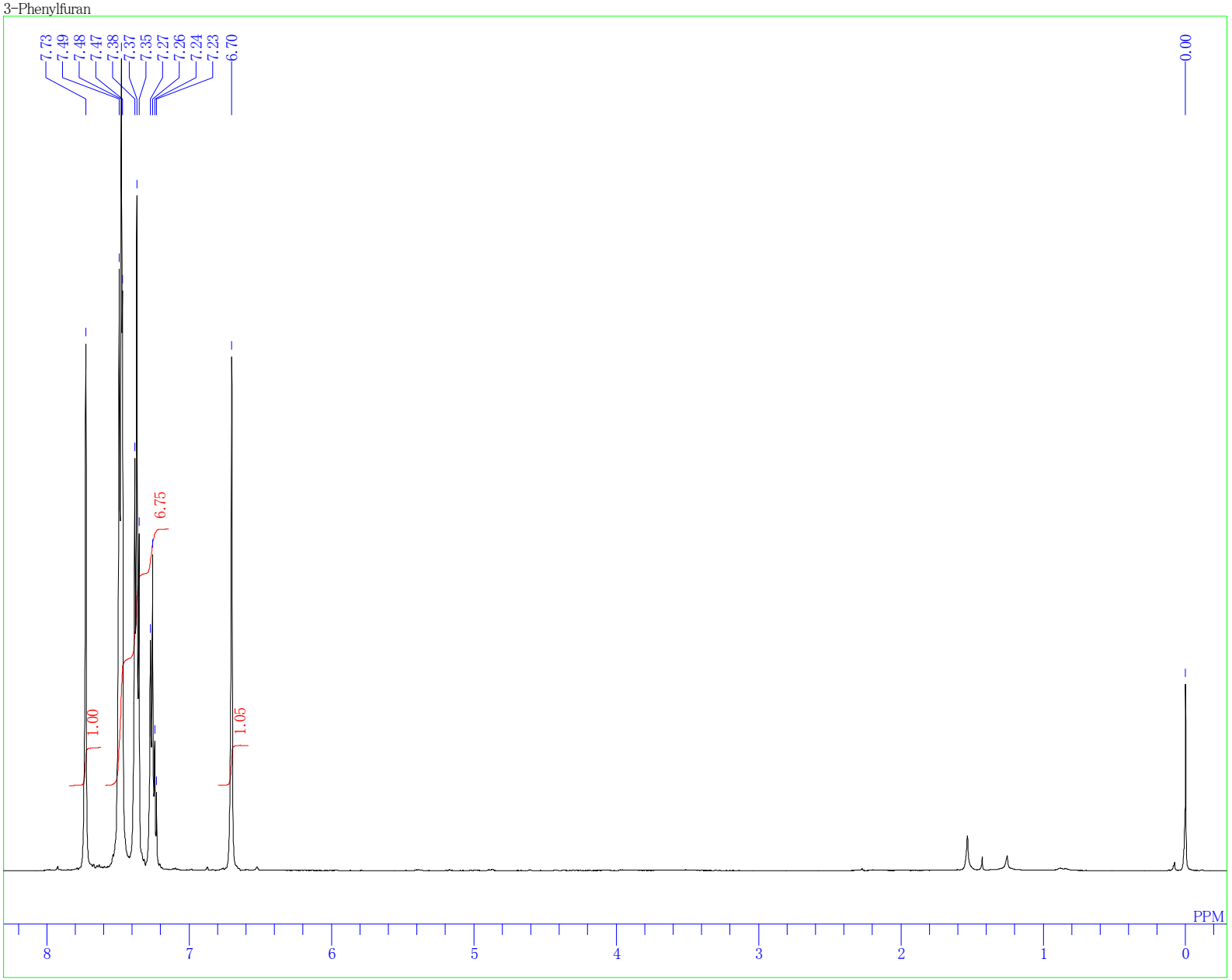


2a

F:¥furan¥NMR (furan)¥3-phenylfuran (H).als
3-Phenylfuran



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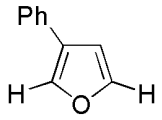
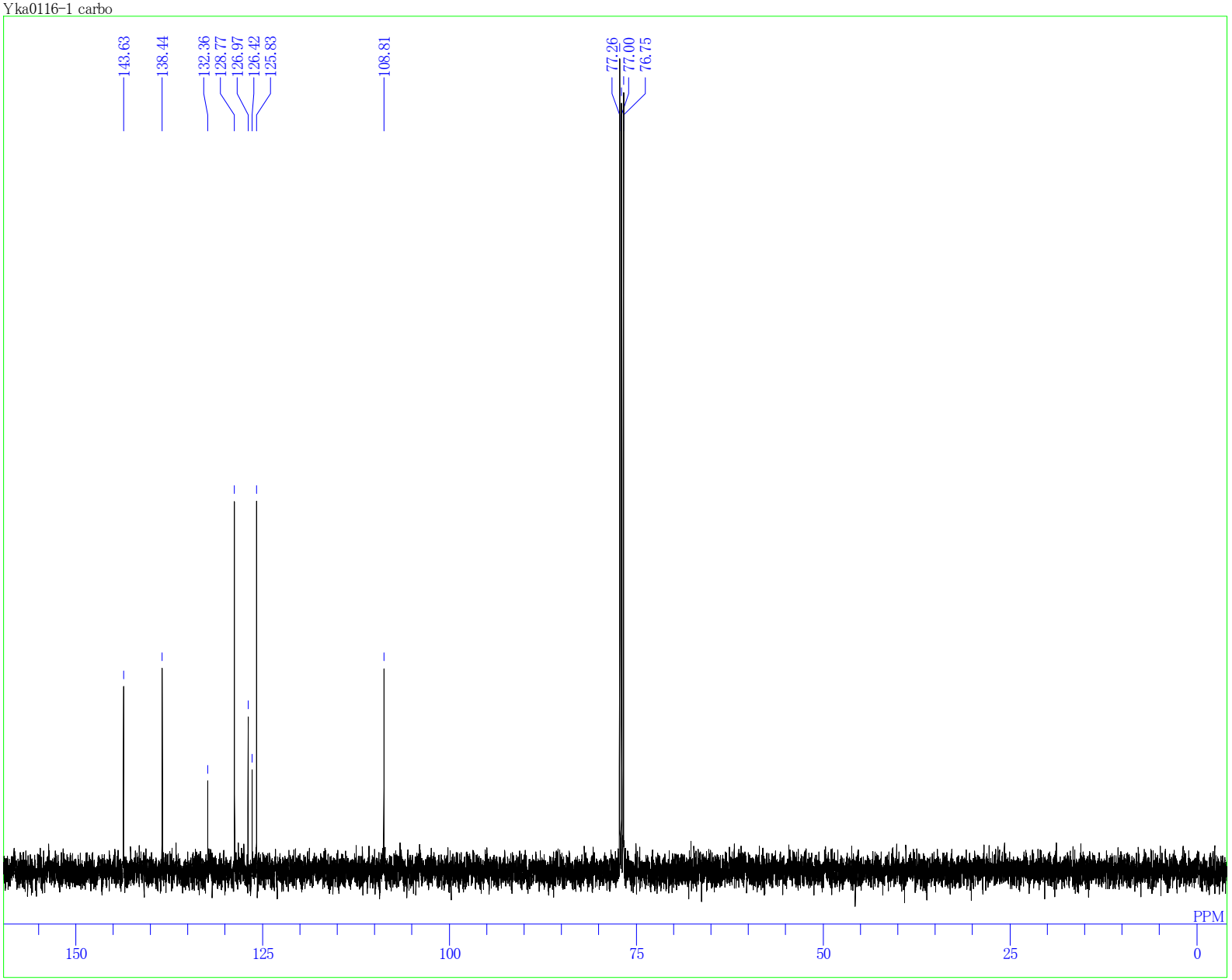


2b

F:¥furan¥NMR (furan)¥3-phenylfuran (C).als
Yka0116-1 carbo



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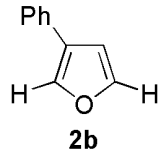
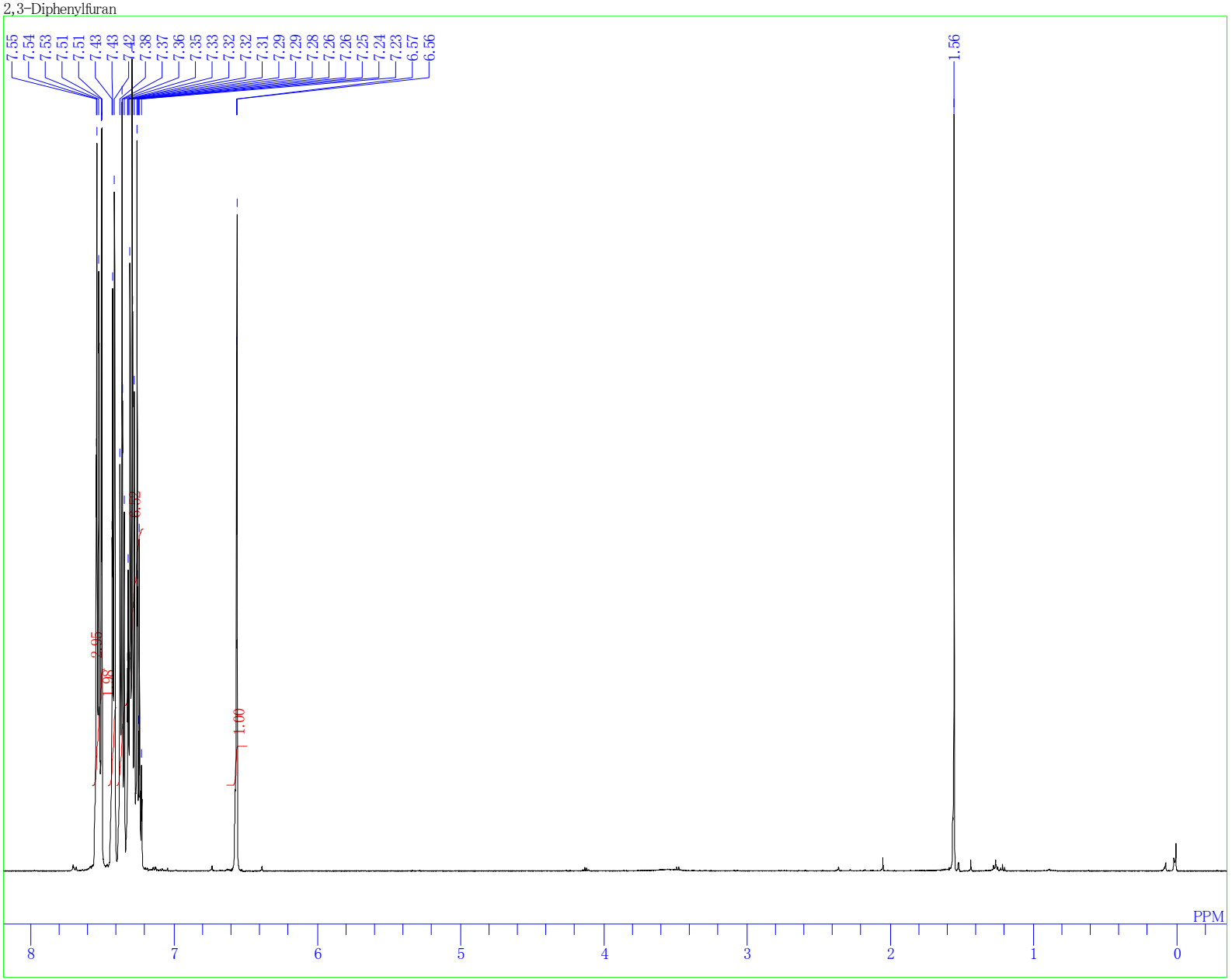


2b

F:¥furan¥NMR (furan)¥Yka0229-1.als
2,3-Diphenylfuran



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SPO	16384
SCANS	8
FREQU	9384.38 Hz
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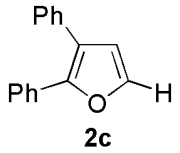
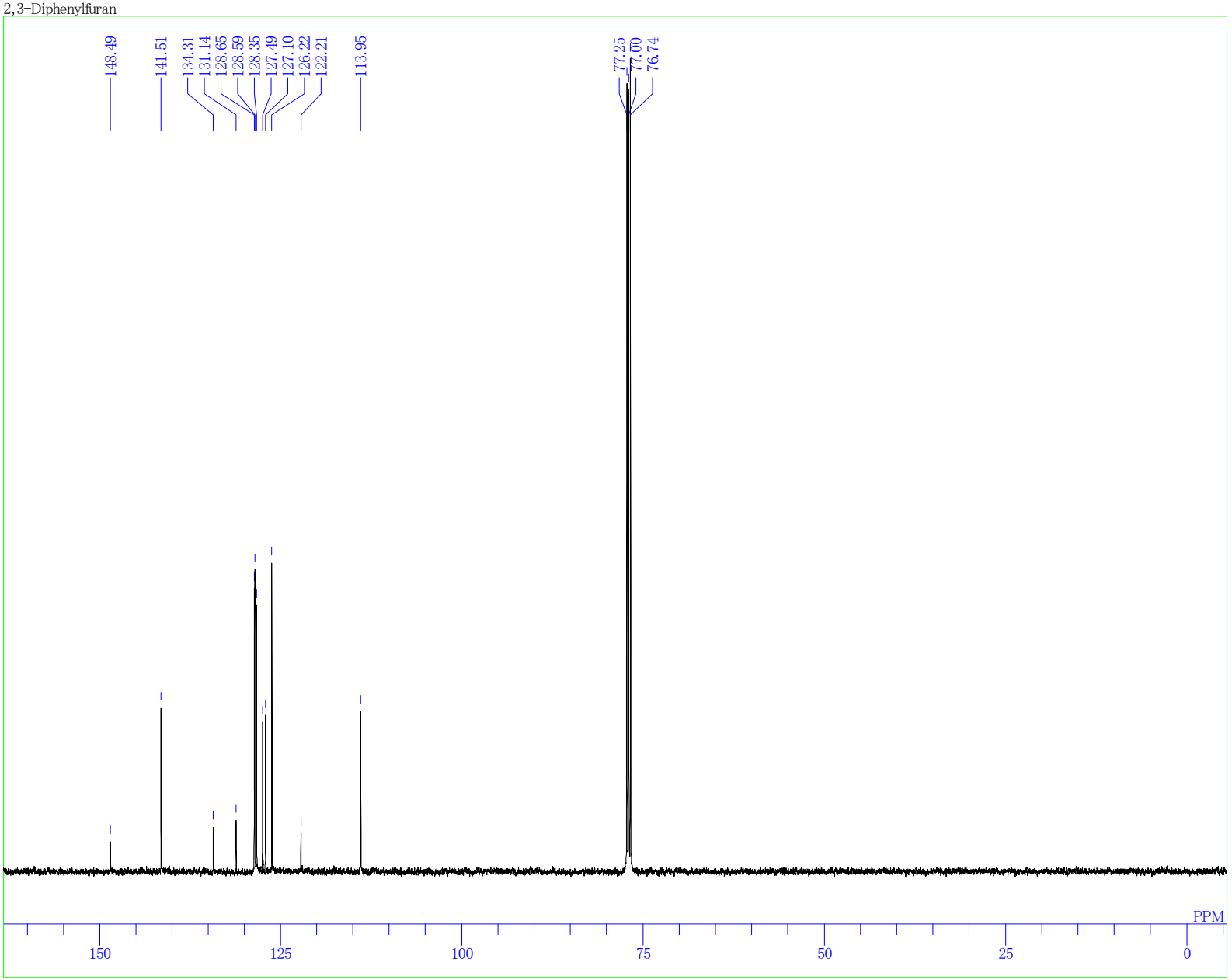


2c

F:¥furan¥NMR (furan)¥Yka0229-1 (13C).als
2,3-Diphenylfuran



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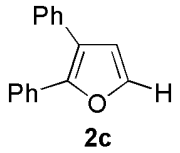
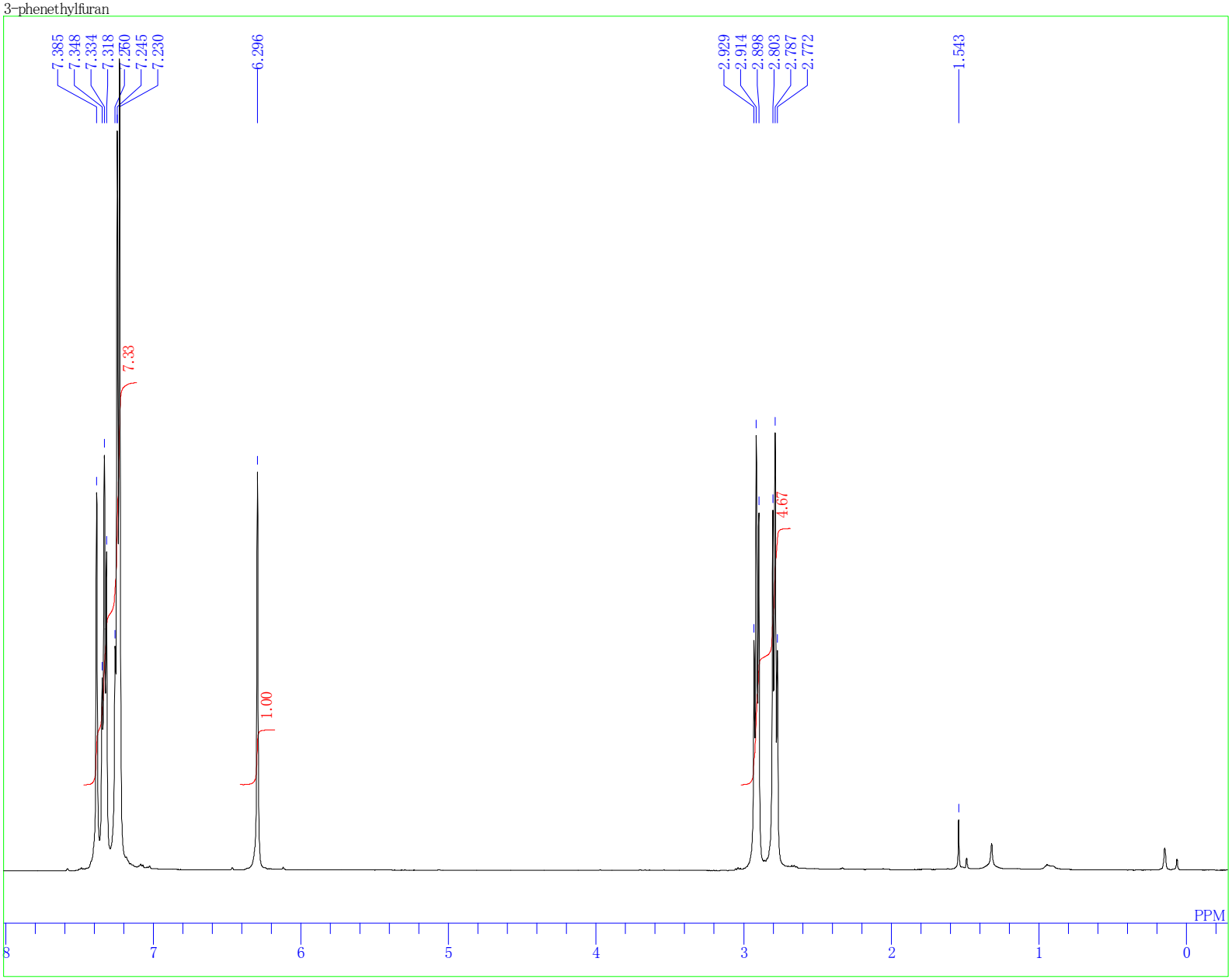
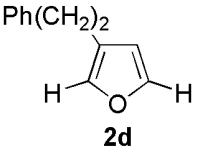


2c

F:¥Yka0264-1.als
3-phenethylfuran

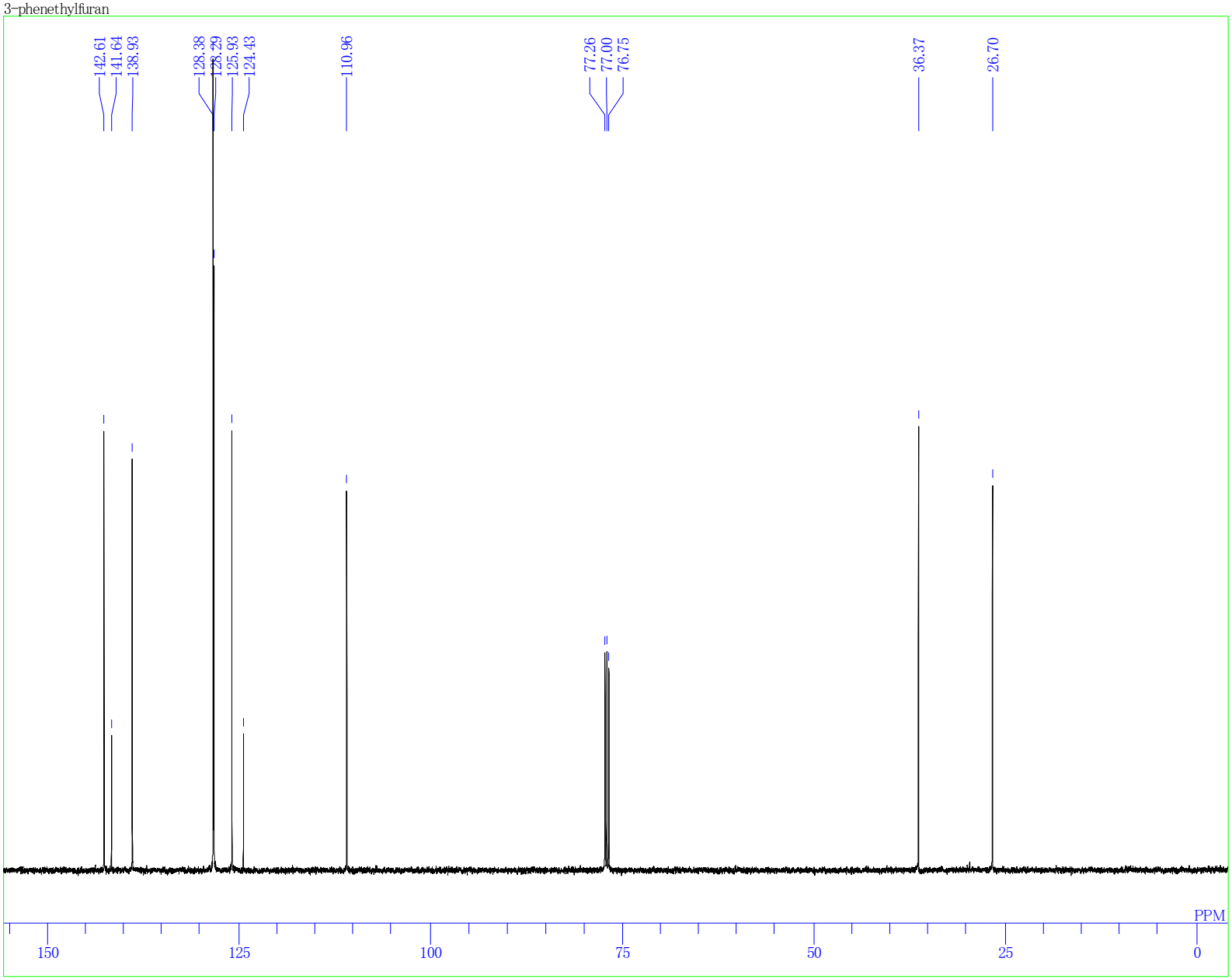


DATIM	09-06-2009 15:19:21
EXMOD	single_pulse.ex2
OBNUC	1H
OFR	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
OBATN	0
PW1	4.50 usec
PW2	0.00 usec
POINT	16384
SPO	16384
SCANS	8
FREQU	9384.38 Hz
PD	1.5000 sec
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	7.26 ppm
TMSP	4577

