Supporting Information

An Organocatalytic Asymmetric Diels-Alder Strategy for the Enantioselective Synthesis of Spirocyclic Oxindole-Cyclohexenones

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General

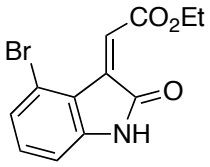
All reactions were performed in oven-dried glassware under a positive pressure of nitrogen or argon. All melting points were measured on a Yanagimoto MP-S3 micro-melting point apparatus and are uncorrected. The NMR spectra were recorded on a JEOL ECA–500 (500 MHz for ^{1}H NMR analysis and 125.8 MHz for ^{13}C NMR analysis) instrument in $CDCl_3$ unless otherwise stated and are reported in parts per million (δ) downfield from TMS as an internal standard. Mass spectral analyses were performed on a JEOL JMS-700/MStaion mass spectrometer. The infrared spectra were measured with a JASCO FTIR-4100 Fourier Transform Infrared Spectrophotometer and are reported in wave-numbers (cm^{-1}). Optical rotations were measured on a JASCO DIP-370 digital polarimeter. HPLC analysis was carried out using a Hitachi L-6200 HPLC system.

Thin-layer chromatography (TLC) was conducted using Merck Kieselgel 60F-254 plates (0.25 mm). Kanto Chemicals silica gel 60N (spherical, neutral 63–210 mm) was used for column chromatography.

All dried solvents were stored over molecular sieves 3Å or 4Å.

The chiral thiourea catalyst was prepared by the procedure as reported by Lattanzi.[1] The starting 3-alkylidene oxindoles **2** were prepared from isatins **1** as precursors following the literature procedure.[2] Compounds **2g**[3] and **2h**[4] are known.

Ethyl (*Z*)-2-(4-Bromo-2-oxoindolin-3-ylidene)acetate (2a)



2a

Yellow powder, mp 187–189 °C (sublimed, recryst from hexane/Et_2O); FTIR (KBr) ν 3439, 1720, 1646, 1614, 1577 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.40 (3H, t, *J* = 7.5 Hz), 4.41 (2H, q, *J* = 7.5 Hz), 6.82 (1H, d, *J* = 8.0 Hz), 7.12 (1H, t, *J* = 8.0 Hz), 7.18 (1H, d, *J* = 8.0 Hz), 7.70 (1H, s), 8.86 (1H, br); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ 13.99, 61.78, 109.31, 118.99, 119.66, 127.13, 127.48, 131.29, 131.49, 143.33, 166.19, 166.40; Anal. Calcd for $C_{12}H_{10}BrNO_3$: C, 48.67%; H, 3.40%; N, 4.73%. Found: C, 48.89%; H, 3.38%; N, 4.68%.

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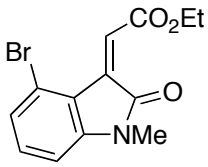
(1) Lattanzi, A. *Synlett* **2007**, 2106.

(2) (a) Brandman, H. A. *J. Heterocycl. Chem.* **1973**, *10*, 383. (b) Shimazawa, R.; Kuriyama, M.; Shirai, R. *Bioorg. Med. Chem. Lett.* **2008**, *18*, 3350.

(3) Jiang, T.; Kuhen, K. L.; Wolff, K.; Yin, H.; Bieza, K.; Caldwell, J.; Bursulaya, B.; Wu, T. Y.-H.; He, Y. *Bioorg. Med. Chem. Lett.* **2006**, *16*, 2105.

(4) Malhotra, S.; Balwani, S.; Dhawan, A.; Singh, B. K.; Kumar, S.; Thimmulappa, R.; Biswal, S.; Olsen, C. E.; Van der Eycken, E.; Prasad, A. K.; Ghosh, B.; Parmar, V. S. *Med. Chem. Commun.* **2011**, *2*, 743.

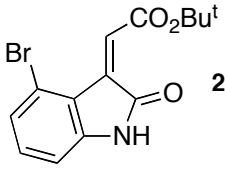
Ethyl (*Z*)-2-(4-Bromo-1-methyl-2-oxoindolin-3-ylidene)-acetate (2b)



2b

Yellow needles, mp 123–124 °C (recryst from hexane/Et_2O); FTIR (KBr) ν 1718, 1645, 1605, 1577 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.40 (3H, t, *J* = 7.5 Hz), 3.21 (3H, s), 4.41 (2H, q, *J* = 7.5 Hz), 6.76 (1H, dd, *J* = 7.0, 2.0 Hz), 7.16-7.20 (2H, m), 7.70 (1H, s); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ 14.01, 26.06, 61.83, 107.30, 118.86, 119.03, 127.11, 127.17, 130.75, 131.31, 145.85, 164.63, 166.29; Anal. Calcd for $C_{13}H_{12}BrNO_3$: C, 50.34%; H, 3.90%; N, 4.52%. Found: C, 50.20%; H, 3.69%; N. 4.44%.

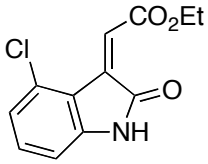
***tert*-Butyl (*Z*)-2-(4-Bromo-2-oxoindolin-3-ylidene)acetate (2c)**



2c

Yellow powder, mp 174–178 °C (sublimed, recryst from hexane/Et_2O); FTIR (KBr) ν 1720, 1647, 1611 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.62 (9H, s), 6.78 (1H, d, *J* = 8.0 Hz), 7.10 (1H, t, *J* = 8.0 Hz), 7.18 (1H, d, *J* = 8.0 Hz), 7.67 (1H, s), 8.54 (1H, br); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ 28.09 (×3), 83.23, 109.10, 118.85, 119.92, 127.02, 128.83, 130.00, 131.18, 143.28, 165.19, 166.69; Anal. Calcd for $C_{14}H_{14}BrNO_3$: C, 51.87%; H, 4.35%; N, 4.32%. Found: C, 52.10%; H, 4.25%; N, 4.22%.

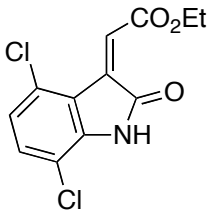
Ethyl (*Z*)-2-(4-Chloro-2-oxoindolin-3-ylidene)acetate (2d)



2d

Yellow needles, mp 182–185 °C (sublimed, recryst from hexane/Et_2O); FTIR (KBr) ν 3439, 1716, 1650, 1616, 1584 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.39 (3H, t, *J* = 7.5 Hz), 4.41 (2H, q, *J* = 7.5 Hz), 6.78 (1H, d, *J* = 8.0 Hz), 7.00 (1H, d, *J* = 8.0 Hz), 7.20 (1H, t, *J* = 8.0 Hz), 7.50 (1H, s), 8.89 (1H, br); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ 14.00, 61.79, 108.78, 118.20, 123.89, 127.90, 130.71, 130.82, 131.42, 143.02, 166.28, 166.36; Anal. Calcd for $C_{12}H_{10}ClNO_3$: C, 57.27%; H, 4.01%; N, 5.57%. Found: C, 57.64%; H, 3.88%; N, 5.51%.

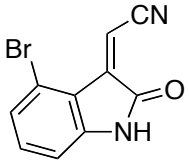
Ethyl (*Z*)-2-(4,7-Dichloro-2-oxoindolin-3-ylidene)acetate (2e)



2e

Yellow powder, mp 195–198 °C (sublimed, recryst from hexane/Et_2O); FTIR (KBr) ν 3446, 1726, 1651, 1611 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.38 (3H, t, *J* = 7.5 Hz), 4.40 (2H, q, *J* = 7.5 Hz), 6.97 (1H, d, *J* = 8.5 Hz), 7.20 (1H, d, *J* = 8.5 Hz), 7.54 (1H, s), 8.35 (1H, br s); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ 13.98, 61.99, 113.93, 119.21, 124.45, 129.07, 129.37, 130.53, 130.85, 140.23, 164.90, 165.73; Anal. Calcd for $C_{12}H_9Cl_2NO_3$: C, 50.38%; H, 3.17%; N, 4.90%. Found: C, 50.76%; H, 3.03%; N, 4.81%.

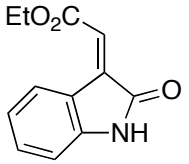
(*Z*)-2-(4-Bromo-2-oxoindolin-3-ylidene)acetonitrile (2f)



2f

Yellow needles, mp 259–260 °C (recryst from $CHCl_3$); FTIR (KBr) ν 2214, 1727, 1608, 1575 cm^{-1}; ^1H NMR (500 MHz, CD_3OD) δ 6.87-6.89 (1H, m), 7.19-7.26 (2H, m), 7.21 (1H, s); ^{13}C NMR (125.8 MHz, CD_3OD) δ 101.39, 110.92, 115.83, 120.28, 120.98, 128.15, 135.17, 144.76, 146.65, 166.00; HRMS for $C_{10}H_5BrN_2O$ + H: 248.9664, found: 248.9668.

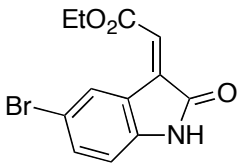
Ethyl (*E*)-2-(5-Bromo-2-oxoindolin-3-ylidene)acetate (2g)[3]



2g

Orange needles, mp 175–178 °C (sublimed, recryst from hexane/Et_2O); FTIR (KBr) ν 1716, 1650, 1613, 1586 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.38 (3H, t, *J* = 7.0 Hz), 4.34 (2H, q, *J* = 7.0 Hz), 6.86 (1H, d, *J* = 8.0 Hz), 6.88 (1H, s), 7.05 (1H, t, *J* = 8.0 Hz), 7.32 (1H, t, *J* = 8.0 Hz), 8.33 (1H, br), 8.54 (1H, d, *J* = 8.0 Hz); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ 14.17, 61.24, 110.08, 120.37, 122.66, 122.89, 129.06, 132.55, 138.05, 143.23, 165.58, 169.17.

Ethyl (*E*)-2-(2-Oxoindolin-3-ylidene)acetate (2h)[4]

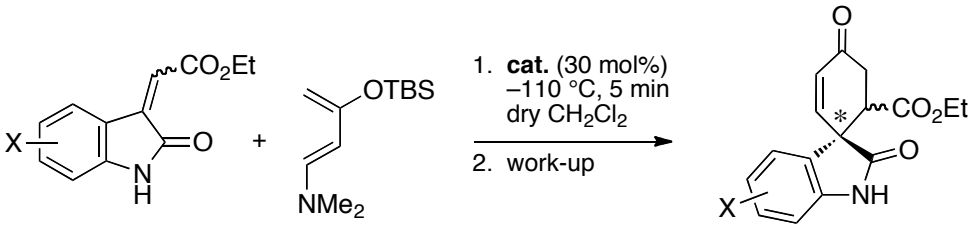


2h

Orange plates, mp 227–229 °C (sublimed, recryst from hexane/Et_2O); FTIR (KBr) ν 1710,

1652, 1611, 1448 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.39 (3H, t, *J* = 7.5 Hz), 4.35 (2H, q, *J* = 7.5 Hz), 6.74 (1H, d, *J* = 8.5 Hz), 6.91 (1H, s), 7.46 (1H, dd, *J* = 8.5, 1.5 Hz), 7.68 (1H, br), 8.74 (1H, d, *J* = 1.5 Hz); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ 14.17, 61.53, 111.25, 115.51, 122.01, 124.20, 131.89, 135.02, 136.80, 141.83, 165.19, 168.03.

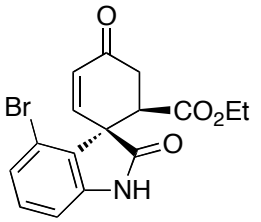
General Procedure for the Diels-Alder Reaction of 2 with 3b



2 **3b** (1.5 eq) **4**

To a solution of 3-alkylidene-oxindole **2** (0.20 mmol) and the thiourea catalyst (0.060 mmol) in CH_2Cl_2 (2.5 mL) was added Rawal's diene **3b** (0.30 mmol) at –110 ºC under Ar, and the resulting orange-colored mixture was stirred at this temperature for a few minutes. The mixture was then exposed to silica gel (ca. 10 mg) at r.t. and stirred for 15 min, filtered and concentrated. The residue was treated with an excess of MeI (ca. 10 eq) at 50 °C for 15 min. After concentration, the mixture was diluted with toluene (1.5 mL) containing 2,6-di(*t*-Bu)$_2$pyridine (20 mg), and the mixture was heated at 110 °C for 15 h. After concentration, the residue was purified by silica gel column chromatography (benzene/acetone) to afford the desired cyclohexenone **4** in a pure form, and its ee was determined by chiral HPLC analysis. The results are summarized in Tables 1 and 2.

Ethyl (1*S*,6*R*)-4'-Bromo-2',4-dioxospiro[cyclohex[2]ene-1,3'-indoline]-6-carboxylate (4a-major)



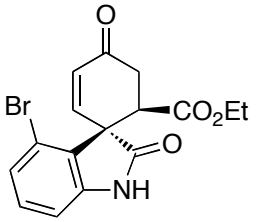
4a-major

White solid; R_f 0.57 (hexane/acetone = 2 : 1); $[\alpha]_D^{19}$ +103.8 (*c* = 0.96, $CHCl_3$, 85% ee); FTIR (KBr) ν 3263, 1736, 1681, 1613, 1581 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.06 (3H, t, *J* = 7.0 Hz), 2.83 (1H, dd, *J* = 17.0, 5.0 Hz), 3.67 (1H, dd, *J* = 17.0, 14.5 Hz), 3.94-4.05 (2H, m), 4.48 (1H, dd, *J* = 14.5, 5.0 Hz), 6.34 (1H, d, *J* = 10.0 Hz), 6.47 (1H, dd, *J* = 10.0, 1.5 Hz), 6.89-6.90 (1H, m), 7.17-7.20 (2H, m), 8.16 (1H, br); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ 13.69, 35.32, 44.90, 53.07, 61.30, 109.53, 118.62, 126.78, 129.27, 130.87, 131.83, 143.22, 145.25, 170.30, 175.12, 196.46; HRMS for $C_{16}H_{14}BrNO_4$: 363.0106, found: 363.0103.

The ee of the product was determined by chiral HPLC analysis with a Chiralpak AD column (0.46 × 25 cm, hexane/2-propanol = 90 : 10, flow rate 1.0 cm^3 min^{-1}, λ = 254 nm): t_R (minor)

= 16.93 min and t_R (major) = 24.62 min.

Ethyl (1*R*,6*R*)-4'-Bromo-2',4-dioxospiro[cyclohex[2]ene-1,3'-indoline]-6-carboxylate (4a-minor)

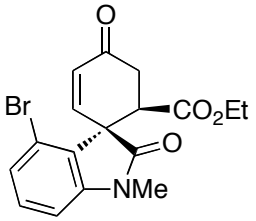


4a-minor

White solid; R_f 0.42 (hexane/acetone = 2 : 1); 83% ee; FTIR (KBr) ν 3319, 1734, 1613, 1578, 1447 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 0.92 (3H, t, *J* = 7.5 Hz), 2.94 (1H, dd, *J* = 18.0, 6.5 Hz), 3.80 (1H, dd, *J* = 18.0, 13.5 Hz), 3.85-3.95 (2H, m), 3.99 (1H, dd, *J* = 13.5, 6.5 Hz), 6.22 (1H, d, *J* = 10.0 Hz), 6.43 (1H, d, *J* = 10.0 Hz), 6.94 (1H, dd, *J* = 7.5, 1.0 Hz), 7.15-7.23 (2H, m), 8.66 (1H, br s); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ 13.53, 36.79, 44.01, 54.24, 61.34, 109.46, 120.12, 127.30, 127.92, 131.06, 132.70, 140.07, 143.36, 169.67, 177.92, 196.72; HRMS for $C_{16}H_{14}BrNO_4$ + H 364.0185, found: 364.0194.

The ee of the product was determined by chiral HPLC analysis with a Chiralpak AD column (0.46 × 25 cm, hexane/2-propanol = 90 : 10, flow rate 1.0 cm^3 min^{-1}, λ = 254 nm): t_R (major) = 25.78 min and t_R (minor) = 29.09 min.

Ethyl 4'-Bromo-1'-methyl-2',4-dioxospiro[cyclohex[2]ene-1,3'-indoline]-6-carboxylate (4b-major)

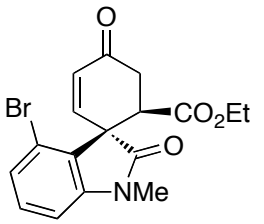


4b-major

Pale yellow solid; R_f 0.46 (hexane/acetone = 2 : 1); $[\alpha]_D^{22}$ +162.4 (*c* = 1.03, $CHCl_3$, 69% ee); FTIR (KBr) ν 1732, 1719, 1683, 1604, 1580, 1457 cm^{-1}; ^1H-NMR (500 MHz, $CDCl_3$) δ 1.04 (3H, t, *J* = 7.0 Hz), 2.82 (1H, dd, *J* = 16.5, 5.0 Hz), 3.26 (3H, s), 3.69 (1H, dd, *J* = 16.5, 14.5 Hz), 3.90-4.01 (2H, m), 4.46 (1H, dd, *J* = 14.5, 5.0 Hz), 6.33 (1H, d, *J* = 9.5 Hz), 6.41 (1H, d, *J* = 9.5 Hz), 6.85 (1H, d, *J* = 7.0 Hz), 7.19-7.26 (2H, m); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ 13.79, 26.95, 35.47, 44.88, 52.41, 61.15, 107.53, 118.57, 126.86, 128.85, 130.83, 131.86, 145.23, 146.03, 170.21, 173.06, 196.45; HRMS for $C_{17}H_{16}BrNO_4$: 377.0263, found: 377.0241.

The ee of the product was determined by chiral HPLC analysis with a Chiralpak AD column (0.46 × 25 cm, hexane/2-propanol = 90 : 10, flow rate 1.0 cm^3 min^{-1}, λ = 254 nm): t_R (minor) = 16.87 min and t_R (major) = 19.07 min.

Ethyl 4'-Bromo-1'-methyl-2',4-dioxospiro[cyclohex[2]ene-1,3'-indoline]-6-carboxylate (4b-minor)

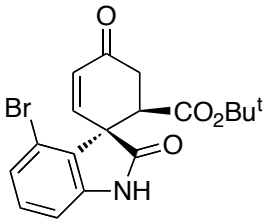


4b-minor

Pale yellow solid; R_f 0.34 (hexane/acetone = 2 : 1); 70% ee; FTIR (KBr) ν 1726, 1681, 1603, 1577, 1456 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 0.88 (3H, t, *J* = 7.5 Hz), 2.92 (1H, dd, *J* = 18.5, 7.0 Hz), 3.31 (3H, s), 3.79 (1H, dd, *J* = 18.5, 13.5 Hz), 3.83 (2H, q, *J* = 7.0 Hz), 3.99 (1H, dd, *J* = 13.5, 7.0 Hz), 6.14 (1H, d, *J* = 10.0 Hz), 6.42 (1H, d, *J* = 10.0 Hz), 6.88 (1H, t, *J* = 4.5 Hz), 7.23 (2H, d, *J* = 4.5 Hz); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ 13.65, 27.26, 36.89, 44.26, 53.63, 61.05, 107.54, 120.09, 126.82, 127.92, 130.96, 132.73, 140.32, 146.30, 169.61, 176.04, 196.69; HRMS for $C_{17}H_{16}BrNO_4$: 377.0263, found: 377.0269.

The ee of the product was determined by chiral HPLC analysis with a Chiralpak AD column (0.46 × 25 cm, hexane/2-propanol = 90 : 10, flow rate 1.0 cm^3 min^{-1}, λ = 254 nm): t_R (minor) = 30.36 min and t_R (major) = 36.73 min.

***tert*-Butyl 4'-Bromo-2',4-dioxospiro[cyclohex[2]ene-1,3'-indoline]-6-carboxylate (4c-major)**

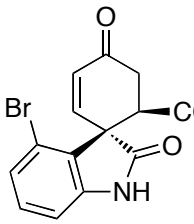


4c-major

Yellow oil; R_f 0.57 (hexane/acetone = 2 : 1); $[\alpha]_D^{20}$ +131.2 (*c* = 0.71, $CHCl_3$, 86% ee); FTIR (KBr) ν 3296, 1733, 1679, 1614, 1582, 1450 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.21 (9H, s), 2.73 (1H, dd, *J* = 16.5, 5.0 Hz), 3.65 (1H, dd, *J* = 16.5, 14.5 Hz), 4.37 (1H, dd, *J* = 14.5, 5.0 Hz), 6.32 (1H, d, *J* = 10.0 Hz), 6.47 (1H, d, *J* = 10.0 Hz), 6.88 (1H, d, *J* = 7.5 Hz), 7.16-7.21 (2H, m), 7.90 (1H, br s); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ 27.51 (×3), 35.19, 45.71, 53.35, 81.97, 109.47, 118.93, 126.83, 129.39, 130.81, 131.92, 143.04, 145.22, 169.31, 174.93, 197.00; HRMS for $C_{18}H_{18}BrNO_4$: 391.0419, found: 391.0417.

The ee of the product was determined by chiral HPLC analysis with a Chiralpak AD column (0.46 × 25 cm, hexane/2-propanol = 90 : 10, flow rate 1.0 cm^3 min^{-1}, λ = 254 nm): t_R (major) = 11.01 min and t_R (minor) = 12.41 min.

***tert*-Butyl 4'-Bromo-2',4-dioxospiro[cyclohex[2]ene-1,3'-indoline]-6-carboxylate (4c-minor)**

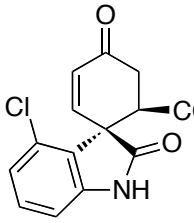


4c-minor

Yellow oil; R_f 0.51 (hexane/acetone = 2 : 1); 76% ee; FTIR (KBr) ν 1736, 1681, 1613, 1578, 1518, 1446 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.10 (9H, s), 2.86 (1H, dd, *J* = 18.0, 6.5 Hz), 3.77 (1H, dd, *J* = 18.0, 13.5 Hz), 3.91 (1H, dd, *J* = 13.5, 6.5 Hz), 6.17 (1H, d, *J* = 10.0 Hz), 6.41 (1H, d, *J* = 10.0 Hz), 6.94 (1H, d, *J* = 7.5 Hz), 7.17-7.23 (2H, m), 8.15 (1H, br s); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ 27.25 (×3), 36.87, 44.03, 54.52, 82.27, 109.55, 120.19, 127.82, 129.90, 131.01, 132.55, 140.17, 143.62, 168.60, 178.36, 197.22; HRMS for $C_{18}H_{18}BrNO_4$: 391.0419, found: 391.0410.

The ee of the product was determined by chiral HPLC analysis with a Chiralpak AD column (0.46 × 25 cm, hexane/2-propanol = 90 : 10, flow rate 1.0 cm^3 min^{-1}, λ = 254 nm): t_R (minor) = 20.36 min and t_R (major) = 21.97 min.

Ethyl 4'-Chloro-2',4-dioxospiro[cyclohex[2]ene-1,3'-indoline]-6-carboxylate (4d-major)

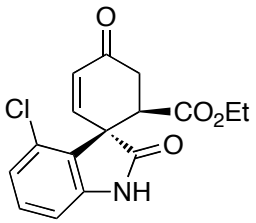


4d-major

Yellow oil; R_f 0.51 (hexane/acetone = 2 : 1); $[\alpha]_D^{21}$ +83.63 (*c* = 2.72, $CHCl_3$, 60% ee); FTIR (KBr) ν 1736, 1682, 1616, 1588, 1454 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.06 (3H, t, *J* = 7.5 Hz), 2.82 (1H, dd, *J* = 17.0, 5.0 Hz), 3.65 (1H, dd, *J* = 17.0, 14.5 Hz), 3.94-4.05 (2H, m), 4.37 (1H, dd, *J* = 14.5, 5.0 Hz), 6.32 (1H, d, *J* = 10.0 Hz), 6.47 (1H, d, *J* = 10.0 Hz), 6.85 (1H, d, *J* = 8.0 Hz), 7.01 (1H, d, *J* = 8.0 Hz), 7.24 (1H, t, *J* = 8.0 Hz), 8.24 (1H, br s); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ 13.70, 35.44, 44.93, 52.20, 61.34, 108.94, 123.76, 127.67, 130.48, 130.69, 131.68, 142.91, 145.14, 170.35, 175.14, 196.52; HRMS for $C_{16}H_{14}ClNO_4$: 319.0611, found: 319.0617.

The ee of the product was determined by chiral HPLC analysis with a Chiralpak AD column (0.46 × 25 cm, hexane/2-propanol = 90 : 10, flow rate 1.0 cm^3 min^{-1}, λ = 254 nm): t_R (minor) = 19.10 min and t_R (major) = 29.08 min.

Ethyl 4'-Chloro-2',4-dioxospiro[cyclohex[2]ene-1,3'-indoline]-6-carboxylate (4d-minor)

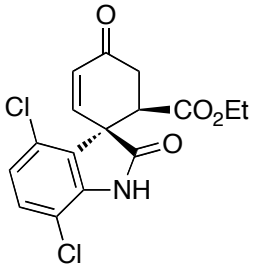


4d-minor

Yellow oil; R_f 0.43 (hexane/acetone = 2 : 1); 61% ee; FTIR (KBr) ν 1739, 1684, 1616, 1585, 1449 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 0.92 (3H, t, *J* = 7.5 Hz), 2.94 (1H, dd, *J* = 18.5, 6.5 Hz), 3.63 (1H, dd, *J* = 18.5, 14.0 Hz), 3.84-3.98 (3H, m), 6.23 (1H, d, *J* = 10.5 Hz), 6.41 (1H, d, *J* = 10.5 Hz), 6.91 (1H, d, *J* = 7.5 Hz), 7.04 (1H, d, *J* = 7.5 Hz), 7.26 (1H, t, *J* = 7.5 Hz), 8.72 (1H, br s); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ 13.55, 36.51, 44.06, 53.48, 61.34, 108.86, 124.61, 125.12, 130.91, 131.80, 132.26, 140.16, 143.05, 169.67, 177.82, 196.67; HRMS for $C_{16}H_{14}ClNO_4$: 319.0611, found: 319.0618.

The ee of the product was determined by chiral HPLC analysis with a Chiralpak AD column (0.46 × 25 cm, hexane/2-propanol = 90 : 10, flow rate 1.0 cm^3 min^{-1}, λ = 254 nm): t_R (major) = 28.78 min and t_R (minor) = 33.51 min.

Ethyl 4',7'-Dichloro-2',4-dioxospiro[cyclohex[2]ene-1,3'-indoline]-6-carboxylate (4e-major)

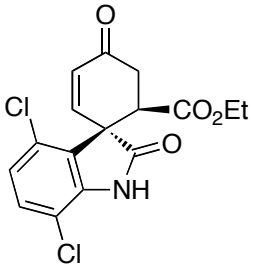


4e-major

Pale yellow solid; R_f 0.38 (hexane/acetone = 2 : 1); $[\alpha]_D^{23}$ +96.7 (*c* = 1.75, $CHCl_3$, 72% ee); FTIR (KBr) ν 1737, 1667, 1612, 1567, 1472 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.08 (3H, t, *J* = 7.5 Hz), 2.85 (1H, dd, *J* = 17.0, 5.0 Hz), 3.64 (1H, dd, *J* = 17.0, 14.5 Hz), 3.98-4.07 (2H, m), 4.35 (1H, dd, *J* = 14.5, 5.0 Hz), 6.34 (1H, d, *J* = 10.0 Hz), 6.45 (1H, d, *J* = 10.0 Hz), 6.98 (1H, d, *J* = 9.0 Hz), 7.25 (1H, d, *J* = 9.0 Hz), 8.77 (1H, br); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ 13.68, 35.36, 44.88, 53.23, 61.46, 114.10, 124.39, 128.6(6), 128.7(2), 130.42, 131.87, 140.68, 144.02, 170.02, 174.12, 196.13; HRMS for $C_{16}H_{13}Cl_2NO_4$: 353.0222, found: 353.0225.

The ee of the product was determined by chiral HPLC analysis with a Chiralpak AD column (0.46 × 25 cm, hexane/2-propanol = 90 : 10, flow rate 1.0 cm^3 min^{-1}, λ = 254 nm): t_R (minor) = 18.06 min and t_R (major) = 22.27 min.

Ethyl 4',7'-Dichloro-2',4-dioxospiro[cyclohex[2]ene-1,3'-indoline]-6-carboxylate (4e-minor)

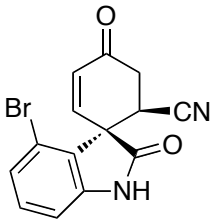


4e-minor

Pale yellow solid; R_f 0.34 (hexane/acetone = 2 : 1); 87% ee; FTIR (KBr) ν 1737, 1667, 1616, 1612, 1567, 1472 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 0.95 (3H, t, *J* = 7.0 Hz), 2.95 (1H, dd, *J* = 18.0, 6.0 Hz), 3.56 (1H, dd, *J* = 18.0, 13.5 Hz), 3.86-4.00 (3H, m), 6.23 (1H, d, *J* = 9.5 Hz), 6.41 (1H, d, *J* = 9.5 Hz), 7.00 (1H, d, *J* = 8.5 Hz), 7.26 (1H, d, *J* = 8.5 Hz), 8.32 (1H, br s); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ 13.51, 36.45, 44.18, 54.50, 61.46, 114.10, 125.13, 130.04, 130.54, 132.53, 139.24, 140.63, 169.47, 176.32, 196.29; HRMS for $C_{16}H_{13}Cl_2NO_4$: 353.0222, found: 353.0219.

The ee of the product was determined by chiral HPLC analysis with a Chiralpak AD column (0.46× 25 cm, hexane/2-propanol = 90 : 10, flow rate 1.0 cm^3 min^{-1}, λ = 254 nm): t_R (major) = 20.60 min and t_R (minor) = 25.16 min.

4'-Bromo-2',4-dioxospiro[cyclohex[2]ene-1,3'-indoline]-6-carbonitrile (4f-major)

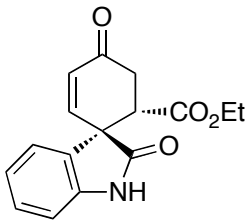


4f-major

White crysts; R_f 0.21 (hexane/acetone = 2 : 1); $[\alpha]_D^{25}$ +4.57 (*c* = 0.67, $CHCl_3$, 4% ee); FTIR (KBr) ν 2246, 1732, 1690, 1614, 1582, 1450 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 2.85 (1H, dd, *J* = 17.0, 5.0 Hz), 3.71 (1H, dd, *J* = 17.0, 14.0 Hz), 4.43 (1H, dd, *J* = 14.0, 5.0 Hz), 6.42 (1H, d, *J* = 10.5 Hz), 6.60 (1H, d, *J* = 10.5 Hz), 6.98-7.01 (1H, m), 7.28-7.29 (2H, m), 8.30 (1H, br); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ 31.42, 34.63, 52.81, 110.04, 116.57, 119.10, 126.83, 127.59, 132.17, 132.48, 142.44, 143.32, 172.13, 192.87; HRMS for $C_{16}H_9BrN_2O_2$: 315.9847, found: 315.9844.

The ee of the product was determined by chiral HPLC analysis with a Chiralpak AD column (0.46 × 25 cm, hexane/2-propanol = 90 : 10, flow rate 1.0 cm^3 min^{-1}, λ = 254 nm): t_R (minor) = 25.80 min and t_R (major) = 32.21 min.

Ethyl 2',4-Dioxospiro[cyclohex[2]ene-1,3'-indoline]-6-carboxylate (4g-major)

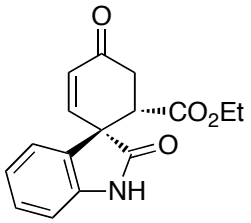


4g-major

Yellow oil; R_f 0.51 (hexane/acetone = 2 : 1); $[\alpha]_D^{24}$ –21.4 (*c* = 0.94, $CHCl_3$, 25% ee); FTIR (KBr) ν 1747, 1718, 1680, 1609, 1470 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 0.92 (3H, t, *J* = 7.5 Hz), 3.00 (1H, dd, *J* = 18.0 5.0 Hz), 3.11 (1H, dd, *J* = 18.0 14.0 Hz), 3.82-3.95 (3H, m), 6.25 (1H, d, *J* = 10.0 Hz), 6.46 (1H, d, *J* = 10.0 Hz), 6.99 (1H, d, *J* = 8.0 Hz), 7.04 (1H, t, *J* = 8.0 Hz), 7.21 (1H, d, *J* = 8.0 Hz), 7.30 (1H, t, *J* = 8.0 Hz), 9.15 (1H, br); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ 13.53, 35.68, 44.37, 52.53, 61.32, 110.52, 123.22, 124.85, 126.77, 129.58, 129.84, 141.09, 144.86, 169.62, 178.87, 196.43; HRMS for $C_{16}H_{15}NO_4$: 285.1001, found: 285.1008.

The ee of the product was determined by chiral HPLC analysis with a Chiralpak AD column (0.46 × 25 cm, hexane/2-propanol = 90 : 10, flow rate 1.0 cm^3 min^{-1}, λ = 254 nm): t_R (minor) = 24.62 min and t_R (major) = 28.73 min.

Ethyl 2',4-Dioxospiro[cyclohex[2]ene-1,3'-indoline]-6-carboxylate (4g-minor)

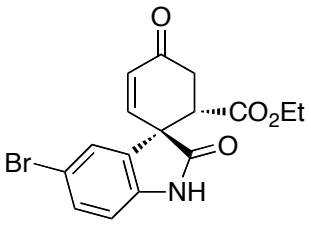


4g-minor

Yellow oil; R_f 0.57 (hexane/acetone = 2 : 1); 17% ee; FTIR (KBr) ν 3282, 1732, 1678, 1620, 1487, 1472 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.02 (3H, t, *J* = 7.0 Hz), 2.78-2.86 (1H, m), 3.62-3.70 (2H, m), 3.92-4.00 (2H, m), 6.26 (1H, d, *J* = 10.0 Hz), 6.42 (1H, d, *J* = 10.0 Hz), 6.97 (1H, d, *J* = 7.5 Hz), 7.07 (1H, t, *J* = 7.5 Hz), 7.14 (1H, d, *J* = 7.5 Hz), 7.29 (1H, dt, *J* = 7.5, 1.0 Hz), 9.16 (1H, br s); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ 13.62, 35.83, 47.85, 51.31, 61.25, 110.48, 122.80, 122.95, 129.27, 130.49, 131.47, 141.26, 146.81, 170.09, 176.33, 196.76; HRMS for $C_{16}H_{15}NO_4$ + H: 286.1079, found: 286.1079.

The ee of the product was determined by chiral HPLC analysis with a Chiralpak AD column (0.46 × 25 cm, hexane/2-propanol = 90 : 10, flow rate 1.0 cm^3 min^{-1}, λ = 254 nm): t_R (minor) = 28.68 min and t_R (major) = 32.20 min.

Ethyl 5'-Bromo-2',4-dioxospiro[cyclohex[2]ene-1,3'-indoline]-6-carboxylate (4h-major)

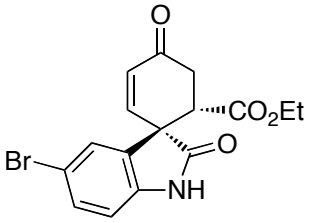


4h-major

Yellow oil; R_f 0.49 (hexane/acetone = 2 : 1); $[\alpha]_D^{26}$ –30.86 (*c* = 1.33, $CHCl_3$, 17% ee); FTIR (KBr) ν 1743, 1719, 1680, 1609, 1472 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 0.99 (3H, t, *J* = 7.5 Hz), 3.03-3.09 (2H, m), 3.86 (1H, dd, *J* = 12.5, 7.0 Hz), 3.89-4.01 (2H, m), 6.27 (1H, d, *J* = 9.5 Hz), 6.43 (1H, d, *J* = 9.5 Hz), 6.87 (1H, d, *J* = 8.5 Hz), 7.30 (1H, s), 7.43 (1H, dd, *J* = 8.5, 1.5 Hz), 8.68 (1H, br s); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ 13.64, 35.57, 44.55, 52.42, 61.52, 111.86, 115.57, 127.94, 128.99, 130.04, 132.68, 140.13, 143.98, 169.38, 178.10, 195.87; HRMS for $C_{16}H_{14}BrNO_4$: 363.0106, found: 363.0094.

The ee of the product was determined by chiral HPLC analysis with a Chiralpak AD column (0.46 × 25 cm, hexane/2-propanol = 95 : 5, flow rate 1.0 cm^3 min^{-1}, λ = 254 nm): t_R (minor) = 50.88 min and t_R (major) = 56.41 min.

Ethyl 5'-Bromo-2',4-dioxospiro[cyclohex[2]ene-1,3'-indoline]-6-carboxylate (4h-minor)

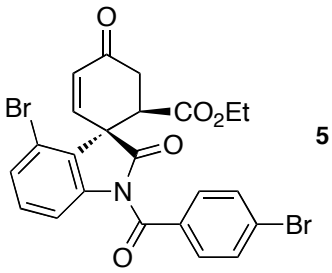


4h-minor

Yellow oil; R_f 0.57 (hexane/acetone = 2 : 1); 7% ee; FTIR (KBr) ν 3269, 1735, 1678, 1618, 1477 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.10 (3H, t, *J* = 7.5 Hz), 2.81-2.88 (1H, m), 3.57-3.65 (2H, m), 3.94-4.07 (2H, m), 6.26 (1H, d, *J* = 10.0 Hz), 6.37 (1H, d, *J* = 10.0 Hz), 6.85 (1H, d, *J* = 8.5 Hz), 7.25 (1H, d, *J* = 2.0 Hz), 7.43 (1H, dd, *J* = 8.5, 2.0 Hz), 8.31 (1H, br s); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ 13.74, 35.79, 47.81, 51.27, 61.54, 111.82, 115.36, 126.31, 130.83, 132.19, 133.67, 140.20, 145.64, 169.96, 175.64, 196.21; HRMS for $C_{16}H_{14}BrNO_4$: 363.0106, found: 363.0103.

The ee of the product was determined by chiral HPLC analysis with a Chiralpak AD column (0.46 × 25 cm, hexane/2-propanol = 95 : 5, flow rate 1.0 cm^3 min^{-1}, λ = 254 nm): t_R (major) = 46.38 min and t_R (minor) = 56.92 min.

Ethyl (1*S*,6*R*)-4'-Bromo-1'-(4-bromobenzoyl)-2',4-dioxospiro[cyclohex[2]ene-1,3'-indoline]-6-carboxylate (5)



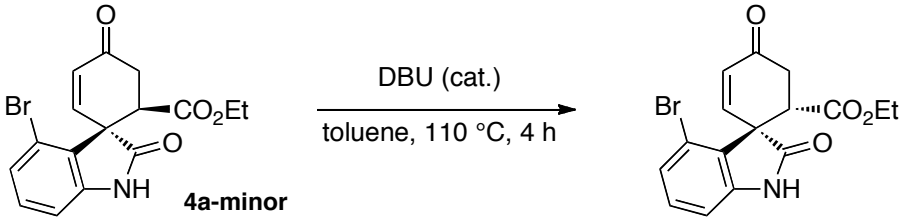
5

To a solution of **4a** (30.0 mg, 0.10 mmol) in CH_2Cl_2 (1.0 mL) were added 4-BrC_6H_4COCl (22.0 mg, 0.10 mmol) and Et_3N (0.03 mL, 0.20 mmol) at r.t., and the mixture was stirred for 72 h. The mixture was diluted with CH_2Cl_2, washed with sat. $NaHCO_3$ and brine, and dried (Na_2SO_4). After evaporation, the crude product was purified by silica gel column chromatography (elution with hexane-acetone) and recrystallized from hexane-Et_2O to afford **5** (20 mg, 50%).

