Electronic Supplementary Information for

Fluorescence-labeled neopeltolide derivatives for subcellular localization imaging

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Contents

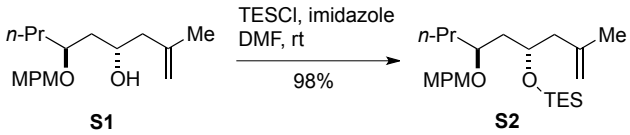
Copies of NMR spectra and HPLC chromatograms

Chemistry

General remarks

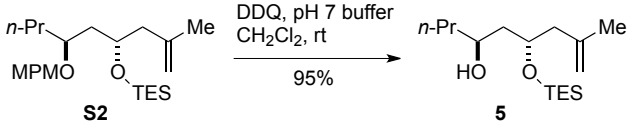
All reactions sensitive to moisture and/or air were carried out under an atmosphere of argon in dry, freshly distilled solvents under anhydrous conditions using oven-dried glassware unless otherwise noted. Anhydrous dichloromethane (CH_2Cl_2) was purchased from Kanto Chemical Co. Inc. and used directly without further drying. Anhydrous tetrahydrofuran (THF) and toluene were purchased from Wako Pure Chemical Industries, Ltd. and further purified by a Glass Contour solvent purification system under an atmosphere of argon immediately prior to use. Where appropriate, solvents were degassed by the freeze–thaw technique immediately prior to use. Acetonitrile, boron trifluoride diethyl etherate ($BF_3 \cdot OEt_2$), methanol, and triethylamine were distilled from calcium hydride under an atmosphere of argon. *N*,*N*-Dimethylformamide (DMF) was distilled over $MgSO_4$ under an atmosphere of argon. All other chemicals were purchased at highest commercial grade and used directly. Analytical thin-layer chromatography was performed using E. Merck silica gel 60 F_{254} plates (0.25-mm thickness). Flash column chromatography was carried out using Fuji Silysia silica gel BW-300 (200–400 mesh). Optical rotations were recorded on a JASCO P-1020 digital polarimeter. IR spectra were recorded on a JASCO FT/IR-4100 spectrometer. ^1H and ^{13}C NMR spectra were recorded on a JEOL JNM ECA-600 spectrometer, and chemical shift values are reported in ppm (δ) downfield from tetramethylsilane with reference to internal residual solvent [^1H NMR, C*H*Cl_3 (7.24), C_6HD_5 (7.15), C*H*D_2OD (3.31); ^{13}C NMR, $CDCl_3$ (77.0), C_6D_6 (128.0), CD_3OD (49.8)] unless otherwise noted. Coupling constants (*J*) are reported in Hertz (Hz). The following abbreviations were used to designate the multiplicities: s = singlet; d = doublet; t = triplet; q = quartet; m = multiplet; br = broad. ESI-TOF mass spectra were measured on a Bruker microTOFfocus spectrometer.

Synthesis and characterization data of new compounds



S1 S2

Silyl ether S2. To a solution of alcohol **S1**[1] (147 mg, 0.503 mmol) in DMF (5 mL) at 0 °C were added imidazole (102 mg, 1.50 mmol) and TESCl (0.125 mL, 0.745 mmol), and the resultant solution was stirred at room temperature for 1.5 h. The reaction was quenched with saturated aqueous $NaHCO_3$ solution. The resultant mixture was extracted with EtOAc, and the organic layer was washed with brine, dried (Na_2SO_4), filtered, and concentrated under reduced pressure. Purification of the residue by flash column chromatography (silica gel, 3% EtOAc/hexanes) gave silyl ether **S2** (199 mg, 98%) as a colorless oil: $[\alpha]_D^{24}$ +24.0 (*c* 1.00, $CHCl_3$); IR (film) 2955, 2875, 1513, 1247, 1084, 740 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 7.25–7.22 (m, 2H), 6.86–6.83 (m, 2H), 4.73 (s, 1H), 4.68 (s, 1H), 4.46 (d, *J* = 11.0 Hz, 1H), 4.34 (d, *J* = 11.0 Hz, 1H), 4.02 (dddd, *J* = 8.3, 8.2, 3.2, 1.9 Hz, 1H), 3.78 (s, 3H), 3.56 (ddt, *J* = 8.7, 5.9, 3.2 Hz, 1H), 2.24 (dd, *J* = 13.3, 4.6 Hz, 1H), 2.12 (dd, *J* = 13.3, 7.3 Hz, 1H), 1.75–1.69 (m, 4H), 1.60–1.53 (m, 1H), 1.49–1.32 (m, 4H), 0.93 (t, *J* = 7.7 Hz, 9H), 0.90 (t, *J* = 7.3 Hz, 3H), 0.57 (q, *J* = 7.7 Hz, 6H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 158.9, 142.6, 131.4, 129.0 (2C), 113.7, 112.9, 75.8, 69.9, 68.1, 55.2, 47.1, 42.1, 36.3, 22.9, 18.3, 14.3, 7.0 (3C), 5.2 (3C) ; HRMS (ESI) *m/z* calcd for $C_{24}H_{42}O_3Si$ [(M + Na)$^+$] 429.2795, found 429.2794.

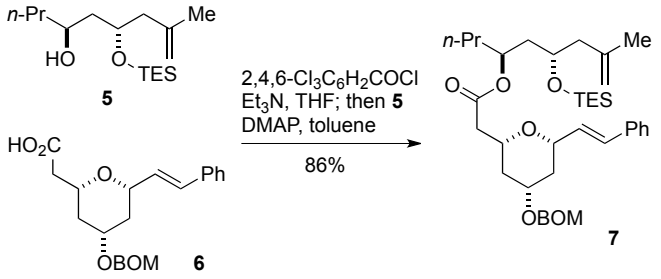


S2 5

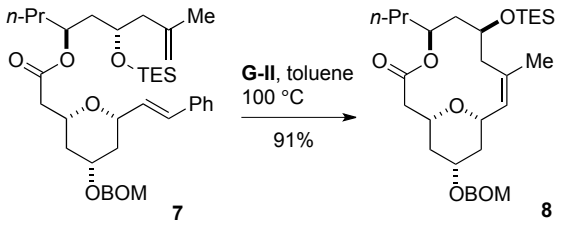
Alcohol 5. To a solution of silyl ether **S2** (189 mg, 0.466 mmol) in CH_2Cl_2/pH 7 buffer (10:1, v/v, 5 mL) at 0 °C was added DDQ (116 mg, 0.511 mmol), and the resultant mixture was stirred at room temperature for 30 min. The reaction was quenched with saturated aqueous $NaHCO_3$ solution. The resultant mixture was extracted with EtOAc, and the organic layer was washed with brine, dried (Na_2SO_4), filtered, and concentrated under reduced pressure. Purification of the residue by flash column chromatography (silica gel, 2% EtOAc/hexanes) gave alcohol **5** (127 mg, 95%) as a colorless oil: $[\alpha]_D^{24}$ +1.2 (*c* 1.00, $CHCl_3$); IR (film) 3454, 2956, 2876, 1457, 1414, 1377, 1239, 1080, 1005, 890, 742 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 4.76 (s, 1H), 4.69 (s, 1H), 4.17 (ddd, *J* = 13.7, 8.7, 4.1 Hz, 1H), 3.97 (m, 1H), 3.40 (d, *J* =

[1] H. Fuwa, M. Kawakami, K. Noto, T. Muto, Y. Suga, K. Konoki, M. Yotsu-Yamashita and M. Sasaki, *Chem. Eur. J.*, 2013, **19**, 8100.

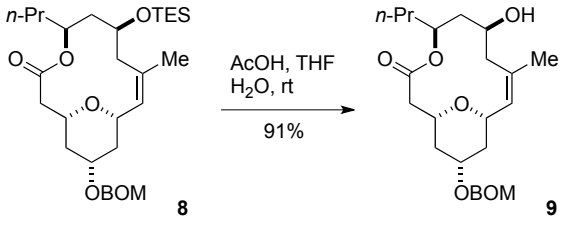
2.3 Hz, 1H), 2.33 (dd, *J* = 13.7, 8.3 Hz, 1H), 2.29 (dd, *J* = 13.7, 5.5 Hz, 1H), 1.69 (s, 3H), 1.61–1.51 (m, 2H), 1.48–1.27 (m, 4H), 0.95 (t, *J* = 8.3 Hz, 9H), 0.89 (t, *J* = 7.3 Hz, 3H), 0.61 (q, *J* = 8.3 Hz, 6H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 142.1, 113.3, 69.9, 67.8, 44.9, 40.7, 40.1, 22.6, 18.7, 14.0, 6.8 (3C), 4.8 (3C); HRMS (ESI) *m/z* calcd for $C_{16}H_{34}O_2Si$ [(M + Na)$^+$] 309.2220, found 309.2243.



Diene 7. To a solution of carboxylic acid **6** (37.0 mg, 0.0968 mmol) in THF (2 mL) at 0 °C were added Et_3N (0.08 mL, 0.6 mmol) and 2,4,6-$Cl_3C_6H_2COCl$ (0.045 mL, 0.28 mmol), and the resultant mixture was stirred at room temperature for 1.5 h. The resultant mixture was concentrated under reduced pressure and taken up in toluene (0.6 mL). To this mixture was added a solution of alcohol **5** (28.3 mg, 0.098 mmol) and DMAP (40 mg, 0.33 mmol) in toluene (1 mL + 0.4 mL rinse), and the resultant mixture was stirred at room temperature for 30 min. The reaction mixture was diluted with EtOAc, washed with H_2O and brine, dried (Na_2SO_4), filtered, and concentrated under reduced pressure. Purification of the residue by flash column chromatography (silica gel, 4 to 10% EtOAc/hexanes) gave diene **7** (54.3 mg, 86%) as a colorless oil: $[\alpha]_D^{24}$ +12.0 (*c* 1.00, $CHCl_3$); IR (film) 2955, 2875, 1731, 1455, 1376, 1247, 1040, 744 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.36–7.32 (m, 6H), 7.29–7.26 (m, 3H), 7.20 (m, 1H), 6.54 (d, *J* = 15.6 Hz, 1H), 6.16 (dd, *J* = 15.6, 5.9 Hz, 1H), 5.02 (ddt, *J* = 9.1, 5.9, 2.7 Hz, 1H), 4.83 (d, *J* = 8.7 Hz, 1H), 4.82 (d, *J* = 8.7 Hz, 1H), 4.75 (s, 1H), 4.68 (s, 1H), 4.61 (s, 2H), 4.02 (m, 1H), 3.92–3.79 (m, 3H), 2.63 (dd, *J* = 15.1, 7.8 Hz, 1H), 2.43 (dd, *J* = 15.1, 5.4 Hz, 1H), 2.25 (dd, *J* = 13.2, 4.5 Hz, 1H), 2.13–2.08 (m, 3H), 1.75 (ddd, *J* = 14.7, 9.2, 2.3 Hz, 1H), 1.69 (s, 3H), 1.58–1.38 (m, 4H), 1.32–1.23 (m, 3H), 0.93 (t, *J* = 7.8 Hz, 9H), 0.83 (t, *J* = 7.3 Hz, 3H), 0.57 (q, *J* = 7.3 Hz, 6H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 170.7, 142.3, 137.7, 136.7, 130.4, 129.2, 128.4 (5C), 127.9 (2C), 127.8, 127.6, 126.4 (2C), 113.0, 92.4, 76.0, 72.5 (2C), 72.2, 69.5, 67.6, 46.9, 41.6, 41.2, 38.3, 37.9, 37.1, 22.9, 18.4, 13.9, 6.9 (3C), 5.1 (3C); HRMS (ESI) *m/z* calcd for $C_{39}H_{58}O_6Si$ [(M + Na)$^+$] 673.3895, found 673.3911.

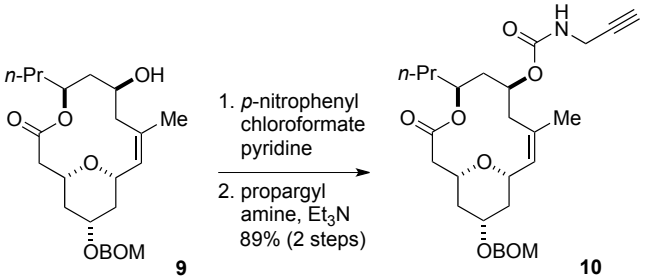


Macrocycle 8. To a solution of diene **7** (112 mg, 0.172 mmol) and 1,4-benzoquinone (16 mg, 0.15 mmol) in toluene (19.5 mL) at 100 °C was added a solution of the second-generation Grubbs catalyst (**G-II**) (44.0 mg, 0.0518 mmol) in toluene (6 mL) over a period of 6 h. The resultant solution was stirred at 100 °C for 15 h. The reaction mixture was cooled to room temperature and then exposed to air with stirring for 1 h. The resultant mixture was concentrated under reduced pressure. Purification of the residue by flash column chromatography (silica gel, first round: 5 to 10% EtOAc/hexanes; second round: 1 to 20% Et_2O/benzene) gave macrocycle **8** (85.5 mg, 91%) as a yellow oil: $[\alpha]_D^{24}$ –38.9 (*c* 1.00, $CHCl_3$); IR (film) 2953, 2875, 1732, 1455, 1374, 1264, 1203, 1039, 738 cm^{-1} ; ^1H NMR (600 MHz, $CDCl_3$) δ 7.35–7.26 (m, 5H), 5.28 (m, 1H), 5.21 (d, *J* = 5.1 Hz, 1H) , 4.80 (s, 2H), 4.59 (s, 2H), 3.86–3.79 (m, 4H), 2.54 (dd, *J* = 15.1, 3.6 Hz, 1H), 2.45 (dd, *J* = 15.1, 8.3 Hz, 1H), 2.33 (d, *J* = 12.8 Hz, 1H), 2.02–1.92 (m, 3H), 1.80 (s, 3H), 1.78–1.75 (m, 2H), 1.59 (br s, 1H), 1.53–1.36 (m, 3H), 1.32–1.23 (m, 5H), 0.92 (t, *J* = 8.2 Hz, 9H), 0.89 (t, *J* = 7.4 Hz, 3H), 0.60 (q, *J* = 8.2 Hz, 6H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 170.2, 146.0, 137.8, 128.4 (2C), 127.8 (2C), 127.7, 125.2, 92.4, 73.6, 72.9, 72.8, 72.6, 71.7, 69.5, 46.5, 44.1, 42.0, 37.7, 37.6, 37.5, 25.3, 18.6, 13.9, 6.9 (3C), 5.1 (3C); HRMS (ESI) *m/z* calcd for $C_{31}H_{50}O_6Si$ [$(M + Na)^+$] 569.3269, found 569.3252.



Alcohol 9. To a solution of macrocycle **8** (72.3 mg, 0.132 mmol) in THF/H_2O (1:1, v/v, 1.76 mL) at 0 °C was added AcOH (2.64 mL), and the resultant solution was stirred at room temperature for 30 min. The reaction was quenched with saturated aqueous $NaHCO_3$ solution. The resultant mixture was extracted with EtOAc, and the organic layer was washed with brine, dried (Na_2SO_4), filtered, and concentrated under reduced pressure. Purification of the residue by flash column chromatography (silica gel, 15 to 30% EtOAc/hexanes) gave alcohol **9** (52.3 mg, 91%) as a colorless oil: $[\alpha]_D^{24}$ –30.4 (*c* 1.00, $CHCl_3$); IR (film) 3477, 2957, 2872, 1729,

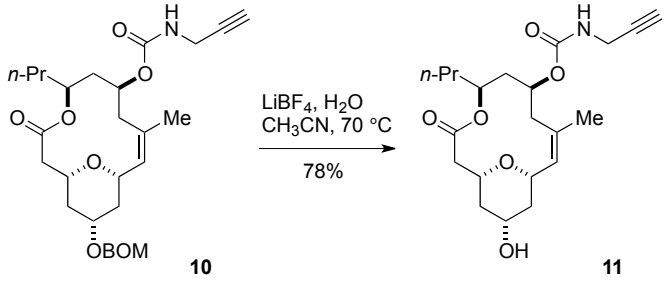
1453, 1373, 1266, 1202, 1025, 876, 737 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 7.35–7.26 (m, 5H), 5.29 (d, *J* = 6.9 Hz, 1H), 5.24 (m, 1H), 4.80 (s, 2H), 4.59 (s, 2H), 4.12 (br s, 1H), 3.86–3.78 (m, 3H), 2.57 (dd, *J* = 14.2, 3.2 Hz, 1H), 2.41 (dd, *J* = 14.2, 11.0 Hz, 1H), 2.25 (d, *J* = 13.3 Hz, 1H), 2.23 (br s, 1H), 2.07–2.01 (m, 2H), 1.98 (m, 1H), 1.84 (s, 3H), 1.78–1.65 (m, 2H), 1.57–1.42 (m, 3H), 1.37–1.28 (m, 3H), 0.88 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 170.6, 145.9, 137.7, 128.4 (2C), 127.8 (2C), 127.7, 125.3, 92.5, 72.9 (2C), 72.7, 72.6, 71.7, 69.5, 44.9, 42.1, 41.8, 37.9, 37.4, 37.0, 25.9, 18.6, 13.8 ; HRMS (ESI) *m/z* calcd for $C_{25}H_{36}O_6$ [$(M + Na)^+$] 455.2404, found 455.2426.



Alkyne 10. To a solution of alcohol **9** (52.3 mg, 0.121 mmol) in CH_2Cl_2 (4 mL) at 0 °C were added pyridine (0.1 mL, 1 mmol) and *p*-nitrophenyl chloroformate (48.8 mg, 0.242 mmol), and the resultant mixture was stirred at room temperature for 30 min. The reaction mixture was diluted with EtOAc and washed with H_2O and brine, dried (Na_2SO_4), filtered, and concentrated under reduced pressure. Purification of the residue by flash column chromatography (silica gel, 10 to 30% EtOAc/hexanes) gave an activated ester (67.8 mg, 94%) as a colorless oil, which was immediately used in the next reaction.

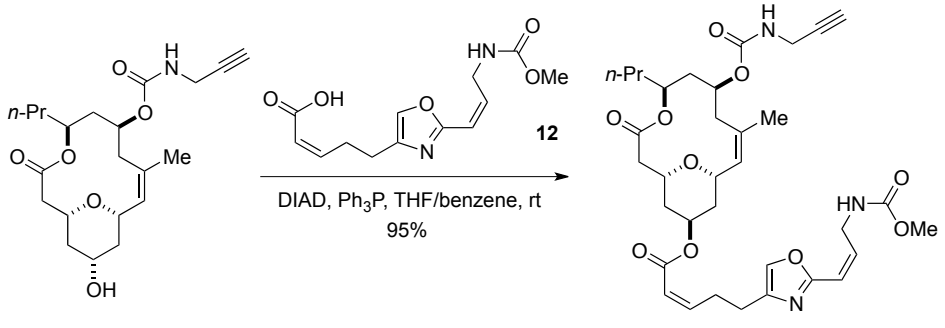
To a solution of the above activated ester (67.8 mg, 0.114 mmol) in DMF (3.8 mL) at 0 °C were added Et_3N (0.08 mL, 0.6 mmol) and propargyl amine (0.015 mL, 0.23 mmol), and the resultant solution was stirred at room temperature for 3 h. The reaction was quenched with saturated aqueous NH_4Cl solution. The resultant mixture was extracted with Et_2O, and the organic layer was washed with brine, dried ($MgSO_4$), filtered, and concentrated under reduced pressure. Purification of the residue by flash column chromatography (silica gel, 15 to 30% EtOAc/hexanes) gave alkyne **10** (55.7 mg, 95%) as a colorless oil: $[\alpha]_D^{24}$ –45.5 (*c* 1.00, $CHCl_3$); IR (film) 3303, 2956, 2872, 1725, 1517, 1453, 1377, 1317, 1245, 1037, 738 cm^{-1} ; ^1H NMR (600 MHz, $CDCl_3$) δ 7.35–7.26 (m, 5H), 5.45 (m, 1H), 5.27 (d, *J* = 6.0 Hz, 1H), 4.80 (s, 2H), 4.71 (br s, 1H), 4.59 (s, 2H), 3.95 (s, 2H), 3.89–3.79 (m, 3H), 2.55 (dd, *J* = 15.6, 4.1 Hz, 1H), 2.51 (dd, *J* = 15.6, 10.5 Hz, 1H), 2.47 (d, *J* = 12.7 Hz, 1H), 2.19 (t, *J* = 2.7 Hz, 1H), 2.13 (t, *J* = 11.4 Hz, 1H), 2.03–1.76 (m, 4H), 1.71 (s, 3H), 1.64–1.60 (m, 2H), 1.54–1.38 (m, 3H), 1.32–1.23 (m, 3H), 0.87 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 169.6, 155.2,

143.8, 137.7, 128.4 (2C), 127.8 (2C), 127.7, 126.4, 92.5, 79.8, 73.3, 72.8, 72.2, 71.5, 71.4, 69.5, 43.1, 41.9, 40.1, 37.5 (2C), 37.2, 30.7, 24.5, 18.5, 13.9; HRMS (ESI) *m/z* calcd for $C_{29}H_{39}NO_7$ [(M + Na)$^+$] 536.2619, found 536.2600.



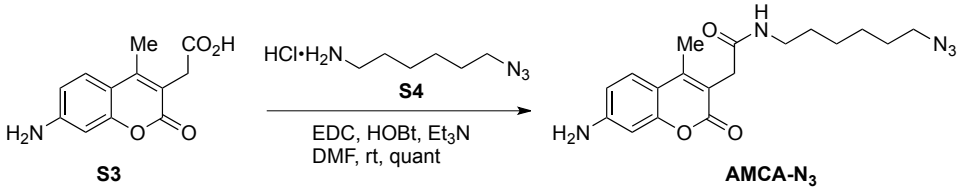
10 11

Alcohol 11. To a solution of alkyne **9** (55.6 mg, 0.108 mmol) in CH_3CN (3 mL) was added a solution of $LiBF_4$ (304 mg, 3.24 mmol) in H_2O (0.3 mL), and the resultant mixture was stirred at 70 °C for 13 h. The reaction mixture was cooled to room temperature, diluted with H_2O, and extracted with EtOAc. The organic layer was washed with brine, dried ($MgSO_4$), filtered, and concentrated under reduced pressure. Purification of the residue by flash column chromatography (silica gel, 50% EtOAc/hexanes to 100% EtOAc) gave alcohol **11** (33 mg, 78%) as a colorless oil: $[\alpha]_D^{24}$ –57.0 (*c* 1.00, $CHCl_3$); IR (film) 3303, 2957, 2921, 2854, 1715, 1524, 1446, 1375, 1258, 1147, 1034, 980 cm^{-1} ; ^{1}H NMR (600 MHz, $CDCl_3$) δ 5.94 (m, 1H), 5.27 (d, *J* = 5.0 Hz, 1H), 4.76 (s, 1H), 4.69 (d, *J* = 9.7 Hz, 1H), 3.93 (s, 2H), 3.90–3.78 (m, 3H), 2.57 (dd, *J* = 16.0, 3.7 Hz, 1H), 2.50 (dd, *J* = 16.0, 10.5 Hz, 1H), 2.48 (d, *J* = 15.0 Hz, 1H), 2.19 (t, *J* = 2.3 Hz, 1H), 2.13 (t, *J* = 11.9 Hz, 1H), 1.99–1.90 (m, 2H), 1.88 (dt, *J* = 15.6, 3.2 Hz, 1H), 1.85–1.75 (m, 2H), 2.51–2.40 (m, 2H), 1.71 (s, 3H), 1.49 (td, *J* = 15.2, 7.3 Hz, 1H), 1.44–1.36 (m, 2H), 1.28 (td, *J* = 14.6, 7.3 Hz, 2H), 1.20 (q, *J* = 11.4 Hz, 1H), 0.86 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 169.7, 155.3, 143.7, 128.3, 126.4 , 79.8, 73.3, 72.2, 71.5, 68.0, 43.1, 41.8, 40.3, 40.2, 40.1, 37.2, 30.7, 24.6, 18.5, 13.9; HRMS (ESI) *m/z* calcd for $C_{21}H_{31}NO_6$ [(M + Na)$^+$] 416.2044, found 416.2050.



11 2

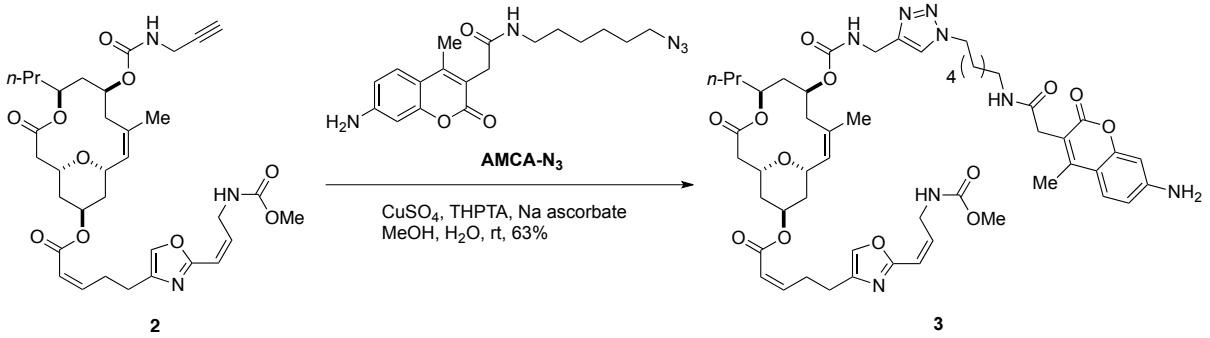
Common precursor 2. To a solution of alcohol **11** (29.7 mg, 0.0754 mmol) and carboxylic acid **12** (56.0 mg, 0.200 mmol) in THF/benzene (1:30, v/v, 3.1 mL) were added Ph_3P (75.9 mg, 0.289 mmol) and DIAD (1.9 M solution in toluene, 0.15 mL, 0.29 mmol), and the resultant mixture was stirred at room temperature for 1.5 h. To the reaction mixture were added additional portions of Ph_3P (38 mg, 0.14 mmol) and DIAD (1.9 M solution in toluene, 0.075 mL, 0.14 mmol), and the resultant mixture was stirred at room temperature for 1 h before it was concentrated under reduced pressure. Purification of the residue by flash column chromatography (silica gel, 40 to 80% EtOAc/hexanes) followed by preparative TLC (80% EtOAc/hexanes) gave common precursor **2** (47.0 mg, 95%) as a colorless oil: $[\alpha]_D^{24}$ –52.6 (*c* 1.00, CH_3OH); IR (film) 3583, 3303, 2957, 2922, 2853, 1714, 1519, 1436, 1317, 1247, 1175, 1073, 1036 cm^{-1}; ^1H NMR (600 MHz, CD_3OD) δ 7.65 (s, 1H), 6.37 (dt, *J* = 11.4, 7.4 Hz, 1H), 6.27 (dt, *J* = 11.9, 2.3 Hz, 1H), 6.03 (dt, *J* = 11.9, 6.4 Hz, 1H), 5.89 (dt, *J* = 11.4, 1.4 Hz, 1H), 5.46 (m, 1H), 5.24 (t, *J* = 3.2 Hz, 1H), 5.21 (d, *J* = 5.5 Hz, 1H), 4.66 (d, *J* = 10.1 Hz, 1H), 4.30 (d, *J* = 5.9 Hz, 2H), 4.29 (s, 1H), 4.18 (t, *J* = 11.4 Hz, 1H), 3.84 (s, 2H), 3.64 (s, 3H), 3.02 (d, *J* = 6.9 Hz, 1H), 3.00 (d, *J* = 6.9 Hz, 1H), 2.72 (t, *J* = 7.8 Hz, 2H), 2.67 (dd, *J* = 16.0, 3.2 Hz, 1H), 2.56 (d, *J* = 13.7 Hz, 1H), 2.54 (s, 1H), 2.34 (dd, *J* = 16.0, 11.5 Hz, 1H), 2.12 (t, *J* = 11.5 Hz , 1H), 1.87–1.70 (m, 8H), 1.57–1.43 (m, 3H), 1.32 (td, *J* = 15.1, 7.3 Hz, 2H), 0.92 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (150 MHz, CD_3OD) δ 172.0, 166.8, 161.8, 159.6, 158.2, 150.0, 145.6, 142.2, 139.2, 135.9, 127.6, 121.7, 115.9, 81.3, 78.0, 74.8, 71.8, 70.6, 70.4, 69.0, 52.5, 44.3, 42.9, 41.2, 41.0, 38.3, 36.0, 35.8, 30.9, 29.0, 26.3, 24.8, 19.6, 14.2; HRMS (ESI) *m/z* calcd for $C_{34}H_{45}N_3O_{10}$ [(M + H)$^+$] 656.3178, found 656.3164; calcd for $C_{34}H_{44}N_3O_{10}Na$ [(M + Na)$^+$] 678.2997, found 678.3021.



