

Supporting Information

The Bulky Side Chain of Antillatoxin is Important for Potent Toxicity: Rational Design of Photoresponsive Cytotoxins Based on SAR Study

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67 pages

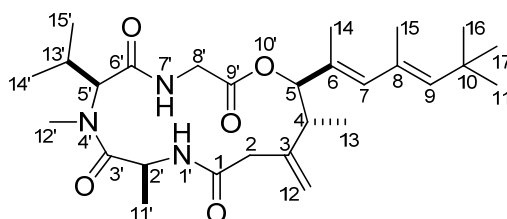
Contents:

S2-S17: Experimental

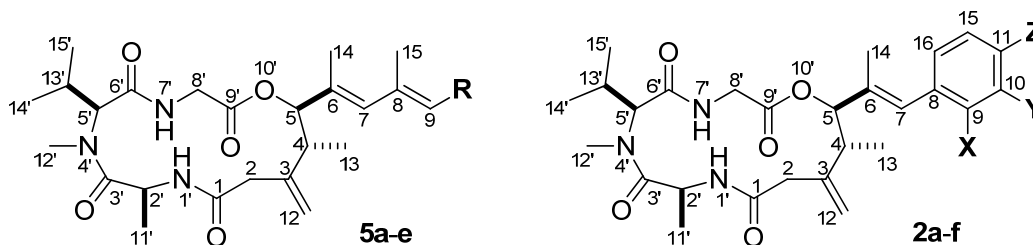
S18-S67: ^1H and ^{13}C NMR spectra of newly synthesized compounds

General Methods. ^1H and ^{13}C NMR spectra were recorded on a Varian INOVA 500 (500 MHz for ^1H NMR, 125 MHz for ^{13}C NMR) spectrometer, a JEOL ECX 500 (500 MHz for ^1H NMR, 125 MHz for ^{13}C NMR) spectrometer or a JEOL ECA 500 (500 MHz for ^1H NMR, 125 MHz for ^{13}C NMR) spectrometer. Chemical shifts are denoted in δ (ppm) relative to residual solvent peaks as internal standard (CDCl_3 , ^1H δ 7.26, ^{13}C δ 77.0; CD_3OD , ^1H δ 3.31, ^{13}C δ 49.0). IR spectra were recorded on a JASCO FT/IR-410 spectrometer or a JASCO FT/IR-4100 spectrometer. Mass spectra were recorded on a PerSeptive Biosystems Mariner Biospectrometry Workstation instrument or Bruker BioTOF-Q spectrometer. Optical rotations were recorded on a JASCO P-1010 polarimeter or a JASCO P-2100 polarimeter. All reactions were monitored by TLC on MERCK TLC plates silica gel 60 F₂₅₄ under UV light (254 nm), and/or developed by 10% ethanolic phosphomolybdic acid or 5% (v/v) *p*-anisaldehyde solution in ethanol with 5% H_2SO_4 . Flash column chromatography was performed using MERCK Silica gel 60 particle size 0.040-0.063 mm (230-400 mesh ASTM). Dry hexane and toluene were dried over $\text{MS4}\text{\AA}$. Dry DMF, pyridine and DMSO were distilled over CaH_2 .

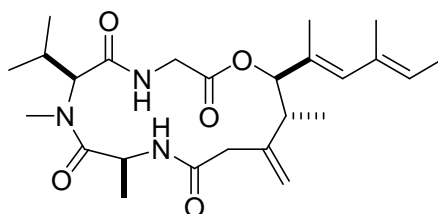
The atom numbering of compounds is shown below.



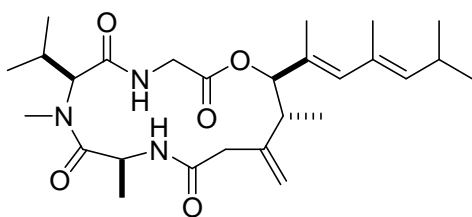
antillatoxin (**1a**)



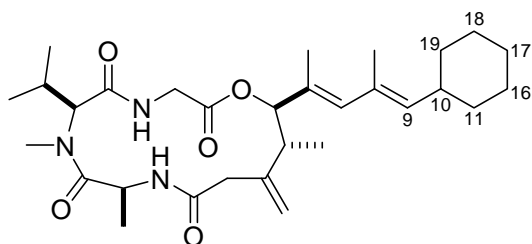
Antillatoxin (1a).^{S 1} To a mixture of **3** (48.6 mg, 91.1 μmol) and (Z)-2-(4,4-dimethylpent-2-en-2-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (81.7 mg, 364 μmol) were added dry THF (300 μL), $\text{PdCl}_2(\text{dppf})\cdot\text{CH}_2\text{Cl}_2$ (18.6 mg, 22.8 μmol), Cs_2CO_3 (119 mg, 364 μmol) and Ph_3As (14.0 mg, 45.6 μmol) at room temperature. The reaction mixture was stirred at room temperature for 24 h and filtrated through a pad of Celite with Et_2O . The filtrate was concentrated. The residue was purified with flash column chromatography (hexane/AcOEt = 3/1 to 1.5/1) and HPLC (column: Inertsil ODS-3 $\phi 10 \times 250$ mm, flow rate: 2 mL/min, detection: UV 210 nm, eluent: MeOH/ H_2O = 4/1, T_R = 25.2 min) to give 35.7 mg of **1** (78%): colorless solid; $[\alpha]_D^{26.7} = -163^\circ$ (c 0.55, MeOH); IR (film) ν 3282, 2962, 1735, 1644, 1556, 1457, 1262, 1166, 960, 756 cm^{-1} ; ^1H NMR (CDCl_3 , 500 MHz) δ 0.86 (d, J = 6.9 Hz, 3H, H15'), 0.87 (d, J = 6.9 Hz, 3H, H13), 0.97 (d, J = 6.9 Hz, 3H, H14'), 1.12 (s, 9H, H11, H16, H17), 1.42 (d, J = 6.9 Hz, 3H, H11'), 1.55 (s, 3H, H14), 1.79 (s, 3H, H15), 2.17 (dq, J = 11.5, 6.9 Hz, 1H, H4), 2.43 (dq, J = 10.9, 6.9, 6.9 Hz, 1H, H13'), 2.81 (d, J = 13.2 Hz, 1H, H2), 2.86 (s, 3H, H12'), 2.99 (d, J = 13.2 Hz, 1H, H2), 3.48 (d, J = 18.4 Hz, 1H, H8'), 4.25 (d, J = 10.9 Hz, 1H, H5'), 4.69 (dd, J = 18.4, 10.3 Hz, 1H, H8'), 5.01 (s, 1H, H12), 5.05 (s, 1H, H12), 5.17 (d, J = 11.5 Hz, 1H, H5), 5.29 (s, 1H, H9), 5.34 (dq, J = 9.2, 6.9 Hz, 1H, H2'), 5.93 (s, 1H, H7), 6.57 (d, J = 9.2 Hz, 1H, H1'), 7.96 (d, J = 10.3 Hz, 1H, H7'); ^{13}C NMR (125 MHz, CDCl_3) δ 12.4, 17.8, 18.5, 18.6, 18.9, 19.3, 26.1, 28.8, 30.8, 32.6, 38.9, 41.1, 43.0, 46.6, 67.1, 83.4, 113.8, 129.1, 130.4, 137.3, 141.5, 144.7, 167.6, 167.8, 171.0, 173.0; HRMS (ESI-TOF) calcd for $\text{C}_{28}\text{H}_{45}\text{N}_3\text{O}_5\text{Na}$ 526.3251 ($\text{M}+\text{Na}^+$), found 526.3249.



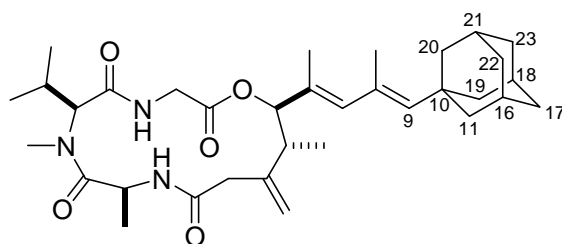
9-Methyl-analogue 5a. **5a** was obtained in 75% yield by following the procedure of **1a**: Colorless solid; $[\alpha]_D^{25.2} = -201^\circ$ (c 0.52, MeOH); IR (film) ν 3281, 2968, 1737, 1641, 1550, 1444, 1267, 960, 756 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 0.86 (3H, d, J = 7.4 Hz, H15'), 0.87 (3H, d, J = 7.5 Hz, H13), 0.97 (3H, d, J = 6.9 Hz, H14'), 1.41 (3H, d, J = 6.9 Hz, H11'), 1.58 (3H, s, H14), 1.65 (3H, d, J = 6.9 Hz, H10), 1.78 (3H, s, H15), 2.18 (1H, dq, J = 11.5, 7.5 Hz, H4), 2.43 (1H, dq, J = 10.9, 7.4, 6.9 Hz, H13'), 2.81 (1H, d, J = 13.2 Hz, H2), 2.85 (3H, s, H12'), 2.98 (1H, d, J = 13.2 Hz, H2), 3.48 (1H, dd, J = 18.3, 1.7 Hz, H8'), 4.24 (1H, d, J = 10.9 Hz, H5'), 4.67 (1H, dd, J = 18.3, 10.3 Hz, H8'), 5.01, (1H, s, H12'), 5.05 (1H, s, H12'), 5.18 (1H, d, J = 11.5 Hz, H5), 5.34 (1H, dq, J = 9.2, 6.9 Hz, H12'), 5.42 (1H, q, J = 6.9 Hz, H9), 5.97 (1H, s, H7), 6.62 (1H, d, J = 9.2 Hz, H1'), 7.95 (1H, dd, J = 10.3, 1.7 Hz, H7'); ^{13}C NMR (125 MHz, CDCl_3) δ 12.6, 13.6, 16.3, 18.5, 18.6, 18.8, 19.3, 26.1, 28.7, 38.9, 41.1, 43.0, 46.6, 67.1, 83.6, 113.8, 126.1, 129.2, 132.6, 135.5, 144.7, 167.6, 167.8, 171.0, 173.1; HRMS (ESI-TOF) calcd for $\text{C}_{25}\text{H}_{39}\text{N}_3\text{O}_5\text{Na}$ 484.2782 ($\text{M}+\text{Na}^+$), found 484.2781.



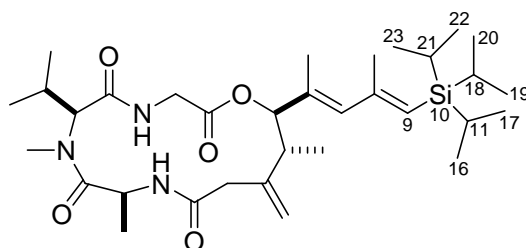
9-Isopropyl-analogue 5b. **5b** was obtained in 86% yield by following the procedure of **1a**: Colorless solid; $[\alpha]_D^{25.3} = -178^\circ$ (*c* 0.73, MeOH); IR (film) ν 3282, 2963, 1736, 1641, 1552, 1459, 1268, 757 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 0.86 (3H, d, $J = 6.9$ Hz, H15'), 0.88 (3H, d, $J = 7.4$ Hz, H13), 0.94 (3H, d, $J = 5.1$ Hz, H16), 0.96 (3H, d, $J = 5.1$ Hz, H11), 0.97 (3H, d, $J = 6.3$ Hz, H14'), 1.41 (3H, d, $J = 6.9$ Hz, H11'), 1.57 (3H, s, H14), 1.71 (3H, s, H15), 2.18 (1H, dq, $J = 10.9, 7.4$ Hz, H4), 2.43 (1H, dq, $J = 10.9, 6.9, 6.3$ Hz, H13'), 2.54 (1H, dq, $J = 9.7, 5.1, 5.1$ Hz, H10), 2.82 (1H, d, $J = 13.2$ Hz, H2), 2.85 (3H, s, H12'), 2.98 (1H, d, $J = 13.2$ Hz, H2), 3.48 (1H, dd, $J = 18.3, 1.7$ Hz, H8'), 4.25 (1H, d, $J = 10.9$ Hz, H5'), 4.68 (1H, dd, $J = 18.3, 10.4$ Hz, H8'), 5.00 (1H, s, H12'), 5.05 (1H, s, H12'), 5.14 (1H, d, $J = 9.7$ Hz, H9), 5.18 (1H, d, $J = 10.9$ Hz, H5), 5.34 (1H, dq, $J = 9.1, 6.9$ Hz, H12'), 5.94 (1H, s, H7), 6.70 (1H, d, $J = 9.1$ Hz, H1'), 7.96 (1H, dd, $J = 10.4, 1.7$ Hz, H7'); ^{13}C NMR (125 MHz, CDCl_3) δ 12.5, 16.6, 18.5, 18.6, 18.8, 19.3, 22.80, 22.83, 26.1, 27.3, 28.8, 38.9, 41.1, 43.0, 46.5, 67.1, 83.6, 113.7, 129.2, 129.4, 135.6, 139.5, 144.8, 167.6, 167.8, 171.0, 173.1; HRMS (ESI-TOF) calcd for $\text{C}_{27}\text{H}_{43}\text{N}_3\text{O}_5\text{Na}$ 512.3095 ($\text{M}+\text{Na}^+$), found 512.3092.



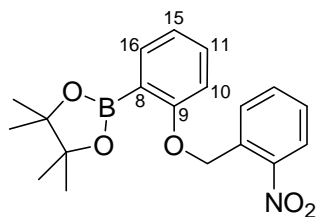
9-Cyclohexyl-analogue 5c. **5c** was obtained in 80% yield by following the procedure of **1a**: Colorless solid; $[\alpha]_D^{25.2} = -165^\circ$ (*c* 0.53, MeOH); IR (film) ν 3283, 2926, 1736, 1643, 1552, 1448, 1267, 756 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 0.86 (3H, d, $J = 6.8$ Hz, H15'), 0.88 (3H, d, $J = 6.8$ Hz, H13), 0.97 (3H, d, $J = 6.9$ Hz, H14'), 1.05 (2H, m, H11ax, H19ax), 1.18 (1H, tt, $J = 12.6, 3.4$ Hz, H17ax), 1.2.31 (2H, m, H16ax, H18ax), 1.41 (3H, d, $J = 6.9$ Hz, H11'), 1.57 (3H, s, H14), 1.60-1.63 (3H, m, H11eq, H17eq, H19eq), 1.69 (2H, m, H16eq, H18eq), 1.71 (3H, s, H15), 2.18 (1H, dq, $J = 11.5, 6.8$ Hz, H4), 2.19 (1H, m, H10), 2.43 (1H, dq, $J = 10.9, 6.9, 6.8$ Hz, H13'), 2.82 (1H, d, $J = 13.2$ Hz, H2), 2.85 (3H, s, H12'), 2.98 (1H, d, $J = 13.2$ Hz, H2), 3.48 (1H, dd, $J = 18.3, 1.7$ Hz, H8'), 4.25 (1H, d, $J = 10.9$ Hz, H5'), 4.68 (1H, dd, $J = 18.3, 10.3$ Hz, H8'), 5.00 (1H, s, H12), 5.05 (1H, s, H12), 5.16 (1H, d, $J = 9.1$ Hz, H9), 5.18 (1H, d, $J = 11.5$ Hz, H5), 5.34 (1H, dq, $J = 9.1, 6.9$ Hz, H2'), 5.95 (1H, s, H7), 6.70 (1H, d, $J = 9.1$ Hz, H1'), 7.95 (1H, dd, $J = 10.3, 1.7$ Hz, H7'); ^{13}C NMR (125 MHz, CDCl_3) δ 12.6, 16.7, 18.5, 18.6, 18.9, 19.3, 25.93, 25.94, 26.05, 26.09, 28.8, 32.90, 32.96, 37.2, 38.9, 41.1, 43.0, 46.6, 67.1, 83.6, 113.7, 129.3, 129.6, 135.7, 138.1, 144.8, 167.6, 167.8, 171.0, 173.1; HRMS (ESI-TOF) calcd for $\text{C}_{30}\text{H}_{47}\text{N}_3\text{O}_5\text{Na}$ 552.3408 ($\text{M}+\text{Na}^+$), found 552.3400.



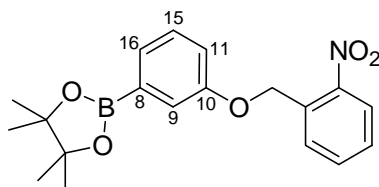
9-(1-Adamantyl)-analogue 5d. **5d** was obtained in 80% yield by following the procedure of **1a**: Colorless solid; $[\alpha]_D^{24.7} = -147^\circ$ (*c* 0.55, MeOH); IR (film) ν 3282, 2904, 1736, 1643, 1552, 1450, 1269, 756 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 0.86 (3H, d, $J = 6.3$ Hz, H15'), 0.87 (3H, d, $J = 7.5$ Hz, H13), 0.97 (3H, $J = 6.9$ Hz, H14'), 1.42 (3H, d, $J = 6.8$ Hz, H11'), 1.55 (3H, s, H14), 1.68 (6H, m, H17, H22, H23), 1.77 (6H, d, $J = 2.9$ Hz, H11, H19, H20), 1.80 (3H, s, H15), 1.95 (3H, m, H16, H18, H21), 2.17 (1H, dq, $J = 11.5, 7.5$ Hz, H4), 2.43 (1H, dq, $J = 11.5, 6.9, 6.3$ Hz, H13'), 2.81 (1H, d, $J = 13.2$ Hz, H2), 2.86 (3H, s, H12'), 2.98 (1H, d, $J = 13.2$ Hz, H2), 3.48 (1H, dd, $J = 18.9, 1.7$ Hz, H8'), 4.24 (1H, d, $J = 11.5$ Hz, H5'), 4.68 (1H, dd, $J = 18.9, 10.3$ Hz, H8'), 5.00 (1H, s, H12), 5.02 (1H, s, H9), 5.05 (1H, s, H12), 5.16 (1H, d, $J = 11.5$ Hz, H5), 5.34 (1H, dq, $J = 9.1, 6.8$ Hz, H2'), 5.92 (1H, s, H7), 6.58 (1H, d, $J = 9.1$ Hz, H1'), 7.94 (1H, dd, $J = 10.3, 1.7$ Hz, H7'); ^{13}C NMR (125 MHz, CDCl_3) δ 12.5, 18.2, 18.55, 18.63, 18.9, 19.3, 26.1, 28.8, 35.2, 36.9, 39.0, 41.1, 42.6, 43.0, 46.6, 67.1, 83.4, 113.7, 129.1, 130.1, 137.6, 141.5, 144.7, 167.6, 167.8, 171.0, 173.0; HRMS (ESI-TOF) calcd for $\text{C}_{34}\text{H}_{51}\text{N}_3\text{O}_5\text{Na}$ 604.3721 ($\text{M}+\text{Na}^+$), found 604.3721.



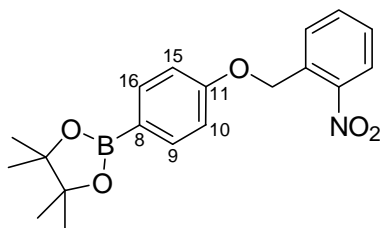
9-Triisopropylsilyl-analogue 5e. **5e** was obtained in 78% yield by following the procedure of **1a**: Colorless solid; $[\alpha]_D^{25.2} = -140^\circ$ (*c* 0.59, MeOH); IR (film) ν 3282, 2963, 2865, 1738, 1645, 1552, 1461, 1264, 884 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 0.86 (3H, d, $J = 6.9$ Hz, H15'), 0.89 (3H, d, $J = 6.9$ Hz, H13), 0.97 (3H, d, $J = 6.3$ Hz, H14'), 1.04 (18H, d, $J = 7.4$ Hz, H16, H17, H19, H20, H22, H23), 1.13 (3H, qq, $J = 7.4, 7.4$ Hz, H11, H18, H21), 1.42 (3H, d, $J = 6.9$ Hz, H11'), 1.59 (3H, s, H14), 1.84 (3H, s, H15), 2.19 (1H, dq, $J = 11.5, 6.9$ Hz, H4), 2.43 (1H, dq, $J = 10.9, 6.9, 6.3$ Hz, H13'), 2.81 (1H, d, $J = 13.2$ Hz, H2), 2.85 (3H, s, H12'), 2.99 (1H, d, $J = 13.2$ Hz, H2), 3.49 (1H, dd, $J = 18.9, 1.7$ Hz, H8'), 4.25 (1H, d, $J = 10.9$ Hz, H5'), 4.71 (1H, dd, $J = 18.3, 10.3$ Hz, H8'), 5.01, (1H, s, H12), 5.06 (1H, s, H12), 5.20 (1H, d, $J = 11.5$ Hz, H5), 5.24 (1H, s, H9), 5.34 (1H, dq, $J = 9.1, 6.9$ Hz, H2'), 6.08 (1H, s, H7), 6.60 (1H, d, $J = 9.1$ Hz, H1'), 7.97 (1H, dd, $J = 10.3, 1.7$ Hz, H7'); ^{13}C NMR (125 MHz, CDCl_3) δ 12.2, 12.7, 18.5, 18.6, 18.9, 19.0, 19.3, 23.5, 26.1, 28.7, 39.1, 41.1, 43.1, 46.5, 67.1, 83.0, 113.8, 125.5, 130.6, 137.4, 144.6, 150.4, 167.6, 167.9, 171.0, 173.1; HRMS (ESI-TOF) calcd for $\text{C}_{33}\text{H}_{57}\text{N}_3\text{O}_5\text{Na}$ 626.3960 ($\text{M}+\text{Na}^+$), found 626.3961.



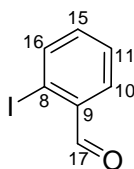
***o*-(*o*-Nitrobenzyloxy)phenylboronic ester 6a.** To a solution of 2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenol (110 mg, 500 μmol) in dry DMF (2.5 mL) were added *o*-nitrobenzylbromide **5-1** (97.2 mg, 450 μmol) and K_2CO_3 (76 mg, 550 μmol) at 0 $^\circ\text{C}$. The reaction mixture was stirred in the dark at room temperature for 4 h and at 45 $^\circ\text{C}$ for another 3 h. The reaction was quenched with H_2O . The aqueous layer was extracted six times with a mixture of hexane/ Et_2O = 2/1. The combined organic layer was washed with H_2O and brine, dried over Na_2SO_4 , filtrated and concentrated. The residue was purified with flash column chromatography on silica gel (hexane/ AcOEt = 25) to give 64.0 mg of **6a** (34%): colorless solid; IR (film) ν 2976, 1604, 1525, 1450, 1353, 1251, 1147, 1075, 860, 728 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 1.41 (12H, s, pinacol), 5.50 (2H, s, $\text{OCH}_2\text{C}_6\text{H}_4\text{NO}_2$), 7.02 (1H, t, J = 7.4 Hz, H15), 7.05 (1H, dd, J = 7.4, 1.7 Hz, H10), 7.47 (1H, td, J = 7.4, 1.7 Hz, H11), 7.49 (1H, td, J = 8.0, 1.1 Hz, $\text{OCH}_2\text{C}_6\text{H}_4\text{NO}_2$), 7.73 (1H, td, J = 8.0, 1.1 Hz, $\text{OCH}_2\text{C}_6\text{H}_4\text{NO}_2$), 7.79 (1H, dd, J = 7.4, 1.7 Hz, H16), 8.20 (1H, dd, J = 8.0, 1.1 Hz, $\text{OCH}_2\text{C}_6\text{H}_4\text{NO}_2$), 8.71 (1H, dd, J = 8.0, 1.1 Hz, $\text{OCH}_2\text{C}_6\text{H}_4\text{NO}_2$); ^{13}C NMR (125 MHz, CDCl_3) δ 25.0, 66.8, 83.4, 111.5, 120.9, 124.6, 127.8, 129.4, 133.0, 133.9, 134.7, 137.2, 146.2, 162.6; HRMS (ESI-TOF) calcd for $\text{C}_{20}\text{H}_{25}\text{BNO}_6$ 386.1780 ($\text{M}+\text{OCH}_3^-$), found 386.1780.



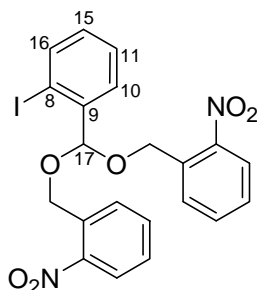
***m*-(*o*-Nitrobenzyloxy)phenylboronic ester 6b.** **6b** was obtained in 82% yield by following the procedure of **6a**. Colorless solid; IR (film) ν 2979, 1526, 1429, 1355, 1223, 1145, 855, 788 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 1.36 (12H, s, pinacol), 5.51 (2H, s, $\text{OCH}_2\text{C}_6\text{H}_4\text{NO}_2$), 7.09 (1H, ddd, J = 8.6, 2.9, 1.1 Hz, H11), 7.33 (1H, t, J = 8.6 Hz, H15), 7.46 (1H, m, H16), 7.47 (1H, m, H9), 7.48 (1H, t, J = 8.0 Hz, $\text{OCH}_2\text{C}_6\text{H}_4\text{NO}_2$), 7.68 (1H, td, J = 8.0, 1.1 Hz, $\text{OCH}_2\text{C}_6\text{H}_4\text{NO}_2$), 7.94 (1H, d, J = 8.0 Hz, $\text{OCH}_2\text{C}_6\text{H}_4\text{NO}_2$), 8.16 (1H, dd, J = 8.0, 1.1 Hz, $\text{OCH}_2\text{C}_6\text{H}_4\text{NO}_2$); ^{13}C NMR (125 MHz, CDCl_3) δ 24.8, 66.6, 83.9, 117.9, 120.5, 124.9, 127.9, 128.2, 128.6, 129.1, 133.9, 134.0, 146.9, 157.6; HRMS (ESI-TOF) calcd for $\text{C}_{20}\text{H}_{25}\text{BNO}_6$ 386.1780 ($\text{M}+\text{OCH}_3^-$), found 386.1785.



***p*-(*o*-Nitrobenzyloxy)phenylboronic ester 6c.** **6c** was obtained in 77% yield by following the procedure of **6a**. Colorless solid; IR (film) ν 2978, 1605, 1528, 1361, 1242, 1144, 1093, 1033, 860, 729 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 1.34 (12H, s, pinacol), 5.52 (2H, s, $\text{OCH}_2\text{C}_6\text{H}_4\text{NO}_2$), 6.99 (2H, dt, $J = 9.1, 2.3$ Hz, H9, H16), 7.49 (1H, t, $J = 8.0$ Hz, $\text{OCH}_2\text{C}_6\text{H}_4\text{NO}_2$), 7.67 (1H, td, $J = 8.0, 1.1$ Hz, $\text{OCH}_2\text{C}_6\text{H}_4\text{NO}_2$), 7.78 (2H, dt, $J = 9.1, 2.3$ Hz, H10, H15), 7.88 (1H, d, $J = 8.0$ Hz, $\text{OCH}_2\text{C}_6\text{H}_4\text{NO}_2$), 8.17 (1H, dd, $J = 8.0, 1.1$ Hz, $\text{OCH}_2\text{C}_6\text{H}_4\text{NO}_2$); ^{13}C NMR (125 MHz, CDCl_3) δ 24.8, 66.4, 83.6, 114.1, 125.0, 128.3, 128.4, 133.7, 134.0, 136.6, 146.8, 160.5; HRMS (ESI-TOF) calcd for $\text{C}_{20}\text{H}_{25}\text{BNO}_6$ 386.1780 ($\text{M}+\text{OCH}_3^-$), found 386.1770.

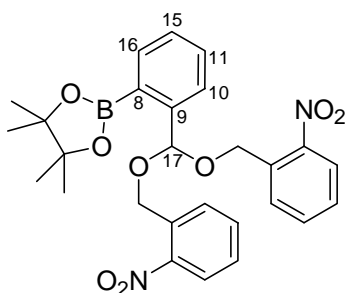


***o*-Iodobenzaldehyde 9.** To a solution of *o*-iodobenzylalcohol (1.0 g, 4.27 mmol) in dry MeCN (4.3 mL) were added MS4Å (500 mg), NMO (551 mg, 4.70 mmol) and TPAP (75 mg, 2.14 μmol) at 0 $^\circ\text{C}$. The reaction mixture was stirred at room temperature for 1.5 h and filtrated through short flash column chromatography on silica gel with AcOEt. The filtrate was concentrated. The residue was purified with flash column chromatography on silica gel (hexane/AcOEt = 50/1) to give 804 mg of **9** (81%, a mixture of aldehyde and hydrate): colorless oil; IR (film) ν 2853, 1696, 1580, 1438, 1262, 1200, 1015, 822, 754 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 7.26 (1H, td, $J = 8.0, 2.3$ Hz, H11), 7.44 (1H, td, $J = 8.0, 1.1$ Hz, H16), 7.86 (1H, dd, $J = 8.0, 2.3$ Hz, H16), 7.93 (1H, dd, $J = 8.0, 1.1$ Hz, H15), 10.0 (1H, s, H17); ^{13}C NMR (125 MHz, CDCl_3) δ 100.7, 128.6, 130.2, 135.0, 135.4, 140.5, 195.7; HRMS (ESI-TOF) calcd for $\text{C}_{14}\text{H}_{10}\text{I}_2\text{O}_2\text{Na}$ 486.8668 ($2\text{M}+\text{Na}^+$), found 486.8661.

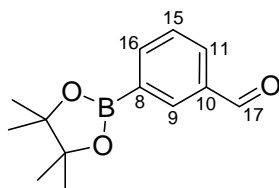


***o*-[Bis(*o*-nitrobenzyloxy)methyl]iodobenzene 10.** To a solution of **9** (154 mg, 665 μmol) in dry benzene (6.7 mL) were added *o*-nitrobenzylalcohol (204 mg, 1.33 mmol) and TsOH \cdot H $_2$ O (6.3 mg, 33.3 μmol) at room temperature. The reaction mixture was stirred at 95 $^\circ\text{C}$ for 20 h using Dean-Stark trap in the dark. TsOH was removed from the mixture using a pad of Florisil with a mixture of hexane/AcOEt = 5/1. The filtrate was

concentrated. The residue was purified with flash column chromatography on Florisil (hexane/AcOEt = 40/1) to give 189 mg of **10** (55%): colorless solid; IR (film) ν 2921, 1613, 1519, 1337, 1202, 1051, 856, 790, 729, 682 cm^{-1} ; ^1H NMR (500 MHz, C_6D_6) δ 4.90 (2H, d, $J = 14.9$ Hz, $\text{OCH}_2\text{C}_6\text{H}_4\text{NO}_2$), 4.96 (2H, d, $J = 14.9$ Hz, $\text{OCH}_2\text{C}_6\text{H}_4\text{NO}_2$), 5.92 (1H, s, H17), 6.55 (1H, td, $J = 8.0, 1.7$ Hz, H15), 6.66 (2H, t, $J = 8.0$ Hz, $\text{OCH}_2\text{C}_6\text{H}_4\text{NO}_2$), 6.96 (2H, t, $J = 8.0$ Hz, $\text{OCH}_2\text{C}_6\text{H}_4\text{NO}_2$), 7.01 (1H, t, $J = 8.0$ Hz, H11), 7.61-7.63 (5H, m, H16, $\text{OCH}_2\text{C}_6\text{H}_4\text{NO}_2$), 7.73 (1H, dd, $J = 8.0, 1.7$ Hz, H10); ^{13}C NMR (125 MHz, C_6D_6) δ 65.4, 98.0, 105.6, 124.6, 128.3, 128.6, 128.8, 130.9, 133.3, 134.4, 139.6, 140.1, 147.4; HRMS (ESI-TOF) calcd for $\text{C}_{21}\text{H}_{17}\text{N}_2\text{O}_6\text{Na}$ 543.0024 ($\text{M}+\text{Na}^+$), found 543.0012.

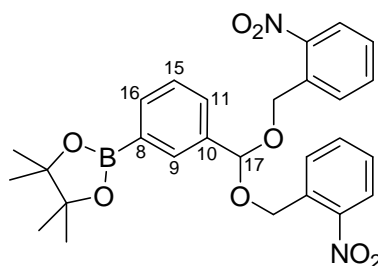


***o*-[Bis(*o*-nitrobenzyloxy)methyl]phenylboronic ester **6d**.** To a solution of **10** (96.8 mg, 186 μmol) in dry DMF (930 μL) were added bispinacolato diboron (87 mg, 343 μmol), $\text{Pd}(\text{OAc})_2$ (4.2 mg, 18.6 μmol) and KOAc (65 mg, 662 μmol) at room temperature. The reaction mixture was stirred at 100 $^\circ\text{C}$ for 12 h in the dark. The reaction was quenched with 0.2 M aqueous phosphate buffer (pH 6.9) at 0 $^\circ\text{C}$. The aqueous layer was extracted five times with a mixture of hexane/AcOEt = 4/1. The combined organic layer was washed with H_2O and brine, dried over Na_2SO_4 , filtrated and concentrated. The residue was purified with flash column chromatography on Florisil (hexane/toluene = 4/1 and hexane/*i*-PrOH = 100) to give 54.7 mg of **6d** (42%, a mixture of **6d/10** = 3/1): colorless oil; IR (film) ν 2979, 1695, 1525, 1346, 1145, 1036, 858, 729 cm^{-1} ; ^1H NMR (500 MHz, C_6D_6) δ 1.08 (12H, s, pinacol), 5.10 (2H, $J = 15.5$ Hz, $\text{OCH}_2\text{C}_6\text{H}_4\text{NO}_2$), 5.19 (2H, $J = 15.5$ Hz, $\text{OCH}_2\text{C}_6\text{H}_4\text{NO}_2$), 6.67 (2H, m, $\text{OCH}_2\text{C}_6\text{H}_4\text{NO}_2$), 6.75 (1H, s, H17), 6.95 (2H, m, $\text{OCH}_2\text{C}_6\text{H}_4\text{NO}_2$), 7.08-7.19 (2H, m, $\text{BC}_6\text{H}_4\text{CH}$), 7.64 (2H, m, $\text{OCH}_2\text{C}_6\text{H}_4\text{NO}_2$), 7.69 (2H, d, $J = 8.0$ Hz, $\text{OCH}_2\text{C}_6\text{H}_4\text{NO}_2$), 7.95 (1H, m, $\text{BC}_6\text{H}_4\text{CH}$), 8.12 (1H, d, $J = 7.4$ Hz, $\text{BC}_6\text{H}_4\text{CH}$); ^{13}C NMR (125 MHz, C_6D_6) δ 24.7, 66.0, 83.9, 102.4, 124.5, 126.2, 127.6, 128.4, 128.5, 128.8, 129.0, 131.0, 131.3, 133.2, 133.29, 133.30, 135.2, 136.0, 147.5; HRMS (ESI-TOF) calcd for $\text{C}_{28}\text{H}_{32}\text{BN}_2\text{O}_9$ 551.2206 ($\text{M}+\text{OCH}_3^-$), found 551.2197.

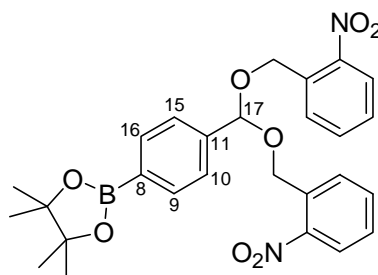


***m*-Formylphenylboronic ester **11e**.** To a solution of *m*-iodobenzaldehyde **11e** (348 mg, 1.50 mmol) in dry DMF (5 mL) were added bispinacolato diboron (381 mg, 1.50 mmol), $\text{Pd}(\text{OAc})_2$ (33.7 mg, 150 μmol) and KOAc (294 mg, 3.00 mmol) at room temperature. The reaction mixture was stirred at 80 $^\circ\text{C}$ for 5 h. The reaction was

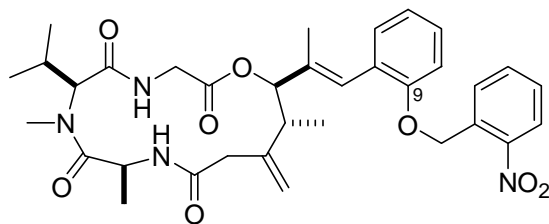
quenched with H₂O at 0 °C. The aqueous layer was acidified with saturated aqueous NH₄Cl and extracted twice with a mixture of hexane/AcOEt = 4/1. The combined organic layer was washed with H₂O and brine, dried over Na₂SO₄, filtrated and concentrated. The residue was purified with flash column chromatography on silica gel (hexane/AcOEt = 50/1 to 20/1) to give 244 mg of **11e** (70%): colorless solid; IR (film) ν 2979, 1699, 1602, 1359, 1196, 1143, 851, 701 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 1.35 (12H, s, pinacol), 7.52 (1H, dd, *J* = 8.0, 7.4 Hz, H15), 7.97 (1H, d, *J* = 8.0 Hz, H11), 8.05 (1H, d, *J* = 7.4 Hz, H16), 8.30 (1H, s, H9), 10.0 (1H, s, H17); ¹³C NMR (125 MHz, CDCl₃) δ 24.8, 84.3, 128.4, 131.3, 135.6, 137.2, 140.7, 192.8; HRMS (ESI-TOF) calcd for C₁₄H₂₀BO₄ 263.1460 (M+OCH₃⁻), found 263.1457.



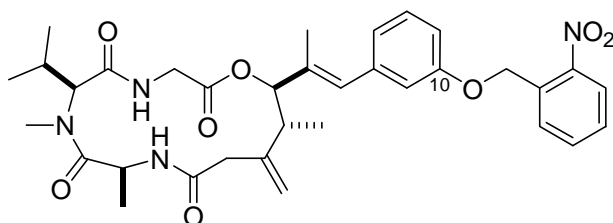
***m*-[Bis(*o*-nitrobenzyloxy)methyl]phenylboronic ester 6e.** A mixture of **6e/11e** = 4/1 was obtained in 31% yield by following the procedure of **10**. Colorless oil; IR (film) ν 2979, 1704, 1609, 1530, 1361, 1202, 1144, 1041, 855, 790 cm⁻¹; ¹H NMR (500 MHz, C₆D₆) δ 1.12 (12H, s, pinacol), 4.84 (2H, d, *J* = 15.5 Hz, OCH₂C₆H₄NO₂), 4.93 (2H, d, *J* = 15.5 Hz, OCH₂C₆H₄NO₂), 5.59 (1H, s, H17), 6.70 (2H, td, *J* = 8.0, 1.1 Hz, OCH₂C₆H₄NO₂), 6.95 (2H, td, *J* = 8.0, 1.1 Hz, OCH₂C₆H₄NO₂), 7.27 (1H, t, *J* = 8.0 Hz, H15), 7.57 (2H, dd, *J* = 8.0, 1.1 Hz, OCH₂C₆H₄NO₂), 7.62 (2H, dd, *J* = 8.0, 1.1 Hz, OCH₂C₆H₄NO₂), 7.68 (1H, d, *J* = 8.0 Hz, H11), 8.10 (1H, d, *J* = 8.0 Hz, H16), 8.41 (1H, s, H9); ¹³C NMR (125 MHz, C₆D₆) δ 24.9, 64.8, 83.9, 102.8, 124.5, 127.7, 128.8, 130.0, 133.3, 133.8, 134.8, 136.1, 137.5, 147.4, 191.5; HRMS (ESI-TOF) calcd for C₂₈H₃₂BN₂O₉ 551.2206 (M+OCH₃⁻), found 551.2214.



***p*-[Bis(*o*-nitrobenzyloxy)methyl]phenylboronic ester 6f.** **6f** was obtained in 32% yield by following the procedure of **10**. Colorless solid; IR (film) ν 2979, 1614, 1525, 1361, 1209, 1144, 1090, 1042, 858, 729 cm⁻¹; ¹H NMR (500 MHz, C₆D₆) δ 1.13 (12H, s, pinacol), 4.85 (2H, d, *J* = 15.5 Hz, OCH₂C₆H₄NO₂), 4.93 (2H, d, *J* = 15.5 Hz, OCH₂C₆H₄NO₂), 5.56 (1H, s, H17), 6.69 (2H, m, OCH₂C₆H₄NO₂), 6.96 (2H, m, OCH₂C₆H₄NO₂), 7.56 (2H, dd, *J* = 8.0, 1.1 Hz, BC₆H₄CH), 7.62 (4H, d, *J* = 8.0 Hz, BC₆H₄CH, OCH₂C₆H₄NO₂), 8.20 (2H, m, OCH₂C₆H₄NO₂); ¹³C NMR (125 MHz, C₆D₆) δ 24.9, 64.7, 83.9, 102.4, 124.6, 126.5, 128.5, 128.7, 133.3, 134.7, 135.6, 141.0, 147.5; HRMS (ESI-TOF) calcd for C₂₈H₃₂BN₂O₉ 551.2206 (M+OCH₃⁻), found 551.2217.

