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–Supporting Information for–

Multifunctionalization of Alkenes via Aerobic Oxynitration and sp^3 C-H Oxidation

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General methods: All glassware was oven dried. All reagents purchased commercially were used without further purification. All solvents were used after dried by distillation from appropriate dehydrating reagents such as calcium hydride, sodium/benzophenone, or molecular sieves. Deuterated chloroform was passed through an alumina column before the use. IR spectra were recorded on Horiba IR-710 FT/IR spectrometer. ^{1}H and ^{13}C NMR spectra were recorded on JEOL JNM ECS400 (400 MHz and 100 MHz, respectively) and JEOL JNM ECA600 (600 MHz and 150 MHz, respectively) spectrometers. Chemical shifts (δ) are quoted relative to tetramethylsilane (^{1}H NMR) and the residual signals of chloroform (^{13}C NMR). Silica gel column chromatography was carried out on silica gel 60N (Kanto Kagaku Co., Ltd., spherical, neutral, 63-210 μm). Mass spectra were recorded on JEOL JMS-T100TD (direct analysis in real time, DART) or JEOL JMS-700 spectrometers (fast atom bombardment, FAB).

Starting meterials

1a-d were commercially available. **1e,**[1] **g,**[2] **h,**[3] **i,**[4] **j,**[5] **k**[6] were prepared according to literatures. **1f, l, m** were prepared by benzoylation of the corresponding commercially available or known alcohols[7,8].

2,4-Dimethylpent-4-en-2-yl benzoate (1f):

To a solution of 2,4-dimethylpent-4-penten-2-ol (50.0 mg, 0.458 mmol) in THF (0.9 mL) was added a solution of *n*-butyllithium (1.6 M, 0.30 mL, 0.482 mmol) at 0 °C, and the mixture was stirred for 15 min at same temperature. After benzoyl chloride (73.9 mg, 0.525 mmol) was added at 0 °C, the mixture was stirred for 18 h at room temperature. The reaction mixture was poured into a saturated solution of NH_4Cl and extracted with CH_2Cl_2. The organic layer was washed with brine and dried with Na_2SO_4. After the solvent was removed under reduced pressure, the residue was purified by silica gel chromatography (hexane/EtOAc, 20:1) to give **1f** (50.0 mg, 53%) as a colorless oil. IR ($CHCl_3$) υ 1707, 1286 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$) δ 1.61 (6H, s), 1.81 (3H, s), 2.67 (2H, s), 4.77 (1H, s), 4.90 (1H, s), 7.42 (2H, t, J = 8.0 Hz), 7.52 (1H, t, J = 7.9 Hz), 8.00 (2H, d, J = 7.9 Hz); HRFABMS calcd for $C_{14}H_{19}NO$ $[M+H]^+$ 219.1385, found: 219.1390.

2-(2-Methylallyl)propane-1,3-diyl dibenzoate (1l):

To a solution of 2-(2-methylallyl)propane-1,3-diol[7] (250 mg, 1.92 mmol) in CH_2Cl_2 (4 mL) was added triethylamine (780 mg, 7.68 mmol) and benzoyl chloride (1080 mg, 7.68 mmol) at 0 °C, and the mixture was stirred for 2 h at room temperature. The

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reaction mixture was poured into water and extracted with CH_2Cl_2. The organic layer was washed with brine and dried with Na_2SO_4. After the solvent was removed under reduced pressure, the residue was purified by silica gel chromatography (hexane/EtOAc, 10:1) to give **1l** (334.7 mg, 52%) as colorless crystals. Mp 43.0–43.5 °C (hexane); IR ($CHCl_3$) υ 1716, 1271 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 1.81 (3H, s), 2.29 (2H, d, J = 7.6 Hz), 2.51–2.57 (1H, m), 4.37 (2H, dd, J = 11.0, 6.2 Hz), 4.47 (2H, dd, J = 11.0, 4.5 Hz), 4.80 (1H, s), 4.87 (1H, s), 7.43 (4H, t, J = 7.6 Hz), 7.56 (2H, t, J = 7.6 Hz), 8.04 (4H, t, J = 7.2 Hz); ^{13}C NMR (150 MHz, $CDCl_3$) δ 22.2, 35.5, 37.0, 64.7, 113.3, 128.4, 129.6, 130.0, 133.0, 141.9, 166.5; HRFABMS calcd for $C_{21}H_{23}O_4$ $[M+H]^+$ 339.1596, found: 339.1591.

2-Methyl-2-(2-methylallyl)propane-1,3-diyl dibenzoate (1m):

To a solution of 2-methyl-2-(2-methylallyl)propane-1,3-diol[8] (120 mg, 0.832 mmol) in CH_2Cl_2 (3.0 mL) was added triethylamine (337 mg, 3.33 mmol) and benzoyl chloride (468 mg, 3.33 mmol) at 0 °C, and the mixture was stirred for 24 h at room temperature. The reaction mixture was poured into water and extracted with CH_2Cl_2. The organic layer was washed with brine and dried with Na_2SO_4. After the solvent was removed under reduced pressure, the residue was purified by silica gel chromatography (hexane/EtOAc, 10:1) to give **1m** (177.8 mg, 61%) as a colorless oil. IR ($CHCl_3$) υ 1718, 1281 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 1.17 (3H, s), 1.83 (3H, s), 2.31 (2H, s), 4.29 (2H, d, J = 11.0 Hz), 4.32 (2H, d, J = 11.0 Hz), 4.78 (1H, s), 4.97 (1H, s), 7.43 (4H, t, J = 7.7 Hz), 7.55 (2H, t, J = 7.6 Hz), 8.04 (4H, d, J = 8.2 Hz); ^{13}C NMR (150 MHz, $CDCl_3$) δ 19.9, 25.3, 38.4, 42.2, 68.4, 116.0, 128.4, 129.5, 129.9, 133.0, 140.7, 166.2; HRFABMS calcd for $C_{22}H_{25}O_4$ $[M+H]^+$ 353.1753, found: 353.1748.

General procedure for multifunctionalization reactions using *tert*-butyl nitrite and oxygen:

To a stirred solution of alkene (0.60 mmol) in dry DMSO (3.0 mL) was added *tert*-butyl nitrite (310 mg, 3.0 mmol), and the mixture was stirred at room temperature under O_2 atmosphere (1 atm, balloon). The mixture was diluted with water and extracted with Et_2O. The organic layer was washed with brine and dried with Na_2SO_4. After the solvent was removed under reduced pressure, the residue was purified by silica gel chromatography (hexane/EtOAc) to give the product. Since several products were unstable, these needed to be analyzed as soon as possible after purification.

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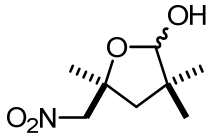
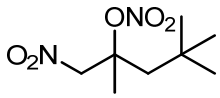


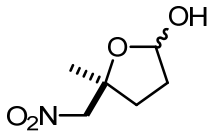
3,3,5-Trimethyl-5-nitromethyltetrahydro-2-furanol (2a):

Time: 5 h. Elute: hexane/EtOAc, 5:1 (second fraction). 11–52% yield. Colorless oil. Mixture of two isomers (75:25). IR ($CHCl_3$) υ 3587, 3408, 1550, 1383 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 1.11 (3H, s, [25/100]), 1.140 (3H, s, [75/100]), 1.146 (3H, s, [25/100]), 1.150 (3H, s), 1.48 (3H, s, [75/100]), 1.56 (3H, s, [25/100]), 1.74 (1H, d, J =12.7 Hz, [75/100]), 1.96 (1H, d, J =13.4 Hz, [25/100]), 2.05 (1H, d, J =13.4 Hz, [25/100]), 2.09 (1H, d, J =13.1 Hz, [75/100]), 2.72 (1H, br, [25/100]), 2.87 (1H, br, [75/100]), 4.42 (1H, d, J =11.0 Hz, [25/100]), 4.50 (1H, d, J =11.0 Hz, [25/100]), 4.52 (1H, d, J =11.0 Hz, [75/100]), 4.67 (1H, d, J =11.0 Hz, [75/100]), 5.01 (1H × 2, br, [25/100] and [75/100]); ^{13}C NMR (150 MHz, $CDCl_3$) δ 22.71, 22.77, 26.0, 27.2, 27.6, 28.4, 44.0, 44.5, 47.8, 47.9, 81.0, 81.5, 84.0, 86.0, 105.1, 105.4; HRFABMS calcd for $C_8H_{15}NNaO_4$ $[M+Na]^+$ 212.0899, found: 212.0897.



4,4-Dimethyl-2-nitromethyl-pent-2-yl nitrate (3a):

Elute: hexane/EtOAc, 5:1 (first fraction). 5–44% yield. Colorless oil. IR ($CHCl_3$) υ 1635, 1558, 1373, 1292 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 1.08 (9H, s), 1.70 (3H, s), 1.74 (1H, d, J = 15.5 Hz), 2.13 (1H, d, J = 15.5 Hz) 4.78 (1H, d, J = 11.7 Hz) 4.99 (1H, d, J = 11.7 Hz); ^{13}C NMR (150 MHz, $CDCl_3$) δ 22.1, 31.1, 31.4, 47.1, 80.5, 88.8; HRFABMS calcd for $C_8H_{16}N_2NaO_5$ $[M+Na]^+$ 243.0957, found: 243.0953.



5-Methyl-5-nitromethyltetrahydro-2-furanol (2b):

Time: 6 h. Elute: hexane/EtOAc, 2:1. 15% yield. Colorless oil. Mixture of two isomers (60:40). This compound was unstable. ^1H NMR (400 MHz, $CDCl_3$) δ 1.38 (3H, s, [60/100]), 1.55 (3H, s, [40/100]), 1.84–1.90 (2H, m, [40/100]), 2.00–2.17 and 2.22–2.34 (4H [60/100] + 2H [40/100] both m), 3.20 (1H × 2, br, [60/100] and [40/100]), 4.35 (1H, d, J = 11.0 Hz, [40/100]), 4.38 (1H, d, J = 11.0 Hz, [40/100]), 4.58 (1H, d, J = 11.0 Hz, [60/100]), 4.63 (1H, d, J =11.0 Hz, [60/100]), 5.56–5.59 (1H × 2, m, [60/100] and

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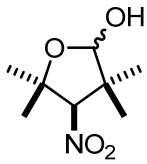
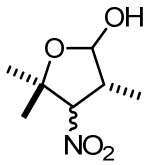
[40/100]).



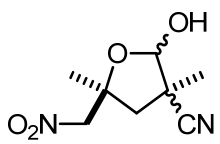
3,3,5,5-Tetramethyl-4-nitrotetrahydro-2-furanol (2c):

Time: 6 h. Elute: hexane/EtOAc, 5:1. 44% yield. Colorless oil. Mixture of two isomers (75:25). IR ($CHCl_3$) υ 3602, 3516, 3410, 1550, 1371 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 1.23 (3H, s, [25/100]), 1.27 (3H, s, [75/100]), 1.30 (3H, s, [75/100]), 1.36 (3H, s, [75/100]), 1.37 (3H, s, [25/100]), 1.45 (3H, s, [25/100]), 1.53 (3H, s, [25/100]), 1.62 (3H, s, [75/100]), 3.33 (1H, br-s, [75/100]), 4.36 (1H, d, J = 11.7 Hz, [25/100]), 4.73 (1H, s, [25/100]), 4.86 (1H, s, [75/100]), 4,95 (1H, d, J = 11.7 Hz, [25/100]), 5.12 (1H, d, J = 2.7 Hz, [75/100]); ^{13}C NMR (150 MHz, $CDCl_3$) δ 18.9, 21.1, 21.8, 25.3, 27.0, 28.3, 31.3, 32.4, 47.7, 48.7, 81.2, 83.5, 99.0, 103.6, 103.9, 105.6; HRDARTMS calcd for $C_8H_{16}NO_4$ $[M+H]^+$ 190.1079, found: 190.1050.



3,5,5-Trimethyl-4-nitrotetrahydro-2-furanol (2d):

Time: 5 h. Elute: hexane/EtOAc, 5:1. 54% yield. Colorless oil. Mixture of two isomers (75:25). IR ($CHCl_3$) υ 3604, 3423, 1550, 1371 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 1.15 (3H, d, J = 7.2 Hz, [75/100]), 1.16 (3H, s, [75/100]), 1.27 (3H, d, J = 7.2 Hz, [25/100]), 1.33 (3H, s, [25/100]), 1.51 (3H, s, [25/100]), 1.64 (3H, s, [75/100]), 3.03-3.10 (1H × 2, m, [75/100] and [25/100]), 3.41 (1H × 2, br, [75/100] and [25/100]), 4.43 (1H, d, J = 7.9 Hz, [25/100]), 4.75 (1H, d, J = 11.0 Hz, [75/100]), 5.10 (1H, d, J = 4.5 Hz, [25/100]), 5.36 (1H, d, 4.8 Hz, [75/100]); ^{13}C NMR (150 MHz, $CDCl_3$) δ 11.0, 15.4, 23.7, 24.0, 28.5, 30.7, 42.1, 44.9, 82.2, 82.4, 96.5, 97.5, 97.7, 102.4; HRDARTMS calcd for $C_7H_{14}NO_4$ $[M+H]^+$ 176.0923, found: 176.0972.



2-Hydroxy-3,5-dimethyl-5-(nitromethyl)tetrahydrofuran-3-carbonitrile (2e):

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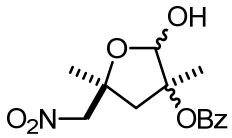
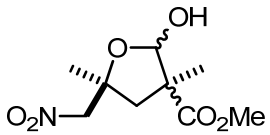
Time: 10 h. Elute: hexane/EtOAc, 2.5:1. 51% yield. Colorless oil. Mixture of four isomers (40:33:20:7). A small amount of impurities based on degradation were detected in NMR spectra because this compound was relatively unstable. IR ($CHCl_3$) υ 3595, 3405, 2245, 1554, 1381 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$, data of the minor isomer [8/100] are only characteristic peaks) δ 1.50 (3H, s, [40/100]), 1.52 (3H, s, [33/100]), 1.53 (3H, s, [19/100]), 1.54 (3H, s, [40/100]), 1.58 (3H, s, [19/100]), 1.60 (3H, s, [33/100]), 2.09 (1H, d, J = 13.7 Hz, [33/100]), 2.14 (1H, d, J = 13.7 Hz, [19/100]), 2.37 (1H, d, J = 13.7 Hz, [40/100]), 2.44 (1H, d, J = 13.7 Hz, [40/100]), 2.47 (1H, d, J = 14.1 Hz, [8/100]), 2.62 (1H, d, J = 14.1 Hz, [8/100]), 2.84 (1H, d, J = 14.1 Hz, [19/100]), 2.85 (1H, d, J = 13.7 Hz, [33/100]), 3.29 (1H, br, [8/100]), 3.85 (1H, br, [19/100]), 4.04 (1H, br, [33/100]), 4.31 (1H, br, [40/100]), 4.58 (1H, d, J = 11.3 Hz, [40/100]), 4.61 (1H, d, J = 11.3 Hz, [40/100]), 4.62 (1H, d, J = 11.4 Hz, [19/100]), 4.65 (1H, d, J = 11.7 Hz, [33/100]), 4.66 (1H, d, J = 11.4 Hz, [19/100]), 4.73 (1H, d, J = 11.7 Hz, [33/100]), 5.21 (1H, s, [8/100]), 5.32 (1H, s, [40/100]), 5.507 (1H, s, [33/100]), 5.513 (1H, s, [19/100]); ^{13}C NMR (150 MHz, $CDCl_3$, several peaks of the minor isomer [8/100] were overlapped or not distinguished) δ 18.8, 22.4, 23.4, 25.2, 25.9, 26.5, 27.2, 27.5, 28.2, 43.7, 44.1, 44.7, 44.8, 45.0, 45.5, 81.7, 81.8, 82.2, 84.6, 84.7, 100.0, 100.2, 1002.2, 120.7, 120.9, 122.77, 122.83; HRDARTMS calcd for $C_8H_{13}N_2O_4$ $[M+H]^+$ 201.0875, found: 201.0830.



2-Hydroxy-3,5-dimethyl-5-(nitromethyl)tetrahydrofuran-3-yl benzoate (2f):

Time: 8 h. Elute: hexane/EtOAc, 3:1. 52% yield. Colorless oil. Mixture of four isomers (47:25:25:3). IR ($CHCl_3$) υ 3600, 3419, 1716, 1552, 1380 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$, data of the minor isomer [3/100] are only characteristic peaks) δ 1.46 (3H, s, [25/100]), 1.52 (3H, s, [25/100]), 1.57 (3H, s, [3/100]), 1.61 (3H, s, [47/100]), 1.70 (3H, s, [3/100]), 1.72 (3H, s, [25/100]), 1.78 (3H, s, [25/100]), 1.79 (3H, s, [47/100]), 2.22 (1H, d, J = 14.8 Hz, [47/100]), 2.31 (1H, d, J = 13.4 Hz, [25/100]), 2.42 (1H, d, J = 14.4 Hz, [25/100]), 2.66 (1H, d, J = 14.4 Hz, [3/100]), 2.73 (1H, d, J = 14.4 Hz, [25/100]), 2.82 (1H, d, J = 13.4 Hz, [25/100]), 3.00 (1H, d, J = 14.8 Hz, [47/100]), 3.36 (1H, br, [47/100]), 3.42 (1H, br, [25/100]), 3.53 (1H, br, [25/100]), 4.47 (1H, d, J = 10.7 Hz, [47/100]), 4.52 (1H, d, J = 10.7 Hz, [47/100]), 4.59 (1H, d, J = 11.0 Hz, [25/100]), 4.62 (1H, d, J = 11.0 Hz, [25/100]), 4.67 (1H, d, J = 11.3 Hz, [25/100]), 4.69 (1H, d, J = 11.3 Hz, [25/100]), 5.48 (1H, s, [3/100]), 5.65 (1H, s, [25/100]), 5.79 (1H, s, [25/100]), 5.84

(1H, s, [47/100]), 7.44–7.48 (2H × 4, m, all isomers), 7.56–7.60 (1H × 4, m, all isomers), 7.99–8.03 (2H × 4, m, all isomers); ^{13}C NMR (150 MHz, $CDCl_3$, several peaks of the minor isomer [3/100] were overlapped or not distinguished) δ 18.2, 22.6, 23.3, 25.6, 27.6, 44.5, 44.7, 45.7, 78.8, 81.4, 81.5, 83.2, 84.9, 85.1, 85.2, 90.4, 90.7, 100.7, 100.9, 101.2, 128.4, 128.5, 128.6, 129.49, 129.51, 129.7, 130.0, 130.3, 130.5, 133.2, 133.3, 133.4, 165.38, 165.40, 165.45; HRFABMS calcd for $C_{14}H_{18}NO_6$ $[M+H]^+$ 296.1134, found: 296.1132.



Methyl 2-hydroxy-3,5-dimethyl-5-(nitromethyl)tetrahydrofuran-3-carboxylate (2g):

Time: 19 h. Elute: hexane/EtOAc, 2:1. 45% yield. Colorless oil. Mixture of four isomers (35:35:25:5). A small amount of impurities based on degradation were detected in NMR spectra because this compound was relatively unstable. IR ($CHCl_3$) υ 3599, 3406, 1732, 1552, 1381 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$, data of the minor isomer [5/100] are only characteristic peaks) δ 1.35 (3H, s, [35/100]), 1.36 (3H, s, [35/100]), 1.38 (3H, s, [25/100]), 1.39 (3H, s, [5/100]), 1.44 (3H, s, [35/100]), 1.51 (3H, s, [35/100]), 1.58 (3H, s, [25/100]), 1.60 (3H, s, [5/100]), 1.82 (1H, d, J = 13.4 Hz, [35/100]), 1.93 (1H, d, J = 13.4 Hz, [25/100]), 2.09 (1H, d, J = 13.1 Hz, [35/100]), 2.15 (1H, d, J = 13.7 Hz, [5/100]), 2.71 (1H, d, J = 13.4 Hz, [35/100]), 2.79 (1H, d, J = 13.7 Hz, [5/100]), 2.93 (1H × 2, d, J = 13.4 Hz, [35/100] and [25/100]), 3.54 (1H, br, [25/100]), 3.65 (1H, br, [35/100]), 3.745 (3H, s, [35/100]), 3.751 (3H, s, [35/100]), 3.757 (3H, s, [5/100]), 3.765 (3H, s, [25/100]), 3.88 (1H, br, [35/100]), 4.02 (1H, br, [5/100]), 4.39 (1H, d, J = 11.3 Hz, [25/100]), 4.45 (1H, d, J = 11.3 Hz, [25/100]), 4.56 (1H, d, J = 11.0 Hz, [35/100]), 4.59 (1H, d, J = 11.1 Hz, [35/100]), 4.64 (1H, d, J = 11.0 Hz, [35/100]), 4.74 (1H, d, J = 11.0 Hz, [35/100]), 5.23 (1H, d, J = 4.2 Hz, [5/100]), 5.28 (1H, d, J = 3.0 Hz, [35/100]), 5.67 (1H, d, J = 2.8 Hz, [25/100]), 5.71 (1H, d, J = 2.4 Hz, [35/100]); ^{13}C NMR (150 MHz, $CDCl_3$, several peaks of the minor isomer [5/100] were overlapped or not distinguished) δ 19.1, 19.2, 22.7, 23.3, 24.7, 26.1, 27.9, 42.5, 42.7, 42.9, 43.3, 52.4, 52.6, 52.7, 55.67, 55.72, 55.82, 81.2, 81.3, 81.9, 82.8, 83.7, 85.3, 85.4, 100.9, 103.6, 103.7, 173.5, 174.1, 175.1, 175.4; HRFABMS calcd for $C_9H_{15}NNaO_6$ $[M+Na]^+$ 256.0797, found: 256.0797.

Oxidation of 2e–g:

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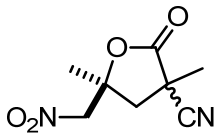
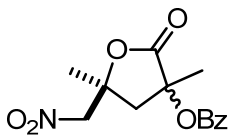
2e–g were relatively unstable and obtained as the mixture of four isomers, and these complicated NMR spectra. Therefore, **2e–g** were oxidized into the corresponding lactones in order to confirm production of **2e–g** and estimate diastereomeric ratios except the hemiacetal stereocenter. An excessive amount of Dess-Martin periodinane was used for oxidation of **2e–g** because IBX oxidation did not proceed in these cases.

To a solution of **2e–g** (0.25 mmol) and *t*BuOH (55.6 mg, 0.75 mmol) in CH_2Cl_2 (2.5 ml) was added Dess-Martin periodinane (530.2 mg, 1.25 mmol), and the mixture was stirred at room temperature for 10 h. After a 10% aqueous solution of $Na_2S_2O_3$ was added to the reaction mixture, the mixture was extracted with Et_2O. The organic layer was successively washed with brine and dried with Na_2SO_4. After removal of solvent under reduced pressure, the residue was purified by silica gel chromatography (hexane/EtOAc, 2.5:1) to give lactone products. Although contamination by a small amount of impurities based on Dess-Martin periodinane was observed due to the use of the excessive amount of the reagent and the relatively high polarity of products, this did not affect identification of products and estimation of diastereomeric ratios.



3,5-Dimethyl-5-(nitromethyl)-2-oxotetrahydrofuran-3-carbonitrile:

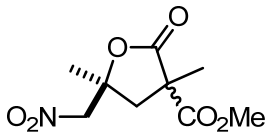
<65% yield. Colorless oil. Mixture of two isomers (50:50). Elute: hexane/EtOAc (1:1). IR ($CHCl_3$) υ 2247, 1797, 1562, 1379, 1290 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.66 (3H, s), 1.74 (3H, s), 1.77 (3H, s), 1.80 (3H, s), 2.28 (1H, d, J = 14.2 Hz), 2.69 (1H, d, J = 15.2 Hz), 2.84 (1H, d, J = 14.2 Hz), 3.28 (1H, d, J = 14.2 Hz), 4.61 (1H, d, J = 13.6 Hz), 4.67 (1H, d, J = 5.2 Hz), 4.70 (1H, d, J = 5.2 Hz), 4.77 (1H, d, J = 13.6 Hz). ^{13}C NMR (150 MHz, $CDCl_3$) δ 22.9, 24.4, 25.0, 25.9, 38.5, 39.0, 43.0, 43.3, 79.9, 80.1, 80.2, 80.3, 118.3, 118.7, 169.0, 169.6; HRFABMS calcd for $C_8H_{11}N_2O_4$ $[M+H]^+$ 199.0719, found: 199.0726.



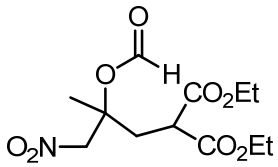
3,5-Dimethyl-5-(nitromethyl)-2-oxotetrahydrofuran-3-yl benzoate:

<76% yield. Colorless oil. Mixture of two isomers (75:25). Elute: hexane/EtOAc (3:1). IR ($CHCl_3$) υ 1793, 1718, 1558, 1380, 1277 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 1.66 (3H, s, [75/100]), 1.76 (3H, s, [25/100]), 1.80 (3H, s, [25/100]), 1.81 (3H, s, [75/100]),

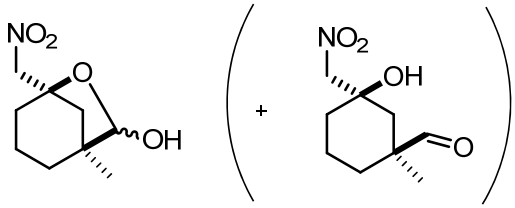
2.38 (1H, d, J = 14.8 Hz, [75/100]), 2.70 (1H, d, J = 14.8 Hz, [25/100]), 2.78 (1H, d, J = 14.8 Hz, [25/100]), 3.05 (1H, d, J = 14.8 Hz, [60/100]), 4.61 (2H, s, [25/100]), 4.84 (1H, d, J = 12.6 Hz, [75/100]), 4.99 (1H, d, J = 12.6 Hz, [75/100]), 7.43–7.47 (2H × 2, m, all isomers), 7.59–7.62 (1H × 2, m, all isomers), 8.00–8.06 (2H × 2, m, all isomers). ^{13}C NMR (150 MHz, $CDCl_3$) δ 24.9, 25.5, 25.7, 26.0, 42.1, 43.8, 77.4, 78.3, 81.7, 82.0, 128.0, 128.3, 128.51, 128.53, 129.87, 129.93, 131.9, 133.4, 133.9, 134.0, 165.3, 165.8, 173.0, 173.2; HRFABMS calcd for $C_{14}H_{16}NO_6$ $[M+H]^+$ 294.0978, found: 294.0971.



Methyl 3,5-dimethyl-5-(nitromethyl)-2-oxotetrahydrofuran-3-carboxylate:
<77% yield. Colorless oil. Mixture of two isomers (60:40). Elute: hexane/EtOAc (1:1). IR ($CHCl_3$) υ 1786, 1734, 1560, 1381, 1284 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.59 (3H, s, [60/100]), 1.61 (3H, s, [60/100]), 1.63 (3H, s, [40/100]), 1.66 (3H, s, [40/100]), 2.11 (1H, d, J = 14.7 Hz, [40/100]), 2.49 (1H, d, J = 14.2 Hz, [60/100]), 2.81 (1H, d, J = 14.2 Hz, [60/100]), 3.17 (1H, d, J = 14.7 Hz, [40/100]), 3.81 (3H, s, [60/100]), 3.82 (3H, s, [40/100]), 4.57 (1H, d, J = 13.2 Hz, [60/100]), 4.65 (1H × 2, d, J = 12.8 Hz, [60/100] and [40/100]), 4.74 (1H, d, J = 12.4 Hz, [60/100]). ^{13}C NMR (150 MHz, $CDCl_3$) δ 22.2, 22.8, 25.3, 26.5, 42.3, 43.3, 51.4, 51.7, 53.5, 53.6, 79.4, 79.7, 81.1, 81.4, 170.8, 171.1, 173.4, 173.5. HRFABMS calcd for $C_9H_{14}NO_6$ $[M+H]^+$ 232.0821, found: 232.0822.

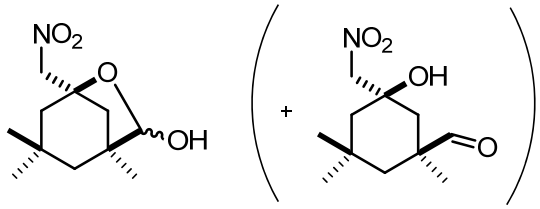


Diethyl (2-formyloxy-2-nitromethylbutyl) malonate (2h):
Time: 24 h. Elute: hexane/EtOAc, 3:1. 54% yield. Colorless oil. This compound was unstable. ^{1}H NMR (600 MHz, $CDCl_3$) δ 1.25–1.30 (6H, m), 1.61 (3H, s), 2.47 (1H, dd, J = 15.1, 6.5 Hz), 2.73 (1H, dd, J = 15.1, 6.5 Hz), 3.56 (1H, t, J = 6.5 Hz), 4.19–4.27 (4H, m), 4.94 (1H, d, J = 11.7 Hz), 4.96 (1H, d, J = 11.7 Hz), 7.93 (1H, s). ^{13}C NMR (150 MHz, $CDCl_3$) δ 13.9, 21.8, 35.4, 47.1, 61.9, 62.0, 79.69, 79.72, 159.4, 168.5, 168.7.



