Supplementary Material (ESI) for Organic & Biomolecular Chemistry

Supporting Information

Iron-mediated one-pot formal nitrocyclization onto unactivated alkenes

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General. All reactions were carried out in a flame-dried glassware under nitrogen atmosphere. Amines were distilled over calcium hydride. All reagents were purchased commercially and used without further purification. Melting points are uncorrected. IR spectra were recorded on a commercial FT/IR spectrometer. ^{1}H NMR spectra were recorded at 600 and 400 MHz spectrometers; chemical shifts (δ) are quoted relative to tetramethylsilane. ^{13}C NMR spectra were recorded at 150 and 100 MHz spectrometers with complete proton decoupling; chemical shift (δ) are quoted relative to the residual signals of chloroform. Silica gel column chromatography was carried out on silica gel 60N. Mass spectra were recorded on a high-resolution mass spectrometer in fast atom bombardment mode (FAB).

Starting materials. 1l was commercially available. **1a**,[1] **1b**,[2] **1c**,[2] **1d**,[2] **1e**,[2] **1f**,[3] **1g**,[2] **1h**,[3] **1i**,[4] **1j**,[5] **1k**,[1] **1m**,[2] **1n**,[2] **1o**,[6] **1p**,[7] **1q**[8] and **1r**[6] were prepared according to literatures.

1-(4-Methylphenylsulfonyl)-2-nitromethylpyrrolidine (2a). 70% yield. Colourless crystals, mp 91.5–92 °C (hexane-EtOAc). IR ($CHCl_3$) υ 1560, 1352, 1161 cm^{-1} ;^{1}H NMR (400 MHz, $CDCl_3$) δ 1.63–1.68 (1H, m), 1.80–1.87 (3H, m), 2.46 (3H, s), 3.09–3.15 (1H, m), 3,47–3.52 (1H, m), 4.16–4.21 (1H, m), 4.41 (1H, dd, *J* = 12.7, 9.8 Hz), 4.93 (1H, dd, *J* = 12.7, 3.9 Hz), 7.37 (2H, d, *J* = 8.0 Hz), 7.76 (2H, d, *J* = 8.0 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.5, 23.5, 29.8, 49.3, 56.8, 78.5, 127.6, 130.0, 133.1, 144.2. Anal. Calcd for $C_{12}H_{16}N_2O_4S$: C, 50.69; H, 5.67; N, 9.85. Found: C, 50.49; H, 5.62; N, 9.82.

4,4-Dimethyl-1-(4-methylphenylsulfonyl)-2-nitromethylpyrrolidine (2b) :75% yield. Colourless crystals, mp 117.5–118 °C (hexane-EtOAc). IR ($CHCl_3$) υ 1553, 1352, 1219, 1159 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.52 (3H, s), 1.07 (3H, s), 1.67 (1H, dd, *J* = 12.7, 8.0 Hz), 1.86 (1H, dd, *J* = 12.7, 7.3 Hz), 2.46 (3H, s), 3.06 (1H, d, *J* = 10.5 Hz), 3.22 (1H, d, *J* = 10.5 Hz), 4.12–4.20 (1H, m), 4.49 (1H, dd, *J* = 12.7, 9.0 Hz), 5.20 (1H, dd, *J* = 12.7, 8.5 Hz), 7.37 (2H, d, *J* = 8.0 Hz), 7.76 (2H, d, *J* = 8.0 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.6, 25.5, 26.2, 37.6, 44.6, 56.7, 61.4, 79.7, 127.7, 129.9, 133.4, 144.3 . Anal. Calcd for $C_{14}H_{20}N_2O_4S$: C, 53.83; H, 6.45; N, 8.97. Found: C, 53.80; H, 6.41; N, 8.88.

4,4-Diphenyl-1-(4-methylphenylsulfonyl)-2-nitromethylpyrrolidine (2c). 48% yield. Colourless crystals, mp 202.5–203 °C (hexane-EtOAc). IR ($CHCl_3$) υ 1553, 1352, 1228, 1163 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.42 (3H, s), 2.57 (1H, d, *J* = 13.2 Hz), 2.74

(1H, dd, *J* = 13.2, 8.3 Hz), 3.39 (1H, d, *J* = 10.2 Hz), 3.77 (1H, dd, *J* = 13.2, 10.2 Hz), 4.25–4.31 (1H, m), 4.54 (1H, d, *J* = 10.0 Hz), 4.86 (1H, dd, *J* = 13.2, 3.9 Hz), 7.05 (2H, d, *J* = 7.6 Hz), 7.14–7.32 (10H, m), 7.67 (2H, d, *J* = 8.3 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.6, 41.5, 52.2, 56.1, 57.8, 78.1, 126.2, 126.6, 126.8, 127.2, 127.7, 128.8, 129.0, 130.0, 132.3, 143.7, 144.0, 144.3; HRFABMS calcd for $C_{24}H_{25}N_2O_4S$ (M^++H) 437.1535, found: 437.1527.

1-(4-Methylphenylsulfonyl)-2-nitromethyl-4-phenylpyrrolidine (2d). 75% yield (as a mixture of two isomers, 57:43). Colourless oil. IR ($CHCl_3$) υ 1552, 1352, 1224, 1161 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.89 (1H, td, *J* = 12.9, 8.8 Hz, major), 2.00 (1H, td, *J* = 11.5, 8.8 Hz, minor), 2.21 (1H, dd, *J* = 13.2, 6.1 Hz, major), 2.46 (3H, s, major), 2.47 (3H, s, minor), 2.48–2.53 (1H, m, minor), 2.67–2.73 (1H, m, minor), 2.96 (1H, dd, *J* = 10.7, 9.0 Hz, major), 3.42 (1H, t, *J* = 10.7 Hz, major), 3.43–3.51 (1H, m, minor), 3.82 (1H, dd, *J* = 11.7, 7.3 Hz, minor), 3.89 (1H, t-like, *J* = 8.0 Hz, major), 4.23–4.30 (1H, m, minor), 4.36 (1H, td, *J* = 8.8, 3.9 Hz, major), 4.53 (1H, d, *J* = 12.7 Hz, major), 4.55 (1H, dd, *J* = 12.9, 2.0 Hz, minor), 4.99 (1H, dd, *J* = 12.9, 3.9 Hz, major), 5.08, (1H, dd, *J* = 12.9, 4.1 Hz, minor), 7.03 (2H, d, *J* = 6.3 Hz, major), 7.08 (2H, d, *J* = 7.0 Hz, minor), 7.22–7.29 (5H, m), 7.35–7.41 (5H, m), 7.76 (2H, d, *J* = 8.3 Hz, major), 7.80 (2H, d, *J* = 8.6 Hz, minor); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.5, 21.6, 35.8, 38.0, 41.0, 42.6, 54.9, 55.0, 56.6, 57.2, 78.3, 79.1, 126.8, 126.8, 127.4, 127.6, 127.7, 128.3, 128.7, 128.7, 130.0, 132.4, 133.5, 138.0, 138.1, 144.4, 144.5; HRFABMS calcd for $C_{18}H_{21}N_2O_4S$ (M^++H) 361.1222, found: 361.1226.

2-(4-Methylphenylsulfonyl)-3-nitromethylspiro[4,5]decane (2e). 69% yield. Colourless crystals, mp 118.5–119 °C (hexane-EtOAc). IR ($CHCl_3$) υ 1553, 1352, 1221, 1159 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.59–0.63 (1H, m), 0.78 (1H,ddd, *J* = 13.3, 9.2, 3.9 Hz), 1.10–1.50 (8H, m), 1.61 (1H, dd, *J* = 13.3, 8.3 Hz), 1.91 (1H, dd, *J* = 12.9, 7.6), 2.45 (3H, s), 3.19 (1H, d, *J* = 11.0 Hz), 3.24 (1H, d, *J* = 11.0 Hz), 4.09 (1H, ddd, *J* = 16.8, 7.8, 4.1 Hz), 4.45 (1H, dd, *J* = 12.7, 9.0 Hz), 5.19 (1H, dd, *J* = 12.7, 4.1 Hz), 7.36 (2H, d, *J* = 8.0 Hz), 7.76 (2H, d, *J* = 8.0 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.6, 22.8, 23.5, 25.6, 33.9, 36.0, 41.5, 42.8, 56.0, 58.6, 79.9, 127.7, 129.9, 133.3, 144.2. Anal. Calcd for $C_{17}H_{24}N_2O_4S$: C, 57.93; H, 6.86; N, 7.95. Found: C, 58.01; H, 6.75; N, 8.02.

(2*R,5S*)-1-(4-Methylphenylsulfonyl)-2-nitromethyl-5-phenylpyrrolidine (2f).** 80% yield. Colourless crystals, mp 117–117.5 °C (hexane-EtOAc). IR ($CHCl_3$) υ 1553, 1356, 1225, 1163 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.71–1.76 (1H, m), 1.86–1.93 (3H, m),

2.43 (3H, s), 4.36–4.43 (1H, m), 4.49 (1H, dd, *J* = 12.4, 9.3 Hz), 4.71 (1H, t-like, *J* = 6.3 Hz), 5.04 (1H, dd, *J* = 12.4, 4.4 Hz), 7.25–7.34 (7H, m), 7.71 (2H, d, *J* = 8.0 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.4, 28.6, 33.5, 58.4, 64.8, 78.9, 125.9, 127.4, 127.6, 128.4, 129.9, 133.4, 140.9, 144.3. Anal. Calcd for $C_{18}H_{20}N_2O_4S$: C, 59.98; H,5.59; N, 7.77. Found: C, 59.70; H, 5.55; N, 7.65.

1-(4-Methylphenylsulfonyl)-2-nitromethyl-2,4,4-trimethylpyrrolidine (2g). 45% yield. Colourless oil. IR ($CHCl_3$) υ 1553, 1343, 1221, 1156 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.99 (3H, s). 1.07 (3H, s), 1.62 (3H, s), 1.65 (1H, d, *J* = 13.7 Hz), 2.36 (1H, d, *J* = 13.7 Hz), 2.44 (3H, s), 3.07 (1H, d, *J* = 10.0 Hz), 3.12 (1H, d, *J* = 10.0 Hz), 4.80 (1H, d, *J* = 11.2 Hz), 5.03 (1H, d, *J* = 11.2 Hz), 7.31 (2H, d, *J* = 8.0 Hz), 7.75 (2H, d, *J* = 8.0 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.5, 25.2, 27.1, 27.3, 36.2, 51.4, 61.2, 65.9, 83.3, 127.4, 129.6, 137.0, 143.7; HRFABMS calcd for $C_{15}H_{23}N_2O_4S$ (M^++H) 327.1379, found: 327.1372.

1-(Benzyloxycarbonyl)-2-nitromethylpyrrolidine (2h). 31% yield. Colourless oil. IR ($CHCl_3$) υ 1703, 1559, 1414, 1358, 1129 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.88-1.97 (3H, m), 2.11–2.19 (1H, m), 3.45 (2H, br-t, *J* = 6.1 Hz), 4.27 (1H, br-t, *J* = 10.5 Hz), 4.41–5.56 (2H, m), 4.61 (1H, br-d, *J* = 11.5 Hz). 4.81 (1H, d, *J* = 8.3 Hz), 5.15 (2H, br-s), 7.30–7.27 (5H, m); ^{13}C NMR (100 MHz, $CDCl_3$) δ 22.6, 23.4, 28.6, 29.6, 46.7, 46.9, 55.1, 55.6, 67.1, 67.3, 76.1, 77.1, 127.9, 128.1, 128.2, 128.5, 128.6, 136.1, 136.4, 154.3, 154.8; HRFABMS calcd for $C_{13}H_{17}N_2O_4$ (M^++H) 265.1188, found: 265.1182.

5-Nitromethyl-1-phenylpyrrolidin-2-one (2i). 47% yield. Colourless crystals, mp 71.5–72 °C (hexane-EtOAc). IR ($CHCl_3$) υ 1703, 1557, 1498, 1383, 1231 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$) δ 2.06–2.15 (1H, m), 2.48-2.56 (1H, m), 2.61 (1H, ddd, *J* = 17.2, 9.6, 5.0 Hz), 2.69 (1H, ddd, *J* = 17.2, 9.6, 8.2 Hz), 4.37 (1H, dd, *J* = 8.7, 7.6 Hz), 4.53 (1H, dd, *J* = 8.7, 4.1 Hz), 4.83–4.86 (1H, m), 7.25–7.30 (1H, m), 7.38-7.45 (4H, m); ^{13}C NMR (100 MHz, $CDCl_3$) δ 22.0, 30.1, 57.2, 76.1, 124.0, 126.9, 129.5, 136.0, 173.5; HRFABMS calcd for $C_{11}H_{13}N_2O_3$ (M^++H) 221.0926, found: 221.0923. Anal. Calcd for $C_{11}H_{12}N_2O_3$: C, 59.99; H, 5.49; N, 12.72. Found: C, 59.55; H, 5.74; N, 12.78.

1-(4-Methylphenylsulfonyl)-5-nitromethylpyrrolidin-2-one (2j). 60% yield. Colourless crystals, mp 138.5–139 °C (hexane-EtOAc). IR ($CHCl_3$) υ 1748, 1557, 1366, 1171, 1125 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.05–2.13 (1H, m), 2.35–2.45 (2H, m),

2.46 (3H, s), 2.56–2.63 (1H, m), 4.71 (1H, dd, *J* = 13.2, 8.0 Hz), 4.87–4.93 (1H, m), 4.99 (1H, dd, *J* = 13.2, 3.4 Hz), 7.37 (2H, d, *J* = 8.1), 7.95 (2H, d, *J* = 8.1 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.7, 22.5, 29.7, 55.9, 76.6, 128.4, 129.8, 134.7, 145.9, 172.6. Anal. Calcd for $C_{12}H_{14}N_2O_5S$: C, 48.31; H,4.73; N, 9.39. Found: C, 48.22; H, 4.72; N, 9.24.

1-(4-Methylphenylsulfonyl)-2-nitromethylpiperidine (2k). 55% yield. Colourless oil. IR ($CHCl_3$) υ 1560, 1354, 1229, 1159 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.33-1.52 (2H, m), 1.59-1.68 (4H, m), 2.43 (3H, s), 2.95 (1H, dd, *J* = 14.6, 12.7, 2.9 Hz), 3.82 (1H, br-d, *J* = 12.7 Hz), 4.53 (1H, dd, *J* = 11.7, 8.0 Hz), 4.60 (1H, dd, *J* = 11.7, 7.1 Hz), 4.79–4.85 (1H, m), 7.31 (2H, d, *J* = 8.0 Hz), 7.70 (2H, d, *J* = 8.0 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 18.4, 21.5, 24.0, 25.7, 41.2, 50.5, 74.3, 127.0, 129.8, 137.2, 143.7; HRFABMS calcd for $C_{13}H_{19}N_2O_4S$ (M^++H) 299.1066, found: 299.1062.

2-Nitromethyltetrahydrofuran (2l). 53% yield. Colourless oil. IR ($CHCl_3$) υ 1557, 1389, 1082 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.63-1.72 (1H, m), 1.97 (2H, dt, *J* = 14.6, 6.8 Hz), 2.11–2.20 (1H, m), 3.84 (1H, dt, *J* = 8.3, 6.8 Hz), 3.91 (1H, dt, *J* = 8.5, 6.8 Hz), 4.41 (1H, dd, *J* = 12.2, 5.1 Hz), 4.45 (1H, dd, *J* = 12.2, 7.1 Hz), 4.53–4.61 (1H, m); ^{13}C NMR (150 MHz, $CDCl_3$) δ 25.3, 28.9, 68.6, 75.1, 78.9; HRFABMS calcd for $C_5H_{10}NO_3$ (M^++H) 132.0661, found:132.0666.

***cis*-1-(4-Methylphenylsulfonyl)-2-nitromethyloctahydro-1*H*-indole (2m).** 78% yield. (as a mixture of two isomers, 95:5). Colourless oil. IR ($CHCl_3$) υ 1553, 1348, 1229, 1163 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$, for major isomer including the partial peaks of minor isomer) δ 1.09–1.31 (3H, m), 1.40–1.50 (2H, m), 1.55–1.61 (2H, m), 1.65–1.72 (1H, m), 1.90-2.00 (3H, m), 2.45 (3H, s), 3.62 (1H, dt, *J* = 11.0, 6.3 Hz), 3.88 (1H, dt, *J* = 11.2, 5.6 Hz, for minor isomer), 4.04–4.12 (1H, m), 4.30-4.41 (2H, m, for minor isomer), 4.48 (1H, dd, *J* = 12.7, 9.3 Hz), 5.15 (1H, dd, *J* = 12.7, 4.4 Hz), 7.37 (2H, d, *J* = 8.0 Hz), 7.77 (2H, d, *J* = 8.0 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.8 (minor), 20.0, 21.45 (minor), 21.48, 23.4 (minor), 24.0, 25.3 (minor), 25.4, 27.5 (minor), 31.1, 31.2 (minor), 32.6, 34.4 (minor), 36.1, 55.3 (minor), 57.1, 60.9, 61.0 (minor), 78.3 (minor), 80.6, 127.37 (minor), 127.42, 129.7 (minor), 129.9, 134.0, 143.6 (minor), 144.0; HRFABMS calcd for $C_{16}H_{23}N_2O_4S$ (M^++H) 339.1379, found: 339.1383.

***trans*-1-(4-Methylphenylsulfonyl)-2-nitromethyloctahydro-1*H*-indole (2n).** 72% yield. Colourless oil. IR ($CHCl_3$) υ 1553, 1348, 1225, 1162 cm^{-1} ; ^{1}H NMR (400 MHz,

$CDCl_3$) δ 0.91–1.02 (1H, m), 1.16-1.28 (3H, m), 1.35–1.44 (1H, m), 1.57–1.63 (1H, m), 1.70–1.78 (2H, m), 1.80–1.87 (2H, m), 2.30 (1H, td, *J* = 10.7, 3.4 Hz), 2.47 (3H, s), 2.50–2.56 (1H, m), 4.19 (1H, td, *J* = 10.2, 3.9 Hz), 4.41 (1H, dd, *J* = 12.7, 10.8 Hz), 4.91 (1H, dd, *J* =12.7, 3.7 Hz), 7.38 (2H, d, *J* = 8.3 Hz), 7.72 (2H, d, *J* = 8.3 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.6, 24.4, 25.0, 29.3, 32.3, 33.0, 42.7, 57.3, 66.7, 78.8, 127.9, 129.9, 132.3, 144.2; HRFABMS calcd for $C_{16}H_{23}N_2O_4S$ (M^++H) 339.13786, found: 339.13825.

1-Acetyl-2-nitromethylindoline (2p). 69% yield. Colourless crystals, mp 70–70.5 °C (hexane-EtOAc). IR ($CHCl_3$) υ 1659, 1557, 1485, 1393, 1323 cm^{-1}; Rotamers were observed in NMR spectra of **2p**. ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.35 and 2.44 (total 3H, br and s), 2.95 (1H, d, *J* = 17.0 Hz), 3.45 (1H, br-dd, *J* = 16.0, 9.6 Hz), 4.40 (1H, dd, *J* = 11.9, 9.2 Hz), 4.55 and 4.79 (total 1H, br and br-d, *J* = 9.6 Hz), 5.05 and 5.38 (total 1H, both br), 7.06–7.27 and 8.07 (total 4H, m and br); ^{13}C NMR (150 MHz, $CDCl_3$) δ 24.2, 31.2, 56.7, 75.4, 114.7, 124.2, 126.2, 127.9, 130.7, 140.4, 168.6; HRFABMS calcd for $C_{11}H_{13}N_2O_3$ (M^++H) 221.0926, found: 221.0931.