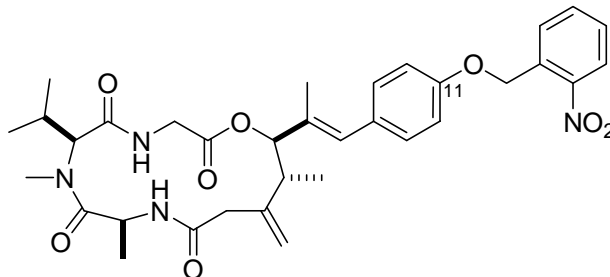


***o*-(*o*-Nitrobenzyloxy)-styrene-analogue 2a.** **2a** was obtained in 60% yield by following the procedure of **1a**: Colorless solid; $[\alpha]_D^{26.8} = -152^\circ$ (c 0.47, MeOH); IR (film) ν 3277, 2967, 1734, 1644, 1525, 1454, 1245, 753 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 0.86 (3H, d, $J = 6.9$ Hz, H15'), 0.97 (3H, d, $J = 6.9$ Hz, H14'), 0.98 (3H, d, $J = 6.9$ Hz, H13) 1.43 (3H, d, $J = 6.9$ Hz, H11'), 1.66 (3H, d, $J = 1.2$ Hz, H14), 2.30 (1H, dq, $J = 10.9, 6.9$ Hz, H4), 2.42 (1H, dq, $J = 11.5, 6.9, 6.9$ Hz, H13'), 2.83 (3H, s, H12'), 2.86 (1H, d, $J = 13.7$ Hz, H2), 3.03 (1H, d, $J = 13.7$ Hz, H2), 3.54 (1H, dd, $J = 18.3, 1.7$ Hz, H8'), 4.25 (1H, d, $J = 11.5$ Hz, H5'), 4.70 (1H, dd, $J = 18.3, 10.3$ Hz, H8'), 5.10 (1H, s, H12), 5.12 (1H, s, H12), 5.36 (1H, dq, $J = 9.1, 6.9$ Hz, H2'), 5.45 (1H, d, $J = 15.5$ Hz, OCH2Ar), 5.48 (1H, d, $J = 10.9$ Hz, H5), 5.49 (1H, d, $J = 15.5$ Hz, OCH2C6H4NO2), 6.62 (1H, d, $J = 9.1$ Hz, H1'), 6.85 (1H, d, $J = 1.2$ Hz, H7), 6.97 (1H, t, $J = 7.4$ Hz, H15), 6.98 (1H, d, $J = 7.4$ Hz, H10), 7.20 (1H, d, $J = 7.4$ Hz, H16), 7.25 (1H, t, $J = 7.4$ Hz, H11), 7.48 (1H, t, $J = 8.0$ Hz, OCH2C6H4NO2), 7.79 (1H, td, $J = 8.0, 1.1$ Hz, OCH2C6H4NO2), 8.01 (1H, d, $J = 10.3, 1.7$ Hz, H7'), 8.04 (1H, d, $J = 8.0$ Hz, OCH2C6H4NO2), 8.18 (1H, dd, $J = 8.0, 1.1$ Hz, OCH2C6H4NO2); ^{13}C NMR (125 MHz, CDCl_3) δ 12.7, 18.57, 18.62, 18.9, 19.3, 26.1, 28.7, 38.8, 41.1, 43.1, 46.6, 66.7, 67.1, 83.0, 111.9, 113.9, 120.7, 124.7, 126.0, 127.6, 128.0, 128.7, 128.8, 130.3, 133.3, 134.1, 134.6, 144.7, 146.4, 155.7, 167.6, 167.8, 171.0, 173.1; HRMS (ESI-TOF) calcd for $\text{C}_{34}\text{H}_{42}\text{N}_4\text{O}_8\text{Na}$ 657.2895 ($\text{M}+\text{Na}^+$), found 657.2898.



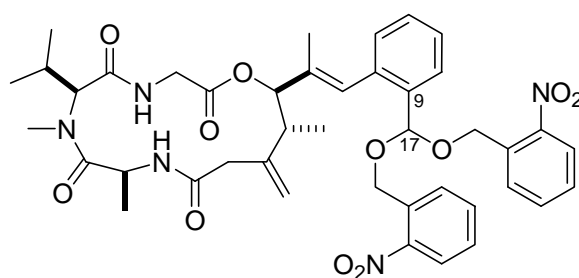
***m*-(*o*-Nitrobenzyloxy)-styrene-analogue 2b.** **2b** was obtained in 83% yield by following the procedure of **1a**: Colorless solid; $[\alpha]_D^{26.1} = -125^\circ$ (c 0.66, MeOH); IR (film) ν 3280, 2967, 1734, 1644, 1526, 1341, 1262, 757 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 0.86 (3H, d, $J = 6.9$ Hz, H15'), 0.94 (3H, d, $J = 7.5$ Hz, H13), 0.97 (3H, d, $J = 6.3$ Hz, H14'), 1.43 (3H, d, $J = 6.9$ Hz, H11'), 1.70 (3H, d, $J = 1.2$ Hz, H14), 2.26 (1H, dq, $J = 11.4, 7.5$ Hz, H4), 2.42 (1H, dq, $J = 11.5, 6.9, 6.3$ Hz, H13'), 2.83 (3H, s, H12'), 2.85 (1H, d, $J = 13.2$ Hz, H2), 3.01 (1H, d, $J = 13.2$ Hz, H2), 3.51 (1H, dd, $J = 18.3, 1.7$ Hz, H8'), 4.24 (1H, d, $J = 11.5$ Hz, H5'), 4.70 (1H, dd, $J = 18.3, 10.3$ Hz, H8'), 5.06 (1H, s, H12), 5.09 (1H, s, H12), 5.33 (1H, d, $J = 11.4$ Hz, H5), 5.36 (1H, dq, $J = 9.7, 6.9$ Hz, H2'), 5.49 (2H, s, OCH2C6H4NO2), 6.56 (1H, d, $J = 1.2$ Hz, H7), 6.67 (1H, d, $J = 9.7$ Hz, H1'), 6.85 (1H, dd, $J = 8.0, 1.7$ Hz, H11), 6.90 (1H, d, $J = 1.7$ Hz, H9), 6.91 (1H, d, $J = 8.0$ Hz, H16), 7.25 (1H, t, $J = 8.0$ Hz, H15), 7.48 (1H, t, $J = 8.0$ Hz, OCH2C6H4NO2), 7.68 (1H, td, $J = 8.0, 1.1$ Hz, OCH2C6H4NO2), 7.88 (1H, d, $J = 8.0$ Hz, OCH2C6H4NO2), 7.99 (1H, dd, $J = 10.3, 1.7$ Hz, H7'), 8.16 (1H, dd, $J = 8.0, 1.1$ Hz, OCH2C6H4NO2); ^{13}C

NMR (125 MHz, CDCl₃) δ 13.0, 18.56, 18.63, 18.8, 19.3, 26.1, 28.7, 39.1, 41.1, 43.1, 46.6, 66.8, 67.1, 82.9, 113.3, 134.0, 115.9, 122.4, 125.0, 128.3, 128.6, 129.3, 131.0, 133.90, 133.94, 134.0, 138.2, 144.6, 147.0, 157.9, 167.6, 167.9, 171.0, 173.2; HRMS (ESI-TOF) calcd for C₃₄H₄₂N₄O₈Na 657.2895 (M+Na⁺), found 657.2888.



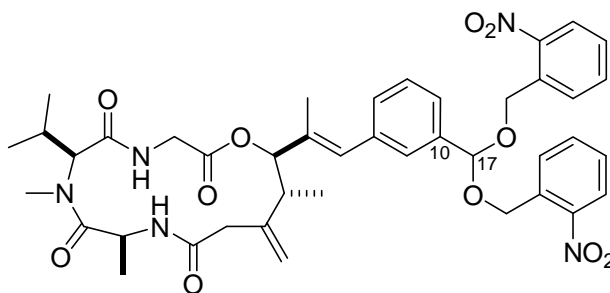
***p*-(*o*-Nitrobenzyloxy)-styrene-analogue 2c.** **2c** was obtained in 77% yield by following the procedure of **1a**:

Colorless solid; $[\alpha]_D^{25.6} = -141^\circ$ (*c* 0.59, MeOH); IR (film) ν 3279, 2967, 1735, 1638, 1526, 1509, 1341, 1244, 756 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 0.86 (3H, d, *J* = 6.3 Hz, H15'), 0.93 (3H, d, *J* = 7.5 Hz, H13), 0.97 (3H, d, *J* = 6.3 Hz, H14'), 1.43 (3H, d, *J* = 6.9 Hz, H11'), 1.71 (3H, s, H14), 2.26 (1H, dq, *J* = 11.4, 7.5 Hz, H4), 2.42 (1H, dq, *J* = 10.9, 6.3, 6.3 Hz, H13'), 2.84 (3H, s, H12'), 2.85 (1H, d, *J* = 13.2 Hz, H2), 3.01 (1H, d, *J* = 13.2 Hz, H2), 3.50 (1H, d, *J* = 18.3 Hz, H8'), 4.24 (1H, d, *J* = 10.9 Hz, H5'), 4.69 (1H, dd, *J* = 18.3, 10.3 Hz, H8'), 5.06 (1H, s, H12), 5.09 (1H, s, H12), 5.33 (1H, d, *J* = 11.4 Hz, H5), 5.36 (1H, dq, *J* = 9.8, 6.9 Hz, H2'), 5.50 (2H, s, OCH₂C₆H₄NO₂), 6.53 (1H, s, H7), 6.65 (1H, d, *J* = 9.8 Hz, H1'), 6.94 (2H, d, *J* = 8.6 Hz, H10, H15), 7.23 (2H, d, *J* = 8.6 Hz, H9, H16), 7.48 (1H, t, *J* = 8.0 Hz, OCH₂C₆H₄NO₂), 7.67 (1H, t, *J* = 8.0 Hz, OCH₂C₆H₄NO₂), 7.87 (1H, d, *J* = 8.0 Hz, OCH₂C₆H₄NO₂), 7.98 (1H, d, *J* = 10.3 Hz, H7'), 8.17 (1H, d, *J* = 8.0 Hz, OCH₂C₆H₄NO₂); ¹³C NMR (125 MHz, CDCl₃) δ 12.9, 18.57, 18.63, 18.9, 19.3, 26.1, 28.7, 39.1, 41.1, 43.1, 46.6, 66.8, 67.1, 83.2, 113.9, 114.6, 125.0, 128.3, 128.5, 130.1, 130.5, 130.7, 131.9, 133.9, 134.0, 144.6, 146.9, 157.0, 167.6, 167.9, 171.0, 173.2; HRMS (ESI-TOF) calcd for C₃₄H₄₂N₄O₈Na 657.2895 (M+Na⁺), found 657.2899.

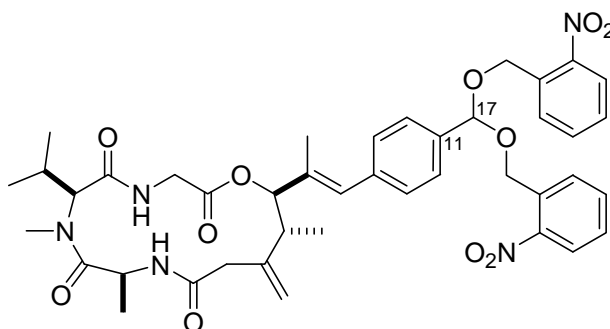


***o*-[Bis(*o*-nitrobenzyloxy)methyl]-styrene-analogue 2d.** To a mixture of **3** (7.7 mg, 14.4 μ mol) and boronic ester **6d** (20 mg, 38.5 μ mol) were added dry THF (150 μ L), PdCl₂(dppf)·CH₂Cl₂ (3.0 mg, 3.6 μ mol), Cs₂CO₃ (9.4 mg, 28.9 μ mol) and Ph₃As (2.2 mg, 7.2 μ mol) at room temperature. The reaction mixture was stirred at room temperature for 24 h in the dark and filtrated through a pad of Celite with Et₂O. The filtrate was concentrated. The residue was purified with flash column chromatography on Florisil (hexane/AcOEt = 5/1 to 1/2) and HPLC (column: Inertsil ODS-3 ϕ 10 x 250 mm, flow rate: 2 mL/min, detection: UV 210 nm, eluent: MeCN/H₂O = 4/1, *T_R* = 21.8 min) to give 5.5 mg of **2d** (48%): colorless solid; $[\alpha]_D^{17.6} = -86^\circ$ (*c* 0.28, MeOH); IR (film) ν 3275, 2964, 1734, 1685, 1647, 1525, 1457, 1340, 1258 cm⁻¹; ¹H NMR (500 MHz, CD₃OD) δ 0.86 (3H, d, *J* = 6.9 Hz,

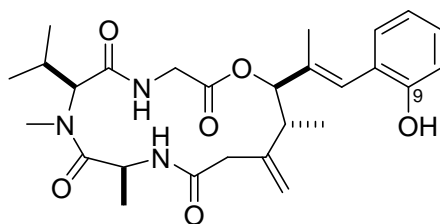
H15'), 0.95 (3H, d, $J = 6.9$ Hz, H14'), 0.97 (3H, d, $J = 7.5$ Hz, H13), 1.36 (3H, d, $J = 6.9$ Hz, H11'), 1.46 (3H, d, $J = 1.1$ Hz, H14), 2.29 (1H, dq, $J = 10.9, 6.9, 6.9$ Hz, H13'), 2.36 (1H, dq, $J = 11.4, 7.5$ Hz, H4), 2.68 (3H, s, H12'), 2.90 (1H, d, $J = 13.2$ Hz, H2), 3.15 (1H, d, $J = 13.2$ Hz, H2), 3.51 (1H, d, $J = 18.3$ Hz, H8'), 4.449 (1H, d, $J = 10.9$ Hz, H5'), 4.453 (1H, d, $J = 18.3$ Hz, H8'), 4.91 (1H, s, OCH₂C₆H₄NO₂), 4.92 (1H, s, OCH₂C₆H₄NO₂), 4.946 (1H, s, OCH₂C₆H₄NO₂), 4.954 (1H, s, OCH₂C₆H₄NO₂), 5.02 (1H, s, H12), 5.05 (1H, s, H12), 5.30 (1H, d, $J = 11.4$ Hz, H5), 5.37 (1H, q, $J = 6.9$ Hz, H2'), 5.86 (1H, s, H17), 6.75 (1H, d, $J = 1.1$ Hz, H7), 7.11 (1H, dd, $J = 5.7, 3.4$ Hz, H16), 7.35 (2H, dd, $J = 5.7, 3.4$ Hz, H11, H15), 7.48 (2H, td, $J = 8.0, 1.7$ Hz, OCH₂C₆H₄NO₂), 7.63 (1H, td, $J = 8.0, 1.7$ Hz, OCH₂C₆H₄NO₂), 7.65 (1H, td, $J = 8.0, 1.7$ Hz, OCH₂C₆H₄NO₂), 7.68 (1H, dd, $J = 8.0, 1.7$ Hz, OCH₂C₆H₄NO₂), 7.70 (1H, dd, $J = 5.7, 3.4$ Hz, H10), 7.74 (1H, dd, $J = 8.0, 1.7$ Hz, OCH₂C₆H₄NO₂), 7.99 (1H, dd, $J = 8.0, 1.7$ Hz, OCH₂C₆H₄NO₂), 8.00 (1H, dd, $J = 8.0, 1.7$ Hz, OCH₂C₆H₄NO₂); ¹³C NMR (125 MHz, CD₃OD) δ 13.2, 18.6, 18.8, 19.1, 19.3, 27.5, 29.3, 39.7, 41.9, 44.5, 53.6, 66.5, 67.8, 84.4, 102.2, 125.5, 125.6, 127.56, 127.62, 129.4, 129.5, 129.8, 130.4, 134.6, 135.3, 135.4, 136.6, 137.1, 147.6, 149.1, 147.6, 149.07, 149.09, 169.4, 169.7, 173.0, 175.3; HRMS (ESI-TOF) calcd for C₄₂H₄₉N₅O₁₁Na 822.3321 (M+Na⁺), found 822.3327.



***m*-[Bis-(*o*-nitrobenzyloxy)methyl]-styrene-analogue 2e.** 2e was obtained in 36% yield by following the procedure of 2d. Colorless solid; $[\alpha]_D^{26.8} = -142^\circ$ (c 0.065, MeOH); IR (film) ν 3285, 2962, 1729, 1641, 1527, 1342, 1261, 1032 cm⁻¹; ¹H NMR (500 MHz, CD₃OD) δ 0.90 (3H, d, $J = 6.9$ Hz, H15'), 0.97 (3H, d, $J = 6.3$ Hz, H14'), 1.00 (3H, d, $J = 6.9$ Hz, H13), 1.39 (3H, d, $J = 6.8$ Hz, H11'), 1.70 (3H, d, $J = 1.1$ Hz, H14), 2.35 (1H, dq, $J = 10.9, 6.9, 6.3$ Hz, H13'), 2.41 (1H, dq, $J = 11.5, 6.9$ Hz, H4), 2.81 (3H, s, H12'), 2.91 (1H, d, $J = 12.6$ Hz, H2), 3.17 (1H, d, $J = 12.6$ Hz, H2), 3.55 (1H, d, $J = 18.3$ Hz, H8'), 4.49 (1H, d, $J = 10.9$ Hz, H5'), 4.53 (1H, d, $J = 18.3$ Hz, H8'), 4.94 (4H, s, OCH₂C₆H₄NO₂), 5.04 (1H, s, H12), 5.07 (1H, s, H12), 5.35 (1H, d, $J = 11.5$ Hz, H5), 5.43 (1H, q, $J = 6.9$ Hz, H2'), 5.88 (1H, s, H17), 6.62 (1H, d, $J = 1.1$ Hz, H7), 7.26 (1H, d, $J = 7.4$ Hz, H16), 7.37 (1H, t, $J = 7.4$ Hz, H15), 7.41 (1H, d, $J = 7.4$ Hz, H11), 7.45 (1H, s, H9), 7.49 (2H, td, 8.0, 1.1 Hz, OCH₂C₆H₄NO₂), 7.66 (2H, td, $J = 8.0, 1.1$ Hz, OCH₂C₆H₄NO₂), 7.75 (2H, dd, $J = 8.0, 1.1$ Hz, OCH₂C₆H₄NO₂), 7.99 (2H, dd, $J = 8.0, 1.1$ Hz, OCH₂C₆H₄NO₂); ¹³C NMR (125 MHz, CD₃OD) δ 13.8, 18.6, 18.8, 19.1, 19.2, 27.5, 29.5, 39.9, 41.9, 44.4, 46.4, 65.65, 65.68, 67.9, 84.9, 103.5, 113.5, 114.4, 125.6, 126.5, 128.3, 129.4, 129.51, 129.55, 130.5, 130.7, 131.9, 134.6, 135.1, 135.2, 135.6, 138.3, 139.0, 147.6, 148.0, 149.21, 149.24, 169.4, 170.3, 173.1, 175.3; HRMS (ESI-TOF) calcd for C₄₂H₄₉N₅O₁₁Na 822.3321 (M+Na⁺), found 822.3327.

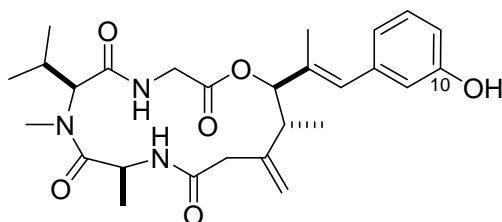


***p*-[Bis(*o*-nitrobenzyloxy)methyl]-styrene-analogue 2f.** **2f** was obtained in 73% yield by following the procedure of **2d**. Colorless solid; $[\alpha]_D^{15.2} = -87^\circ$ (c 0.42, MeOH); IR (film) ν 3276, 2964, 1735, 1644, 1526, 1341, 1268, 1034 cm^{-1} ; ^1H NMR (500 MHz, CD_3OD) δ 0.89 (3H, d, $J = 6.9$ Hz, H15'), 0.97 (3H, d, $J = 6.8$ Hz, H14'), 0.99 (3H, d, $J = 6.9$ Hz, H13), 1.39 (3H, d, $J = 6.8$ Hz, H11'), 1.71 (3H, d, $J = 1.1$ Hz, H14), 2.36 (1H, dq, $J = 10.9, 6.9, 6.8$ Hz, H13'), 2.40 (1H, dq, $J = 10.9, 6.9$ Hz, H4), 2.83 (3H, s, H12'), 2.91 (1H, d, $J = 13.2$ Hz, H2), 3.16 (1H, d, $J = 13.2$ Hz, H2), 3.54 (1H, d, $J = 18.9$ Hz, H8'), 4.49 (1H, d, $J = 10.9$ Hz, H5'), 4.52 (1H, d, $J = 18.9$ Hz, H8'), 4.92 (4H, s, OCH₂C₆H₄NO₂), 5.03 (1H, s, H12), 5.06 (1H, s, H12), 5.35 (1H, d, $J = 10.9$ Hz, H5), 5.43 (1H, q, $J = 6.9$ Hz, H2'), 5.85 (1H, s, H17), 6.61 (1H, d, $J = 1.1$ Hz, H7), 7.30 (2H, d, $J = 8.0$ Hz, H9, H16), 7.46 (2H, td, $J = 8.6, 1.1$ Hz, OCH₂C₆H₄NO₂), 7.49 (2H, d, $J = 8.0$ Hz, H10, H15), 7.64 (2H, td, $J = 8.6, 1.1$ Hz, OCH₂C₆H₄NO₂), 7.75 (2H, dd, $J = 8.6, 1.1$ Hz, OCH₂C₆H₄NO₂), 7.97 (2H, dd, $J = 8.6, 1.1$ Hz, OCH₂C₆H₄NO₂); ^{13}C NMR (125 MHz, CD_3OD) δ 13.8, 18.6, 18.8, 19.1, 19.2, 27.5, 29.4, 39.9, 41.9, 44.4, 46.4, 65.6, 67.9, 84.9, 85.2, 103.4, 113.5, 125.5, 127.8, 129.5, 130.1, 130.5, 131.8, 134.6, 135.2, 135.7, 137.7, 138.8, 147.6, 149.2, 169.3, 170.2, 173.0, 175.3; HRMS (ESI-TOF) calcd for C₄₂H₄₉N₅O₁₁Na 822.3321 (M+Na⁺), found 822.3317.

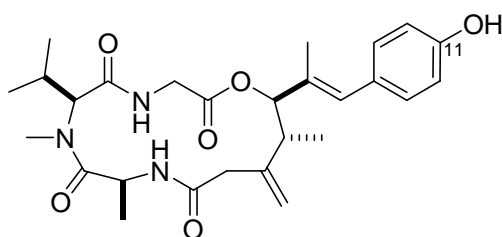


***o*-Hydroxy-styrene-analogue 7a.** A solution of **2a** (1.56 mg, 2.46 μmol) in CD_3OD (600 μL) in NMR tube was irradiated with UV light ($\lambda = 365$ nm) using handy UV lamp (AS ONE, SLUV-6, 6 W) at room temperature for 1.5 h. The mixture was concentrated. The residue was purified with flash column chromatography on silica gel (hexane/AcOEt = 1.5/1 to 1/1) and HPLC (column: Inertsil ODS-3 ϕ 10 x 250 mm, flow rate: 2 mL/min, detection: UV 254 nm, eluent: MeOH/H₂O = 2.5/1, $T_R = 19.9$ min) to give 0.73 mg of **7a** (60%): colorless solid; $[\alpha]_D^{23.7} = -139^\circ$ (c 0.063, MeOH); IR (film) ν 3280, 2966, 1734, 1625, 1558, 1454, 1261, 960, 756 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 0.86 (3H, d, $J = 6.8$ Hz, H15'), 0.97 (3H, d, $J = 6.8$ Hz, H14'), 1.03 (3H, d, $J = 7.5$ Hz, H13), 1.44 (3H, d, $J = 6.9$ Hz, H11'), 1.70 (3H, s, H14), 2.37 (1H, dq, $J = 11.5, 7.5$ Hz, H4), 2.41 (1H, dq, $J = 10.9, 6.8, 6.8$ Hz, H13'), 2.74 (3H, s, H12'), 2.88 (1H, d, $J = 13.2$ Hz, H2), 3.03 (1H, d, $J = 13.2$ Hz, H2), 3.52 (1H, dd, $J = 18.9, 1.7$ Hz, H8'), 4.21 (1H, d, $J = 10.9$ Hz, H5'), 4.67 (1H, dd, $J = 18.9, 10.3$ Hz, H8'), 5.04 (1H, s, H12), 5.11 (1H, s, H12), 5.33 (1H, d, $J = 11.5$ Hz, H5), 5.35 (1H, dq, $J = 9.8, 6.9$ Hz, H2'), 6.55 (1H, s, H7), 6.58 (1H, br, OH), 6.62

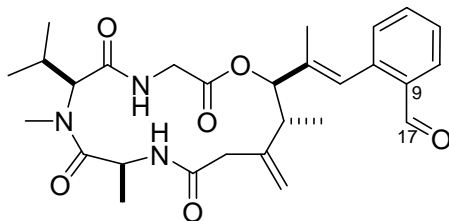
(1H, d, $J = 9.8$ Hz, H1'), 6.87 (1H, t, $J = 7.4$ Hz, CHC6H4OH), 6.90 (1H, d, $J = 8.6$ Hz, CHC6H4OH), 7.10 (1H, dd, $J = 7.4, 1.1$ Hz, CHC6H4OH), 7.16 (1H, ddd, $J = 8.6, 7.4, 1.1$ Hz, CHC6H4OH), 8.00 (1H, dd, $J = 10.3, 1.7$ Hz, H7'); ^{13}C NMR (125 MHz, CDCl_3) δ 15.2, 18.58, 18.64, 19.1, 19.3, 25.9, 29.0, 39.7, 41.2, 43.3, 46.6, 67.4, 81.1, 114.2, 116.3, 119.9, 123.0, 124.3, 128.9, 129.8, 136.8, 144.7, 153.9, 167.6, 168.3, 171.2, 173.8; HRMS (ESI-TOF) calcd for $\text{C}_{27}\text{H}_{37}\text{N}_3\text{O}_6\text{Na}$ 522.2575 ($\text{M}+\text{Na}^+$), found 522.2578.



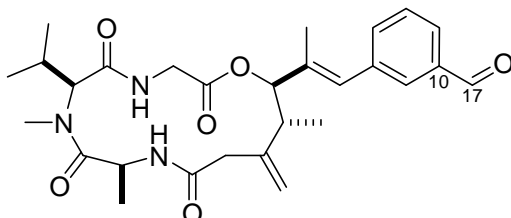
***m*-Hydroxy-styrene-analogue 7b.** **7b** was obtained in 59% yield by following the procedure of **7a**. Colorless solid; $[\alpha]_{\text{D}}^{17.5} = -285^\circ$ (c 0.016, MeOH); IR (film) ν 3283, 2967, 1734, 1623, 1456, 1269, 962, 909, 731 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 0.87 (3H, d, $J = 6.9$ Hz, H15'), 0.88 (3H, d, $J = 6.9$ Hz, H13), 0.98 (3H, d, $J = 6.9$ Hz, H14'), 1.44 (3H, d, $J = 6.8$ Hz, H11'), 1.70 (3H, d, $J = 1.1$ Hz, H14), 2.26 (1H, dq, $J = 11.5, 6.9$ Hz, H4), 2.44 (1H, dq, $J = 10.9, 6.9, 6.9$ Hz, H13'), 2.86 (3H, s, H12'), 2.87 (1H, d, $J = 13.2$ Hz, H2), 3.02 (1H, d, $J = 13.2$ Hz, H2), 3.52 (1H, dd, $J = 18.3, 1.7$ Hz, H8'), 4.27 (1H, d, $J = 10.9$ Hz, H5'), 4.70 (1H, dd, $J = 18.3, 10.3$ Hz, H8'), 5.05 (1H, s, H12), 5.09 (1H, s, H12), 5.32 (1H, d, $J = 11.5$ Hz, H5), 5.37 (1H, dq, $J = 9.1, 6.8$ Hz, H2'), 6.18 (1H, br, OH), 6.51 (1H, d, $J = 1.1$ Hz, H7), 6.71 (1H, dd, $J = 8.0, 1.7$ Hz, H11), 6.75 (1H, d, $J = 9.1$ Hz, H1'), 6.77 (1H, d, $J = 8.0$ Hz, H16), 6.80 (1H, d, $J = 1.7$ Hz, H9), 7.17 (1H, t, $J = 8.0$ Hz, H15), 8.03 (1H, dd, $J = 10.3, 1.7$ Hz, H7'); ^{13}C NMR (125 MHz, CDCl_3) δ 13.1, 18.5, 18.6, 18.8, 19.3, 26.1, 28.8, 39.0, 41.1, 43.2, 50.9, 67.2, 83.0, 113.9, 114.0, 115.6, 121.7, 129.4, 130.8, 133.5, 138.1, 144.6, 155.5, 167.5, 167.8, 171.1, 179.0; HRMS (ESI-TOF) calcd for $\text{C}_{27}\text{H}_{37}\text{N}_3\text{O}_6\text{Na}$ 522.2575 ($\text{M}+\text{Na}^+$), found 522.2570.



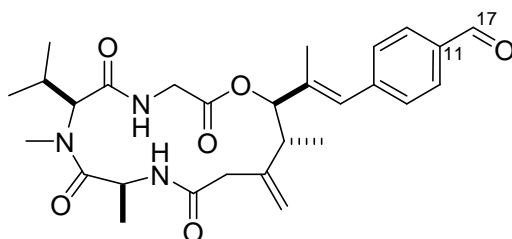
***p*-Hydroxy-styrene-analogue 7c.** **7c** was obtained in 62% yield by following the procedure of **7a**. Colorless solid; $[\alpha]_{\text{D}}^{17.2} = -177^\circ$ (c 0.020, MeOH); IR (film) ν 3280, 2927, 1624, 1513, 1456, 1263, 960, 906, 731 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 0.78 (3H, d, $J = 6.8$ Hz, H13), 0.87 (3H, d, $J = 6.9$ Hz, H15'), 0.98 (3H, d, $J = 6.8$ Hz, H14'), 1.45 (3H, d, $J = 6.9$ Hz, H11'), 1.63 (3H, s, H14), 2.15 (1H, dq, $J = 11.5, 6.8$ Hz, H4), 2.44 (1H, dq, $J = 10.9, 6.9, 6.8$ Hz, H13'), 2.84 (1H, d, $J = 13.2$ Hz, H2), 2.89 (3H, s, H12'), 3.01 (1H, d, $J = 13.2$ Hz, H2), 3.51 (1H, dd, $J = 18.9, 1.7$ Hz, H8'), 4.29 (1H, d, $J = 10.9$ Hz, H5'), 4.70 (1H, dd, $J = 18.9, 10.3$ Hz, H8'), 5.03 (1H, s, H12), 5.07 (1H, s, H12), 5.29 (1H, d, $J = 11.5$ Hz, H5), 5.38 (1H, dq, $J = 6.9$ Hz, H2'), 6.46 (1H, s, H7), 6.81 (3H, d, $J = 9.1$ Hz, H1', CHC6H4OH), 7.13 (2H, d, $J = 9.1$ Hz, CHC6H4OH), 8.03 (1H, dd, $J = 10.3, 1.7$ Hz, H7'); HRMS (ESI-TOF) calcd for $\text{C}_{27}\text{H}_{37}\text{N}_3\text{O}_6\text{Na}$ 522.2575 ($\text{M}+\text{Na}^+$), found 522.2575.



***o*-Formyl-styrene-analogue 7d.** **7d** was obtained as a 3:2 mixture of *E*- and *Z*-isomers at C6-C7-olefin by following the procedure of **7a**. *E*-isomer: Colorless solid; $[\alpha]_D^{18.7} = -144^\circ$ (*c* 0.10, CHCl₃); IR (film) ν 3281, 2924, 1687, 1640, 1542, 1456, 1259, 755 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 0.86 (3H, d, *J* = 6.8 Hz, H15'), 0.98 (3H, d, *J* = 6.9 Hz, H14'), 1.02 (3H, d, *J* = 7.4 Hz, H13), 1.42 (3H, d, *J* = 6.8 Hz, H11'), 1.52 (3H, s, H14), 2.28 (1H, dq, *J* = 11.5, 7.4 Hz, H4), 2.42 (1H, dqq, *J* = 11.5, 6.9, 6.8 Hz, H13'), 2.80 (3H, s, H12'), 2.86 (1H, d, *J* = 13.2 Hz, H2), 3.04 (1H, d, *J* = 13.2 Hz, H2), 3.54 (1H, dd, *J* = 18.3, 1.7 Hz, H8'), 4.24 (1H, d, *J* = 11.5 Hz, H5'), 4.73 (1H, dd, *J* = 18.3, 10.3 Hz, H8'), 5.09 (1H, s, H12), 5.12 (1H, s, H12), 5.35 (1H, dq, *J* = 9.7, 6.8 Hz, H2'), 5.41 (1H, d, *J* = 11.5 Hz, H5), 6.60 (1H, d, *J* = 9.7 Hz, H1'), 7.00 (1H, s, H7), 7.23 (1H, d, *J* = 8.0 Hz, H10), 7.40 (1H, t, *J* = 8.0 Hz, H15), 7.55 (1H, t, *J* = 8.0 Hz, H11), 7.89 (1H, d, *J* = 8.0 Hz, H16), 8.00 (1H, dd, *J* = 10.3, 1.7 Hz, H7'); ¹³C NMR (125 MHz, CDCl₃) δ 12.7, 18.58, 18.64, 18.8, 19.3, 26.1, 28.7, 39.0, 41.1, 43.1, 46.5, 67.1, 82.2, 114.1, 127.7, 128.1, 129.0, 130.5, 133.6, 133.7, 136.8, 139.6, 144.4, 167.7, 167.9, 170.9, 173.2, 191.9; HRMS (ESI-TOF) calcd for C₂₈H₃₇N₃O₆Na 534.2575 (M+Na⁺), found 534.2571.



***m*-Formyl-styrene-analogue 7e** was obtained as a 3:1 mixture of *E*- and *Z*-isomers at C6-C7-olefin by following the procedure of **7a**. *E*-isomer: Colorless solid; $[\alpha]_D^{19.4} = -172^\circ$ (*c* 0.10, CHCl₃); IR (film) ν 3277, 2967, 1685, 1637, 1541, 1457, 1259 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 0.87 (3H, d, *J* = 6.9 Hz, H15'), 0.97 (3H, d, *J* = 5.7 Hz, H13), 0.98 (3H, d, *J* = 6.9 Hz, H14'), 1.44 (3H, d, *J* = 6.9 Hz, H11'), 1.74 (3H, d, *J* = 1.1 Hz, H14), 2.28 (1H, dq, *J* = 11.5, 5.7 Hz, H4), 2.43 (1H, dqq, *J* = 10.9, 6.9, 6.9 Hz, H13'), 2.84 (3H, s, H12'), 2.85 (1H, d, *J* = 13.2 Hz, H2), 3.04 (1H, d, *J* = 13.2 Hz, H2), 3.53 (1H, d, *J* = 18.9, 1.8 Hz, H8'), 4.24 (1H, d, *J* = 10.9 Hz, H5'), 4.71 (1H, d, *J* = 18.9, 10.3 Hz, H8'), 5.08 (1H, s, H12), 5.12 (1H, s, H12), 5.36 (1H, d, *J* = 11.5 Hz, H5), 5.37 (1H, dq, *J* = 9.7, 6.9 Hz, H2'), 6.59, (1H, d, *J* = 9.7 Hz, H1'), 6.65 (1H, s, H7), 7.50 (1H, t, *J* = 7.4 Hz, H15), 7.53 (1H, d, *J* = 7.4 Hz, H16), 7.75 (1H, d, *J* = 7.4 Hz, H11), 7.77 (1H, s, H9), 8.01 (1H, dd, *J* = 10.9, 1.8 Hz, H7'), 10.02 (1H, d, *J* = 4.0 Hz, H17); ¹³C NMR (125 MHz, CDCl₃) δ 13.0, 18.61, 18.64, 18.9, 19.3, 26.1, 28.7, 39.1, 41.1, 43.1, 46.6, 67.1, 82.7, 114.1, 128.1, 128.9, 129.9, 130.2, 135.0, 135.3, 136.3, 137.6, 144.4, 167.7, 167.9, 170.9, 173.2, 192.3; HRMS (ESI-TOF) calcd for C₂₈H₃₇N₃O₆Na 534.2575 (M+Na⁺), found 534.2573.



***p*-Formylstyrene-analogue 7f.** **7f** was obtained as a 2:1 mixture of *E*- and *Z*-isomers at C6-C7-olefin by following the procedure of **7a**. *E*-isomer: colorless solid; IR (film) ν 3280, 2927, 1688, 1640, 1552, 1457, 1259, 1169, 977, 757 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 0.87 (3H, d, $J = 7.5$ Hz, H15'), 0.96 (3H, d, $J = 6.9$ Hz, H13), 0.98 (3H, d, $J = 6.9$ Hz, H14'), 1.44 (3H, d, $J = 6.9$ Hz, H11'), 1.76 (3H, d, $J = 1.1$ Hz, H14), 2.27 (1H, dq, $J = 11.5, 6.9$ Hz, H4), 2.43 (1H, dq, $J = 11.5, 7.5, 6.9$ Hz, H13'), 2.84 (3H, s, H12'), 2.85 (1H, d, $J = 13.2$ Hz, H2), 3.04 (1H, d, $J = 13.2$ Hz, H2), 3.53 (1H, dd, $J = 18.9, 1.7$ Hz, H8'), 4.24 (1H, d, $J = 11.5$ Hz, H5'), 4.71 (1H, d, $J = 18.9, 10.3$ Hz, H8'), 5.07 (1H, s, H12), 5.12 (1H, s, H12), 5.35 (1H, d, $J = 11.5$ Hz, H5), 5.37 (1H, dq, $J = 9.1, 6.9$ Hz, H2'), 6.59 (1H, d, $J = 9.1$ Hz, H1'), 6.64 (1H, d, $J = 1.1$ Hz, H7), 7.43 (2H, d, $J = 8.6$ Hz), 7.84 (2H, d, $J = 8.6$ Hz), 8.01 (1H, dd, $J = 10.3, 1.7$ Hz, H7'), 9.99 (1H, s, H17); HRMS (ESI-TOF) calcd for $\text{C}_{28}\text{H}_{37}\text{N}_3\text{O}_6\text{Na}$ 534.2575 ($\text{M}+\text{Na}^+$), found 534.2578.

Neurotoxicity assay against Neuro 2a mouse neuroblastoma

Neuro 2a cells (ATCC, CCL131), obtained from Institute of Development Aging and Cancer (Tohoku university), were grown and maintained in 75 cm^2 tissue culture flasks (Falcon) at 37 $^\circ\text{C}$ in a humidified 5% CO_2 atmosphere using a growth medium, which was composed of RPMI 1640 medium supplemented with 10% heat inactivated fetal bovine serum (Gibco), 2 mM L-glutamine, and 1% of antibiotic antifungal solution (10000 U/ml penicillin G, 10 mg/ml streptomycin).

Cells were harvested in trypsin-EDTA solution (0.5% – 0.2%, 5 min at room temperature), and diluted to a concentration of 4×10^5 cell/mL with the growth medium. 100 μL of the cell suspension was inoculated into each well of a 96-well microplate (Falcon) and mixed with 100 μL of a solution of antillatoxin analogue to give a range of final concentrations between 10^{-4} and 10^{-9} M. The solutions of antillatoxin analogue were prepared from 100 μM DMSO stock solutions by sequential dilution with the growth media containing veratridine (40 μM), a site 2-specific sodium channel activator, ouabain (88 μM), a blocker of the Na^+/K^+ ATPase, and DMSO (2 v/v%). Three replicate samples were prepared for each antillatoxin analogues. For the experiments of Figure 2, the 96-well microplate was irradiated with UV light ($\lambda = 365$ nm) using handy UV lamp (AS ONE, SLUV-6, 6 W) at room temperature for 30 min (Figure S1). After incubation for 20 hours at 37 $^\circ\text{C}$, cells were treated with 50 μL of 100 μM /3 mM PMS/XTT-containing growth medium, followed by further incubation for 4 hours. Absorbance at 490 nm was measured on the microplate reader Model 550 (Bio Rad). The EC_{50} values were calculated using Prism v. 4.0 (GraphPad).