(1*R,5*S**)-1-Methyl-5-nitromethyl-6-oxabicyclo[3.2.1]octan-7-ol (2i):**

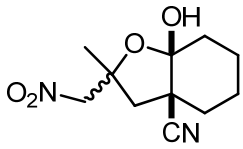
Time: 8 h. Elute: hexane/EtOAc, 3:1. 34% yield. Mixture of two isomer (90:10, and a small amount of (1*R**,3*S**)-3-hydroxy-1-methyl-3-(nitromethyl)cyclohexanecarbaldehyde was included (92:8)), 46% NMR yield (internal standard: mesitylene). Colorless oil. This compound was unstable. IR ($CHCl_3$) υ 3597, 3430, 1552, 1383 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$, data of the minor isomer [10/100] and the ring-opening aldehyde are only characteristic peaks) δ 0.88 and 1.02 (each 3H, s, [10/100] and [aldehyde]), 1.06 (3H, s, [90/100]), 1.29–1.43 (2H, m, [90/100]), 1.44 (1H, d, J = 11.7 Hz, [90/100]), 1.57–1.60 (1H, m, [90/100]), 1.62–1.71 (3H × 2, m, [90/100] and [10/100]), 1.88 (1H, d-like, J = 13.1 Hz, [10/100] or [aldehyde]), 1.98 (2H, d, J = 11.7 Hz, [10/100] and/or [aldehyde]), 2.06 (1H, d, J = 11.4 Hz, [90/100]), 2.18–2.23 (3H, m, [10/100] and/or [aldehyde]), 3.33 (1H, br, [90/100]), 3.89 (1H, br, [10/100]), 4.42 (2H, s, [10/100] or [aldehyde]), 4.48 (1H, d, J = 11.0 Hz, [10/100] or [aldehyde]), 4.53 (1H, d, J = 11.0 Hz, [10/100] or [aldehyde]), 4.56 (1H, d, J = 11.0 Hz, [90/100]), 4.60 (1H, d, J = 11.0 Hz, [90/100]), 5.02 (1H, s, [90/100]), 5.17 (1H, d, J = 4.8 Hz, [10/100]), 9.44 (1H, s, [aldehyde]); ^{13}C NMR (150 MHz, $CDCl_3$, several peaks of the minor isomer [10/100] and aldehydes were overlapped or not distinguished) δ 17.6, 19.12, 19.15 (major), 20.3 (major), 21.9, 24.1, 31.4, 31.8, 32.6 (major), 33.1, 33.8, 34.7 (major), 41.7, 42.8, 43.3 (major), 44.9 (major), 45.3, 46.3, 70.6, 82.0 (major), 82.2 (major), 82.3, 85.3, 102.4 (major), 105.3, 205.0 (aldehyde); HRFABMS calcd for $C_9H_{16}NO_4$ $[M+H]^+$ 202.1079, found: 202.1083.



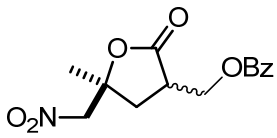
(1*R,5*S**)-1,3,3-Trimethyl-5-nitromethyl-6-oxabicyclo[3.2.1]octan-7-ol (2j):**

Time: 9 h. Elute: hexane/EtOAc, 4:1. 46% yield (A small amount of (1*R**,3*S**)-3-hydroxy-1,5,5-methyl-3-(nitromethyl)cyclohexanecarbaldehyde was included (93:7)). Colorless oil. IR ($CHCl_3$) υ 3597, 3442, 1552, 1383 cm^{-1}; ^1H NMR

(600 MHz, $CDCl_3$, data of the ring-opening aldehyde are only characteristic peaks) δ 0.97 (3H, s, [aldehyde]), 0.98 (3H, s, [aldehyde]), 1.01 (3H, s, [aldehyde]), 1.02 (3H, s), 1.06 (3H, s), 1.14 (3H, s), 1.36 (1H, d, J = 13.7 Hz), 1.39 (1H, d, J = 11.3 Hz), 1.41 (1H, d, J = 14.1 Hz), 1.60 (1H, dd, J = 14.4, 1.0 Hz), 1.66 (1H, dd, J = 13.7, 1.7 Hz), 2.11 (1H, dt, J = 11.3, 2.4 Hz), 3.12 (1H, br d, J = 13.4 Hz), 4.36 (1H, d, J = 11.3 Hz, [aldehyde]), 4.40 (1H, d, J = 11.3 Hz, [aldehyde]), 4.54 (1H, d, J = 11.0 Hz), 4.59 (1H, d, J = 11.0 Hz), 5.08 (1H, d, J = 3.8 Hz), 9.53 (1H, s, [aldehyde]); ^{13}C NMR (150 MHz, $CDCl_3$) δ 20.4, 25.5 (aldehyde), 27.3 (aldehyde), 30.5, 32.0, 34.7 (aldehyde), 36.0, 39.6 (aldehyde), 42.0, 45.2 (aldehyde), 45.5, 45.9 (aldehyde), 46.0 (aldehyde), 49.4, 82.4, 86.2 (aldehyde), 102.0, 206.4 (aldehyde); HRDARTMS calcd for $C_{11}H_{20}NO_4$ $[M+H]^+$ 230.1392, found: 236.1393.

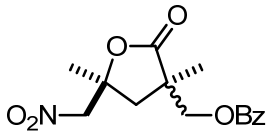


Octahydro-7a-hydroxy-2-methyl-2-nitromethyl-benzofuran-3a-carbonitrile (2k):
Time: 10 h. Elute: hexane/EtOAc, 3:1. 55% yield. White solids. Mixture of two diastereoisomers (55:45). IR ($CHCl_3$) υ 3587, 3375, 2245, 1554, 1379 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$) δ 1.35–1.62 (3H × 2, m, [55/100] and [45/100]), 1.55 (3H, s), 1.68 (3H, s, [55/100]), 1.70–1.82 (3H × 2, m, [55/100] and [45/100]), 2.08–2.18 (2H × 2, m, [55/100] and [45/100]), 2.11 (1H, d, J = 13.1 Hz, [45/100]), 2.44 (1H, d, J = 13.4 Hz, [55/100]), 2.71 (1H, d, J = 13.4 Hz, [55/100]), 2.84 (1H, br-s, [55/100]), 2.87 (1H, d, J = 13.4 Hz, [45/100]), 3.09 (1H, br-s, [45/100]), 4.47 (1H, d, J = 11.3 Hz, [55/100]), 4.55 (1H, d, J = 11.3 Hz, [55/100]), 4.67 (1H, d, J = 11.0 Hz, [45/100]), 4.78 (1H, d, J = 11.0 Hz, [45/100]); ^{13}C NMR (150 MHz, $CDCl_3$) δ 22.1, 22.2, 22.27, 22.34, 26.1, 28.4, 33.5, 33.9, 35.1, 35.2, 45.1, 46.0, 46.1, 46.4, 80.2, 80.6, 83.7, 85.1, 104.4, 104.9, 120.5, 120.6; HRDARTMS calcd for $C_{11}H_{17}N_2O_4$ $[M+H]^+$ 241.1188, found: 241.1212.



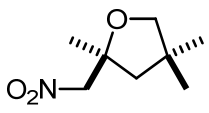
3-Benzoyloxymethyl-5-methyl-5-nitromethyltetrahydro-2-furanone (2l):
Time: 30 h. Elute: hexane/EtOAc, 3:1. 47% NMR yield (internal standard: mesitylene). White solid. Mixture of two diastereoisomers (65:35). Two isomers were inseparable, and one isomer was likely to be somewhat unstable because the diastereomer ratio

significantly changed after silica gel chromatography. Therefore, the yield was determined by ^{1}H NMR analysis, and the products were partially purified for identification. IR ($CHCl_3$) υ 3030, 1786, 1724, 1560, 1560, 1377, 1271, 1115 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$) δ 1.59 (3H, s, for minor isomer), 1.61 (3H, s, for major isomer), 2.20 (1H, dd, J = 13.7, 10.0 Hz, for major isomer), 2.45 (1H, dd, J = 13.1, 9.6 Hz, for minor isomer), 2.54 (1H, t, J = 11.0 Hz, for minor isomer), 2.84 (1H, dd, J = 13.7, 10.3 Hz, for major isomer), 3.29–3.35 (1H × 2, m, for major and nimor isomers), 4.54–4.71 (4H × 2, m, for major and nimor isomers), 7.46 (2H × 2, t, J = 7.6 Hz, for major and nimor isomers), 7.57–7.61 (1H × 2, m, for major and nimor isomers), 8.01 (2H × 2, t, J = 6.9 Hz, for major and nimor isomers); ^{13}C NMR (150 MHz, $CDCl_3$, one peak was overlapped or not distinguished) δ 24.4, 26.5, 34.3, 34.5, 39.8, 40.5, 62.3, 62.8, 79.9, 80.3, 80.9, 81.6, 128.5, 128.5, 129.20, 129.22, 129.6, 129.7, 133.4, 133.5, 166.07, 166.13, 173.4; HRFABMS calcd for $C_{14}H_{16}NO_6$ $[M+H]^+$ 294.0978, found: 294.0982.



3-Benzoyloxymethyl-3,5-dimethyl-5-nitromethyltetrahydro-2-furanone (2m):

Time: 29 h. Elute: hexane/EtOAc, 2:1. 50% yield. Colorless oil. Mixture of two diastereoisomers (50:50). IR ($CHCl_3$) υ 3032, 1782, 1724, 1558, 1375, 1269, 1111 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$) δ 1.43 (3H, s), 1.49 (3H, s), 1.58 (3H, s), 1.67 (3H, s), 2.15 (1H, d, J = 14.1 Hz), 2.39 (1H, d, J = 14.1 Hz), 2.51 (1H, d, J = 14.1 Hz), 2.72 (1H, d, J = 14.1 Hz), 4.34 (1H, d, J = 11.0 Hz), 4.37 (1H, d, J = 11.0 Hz), 4.41 (1H, d, J = 11.0 Hz), 4.43 (1H, d, J = 11.0 Hz), 4.55 (1H, d, J = 12.0 Hz), 4.59 (1H, d, J = 12.7 Hz) 4.60 (1H, d, J = 12.0 Hz), 4.64 (1H, d, J = 12.7 Hz), 7.44–7.47 (4H, m), 7.58–7.60 (2H, m), 7.99–8.01 (4H, m); ^{13}C NMR (150 MHz, $CDCl_3$, four peaks were overlapped) δ 22.2, 23.0, 26.6, 40.4, 41.0, 44.7, 45.1, 68.4, 68.9, 78.9, 79.0, 82.0, 82.1, 128.56, 128.58, 129.1, 129.5, 129.6, 133.5, 165.9, 177.2, 177.4; HRDARTMS calcd for $C_{15}H_{18}NO_6$ $[M+H]^+$ 308.1134, found: 308.1163.



2,4,4-Trimethyl-2-nitromethyltetrahydrofuran (4a):

To a stirred mixture of **2a** (30.0 mg, 0.16 mmol) and triethylsilane (37.2 mg, 0.32 mmol) in CH_2Cl_2 (1.6 ml) was added BF_3·OEt_2 (45.4 mg, 0.32 mmol) at -78 °C and

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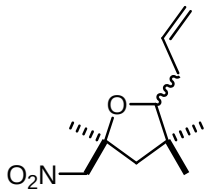
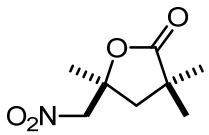
stirred for 30 min at room temperature. After addition of water, the mixture was extracted with CH_2Cl_2. The organic layer was successively washed with brine and dried with Na_2SO_4. After removal of solvent under reduced pressure, the residue was purified by silica gel chromatography (hexane/EtOAc, 5:1) to give **4a** (22.5 mg, 82%) as a colorless oil. IR ($CHCl_3$) υ 1550, 1381, 1041 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 1.12 (3H, s), 1.17 (3H, s), 1.45 (3H, s), 1.70 (1H, d, J = 13.4 Hz), 1.98 (1H, d, J = 13.4 Hz), 3.55 (1H, d, J = 8.6 Hz), 3.61 (1H, d, J = 8.6 Hz) 4.39 (1H, d, J = 11.0 Hz), 4.48 (1H, d, 11.0 Hz); ^{13}C NMR (150 MHz, $CDCl_3$) δ 25.6, 26.7, 27.5, 40.4, 49.9, 79.5, 81.4, 83.0; HRFABMS calcd for $C_8H_{16}NO_3$ $[M+H]^+$ 174.1130, found: 174.1129.



5-Allyl-2,4,4-trimethyl-2-nitromethyltetrahydrofuran (5a):

To a stirred mixture of **2a** (30.0 mg, 0.16 mmol) and allyltrimethylsilane (36.3 mg, 0.32 mmol) in CH_2Cl_2 (1.6 ml) was added $BF_3{\cdot}OEt_2$ (45.4 mg, 0.32 mmol) at -78 °C and stirred for 30 min at room temperature. After addition of water, the mixture was extracted with CH_2Cl_2. The organic layer was successively washed with brine and dried with Na_2SO_4. After removal of solvent under reduced pressure, the residue was purified by silica gel chromatography (hexane/EtOAc, 6:1) to give **5a** (18.3 mg, 55%) as a colorless oil. Mixture of two diastereoisomers (55:45). IR ($CHCl_3$) υ 1641, 1550, 1381 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 0.96 (3H, s, [55/100]), 1.01 (3H, s, [45/100]), 1.04 (3H, s, [45/100]), 1.06 (3H, s, [55/100]), 1.38 (3H, s, [55/100]), 1.46 (3H, s, [45/100]), 1.71 (1H, d, J = 13.7 Hz, [45/100]), 1.74 (1H, d, J = 13.4 Hz, [55/100]), 2.03 (1H, d, J = 13.1 Hz, [45/100]), 2.13–2.26 (2H × 2, m, [55/100] and [45/100]), 2.17 (1H, d, J = 13.4 Hz, [55/100]), 3.55 (1H, dd, J = 8.9, 3.8 Hz, [45/100]), 3.61 (1H, dd, J = 8.6, 4.5 Hz, [55/100]), 4.35 (1H, d, J = 10.7 Hz, [45/100]), 4.42 (1H, d, J = 11.0 Hz, [55/100]), 4.45 (1H, d, J = 11.0 Hz, [55/100]), 4.47(1H, d, J = 10.7 Hz, [45/100]), 5.04–5.15 (2H × 2, m, [55/100] and [45/100]), 5.81–5.88 (1H × 2, m, [55/100] and [45/100]); ^{13}C NMR (150 MHz, $CDCl_3$) δ 22.8, 22.9, 25.6, 25.7, 26.3, 26.4, 33.7, 33.9, 41.8, 42.0, 51.1, 51.2, 78.0, 78.4, 83.1, 84.0, 85.4, 86.2, 116.3, 116.4, 135.4, 135.5; HRDARTMS calcd for $C_{11}H_{20}NO_3$ $[M+H]^+$ 214.1443, found: 214.1396.



3,3,5-Trimethyl-5-nitromethyltetrahydro-2-furanone (6a):

To a solution of **2a** (34.6 mg, 0.18 mmol) in DMSO (1.0 ml) was added IBX (256 mg, 0.91 mmol) and the mixture was stirred at room temperature for 48 h. After addition of 10% aqueous solution of $Na_2S_2O_3$ to reaction mixture, the mixture was extracted with Et_2O. The organic layer was successively washed with brine and dried with Na_2SO_4. After removal of solvent under reduced pressure, the residue was purified by silica gel chromatography (hexane/EtOAc, 2.5:1) to give **6a** (28.0 mg, 82%) as a colorless oil. IR ($CHCl_3$) υ 1782, 1558, 1385 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 1.34 (3H, s), 1.39 (3H, s), 1.60 (3H, s), 2.12 (1H, d, J = 13.7 Hz), 2.47 (1H, d, J = 13.7 Hz), 4.57 (1H, d, J = 12.7 Hz) 4.61 (1H, d, J = 12.7 Hz); ^{13}C NMR (150 MHz, $CDCl_3$) δ 26.6, 26.7, 27.9, 40.4, 45.0, 78.3, 82.0, 180.4; HRFABMS calcd for $C_8H_{14}NO_4$ $[M+H]^+$ 188.0923, found: 188.0907.

References

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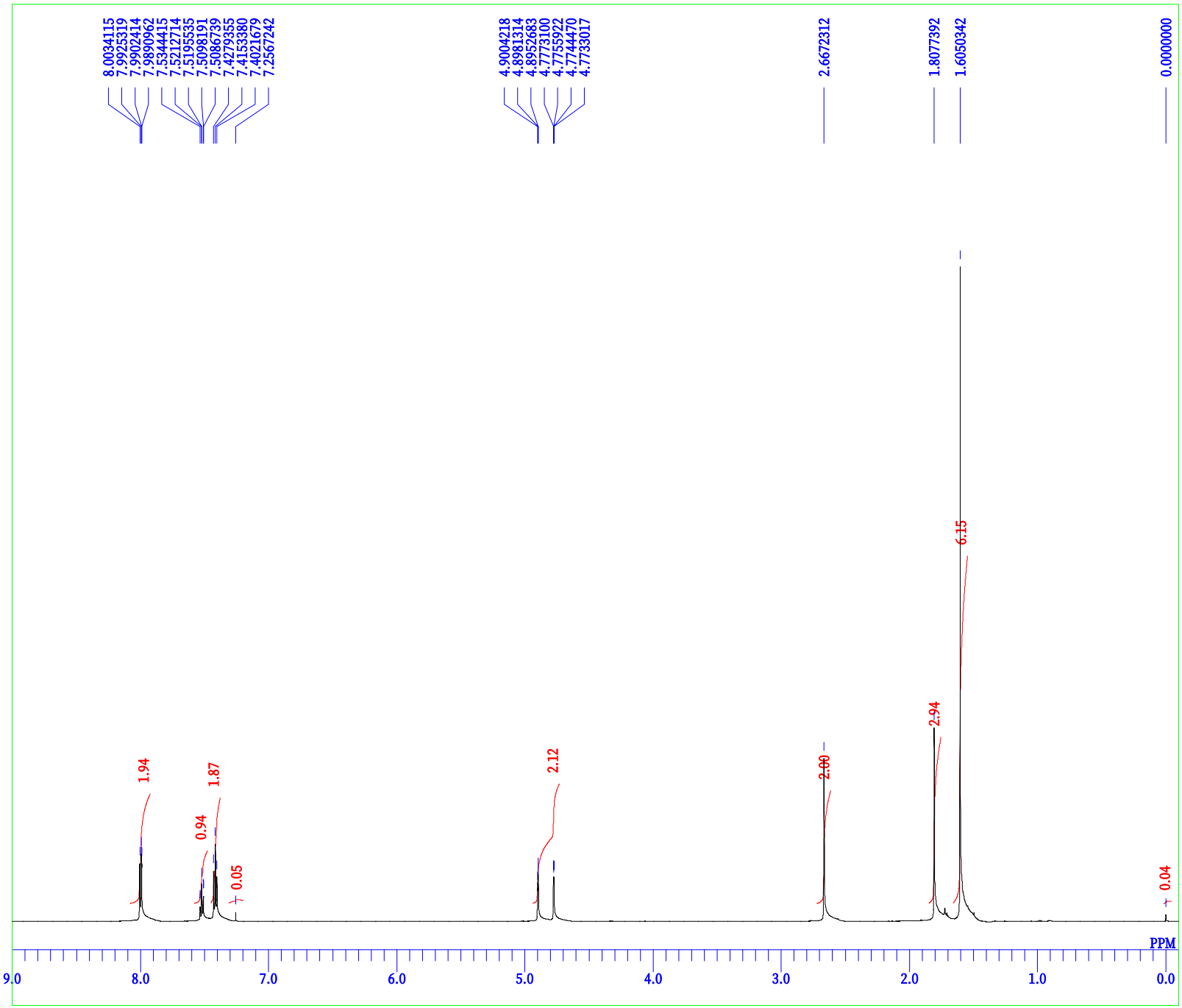
1f



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PD	2.0000 sec
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EXREF	0.00 ppm
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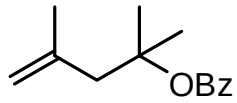
RGAIN	26



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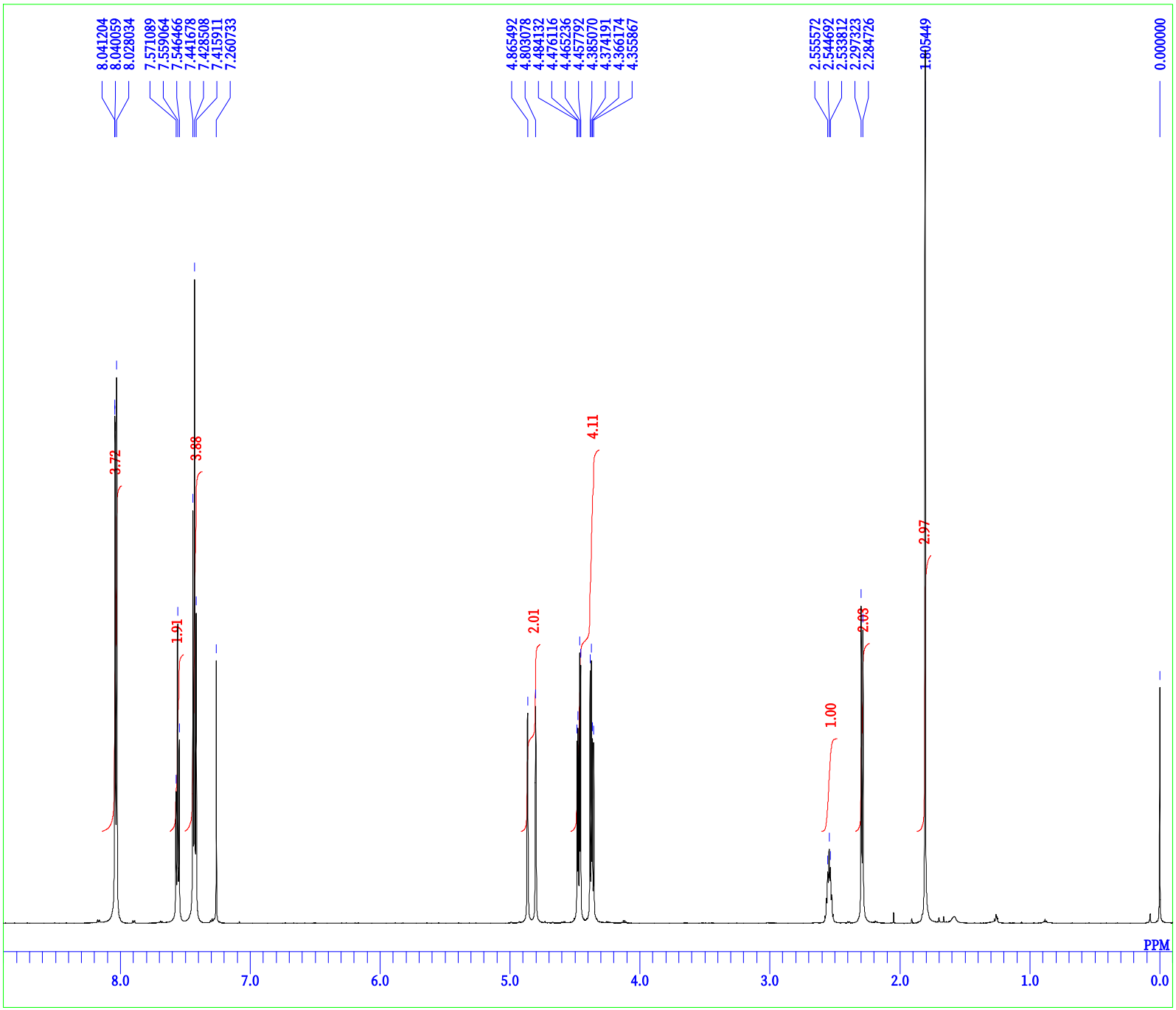

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OBFIN	5.47 Hz
POINT	26214
FREQU	9008.87 Hz
SCANS	16
ACQTM	2.9098 sec
PD	2.0000 sec
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IRNUC	1H
CTEMP	18.7 c
SLVNT	CDCL3
EXREF	0.00 ppm
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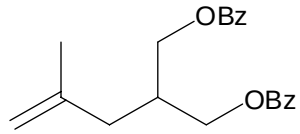
RGAIN	44



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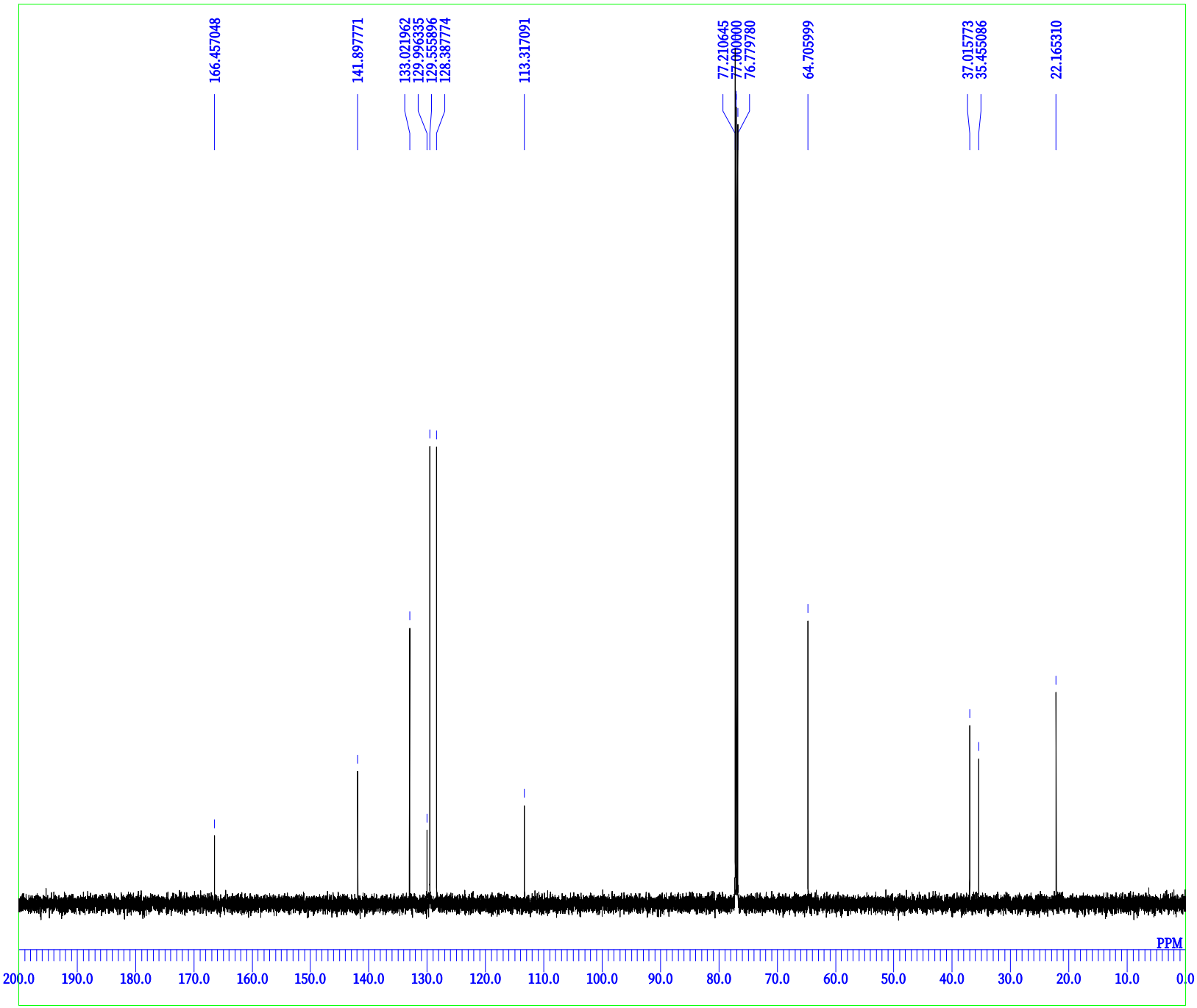
1l_13C



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POINT	26214
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SCANS	250
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PD	1.2000 sec
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SLVNT	CDCL3
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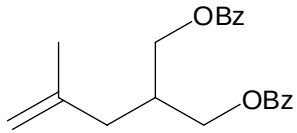
RGAIN	52