2-Nitromethyl-1-trifluoroacetyl-indoline (2q). 56% yield. Colourless oil. IR ($CHCl_3$) υ 1701, 1560, 1483, 1431, 1379, 1263, 1149 cm^{-1}; Rotamers were observed in NMR spectra of **2q**. ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.06 (1H, d, *J* = 16.3 Hz), 3.57 (1H, dd, *J* = 16.3, 7.6 Hz), 4.42 (1H, dd, *J* = 12.7, 10.5 Hz), 4.62 (1H, d, *J* = 12.7 Hz), 5.34 (1H, br), 7.22-7.35 and 8.04 (total 4H, m and br-d, *J* = 7.1 Hz); ^{13}C NMR (150 MHz, $CDCl_3$) δ 33.7, 57.1, 76.3, 115.9 (q, $J_{C\text{-}F}$ = 286 Hz), 119.0, 125.7, 127.0, 128.5, 128.9, 154.0 (q, $J_{C\text{-}F}$ = 77.6 Hz) HRFABMS calcd for $C_{11}H_{10}F_3N_2O_3$ (M^++H) 275.0644, found: 275.0645.

1-(Benzyloxycarbonyl)-2-nitromethylindoline (2r). 42% yield. Colourless crystals, mp 106.5–107 °C (hexane-EtOAc). IR ($CHCl_3$) υ 1712, 1556, 1485, 1406, 1327, 1281, 1140 cm^{-1} ; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.99 (1H, d, *J* = 16.5 Hz), 3.49 (1H, dd, *J* = 17.2, 9.6 Hz), 4.40 (1H, br), 4.70–4.81 (1H, br), 5.14 (1H, br), 5.31 (2H, br), 7.02 (1H, br), 7.18 (1H, d, *J* = 7.6 Hz), 7.21 (2/3 H, br), 7.35–7.44 (6H,, m), 7.84 (1/3H, br); ^{13}C NMR (150 MHz, $CDCl_3$) δ 31.9 (br), 32.6 (br), 56.7, 67.8 (br), 75.9 (br), 115.6, 123.8, 125.2, 128.1, 128.4, 128.6, 128.7, 135.5, 141.0 (br), 151.9 (br); HRFABMS calcd for $C_{17}H_{17}N_2O_4$ (M^++H) 313.1188, found: 313.1191.

2-Nitromethyl-1-phenylpyrrolidine (3). To a solution of **2i** (5.0 mg, 23 μmol) in THF

(1 mL) was added a 1M solution of BH_3•THF (0.7 mL, 0.70 mmol) at room temperature, and the mixture was heated at reflux for 2 h. To the reaction mixture was added MeOH, and the mixture was further stirred at room temperature for 30 min. 1*N* HCl was added and heated reflux for 1 h. The reaction mixture was basified by addition of a saturate solution of $NaHCO_3$, and the mixture was extracted with EtOAc. The organic phase was washed with brine and dried with $MgSO_4$. The mixture was concentrated and the resultant residue was purified with silica gel chromatography (hexane/EtOAc, 5:1) to give **3** (4.1 mg, 88%) as a colourless oil. IR ($CHCl_3$) υ 1600, 1549, 1485, 1342 cm^{-1} ; ^1H NMR (600 MHz, $CDCl_3$) δ 2.05 (4H, m), 3.19–3.24 (1H, m), 3.49 (1H, t, J = 8.3), 4.19 (1H, dd, J = 11.0, 9.6 Hz), 4.40–4.44 (1H, m), 4.63 (1H, dd, J = 11.0, 2.7 Hz), 6.70 (2H, d, J = 7.6 Hz), 6.78 (1H, t, J = 7.6 Hz), 7.29 (2H, t, J = 8.2 Hz); ^{13}C NMR (150 MHz, $CDCl_3$) δ 22.8, 29.3, 48.1, 57.4, 75.8, 111.9, 117.3, 129.7, 145.7; HRFABMS calcd for $C_{11}H_{14}N_2O_2$ (M^++H) 206.1055, found: 206.1056.

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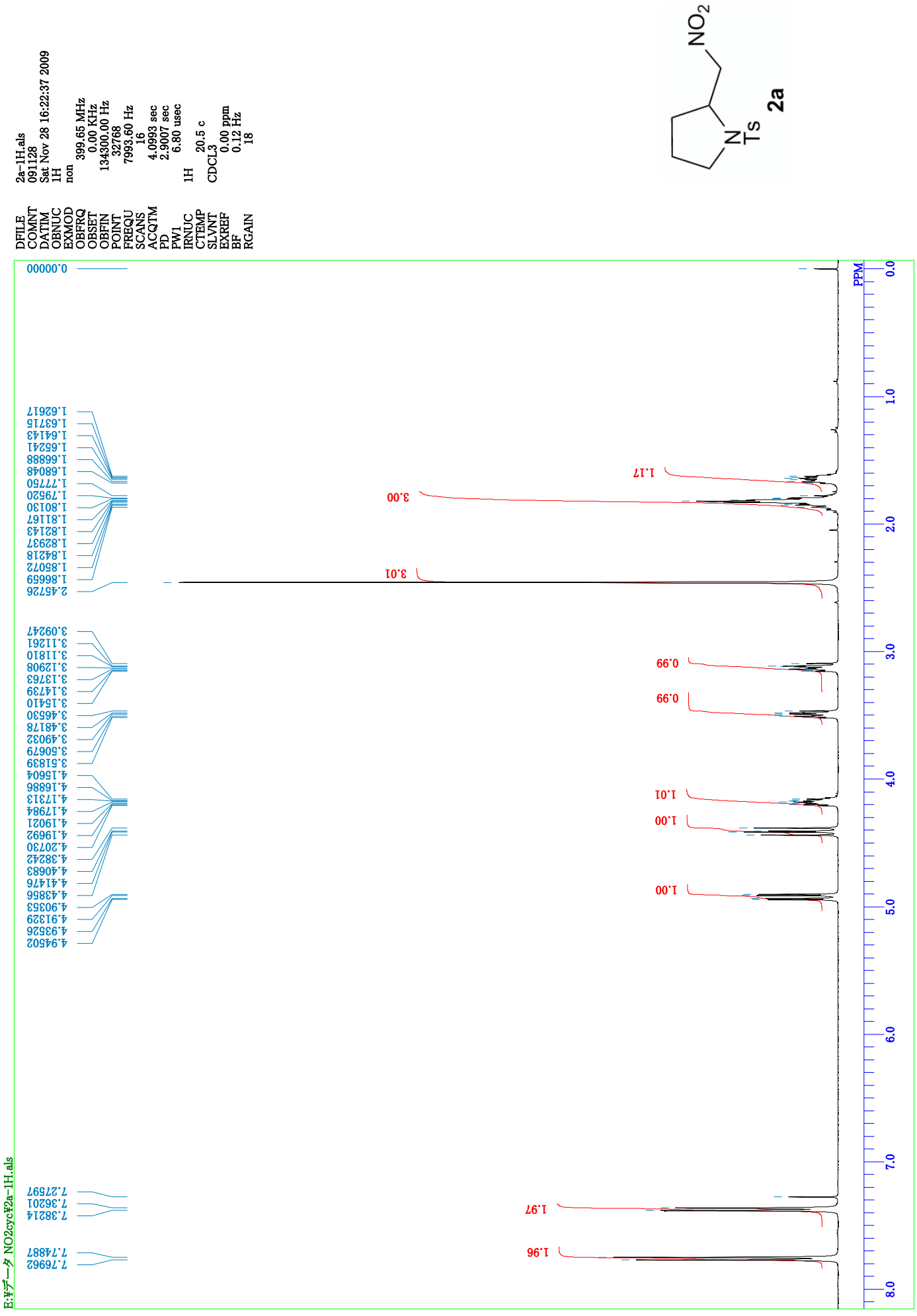
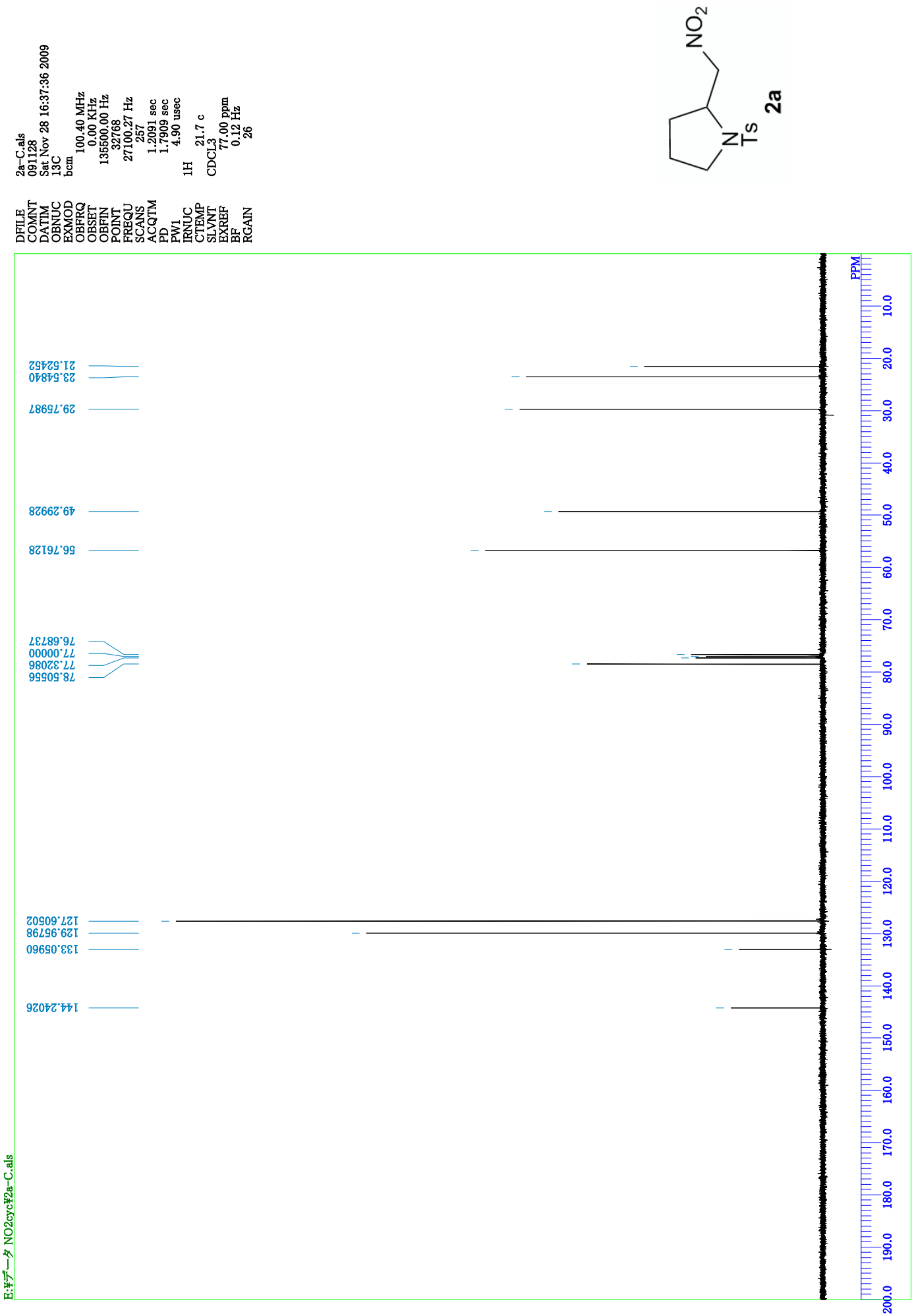
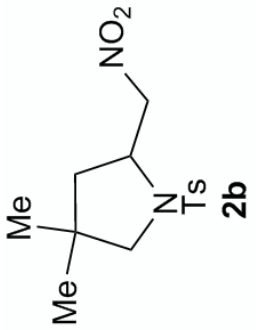

2a
Ts
N
NO2
2a-1H.als
091128
Sat Nov 28 16:22:37 2009
1H
non
399.65 MHz
0.00 KHz
134300.00 Hz
32768
7993.60 Hz
16
4.0993 sec
2.9007 sec
6.80 usec
1H
20.5 c
CDCL3
0.00 ppm
0.12 Hz
18
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
PPM
1.17
3.00
3.01
0.99
0.99
1.01
1.00
1.00
1.97
1.96
E:¥データ NO2cyc¥2a-1H.als
091128

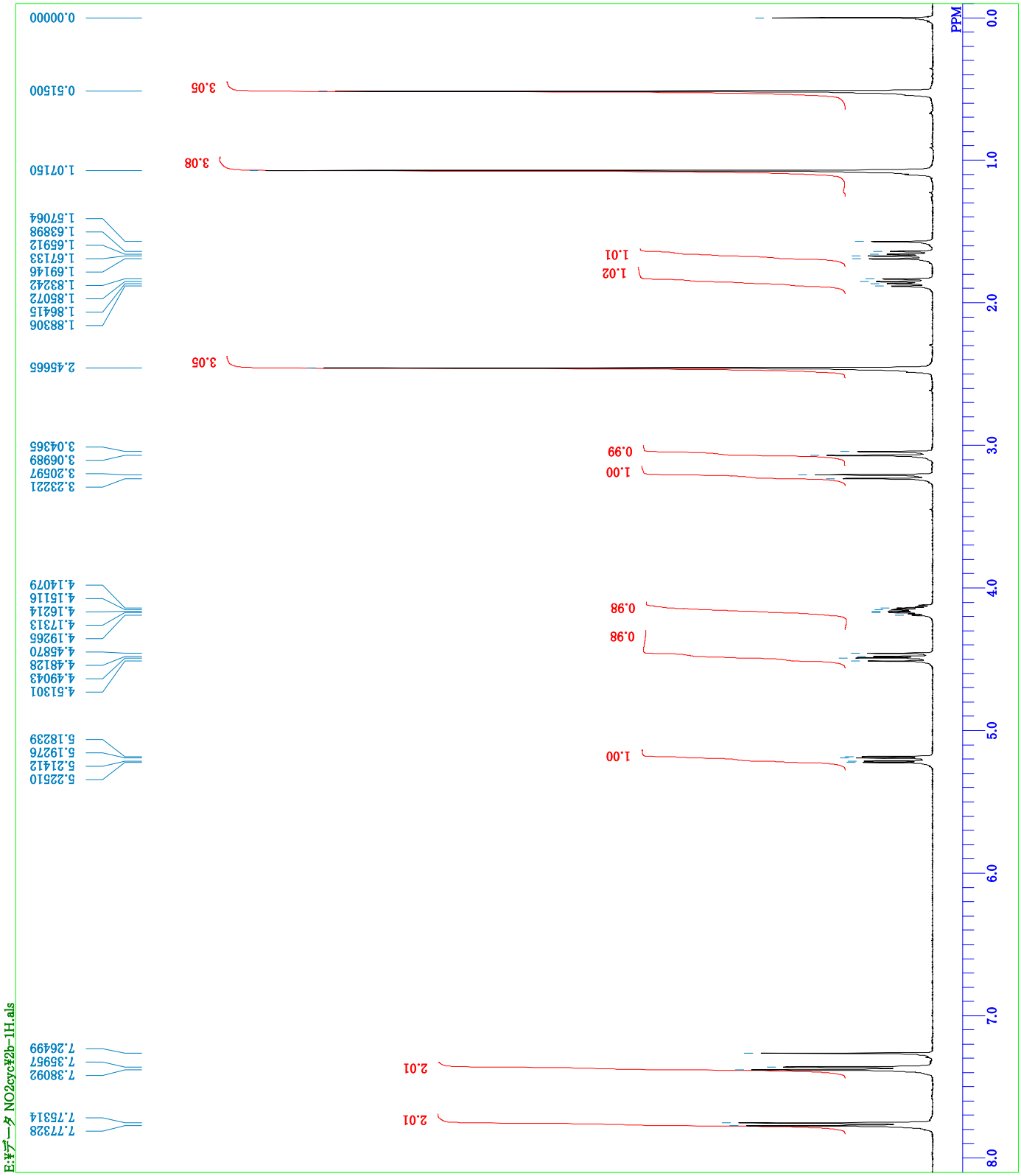

DFILE 2a-C.als
COMNT 091128
DATIM Sat Nov 28 16:37:36 2009
OBNUC 13C
EXMOD bcm
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 257
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 4.90 usec
IRNUC 1H
CTEMP 21.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 26
NO2
N
Ts
2a
21.52452
23.54840
29.75987
49.29928
56.76128
76.68737
77.00000
77.32086
78.50556
127.60502
129.95798
133.05960
144.24026
PPM
10.0
20.0
30.0
40.0
50.0
60.0
70.0
80.0
90.0
100.0
110.0
120.0
130.0
140.0
150.0
160.0
170.0
180.0
190.0
200.0
E:¥データ NO2cyc¥2a-C.als

091128

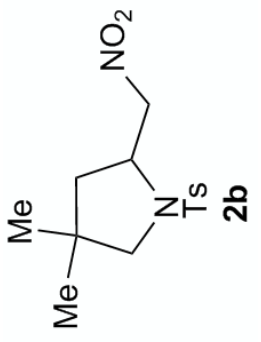


2b-1H.als
091130
Mon Nov 30 21:30:36 2009
1H
non
399.65 MHz
0.00 KHz
134300.00 Hz
32768
7993.60 Hz
16
4.0993 sec
2.9007 sec
6.80 usec
1H
21.7 c
CDCL3
0.00 ppm
0.12 Hz
23

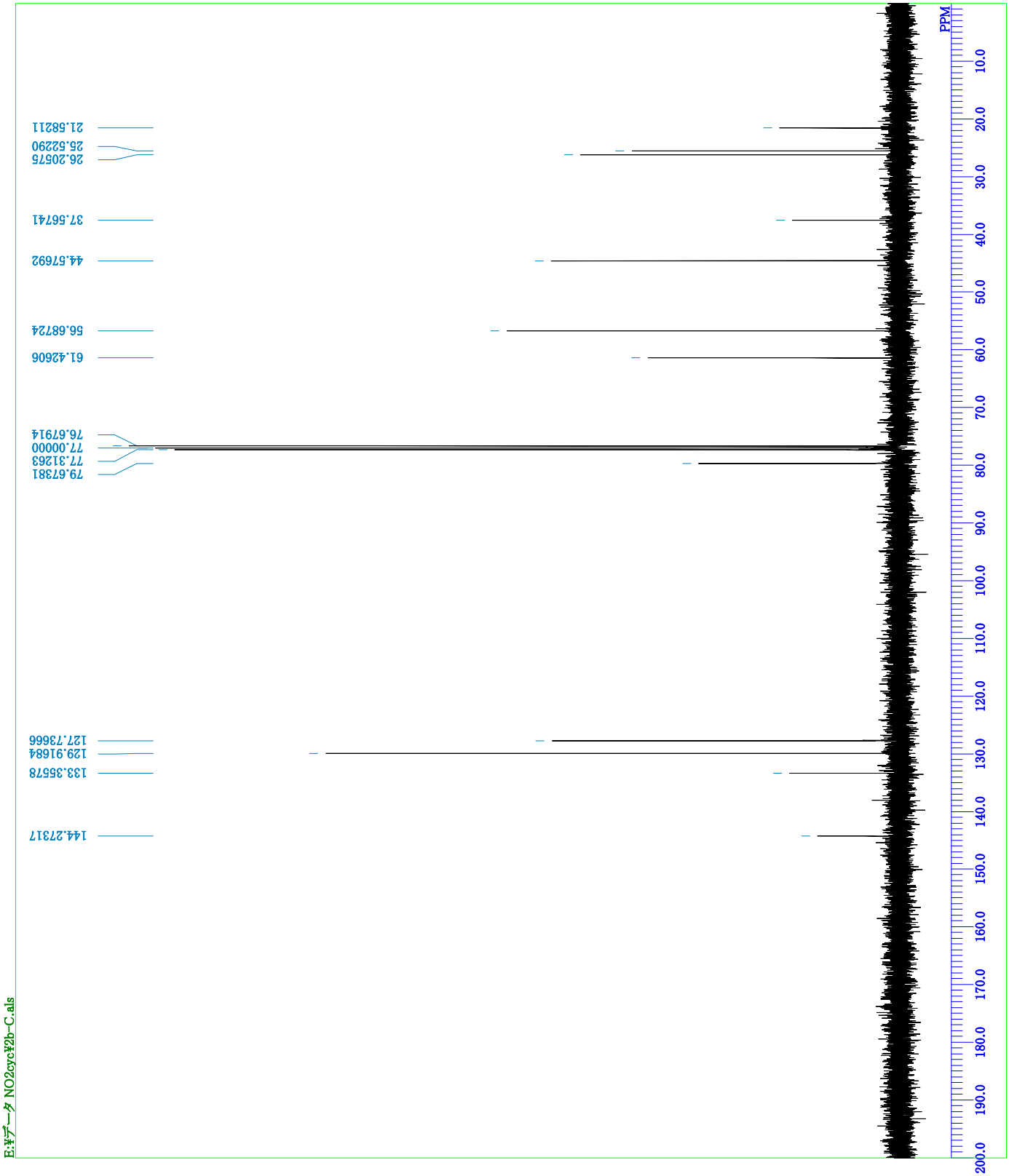
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN



E:¥データ¥NO2cyc¥2b-1H.als
091130

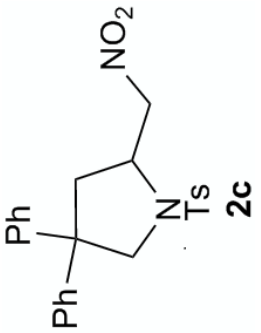


DFILE 2b-C.als
COMNT 091130
DATIM Mon Nov 30 21:56:59 2009
OBNUC 13C
EXMOD bcm
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 512
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 4.90 usec
IRNUC 1H
CTEMP 23.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 27

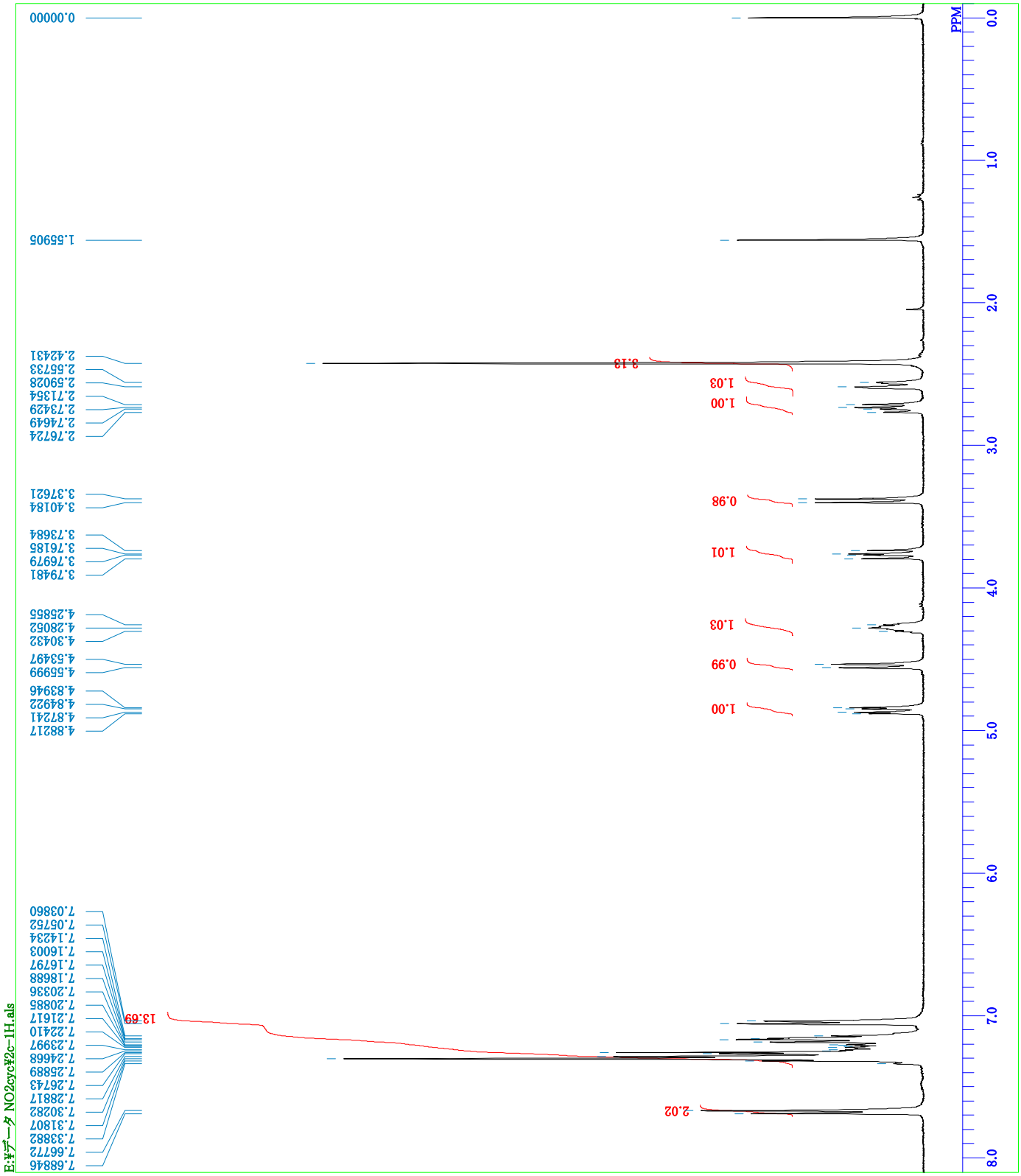


E:¥データ¥NO2cyc¥2b-C.als

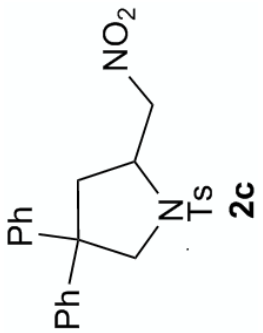
091130



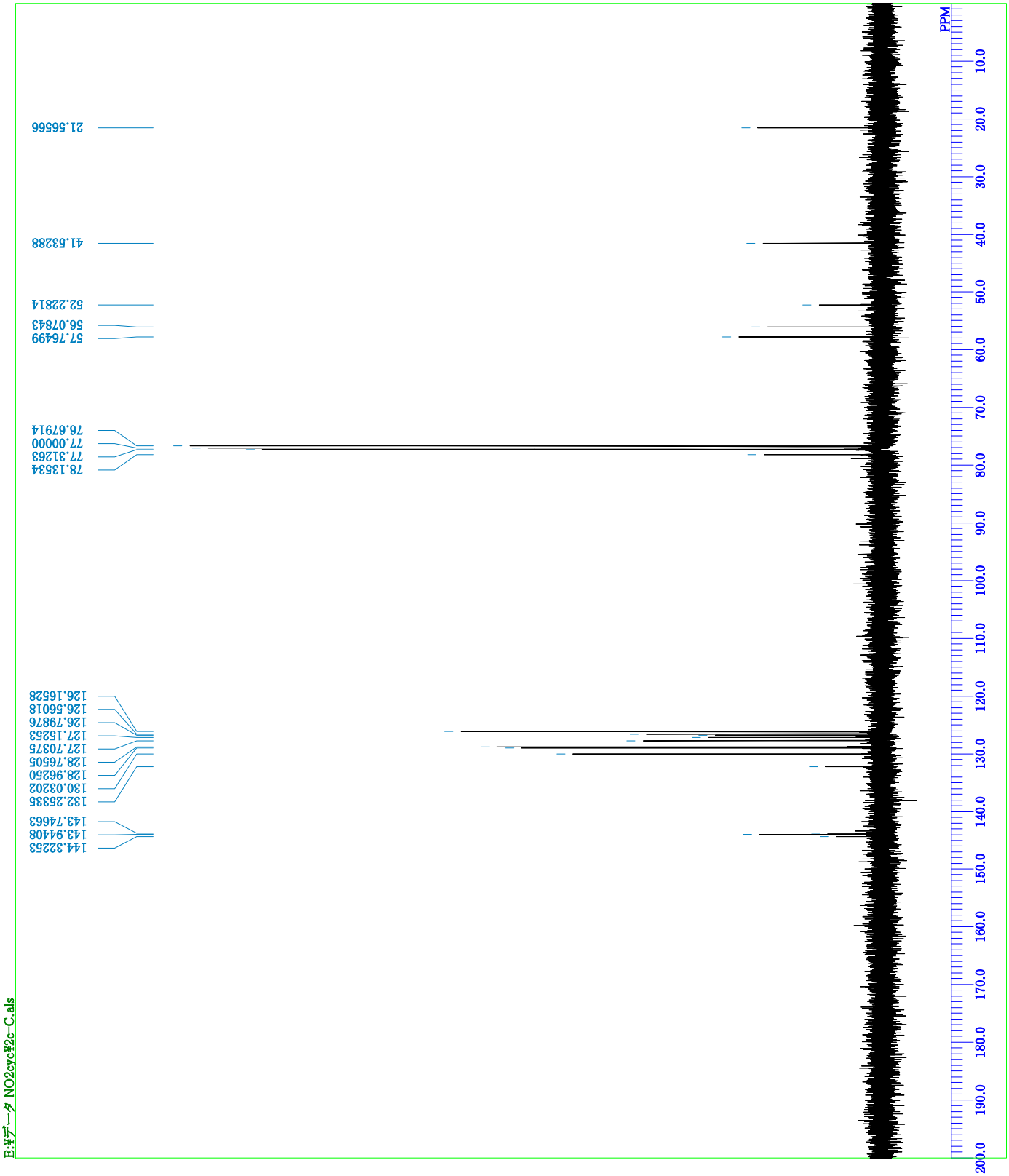
DFILE 2c-1H.als
COMNT 091201
DATIM Tue Dec 01 21:44:10 2009
OBNUC 1H
EXMOD non
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134300.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 16
ACQTM 4.0993 sec
PD 2.9010 sec
PW1 6.80 usec
IRNUC 1H
CTEMP 21.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 24



E:¥データ NO2cyc¥2c-1H.als
091201

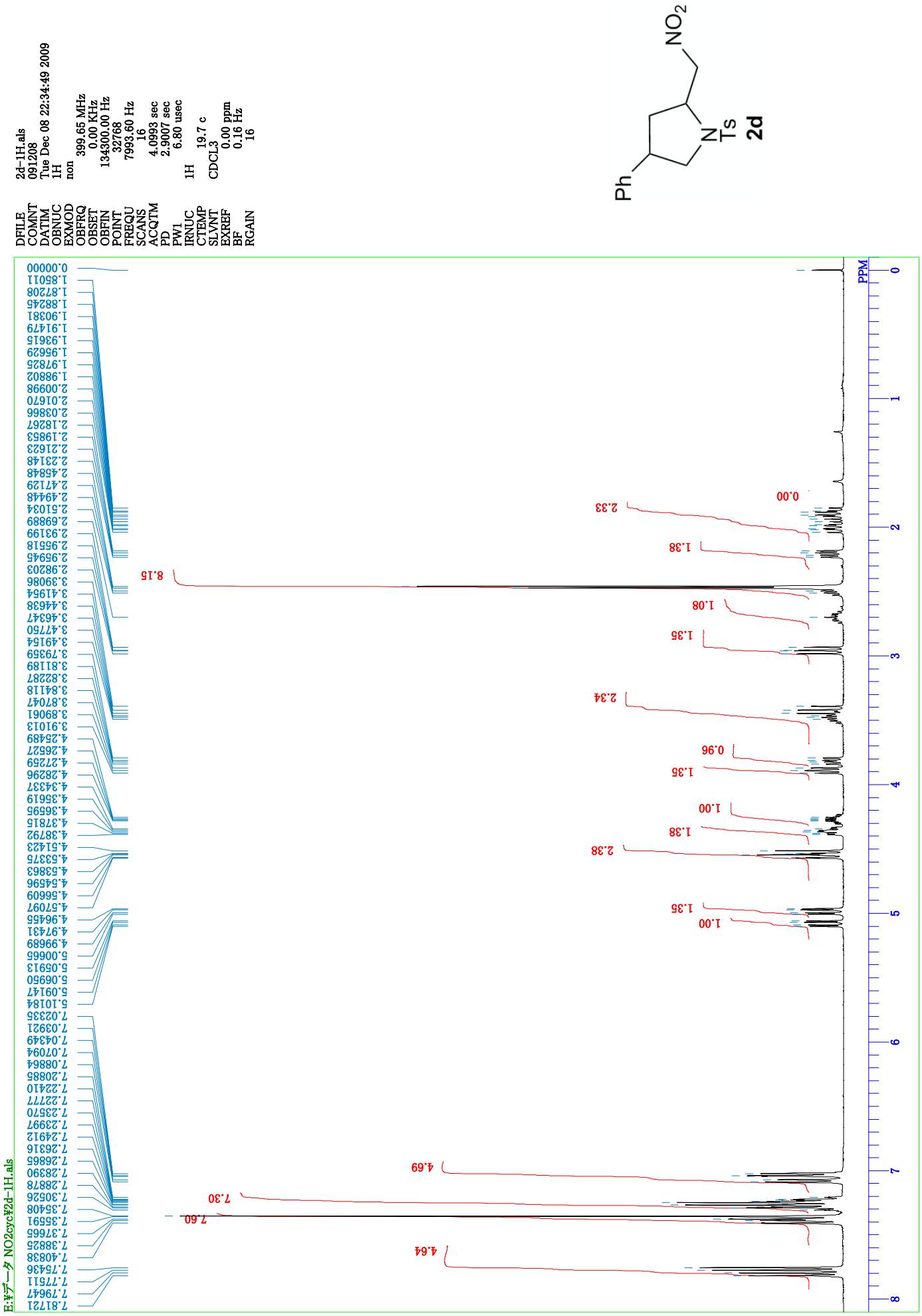


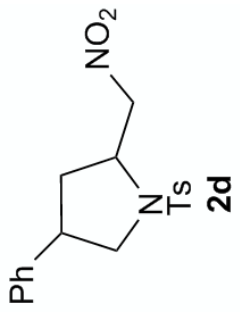
DFILE 2c-C.als
COMNT 091201
DATIM Tue Dec 01 21:57:56 2009
OBNUC 13C
EXMOD bcm
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 257
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 4.90 usec
IRNUC 1H
CTEMP 23.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 19



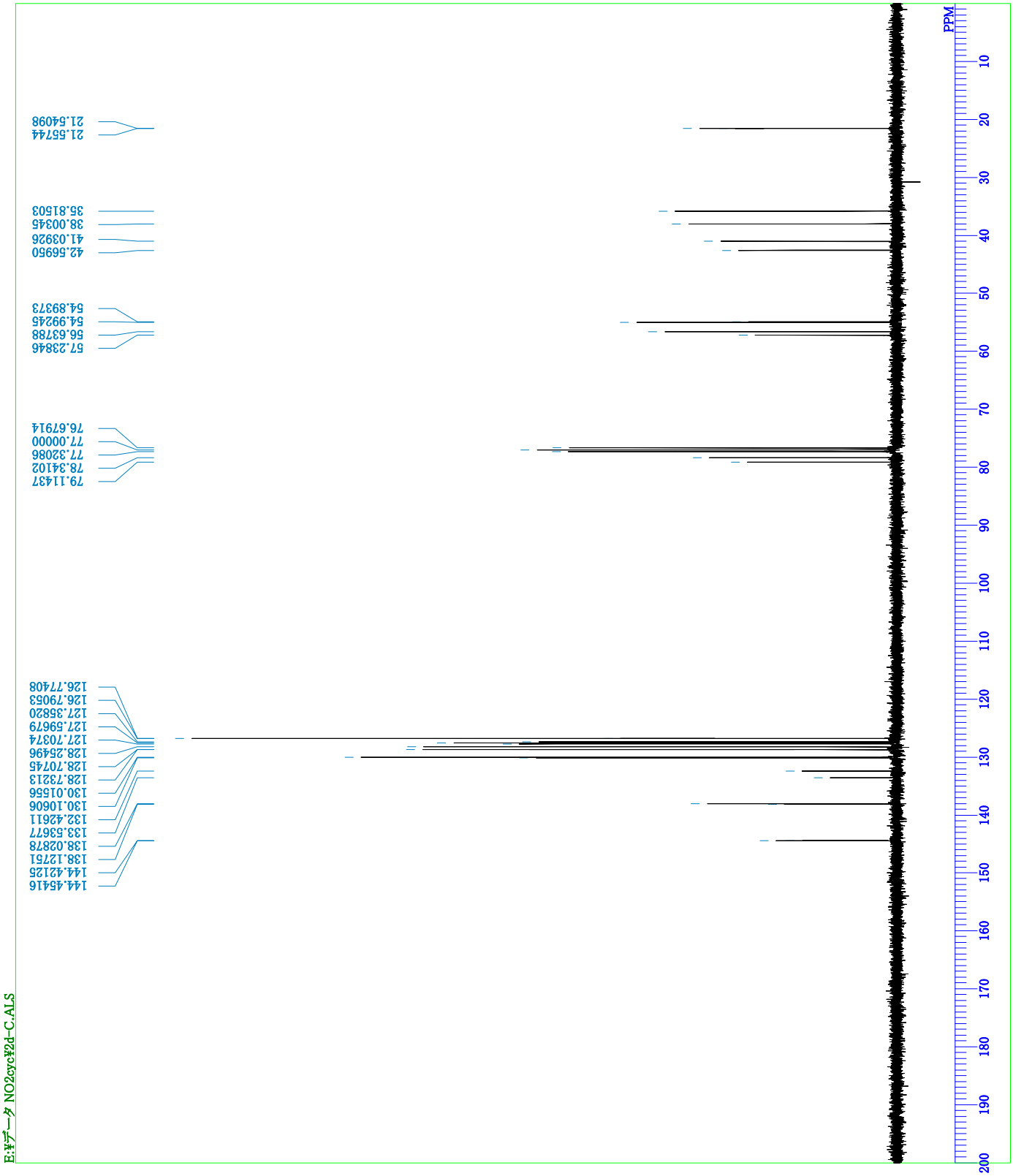
E:¥データ¥NO2cyc¥2c-C.als

091201


DFILE 2d-1H.als
COMNT 091208
DATIM Tue Dec 08 22:34:49 2009
OBNUC 1H
EXMOD non
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134300.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 16
ACQTM 4.0993 sec
PD 2.9007 sec
PW1 6.80 usec
IRNUC 1H
CTEMP 19.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.16 Hz
RGAIN 16
NO2
N
Ts
Ph
2d
PPM
0.00
2.33
1.38
8.15
1.08
1.35
2.34
0.96
1.35
1.00
1.38
2.38
1.35
1.00
4.69
7.30
7.60
4.64
091208
E:¥データ NO2cyc¥2d-1H.als