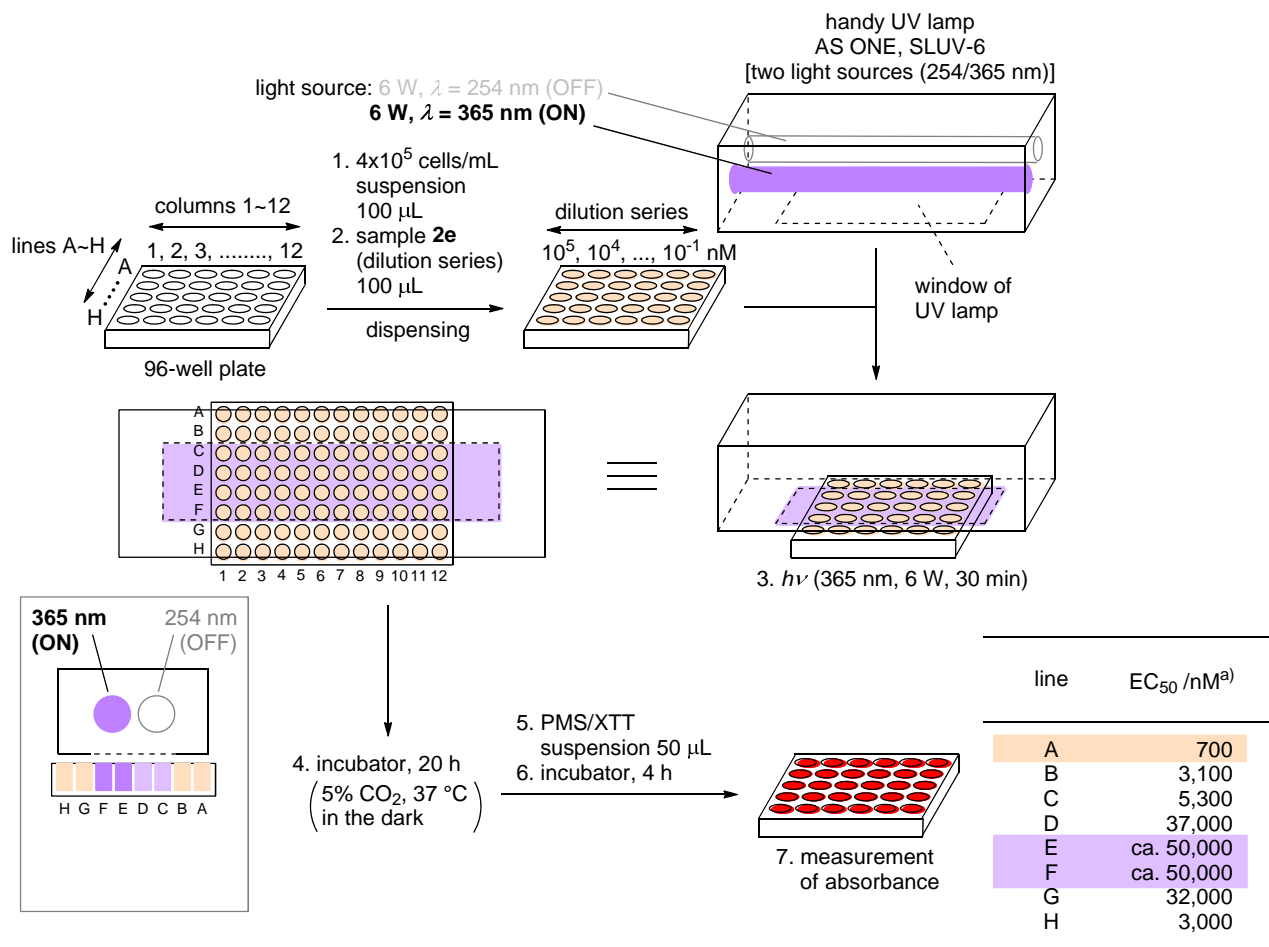
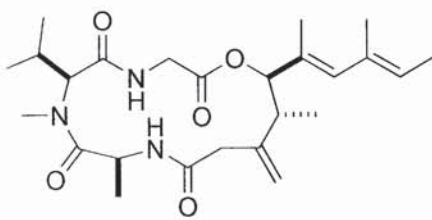
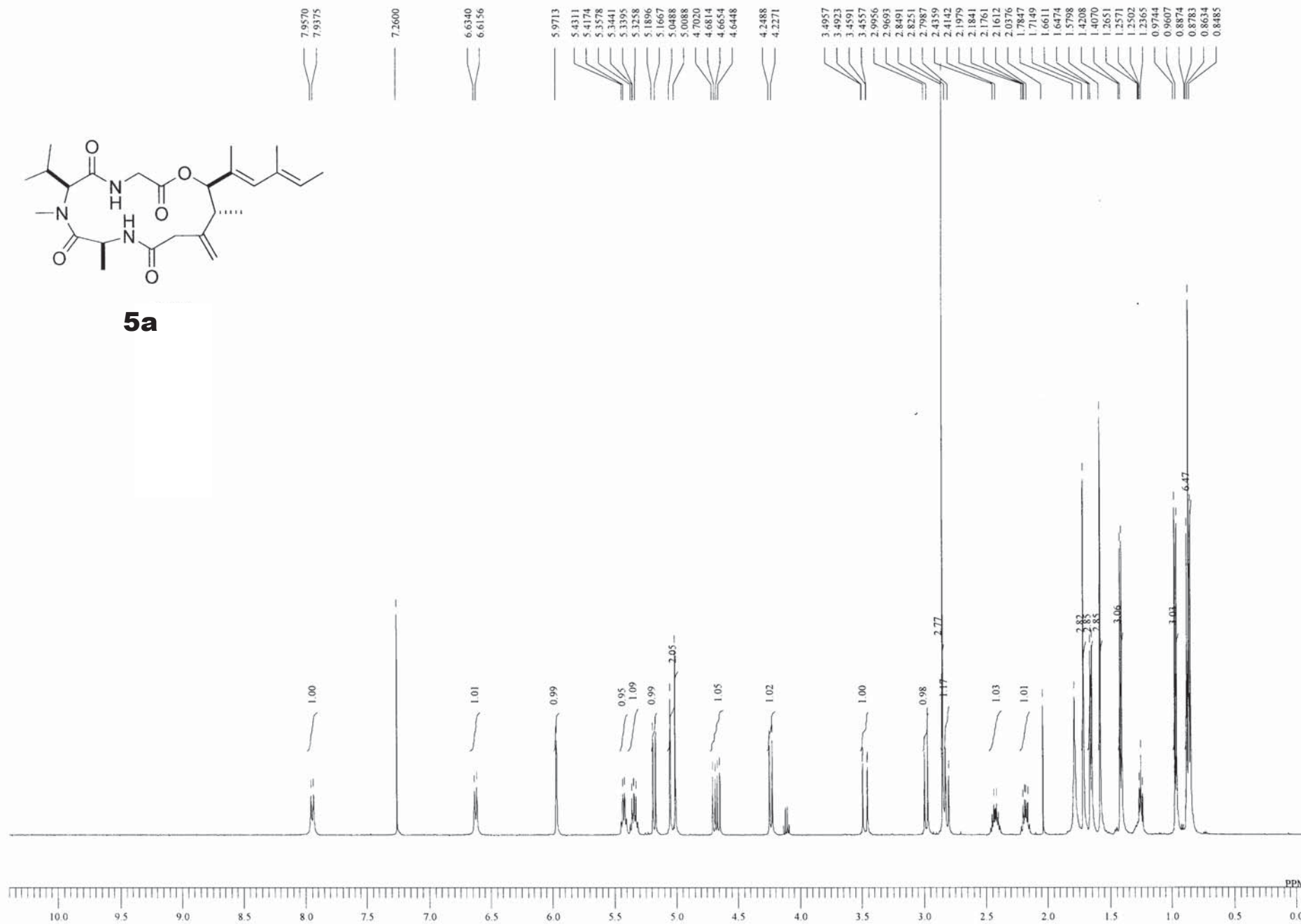


Figure S1. Experimental procedure for the cytotoxicity assay of UV-pre-irradiated **2e**. Magnitude of the EC₅₀ values correlated to distance between the lines and the light source of 365 nm. The data of line A, which was kept in the dark, represented the control experiment, and the data of lines E and F, which were closest from the light source, represented the UV pre-irradiated experiment.

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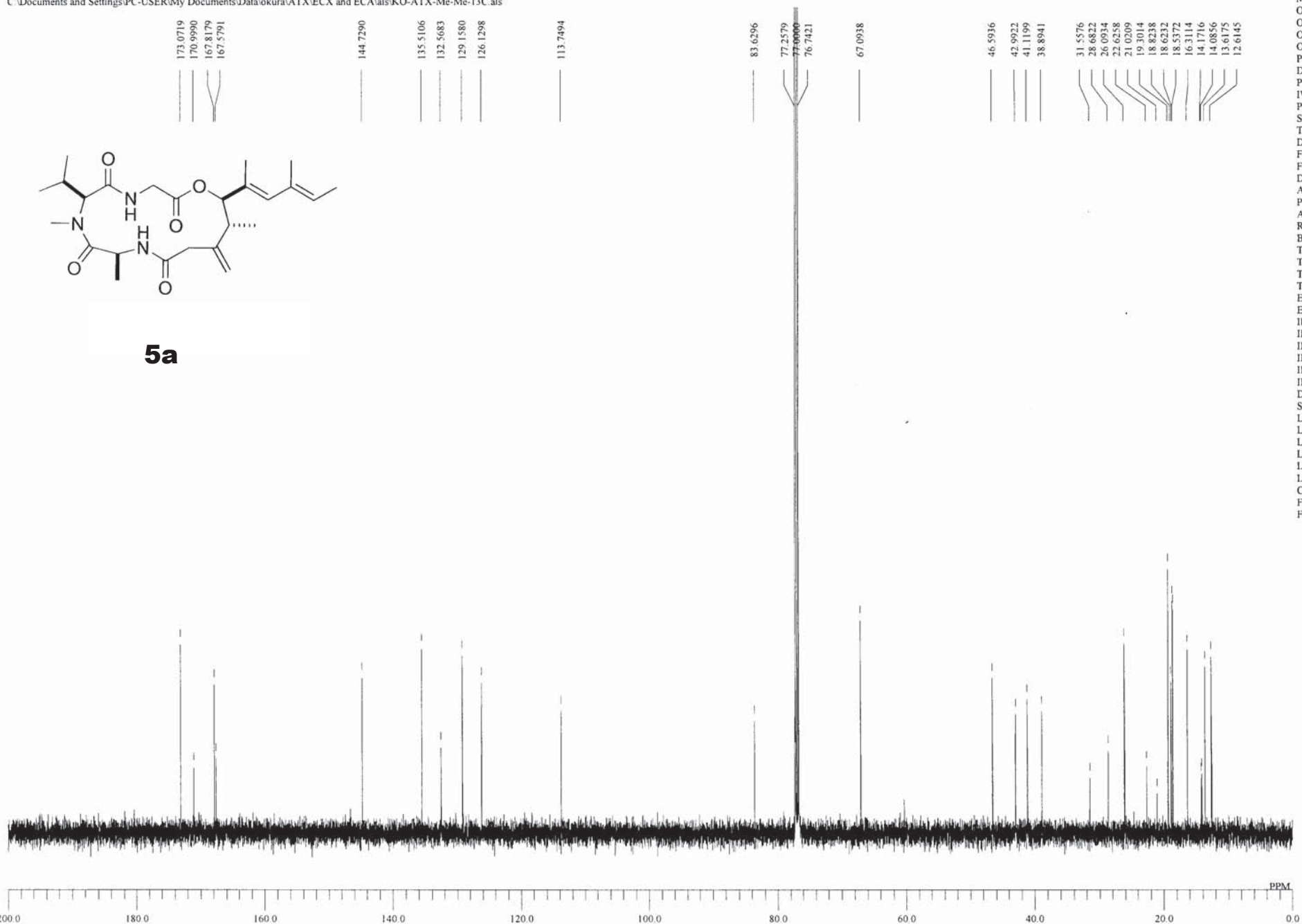


5a



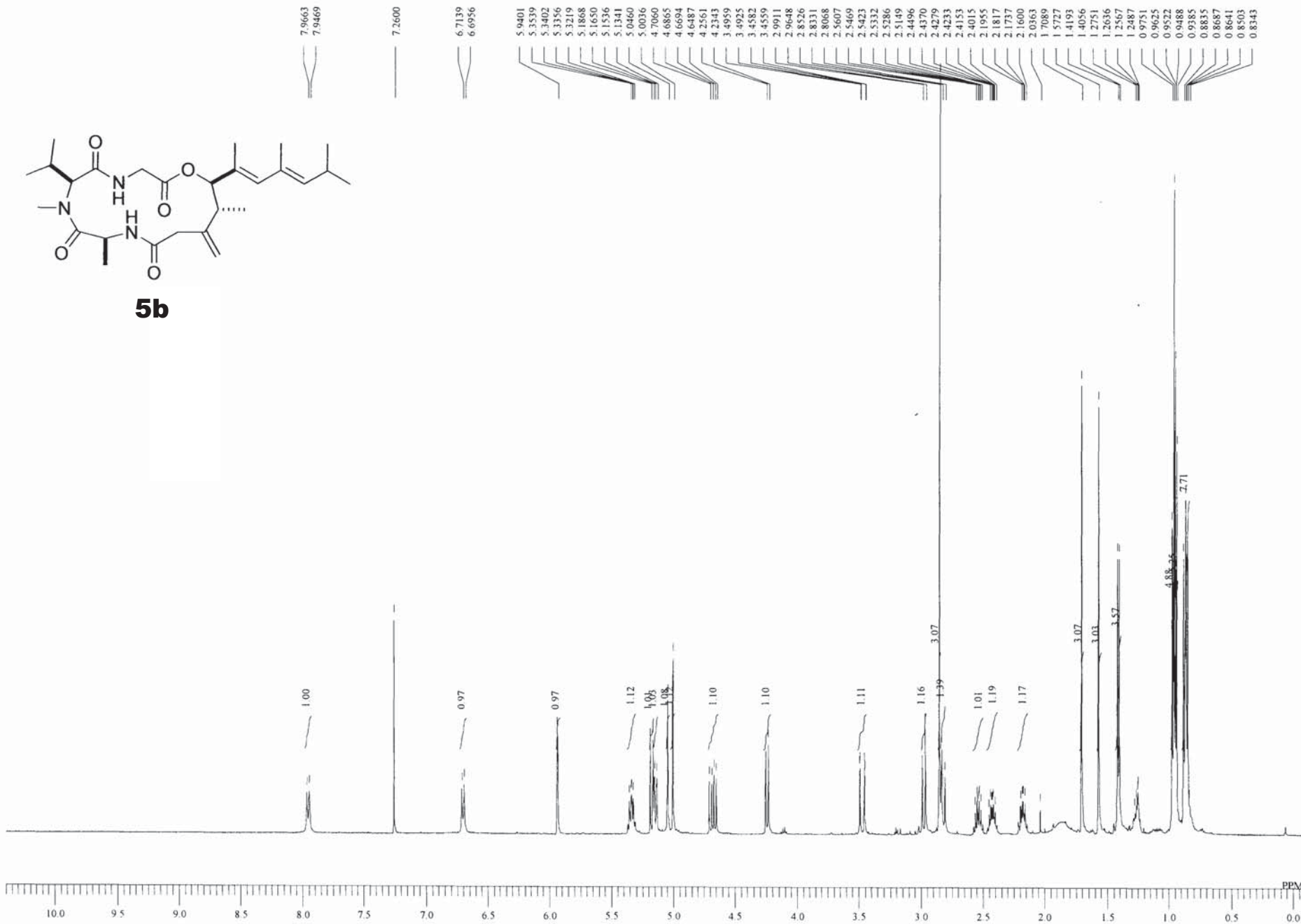
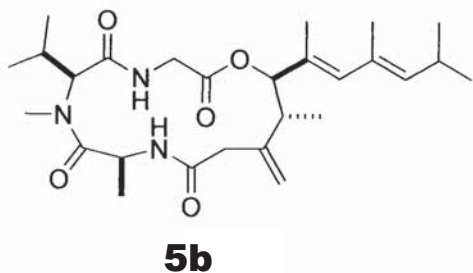
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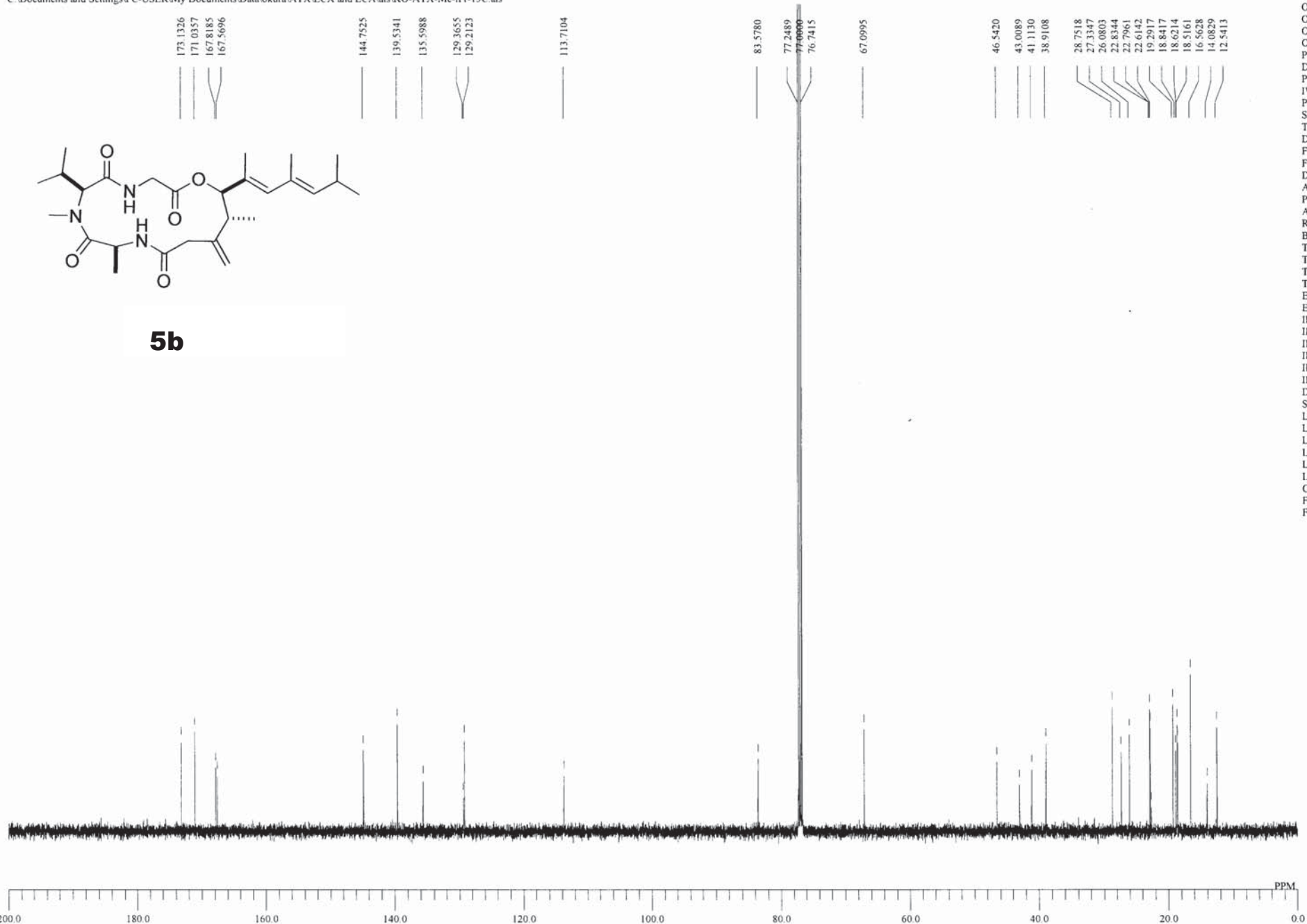
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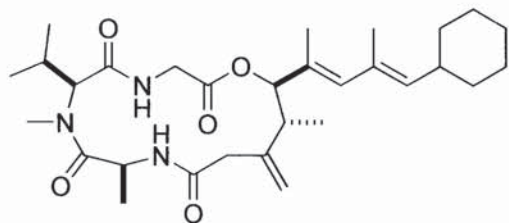
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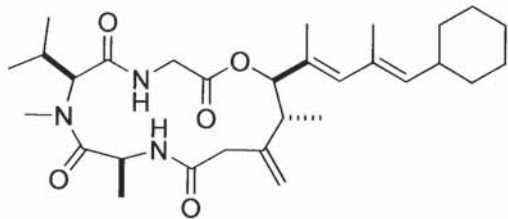
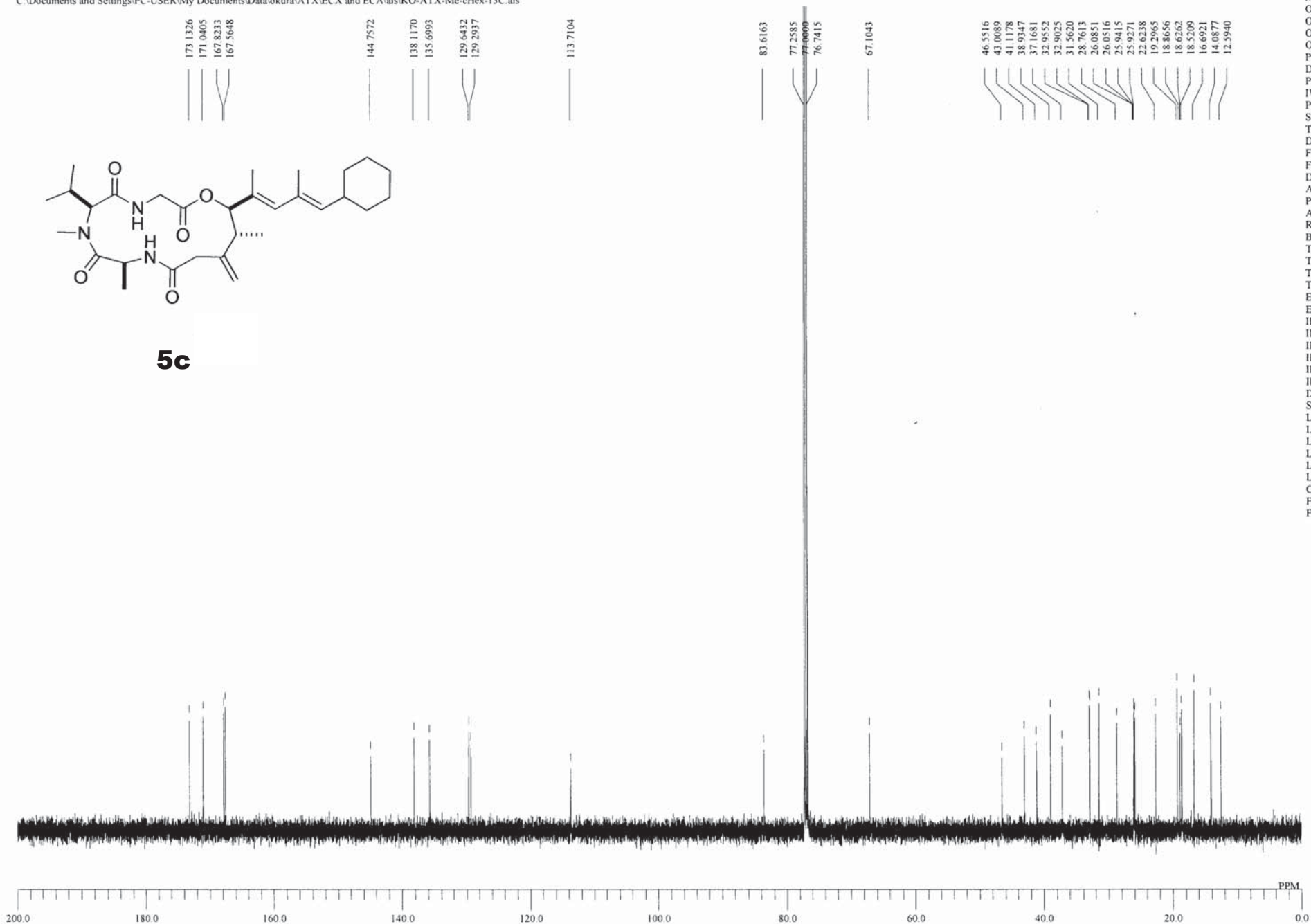


5c



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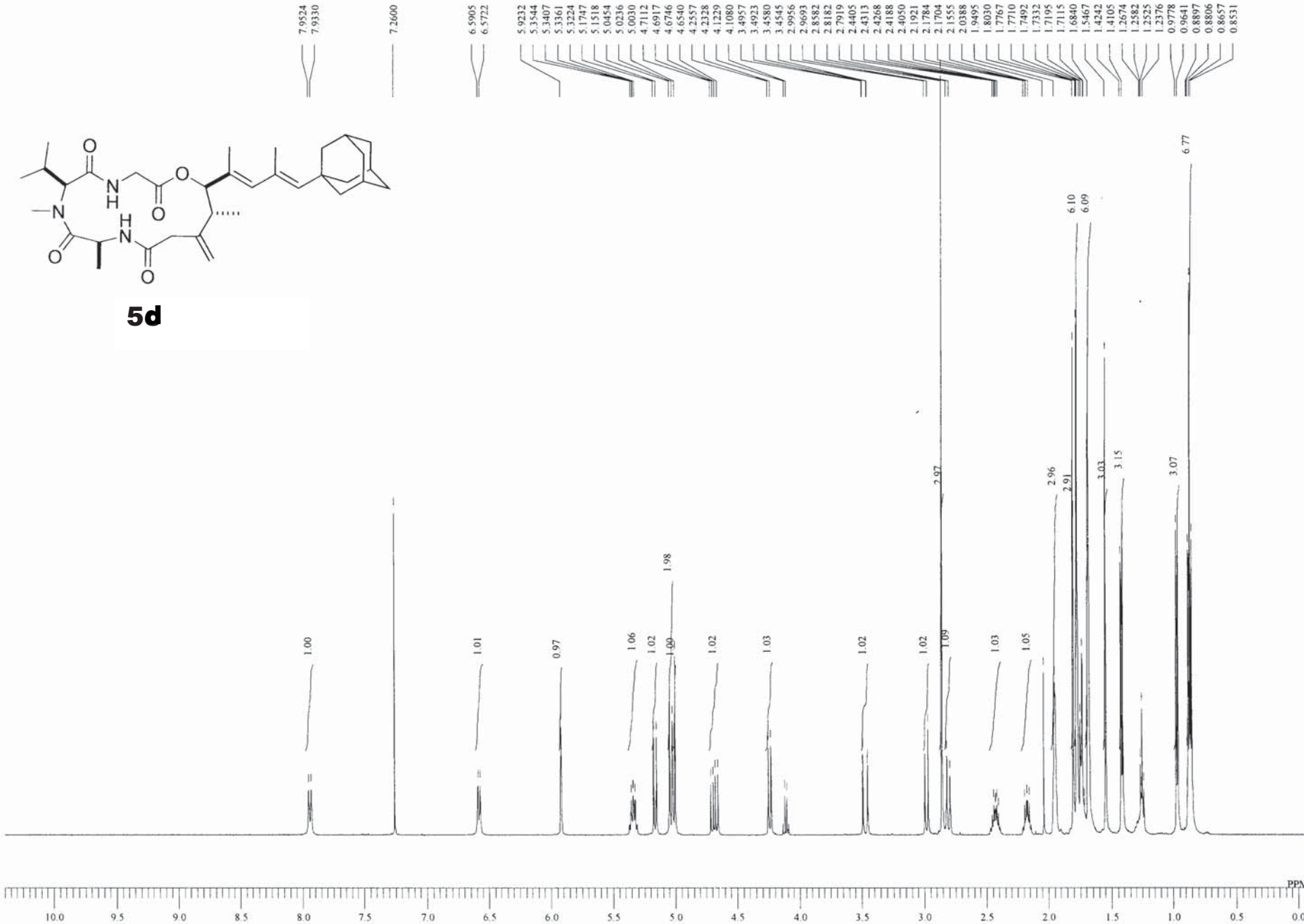
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5c

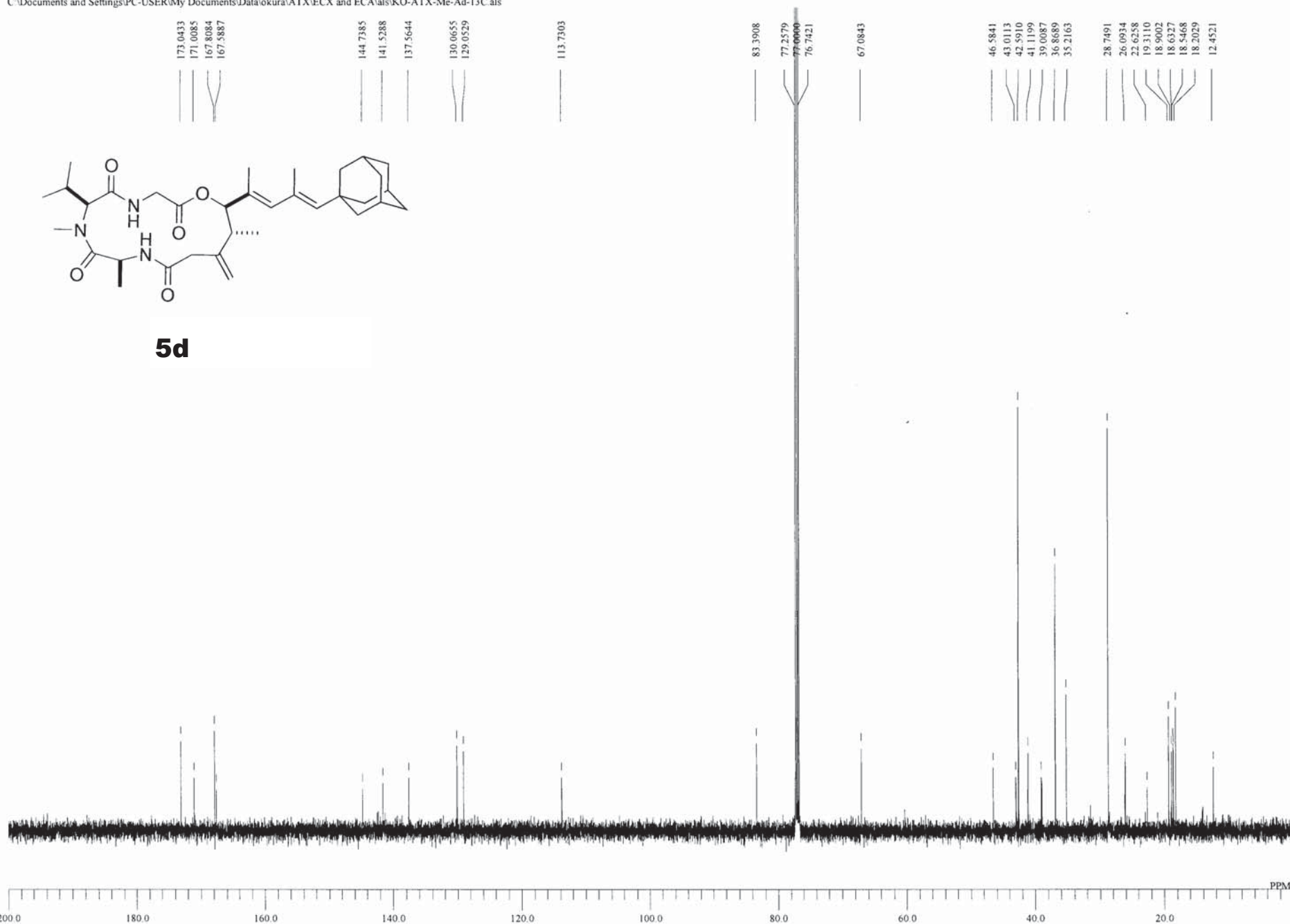
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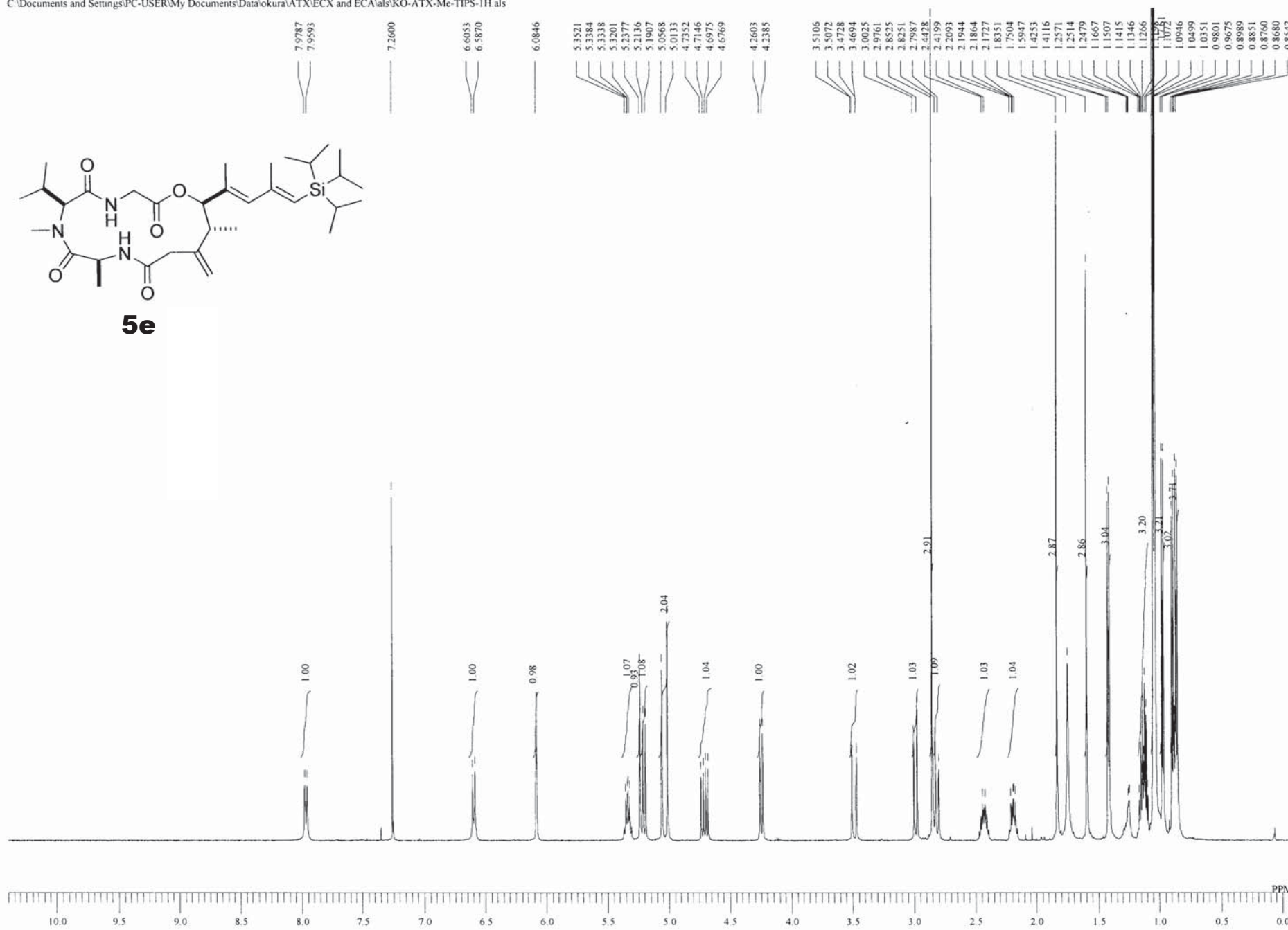
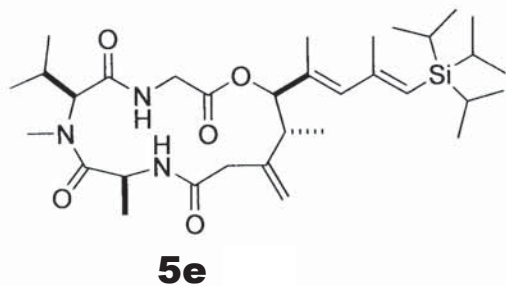
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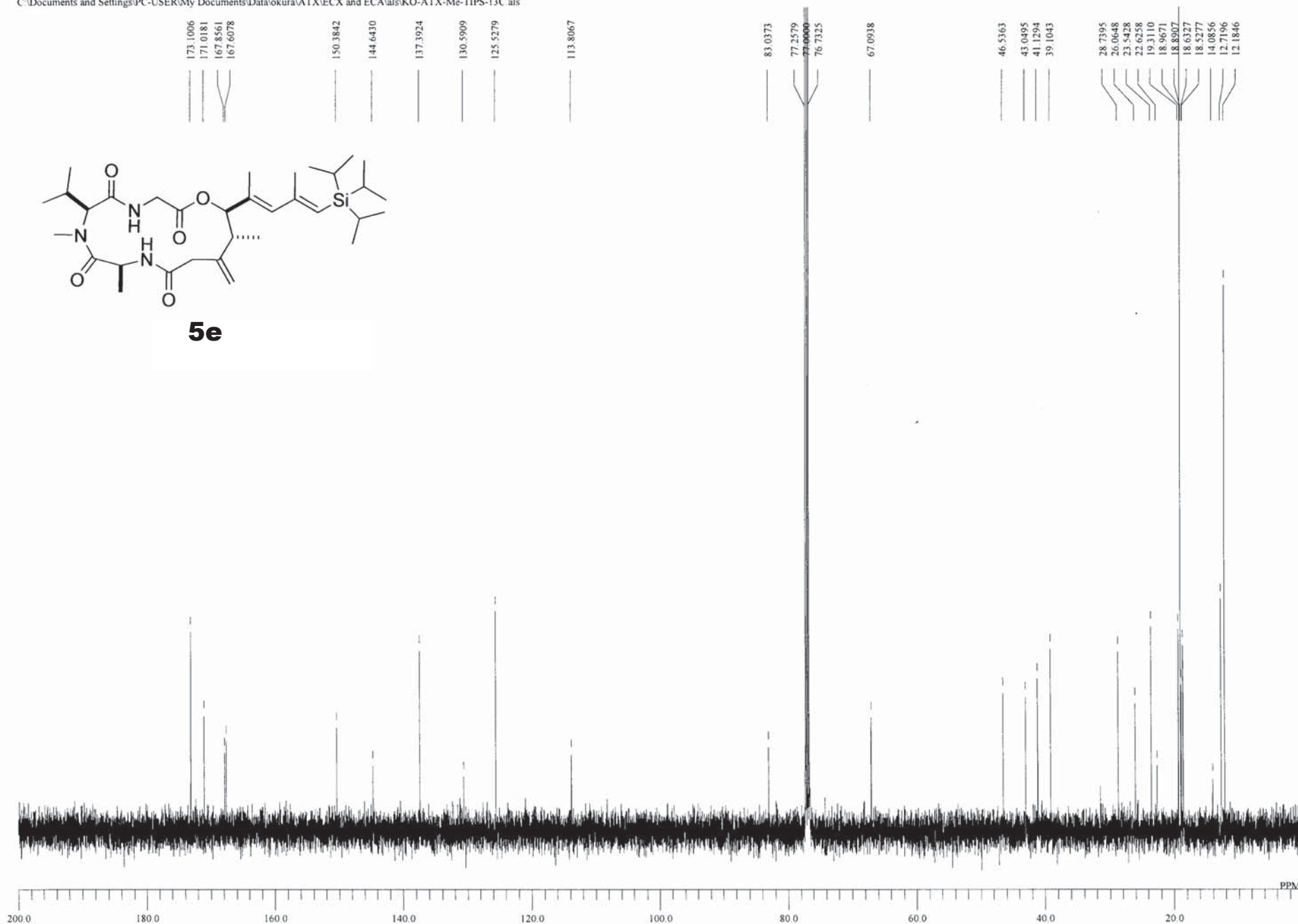
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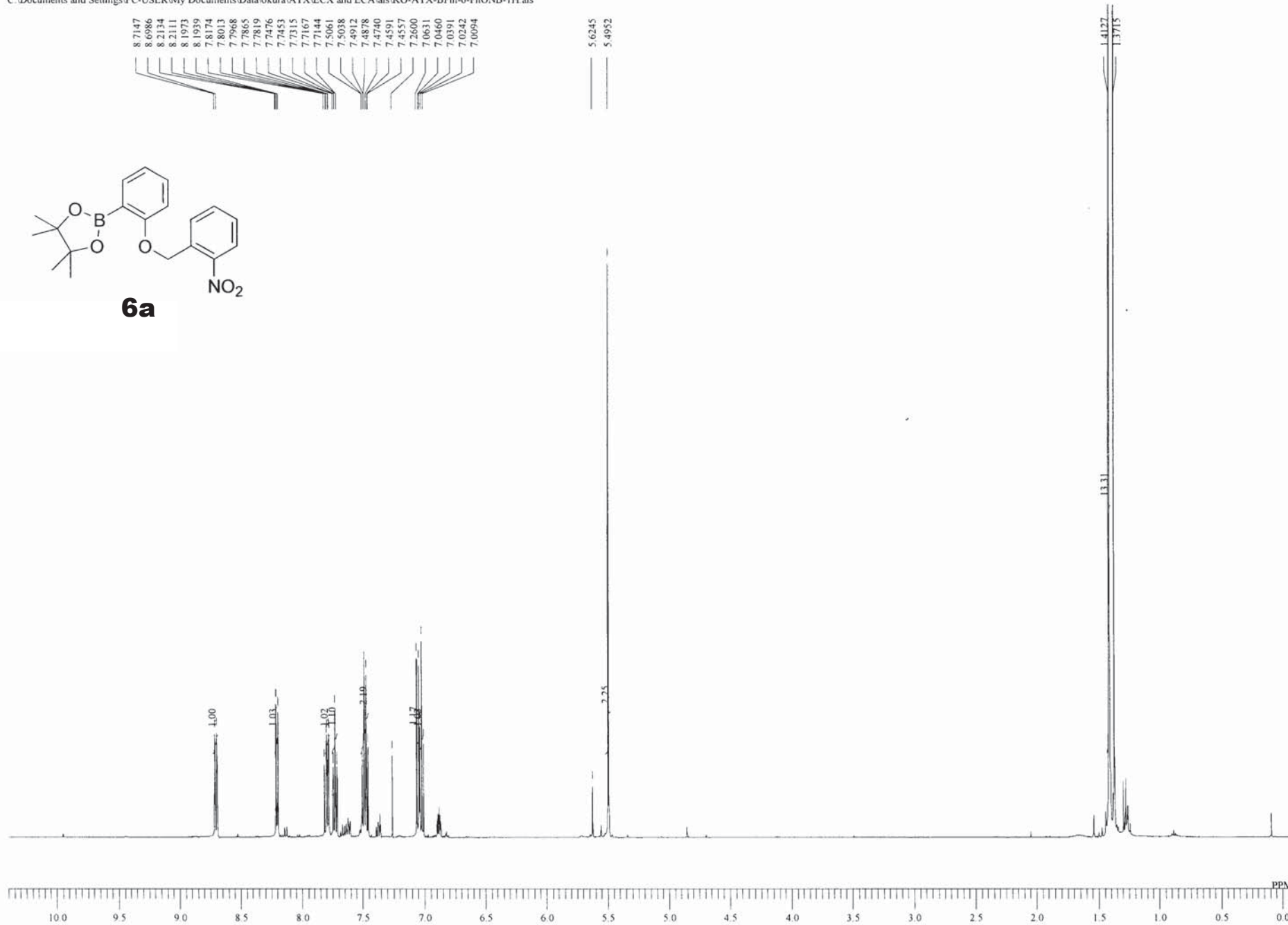
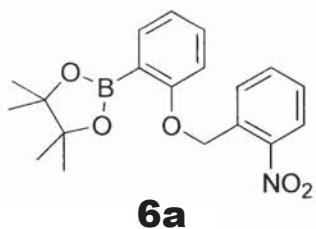
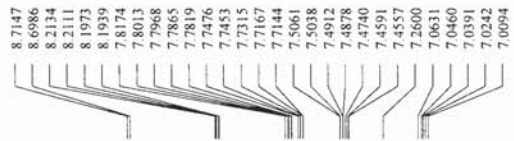
DATIM	23-08-2010 19:21:11
MENUF	
OBNUC	¹ H
OFR	490.15 MHz
OBSET	9.16 KHz
OBFIN	7.60 Hz
PW1	8.55 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	13107
SPO	13107
TIMES	8
DUMMY	1
FREQU	7352.83 Hz
FLT	37000 Hz
DELAY	13.52 usec
ACQTM	1.7826 sec
PD	1.5000 sec
ADBIT	16
RGAIN	38
BF	0.01 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	¹ H
IFR	490.15 MHz
IRSET	9.16 KHz
IRFIN	7.60 Hz
IRRPW	118 usec
IRATN	79
DFILE	KO-ATX-Me-TIPS-1H.als
SF	
LKSET	70.30 KHz
LKFIN	32.5 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	