AMCA-N_3. To a solution of 7-amino-4-methylcoumarin-3-acetic acid (**S3**) (6.0 mg, 0.026 mmol) in DMF (0.9 mL) at 0 °C were added EDC (7.5 mg, 0.039 mmol) and HOBt (6.0 mg, 0.044 mmol), and the resultant mixture was stirred at 0 °C for 30 min. To the reaction mixture were added a solution of 6-azido-1-hexanamine hydrochloride salt (**S4**)[2] (30 mg, 0.17 mmol) in DMF (0.1 mL + 0.1 mL rinse) and Et_3N (0.020 mL, 0.14 mmol), and the resultant mixture

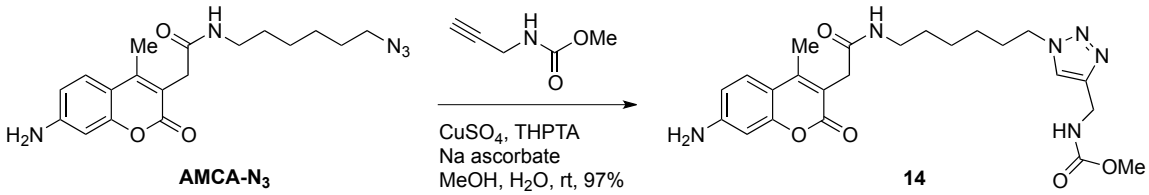
[2] P. M. Weerawarna, Y. Kim, A. C. G. Kankanamalage, V. C. Damalanka, G. H. Lushington, K. R. Alliston, N. Mehzabeen, K. P. Battaile, S. Lovell, K.-O. Chang and W. C. Groutas, *Eur. J. Med. Chem.*, 2016, **119**, 300.

was stirred at room temperature for 8.5 h. The reaction mixture was diluted with EtOAc and washed with H_2O and brine. The organic layer was dried ($MgSO_4$), filtered, and concentrated under reduced pressure. Purification of the residue by flash column chromatography (silica gel, 20 to 40% EtOAc/benzene) gave **AMCA-N$_3$** (9.2 mg, quant) as a yellow solid: ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.39 (d, *J* = 8.7 Hz, 1H), 6.57 (dd, *J* = 8.3, 2.3 Hz, 1H), 6.51 (d, *J* = 2.3 Hz, 1H), 6.45 (m, 1H), 4.02 (br s, 2H), 3.49 (s, 2H), 3.19 (dd, *J* = 6.8, 6.9 Hz, 2H), 3.15 (dd, *J* = 12.8, 6.8 Hz, 2H), 1.52 (ddd, *J* = 7.4, 7.4, 6.8 Hz, 2H), 1.45 (ddd, *J* = 7.4, 7.4, 6.8 Hz, 2H), 1.34–1.21 (m, 5H), three protons missing due to H/D exchange; ^{13}C NMR (150 MHz, $CDCl_3$) δ 170.2, 163.5, 154.1, 150.5, 150.0, 126.3, 115.2, 112.2, 112.0, 100.8, 51.3, 39.4, 35.8, 29.3, 28.7, 26.2 (2C), 15.4; HRMS (ESI) *m/z* calcd for $C_{18}H_{23}N_5O_3$ [(M + H)$^+$] 358.1874, found 358.1877.

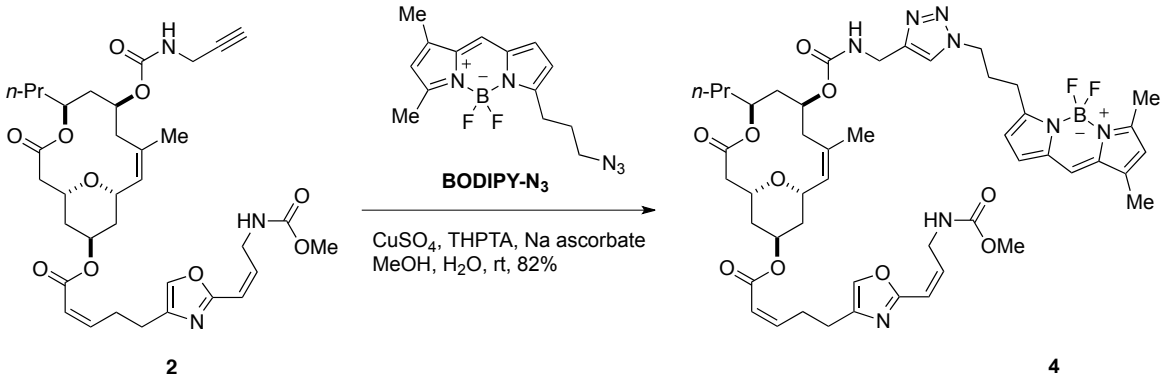


AMCA derivative 3. To a solution of common precursor **2** (6.4 mg, 0.0097 mmol) and **AMCA-N$_3$** (5.2 mg, 0.015 mmol) in MeOH (0.97 mL) were added 0.2 M aqueous sodium ascorbate solution (0.097 mL, 0.019 mmol), 0.1 M aqueous $CuSO_4$ solution (0.097 mL, 0.0097 mmol), and a solution of THPTA (0.1 M solution in MeOH, 0.097 mL, 0.0097 mmol), and the resultant mixture was stirred at room temperature for 9 h 40 min. The reaction mixture was concentrated under reduced pressure, and the residue was diluted with EtOAc, washed with H_2O and brine, dried ($MgSO_4$), filtered, and concentrated under reduced pressure. Purification of the residue by flash column chromatography (silica gel, 80% EtOAc/hexanes to EtOAc to 20% MeOH/EtOAc) gave AMCA derivative **3** (6.2 mg, 63%) as a colorless solid. This material was further purified by preparative reverse-phase HPLC (Cosmosil 5C18 AR-II column: 20 mm I.D. × 250 mm; eluent: 55% CH_3CN/H_2O; flow rate: 8 mL/min; UV detection: 254 nm) before its use in biological experiments. Data for **3**: $[\alpha]_D^{24}$ –34.0 (*c* 0.5, CH_3OH); ^{1}H NMR (600 MHz, CD_3OD) δ 7.84 (s, 1H), 7.64 (s, 1H), 7.49 (d, *J* = 8.7 Hz, 1H), 6.65 (dd, *J* = 8.7, 2.3 Hz, 1H), 6.50 (d, *J* = 2.2 Hz, 1H), 6.36 (ddd, *J* = 14.6, 11.0, 7.3 Hz, 1H), 6.26 (ddd *J* = 11.9, 2.3, 1.8 Hz, 1H), 6.03 (ddd, *J* = 11.9, 5.9, 5.9 Hz, 1H), 5.89 (d, *J* = 11.4 Hz, 1H), 5.43 (m, 1H), 5.23 (m, 1H), 5.17 (d, *J* = 5.04, 1H), 4.70 (m, 1H), 4.60 (s, 2H), 4.35

(dd, J = 7.3, 6.8 Hz, 2H), 4.33–4.26 (m, 5H), 4.18 (dd, J = 12.4, 10.6 Hz, 1H), 3.64 (s, 3H), 3.54 (s, 2H), 3.17 (dd, J = 6.9, 6.8 Hz, 2H), 3.01 (dd, J = 14.6, 7.8 Hz, 2H), 2.71 (dd, J = 7.3, 7.3 Hz, 2H), 2.67 (dd, J = 16.1, 3.2 Hz, 1H), 2.54 (d, J = 13.7 Hz, 1H), 2.39 (s, 3H), 2.32 (dd, J = 15.6, 11.5 Hz, 1H), 2.09 (dd, J = 13.7, 11.5 Hz, 1H), 1.90–1.75 (m, 6H), 1.69 (s, 3H), 1.55–1.27 (m, 11H), 0.90 (dd, J = 7.3, 7.3 Hz, 3H), five protons missing due to H/D exchange; ^{13}C NMR (150 MHz, CD_3OD) δ 173.0, 172.0, 166.8, 164.8, 161.9, 158.5, 155.9, 154.0, 152.9, 150.0, 145.7, 142.2, 139.2, 135.9, 127.6, 127.4, 124.2, 121.7, 115.9, 114.5, 113.2, 111.8, 101.3, 100.5, 77.8, 74.8, 70.6, 70.5, 69.0, 52.6, 51.1, 49.8, 49.5, 44.3, 42.9, 41.2, 41.0, 40.3, 38.3, 37.0, 36.0, 35.8, 35.2, 31.1, 30.1, 27.1, 27.0, 26.4, 24.9, 19.7, 15.4, 14.3; HRMS (ESI) *m/z* calcd for $C_{52}H_{68}N_8O_{13}$ [(M + H)$^+$] 1013.4979, found 1013.4960.

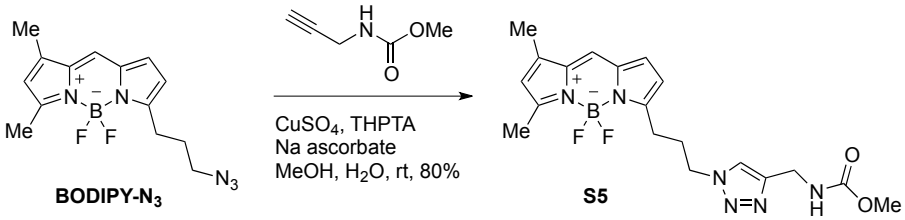


AMCA derivative 14. To a solution of **AMCA-N$_3$** (0.90 mg, 0.0025 mmol) and methyl *N*-(prop-2-yn-1-yl)carbamate (3.1 mg, 0.027 mmol) in MeOH (0.5 mL) were added 0.2 M aqueous sodium ascorbate solution (0.025 mL, 0.0050 mmol), 0.1 M aqueous $CuSO_4$ solution (0.025 mL, 0.0025 mmol), and a solution of THPTA (0.1 M solution in MeOH, 0.025 mL, 0.0025 mmol), and the resultant mixture was stirred at room temperature for 10 min. The reaction mixture was concentrated under reduced pressure. Purification of the residue by flash column chromatography (silica gel, 50% EtOAc/hexanes to EtOAc to 10% MeOH/EtOAc) gave AMCA derivative **14** (1.15 mg, 97%) as a yellow solid. This material was further purified by preparative reverse-phase HPLC (Cosmosil 5C18 AR-II column: 20 mm I.D. × 250 mm; eluent: 30% CH_3CN/H_2O; flow rate: 8 mL/min; UV detection: 254 nm) before its use in biological experiments. Data for **14**: ^{1}H NMR (600 MHz, CD_3OD) δ 7.81 (s, 1H), 7.50 (d, J = 8.7 Hz, 1H), 6.65 (dd, J = 8.7, 2.3 Hz, 1H), 6.50 (d, J = 2.3 Hz, 1H), 4.35 (dd, J = 7.3, 7.3 Hz, 2H), 4.34 (s, 2H), 3.64 (s, 3H), 3.53 (s, 2H), 3.16 (dd, J = 6.8, 6.8 Hz, 2H), 2.38 (s, 3H), 1.87 (dddd, J = 7.3, 7.3, 7.3, 6.9 Hz, 2H), 1.49 (dddd, J = 7.3, 7.3, 6.9, 6.9 Hz, 2H), 1.39–1.28 (m, 4H), four protons missing due to H/D exchange; ^{13}C NMR (150 MHz, CD_3OD) δ 173.0, 164.9, 161.9, 155.9, 154.0, 152.9, 127.4, 124.0, 114.5, 113.2, 111.8, 100.4, 52.6, 51.2, 40.3, 37.1, 35.1, 31.1, 30.1, 27.0 (2C), 15.4, signal for the carbamate carbon missing; HRMS (ESI) *m/z* calcd for $C_{23}H_{30}N_6O_5$ [(M + H)$^+$] 471.2350, found 471.2373.



BODIPY derivative 4. To a solution of common precursor **2** (3.7 mg, 0.0056 mmol) and **BODIPY-N_3**[3] (8.6 mg, 0.028 mmol) in MeOH (0.56 mL) were added 0.2 M aqueous sodium ascorbate solution (0.056 mL, 0.011 mmol), 0.1 M aqueous $CuSO_4$ solution (0.056 mL, 0.0056 mmol), and a solution of THPTA (0.1 M solution in MeOH, 0.056 mL, 0.0056 mmol), and the resultant mixture was stirred at room temperature overnight. The reaction mixture was concentrated under reduced pressure. Purification of the residue by flash column chromatography (silica gel, 40% EtOAc/hexanes to EtOAc) gave BODIPY derivative **4** (4.4 mg, 82%) as a red oil. This material was further purified by preparative reverse-phase HPLC (Cosmosil 5C18 AR-II column: 20 mm I.D. × 250 mm; eluent: 65% CH_3CN/H_2O; flow rate: 8 mL/min; UV detection: 254 nm) before its use in biological experiments. Data for **4**: $[\alpha]_D^{24}$ –47.5 (*c* 0.25, CH_3OH); ^{1}H NMR (600 MHz, CD_3OD) δ 7.86 (s, 1H), 7.64 (s, 1H), 7.44 (s, 1H), 7.01 (d, *J* = 4.1 Hz, 1H), 6.36 (ddd, *J* =14.6, 9.3, 7.3Hz, 1H), 6.34 (d, *J* = 4.1 Hz, 1H), 6.26 (ddd, *J* = 11.9, 2.3, 1.8 Hz, 1H), 6.21 (s, 1H), 6.0 (dd, *J* = 11.9, 5.9 Hz, 1H), 5.89 (ddd, *J* = 11.4, 1.8, 1.4 Hz, 1H), 5.40 (m 1H), 5.23 (dd, *J* = 2.8, 2.8 Hz, 1H), 5.16 (dd, *J* = 5.9, 1.4 Hz, 1H), 4.71 (m, 1H), 4.45 (dd, *J* = 6.9, 6.8 Hz, 2H), 4.31–4.25 (m, 5H), 4.17 (dd, *J* = 11.9, 10.1 Hz, 1H), 3.64 (s, 3H), 3.00 (dd, *J* = 14.6, 7.3 Hz, 2H), 2.94 (dd, *J* = 7.4, 7.7 Hz, 2H), 2.71 (dd, *J* = 7.8, 7.3 Hz, 2H), 2.65 (dd, *J* = 15.6, 3.2, 1H), 2.53 (d, *J* = 13.7, 1H), 2.51 (s, 3H), 2.34–2.30 (m, 3H), 2.28 (s, 3H), 2.10 (dd, *J* = 13.7, 13.8 Hz, 1H), 1.84–1.64 (m, 5H), 1.66 (s, 3H), 1.54–1.46 (m, 2H), 1.42 (m, 1H), 1.28 (m, 2H), 0.87 (dd, *J* = 7.4, 7.3 Hz, 3H), two protons missing due to H/D exchange; ^{13}C NMR (150 MHz, CD_3OD) δ 172.0, 166.8, 161.8, 161.4, 159.6, 158.5, 158.2, 149.9, 147.1, 145.9, 145.7, 142.2, 139.2, 136.5, 135.9, 134.8, 129.7, 127.6, 125.8, 124.3, 121.7, 121.4, 117.7, 115.9, 77.7, 74.8, 70.6, 70.5, 69.0, 52.6, 50.9, 44.3, 42.9, 41.2, 41.0, 38.3, 37.0, 36.0, 35.8, 30.7, 29.0, 26.5, 26.4, 24.9, 19.7, 14.9, 14.2, 11.2; HRMS (ESI) *m/z* calcd for $C_{48}H_{61}BF_2N_8O_{10}$ [$(M + H)^+$] 959.8763, found 959.8774

[3] A. M. Hansen, A. L. Sewell, R. H. Pedersen, D.-L. Long, N. Gadegaard and R. Marquez, *Tetrahedron*, 2014, **69**, 8527.



BODIPY derivative S5. To a solution of **BODIPY-N_3** (5.0 mg, 0.016 mmol) and methyl *N*-(prop-2-yn-1-yl)carbamate (5.0 mg, 0.044 mmol) in MeOH (0.4 mL) were added 0.2 M aqueous sodium ascorbate solution (0.033 mL, 0.0066 mmol), 0.1 M aqueous $CuSO_4$ solution (0.033 mL, 0.0033 mmol), and a solution of THPTA (0.1 M solution in MeOH, 0.033 mL, 0.0033 mmol), and the resultant mixture was stirred at room temperature for 9.5 h. The reaction mixture was concentrated under reduced pressure. Purification of the residue by flash column chromatography (silica gel, 30 to 60% EtOAc/hexanes to EtOAc) gave BODIPY derivative **S5** (5.5 mg, 80%) as a red oil. This material was further purified by preparative reverse-phase HPLC (Cosmosil 5C18 AR-II column: 20 mm I.D. × 250 mm; eluent: 45% CH_3CN/H_2O; flow rate: 8 mL/min; UV detection: 254 nm) before its use in biological experiments. Data for **S5**: ^{1}H NMR (600 MHz, CD_3OD) δ 7.85 (s, 1H), 7.43 (s, 1H), 7.01 (d, J = 3.7 Hz, 1H), 6.34 (d, J = 3.7 Hz, 1H), 6.21 (s, 1H), 4.46 (dd, J = 6.8, 6.8 Hz, 2H), 4.34 (s, 2H), 3.64 (s, 3H), 2.95 (dd, J = 7.8, 7.3 Hz, 2H), 2.50 (s, 3H), 2.33 (dddd, J = 7.3, 7.3, 7.3, 7.3 Hz, 2H), 2.29 (s, 3H), one proton missing due to H/D exchange; ^{13}C NMR (150 MHz, CD_3OD) δ 161.4, 159.5, 158.2, 146.8, 129.6, 125.8, 124.1, 121.4, 117.6, 52.6, 50.9, 37.1, 30.5, 26.5, 14.8, 11.2; HRMS (ESI) *m/z* calcd for $C_{19}H_{23}BF_2N_6O_2$ [(M + H)$^+$] 417.2016 , found 417.1996.

Fluorescence excitation and emission spectra of compounds 3 and 4

Fluorescence spectra were recorded for **3** and **4** as 1 μM solution in pH 7 phosphate buffer, pH 6 phosphate buffer or pH 5 acetate buffer. The excitation or emission spectra for an appropriate spectra window were recorded on FluoroMax-4 spectrofluorometer (HORIBA scientific) using the excitation scan or emission scan mode. Fluorescence excitation spectra for **3** were recorded with an emission at 480 nm and emission spectra were recorded with an excitation at 350 nm. Fluorescence excitation spectra for **4** were recorded with an emission at 580 nm and emission spectra were recorded with an excitation at 450 nm. Absolute photoluminescent quantum yield (PLQY) of **3** and **4** at 1 μM solution in pH 7 phosphate buffer was recorded by PMA-12 equipped with a "C9920-02G" Integrating Sphere (Hamamatsu photonics). The PLQY of **3** was 0.995 and **4** was 0.101.

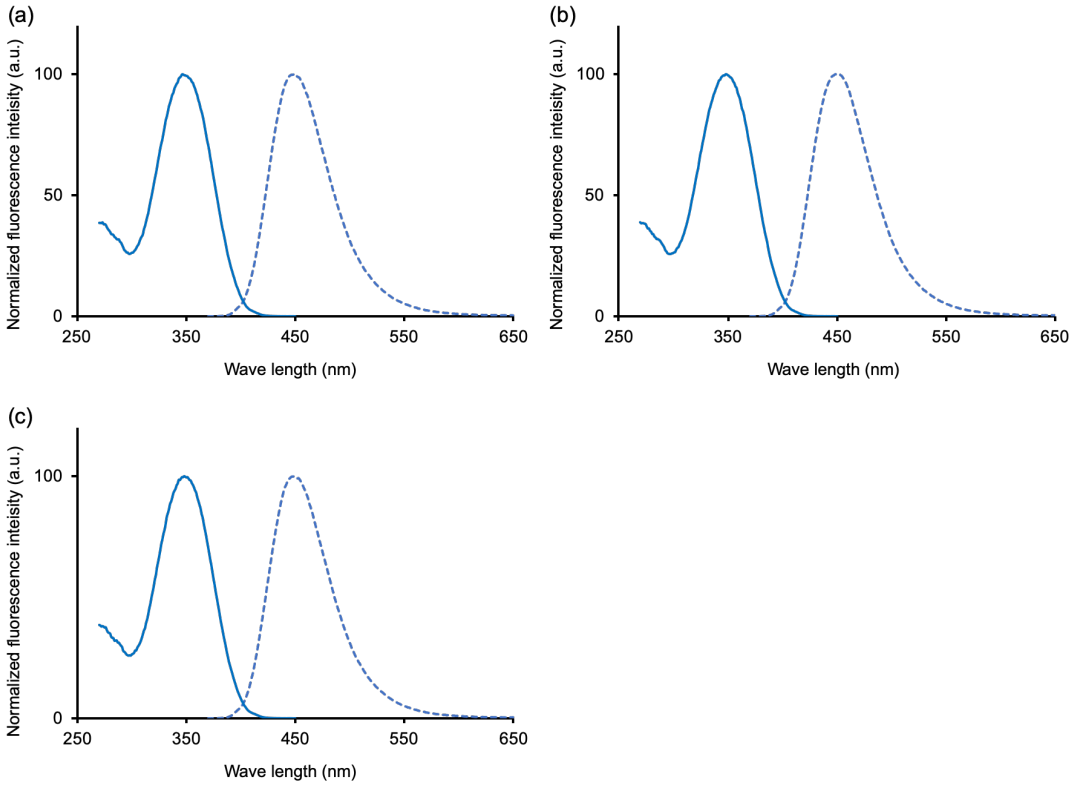


Fig. S1 Fluorescence excitation (single line) and emission spectra (dashed line) of **3** at 1 μM in (a) pH 7 phosphate buffer, (b) pH 6 phosphate buffer, and (c) pH 5 acetate buffer.

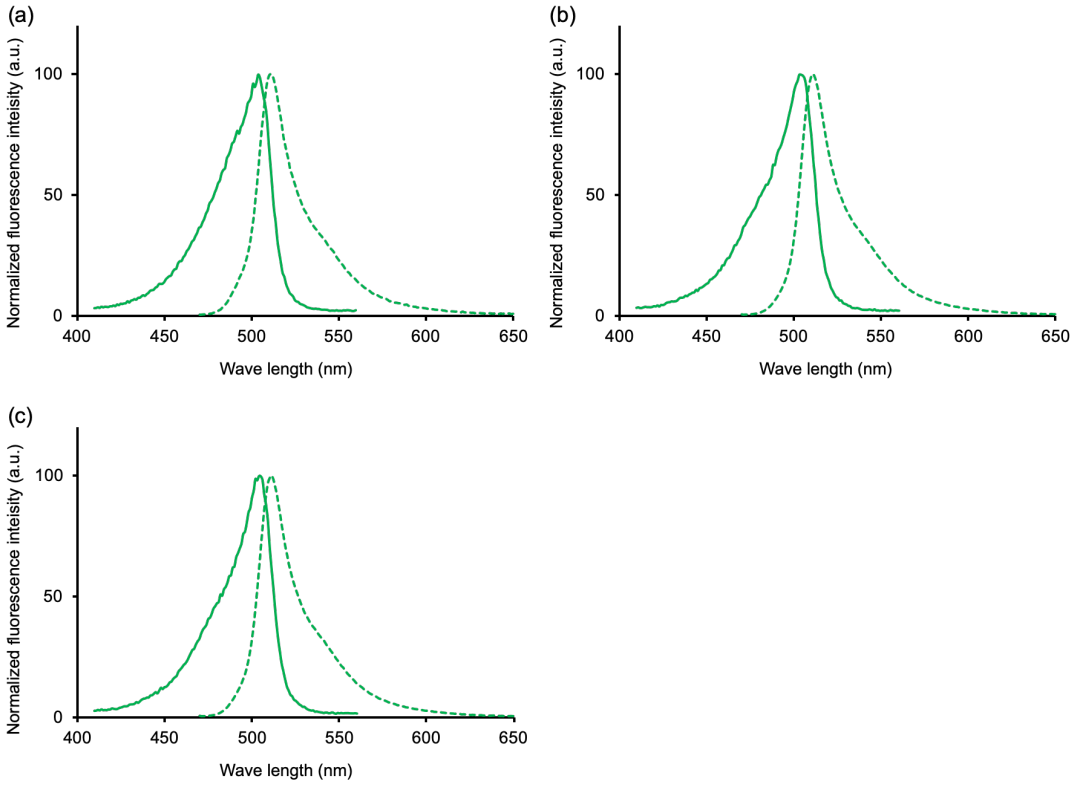


Fig. S2 Fluorescence excitation (single line) and emission spectra (dashed line) of **4** at 1 μM in (a) pH 7 phosphate buffer, (b) pH 6 phosphate buffer, and (c) pH 5 acetate buffer.