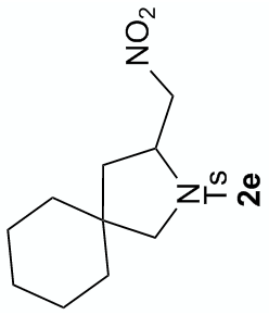


DFILE 2d-C.ALS
COMNT 091208
DATIM Tue Dec 08 22:48:15 2009
OBNUC 13C
EXMOD bcm
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 257
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 4.90 usec
IRNUC 1H
CTEMP 21.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.16 Hz
RGAIN 26

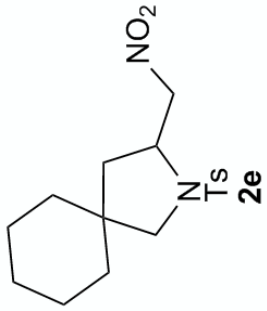


E:¥データ NO2cyc¥2d-C.ALS

091208

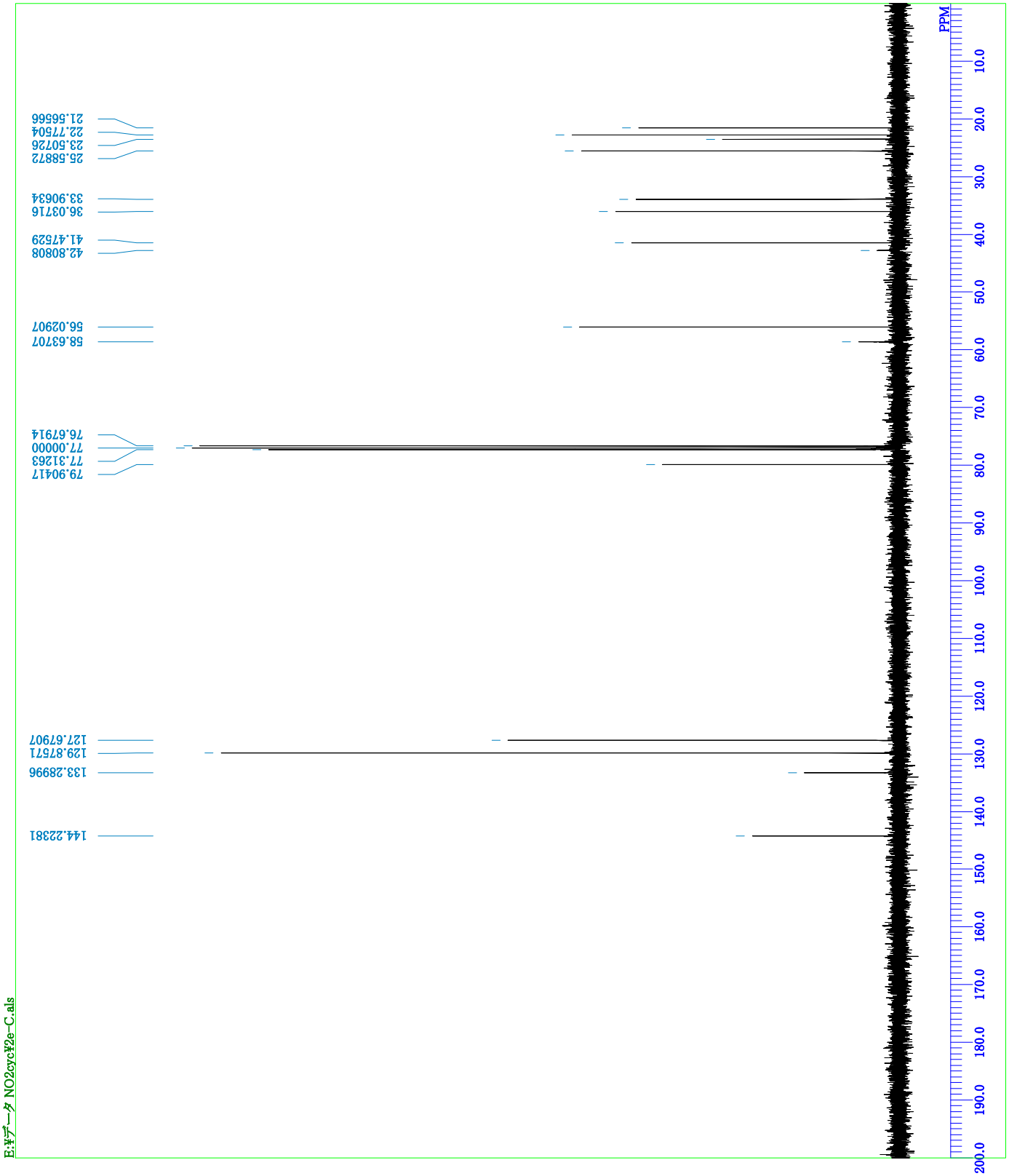

NO2
N
Ts
2e

2e-1H.als
09123
Wed Dec 02 13:19:07 2009
1H
non
399.65 MHz
0.00 KHz
134300.00 Hz
32768
7993.60 Hz
16
4.0993 sec
2.9007 sec
6.80 usec
1H
21.7 c
CDCL3
0.00 ppm
0.12 Hz
23
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
PPM
1.03
1.08
8.28
1.02
1.00
3.10
1.99
1.00
1.00
1.00
1.89
1.83
E:¥データ¥NO2cyc¥2e-1H.als
09123



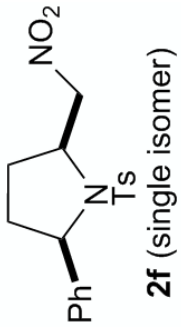
2e-C.als
091203
Thu Dec 03 10:50:01 2009
13C
bcm
100.40 MHz
0.00 KHz
135500.00 Hz
32768
27100.27 Hz
512
1.2091 sec
1.7909 sec
4.90 usec
1H
23.4 c
CDCL3
77.00 ppm
0.16 Hz
24

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN



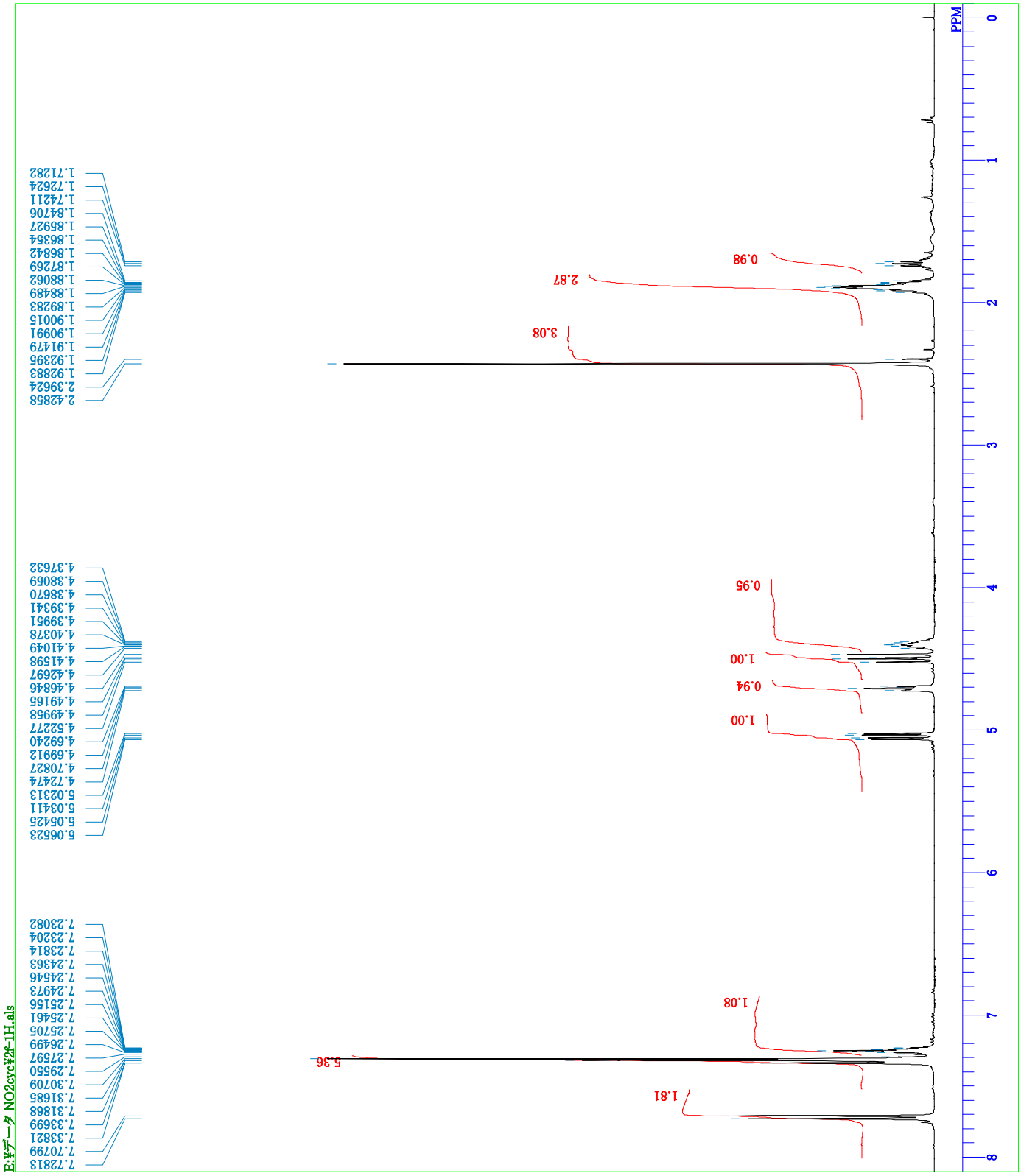
E:¥データ¥NO2cyc¥2e-C.als

091203



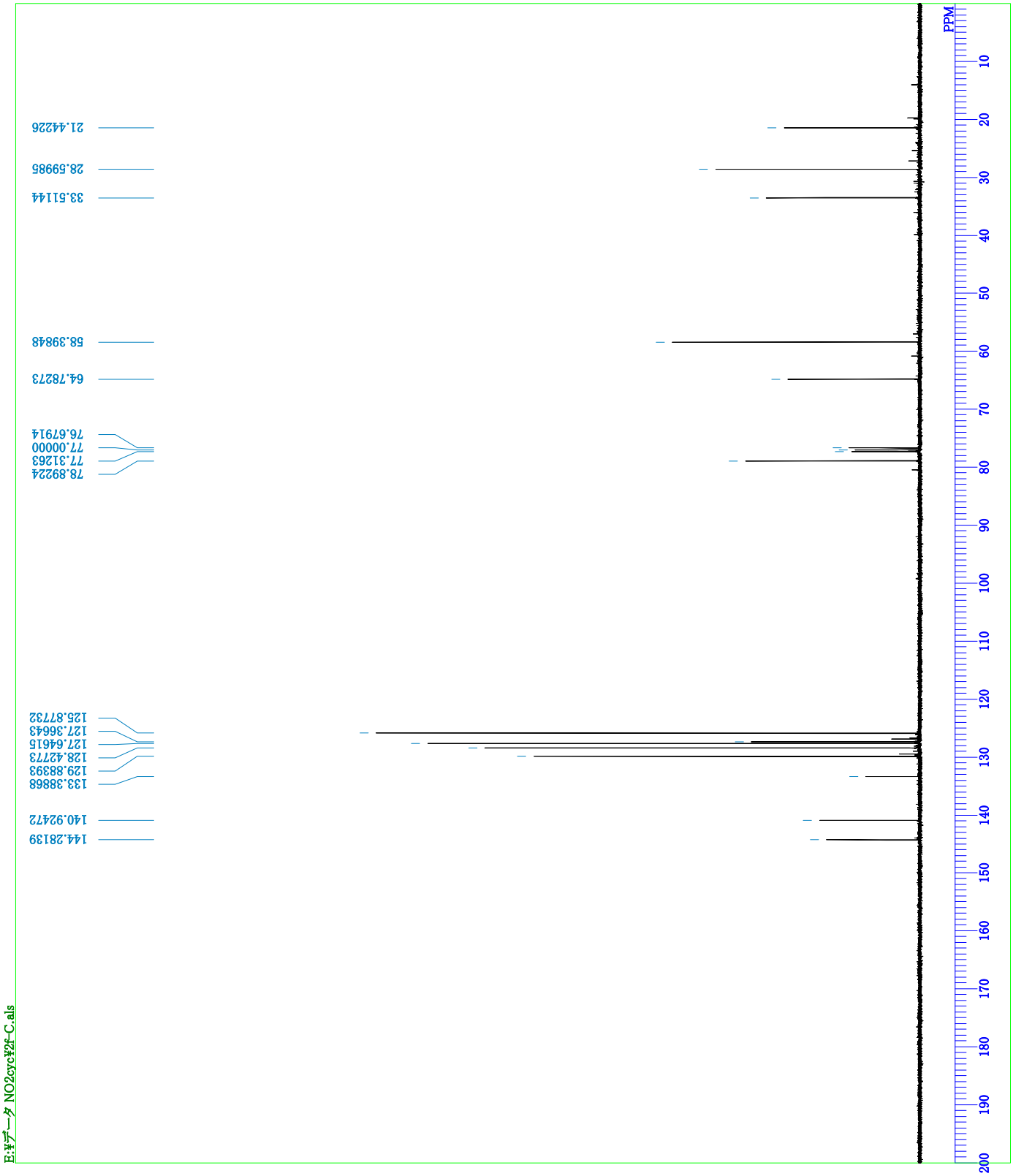
2f (single isomer)

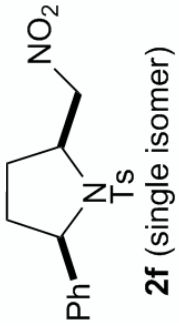
DFILE 2f-1H.als
COMNT 091209
DATIM Wed Dec 09 15:52:53 2009
OBNUC 1H
EXMOD non
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134300.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 16
ACQTM 4.0993 sec
PD 2.9010 sec
PW1 6.80 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.16 Hz
RGAIN 15