C:\Documents and Settings\PC-USER\My Documents\Data\okura\ATX\ECX and ECA\als\KO-ATX-Me-TIPS-13C.als



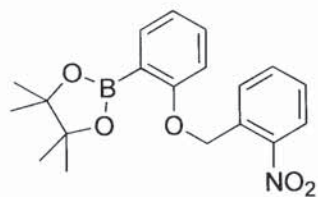
DATIM 23-08-2010 20:55:11
 MENUF
 OBNUC 13C
 OFR 123.26 MHz
 OBSSET 2.31 KHz
 OBFIN 6.71 Hz
 PW1 3.20 usec
 DEADT 0.00 usec
 PREDL 0.00000 msec
 IWT 1.0000 sec
 POINT 26214
 SPO 26214
 TIMES 512
 DUMMY 4
 FREQU 30863.73 Hz
 FLT 155000 Hz
 DELAY 21.06 usec
 ACQTM 0.8493 sec
 PD 10.0000 sec
 ADBIT 16
 RGAIN 60
 BF 0.10 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD single_pulse_dec
 EXPCM
 IRNUC 1H
 IFR 490.15 MHz
 IRSET 9.16 KHz
 IRFIN 7.60 Hz
 IRRPW 92 usec
 IRATN 79
 DFILE KO-ATX-Me-TIPS-13C.a
 SF
 LKSET 70.30 KHz
 LKFIN 32.5 Hz
 LKLEV 0
 LGAIN 0
 LKPHS 0
 LKSIG 0
 CSPED 0 Hz
 FILDC
 FILDF

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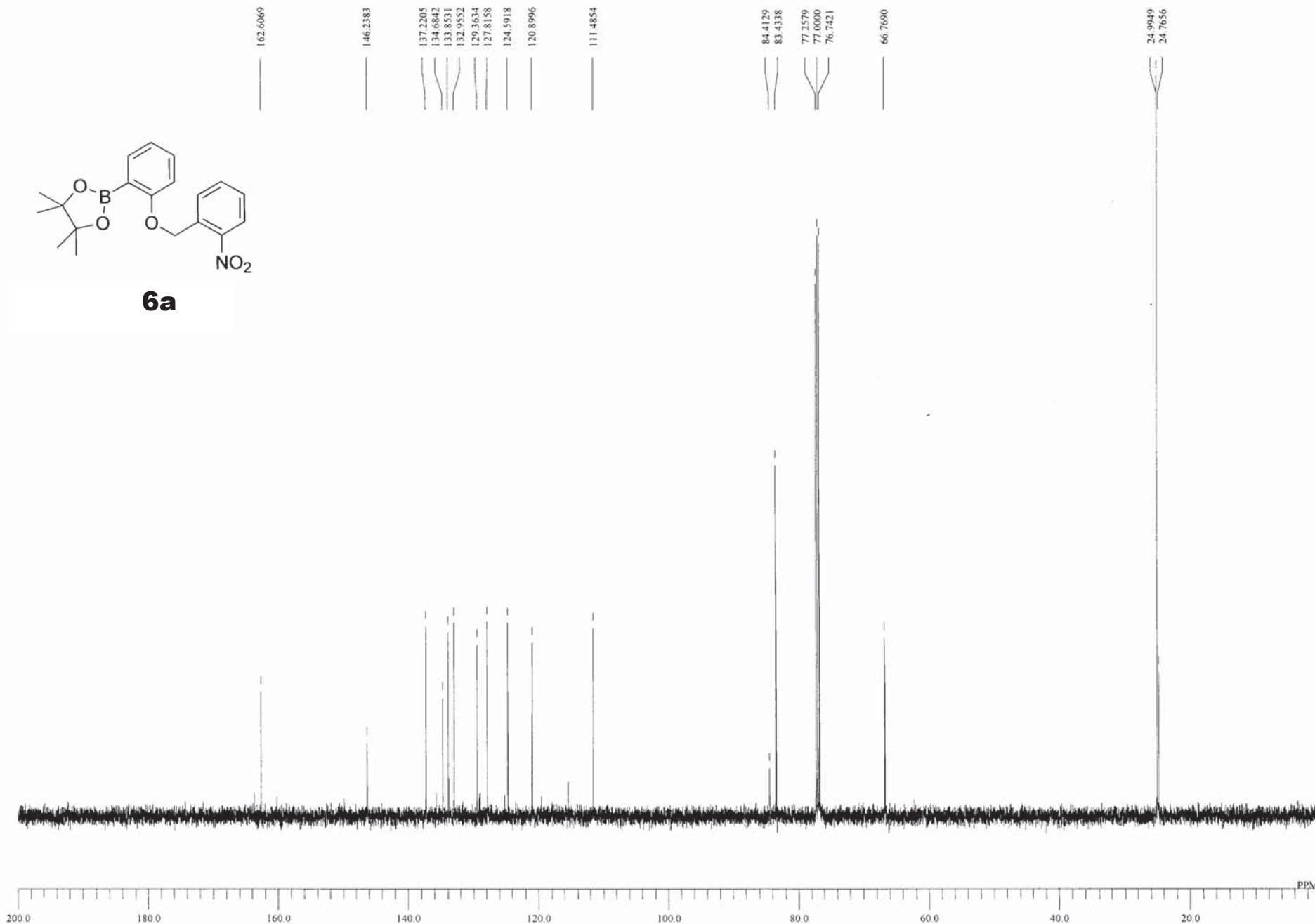


DATIM	02-03-2011 21:25:44
MENUF	
OBNUC	¹ H
OFR	490.15 MHz
OBSET	9.16 KHz
OBFIN	7.60 Hz
PW1	8.55 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	13107
SPO	13107
TIMES	8
DUMMY	1
FREQU	7352.83 Hz
FLT	37000 Hz
DELAY	13.52 usec
ACQTM	1.7826 sec
PD	1.5000 sec
ADBIT	16
RGAIN	30
BF	0.01 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse ex2
EXPCM	
IRNUC	¹ H
IFR	490.15 MHz
IRSET	9.16 KHz
IRFIN	7.60 Hz
IRRPW	118 usec
IRATN	79
DFILE	KO-ATX-BPin-o-PhONB
SF	
LKSET	70.30 KHz
LKFIN	32.5 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	

C:\Documents and Settings\PC-USER\My Documents\Data\okura\ATX\ECX and ECA\als\KO-ATX-BPin-o-PhONB-13C.als

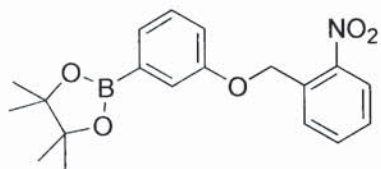
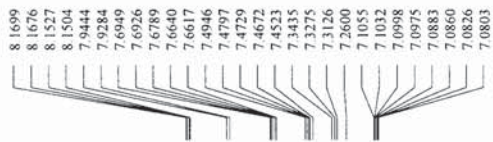


6a

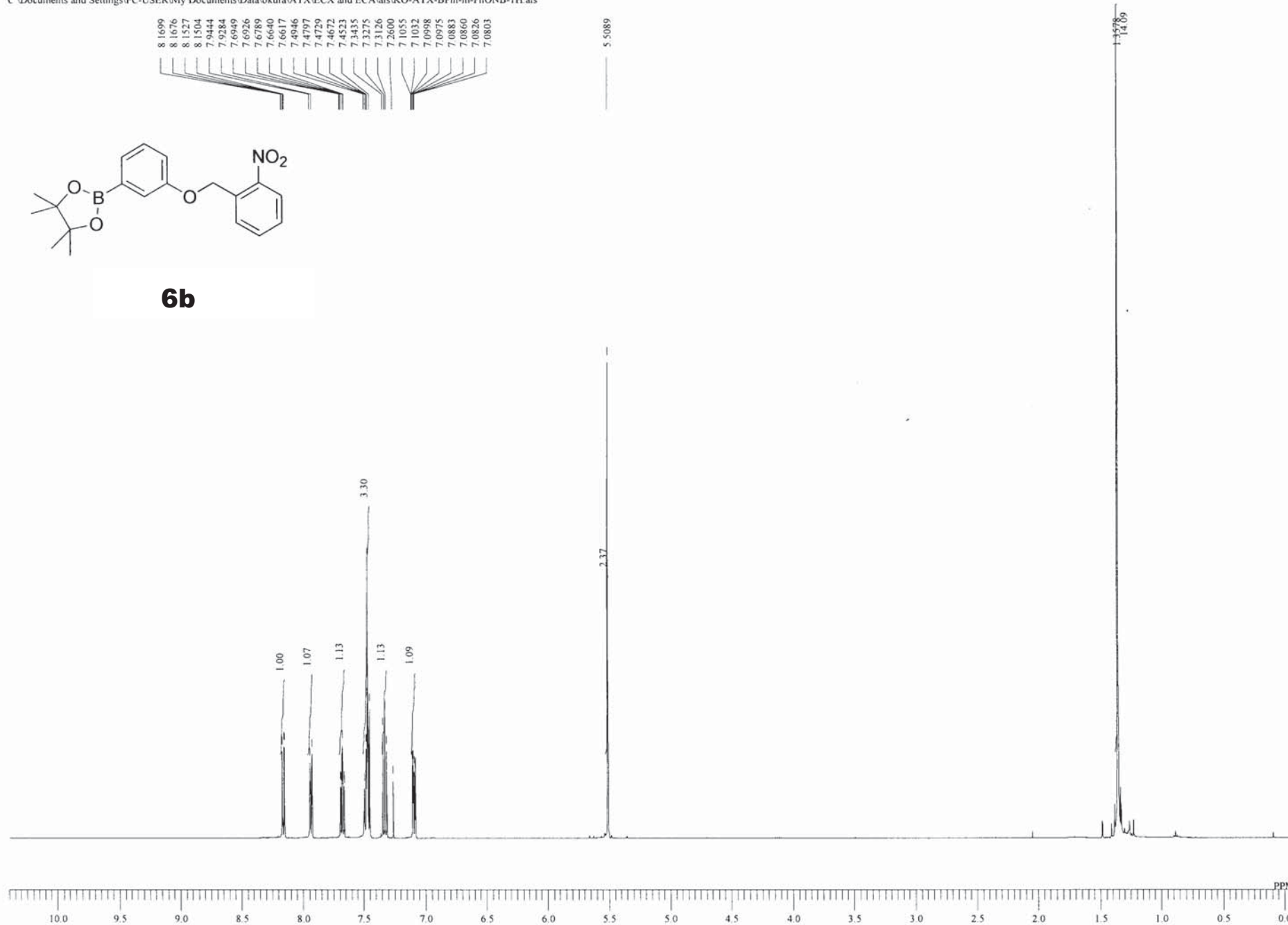


DATIM	02-03-2011 18:32:03
MENUP	
OBNUC	13C
OFR	123.26 MHz
OBSET	2.31 KHz
OBFIN	6.71 Hz
PW1	3.20 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	52428
SPO	52428
TIMES	16
DUMMY	4
FREQU	30863.73 Hz
FLT	155000 Hz
DELAY	21.06 usec
ACQTM	0.8493 sec
PD	10.0000 sec
ADBIT	16
RGAIN	60
BF	1.00 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	490.15 MHz
IRSET	9.16 KHz
IRFIN	7.60 Hz
IRRPW	92 usec
IRATN	79
DFILE	KO-ATX-BPin-o-PhONB
SF	
LKSET	70.30 KHz
LKFIN	32.5 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	

C:\Documents and Settings\PC-USER\My Documents\Data\okura\ATX\ECX and ECA\als\KO-ATX-BPin-m-PhONB-1H.als

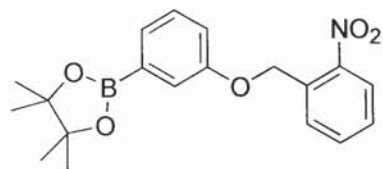


6b

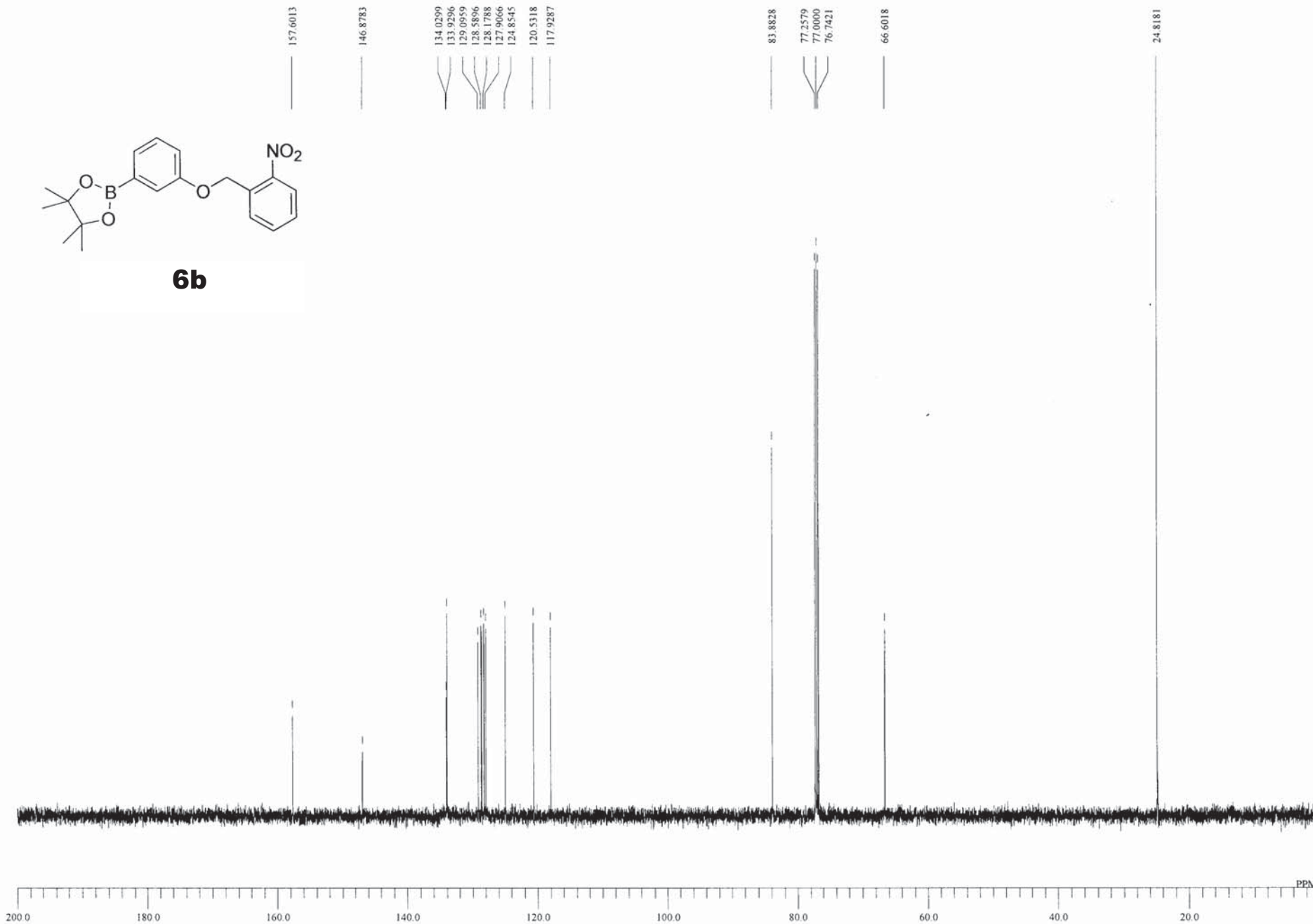


DATIM 02-03-2011 21:36:31
MENUF
OBNUC 1H
OFR 490.15 MHz
OBSET 9.16 KHz
OBFIN 7.60 Hz
PW1 8.55 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 13107
SPO 13107
TIMES 8
DUMMY 1
FREQU 7352.83 Hz
FLT 37000 Hz
DELAY 13.52 usec
ACQTM 1.7826 sec
PD 1.5000 sec
ADBIT 16
RGAIN 30
BF 0.01 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 490.15 MHz
IRSET 9.16 KHz
IRFIN 7.60 Hz
IRRPW 118 usec
IRATN 79
DFILE KO-ATX-BPin-m-PhONE
SF
LKSET 70.30 KHz
LKFIN 32.5 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF

C:\Documents and Settings\PC-USER\My Documents\Data\okura\ATX\ECX and ECA\als\KO-ATX-BPin-m-PhONB-13C.als

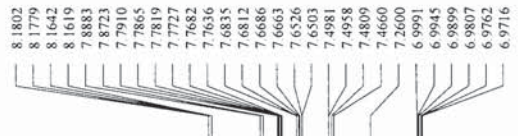
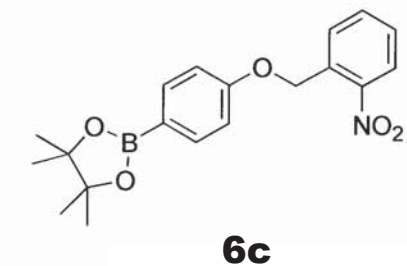


6b



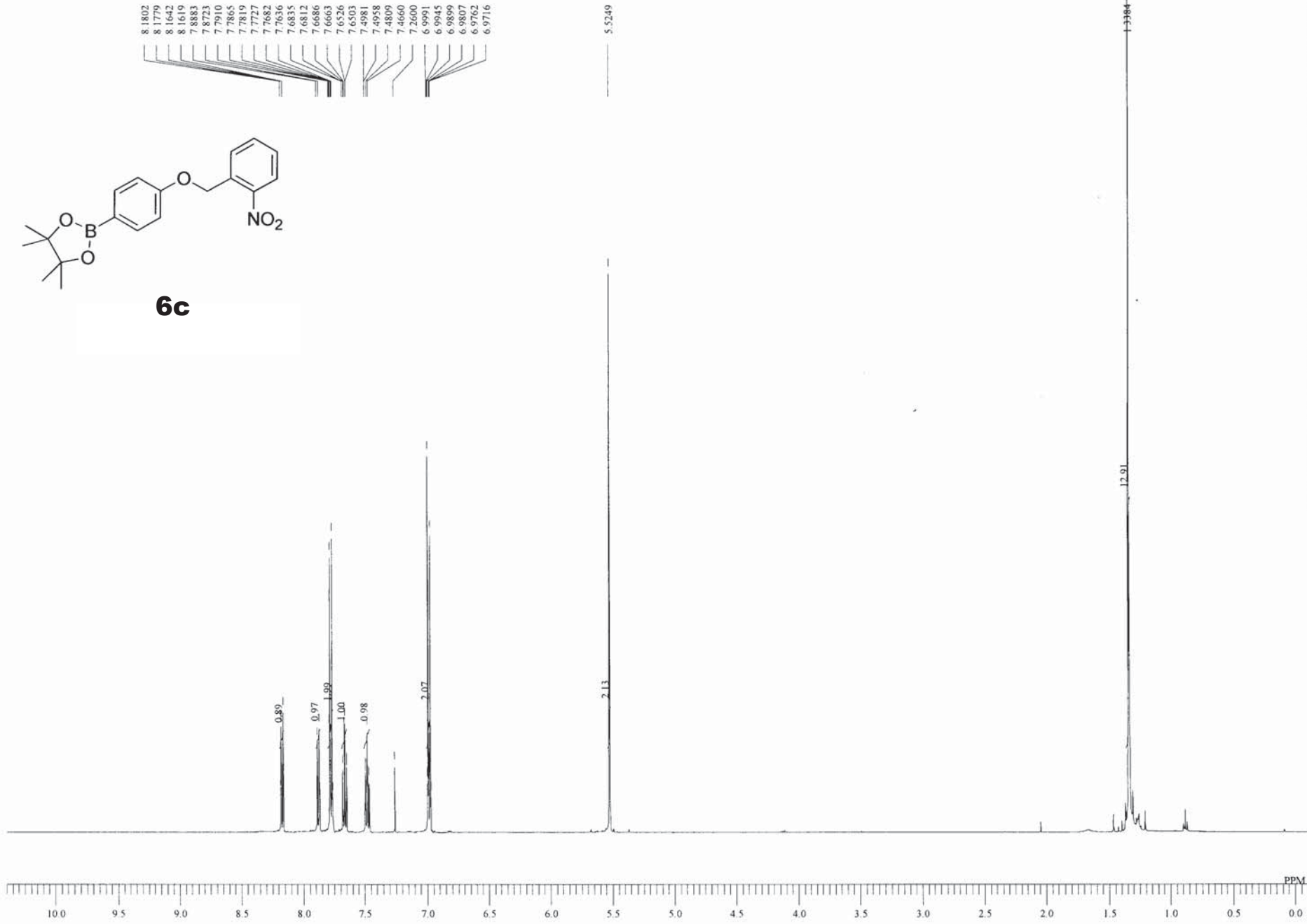
DATIM	02-03-2011 18:43:59
MENUF	
OBNUC	13C
OFR	123.26 MHz
OBSET	2.31 KHz
OBFIN	6.71 Hz
PW1	3.20 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	52428
SPO	52428
TIMES	16
DUMMY	4
FREQU	30863.73 Hz
FLT	155000 Hz
DELAY	21.06 usec
ACQTM	0.8493 sec
PD	10.0000 sec
ADBIT	16
RGAIN	60
BF	1.00 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	13H
IFR	490.15 MHz
IRSET	9.16 KHz
IRFIN	7.60 Hz
IRRPW	92 usec
IRATN	79
DFILE	KO-ATX-BPin-m-PhONE
SF	
LKSET	70.30 KHz
LKFIN	32.5 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	

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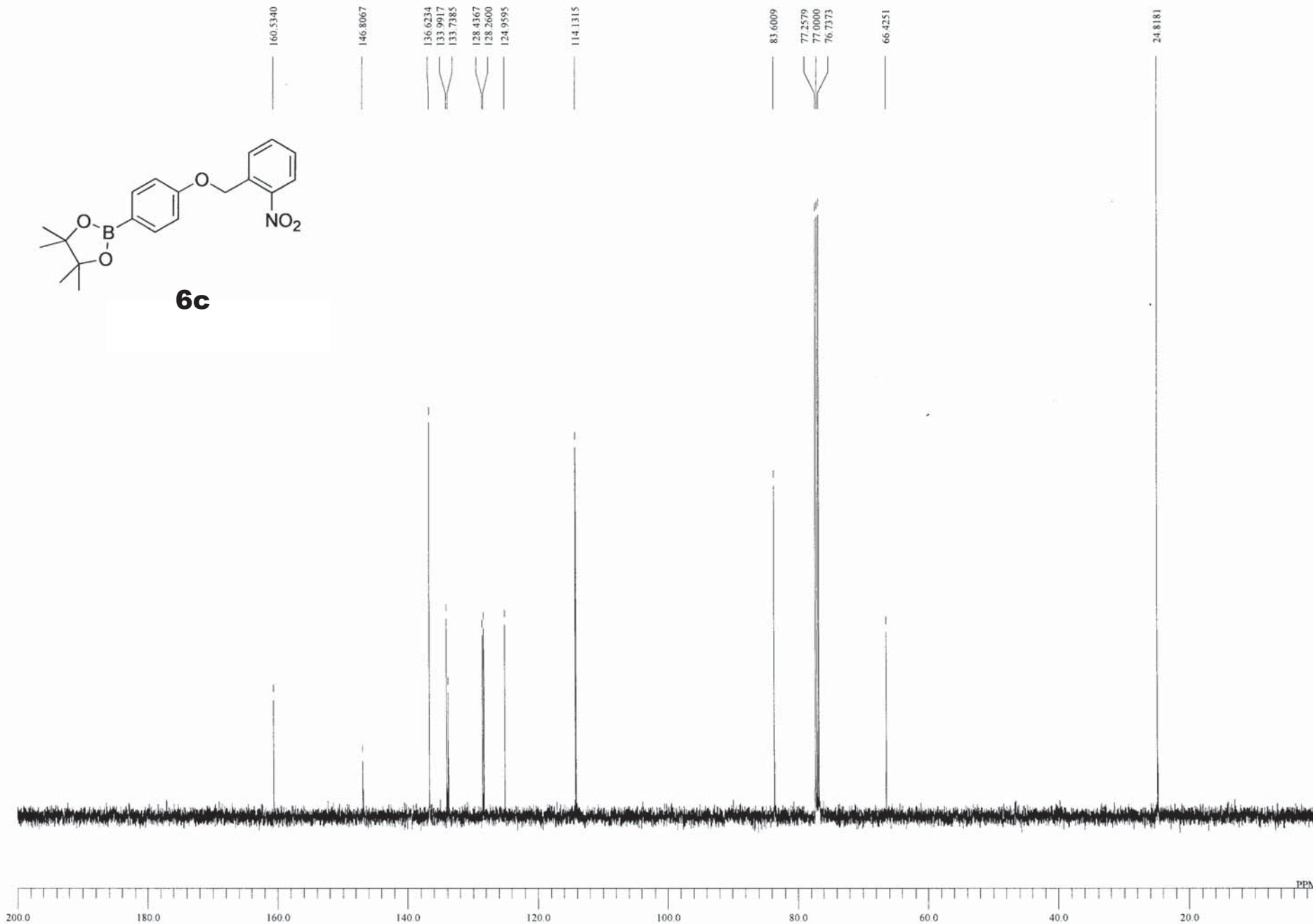
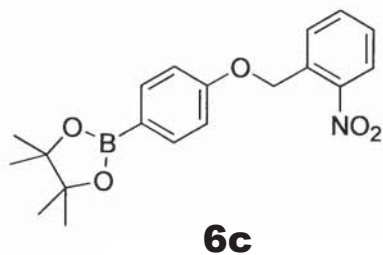
5.5249

1.3384



DATIM	02-03-2011 21:44:37
MENUF	
OBNUC	1H
OFR	490.15 MHz
OBSET	9.16 KHz
OBFIN	7.60 Hz
PW1	8.55 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	13107
SPO	13107
TIMES	8
DUMMY	1
FREQU	7352.83 Hz
FLT	37000 Hz
DELAY	13.52 usec
ACQTM	1.7826 sec
PD	1.5000 sec
ADBIT	16
RGAIN	30
BF	0.01 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	1H
IFR	490.15 MHz
IRSET	9.16 KHz
IRFIN	7.60 Hz
IRRPW	118 usec
IRATN	79
DFILE	KO-ATX-Bpin-p-PhONB-
SF	
LKSET	70.30 KHz
LKFIN	32.5 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	

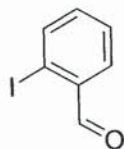
C:\Documents and Settings\PC-USER\My Documents\Data\okura\ATX\ECX and ECA\als\KO-ATX-Bpin-p-PhONB-13C.als



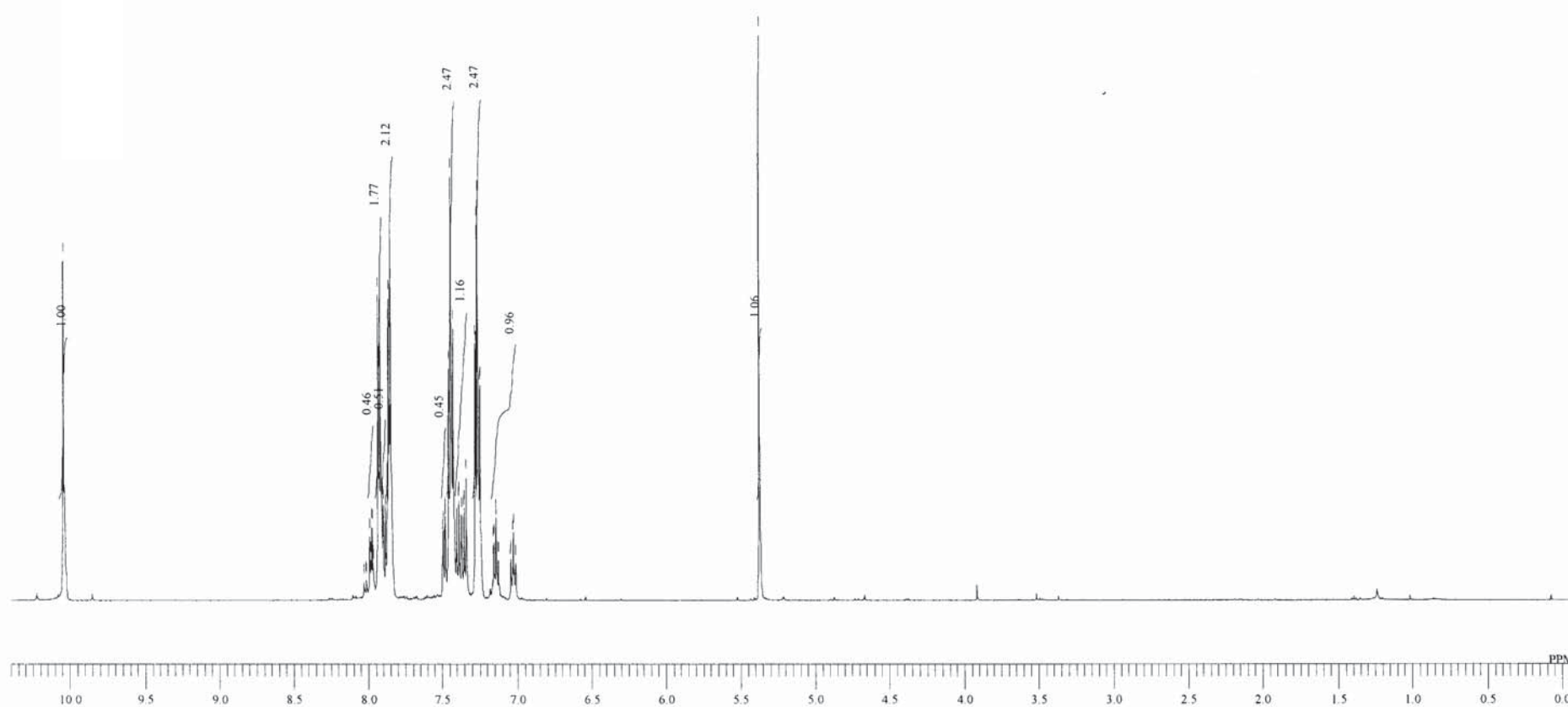
DATIM	02-03-2011 20:54:41
MENUF	
OBNUC	13C
OFR	123.26 MHz
OBSET	2.31 KHz
OBFIN	6.71 Hz
PW1	3.20 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	52428
SPO	52428
TIMES	16
DUMMY	4
FREQU	30863.73 Hz
FLT	155000 Hz
DELAY	21.06 usec
ACQTM	0.8493 sec
PD	10.0000 sec
ADBIT	16
RGAIN	60
BF	1.00 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	490.15 MHz
IRSET	9.16 KHz
IRFIN	7.60 Hz
IRRPW	92 usec
IRATN	79
DFILE	KO-ATX-Bpin-p-PhONB
SF	
LKSET	70.30 KHz
LKFIN	32.5 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	

C:\Documents and Settings\PC-USER\My Documents\Data\okura\ATX\ECX and ECA\als\KO-ATX-o-I-CHO-1H.als

10.0423
8.0314
8.0291
8.0154
8.0131
7.9925
7.9902
7.9845
7.9810
7.9765
7.9742
7.9684
7.9650
7.9353
7.9330
7.9192
7.9169
7.9021
7.8986
7.8872
7.8838
7.8723
7.8700
7.8654
7.8609
7.8563
7.8540
7.8494
7.8460
7.4981
7.4946
7.4820
7.4797
7.4569
7.4420
7.4260
7.4054
7.4031
7.3893
7.3870
7.3745
7.3710
7.3687
7.3550
7.3527
7.3390
7.2806
7.2760
7.2646
7.2611
7.2497
7.2451
7.1593
7.1559
7.1433
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7.0151
7.0116
5.3727

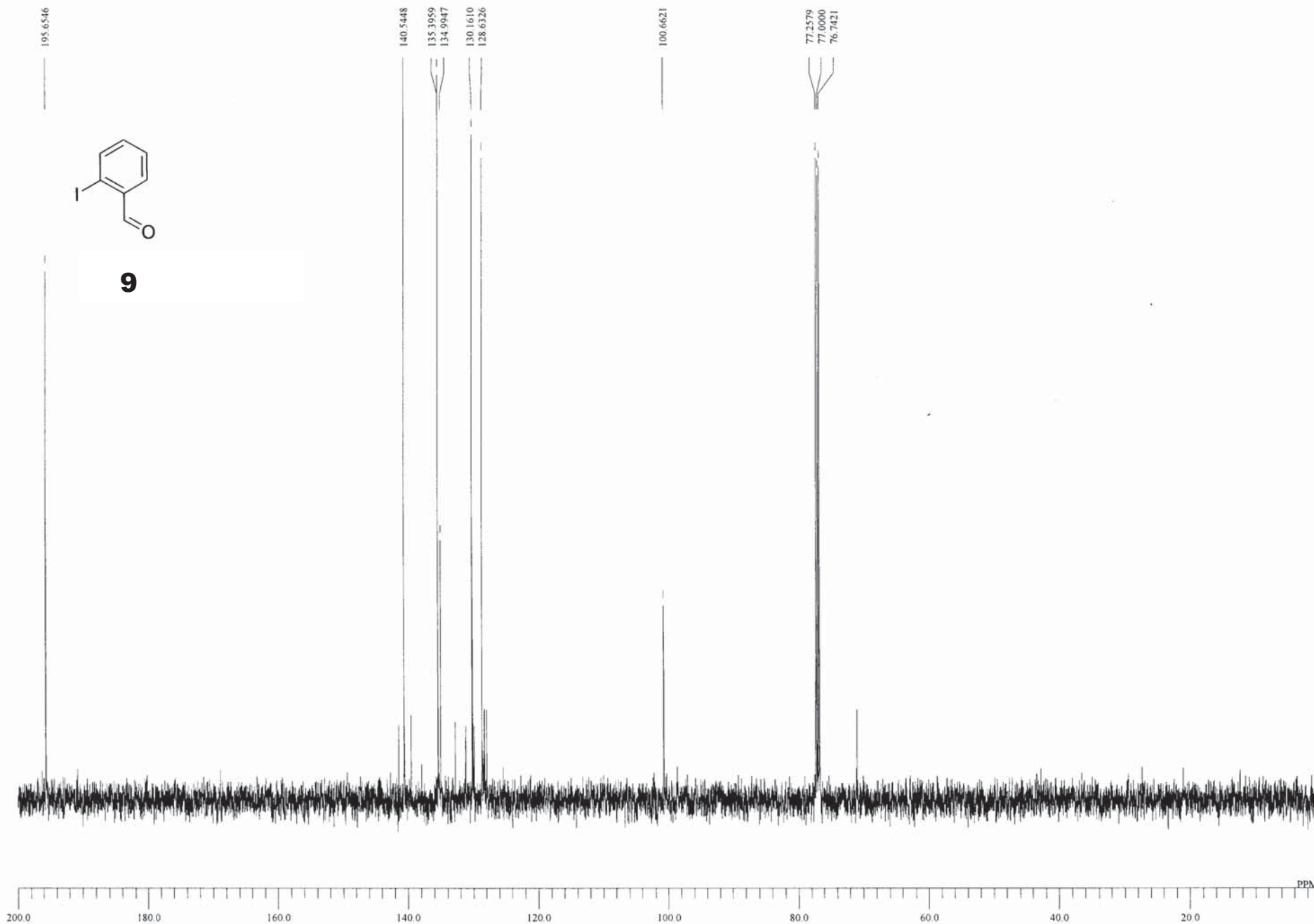


9



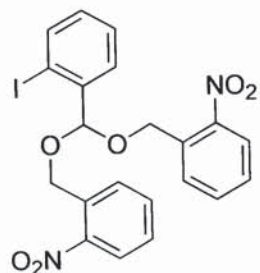
DATIM 08-03-2011 12:55:47
MENUF
OBNUC 1H
OFR 490.15 MHz
OBSET 9.16 KHz
OBFIN 7.60 Hz
PW1 8.55 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 13107
SPO 13107
TIMES 8
DUMMY 1
FREQU 7352.83 Hz
FLT 37000 Hz
DELAY 13.52 usec
ACQTM 1.7826 sec
PD 1.5000 sec
ADBIT 16
RGAIN 40
BF 0.01 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse ex2
EXPCM
IRNUC 1H
IFR 490.15 MHz
IRSET 9.16 KHz
IRFIN 7.60 Hz
IRRPW 118 usec
IRATN 79
DFILE KO-ATX-o-I-CHO-1H.als
SF
LKSET 70.30 KHz
LKFIN 32.5 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF

C:\Documents and Settings\PC-USER\My Documents\Data\okura\ATX\ECX and ECA\als\KO-ATX-o-I-CHO-13C.als