Determination of Log P values of final compounds

The Log P value of synthetic compounds was determined by reverse-phase HPLC using a Cosmosil 5C18 AR-II column (4.6 mm I.D. × 150 mm; eluent: 60% CH_3CN/H_2O; flow rate: 0.5 mL/min; UV detection: 254 nm). A calibration curve was generated using acetanilide (t_R 4.44 min; Log $P_{o/w}$ 1.0), acetophenone (t_R 6.30 min; Log $P_{o/w}$ 1.7), anisole (t_R 8.87 min; Log $P_{o/w}$ 2.1), toluene (t_R 13.2 min; Log $P_{o/w}$ 2.7), bromobenzene (t_R 14.6 min; Log $P_{o/w}$ 3.0), naphthalene (t_R 17.0 min; Log $P_{o/w}$ 3.6), and benzyl benzoate (t_R 18.6 min; Log $P_{o/w}$ 4.0) as reference compounds.

Biology

General remarks

A549 cells were obtained from RIKEN BioResource Center (BRC) and maintained in RPMI1640 medium containing 10% fetal bovine serum and penicillin/streptomycin at 37 °C in a humidified 5% CO_2/air atmosphere. MitoTracker Orange CMTMRos and ER-Tracker Red were obtained from Thermo Fisher Scientific Inc. (Waltham, MA, USA). All other reagents were purchased from Nacalai Tesque, Inc. (Kyoto, Japan). Spectrophotometric measurements were done using a BioTek Synergy HT multimode microplate reader. Cell imaging experiments were performed using a Zeiss confocal laser-scanning microscope LSM-700 equipped with a 63× oil immersion objective or an Olympus inverted microscope IX73 equipped with 20× and 40× dry objectives. Where appropriate, phenol red-free RPMI1640 medium was used for cell imaging experiments.

Complex III inhibition assay

A549 cells (5×10^7 cells) were harvested, washed with ice-cold PBS, and suspended in 800 μL of ice-cold MOPS buffer (250 mM sucrose, 20 mM MOPS, 3 mM EGTA). The suspension was gently pipetted, centrifuged (4 °C, 3,000 × *g*, 20 s), and the resultant supernatant was collected. This procedure was repeated three times. The combined supernatant was centrifuged (4 °C, 16,000 × *g*, 5 min) to give isolated mitochondria as a pellet, which was resupsended in 660 μL of ice-cold mitochondria resuspending buffer (250 mM mannitol, 5 mM HEPES, 0.5 mM EGTA). The total protein concentration was estimated by bicinchoninic acid (BCA) assay to be 0.098 mg/mL.

The inhibitory activity of synthetic compounds against the complex III (ubiquinol–cytochrome *c* reductase) of the mitochondrial electron transport chain was evaluated by following the reduction of cytochrome *c* spectrophotometrically using a 96-well microtiter plate. The suspension of isolated mitochondria (40 μL), 0.25 mM cytochrome *c* (40 μL), and double distilled water (80 μL) were added to each well. After addition of 20 mg/mL bovine serum albumin (1.0 μL/well), background absorbance (ex/em 550/580 nm) was measured every 30 s for 10 min at 30 °C. 100 mM *n*-dodecyl-β-D-maltoside (1.5 μL) and a compound solution in DMSO (2.0 μL) were then added to each well and incubated at room temperature for 5 min. The reaction was initiated by addition of a solution containing 0.25 mM decylubiquinol, 20 mM NaN_3, and 250 mM Tris-HCl buffer (40 μL). The absorbance of cytochrome *c* (ex/em 550/580 nm) was recorded every 30 s for 10 min at 30 °C.

WST-8 assay

A549 cells were harvested, pelleted, and suspended in RPMI1640 medium (2.5×10^5 cells/mL). The suspension (200 μL) was added to each well of a 96-well microtiter plate. Cells were cultured at 37 °C in a humidified 5% CO_2/air atmosphere overnight. On the next day, the medium was replaced with fresh RPMI1640 medium (99 μL), and a compound solution in DMSO (1 μL) was added to each well. Cells were incubated at 37 °C in a humidified 5% CO_2/air atmosphere for 96 h. After addition of WST-8 reagent (5 μL) to each well, cells were incubated at 37 °C in a humidified 5% CO_2/air atmosphere for several hours. The absorbance spectrum of WST-8 formazan was measured at room temperature.

Cell imaging experiments

Co-localization analysis (mitochondria):

A549 cells (8×10^3 cells) were seeded onto a 35-mm glass bottom dish and cultured in RPMI1640 medium (1,500 μL) at 37 °C in a humidified 5% CO_2/air atmosphere overnight. On the next day, the medium was replaced with fresh medium (1,000 μL). Cells were treated with MitoTracker Orange (10 nM) at 37 °C for 30 min, rinsed with phosphate buffer saline, and then treated with the AMCA derivative **3** (10 μM) or the BODIPY derivative **4** (1 μM) in RPMI1640 medium (1,000 μL) at 37 °C for 30 min. Cells were rinsed with PBS and then examined using a confocal laser-scanning microscope.

Co-localization analysis (endoplasmic reticulum (ER)):

A549 cells (8×10^3 cells) were seeded onto a 35-mm glass bottom dish and cultured in RPMI1640 medium (1,500 μL) at 37 °C in a humidified 5% CO_2/air atmosphere overnight. On the next day, the medium was replaced with fresh medium (1,000 μL). Cells were treated with the AMCA derivative **3** (10 μM) or the BODIPY derivative **4** (1 μM) in the presence of ER-Tracker Red (1 μM) in RPMI1640 medium (1,000 μL) at 37 °C for 30 min, rinsed with PBS, and then examined using a confocal laser-scanning microscope.

Competition experiments:

A549 cells (8×10^3 cells) were seeded onto a 35-mm glass bottom dish and cultured in RPMI1640 medium (1,500 μL) at 37 °C in a humidified 5% CO_2/air atmosphere overnight. On the next day, the medium was replaced with fresh medium (1,000 μL). Cells were treated with the AMCA derivative **3** (1 μM) or the BODIPY derivative **4** (10 nM) in RPMI1640 medium (1,000 μL) at 37 °C for 30 min, and then incubated with or without 8,9-dehydroneopeltolide **13** (1 μM for **3**, 10 nM for **4**) in RPMI1640 medium (1,000 μL) at

37 °C for 5 min. Cells were rinsed with PBS and examined with a confocal laser-scanning microscope.

ER morphology analysis:

A549 cells (8.0×10^3 cells/mL) were seeded onto a 35-mm glass bottom dish and cultured in RPMI1640 medium (1,500 μL) at 37 °C in a humidified 5% CO_2/air atmosphere overnight. On the next day, the medium was replaced with fresh medium (1,000 μL). Cells were treated with a solution of compound of interest (10 μL) in RPMI1640 medium (1,000 μL) at 37 °C for 1 or 24 h. Cells were rinsed with PBS thrice and treated with ER-Tracker Red (0.1 mM solution in DMSO, 10 μL) in RPMI1640 medium (1,000 μL) at 37 °C for 30 min. Finally, cells were rinsed with PBS thrice and examined with a confocal laser-scanning microscope.

Mitochondria morphology analysis:

A549 cells (8.0×10^3 cells) were seeded onto a 35-mm glass bottom dish and cultured in RPMI1640 medium (1,500 μL) at 37 °C in a humidified 5% CO_2/air atmosphere overnight. On the next day, the medium was replaced with fresh medium (1,000 μL). Cells were treated with MitoTracker Orange (10 μM solution in DMSO, 10 μL) at 37 °C for 15 min. Cells were rinsed with PBS thrice and treated with a solution of compound of interest (10 μL) in RPMI1640 medium (1,000 μL) at 37 °C for 1 h. Finally, cells were rinsed with PBS thrice and examined with a confocal laser-scanning microscope.

Mitochondrial membrane potential analysis (JC-1 assay):

A549 cells (1.0×10^5 cells) were seeded onto a 35-mm dish and cultured in RPMI1640 medium (1,000 μL) at 37 °C in a humidified 5% CO_2/air atmosphere overnight. On the next day, the medium was replaced with fresh medium (800 μL). Cells were treated with a solution of compound of interest (8 μL) at 37 °C for 1 h. Cells were rinsed with PBS thrice and treated with JC-1 dye (250 μg/mL solution in DMSO, 8 μL) in HBSS (800 μL) for 30 min. Finally, cells were rinsed with PBS thrice and examined with an inverted microscope.

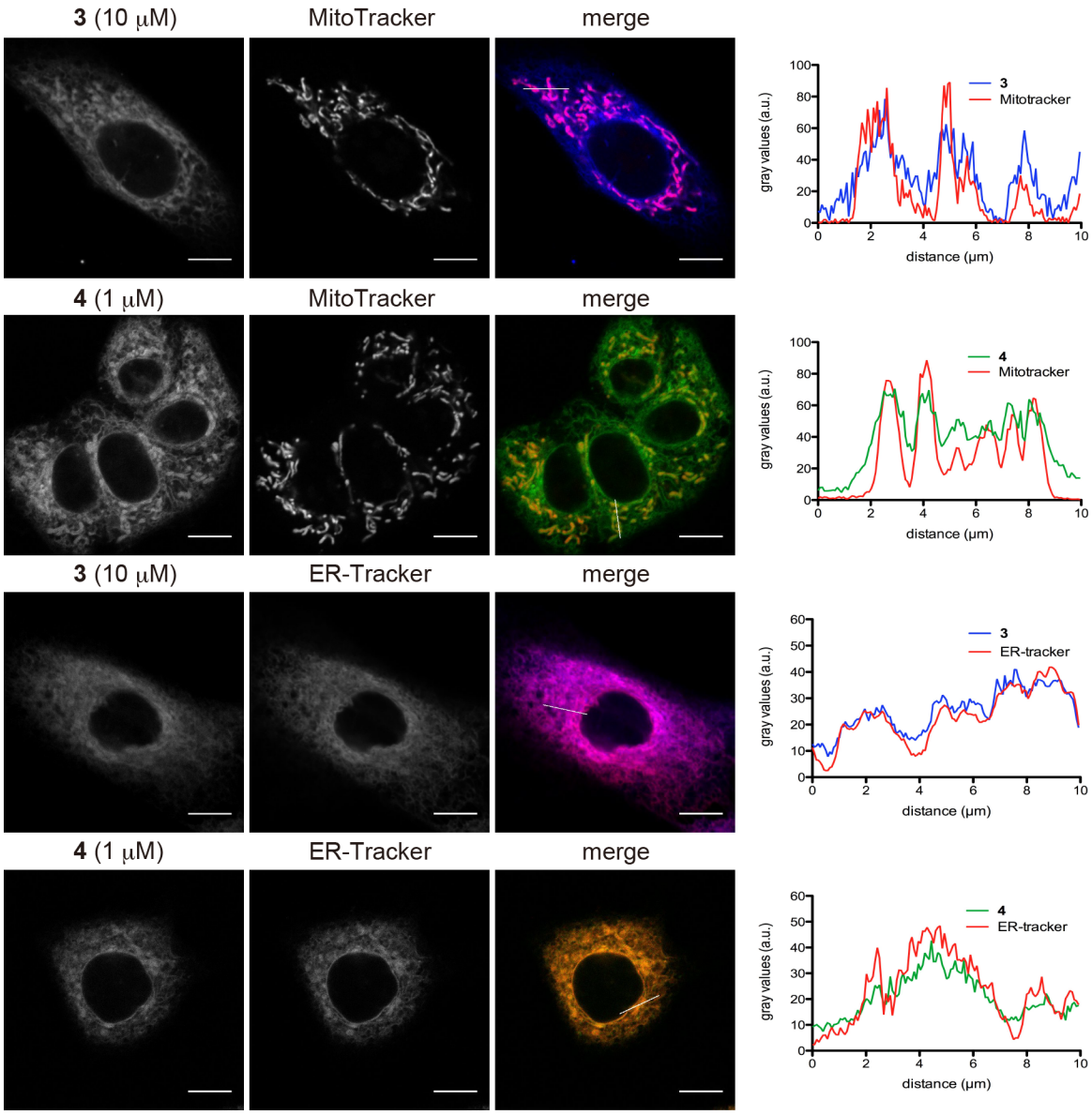
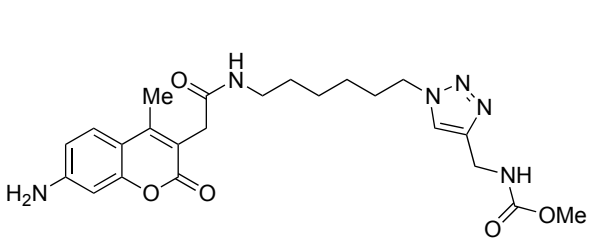


Fig. S3. Colocalization analysis of the AMCA derivative **3** and the BODIPY derivative **4** in A549 cells co-stained with MitoTracker Orange or ER-Tracker Red. The graphs next to the merged photographs represent the fluorescence distribution determined for indicated sections of the cell.

(a)



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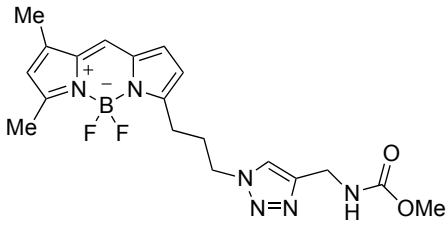


S5

(b)

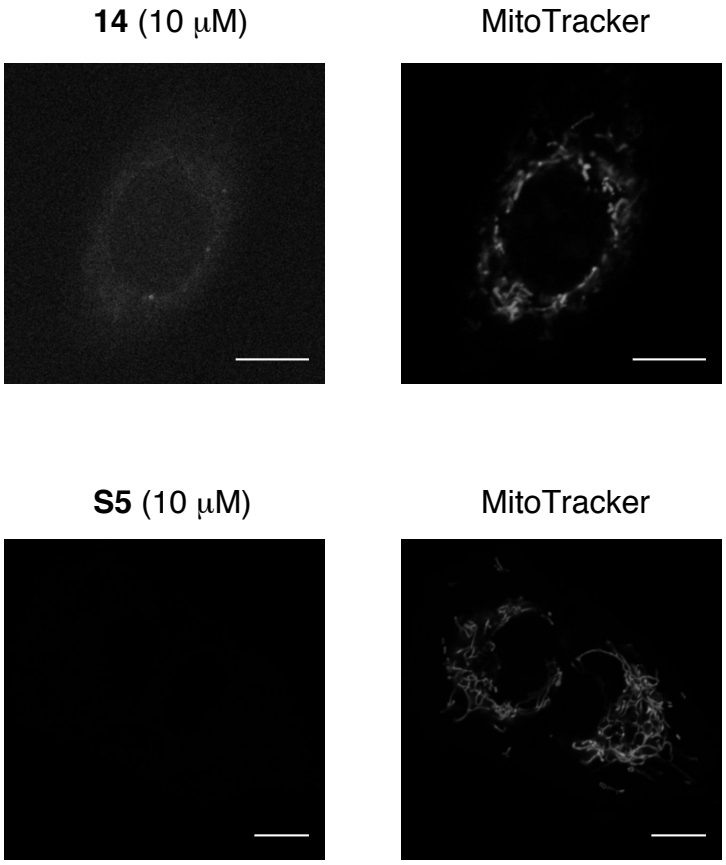


Fig. S4. Cell imaging experiments using negative controls. (a) Structures of AMCA derivative **14** and BODIPY derivative **S5**. (b) A549 cells were treated with MitoTracker Orange (10 nM) at 37 °C for 30 min and then with compound **14** (10 μM) or **S5** (10 μM) at 37 °C for 30 min, and examined with a confocal laser-scanning microscope. Bar = 10 μm.

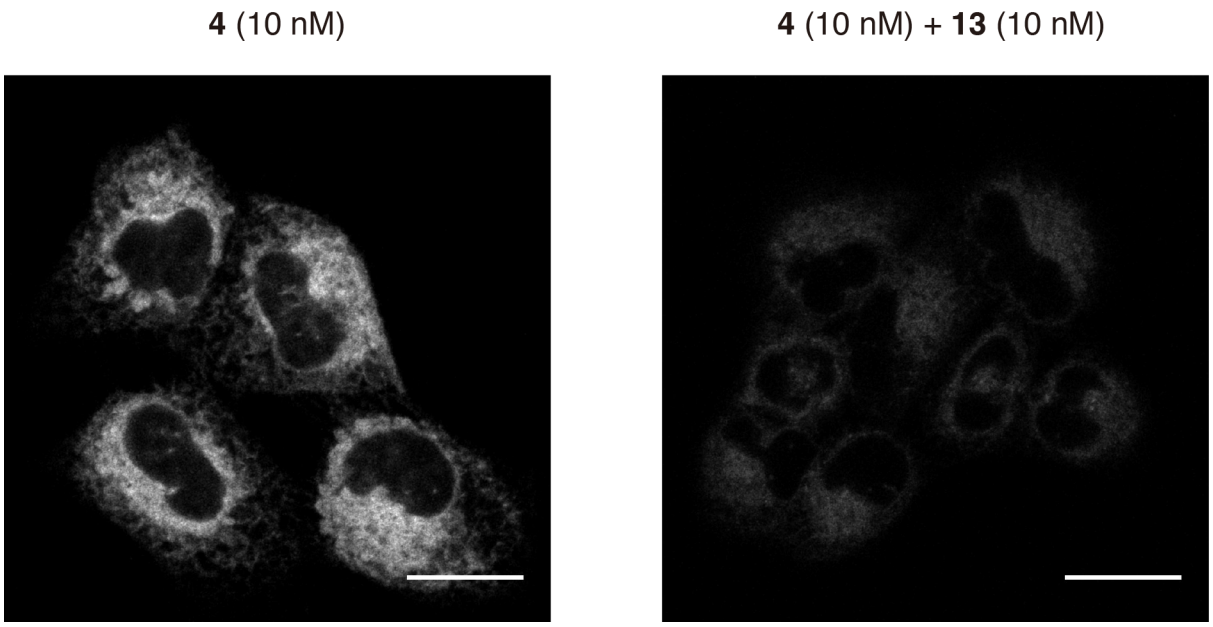


Fig. S5. Competition experiments using **4** and **13**. A549 cells were treated with the BODIPY derivative **4** (10 nM) at 37 °C for 30 min and then incubated in the absence or presence of 8,9-dehydroneopeltolide **13** (10 nM) at 37 °C for 5 min, and examined with a confocal laser-scanning microscope. Bar = 30 μm.

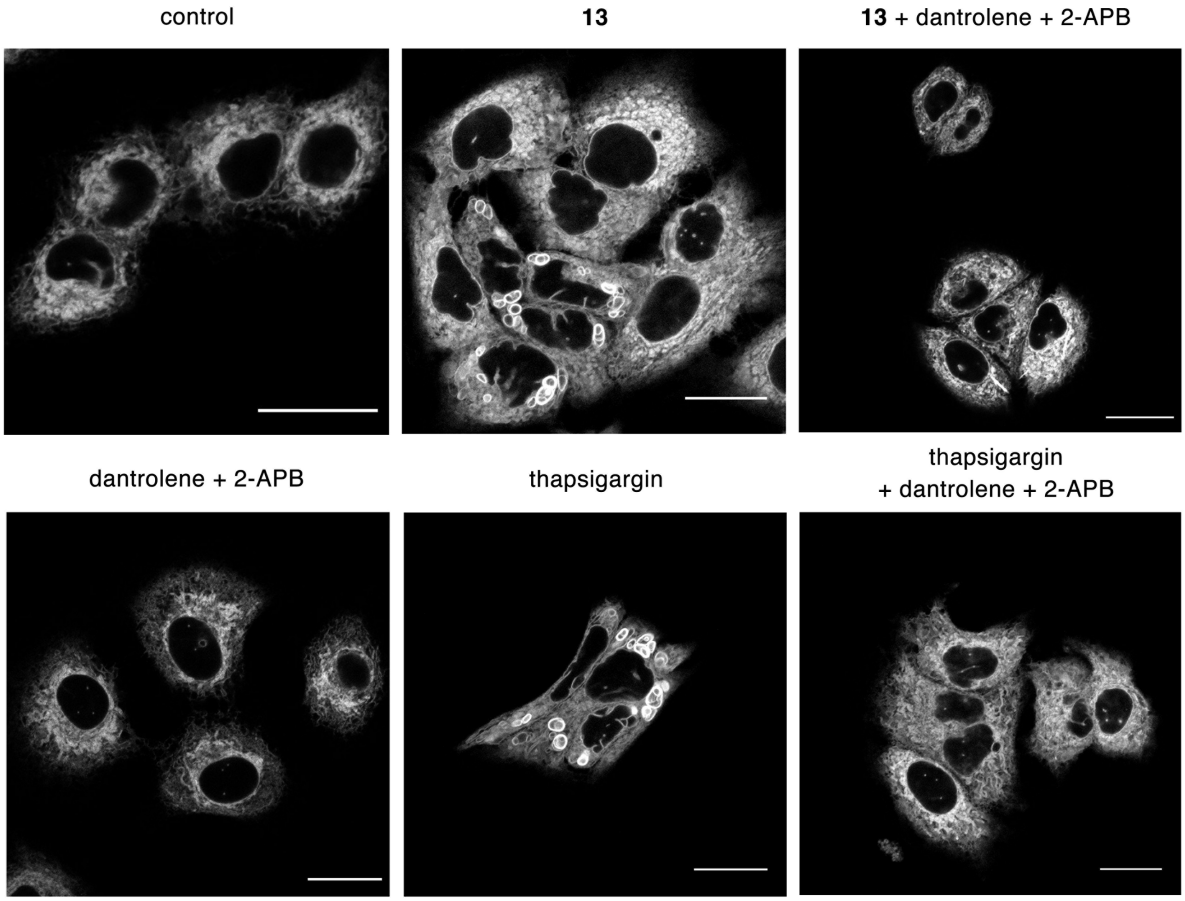


Fig. S6. ER morphology analysis of A549 cells treated with **4** (1 μM) for 1 h, **13** (100 nM) for 1 h, **13** (100 nM) for 24 h, dantrolene (20 μM) + 2-APB (100 μM) for 24 h, **13** (100 nM) + dantrolene (20 μM) + 2-APB (100 μM) for 24 h, thapsigargin (10 nM) for 24 h or thapsigargin (10 nM) + dantrolene (20 μM) + 2-APB (100 μM) for 24 h, stained with ER-Tracker Red, and then examined with a confocal laser-scanning microscope. Bar = 20 μm. Thapsigargin is a sarcoplasmic/endoplasmic reticulum Ca^{2+} ATPase inhibitor. Dantrolene is a ryanodine receptor antagonist. 2-APB is an inositol 1,4,5-triphosphate receptor (IP3R) antagonist. Compounds **4** and **13** and thapsigargin induced formation of characteristic small vesicles, which was blocked by co-incubation with dantrolene and 2-APB.

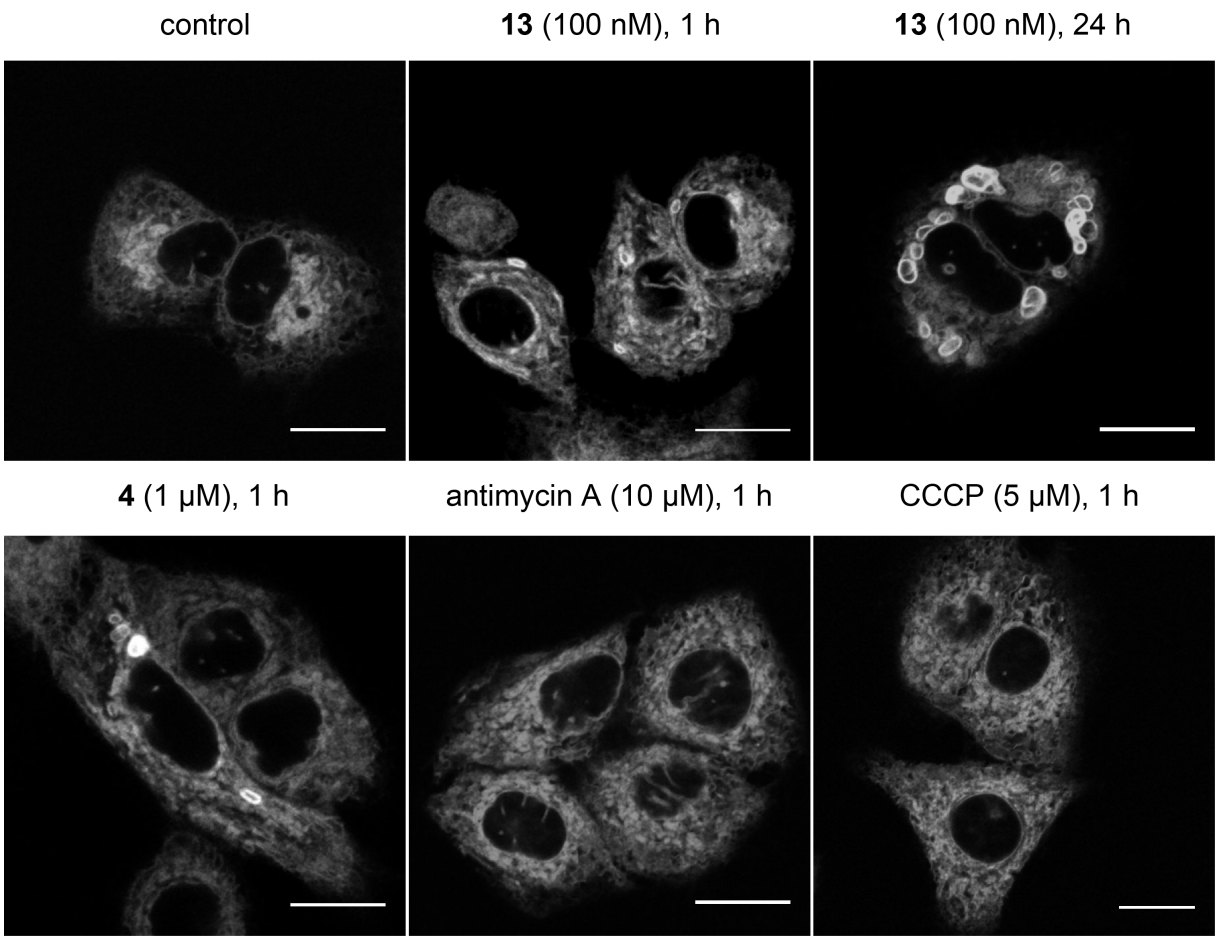


Fig. S7. ER morphology analysis of A549 cells treated with the BODIPY derivative **4** (1 μM), 8,9-dehydroneopeltolide **13** (100 nM), antimycin A (10 μM) or CCCP (5 μM). Cells were stained with ER-Tracker Red and examined with a confocal laser-scanning microscope. Bar = 30 μm. Compounds **4** and **13** induced ER morphology alterations indicative of ER stress. Meanwhile, antimycin A and CCCP did not cause significant ER morphological change.