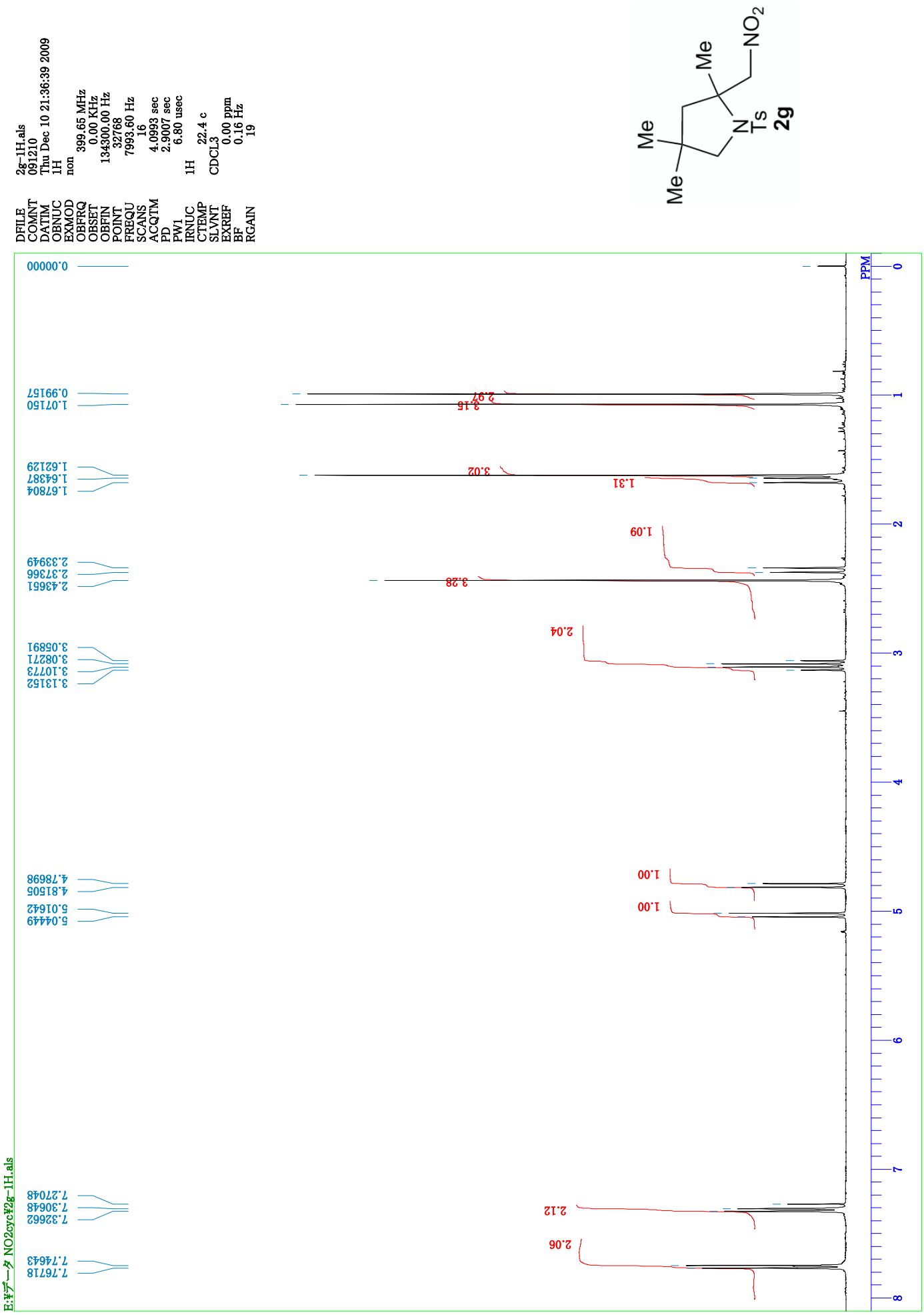
091209

E:¥データ NO2cyc¥2f-1H.als

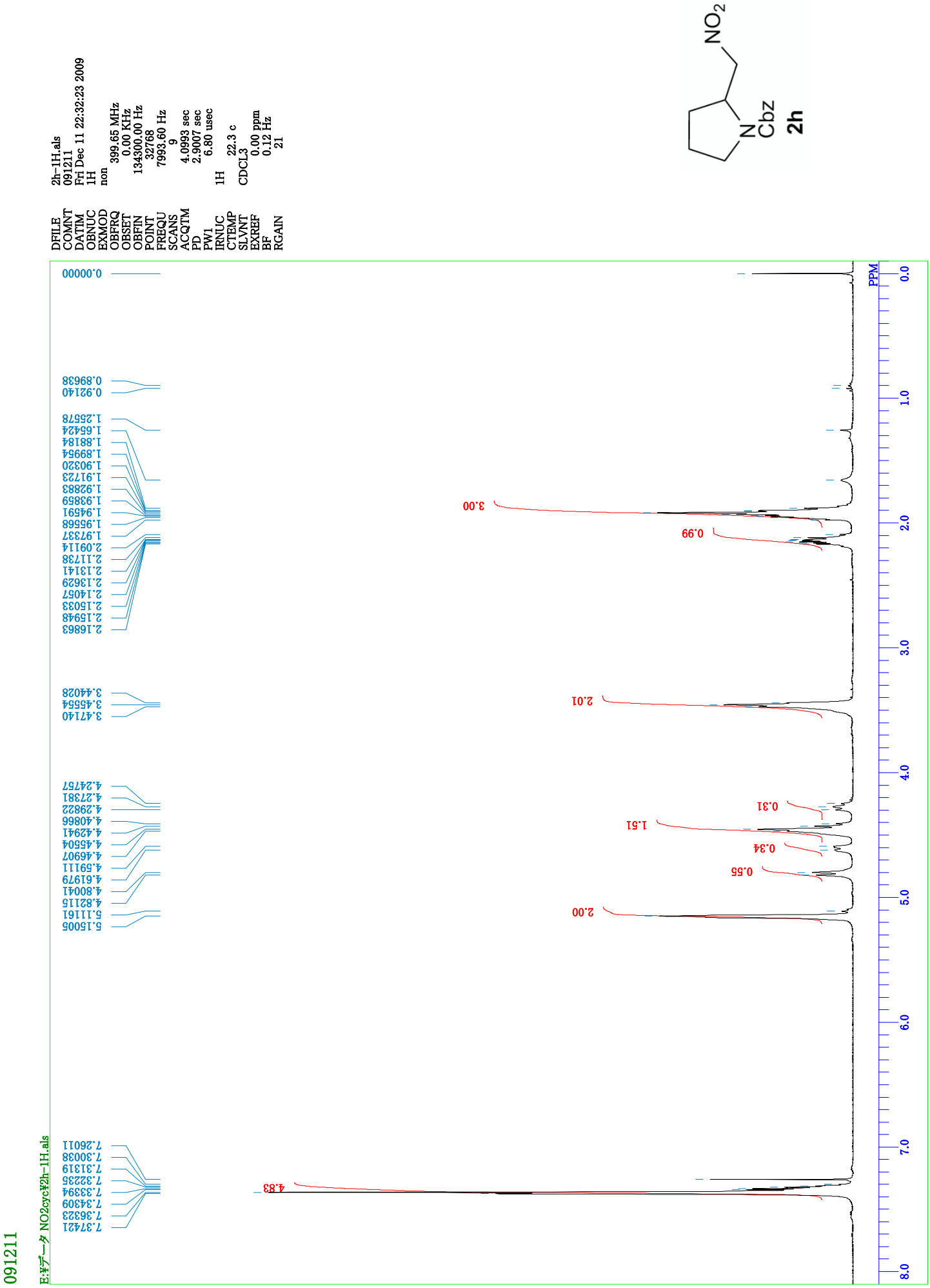


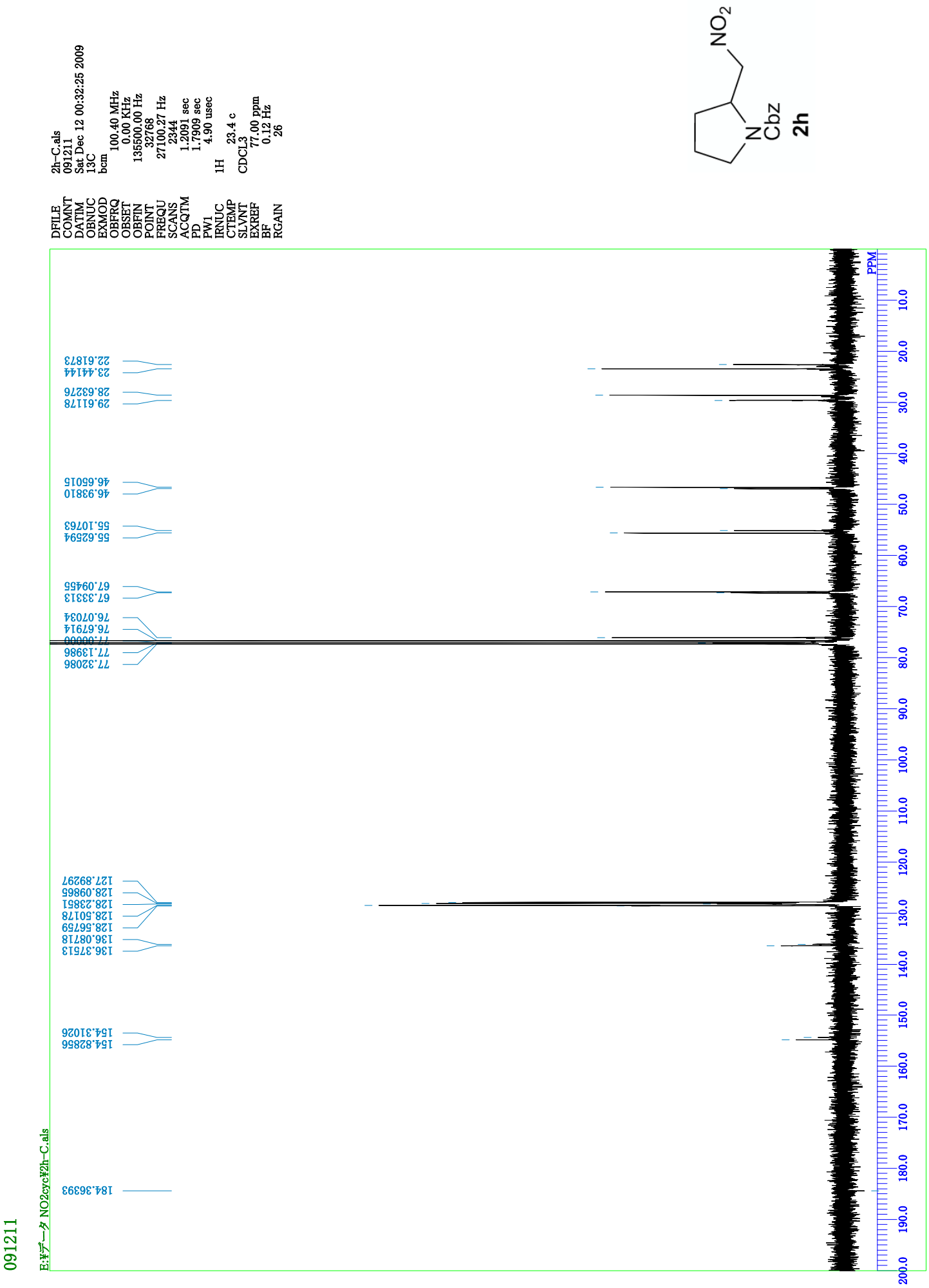


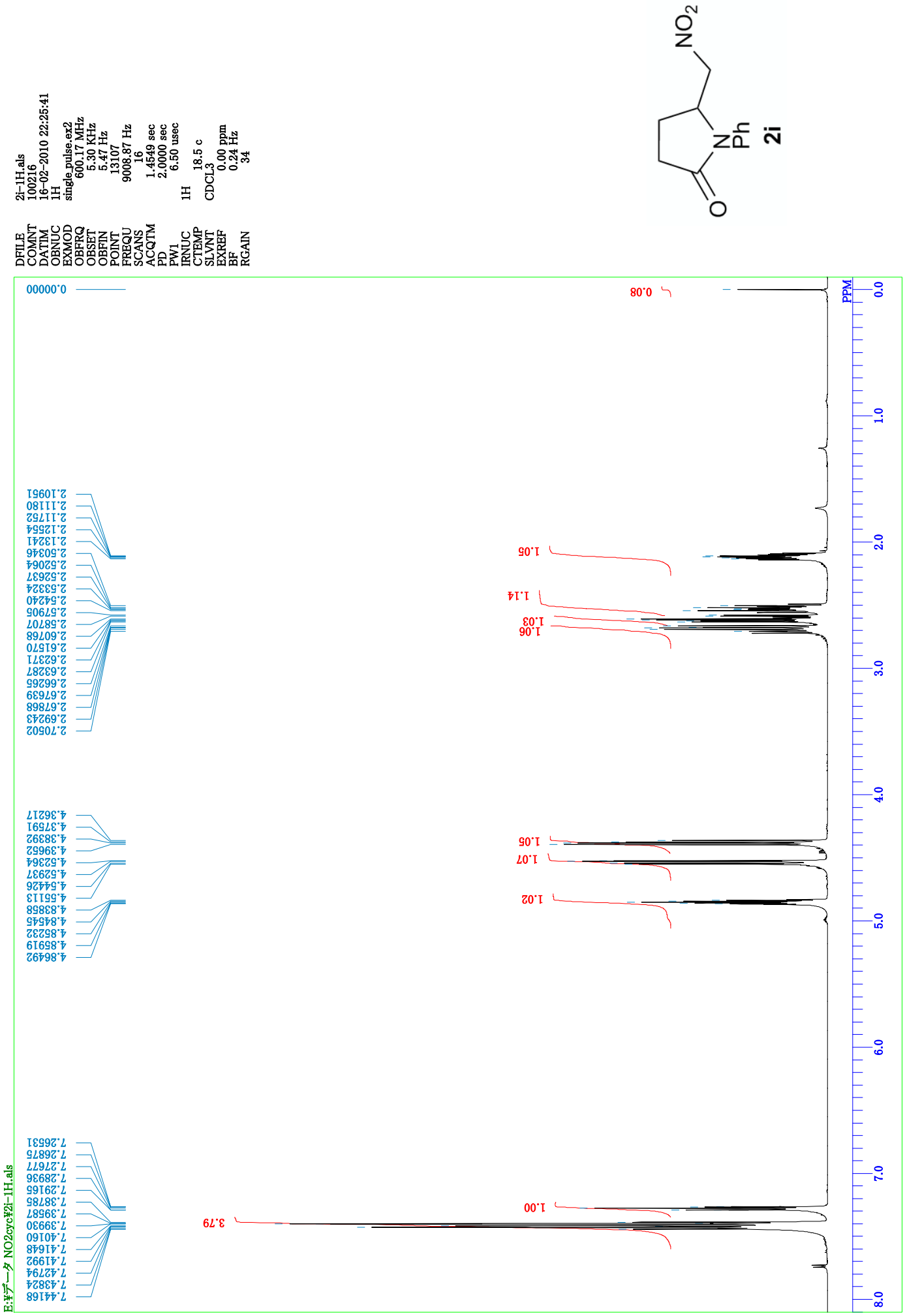
2f (single isomer)

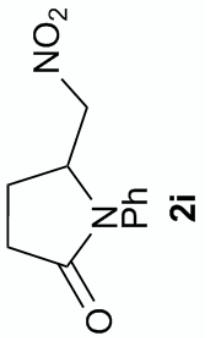

DFILE 2g-1H.als
COMNT 091210
DATIM Thu Dec 10 21:36:39 2009
OBNUC 1H
EXMOD non
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134300.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 16
ACQTM 4.0993 sec
PD 2.9007 sec
PW1 6.80 usec
IRNUC 1H
CTEMP 22.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.16 Hz
RGAIN 19
Me
Me
N
Ts
NO2
2g
PPM
0.00000
0.99157
1.07150
1.62129
1.64387
1.67804
2.33949
2.37366
2.43651
3.05891
3.08271
3.10773
3.13162
4.78698
4.81505
5.01642
5.04449
7.27048
7.30648
7.32662
7.74643
7.76718
2.97
3.15
3.02
1.31
1.09
3.28
2.04
1.00
1.00
2.12
2.06
E:¥データ¥NO2cyc¥2g-1H.als
091210


DFILE 2g-C.als
COMNT 091210
DATIM Thu Dec 10 21:50:05 2009
OBNUC 13C
EXMOD bcm
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 257
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 4.90 usec
IRNUC 1H
CTEMP 23.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.16 Hz
RGAIN 26
Me
Me
N
Ts
NO2
2g
21.49984
25.16914
27.06138
27.26705
36.24284
51.39720
61.20393
65.86048
76.67914
77.00000
77.31263
83.26907
127.43225
129.58776
137.00862
143.66436
PPM
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
E:¥データ NO2cyc¥2g-C.als
091210

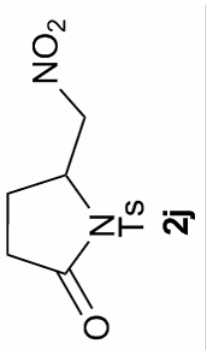

2h
Cbz
N
NO2
2h-1H.als
091211
Fri Dec 11 22:32:23 2009
1H
non
399.65 MHz
0.00 KHz
134300.00 Hz
32768
7993.60 Hz
9
4.0993 sec
2.9007 sec
6.80 usec
1H
22.3 c
CDCL3
0.00 ppm
0.12 Hz
21
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
PPM
3.00
0.99
2.01
0.31
1.51
0.34
0.55
2.00
4.83
E:¥データ NO2cyc¥2h-1H.als
091211


DFILE 2h-C.als
COMNT 091211
DATIM Sat Dec 12 00:32:25 2009
OBNUC 13C
EXMOD bcm
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 2344
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 4.90 usec
IRNUC 1H
CTEMP 23.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 26
NO2
N
Cbz
2h
22.61873
23.44144
28.63276
29.61178
46.93810
46.65015
55.62594
55.10763
67.33313
67.09455
76.67914
76.07034
77.32086
77.13986
127.89297
128.09865
128.23851
128.50178
128.56759
136.08718
136.37513
154.82856
154.31026
184.36393
PPM
10.0
20.0
30.0
40.0
50.0
60.0
70.0
80.0
90.0
100.0
110.0
120.0
130.0
140.0
150.0
160.0
170.0
180.0
190.0
200.0
E:¥データ NO2cyc¥2h-C.als
091211

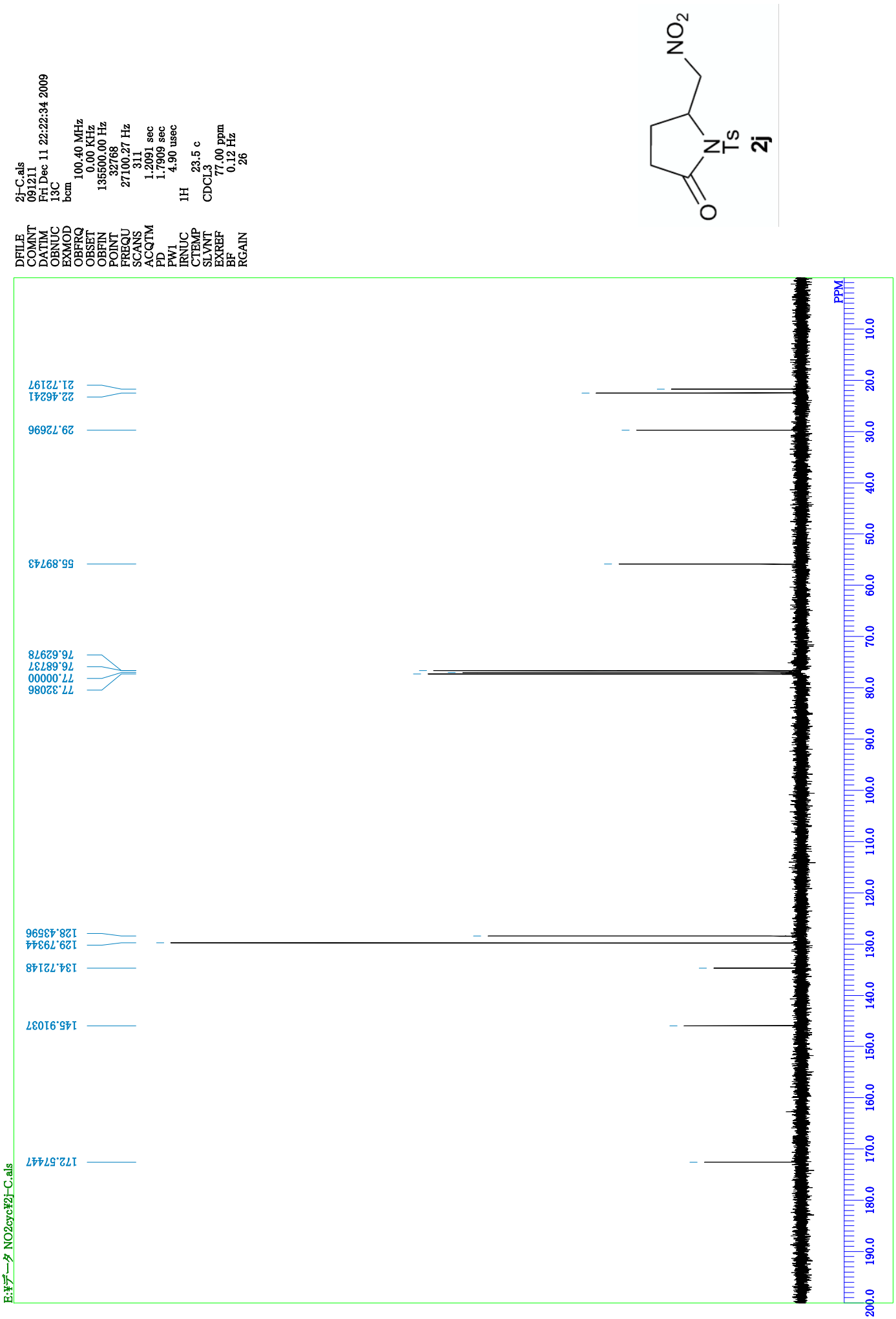

DFILE 2i-1H.als
COMNT 100216
DATIM 16-02-2010 22:25:41
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 600.17 MHz
OBSET 5.30 KHz
OBFIN 5.47 Hz
POINT 13107
FREQU 9008.87 Hz
SCANS 16
ACQTM 1.4549 sec
PD 2.0000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 18.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.24 Hz
RGAIN 34
NO2
N
Ph
O
2i
E:¥データ NO2cyc¥2i-1H.als
100216
PPM
0.08
1.05
1.14
1.03
1.06
1.05
1.07
1.02
1.00
3.79

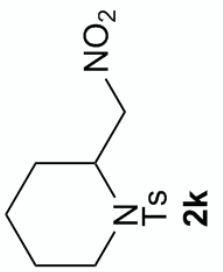

NO2
N
Ph
O
2i

DFILE 2i-C.als
COMNT 091214
DATIM 14-12-2009 22:48:31
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 20970
FREQU 20099.89 Hz
SCANS 258
ACQTM 1.0433 sec
PD 1.2000 sec
PW1 2.87 usec
IRNUC 1H
CTEMP 20.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.00 Hz
RGAIN 60
22.02131
30.04976
57.17678
76.09418
76.68535
77.00000
77.31465
123.96931
126.91562
129.49960
136.04059
173.52259
PPM
0.0
10.0
20.0
30.0
40.0
50.0
60.0
70.0
80.0
90.0
100.0
110.0
120.0
130.0
140.0
150.0
160.0
170.0
180.0
190.0
E:¥データ NO2cyc¥2i-C.als
091214


NO2
N
Ts
O
2j

DFILE 2j-1H.als
COMNT 091211
DATIM Fri Dec 11 22:06:04 2009
OBNUC 1H
EXMOD non
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134300.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 16
ACQTM 4.0993 sec
PD 2.9007 sec
PW1 6.80 usec
IRNUC 1H
CTEMP 22.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 22
0.00000
0.00183
1.59627
2.06490
2.07161
2.09724
2.10151
2.11555
2.12104
2.34803
2.37183
2.37671
2.38586
2.40112
2.41393
2.41881
2.43661
2.44322
2.45726
2.54879
2.58296
2.60126
2.60675
2.62506
2.63543
4.67715
4.69728
4.71010
4.73024
4.88461
4.89194
4.89926
4.91879
4.96882
4.97736
5.00116
5.00970
7.26621
7.35713
7.37726
7.37848
7.93986
7.96122
1.00
2.11
2.84
1.04
1.00
0.98
1.00
1.94
1.90
PPM
0.0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
E:¥データ NO2cyc¥2j-1H.als
091211