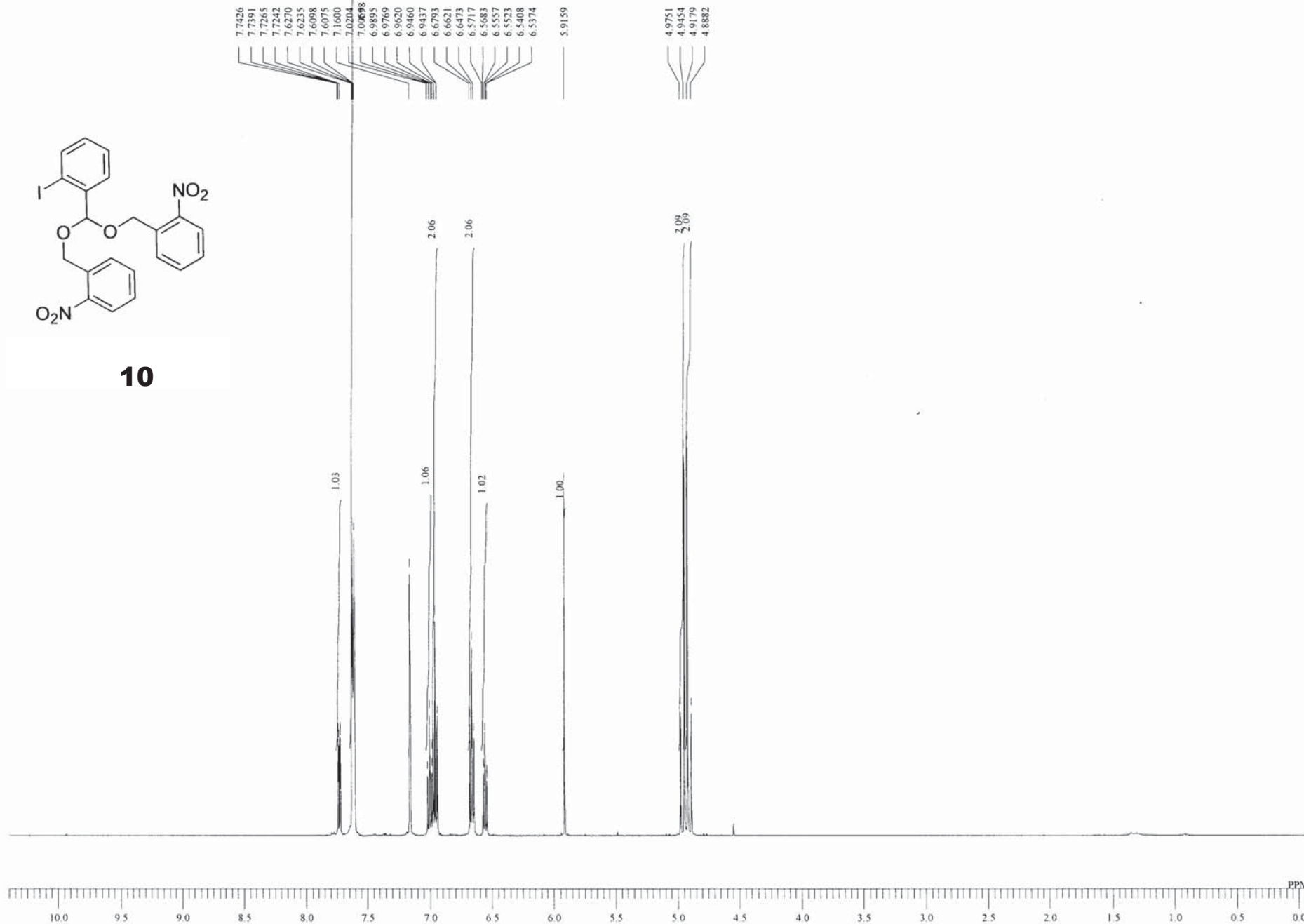


DATIM	08-03-2011 12:59:29
MENUF	
OBNUC	13C
OFR	123.26 MHz
OBSET	2.31 KHz
OBFIN	6.71 Hz
PW1	3.20 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	26214
SPO	26214
TIMES	16
DUMMY	4
FREQU	30863.73 Hz
FLT	155000 Hz
DELAY	21.06 usec
ACQTM	0.8493 sec
PD	10.0000 sec
ADBIT	16
RGAIN	60
BF	2.00 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	490.15 MHz
IRSET	9.16 KHz
IRFIN	7.60 Hz
IRRPW	92 usec
IRATN	79
DFILE	KO-ATX-o-I-CHO-13C.a
SF	
LKSET	70.30 KHz
LKFIN	32.5 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	

C:\Documents and Settings\PC-USER\My Documents\Data\okura\ATX\ECX and ECA\als\KO-ATX-1-o-CH(ONB)2-1H.als

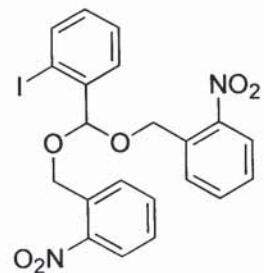


10

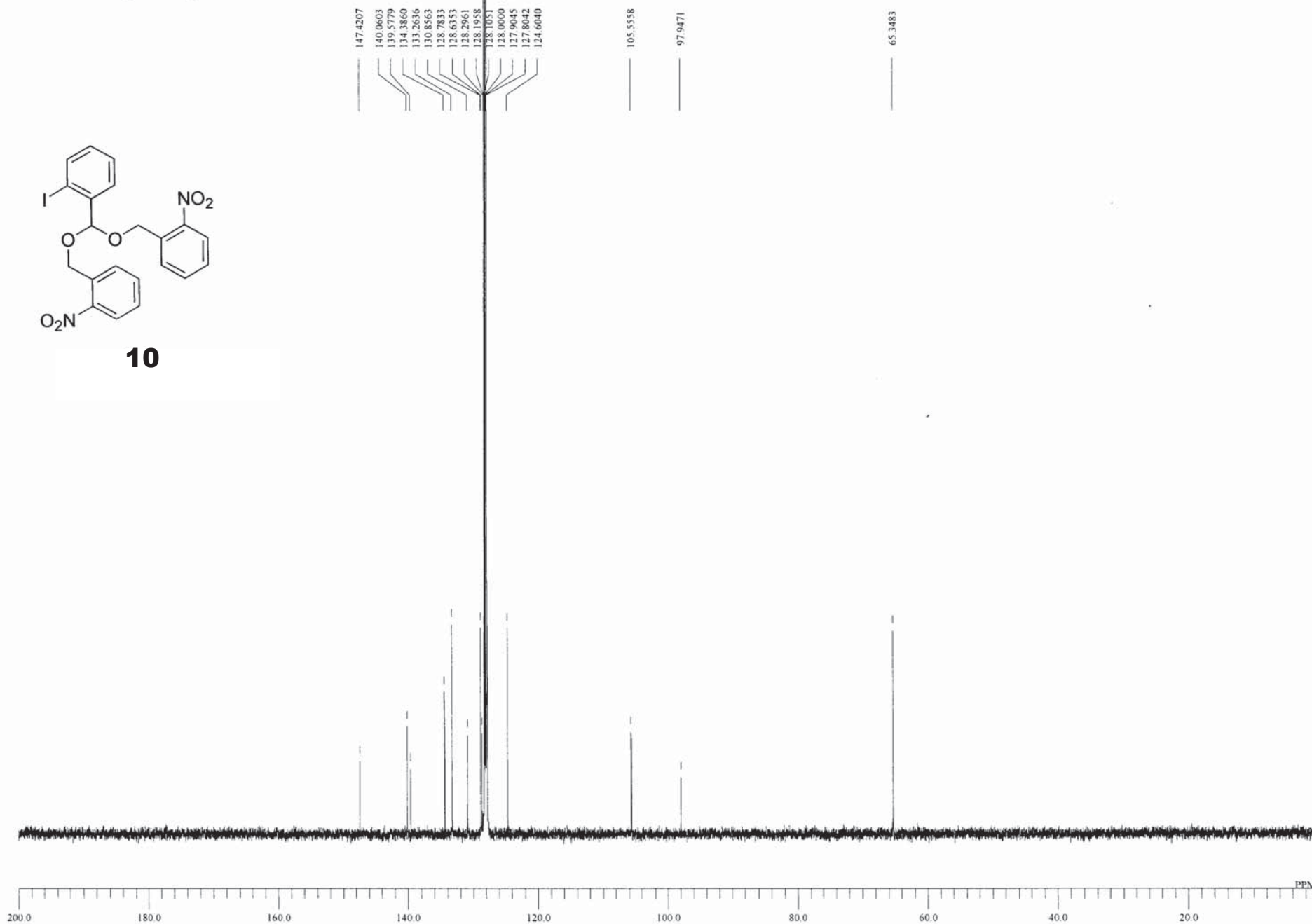


DATIM	04-03-2011 16:43:00
MENUF	
OBNUC	¹ H
OFR	490.15 MHz
OBSET	9.16 KHz
OBFIN	7.60 Hz
PW1	8.55 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	13107
SPO	13107
TIMES	8
DUMMY	1
FREQU	7352.83 Hz
FLT	37000 Hz
DELAY	13.52 usec
ACQTM	1.7826 sec
PD	5.0000 sec
ADBIT	16
RGAIN	34
BF	0.01 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse ex2
EXPCM	
IRNUC	¹ H
IFR	490.15 MHz
IRSET	9.16 KHz
IRFIN	7.60 Hz
IRRPW	118 usec
IRATN	79
DFILE	KO-ATX-1-o-CH(ONB)2-
SF	
LKSET	70.30 KHz
LKFIN	29.2 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	

C:\Documents and Settings\PC-USER\My Documents\Data\okura\ATX\ECX and ECA\als\KO-ATX-I-o-CH(ONB)2-13C.als

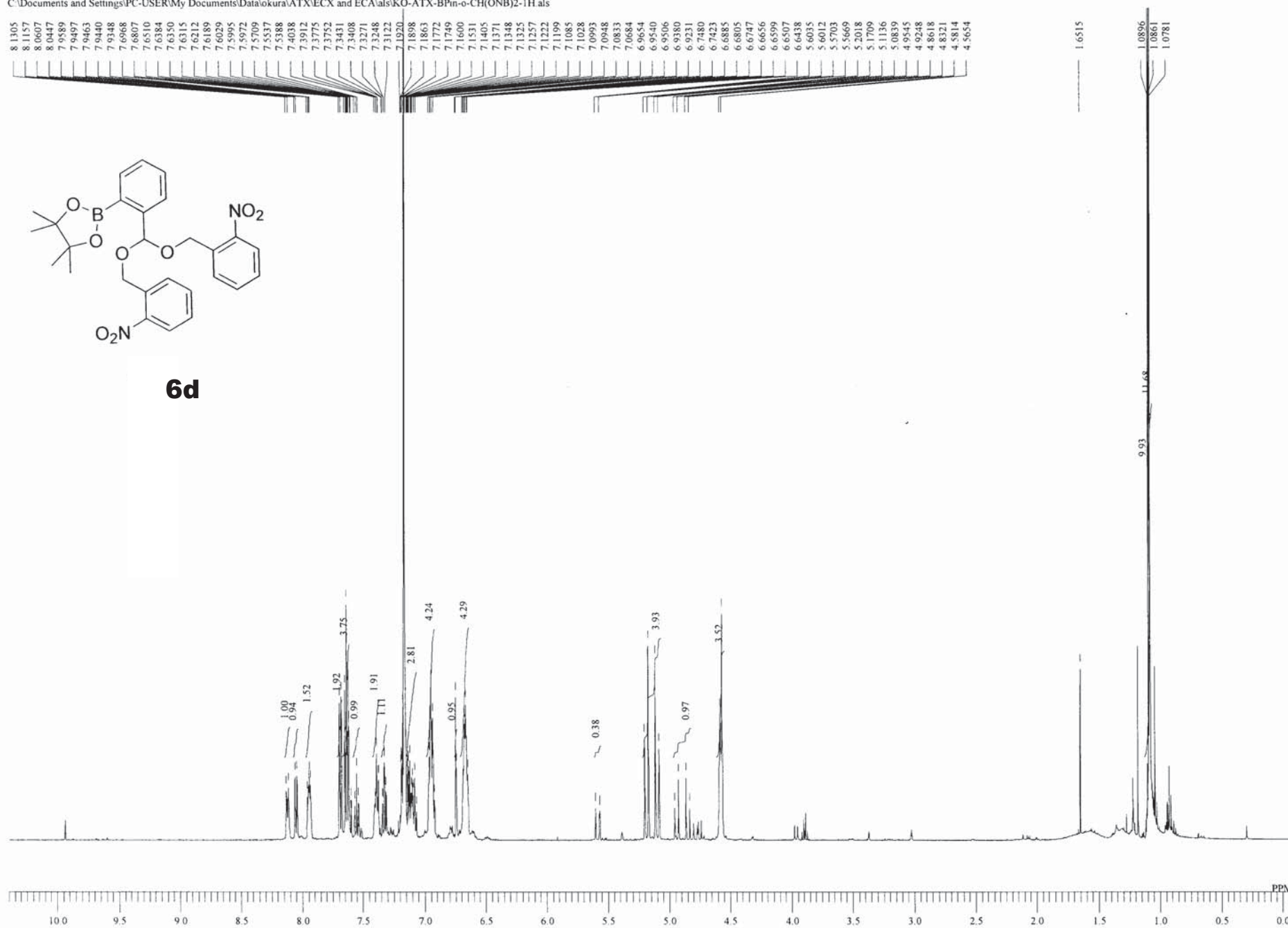


10



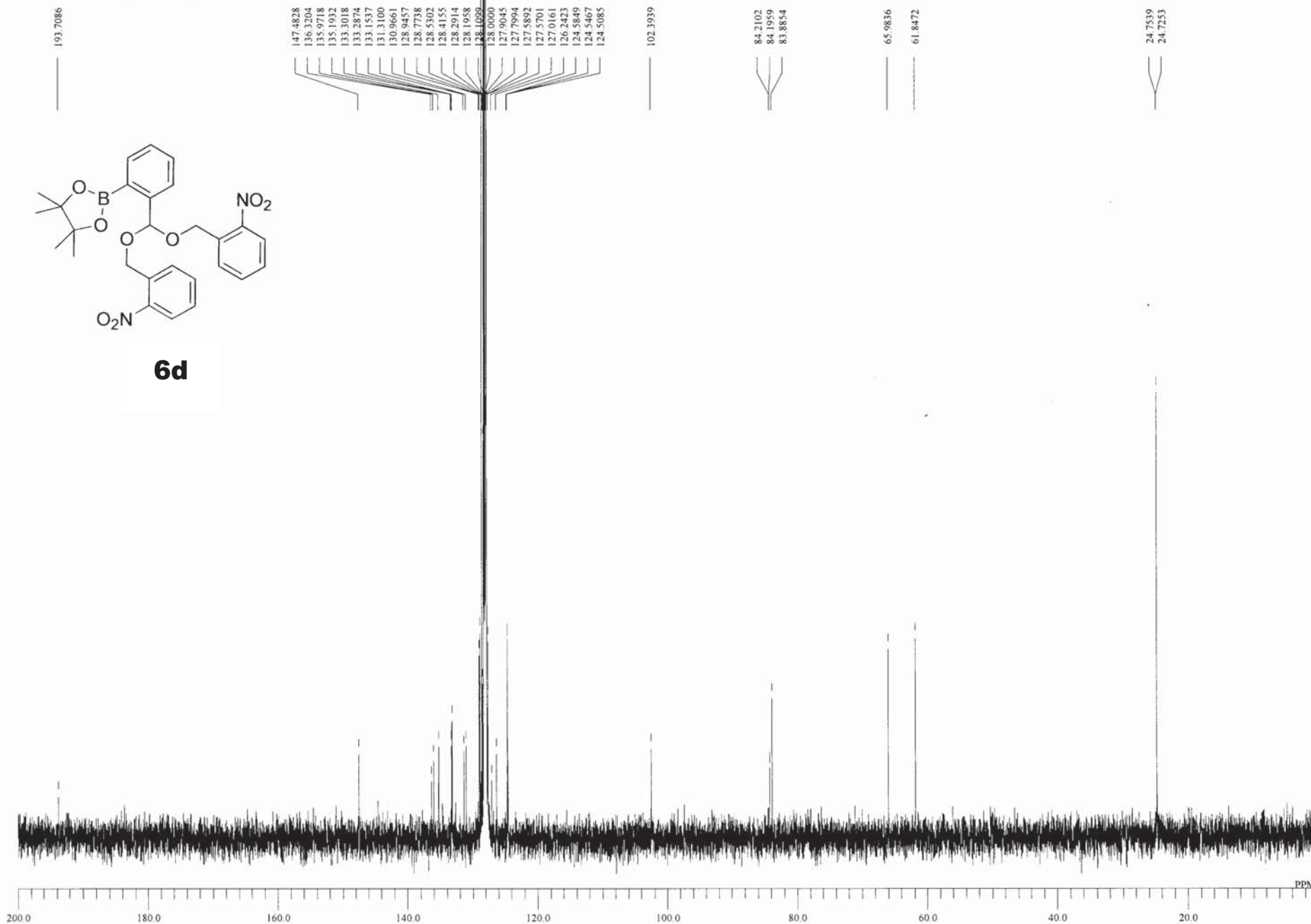
DATIM	04-03-2011 16:49:37
MENUF	
OBNUC	13C
OFR	123.26 MHz
OBSET	2.31 KHz
OBFIN	6.71 Hz
PW1	3.20 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	52428
SPO	52428
TIMES	32
DUMMY	4
FREQU	30863.73 Hz
FLT	155000 Hz
DELAY	21.06 usec
ACQTM	0.8493 sec
PD	10.0000 sec
ADBIT	16
RGAIN	60
BF	1.00 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	490.15 MHz
IRSET	9.16 KHz
IRFIN	7.60 Hz
IRRPW	92 usec
IRATN	79
DFILE	KO-ATX-I-o-CH(ONB)2-
SF	
LKSET	70.30 KHz
LKFIN	29.2 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	

C:\Documents and Settings\PC-USER\My Documents\Data\okura\ATX\ECX and ECA\als\KO-ATX-BPin-o-CH(ONB)2-1H.als



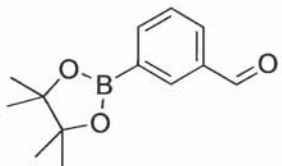
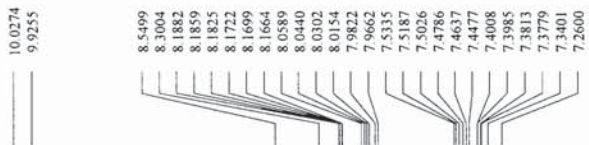
DATIM	04-03-2011 17:15:42
MENUF	
OBNUC	¹ H
OFR	490.15 MHz
OBSET	9.16 KHz
OBFIN	7.60 Hz
PWI	8.55 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	13107
SPO	13107
TIMES	8
DUMMY	1
FREQU	7352.83 Hz
FLT	37000 Hz
DELAY	13.52 usec
ACQTM	1.7826 sec
PD	5.0000 sec
ADBIT	16
RGAIN	32
BF	0.01 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse ex2
EXPCM	
IRNUC	¹ H
IFR	490.15 MHz
IRSET	9.16 KHz
IRFIN	7.60 Hz
IRRPW	118 usec
IRATN	79
DFILE	KO-ATX-BPin-o-CH(ON
SF	
LKSET	70.30 KHz
LKFIN	29.2 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	

C:\Documents and Settings\PC-USER\My Documents\Data\okura\ATX\ECX and ECA\als\KO-ATX-BPin-o-CH(ONB)2-13C.als

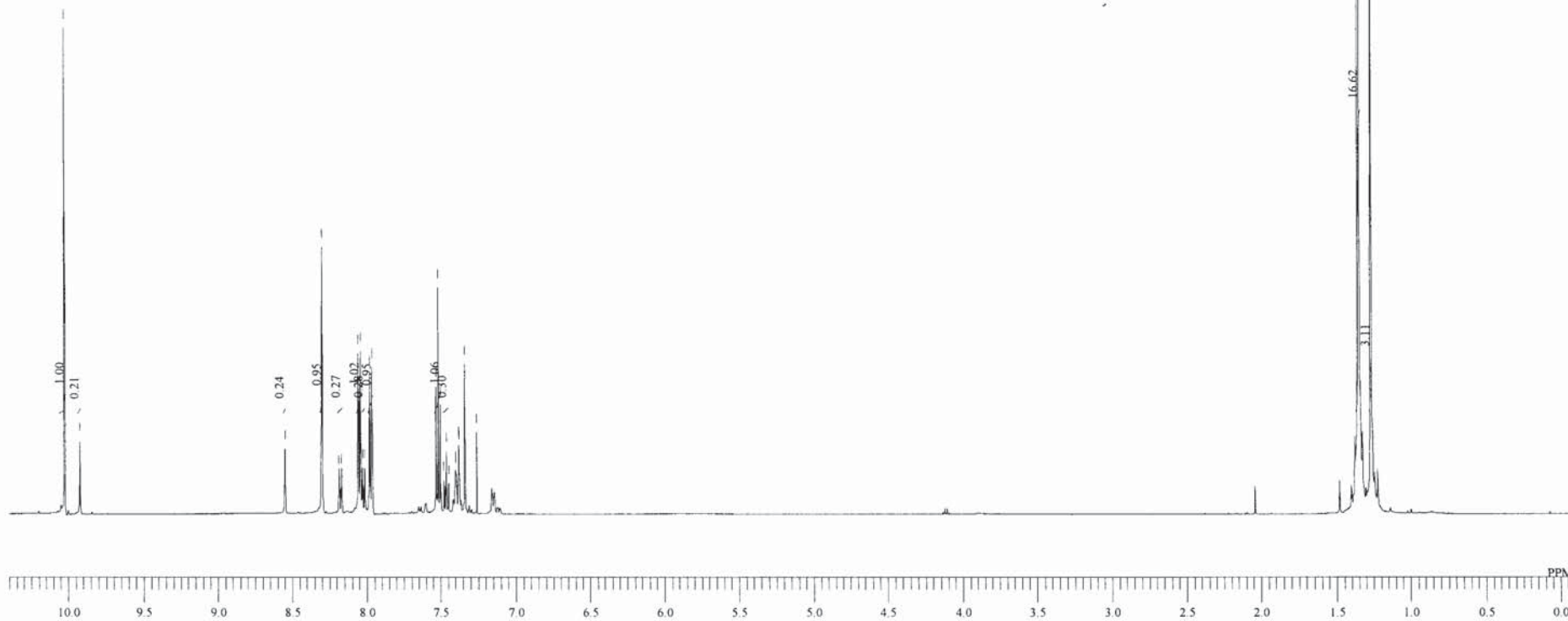


DATIM 04-03-2011 17.22.19
 MENU/ OBNUC 13C
 OFR 123.26 MHz
 OBSET 2.31 KHz
 OBFIN 6.71 Hz
 PW1 3.20 usec
 DEADT 0.00 usec
 PREDL 0.00000 msec
 IWT 1.0000 sec
 POINT 52428
 SPO 52428
 TIMES 32
 DUMMY 4
 FREQU 30863.73 Hz
 FLT 155000 Hz
 DELAY 21.06 usec
 ACQTM 0.8493 sec
 PD 10.0000 sec
 ADBIT 16
 RGAIN 60
 BF 1.00 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD single_pulse_dec
 EXPCM
 IRNUC 1H
 IFR 490.15 MHz
 IRSET 9.16 KHz
 IRFIN 7.60 Hz
 IRRPW 92 usec
 IRATN 79
 DFILE KO-ATX-BPin-o-CH(ON
 SF
 LKSET 70.30 KHz
 LKFIN 29.2 Hz
 LKLEV 0
 LGAIN 0
 LKPHS 0
 LKSIG 0
 CSPED 0 Hz
 FILDC
 FILDF

C:\Documents and Settings\PC-USER\My Documents\Data\okura\ATX\ECX and ECA\als\KO-ATX-m-BPin-CHO-1H.als

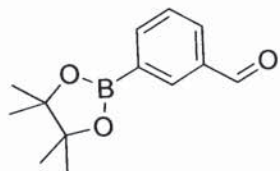


11e

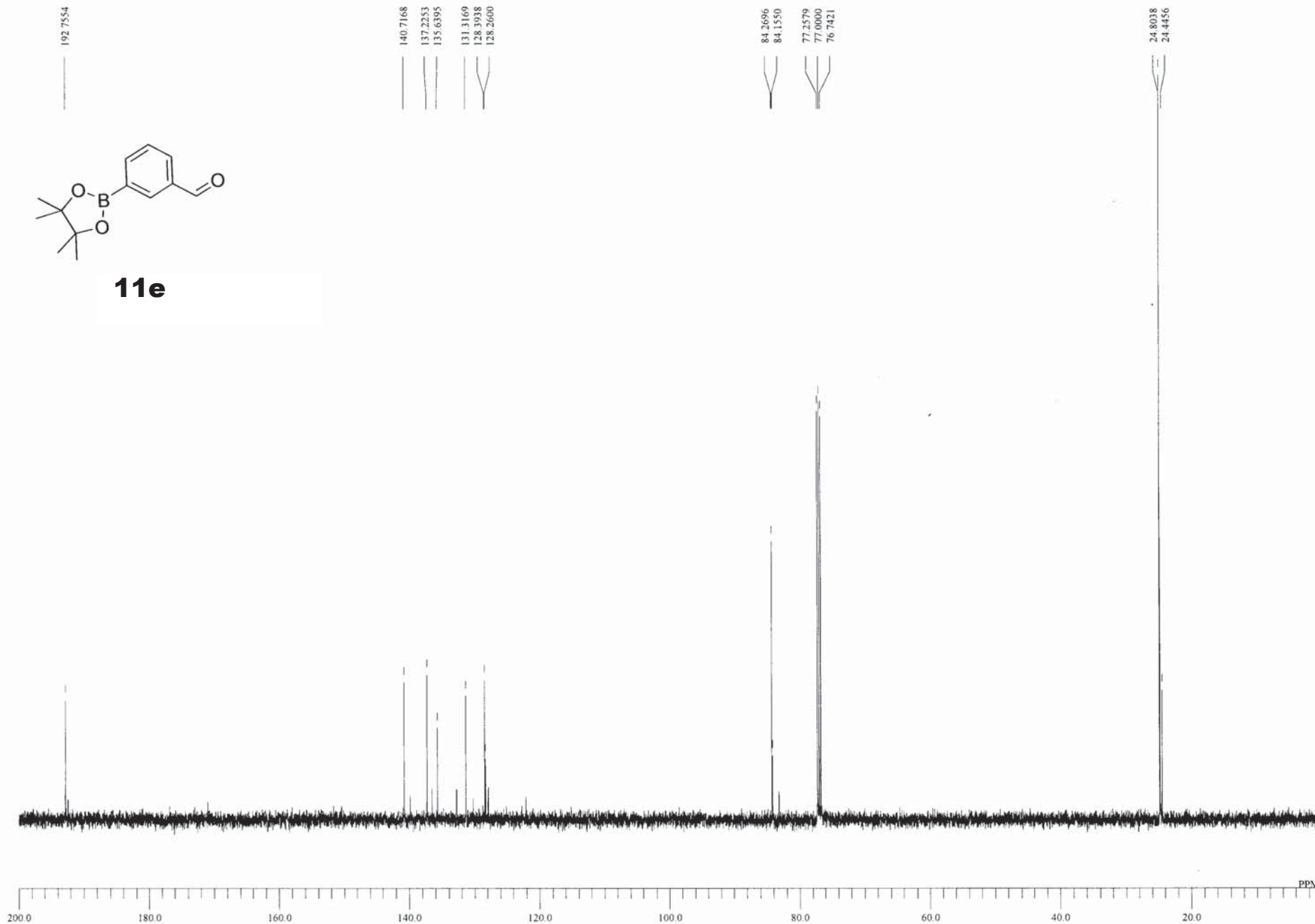


DATIM	08-03-2011 12:24:50
MENUF	
OBNUC	¹ H
OFR	490.15 MHz
OBSET	9.16 KHz
OBFIN	7.60 Hz
PW1	8.55 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	13107
SPO	13107
TIMES	8
DUMMY	1
FREQU	7352.83 Hz
FLT	37000 Hz
DELAY	13.52 usec
ACQTM	1.7826 sec
PD	1.5000 sec
ADBIT	16
RGAIN	30
BF	0.01 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse ex2
EXPCM	
IRNUC	¹ H
IFR	490.15 MHz
IRSET	9.16 KHz
IRFIN	7.60 Hz
IRRPW	118 usec
IRATN	79
DFILE	KO-ATX-m-BPin-CHO-1
SF	
LKSET	70.30 KHz
LKFIN	32.5 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	

C:\Documents and Settings\PC-USER\My Documents\Data\okura\ATX\ECX and ECA\als\KO-ATX-m-BPin-CHO-13C.als

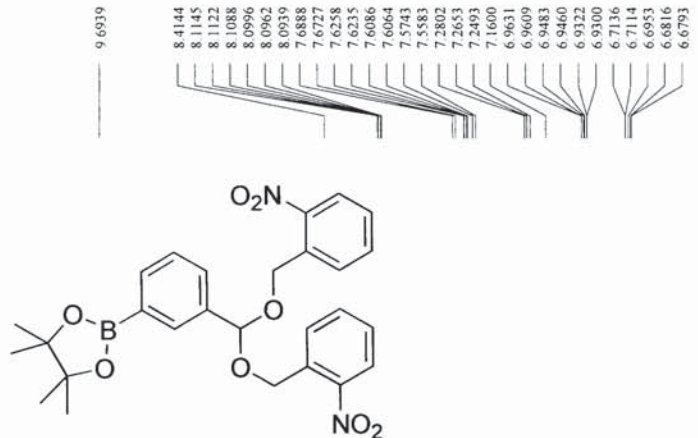


11e

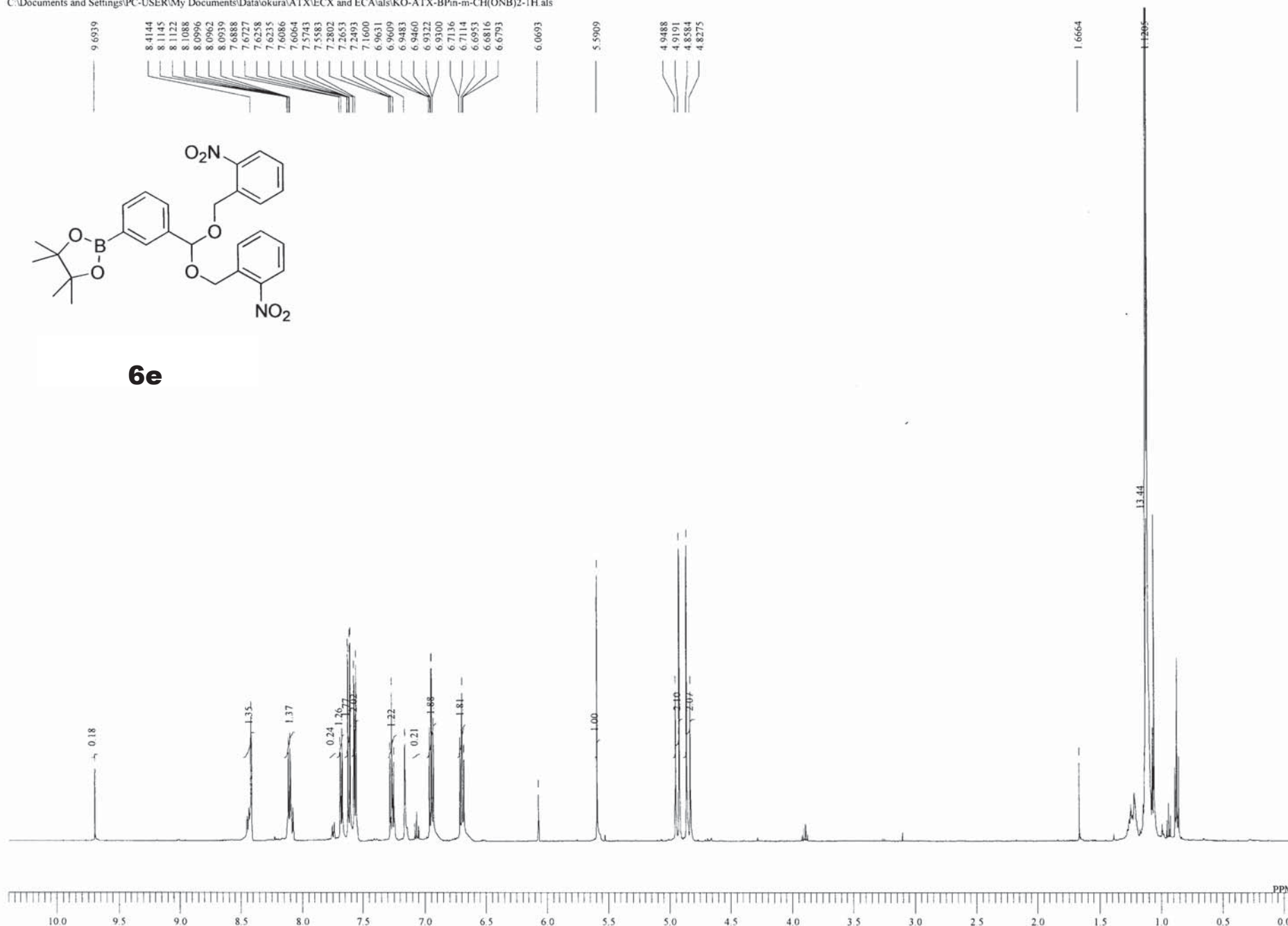


DATIM	08-03-2011 12:28:35
MENUF	
OBNUC	13C
OFR	123.26 MHz
OBSET	2.31 KHz
OBFIN	6.71 Hz
PW1	3.20 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	52428
SPO	52428
TIMES	16
DUMMY	4
FREQU	30863.73 Hz
FLT	155000 Hz
DELAY	21.06 usec
ACQTM	0.8493 sec
PD	10.0000 sec
ADBIT	16
RGAIN	60
BF	1.00 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	490.15 MHz
IRSET	9.16 KHz
IRFIN	7.60 Hz
IRRPW	92 usec
IRATN	79
DFILE	KO-ATX-m-BPin-CHO-1
SF	
LKSET	70.30 KHz
LKFIN	32.5 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	

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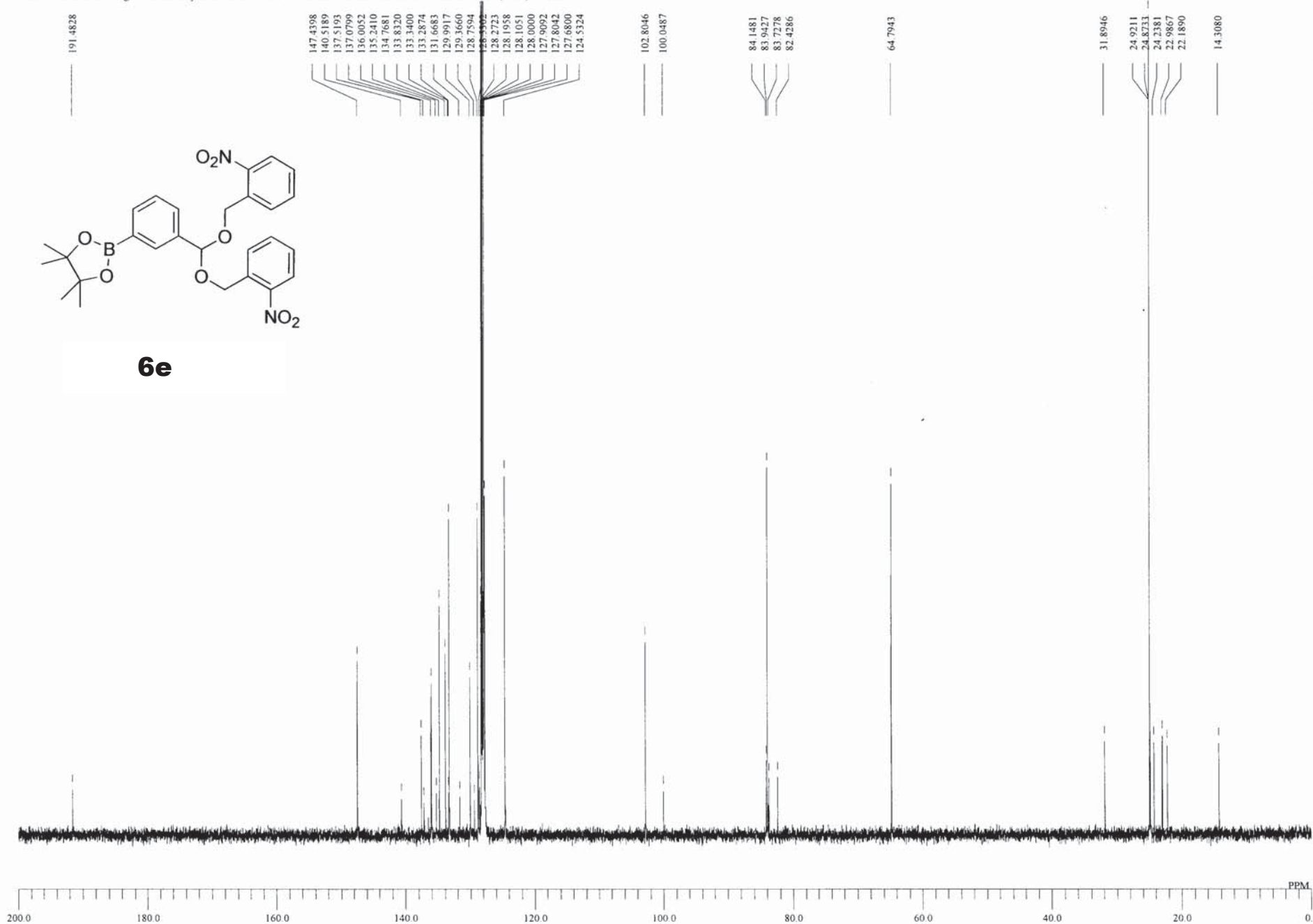


6e



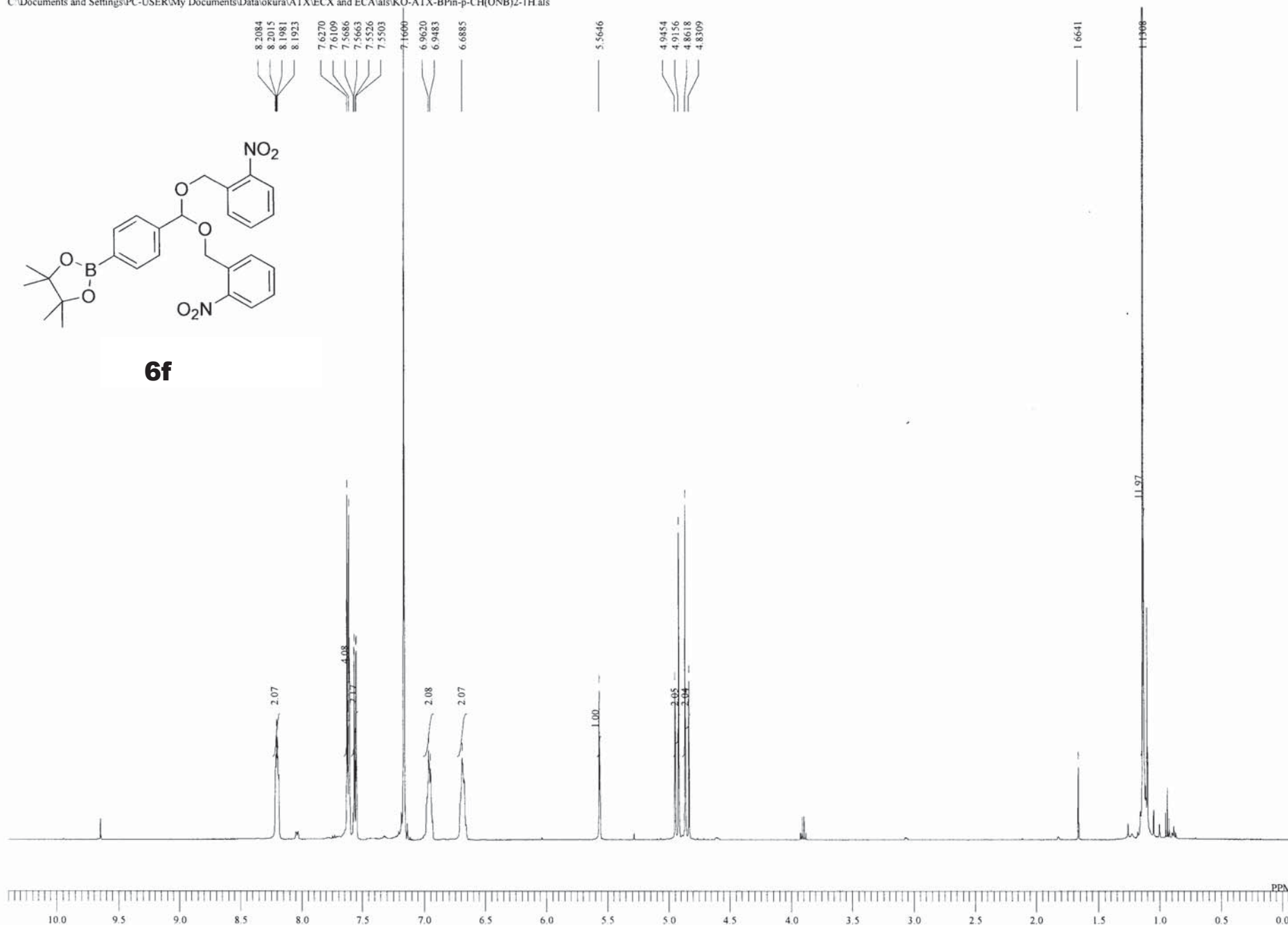
DATIM	03-03-2011 23:37:13
MENUP	
OBNUC	¹ H
OFR	490.15 MHz
OBSET	9.16 KHz
OBFIN	7.60 Hz
PWI	8.55 usec
DEADT	0.00 usec
FREDL	0.00000 msec
IWT	1.0000 sec
POINT	13107
SPO	13107
TIMES	8
DUMMY	1
FREQU	7352.83 Hz
FLT	37000 Hz
DELAY	13.52 usec
ACQTM	1.7826 sec
PD	1.5000 sec
ADBIT	16
RGAIN	26
BF	0.01 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	¹ H
IFR	490.15 MHz
IRSET	9.16 KHz
IRFIN	7.60 Hz
IRRPW	118 usec
IRATN	79
DFILE	KO-ATX-BPin-m-CH(ON
SF	
LKSET	70.30 KHz
LKFIN	29.2 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	