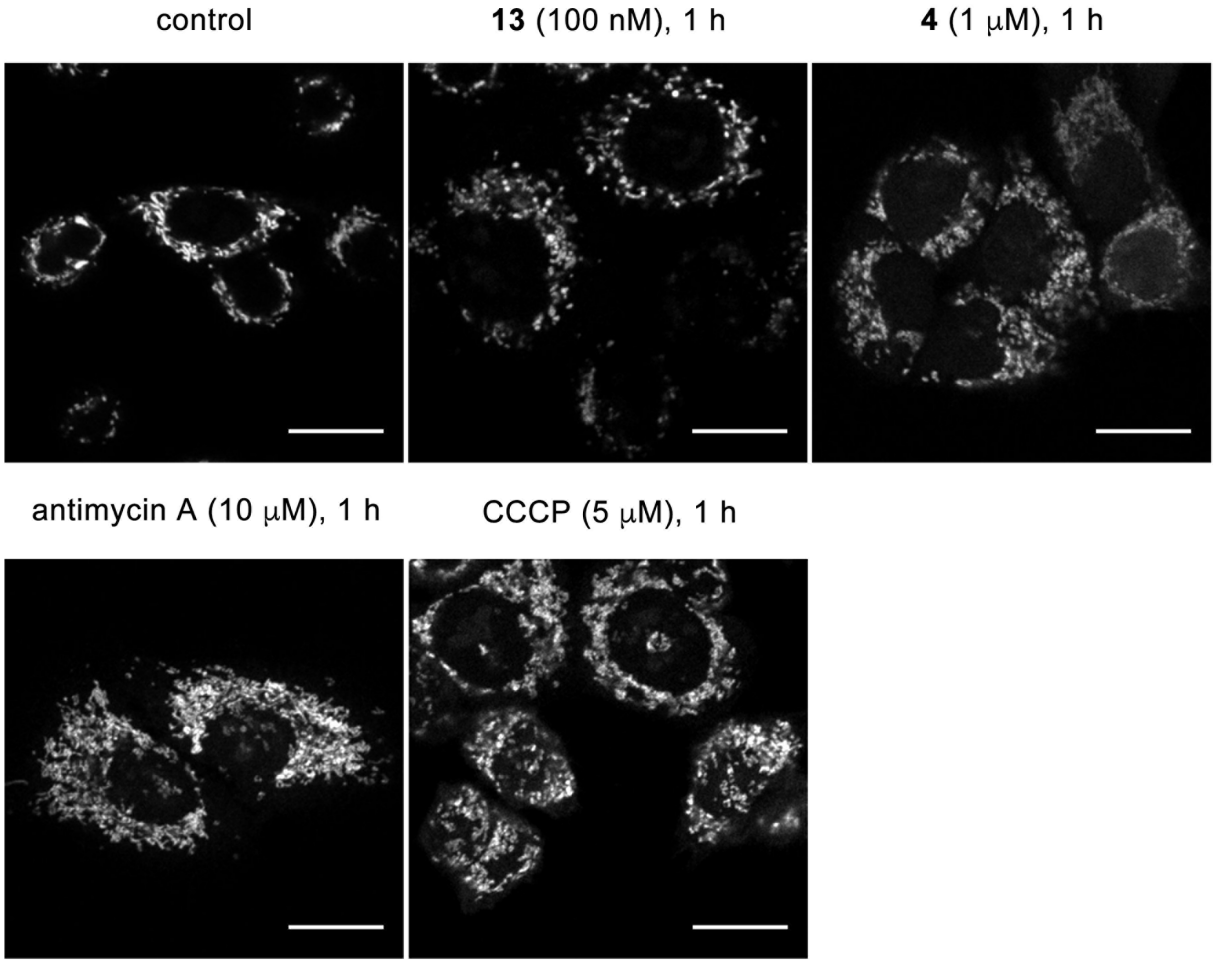


Fig. S8. Mitochondria morphology analysis of A549 cells treated with the BODIPY derivative **4** (1 μM), 8,9-dehydroneopeltolide **13** (100 nM), antimycin A (10 μM) or CCCP (5 μM) for 1 h. Cells were stained with MitoTracker Orange and examined with a confocal laser-scanning microscope. Bar = 30 μm. All these compounds induced fission of mitochondria, indicative of mitochondrial damage.

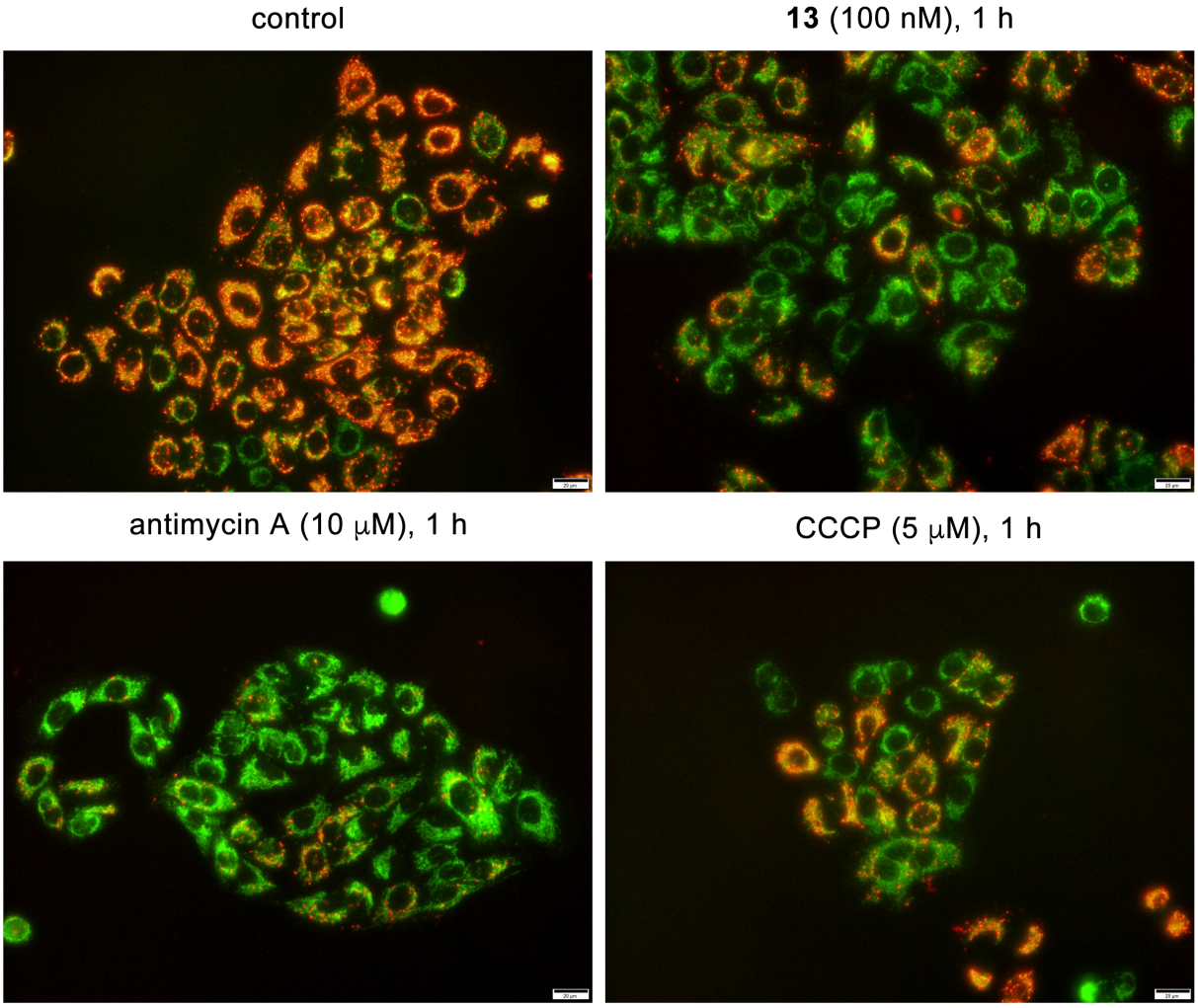
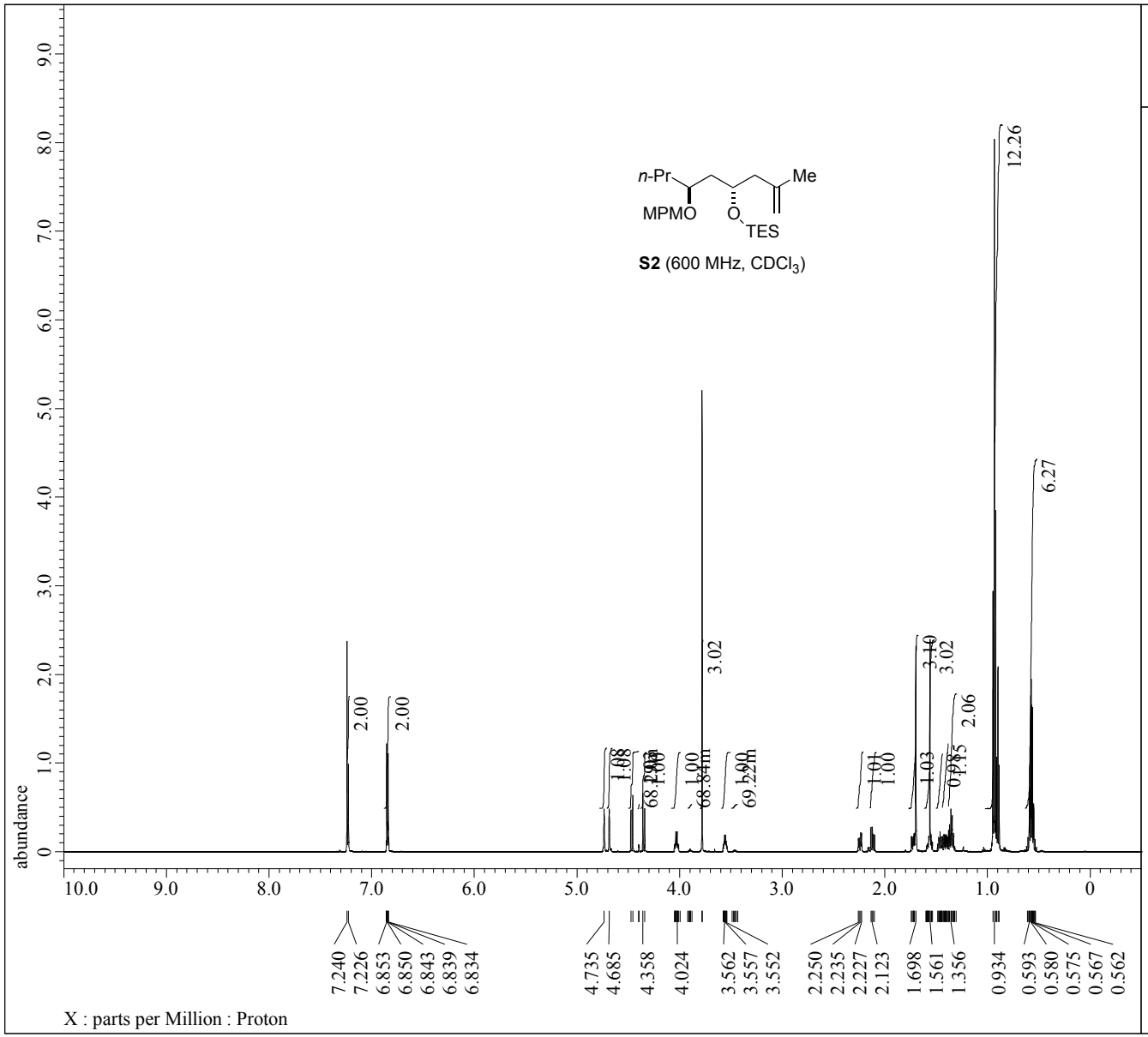
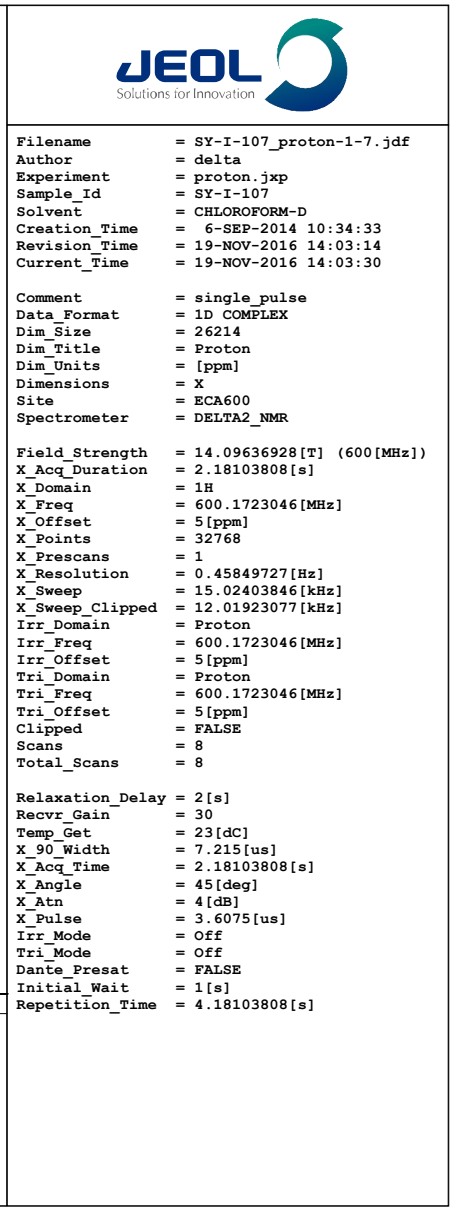


Fig. S9. Mitochondrial membrane potential assay. A549 cells were treated with 8,9-dehydroneopeltolide **13** (100 nM), antimycin A (10 μM) or CCCP (5 μM) for 1 h and then with JC-1 (2.5 μg/mL) for 30 min. Cells were examined with a fluorescence microscope. All these compounds caused dissipation of mitochondrial membrane potential as indicated by the increase of green fluorescent signal of JC-1 monomers.

Copies of NMR spectra and HPLC chromatograms





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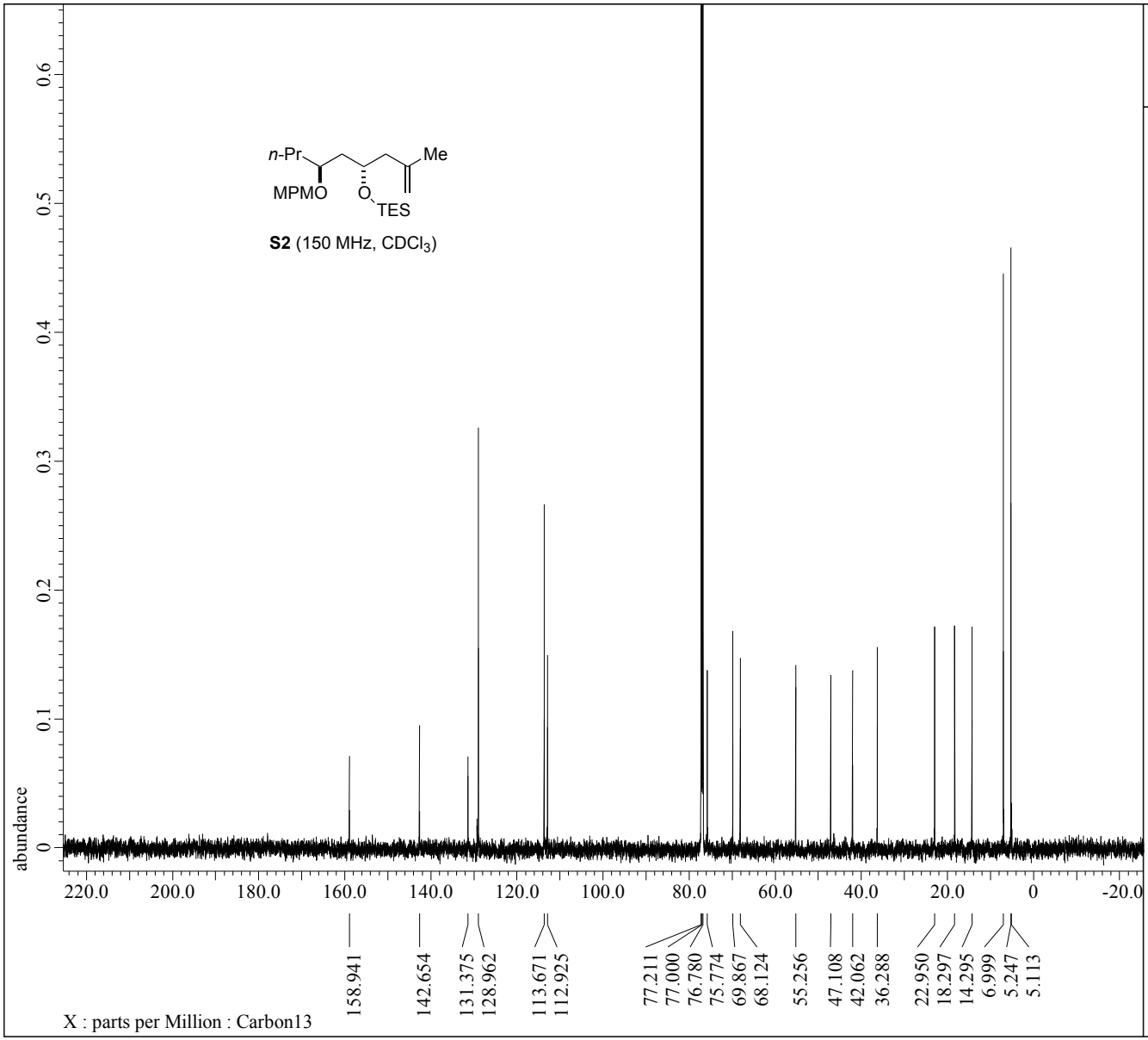

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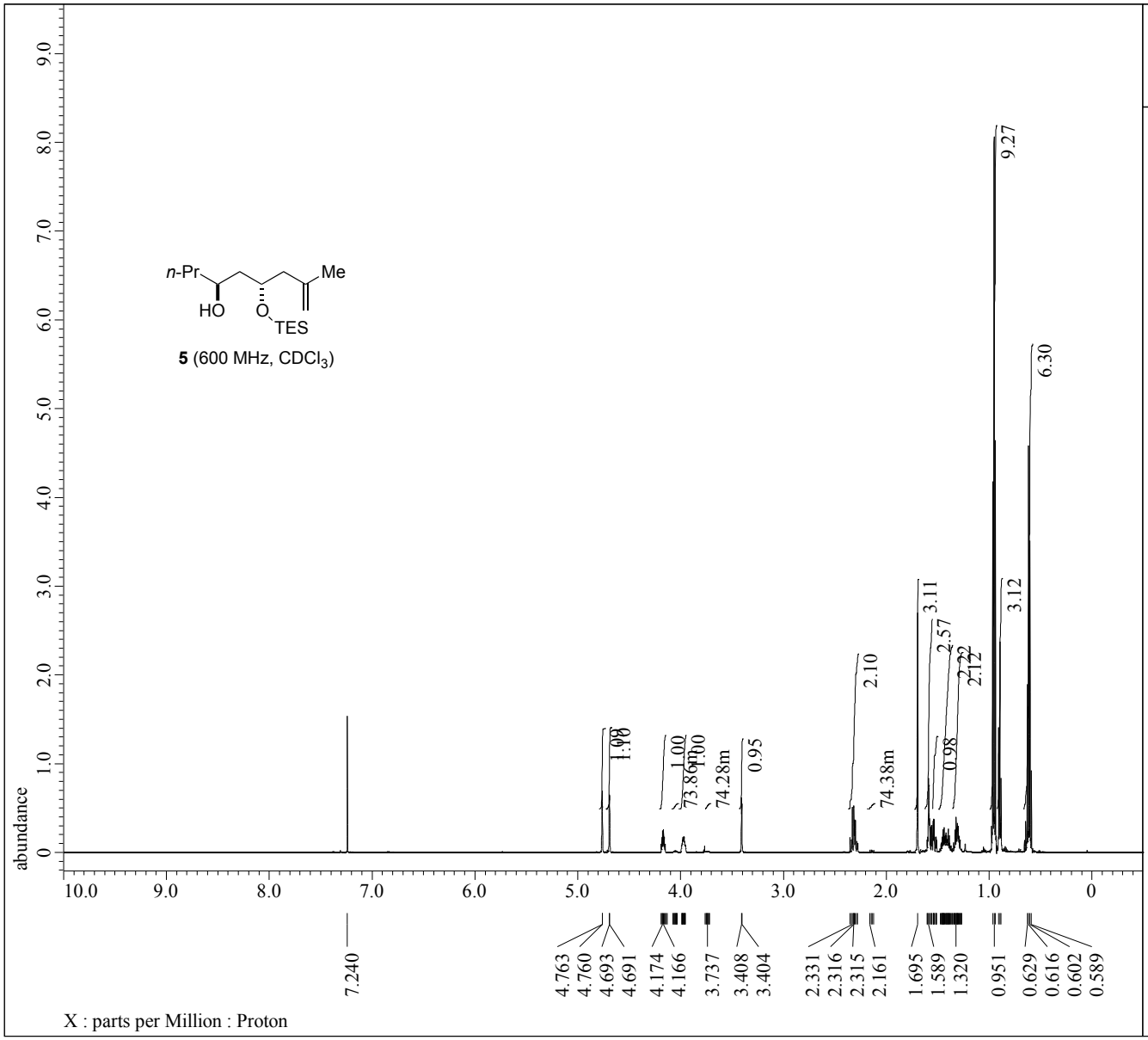


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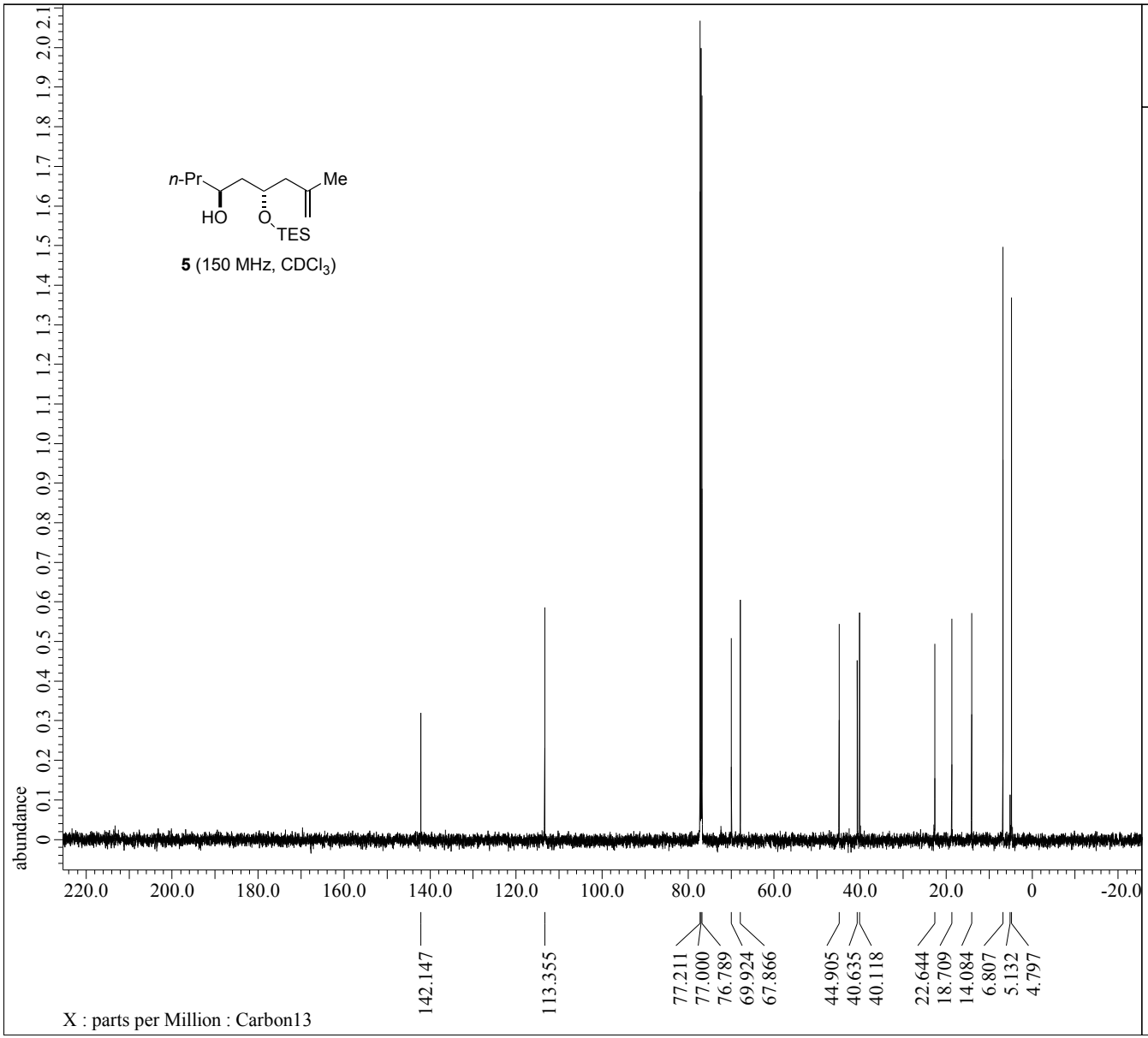