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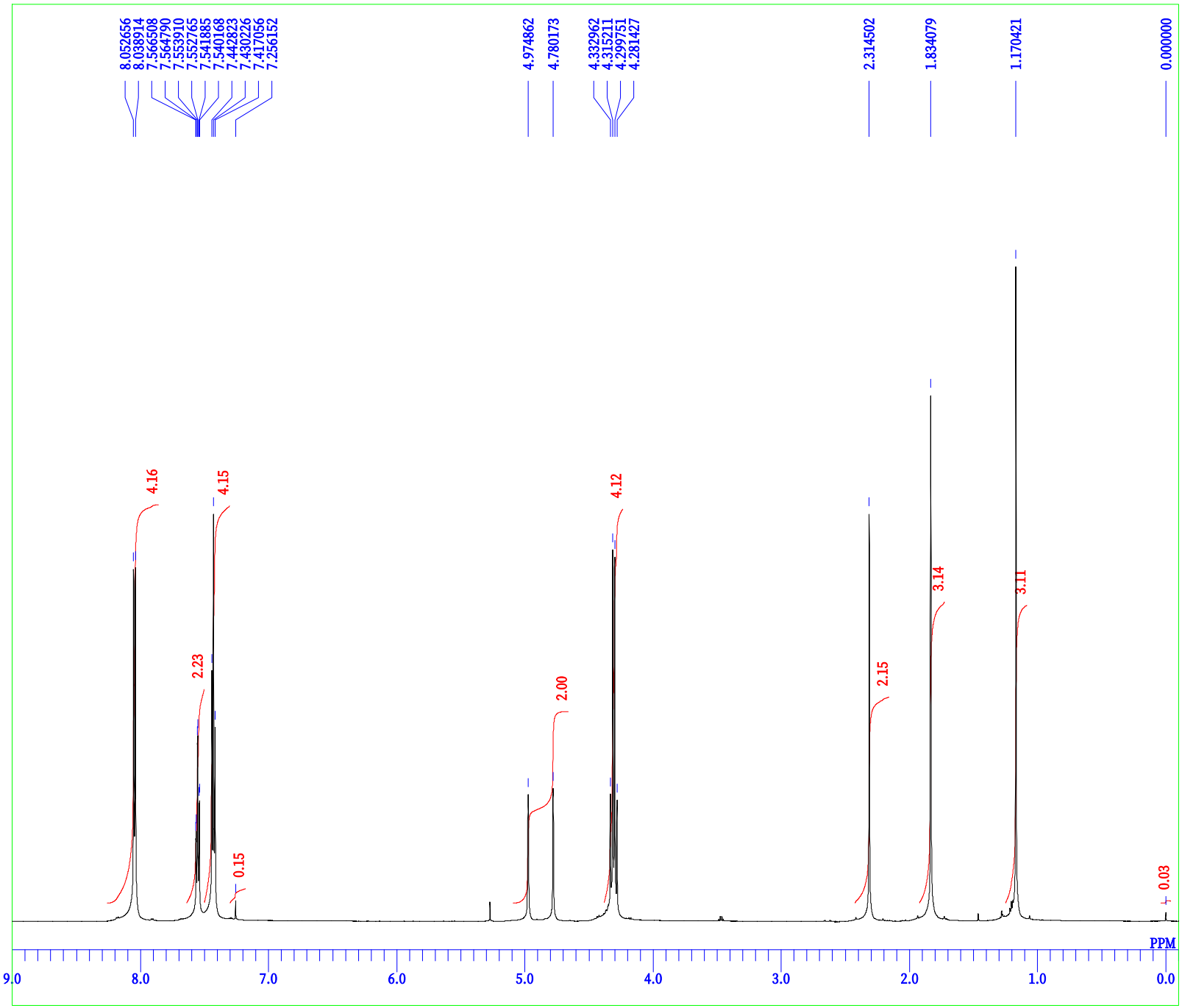

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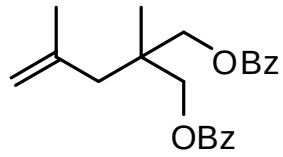
RGAIN	26



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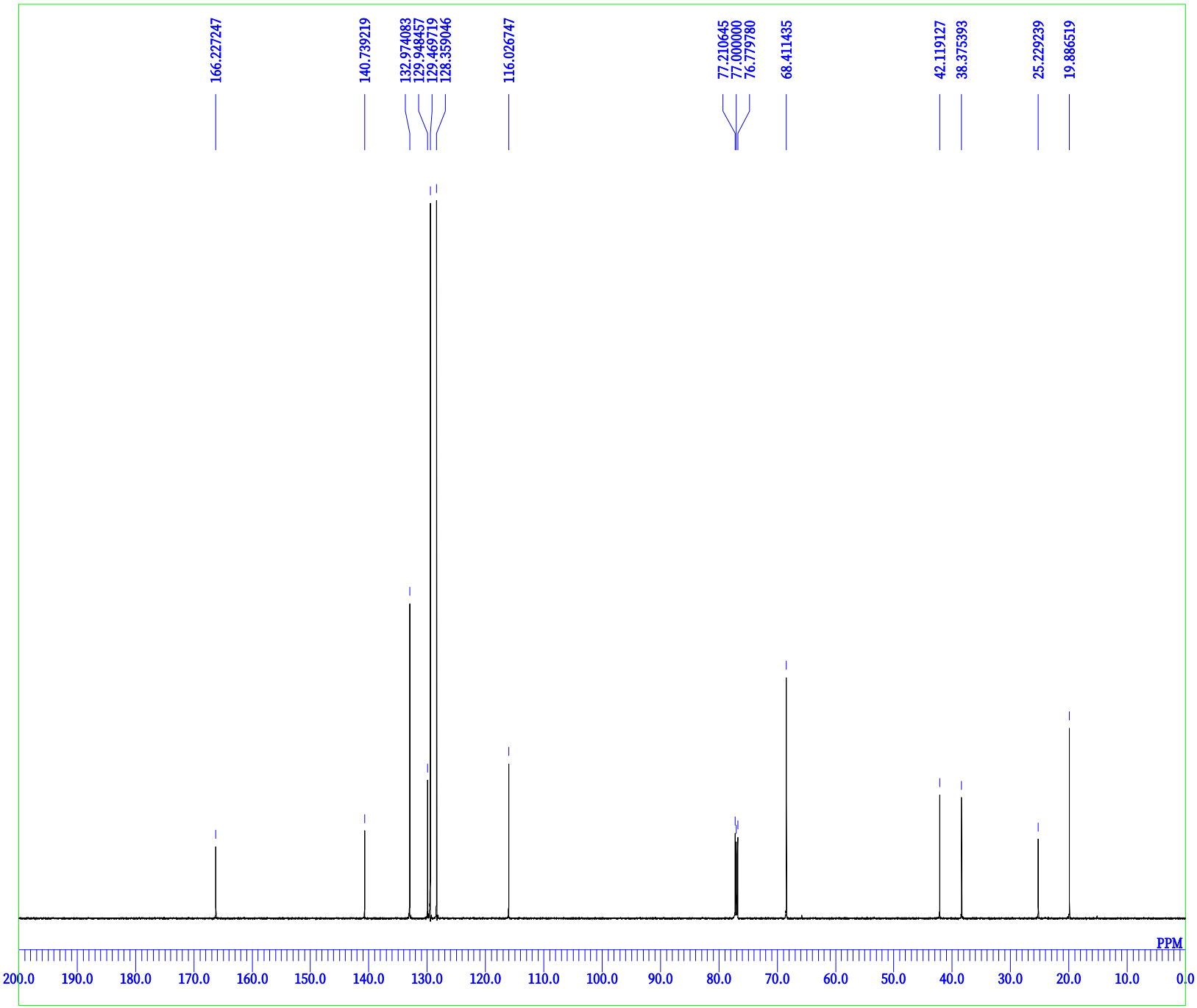

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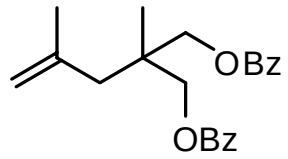

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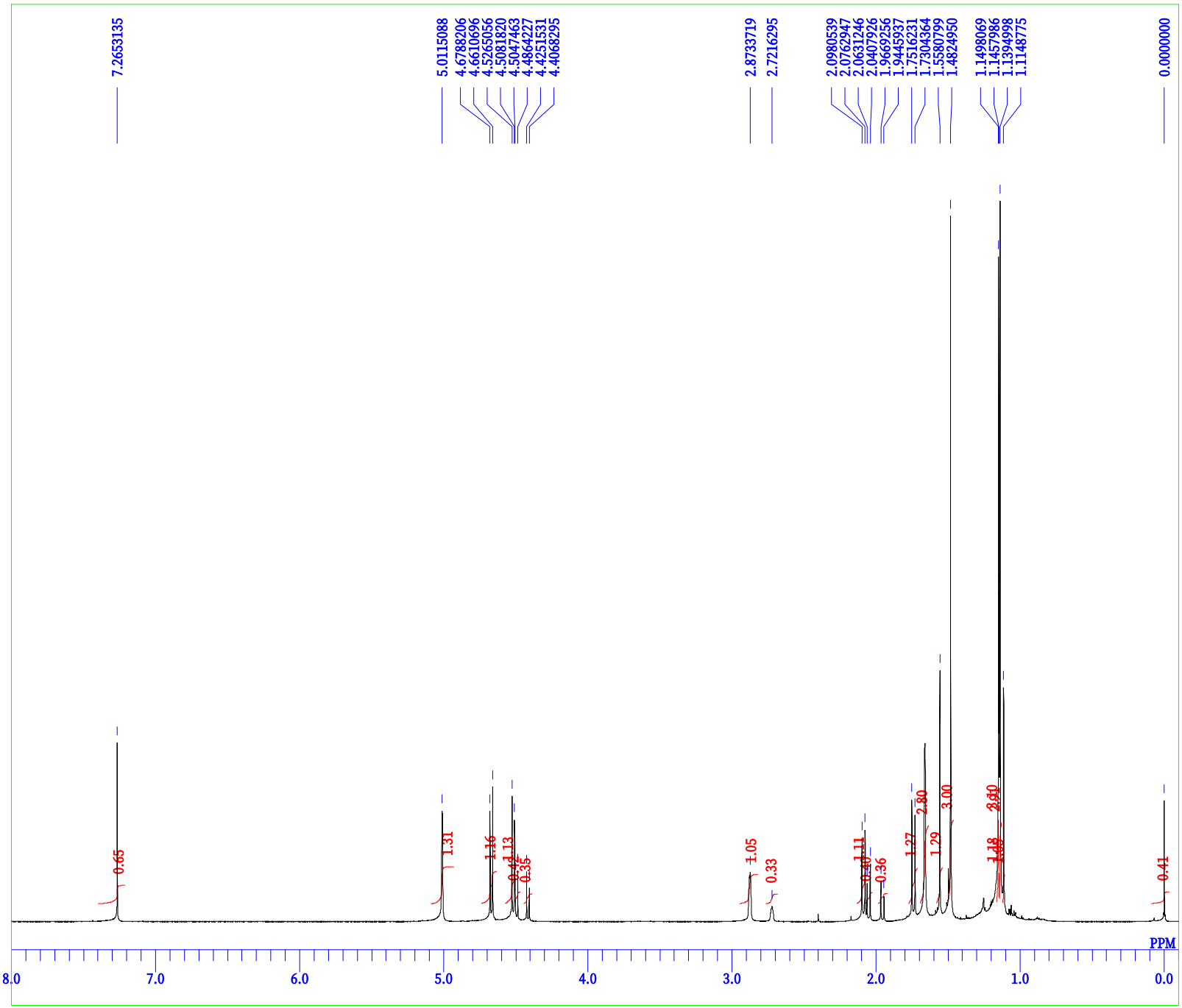

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OBFIN 5.47 Hz
POINT 26214
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SCANS 16
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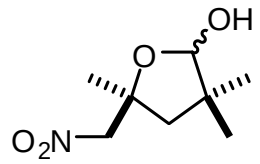
RGAIN 42



2a

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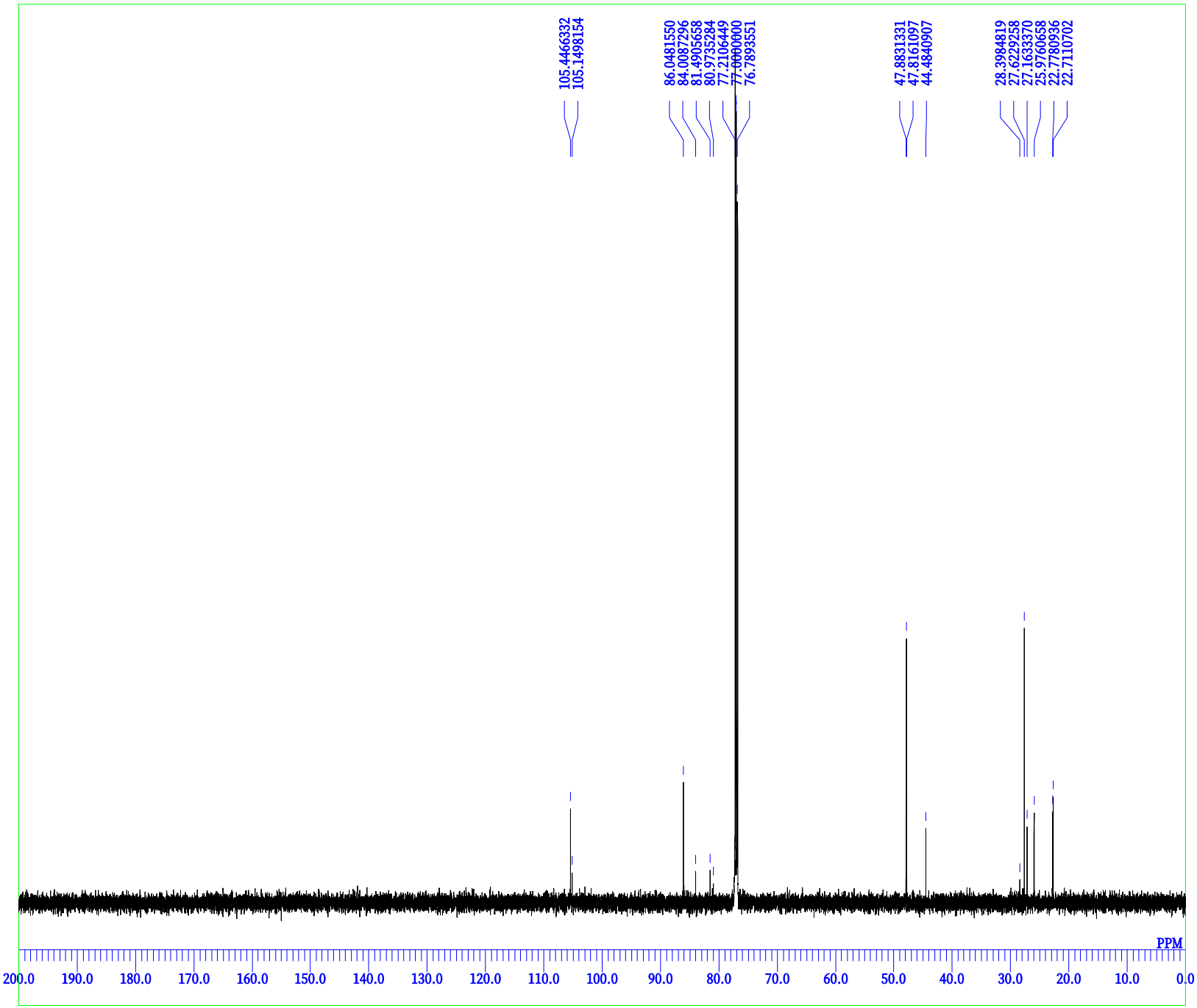
110901



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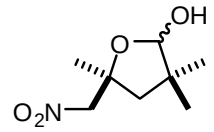
PD	1.2000 sec
PW1	2.77 usec
IRNUC	1H
CTEMP	24.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	56



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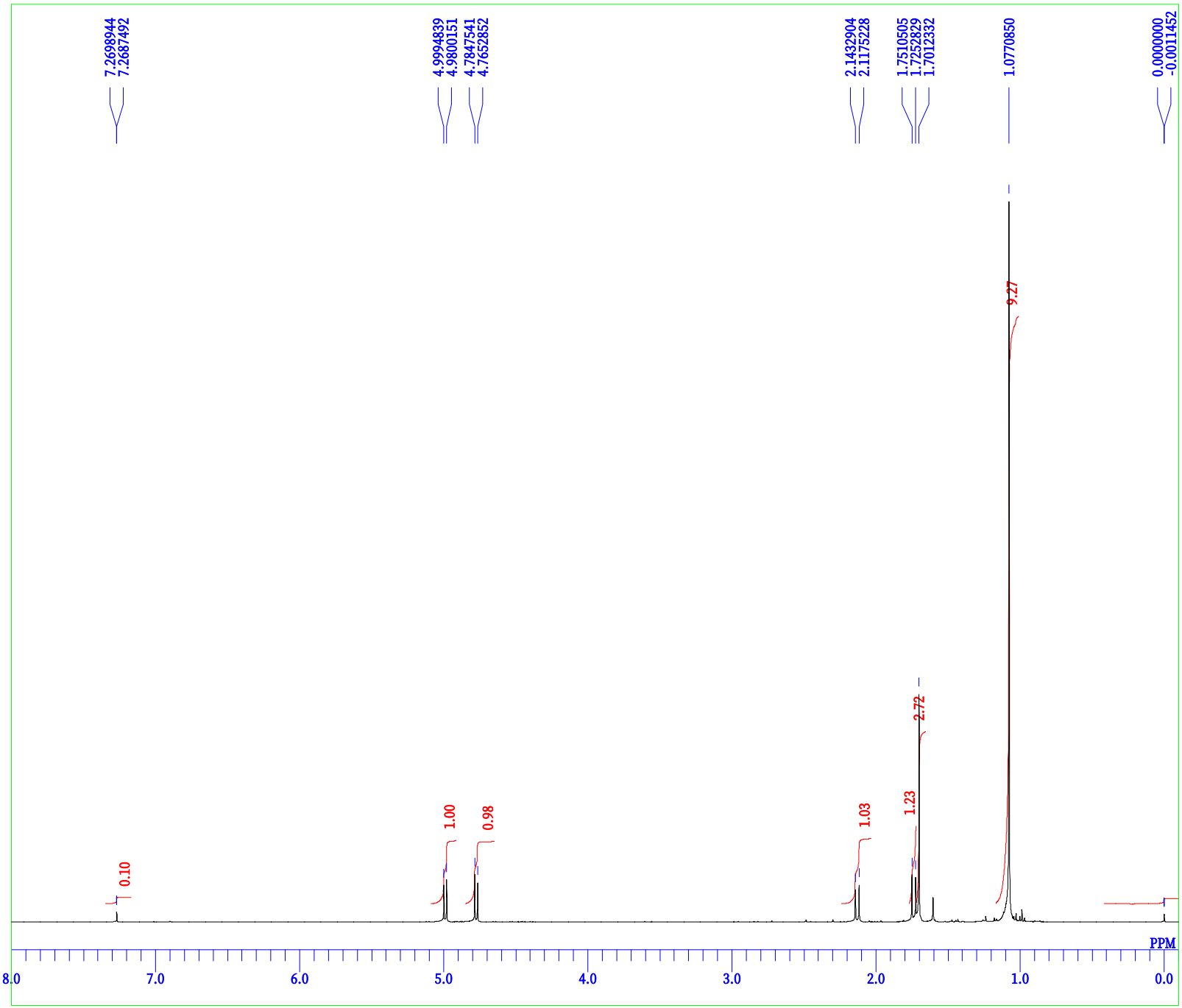
3a



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POINT 26214
FREQU 9008.87 Hz
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PD 2.0000 sec
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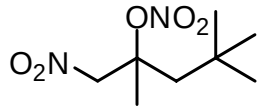
CTEMP 23.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 30



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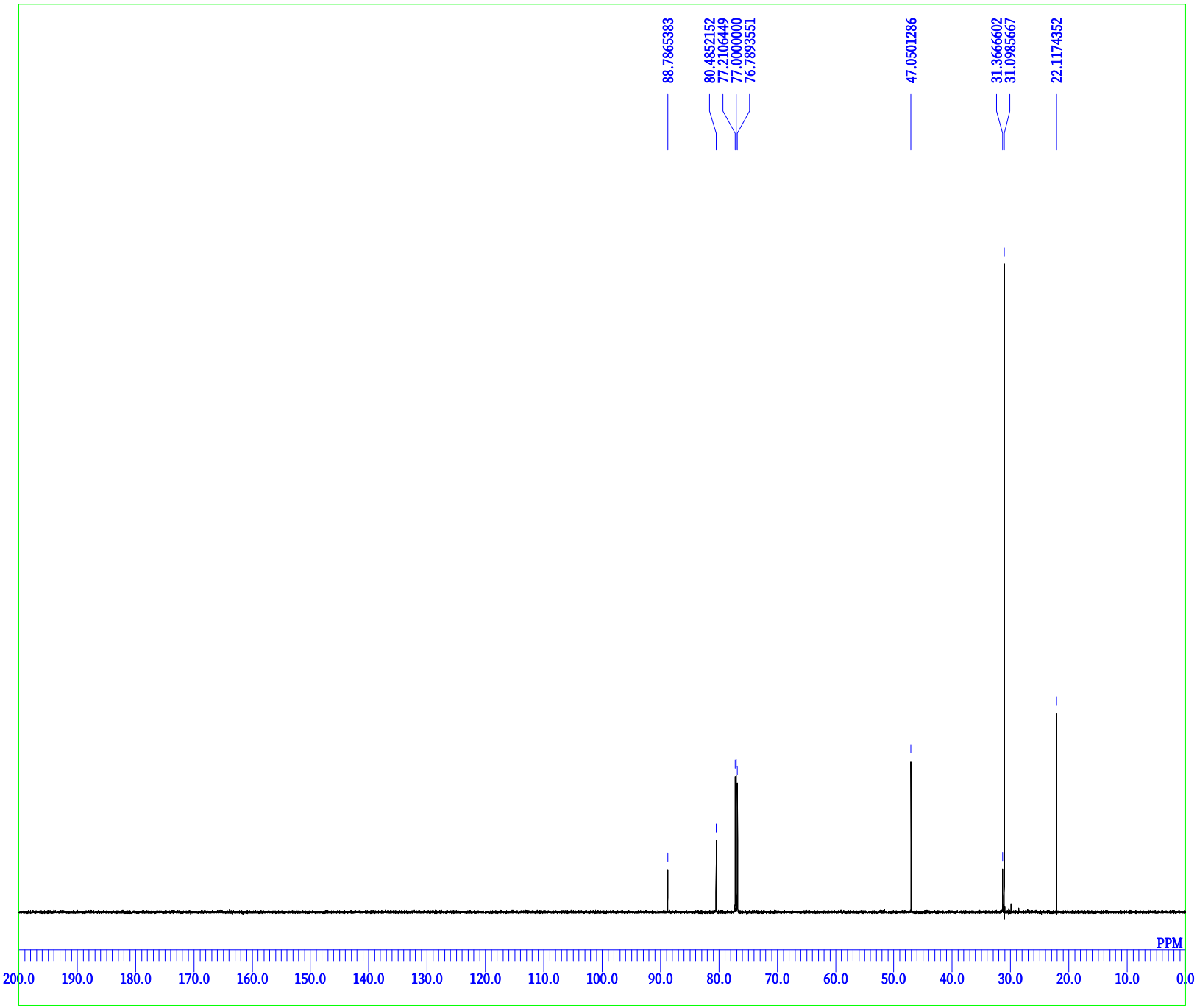
3a



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SCANS	512
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PD	1.2000 sec
PW1	2.77 usec
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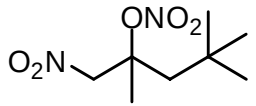
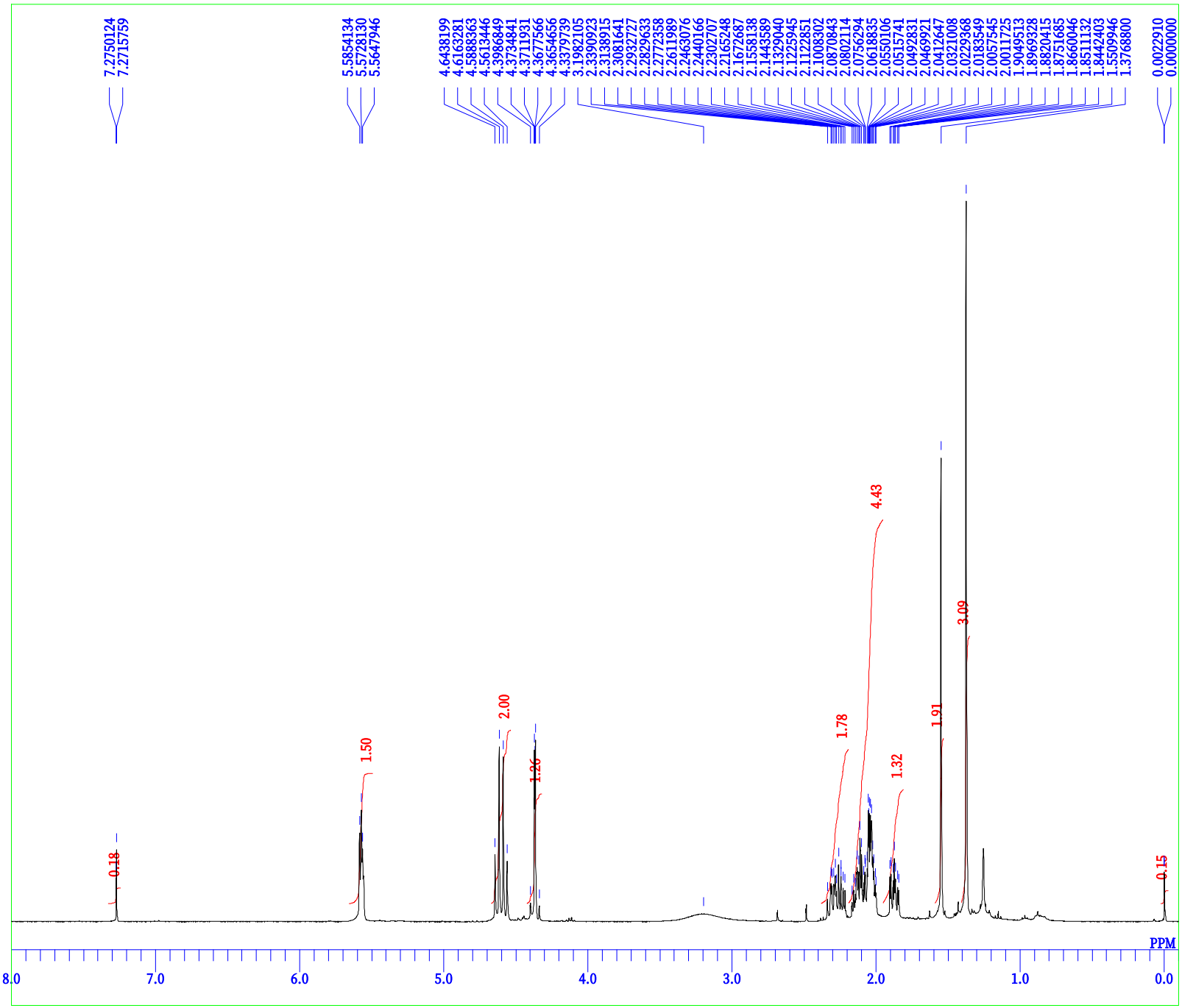
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	54



3a

2b



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PD	2.0000 sec
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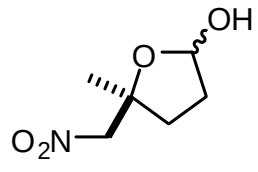