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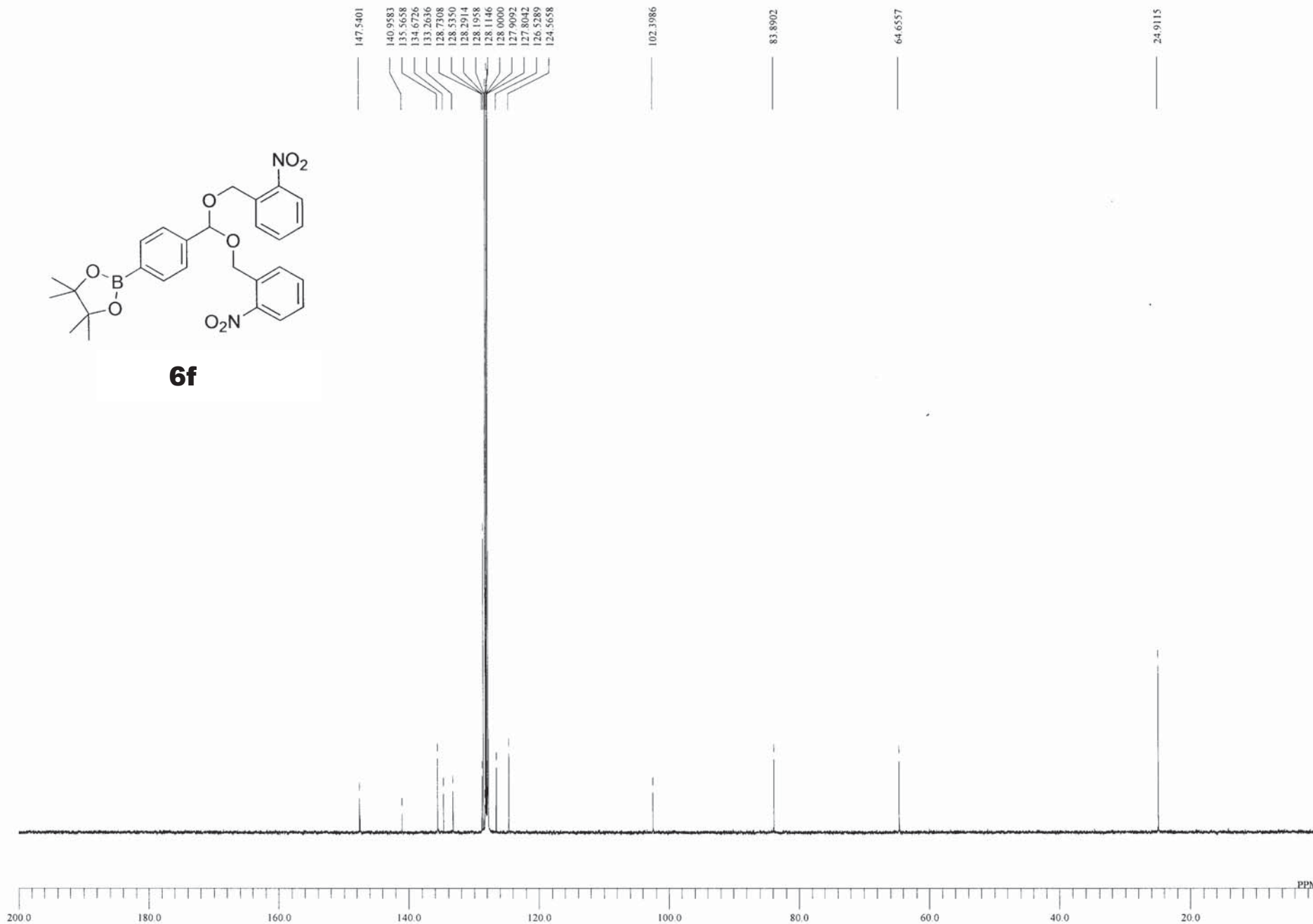
DATIM	03-03-2011 23:43:50
MENUF	
OBNUC	13C
OFR	123.26 MHz
OBSET	2.31 KHz
OBFIN	6.71 Hz
PW1	3.20 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	52428
SPO	52428
TIMES	32
DUMMY	4
FREQU	30863.73 Hz
FLT	155000 Hz
DELAY	21.06 usec
ACQTM	0.8493 sec
PD	10.0000 sec
ADBIT	16
RGAIN	60
BF	1.00 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	490.15 MHz
IRSET	9.16 KHz
IRFIN	7.60 Hz
IRRPW	92 usec
IRATN	79
DFILE	KO-ATX-BPin-m-CH(ON
SF	
LKSET	70.30 KHz
LKFIN	29.2 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	

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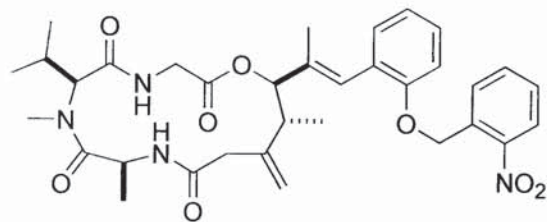
DATIM	04-03-2011 17:47:29
MENUF	
OBRUC	1H
OFR	490.15 MHz
OBSET	9.16 KHz
OBFIN	7.60 Hz
PW1	8.55 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	13107
SPO	13107
TIMES	8
DUMMY	1
FREQU	7352.83 Hz
FLT	37000 Hz
DELAY	13.52 usec
ACQTM	1.7826 sec
PD	5.0000 sec
ADBIT	16
RGAIN	26
BF	0.01 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse ex2
EXPCM	
IRNUC	1H
IFR	490.15 MHz
IRSET	9.16 KHz
IRFIN	7.60 Hz
IRRPW	118 usec
IRATN	79
DFILE	KO-ATX-BPin-p-CH(ON
SF	
LKSET	70.30 KHz
LKFIN	29.2 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	

C:\Documents and Settings\PC-USER\My Documents\Data\okura\ATX\ECX and ECA\als\KO-ATX-BPin-p-CH(ONB)2-13C.als

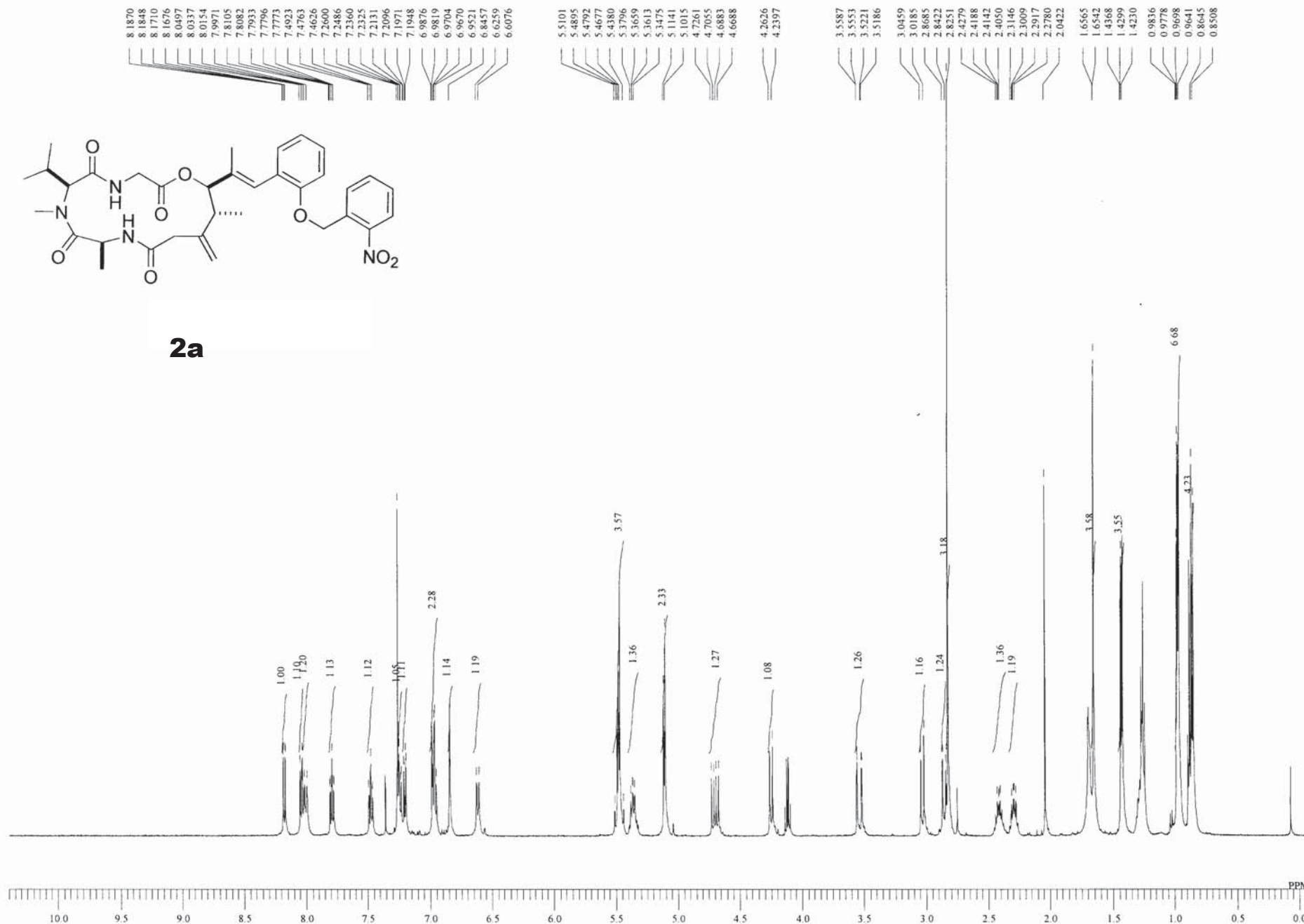


DATIM	04-03-2011 17:54:06
MENUF	
OBNUC	13C
OFR	123.26 MHz
OBSET	2.31 KHz
OBFIN	6.71 Hz
PW1	3.20 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	52428
SPO	52428
TIMES	32
DUMMY	4
FREQU	30863.73 Hz
FLT	155000 Hz
DELAY	21.06 usec
ACQTM	0.8493 sec
PD	10.0000 sec
ADBIT	16
RGAIN	60
BF	1.00 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	490.15 MHz
IRSET	9.16 KHz
IRFIN	7.60 Hz
IRRPW	92 usec
IRATN	79
DFILE	KO-ATX-BPin-p-CH(ON
SF	
LKSET	70.30 KHz
LKFIN	29.2 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	

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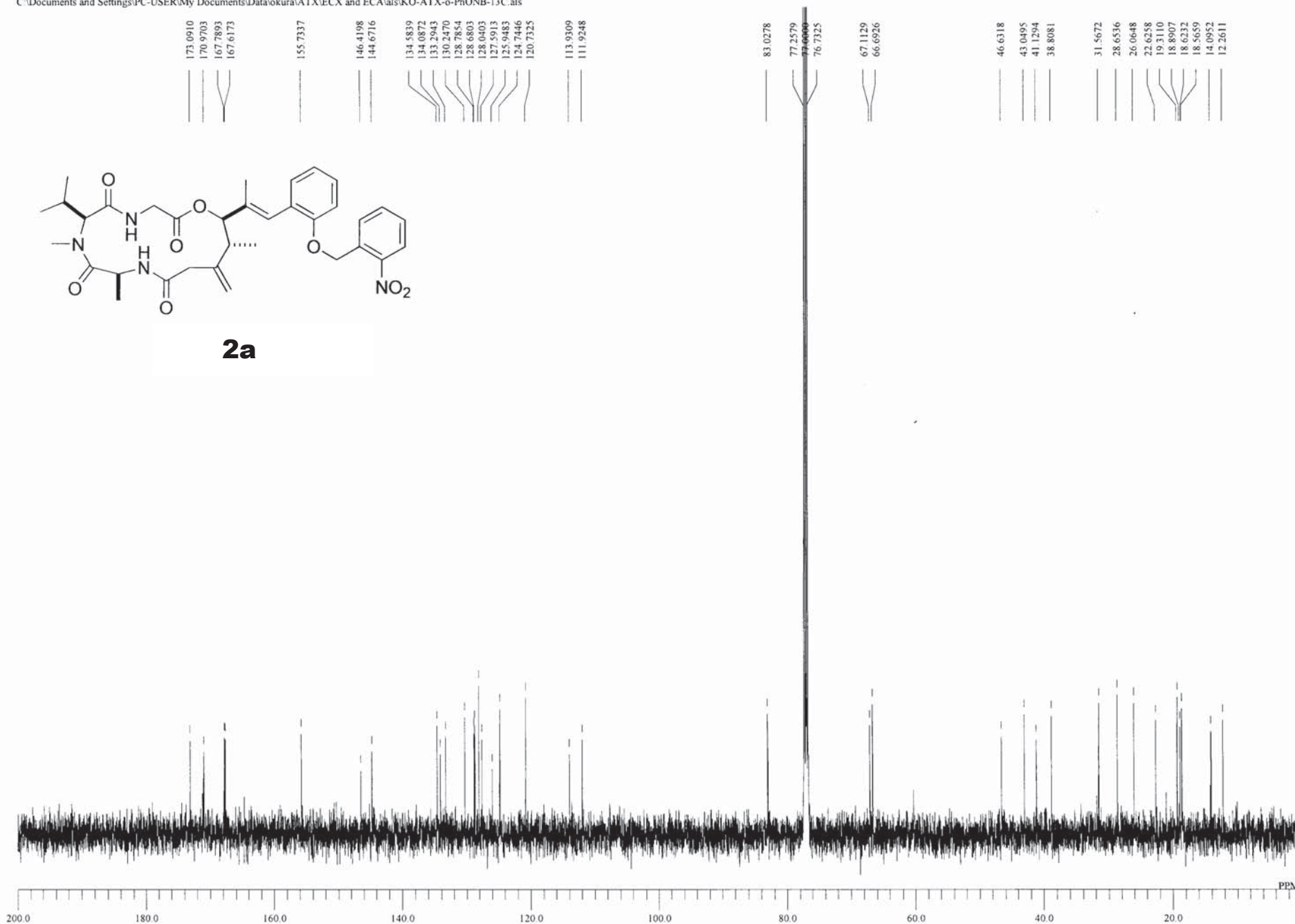


2a



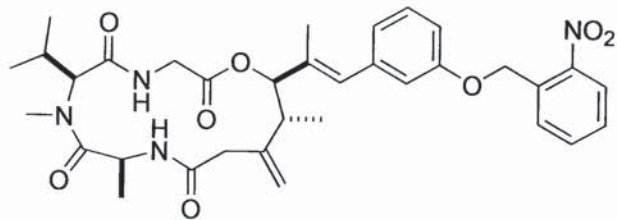
DATIM 18-09-2010 15:59:07
 MENUF 1H
 OBNUC 1H
 OFR 490.15 MHz
 OBSET 9.16 KHz
 OBFIN 7.60 Hz
 PWI 8.55 usec
 DEADT 0.00 usec
 PREDL 0.00000 msec
 IWT 1.0000 sec
 POINT 13107
 SPO 13107
 TIMES 8
 DUMMY 1
 FREQU 7352.83 Hz
 FLT 37000 Hz
 DELAY 13.52 usec
 ACQTM 1.7826 sec
 PD 1.5000 sec
 ADBIT 16
 RGAIN 40
 BF 0.01 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD single_pulse.ex2
 EXPCM 1H
 IRNUC 1H
 IFR 490.15 MHz
 IRSET 9.16 KHz
 IRFIN 7.60 Hz
 IRRPW 118 usec
 IRATN 79
 DFILE KO-ATX-o-PhONB-1H.al
 SF 70.30 KHz
 LKSET 32.5 Hz
 LKFIN 0
 LKLEV 0
 LGAIN 0
 LKPHS 0
 LKSIG 0
 CSPED 0 Hz
 FILDC
 FILDF

C:\Documents and Settings\PC-USER\My Documents\Data\okura\ATX\ECX and ECA\als\KO-ATX-o-PhONB-13C.als

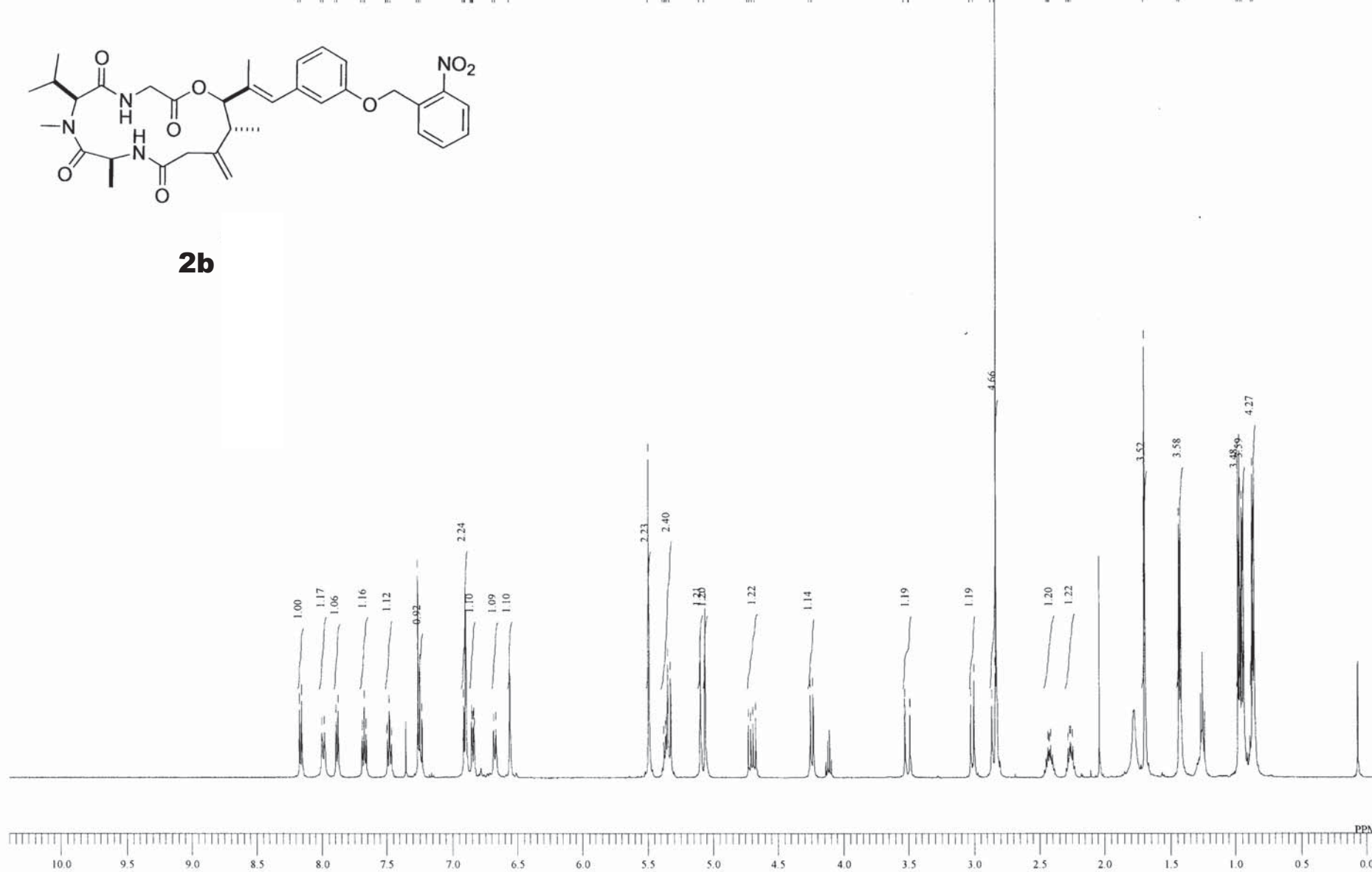
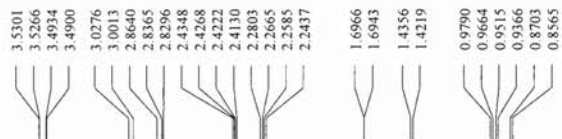
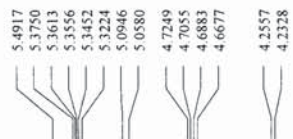
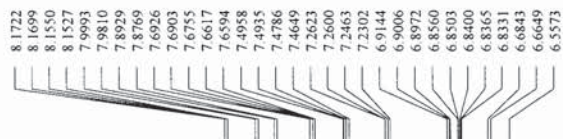


DATIM 18-09-2010 17:32:30
MENUF
OBNUC 13C
OFR 123.26 MHz
OBSET 2.31 KHz
OBFIN 6.71 Hz
PW1 3.20 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 26214
SPO 26214
TIMES 512
DUMMY 4
FREQU 30863.73 Hz
FLT 155000 Hz
DELAY 21.06 usec
ACQTM 0.8493 sec
PD 10.0000 sec
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 490.15 MHz
IRSET 9.16 KHz
IRFIN 7.60 Hz
IRRPW 92 usec
IRATN 79
DFILE KO-ATX-o-PhONB-13C.i
SF
LKSET 70.30 KHz
LKFIN 32.5 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF

C:\Documents and Settings\PC-USER\My Documents\Data\okura\ATX\ECX and ECA\als\KO-ATX-m-PhONB-1H.als

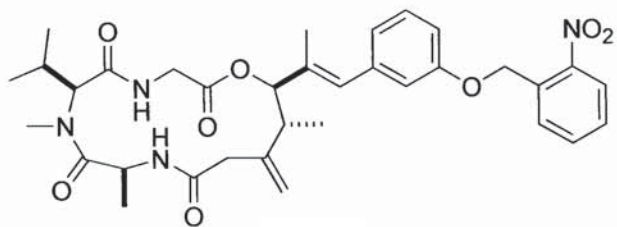


2b

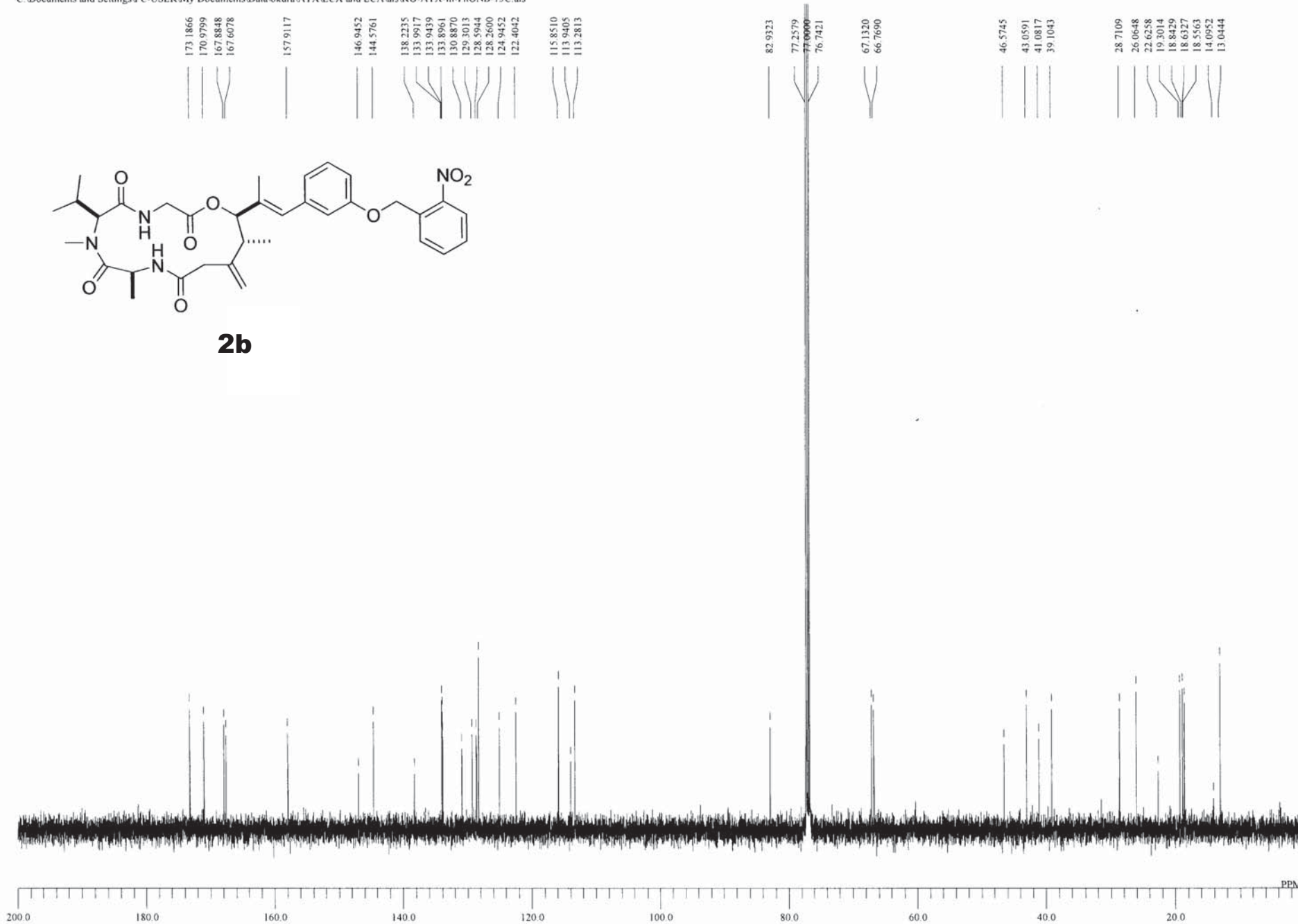


DATIM 18-09-2010 13:16:42
 MENUF 1H
 OBNUC 1H
 OFR 490.15 MHz
 OBSET 9.16 KHz
 OFBIN 7.60 Hz
 PW1 8.55 usec
 DEADT 0.00 usec
 PREDL 0.00000 msec
 IWT 1.0000 sec
 POINT 13107
 SPO 13107
 TIMES 8
 DUMMY 1
 FREQU 7352.83 Hz
 FLT 37000 Hz
 DELAY 13.52 usec
 ACQTM 1.7826 sec
 PD 1.5000 sec
 ADBIT 16
 RGAIN 40
 BF 0.01 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD single_pulse ex2
 EXPCM 1H
 IRNUC 490.15 MHz
 IRSET 9.16 KHz
 IRFIN 7.60 Hz
 IRRPW 118 usec
 IRATN 79
 DFILE KO-ATX-m-PhONB-1H.a
 SF 70.30 KHz
 LKSET 32.5 Hz
 LKFIN 0
 LKLEV 0
 LGAIN 0
 LKPHS 0
 LKSIG 0
 CSPED 0 Hz
 FILDC
 FILDF

C:\Documents and Settings\PC-USER\My Documents\Data\okura\ATX\ECX and ECA\als\KO-ATX-m-PhONB-13C.als

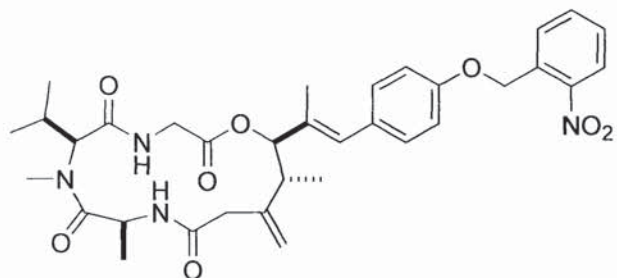


2b

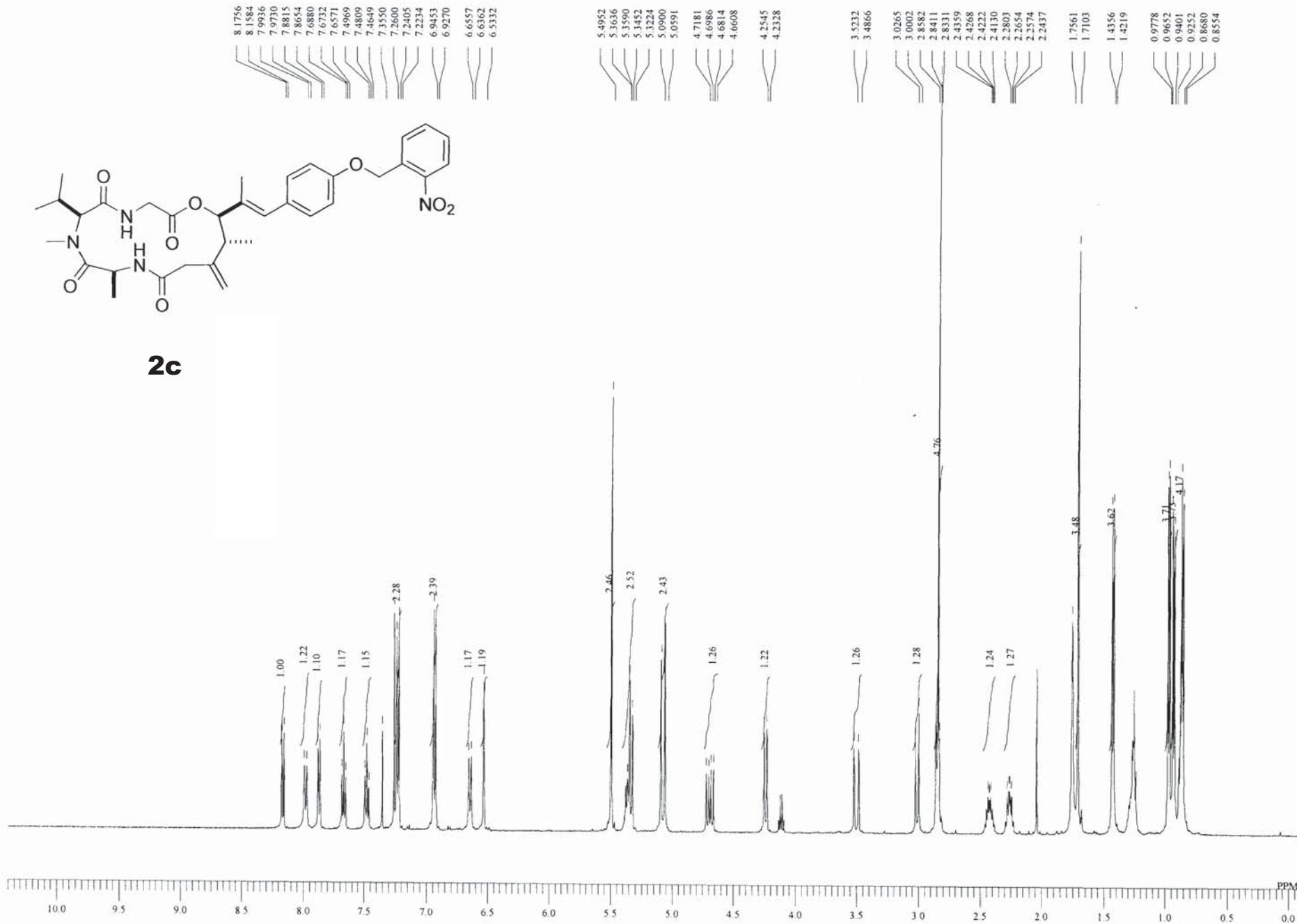


DATIM 18-09-2010 14:50:05
 MENUF
 OBNUC 13C
 OFR 123.26 MHz
 OBSET 2.31 KHz
 OFBIN 6.71 Hz
 PW1 3.20 usec
 DEADT 0.00 usec
 PREDL 0.00000 msec
 IWT 1.0000 sec
 POINT 26214
 SPO 26214
 TIMES 512
 DUMMY 4
 FREQU 30863.73 Hz
 FLT 155000 Hz
 DELAY 21.06 usec
 ACQTM 0.8493 sec
 PD 10.0000 sec
 ADBIT 16
 RGAIN 60
 BF 0.10 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD single_pulse_dec
 EXPCM
 IRNUC 1H
 IFR 490.15 MHz
 IRSET 9.16 KHz
 IRFIN 7.60 Hz
 IRRPW 92 usec
 IRATN 79
 DFILE KO-ATX-m-PhONB-13C
 SF
 LKSET 70.30 KHz
 LKFIN 32.5 Hz
 LKLEV 0
 LGAIN 0
 LKPHS 0
 LKSIG 0
 CSPED 0 Hz
 FILDC
 FILDF

C:\Documents and Settings\PC-USER\My Documents\Data\okura\ATX\ECX and ECA\als\KO-ATX-p-PhONB-1H.als

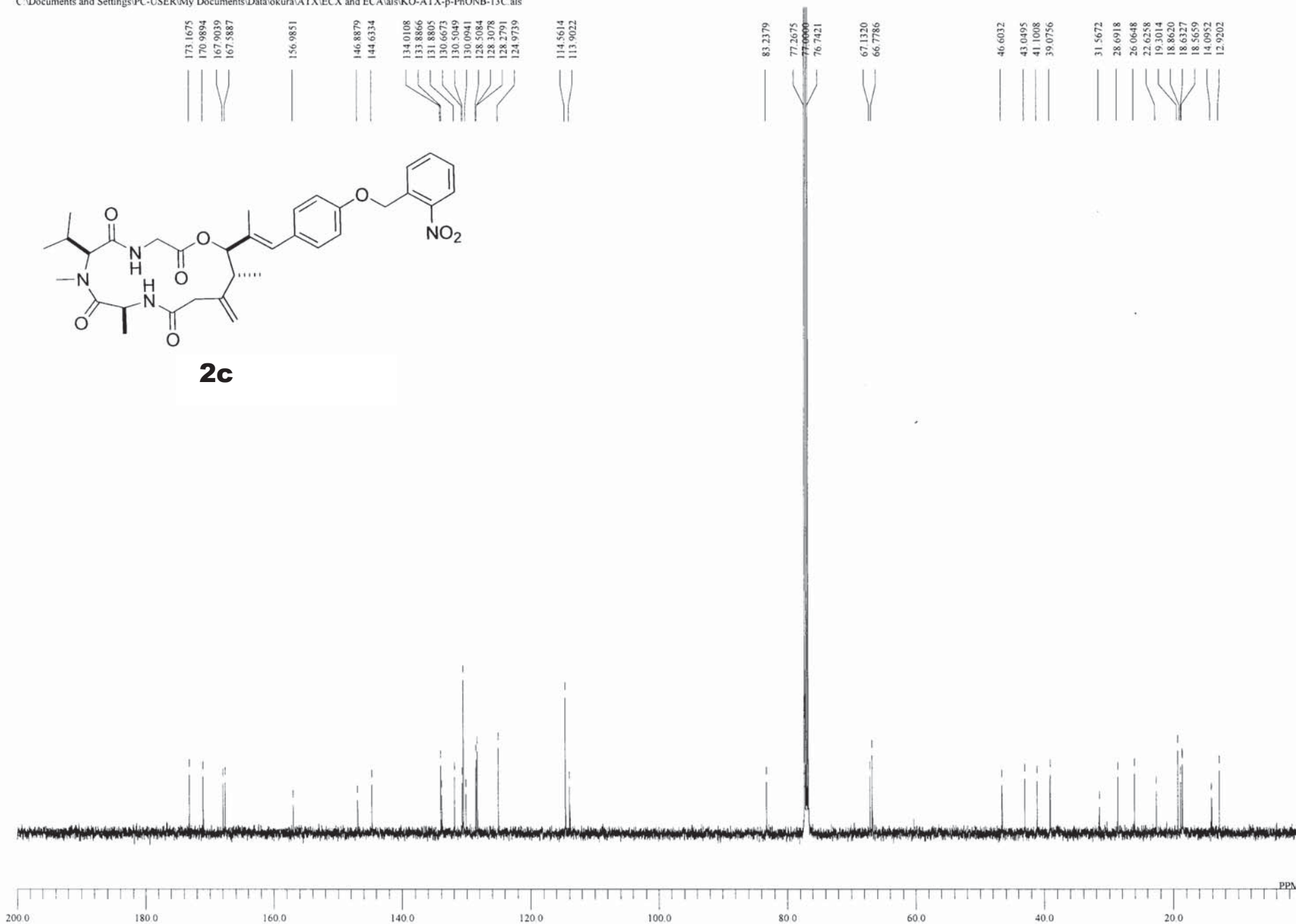
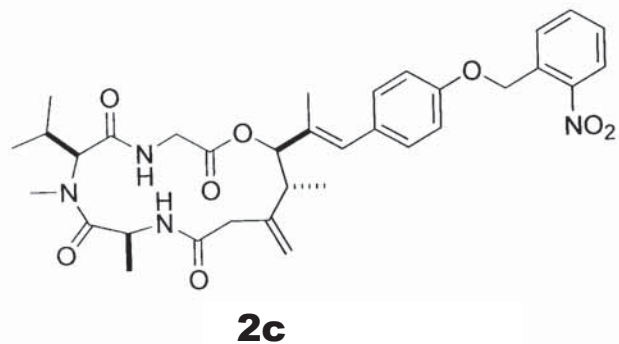


2c



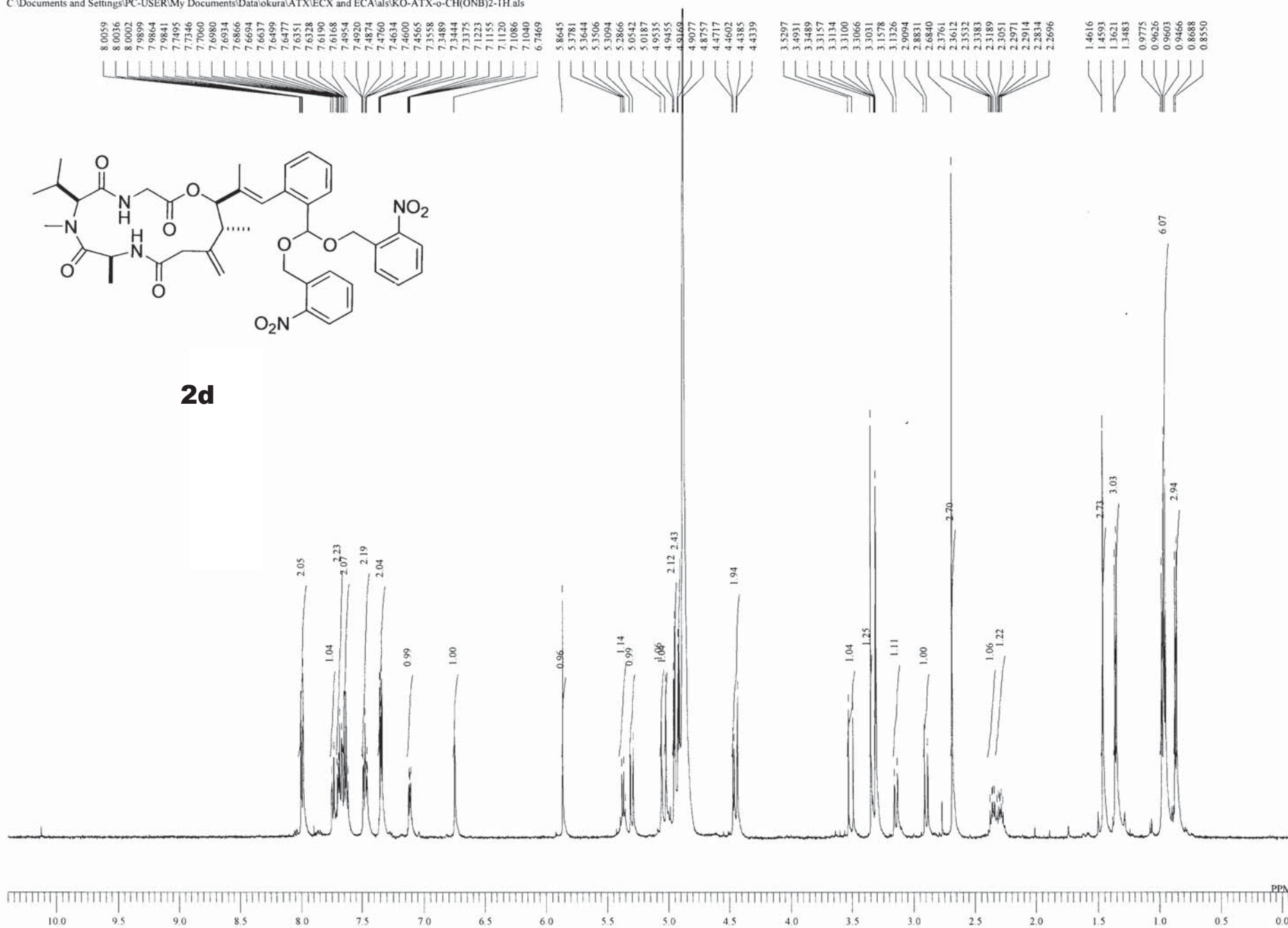
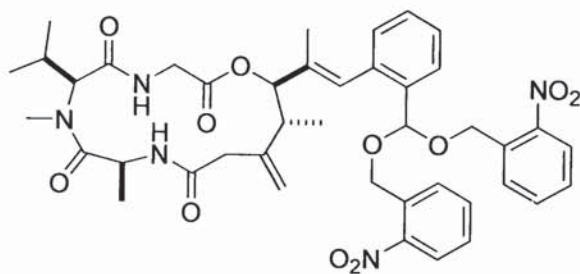
DATIM 18-09-2010 07:33:47
 MENUF 1H
 OBNUC 1H
 OFR 490.15 MHz
 OBSET 9.16 KHz
 OBFIN 7.60 Hz
 PW1 8.55 usec
 DEADT 0.00 usec
 PREDL 0.00000 msec
 IWT 1.0000 sec
 POINT 13107
 SPO 13107
 TIMES 8
 DUMMY 1
 FREQU 7352.83 Hz
 FLT 37000 Hz
 DELAY 13.52 usec
 ACQTM 1.7826 sec
 PD 1.5000 sec
 ADBIT 16
 RGAIN 40
 BF 0.01 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD single_pulse.ex2
 EXPCM 1H
 IRNUC 1H
 IFR 490.15 MHz
 IRSET 9.16 KHz
 IRFIN 7.60 Hz
 IRRPW 92 usec
 IRATN 79
 DFILE KO-ATX-p-PhONB-1H.als
 SF 70.30 KHz
 LKSET 32.5 Hz
 LKFIN 0
 LKLEV 0
 LGAIN 0
 LKPHS 0
 LKSIG 0
 CSPED 0 Hz
 FILDC
 FILDF