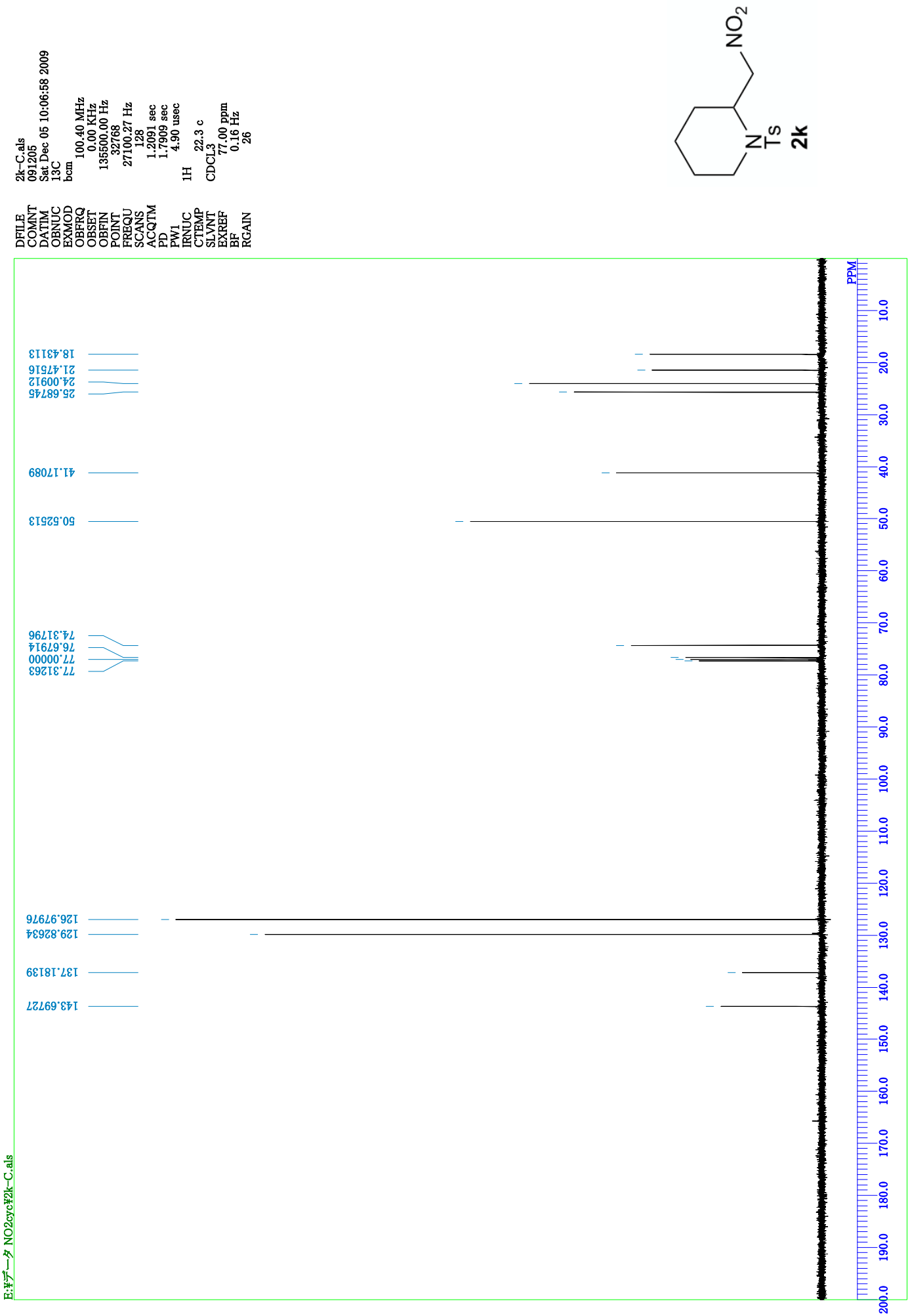

DFILE 2j-C.als
COMNT 091211
DATIM Fri Dec 11 22:22:34 2009
OBNUC 13C
EXMOD bcm
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 311
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 4.90 usec
IRNUC 1H
CTEMP 23.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 26
NO2
N
Ts
O
2j
21.72197
22.46241
29.72696
55.89743
76.62978
76.68737
77.00000
77.32086
128.43596
129.79344
134.72148
145.91037
172.57447
PPM
10.0
20.0
30.0
40.0
50.0
60.0
70.0
80.0
90.0
100.0
110.0
120.0
130.0
140.0
150.0
160.0
170.0
180.0
190.0
200.0
E:¥データ NO2cyc¥2j-C.als
091211

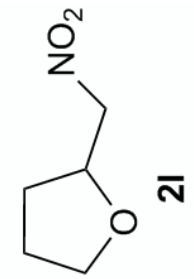


2k-1H.als
091205
Sat Dec 05 09:59:58 2009
1H
non
399.65 MHz
0.00 KHz
134300.00 Hz
32768
7993.60 Hz
16
4.0993 sec
2.9007 sec
6.80 usec
1H
21.3 c
CDCL3
0.00 ppm
0.16 Hz
18

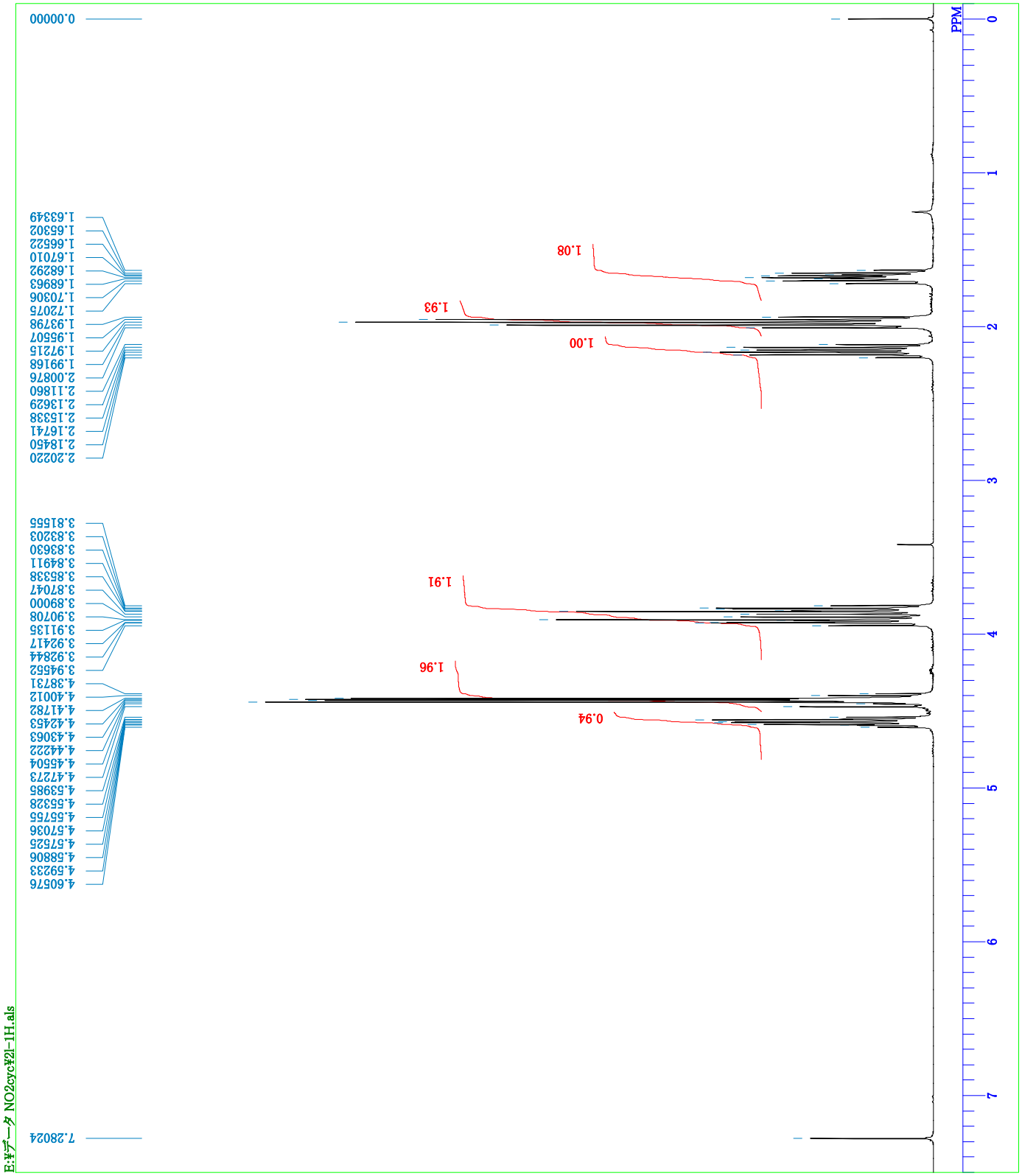
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

E:¥データ¥NO2cyc¥2k-1H.als
091205

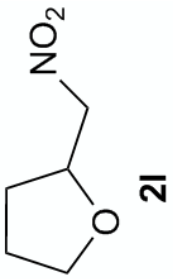

2k-C.als
091205
Sat Dec 05 10:06:58 2009
13C
bcm
100.40 MHz
0.00 KHz
135500.00 Hz
32768
27100.27 Hz
128
1.2091 sec
1.7909 sec
4.90 usec
1H
22.3 c
CDCL3
77.00 ppm
0.16 Hz
26
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
NO2
N
Ts
2k
18.43113
21.47516
24.00912
25.68745
41.17089
50.52513
74.31796
76.67914
77.00000
77.31263
126.97976
129.82634
137.18139
143.69727
PPM
10.0
20.0
30.0
40.0
50.0
60.0
70.0
80.0
90.0
100.0
110.0
120.0
130.0
140.0
150.0
160.0
170.0
180.0
190.0
200.0
E:¥データ NO2cyc¥2k-C.als
091205



DFILE 2l-1H.als
COMNT 091209
DATIM Wed Dec 09 16:05:26 2009
OBNUC 1H
EXMOD non
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134300.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 8
ACQTM 4.0993 sec
PD 2.9007 sec
PW1 6.80 usec
IRNUC 1H
CTEMP 22.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.16 Hz
RGAIN 19

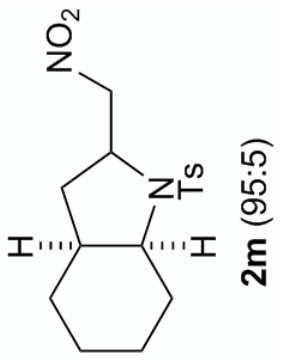


091209
E:¥データ NO2cyc¥2l-1H.als

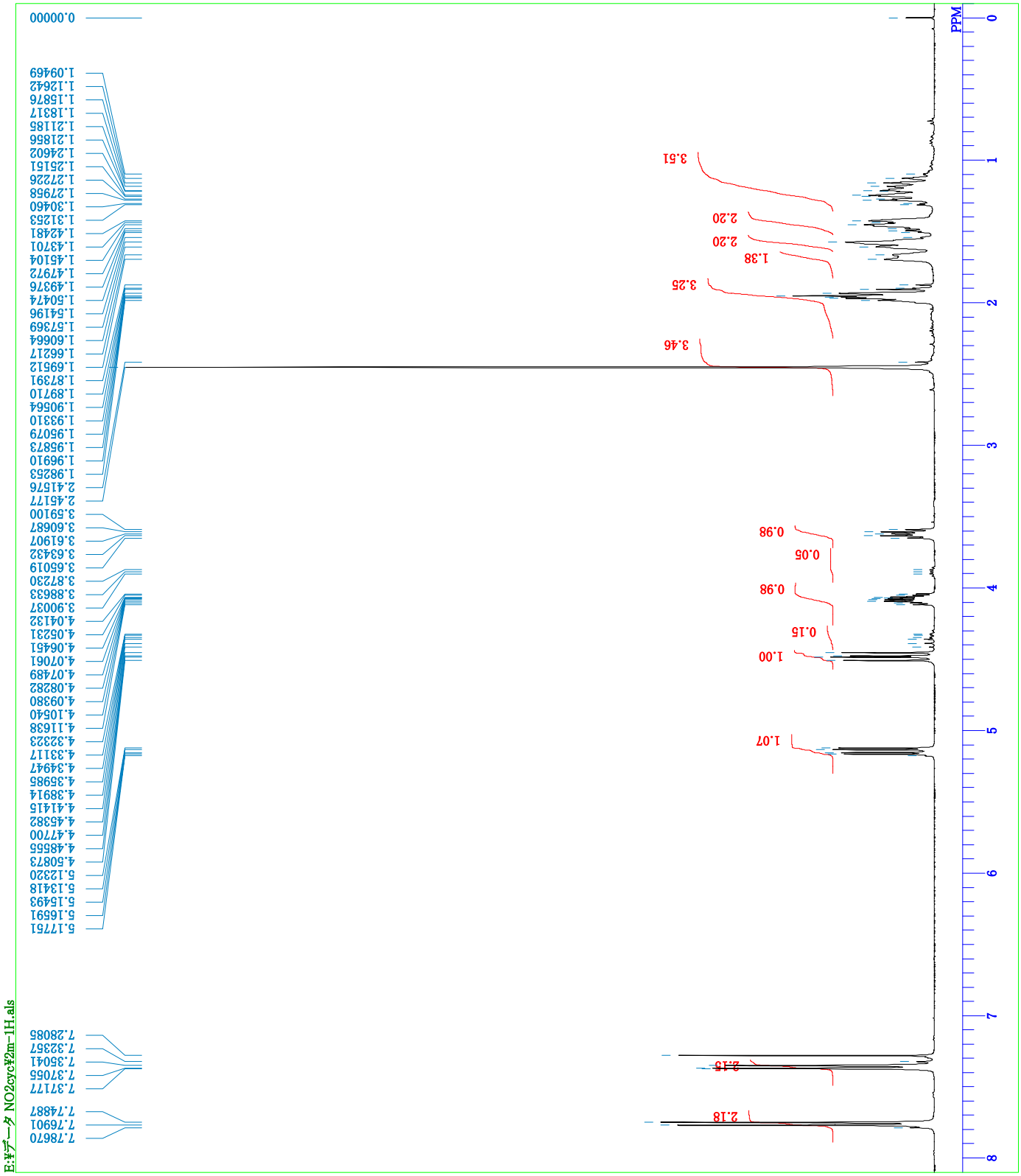

NO2
O

2l

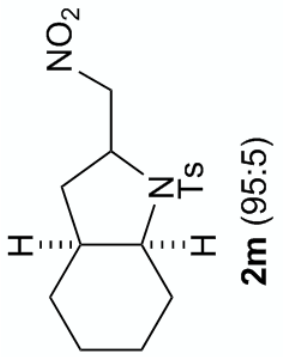
2l-C.als
091209
Wed Dec 09 16:18:57 2009
13C
bcm
100.40 MHz
0.00 KHz
135500.00 Hz
32768
27100.27 Hz
257
1.2091 sec
1.7909 sec
4.90 usec
1H
23.0 c
CDCL3
77.00 ppm
0.16 Hz
26
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
25.34191
28.87134
68.57643
75.10776
76.67914
77.00000
77.31263
78.87578
PPM
E:¥データ¥NO2cyc¥2l-C.als
091209



DFILE 2m-1H.als
COMNT 091205
DATIM Sat Dec 05 14:33:53 2009
OBNUC 1H
EXMOD non
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134300.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 16
ACQTM 4.0993 sec
PD 2.9007 sec
PW1 6.80 usec
IRNUC 1H
CTEMP 21.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 14



091205
E:¥データ¥NO2cyc¥2m-1H.als



2m (95:5)

2m-C.als
091205
Sat Dec 05 14:47:29 2009
13C
bcm
100.40 MHz
0.00 KHz
135500.00 Hz
32768
27100.27 Hz
258
1.2091 sec
1.7909 sec
4.90 usec
1H
23.5 c
CDCL3
77.00 ppm
0.12 Hz
26

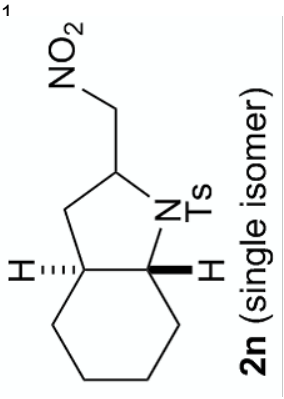
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

19.79683
19.99428
21.45048
21.48339
23.35095
24.02557
25.32546
25.42418
27.52210
31.08444
31.15848
32.59823
34.39174
36.10298
55.26395
57.05746
60.90775
60.99002
76.67914
77.00000
77.32086
78.27520
80.56234
127.37466
127.41580
129.71939
129.92507
133.98926
143.64790
144.01812

PPM

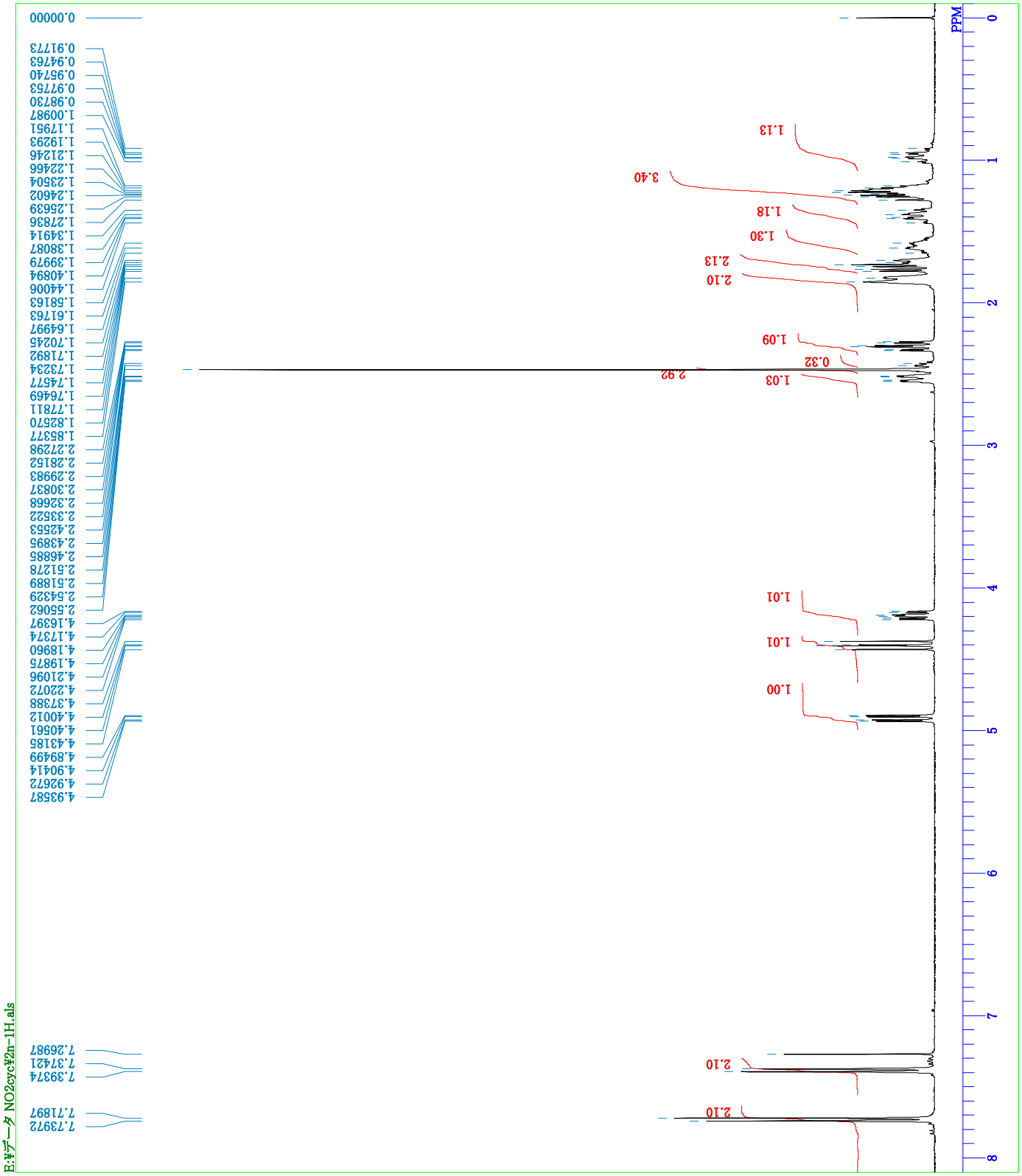
E:¥データ¥NO2cyc¥2m-C.als

091205

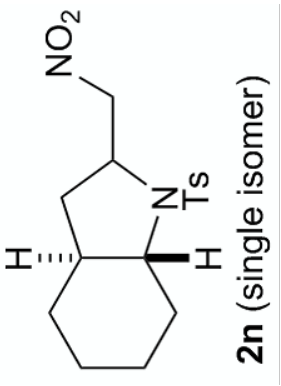


2n (single isomer)