2b

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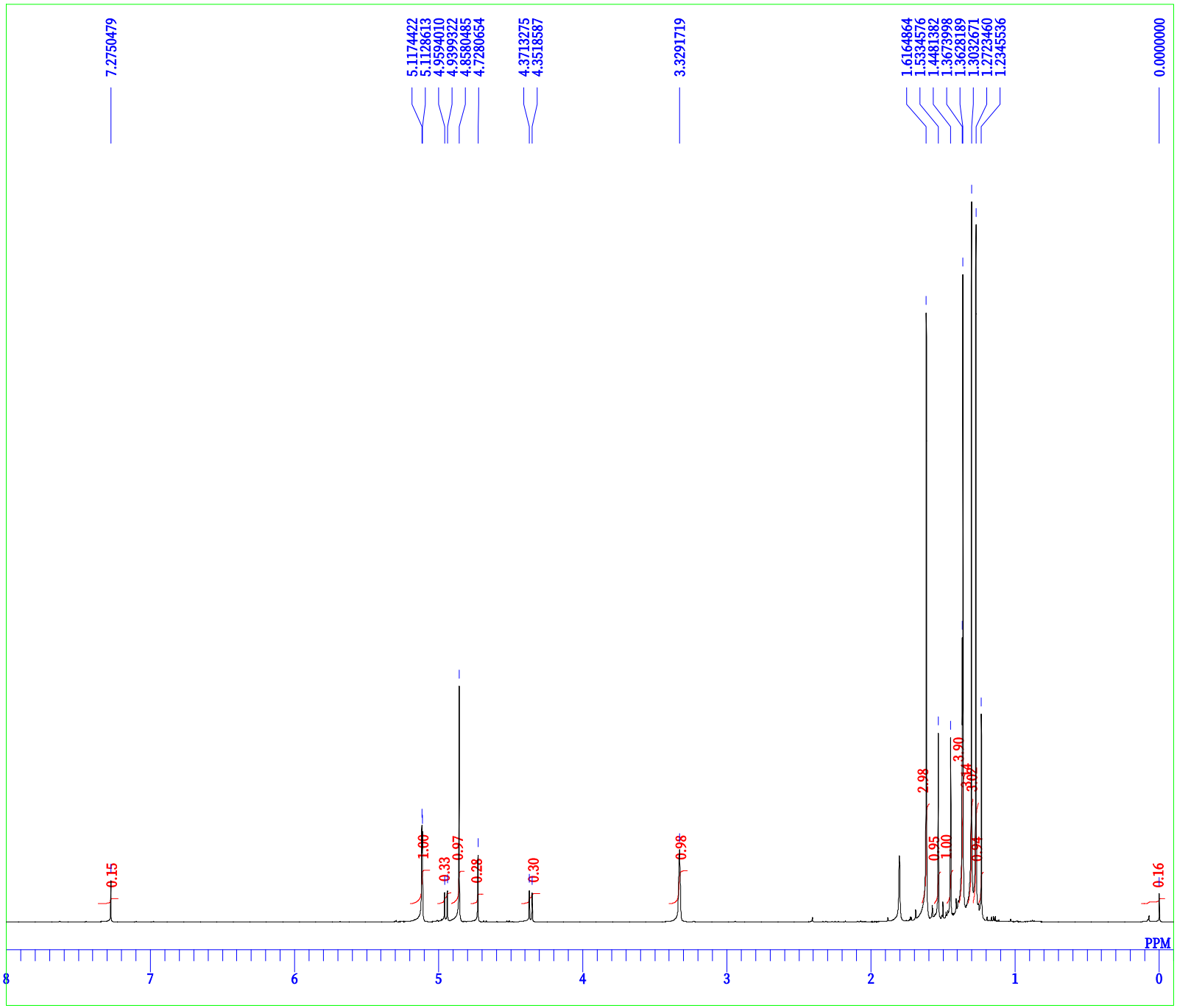
2c



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OBSET 5.30 KHz
OBFIN 5.47 Hz
POINT 26214
FREQU 9008.87 Hz
SCANS 16
ACQTM 2.9098 sec
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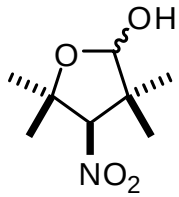
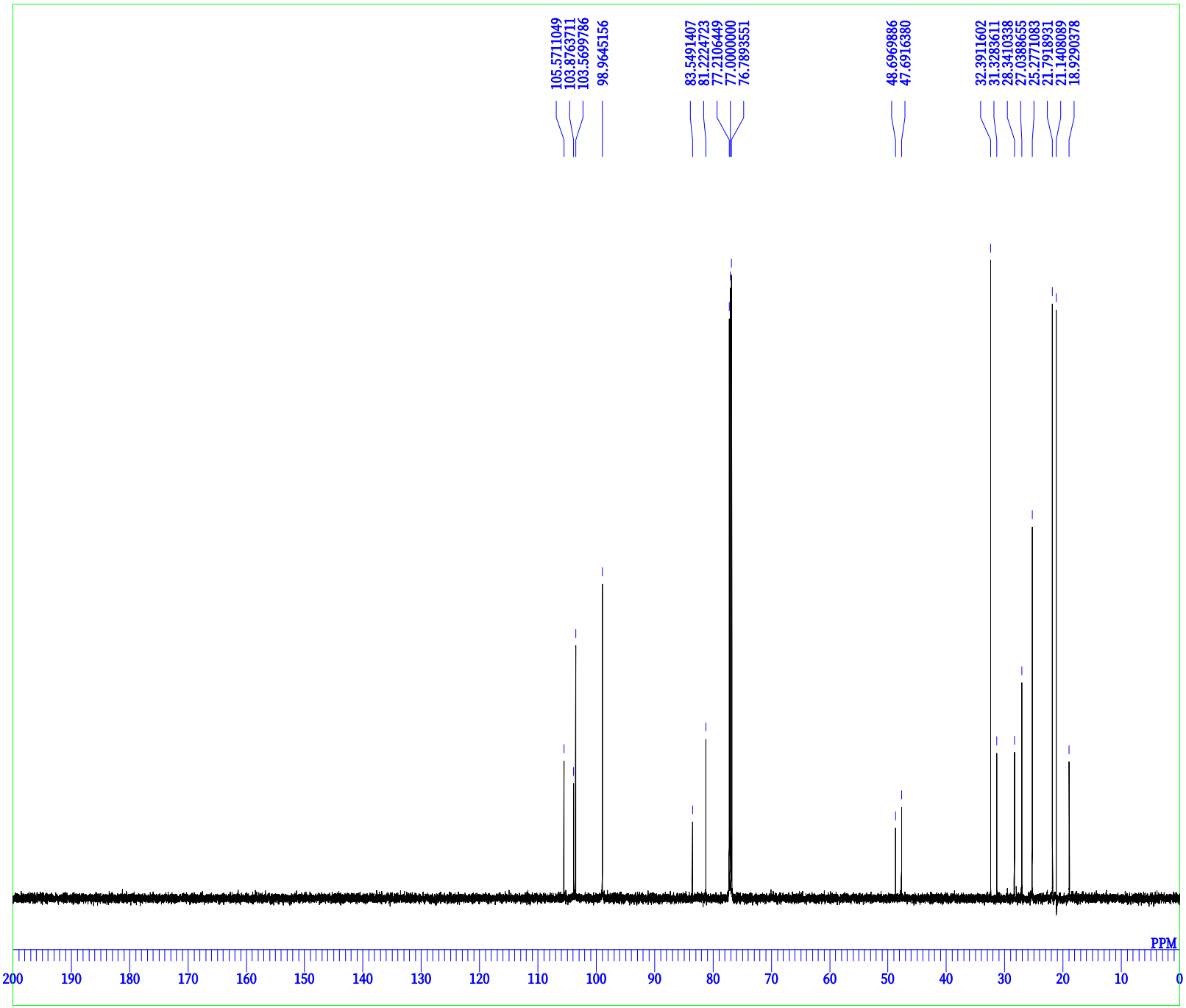
PW1 5.60 usec
IRNUC 1H
CTEMP 23.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.25 Hz
RGAIN 30



2c

2c



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OBFIN	1.74 Hz
POINT	26214
FREQU	37878.21 Hz
SCANS	512
ACQTM	0.6921 sec
PD	1.2000 sec
PW1	2.77 usec
IRNUC	1H
CTEMP	23.7 c
SLVNT	CDCL3
EXREF	77.00 ppm
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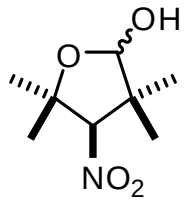
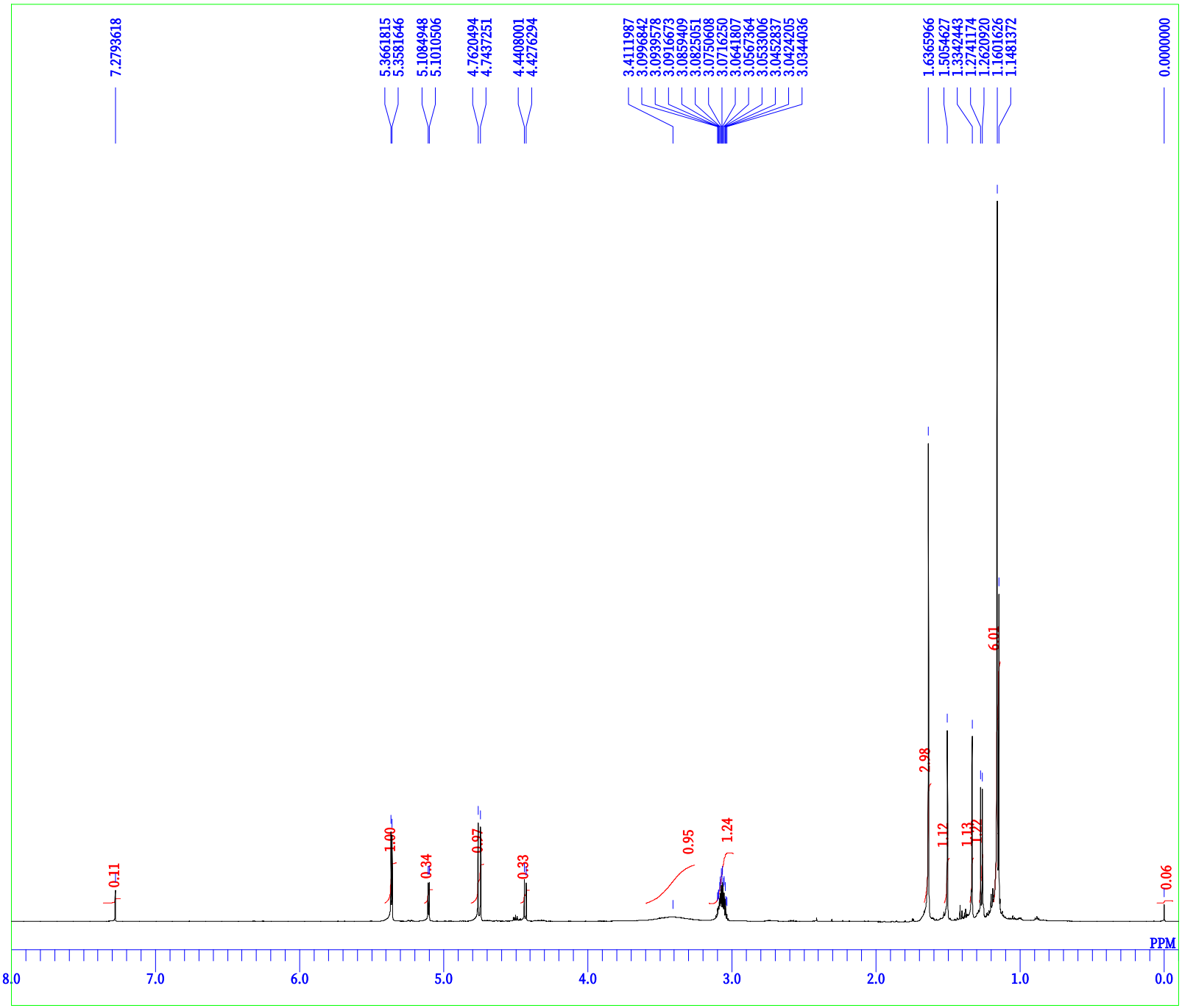
RGAIN	54



2c

2d



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OBSET 5.30 KHz
OBFIN 5.47 Hz
POINT 20970
FREQU 7206.99 Hz
SCANS 16
ACQTM 2.9098 sec
PD 2.0000 sec
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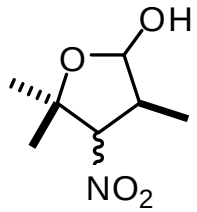
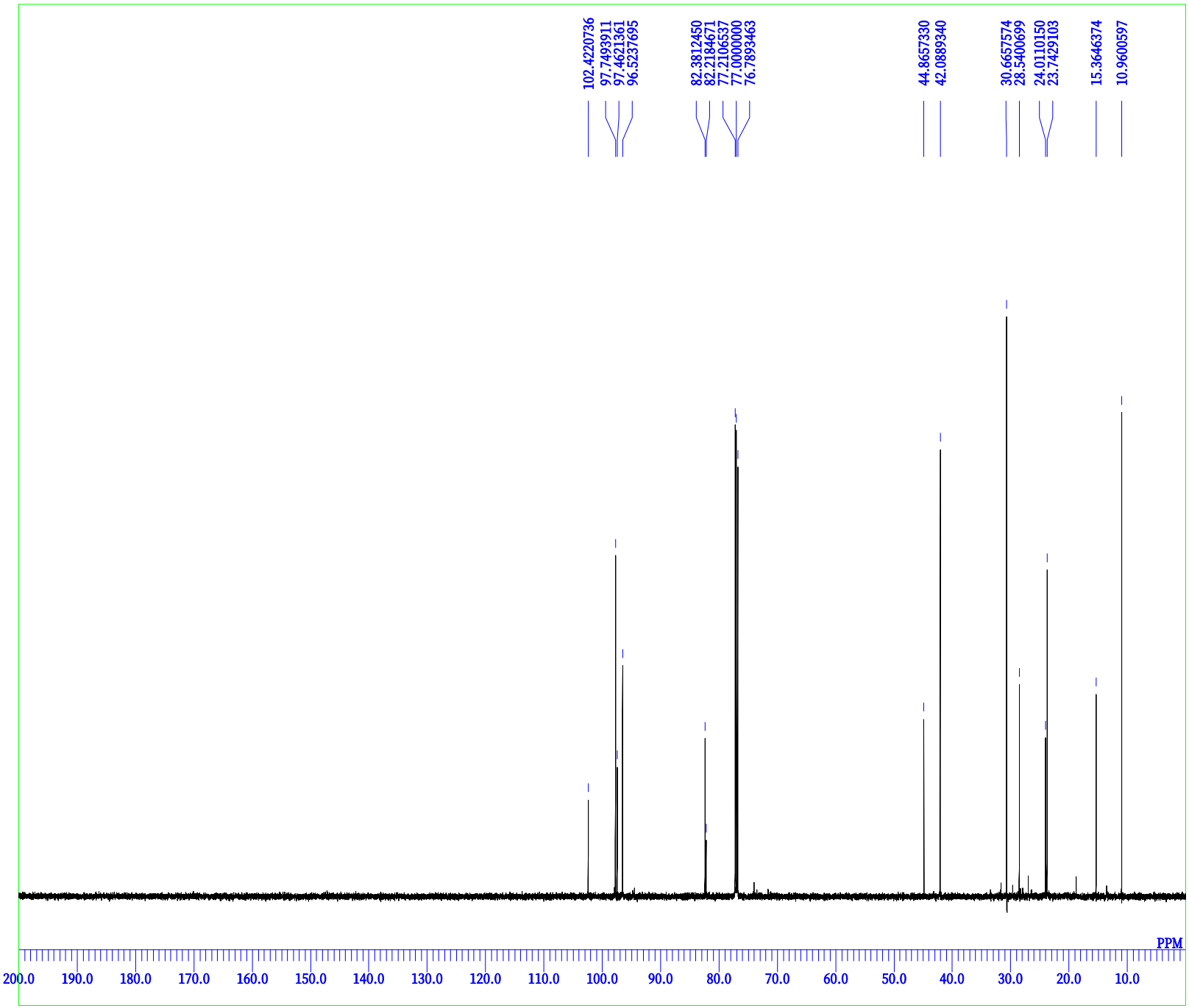
RGAIN 30



2d

2d



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OBFIN	1.74 Hz
POINT	20970
FREQU	30302.11 Hz
SCANS	537
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PD	1.2000 sec
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CTEMP	20.2 c
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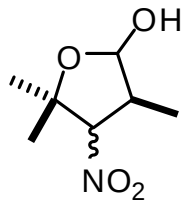
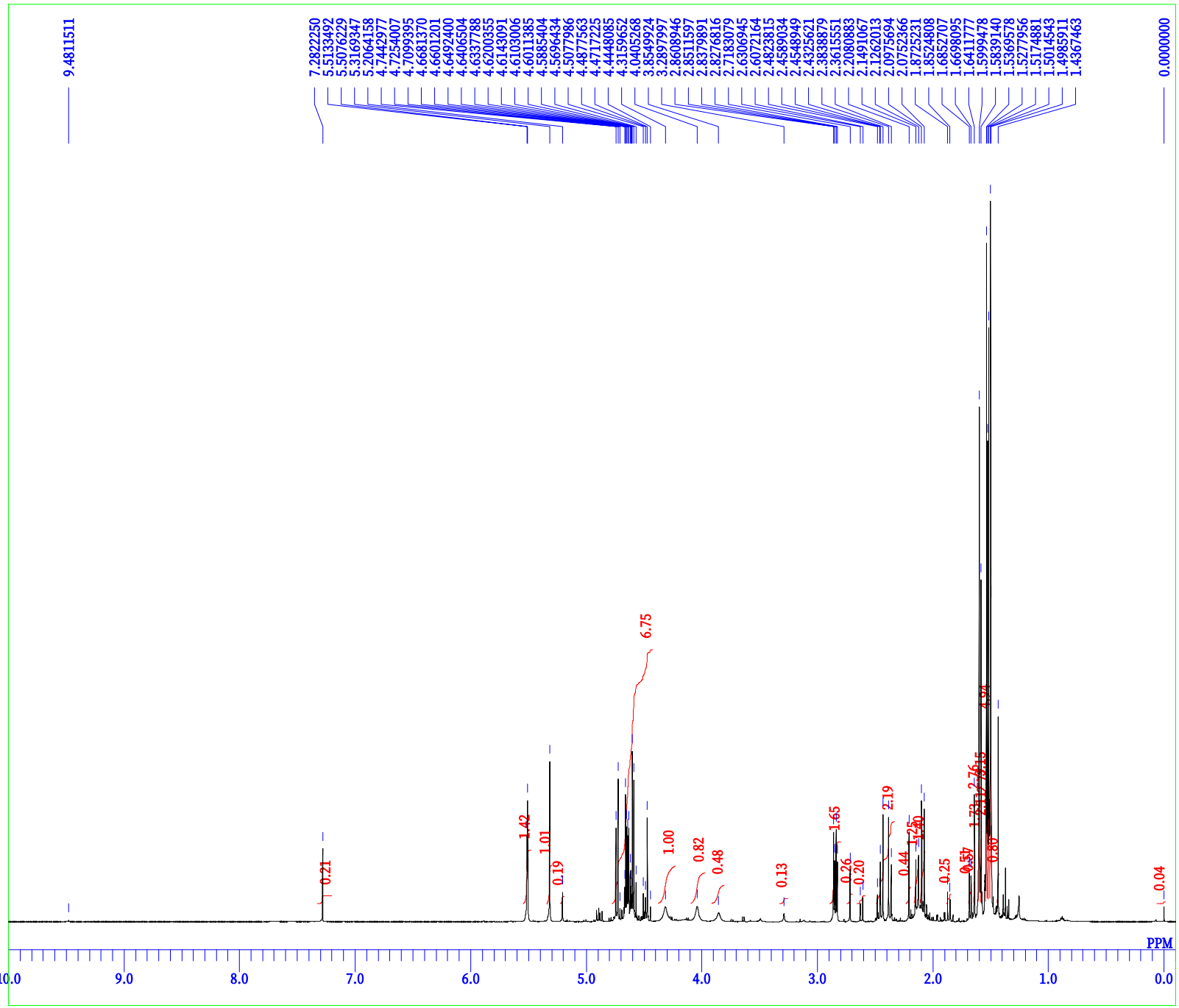
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	56



2d

2e



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OBFIN 5.47 Hz
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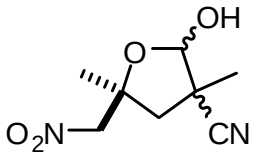
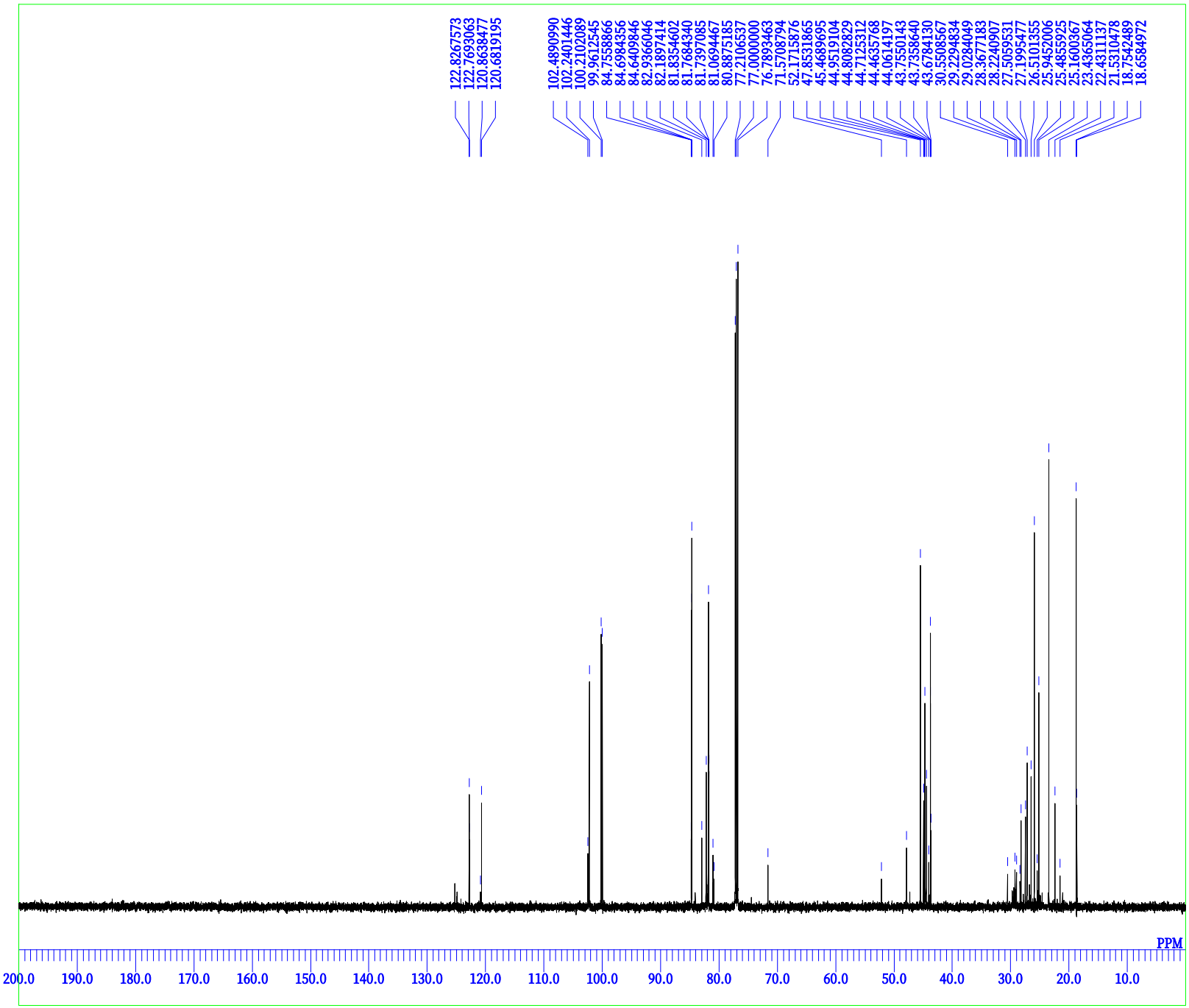
BF 0.24 Hz
RGAIN 30



2e

2e



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OBFIN	1.74 Hz
POINT	20970
FREQU	30302.11 Hz
SCANS	512
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PD	1.2000 sec
PW1	2.73 usec
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CTEMP	24.3 c
SLVNT	CDCL3
EXREF	77.00 ppm
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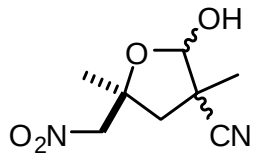
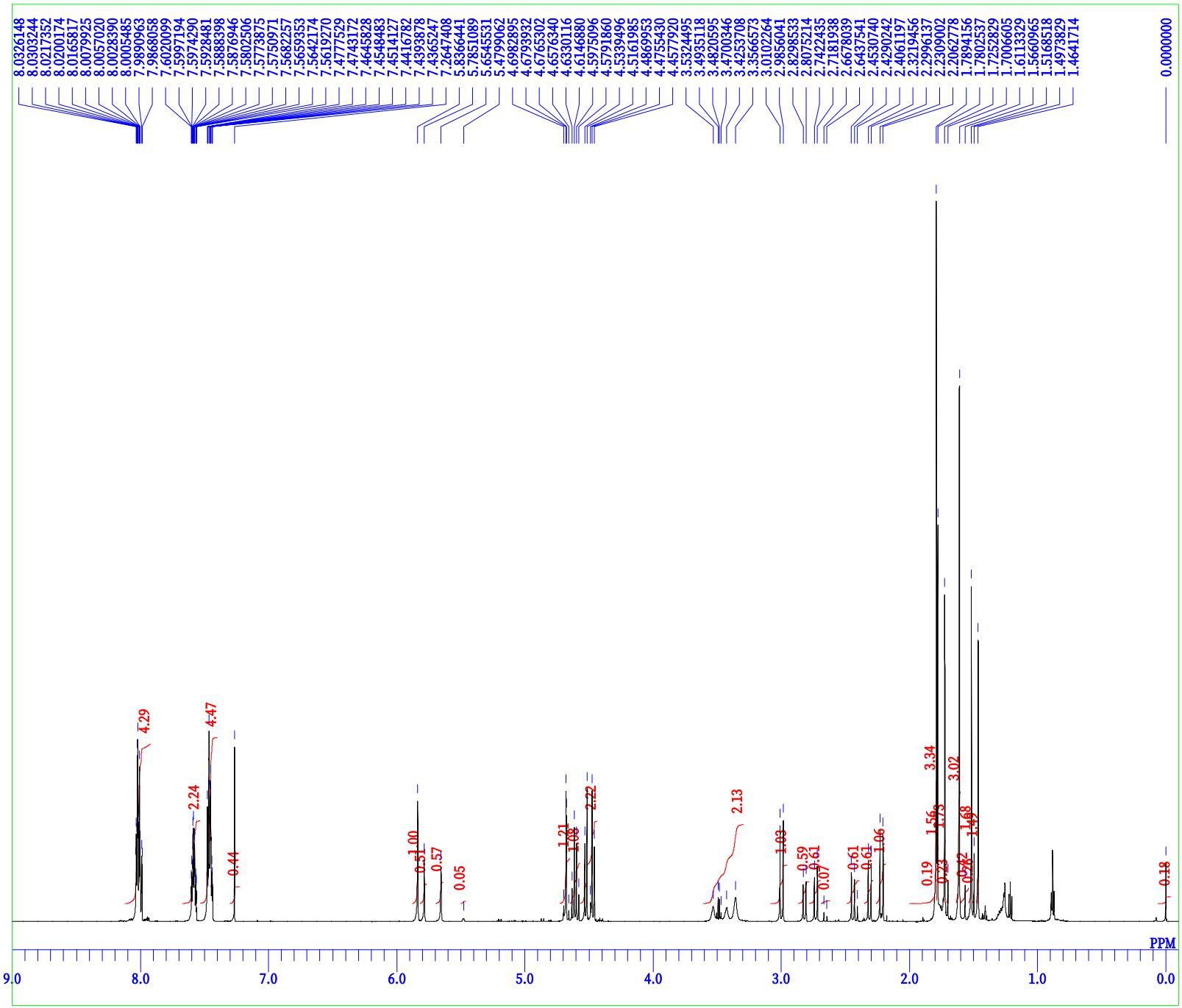
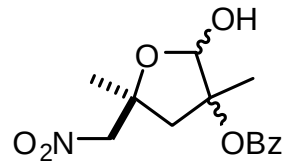
RGAIN	58