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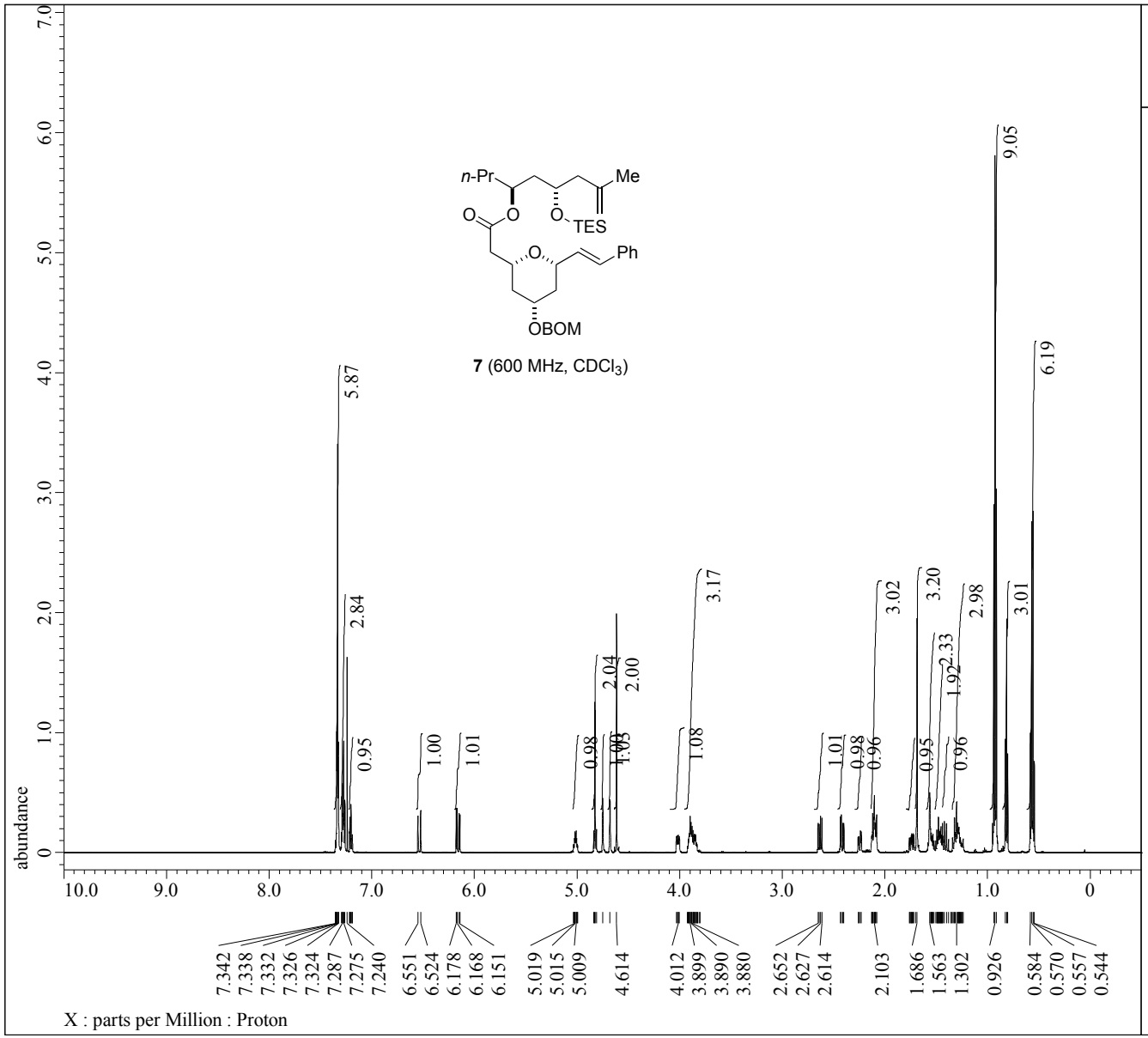


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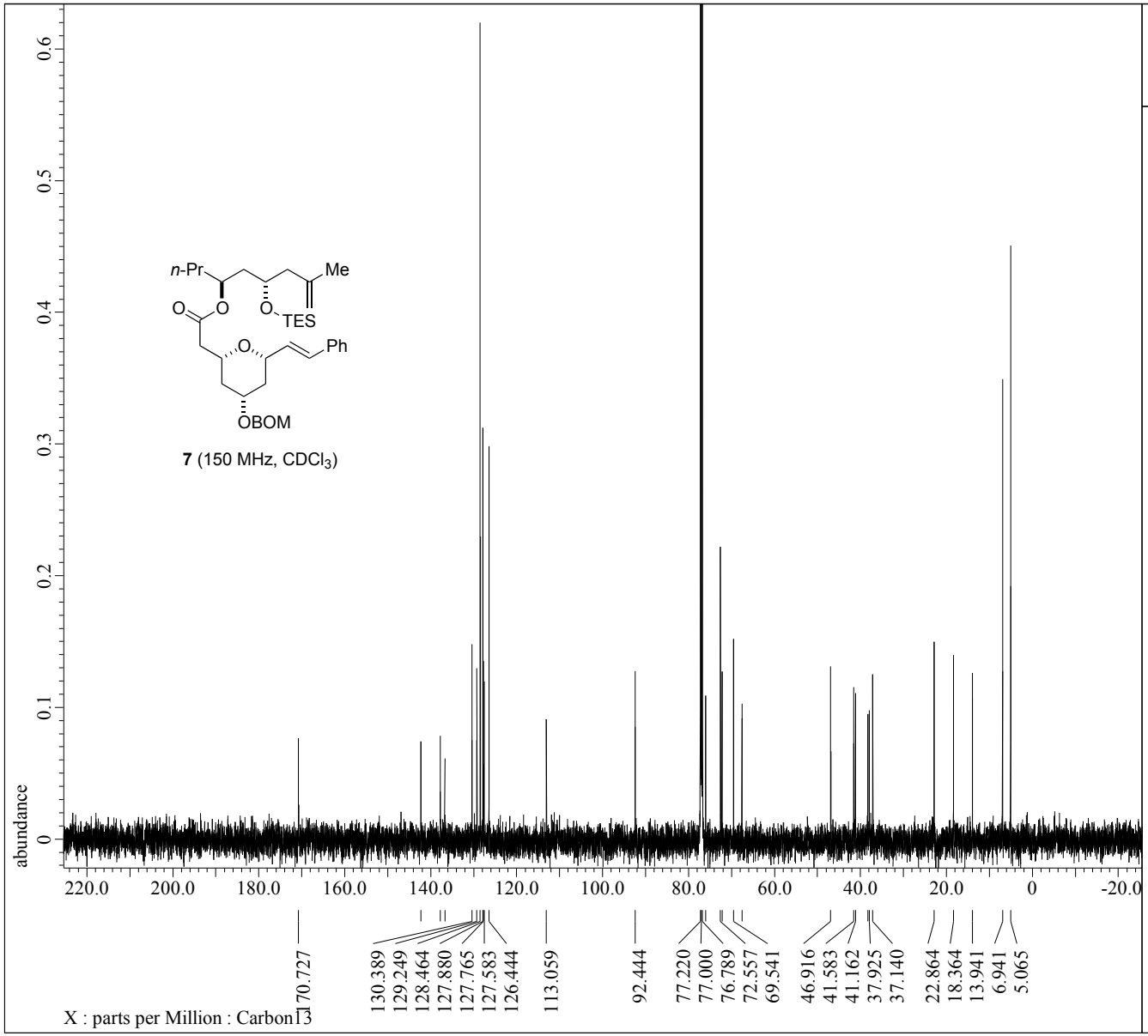

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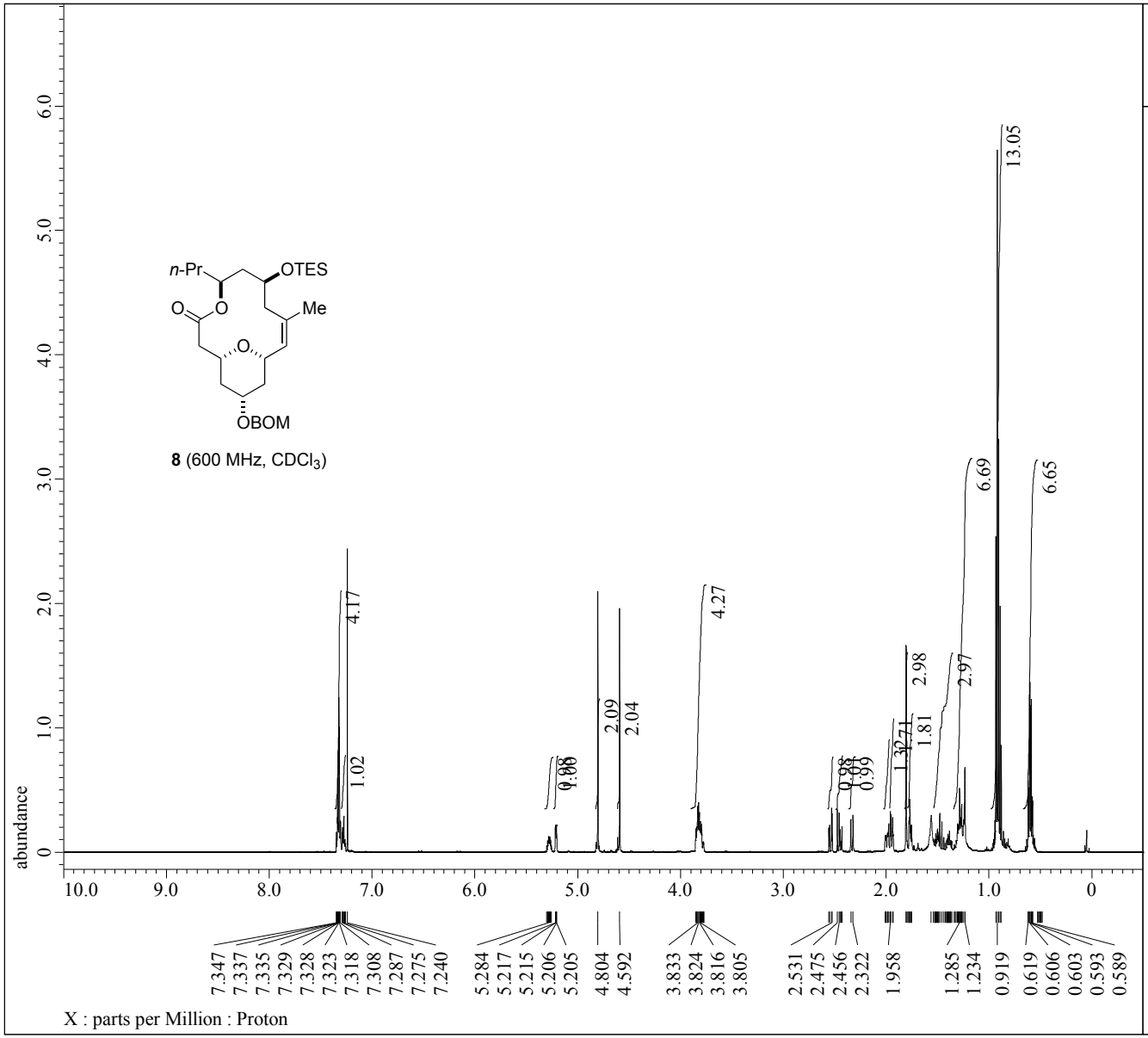


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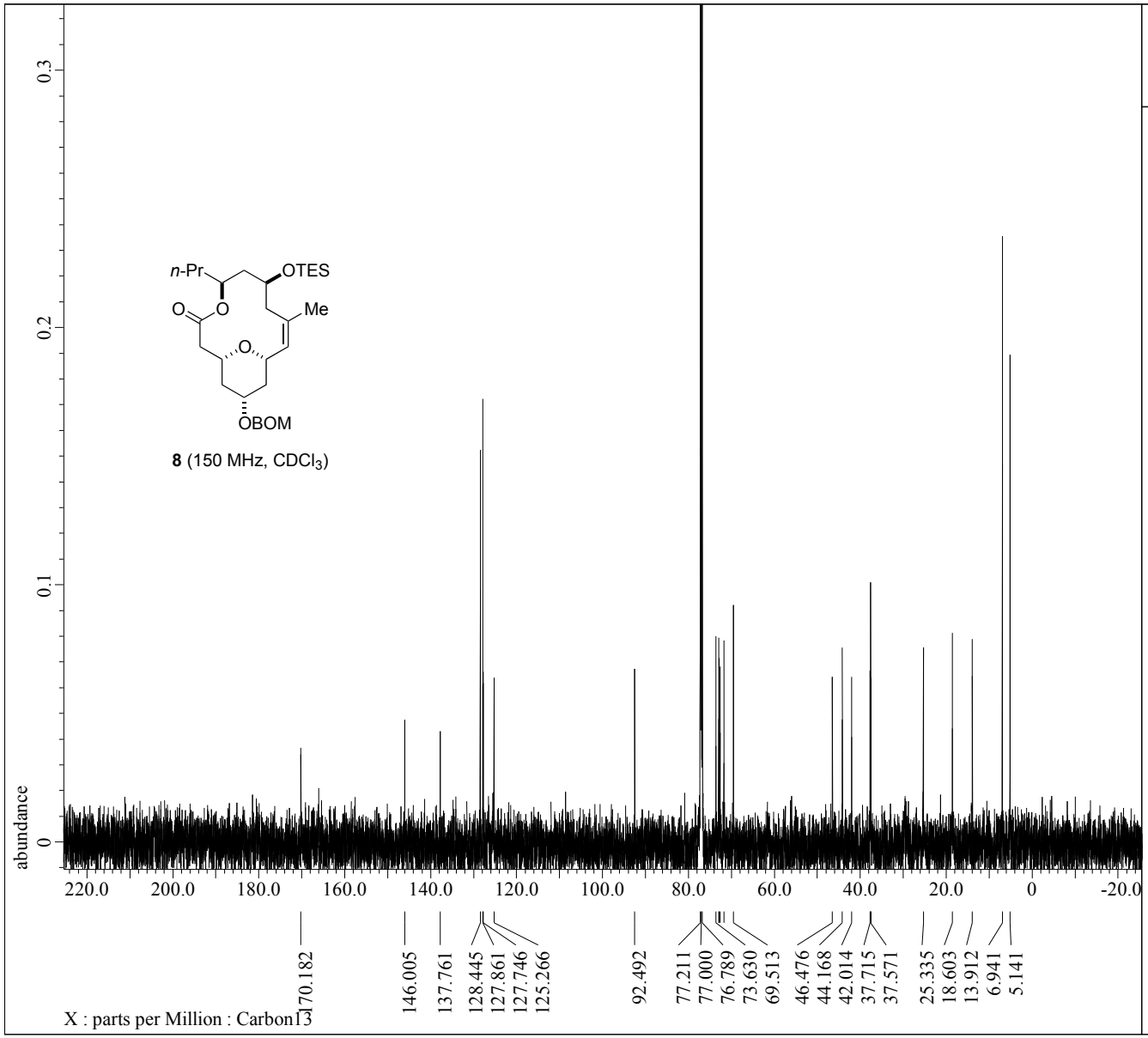
Total_Scans       = 8

Relaxation_Delay  = 2[s]
Recvr_Gain        = 34
Temp_Get          = 23.1[dC]
X_90_Width        = 7.215[us]
X_Acq_Time        = 2.18103808[s]
X_Angle           = 45[deg]
X_Atn             = 4[dB]
X_Pulse           = 3.6075[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 4.18103808[s]
```



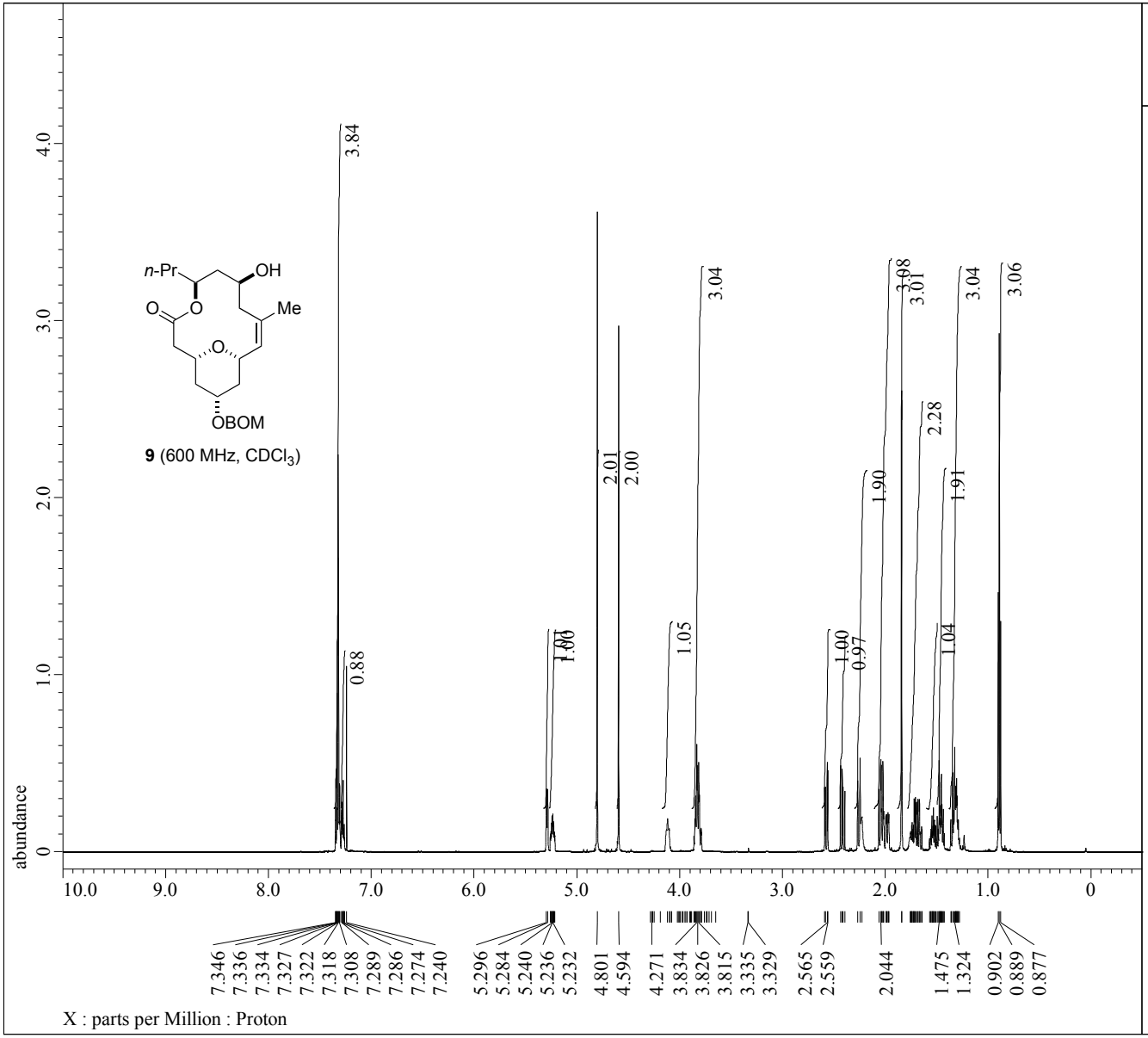
JEOL Solutions for Innovation

```
Filename          = SY-I-116_Carbon-1-3.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = SY-I-116 1
Solvent           = CHLOROFORM-D
Creation_Time     = 22-SEP-2014 14:23:39
Revision_Time     = 21-NOV-2016 12:38:59
Current_Time      = 21-NOV-2016 12:39:10

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 0.69206016[s]
X_Domain          = 13C
X_Freq            = 150.91343039[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.44496109[Hz]
X_Sweep           = 47.34848485[kHz]
X_Sweep_Clipped   = 37.87878788[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 236
Total_Scans       = 236

Relaxation_Delay = 2[s]
Recvr_Gain        = 56
Temp_Get          = 23.3[dC]
X_90_Width        = 10.5[us]
X_Acq_Time        = 0.69206016[s]
X_Angle           = 30[deg]
X_Atn             = 7.4[dB]
X_Pulse           = 3.5[us]
Irr_Atn_Dec       = 24.452[dB]
Irr_Atn_Noe       = 24.452[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69206016[s]
```

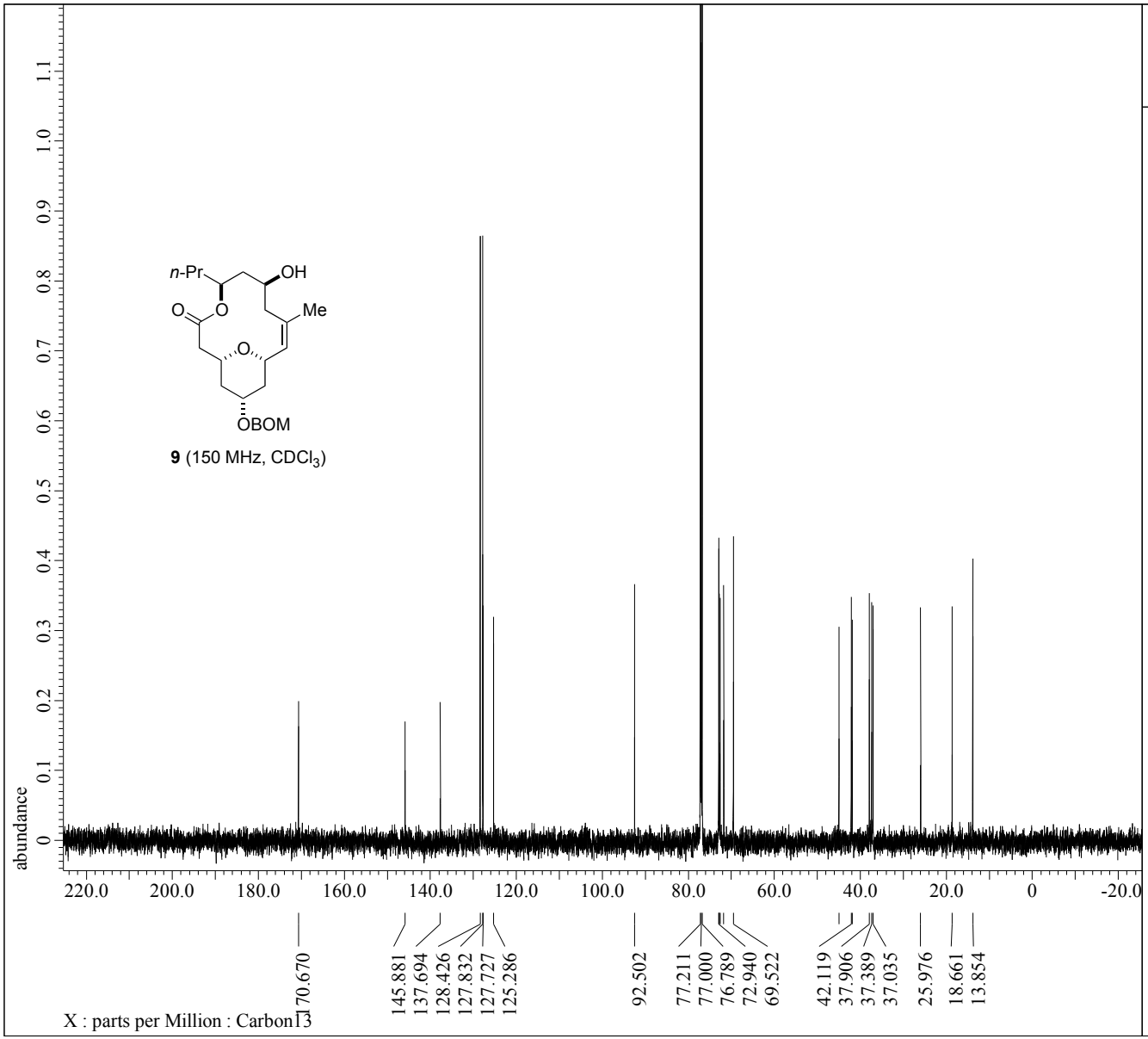



```
Filename          = SY-I-119_proton-1-3.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = SY-I-119
Solvent           = CHLOROFORM-D
Creation_Time     = 27-SEP-2014 18:28:59
Revision_Time     = 19-NOV-2016 14:42:20
Current_Time      = 19-NOV-2016 14:42:46

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 2.18103808[s]
X_Domain          = 1H
X_Freq            = 600.1723046[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.45849727[Hz]
X_Sweep           = 15.02403846[kHz]
X_Sweep_Clipped   = 12.01923077[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 600.1723046[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 2[s]
Recvr_Gain        = 24
Temp_Get          = 22.9[dC]
X_90_Width        = 7.215[us]
X_Acq_Time        = 2.18103808[s]
X_Angle           = 45[deg]
X_Atn             = 4[dB]
X_Pulse           = 3.6075[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 4.18103808[s]
```

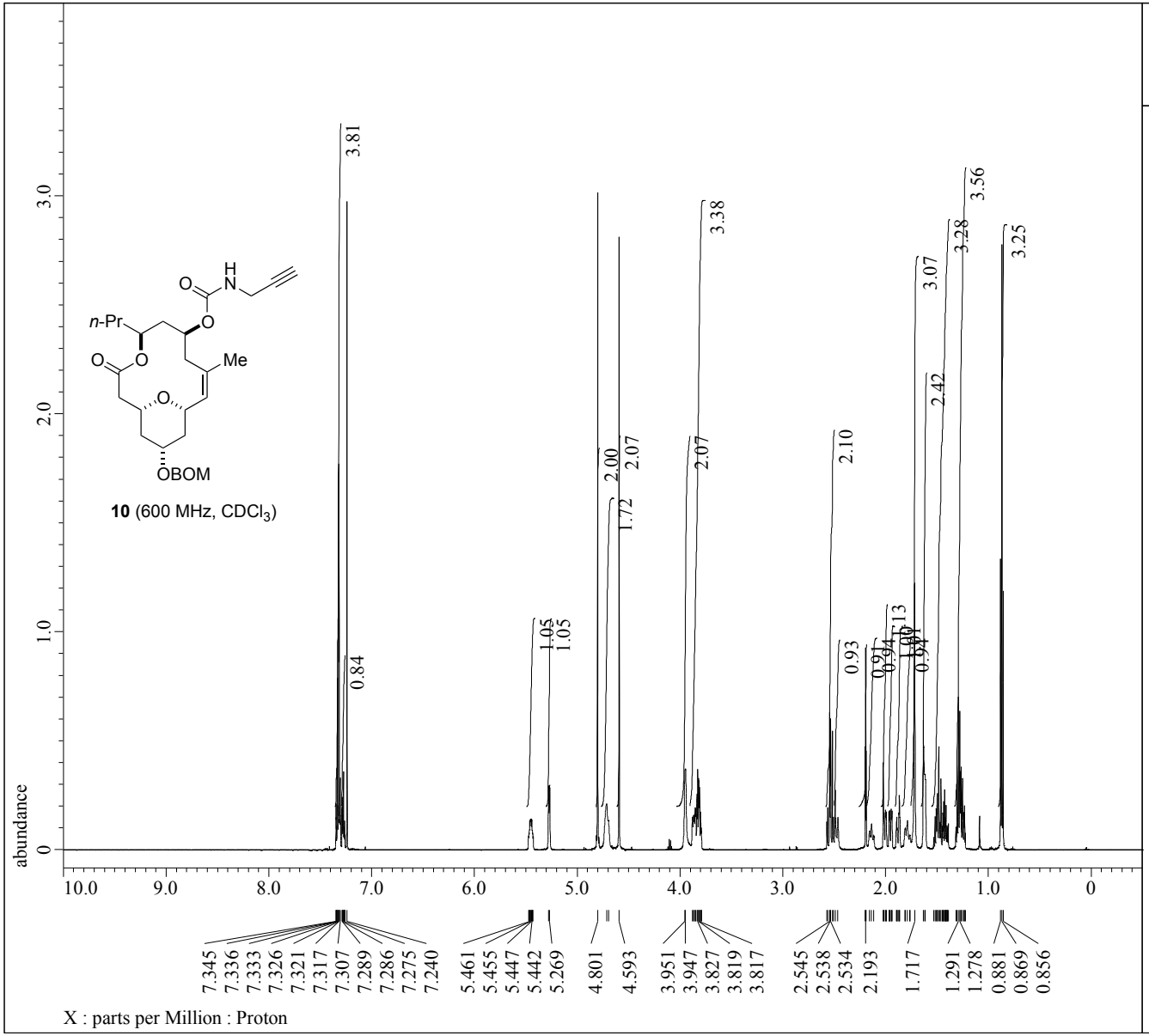



```
Filename          = SY-I-119_Carbon-1-3.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = SY-I-119
Solvent           = CHLOROFORM-D
Creation_Time     = 27-SEP-2014 18:32:35
Revision_Time     = 19-NOV-2016 13:06:21
Current_Time      = 19-NOV-2016 13:06:35