Colorless plates; mp 187-189 °C (sublimed, recryst from hexane/Et_2O); R_f 0.71 (hexane/acetone = 2 : 1); 98% ee; FTIR (KBr) ν 1762, 1734, 1694, 1683, 1588, 1447 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.09 (3H, t, *J* = 7.0 Hz), 2.81 (1H, dd, *J* = 17.0, 5.0 Hz), 3.43 (1H, dd, *J* = 17.0, 14.5 Hz), 4.00-4.10 (2H, m), 4.57 (1H, dd, *J* = 14.5, 5.0 Hz), 6.32 (1H, d, *J* = 10.0 Hz), 6.52 (1H, d, *J* = 10.0 Hz), 7.32 (1H, t, *J* = 8.0 Hz), 7.39 (1H, d, *J* = 8.0 Hz), 7.61-7.63 (2H, m), 7.76-7.78 (2H, m), 7.85 (1H, d, *J* = 8.0 Hz); ^{13}C NMR (125.8 MHz, $CDCl_3$) δ 13.83, 35.31, 46.22, 52.89, 61.74, 114.53, 118.43, 128.11, 128.48, 129.46, 131.15, 131.18 (×2), 131.62 (×2), 132.26, 132.39, 142.06, 144.14, 168.03, 170.73, 172.64, 195.24; Anal. Calcd for $C_{23}H_{17}Br_2NO_5$: C, 50.48%; H, 3.13%; N, 2.56%. Found: C, 50.32%; H, 2.94%; N, 2.42%.

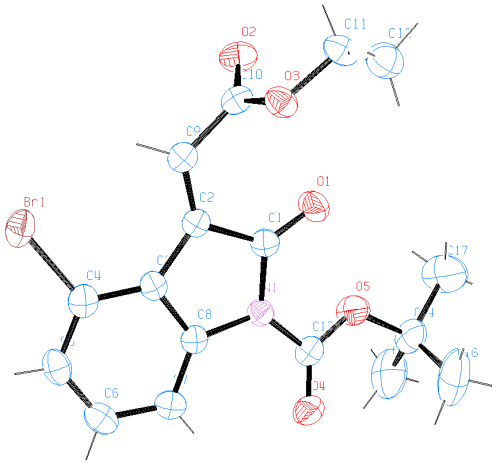
The ee of the product was determined by chiral HPLC analysis with a Chiralpak AD column (0.46 × 25 cm, hexane/2-propanol = 90 : 10, flow rate 1.0 cm^3 min^{-1}, λ = 254 nm): t_R (minor) = 17.69 min and t_R (major) = 19.40 min.

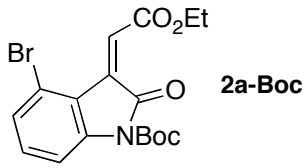
DBU-catalyzed Isomerisation of 4a-minor



4a-minor

To a solution of **4a**-minor (27 mg, 0.08 mmol) in toluene (2.0 mL) was added DBU (3.6 mL, 0.02 mmol) at r.t., and the mixture was stirred at 110 °C for 4 h. After concentration, the residue was purified by silica gel column chromatography (hexane/acetone = 1 : 1) to afford the desired product (27 mg, >99% yield) in a pure form. This was indistinguishable from **4a**-major with respect to ^1H NMR data, but the ee was found to be reversed by chiral HPLC analysis: Chiralpak AD column (0.46 × 25 cm, hexane/2-propanol = 90 : 10, flow rate 1.0 cm^3 min^{-1}, λ = 254 nm): t_R (major) = 17.86 min and t_R (minor) = 25.92 min.





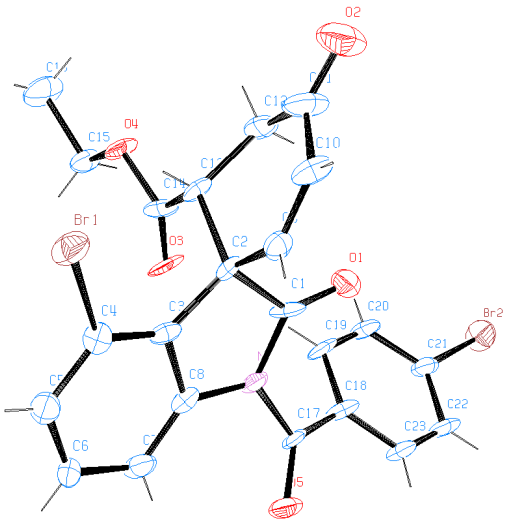
2a-Boc

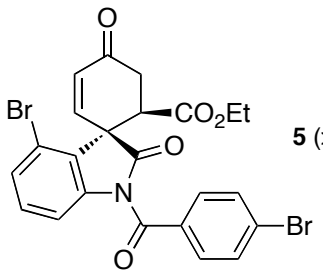
mp 127-129 °C

Datablock:

Empirical Formula	$C_{17}H_{18}BrNO_5$
Formula Weight	396.24
Temperature	0 °C
Crystal Dimensions	0.200 × 0.200 × 0.100 mm
Radiation	MoKα (λ = 0.71075 Å)
Crystal System	triclinic
Lattice Type	primitive
Lattice Parameters	a = 7.226(8) Å
	b = 11.001(12) Å
	c = 12.114(13) Å
	α = 69.75(3)°
	β = 80.82(4)°
	γ = 84.36(4)°
	V = 891(2) Å^3
Space Group	P-1 (#2)
Z value	2
D_{calc}	1.477 g / cm^3
F_{000}	404.00
μ(MoKα)	23.386 cm^{-1}
Reflection / Parameter Ratio	17.24
Residuals: R (I>2.00σ(I))	0.0471
Residuals: R (All reflections)	0.0534
Residuals: wR (All reflections)	0.0564
Goodness of Fit Indicator	5.670
Max Shift / Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.85 e$^-$ / Å^3
Minimum peak in Final Diff. Map	–0.65 e$^-$ / Å^3

Figure 1. X-Ray Structure of **2a-Boc**.





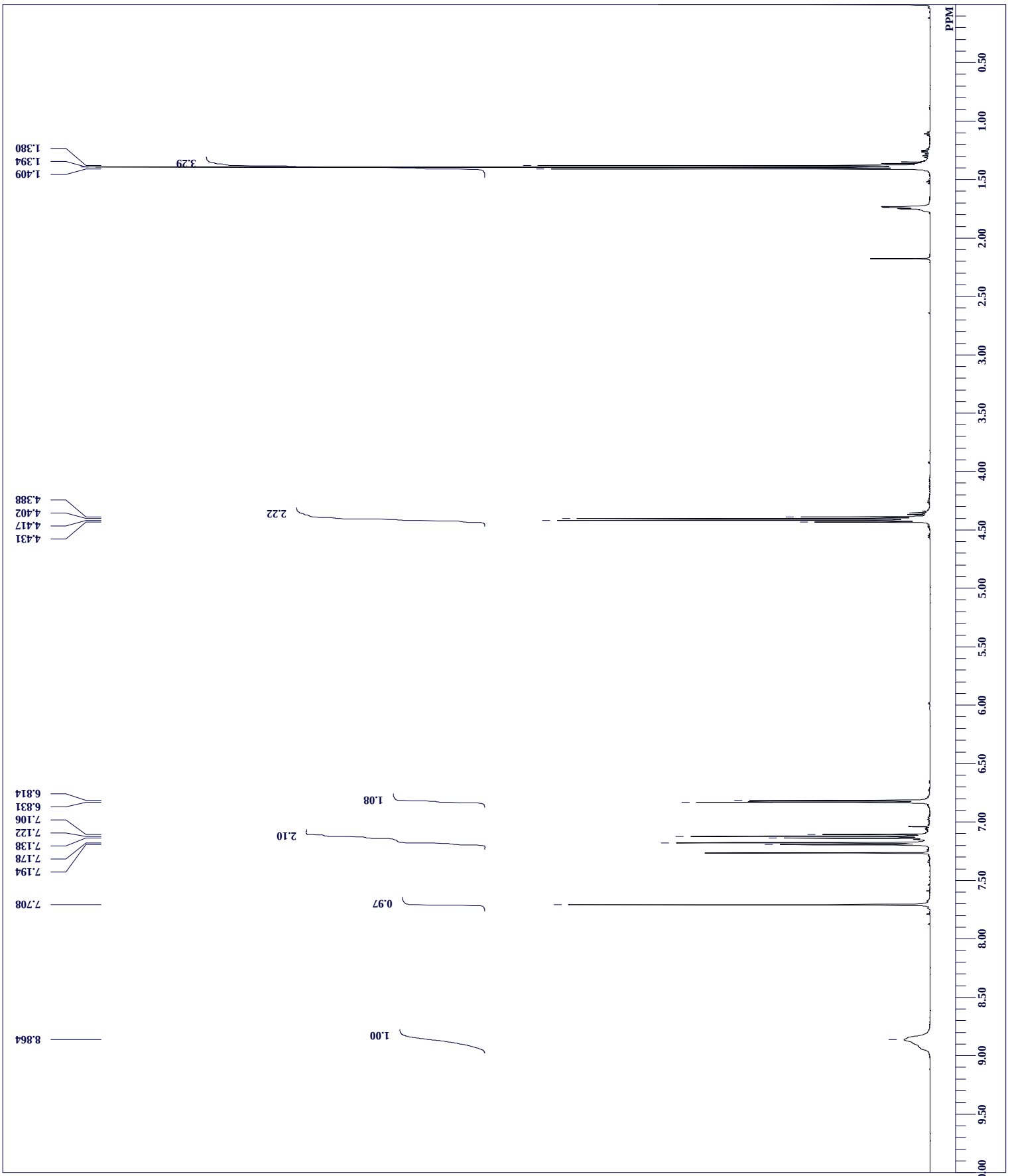
5 (>95% ee)

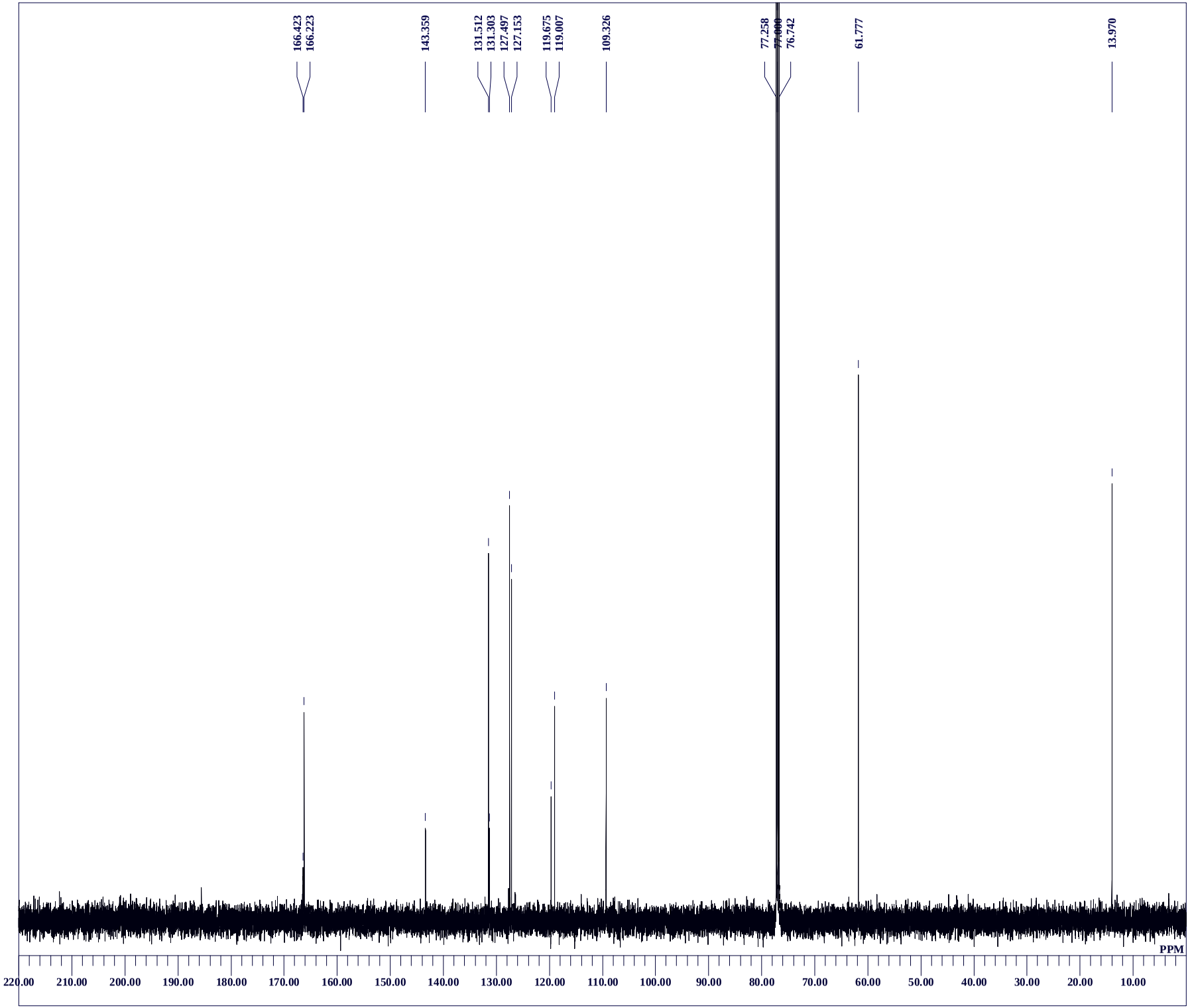
mp 187-189 °C

Datablock:

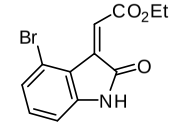
Empirical Formula	$C_{23}H_{16}Br_2NO_5$
Formula Weight	546.19
Temperature	0 °C
Crystal Dimensions	0.200 × 0.200 × 0.200 mm
Radiation	MoKα (λ = 0.71075 Å)
Crystal System	monoclinic
Lattice Type	primitive
Lattice Parameters	a = 12.307(3) Å
	b = 11.266(2) Å
	c = 16.704(4) Å
	β = 109.865(9)°
	V = 2178.1(8) Å^3
Space Group	$P2_1$ / c (#14)
Z value	4
D_{calc}	1.665 g / cm^3
F_{000}	1084.00
μ(MoKα)	37.677 cm^{-1}
Reflection / Parameter Ratio	16.76
Residuals: R (I>2.00σ(I))	0.1405
Residuals: R (All reflections)	0.1559
Residuals: wR (All reflections)	0.1764
Goodness of Fit Indicator	4.426
Max Shift / Error in Final Cycle	0.143
Maximum peak in Final Diff. Map	3.11 e$^-$ / Å^3
Minimum peak in Final Diff. Map	–3.40 e$^-$ / Å^3

Figure 2. X-Ray Structure of **5**.


DFILE 2a_proton.als
COMNT single_pulse
DATIM 2013-10-22 15:14:34
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 4.68 usec
IRNUC 1H
CTEMP 21.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 48
CO2Et
O
NH
Br
2a
1H NMR
(500 MHz, CDCl3)
PPM
0.50
1.00
1.50
2.00
2.50
3.00
3.50
4.00
4.50
5.00
5.50
6.00
6.50
7.00
7.50
8.00
8.50
9.00
9.50
10.00
1.380
1.394
1.409
3.29
4.388
4.402
4.417
4.431
2.22
6.814
6.831
7.106
7.122
7.138
7.178
7.194
1.08
2.10
7.708
0.97
8.864
1.00



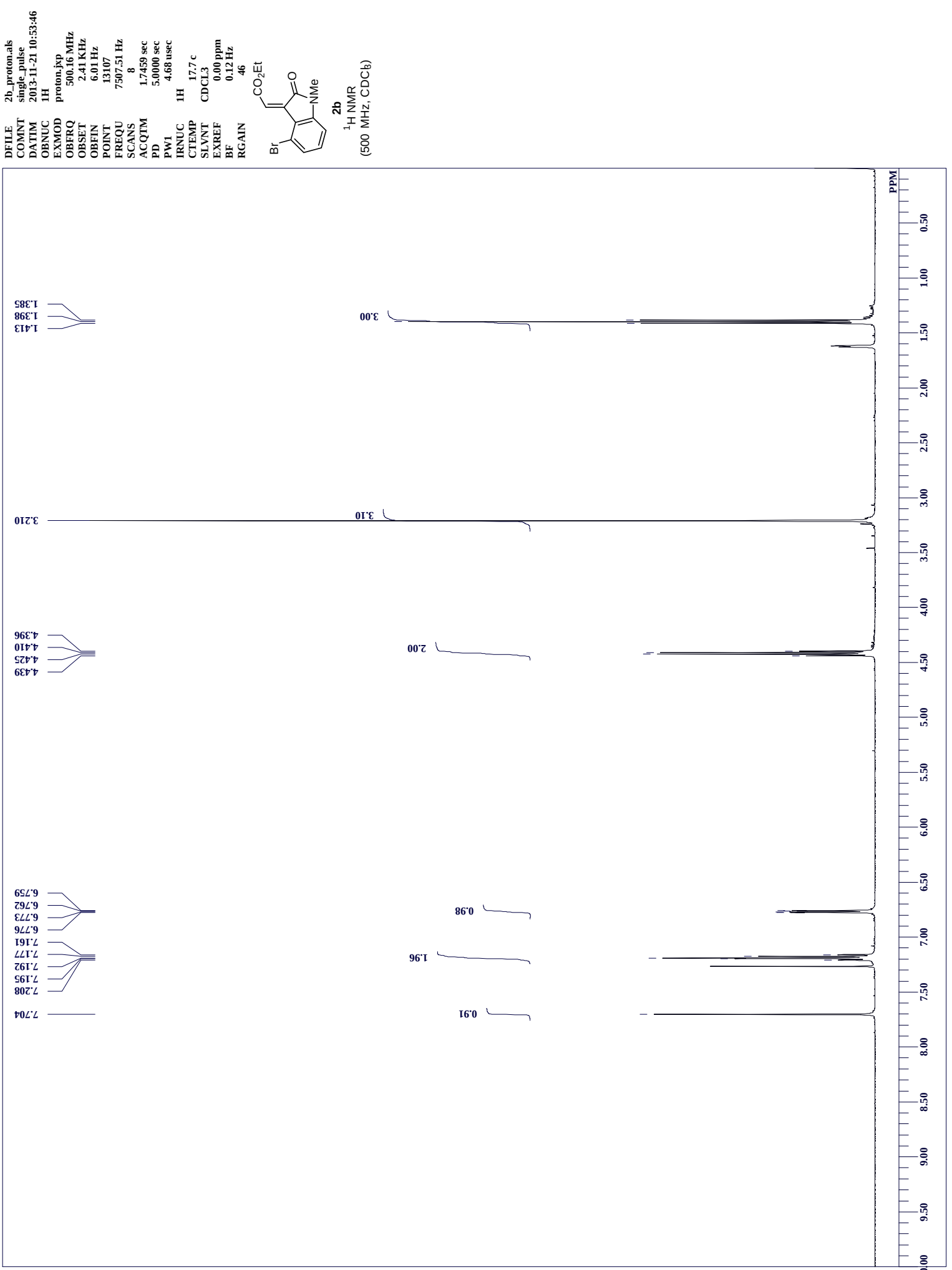
DFILE	2a_carbon.als
COMNT	single pulse decoupled gat
DATIM	2013-10-22 15:16:43
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.54 Hz
SCANS	512
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	22.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

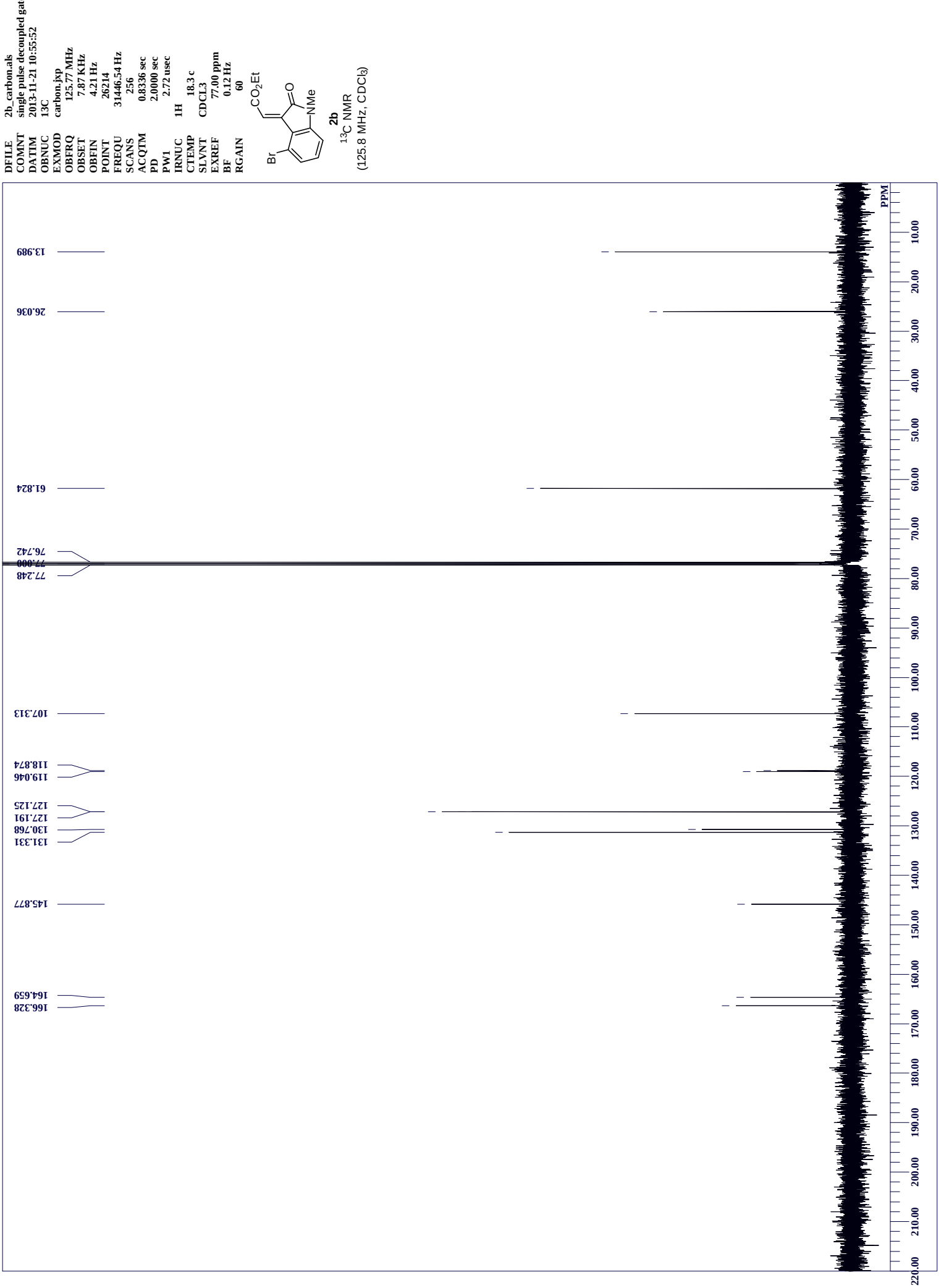


2a

^{13}C NMR

(125.8 MHz, $CDCl_3$)


DFILE 2b_proton.als
COMNT single_pulse
DATIM 2013-11-21 10:53:46
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 4.68 usec
IRNUC 1H
CTEMP 17.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 46
2b
1H NMR
(500 MHz, CDCl3)
PPM
1.385
1.398
1.413
3.210
4.396
4.410
4.425
4.439
6.759
6.762
6.773
6.776
7.161
7.177
7.192
7.195
7.208
7.704
3.00
3.10
2.00
0.98
1.96
0.91


DFILE 2b_carbon.als
COMNT single pulse decoupled gat
DATIM 2013-11-21 10:55:52
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 256
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 18.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60
CO2Et
O
NMe
Br
2b
13C NMR
(125.8 MHz, CDCl3)
13.989
26.036
61.824
76.742
77.000
77.248
107.313
118.874
119.046
127.125
127.191
130.768
131.331
145.877
164.659
166.328
PPM
10.00
20.00
30.00
40.00
50.00
60.00
70.00
80.00
90.00
100.00
110.00
120.00
130.00
140.00
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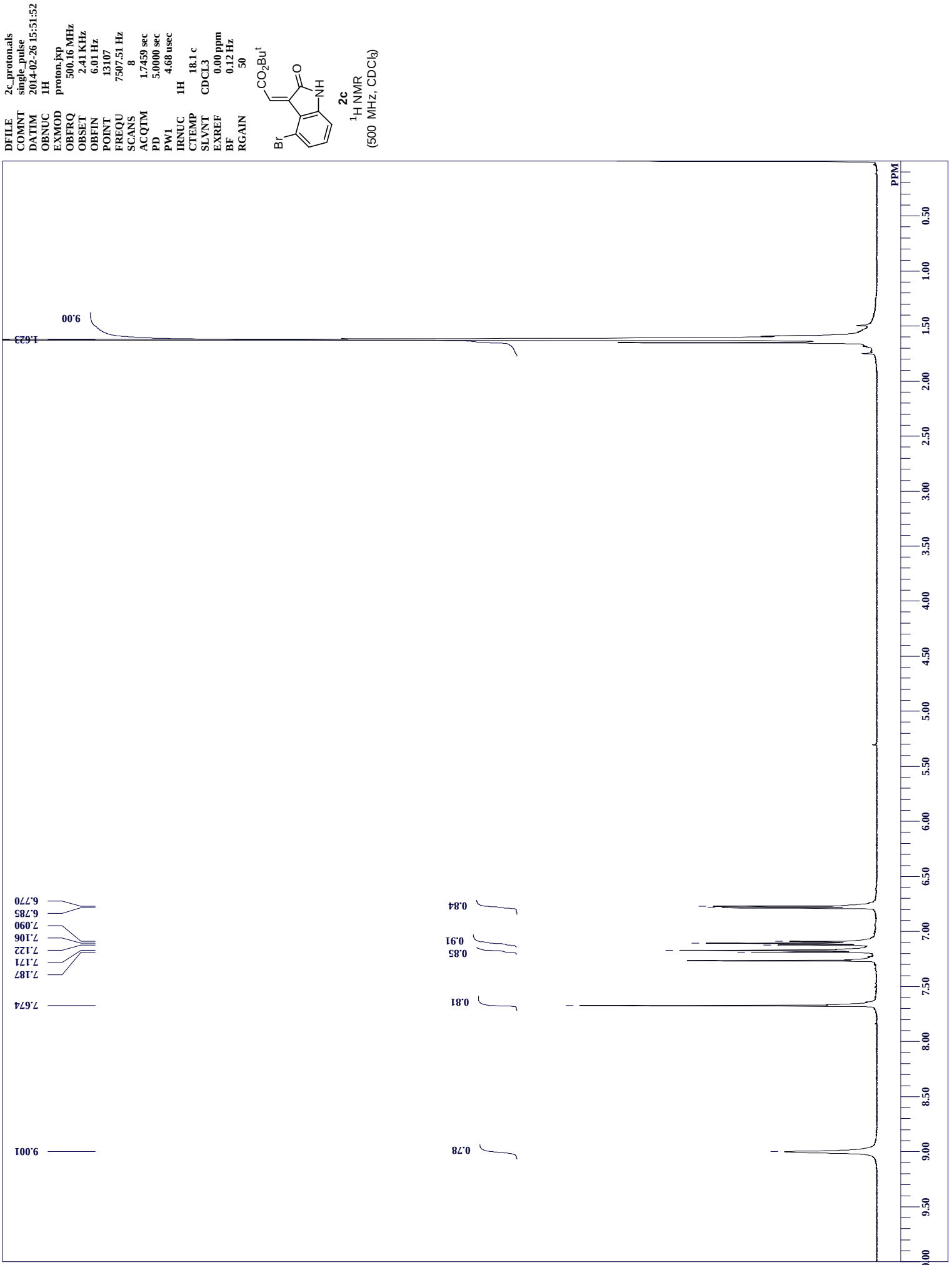
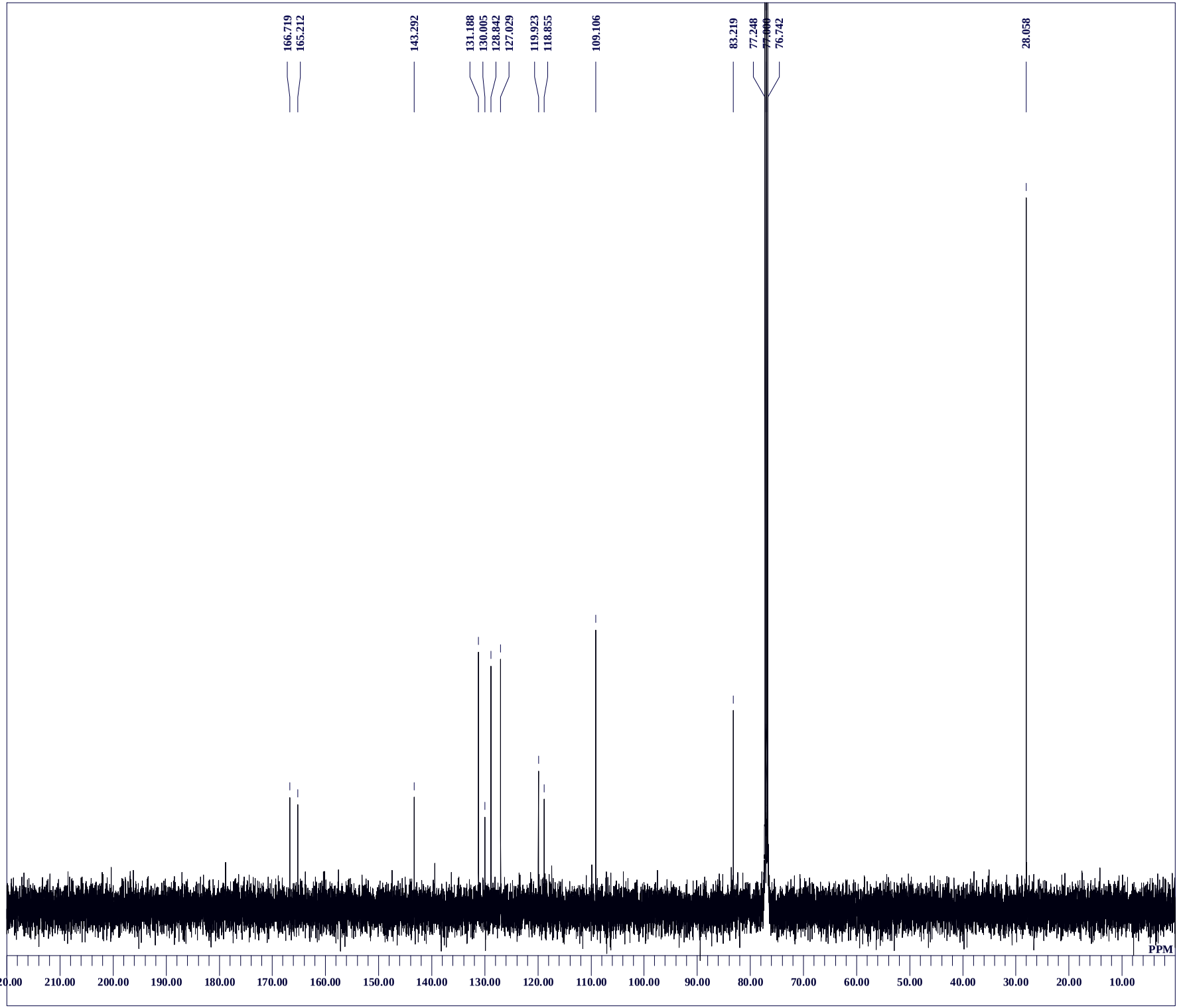
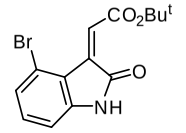
170.00
180.00
190.00
200.00
210.00
220.00


DFILE 2c_proton.als
COMNT single_pulse
DATIM 2014-02-26 15:51:52
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 4.68 usec
IRNUC 1H
CTEMP 18.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 50
CO2But
O
NH
Br
2c
1H NMR
(500 MHz, CDCl3)
9.00
1.623
9.001
7.674
7.187
7.171
7.122
7.106
7.090
6.785
6.770
0.78
0.81
0.84
0.85
0.91
PPM
0.50
1.00
1.50
2.00
2.50
3.00
3.50
4.00
4.50
5.00
5.50
6.00
6.50
7.00
7.50
8.00
8.50
9.00
9.50
10.00



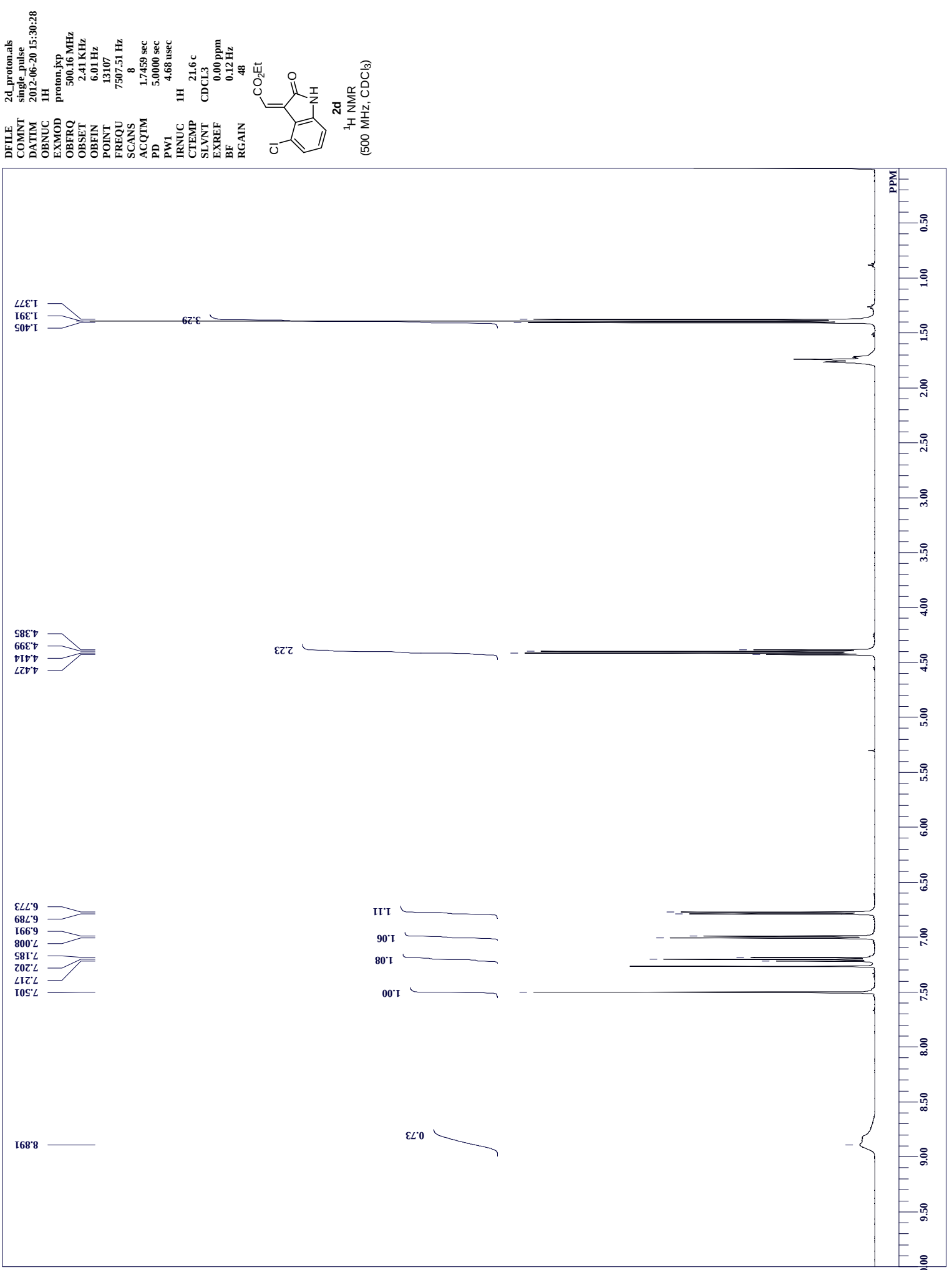
DFILE	2c_carbon.als
COMNT	single pulse decoupled gat
DATIM	2014-02-26 16:08:25
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.54 Hz
SCANS	256
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	18.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	54