2e

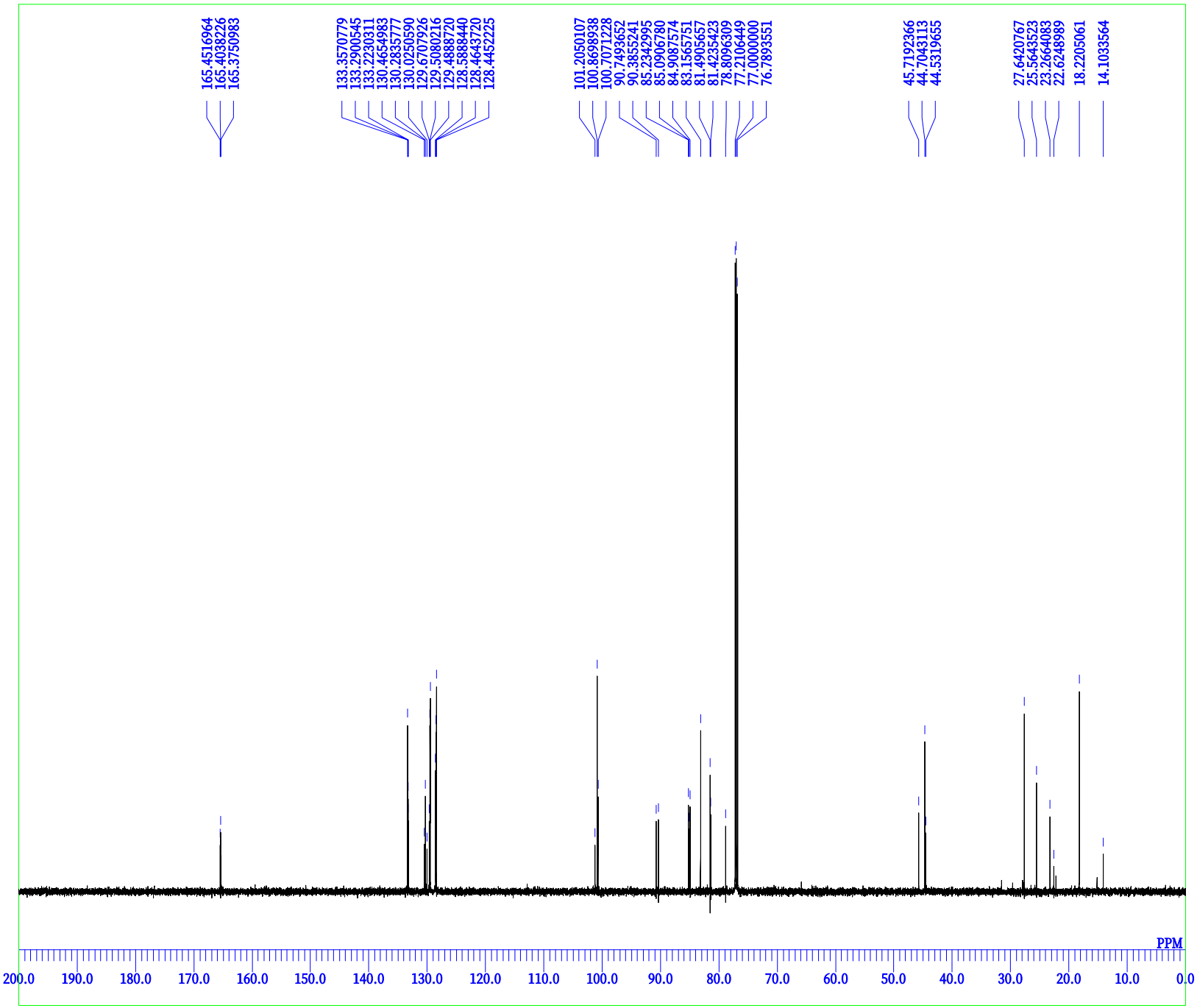
2f


DFILE 2f_1H.als
COMNT 2f
DATIM 18-03-2012 06:22:11
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 600.17 MHz
OBSET 5.30 KHz
OBFIN 5.47 Hz
POINT 26214
FREQU 9008.87 Hz
SCANS 16
ACQTM 2.9098 sec
PD 2.0000 sec
PW1 5.60 usec
IRNUC 1H
CTEMP 18.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 36
PPM
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0

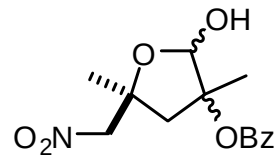

OH
O
O2N
OBz

2f

2f

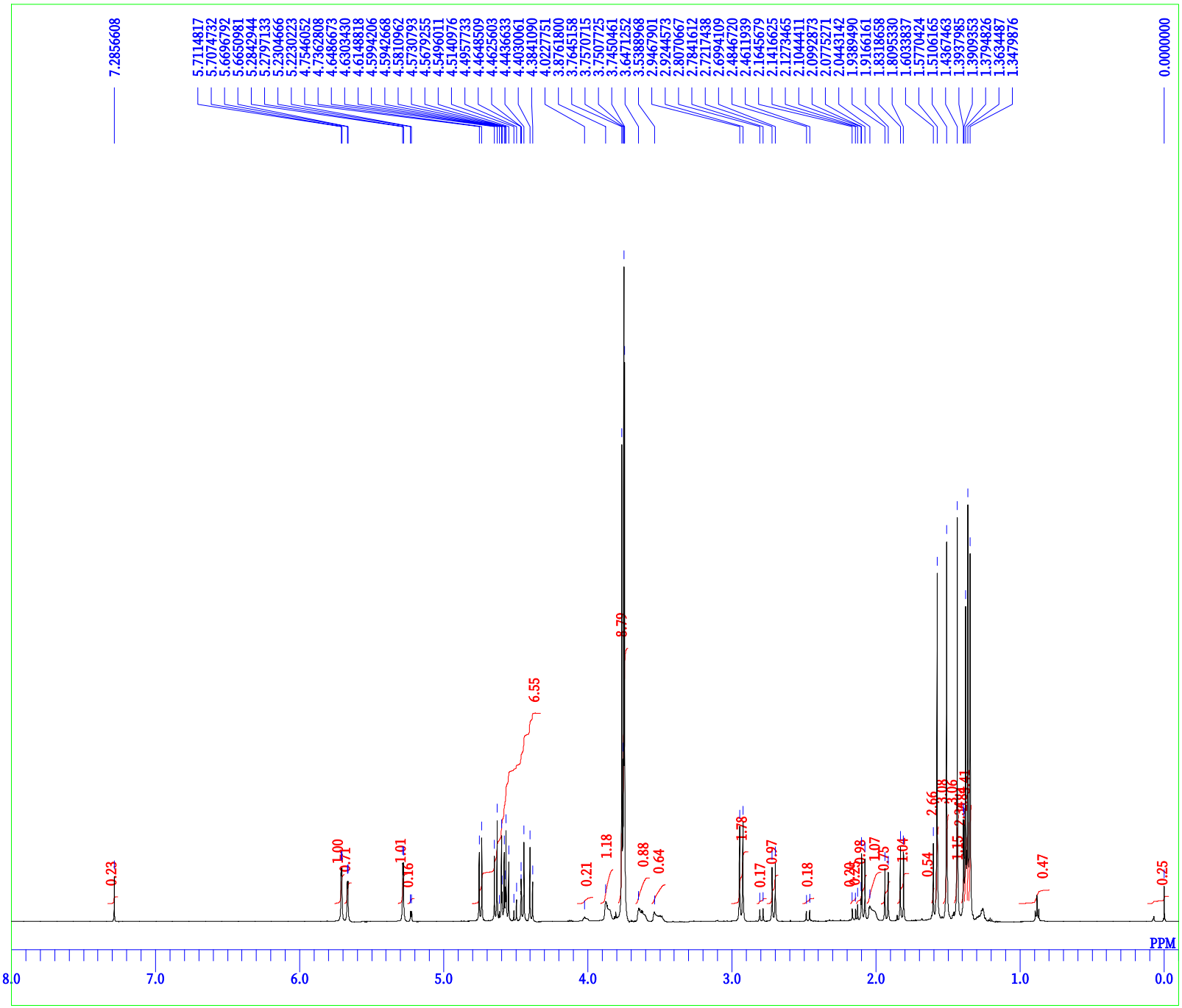


DFILE	2f_13C-1.als
COMNT	2f
DATIM	18-03-2012 06:45:21
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	26214
FREQU	37878.21 Hz
SCANS	722
ACQTM	0.6921 sec
PD	1.2000 sec
PW1	2.77 usec
IRNUC	1H
CTEMP	19.3 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.00 Hz
RGAIN	54

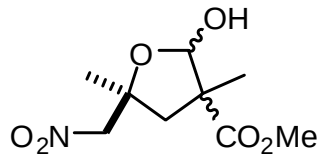


2f

2g

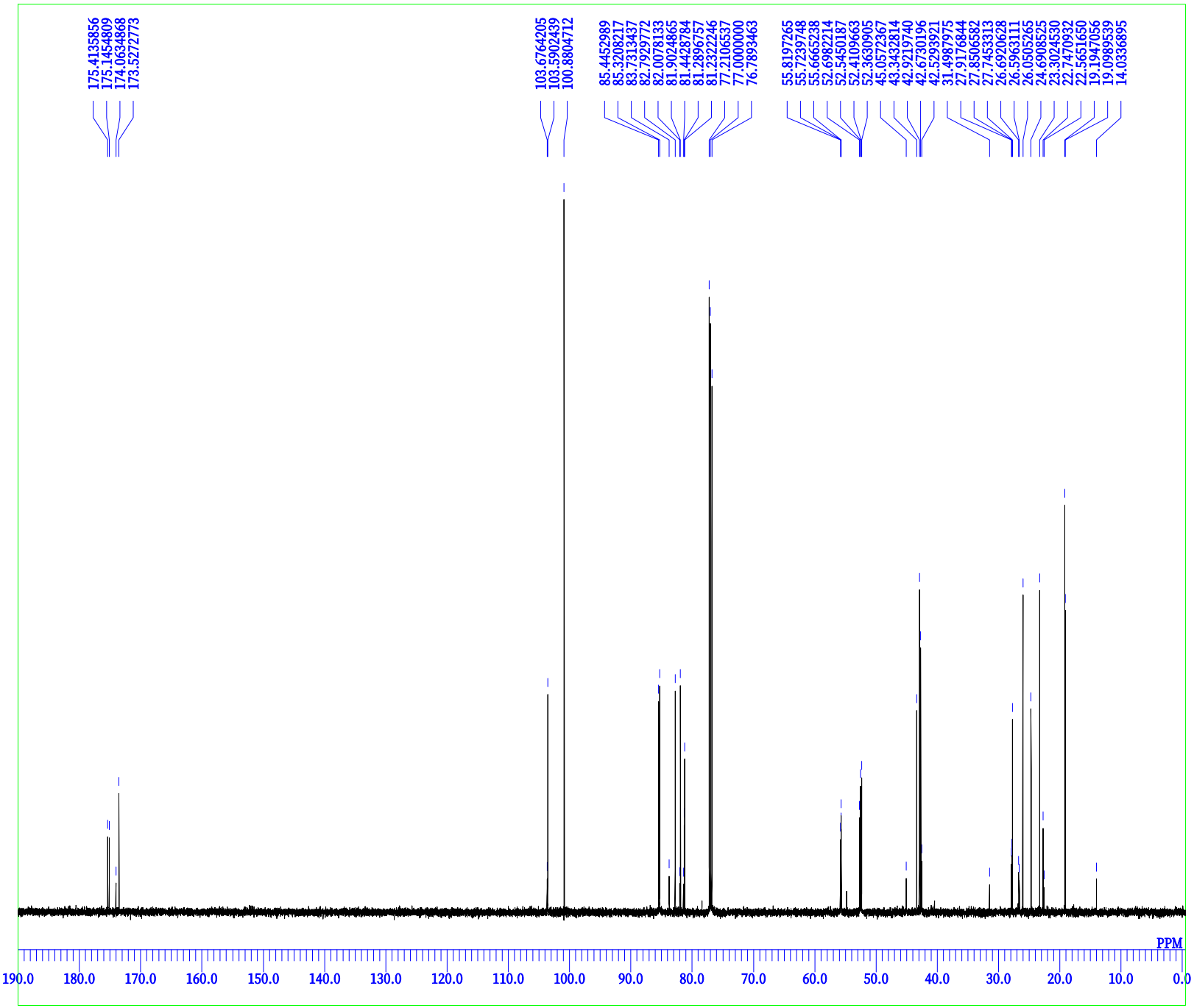


DFILE 2g_1H.als
COMNT 2g
DATIM 06-09-2011 21:20:34
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 600.17 MHz
OBSET 5.30 KHz
OBFIN 5.47 Hz
POINT 20970
FREQU 7206.99 Hz
SCANS 16
ACQTM 2.9098 sec
PD 2.0000 sec
PW1 5.60 usec
IRNUC 1H
CTEMP 23.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.24 Hz
RGAIN 30

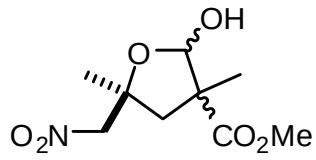


2g

2g

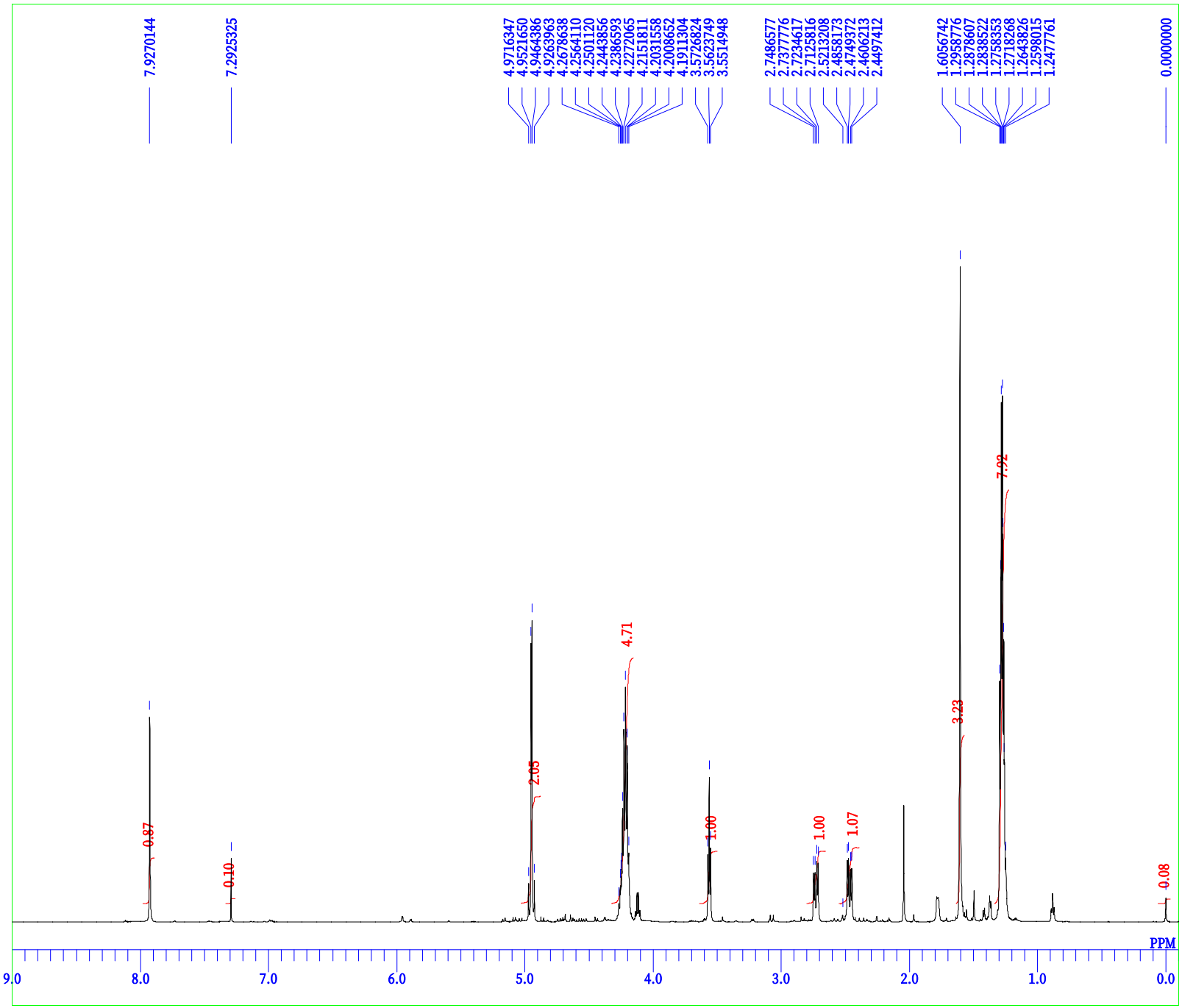


DFILE	2g_13C.als
COMNT	2g
DATIM	06-09-2011 21:37:08
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	20970
FREQU	30302.11 Hz
SCANS	512
ACQTM	0.6921 sec
PD	1.2000 sec
PW1	2.77 usec
IRNUC	1H
CTEMP	23.9 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.24 Hz
RGAIN	54

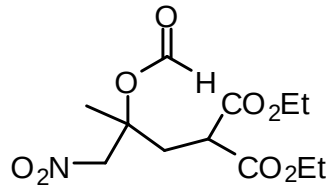


2g

2h



DFILE	2h_1H.als
COMNT	2h
DATIM	08-09-2011 10:31:03
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	20970
FREQU	7206.99 Hz
SCANS	16
ACQTM	2.9098 sec
PD	2.0000 sec
PW1	5.60 usec
IRNUC	1H
CTEMP	23.2 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.24 Hz
RGAIN	28



2h

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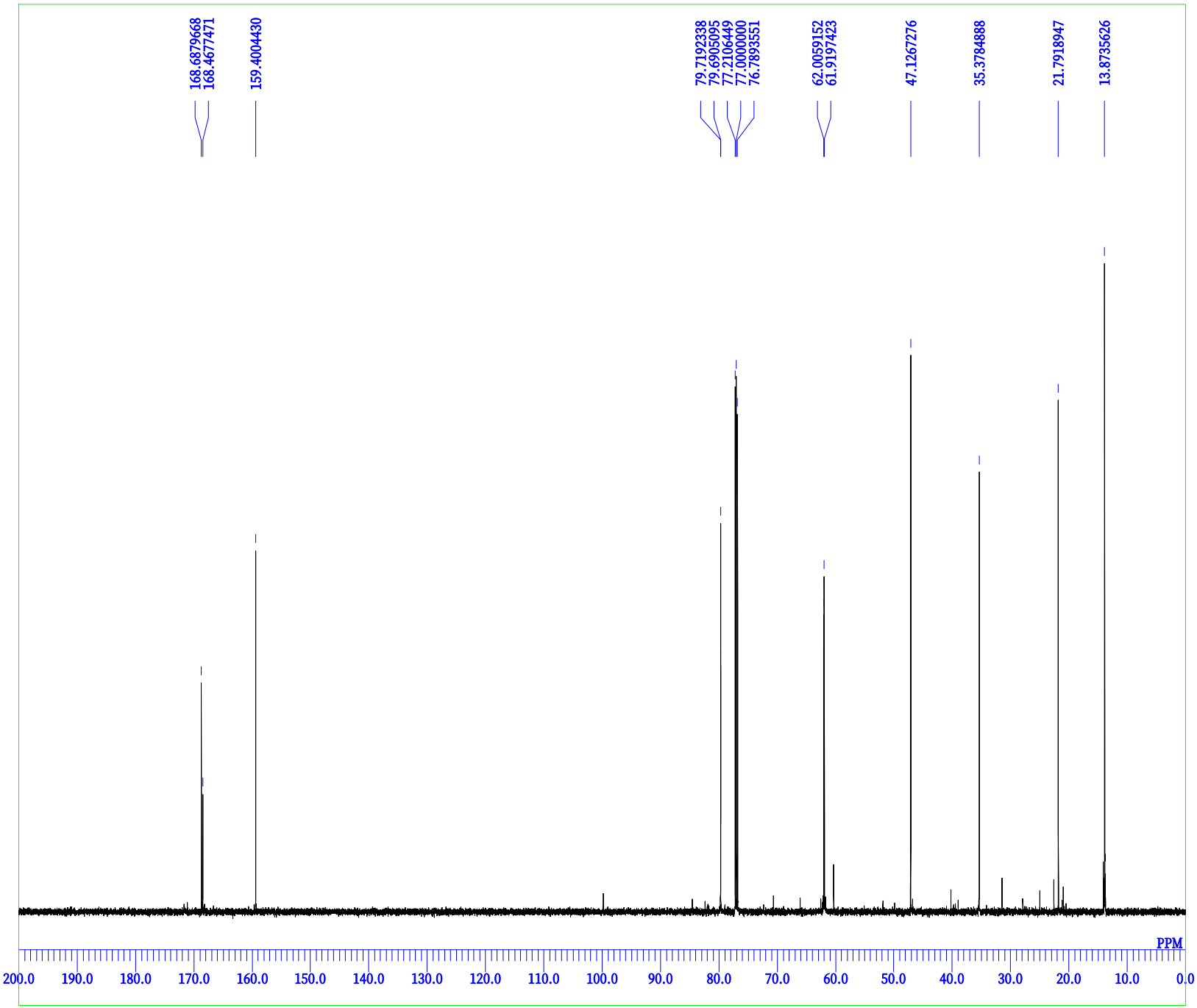
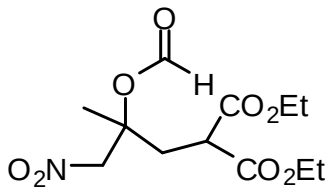
2h

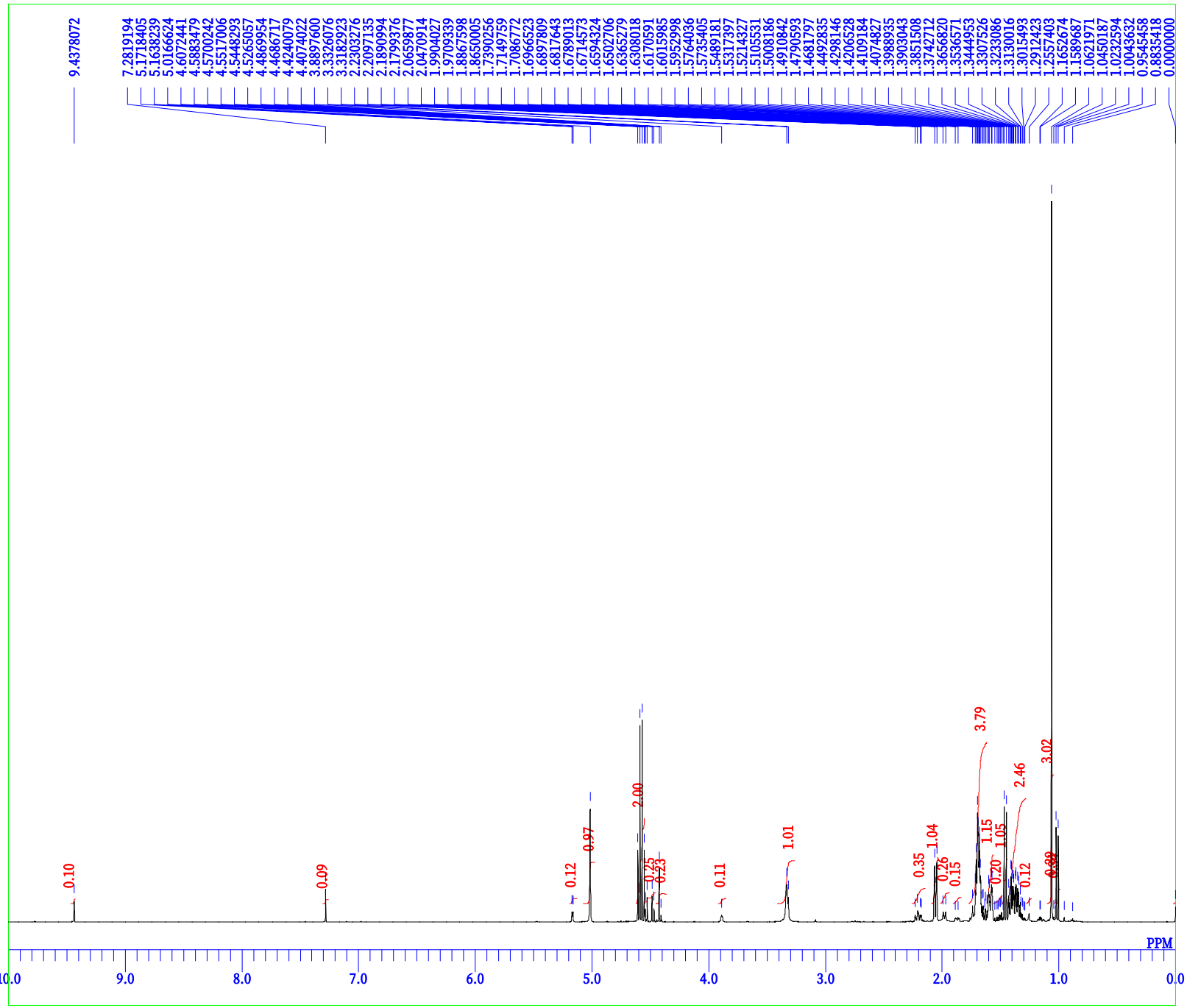


DFILE	2h_13C.als
COMNT	2h
DATIM	08-09-2011 10:48:56
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	26214
FREQU	37878.21 Hz
SCANS	512
ACQTM	0.6921 sec
PD	1.2000 sec
PW1	2.77 usec
IRNUC	1H
CTEMP	23.8 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.24 Hz
RGAIN	54

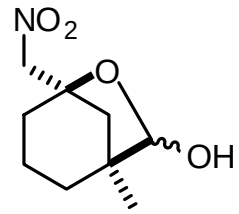


2h

2i

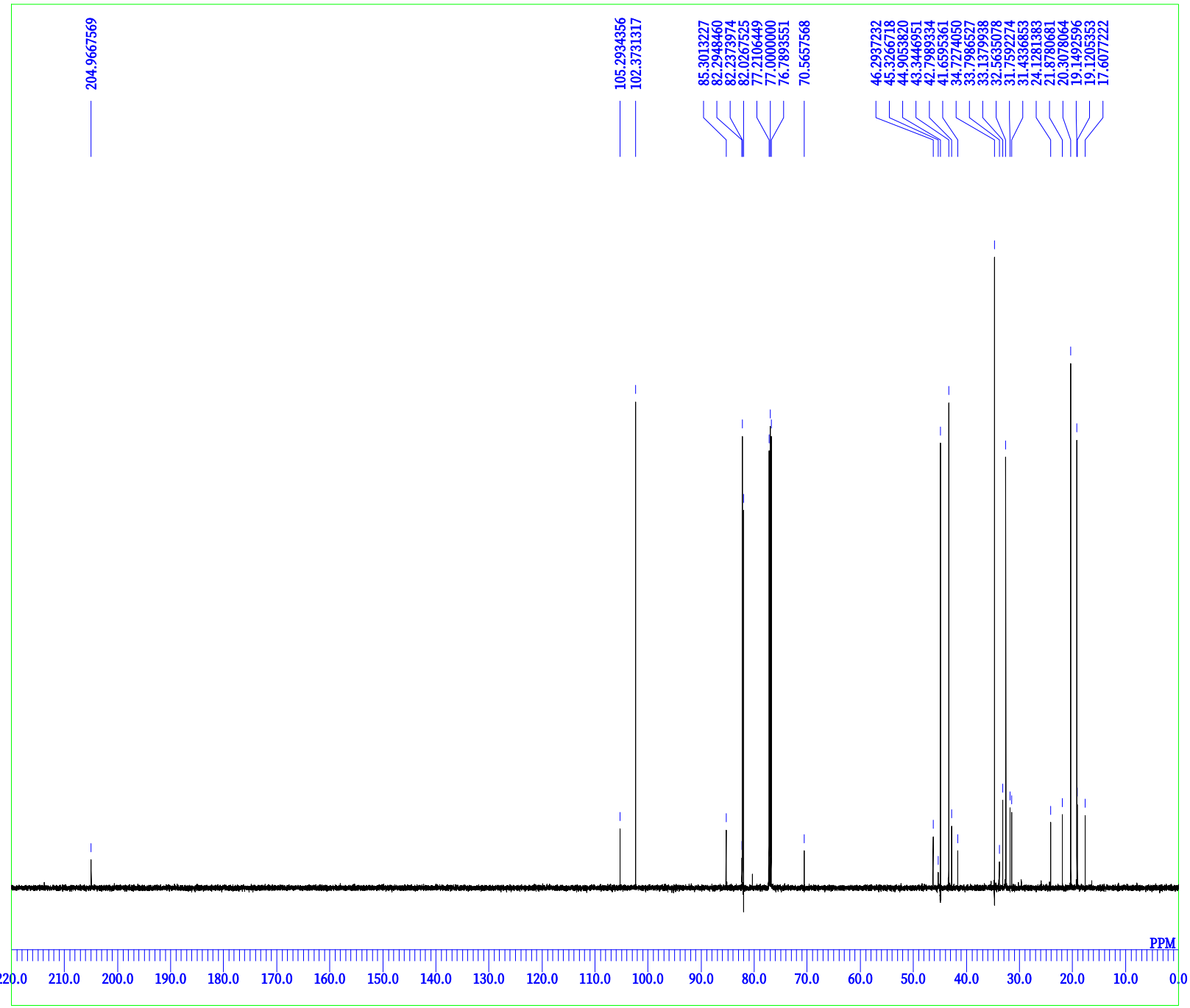


DFILE	2i_1H.als
COMNT	2i
DATIM	15-03-2012 11:21:31
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	26213
FREQU	9008.87 Hz
SCANS	16
ACQTM	2.9098 sec
PD	2.0000 sec
PW1	5.60 usec
IRNUC	1H
CTEMP	20.3 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.00 Hz
RGAIN	30