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 0.69206016[s]
X_Domain          = 13C
X_Freq            = 150.91343039[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.44496109[Hz]
X_Sweep           = 47.34848485[kHz]
X_Sweep_Clipped   = 37.87878788[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 112
Total_Scans       = 112

Relaxation_Delay  = 2[s]
Recvr_Gain        = 56
Temp_Get          = 23.2[dC]
X_90_Width        = 10.5[us]
X_Acq_Time        = 0.69206016[s]
X_Angle           = 30[deg]
X_Atn             = 7.4[dB]
X_Pulse           = 3.5[us]
Irr_Atn_Dec       = 24.452[dB]
Irr_Atn_Noe       = 24.452[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69206016[s]
```

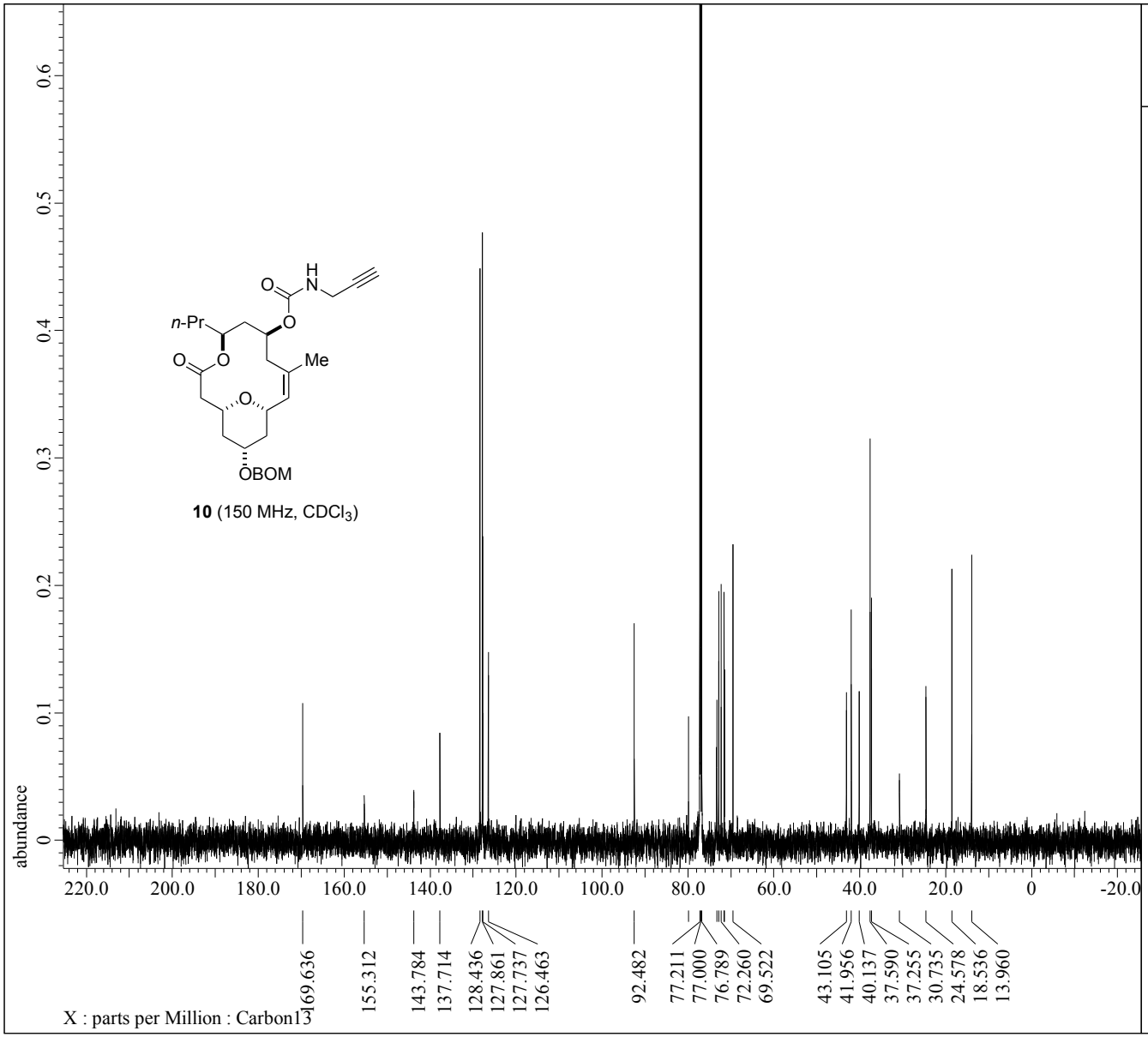



```
Filename          = SY-I-124_proton-1-3.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = SY-I-124
Solvent           = CHLOROFORM-D
Creation_Time     =  3-OCT-2014 22:33:04
Revision_Time     = 19-NOV-2016 15:00:51
Current_Time      = 19-NOV-2016 15:01:25

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 2.18103808[s]
X_Domain          = 1H
X_Freq            = 600.1723046[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.45849727[Hz]
X_Sweep           = 15.02403846[kHz]
X_Sweep_Clipped   = 12.01923077[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 600.1723046[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 2[s]
Recvr_Gain        = 34
Temp_Get          = 23.4[dC]
X_90_Width        = 7.215[us]
X_Acq_Time        = 2.18103808[s]
X_Angle           = 45[deg]
X_Atn             = 4[dB]
X_Pulse           = 3.6075[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 4.18103808[s]
```

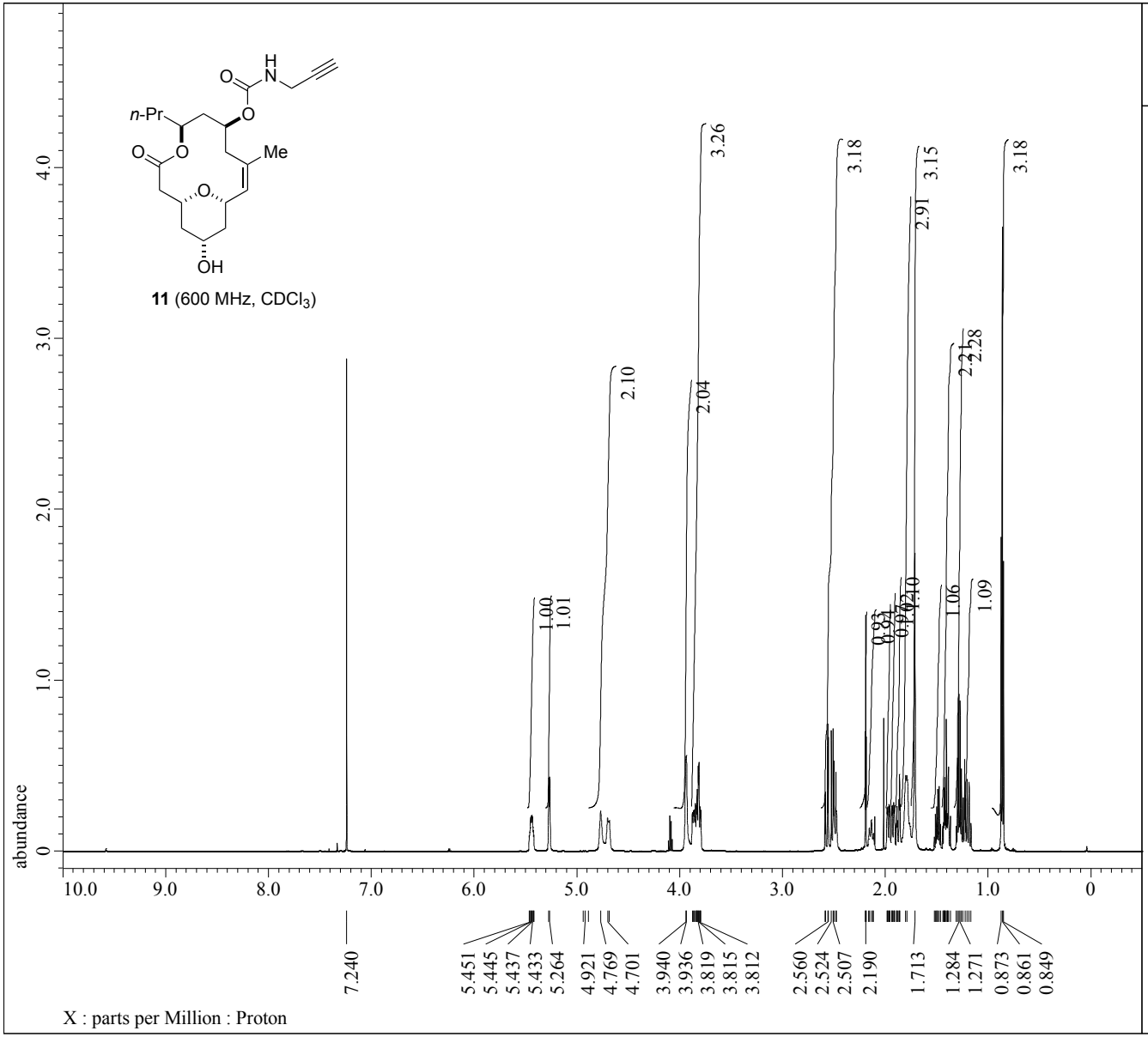



```
Filename          = SY-I-124_Carbon-1-4.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = SY-I-124
Solvent           = CHLOROFORM-D
Creation_Time     =  4-OCT-2014 16:48:27
Revision_Time     = 19-NOV-2016 13:08:09
Current_Time      = 19-NOV-2016 13:08:26

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 0.69206016[s]
X_Domain          = 13C
X_Freq            = 150.91343039[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.44496109[Hz]
X_Sweep           = 47.34848485[kHz]
X_Sweep_Clipped   = 37.87878788[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 180
Total_Scans       = 180

Relaxation_Delay = 2[s]
Recvr_Gain        = 56
Temp_Get          = 23.3[dC]
X_90_Width        = 10.5[us]
X_Acq_Time        = 0.69206016[s]
X_Angle           = 30[deg]
X_Atn             = 7.4[dB]
X_Pulse           = 3.5[us]
Irr_Atn_Dec       = 24.452[dB]
Irr_Atn_Noe       = 24.452[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69206016[s]
```



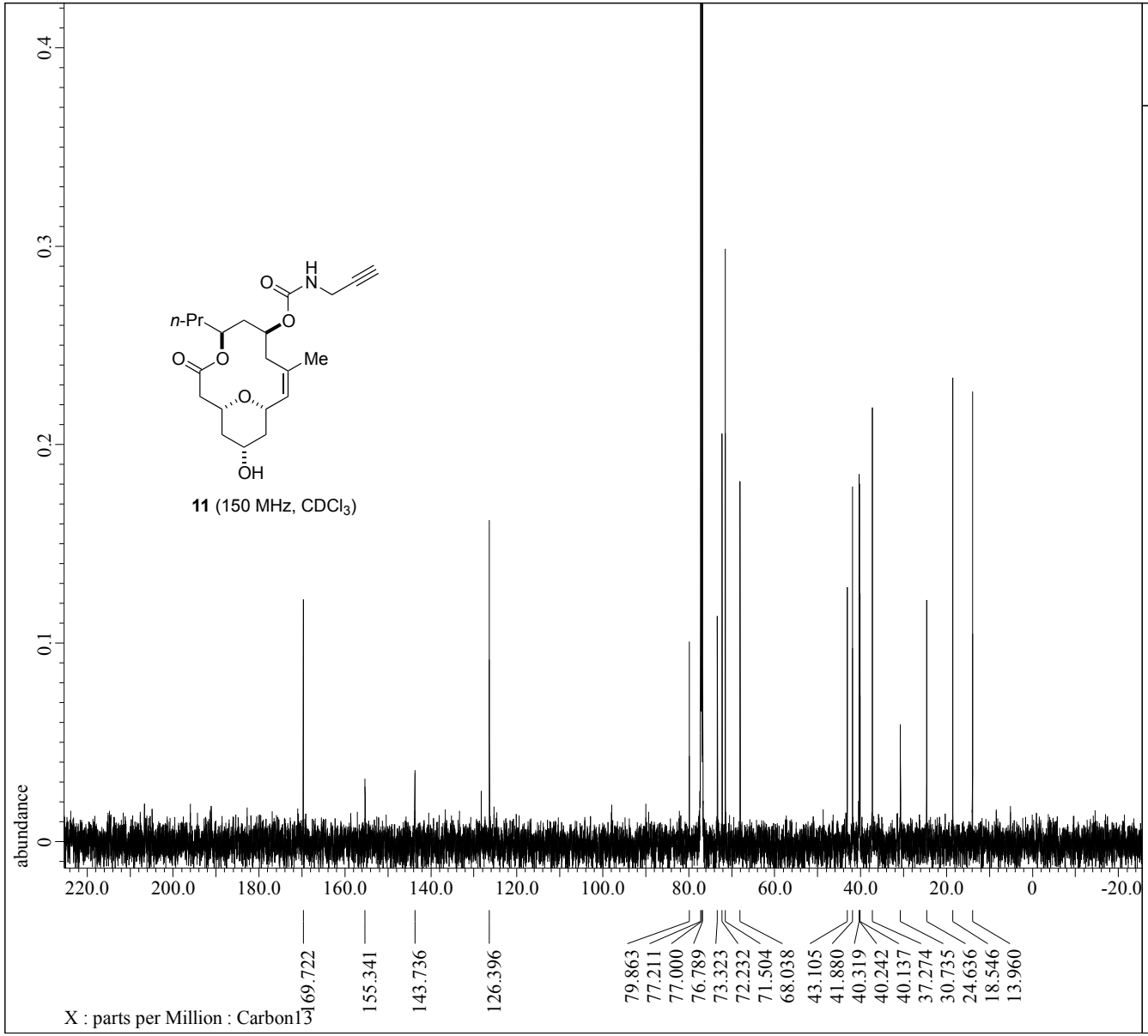
JEOL Solutions for Innovation

```
Filename          = SY-I-126_proton-1-3.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = SY-I-126-c2
Solvent           = CHLOROFORM-D
Creation_Time     =  9-OCT-2014 13:30:24
Revision_Time     = 19-NOV-2016 15:08:53
Current_Time      = 19-NOV-2016 15:09:16

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 2.18103808[s]
X_Domain          = 1H
X_Freq            = 600.1723046[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.45849727[Hz]
X_Sweep           = 15.02403846[kHz]
X_Sweep_Clipped   = 12.01923077[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 600.1723046[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 2[s]
Recvr_Gain        = 30
Temp_Get          = 23.2[dC]
X_90_Width        = 7.215[us]
X_Acq_Time        = 2.18103808[s]
X_Angle           = 45[deg]
X_Atn             = 4[dB]
X_Pulse           = 3.6075[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 4.18103808[s]
```



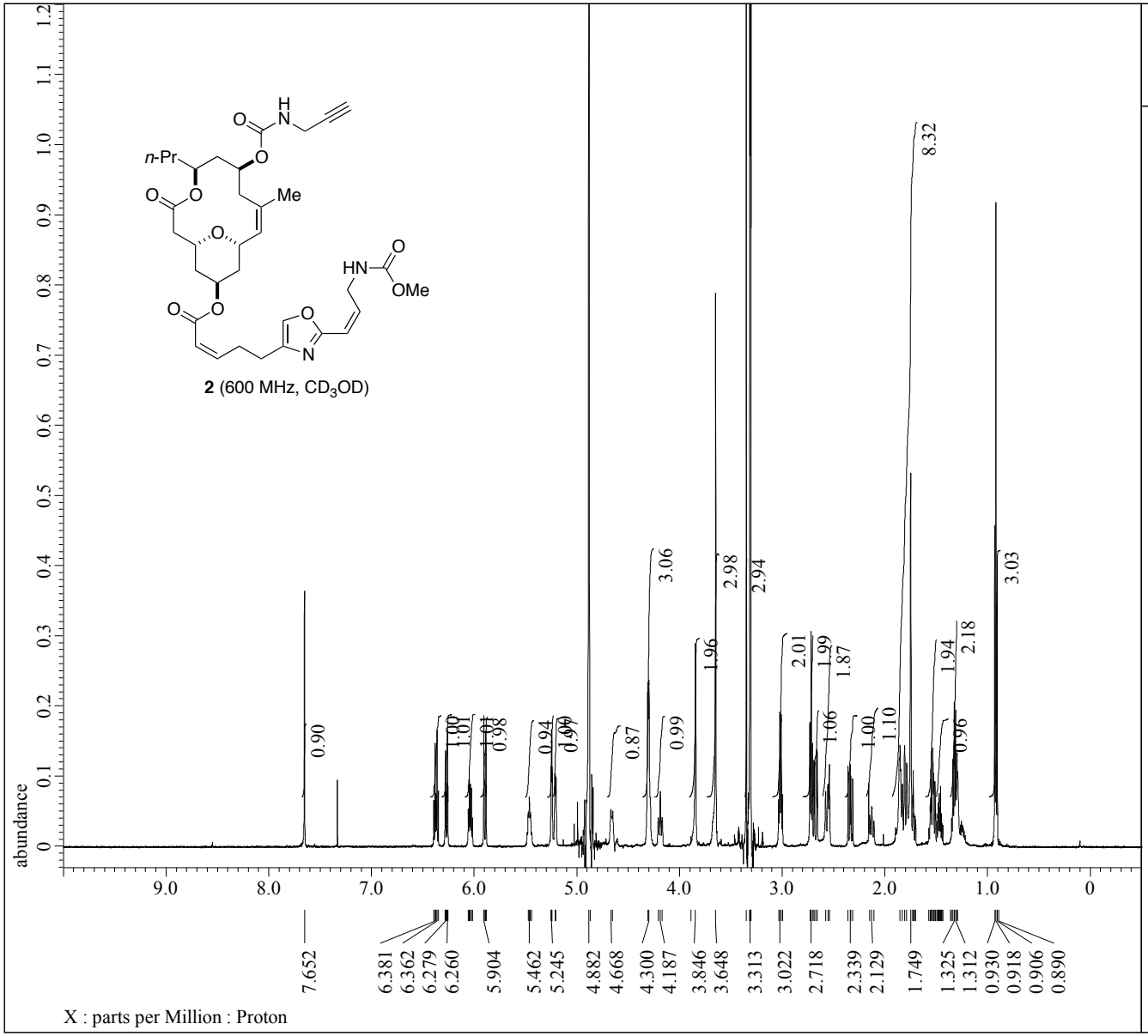
JEOL Solutions for Innovation

```
Filename          = SY-I-126_Carbon-1-4.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = SY-I-126-c2
Solvent           = CHLOROFORM-D
Creation_Time     =  9-OCT-2014 14:50:21
Revision_Time     = 19-NOV-2016 13:09:17
Current_Time      = 19-NOV-2016 13:09:35

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 0.69206016[s]
X_Domain          = 13C
X_Freq            = 150.91343039[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.44496109[Hz]
X_Sweep           = 47.34848485[kHz]
X_Sweep_Clipped   = 37.87878788[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 260
Total_Scans       = 260

Relaxation_Delay = 2[s]
Recvr_Gain        = 56
Temp_Get          = 23.2[dC]
X_90_Width        = 10.5[us]
X_Acq_Time        = 0.69206016[s]
X_Angle           = 30[deg]
X_Atn             = 7.4[dB]
X_Pulse           = 3.5[us]
Irr_Atn_Dec       = 24.452[dB]
Irr_Atn_Noe       = 24.452[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69206016[s]
```



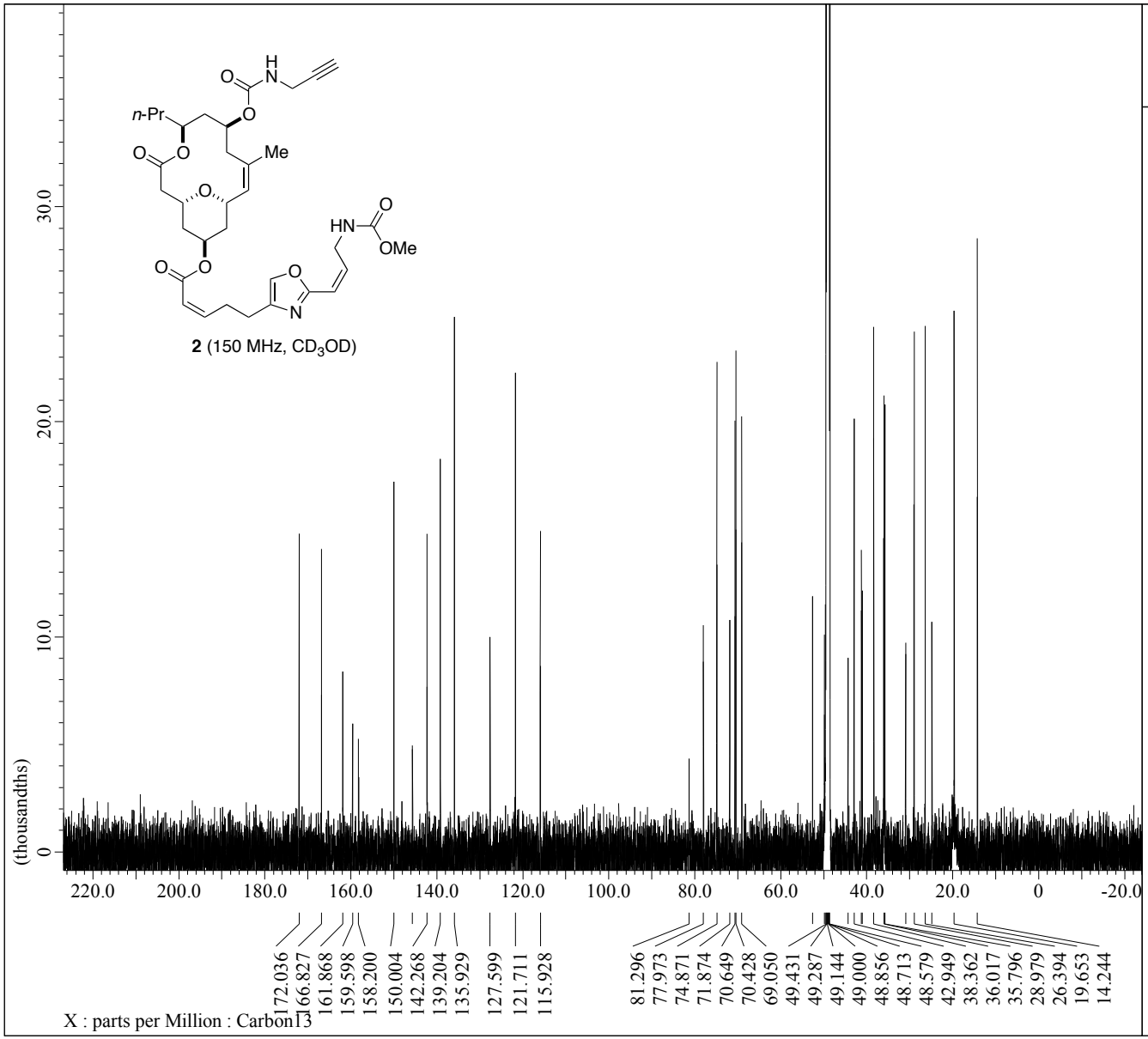
JEOL Solutions for Innovation

```
Filename          = SY-I-140_proton-2-3.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = SY-I-140
Solvent           = METHANOL-D4
Creation_Time     = 18-NOV-2016 00:01:38
Revision_Time     = 19-NOV-2016 17:04:58
Current_Time      = 19-NOV-2016 17:06:04

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 2.18103808[s]
X_Domain          = 1H
X_Freq            = 600.1723046[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.45849727[Hz]
X_Sweep           = 15.02403846[kHz]
X_Sweep_Clipped   = 12.01923077[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 600.1723046[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 2[s]
Recvr_Gain        = 44
Temp_Get          = 20.9[dC]
X_90_Width        = 6.9[us]
X_Acq_Time        = 2.18103808[s]
X_Angle           = 45[deg]
X_Atn             = 4[dB]
X_Pulse           = 3.45[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 4.18103808[s]
```



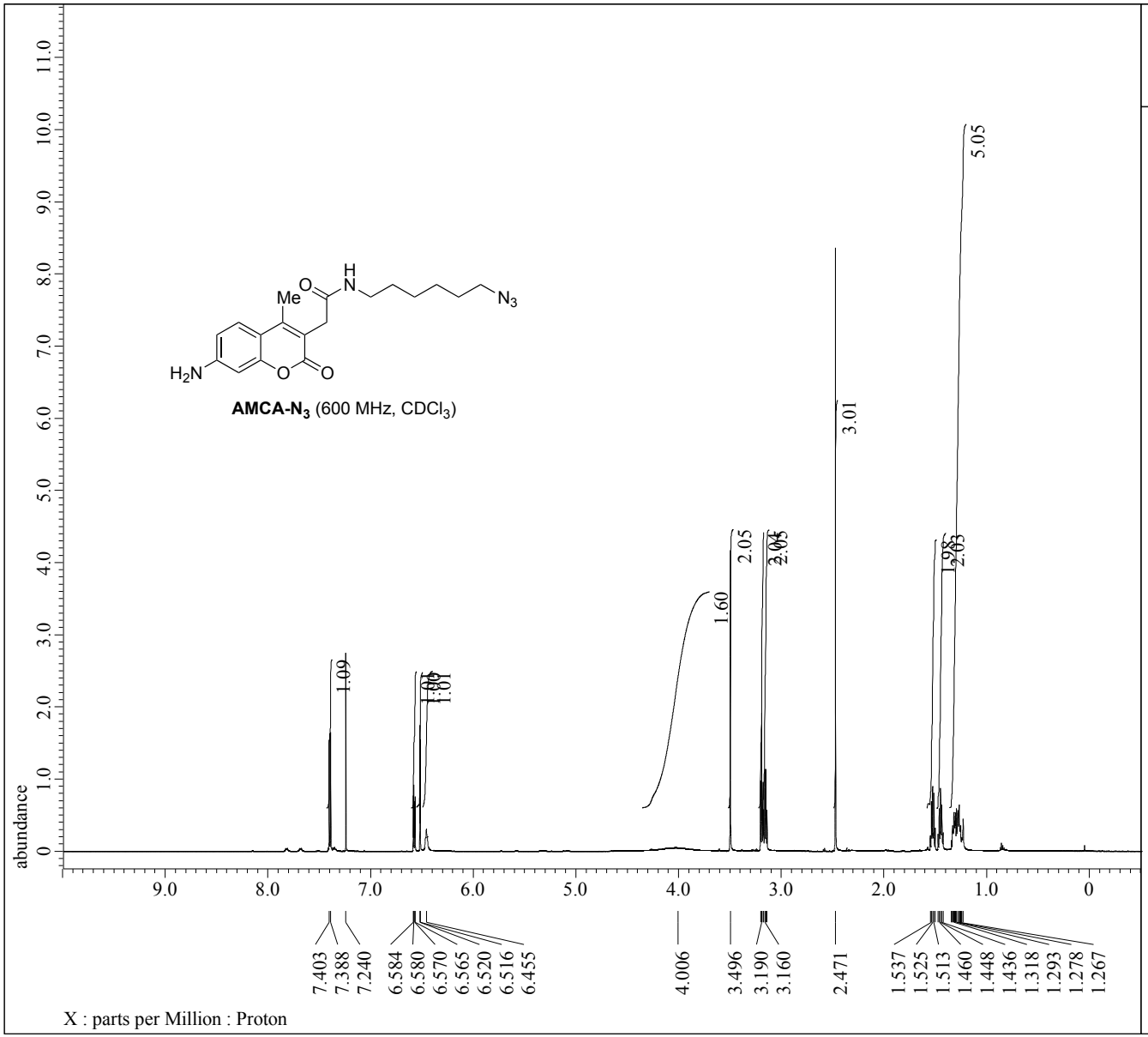
JEOL Solutions for Innovation