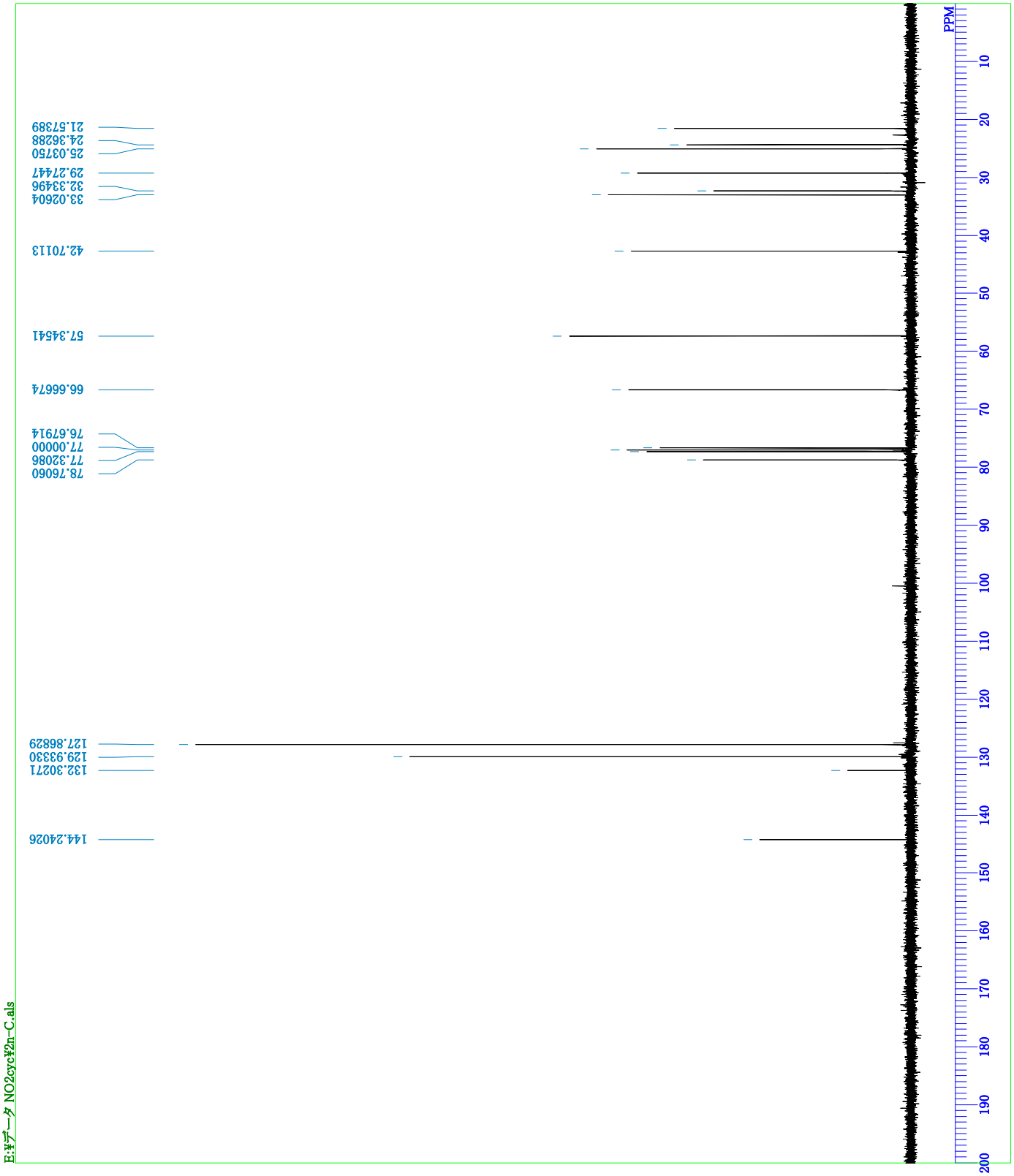
DFILE 2n-1H.als
COMNT 091207
DATIM Mon Dec 07 17:10:07 2009
OBNUC 1H
EXMOD non
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134300.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 16
ACQTM 4.0993 sec
PD 2.9007 sec
PW1 6.80 usec
IRNUC 1H
CTEMP 20.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.16 Hz
RGAIN 18



091207
E:¥データ¥NO2cyc¥2n-1H.als

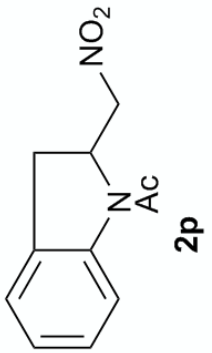


DFILE 2n-C.als
COMNT 091207
DATIM Mon Dec 07 17:23:42 2009
OBNUC 13C
EXMOD bcm
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 257
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 4.90 usec
IRNUC 1H
CTEMP 22.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.16 Hz
RGAIN 26

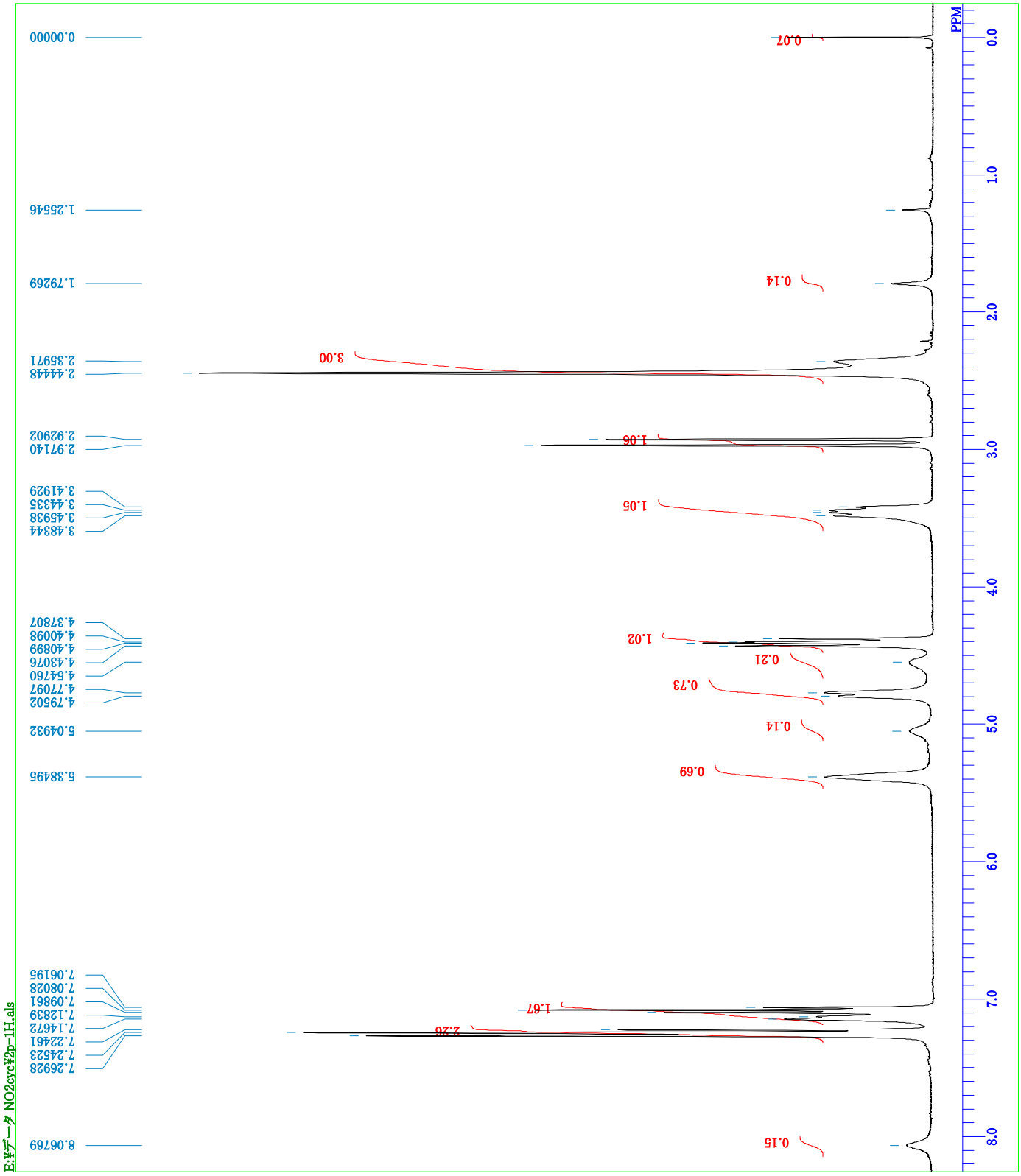


E:¥データ¥NO2cyc¥2n-C.als

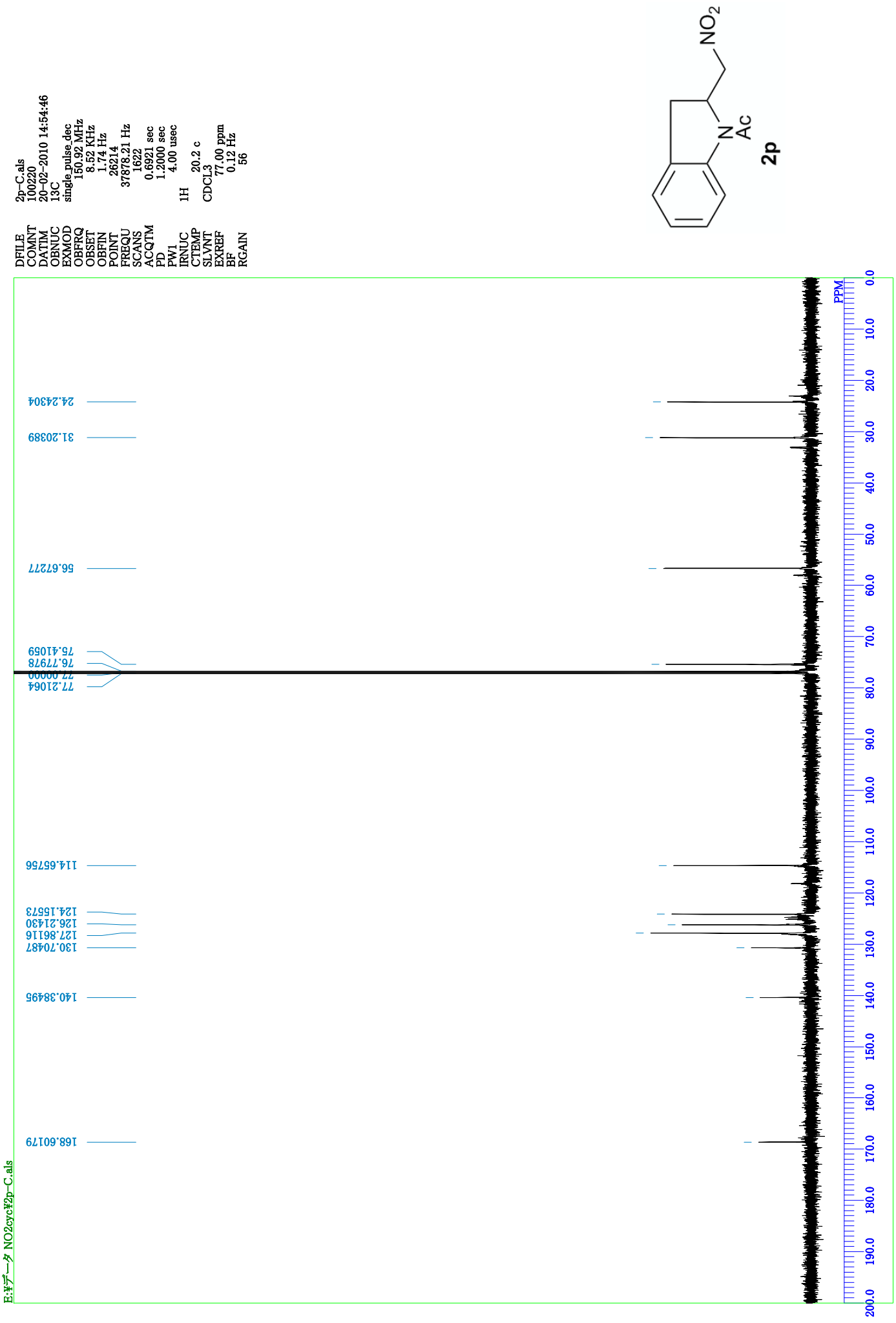
091207

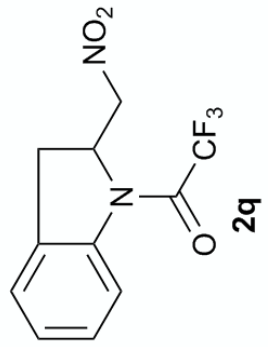


DFILE 2p-1H.als
COMNT single_pulse
DATIM 04-12-2009 18:40:06
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 13107
FREQU 6002.31 Hz
SCANS 16
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 4.90 usec
IRNUC 1H
CTEMP 20.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.16 Hz
RGAIN 50

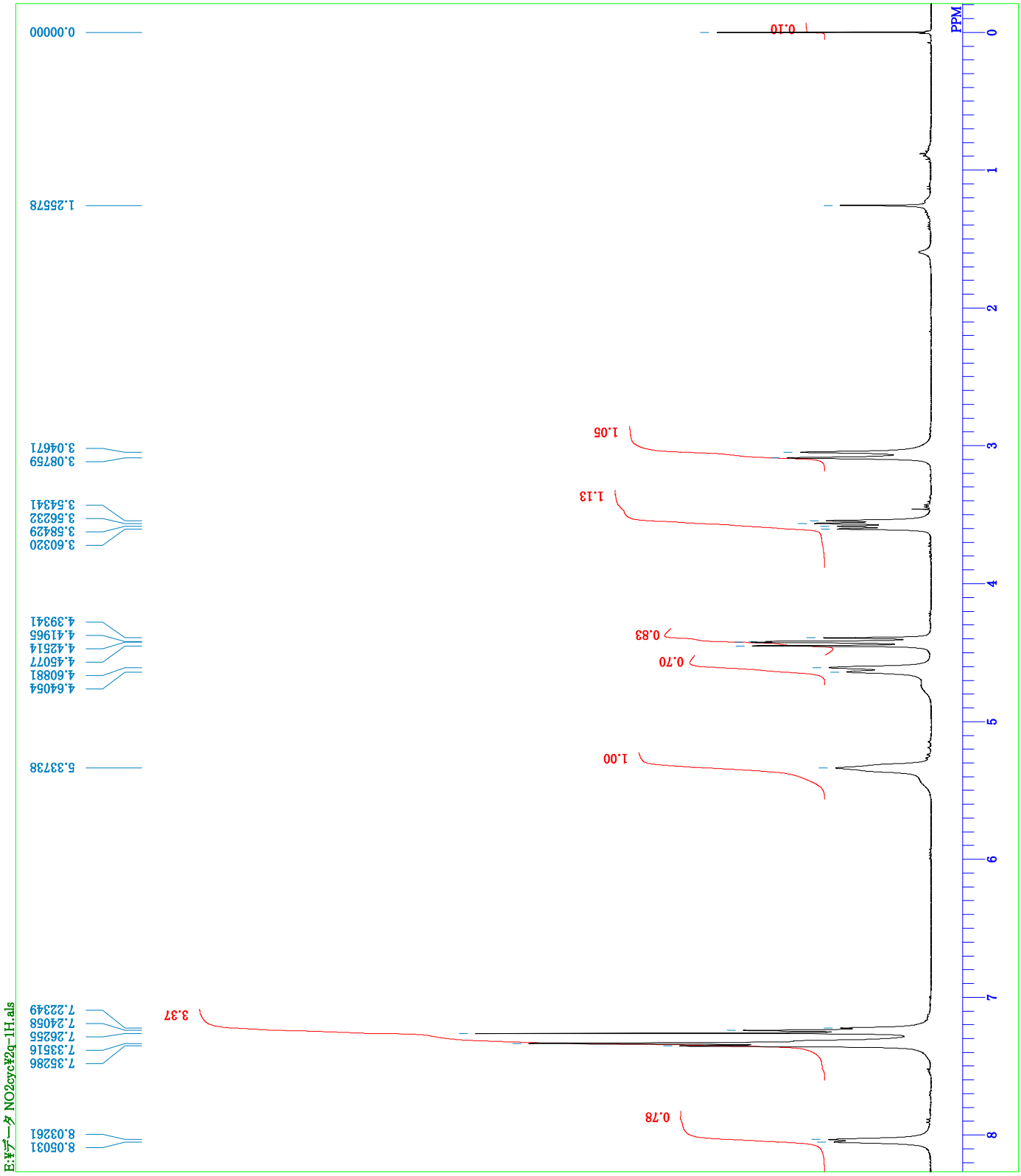


single_pulse
E:¥データ¥NO2cyc¥2p-1H.als


DFILE 2p-C.als
COMNT 100220
DATIM 20-02-2010 14:54:46
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 150.92 MHz
OBSET 8.52 KHz
OBFIN 1.74 Hz
POINT 26214
FREQU 37878.21 Hz
SCANS 1622
ACQTM 0.6921 sec
PD 1.2000 sec
PW1 4.00 usec
IRNUC 1H
CTEMP 20.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 56
NO2
N
Ac
2p
24.24304
31.20389
56.67277
75.41059
76.77978
77.00000
77.21064
114.65756
124.15573
126.21430
127.86116
130.70487
140.38495
168.60179
PPM
0.0
10.0
20.0
30.0
40.0
50.0
60.0
70.0
80.0
90.0
100.0
110.0
120.0
130.0
140.0
150.0
160.0
170.0
180.0
190.0
200.0
100220
E:¥データ NO2cyc¥2p-C.als

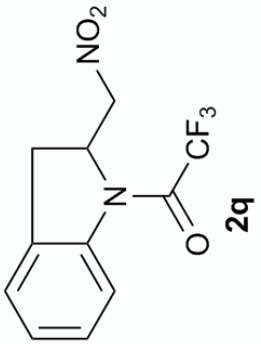


DFILE 2q-1H.als
COMNT 091208
DATIM Tue Dec 08 23:00:28 2009
OBNUC 1H
EXMOD non
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134300.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 16
ACQTM 4.0993 sec
PD 2.9007 sec
PW1 6.80 usec
IRNUC 1H
CTEMP 19.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.16 Hz
RGAIN 21