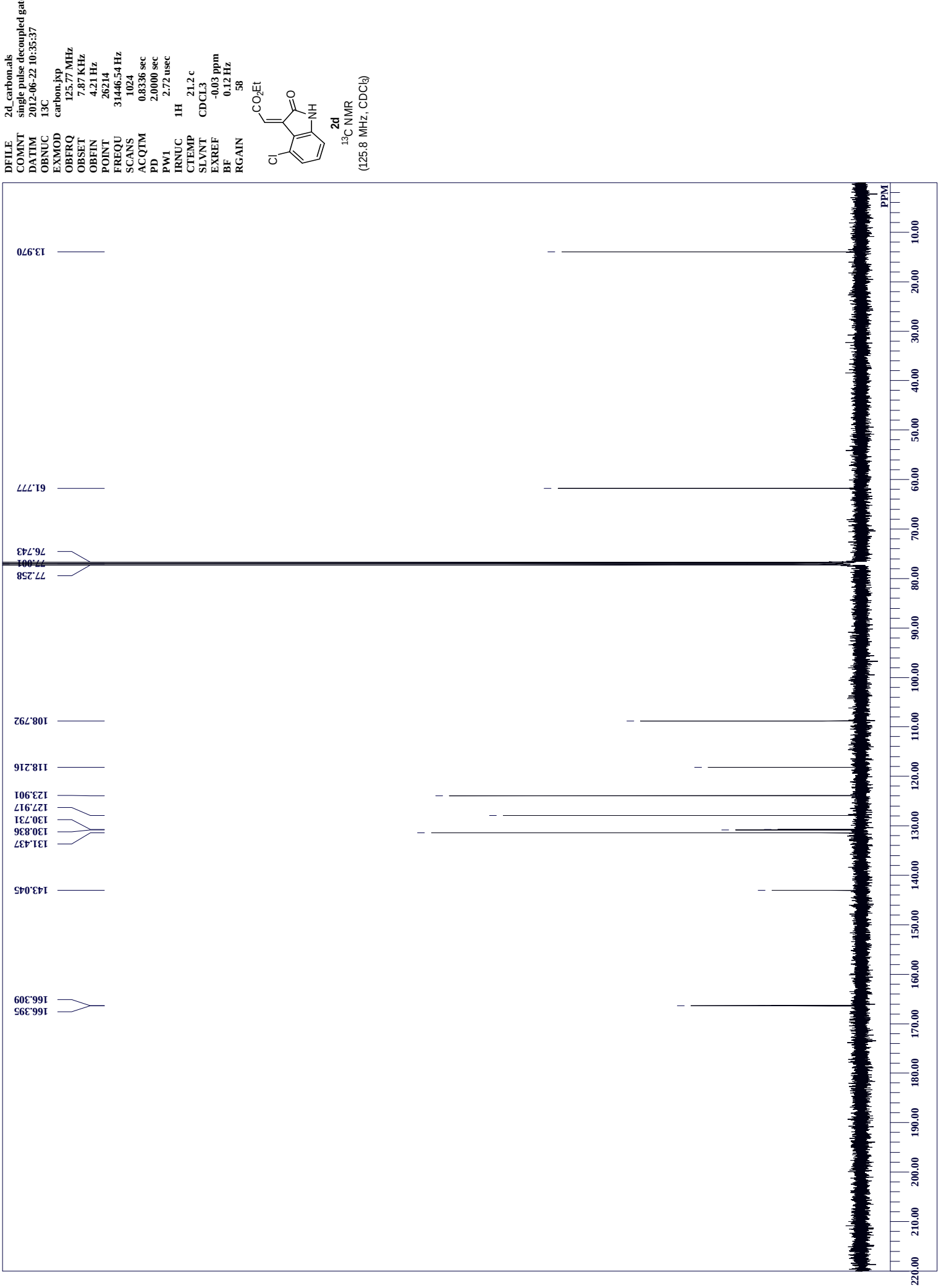


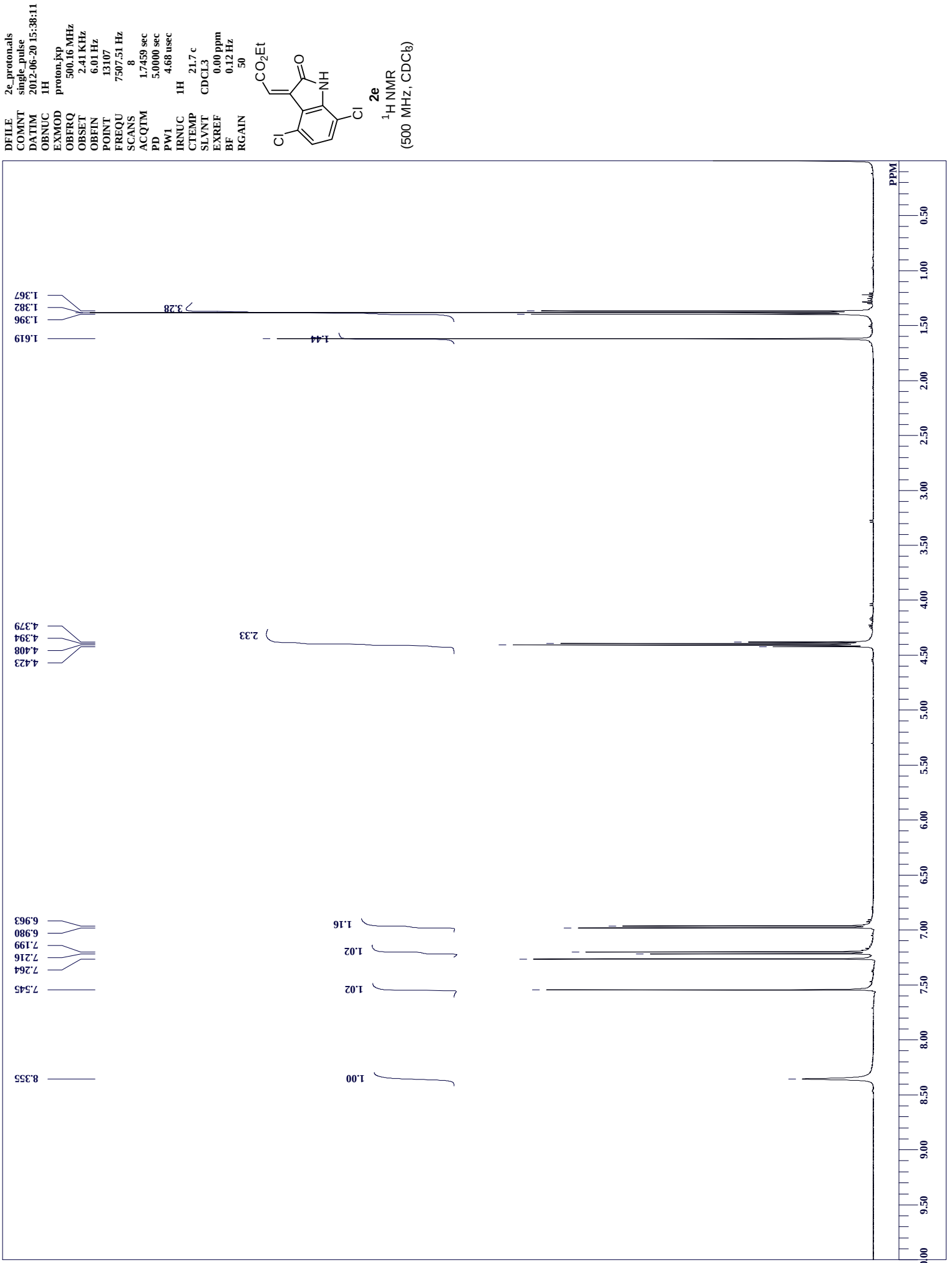
2c

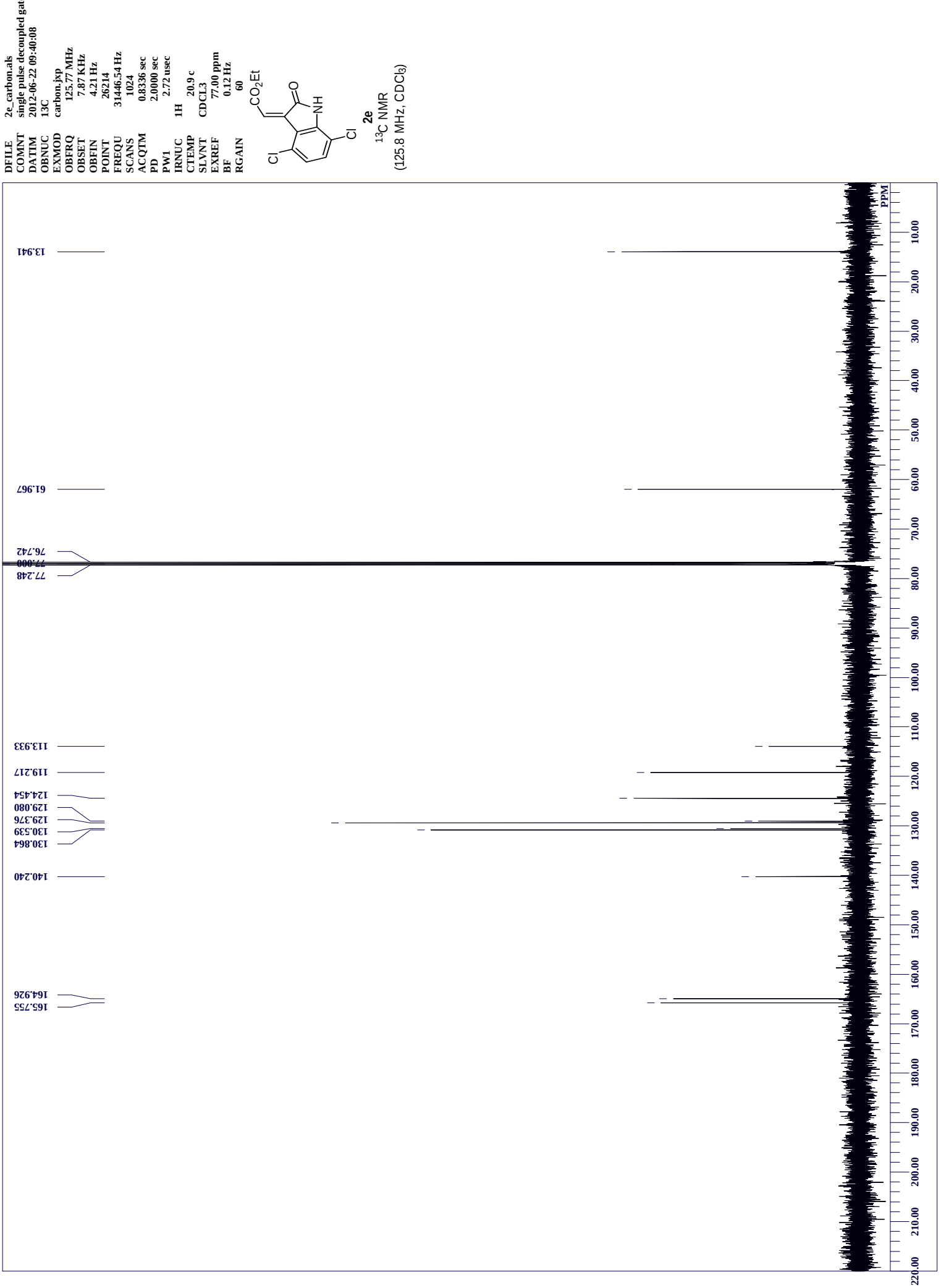
^{13}C NMR

(125.8 MHz, $CDCl_3$)


DFILE 2d_proton.als
COMNT single_pulse
DATIM 2012-06-20 15:30:28
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 4.68 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 48
2d
1H NMR
(500 MHz, CDCl3)
PPM
1.377
1.391
1.405
3.29
4.385
4.399
4.414
4.427
2.23
6.773
6.789
6.991
7.008
7.185
7.202
7.217
7.501
8.891
0.73
1.11
1.06
1.08
1.00


DFILE 2d_carbon.als
COMNT single pulse decoupled gat
DATIM 2012-06-22 10:35:37
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 1024
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 21.2 c
SLVNT CDCL3
EXREF -0.03 ppm
BF 0.12 Hz
RGAIN 58
CO2Et
O
NH
Cl
2d
13C NMR
(125.8 MHz, CDCl3)
13.970
61.777
76.743
77.001
77.258
108.792
118.216
123.901
127.917
130.731
130.836
131.437
143.045
166.309
166.395
PPM
10.00
20.00
30.00
40.00
50.00
60.00
70.00
80.00
90.00
100.00
110.00
120.00
130.00
140.00
150.00
160.00
170.00
180.00
190.00
200.00
210.00
220.00


DFILE 2e_proton.als
COMNT single_pulse
DATIM 2012-06-20 15:38:11
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 4.68 usec
IRNUC 1H
CTEMP 21.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 50
CO2Et
NH
O
Cl
Cl
2e
1H NMR
(500 MHz, CDCl3)
PPM
10.00
9.50
9.00
8.50
8.00
7.50
7.00
6.50
6.00
5.50
5.00
4.50
4.00
3.50
3.00
2.50
2.00
1.50
1.00
0.50
1.367
1.382
1.396
1.619
3.28
1.44
4.379
4.394
4.408
4.423
2.33
6.963
6.980
7.199
7.216
7.264
7.545
8.355
1.16
1.02
1.02
1.00


DFILE 2e_carbon.als
COMNT single pulse decoupled gat
DATIM 2012-06-22 09:40:08
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 1024
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 20.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60
CO2Et
O
NH
Cl
Cl
2e
13C NMR
(125.8 MHz, CDCl3)
13.941
61.967
76.742
77.000
77.248
113.933
119.217
124.454
129.080
129.376
130.539
130.864
140.240
164.926
165.755
PPM
10.00
20.00
30.00
40.00
50.00
60.00
70.00
80.00
90.00
100.00
110.00
120.00
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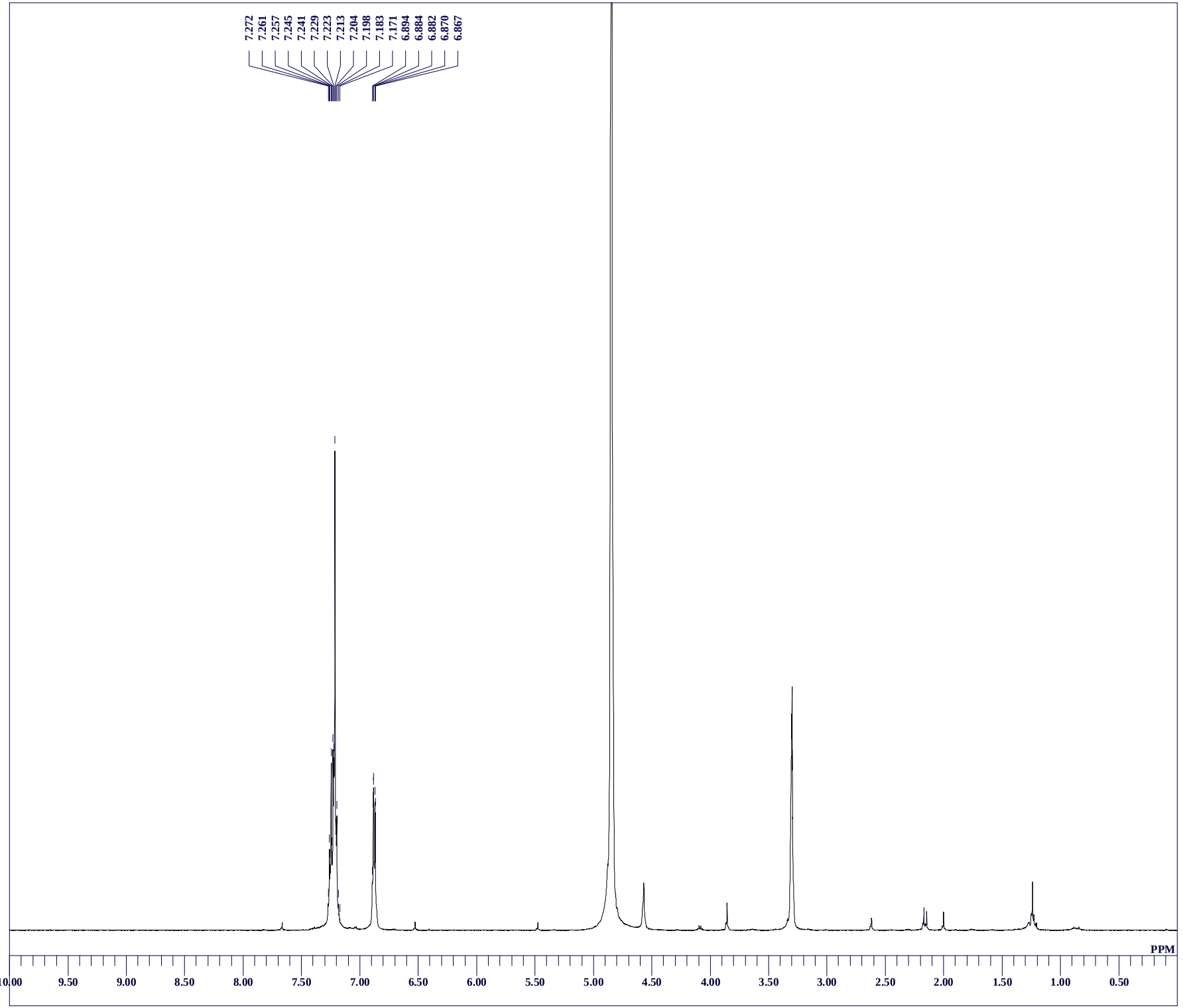
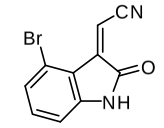
160.00
170.00
180.00
190.00
200.00
210.00
220.00



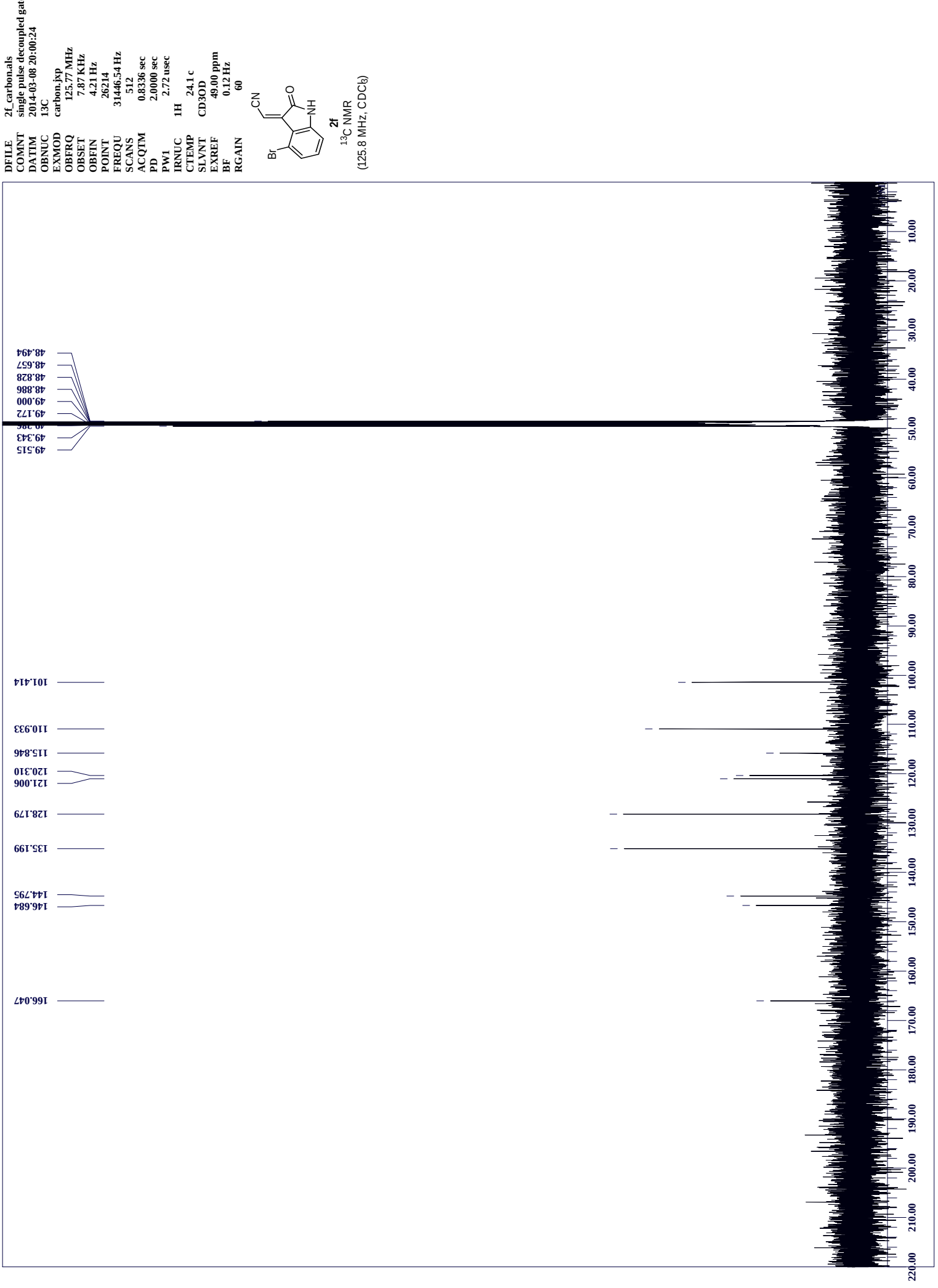
DFILE	2f_proton.als
COMNT	single_pulse
DATIM	2014-03-08 19:57:28
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	4.68 usec
IRNUC	1H
CTEMP	23.8 c
SLVNT	CD3OD
EXREF	3.30 ppm
BF	0.12 Hz
RGAIN	50



2f

^{1}H NMR

(500 MHz, $CDCl_3$)


DFILE 2f_carbon.als
COMNT single pulse decoupled gat
DATIM 2014-03-08 20:00:24
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 512
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 24.1 c
SLVNT CD3OD
EXREF 49.00 ppm
BF 0.12 Hz
RGAIN 60
CN
O
NH
Br
2f
13C NMR
(125.8 MHz, CDCl3)
48.494
48.657
48.828
48.886
49.000
49.172
49.343
49.515
101.414
110.933
115.846
120.310
121.006
128.179
135.199
144.795
146.684
166.047
10.00
20.00
30.00
40.00
50.00
60.00
70.00
80.00
90.00
100.00
110.00
120.00
130.00
140.00
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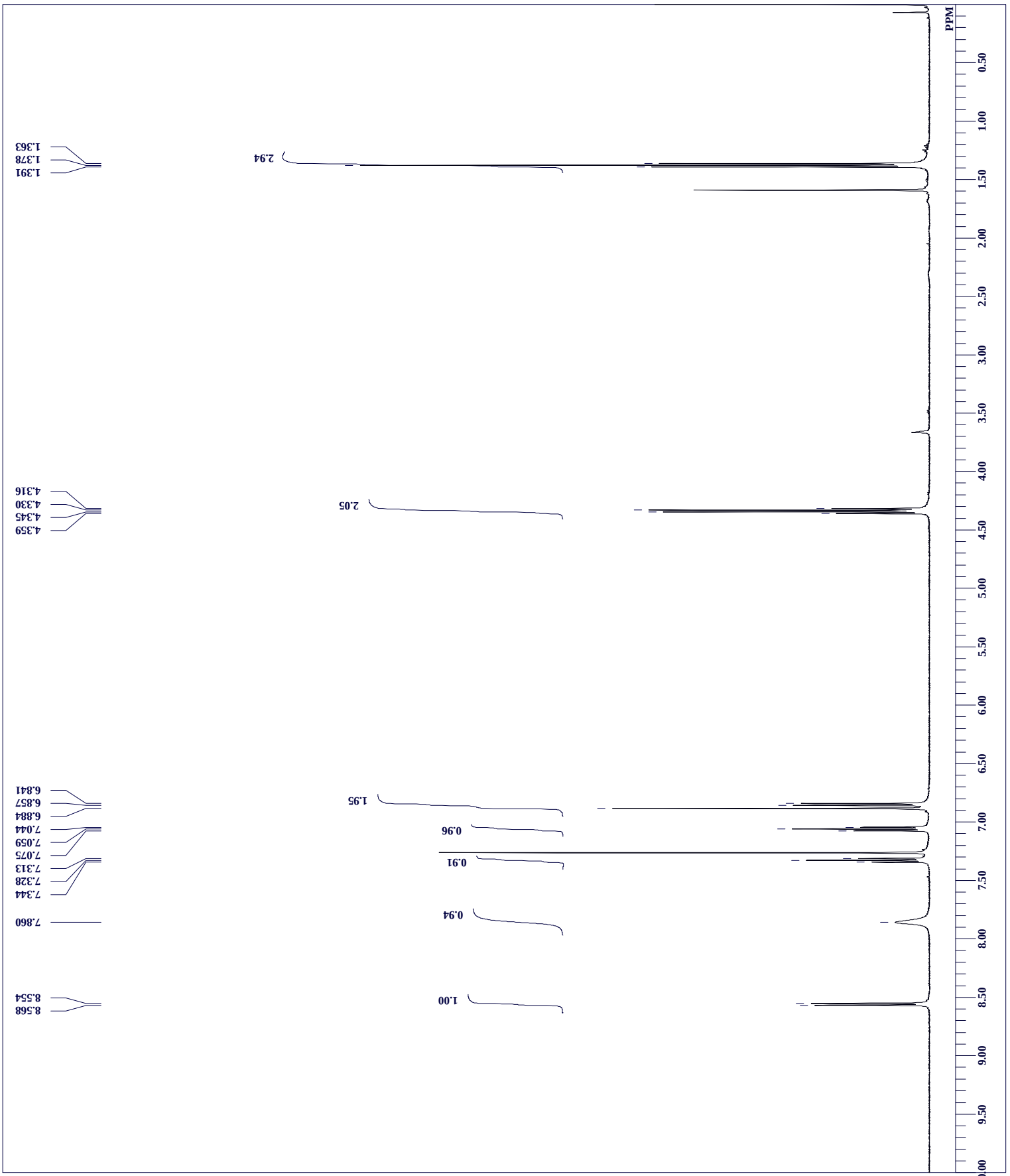
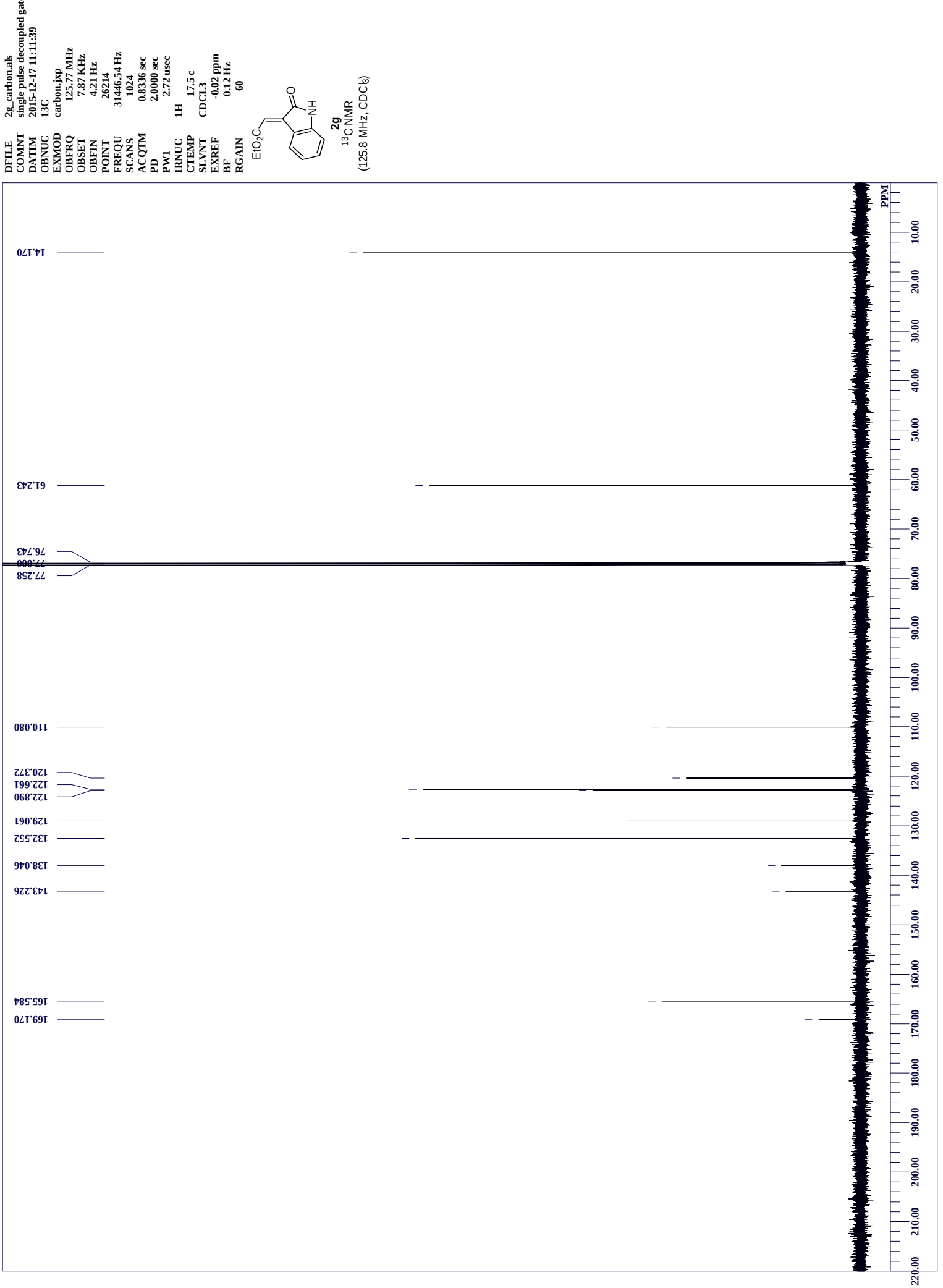
170.00
180.00
190.00
200.00
210.00
220.00


DFILE 2g_proton.als
COMNT single_pulse
DATIM 2015-12-16 22:12:19
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 4.68 usec
IRNUC 1H
CTEMP 18.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 56
EtO2C
O
NH
2g
1H NMR
(500 MHz, CDCl3)
PPM
0.50
1.00
1.50
2.00
2.50
3.00
3.50
4.00
4.50
5.00
5.50
6.00
6.50
7.00
7.50
8.00
8.50
9.00
9.50
10.00
1.363
1.378
1.391
2.94
4.316
4.330
4.345
4.359
2.05
6.841
6.857
6.884
7.044
7.059
7.075
7.313
7.328
7.344
1.95
0.96
0.91
7.860
0.94
8.554
8.568
1.00


DFILE 2g_carbon.als
COMNT single pulse decoupled gat
DATIM 2015-12-17 11:11:39
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 1024
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 17.5 c
SLVNT CDCL3
EXREF -0.02 ppm
BF 0.12 Hz
RGAIN 60
EtO2C
O
NH
2g
13C NMR
(125.8 MHz, CDCl3)
14.170
61.243
76.743
77.000
77.258
110.080
120.372
122.661
122.890
129.061
132.552
138.046
143.226
165.584
169.170
PPM
10.00
20.00
30.00
40.00
50.00
60.00
70.00
80.00
90.00
100.00
110.00
120.00
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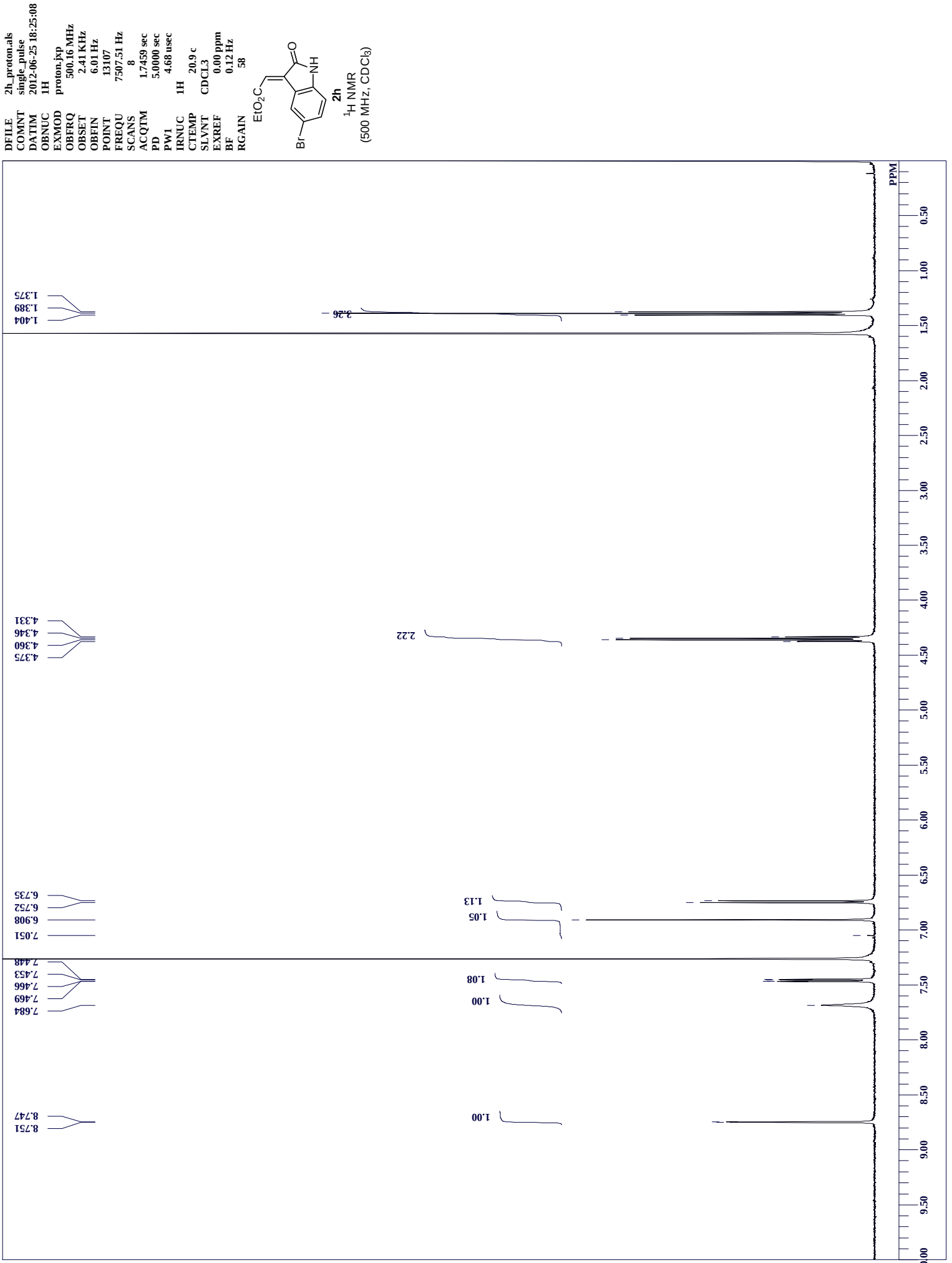
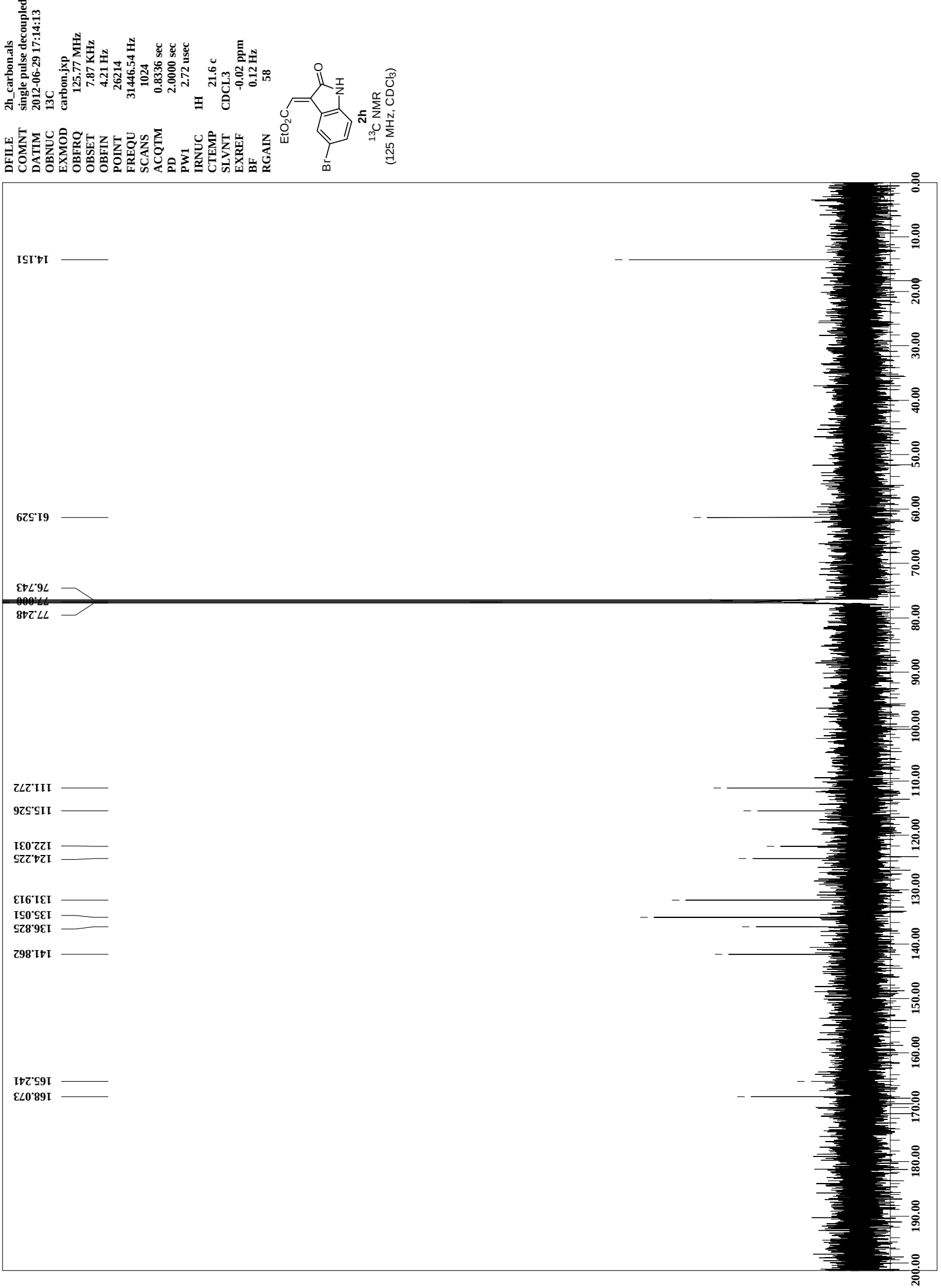
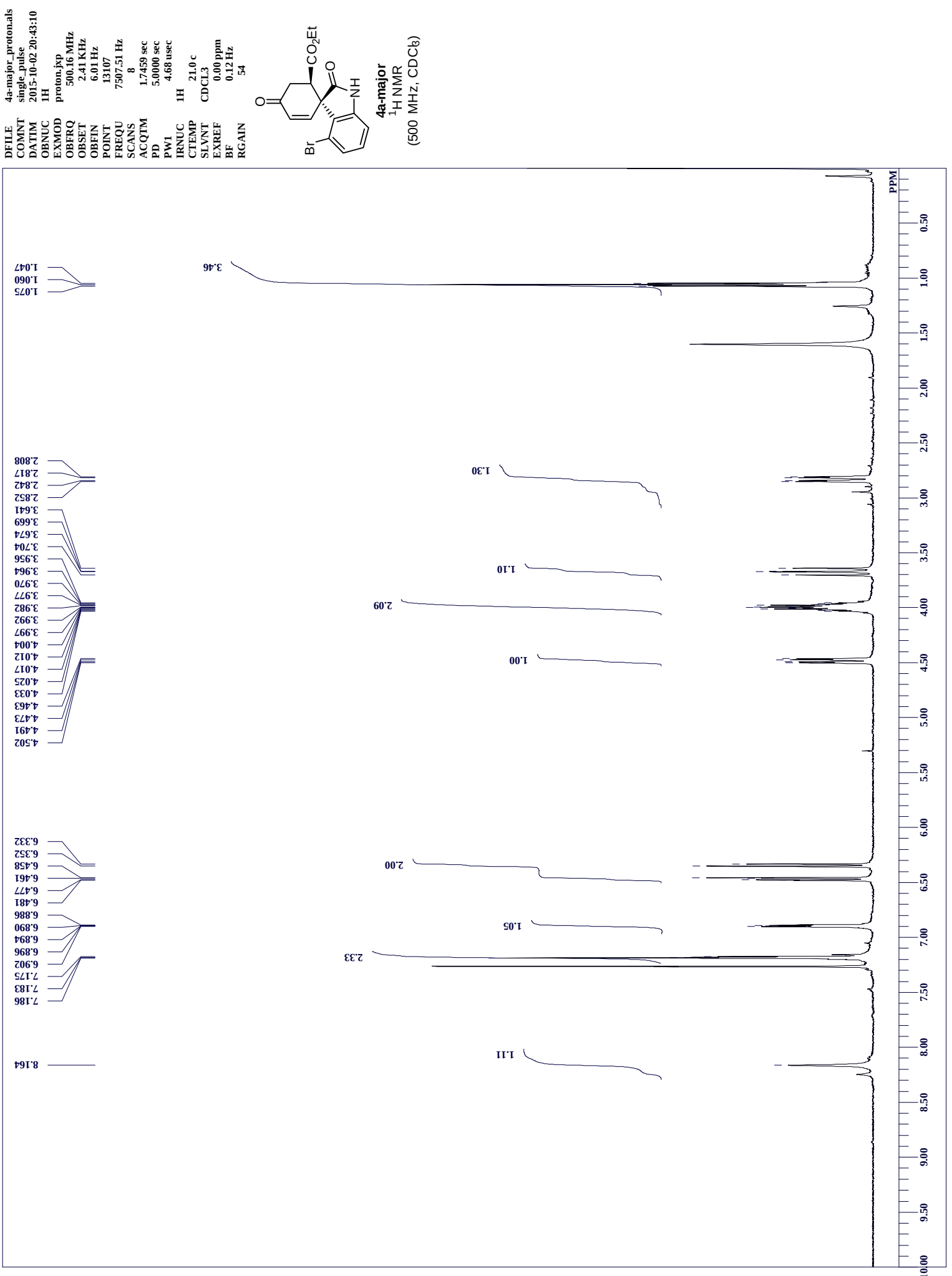
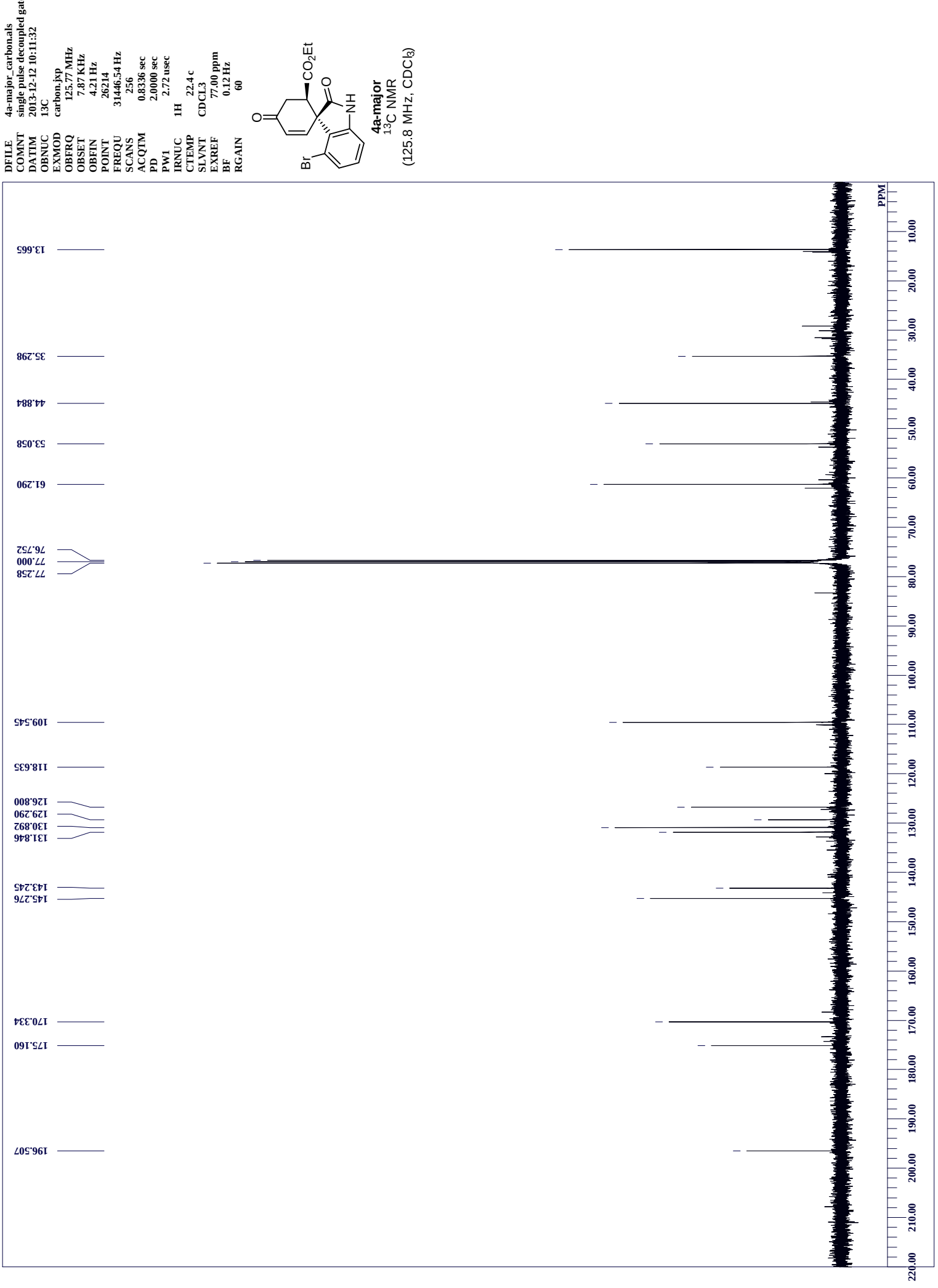
190.00
200.00
210.00
220.00