C:\Documents and Settings\PC-USER\My Documents\Data\okura\ATX\ECX and ECA\als\KO-ATX-p-PhONB-13C.als



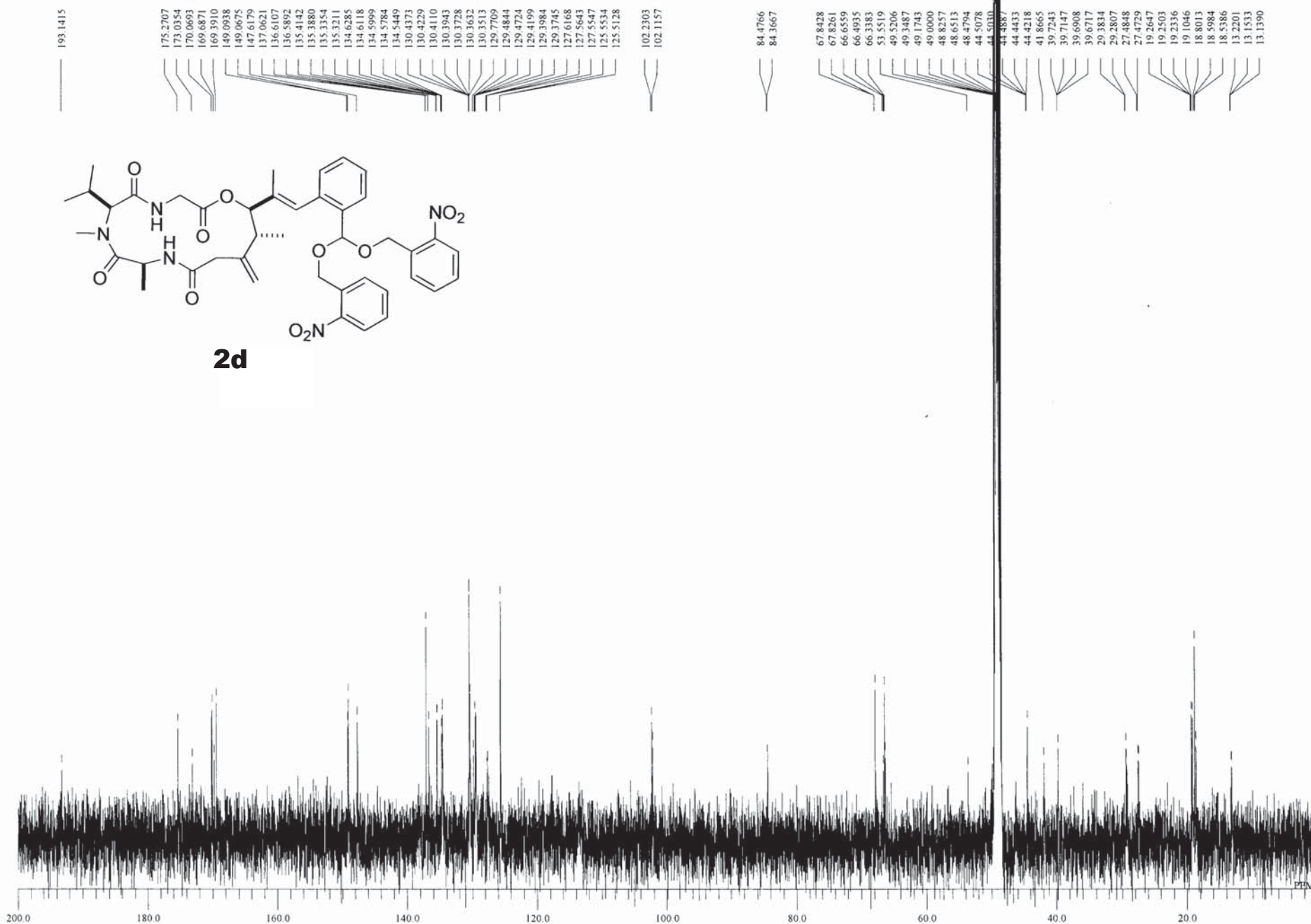
DATIM	18-09-2010 09:07:10
MENUF	
OBNUC	13C
OFR	123.26 MHz
OBSET	2.31 KHz
OBFIN	6.71 Hz
PWI	3.20 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	26214
SPO	26214
TIMES	512
DUMMY	4
FREQU	30863.73 Hz
FLT	155000 Hz
DELAY	21.06 usec
ACQTM	0.8493 sec
PD	10.0000 sec
ADBIT	16
RGAIN	60
BF	1.00 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse_dec
EXPCM	
IRNUC	1H
IFR	490.15 MHz
IRSET	9.16 KHz
IRFIN	7.60 Hz
IRRPW	92 usec
IRATN	79
DFILE	KO-ATX-p-PhONB-13C :
SF	
LKSET	70.30 KHz
LKFIN	32.5 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	

C:\Documents and Settings\PC-USER\My Documents\Data\okura\ATX\ECX and ECA\als\KO-ATX-o-CH(ONB)2-1H.als



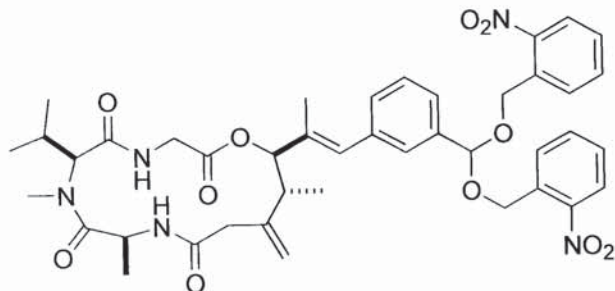
DATIM 16-02-2011 12:29:25
 MENUF
 OBNUC 1H
 OFR 490.15 MHz
 OBSET 9.16 KHz
 OBFIN 7.60 Hz
 PW1 8.55 usec
 DEADT 0.00 usec
 PREDL 0.00000 msec
 IWT 1.0000 sec
 POINT 13107
 SPO 13107
 TIMES 8
 DUMMY 1
 FREQU 7352.83 Hz
 FLT 37000 Hz
 DELAY 13.52 usec
 ACQTM 1.7826 sec
 PD 1.5000 sec
 ADBIT 16
 RGAIN 44
 BF 0.01 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD single_pulse ex2
 IRNUC 1H
 IFR 490.15 MHz
 IRSET 9.16 KHz
 IRFIN 7.60 Hz
 IRRPW 118 usec
 IRATN 79
 DFILE KO-ATX-o-CH(ONB)2-1
 SF
 LKSET 70.00 KHz
 LKFIN 36.6 Hz
 LKLEV 0
 LGAIN 0
 LKPHS 0
 LKSIG 0
 CSPED 0 Hz
 FILDC
 FILDF

C:\Documents and Settings\PC-USER\My Documents\Data\okura\ATX\ECX and ECA\als\KO-ATX-o-CH(ONB)2-13C.als

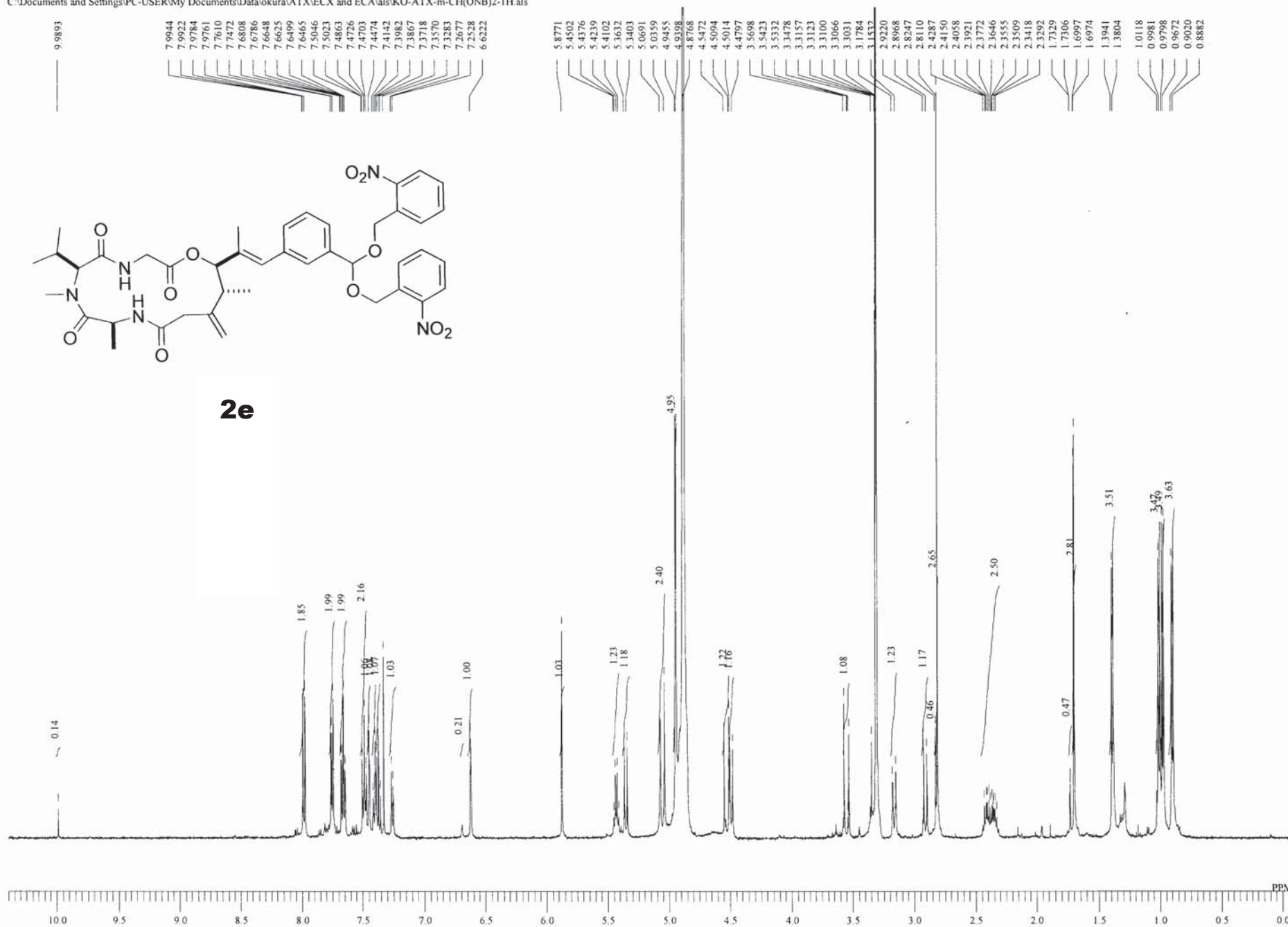


DATIM 16-02-2011 14:12:25
 MENUF
 OBNUC 13C
 OFR 123.26 MHz
 OBSET 2.31 KHz
 OFBIN 6.71 Hz
 PWI 3.20 usec
 DEADT 0.00 usec
 PREDL 0.00000 msec
 IWT 1.0000 sec
 POINT 104856
 SPO 104856
 TIMES 512
 DUMMY 4
 FREQU 30863.73 Hz
 FLT 155000 Hz
 DELAY 21.06 usec
 ACQTM 0.8493 sec
 PD 10.0000 sec
 ADBIT 16
 RGAIN 60
 BF 1.00 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD single_pulse_dec
 EXPCM
 IRNUC 1H
 IFR 490.15 MHz
 IRSET 9.16 KHz
 IRFIN 7.60 Hz
 IRRPW 92 usec
 IRATN 79
 DFILE KO-ATX-o-CH(ONB)2-1
 SF
 LKSET 70.00 KHz
 LKFIN 36.6 Hz
 LKLEV 0
 LGAIN 0
 LKPHS 0
 LKSIG 0
 CSPED 0 Hz
 FILDC
 FILDF

C:\Documents and Settings\PC-USER\My Documents\Data\okura\ATX\ECX and ECA\als\KO-ATX-m-CH(ONB)2-1H.als

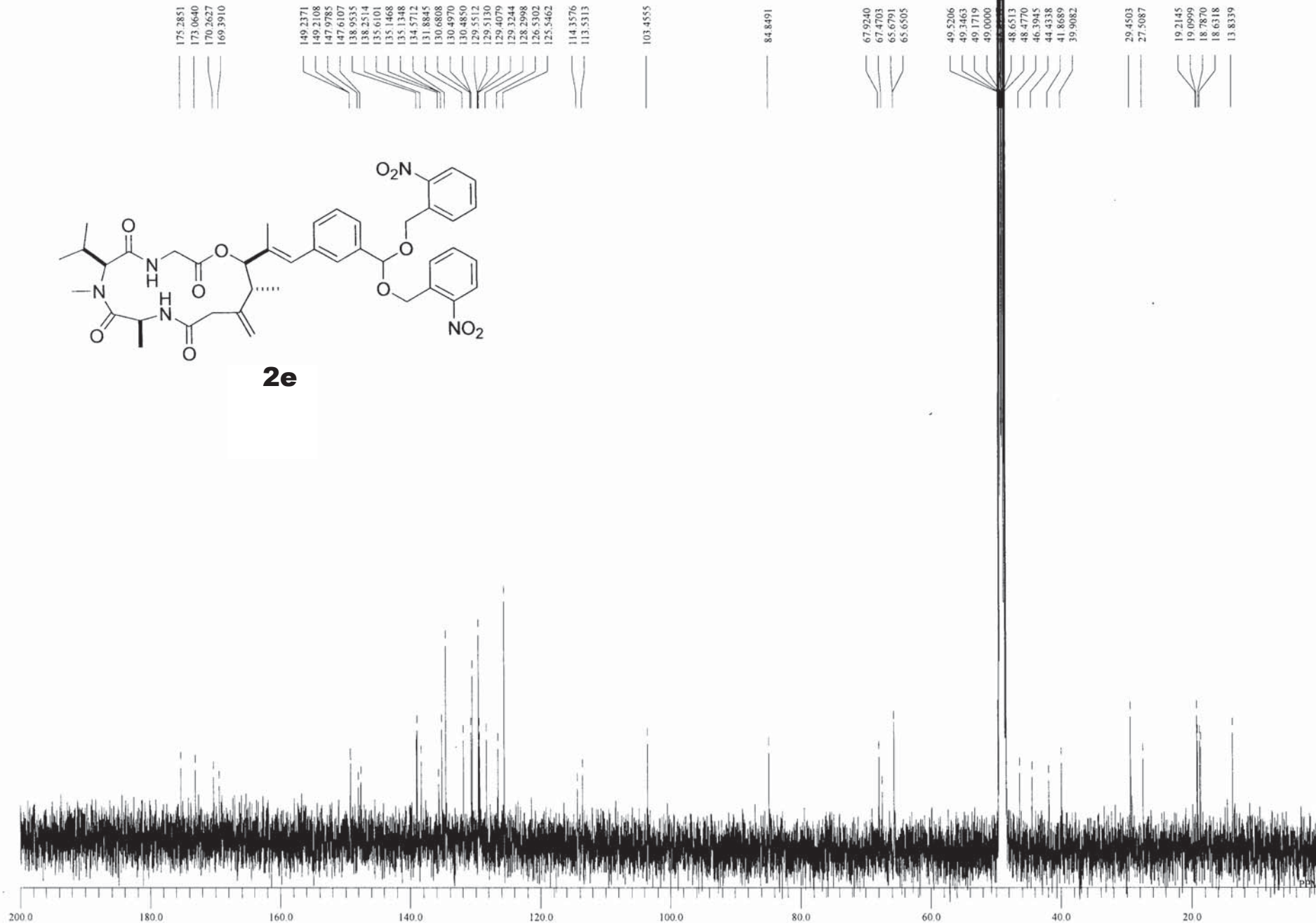
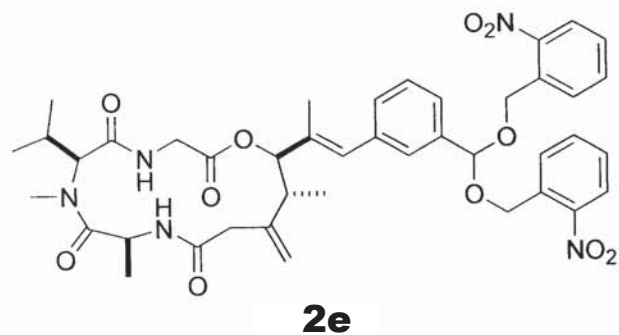


2e



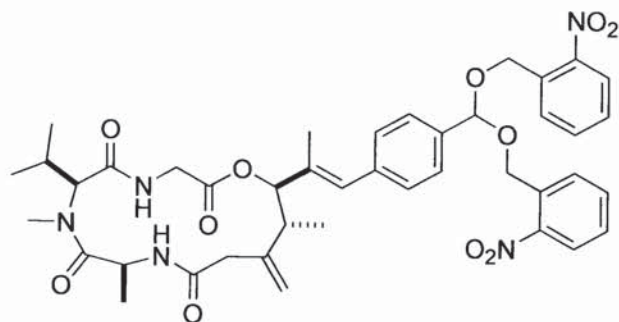
DATIM	30-01-2011 07:53:56
MENUP	
OBNUC	¹ H
OFR	490.15 MHz
OBSET	9.16 KHz
OBFIN	7.60 Hz
PW1	8.55 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	13107
SPO	13107
TIMES	8
DUMMY	1
FREQU	7352.83 Hz
FLT	37000 Hz
DELAY	13.52 usec
ACQTM	1.7826 sec
PD	1.5000 sec
ADBIT	16
RGAIN	44
BF	0.01 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse ex2
EXPCM	
IRNUC	¹ H
IFR	490.15 MHz
IRSET	9.16 KHz
IRFIN	7.60 Hz
IRRPW	118 usec
IRATN	79
DFILE	KO-ATX-m-CH(ONB)2-1
SF	
LKSET	70.00 KHz
LKFIN	36.6 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	

C:\Documents and Settings\PC-USER\My Documents\Data\okura\ATX\ECX and ECA\als\KO-ATX-m-CH(ONB)2-13C-2.als

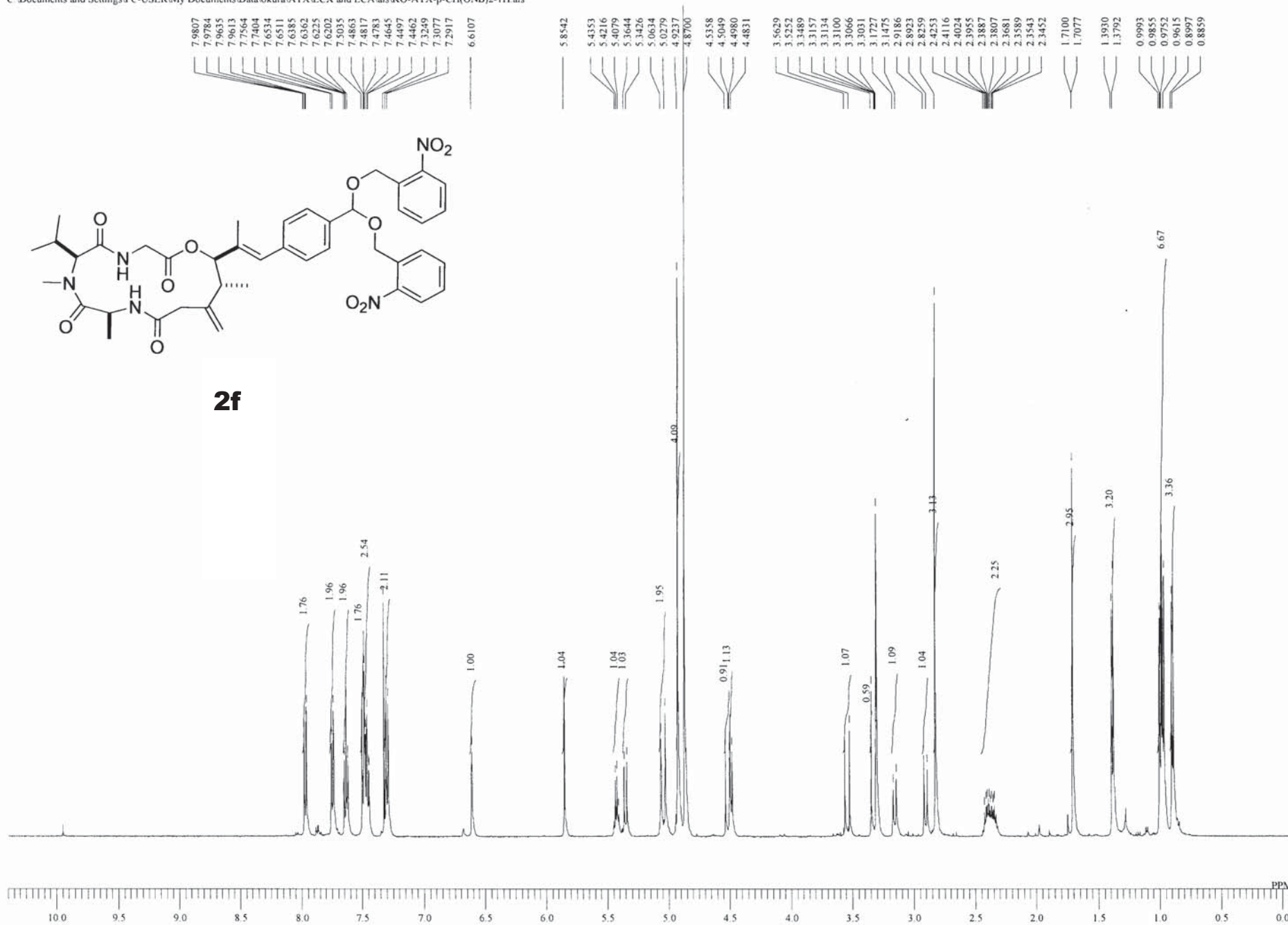


DATIM 30-01-2011 14:07:58
 MENUF
 OBNUC 13C
 OFR 123.26 MHz
 OBSSET 2.31 KHz
 OFBIN 6.71 Hz
 PW1 3.20 usec
 DEADT 0.00 usec
 PREDL 0.00000 msec
 IWT 1.0000 sec
 POINT 104856
 SPO 104856
 TIMES 2048
 DUMMY 4
 FREQU 30863.73 Hz
 FLT 155000 Hz
 DELAY 21.06 usec
 ACQTM 0.8493 sec
 PD 10.0000 sec
 ADBIT 16
 RGAIN 60
 BF 1.00 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD single_pulse_dec
 EXPCM
 IRNUC 1H
 IFR 490.15 MHz
 IRSET 9.16 KHz
 IRFIN 7.60 Hz
 IRRPW 92 usec
 IRATN 79
 DFILE KO-ATX-m-CH(ONB)2-1
 SF
 LKSET 70.00 KHz
 LKFIN 36.6 Hz
 LKLEV 0
 LGAIN 0
 LKPHS 0
 LKSIG 0
 CSPED 0 Hz
 FILDC
 FILDF

C:\Documents and Settings\PC-USER\My Documents\Data\okura\ATX\ECX and ECA\als\KO-ATX-p-CH(ONB)2-1H.als

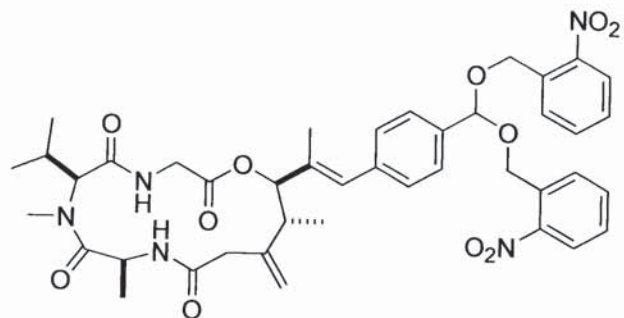


2f

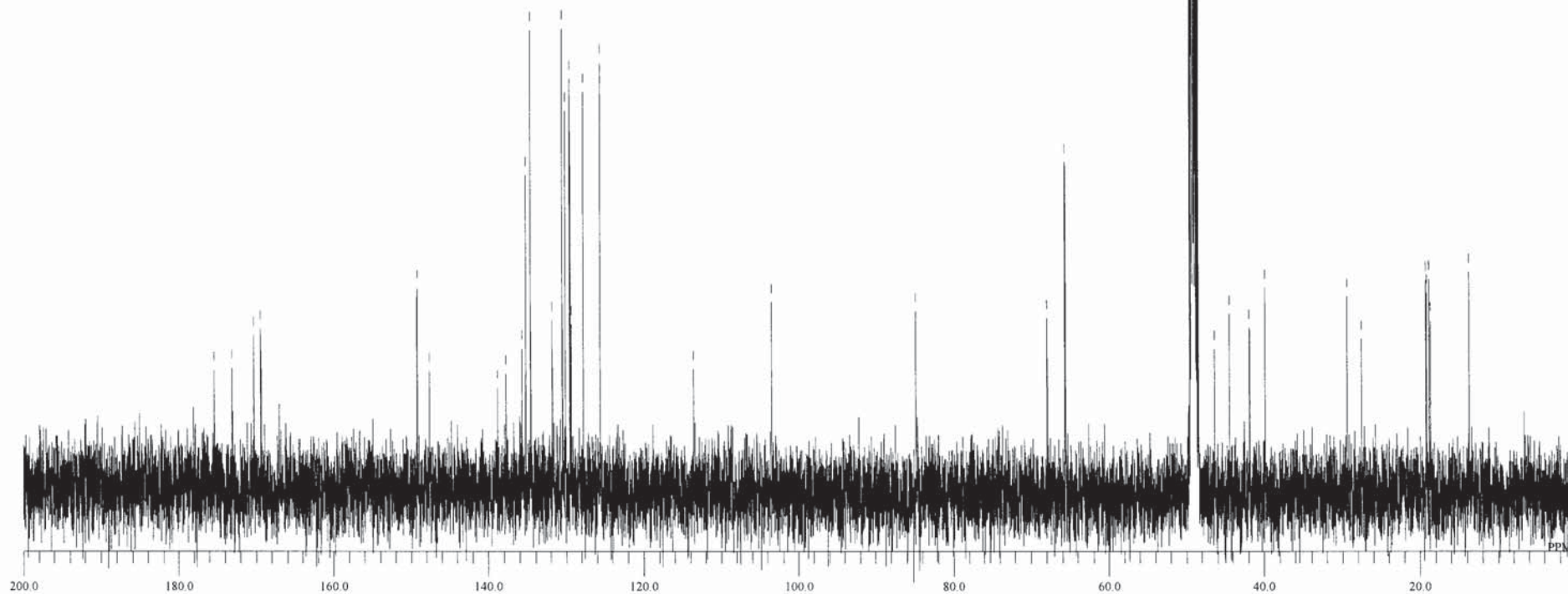


DATIM	30-01-2011 02:02:29
MENUP	
OBNUC	¹ H
OFR	490.15 MHz
OBSET	9.16 KHz
OBFIN	7.60 Hz
PWI	8.55 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	13107
SPO	13107
TIMES	8
DUMMY	1
FREQU	7352.83 Hz
FLT	37000 Hz
DELAY	13.52 usec
ACQTM	1.7826 sec
PD	1.5000 sec
ADBIT	16
RGAIN	36
BF	0.01 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse ex2
EXPCM	
IRNUC	¹ H
IFR	490.15 MHz
IRSET	9.16 KHz
IRFIN	7.60 Hz
IRRPW	92 usec
IRATN	79
DFILE	KO-ATX-p-CH(ONB)2-1
SF	
LKSET	70.00 KHz
LKFIN	36.6 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	

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2f



175.3066
173.0378
170.2388
169.3361

149.1607
147.5797

138.7529
137.6544
135.6817
135.1897
134.5640
131.7746
130.4802
129.4868
129.3196
127.7482
125.5271

113.5384

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67.9097
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49.3487
49.1767
49.0000

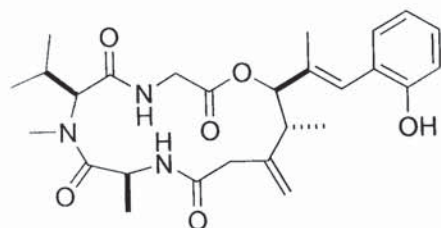
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48.4794
46.3921
44.4386
41.8593
39.8676

29.3977
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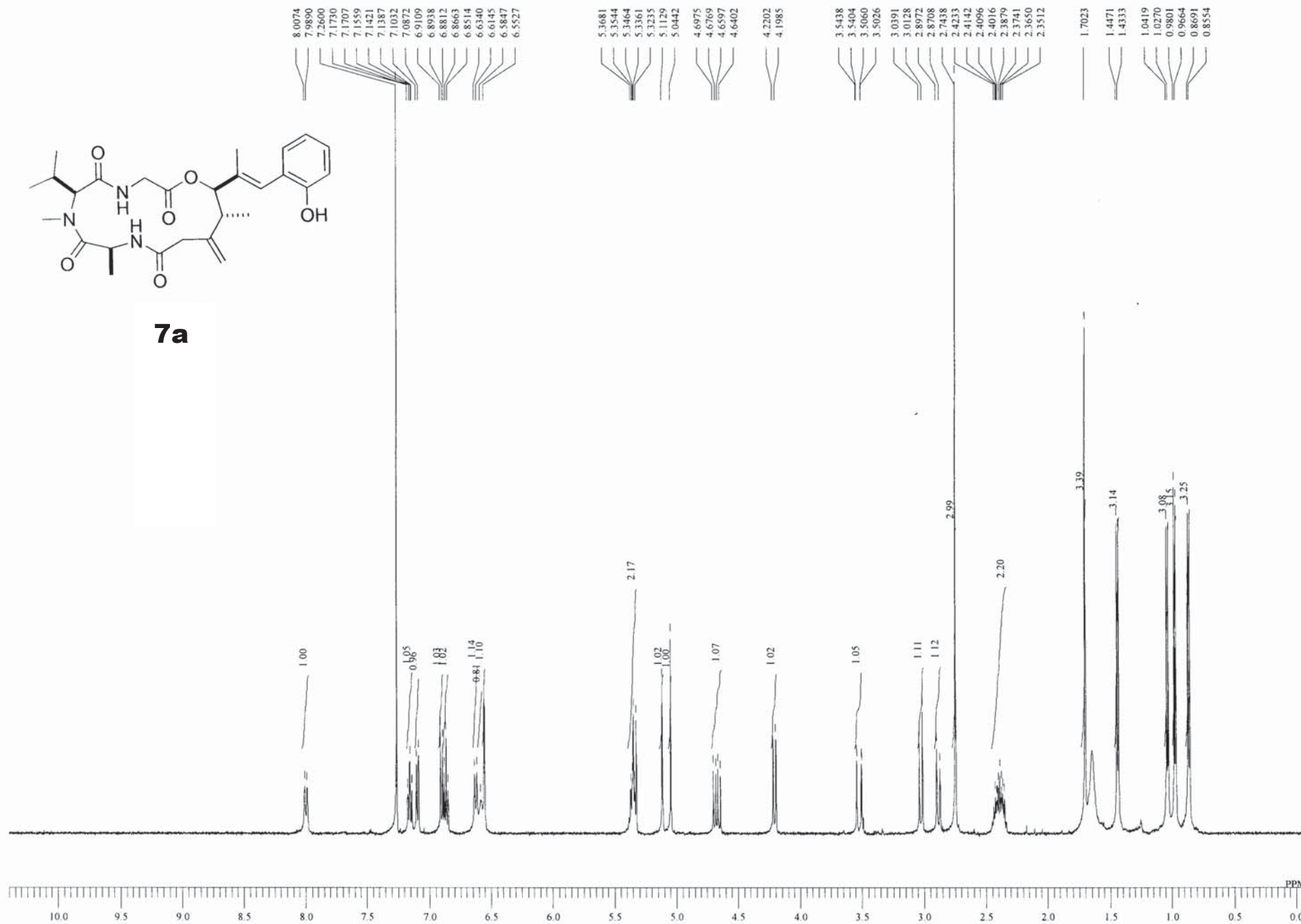
19.2193
19.1046
18.7894
18.6366
13.7503

DATIM 30-01-2011 02:49:34
 MENUF
 OBNUC 13C
 OFR 123.26 MHz
 OBSET 2.31 KHz
 OFBIN 6.71 Hz
 PW1 3.20 usec
 DEADT 0.00 usec
 PREDL 0.00000 msec
 IWT 1.0000 sec
 POINT 52428
 SPO 52428
 TIMES 256
 DUMMY 4
 FREQU 30863.73 Hz
 FLT 155000 Hz
 DELAY 21.06 usec
 ACQTM 0.8493 sec
 PD 10.0000 sec
 ADBIT 16
 RGAIN 60
 BF 1.00 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD single_pulse_dec
 EXPCM
 IRNUC 1H
 IFR 490.15 MHz
 IRSET 9.16 KHz
 IRFIN 7.60 Hz
 IRRPW 92 usec
 IRATN 79
 DFILE KO-ATX-p-CH(ONB)2-1
 SF
 LKSET 70.00 KHz
 LKFIN 36.6 Hz
 LKLEV 0
 LGAIN 0
 LKPHS 0
 LKSIG 0
 CSPED 0 Hz
 FILDC
 FILDF

C:\Documents and Settings\PC-USER\My Documents\Data\okura\ATX\ECX and ECA\als\KO-ATX-o-PhOH-1H.als

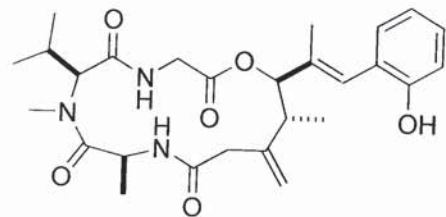


7a

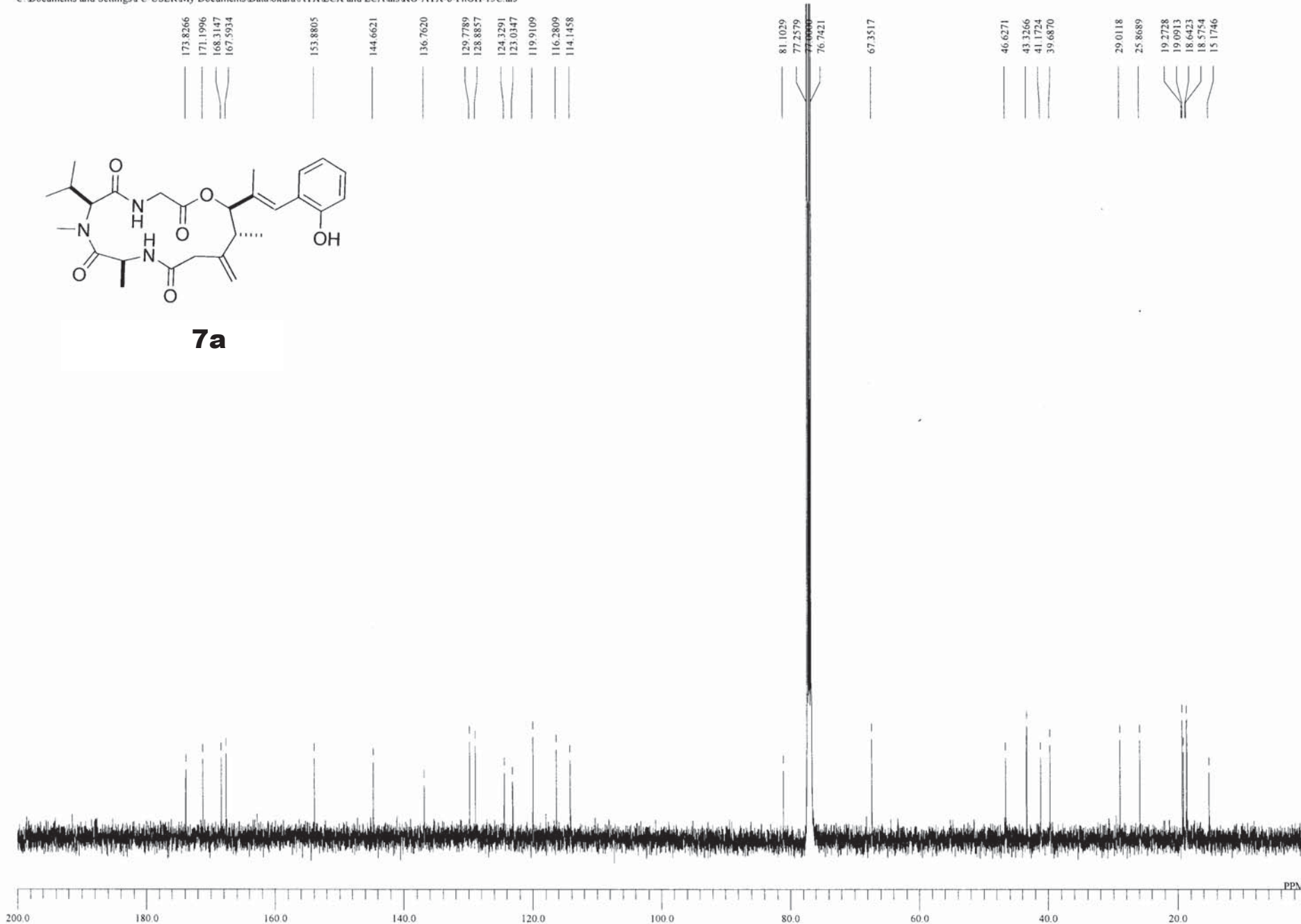


DATIM	04-03-2011 23:42:38
MENUP	
OBNUC	¹ H
OFR	490.15 MHz
OBSET	9.16 KHz
OBFIN	7.60 Hz
PW1	8.55 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	13107
SPO	13107
TIMES	8
DUMMY	1
FREQU	7352.83 Hz
FLT	37000 Hz
DELAY	13.52 usec
ACQTM	1.7826 sec
PD	1.5000 sec
ADBIT	16
RGAIN	46
BF	0.01 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse.ex2
EXPCM	
IRNUC	¹ H
IFR	490.15 MHz
IRSET	9.16 KHz
IRFIN	7.60 Hz
IRRPW	118 usec
IRATN	79
DFILE	KO-ATX-o-PhOH-1H.als
SF	
LKSET	70.30 KHz
LKFIN	32.5 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	

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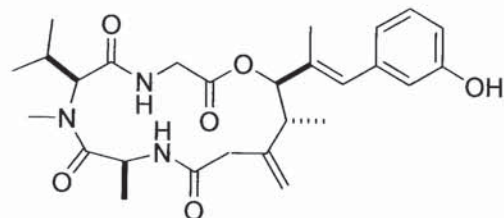