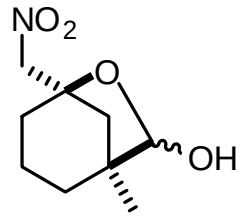


2i

2i

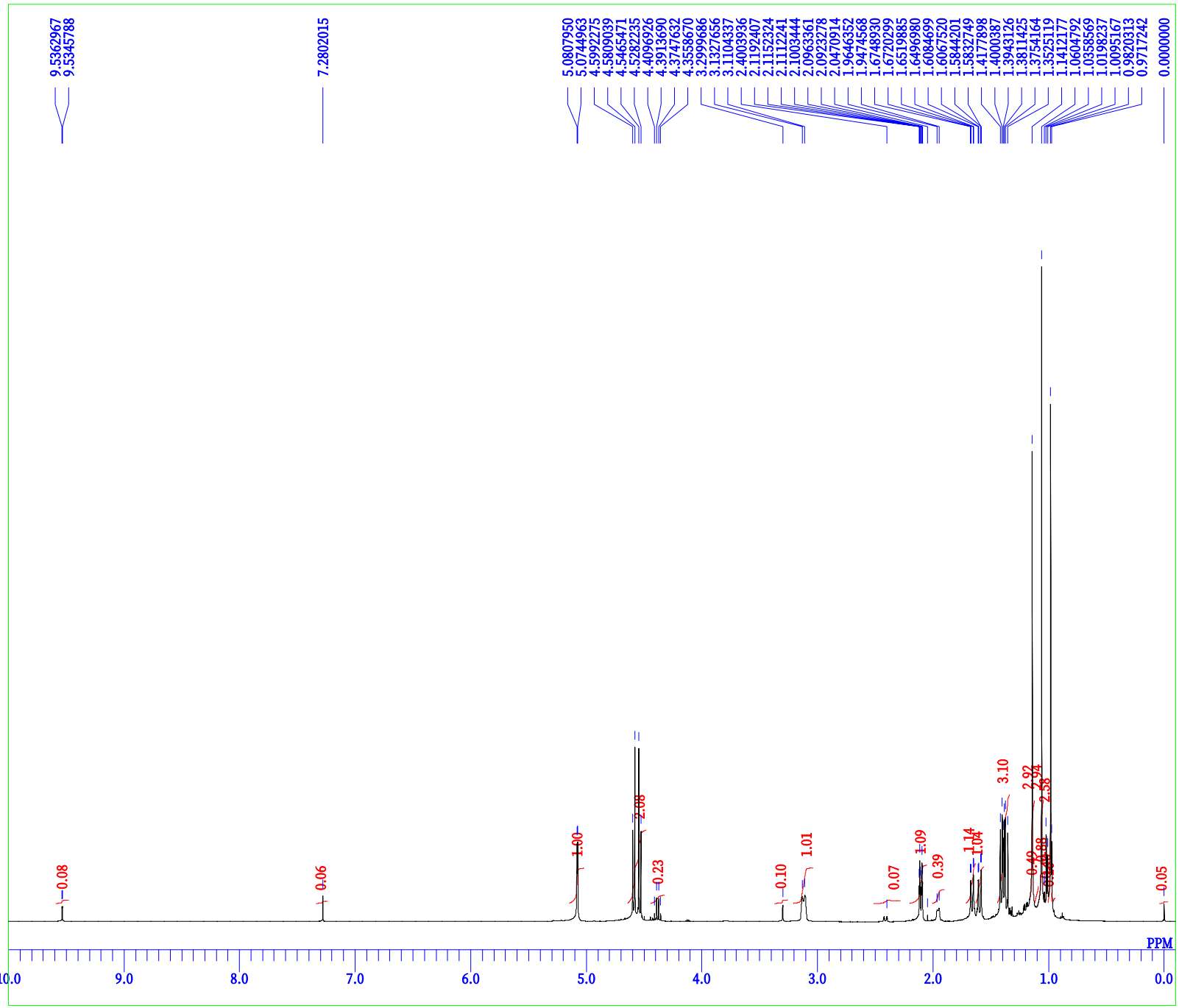


DFILE	2i_13C.als
COMNT	2i
DATIM	15-03-2012 11:39:35
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	32768
FREQU	47348.49 Hz
SCANS	559
ACQTM	0.6921 sec
PD	1.2000 sec
PW1	2.77 usec
IRNUC	1H
CTEMP	21.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	56

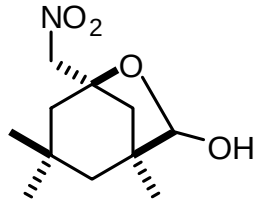


2i

2j

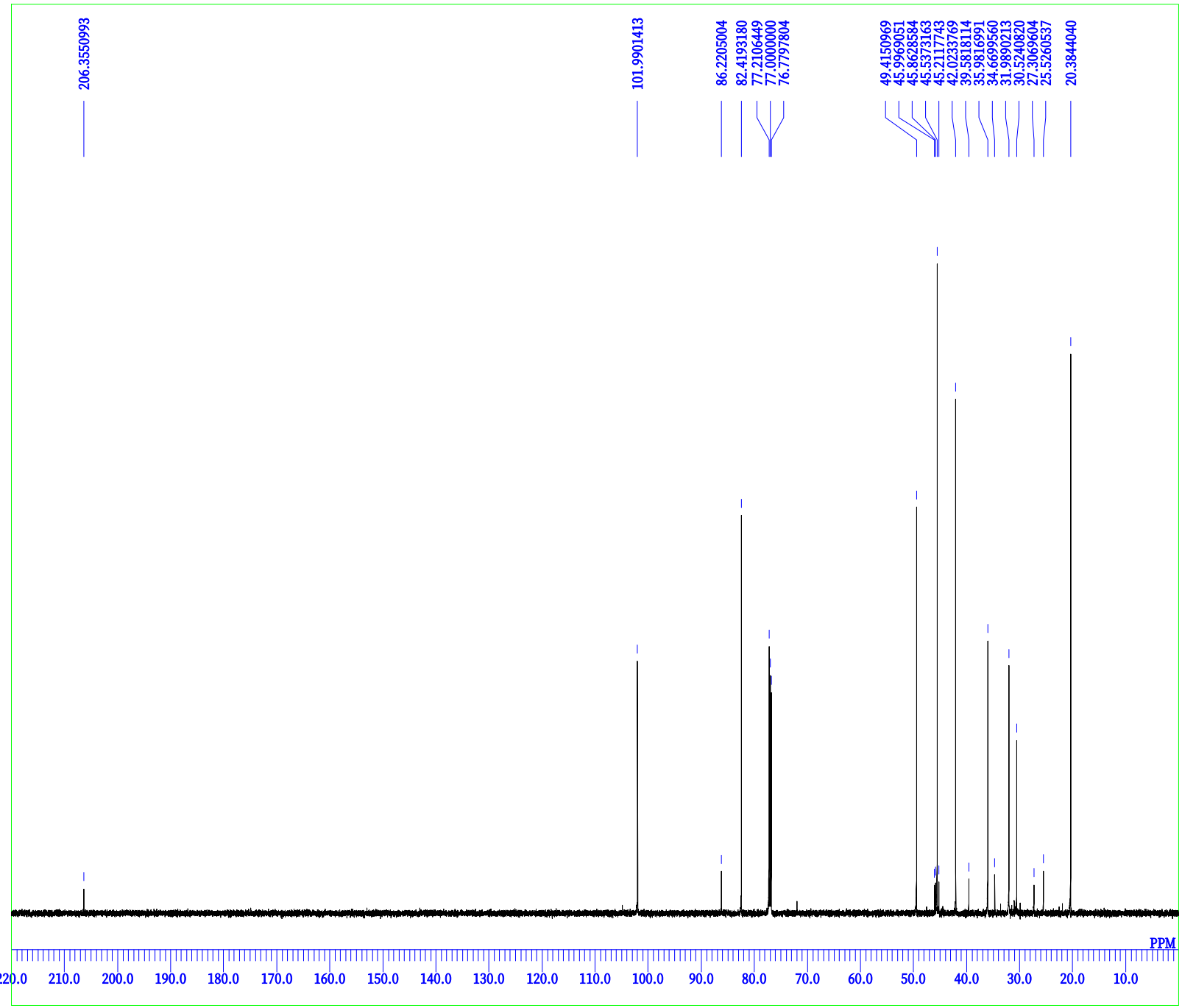


DFILE	2j_1H.als
COMNT	2j
DATIM	02-09-2011 21:16:42
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	26214
FREQU	9008.87 Hz
SCANS	16
ACQTM	2.9098 sec
PD	2.0000 sec
PW1	5.60 usec
IRNUC	1H
CTEMP	22.8 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	26

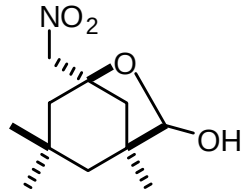


2j

2j

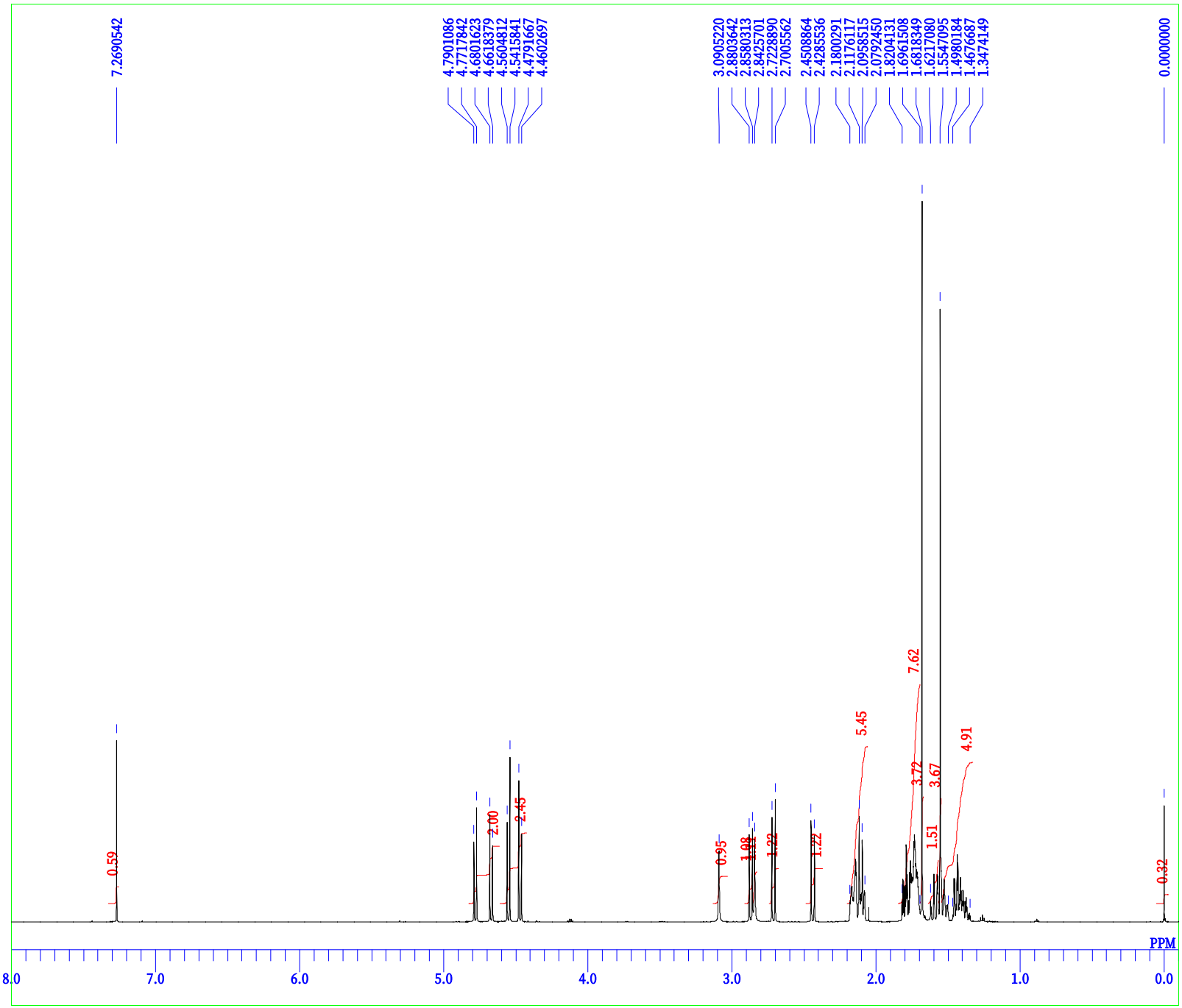


DFILE	2j_13C.als
COMNT	2j
DATIM	02-09-2011 21:33:16
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	26214
FREQU	37878.21 Hz
SCANS	512
ACQTM	0.6921 sec
PD	1.2000 sec
PW1	2.77 usec
IRNUC	1H
CTEMP	23.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	54

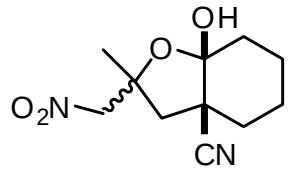


2j

2k



DFILE	2k_1H.als
COMNT	2k
DATIM	20-01-2012 23:15:54
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	20970
FREQU	7206.99 Hz
SCANS	16
ACQTM	2.9098 sec
PD	2.0000 sec
PW1	5.60 usec
IRNUC	1H
CTEMP	19.7 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	40



2k

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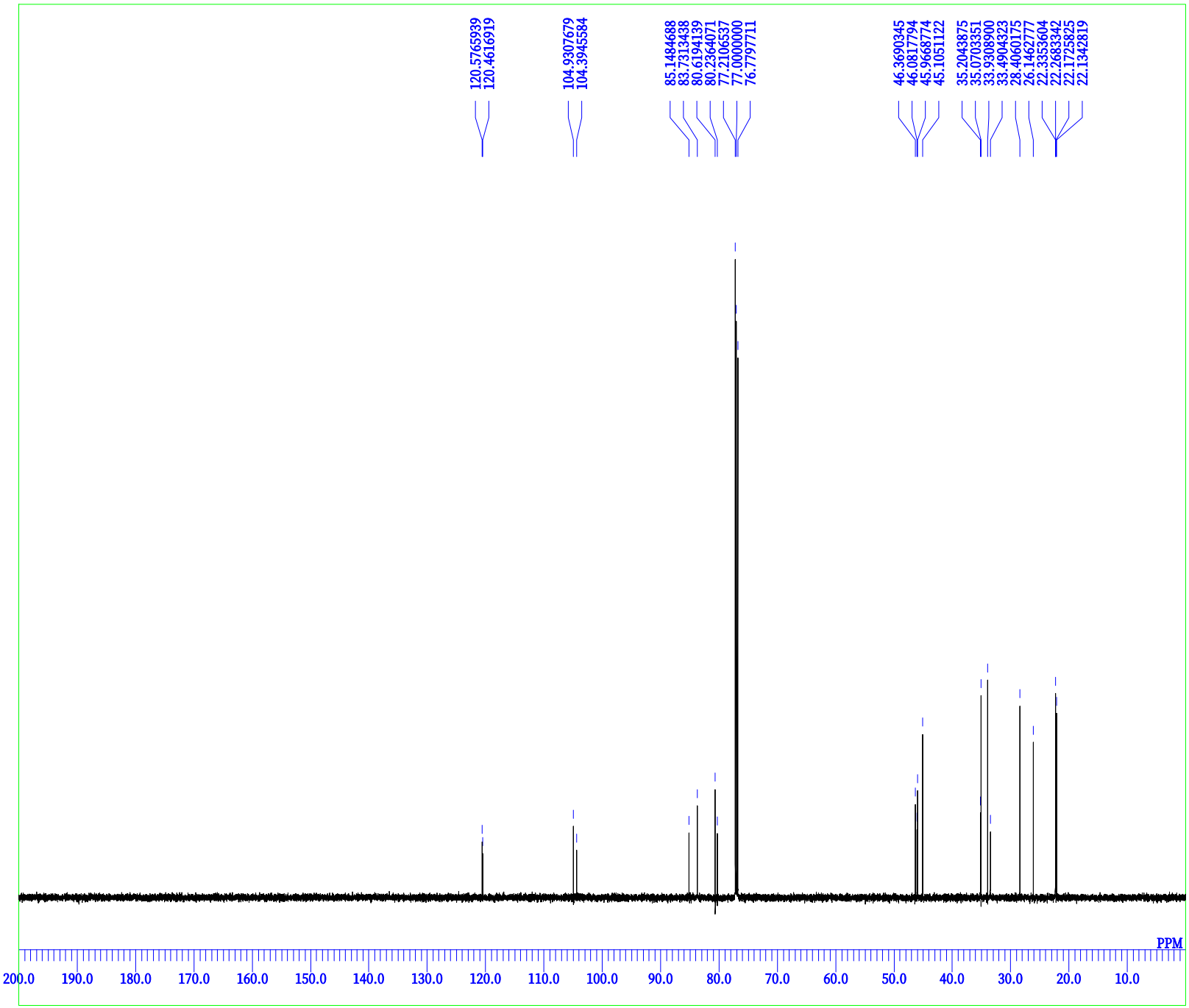
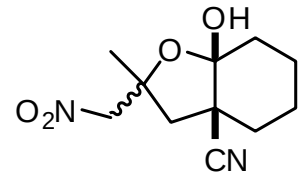
2k

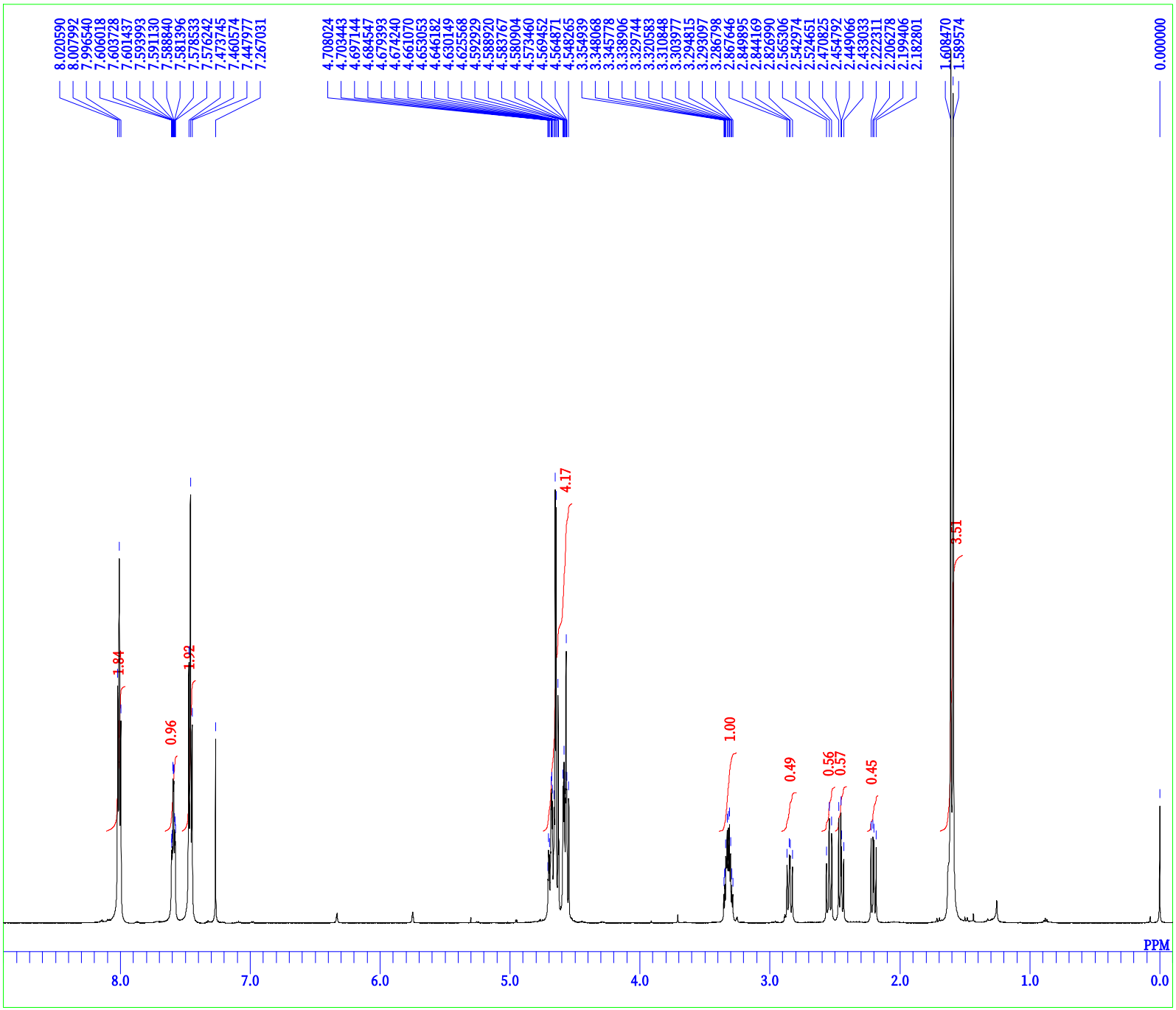


DFILE	2k_13C.als
COMNT	2k
DATIM	20-01-2012 23:33:51
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	20970
FREQU	30302.11 Hz
SCANS	540
ACQTM	0.6921 sec
PD	1.2000 sec
PW1	2.77 usec
IRNUC	1H
CTEMP	20.3 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	56

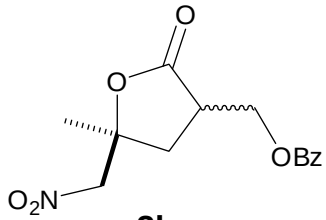


2k

2l_1H

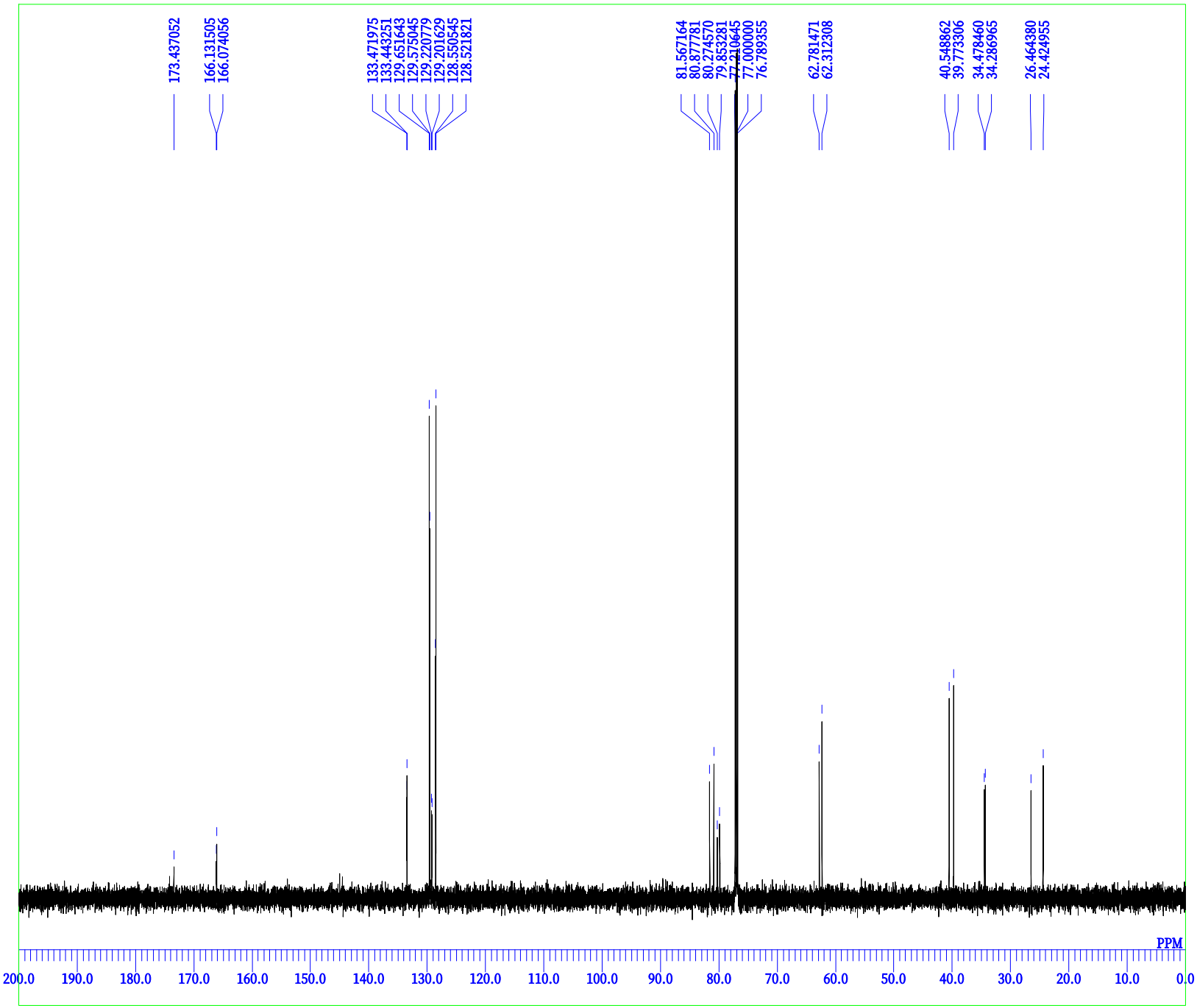


DFILE	th-277-1H-02-1.als
COMNT	2l_1H
DATIM	24-11-2012 10:22:32
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	26214
FREQU	9008.87 Hz
SCANS	16
ACQTM	2.9098 sec
PD	2.0000 sec
PW1	6.75 usec
IRNUC	1H
CTEMP	19.1 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	38

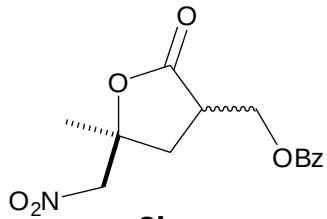


2l

2l_13C

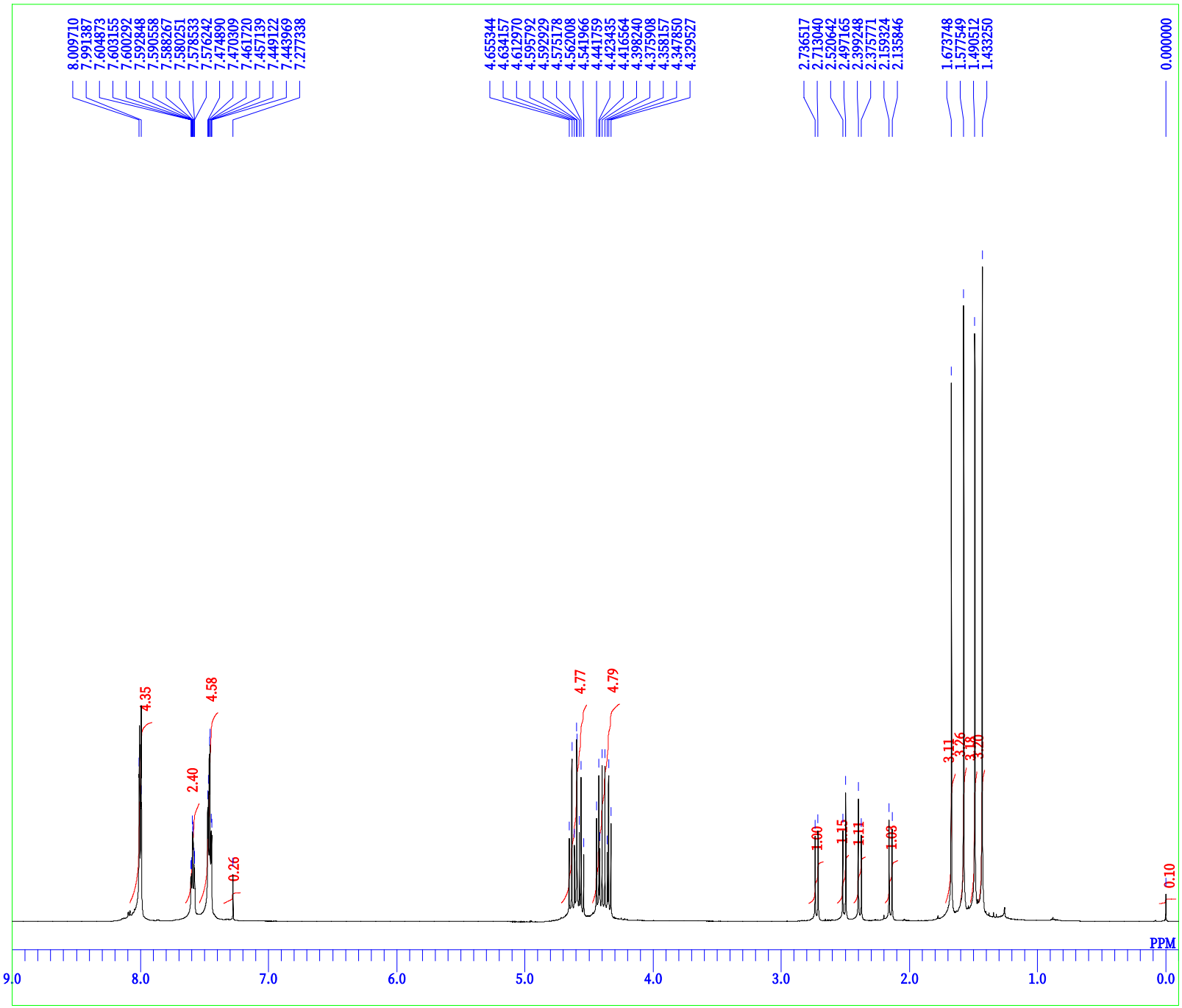


DFILE	th-277-13C-02-1.als
COMNT	2l_13C
DATIM	24-11-2012 10:29:17
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	26214
FREQU	37878.21 Hz
SCANS	200
ACQTM	0.6921 sec
PD	1.2000 sec
PW1	2.77 usec
IRNUC	1H
CTEMP	19.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	54