```
Filename          = SY-I-140_Carbon-1-6.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = SY-I-140
Solvent           = METHANOL-D4
Creation_Time     = 18-NOV-2016 00:05:09
Revision_Time     = 19-NOV-2016 13:11:20
Current_Time      = 19-NOV-2016 13:11:35

Comment           = single pulse decoupled gat
Data_Format       = 1D REAL
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 0.69206016[s]
X_Domain          = 13C
X_Freq            = 150.91343039[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.44496109[Hz]
X_Sweep           = 47.34848485[kHz]
X_Sweep_Clipped   = 37.87878788[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 12400
Total_Scans       = 12400

Relaxation_Delay  = 2[s]
Recvr_Gain        = 56
Temp_Get          = 20.8[dC]
X_90_Width        = 11.2[us]
X_Acq_Time        = 0.69206016[s]
X_Angle           = 30[deg]
X_Atn             = 7.4[dB]
X_Pulse           = 3.73333333[us]
Irr_Atn_Dec       = 24.652[dB]
Irr_Atn_Noe       = 24.652[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69206016[s]
```

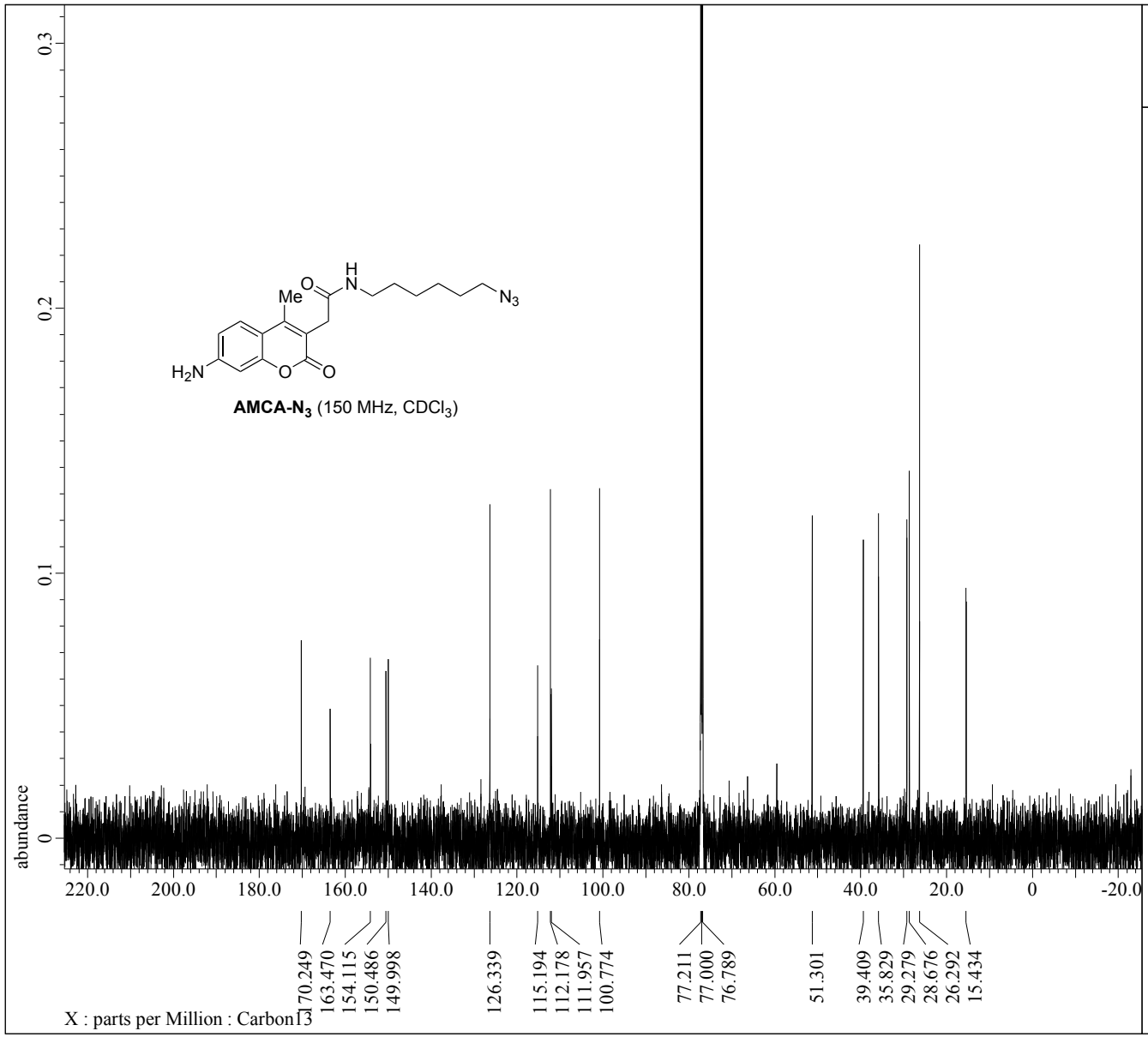



```
Filename          = SY-II-066_proton-1-3.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = SY-II-066
Solvent           = CHLOROFORM-D
Creation_Time     = 21-MAY-2015 18:48:56
Revision_Time     = 19-NOV-2016 16:03:57
Current_Time      = 19-NOV-2016 16:04:21

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 2.18103808[s]
X_Domain          = 1H
X_Freq            = 600.1723046[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.45849727[Hz]
X_Sweep           = 15.02403846[kHz]
X_Sweep_Clipped   = 12.01923077[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 600.1723046[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 2[s]
Recvr_Gain        = 36
Temp_Get          = 22.1[dC]
X_90_Width        = 7.215[us]
X_Acq_Time        = 2.18103808[s]
X_Angle           = 45[deg]
X_Atn             = 4[dB]
X_Pulse           = 3.6075[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 4.18103808[s]
```



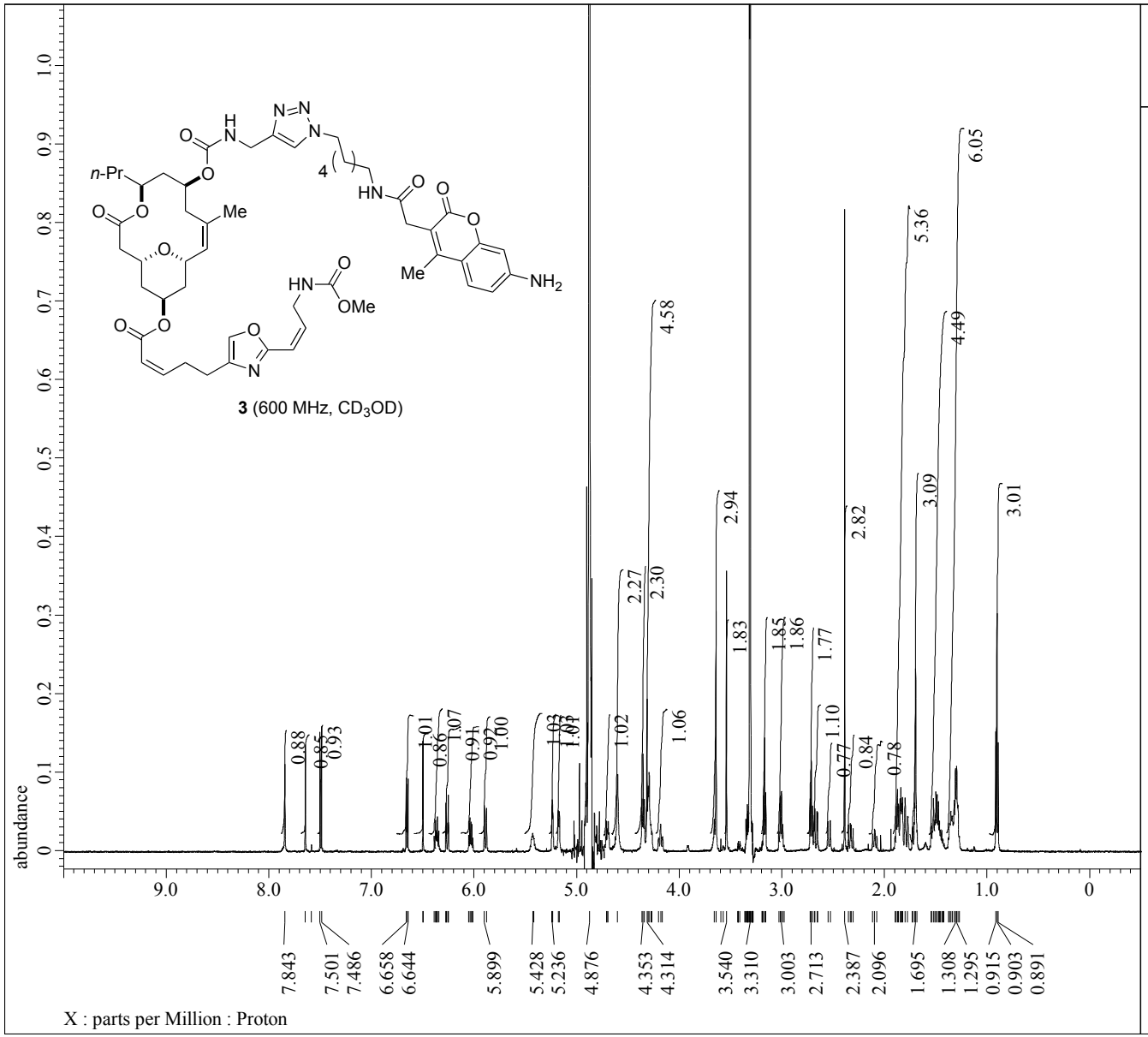
JEOL Solutions for Innovation

```
Filename          = SY-II-066_Carbon-1-4.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = SY-II-066
Solvent           = CHLOROFORM-D
Creation_Time     = 21-MAY-2015 18:52:36
Revision_Time     = 19-NOV-2016 13:13:36
Current_Time      = 19-NOV-2016 13:13:48

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 0.69206016[s]
X_Domain          = 13C
X_Freq            = 150.91343039[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.44496109[Hz]
X_Sweep           = 47.34848485[kHz]
X_Sweep_Clipped   = 37.87878788[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 200
Total_Scans       = 200

Relaxation_Delay = 2[s]
Recvr_Gain        = 56
Temp_Get          = 22.6[dC]
X_90_Width        = 10.5[us]
X_Acq_Time        = 0.69206016[s]
X_Angle           = 30[deg]
X_Atn             = 7.4[dB]
X_Pulse           = 3.5[us]
Irr_Atn_Dec       = 24.452[dB]
Irr_Atn_Noe       = 24.452[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69206016[s]
```

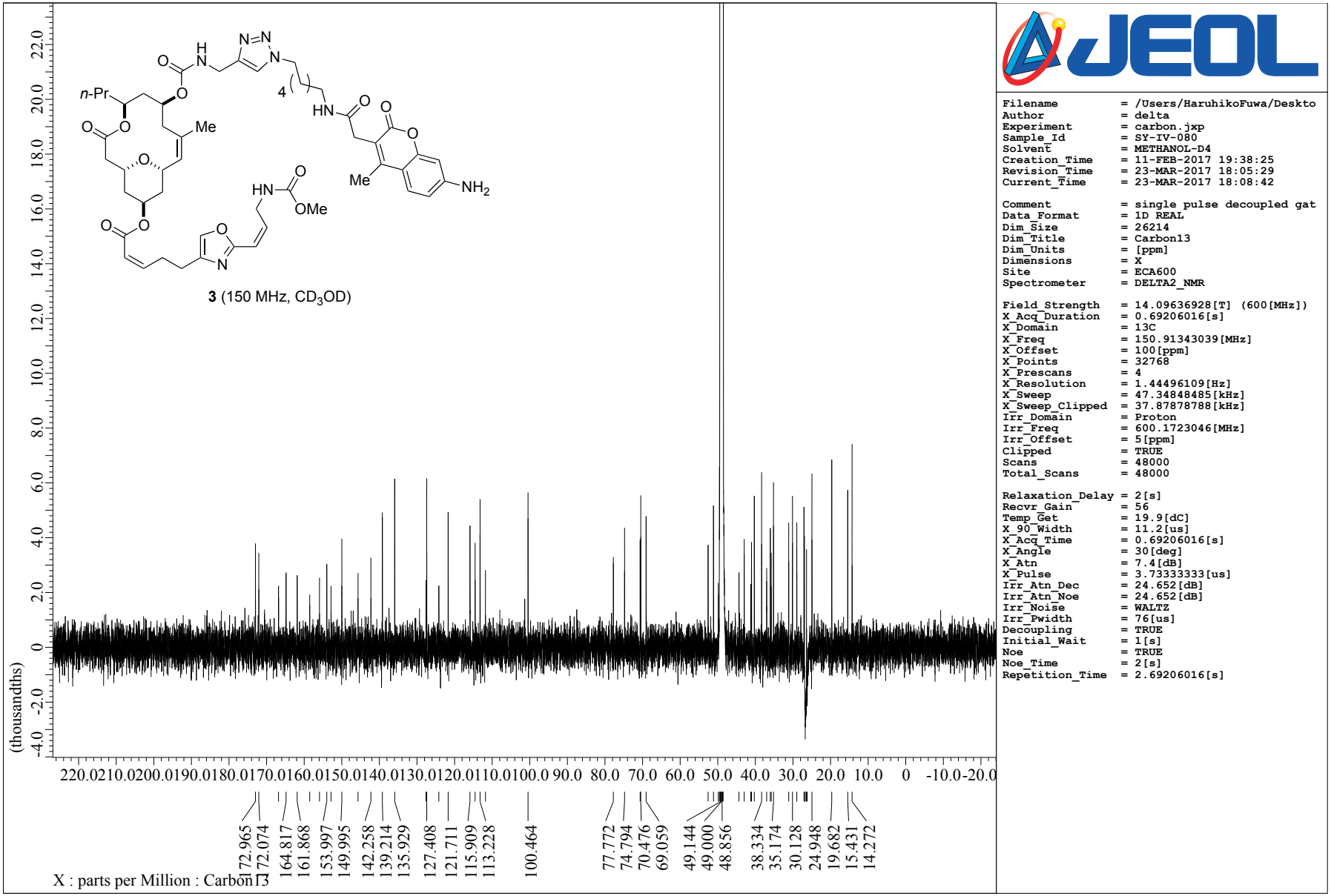


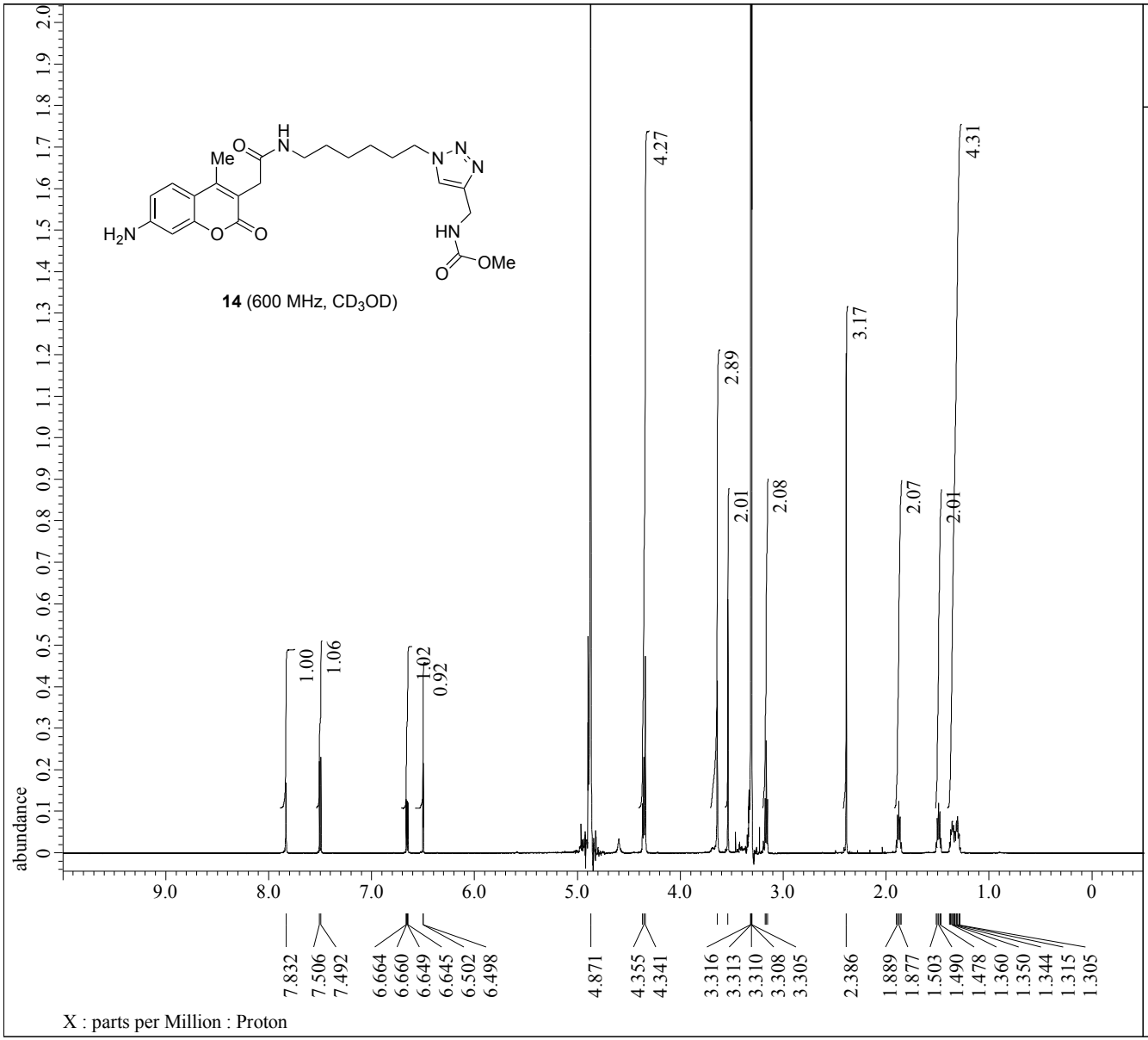

```
Filename          = SY-II-067_proton-1-3.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = SY-II-067HPLC-1
Solvent           = METHANOL-D4
Creation_Time     = 25-MAY-2015 09:56:38
Revision_Time     = 28-NOV-2016 23:15:39
Current_Time      = 28-NOV-2016 23:16:03

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 2.18103808[s]
X_Domain          = 1H
X_Freq            = 600.1723046[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.45849727[Hz]
X_Sweep           = 15.02403846[kHz]
X_Sweep_Clipped   = 12.01923077[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 600.1723046[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 2[s]
Recvr_Gain        = 42
Temp_Get          = 22[dC]
X_90_Width        = 7.215[us]
X_Acq_Time        = 2.18103808[s]
X_Angle           = 45[deg]
X_Atn             = 4[dB]
X_Pulse           = 3.6075[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 4.18103808[s]
```


n-Pr
Me
HN
OMe
4
HN
Me
NH2
3 (150 MHz, CD3OD)
(thousandths)
X : parts per Million : Carbon13
172.965
172.074
164.817
161.868
153.997
149.995
142.258
139.214
135.929
127.408
121.711
115.909
113.228
100.464
77.772
74.794
70.476
69.059
49.144
49.000
48.856
38.334
35.174
30.128
24.948
19.682
15.431
14.272
Filename = /Users/HaruhikoFuwa/Deskto
Author = delta
Experiment = carbon.jxp
Sample_Id = SY-IV-080
Solvent = METHANOL-D4
Creation_Time = 11-FEB-2017 19:38:25
Revision_Time = 23-MAR-2017 18:05:29
Current_Time = 23-MAR-2017 18:08:42
Comment = single pulse decoupled gat
Data_Format = 1D REAL
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 14.09636928[T] (600[MHz])
X_Acq_Duration = 0.69206016[s]
X_Domain = 13C
X_Freq = 150.91343039[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.44496109[Hz]
X_Sweep = 47.34848485[kHz]
X_Sweep_Clipped = 37.87878788[kHz]
Irr_Domain = Proton
Irr_Freq = 600.1723046[MHz]
Irr_Offset = 5[ppm]
Clipped = TRUE
Scans = 48000
Total_Scans = 48000
Relaxation_Delay = 2[s]
Recvr_Gain = 56
Temp_Get = 19.9[dC]
X_90_Width = 11.2[us]
X_Acq_Time = 0.69206016[s]
X_Angle = 30[deg]
X_Atn = 7.4[dB]
X_Pulse = 3.73333333[us]
Irr_Atn_Dec = 24.652[dB]
Irr_Atn_Noe = 24.652[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 76[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.69206016[s]

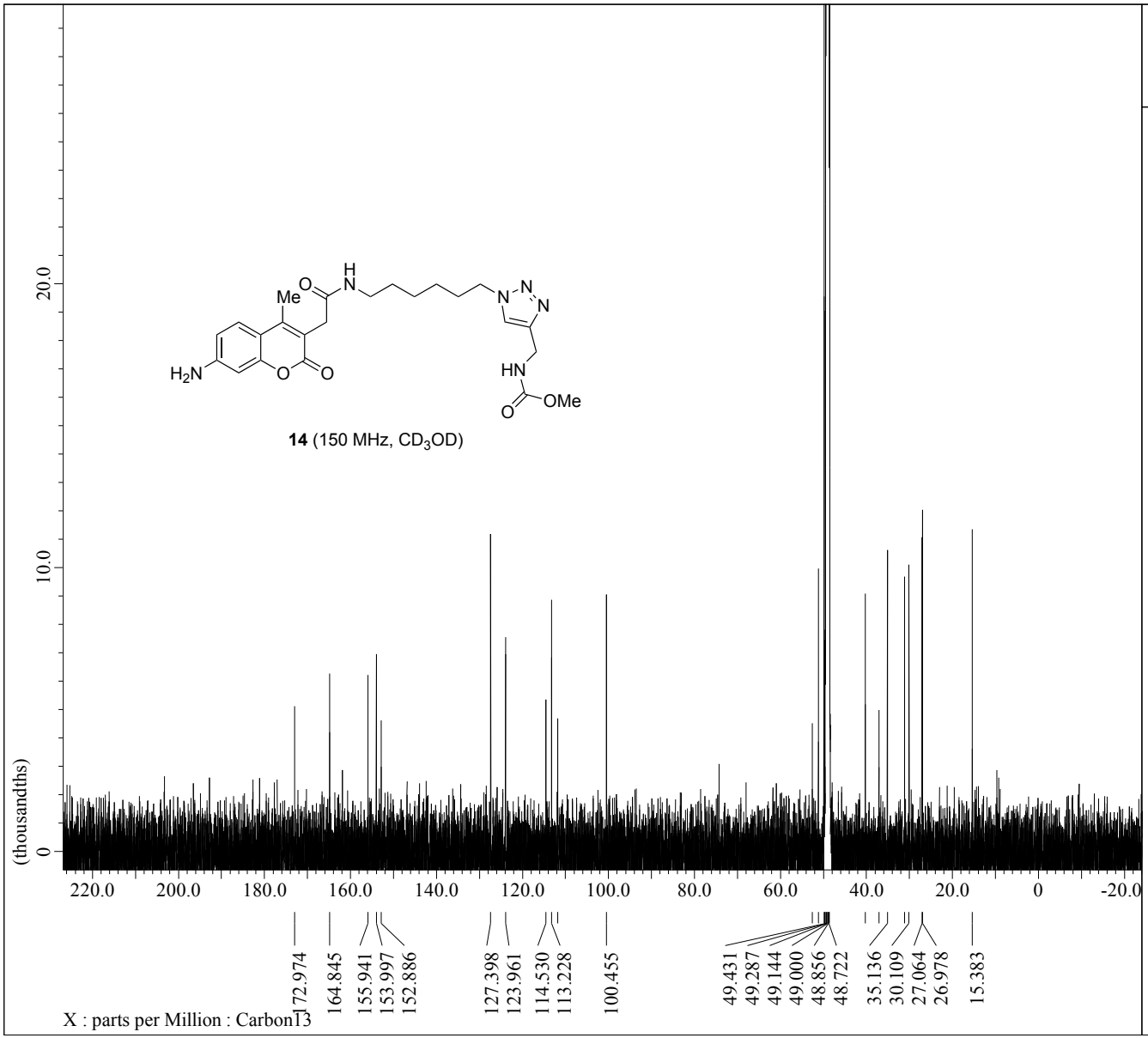



```
Filename          = SY-IV-012_proton-3-4.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = SY-IV-012
Solvent           = METHANOL-D4
Creation_Time     = 19-OCT-2016 23:05:45
Revision_Time     = 19-NOV-2016 16:41:40
Current_Time      = 19-NOV-2016 16:42:02

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 2.18103808[s]
X_Domain          = 1H
X_Freq            = 600.1723046[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.45849727[Hz]
X_Sweep           = 15.02403846[kHz]
X_Sweep_Clipped   = 12.01923077[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 600.1723046[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 32
Total_Scans       = 32