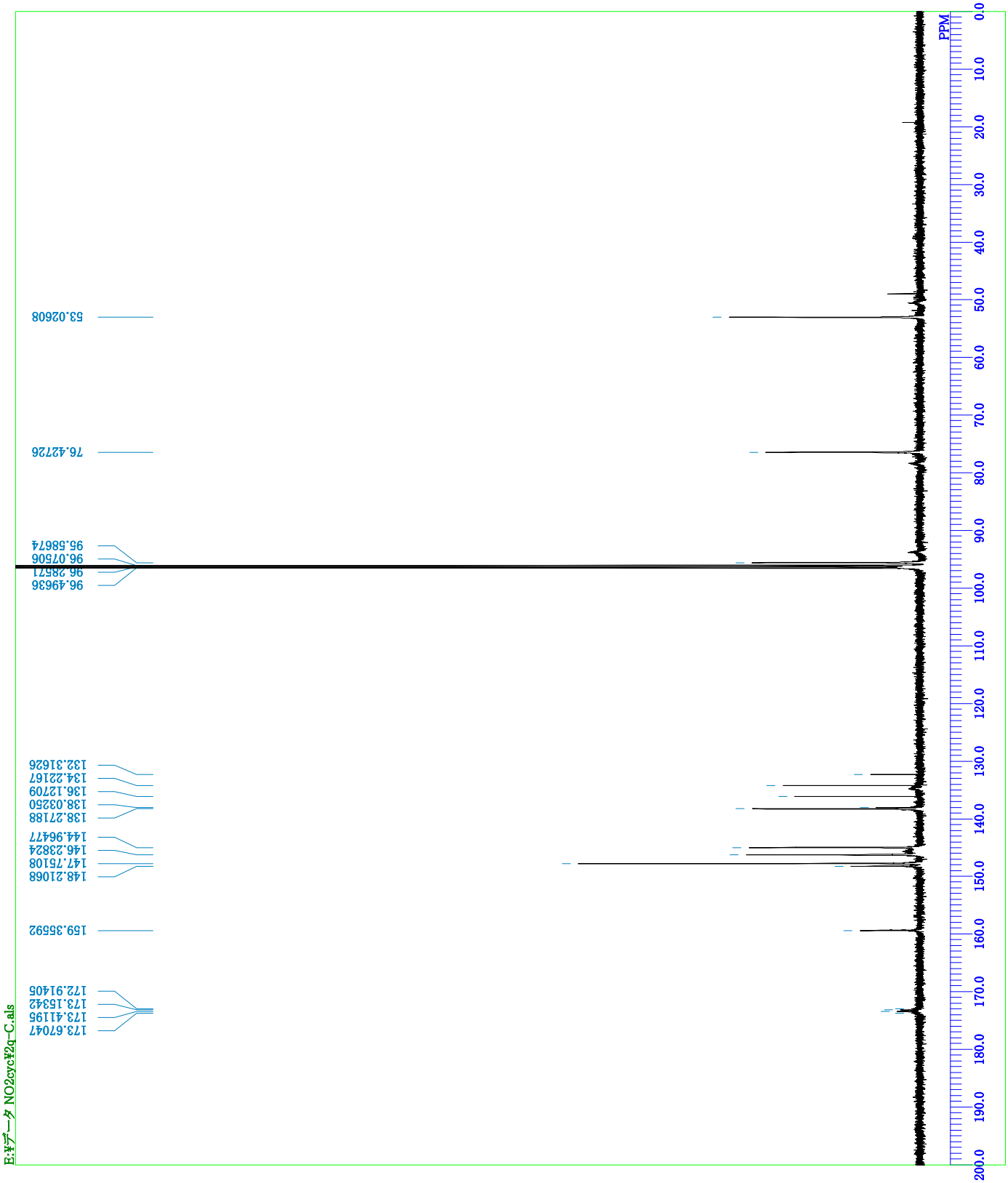


E:¥データ NO2cyc¥2q-1H.als

091208

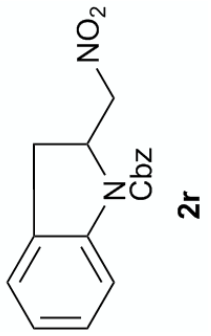


DFILE 2q-C.als
COMNT 100226
DATIM 27-02-2010 08:56:33
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 150.92 MHz
OBSET 8.52 KHz
OBFIN 1.74 Hz
POINT 26214
FREQU 37878.21 Hz
SCANS 18981
ACQTM 0.6921 sec
PD 1.2000 sec
PW1 4.00 usec
IRNUC 1H
CTEMP 21.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 56

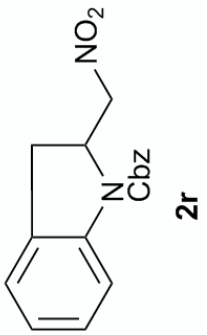


E:¥データ NO2cyc¥2q-C.als

100226

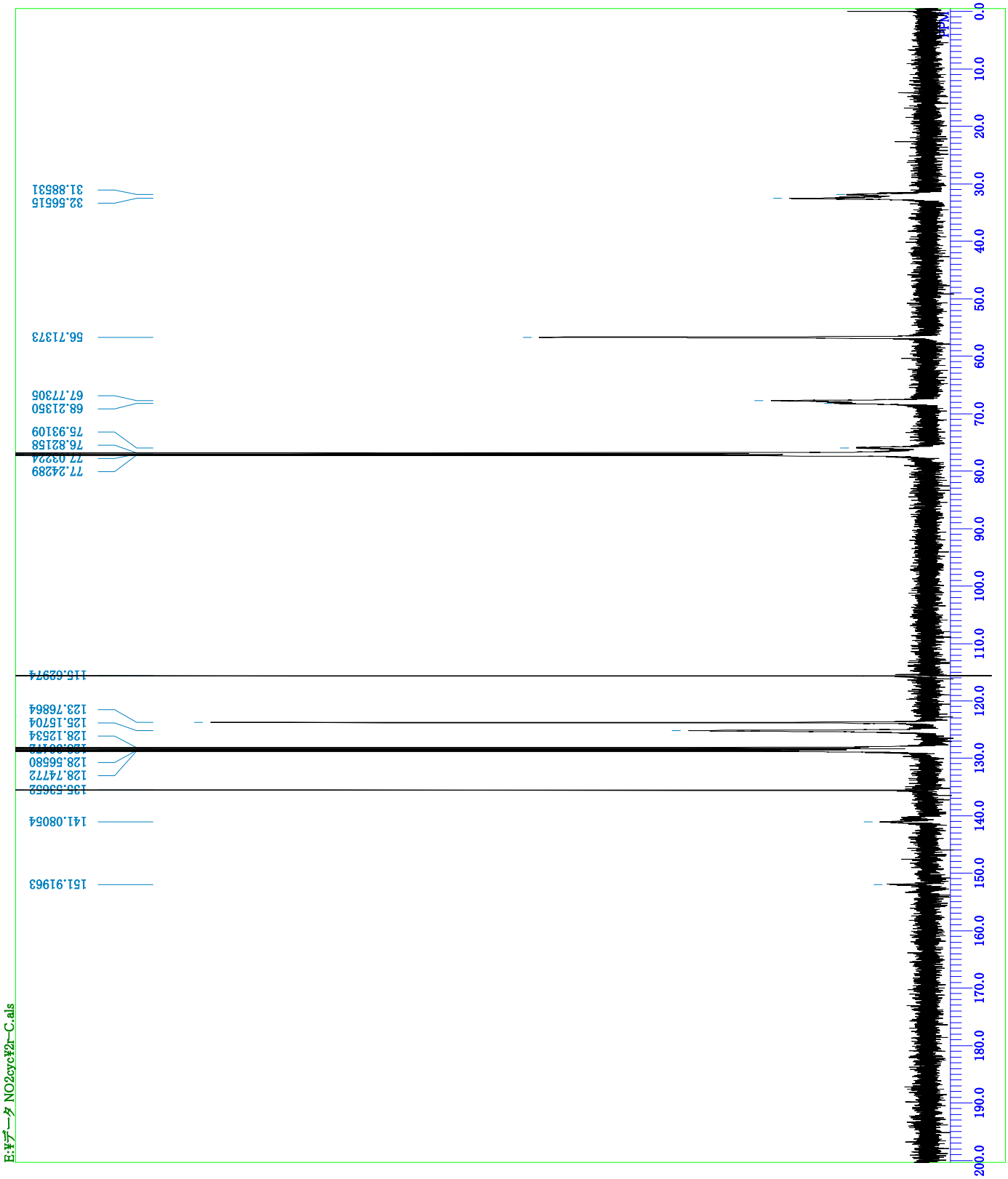

NO2
N
Cbz
2r

DFILE 2r-1H.als
COMNT 100222
DATIM 22-02-2010 15:36:45
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 600.17 MHz
OBSET 5.30 KHz
OBFIN 5.47 Hz
POINT 13107
FREQU 9008.87 Hz
SCANS 16
ACQTM 1.4549 sec
PD 2.0000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 21.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.25 Hz
RGAIN 44
0.00000
-0.00573
1.55178
2.97415
3.00164
3.46660
3.48263
3.49523
3.51126
4.39881
4.70115
5.14321
5.31728
7.02024
7.17828
7.19087
7.25844
7.34891
7.35579
7.36037
7.36495
7.37153
7.37828
7.38786
7.40045
7.40847
7.41190
7.41419
7.43023
7.44168
7.84136
1.30
1.35
1.23
1.00
1.24
2.62
1.06
2.17
0.48
PPM
0.0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
E:¥データ¥NO2cyc¥2r-1H.als
100222



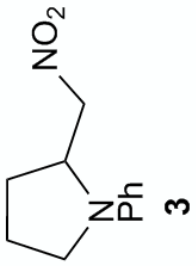
2r

DFILE 2r-C.als
COMNT 100304
DATIM 05-03-2010 08:53:12
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 150.92 MHz
OBSET 8.52 KHz
OBFIN 1.74 Hz
POINT 20970
FREQU 30302.11 Hz
SCANS 20248
ACQTM 0.6921 sec
PD 1.2000 sec
PW1 4.00 usec
IRNUC 1H
CTEMP 20.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.25 Hz
RGAIN 56

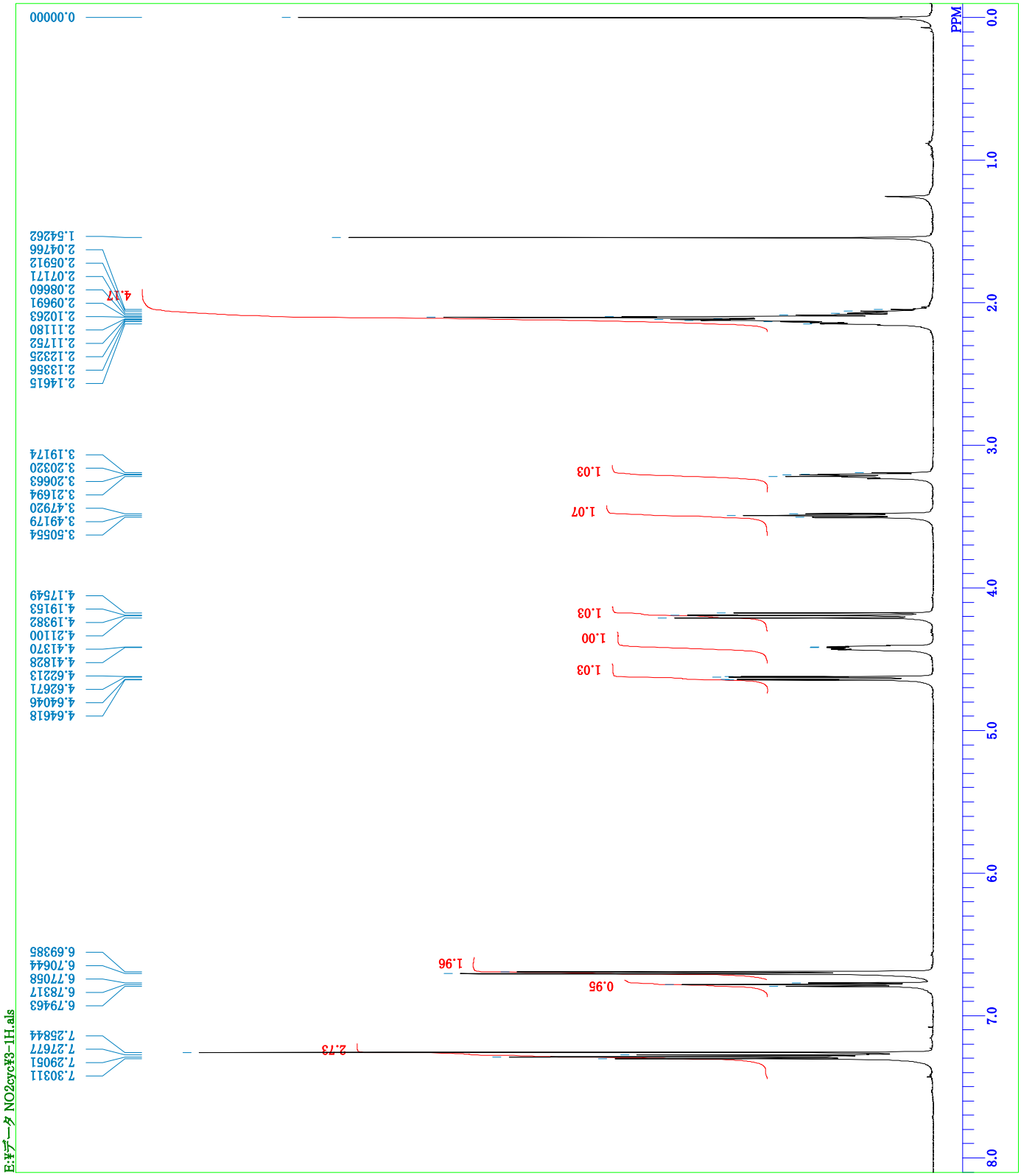


E:¥データ¥NO2cyc¥2r-C.als

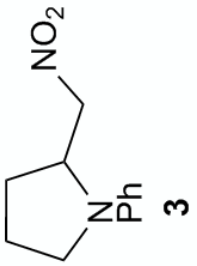
100304

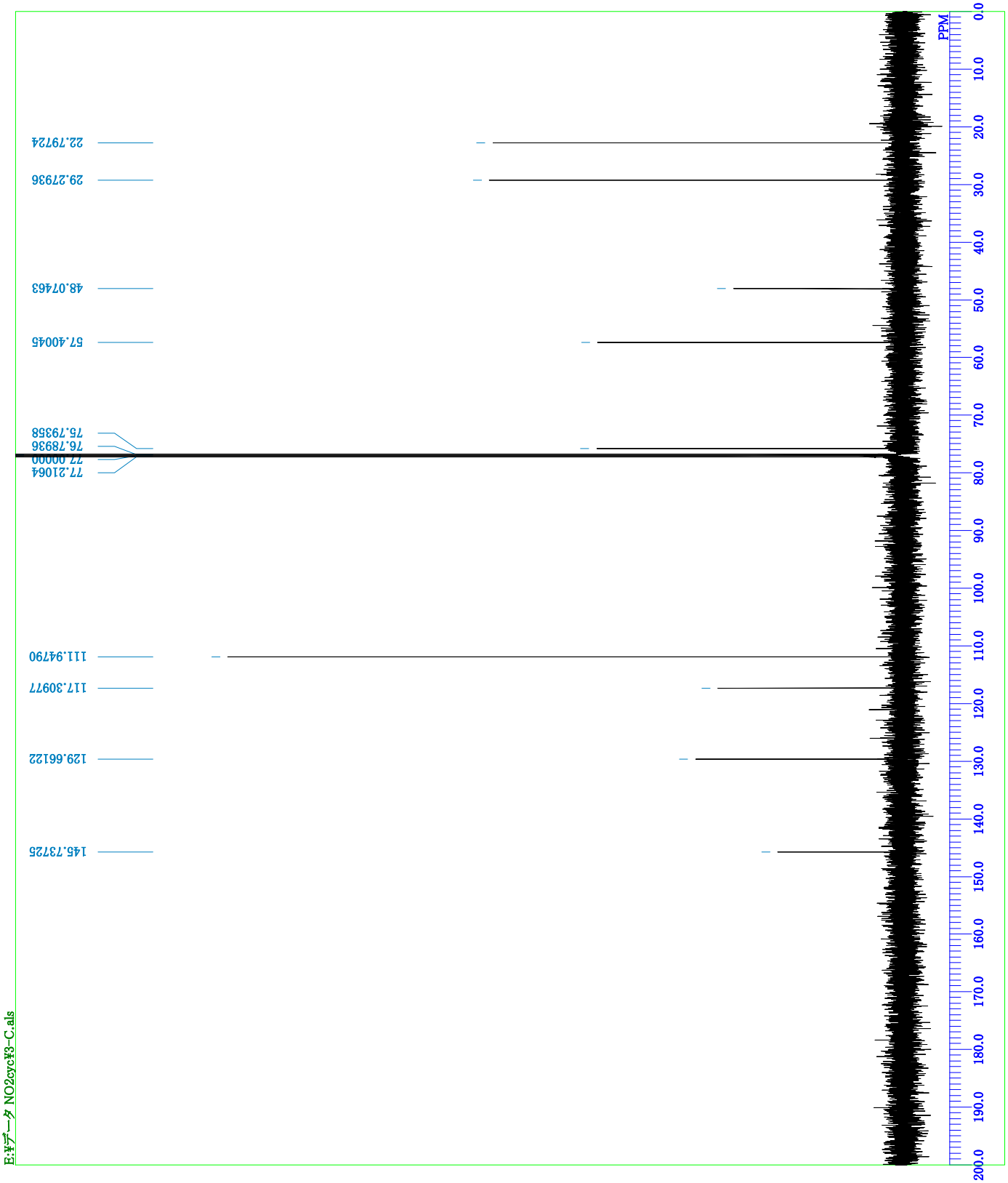


DFILE 3-1H.als
COMNT 100224
DATIM 24-02-2010 21:02:26
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 600.17 MHz
OBSET 5.30 KHz
OBFIN 5.47 Hz
POINT 13107
FREQU 9008.87 Hz
SCANS 16
ACQTM 1.4549 sec
PD 2.0000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 22.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.43 Hz
RGAIN 44



E:¥データ¥NO2cyc¥3-1H.als
100224


NO2
N
Ph
3


DFILE 3-C.als
COMNT 100222
DATIM 22-02-2010 15:31:13
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 150.92 MHz
OBSET 8.52 KHz
OBFIN 1.74 Hz
POINT 26214
FREQU 37878.21 Hz
SCANS 258
ACQTM 0.6921 sec
PD 1.2000 sec
PW1 4.00 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.25 Hz
RGAIN 56
22.79724
29.27936
48.07463
57.40045
75.79358
76.78936
77.00000
77.21064
111.94790
117.30977
129.66122
145.73725
PPM
0.0
10.0
20.0
30.0
40.0
50.0
60.0
70.0
80.0
90.0
100.0
110.0
120.0
130.0
140.0
150.0
160.0
170.0
180.0
190.0
200.0
E:¥データ NO2cyc¥3-C.als
100222