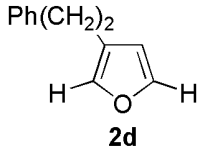


2d

F:¥Yka0264-1 carbon.als
3-phenethylfuran

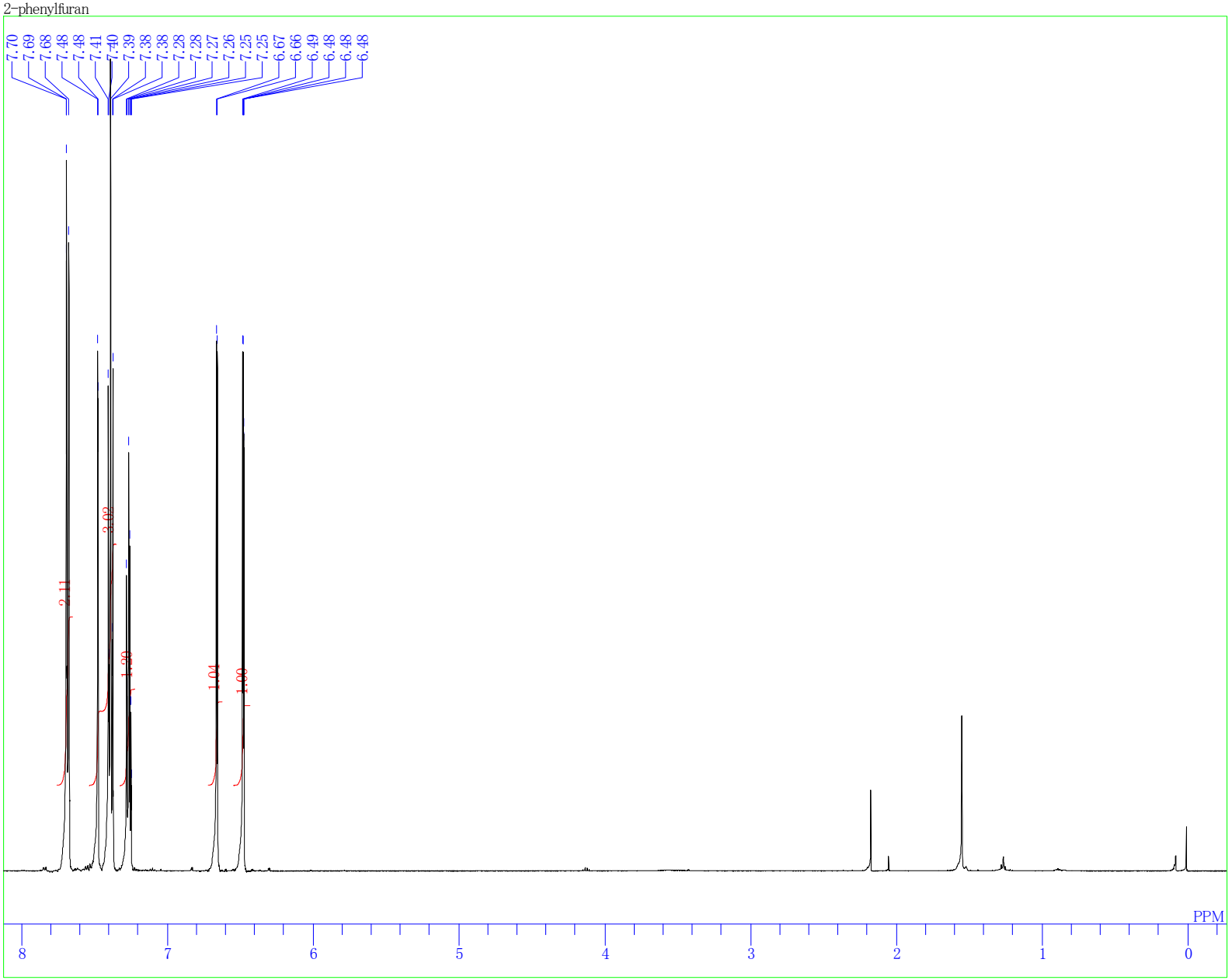


DATIM	09-06-2009 15:26:11
EXMOD	single_pulse_dec
OBNUC	13C
OFR	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
OBATN	0
PW1	3.27 usec
PW2	0.00 usec
POINT	32768
SPO	32768
SCANS	97
FREQU	39308.18 Hz
PD	2.0000 sec
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	77.00 ppm
TMSP	15498

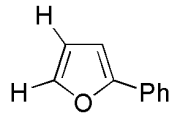


2d

F:¥furan¥NMR (furan)¥Yka0234-1.als
2-phenylfuran

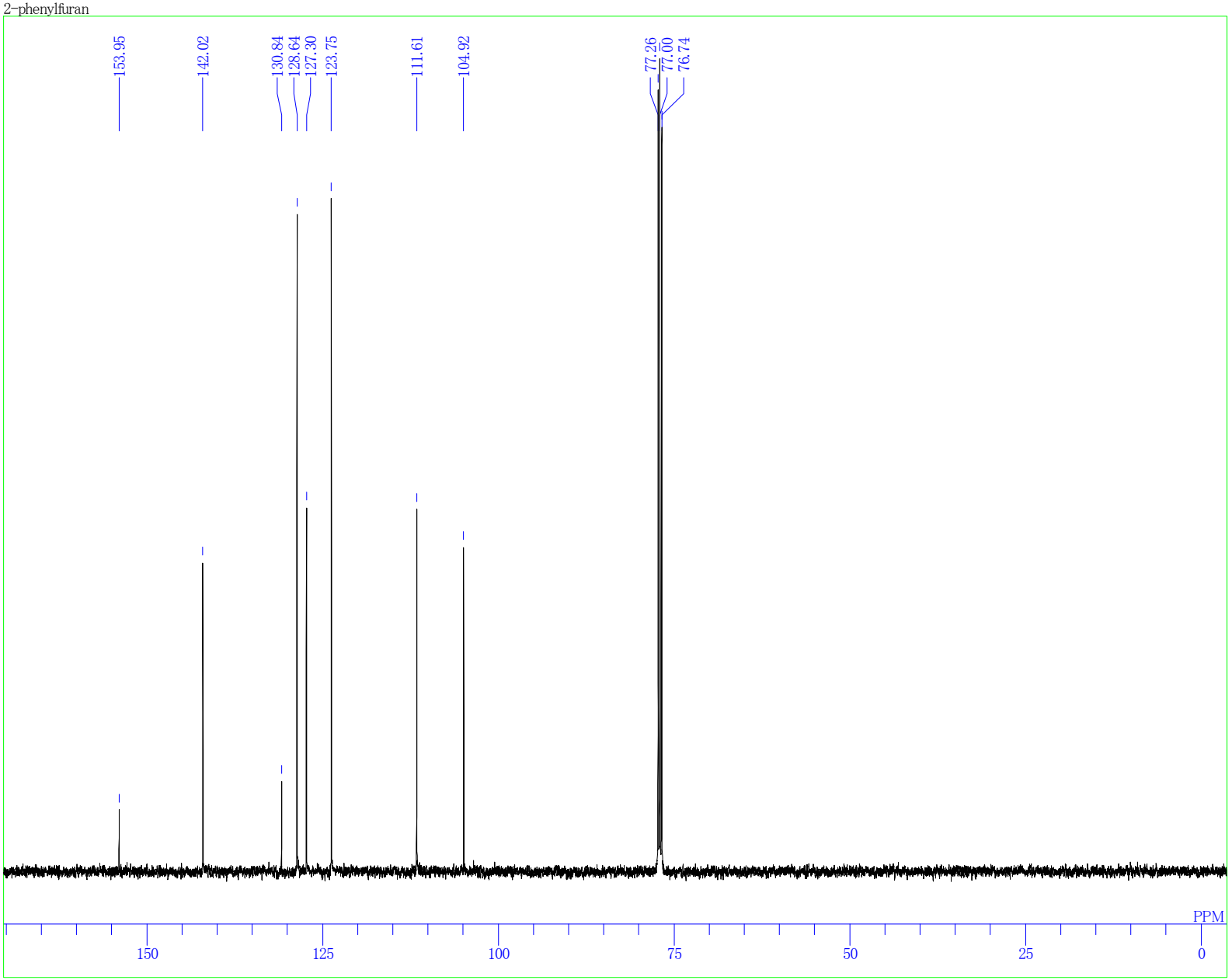


DATIM	05-11-2008 18:22:38
EXMOD	single_pulse.ex2
OBNUC	1H
OFR	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
OBATN	0
PW1	4.50 usec
PW2	0.00 usec
POINT	13107
SPO	13107
SCANS	8
FREQU	7507.39 Hz
PD	1.5000 sec
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	7.26 ppm
TMSP	4584

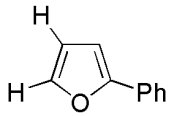


2e

F:¥furan¥NMR (furan)¥Yka0234-1 carbo.als
2-phenylfuran

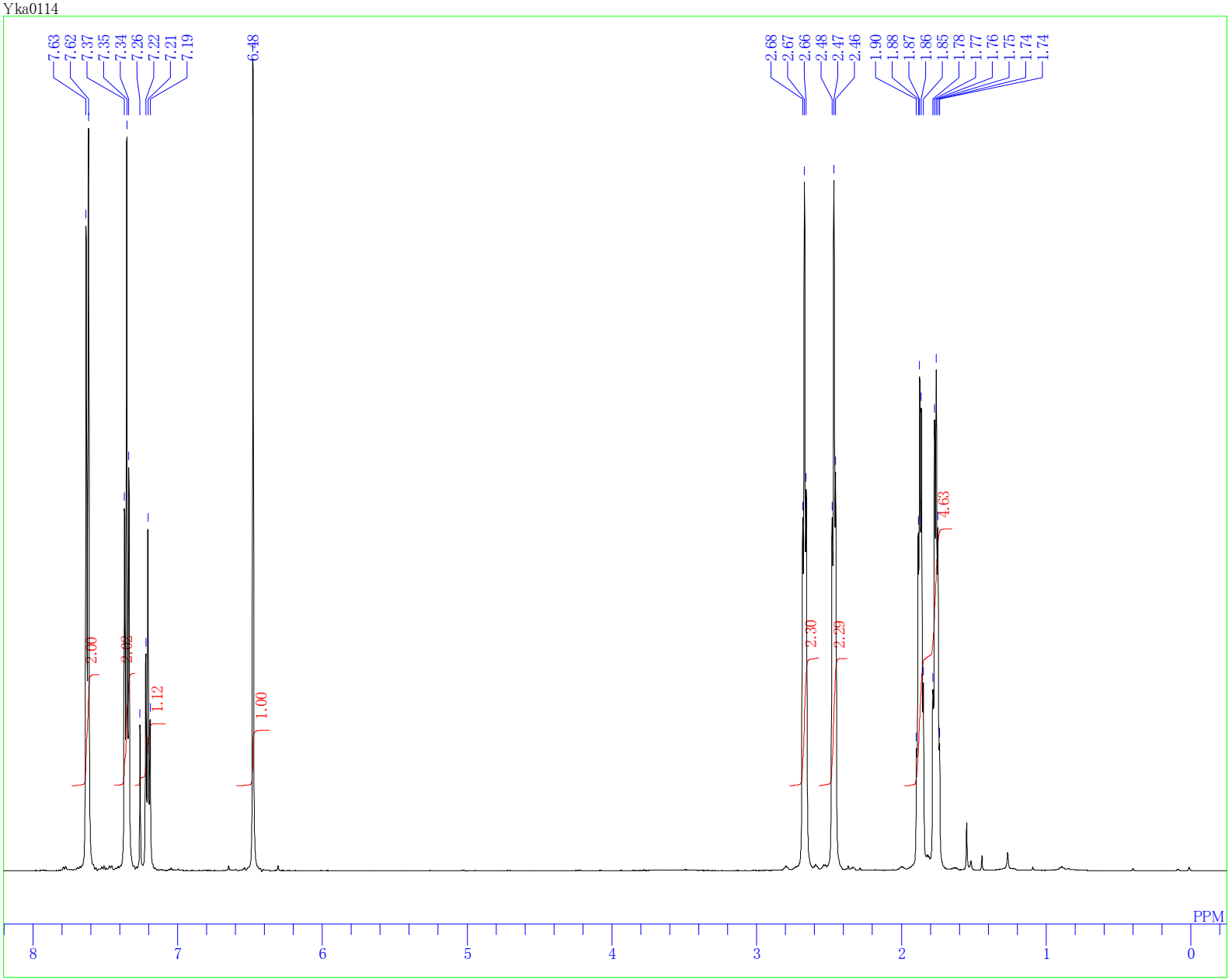


DATIM	05-11-2008 18:39:46
EXMOD	single_pulse_dec
OBNUC	13C
OFR	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
OBATN	0
PW1	3.27 usec
PW2	0.00 usec
POINT	32768
SPO	32768
SCANS	309
FREQU	39308.18 Hz
PD	2.0000 sec
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	77.00 ppm
TMSP	18782

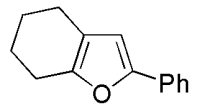


2e

F:¥furan¥NMR (furan)¥2-phenyl-4,5,6,7-tetrahydrobenzofuran (H).als
Yka0114

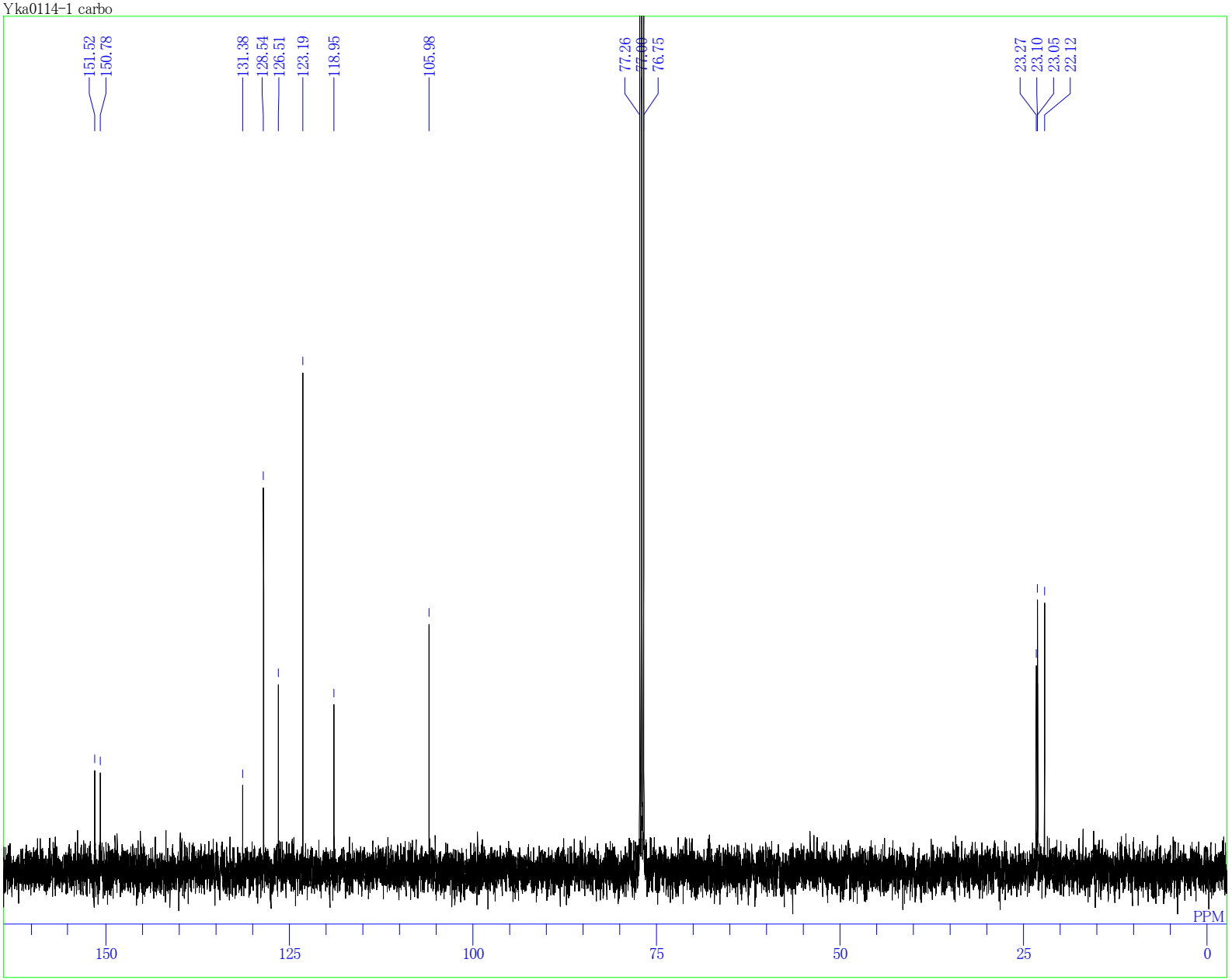


DATIM	05-01-2008 21:12:47
EXMOD	single_pulse.ex2
OBNUC	1H
OFR	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
OBATN	0
PW1	6.00 usec
PW2	0.00 usec
POINT	16384
SPO	16384
SCANS	8
FREQU	9384.38 Hz
PD	1.5000 sec
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	7.26 ppm
TMSP	6223

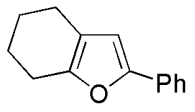


2f

F:¥furan¥NMR (furan)¥2-phenyl-4,5,6,7-tetrahydrobenzofuran (C).als
Yka0114-1 carbo

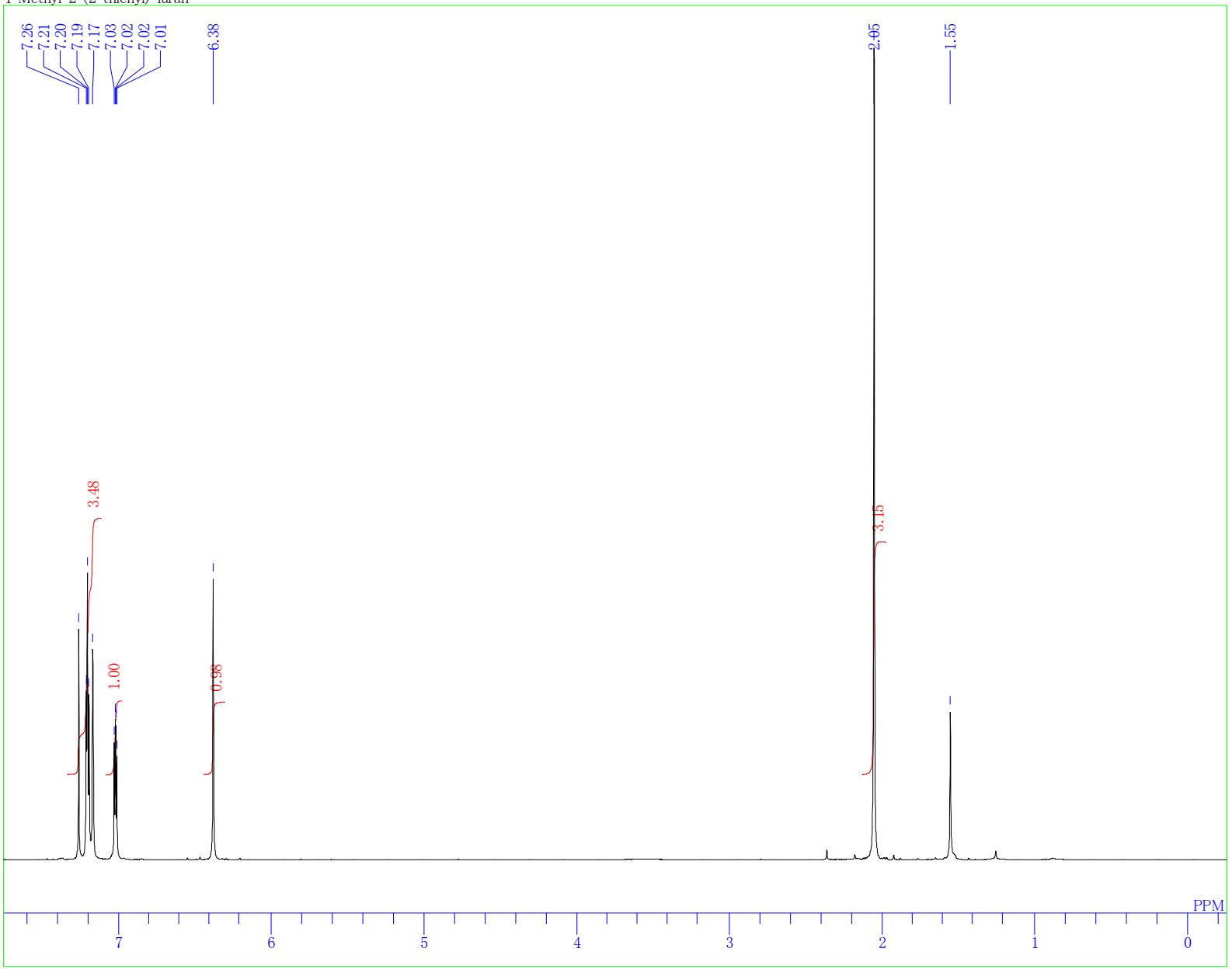


DATIM	05-01-2008 22:47:16
EXMOD	single_pulse_dec
OBNUC	13C
OFR	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
OBATN	0
PW1	3.67 usec
PW2	0.00 usec
POINT	32768
SPO	32768
SCANS	1937
FREQU	39308.18 Hz
PD	2.0000 sec
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	77.00 ppm
TMSP	18781

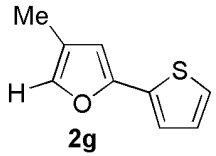


2f

F:¥furan¥NMR (furan)¥Yegi030194.als
4-Methyl-2-(2-thienyl)-furan

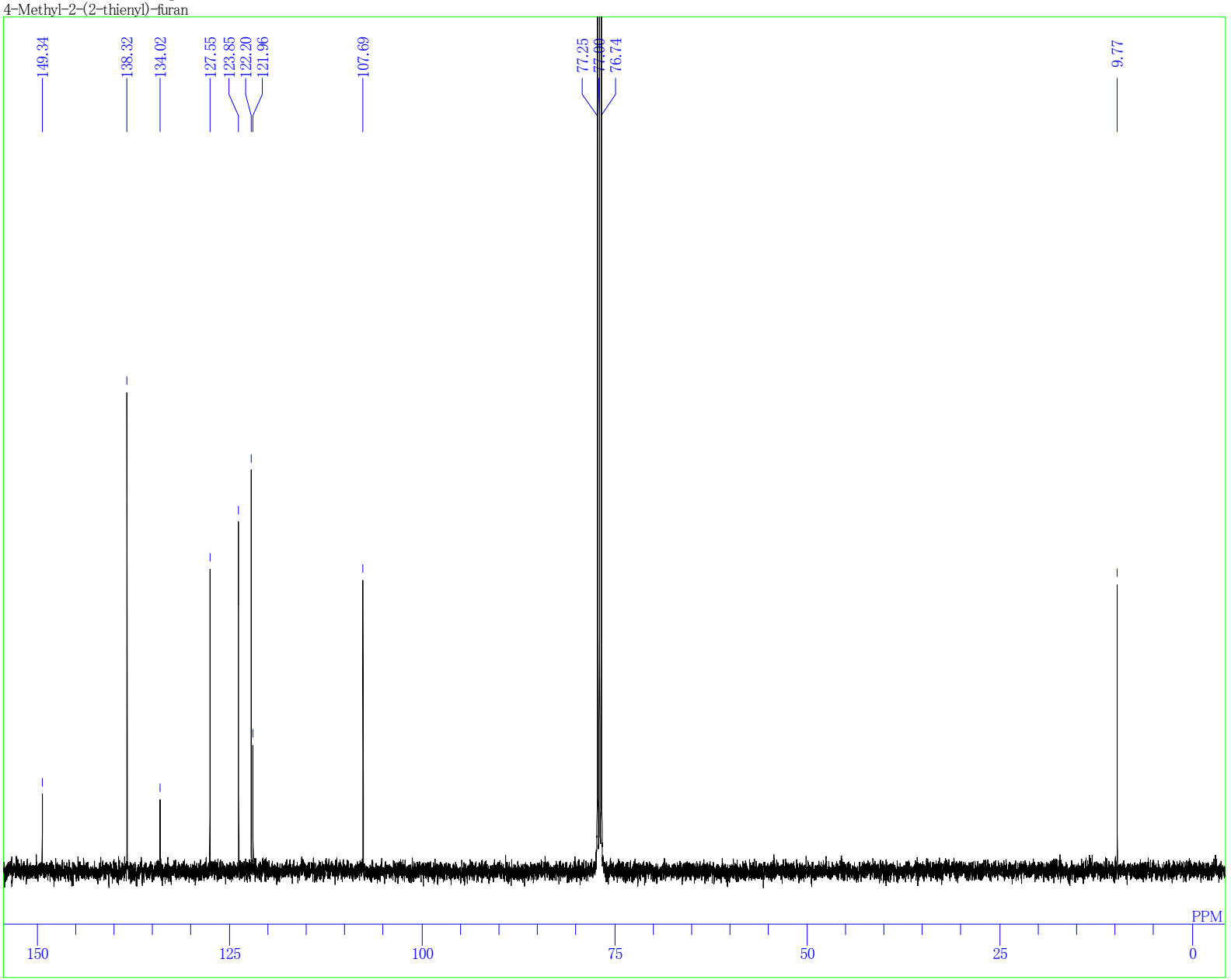


DATIM	01-10-2008 19:36:02
EXMOD	single_pulse.ex2
OBNUC	1H
OFR	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
OBATN	0
PW1	4.50 usec
PW2	0.00 usec
POINT	16384
SPO	16384
SCANS	8
FREQU	9384.38 Hz
PD	5.0000 sec
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	7.26 ppm
TMSP	6222

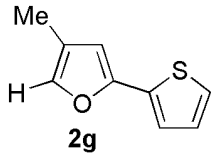


2g

F:¥furan¥NMR (furan)¥Yegi030194-13C.als
4-Methyl-2-(2-thienyl)-furan

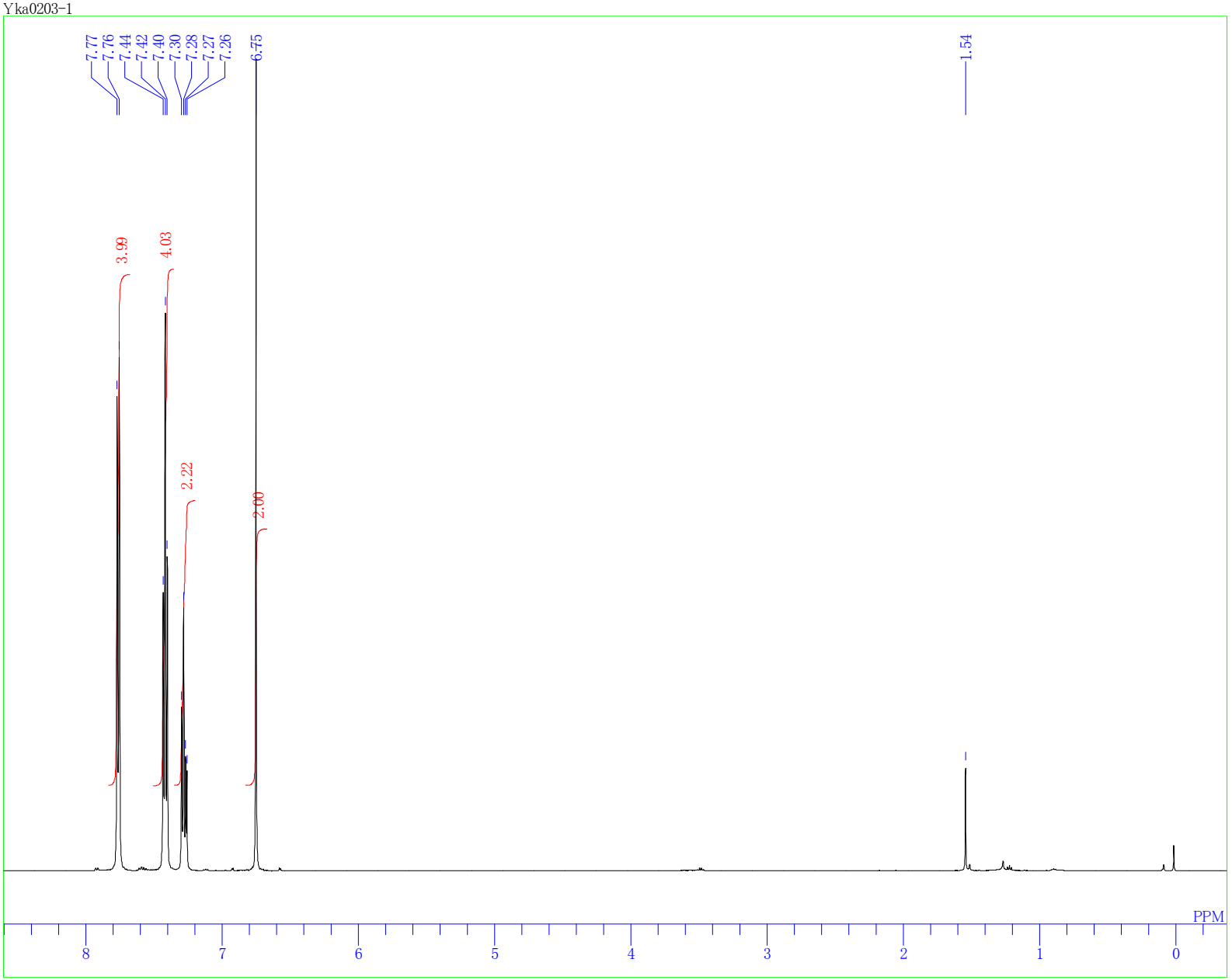


DATIM	01-10-2008 20:15:47
EXMOD	single_pulse_dec
OBNUC	13C
OFR	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
OBATN	0
PW1	3.27 usec
PW2	0.00 usec
POINT	32768
SPO	32768
SCANS	809
FREQU	39308.18 Hz
PD	2.0000 sec
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	77.00 ppm
TMSP	18783

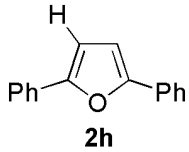


2g

F:¥furan¥NMR (furan)¥2,5-Diphenylfuran (H).als
Yka0203-1

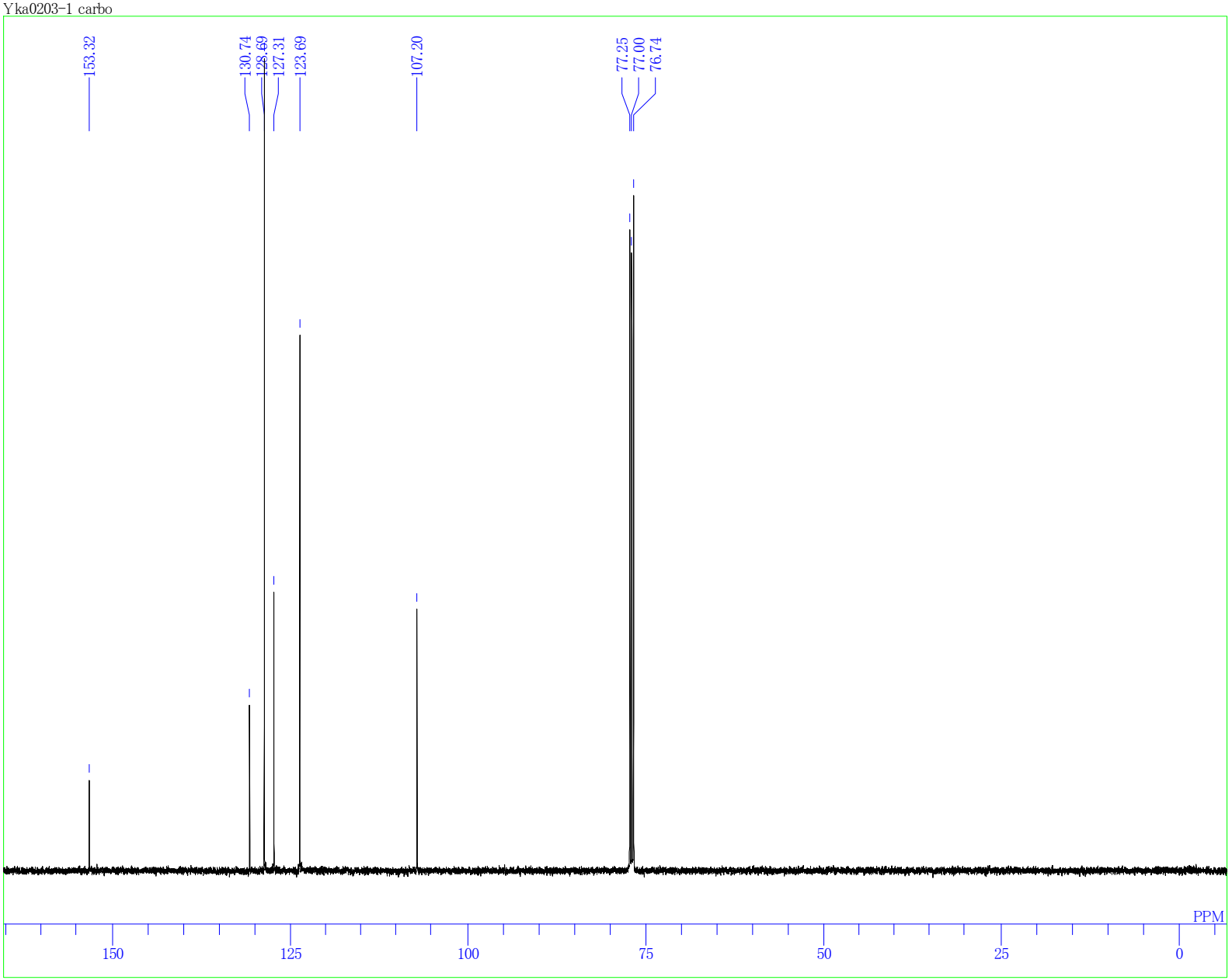


DATIM	01-08-2008 15:55:24
EXMOD	single_pulse.ex2
OBNUC	1H
OFR	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
OBATN	0
PW1	5.75 usec
PW2	0.00 usec
POINT	16384
SPO	16384
SCANS	8
FREQU	9384.38 Hz
PD	1.5000 sec
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	7.26 ppm
TMSP	6223

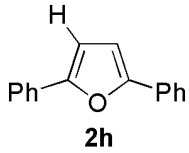


2h

F:¥furan¥NMR (furan)¥2,5-Diphenylfuran (C).als
Yka0203-1 carbo

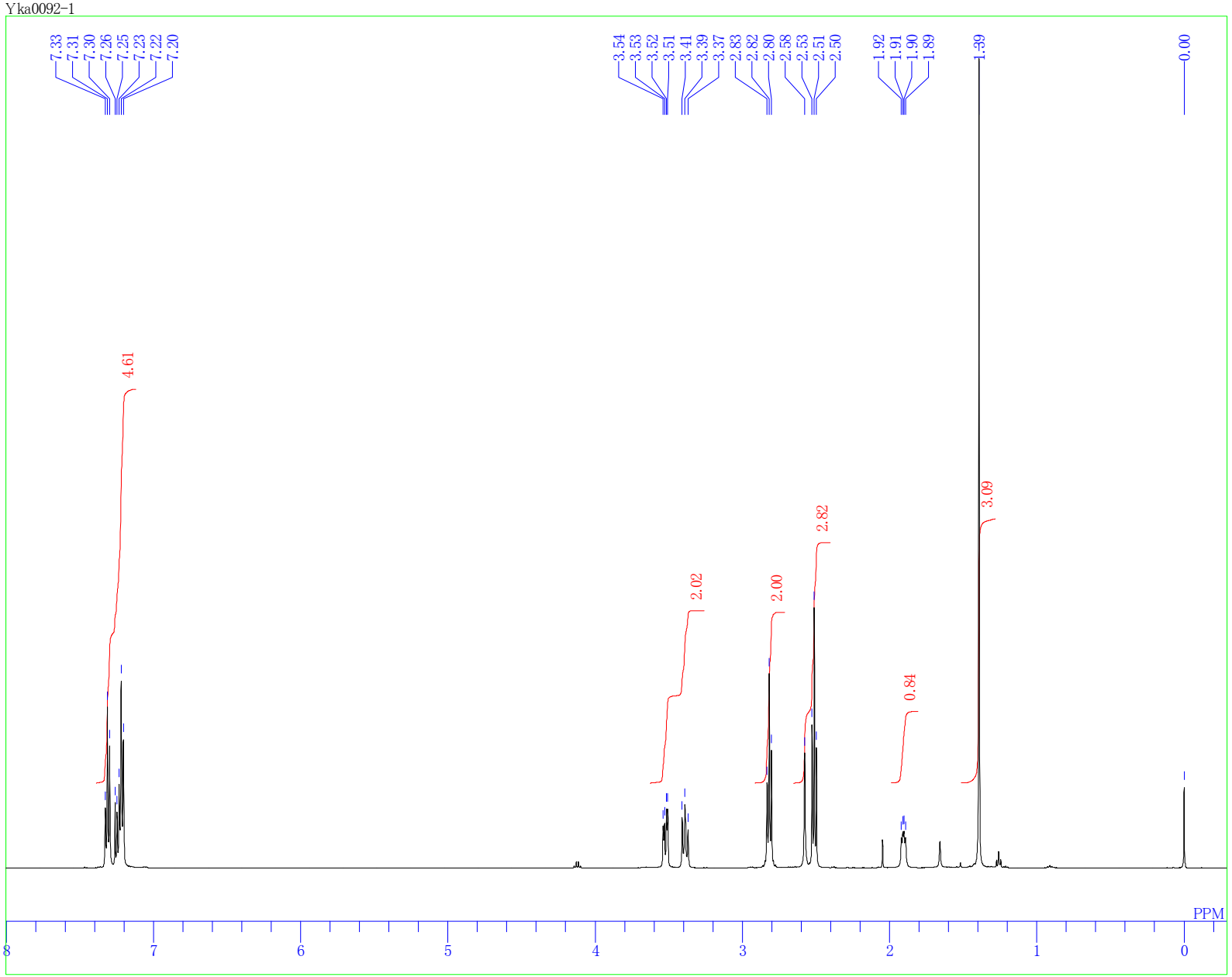


DATIM	01-08-2008 16:33:19
EXMOD	single_pulse_dec
OBNUC	13C
OFR	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
OBATN	0
PW1	3.67 usec
PW2	0.00 usec
POINT	32768
SPO	32768
SCANS	744
FREQU	39308.18 Hz
PD	2.0000 sec
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	77.00 ppm
TMSP	18782

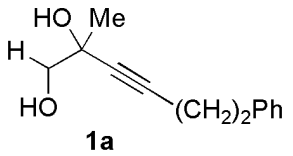


2h

F:¥furan¥NMR (furan)¥2-(4-Phenyl-1-butyn-1-yl)-1,2-propanediol (H).als
Yka0092-1

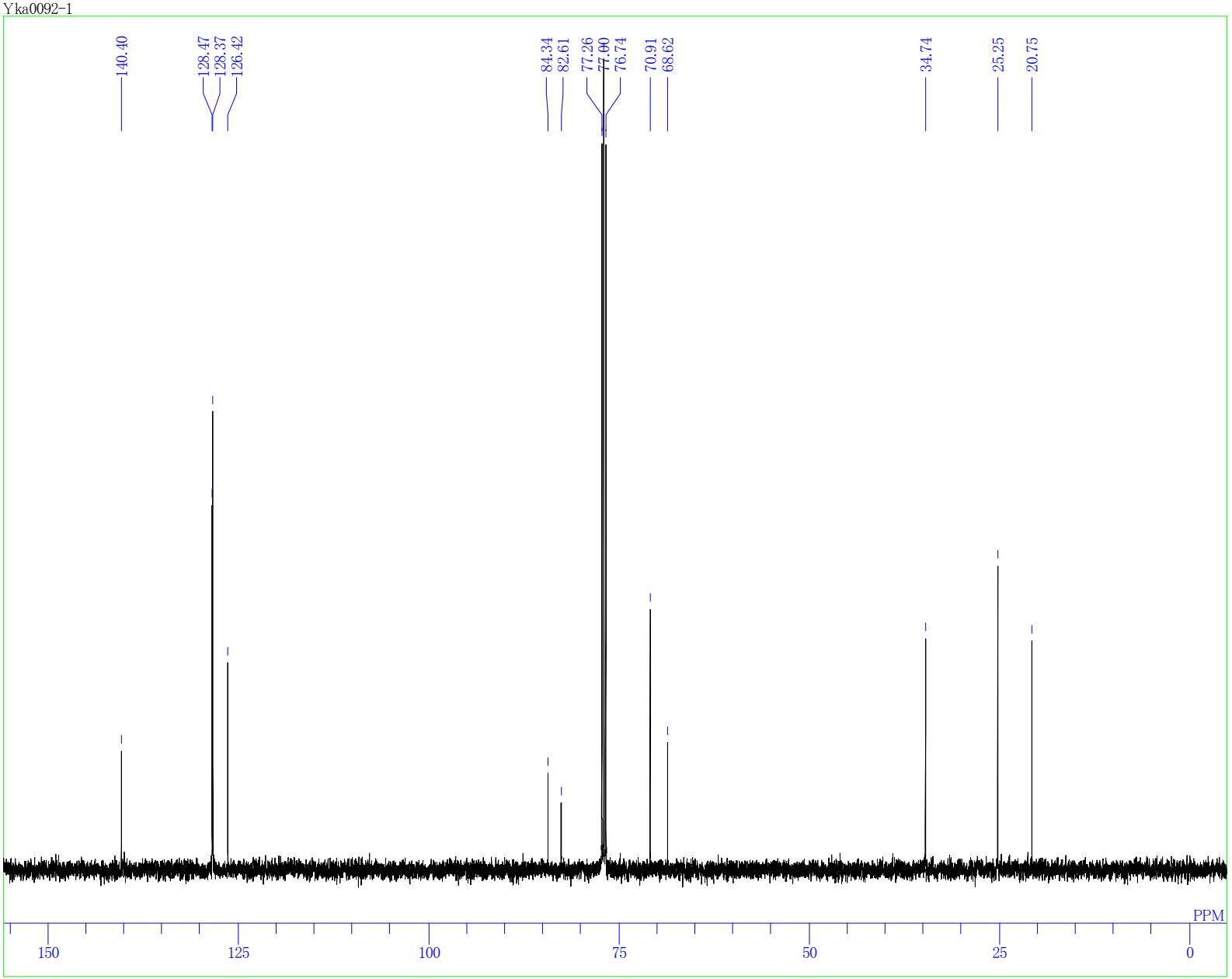


DATIM	03-12-2007 11:56:23
EXMOD	single_pulse.ex2
OBNUC	1H
OFR	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
OBATN	0
PW1	6.00 usec
PW2	0.00 usec
POINT	16384
SPO	16384
SCANS	8
FREQU	9384.38 Hz
PD	1.5000 sec
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	0.00 ppm
TMSP	12564

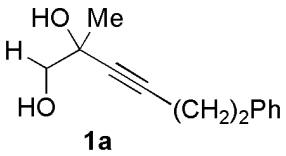


1a

F:¥furan¥NMR (furan)¥2-(4-Phenyl-1-butyn-1-yl)-1,2-propanediol (C).als
Yka0092-1

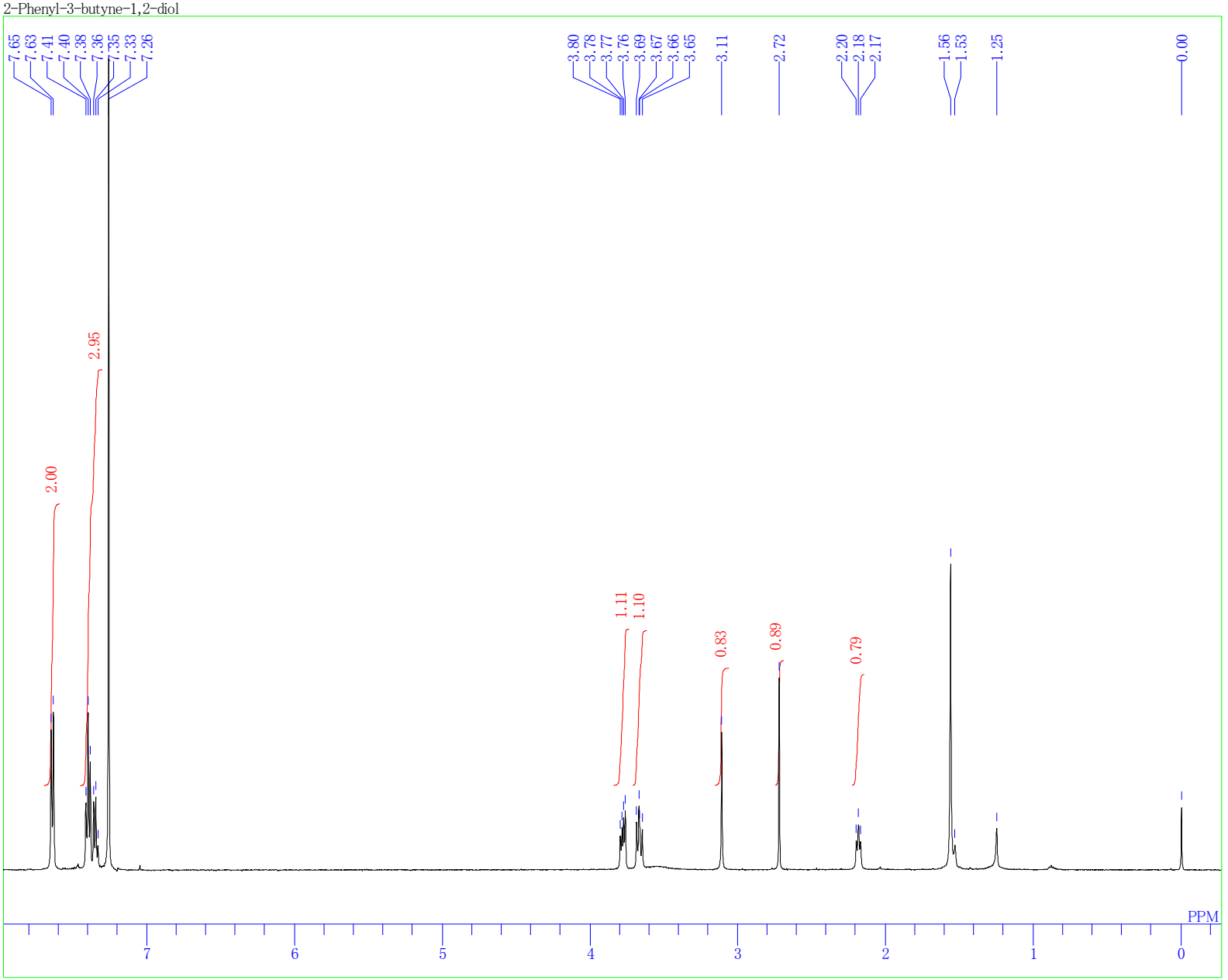


DATIM	03-12-2007 12:06:48
EXMOD	single_pulse_dec
OBNUC	13C
OFR	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
OBATN	0
PW1	3.67 usec
PW2	0.00 usec
POINT	32768
SPO	32768
SCANS	128
FREQU	39308.18 Hz
PD	2.0000 sec
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	77.00 ppm
TMSP	18781

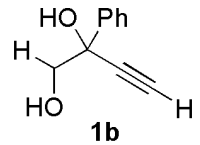


1a

F:¥furan¥NMR (furan)¥Yyy0525.als
2-Phenyl-3-butyne-1,2-diol

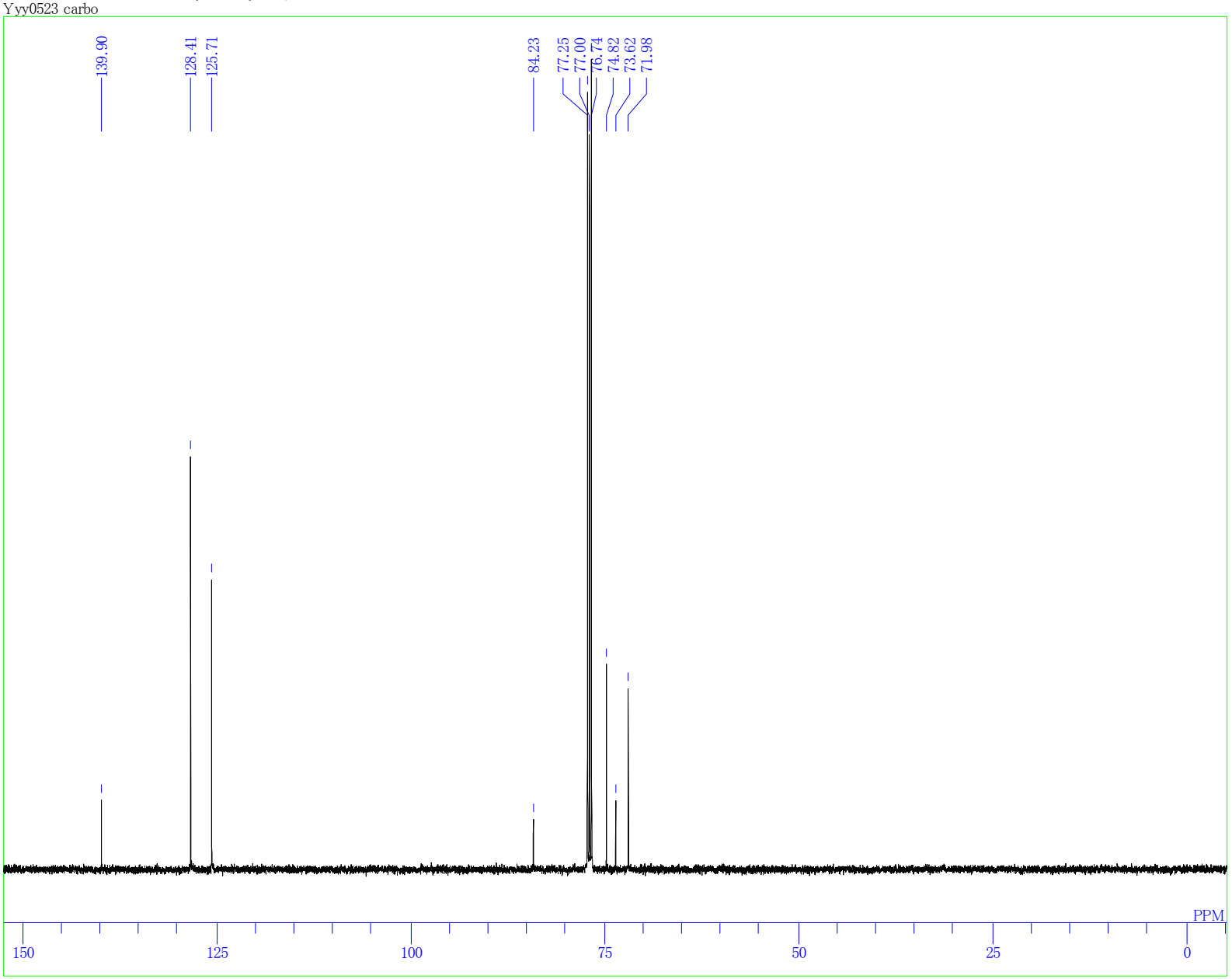


DATIM	20-11-2008 10:34:02
EXMOD	single_pulse.ex2
OBNUC	1H
OFR	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
OBATN	0
PW1	4.50 usec
PW2	0.00 usec
POINT	13107
SPO	13107
SCANS	8
FREQU	7507.39 Hz
PD	1.5000 sec
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	7.26 ppm
TMSP	4584