Relaxation_Delay  = 2[s]
Recvr_Gain        = 44
Temp_Get          = 22.2[dC]
X_90_Width        = 7.05[us]
X_Acq_Time        = 2.18103808[s]
X_Angle           = 45[deg]
X_Atn             = 4[dB]
X_Pulse           = 3.525[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 4.18103808[s]
```



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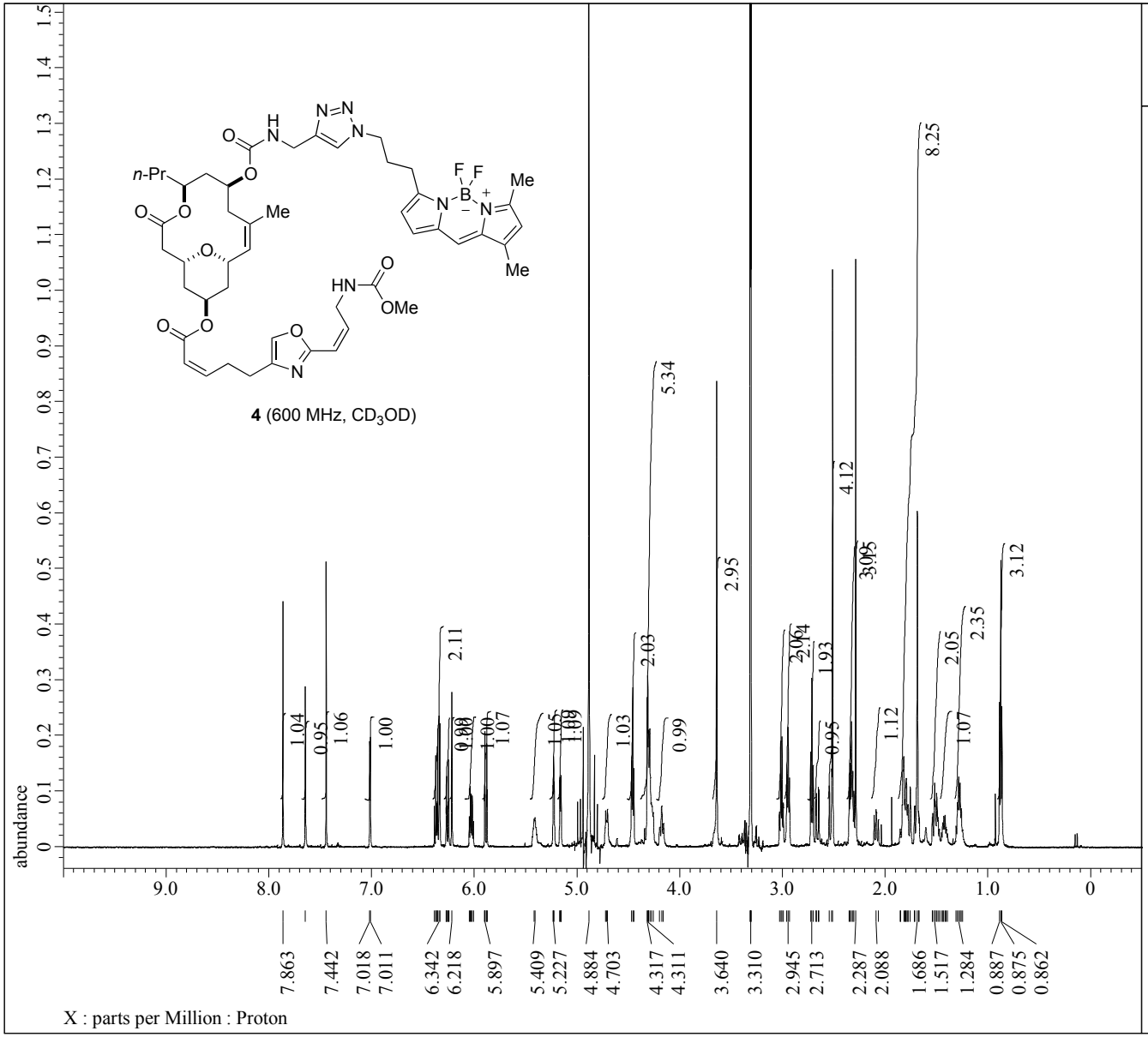
```
Filename          = SY-IV-012_Carbon-2-6.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = SY-IV-012
Solvent           = METHANOL-D4
Creation_Time     = 19-OCT-2016 23:26:06
Revision_Time     = 19-NOV-2016 13:18:38
Current_Time      = 19-NOV-2016 13:20:30

Comment           = single pulse decoupled gat
Data_Format       = 1D REAL
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 0.69206016[s]
X_Domain          = 13C
X_Freq            = 150.91343039[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.44496109[Hz]
X_Sweep           = 47.34848485[kHz]
X_Sweep_Clipped   = 37.87878788[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 13200
Total_Scans       = 13200

Relaxation_Delay  = 2[s]
Recvr_Gain        = 56
Temp_Get          = 22.4[dC]
X_90_Width        = 12[us]
X_Acq_Time        = 0.69206016[s]
X_Angle           = 30[deg]
X_Atn             = 7.4[dB]
X_Pulse           = 4[us]
Irr_Atn_Dec       = 24.652[dB]
Irr_Atn_Noe       = 24.652[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69206016[s]
```

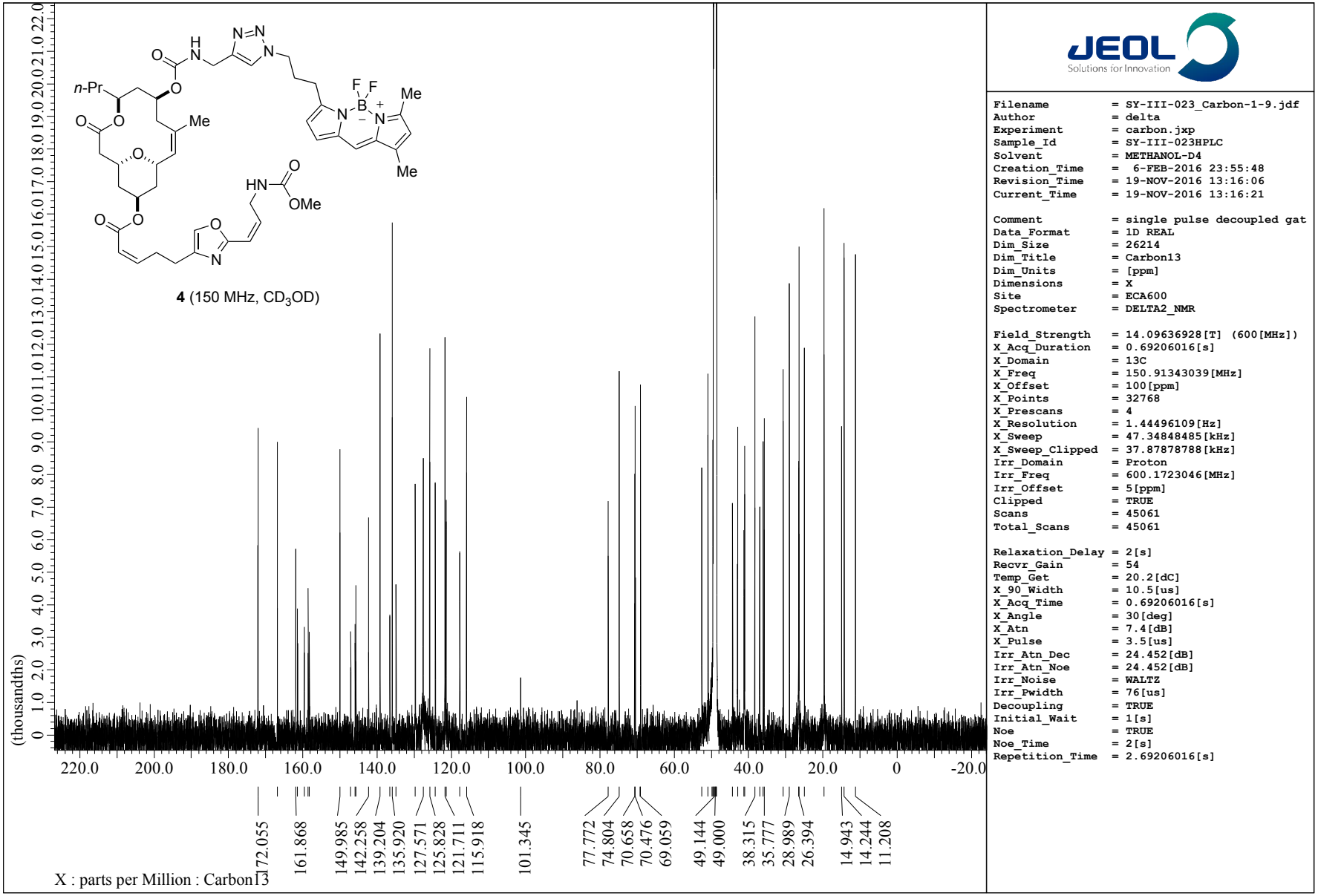


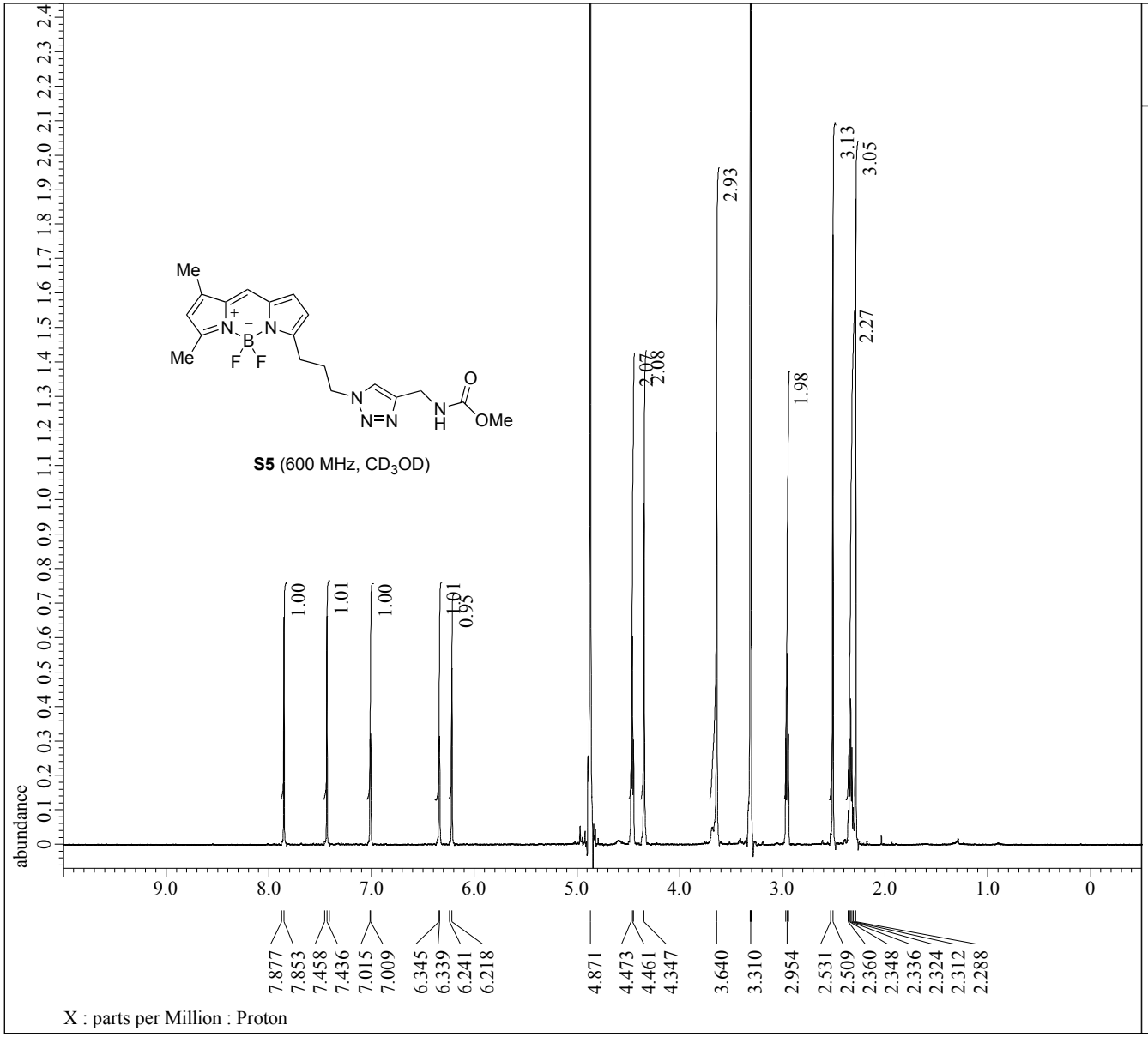

```
Filename          = SY-III-023_proton-1-4.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = SY-III-023HPLC
Solvent           = METHANOL-D4
Creation_Time     =  6-FEB-2016 23:50:53
Revision_Time     = 19-NOV-2016 16:45:49
Current_Time      = 19-NOV-2016 16:46:44

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 2.18103808[s]
X_Domain          = 1H
X_Freq            = 600.1723046[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.45849727[Hz]
X_Sweep           = 15.02403846[kHz]
X_Sweep_Clipped   = 12.01923077[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 600.1723046[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 2[s]
Recvr_Gain        = 42
Temp_Get          = 20.6[dC]
X_90_Width        = 7.215[us]
X_Acq_Time        = 2.18103808[s]
X_Angle           = 45[deg]
X_Atn             = 4[dB]
X_Pulse           = 3.6075[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 4.18103808[s]
```


4 (150 MHz, CD3OD)
(thousandths)
X : parts per Million : Carbon13
172.055
161.868
149.985
142.258
139.204
135.920
127.571
125.828
121.711
115.918
101.345
77.772
74.804
70.658
70.476
69.059
49.144
49.000
38.315
35.777
28.989
26.394
14.943
14.244
11.208
Filename = SY-III-023_Carbon-1-9.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = SY-III-023HPLC
Solvent = METHANOL-D4
Creation_Time = 6-FEB-2016 23:55:48
Revision_Time = 19-NOV-2016 13:16:06
Current_Time = 19-NOV-2016 13:16:21
Comment = single pulse decoupled gat
Data_Format = 1D REAL
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 14.09636928[T] (600[MHz])
X_Acq_Duration = 0.69206016[s]
X_Domain = 13C
X_Freq = 150.91343039[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.44496109[Hz]
X_Sweep = 47.34848485[kHz]
X_Sweep_Clipped = 37.87878788[kHz]
Irr_Domain = Proton
Irr_Freq = 600.1723046[MHz]
Irr_Offset = 5[ppm]
Clipped = TRUE
Scans = 45061
Total_Scans = 45061
Relaxation_Delay = 2[s]
Recvr_Gain = 54
Temp_Get = 20.2[dC]
X_90_Width = 10.5[us]
X_Acq_Time = 0.69206016[s]
X_Angle = 30[deg]
X_Atn = 7.4[dB]
X_Pulse = 3.5[us]
Irr_Atn_Dec = 24.452[dB]
Irr_Atn_Noe = 24.452[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 76[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.69206016[s]



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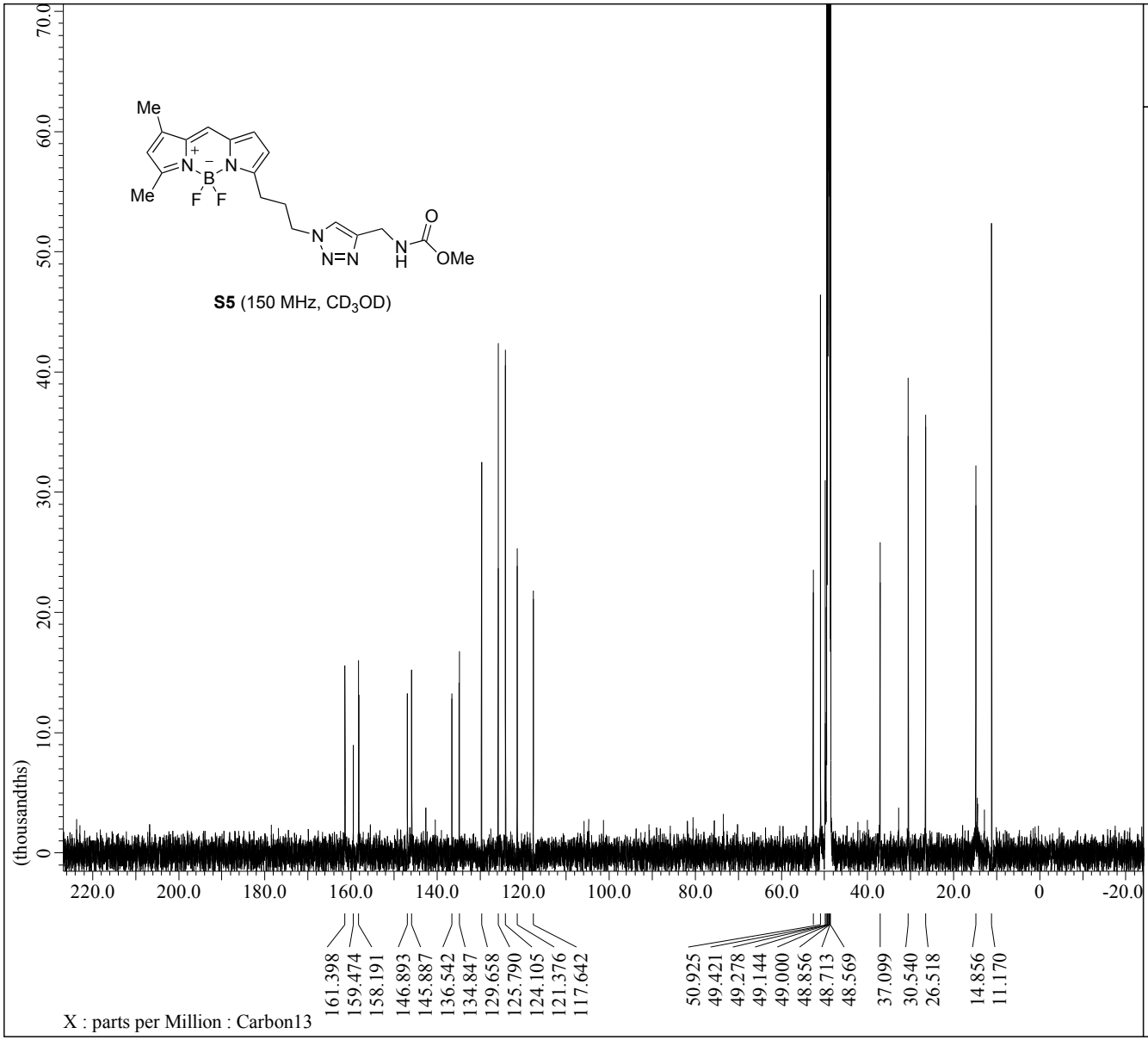
```
Filename          = SY-II-116_proton-1-3.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = SY-II-116-HPLC
Solvent           = METHANOL-D4
Creation_Time     = 25-AUG-2015 12:43:59
Revision_Time     = 19-NOV-2016 16:20:06
Current_Time      = 19-NOV-2016 16:20:52

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 2.18103808[s]
X_Domain          = 1H
X_Freq            = 600.1723046[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.45849727[Hz]
X_Sweep           = 15.02403846[kHz]
X_Sweep_Clipped   = 12.01923077[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 600.1723046[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 2[s]
Recvr_Gain        = 46
Temp_Get          = 22.2[dC]
X_90_Width        = 7.215[us]
X_Acq_Time        = 2.18103808[s]
X_Angle           = 45[deg]
X_Atn             = 4[dB]
X_Pulse           = 3.6075[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 4.18103808[s]
```



JEOL Solutions for Innovation

```
Filename          = SY-II-116_Carbon-2-4.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = SY-II-116
Solvent           = METHANOL-D4
Creation_Time     = 30-OCT-2016 01:52:34
Revision_Time     = 19-NOV-2016 13:14:38
Current_Time      = 19-NOV-2016 13:15:04

Comment           = single pulse decoupled gat
Data_Format       = 1D REAL
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

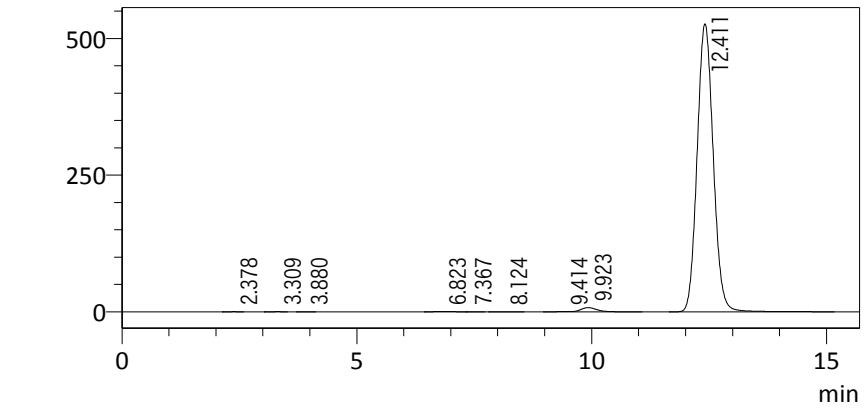
Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 0.69206016[s]
X_Domain          = 13C
X_Freq            = 150.91343039[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.44496109[Hz]
X_Sweep           = 47.34848485[kHz]
X_Sweep_Clipped   = 37.87878788[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 9600
Total_Scans       = 9600

Relaxation_Delay  = 2[s]
Recvr_Gain        = 54
Temp_Get          = 21.9[dC]
X_90_Width        = 12[us]
X_Acq_Time        = 0.69206016[s]
X_Angle           = 30[deg]
X_Atn             = 7.4[dB]
X_Pulse           = 4[us]
Irr_Atn_Dec       = 24.652[dB]
Irr_Atn_Noe       = 24.652[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69206016[s]
```

HPLC chromatogram of AMCA derivative **3**

<<chromatogram peak report>>

Sample : SY-II-067
ID : SY-II-067
Data Name : SY-II-067 .lcd
Method Name : 0.5 ml 55%MeCN.lcm
Inj. Volume : 10 uL
Acquisition Date : 2015/05/27 23:34:32
Modified Date : 2015/05/27 23:50:16



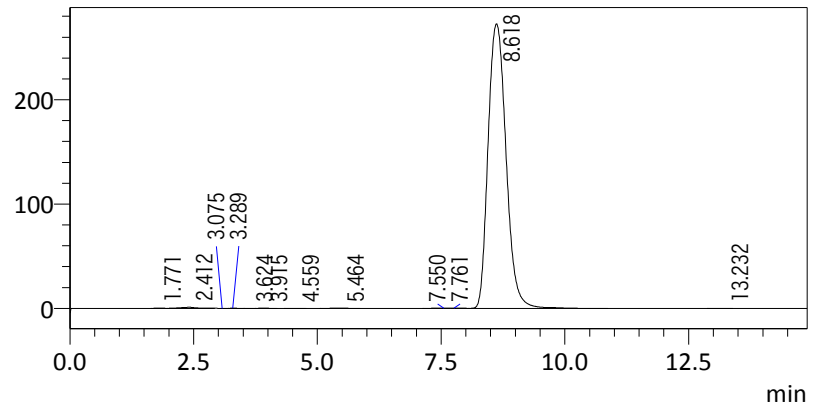
detectorA Ch-1 254nm

peak #	retention time	peak area%
1	2.378	0.021
2	3.309	0.034
3	3.880	0.009
4	6.823	0.138
5	7.367	0.014
6	8.124	0.025
7	9.414	0.056
8	9.923	1.455
9	12.411	98.249

HPLC chromatogram of AMCA derivative **14**

<<chromatogram peak report>>

Sample : SY-IV-012
ID : SY-IV-012
Data Name : SY-IV-012 .lcd
Method Name : 0.5 ml 30%MeCN.lcm
Inj. Volume : 10 uL
Acquisition Date : 2016/10/19 22:22:30
Modified Date : 2016/10/19 22:37:25



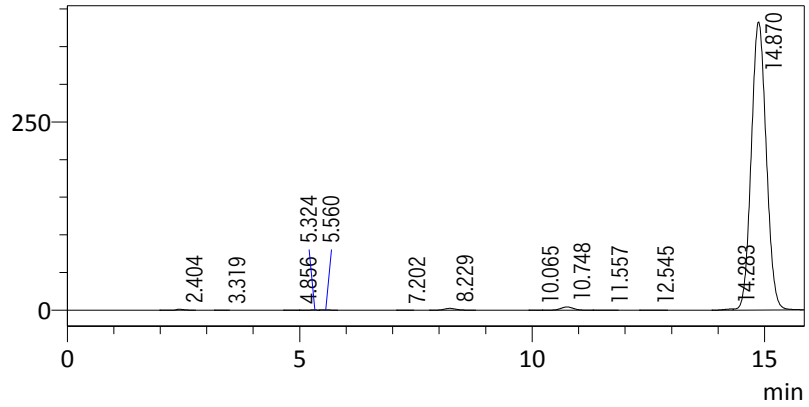
detectorA Ch-1 254nm

peak #	retention time	peak area%
1	1.771	0.047
2	2.412	0.397
3	3.075	0.015
4	3.289	0.031
5	3.624	0.020
6	3.915	0.048
7	4.559	0.020
8	5.464	0.065
9	7.550	0.057
10	7.761	0.044
11	8.618	99.224
12	13.232	0.031

HPLC chromatogram of BODIPY derivative **4**

<<chromatogram peak report>>

Sample : SY-II-102
ID : SY-II-102
Data Name : SY-II-102 .lcd
Method Name :65% 0.5ml min.lcm
Inj. Volume : 10 uL
Acquisition Date : 2015/07/23 18:30:44
Modified Date : 2015/07/23 18:46:36



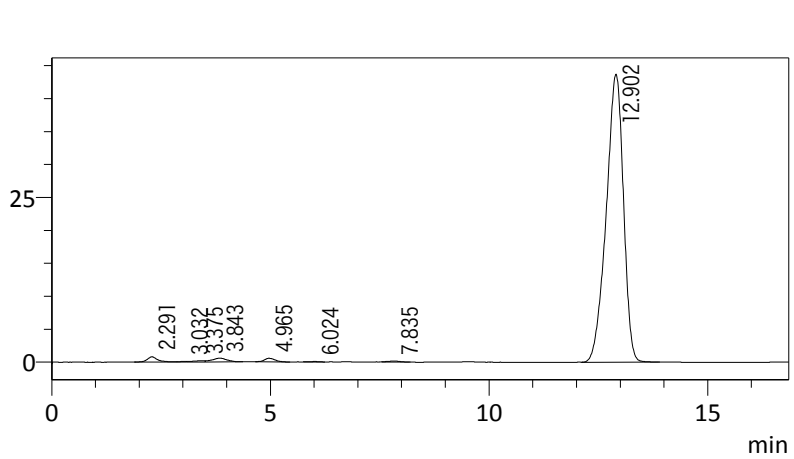
detectorA Ch-1 254nm

peak #	retention time	peak area%
1	2.404	0.177
2	3.319	0.012
3	4.856	0.016
4	5.324	0.043
5	5.560	0.075
6	7.202	0.021
7	8.229	0.490
8	10.065	0.015
9	10.748	0.944
10	11.557	0.020
11	12.545	0.023
12	14.283	0.227
13	14.870	97.937

HPLC chromatogram of BODIPY derivative **S5**

<<chromatogram peak report>>

Sample : SY-II-116
ID : SY-II-116
Data Name : SY-II-116 .lcd
Method Name :0.5 ml 45%MeCN.lcm
Inj. Volume : 10 uL
Acquisition Date : 2015/08/24 20:30:44
Modified Date : 2015/08/24 20:47:36



detectorA Ch-1 254nm

peak #	retention time	peak area%
1	2.291	1.135
2	3.032	0.087
3	3.375	0.294
4	3.843	1.095
5	4.965	0.766
6	6.024	0.100
7	7.835	0.245
8	12.902	96.279