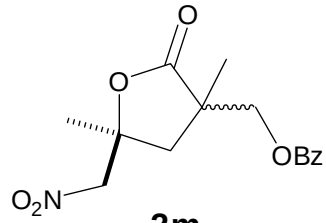


2l

2m_1H

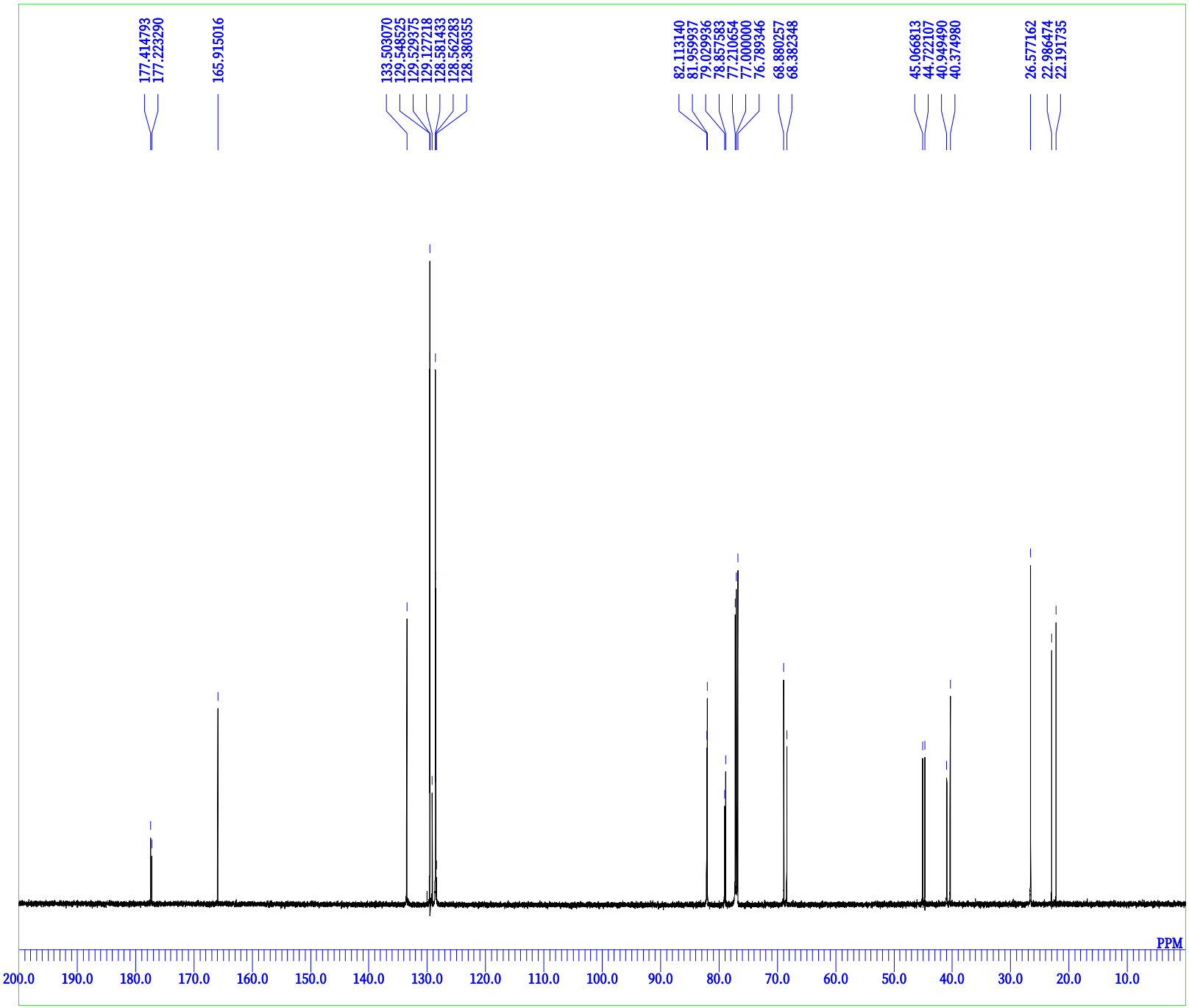


DFILE	2m_1H.als
COMNT	2m_1H
DATIM	22-09-2011 21:30:39
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	26214
FREQU	9008.87 Hz
SCANS	16
ACQTM	2.9098 sec
PD	2.0000 sec
PW1	5.60 usec
IRNUC	1H
CTEMP	22.0 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.24 Hz
RGAIN	30

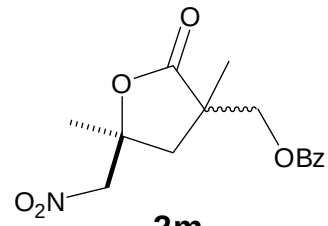


2m

2m_13C

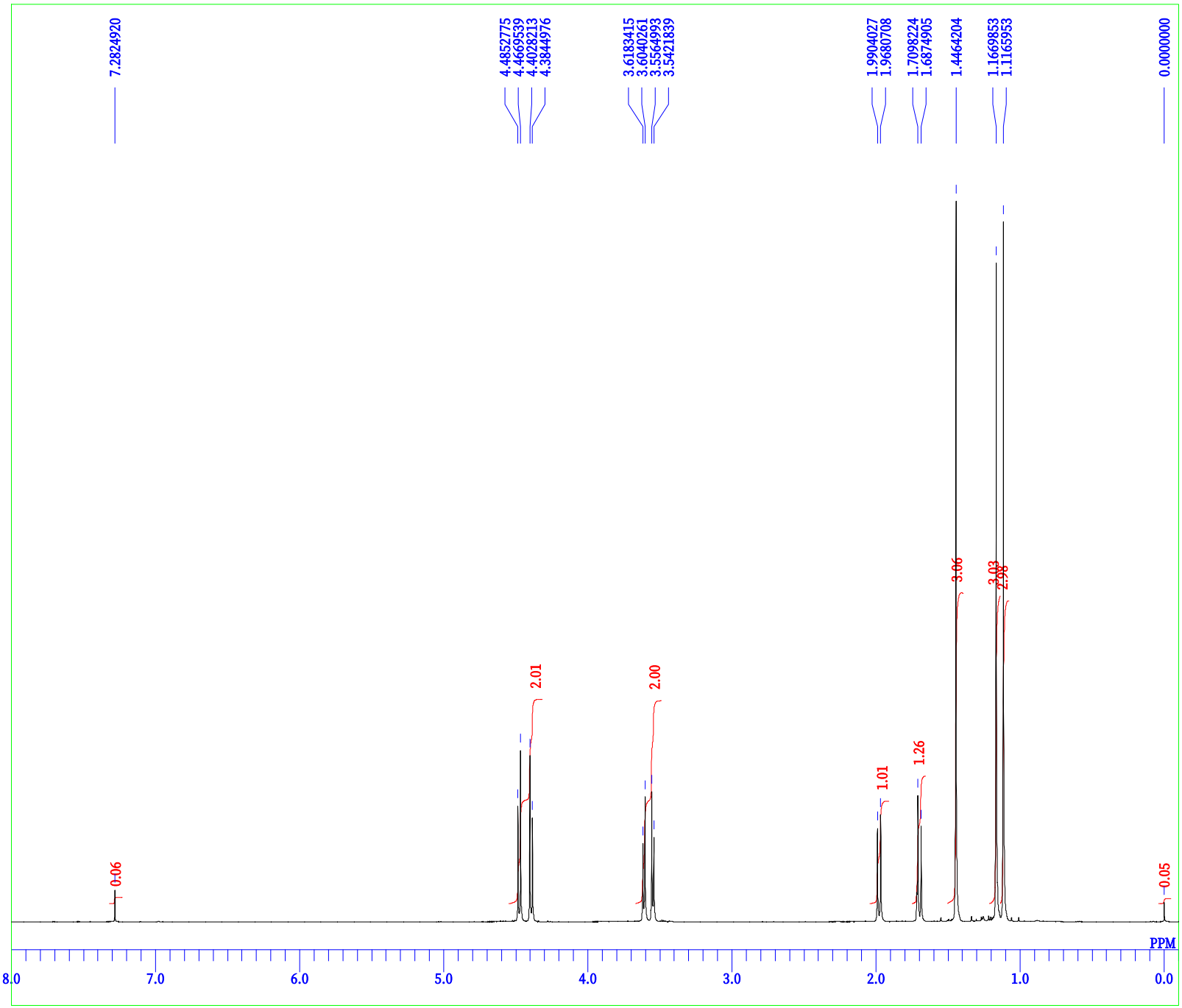


DFILE	2m_13C.als
COMNT	2m_13C
DATIM	22-09-2011 21:54:52
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	20970
FREQU	30302.11 Hz
SCANS	754
ACQTM	0.6921 sec
PD	1.2000 sec
PW1	2.77 usec
IRNUC	1H
CTEMP	22.5 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.24 Hz
RGAIN	54

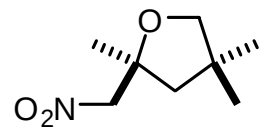


2m

4a

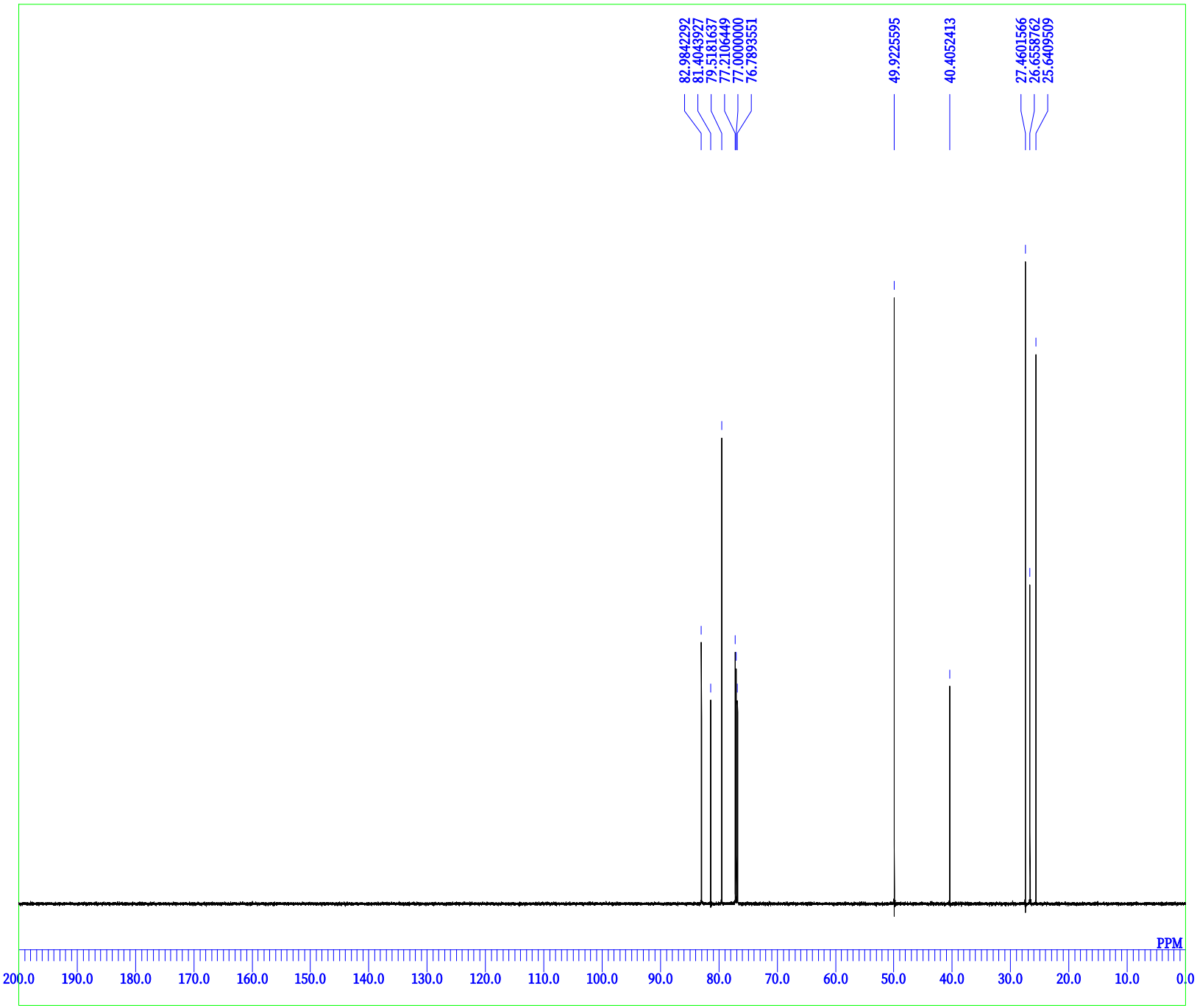


DFILE	4a_1H.als
COMNT	4a
DATIM	26-09-2011 15:44:42
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	26214
FREQU	9008.87 Hz
SCANS	16
ACQTM	2.9098 sec
PD	2.0000 sec
PW1	5.60 usec
IRNUC	1H
CTEMP	22.1 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.24 Hz
RGAIN	26

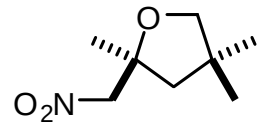


4a

4a

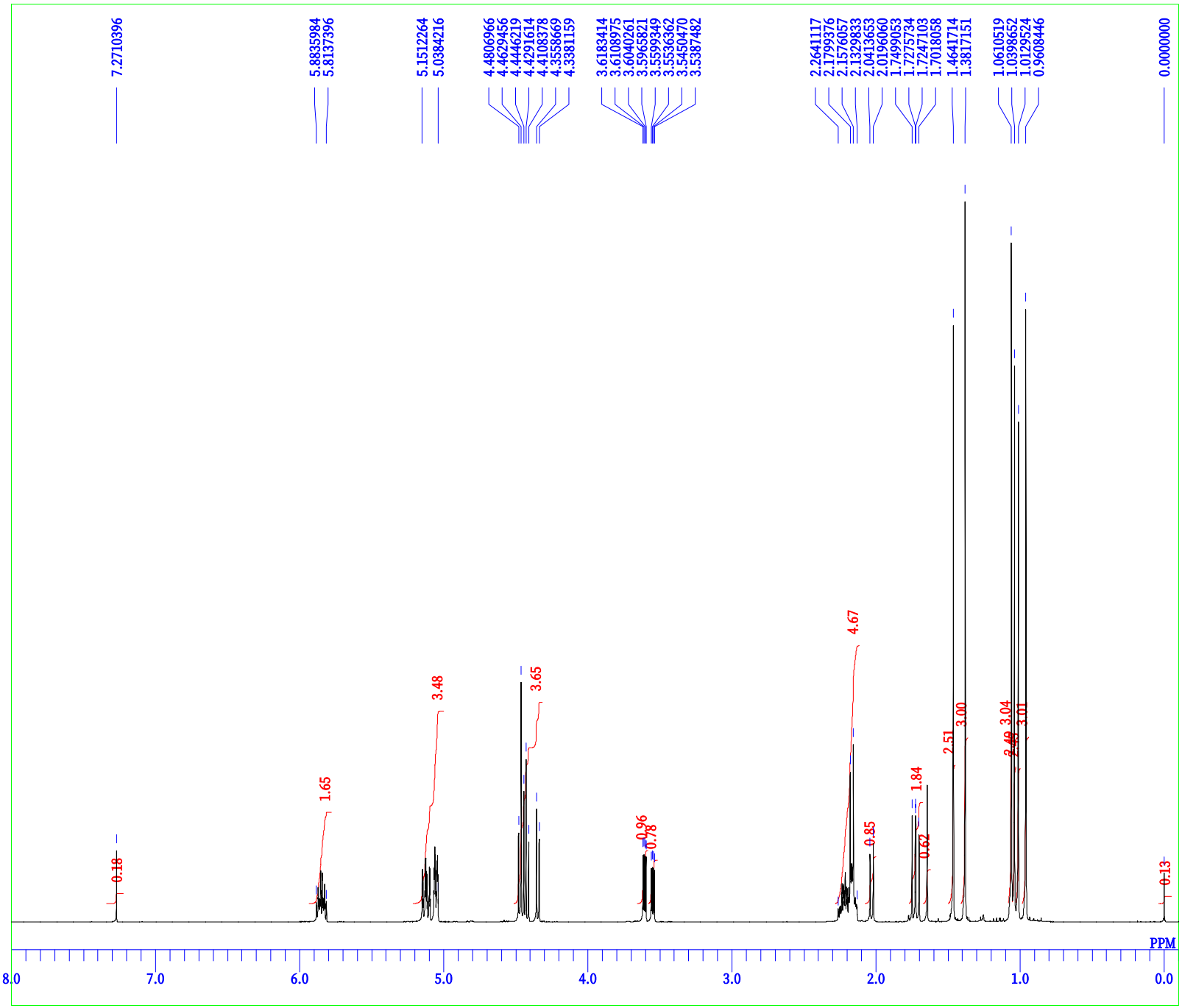


DFILE	4a_13C.als
COMNT	4a
DATIM	26-09-2011 16:09:14
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	26214
FREQU	37878.21 Hz
SCANS	764
ACQTM	0.6921 sec
PD	1.2000 sec
PW1	2.77 usec
IRNUC	1H
CTEMP	22.8 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.24 Hz
RGAIN	54

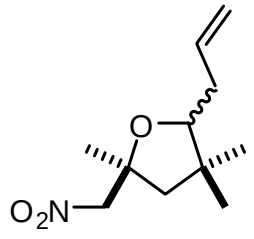


4a

5a

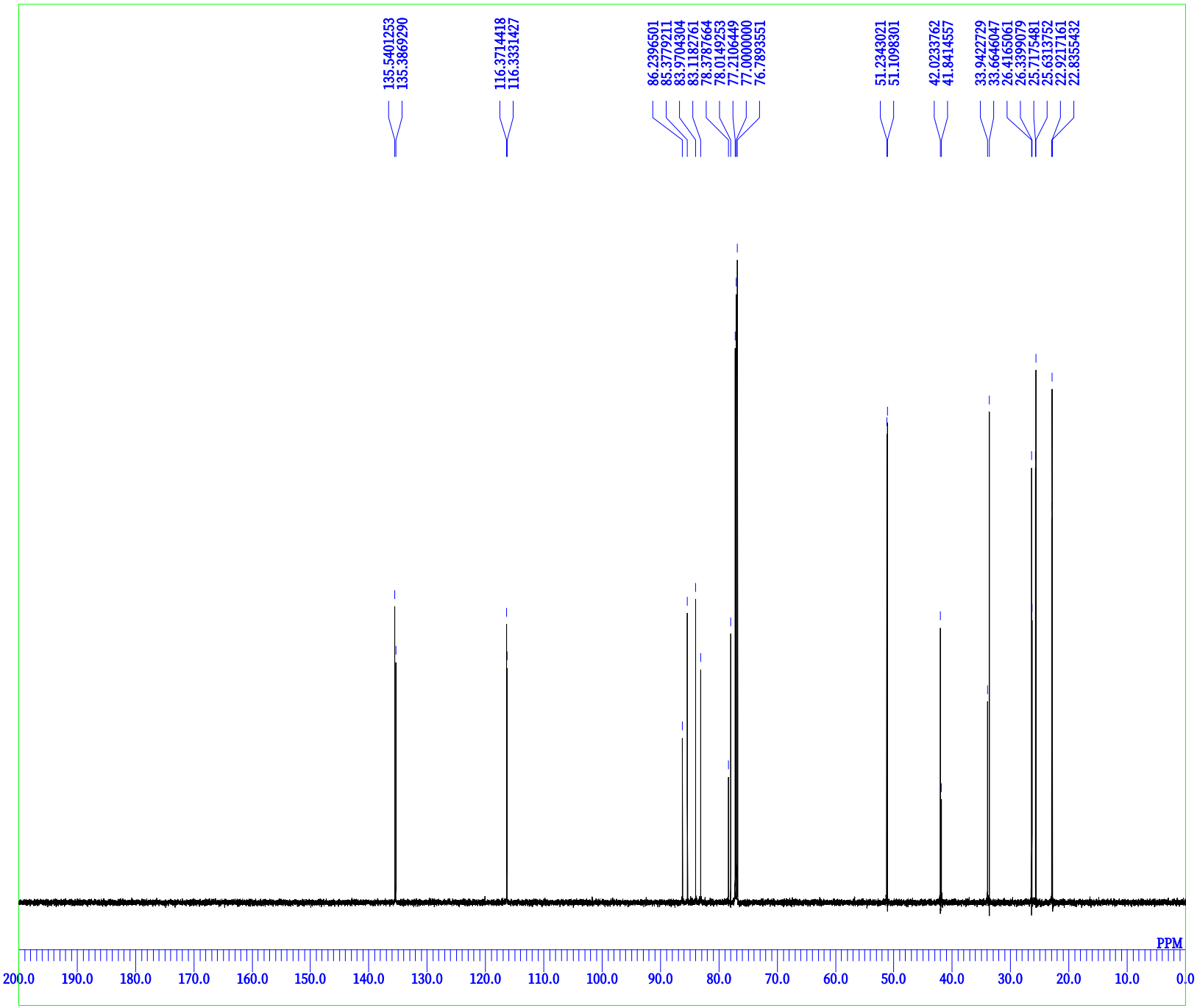


DFILE	5a_1H.als
COMNT	5a
DATIM	22-09-2011 21:59:46
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	26214
FREQU	9008.87 Hz
SCANS	16
ACQTM	2.9098 sec
PD	2.0000 sec
PW1	5.60 usec
IRNUC	1H
CTEMP	22.1 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.24 Hz
RGAIN	30

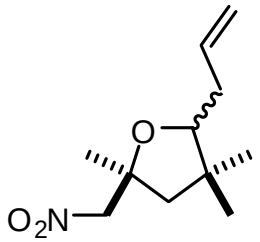


5a

5a

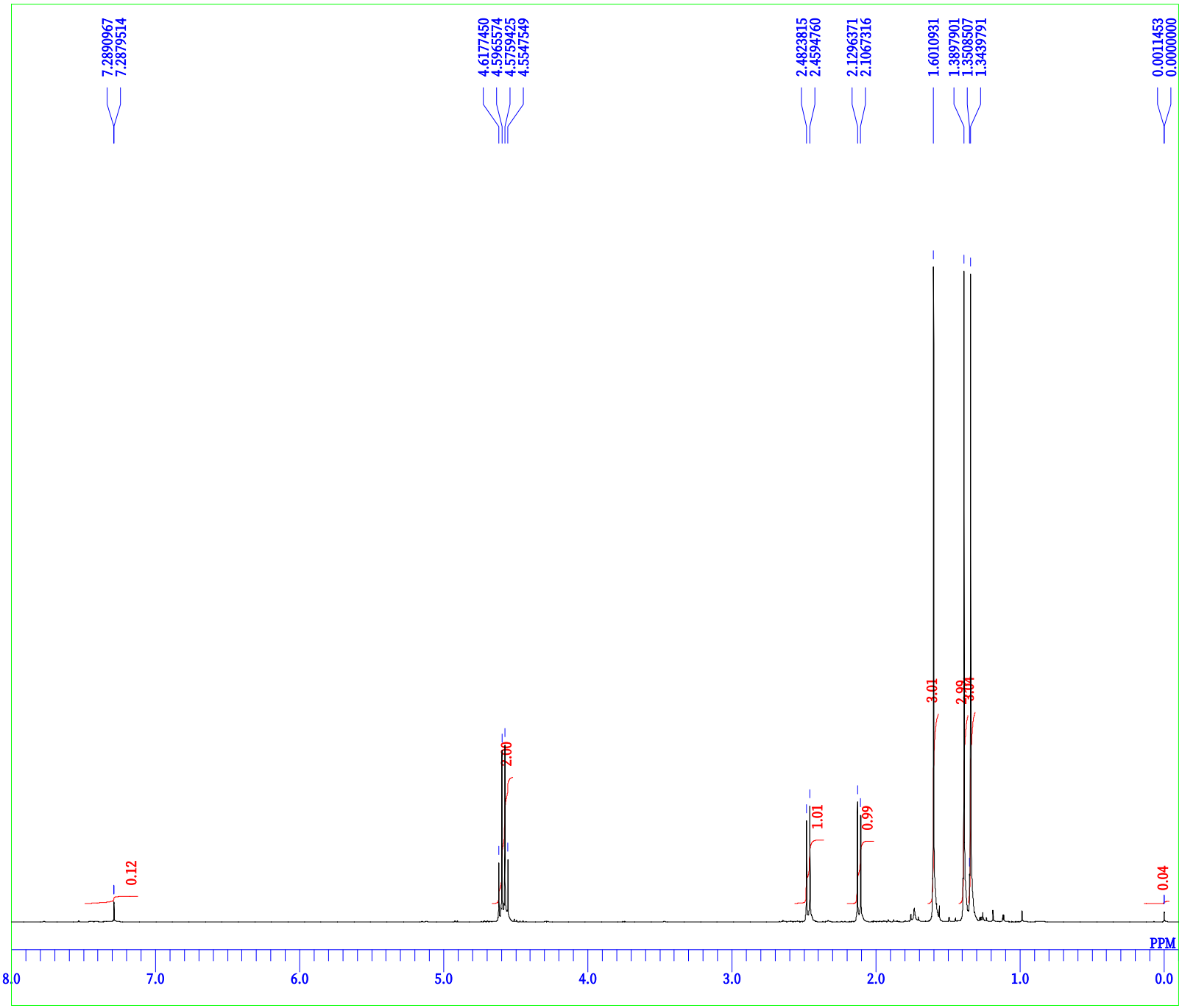


DFILE	5a_13C.als
COMNT	5a
DATIM	22-09-2011 22:31:47
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	26214
FREQU	37878.21 Hz
SCANS	1000
ACQTM	0.6921 sec
PD	1.2000 sec
PW1	2.77 usec
IRNUC	1H
CTEMP	22.7 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.24 Hz
RGAIN	52

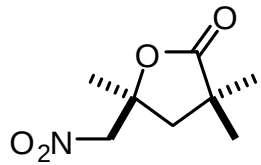


5a

6a

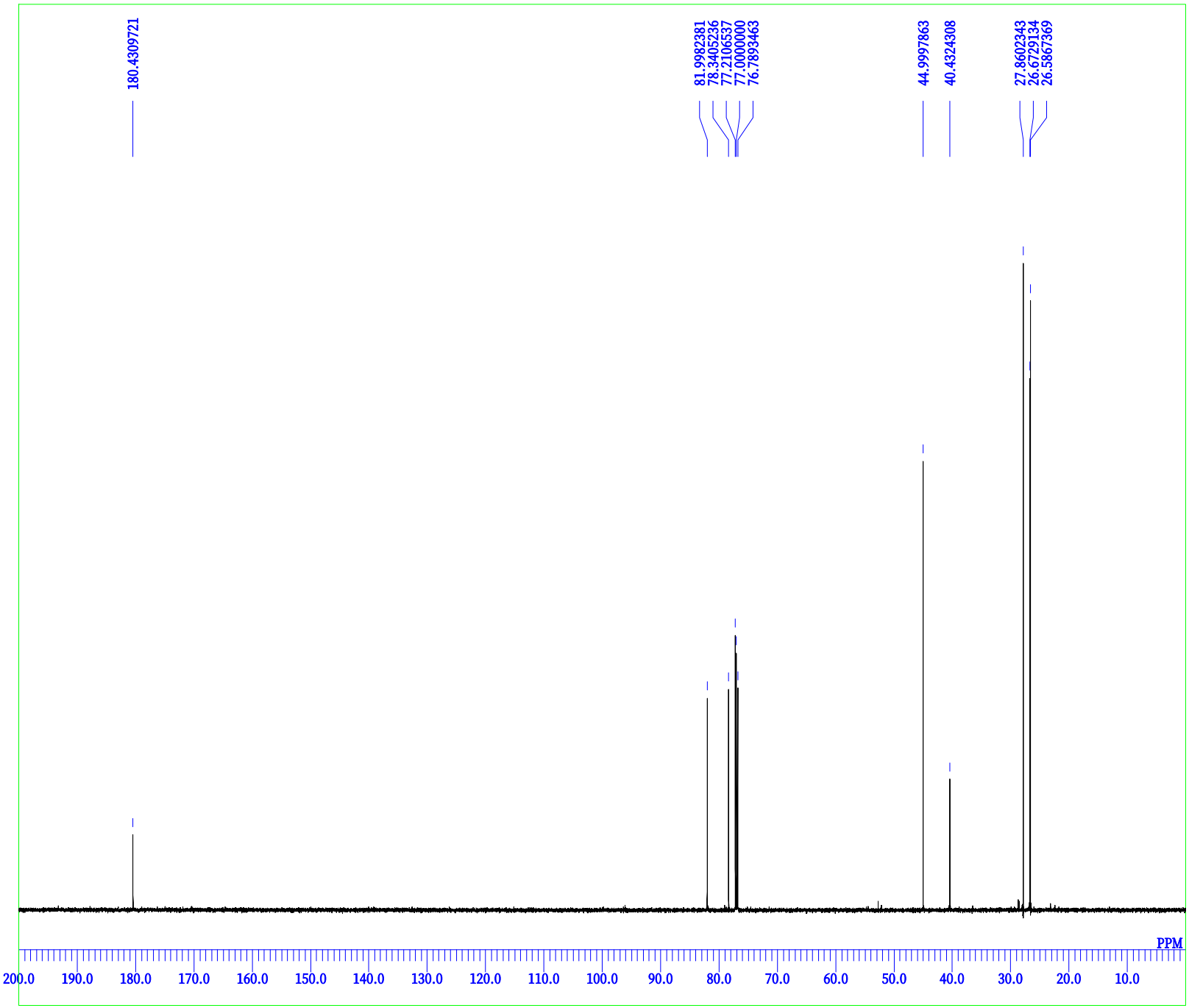


DFILE 6a_1H.als
COMNT 6a
DATIM 01-11-2011 13:43:35
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 600.17 MHz
OBSET 5.30 KHz
OBFIN 5.47 Hz
POINT 20970
FREQU 7206.99 Hz
SCANS 16
ACQTM 2.9098 sec
PD 2.0000 sec
PW1 5.60 usec
IRNUC 1H
CTEMP 21.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 30

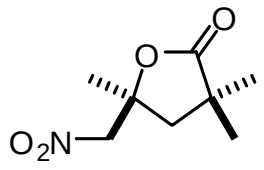


6a

6a

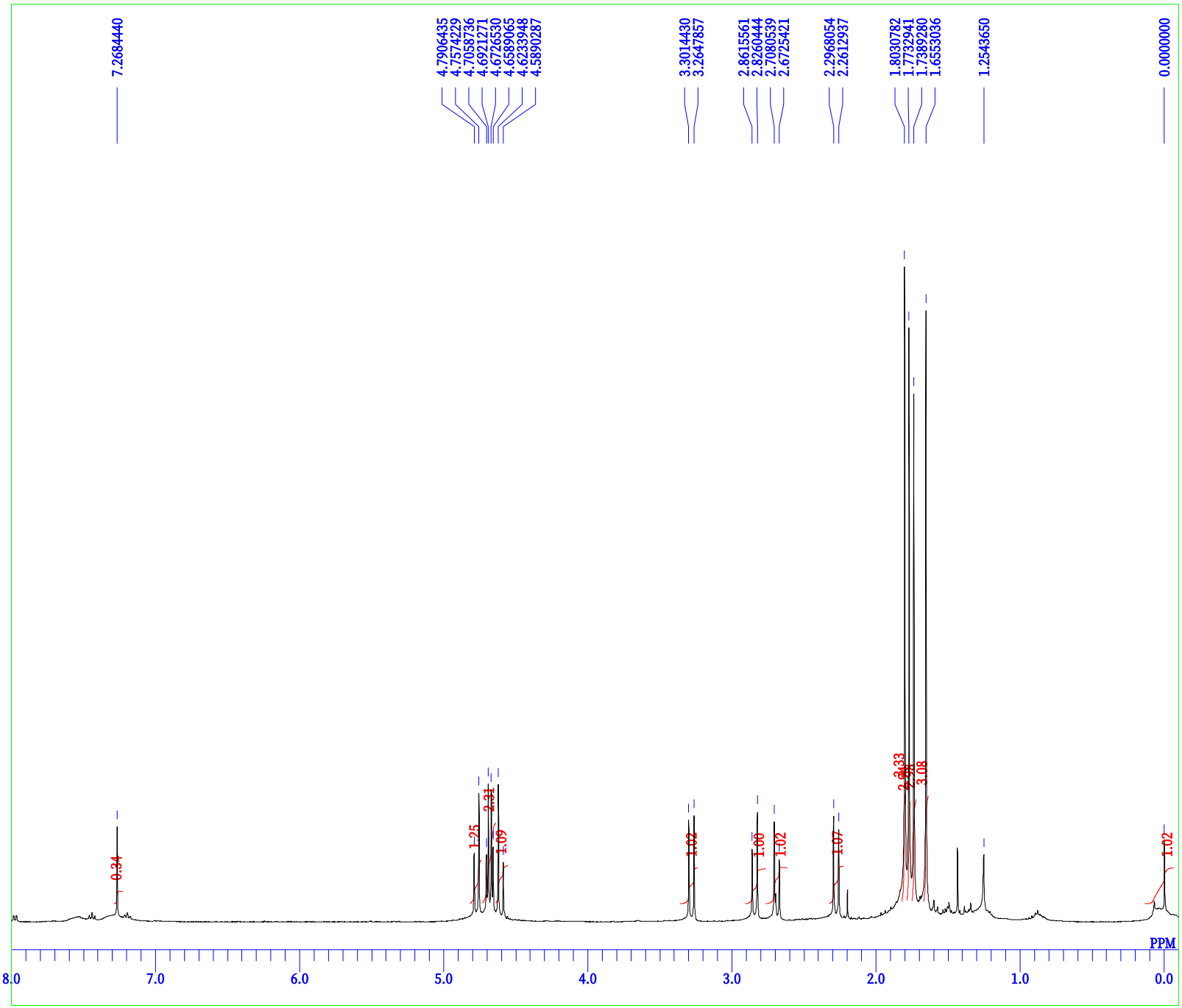


DFILE	6a_13C.als
COMNT	6a
DATIM	01-11-2011 14:04:07
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	20970
FREQU	30302.11 Hz
SCANS	637
ACQTM	0.6921 sec
PD	1.2000 sec
PW1	2.77 usec
IRNUC	1H
CTEMP	22.8 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	56

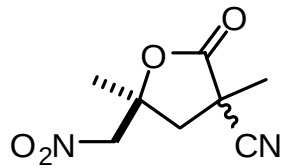


6a

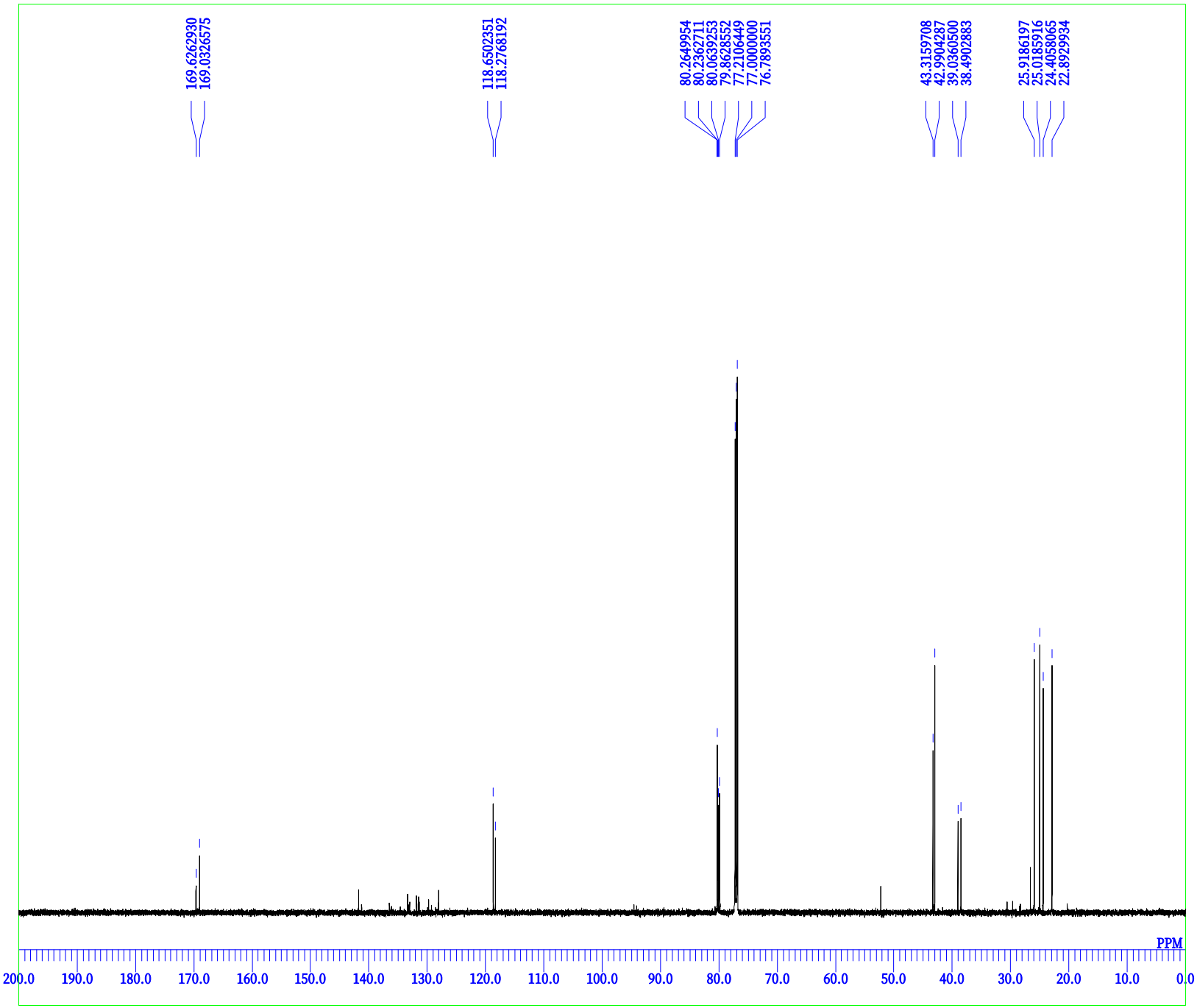
2e_oxi



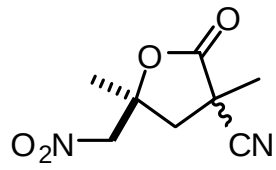
DFILE	2e_oxi_1H.als
COMNT	2e_oxi
DATIM	25-08-2011 10:08:52
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	10485
FREQU	4801.77 Hz
SCANS	16
ACQTM	2.1837 sec
PD	2.0000 sec
PW1	4.75 usec
IRNUC	1H
CTEMP	23.3 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.24 Hz
RGAIN	38



2e_oxi



DFILE	2e_oxi_13C.als
COMNT	2e_oxi
DATIM	17-03-2012 03:49:44
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	26214
FREQU	37878.21 Hz
SCANS	1000
ACQTM	0.6921 sec
PD	1.2000 sec
PW1	2.77 usec
IRNUC	1H
CTEMP	19.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.24 Hz
RGAIN	54



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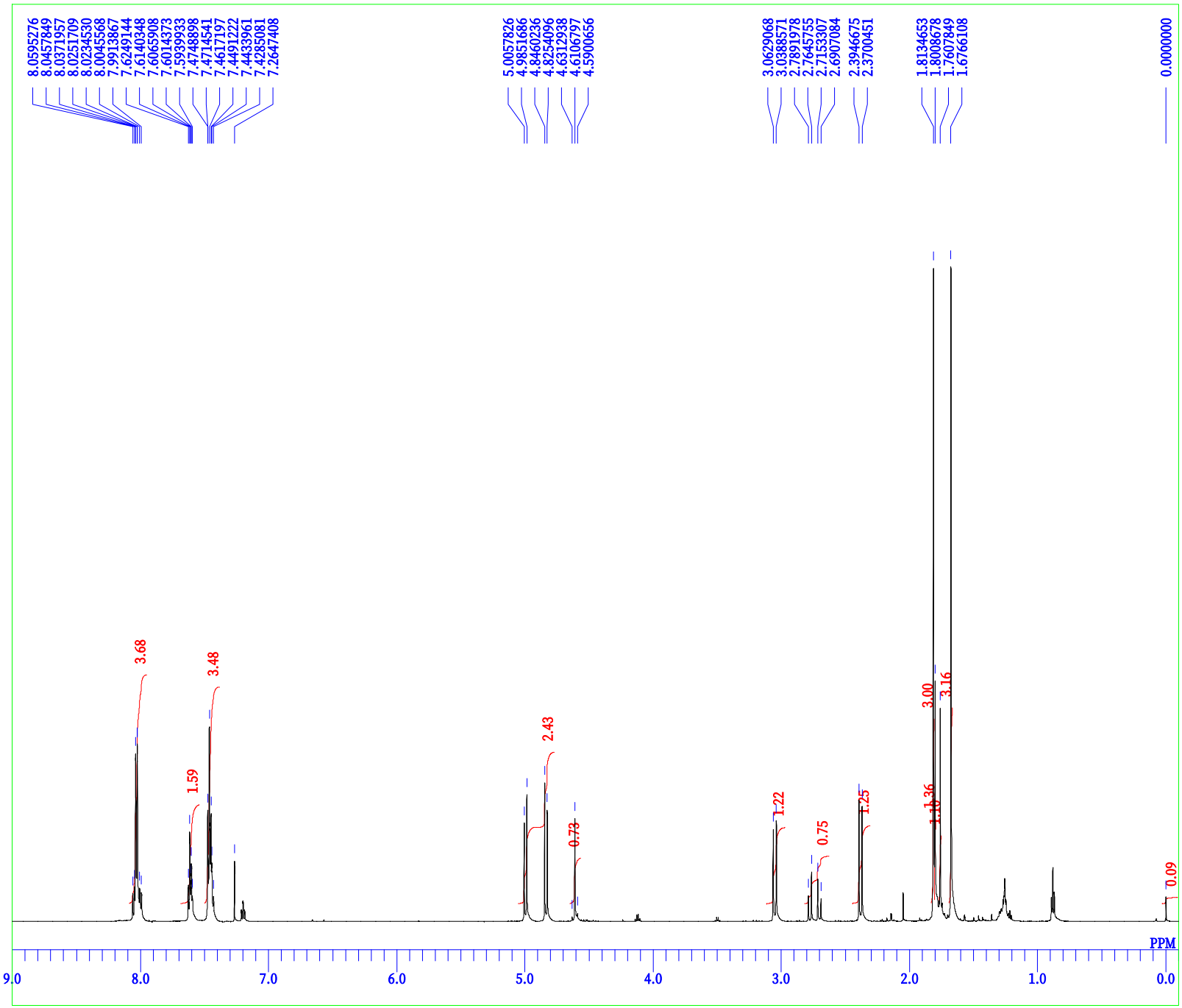
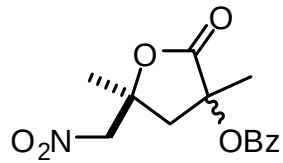
2f_oxi



DFILE	2f_oxi_1H.als
COMNT	2f_oxi
DATIM	18-03-2012 22:15:36
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	26214
FREQU	9008.87 Hz
SCANS	16
ACQTM	2.9098 sec
PD	2.0000 sec
PW1	5.60 usec
IRNUC	1H
CTEMP	18.5 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.00 Hz
RGAIN	34



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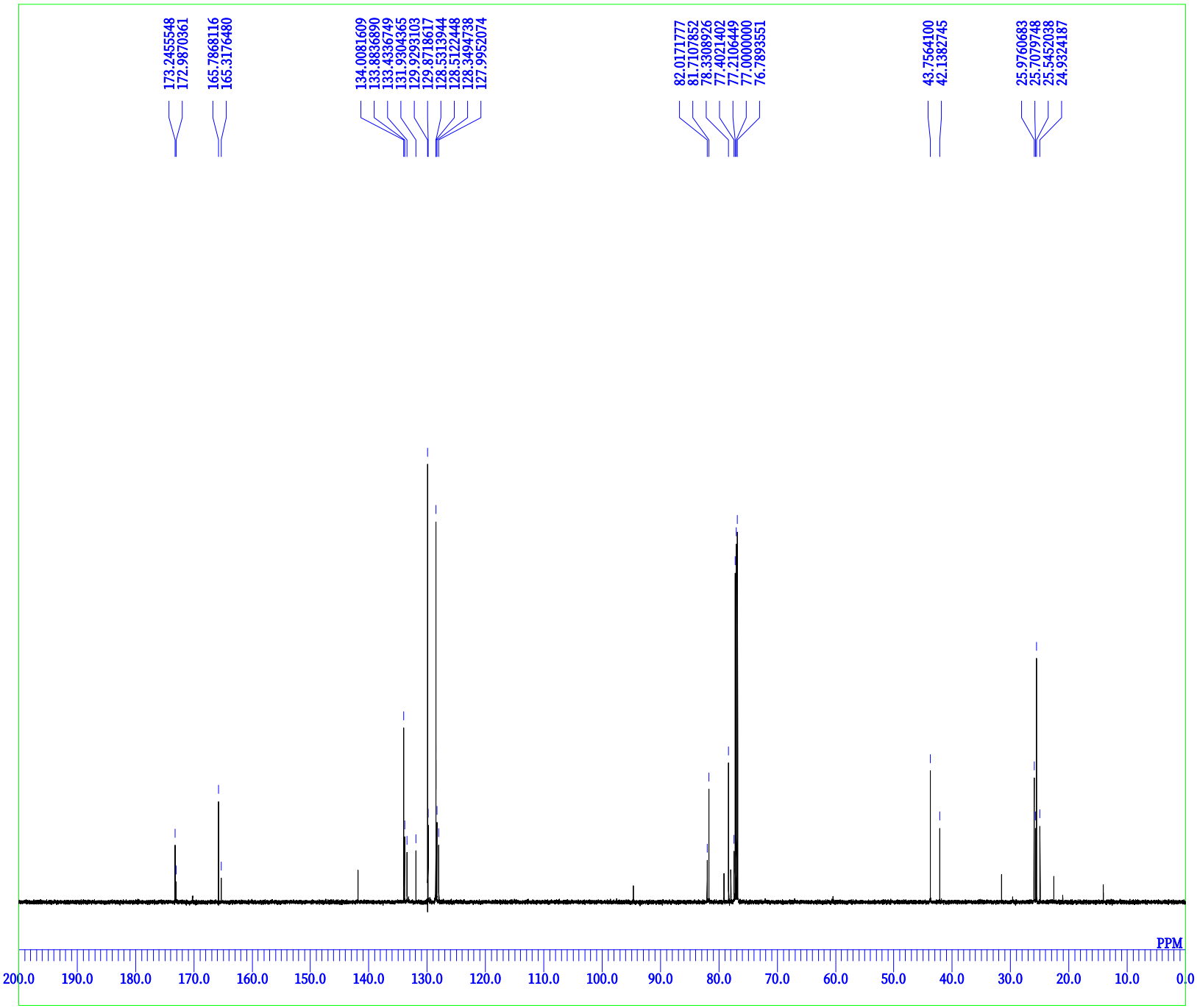
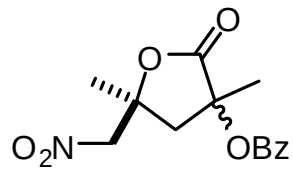
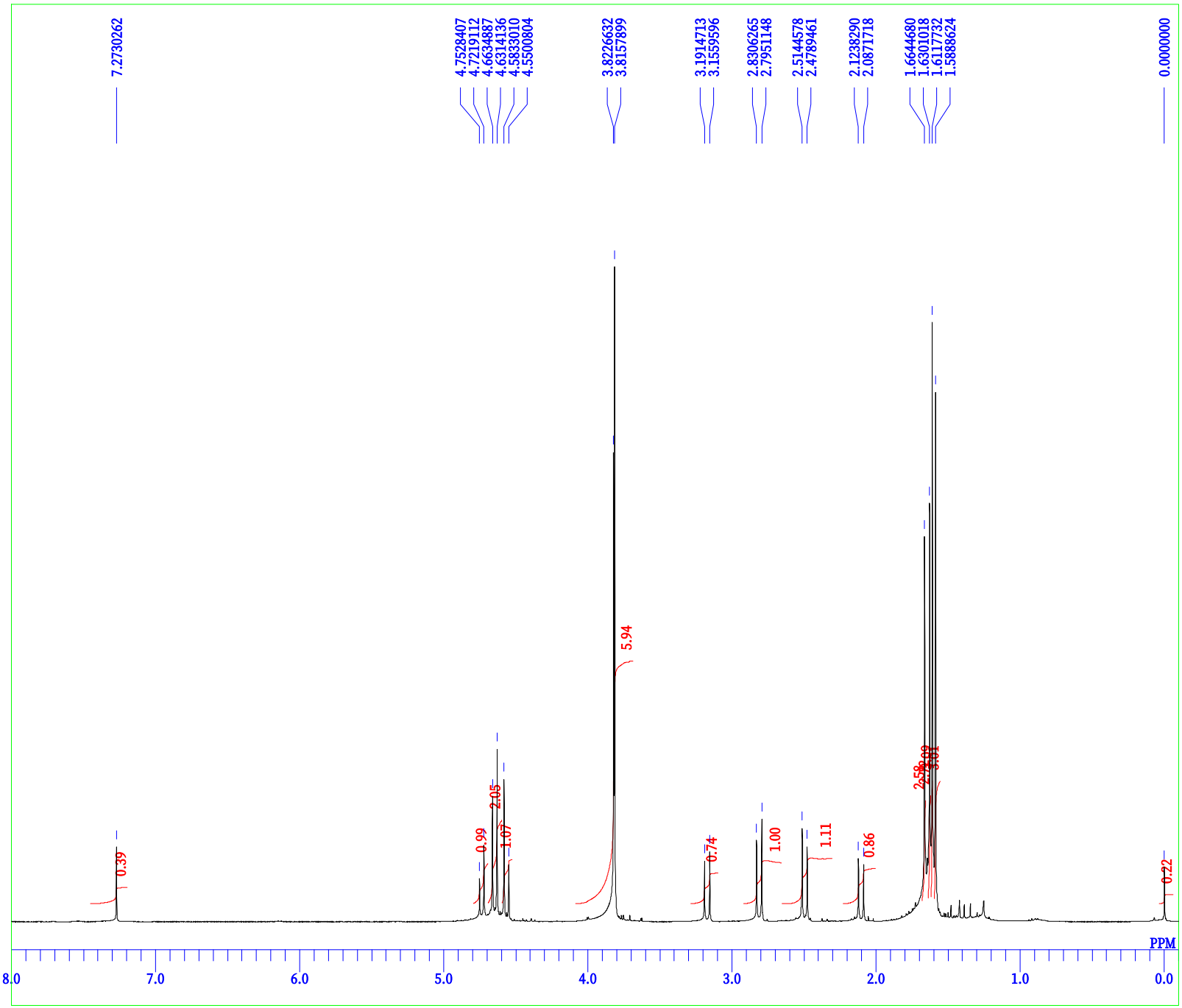
2f



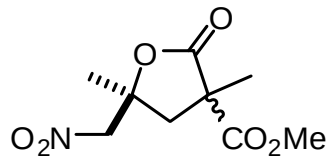
DFILE	2f_oxi_13C-1.jdf
COMNT	2f
DATIM	18-03-2012 22:47:33
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	32768
FREQU	47348.49 Hz
SCANS	1000
ACQTM	0.6921 sec
PD	1.2000 sec
PW1	2.77 usec
IRNUC	1H
CTEMP	19.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	54



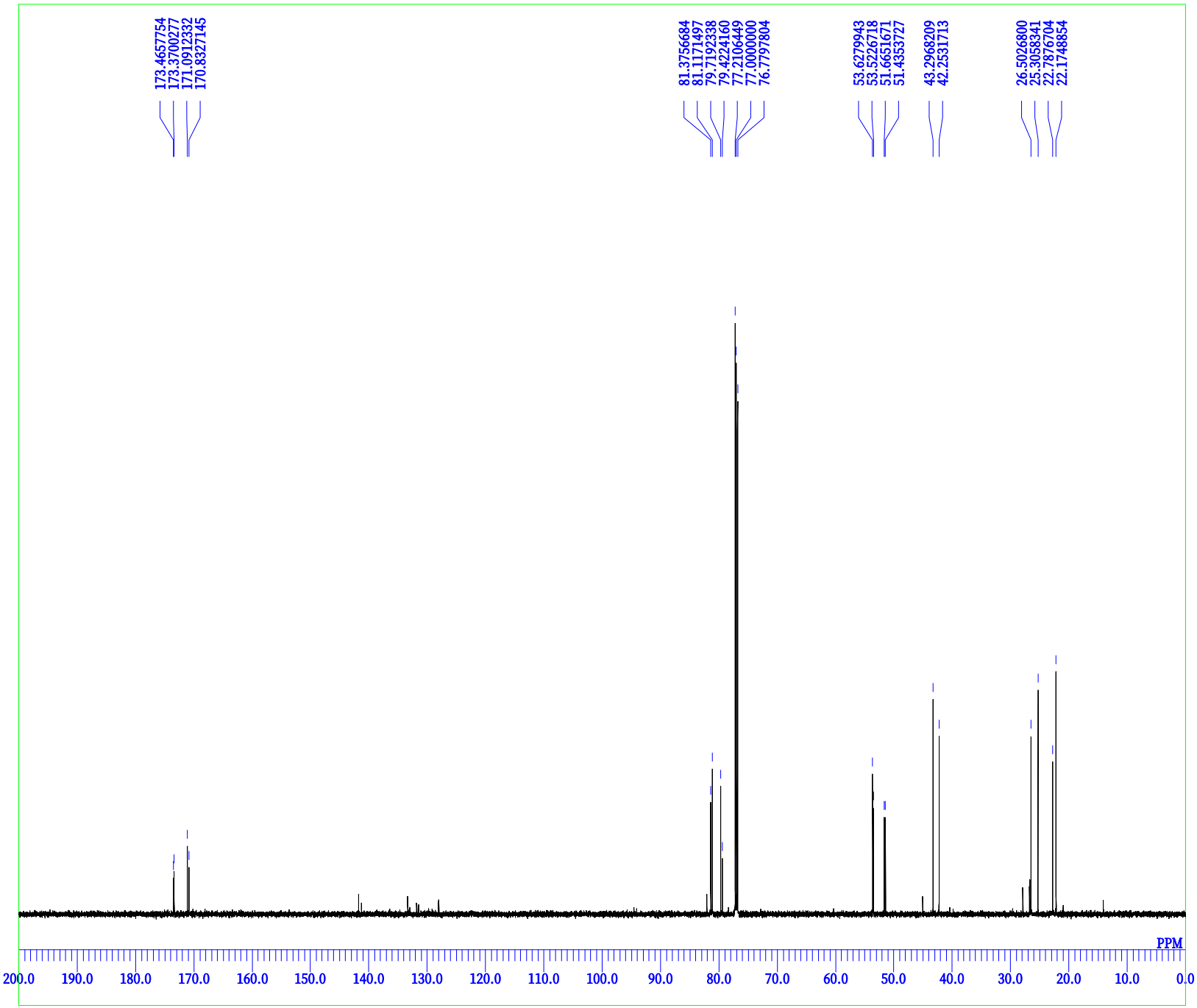
2g_oxi



DFILE 2g_oxi_1H.als
COMNT 2g_oxi
DATIM 25-08-2011 10:17:44
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 10485
FREQU 4801.77 Hz
SCANS 16
ACQTM 2.1837 sec
PD 2.0000 sec
PW1 4.75 usec
IRNUC 1H
CTEMP 23.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.24 Hz
RGAIN 36



2g_oxi



DFILE	2g_oxi_13C.als
COMNT	2g_oxi
DATIM	17-03-2012 07:51:54
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	26214
FREQU	37878.21 Hz
SCANS	1000
ACQTM	0.6921 sec
PD	1.2000 sec
PW1	2.77 usec
IRNUC	1H
CTEMP	19.7 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.24 Hz
RGAIN	54