DFILE 2h_proton.als
COMNT single_pulse
DATIM 2012-06-25 18:25:08
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 4.68 usec
IRNUC 1H
CTEMP 20.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 58
EtO2C
Br
NH
O
2h
1H NMR
(500 MHz, CDCl3)
PPM
1.375
1.389
1.404
3.26
4.331
4.346
4.360
4.375
2.22
6.735
6.752
6.908
7.051
1.13
1.05
7.448
7.453
7.466
7.469
7.684
1.08
1.00
8.747
8.751
1.00


DFILE 2h_carbon.als
COMNT single pulse decoupled
DATIM 2012-06-29 17:14:13
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 1024
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF -0.02 ppm
BF 0.12 Hz
RGAIN 58
EtO2C
NH
O
Br
2h
13C NMR
(125 MHz, CDCl3)
14.151
61.529
76.743
77.000
77.248
111.272
115.526
122.031
124.225
131.913
135.051
136.825
141.862
165.241
168.073
0.00
10.00
20.00
30.00
40.00
50.00
60.00
70.00
80.00
90.00
100.00
110.00
120.00
130.00
140.00
150.00
160.00
170.00
180.00
190.00
200.00


DFILE 4a-major_proton.als
COMNT single_pulse
DATIM 2015-10-02 20:43:10
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 4.68 usec
IRNUC 1H
CTEMP 21.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 54
4a-major
1H NMR
(500 MHz, CDCl3)
CO2Et
O
NH
Br
PPM
1.047
1.060
1.075
3.46
2.808
2.817
2.842
2.852
3.641
3.669
3.674
3.704
3.956
3.964
3.970
3.977
3.982
3.992
3.997
4.004
4.012
4.017
4.025
4.033
4.463
4.473
4.491
4.502
6.332
6.352
6.458
6.461
6.477
6.481
6.886
6.890
6.894
6.896
6.902
7.175
7.183
7.186
8.164
1.30
1.10
2.09
1.00
2.00
1.05
2.33
1.11


DFILE 4a-major_carbon.als
COMNT single pulse decoupled gat
DATIM 2013-12-12 10:11:32
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 256
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 22.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60
CO2Et
O
NH
O
Br
4a-major
13C NMR
(125.8 MHz, CDCl3)
13.665
35.298
44.884
53.058
61.290
76.752
77.000
77.258
109.545
118.635
126.800
129.290
130.892
131.846
143.245
145.276
170.334
175.160
196.507
PPM
10.00
20.00
30.00
40.00
50.00
60.00
70.00
80.00
90.00
100.00
110.00
120.00
130.00
140.00
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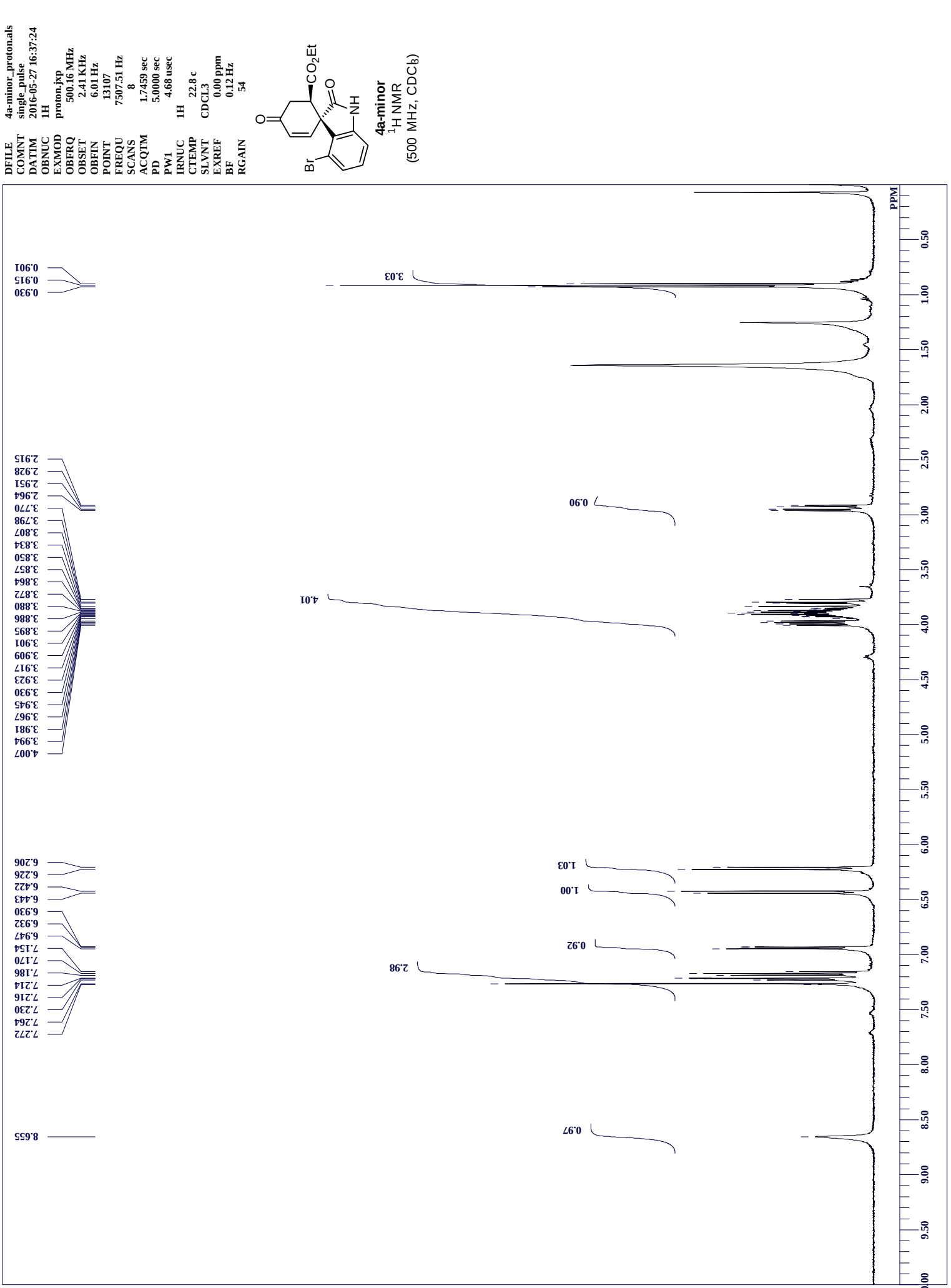
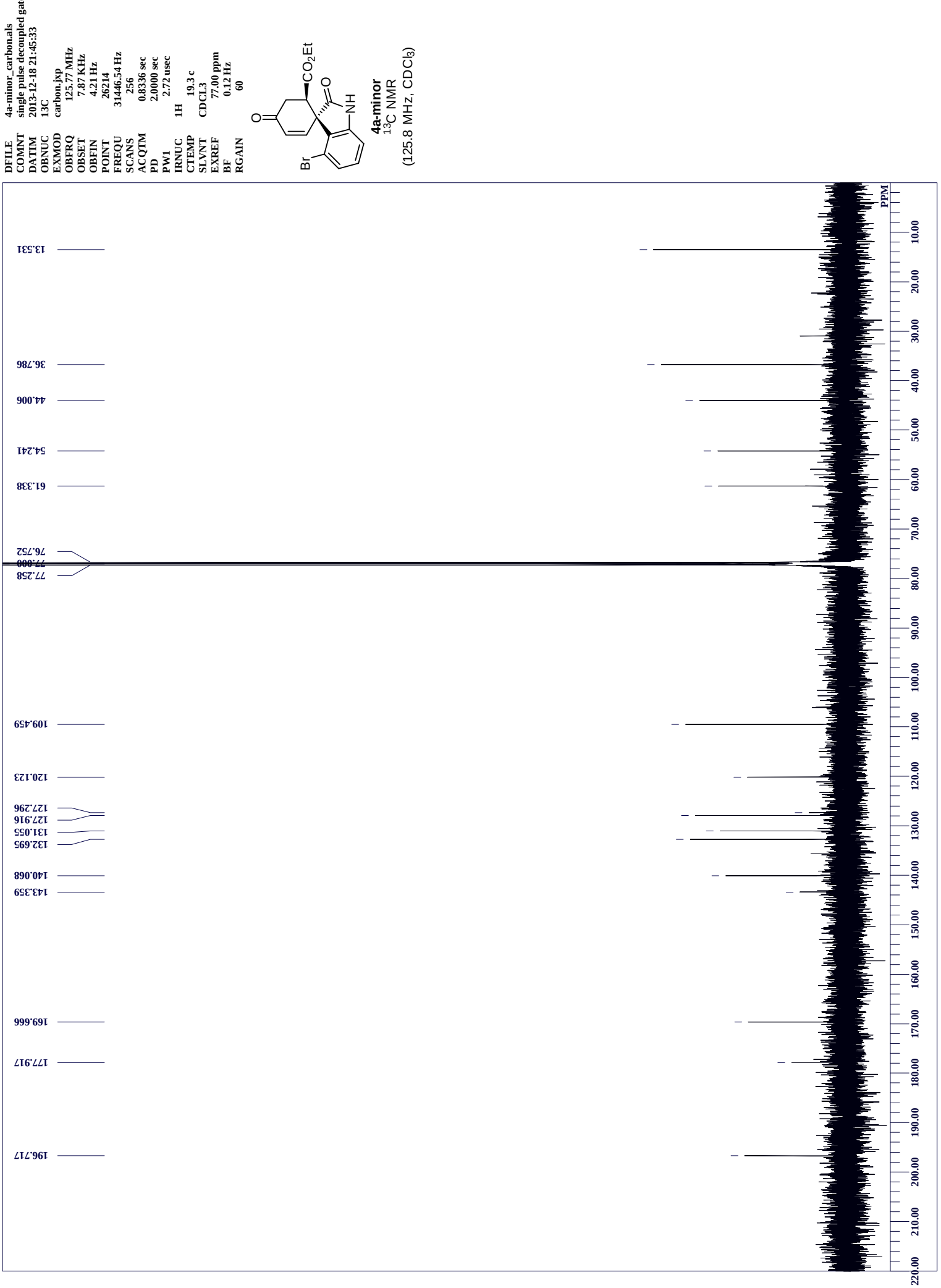
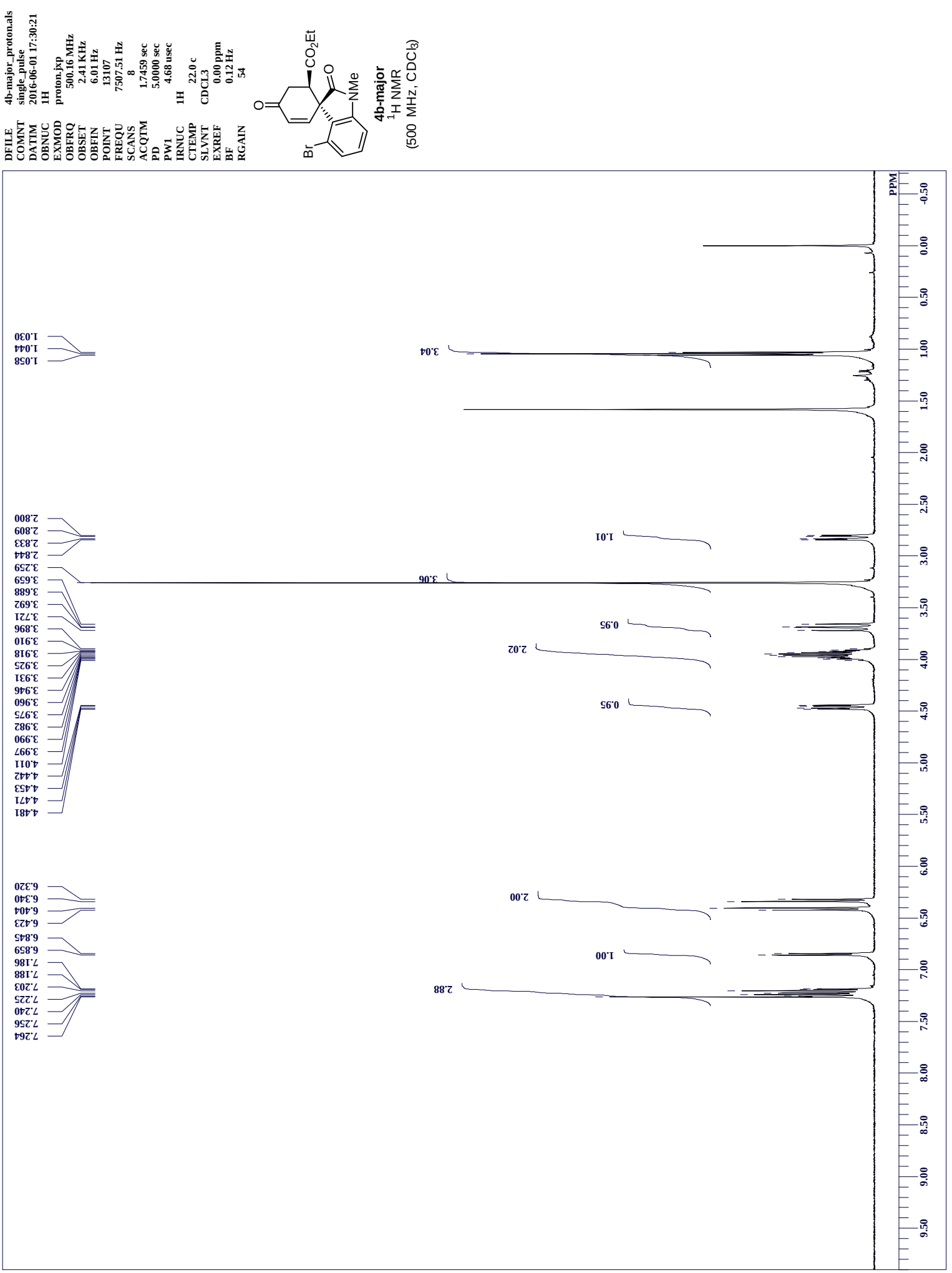
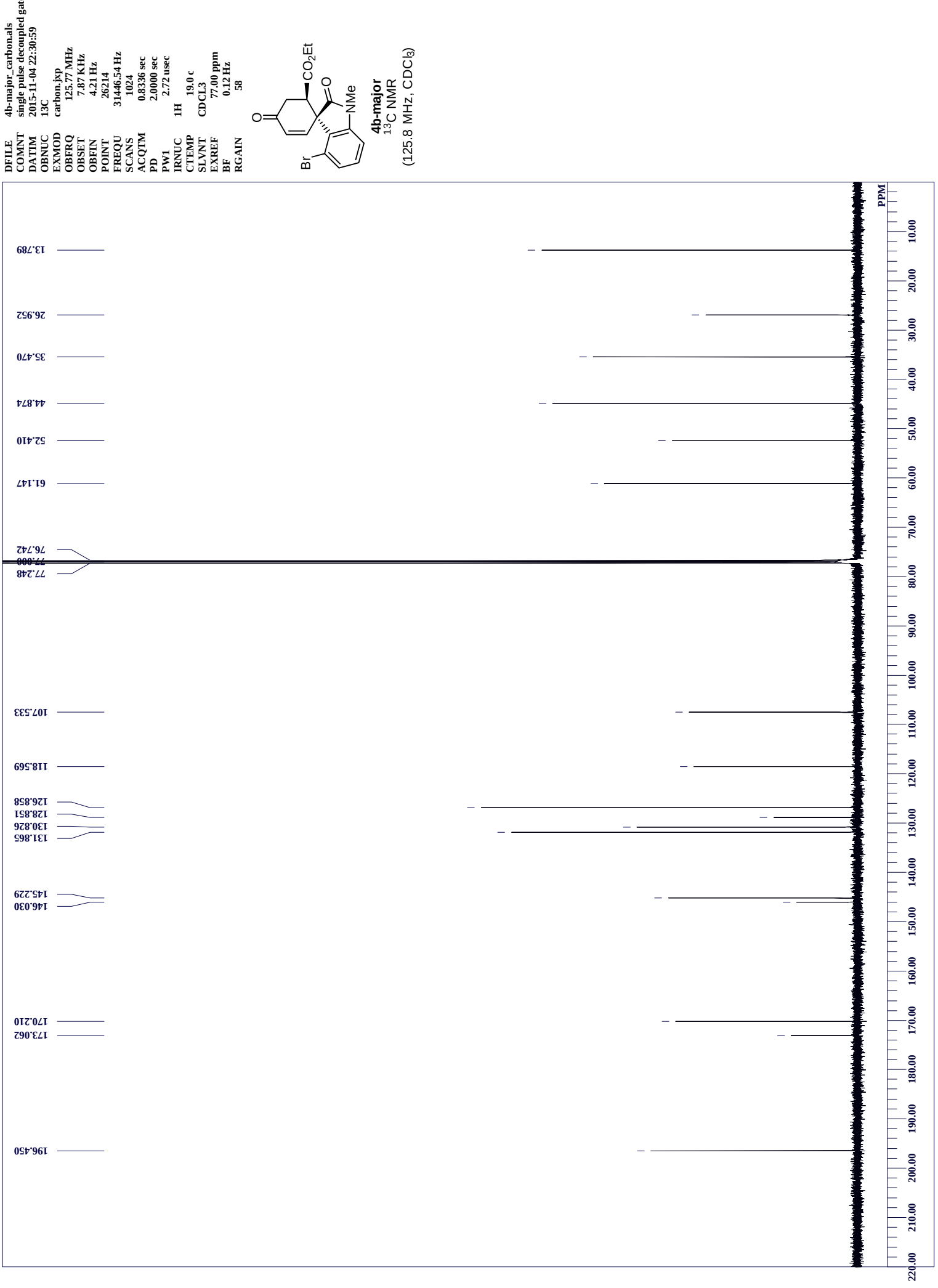
170.00
180.00
190.00
200.00
210.00
220.00


DFILE 4a-minor_proton.als
COMNT single_pulse
DATIM 2016-05-27 16:37:24
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 4.68 usec
IRNUC 1H
CTEMP 22.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 54
4a-minor
1H NMR
(500 MHz, CDCl3)
CO2Et
O
NH
O
Br
PPM
0.50
1.00
1.50
2.00
2.50
3.00
3.50
4.00
4.50
5.00
5.50
6.00
6.50
7.00
7.50
8.00
8.50
9.00
9.50
10.00
3.03
0.90
4.01
1.03
1.00
0.92
2.98
0.97
0.930
0.915
0.901
2.915
2.928
2.951
2.964
3.770
3.798
3.807
3.834
3.850
3.857
3.864
3.872
3.880
3.886
3.895
3.901
3.909
3.917
3.923
3.930
3.945
3.967
3.981
3.994
4.007
6.206
6.226
6.422
6.443
6.930
6.932
6.947
7.154
7.170
7.186
7.214
7.216
7.230
7.264
7.272
8.655


DFILE 4a-minor_carbon.als
COMNT single pulse decoupled gat
DATIM 2013-12-18 21:45:33
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 256
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 19.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60
CO2Et
O
O
NH
Br
4a-minor
13C NMR
(125.8 MHz, CDCl3)
13.531
36.786
44.006
54.241
61.338
76.752
77.000
77.258
109.459
120.123
127.296
127.916
131.055
132.695
140.068
143.359
169.666
177.917
196.717
PPM
10.00
20.00
30.00
40.00
50.00
60.00
70.00
80.00
90.00
100.00
110.00
120.00
130.00
140.00
150.00
160.00
170.00
180.00
190.00
200.00
210.00
220.00


DFILE 4b-major_proton.als
COMNT single_pulse
DATIM 2016-06-01 17:30:21
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 4.68 usec
IRNUC 1H
CTEMP 22.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 54
CO2Et
O
O
NMe
Br
4b-major
1H NMR
(500 MHz, CDCl3)
PPM
1.030
1.044
1.058
2.800
2.809
2.833
2.844
3.259
3.659
3.688
3.692
3.721
3.896
3.910
3.918
3.925
3.931
3.946
3.960
3.975
3.982
3.990
3.997
4.011
4.442
4.453
4.471
4.481
6.320
6.340
6.404
6.423
6.845
6.859
7.186
7.188
7.203
7.225
7.240
7.256
7.264
3.04
1.01
3.06
0.95
2.02
0.95
2.00
1.00
2.88


DFILE 4b-major_carbon.als
COMNT single pulse decoupled gat
DATIM 2015-11-04 22:30:59
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 1024
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 19.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 58
CO2Et
O
O
NMe
Br
4b-major
13C NMR
(125.8 MHz, CDCl3)
13.789
26.952
35.470
44.874
52.410
61.147
76.742
77.000
77.248
107.533
118.569
126.858
128.851
130.826
131.865
145.229
146.030
170.210
173.062
196.450
PPM
10.00
20.00
30.00
40.00
50.00
60.00
70.00
80.00
90.00
100.00
110.00
120.00
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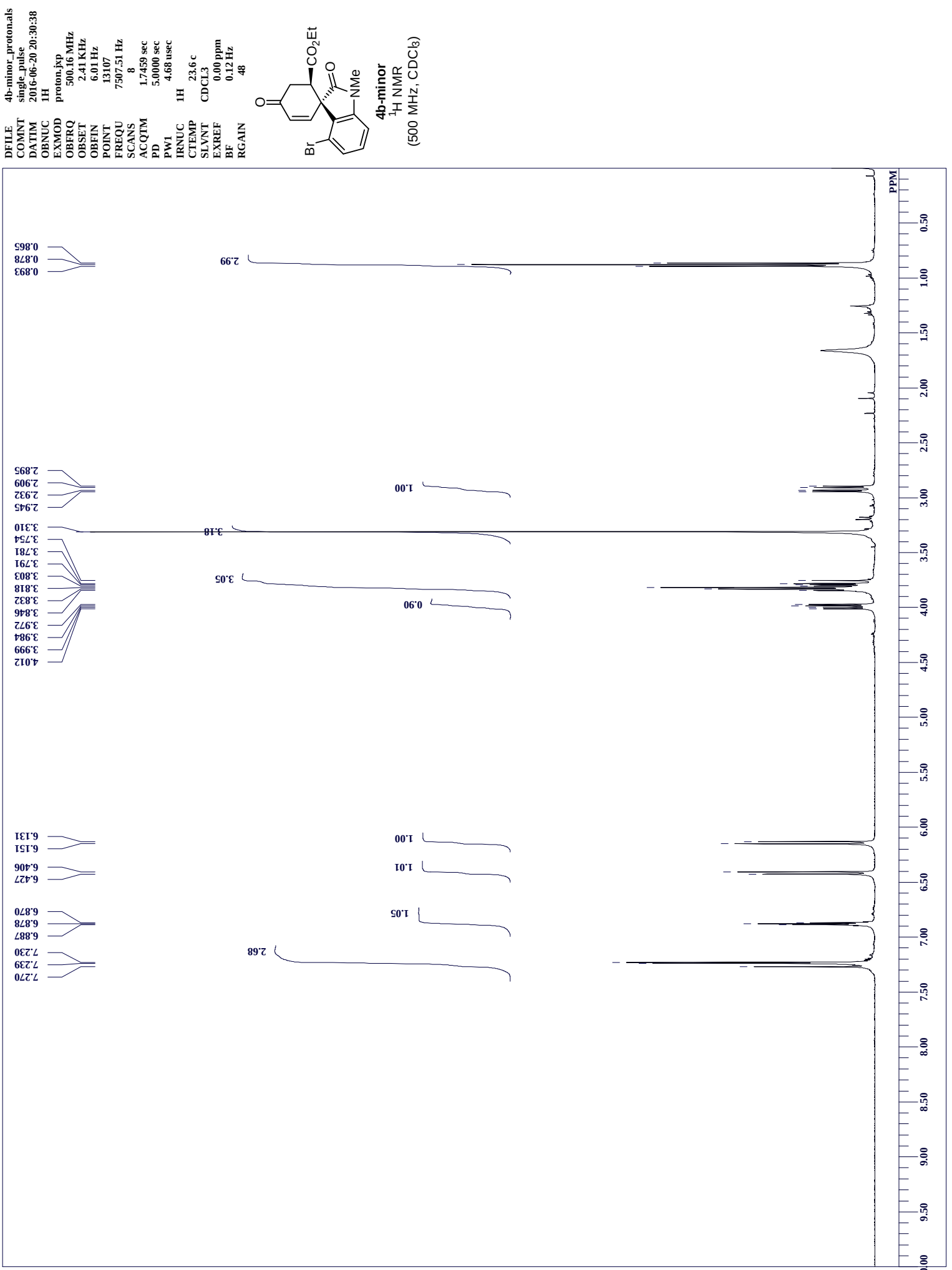
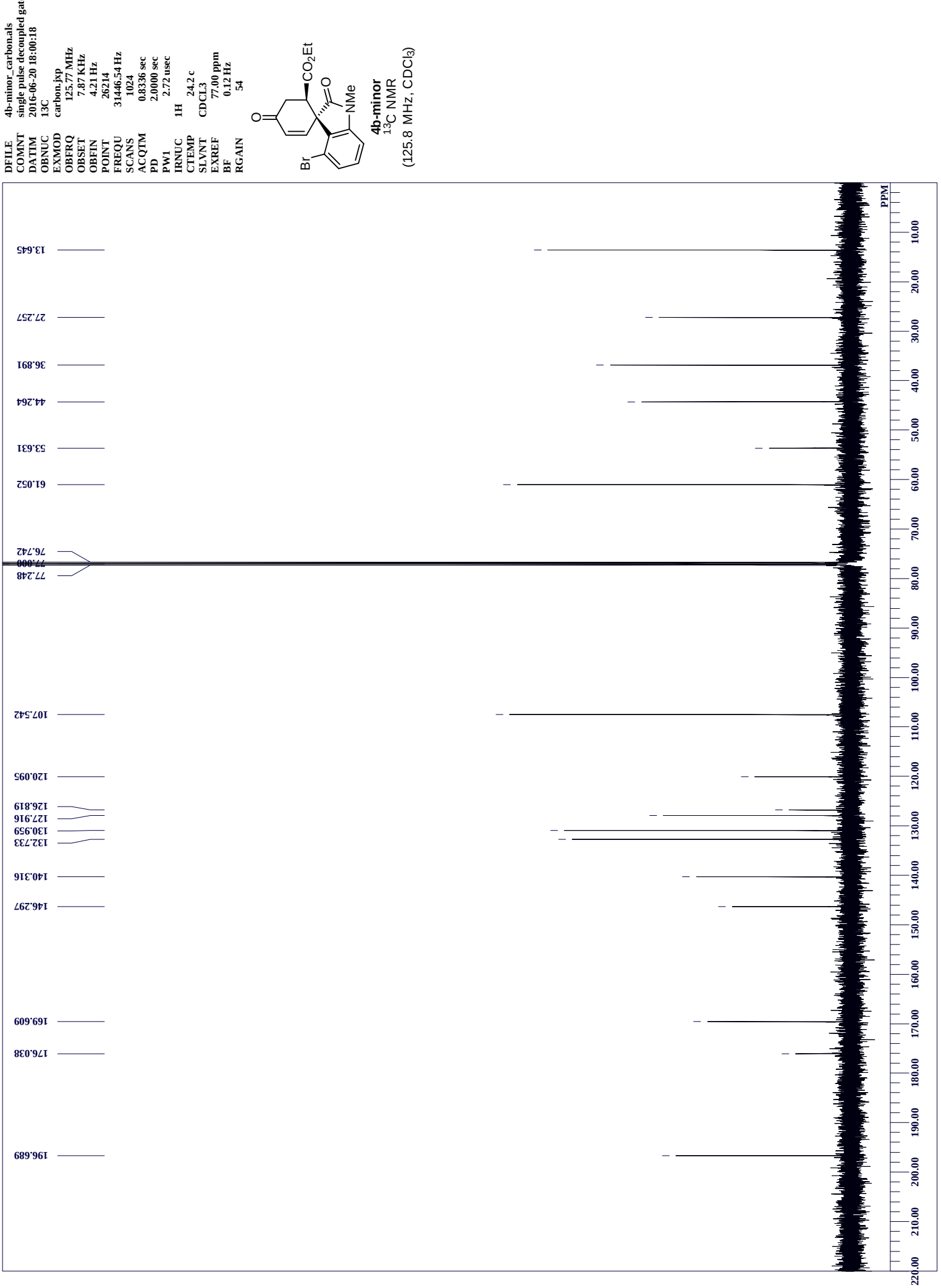
170.00
180.00
190.00
200.00
210.00
220.00


DFILE 4b-minor_proton.als
COMNT single_pulse
DATIM 2016-06-20 20:30:38
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 4.68 usec
IRNUC 1H
CTEMP 23.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 48
4b-minor
1H NMR
(500 MHz, CDCl3)
CO2Et
O
O
NMe
Br
PPM
0.865
0.878
0.893
2.99
2.895
2.909
2.932
2.945
3.310
3.754
3.781
3.791
3.803
3.818
3.832
3.846
3.972
3.984
3.999
4.012
3.18
1.00
3.05
0.90
6.131
6.151
6.406
6.427
6.870
6.878
6.887
7.230
7.239
7.270
1.00
1.01
1.05
2.68
0.50
1.00
1.50
2.00
2.50
3.00
3.50
4.00
4.50
5.00
5.50
6.00
6.50
7.00
7.50
8.00
8.50
9.00
9.50
10.00


DFILE 4b-minor_carbon.als
COMNT single pulse decoupled gat
DATIM 2016-06-20 18:00:18
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 1024
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 24.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 54
CO2Et
O
O
NMe
Br
4b-minor
13C NMR
(125.8 MHz, CDCl3)
13.645
27.257
36.891
44.264
53.631
61.052
76.742
77.000
77.248
107.542
120.095
126.819
127.916
130.959
132.733
140.316
146.297
169.609
176.038
196.689
PPM
10.00
20.00
30.00
40.00
50.00
60.00
70.00
80.00
90.00
100.00
110.00
120.00
130.00
140.00
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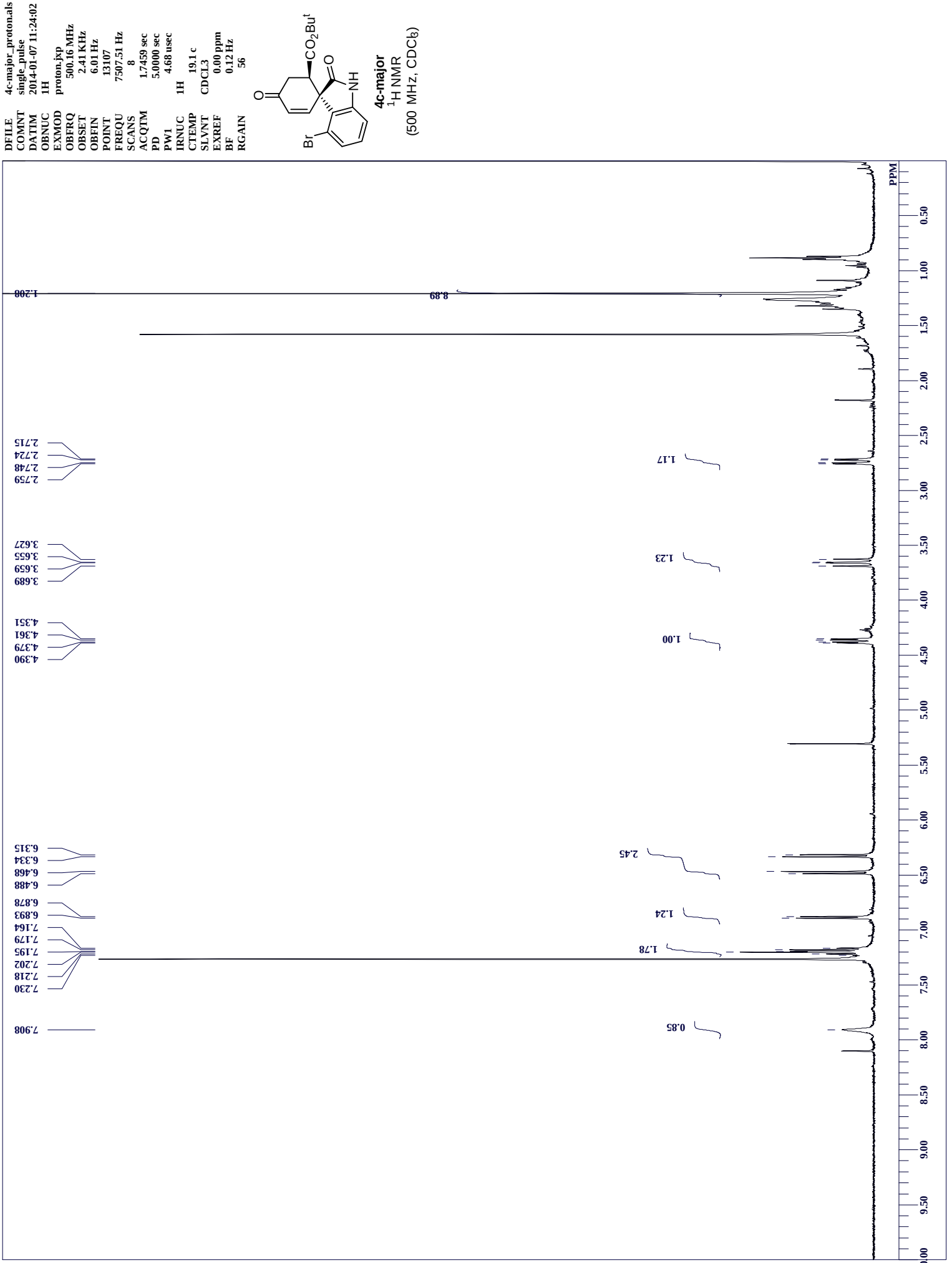
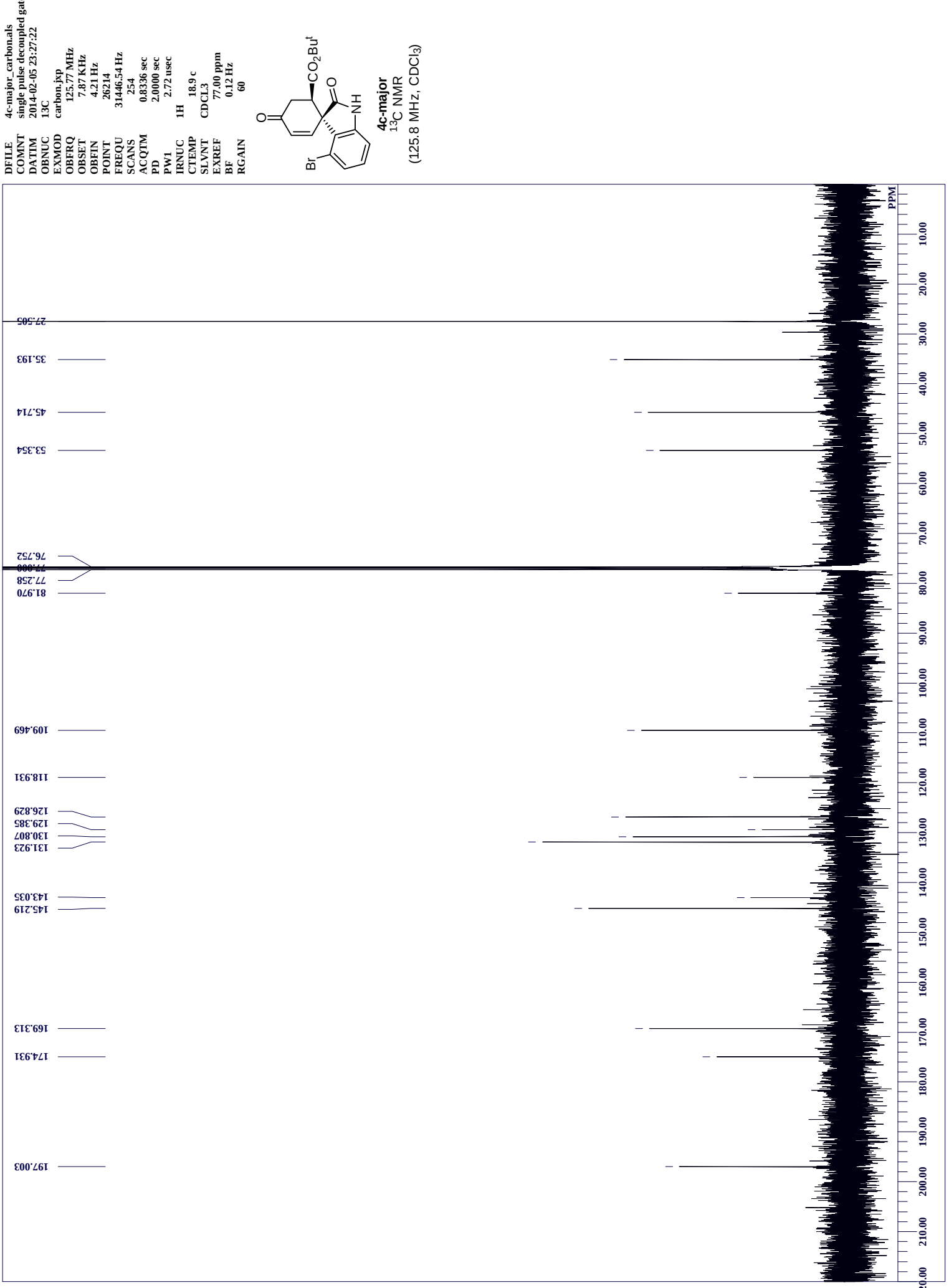
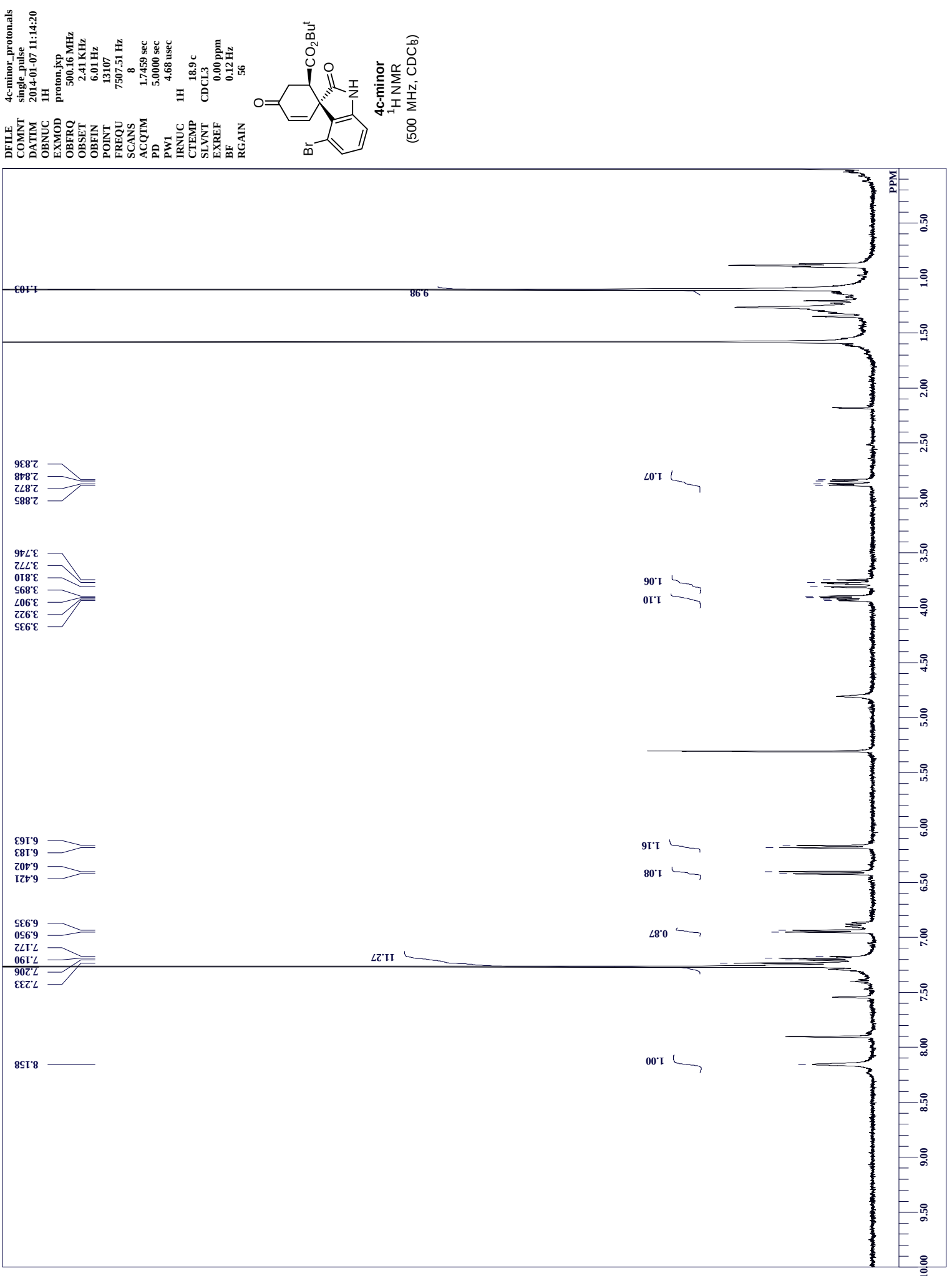
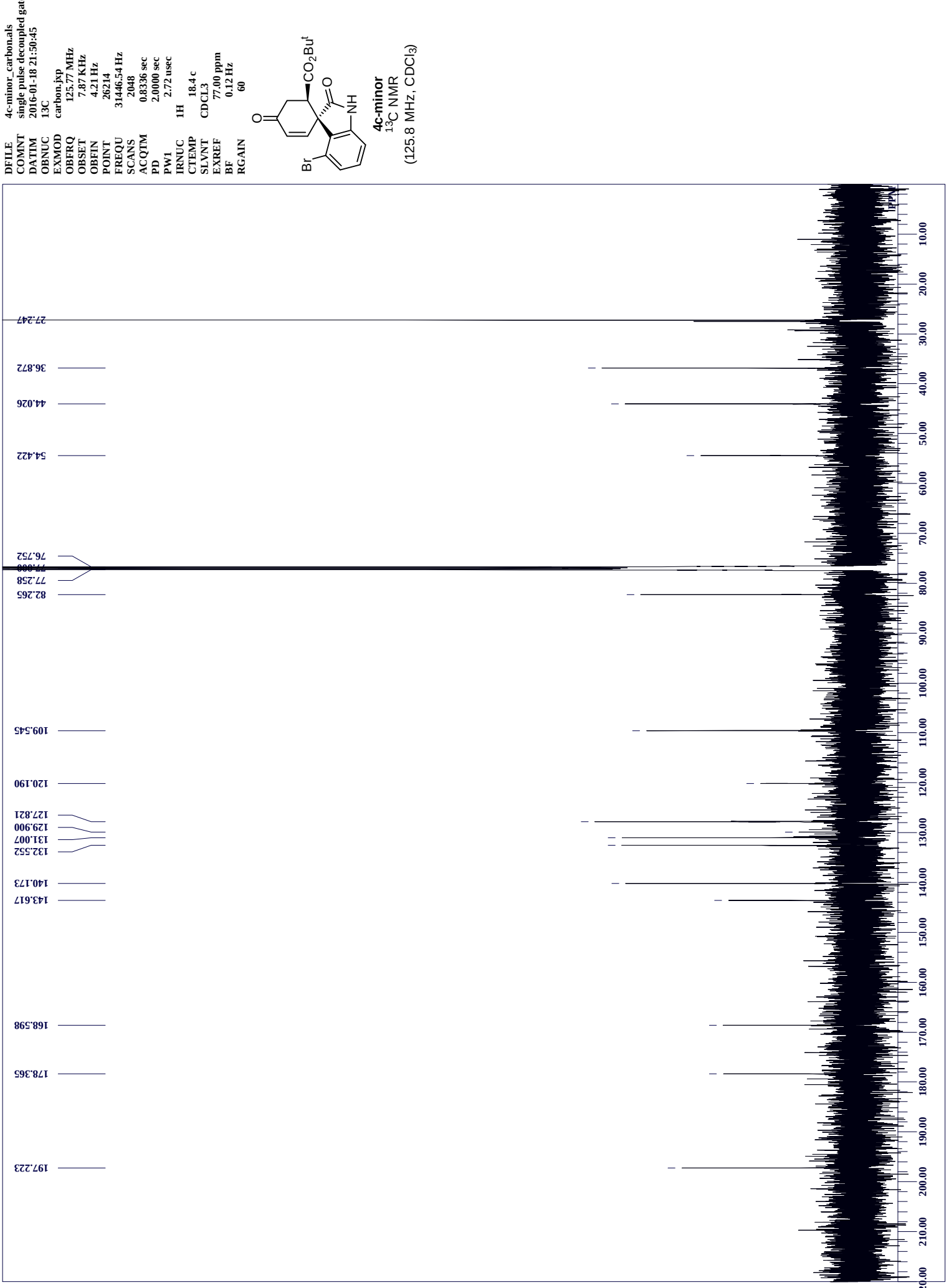
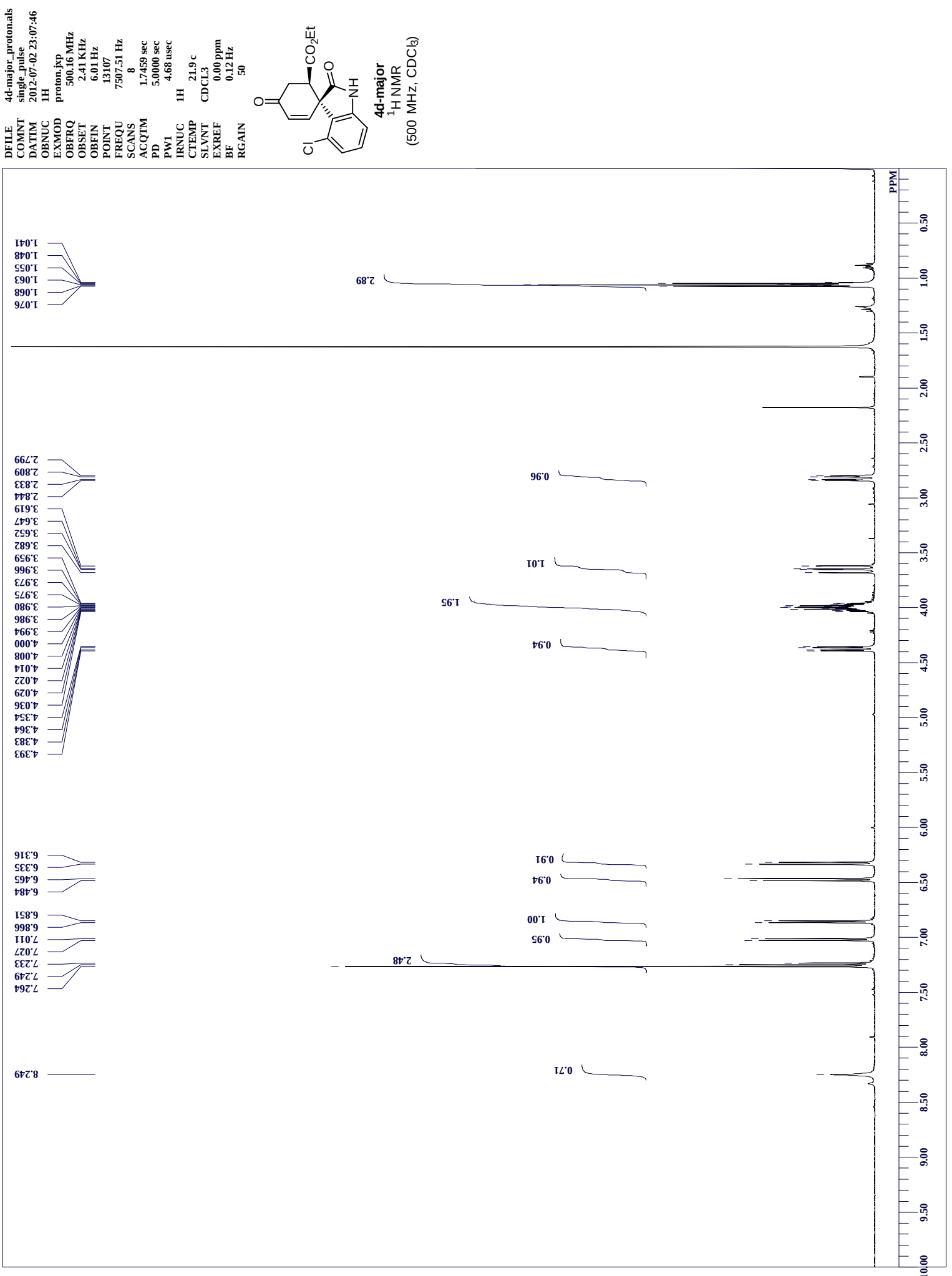
170.00
180.00
190.00
200.00
210.00
220.00

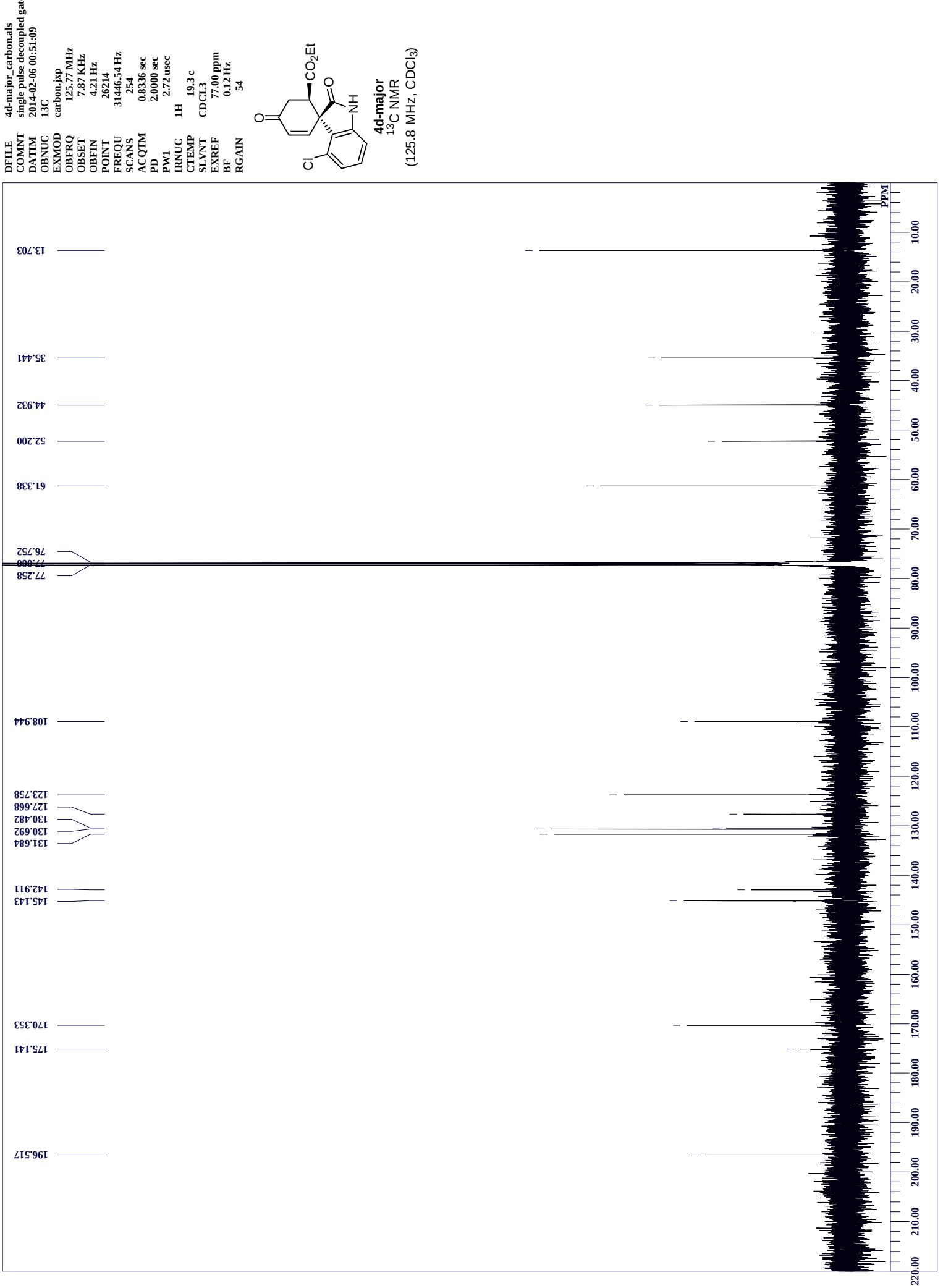

DFILE 4c-major_proton.als
COMNT single_pulse
DATIM 2014-01-07 11:24:02
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 4.68 usec
IRNUC 1H
CTEMP 19.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 56
4c-major
1H NMR
(500 MHz, CDCl3)
PPM
1.208
8.89
2.715
2.724
2.748
2.759
3.627
3.655
3.659
3.689
4.351
4.361
4.379
4.390
6.315
6.334
6.468
6.488
6.878
6.893
7.164
7.179
7.195
7.202
7.218
7.230
7.908
1.17
1.23
1.00
2.45
1.24
1.78
0.85

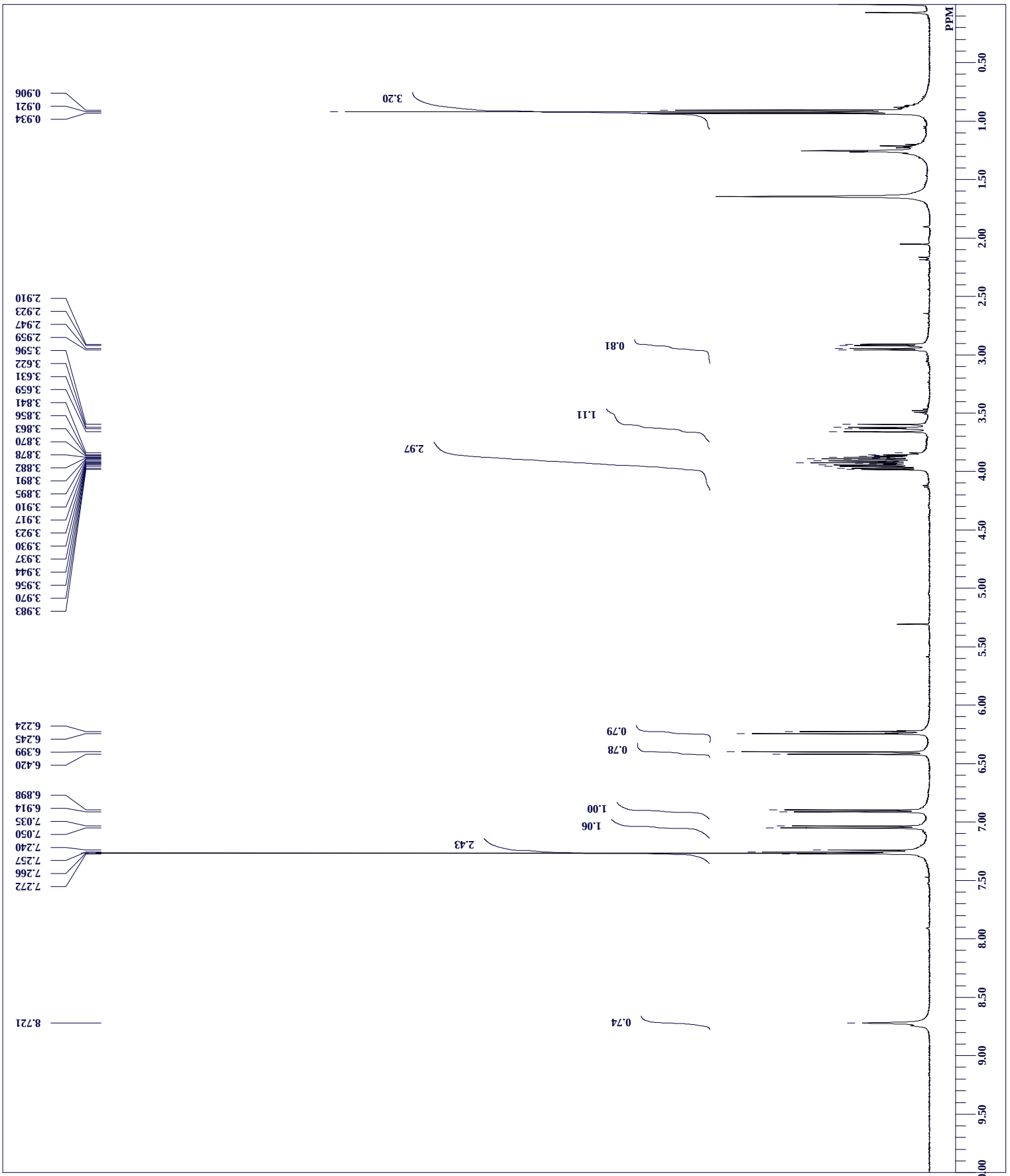

DFILE 4c-major_carbon.als
COMNT single pulse decoupled gat
DATIM 2014-02-05 23:27:22
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 254
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 18.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60
4c-major
13C NMR
(125.8 MHz, CDCl3)
27.505
35.193
45.714
53.354
76.752
77.000
77.258
81.970
109.469
118.931
126.829
129.385
130.807
131.923
143.035
145.219
169.313
174.931
197.003
PPM

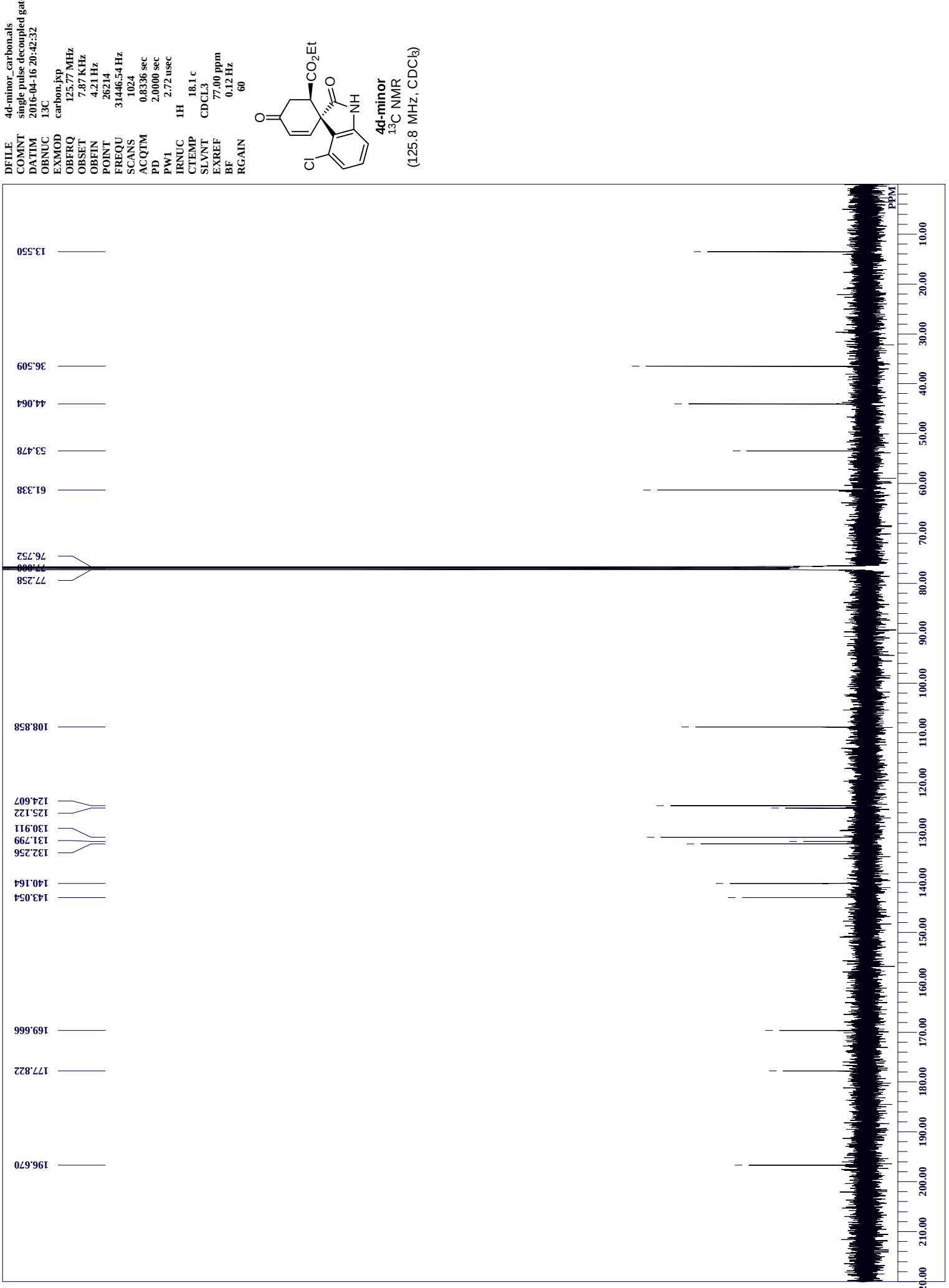

DFILE 4c-minor_proton.als
COMNT single_pulse
DATIM 2014-01-07 11:14:20
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 4.68 usec
IRNUC 1H
CTEMP 18.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 56
4c-minor
1H NMR
(500 MHz, CDCl3)
PPM
1.103
9.98
2.836
2.848
2.872
2.885
3.746
3.772
3.810
3.895
3.907
3.922
3.935
6.163
6.183
6.402
6.421
6.935
6.950
7.172
7.190
7.206
7.233
8.158
1.07
1.06
1.10
1.16
1.08
0.87
11.27
1.00


DFILE 4c-minor_carbon.als
COMNT single pulse decoupled gat
DATIM 2016-01-18 21:50:45
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 2048
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 18.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60
CO2But
O
NH
Br
O
4c-minor
13C NMR
(125.8 MHz, CDCl3)
27.247
36.872
44.026
54.422
76.752
77.008
77.258
82.265
109.545
120.190
127.821
129.900
131.007
132.552
140.173
143.617
168.598
178.365
197.223
ppm
10.00
20.00
30.00
40.00
50.00
60.00
70.00
80.00
90.00
100.00
110.00
120.00
130.00
140.00
150.00
160.00
170.00
180.00
190.00
200.00
210.00
220.00


DFILE 4d-major_proton.als
COMNT single_pulse
DATIM 2012-07-02 23:07:46
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 4.68 usec
IRNUC 1H
CTEMP 21.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 50
4d-major
1H NMR
(500 MHz, CDCl3)
CO2Et
O
NH
O
Cl
PPM
0.50
1.00
1.50
2.00
2.50
3.00
3.50
4.00
4.50
5.00
5.50
6.00
6.50
7.00
7.50
8.00
8.50
9.00
9.50
10.00
1.041
1.048
1.055
1.063
1.068
1.076
2.89
2.799
2.809
2.833
2.844
3.619
3.647
3.652
3.682
3.959
3.966
3.973
3.975
3.980
3.986
3.994
4.000
4.008
4.014
4.022
4.029
4.036
4.354
4.364
4.383
4.393
6.316
6.335
6.465
6.484
6.851
6.866
7.011
7.027
7.233
7.249
7.264
8.249
0.96
1.01
1.95
0.94
0.91
0.94
1.00
0.95
2.48
0.71


DFILE 4d-major_carbon.als
COMNT single pulse decoupled gat
DATIM 2014-02-06 00:51:09
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 254
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 19.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 54
4d-major
13C NMR
(125.8 MHz, CDCl3)
13.703
35.441
44.932
52.200
61.338
76.752
77.000
77.258
108.944
123.758
127.668
130.482
130.692
131.684
142.911
145.143
170.353
175.141
196.517
PPM


DFILE 4d-minor_proton.als
COMNT single_pulse
DATIM 2016-04-16 20:40:37
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 4.68 usec
IRNUC 1H
CTEMP 17.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 50
4d-minor
1H NMR
(500 MHz, CDCl3)
CO2Et
O
NH
Cl
PPM
0.74
2.43
1.06
1.00
0.78
0.79
0.81
1.11
2.97
3.20
8.721
7.272
7.266
7.257
7.240
7.050
7.035
6.914
6.898
6.420
6.399
6.245
6.224
3.983
3.970
3.956
3.944
3.937
3.930
3.923
3.917
3.910
3.895
3.891
3.882
3.878
3.870
3.863
3.856
3.841
3.659
3.631
3.622
3.596
2.959
2.947
2.923
2.910
0.934
0.921
0.906


DFILE 4d-minor_carbon.als
COMNT single pulse decoupled gat
DATIM 2016-04-16 20:42:32
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 1024
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 18.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60
CO2Et
O
O
NH
Cl
4d-minor
13C NMR
(125.8 MHz, CDCl3)
13.550
36.509
44.064
53.478
61.338
76.752
77.000
77.258
108.858
124.607
125.122
130.911
131.799
132.256
140.164
143.054
169.666
177.822
196.670
PPM
10.00
20.00
30.00
40.00
50.00
60.00
70.00
80.00
90.00
100.00
110.00
120.00
130.00
140.00
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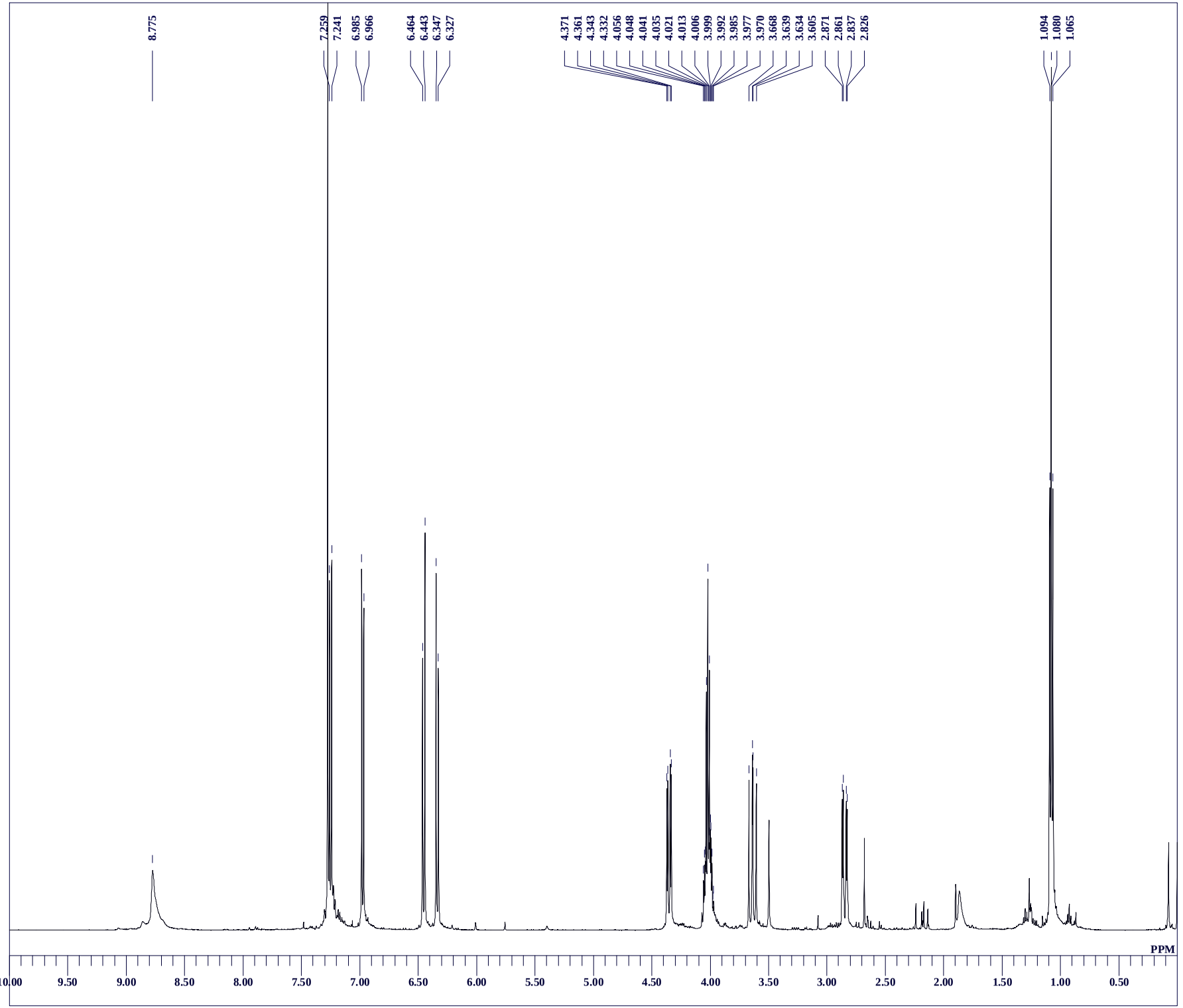
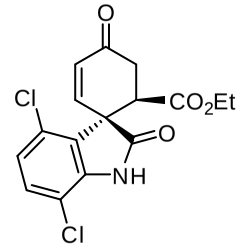
160.00
170.00
180.00
190.00
200.00
210.00
220.00



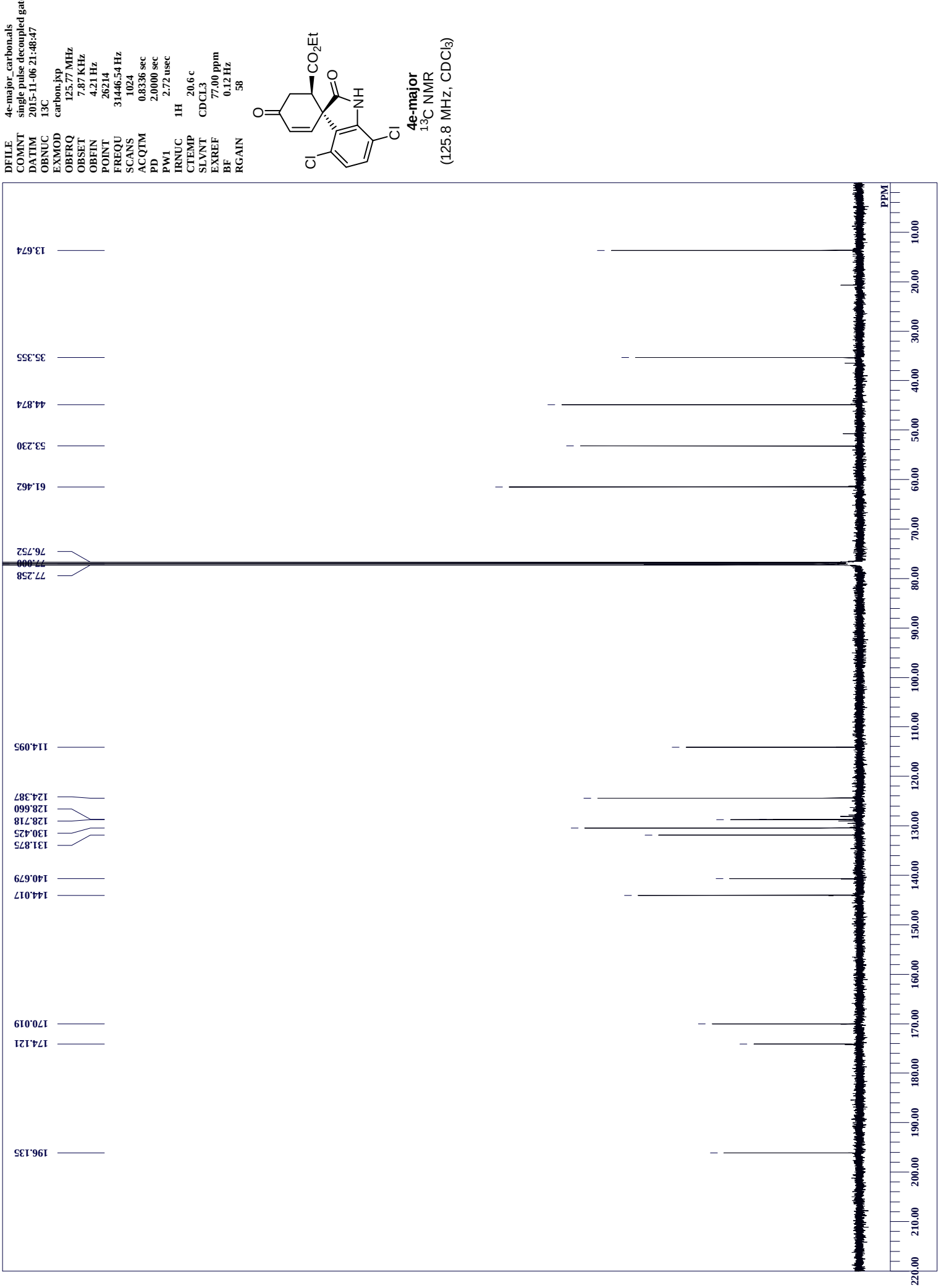
DFILE	4e-major_proton.als
COMNT	single_pulse
DATIM	2015-11-06 15:35:41
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	4.68 usec
IRNUC	1H
CTEMP	19.6 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	40

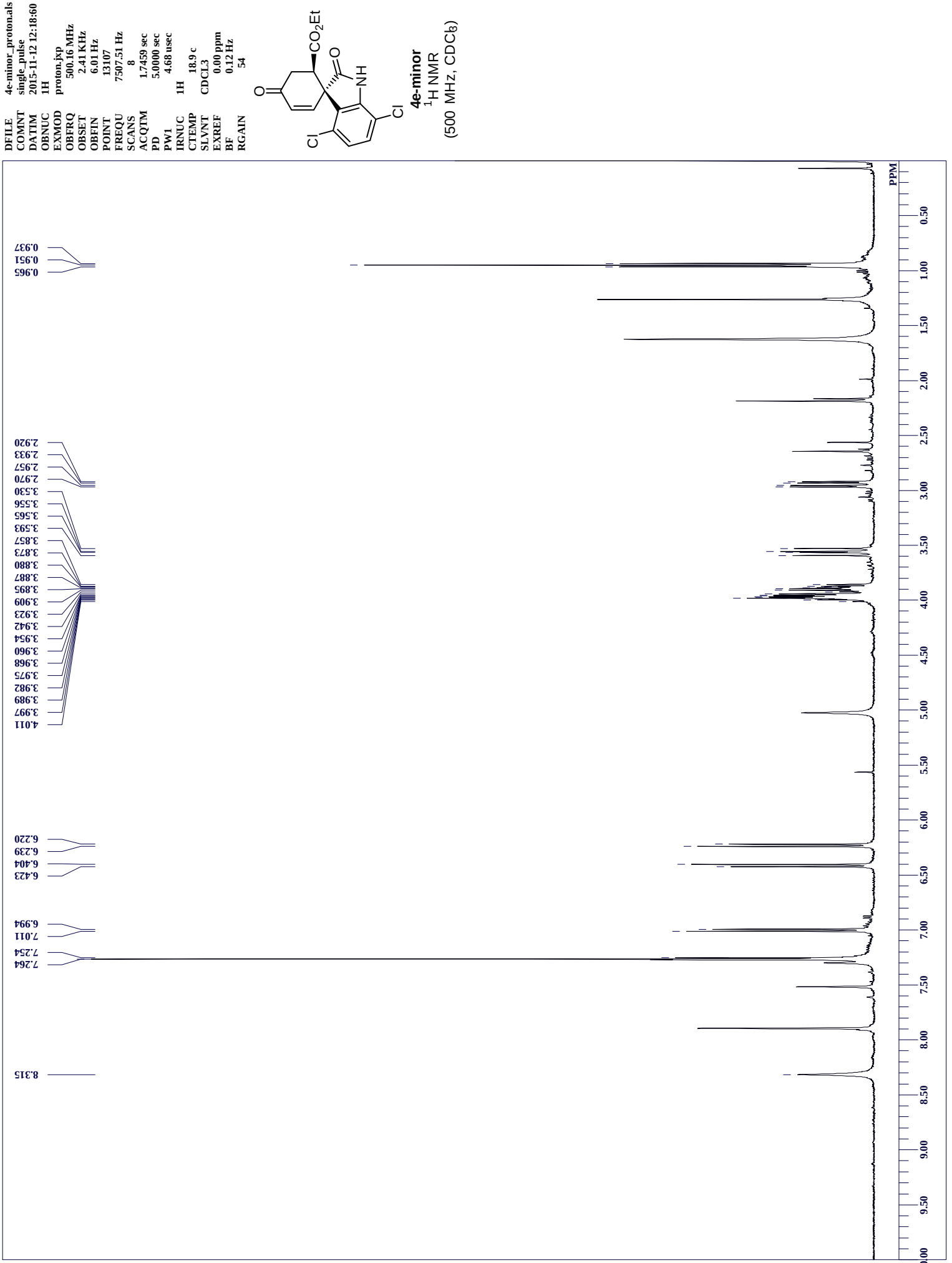


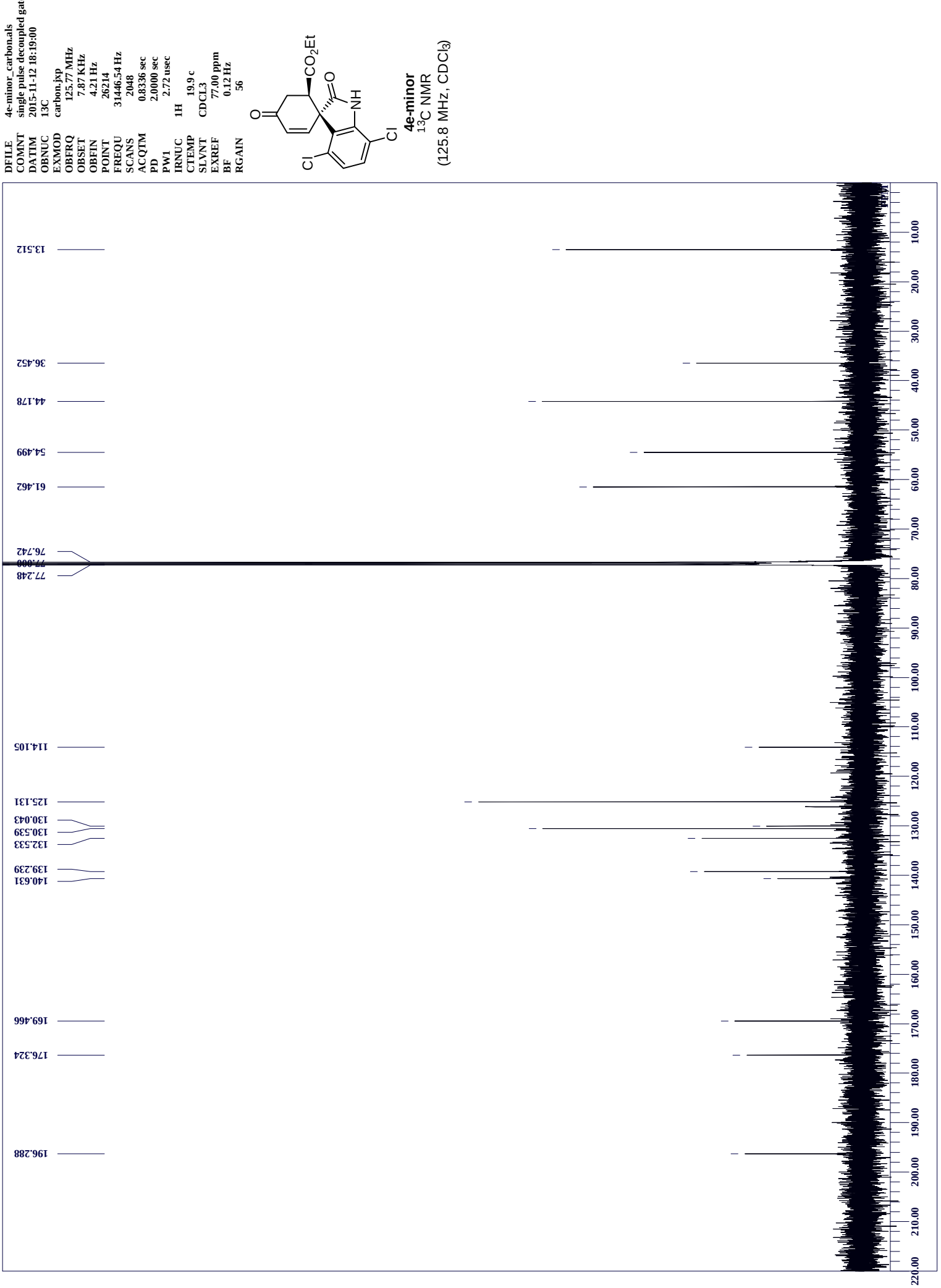
4e-major

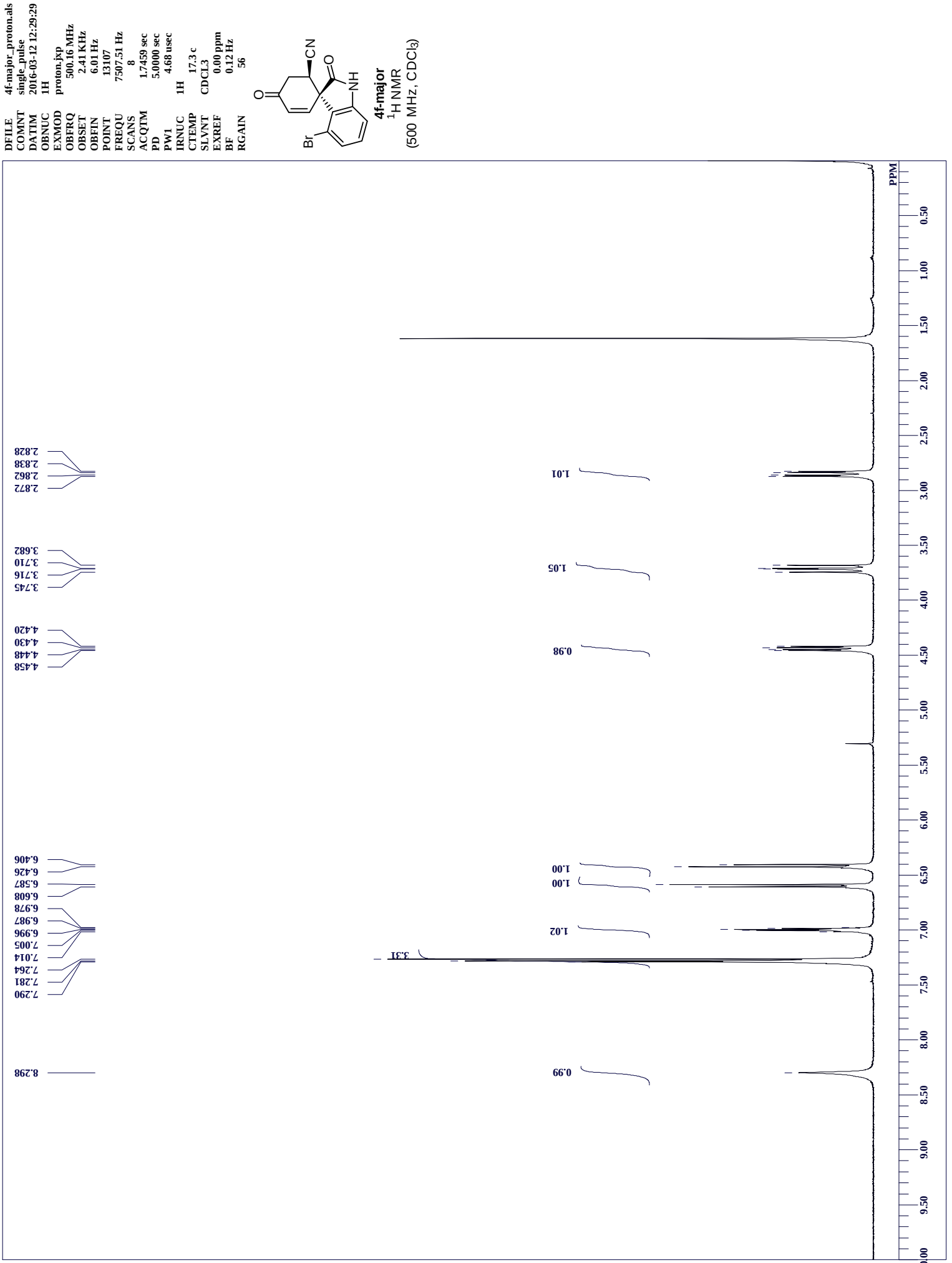
^{1}H NMR

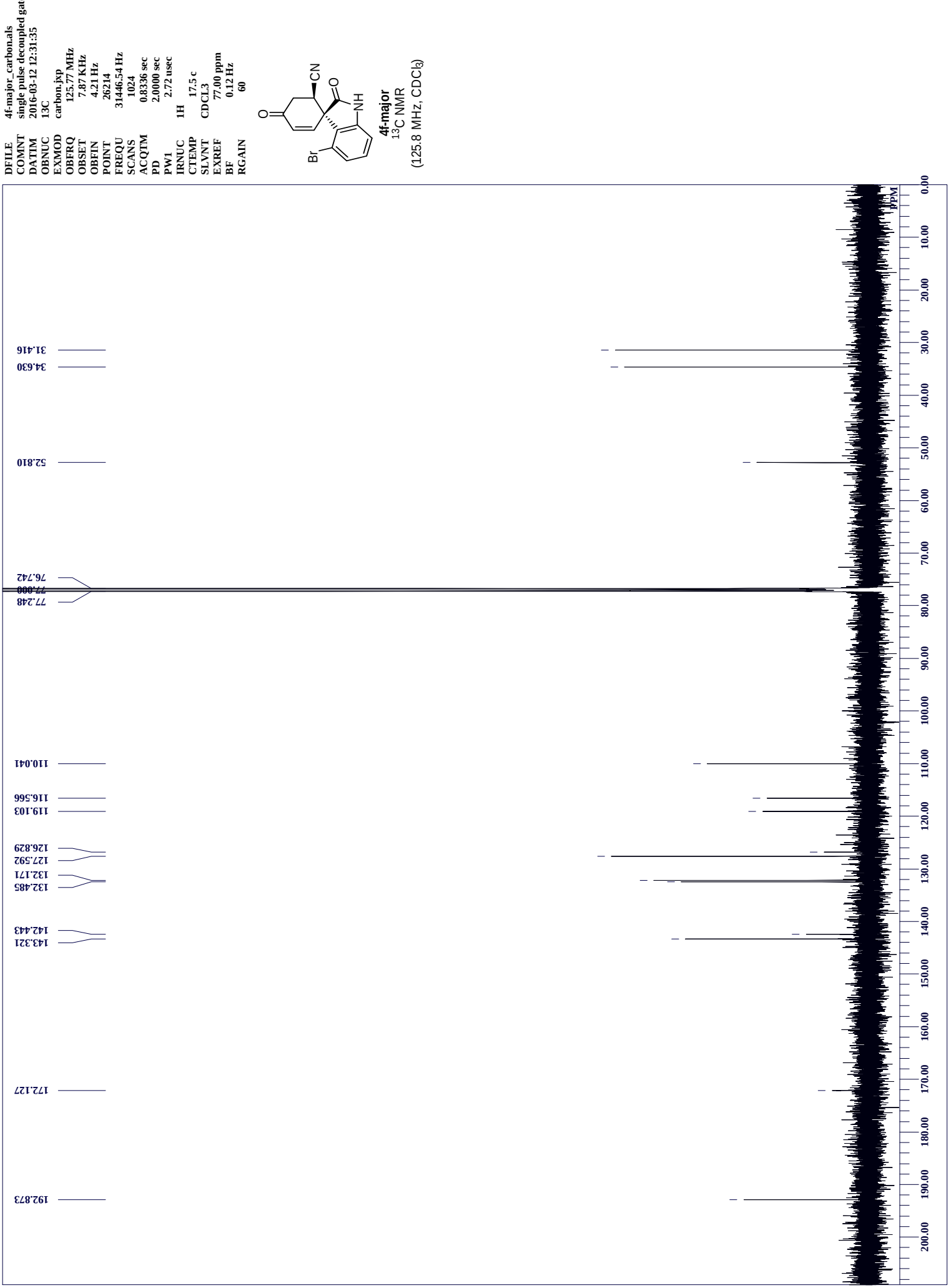
(500 MHz, $CDCl_3$)

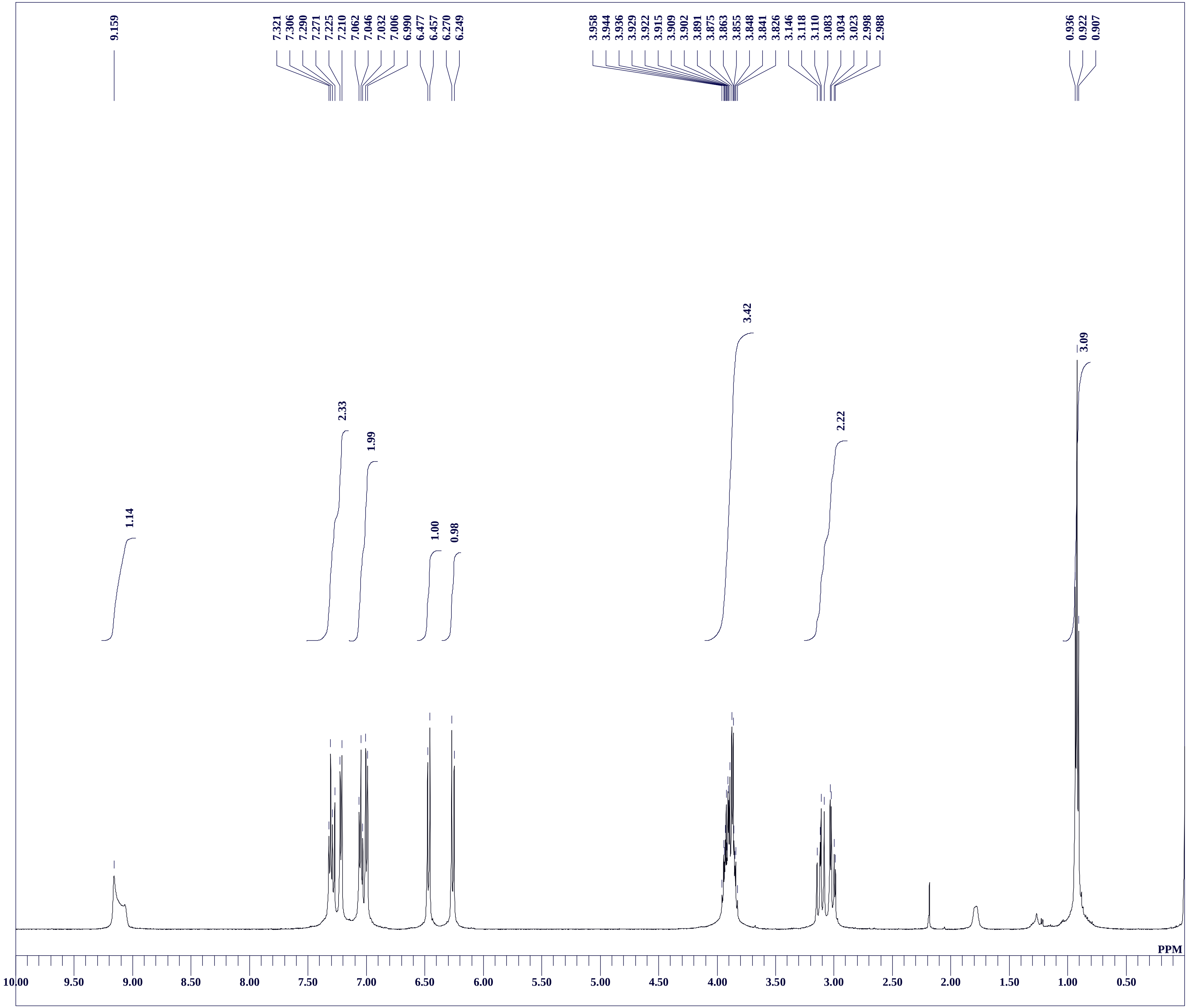

DFILE 4e-major_carbon.als
COMNT single pulse decoupled gat
DATIM 2015-11-06 21:48:47
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 1024
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 20.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 58
CO2Et
O
O
NH
Cl
Cl
4e-major
13C NMR
(125.8 MHz, CDCl3)
13.674
35.355
44.874
53.230
61.462
76.752
77.000
77.258
114.095
124.387
128.660
128.718
130.425
131.875
140.679
144.017
170.019
174.121
196.135
PPM
10.00
20.00
30.00
40.00
50.00
60.00
70.00
80.00
90.00
100.00
110.00
120.00
130.00
140.00
150.00
160.00
170.00
180.00
190.00
200.00
210.00
220.00


DFILE 4e-minor_proton.als
COMNT single_pulse
DATIM 2015-11-12 12:18:60
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 4.68 usec
IRNUC 1H
CTEMP 18.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 54
CO2Et
O
NH
O
Cl
Cl
4e-minor
1H NMR
(500 MHz, CDCl3)
0.937
0.951
0.965
2.920
2.933
2.957
2.970
3.530
3.556
3.565
3.593
3.857
3.873
3.880
3.887
3.895
3.909
3.923
3.942
3.954
3.960
3.968
3.975
3.982
3.989
3.997
4.011
6.220
6.239
6.404
6.423
6.994
7.011
7.254
7.264
8.315
PPM
0.50
1.00
1.50
2.00
2.50
3.00
3.50
4.00
4.50
5.00
5.50
6.00
6.50
7.00
7.50
8.00
8.50
9.00
9.50
10.00

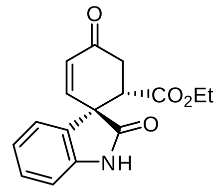

DFILE 4e-minor_carbon.als
COMNT single pulse decoupled gat
DATIM 2015-11-12 18:19:00
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 2048
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 19.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 56
CO2Et
O
NH
Cl
Cl
4e-minor
13C NMR
(125.8 MHz, CDCl3)
13.512
36.452
44.178
54.499
61.462
76.742
77.000
77.248
114.105
125.131
130.043
130.539
132.533
139.239
140.631
169.466
176.324
196.288
10.00
20.00
30.00
40.00
50.00
60.00
70.00
80.00
90.00
100.00
110.00
120.00
130.00
140.00
150.00
160.00
170.00
180.00
190.00
200.00
210.00
220.00


DFILE 4f-major_proton.als
COMNT single_pulse
DATIM 2016-03-12 12:29:29
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 4.68 usec
IRNUC 1H
CTEMP 17.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 56
4f-major
1H NMR
(500 MHz, CDCl3)
PPM
2.828
2.838
2.862
2.872
3.682
3.710
3.716
3.745
4.420
4.430
4.448
4.458
6.406
6.426
6.587
6.608
6.978
6.987
6.996
7.005
7.014
7.264
7.281
7.290
8.298
1.01
1.05
0.98
1.00
1.00
1.02
3.31
0.99

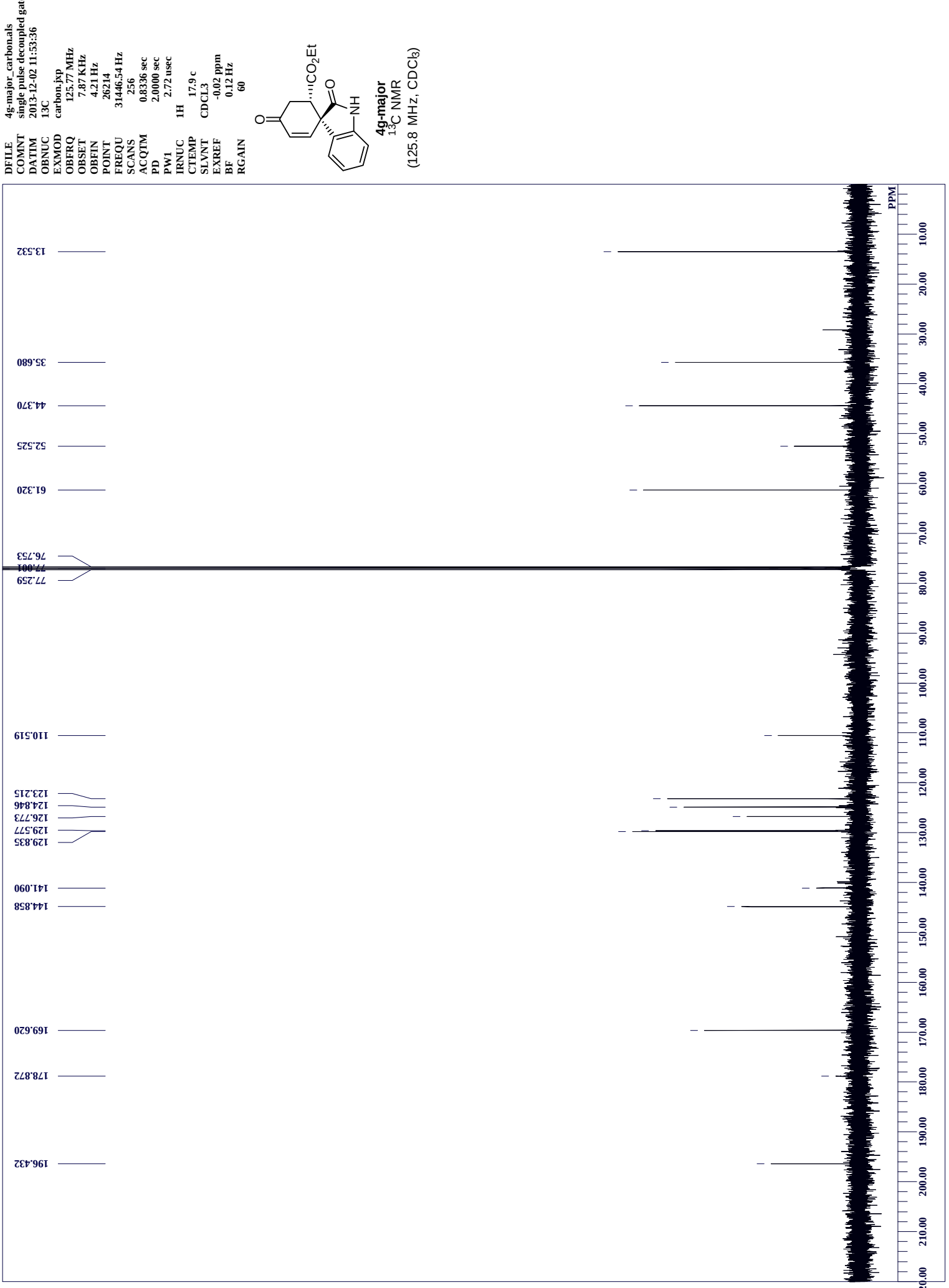

DFILE 4f-major_carbon.als
COMNT single pulse decoupled gat
DATIM 2016-03-12 12:31:35
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 1024
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 17.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60
CN
O
NH
O
Br
4f-major
13C NMR
(125.8 MHz, CDCl3)
31.416
34.630
52.810
76.742
77.000
77.248
110.041
116.566
119.103
126.829
127.592
132.171
132.485
142.443
143.321
172.127
192.873
PPM
0.00
10.00
20.00
30.00
40.00
50.00
60.00
70.00
80.00
90.00
100.00
110.00
120.00
130.00
140.00
150.00
160.00
170.00
180.00
190.00
200.00

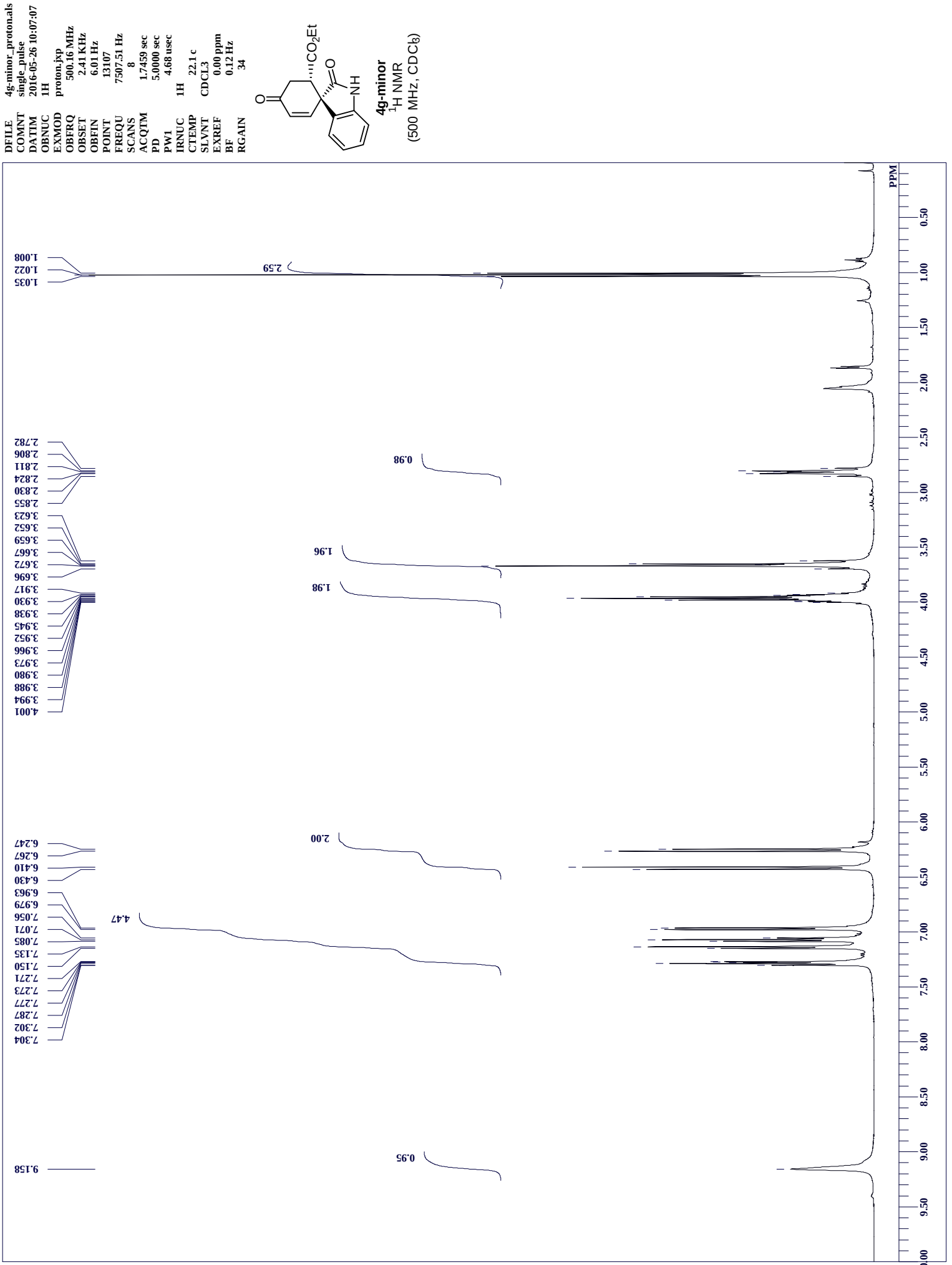


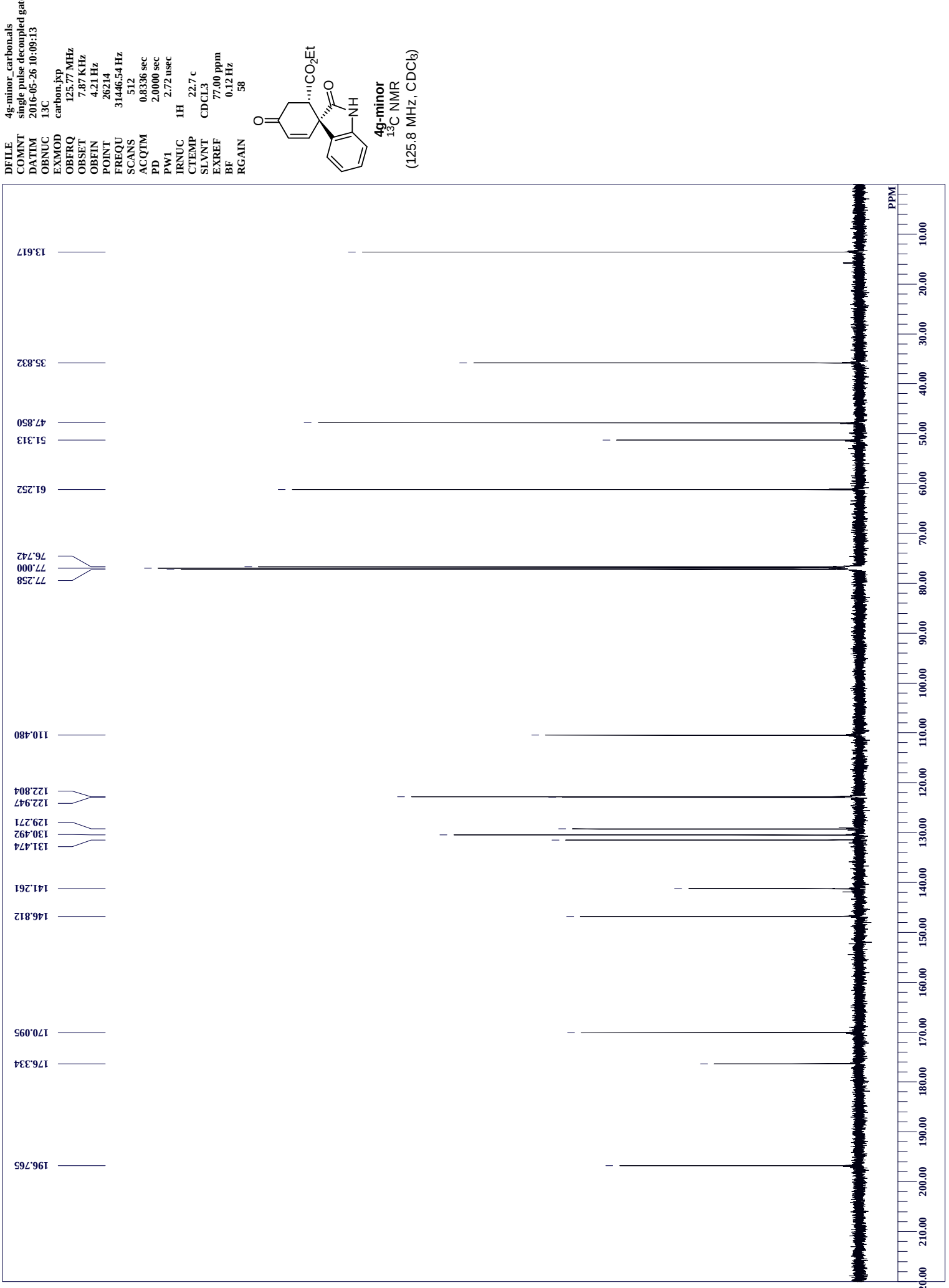
DFILE	4g-major_proton.als
COMNT	single_pulse
DATIM	2014-03-12 19:05:02
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	4.68 usec
IRNUC	1H
CTEMP	18.9 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	40

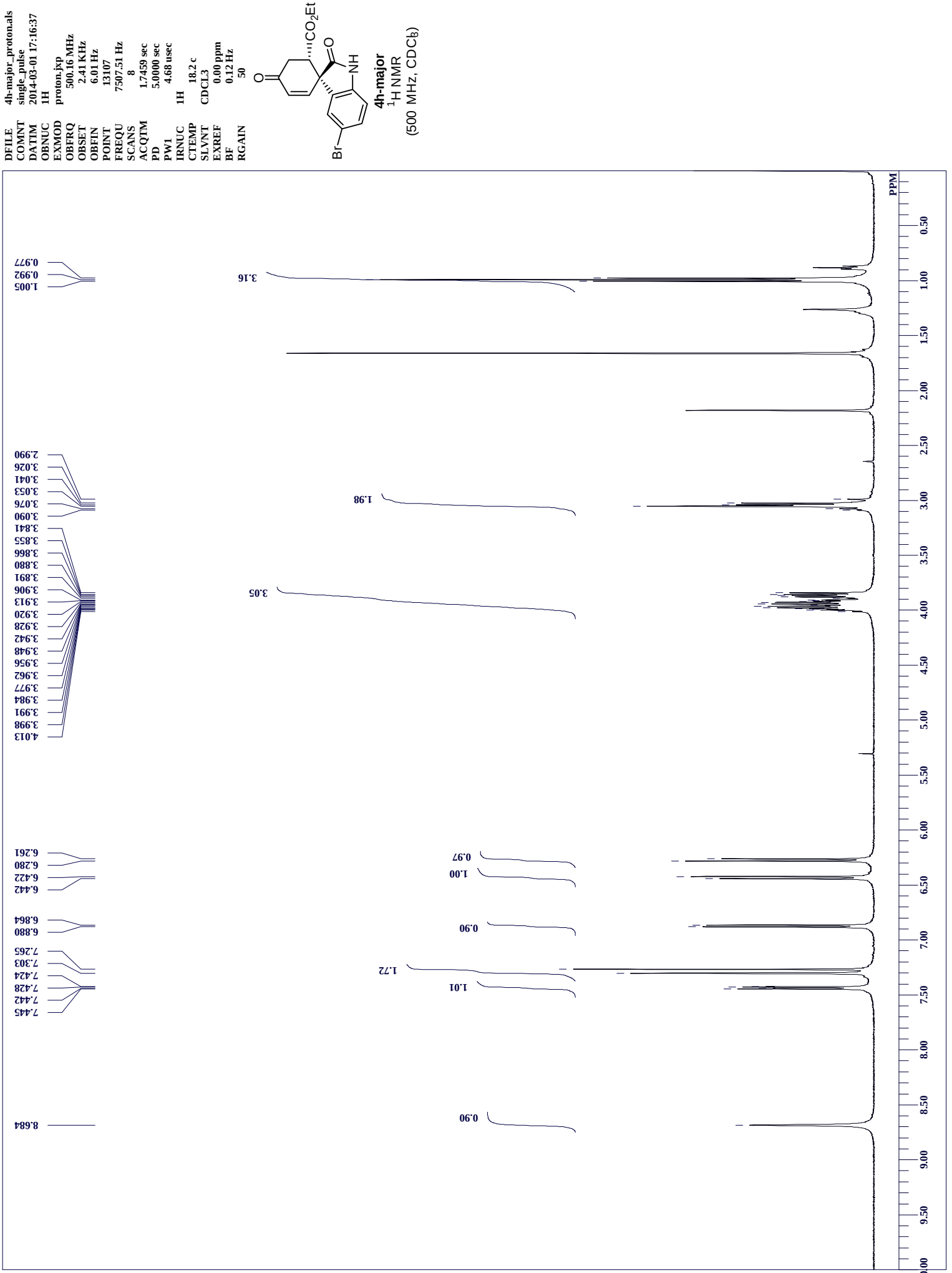


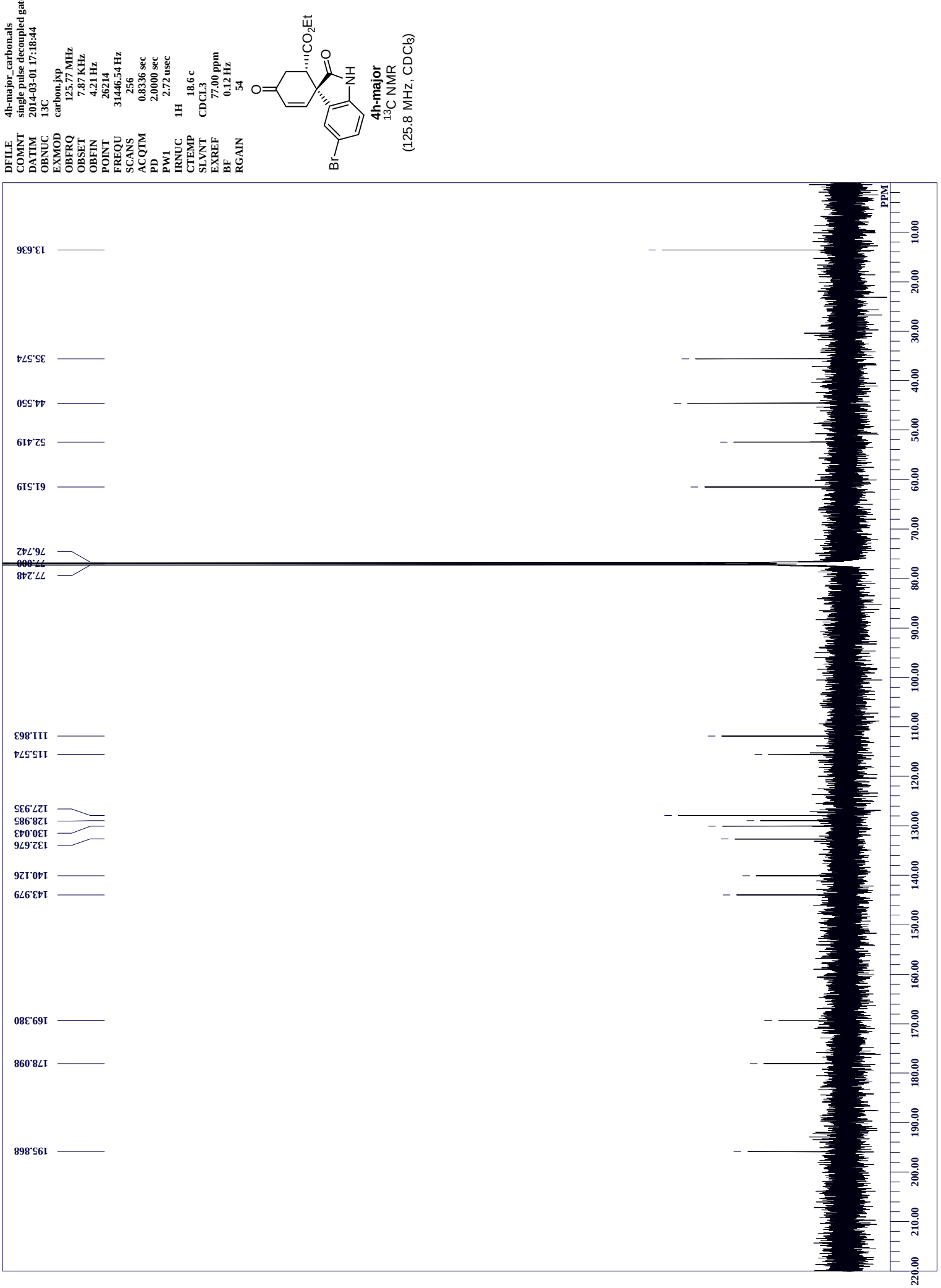
4g-major
^1H NMR
(500 MHz, $CDCl_3$)

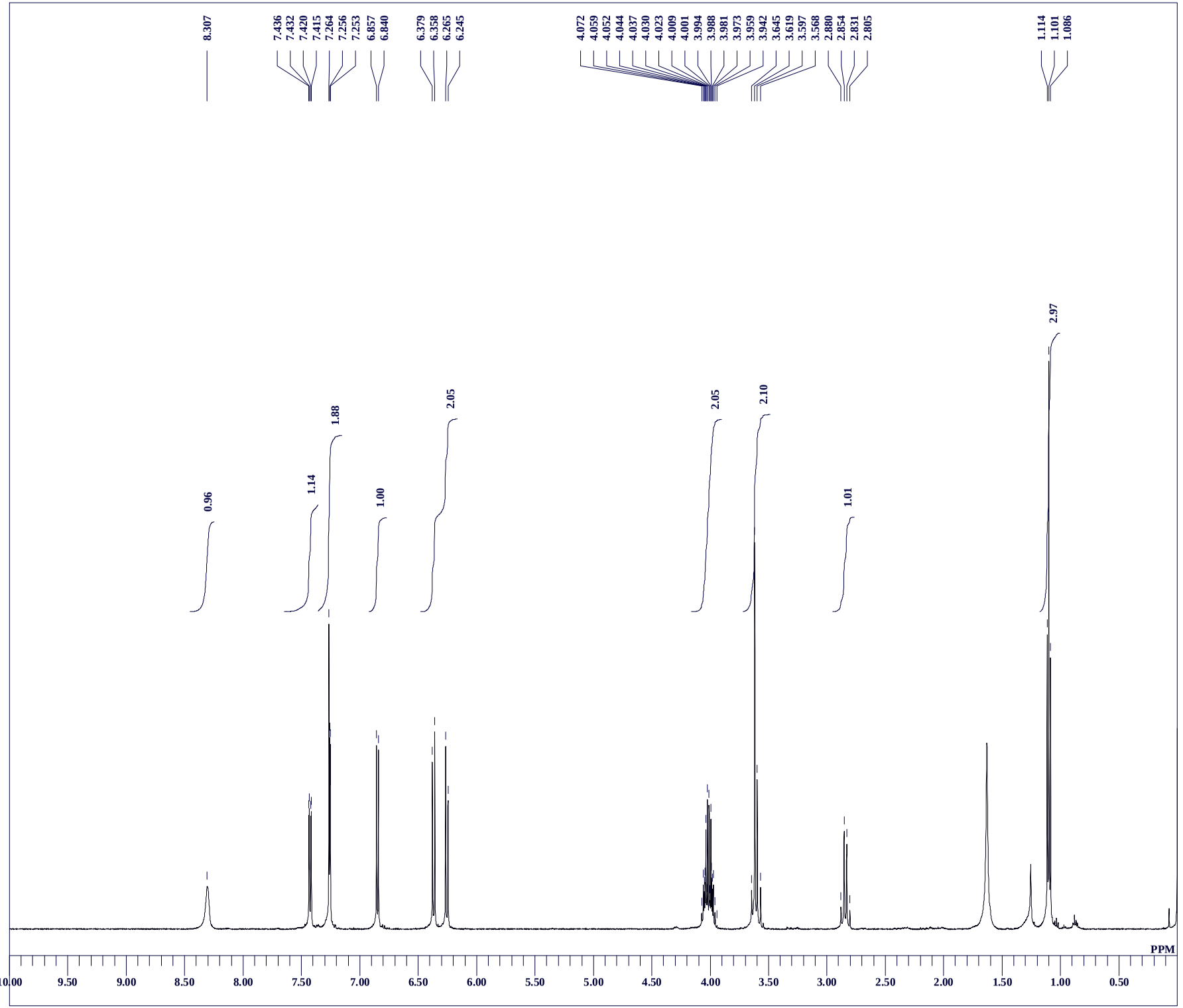

DFILE 4g-major_carbon.als
COMNT single pulse decoupled gat
DATIM 2013-12-02 11:53:36
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 256
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 17.9 c
SLVNT CDCL3
EXREF -0.02 ppm
BF 0.12 Hz
RGAIN 60
4g-major
13C NMR
(125.8 MHz, CDCl3)
CO2Et
NH
O
O
13.532
35.680
44.370
52.525
61.320
76.753
77.001
77.259
110.519
123.215
124.846
126.773
129.577
129.835
141.090
144.858
169.620
178.872
196.432
PPM
10.00
20.00
30.00
40.00
50.00
60.00
70.00
80.00
90.00
100.00
110.00
120.00
130.00
140.00
150.00
160.00
170.00
180.00
190.00
200.00
210.00
220.00


DFILE 4g-minor_proton.als
COMNT single_pulse
DATIM 2016-05-26 10:07:07
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 4.68 usec
IRNUC 1H
CTEMP 22.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 34
4g-minor
1H NMR
(500 MHz, CDCl3)
CO2Et
NH
O
O
PPM
1.008
1.022
1.035
2.59
0.98
1.96
1.98
2.782
2.806
2.811
2.824
2.830
2.855
3.623
3.652
3.659
3.667
3.672
3.696
3.917
3.930
3.938
3.945
3.952
3.966
3.973
3.980
3.988
3.994
4.001
2.00
6.247
6.267
6.410
6.430
6.963
6.979
7.056
7.071
7.085
7.135
7.150
7.271
7.273
7.277
7.287
7.302
7.304
4.47
9.158
0.95

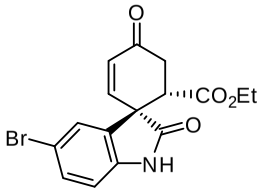

DFILE 4g-minor_carbon.als
COMNT single pulse decoupled gat
DATIM 2016-05-26 10:09:13
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 512
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 22.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 58
4g-minor
13C NMR
(125.8 MHz, CDCl3)
CO2Et
NH
O
O
13.617
35.832
47.850
51.313
61.252
76.742
77.000
77.258
110.480
122.804
122.947
129.271
130.492
131.474
141.261
146.812
170.095
176.334
196.765
PPM
10.00
20.00
30.00
40.00
50.00
60.00
70.00
80.00
90.00
100.00
110.00
120.00
130.00
140.00
150.00
160.00
170.00
180.00
190.00
200.00
210.00
220.00


DFILE 4h-major_proton.als
COMNT single_pulse
DATIM 2014-03-01 17:16:37
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 4.68 usec
IRNUC 1H
CTEMP 18.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 50
4h-major
1H NMR
(500 MHz, CDCl3)
PPM
8.684
7.445
7.442
7.428
7.424
7.303
7.265
6.880
6.864
6.442
6.422
6.280
6.261
4.013
3.998
3.991
3.984
3.977
3.962
3.956
3.948
3.942
3.928
3.920
3.913
3.906
3.891
3.880
3.866
3.855
3.841
3.090
3.076
3.053
3.041
3.026
2.990
1.005
0.992
0.977
0.90
1.01
1.72
0.90
1.00
0.97
3.05
1.98
3.16


DFILE 4h-major_carbon.als
COMNT single pulse decoupled gat
DATIM 2014-03-01 17:18:44
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 256
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 18.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 54
CO2Et
O
NH
Br
O
4h-major
13C NMR
(125.8 MHz, CDCl3)
13.636
35.574
44.550
52.419
61.519
76.742
77.000
77.248
111.863
115.574
127.935
128.985
130.043
132.676
140.126
143.979
169.380
178.098
195.868
PPM
10.00
20.00
30.00
40.00
50.00
60.00
70.00
80.00
90.00
100.00
110.00
120.00
130.00
140.00
150.00
160.00
170.00
180.00
190.00
200.00
210.00
220.00



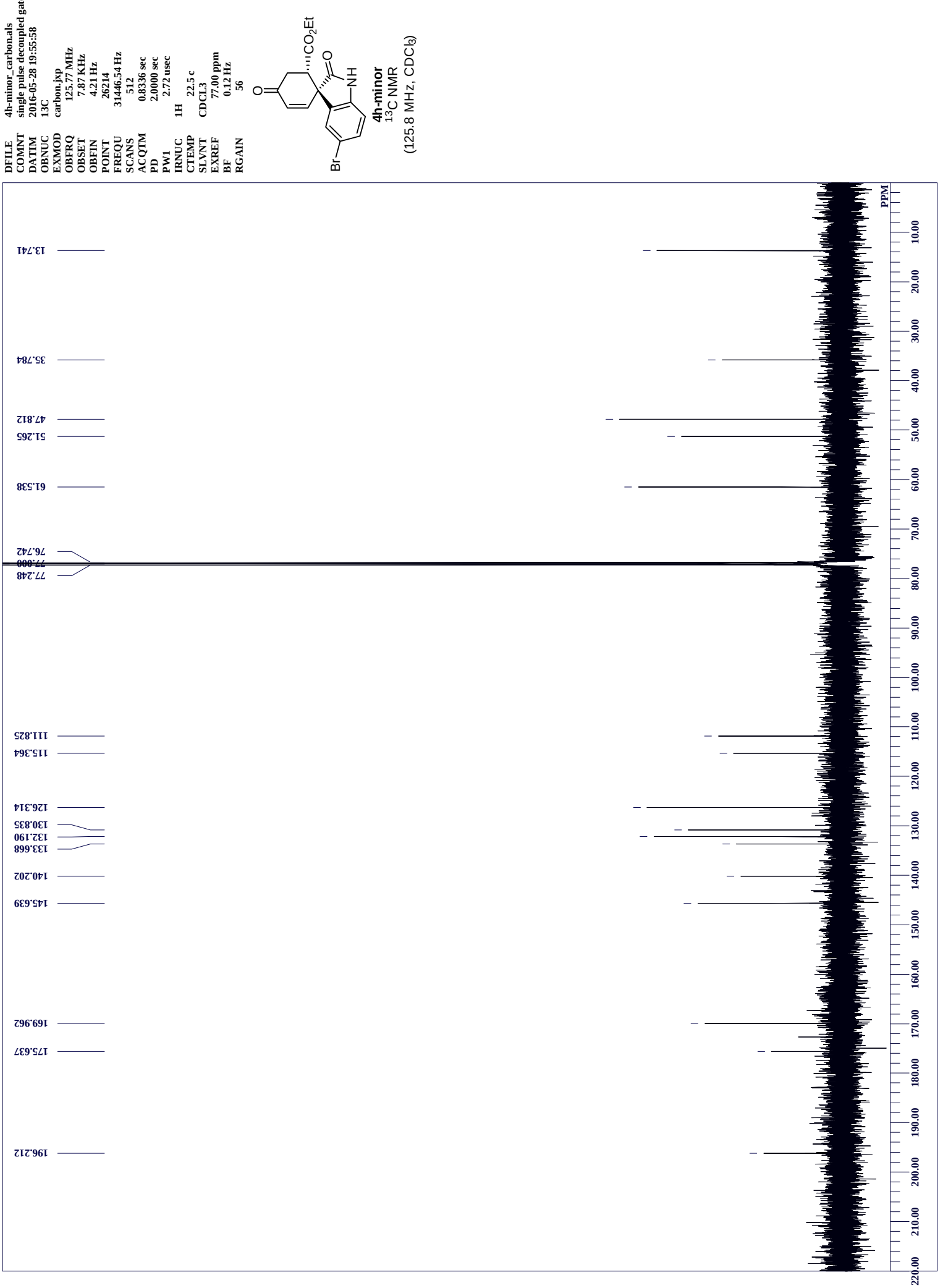
DFILE	4h-minor_proton.als
COMNT	single_pulse
DATIM	2016-05-31 21:10:47
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	4.68 usec
IRNUC	1H
CTEMP	22.7 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	52

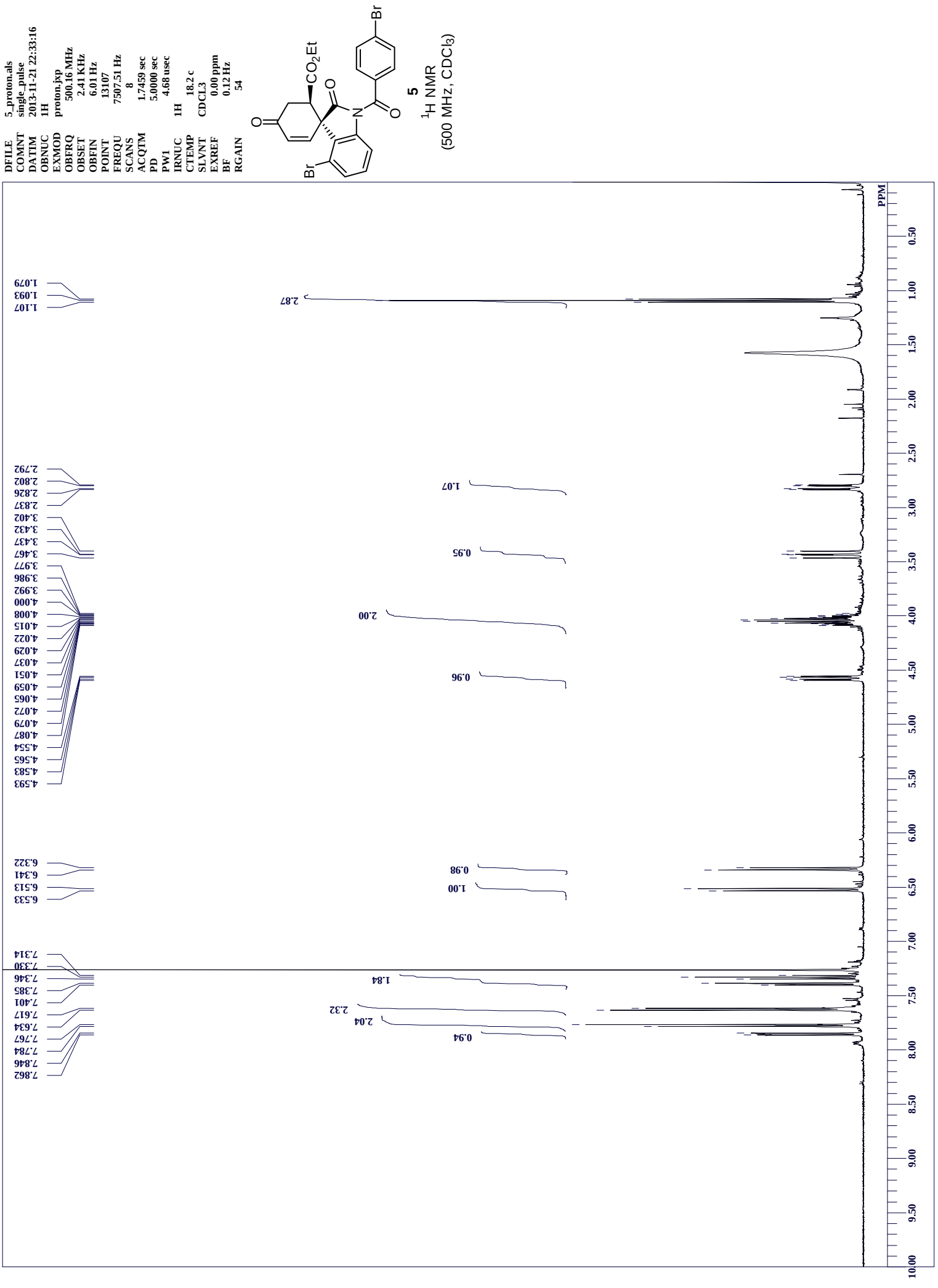


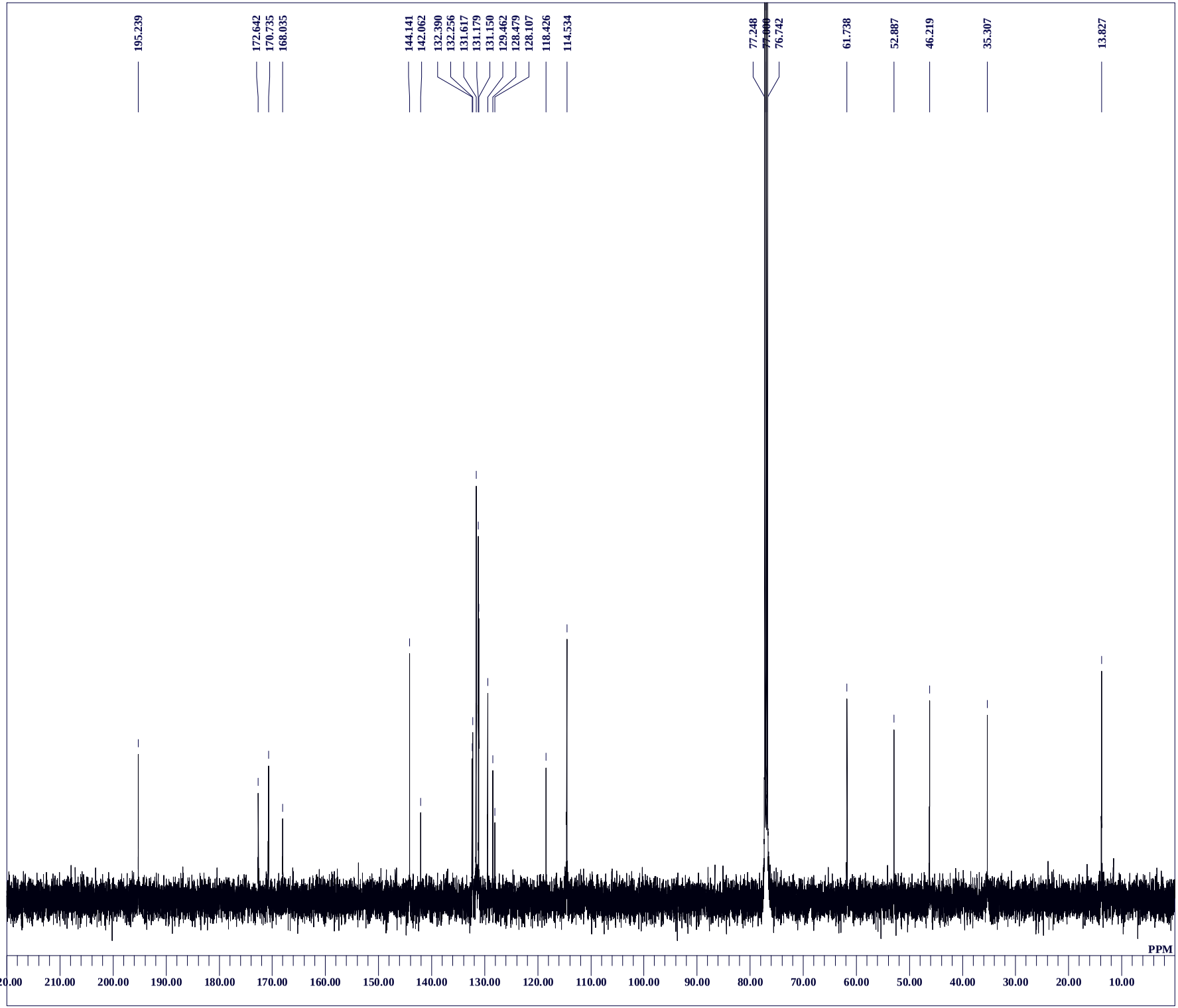
4h-minor

^{1}H NMR

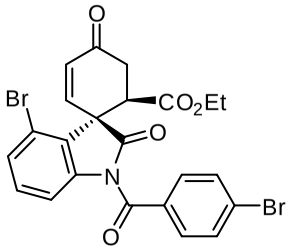
(500 MHz, $CDCl_3$)


DFILE 4h-minor_carbon.als
COMNT single pulse decoupled gat
DATIM 2016-05-28 19:55:58
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 512
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 2.72 usec
IRNUC 1H
CTEMP 22.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 56
4h-minor
13C NMR
(125.8 MHz, CDCl3)
13.741
35.784
47.812
51.265
61.538
76.742
77.000
77.248
111.825
115.364
126.314
130.835
132.190
133.668
140.202
145.639
169.962
175.637
196.212
PPM


DFILE 5_proton.als
COMNT single_pulse
DATIM 2013-11-21 22:33:16
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 4.68 usec
IRNUC 1H
CTEMP 18.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 54
5
1H NMR
(500 MHz, CDCl3)
PPM
2.87
1.07
0.95
2.00
0.96
0.98
1.00
1.84
2.32
2.04
0.94



DFILE	5_carbon.als
COMNT	single pulse decoupled gat
DATIM	2014-03-01 16:29:52
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.54 Hz
SCANS	256
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	18.3 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

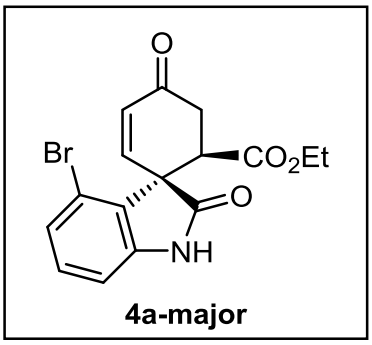


5

^{13}C NMR

(125.8 MHz, $CDCl_3$)

Sample

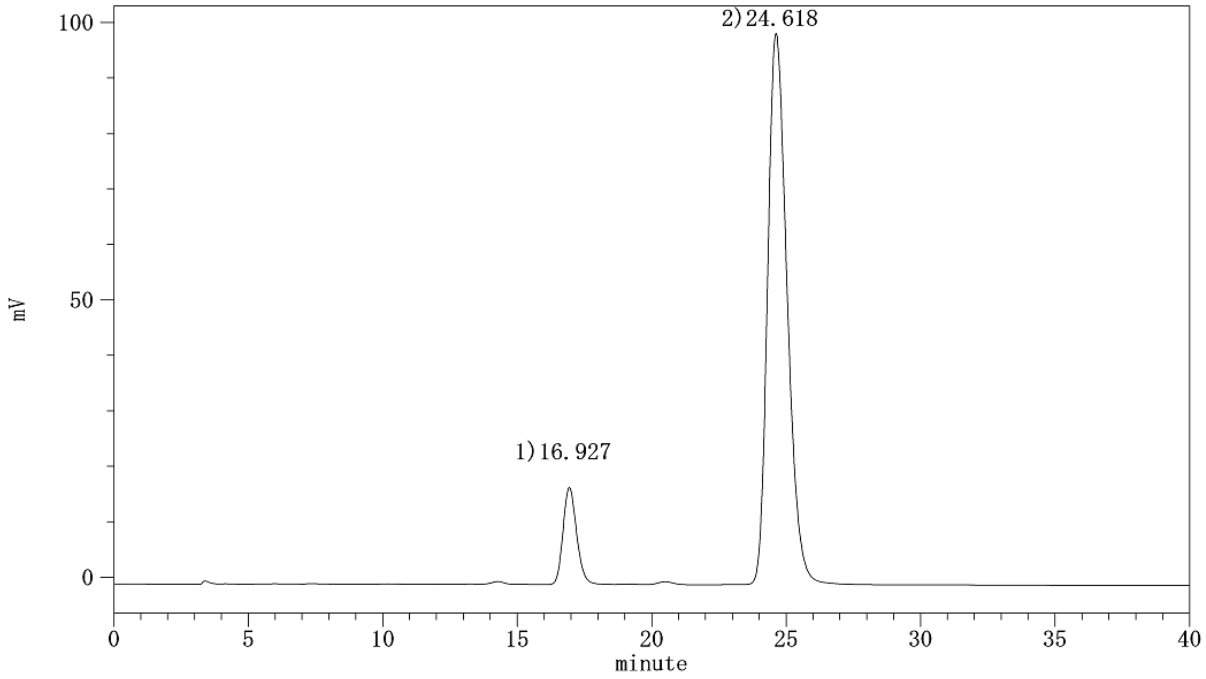


Exptl No.	MTG-511 (major)
Data:	2014/05/14
Column:	CHIRALPAK AD
Solvent:	hexane / 2-propanol = 90 : 10
Flow Speed:	1.0 cm^3 / min
Conc.	3.0 mg / mL
Vol.	3 μL
UV:	254 nm

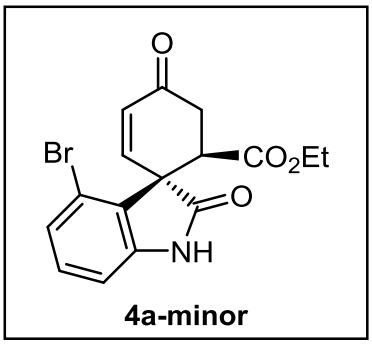
File:A201606111658.CHR
Injection Date:06-11-2016 Time:16:58:52

Integrator Parameter
Up Slope= 2 , Down Slope= 2 , Minimum width= 10.00 sec , Sensitivity= 5 points
Time Event = 0.000 to 2000.000 min.,Minimum area= 100 uv*sec , Baseline Compensative=0

No.	R.T. (minute)	Area%	Height (mv)	Height%	Left Base	Right Base
1	16.927	11.0110	17.5160	14.9926	15.742	19.802
2	24.618	88.9890	99.3149	85.0074	22.945	27.445
Total			116.831			



Sample



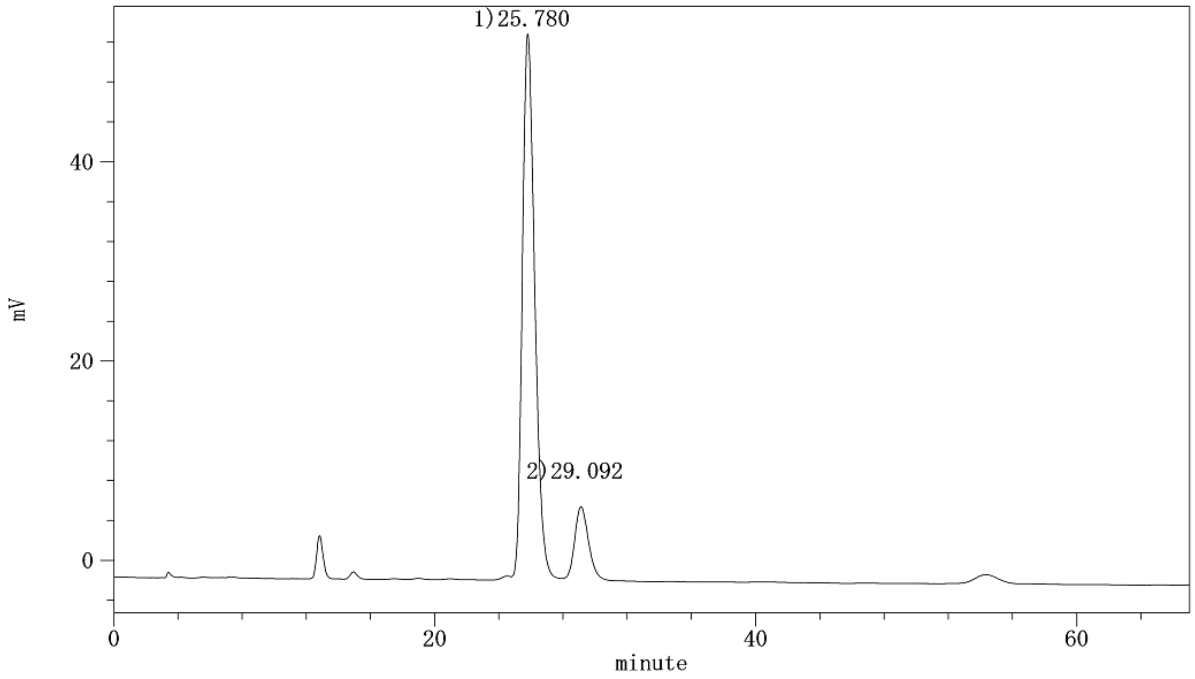
4a-minor

Exptl No.	NNO-566 (minor)
Data:	2016/06/23
Column:	CHIRALPAK AD
Solvent:	hexane / 2-propanol = 90 : 10
Flow Speed:	1.0 cm^3 / min
Conc.	3.0 mg / mL
Vol.	3 μL
UV:	254 nm

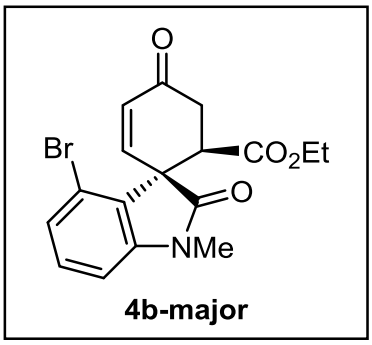
File:A201606221037.CHR
Injection Date:06-22-2016 Time:10:37:37

Integrator Parameter
Up Slope= 2 , Down Slope= 2 , Minimum width= 10.00 sec , Sensitivity= 5 points
Time Event = 0.000 to 2000.000 min.,Minimum area= 100 uv*sec , Baseline Compensative=0

No.	R.T. (minute)	Area%	Height (mv)	Height%	Left Base	Right Base
1	25.780	86.8355	54.8378	88.0699	23.645	31.125
2	29.092	13.1645	7.4285	11.9301	23.645	31.125
Total			62.266			



Sample



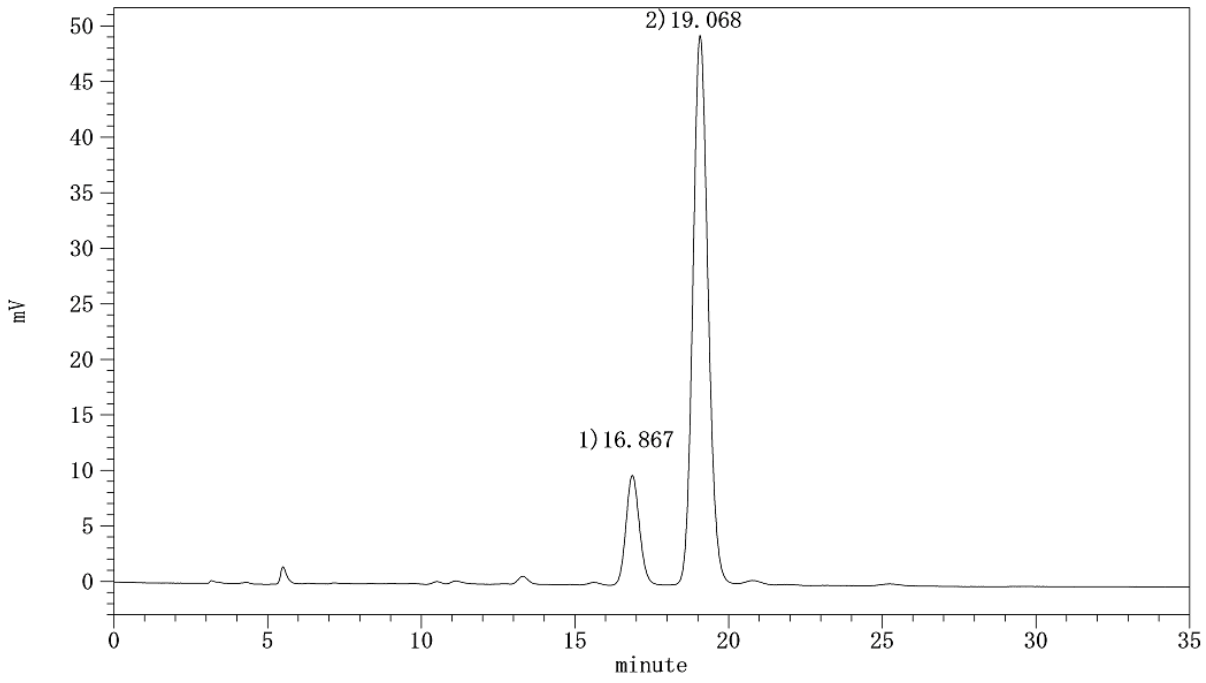
4b-major

Exptl No.	NKT-73 (major)
Data:	2015/10/22
Column:	CHIRALPAK AD
Solvent:	hexane / 2-propanol = 90 : 10
Flow Speed:	1.0 cm^3 / min
Conc.	3.0 mg / mL
Vol.	3 μL
UV:	254 nm

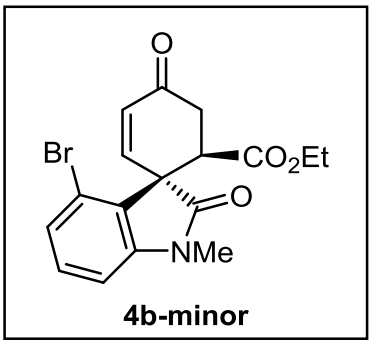
File:NKT-73 major.CHR
Injection Date:10-22-2015 Time:20:54:43

Integrator Parameter
Up Slope= 2 , Down Slope= 2 , Minimum width= 10.00 sec , Sensitivity= 5 points
Time Event = 0.000 to 2000.000 min.,Minimum area= 100 uv*sec , Baseline Compensative=0

No.	R.T. (minute)	Area%	Height (mv)	Height%	Left Base	Right Base
1	16.867	15.1206	9.8886	16.6698	16.140	17.912
2	19.068	84.8794	49.4315	83.3302	18.020	20.203
	Total		59.320			



Sample



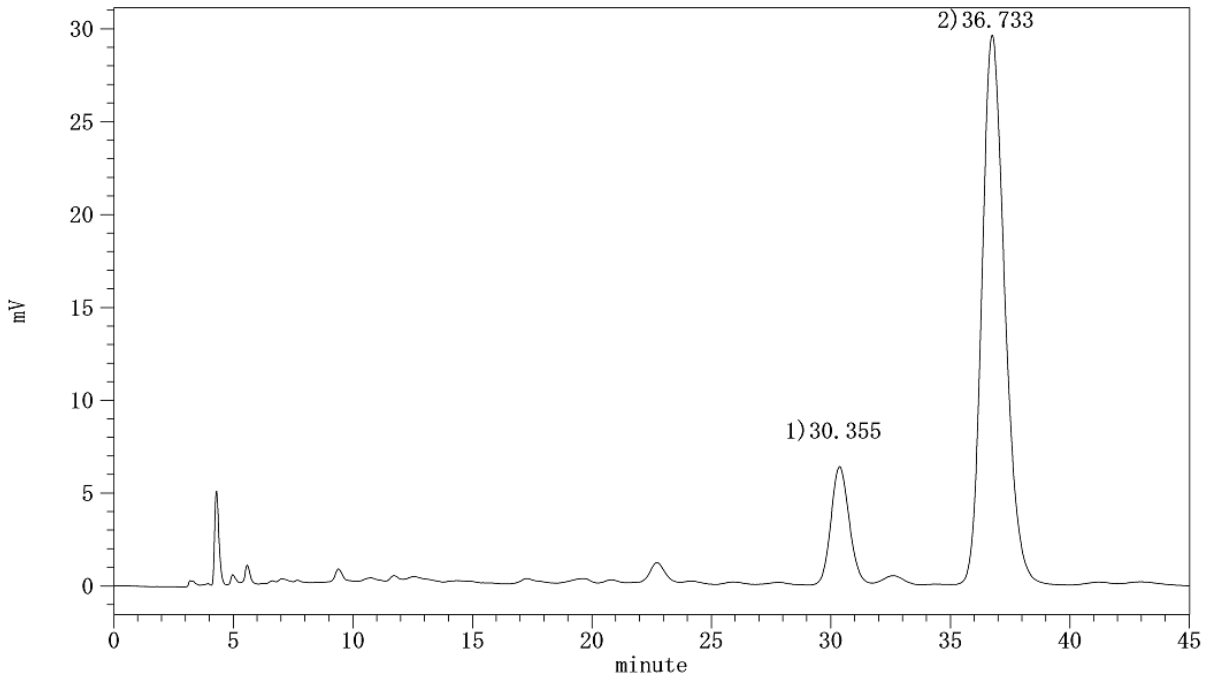
4b-minor

Exptl No.	NKT-73 (minor)
Data:	2015/10/22
Column:	CHIRALPAK AD
Solvent:	hexane / 2-propanol = 90 : 10
Flow Speed:	1.0 cm^3 / min
Conc.	3.0 mg / mL
Vol.	3 μL
UV:	254 nm

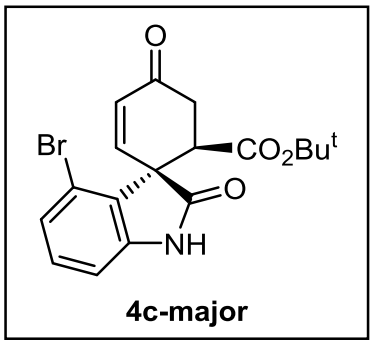
File:NKT-073 minor2.CHR
Injection Date:10-24-2015 Time:14:55:06

Integrator Parameter
Up Slope= 2 , Down Slope= 2 , Minimum width= 10.00 sec , Sensitivity= 5 points
Time Event = 0.000 to 2000.000 min.,Minimum area= 100 uv*sec , Baseline Compensative=0

No.	R.T. (minute)	Area%	Height (mv)	Height%	Left Base	Right Base
1	30.355	14.6996	6.3665	17.7027	29.230	33.825
2	36.733	85.3004	29.5969	82.2973	33.847	39.398
Total			35.963			



Sample



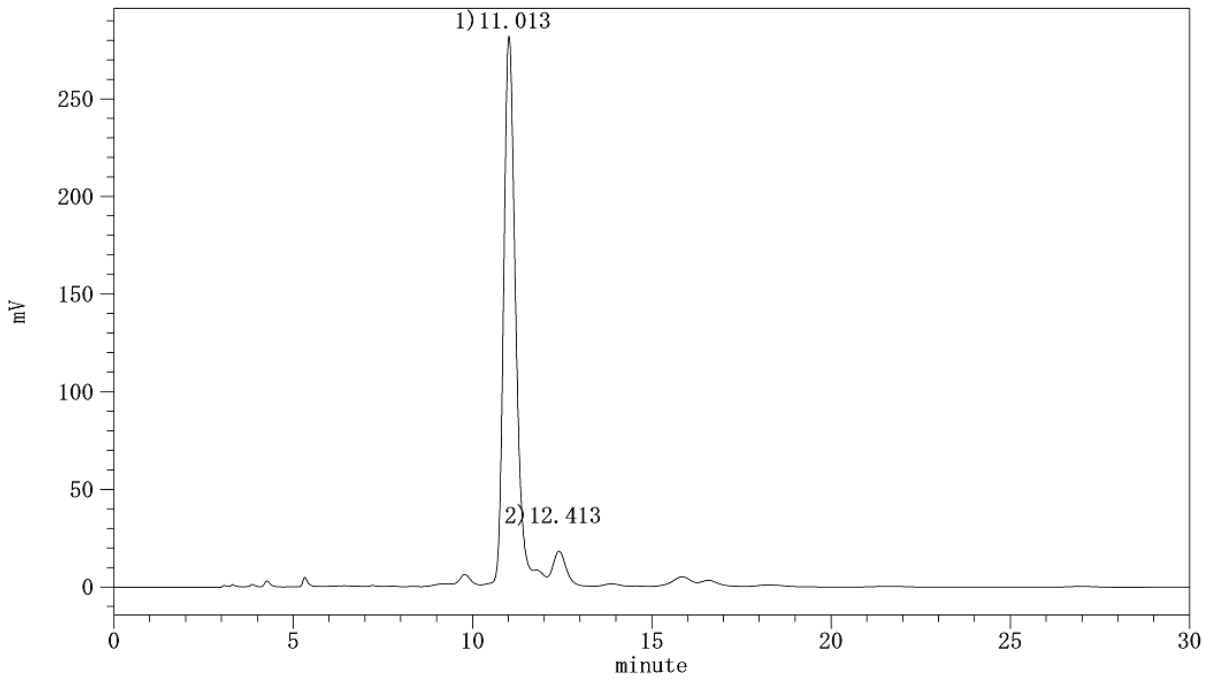
4c-major

Exptl No.	MTG-352 (major)
Data:	2014/05/15
Column:	CHIRALPAK AD
Solvent:	hexane / 2-propanol = 90 : 10
Flow Speed:	1.0 cm^3 / min
Conc.	3.0 mg / mL
Vol.	3 μL
UV:	254 nm

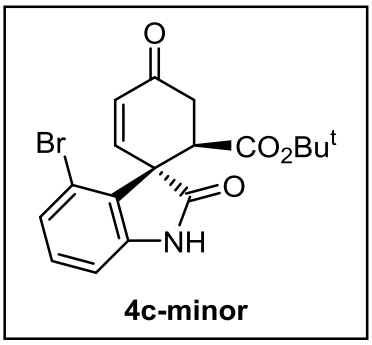
File:A201405152215.CHR
Injection Date:05-15-2014 Time:22:15:08

Integrator Parameter
Up Slope= 2 , Down Slope= 2 , Minimum width= 10.00 sec , Sensitivity= 5 points
Time Event = 0.000 to 2000.000 min.,Minimum area= 100 uv*sec , Baseline Compensative=0

No.	R.T. (minute)	Area%	Height (mv)	Height%	Left Base	Right Base
1	11.013	92.8123	282.1394	93.8699	4.730	22.797
2	12.413	7.1877	18.4249	6.1301	4.730	22.797
Total			300.564			



Sample



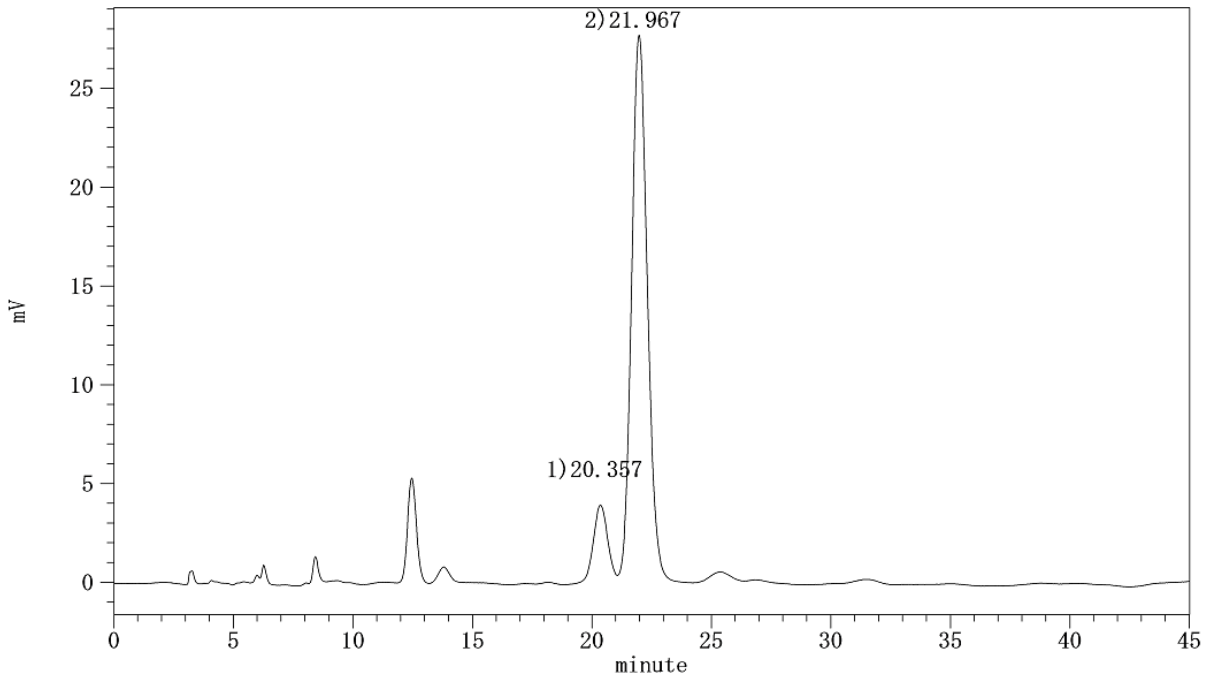
4c-minor

Exptl No.	NKT-107 (minor)
Data:	2016/01/15
Column:	CHIRALPAK AD
Solvent:	hexane / 2-propanol = 90 : 10
Flow Speed:	1.0 cm^3 / min
Conc.	3.0 mg / mL
Vol.	3 μL
UV:	254 nm

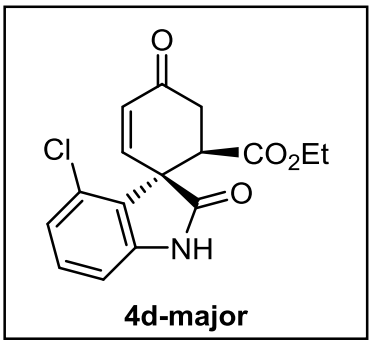
File:NKT-107 down.CHR
Injection Date:01-15-2016 Time:10:42:06

Integrator Parameter
Up Slope= 2 , Down Slope= 2 , Minimum width= 10.00 sec , Sensitivity= 5 points
Time Event = 0.000 to 2000.000 min.,Minimum area= 100 uv*sec , Baseline Compensative=0

No.	R.T. (minute)	Area%	Height (mv)	Height%	Left Base	Right Base
1	20.357	11.8159	3.9927	12.5910	18.873	23.635
2	21.967	88.1841	27.7178	87.4090	18.873	23.635
Total			31.711			



Sample

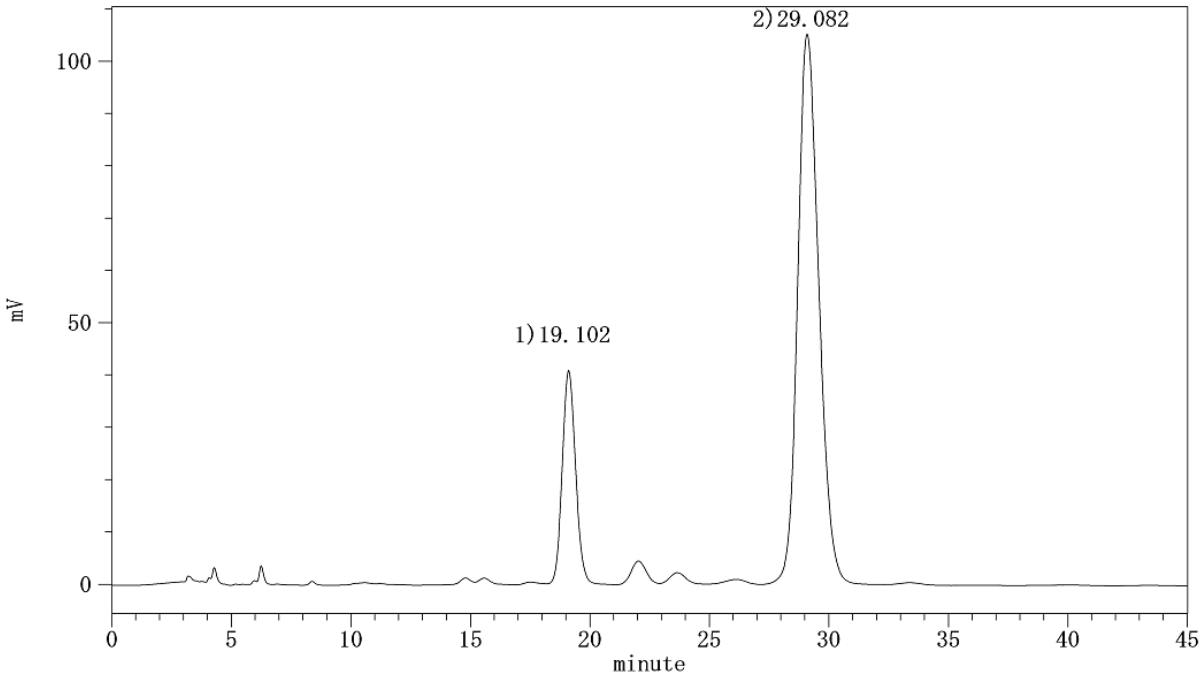


Exptl No.	NKT-105 (major)
Data:	2015/10/22
Column:	CHIRALPAK AD
Solvent:	hexane / 2-propanol = 90 : 10
Flow Speed:	1.0 cm^3 / min
Conc.	3.0 mg / mL
Vol.	3 μL
UV:	254 nm

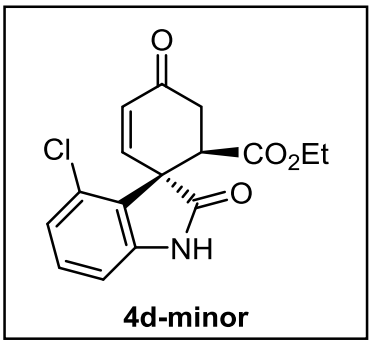
File:NKT-105 fr12-15.CHR
Injection Date:01-09-2016 Time:21:48:08

Integrator Parameter
Up Slope= 2 , Down Slope= 2 , Minimum width= 10.00 sec , Sensitivity= 5 points
Time Event = 0.000 to 2000.000 min.,Minimum area= 100 uv*sec , Baseline Compensative=0

No.	R.T. (minute)	Area%	Height (mv)	Height%	Left Base	Right Base
1	19.102	19.7914	41.0603	28.0421	13.710	35.133
2	29.082	80.2086	105.3637	71.9579	13.710	35.133
Total			146.424			



Sample

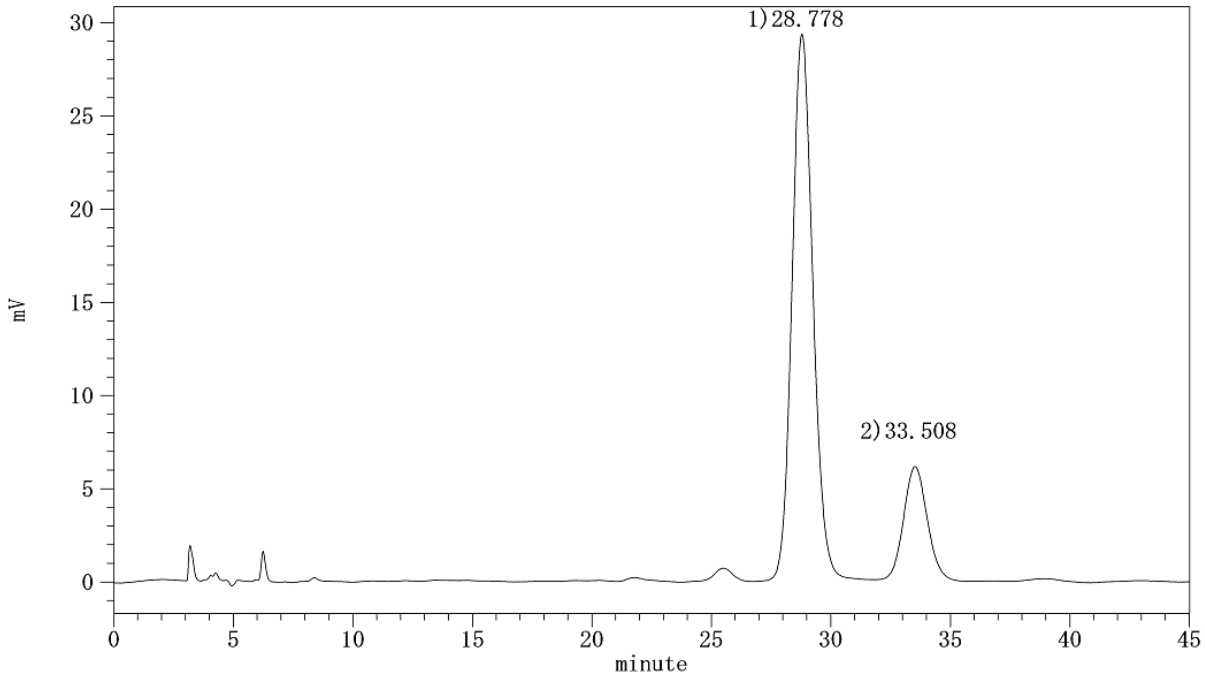


Exptl No.	NKT-105 (minor)
Data:	2015/10/24
Column:	CHIRALPAK AD
Solvent:	hexane / 2-propanol = 90 : 10
Flow Speed:	1.0 cm^3 / min
Conc.	3.0 mg / mL
Vol.	3 μL
UV:	254 nm

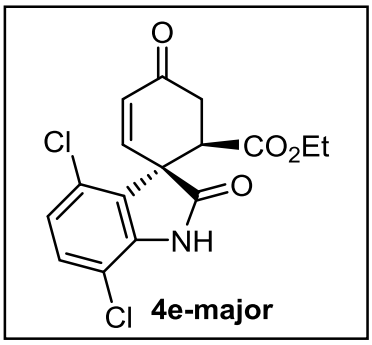
File:NKT-105 major.CHR
Injection Date:01-09-2016 Time:20:43:34

Integrator Parameter
Up Slope= 2 , Down Slope= 2 , Minimum width= 10.00 sec , Sensitivity= 5 points
Time Event = 0.000 to 2000.000 min.,Minimum area= 100 uv*sec , Baseline Compensative=0

No.	R.T. (minute)	Area%	Height (mv)	Height%	Left Base	Right Base
1	28.778	80.6544	29.3461	82.6548	26.845	35.572
2	33.508	19.3456	6.1583	17.3452	26.845	35.572
	Total		35.504			



Sample

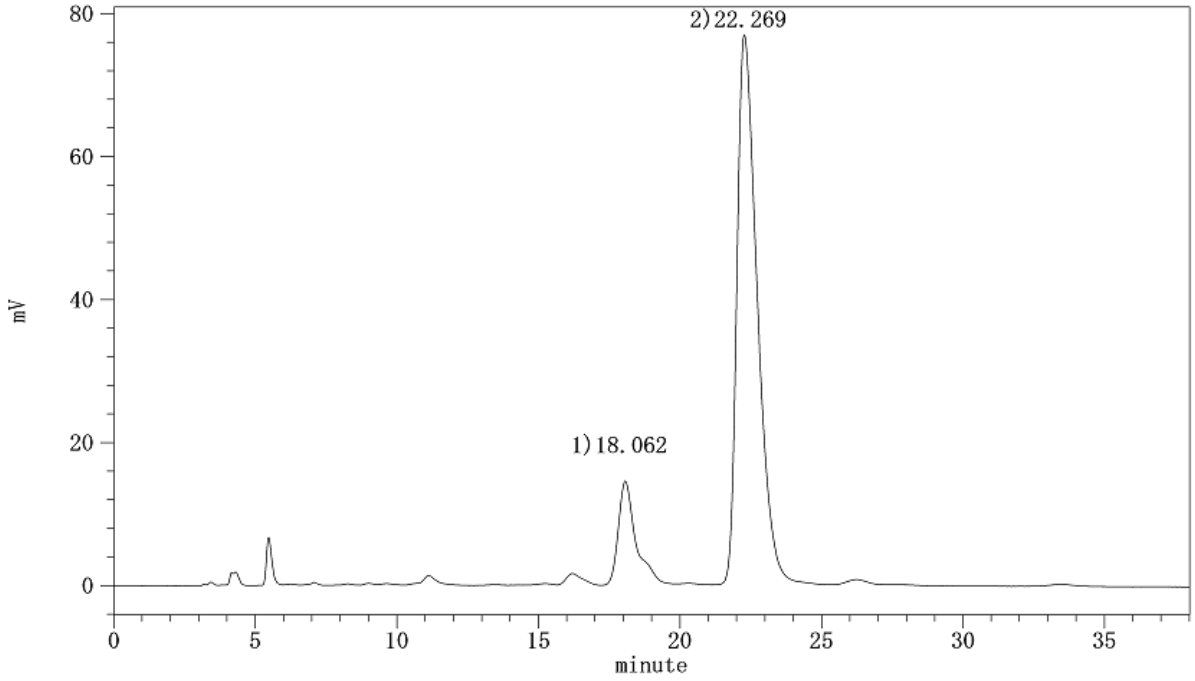


Exptl No.	NKT-75 (major)
Data:	2015/11/06
Column:	CHIRALPAK AD
Solvent:	hexane / 2-propanol = 90 : 10
Flow Speed:	1.0 cm^3 / min
Conc.	3.0 mg / mL
Vol.	3 μL
UV:	254 nm

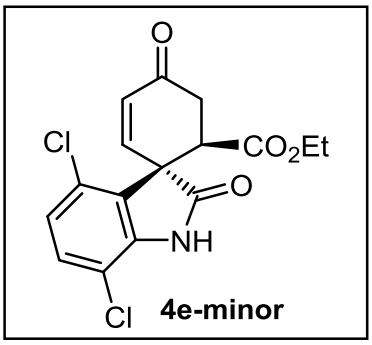
File:NKT-075 major.CHR
Injection Date:11-06-2015 Time:17:33:32

Integrator Parameter
Up Slope= 2 , Down Slope= 2 , Minimum width= 10.00 sec , Sensitivity= 5 points
Time Event = 0.000 to 2000.000 min.,Minimum area= 100 uv*sec , Baseline Compensative=0

No.	R.T. (minute)	Area%	Height (mv)	Height%	Left Base	Right Base
1	18.062	13.9474	14.5356	15.8884	17.263	19.813
2	22.269	86.0526	76.9503	84.1116	21.187	25.209
Total			91.486			



Sample

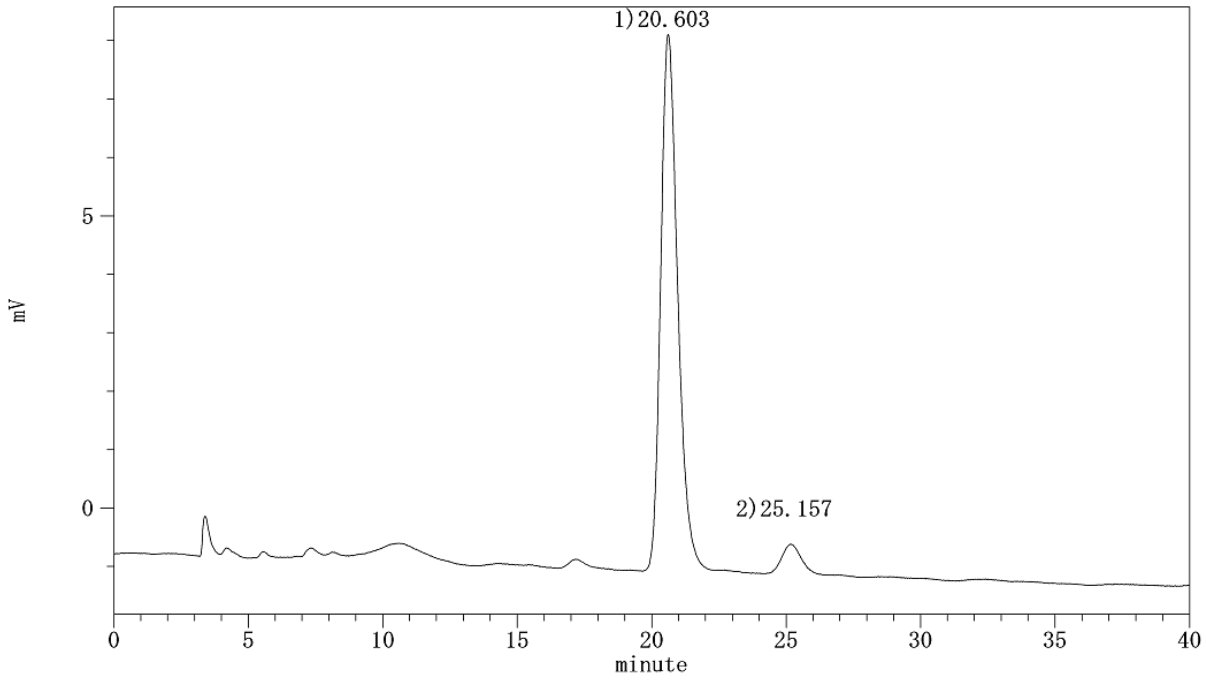


Exptl No.	MTG-334 (minor)
Data:	2016/06/01
Column:	CHIRALPAK AD
Solvent:	hexane / 2-propanol = 90 : 10
Flow Speed:	1.0 cm^3 / min
Conc.	3.0 mg / mL
Vol.	3 μL
UV:	254 nm

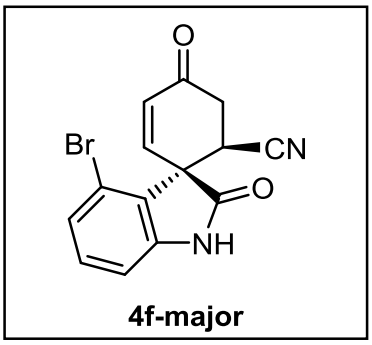
File:A201606092258.CHR
Injection Date:06-09-2016 Time:22:58:35

Integrator Parameter
Up Slope= 2 , Down Slope= 2 , Minimum width= 10.00 sec , Sensitivity= 5 points
Time Event = 0.000 to 2000.000 min.,Minimum area= 100 uv*sec , Baseline Compensative=0

No.	R.T. (minute)	Area%	Height (mv)	Height%	Left Base	Right Base
1	20.603	93.6131	9.1593	94.6669	19.820	23.670
2	25.157	6.3869	0.5160	5.3331	24.352	27.685
Total			9.675			



Sample



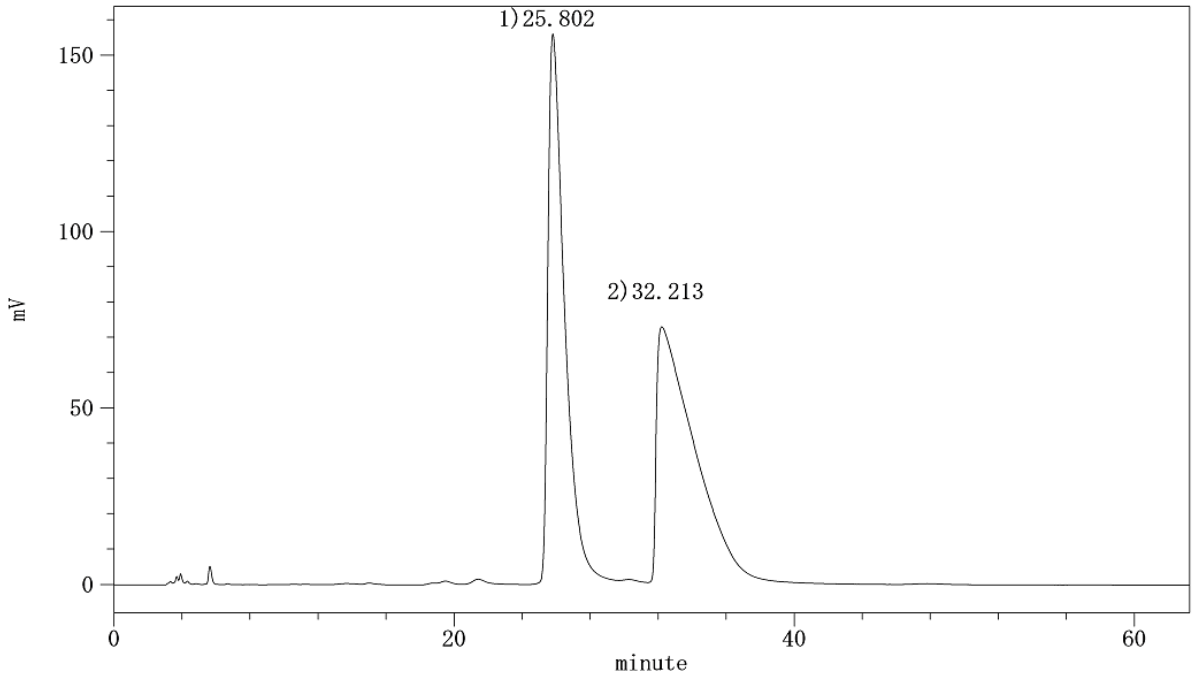
4f-major

Exptl No.	NNO-541 (major)
Data:	2016/05/28
Column:	CHIRALPAK AD
Solvent:	hexane / 2-propanol = 90 : 10
Flow Speed:	1.0 cm^3 / min
Conc.	3.0 mg / mL
Vol.	3 μL
UV:	254 nm

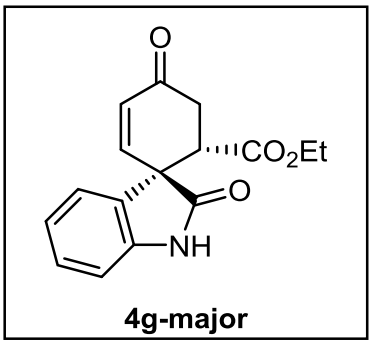
File:A201605282333.CHR
Injection Date:05-28-2016 Time:23:33:05

Integrator Parameter
Up Slope= 2 , Down Slope= 2 , Minimum width= 10.00 sec , Sensitivity= 5 points
Time Event = 0.000 to 2000.000 min.,Minimum area= 100 uv*sec , Baseline Compensative=0

No.	R.T. (minute)	Area%	Height (mv)	Height%	Left Base	Right Base
1	25.802	48.0503	156.0507	68.1151	24.562	53.005
2	32.213	51.9497	73.0479	31.8849	24.562	53.005
Total			229.099			



Sample

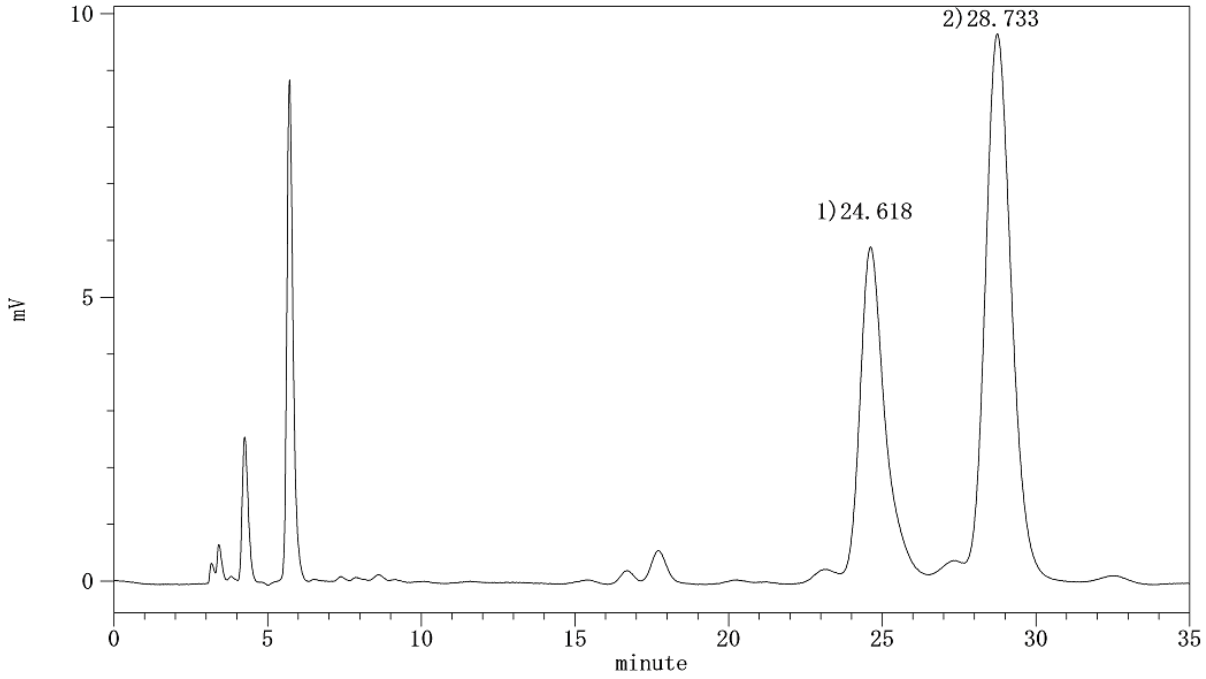


Exptl No.	NKT-101 (major)
Data:	2015/12/21
Column:	CHIRALPAK AD
Solvent:	hexane / 2-propanol = 90 : 10
Flow Speed:	1.0 cm^3 / min
Conc.	3.0 mg / mL
Vol.	3 μL
UV:	254 nm

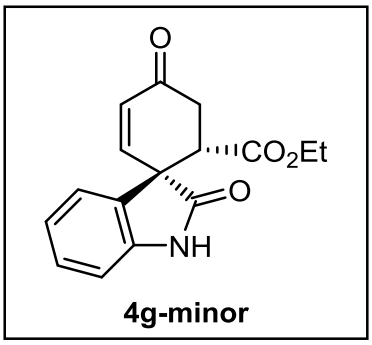
File:NKT-101 re.CHR
Injection Date:12-19-2015 Time:15:25:57

Integrator Parameter
Up Slope= 2 , Down Slope= 2 , Minimum width= 10.00 sec , Sensitivity= 5 points
Time Event = 0.000 to 2000.000 min.,Minimum area= 100 uv*sec , Baseline Compensative=0

No.	R.T. (minute)	Area%	Height (mv)	Height%	Left Base	Right Base
1	24.618	37.6515	5.8457	38.2531	22.062	26.430
2	28.733	62.3485	9.4360	61.7469	27.678	30.693
	Total		15.282			



Sample

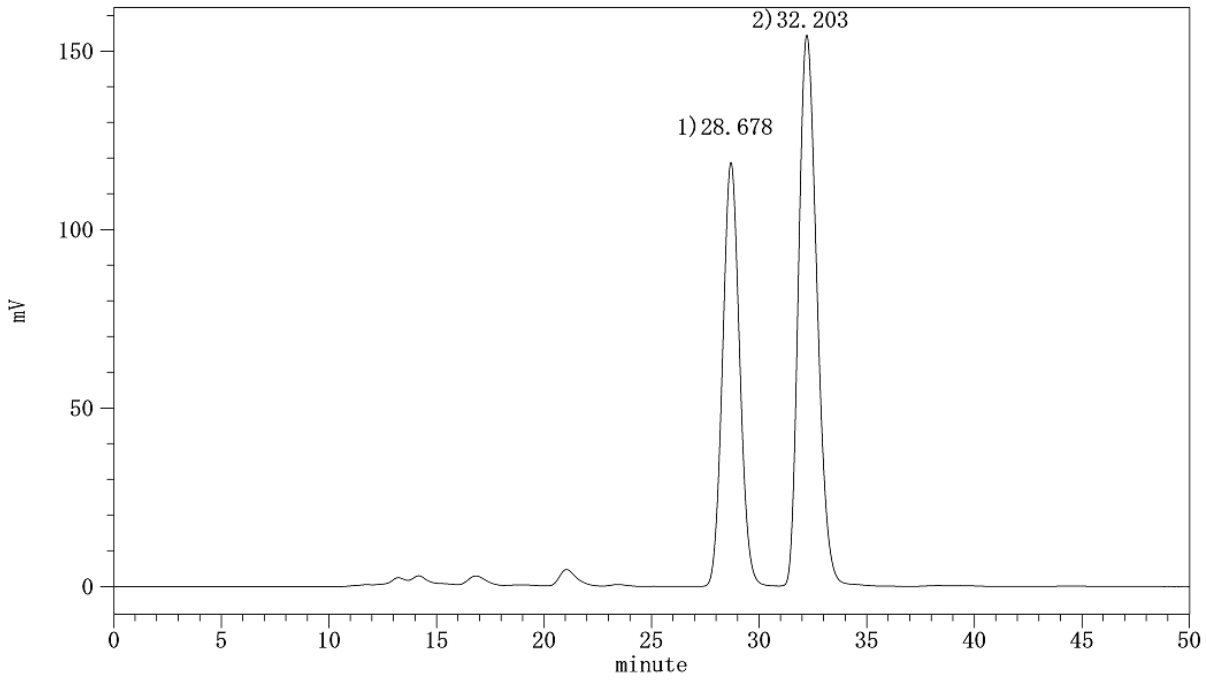


Exptl No.	NNO-555 (minor)
Data:	2016/05/28
Column:	CHIRALPAK AD
Solvent:	hexane / 2-propanol = 90 : 10
Flow Speed:	1.0 cm^3 / min
Conc.	3.0 mg / mL
Vol.	3 μL
UV:	254 nm

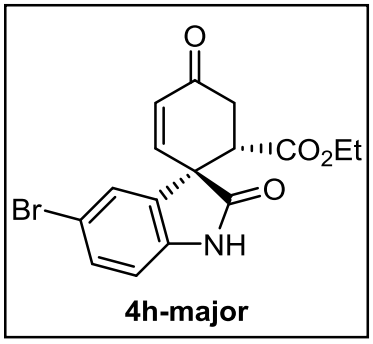
File:A201603032040.CHR
Injection Date:03-03-2016 Time:20:40:40

Integrator Parameter
Up Slope= 2 , Down Slope= 2 , Minimum width= 10.00 sec , Sensitivity= 5 points
Time Event = 0.000 to 2000.000 min.,Minimum area= 100 uv*sec , Baseline Compensative=0

No.	R.T. (minute)	Area%	Height (mv)	Height%	Left Base	Right Base
1	28.678	41.4757	118.7575	43.4759	27.255	30.870
2	32.203	58.5243	154.3995	56.5241	30.870	35.807
Total			273.157			



Sample

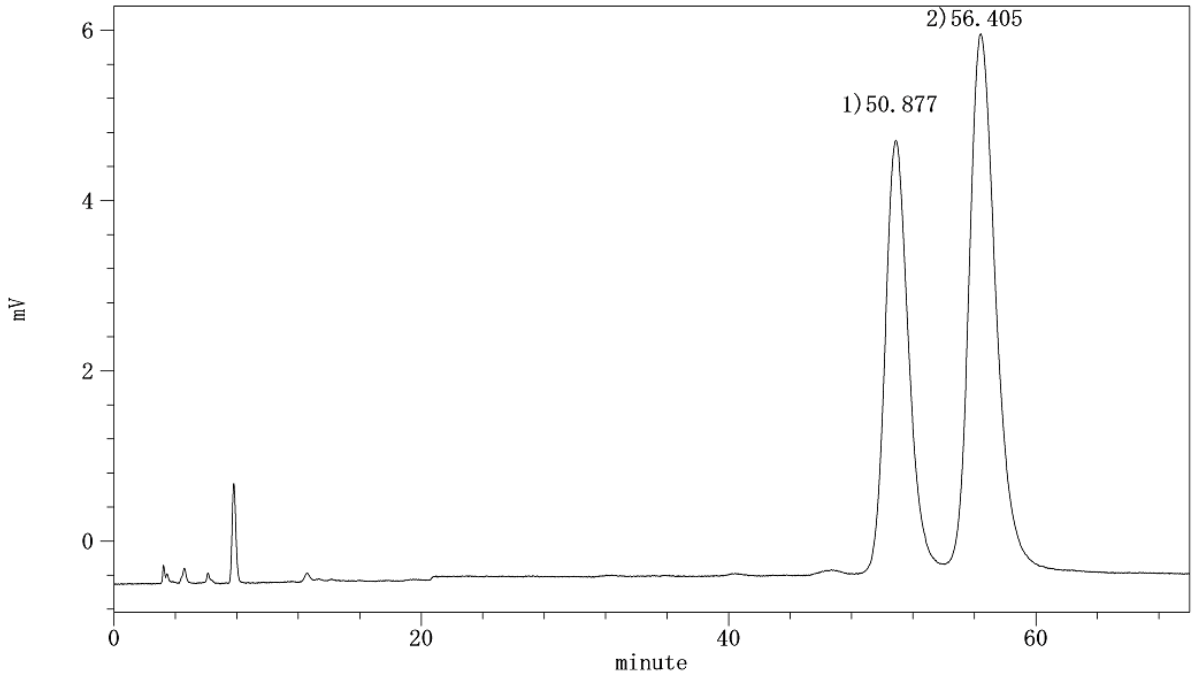


Exptl No.	NNO-557 (major)
Data:	2016/04/28
Column:	CHIRALPAK AD
Solvent:	hexane / 2-propanol = 95 : 5
Flow Speed:	1.0 cm^3 / min
Conc.	3.0 mg / mL
Vol.	3 μL
UV:	254 nm

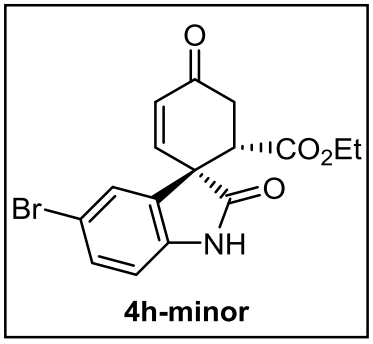
File:A201605281533.CHR
Injection Date:05-28-2016 Time:15:33:38

Integrator Parameter
Up Slope= 2 , Down Slope= 2 , Minimum width= 10.00 sec , Sensitivity= 5 points
Time Event = 0.000 to 2000.000 min.,Minimum area= 100 uv*sec , Baseline Compensative=0

No.	R.T. (minute)	Area%	Height (mv)	Height%	Left Base	Right Base
1	50.877	41.3681	5.0930	44.6019	48.457	62.062
2	56.405	58.6319	6.3259	55.3981	48.457	62.062
	Total		11.419			



Sample



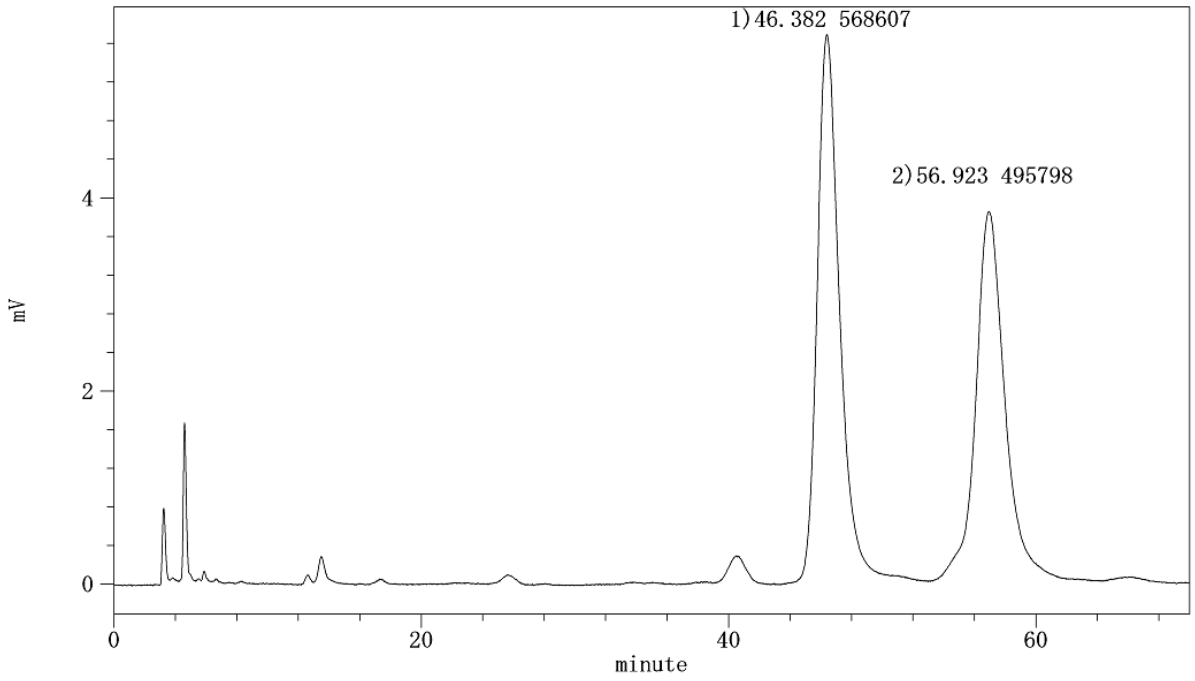
4h-minor

Exptl No.	NNO-557 (minor)
Data:	2016/04/28
Column:	CHIRALPAK AD
Solvent:	hexane / 2-propanol = 95 : 5
Flow Speed:	1.0 cm^3 / min
Conc.	3.0 mg / mL
Vol.	3 μL
UV:	254 nm

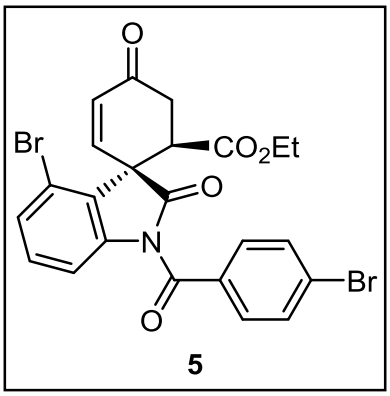
File:A201605281707.CHR
Injection Date:05-28-2016 Time:17:07:03

Integrator Parameter
Up Slope= 2 , Down Slope= 2 , Minimum width= 10.00 sec , Sensitivity= 5 points
Time Event = 0.000 to 2000.000 min.,Minimum area= 100 uv*sec , Baseline Compensative=0

No.	R.T. (minute)	Area%	Height (mv)	Height%	Left Base	Right Base
1	46.382	53.4202	5.6784	59.7461	44.232	52.958
2	56.923	46.5798	3.8258	40.2539	53.465	63.802
Total			9.504			



Sample



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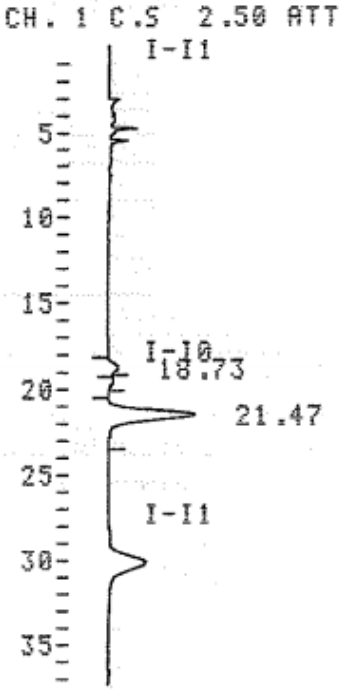
Exptl No. MTG-375
Data: 2014/03/18
Column: CHIRALPAK AD
Solvent: hexane / 2-propanol = 90 : 10
Flow Speed: 1.0 cm^3 / min
Conc. 3.0 mg / mL
Vol. 3 μL
UV: 254 nm



D-2000 00/00/00 00:03

SAMPLE: TEST TAG: 2 CH: 1

FILE: 1 CALC-METHOD: AREA% TABLE: 0 CONC: AREA

NO.	RT	AREA	CONC	BC
1	18.73	12680	5.882	BB
2	19.43	3339	1.549	BB
3	21.47	199534	92.568	BB
TOTAL		215553	1 * 000	

PEAK REJ : 0