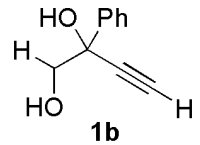


1b

F:¥furan¥NMR (furan)¥2-Phenyl-3-butyne-1,2-diol(C).als
Yyy0523 carbo

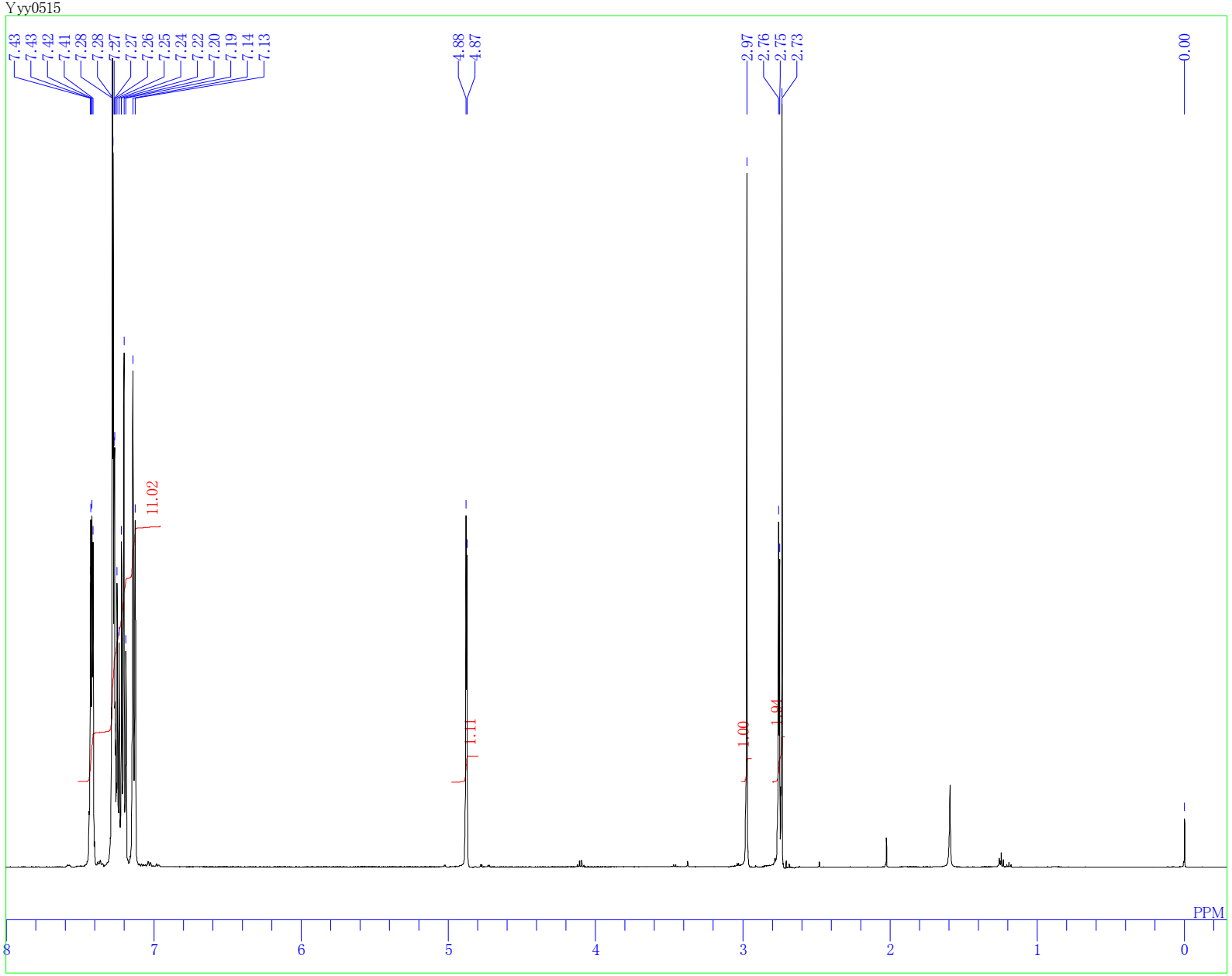


DATIM	03-10-2008 14:27:16
EXMOD	single_pulse_dec
OBNUC	13C
OFR	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
OBATN	0
PW1	3.27 usec
PW2	0.00 usec
POINT	32768
SPO	32768
SCANS	503
FREQU	39308.18 Hz
PD	2.0000 sec
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	77.00 ppm
TMSP	18782

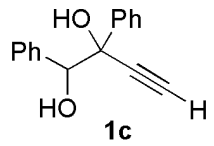


1b

F:¥furan¥NMR (furan)¥1,2-Diphenyl-3-butyne-1,2-diol (H).als
Yyy0515

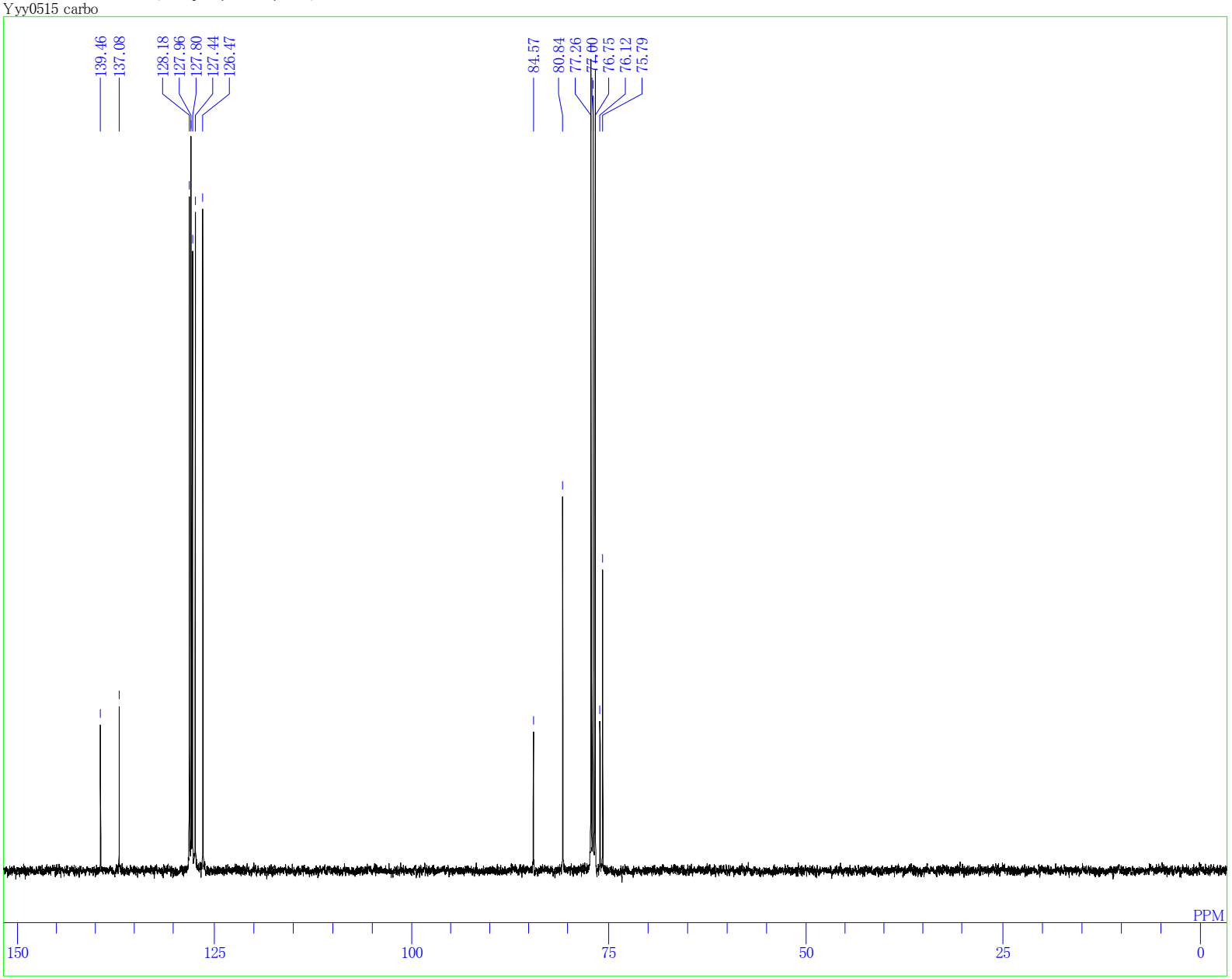


DATIM	03-10-2008 13:39:14
EXMOD	single_pulse.ex2
OBNUC	1H
OFR	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
OBATN	0
PW1	4.50 usec
PW2	0.00 usec
POINT	16384
SPO	16384
SCANS	8
FREQU	9384.38 Hz
PD	1.5000 sec
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	0.00 ppm
TMSP	12550

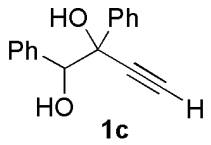


1c

F:¥furan¥NMR (furan)¥1,2-Diphenyl-3-butyne-1,2-diol (C).als
Yyy0515 carbo

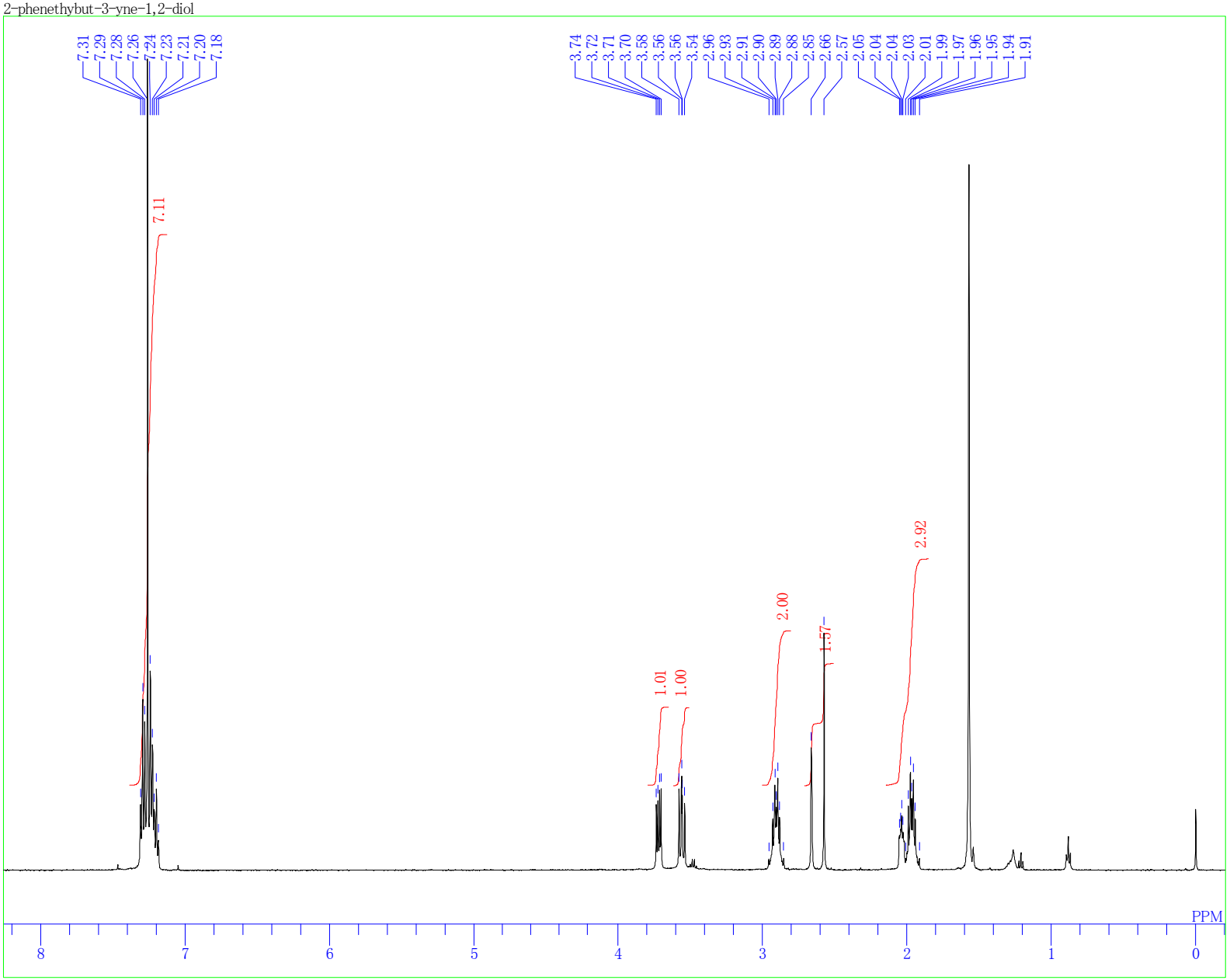


DATIM	03-10-2008 13:55:06
EXMOD	single_pulse_dec
OBNUC	13C
OFR	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
OBATN	0
PW1	3.27 usec
PW2	0.00 usec
POINT	26214
SPO	26214
SCANS	260
FREQU	31446.06 Hz
PD	2.0000 sec
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	77.00 ppm
TMSP	15503

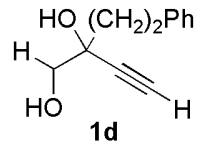


1c

F:¥furan¥NMR (furan)¥Yka0220.als
2-phenethybut-3-yne-1,2-diol

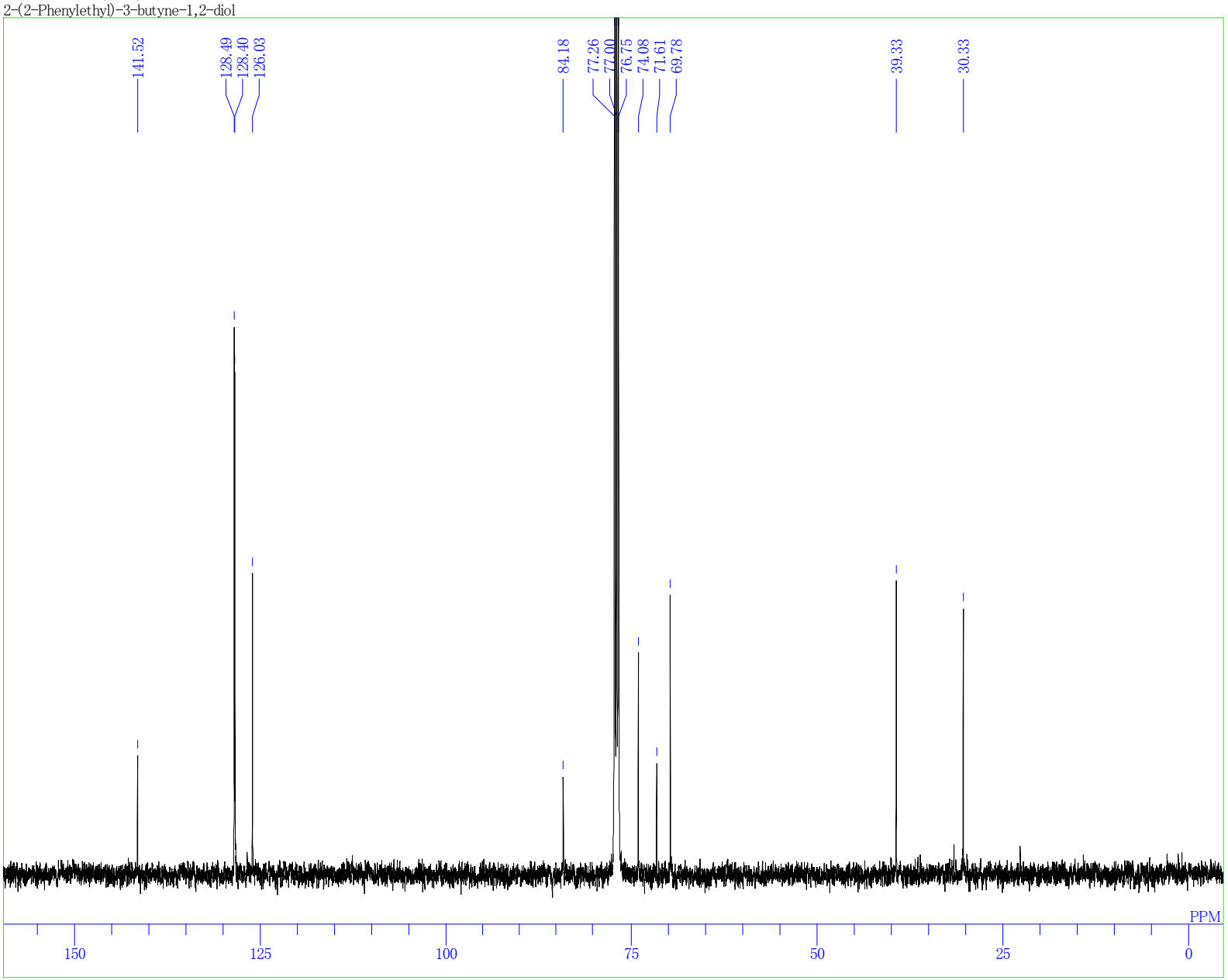


DATIM	14-11-2008 14:57:57
EXMOD	single_pulse.ex2
OBNUC	1H
OFR	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
OBATN	0
PW1	4.50 usec
PW2	0.00 usec
POINT	13107
SPO	13107
SCANS	8
FREQU	7507.39 Hz
PD	1.5000 sec
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	7.26 ppm
TMSP	4584

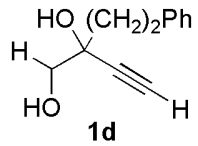


1d

F:¥furan¥NMR (furan)¥Yka0220 carbo.als
2-(2-Phenylethyl)-3-butyne-1,2-diol

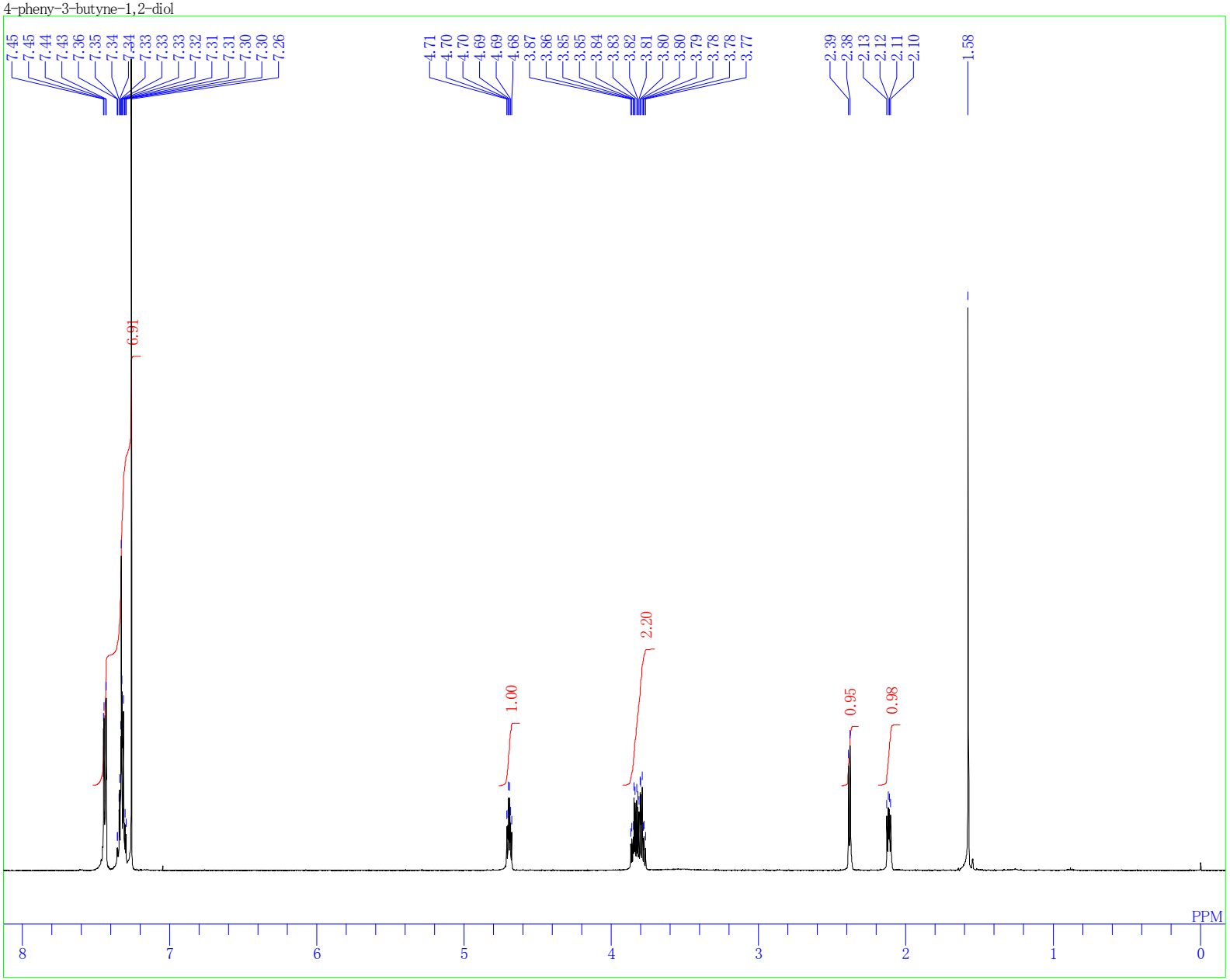


DATIM	19-11-2008 11:39:60
EXMOD	single_pulse_dec
OBNUC	13C
OFR	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
OBATN	0
PW1	3.27 usec
PW2	0.00 usec
POINT	32768
SPO	32768
SCANS	1576
FREQU	39308.18 Hz
PD	2.0000 sec
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	77.00 ppm
TMSP	18783

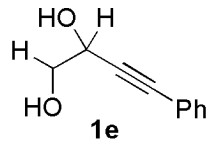


1d

F:¥furan¥NMR (furan)¥Yegi030212-1H.als
4-pheny-3-butyne-1,2-diol

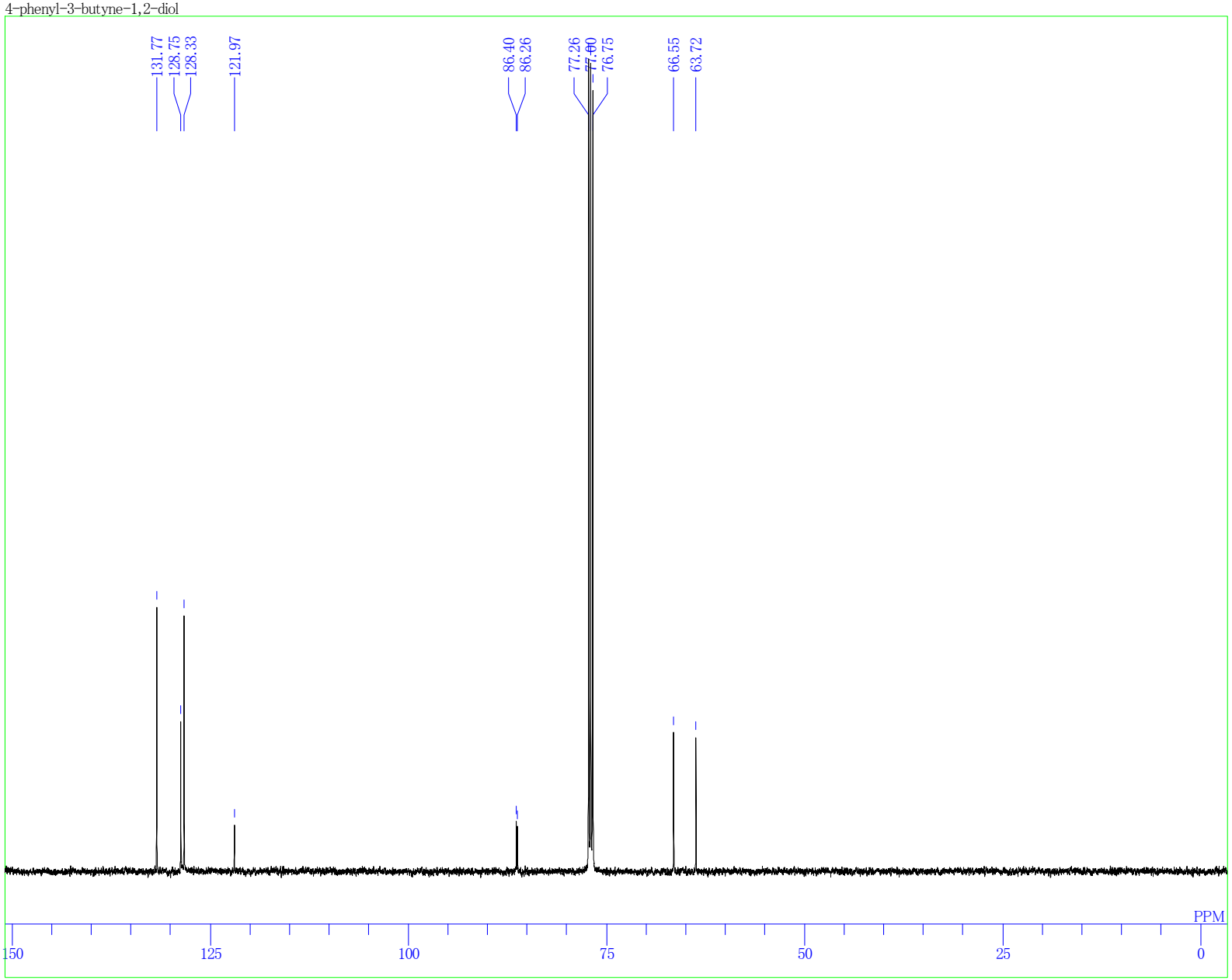


DATIM	29-10-2008 15:49:39
EXMOD	single_pulse.ex2
OBNUC	1H
OFR	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
OBATN	0
PW1	4.50 usec
PW2	0.00 usec
POINT	16384
SPO	16384
SCANS	8
FREQU	9384.38 Hz
PD	5.0000 sec
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	7.26 ppm
TMSP	6222

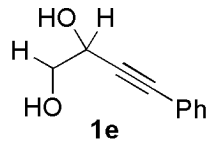


1e

F:¥furan¥NMR (furan)¥Yegi030212-13C.als
4-phenyl-3-butyne-1,2-diol

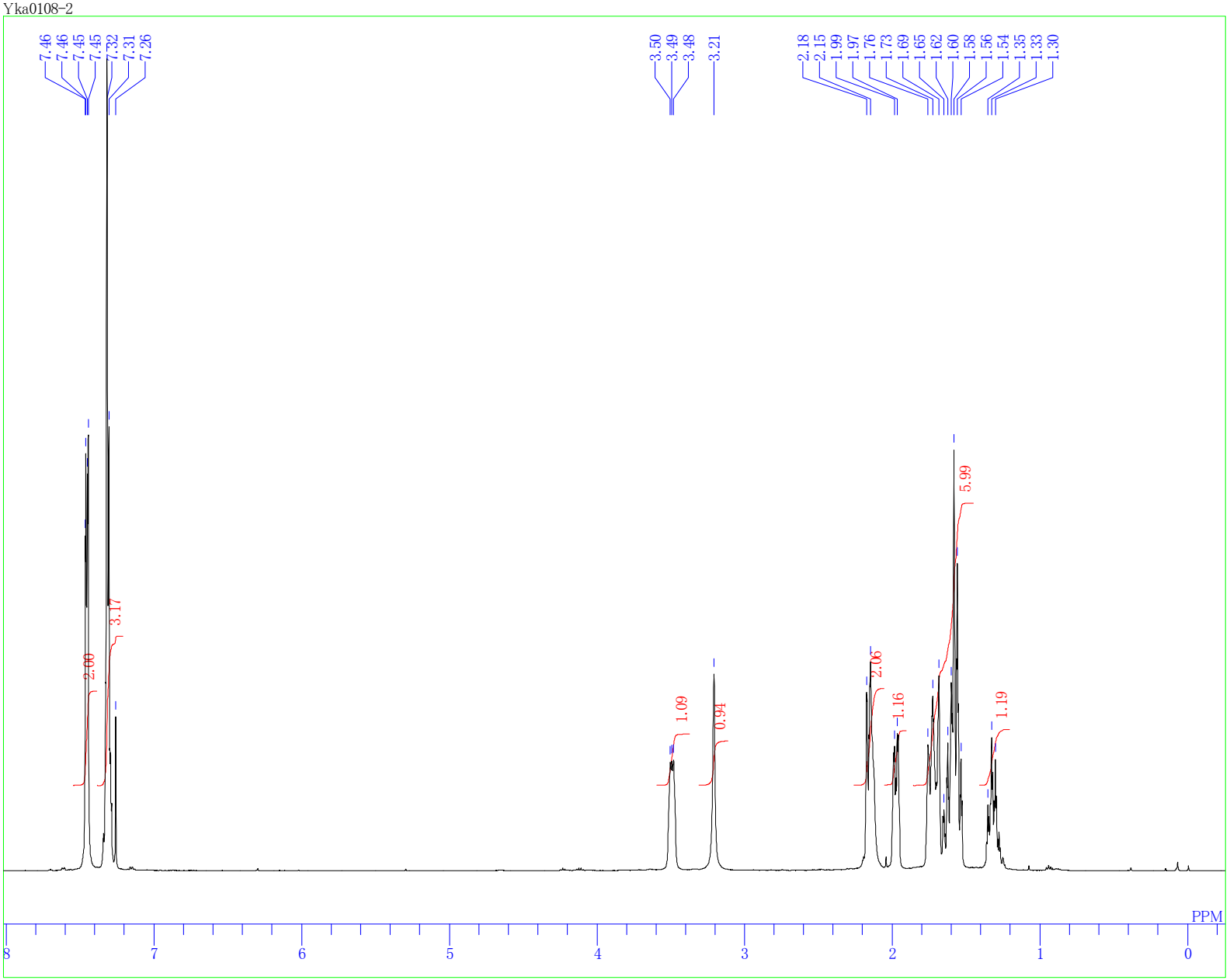


DATIM	29-10-2008 16:26:34
EXMOD	single_pulse_dec
OBNUC	13C
OFR	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
OBATN	0
PW1	3.27 usec
PW2	0.00 usec
POINT	26214
SPO	26214
SCANS	697
FREQU	31446.06 Hz
PD	2.0000 sec
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	77.00 ppm
TMSP	15506

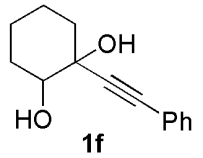


1e

F:¥furan¥NMR (furan)¥1-(2-Phenylethynyl)cyclohexane-1,2-diol (H).als
Yka0108-2

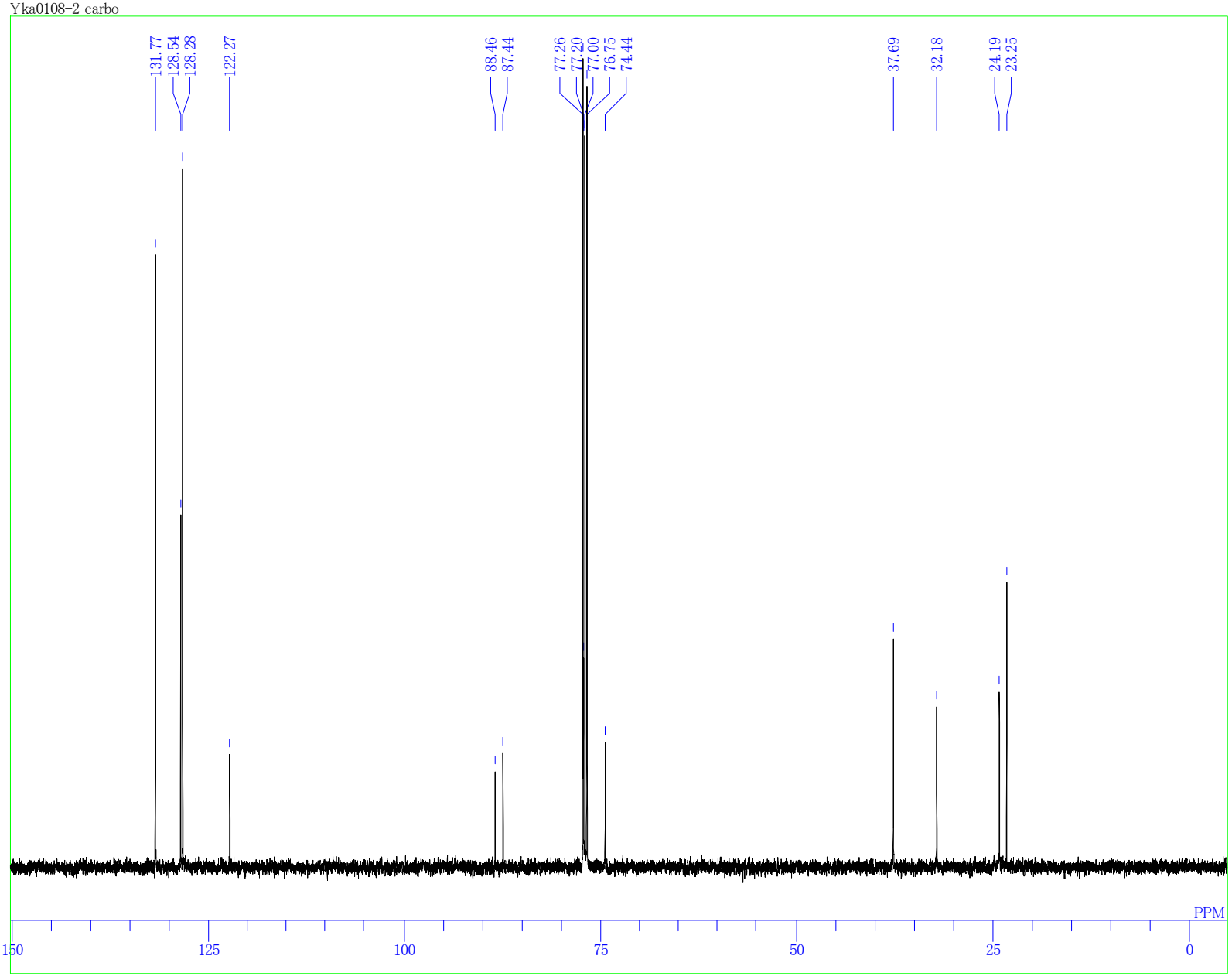


DATIM	25-12-2007 19:33:14
EXMOD	single_pulse.ex2
OBNUC	1H
OFR	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
OBATN	0
PW1	6.00 usec
PW2	0.00 usec
POINT	16384
SPO	16384
SCANS	8
FREQU	9384.38 Hz
PD	1.5000 sec
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	7.26 ppm
TMSP	6223

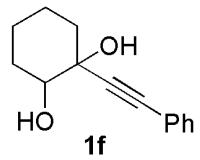


1f

F:¥furan¥NMR (furan)¥1-(2-Phenylethynyl)cyclohexane-1,2-diol (C).als
Yka0108-2 carbo

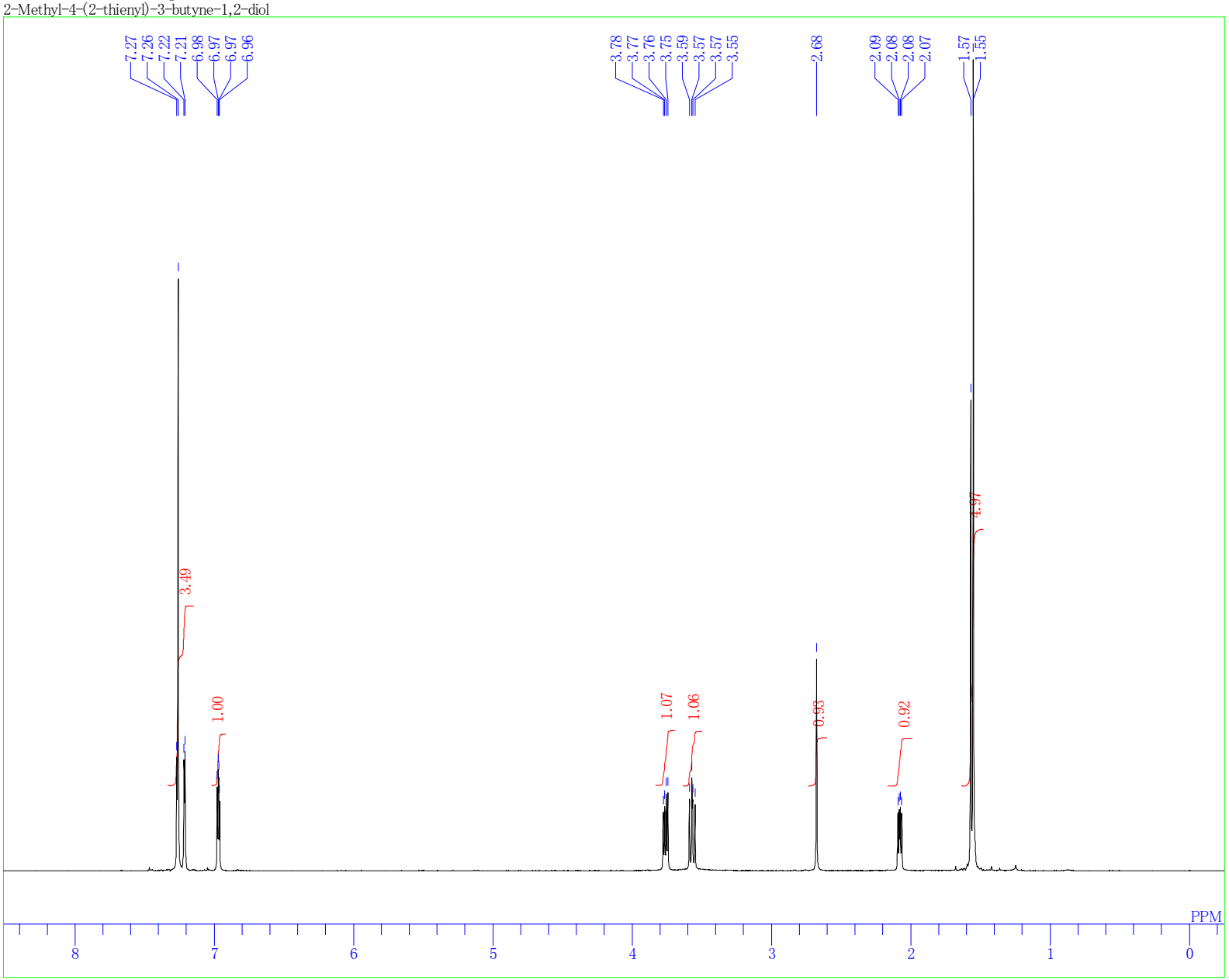


DATIM	25-12-2007 19:46:34
EXMOD	single_pulse_dec
OBNUC	13C
OFR	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
OBATN	0
PW1	3.67 usec
PW2	0.00 usec
POINT	32768
SPO	32768
SCANS	219
FREQU	39308.18 Hz
PD	2.0000 sec
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	77.00 ppm
TMSP	18781

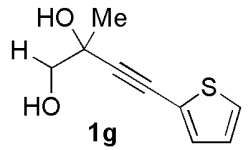


1f

F:¥furan¥NMR (furan)¥Yegi030193.als
2-Methyl-4-(2-thienyl)-3-butyne-1,2-diol

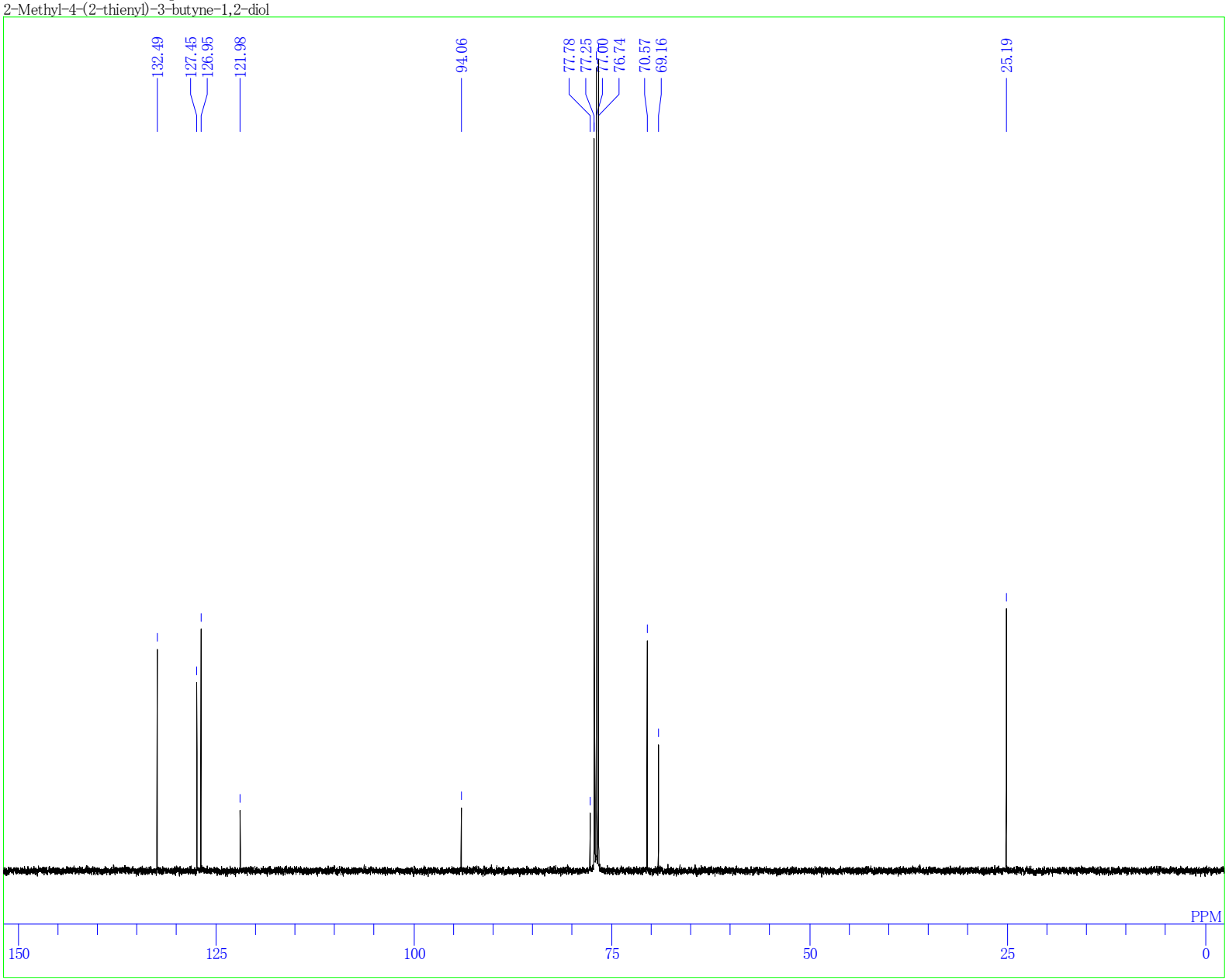


DATIM	02-10-2008 20:51:45
EXMOD	single_pulse.ex2
OBNUC	1H
OFR	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
OBATN	0
PW1	4.50 usec
PW2	0.00 usec
POINT	16384
SPO	16384
SCANS	8
FREQU	9384.38 Hz
PD	5.0000 sec
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	12.51 ppm
TMSP	1638

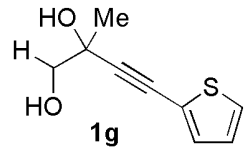


1g

F:¥furan¥NMR (furan)¥Yegi030193-13C.als
2-Methyl-4-(2-thienyl)-3-butyne-1,2-diol

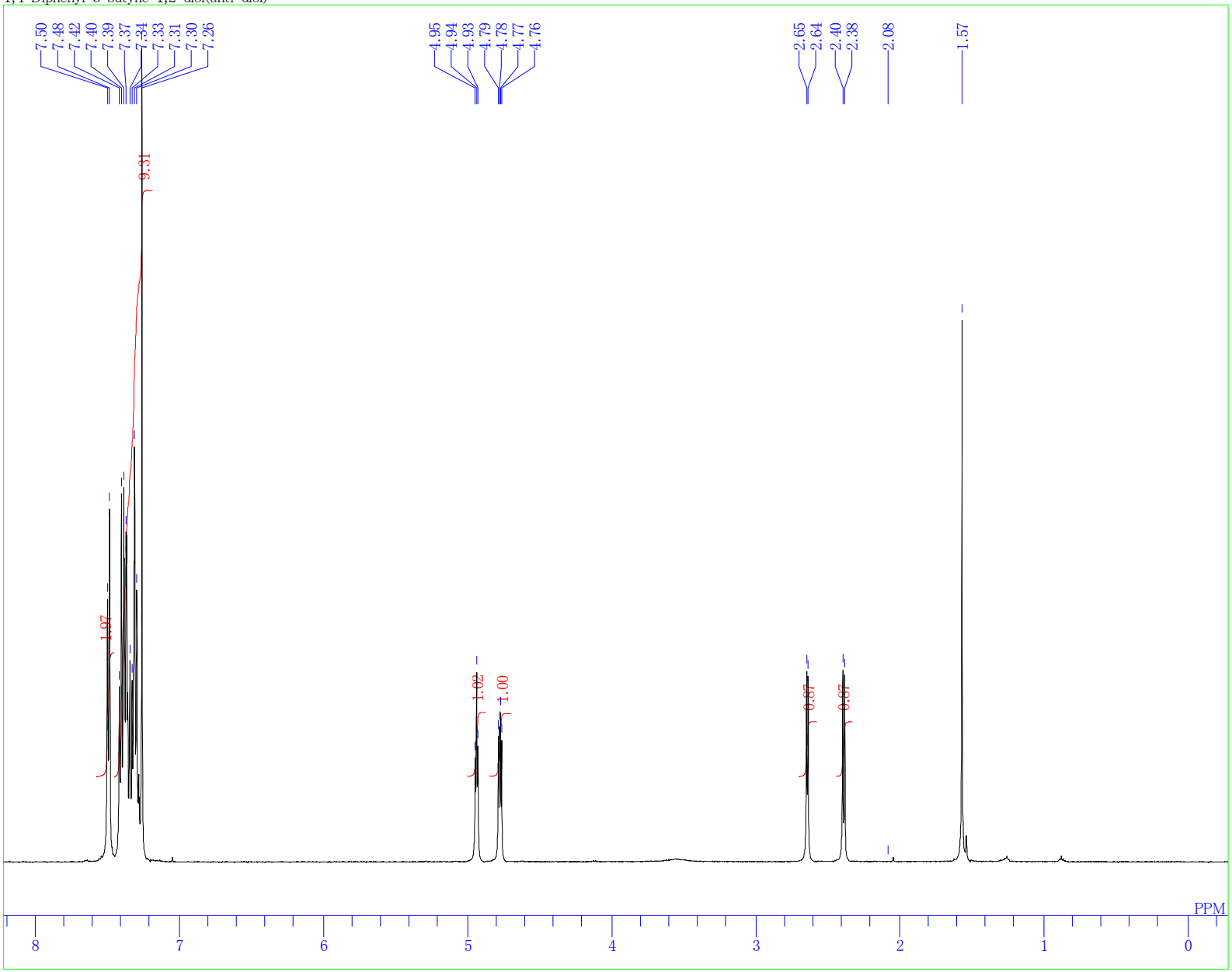


DATIM	01-10-2008 23:02:31
EXMOD	single_pulse_dec
OBNUC	13C
OFR	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
OBATN	0
PW1	3.27 usec
PW2	0.00 usec
POINT	32768
SPO	32768
SCANS	459
FREQU	39308.18 Hz
PD	2.0000 sec
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	77.00 ppm
TMSP	18782

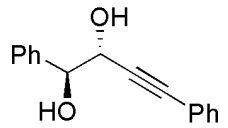


1g

F:¥furan¥NMR (furan)¥Yegi030276.als
1,4-Diphenyl-3-butyne-1,2-diol(anti-diol)

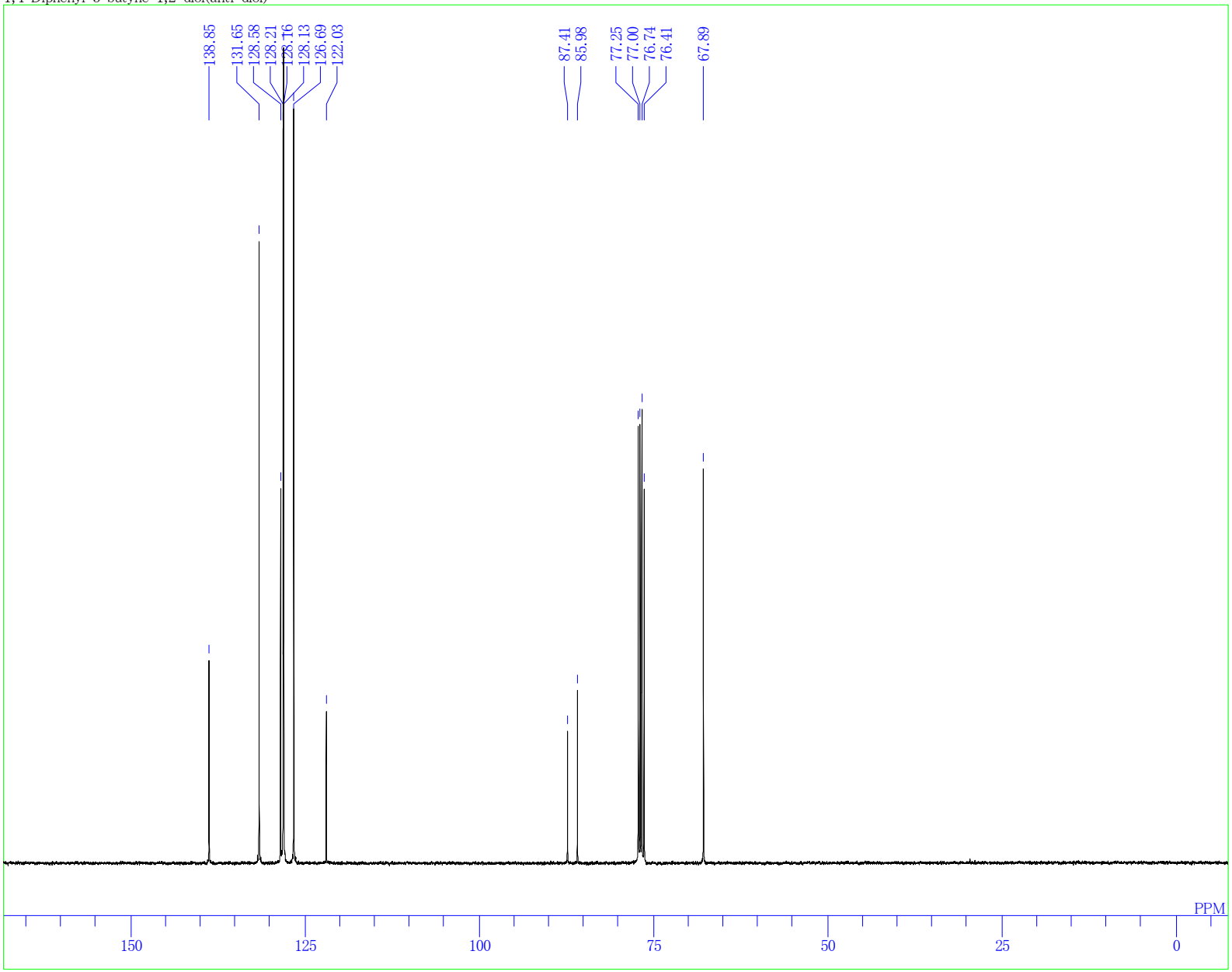


DATIM	23-01-2009 11:41:49
EXMOD	single_pulse.ex2
OBNUC	1H
OFR	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
OBATN	0
PW1	4.50 usec
PW2	0.00 usec
POINT	16384
SPO	16384
SCANS	8
FREQU	9384.38 Hz
PD	5.0000 sec
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	7.26 ppm
TMSP	6222

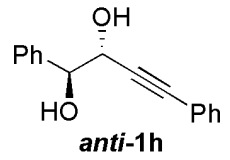


***anti*-1h**

F:¥furan¥NMR (furan)¥Yegi030260-13C.als
1,4-Diphenyl-3-butyne-1,2-diol(anti-diol)

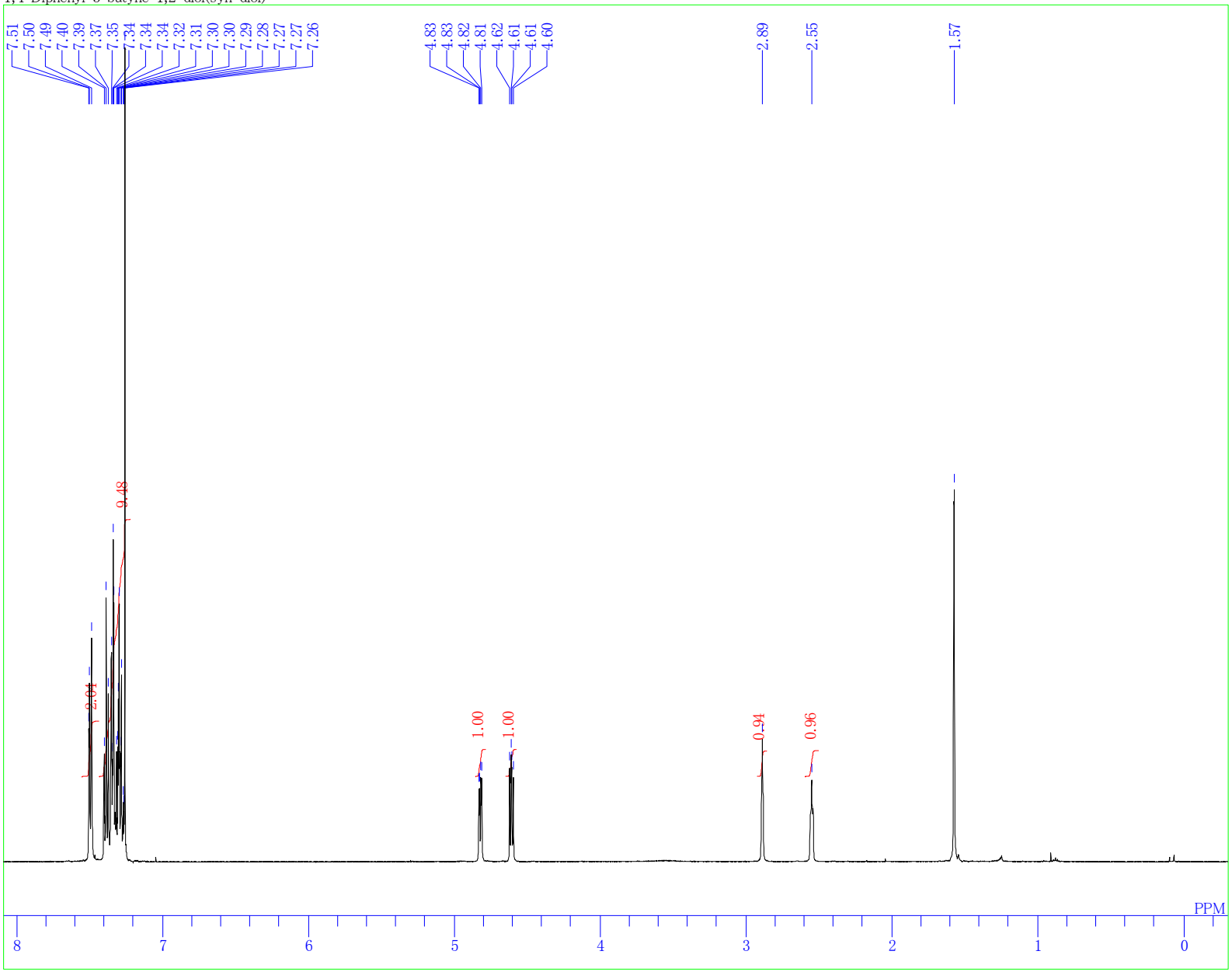


DATIM	19-12-2008 12:09:30
EXMOD	single_pulse_dec
OBNUC	13C
OFR	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
OBATN	0
PW1	3.27 usec
PW2	0.00 usec
POINT	32768
SPO	32768
SCANS	1026
FREQU	39308.18 Hz
PD	2.0000 sec
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	77.00 ppm
TMSP	18775

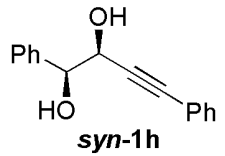


***anti*-1h**

F:¥furan¥NMR (furan)¥Yegi030288pure.als
1,4-Diphenyl-3-butyne-1,2-diol(syn-diol)

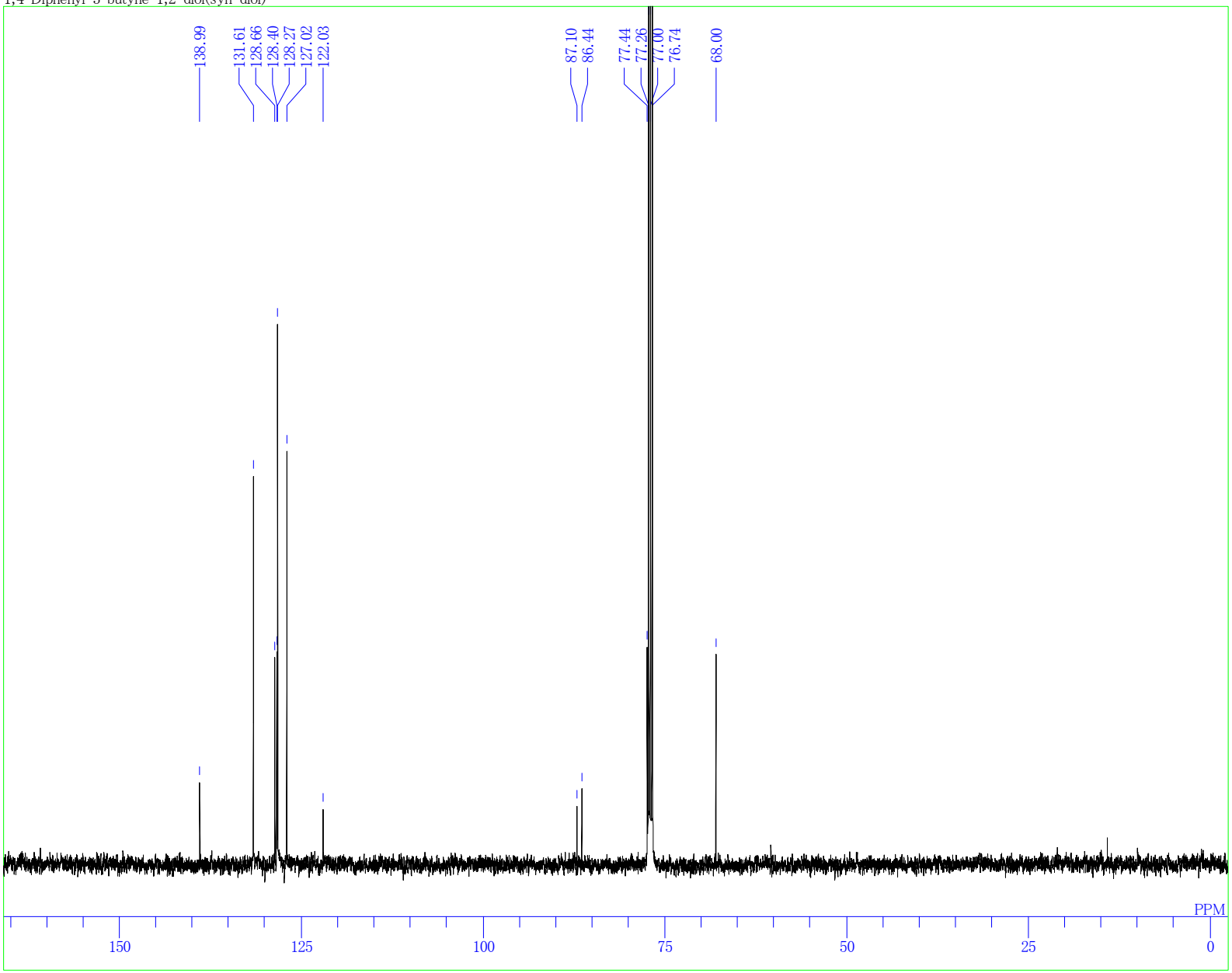


DATIM	04-02-2009 16:50:34
EXMOD	single_pulse.ex2
OBNUC	1H
OFR	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
OBATN	0
PW1	4.50 usec
PW2	0.00 usec
POINT	16384
SPO	16384
SCANS	8
FREQU	9384.38 Hz
PD	5.0000 sec
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	7.26 ppm
TMSP	6222

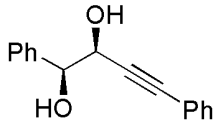


***syn*-1h**

F:¥furan¥NMR (furan)¥Yegi030275-13C.als
1,4-Diphenyl-3-butyne-1,2-diol(syn-diol)

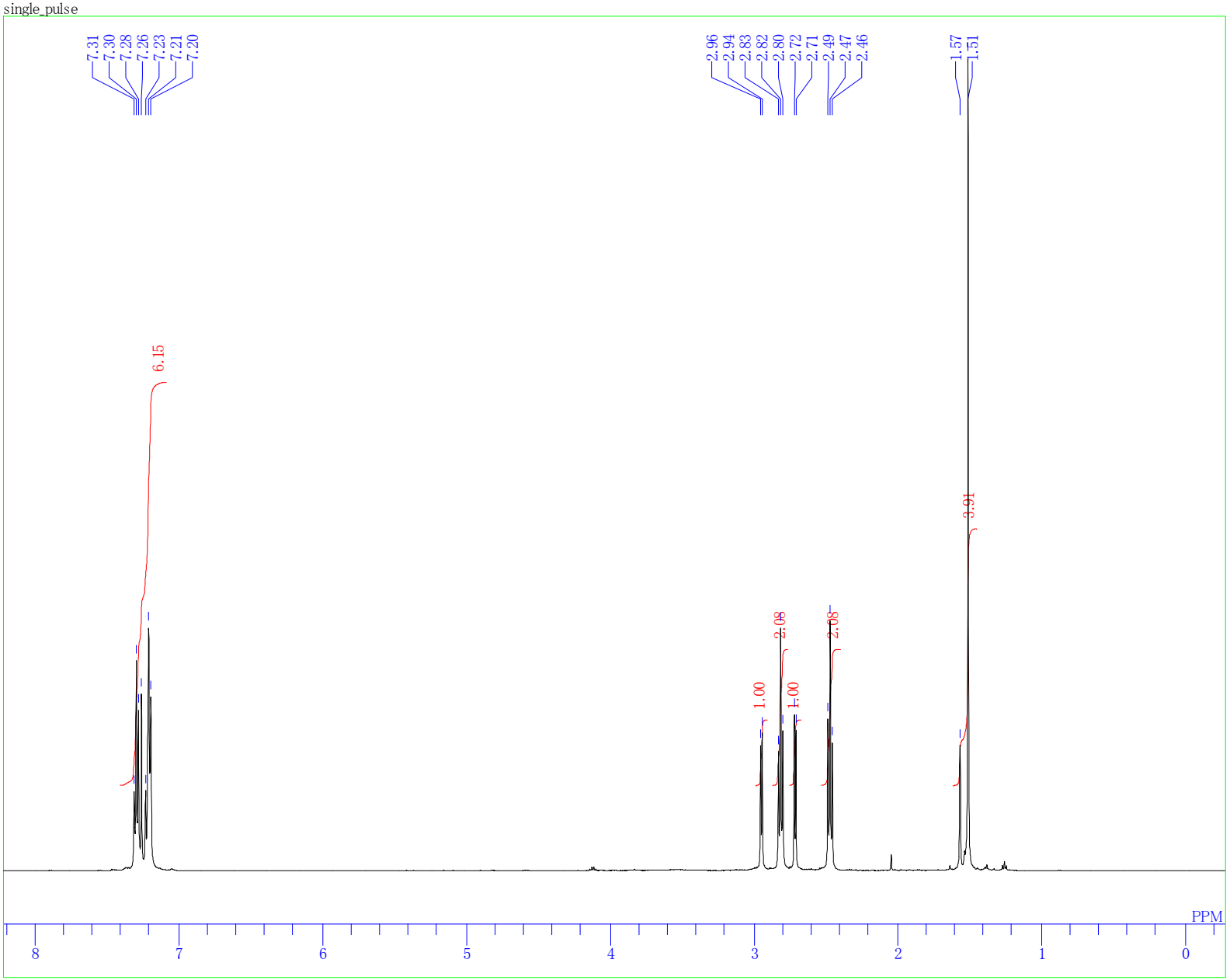


DATIM	23-01-2009 11:37:31
EXMOD	single_pulse_dec
OBNUC	13C
OFR	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
OBATN	0
PW1	3.27 usec
PW2	0.00 usec
POINT	32768
SPO	32768
SCANS	385
FREQU	39308.18 Hz
PD	2.0000 sec
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	77.00 ppm
TMSP	18782

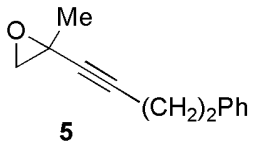


***syn*-1h**

F:¥furan¥NMR (furan)¥Yegi03-epoxide.als
single_pulse

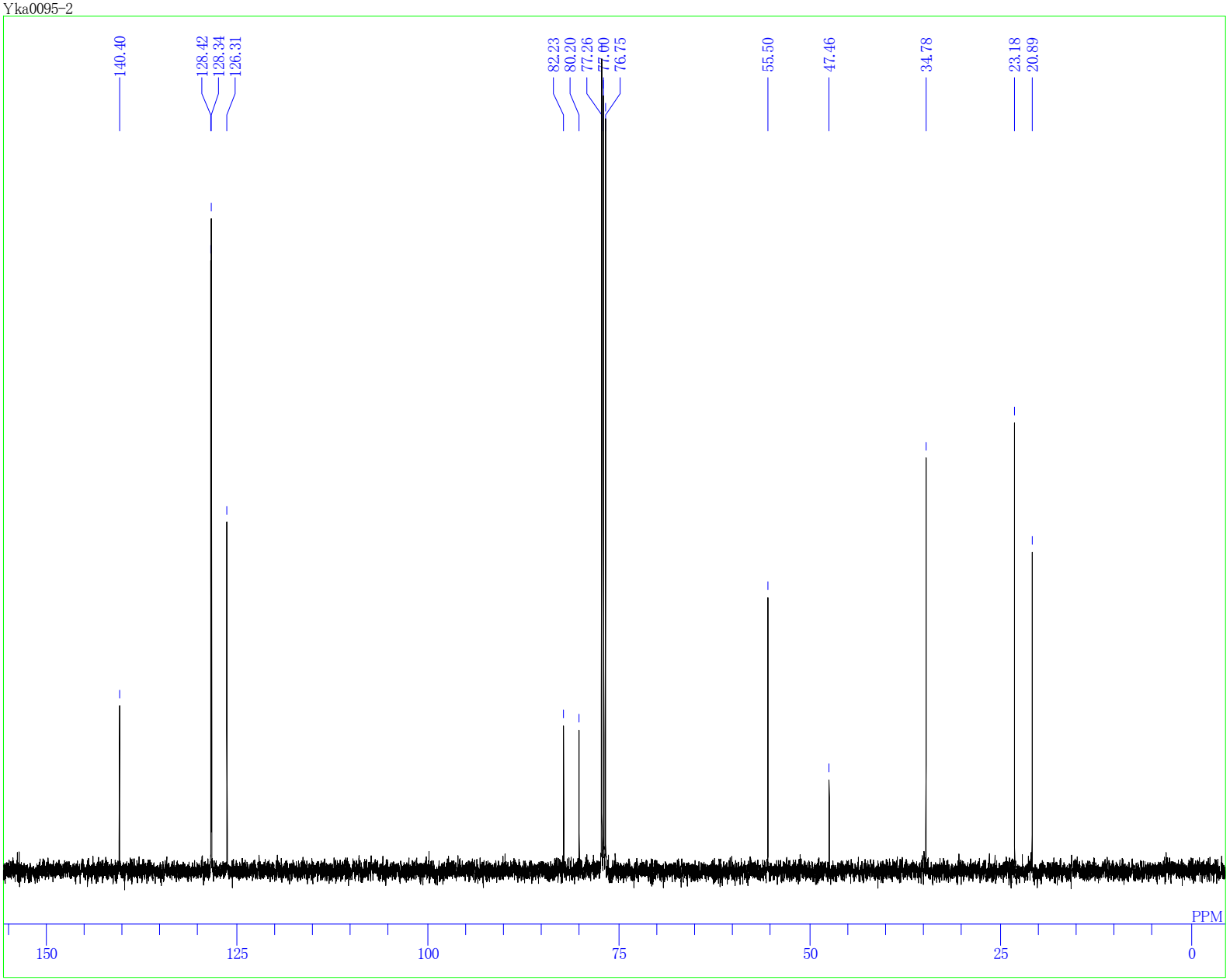


DATIM	01-10-2008 22:33:08
EXMOD	single_pulse.ex2
OBNUC	1H
OFR	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
OBATN	0
PW1	4.50 usec
PW2	0.00 usec
POINT	16384
SPO	16384
SCANS	8
FREQU	9384.38 Hz
PD	5.0000 sec
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	7.26 ppm
TMSP	6222

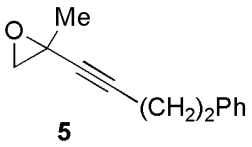


5

F:¥furan¥NMR (furan)¥2-(4-Phenyl-1-butyn-1-yl)-2-methyloxirane (C).als
Yka0095-2

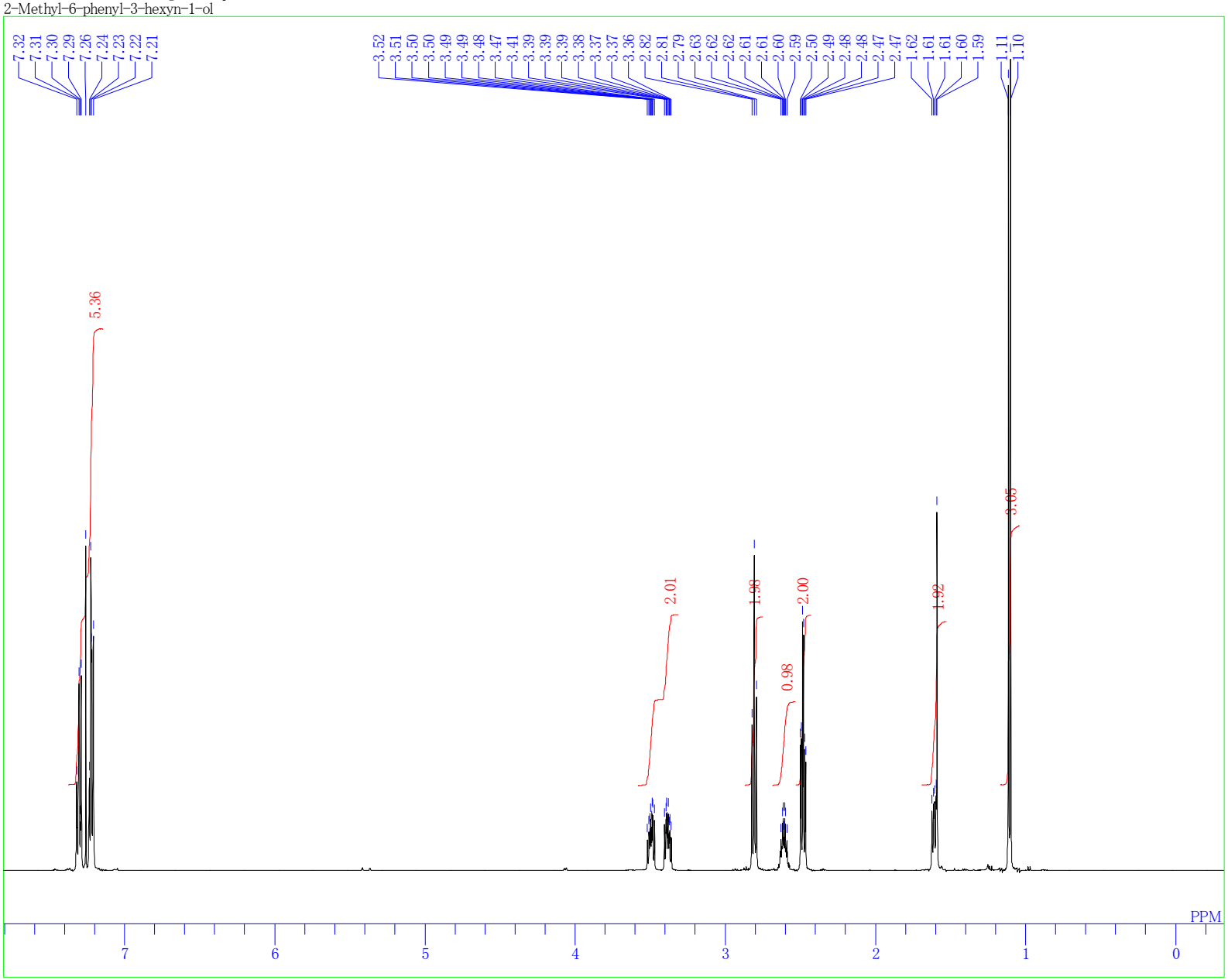


DATIM	08-12-2007 18:45:16
EXMOD	single_pulse_dec
OBNUC	13C
OFR	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
OBATN	0
PW1	3.67 usec
PW2	0.00 usec
POINT	32768
SPO	32768
SCANS	122
FREQU	39308.18 Hz
PD	2.0000 sec
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	77.00 ppm
TMSP	18781

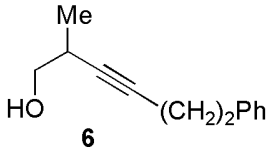


5

F:¥furan¥NMR (furan)¥Yegi030196pure.als
2-Methyl-6-phenyl-3-hexyn-1-ol

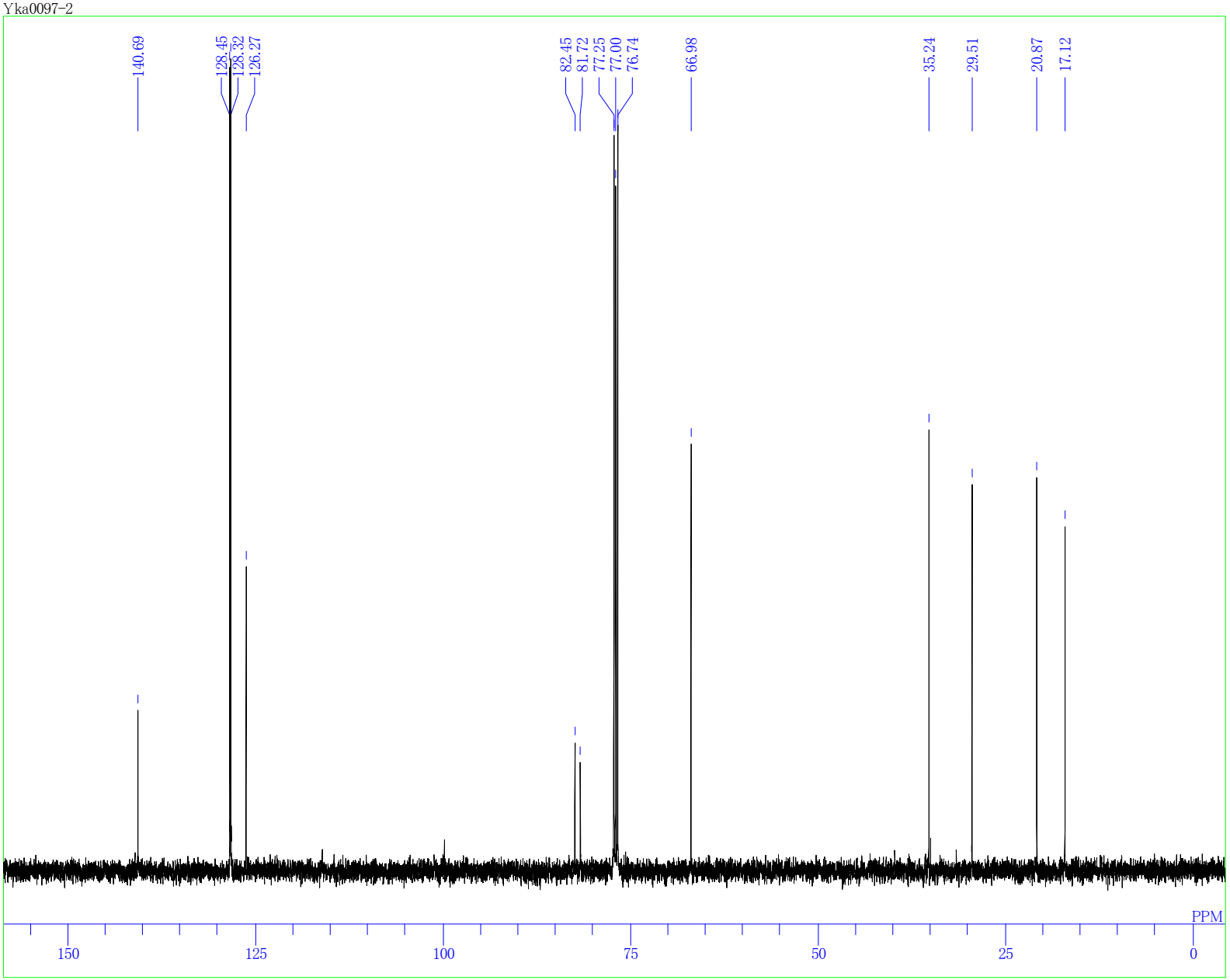


DATIM	09-10-2008 18:00:38
EXMOD	single_pulse.ex2
OBNUC	1H
OFR	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
OBATN	0
PW1	4.50 usec
PW2	0.00 usec
POINT	16384
SPO	16384
SCANS	16
FREQU	9384.38 Hz
PD	5.0000 sec
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	7.26 ppm
TMSP	6222

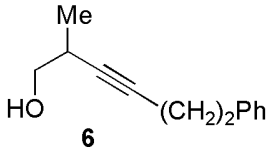


6

F:¥furan¥NMR (furan)¥2-Methyl-6-phenyl-3-hexyn-1-ol (C).als
Yka0097-2

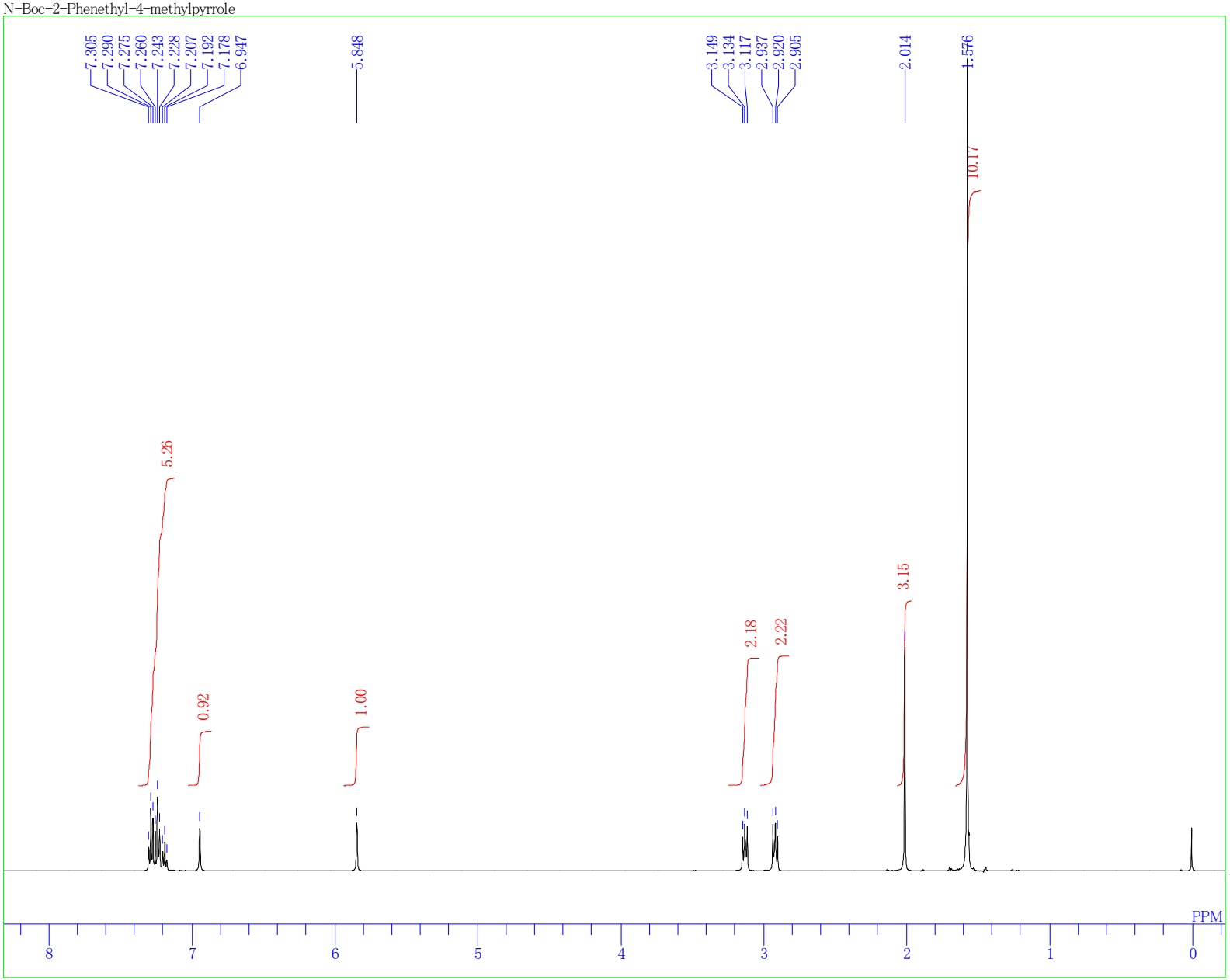


DATIM	12-12-2007 20:11:33
EXMOD	single_pulse_dec
OBNUC	13C
OFR	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
OBATN	0
PW1	3.67 usec
PW2	0.00 usec
POINT	32768
SPO	32768
SCANS	124
FREQU	39308.18 Hz
PD	2.0000 sec
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	77.00 ppm
TMSP	18780

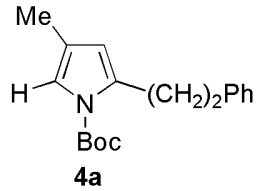


6

F:¥pyrrole¥Yka0289-1.als
N-Boc-2-Phenethyl-4-methylpyrrole

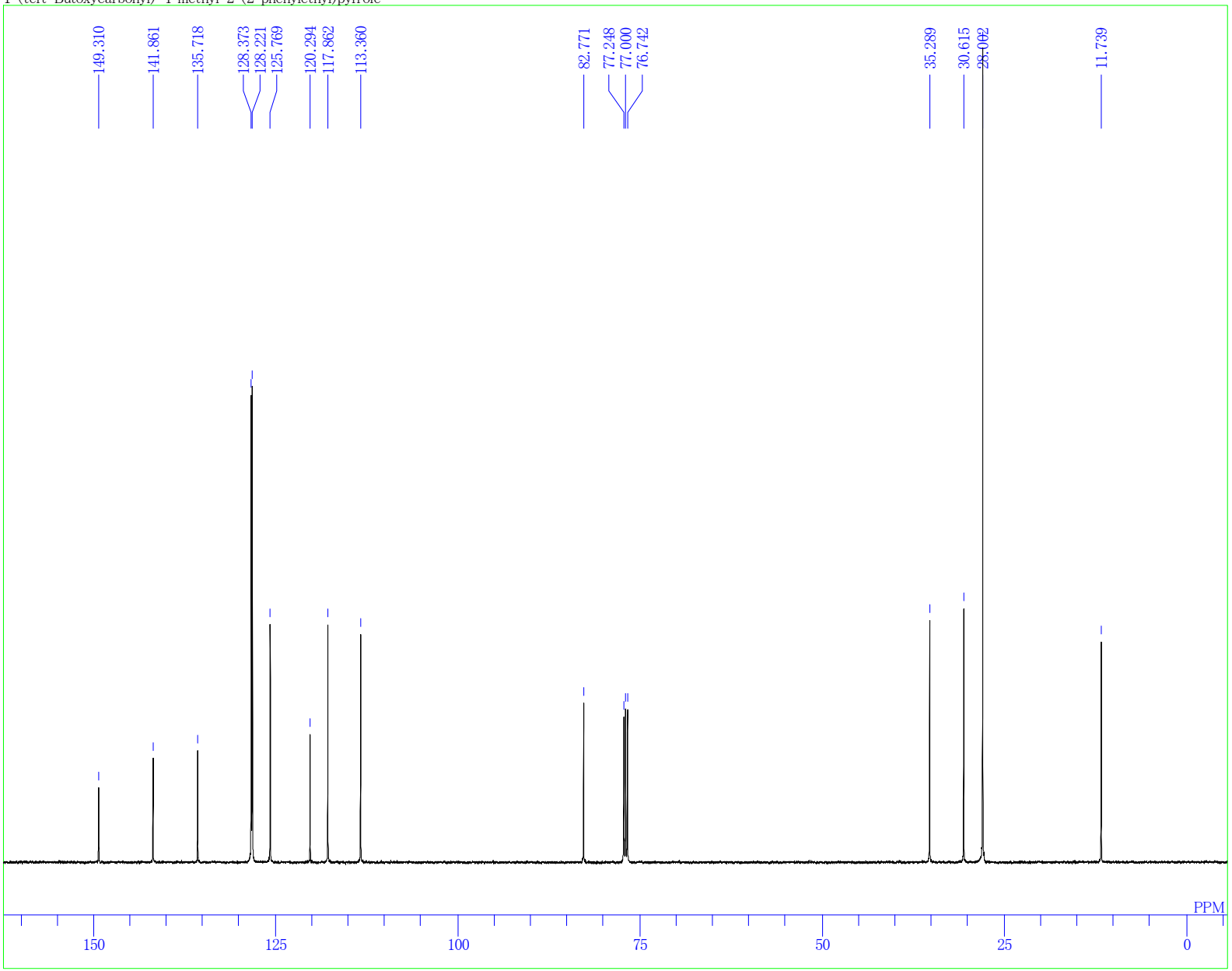


DATIM	28-01-2009 13:53:14
EXMOD	single_pulse.ex2
OBNUC	1H
OFR	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
OBATN	0
PW1	4.50 usec
PW2	0.00 usec
POINT	16384
SPO	16384
SCANS	8
FREQU	9384.38 Hz
PD	1.5000 sec
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	7.26 ppm
TMSP	6222

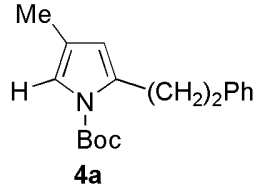


4a

F:¥pyrrole¥Yegi030239-13C.als
1-(tert-Butoxycarbonyl)-4-methyl-2-(2-phenylethyl)pyrrole

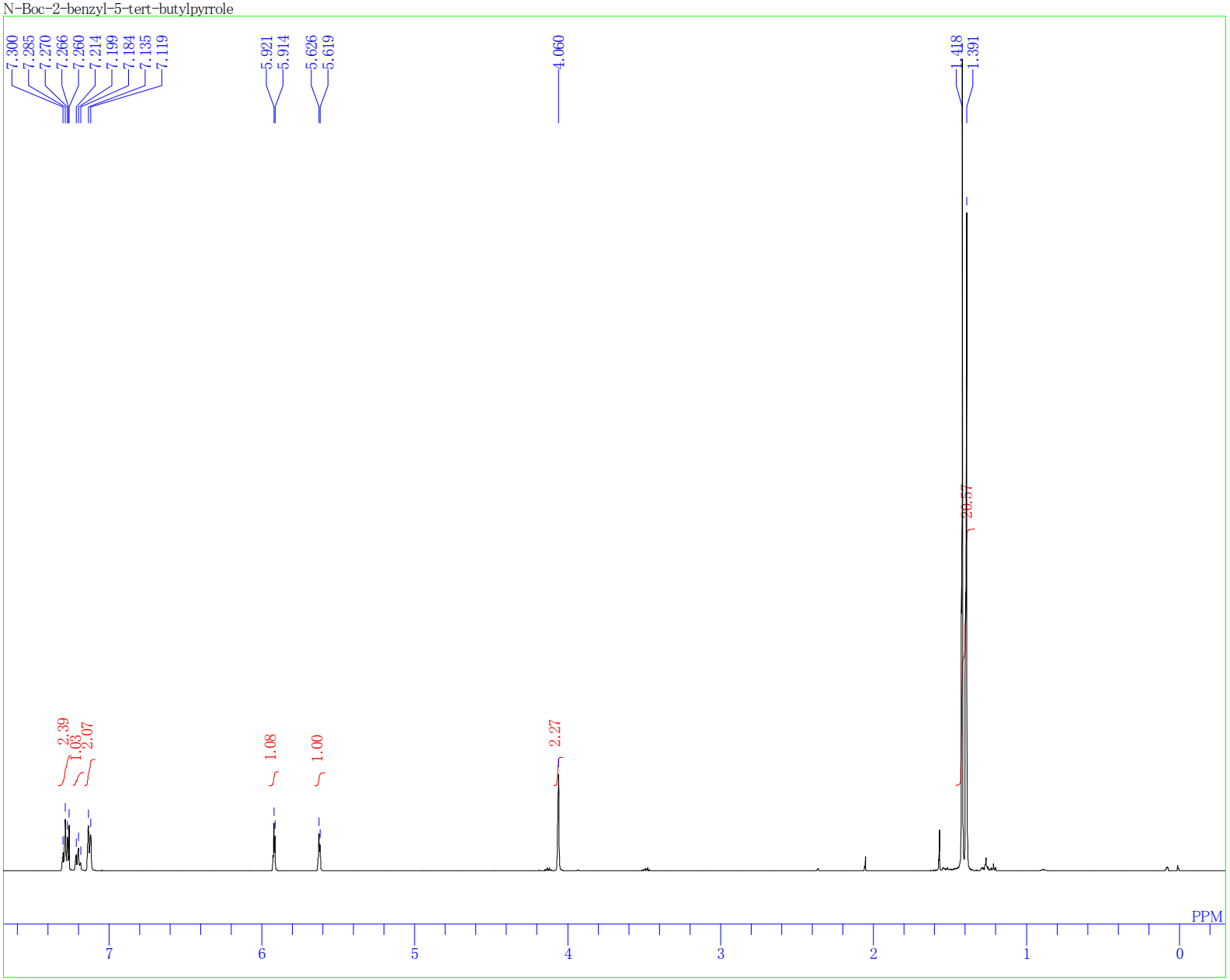


DATIM	28-11-2008 19:26:02
EXMOD	single_pulse_dec
OBNUC	13C
OFR	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
OBATN	0
PW1	3.27 usec
PW2	0.00 usec
POINT	32768
SPO	32768
SCANS	545
FREQU	39308.18 Hz
PD	2.0000 sec
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	77.00 ppm
TMSP	18774

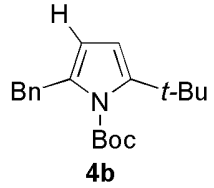


4a

F:¥pyrrole¥Yka0258-1.als
N-Boc-2-benzyl-5-tert-butylpyrrole

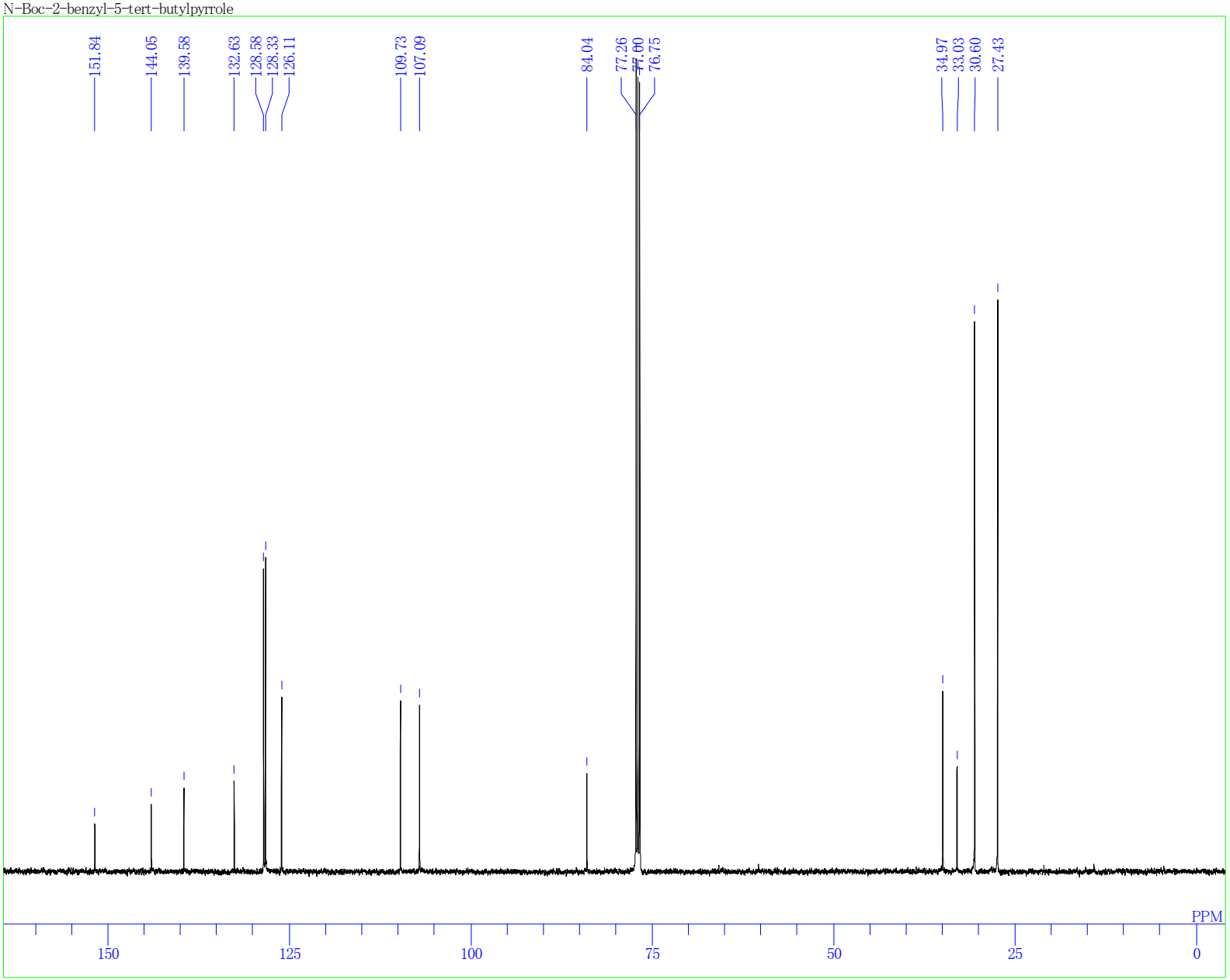


DATIM	08-12-2008 22:35:04
EXMOD	single_pulse.ex2
OBNUC	1H
OFR	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
OBATN	0
PW1	4.50 usec
PW2	0.00 usec
POINT	20480
SPO	20480
SCANS	8
FREQU	11730.66 Hz
PD	1.5000 sec
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	7.26 ppm
TMSP	6223

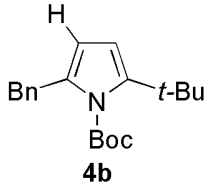


4b

F:¥pyrrole¥Yka0258-1 carbo.als
N-Boc-2-benzyl-5-tert-butylpyrrole

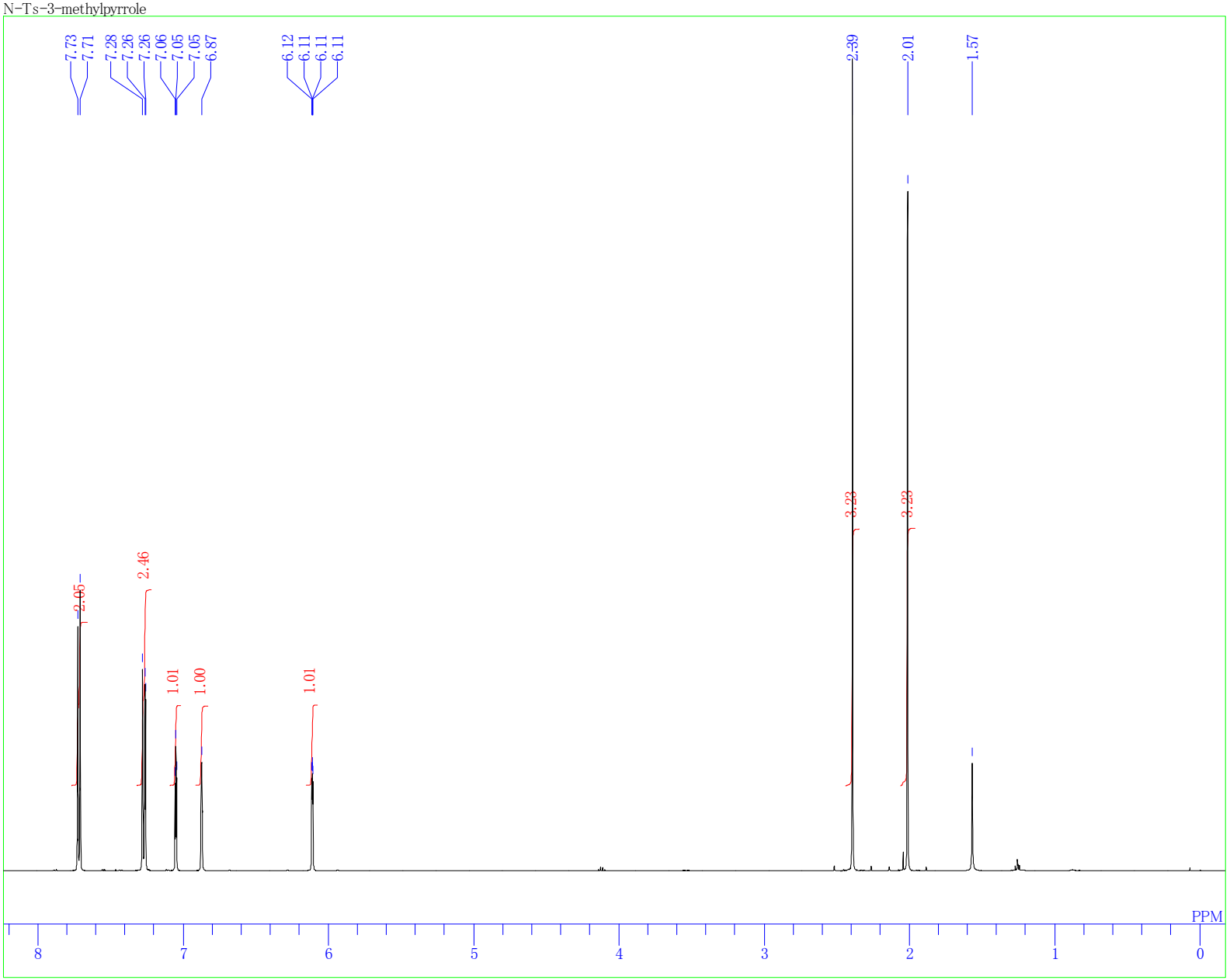


DATIM	08-12-2008 23:17:35
EXMOD	single_pulse_dec
OBNUC	13C
OFR	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
OBATN	0
PW1	3.27 usec
PW2	0.00 usec
POINT	40961
SPO	40961
SCANS	849
FREQU	49135.97 Hz
PD	2.0000 sec
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	77.00 ppm
TMSP	18782

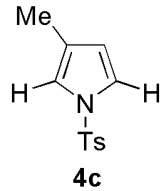


4b

F:¥pyrrole¥Yegi030336.als
N-Ts-3-methylpyrrole

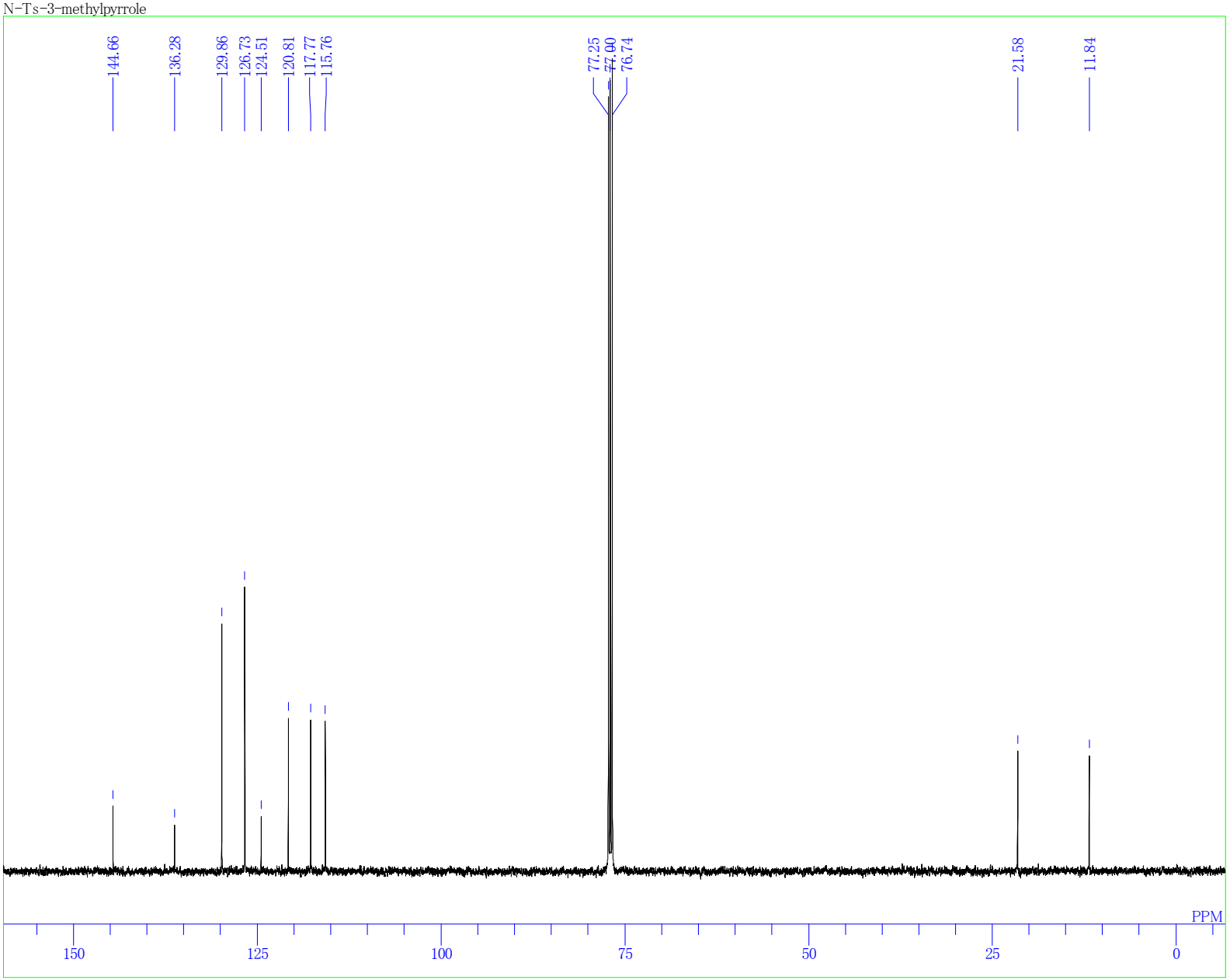


DATIM	09-06-2009 09:33:28
EXMOD	single_pulse.ex2
OBNUC	1H
OFR	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
OBATN	0
PW1	4.50 usec
PW2	0.00 usec
POINT	16384
SPO	16384
SCANS	16
FREQU	9384.38 Hz
PD	5.0000 sec
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	7.26 ppm
TMSP	6222

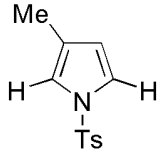


4c

F:¥pyrrole¥Yegi030336-13C.als
N-Ts-3-methylpyrrole

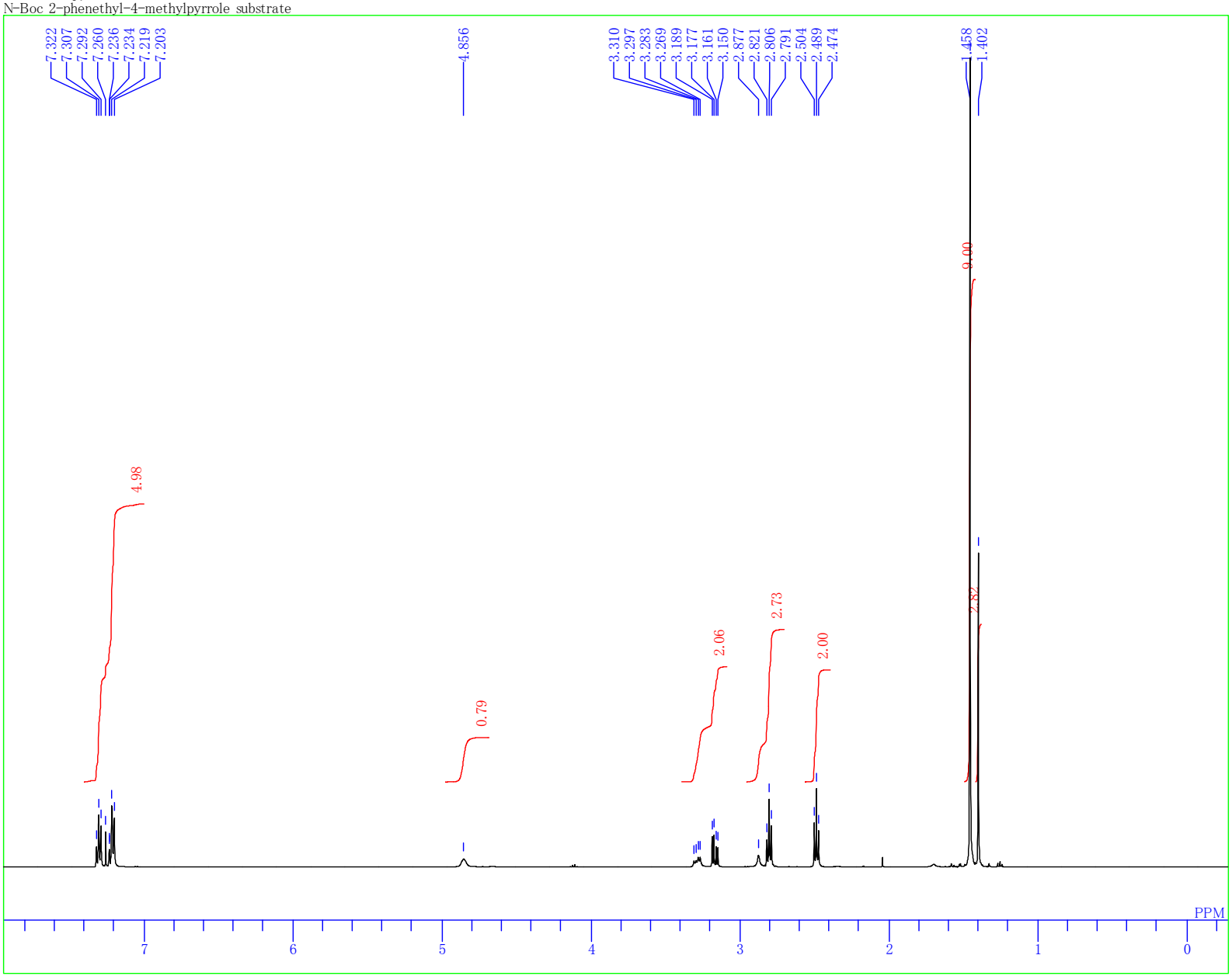


DATIM	09-06-2009 09:51:00
EXMOD	single_pulse_dec
OBNUC	13C
OFR	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
OBATN	0
PW1	3.27 usec
PW2	0.00 usec
POINT	32768
SPO	32768
SCANS	365
FREQU	39308.18 Hz
PD	2.0000 sec
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	77.00 ppm
TMSP	18783

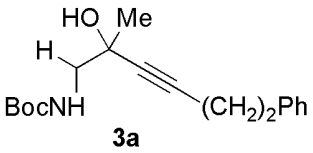


4c

F:¥azechi¥pyrrole¥Yka0288-2.als
N-Boc 2-phenethyl-4-methylpyrrole substrate

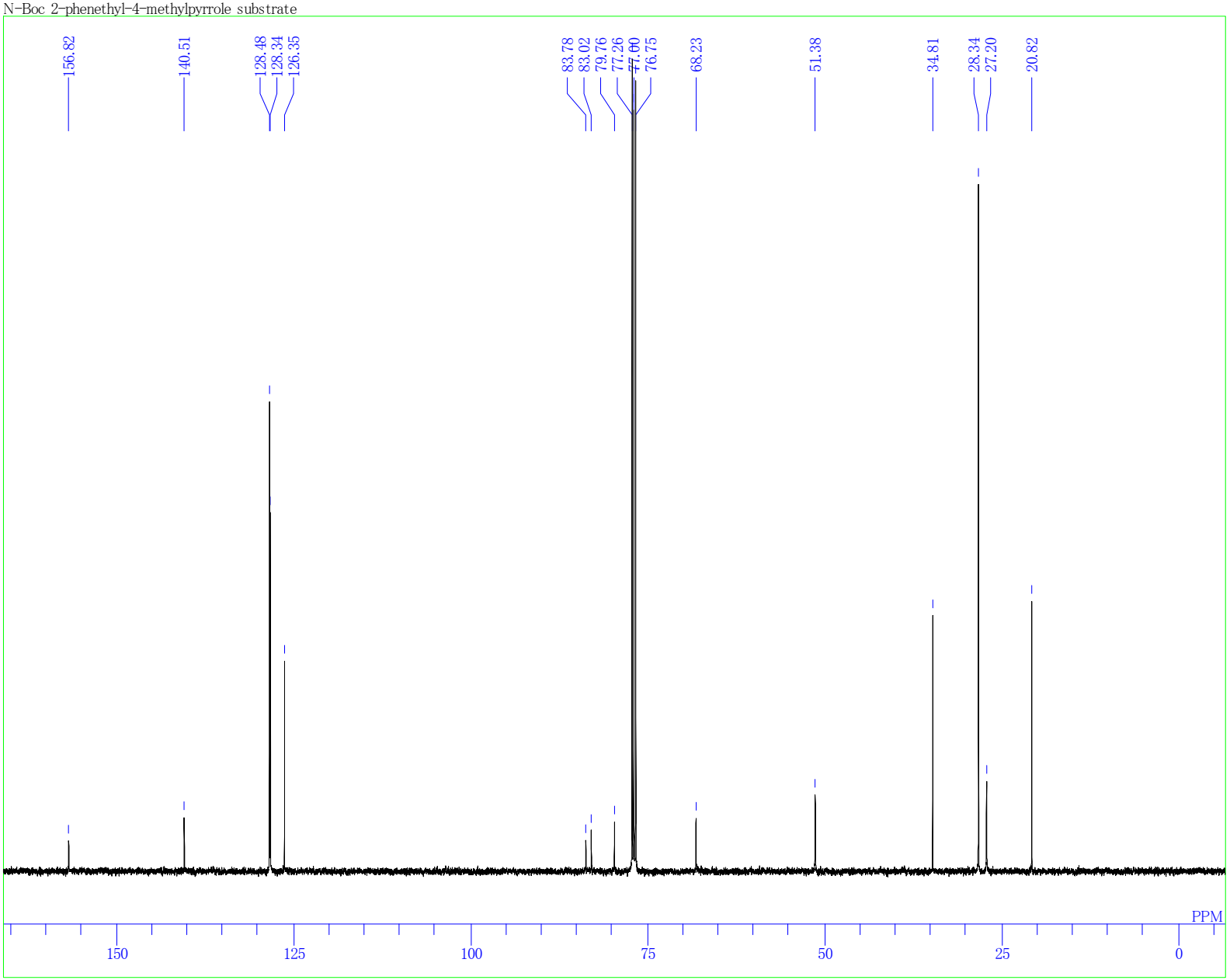


DATIM	20-01-2009 17:21:22
EXMOD	single_pulse.ex2
OBNUC	1H
OFR	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
OBATN	0
PW1	4.50 usec
PW2	0.00 usec
POINT	16384
SPO	16384
SCANS	8
FREQU	9384.38 Hz
PD	1.5000 sec
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	7.26 ppm
TMSP	6222

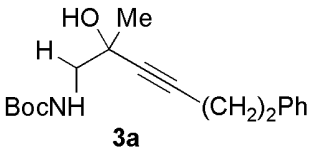


3a

F:¥Yka0288-2 carbon.als
N-Boc 2-phenethyl-4-methylpyrrole substrate

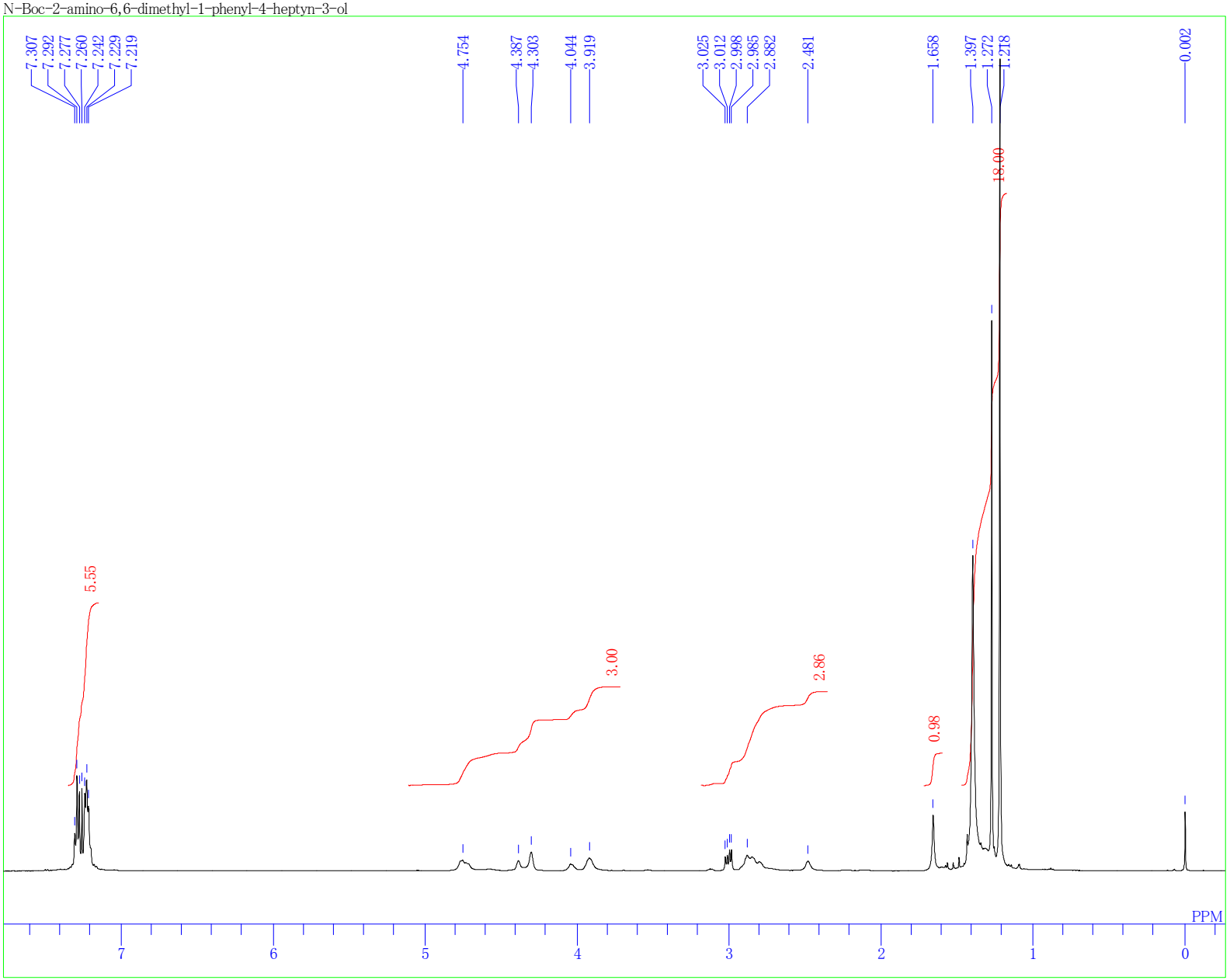


DATIM	20-01-2009 17:53:08
EXMOD	single_pulse_dec
OBNUC	13C
OFR	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
OBATN	0
PW1	3.27 usec
PW2	0.00 usec
POINT	32768
SPO	32768
SCANS	625
FREQU	39308.18 Hz
PD	2.0000 sec
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	77.00 ppm
TMSP	15504

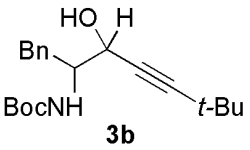


3a

F:¥Yka0240-1.als
N-Boc-2-amino-6,6-dimethyl-1-phenyl-4-heptyn-3-ol

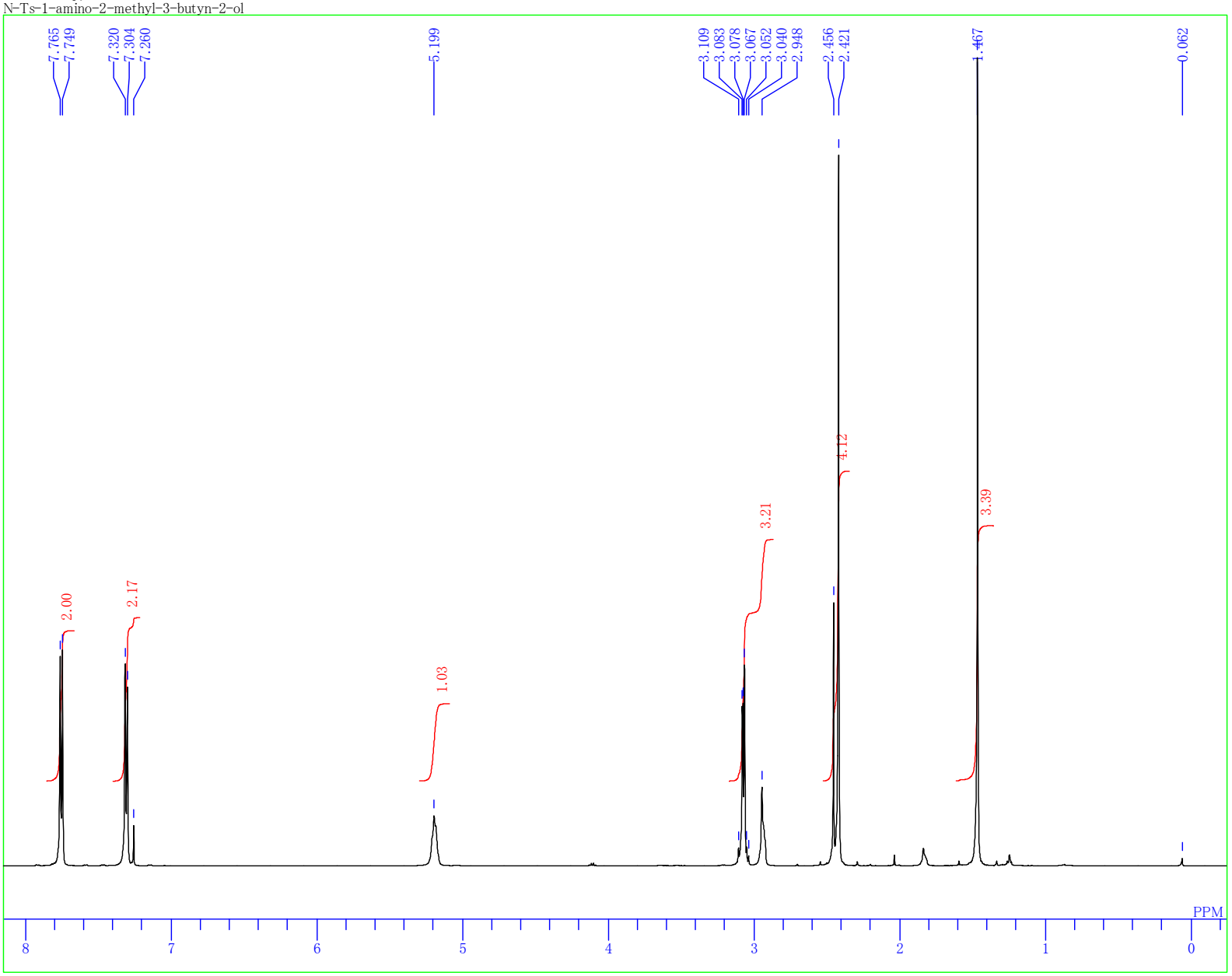


DATIM	24-06-2009 15:08:07
EXMOD	single_pulse.ex2
OBNUC	1H
OFR	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
OBATN	0
PW1	4.50 usec
PW2	0.00 usec
POINT	16384
SPO	16384
SCANS	8
FREQU	9384.38 Hz
PD	1.5000 sec
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	7.26 ppm
TMSP	4585

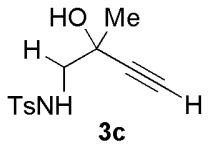


3b

F:¥azechi¥pyrrole¥Yka0366-2.als
N-Ts-1-amino-2-methyl-3-butyn-2-ol

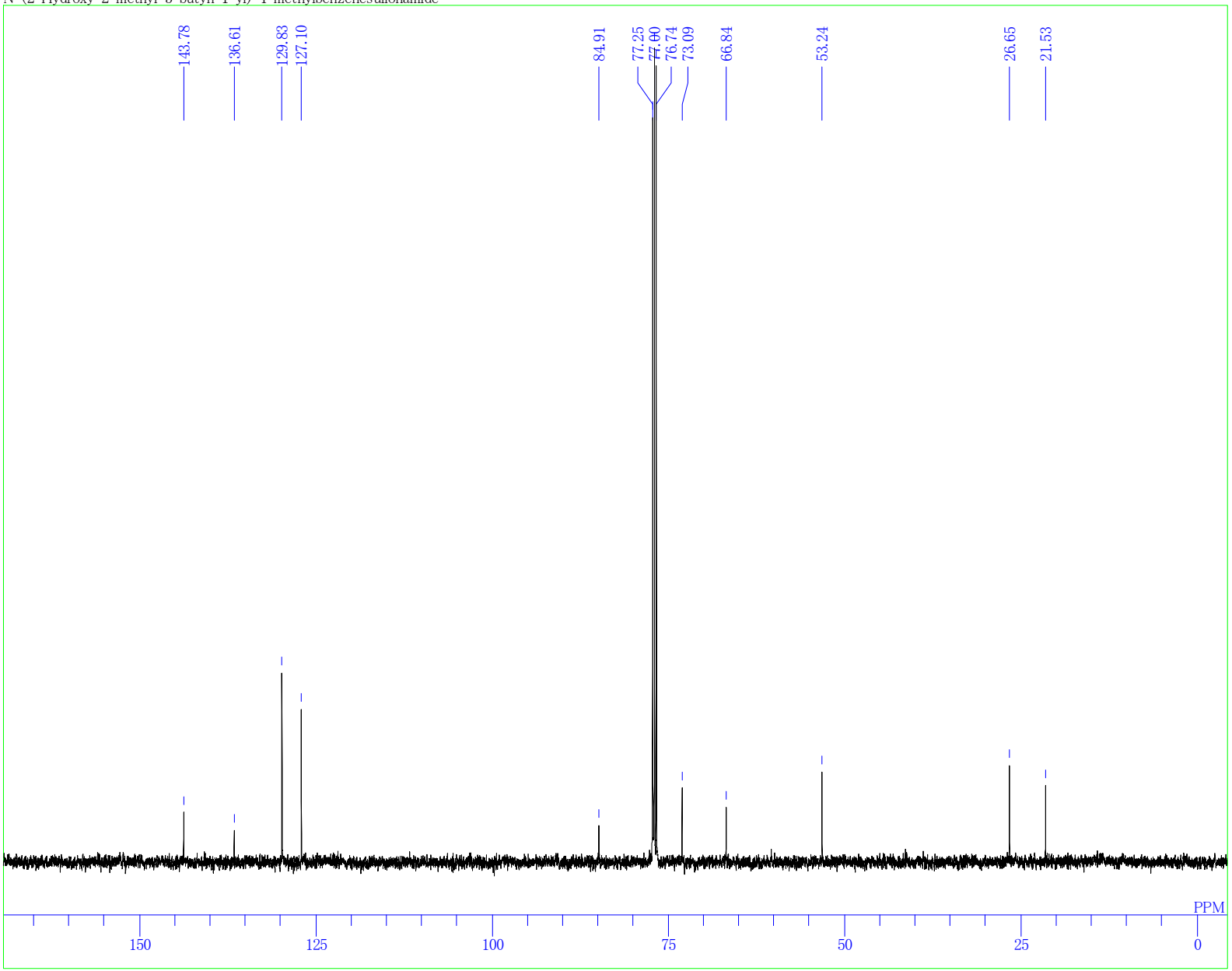


DATIM	11-07-2009 11:03:28
EXMOD	single_pulse.ex2
OBNUC	1H
OFR	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
OBATN	0
PW1	5.10 usec
PW2	0.00 usec
POINT	10485
SPO	10485
SCANS	8
FREQU	6005.82 Hz
PD	1.5000 sec
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	7.26 ppm
TMSP	3274

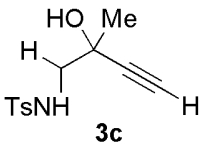


3c

F:¥pyrrole¥Yegi030335-13C.als
N-(2-Hydroxy-2-methyl-3-butyn-1-yl)-4-methylbenzenesulfonamide



DATIM	06-06-2009 19:53:25
EXMOD	single_pulse_dec
OBNUC	13C
OFR	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
OBATN	0
PW1	3.27 usec
PW2	0.00 usec
POINT	32768
SPO	32768
SCANS	136
FREQU	39308.18 Hz
PD	2.0000 sec
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
IRNUC	1H
IRFIN	6.01 Hz
IRATN	0
EXREF	77.00 ppm
TMSP	18783



3c