7a

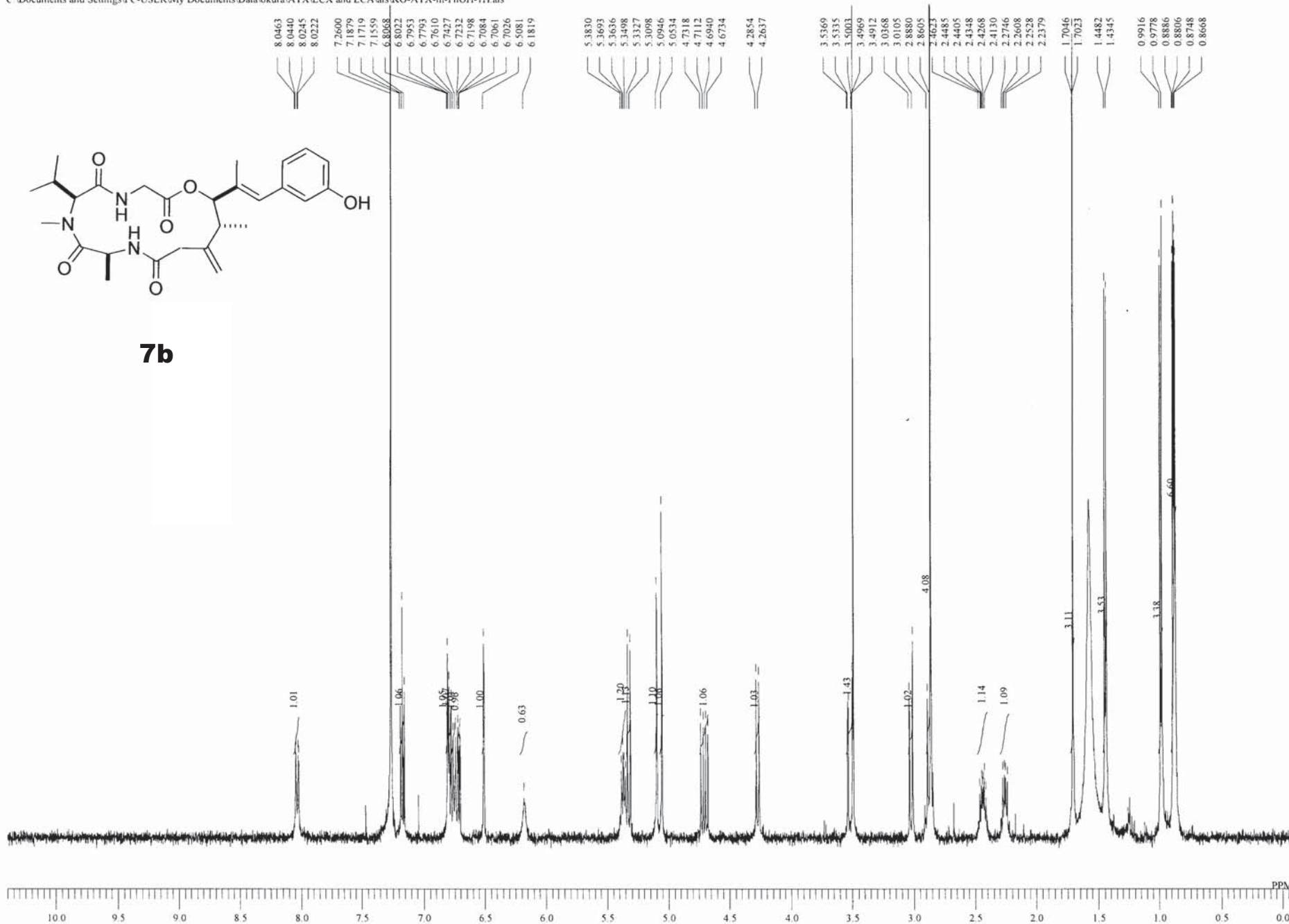


DATIM 05-03-2011 09:11:27
 MENUF
 OBNUC 13C
 OFR 123.26 MHz
 OBSSET 2.31 KHz
 OBFIN 6.71 Hz
 PW1 3.20 usec
 DEADT 0.00 usec
 PREDL 0.00000 msec
 IWT 1.0000 sec
 POINT 52428
 SPO 52428
 TIMES 3072
 DUMMY 4
 FREQU 30863.73 Hz
 FLT 155000 Hz
 DELAY 21.06 usec
 ACQTM 0.8493 sec
 PD 10.0000 sec
 ADBIT 16
 RGAIN 60
 BF 1.00 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD single_pulse_dec
 EXPCM
 IRNUC 1H
 IFR 490.15 MHz
 IRSET 9.16 KHz
 IRFIN 7.60 Hz
 IRRPW 92 usec
 IRATN 79
 DFILE KO-ATX-o-PhOH-13C.als
 SF
 LKSET 70.30 KHz
 LKFIN 32.5 Hz
 LKLEV 0
 LGAIN 0
 LKPHS 0
 LKSIG 0
 CSPED 0 Hz
 FILDC
 FILDF

C:\Documents and Settings\PC-USER\My Documents\Data\okura\ATX\ECX and ECA\als\KO-ATX-m-PhOH-1H.als

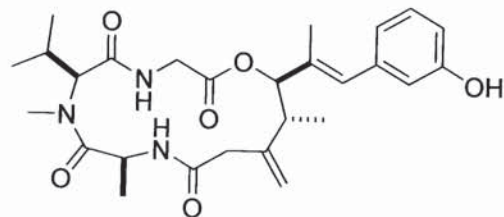
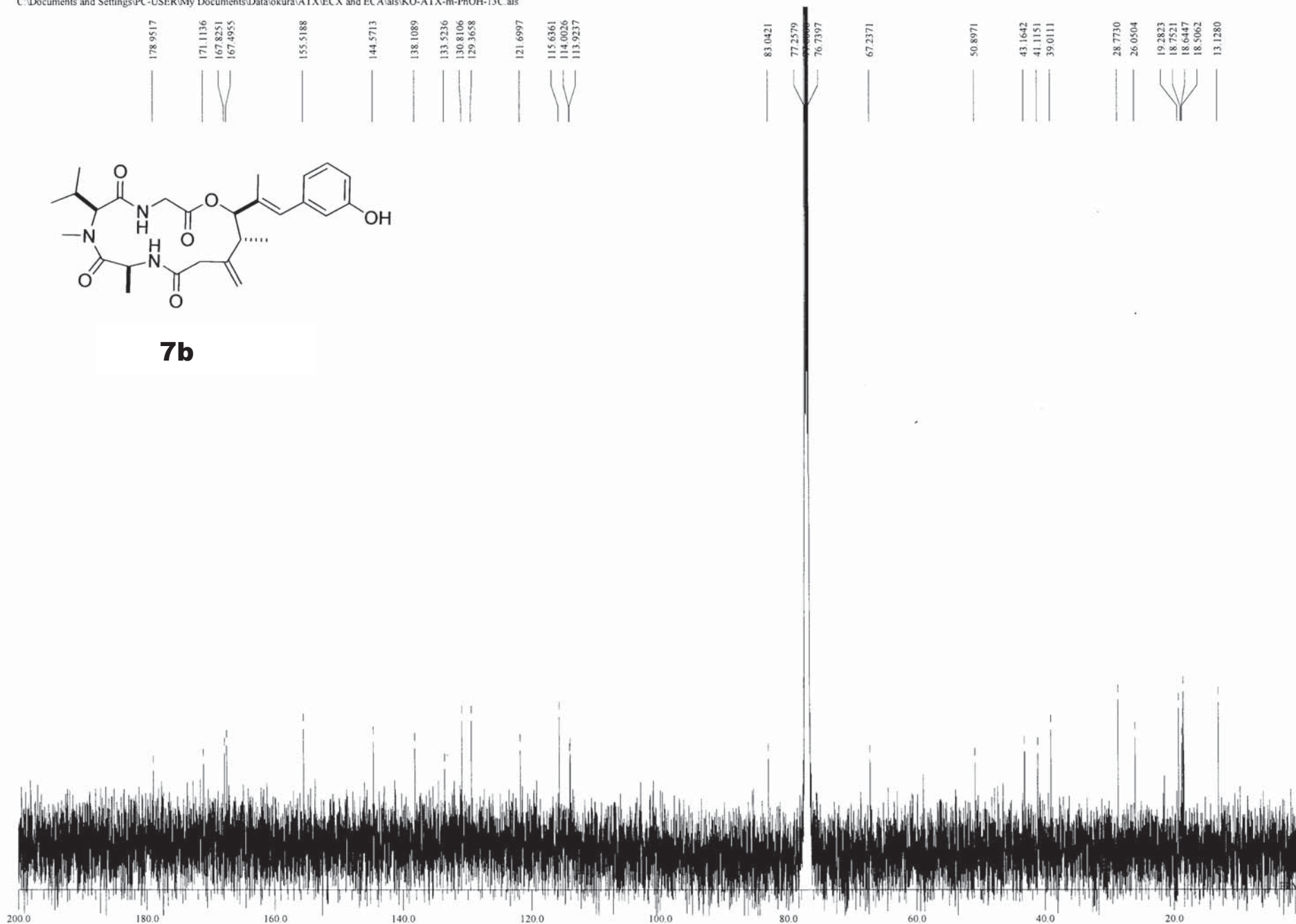


7b



DATIM 02-03-2011 23:35:09
 MENUF
 OBNUC 1H
 OFR 490.15 MHz
 OBSSET 9.16 KHz
 OFBIN 7.60 Hz
 PW1 8.55 usec
 DEADT 0.00 usec
 PREDL 0.00000 msec
 IWT 1.0000 sec
 POINT 13107
 SPO 13107
 TIMES 8
 DUMMY 1
 FREQU 7352.83 Hz
 FLT 37000 Hz
 DELAY 13.52 usec
 ACQTM 1.7826 sec
 PD 1.5000 sec
 ADBIT 16
 RGAIN 48
 BF 0.01 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD single_pulse ex2
 EXPCM
 IRNUC 1H
 IFR 490.15 MHz
 IRSET 9.16 KHz
 IRFIN 7.60 Hz
 IRRPW 118 usec
 IRATN 79
 DFILE KO-ATX-m-PhOH-1H.als
 SF
 LKSET 70.30 KHz
 LKFIN 32.5 Hz
 LKLEV 0
 LGAIN 0
 LKPHS 0
 LKSIG 0
 CSPED 0 Hz
 FILDC
 FILDF

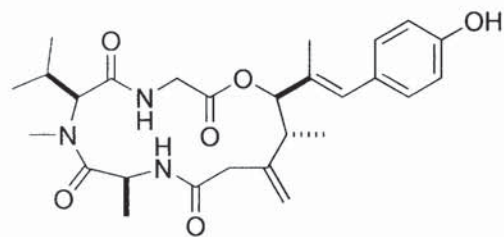
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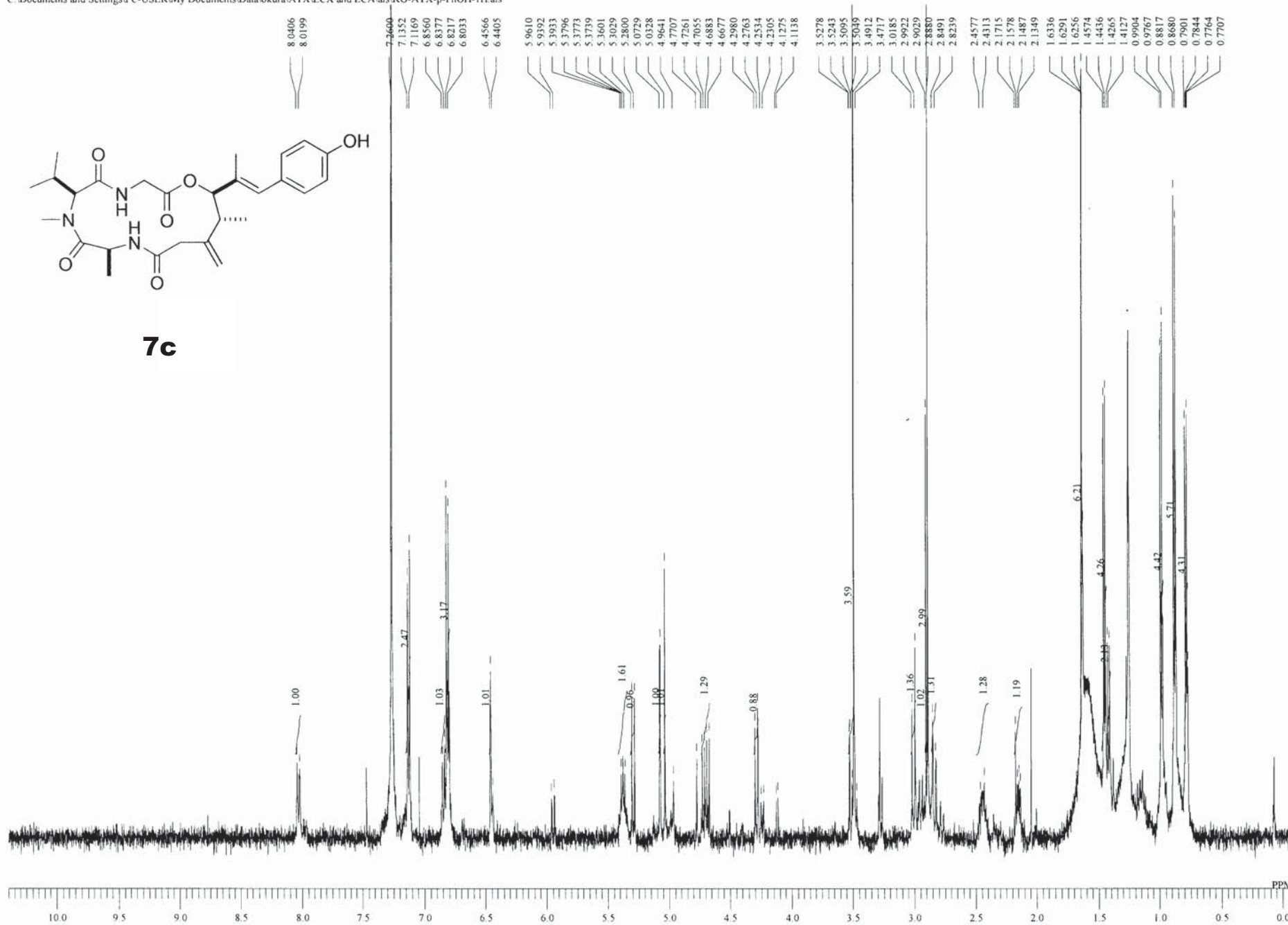
7b

DATIM 03-03-2011 08:59:54
 MENUF
 OBNUC 13C
 OFR 123.26 MHz
 OBSET 2.31 KHz
 OFBIN 6.71 Hz
 PW1 3.20 usec
 DEADT 0.00 usec
 PREDL 0.00000 msec
 IWT 1.0000 sec
 POINT 104856
 SPO 104856
 TIMES 3072
 DUMMY 4
 FREQU 30863.73 Hz
 FLT 155000 Hz
 DELAY 21.06 usec
 ACQTM 0.8493 sec
 PD 10.0000 sec
 ADBIT 16
 RGAIN 60
 BF 1.00 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD single_pulse_dec
 EXPCM
 IRNUC 1H
 IFR 490.15 MHz
 IRSET 9.16 KHz
 IRFIN 7.60 Hz
 IRRPW 92 usec
 IRATN 79
 DFILE KO-ATX-m-PhOH-13C.a
 SF
 LKSET 70.30 KHz
 LKFIN 32.5 Hz
 LKLEV 0
 LGAIN 0
 LKPHS 0
 LKSIG 0
 CSPED 0 Hz
 FILDC
 FILDF

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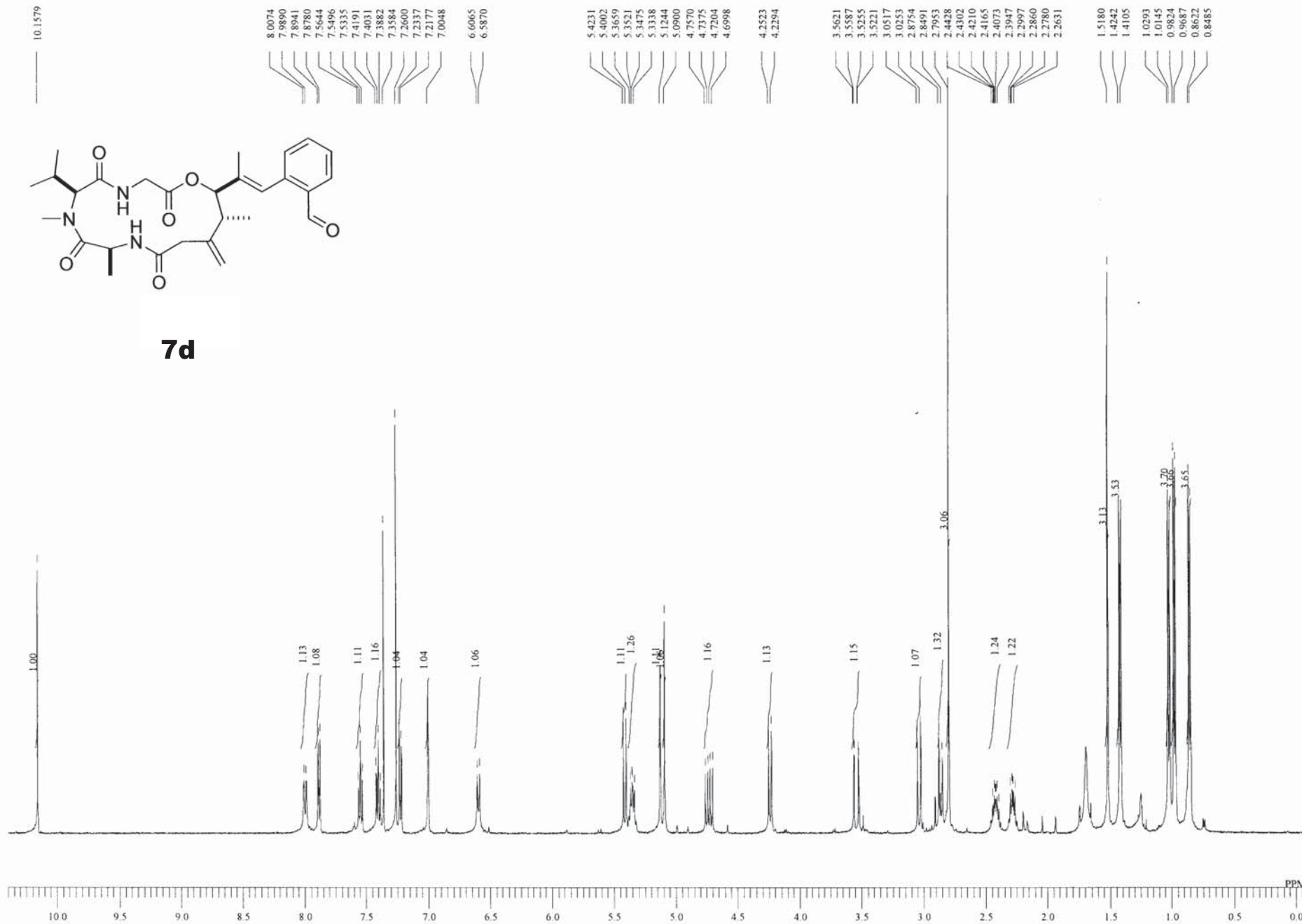


7c



DATIM 04-03-2011 00:08:14
 MENUF
 OBNUC 1H
 OFR 490.15 MHz
 OBSET 9.16 KHz
 OFBIN 7.60 Hz
 PW1 8.55 usec
 DEADT 0.00 usec
 PREDL 0.00000 msec
 IWT 1.0000 sec
 POINT 13107
 SPO 13107
 TIMES 8
 DUMMY 1
 FREQU 7352.83 Hz
 FLT 37000 Hz
 DELAY 13.52 usec
 ACQTM 1.7826 sec
 PD 1.5000 sec
 ADBIT 16
 RGAIN 48
 BF 0.01 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD single_pulse ex2
 EXPCM
 IRNUC 1H
 IFR 490.15 MHz
 IRSET 9.16 KHz
 IRFIN 7.60 Hz
 IRRPW 118 usec
 IRATN 79
 DFILE KO-ATX-p-PhOH-1H.als
 SF
 LKSET 70.30 KHz
 LKFIN 32.5 Hz
 LKLEV 0
 LGAIN 0
 LKPHS 0
 LKSIG 0
 CSPED 0 Hz
 FILDC
 FILDF

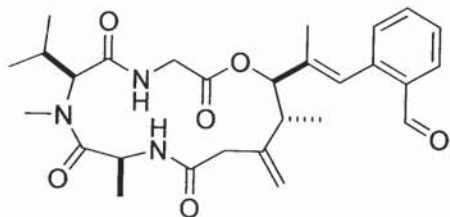
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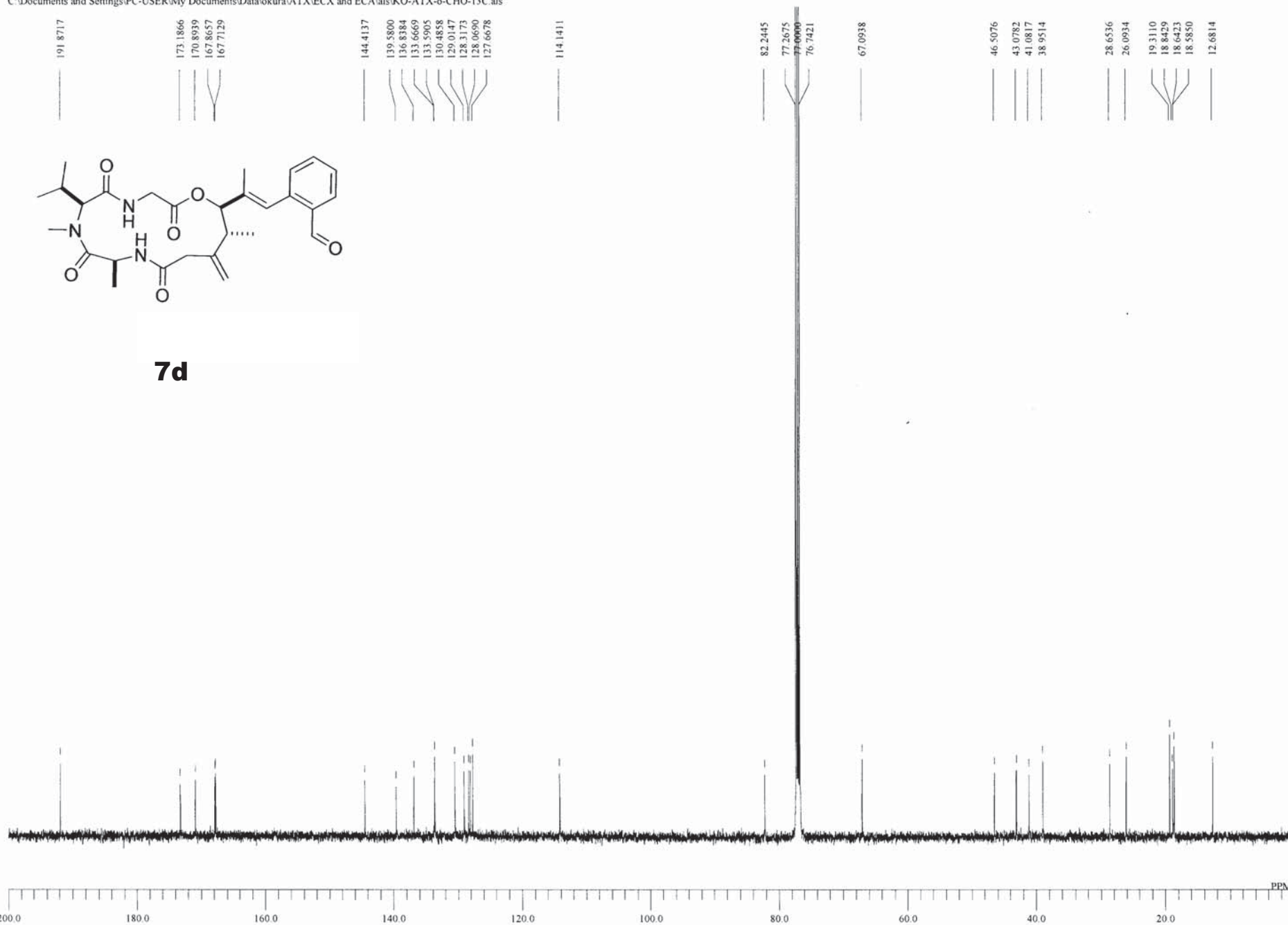
DATIM	06-03-2011 22:29:49
MENUP	
OBNUC	¹ H
OFR	490.15 MHz
OBSET	9.16 KHz
OBFIN	7.60 Hz
PW1	8.55 usec
DEADT	0.00 usec
PREDL	0.00000 msec
IWT	1.0000 sec
POINT	13107
SPO	13107
TIMES	8
DUMMY	1
FREQU	7352.83 Hz
FLT	37000 Hz
DELAY	13.52 usec
ACQTM	1.7826 sec
PD	1.5000 sec
ADBIT	16
RGAIN	44
BF	0.01 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	single_pulse ex2
EXPCM	
IRNUC	¹ H
IFR	490.15 MHz
IRSET	9.16 KHz
IRFIN	7.60 Hz
IRRPW	118 usec
IRATN	79
DFILE	KO-ATX-o-CHO-1H.als
SF	
LKSET	70.30 KHz
LKFIN	32.5 Hz
LKLEV	0
LGAIN	0
LKPHS	0
LKSIG	0
CSPED	0 Hz
FILDC	
FILDF	

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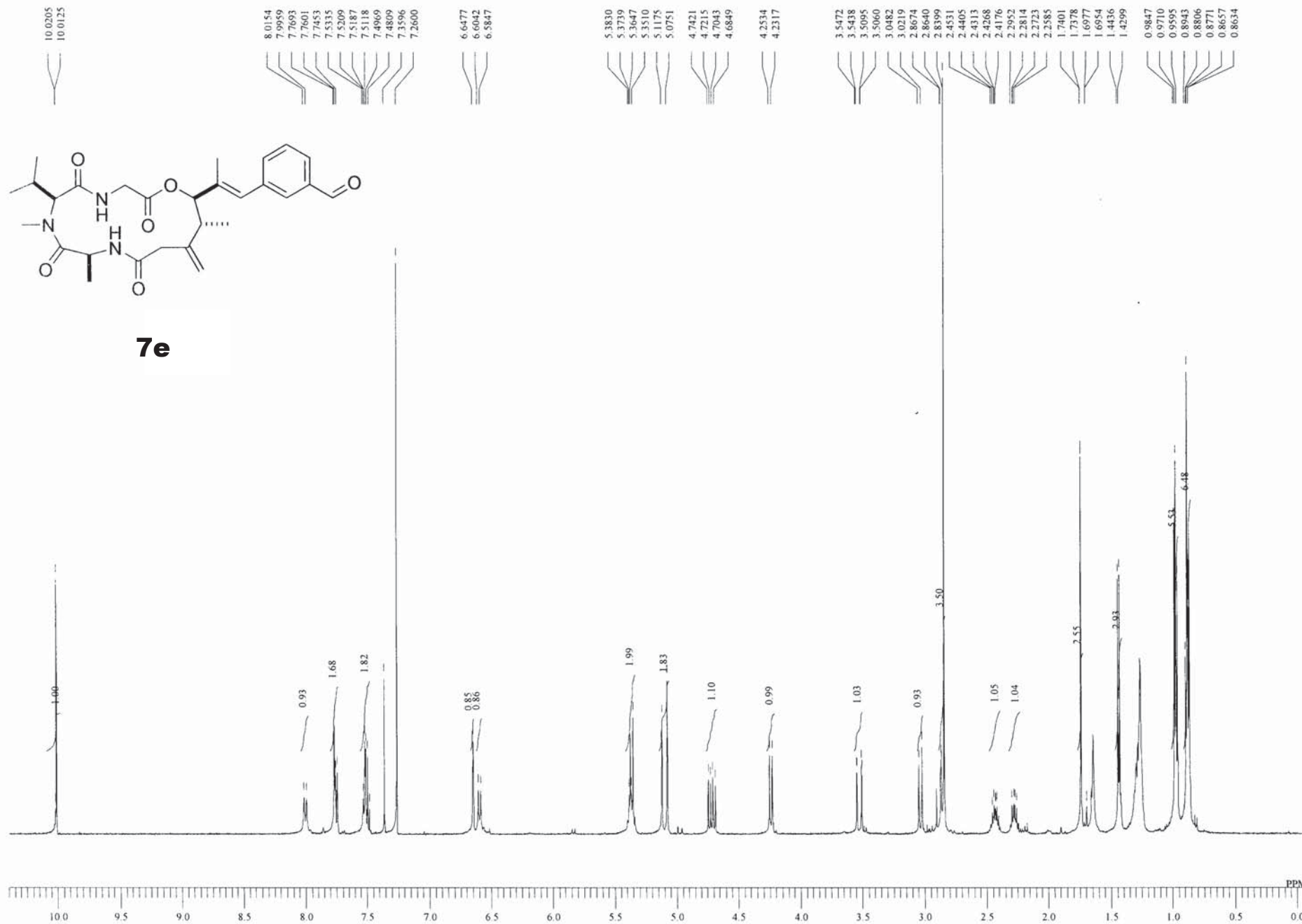
DATIM 07-03-2011 08:00:34
 MENUF
 OBNUC 13C
 OFR 123.26 MHz
 OBSET 2.31 KHz
 OFBIN 6.71 Hz
 PW1 3.20 usec
 DEADT 0.00 usec
 PREDL 0.00000 msec
 IWT 1.0000 sec
 POINT 26214
 SPO 26214
 TIMES 3072
 DUMMY 4
 FREQU 30863.73 Hz
 FLT 155000 Hz
 DELAY 21.06 usec
 ACQTM 0.8493 sec
 PD 10.0000 sec
 ADBIT 16
 RGAIN 60
 BF 1.00 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD single_pulse_dec
 EXPCM
 IRNUC 1H
 IFR 490.15 MHz
 IRSET 9.16 KHz
 IRFIN 7.60 Hz
 IRRPW 92 usec
 IRATN 79
 DFILE KO-ATX-o-CHO-13C.als
 SF
 LKSET 70.30 KHz
 LKFIN 32.5 Hz
 LKLEV 0
 LGAIN 0
 LKPHS 0
 LKSIG 0
 CSPED 0 Hz
 FILDC
 FILDF



7d

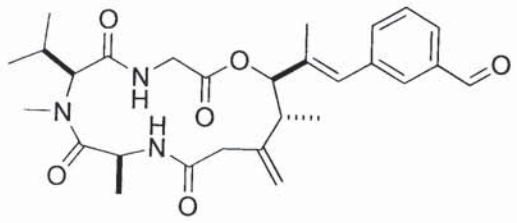
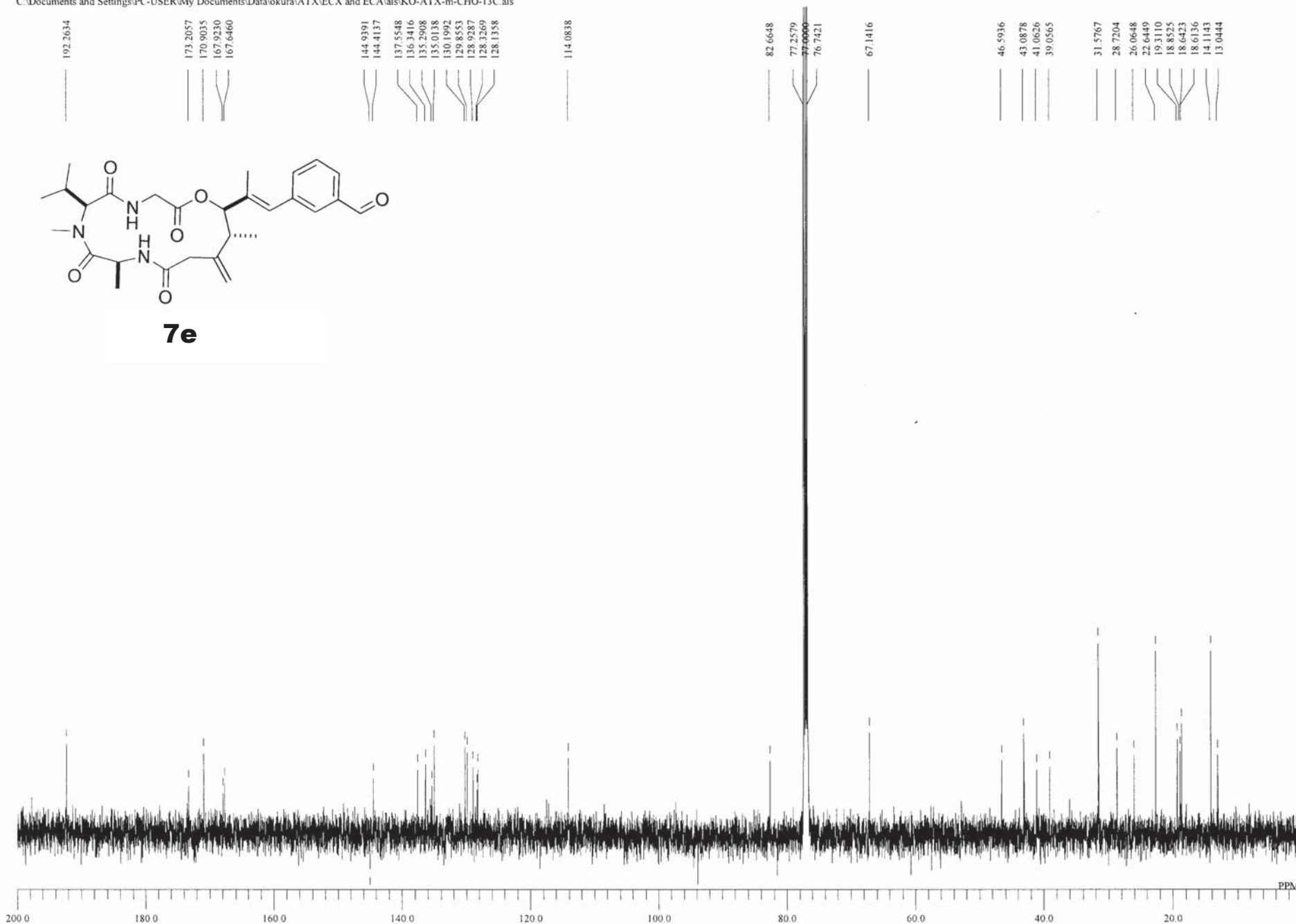


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DATIM 30-01-2011 03:47:30
 MENUF
 OBNUC 1H
 OFR 490.15 MHz
 OBSET 9.16 KHz
 OBFIN 7.60 Hz
 PW1 8.55 usec
 DEADT 0.00 usec
 PREDL 0.00000 msec
 IWT 1.0000 sec
 POINT 13107
 SPO 13107
 TIMES 8
 DUMMY 1
 FREQU 7352.83 Hz
 FLT 37000 Hz
 DELAY 13.52 usec
 ACQTM 1.7826 sec
 PD 1.5000 sec
 ADBIT 16
 RGAIN 44
 BF 0.01 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD single_pulse.ex2
 EXPCM
 IRNUC 1H
 IFR 490.15 MHz
 IRSET 9.16 KHz
 IRFIN 7.60 Hz
 IRRPW 118 usec
 IRATN 79
 DFILE KO-ATX-m-CHO-1H.als
 SF
 LKSET 70.30 KHz
 LKFIN 32.5 Hz
 LKLEV 0
 LGAIN 0
 LKPHS 0
 LKSIG 0
 CSPED 0 Hz
 FILDC
 FILDF

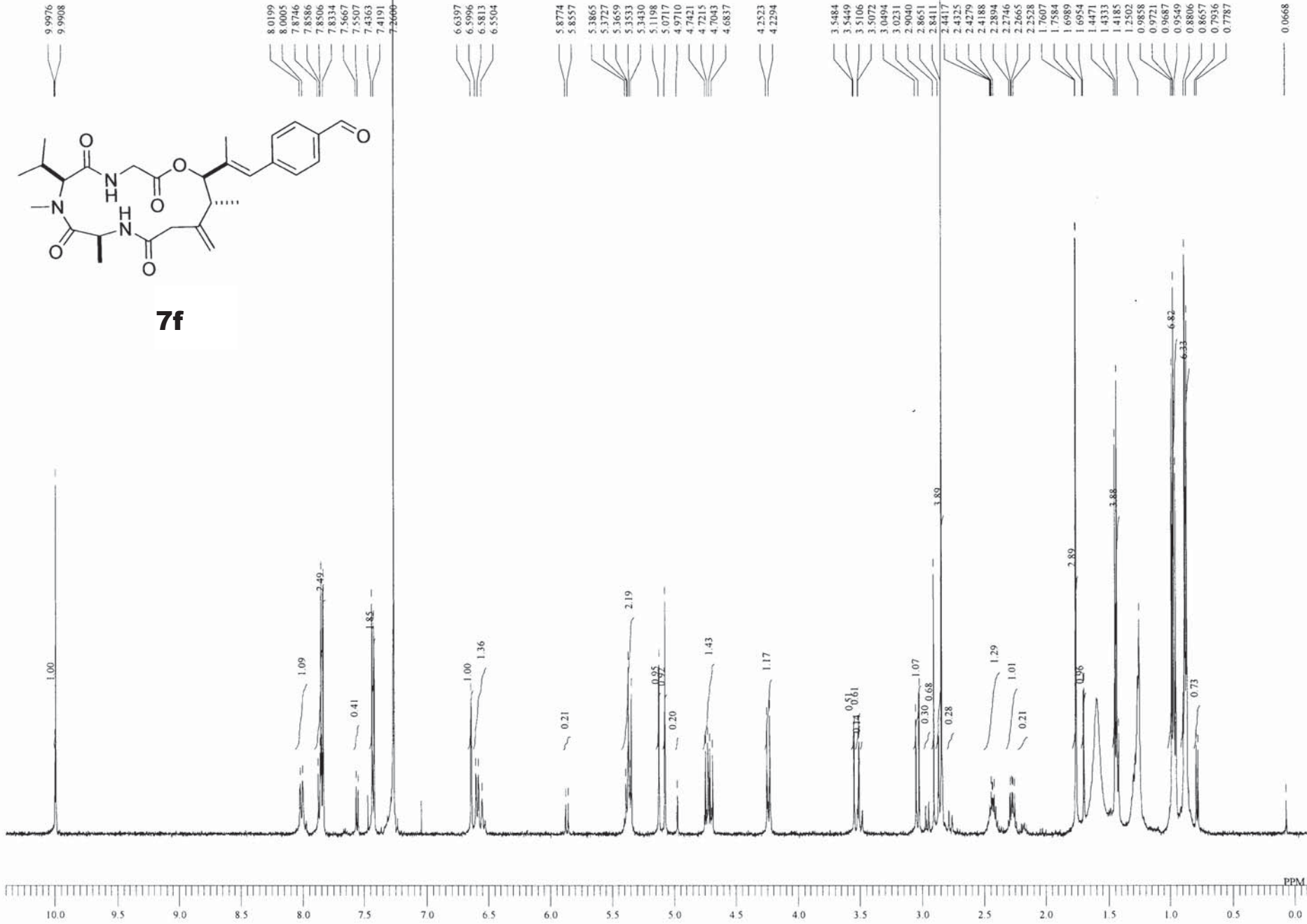
C:\Documents and Settings\PC-USER\My Documents\Data\okura\ATX\ECX and ECA\als\KO-ATX-m-CHO-13C.als



7e

DATIM 30-01-2011 06:56:44
 MENUF
 OBNUC 13C
 OFR 123.26 MHz
 OBSET 2.31 KHz
 OFBIN 6.71 Hz
 PWI 3.20 usec
 DEADT 0.00 usec
 PREDL 0.00000 msec
 IWT 1.0000 sec
 POINT 26214
 SPO 26214
 TIMES 1024
 DUMMY 4
 FREQU 30863.73 Hz
 FLT 155000 Hz
 DELAY 21.06 usec
 ACQTM 0.8493 sec
 PD 10.0000 sec
 ADBIT 16
 RGAIN 60
 BF 1.00 Hz
 T1 0.00
 T2 0.00
 T3 90.00
 T4 100.00
 EXMOD single_pulse_dec
 EXPCM
 IRNUC 1H
 IFR 490.15 MHz
 IRSET 9.16 KHz
 IRFIN 7.60 Hz
 IRRPW 92 usec
 IRATN 79
 DFILE KO-ATX-m-CHO-13C.als
 SF
 LKSET 70.30 KHz
 LKFIN 32.5 Hz
 LKLEV 0
 LGAIN 0
 LKPHS 0
 LKSIG 0
 CSPED 0 Hz
 FILDC
 FILDF

C:\Documents and Settings\PC-USER\My Documents\Data\okura\ATX\ECX and ECA\als\KO-ATX-p-aldehyde-1H.als



DATIM 05-01-2010 22:55:09
MENUF 1H
OBNUC 1H
OFR 490.15 MHz
OBSET 9.16 KHz
OBFIN 7.60 Hz
PWI 8.55 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 13107
SPO 13107
TIMES 32
DUMMY 1
FREQU 7352.83 Hz
FLT 37000 Hz
DELAY 13.52 usec
ACQTM 1.7826 sec
PD 1.5000 sec
ADBIT 16
RGAIN 48
BF 0.01 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM 1H
IRNUC 1H
IFR 490.15 MHz
IRSET 9.16 KHz
IRFIN 7.60 Hz
IRRPW 118 usec
IRATN 79
DFILE KO-ATX-p-aldehyde-1H:
SF 70.30 KHz
LKSET 32.5 Hz
LKFIN 0
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF