Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Supporting information

Unusual (*Z*)-Selective Palladium(II)-Catalysed Addition of Aryl Boronic Acids to Vinylaziridines

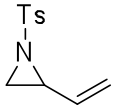
JieXiang Yin, Theresa Mekelburg and Christopher Hyland*

Experimental Section

General procedures

Reagents were purchased from commercial sources and used with no further purification. Reactions were performed under and atmosphere of nitrogen in ovendried glassware. Anhydrous solvents were purified by passage through an Innovative Technologies solvent purification system (purification over activated alumina, copper catalyst, and/or molecular sieves). Flash grade silica gel (32-63 µm) was used for column chromatography and thin layer chromatography was performed on silica gel 60 F254 aluminium backed sheets visualized with a 254 nm UV lamp and stained with either ceric(IV) dip containing phosphomolybdic acid (37.5 g), ceric sulphate (7.5 g), sulphuric acid (37.5 ml) and water (750 ml) or potassium permanganate dip containing potassium permanganate (1.5 g), potassium carbonate (10 g) and sodium hydroxide solution (10%, 1.25 ml) and water (200 ml). ^{1}H NMR and ^{13}C NMR were recorded in deuterated chloroform ($CDCl_3$) on a Mercury 2000 Spectrometer operating at 300 MHz and 75 MHz respectively or on a Varian 500 MHz spectrometer in chloroform-d3. Coupling constants are recorded in Hz and chemical shifts are recorded as δ values in ppm, referenced against residual solvent peaks (^{1}H NMR) or to the ^{13}C resonances of the solvent. The following abbreviations are used in the assignments of 1H NMR signals; s = singlet; d =doublet; td= doublet of triplet; tdd = triplet of doublet of doublet; m= multiplet. Mass spectrometry samples were analyzed by UPLC-MS using a Water Acquity H-series UPLC coupled to a Waters Xeco triple quadrupole mass spectrometer within the Central Science Laboratory at the University of Tasmania or on the at the University of Wollongong. Infrared spectra were recorded on a Shimadzu FTIR 8400s spectrometer as thin films on NaCl plates.

Synthesis of 1-tosyl-2-vinyl aziridine 1



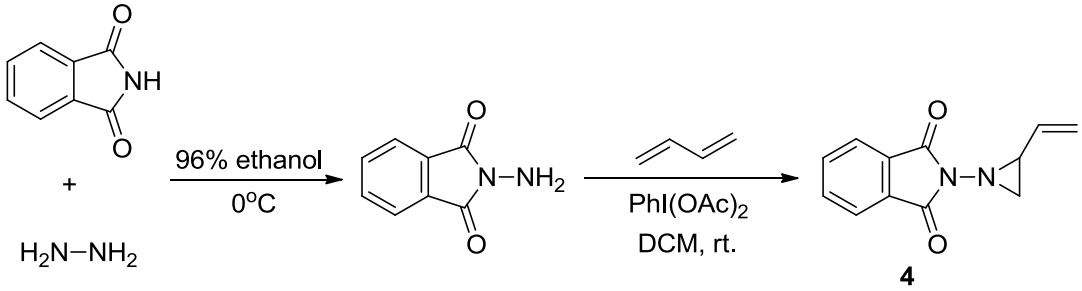
1

Chloramine-T (2.57 g, 11.3 mmol) and phenyltrimethylammonium tribromide (10 mol%) were dissolved in MeCN (42 ml). With vigorous stirring, the reaction flask was then purged with 1,4-butadiene and then sealed. After 24 h, the reaction mixture was passed through a pad of silica gel (5 cm), evaporated and purified by flash column chromatography (30% ethyl acetate/ hexane) to give the desired product as white crystals (813 mg, 60%). The data was in accordance to that previously reported.[1] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.81 (d, *J* = 3.6 Hz, 2H) 7.32 (d, *J* = 3.6 Hz, 2H), 5.50 (m, 2H) 5.25 (m, 1H) 3.26 (m, 1H) 2.79 (d, *J* =7.1 Hz, 1H) 2.47 (s, 3H), 2.21 (d, *J* = 7.1 Hz, 1H).

---

[1] S. L. Ali, M. D. Nikalje and A. Sudalai, *Org. Lett.,* 1999, **1**, 705-707.

Synthesis of 2-(2-vinylaziridin-1-yl)isoindoline-1,3-dione 4:



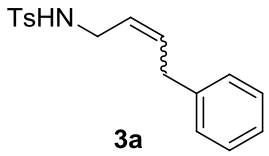
Phthalimide (1.50 g, 10.2 mmol, 1.0 eq) was dissolved in 96% ethanol (10 ml). Hydrazine (0.45 ml, 11.2 mmol, 1.1 eq, 80% water solution of $N_2H_4{\cdot}H_2O$) was subsequently added drop-wise with stirring. The resulting solution was stirred at 0°C for overnight, then diluted with dichloromethane (20 ml), quenched with distilled water (3×20 ml), dried over $MgSO_4$ and concentrated by evaporation. The crude product was recrystalised with 96% ethanol to afford white solids (893 mg, 54%). ^{1}H NMR (500Mz, $CDCl_3$) δ 7.89-7.85 (m, 2H), 7.77-7.73 (m, 2H), 4.14 (s, 2H).

N-amino-phthalimide (893 mg, 5.51 mmol, 1.0 eq) and $PhI(OAc)_2$ (1.33 g, 4.13 mmol, 0.75 eq) were dissolved in anhydrous dichloromethane (27.6 ml). The solution was bubbled with 1,3-butadiene (1 atm) at room temperature for 30 mins with stirring, then diluted with dichloromethane (30 ml), quenched with distilled water (3×30 ml), dried over $MgSO_4$ and concentrated by evaporation. The crude product was finally purified with 35% ethyl acetate in *n*-pentane to give yellow solids (984 mg, 83%). ^{1}H NMR (500MHz, $CDCl_3$ data for major rotamer) δ 7.78-7.77 (m, 2H), 7.69 (m, 2H), 5.80-5.73 (m, 1H), 5.55 (d, *J* = 17.0 Hz, 1H), 5.37 (d, *J* = 10.0 Hz, 1H), 3.08 (q, *J* = 7.0 Hz, 1H), 2.72 (d, *J* = 8.0 Hz, 1H), 2.52 (d, *J* = 5.5 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$ data for major rotamer) δ 165.3, 134.4, 134.3, 130.6, 123.2, 119.9, 119.8, 44.2, 38.9. HRMS (ES): *m/z* 215.0821 $[C_{12}H_{10}N_2O_2+H]^+$, calcd. 215.0815. IR ν(cm^{-1}): 2364, 1715, 1464, 1378, 1184, 1064, 1053, 937, 887, 813, 706.

General procedure for boronic acid addition to 1-tosyl-2-vinyl aziridine 3a-i

In a typical experiment, $Pd(OAc)_2$ (10 mol%), 1,10-phenanthroline (15 mol%) and $AgSbF_6$ (10 mol%) were dissolved in DCE (0.14 M). The resulting mixture was ultrasonically treated for 1 min, and then stirred at room temperature for 1 min. 1-tosyl-2-vinylaziridine (1.0 eq), phenylboronic acid (1.5 eq), KOAc (0.5 eq if needed) were then added. The mixture was then heated at 70℃ for 22 h, and then diluted with ethyl acetate, passed through a short pad of silica gel, evaporated and finally purified with silica gel chromatography (typically 30% ethyl acetate/hexane) to give the allyl sulfonamide.

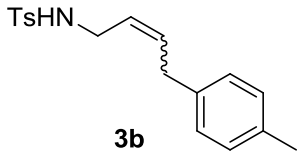
Synthesis of (E/Z)-4-methyl-N-(4-phenylbut-2-enyl)benzenesulfonamide 3a



3a

Following the general procedure, using 30 mg, 0.135 mmol of 1-tosyl-2-vinyl aziridine and phenylboronic acid with no addition of KOAc yielded the title compound as a clear oil (32 mg, 79%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.88-7.71 (m, 2H, *E* and *Z* isomer) 7.31-7.19 (m, 4H, *E* and *Z* isomer), 7.24-7.22 (m, 1H, *E* and *Z* isomer), 7.10-7.06 (m, 2H, *E* and *Z* isomer), 5.80-5.65 (m, 1H, *E* and *Z* isomer), 5.48- 5.35 (m, 1H, *E* and *Z* isomer), 4.48 (s, 1H, *E* and *Z* isomer), 3.70 (t, *J* = 7.0 Hz, 1H, *E* isomer), 3.54 (t, *J* = 7.0 Hz, *Z* isomer), 3.28 (m, *E +Z* isomer), 2.43 (s, 3H, *E* and *Z* isomer). ^{13}C NMR (126 MHz, $CDCl_3$) δ 143.5, 143.4, 139.7, 139.6, 137.0, 133.5, 132.8, 129.7, 129.7, 128.6, 128.5, 128.2, 127.2, 126.5, 126.3, 125.9, 124.9, 45.2, 40.1, 38.5, 33.4, 21.5. IR ν(cm^{-1}): 3274, 2920, 1599, 1404, 1325, 1119, 1093. HRMS (ES): *m/z* 302.1203 $[C_{17}H_{19}N_1O_2S_1 + H]^+$, calcd. 302.1215.

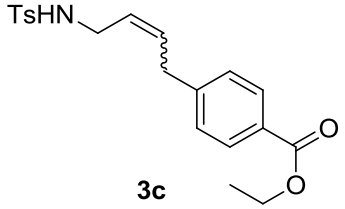
Synthesis of (E/Z)-4-methyl-N-(p-methylphenylbut-2-enyl)benezenesulfonamide 3b



3b

Following the general procedure, using 25 mg, 0.112 mmol of 1-tosyl-2-vinyl aziridine with tolylboronic acid yielded the title compound as a clear oil (14.2 mg, 40%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.75 (d, *J* = 8.5 Hz, 2H, *E* isomer), 7.73 (d, *J* = 9.0 Hz, 2H, *Z* isomer), 7.32-7.28 (m, 2H, *E* and *Z* isomer), 7.08-7.07 (m, 2H, *E* and *Z* isomer), 6.97-6.95 (m, 2H, *E* and *Z* isomer), 5.73-5.67 (m, 1H, *E* and *Z* isomer), 5.44- 5.39 (m, 1H, *E* and *Z* isomer), 4.28-4.27 (m, 1H, *E* and *Z* isomer), 3.71 (t, *J* = 6.5 Hz, 1H, *E* isomer) 3.55 (t, *J* = 6 Hz, *Z* isomer) 3.27-3.23 (m, 2H, *E* and *Z* isomer) 2.43 (s, 3H, *E* and *Z* isomer) 2.31 (s, 3H, *E* and *Z* isomer).^{13}C NMR (125 MHz, $CDCl_3$) δ 143.6, 136.6, 135.8, 133.2, 129.8, 129.7, 129.3, 129.2, 128.4, 128.1, 127.2, 127.2, 124.6, 45.2, 40.3, 38.2, 32.6, 22.0, 21.7. IR ν(cm^{-1}): 3239, 2923, 1663, 1436, 1408, 1327, 1159, 1094. HRMS (ES): *m/z* 338.1197 $[C_{18}H_{21}N_1O_2S_1 + Na]^+$, calcd. 338.1191.

Synthesis of (E/Z)-4-methyl-N-(4-ethoxycarbonylphenylbut-2-enyl)benezenesulfonamide 3c

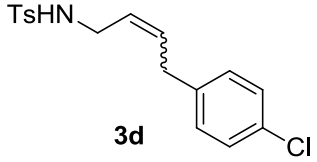


3c

Following the general procedure, using 25 mg, 0.112 mmol of 1-tosyl-2-vinyl aziridine and 4-(ethoxycarbonyl)phenylboronic acid yielded the title compound as a

clear oil (39.3 mg, 94%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.77-7.71 (m, 2H, *E* and *Z* isomer), 7.32-7.26 (m, 2H, *E* and *Z* isomer), 7.09-7.06 (2H, m, *E* and *Z* isomer), 6.99-6.94 (2H, m, *E* and *Z* isomer), 5.80-5.65 (m, 1H, *E* and *Z* isomer), 5.48-5.35 (m, 1H, *E* and *Z* isomer), 4.34 (s, 1H, *E* and *Z* isomer), 3.70 (t, *J* = 7.0 Hz, 1H, *Z* isomer) 3.54 (t, *J* = 7.0 Hz, *E* isomer) 3.24 (t, *J* = 7.0 Hz, *E* and *Z* isomer) 2.42 (s, 3H, *E* and *Z* isomer) 2.31 (s, 3H, *E* and *Z* isomer).^{13}C NMR (125 MHz, $CDCl_3$) δ 166.8, 166.7, 145.1, 145.0, 143.8, 143.7, 137.2, 137.1, 132.5, 131.9, 130.1, 130.0, 129.9, 128.8, 128.7, 128.4, 127.4, 127.4, 127.0, 126.0, 61.1, 45.3, 40.3, 38.6, 33.6, 21.8, 21.7, 14.6. IR ν(cm^{-1}): 3142, 2976, 2921, 2903, 1700, 1609, 1442, 1179, 1107. HRMS (ES): *m/z* 396.1237 $[C_{20}S_1O_4N_1H_{23} + Na]^+$, calcd. 396.1245.

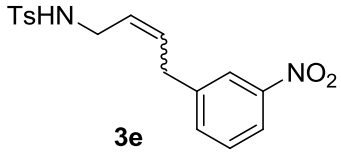
Synthesis of (E/Z)-4-methyl-N-(4-chlorophenylbut-2-enyl)benezenesulfonamide 3d



3d

Following the general procedure, using 25 mg, 0.112 mmol of 1-tosyl-2-vinyl aziridine and 4-chlorophenylboronic acid yielded the title compound as a clear oil (21.4 mg, 57%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.5 Hz, 2H, *E* isomer), 7.76 (d, *J* = 9.0 Hz, 2H, *Z* isomer), 7.35-7.31 (m, 2H, *E* and *Z* isomer), 7.30-7.24 (m, 2H, *E* and *Z* isomer), 7.05-7.03 (m, 2H, *E* and *Z* isomer), 5.74- 5.61 (m, 1H, *E* and *Z* isomer), 5.48-5.35 (m, 1H, *E* and *Z* isomer), 4.35-4.34 (m, 1H, *E* and *Z* isomer), 3.72 (t, *J* = 7.0 Hz, 1H, *E* isomer), 3.60 (t, *J* = 7.0Hz, 1H, *Z* isomer), 3.32 (d, *J* = 7.5 Hz, 2H, *E* isomer), 3.28 (d, *J* = 7.0 Hz, 2H, *Z* isomer), 2.42 (s, 3H, *E* and *Z* isomer). ^{13}C NMR (125 MHz, $CDCl_3$) δ 143.6, 138.0, 137.9, 132.8, 132.2, 132.1, 129.9, 129.8, 129.7, 129.6, 128.7, 128.6, 127.2, 127.2, 126.4, 125.4, 45.1, 40.1, 37.8, 32.8, 21.6. IR ν(cm^{-1}): 3263, 2971, 2923, 1598, 1490, 1404, 1326, 1185, 1093. HRMS (ES): *m/z* 358.0637 $[C_{17}S_1O_2N_1Cl_1H_{18} + Na]^+$, calcd. 358.0644.

Synthesis of (E/Z)-4-methyl-N-(3-nitrophenylbut-2-enyl)benezenesulfonamide 3e

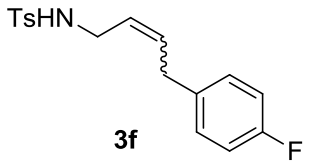


3e

Following the general procedure, using 25 mg, 0.112 mmol of 1-tosyl-2-vinyl aziridine and 4-nitrophenylboronic acid yielded the title compound as a green oil (34.9 mg, 90%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.10-8.08 (m, 1H, *E* and *Z* isomer), 7.98 (br s, 1H, *E* and *Z* isomer), 7.81-7.76 (m, 2H, *E* and *Z* isomer), 7.48-7.47 (2H, m, *E* and *Z* isomer), 7.35-7.32 (2H, m *E* and *Z* isomer), 5.77- 5.63 (m, 1H, *E* and *Z* isomer), 5.58-5.55 (m, 1H, *E* isomer), 5.54-5.47 (m, 1H, *Z* isomer), 4.72 (t, *J* = 5.0 Hz, *E* isomer), 4.67 (t, *J* = 6.0 Hz, *Z* isomer), 3.75 (t, *J* = 6.5 Hz, 1H, *E* isomer), 3.62 (t, *J* = 6.5 Hz, *Z* isomer), 3.49 (d, *J* = 8.0 Hz, 1H, *E* isomer), 3.42 (d, *J* = 6.5 Hz, 2H, *Z* isomer), 2.46 (s, 3H, *E* and *Z* isomer). ^{13}C NMR (125 MHz, $CDCl_3$) δ 148.6, 144.0, 143.9, 141.9, 141.8, 137.2, 137.0, 135.0, 134.8, 131.6, 131.0, 130.4 130.0, 129.9, 129.7, 129.6, 127.9,

127.4, 127.3, 126.8, 123.6, 123.3, 121.7, 45.2, 40.3, 38.2, 33.2, 21.8. IR ν(cm^{-1}): 3410, 2921, 1591, 1383, 1044. HRMS (ES): *m/z* 347.1059 $[C_{17}S_1O_4N_2H_{18} + H]^+$, calcd. 347.1066.

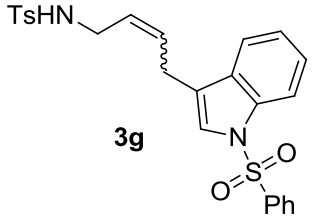
Synthesis of (E/Z)-4-methyl-N-(4-Fluorophenylbut-2-enyl)benezenesulfonamide 3f



3f

Following the general procedure, using 25 mg, 0.112 mmol of 1-tosyl-2-vinyl aziridine and 4-fluorophenylboronic acid yielded the title compound as a clear oil (26.8 mg, 75%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.81-7.75 (m, 2H, *E* and *Z* isomer), 7.40-7.30 (m, 2H, *E* and *Z* isomer), 7.79 (d, *J* = 8.5 Hz, 2H, *E* isomer), 7.77 (d, *J* = 8.0 Hz, 2H, *Z* isomer), 7.35-7.32 (2H, m, *E* and *Z* isomer), 7.08-7.06 (m, 2H, *E* and *Z* isomer), 7.00-6.97 (m, 2H, *E* and *Z* isomer), 5.80- 5.65 (m, 1H, *E* and *Z* isomer), 5.50- 5.38 (m, 1H, *E* and *Z* isomer), 4.42-4.38 (m, 1H, *E* and *Z* isomer), 3.73 (t, *J* = 6.9 Hz, 1H, *E* isomer), 3.60 (t, *J* = 6.2 Hz, 1H, *Z* isomer), 3.32 (d, *J* = 7.5 Hz, 2H, *E* isomer), 3.29 (d, *J* = 7.0 Hz, *Z* isomer), 2.43 (s, 3H, *E* and *Z* isomer). ^{13}C NMR (125 MHz, $CDCl_3$) δ 161.7 (d, *J* = 244 Hz), 161.7 (d, *J* = 242 Hz), 143.8, 143.7, 137.3, 137.1, 135.5 (d, *J* = 2.8 Hz), 135.3 (d, *J* = 2.8 Hz), 133.4, 132.7, 130.1 (d, *J* = 8.4 Hz), 130.0, 129.9 (d, *J* = 9.0 Hz), 129.8, 127.4, 127.4, 126.3, 125.3, 115.5 (d, *J* = 20.5 Hz), 115.4 (d, *J* = 21.4 Hz), 45.3, 40.3, 37.8, 32.8, 21.8. IR ν(cm^{-1}): 3421, 2971, 1655, 1378, 1089, 1047, 881. HRMS (ES): *m/z* 358.0694 $[C_{17}S_1O_2N_1F_1H_{18} + K]^+$, calcd. 358.0679.

Synthesis of (E/Z)-4-methyl-N-(4-(1-(phenylsulfonyl)-1H-indol-3-yl)but-2-en-1-yl)benzenesulfonamide 3g

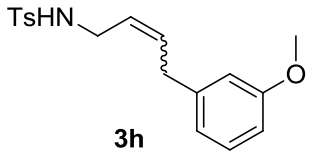


3g

Following the general procedure, using 25 mg, 0.112 mmol of 1-tosyl-2-vinyl aziridine and 4-nitrophenylboronic acid yielded the title compound as a green oil (20.4 mg, 38%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.0 Hz, 1H, *E* and *Z* isomer) 7.85 (d, *J* = 8.0 Hz, 2H, *E* and *Z* isomer) 7.73-7.70 (m, 2H, *E* and *Z* isomer), 7.52-7.21 (m, 1H, *E* and *Z* isomer), 7.43-7.40 (m, 2H, *E* and *Z* isomer), 7.37-7.36 (m, 1H, *E* and *Z* isomer), 7.33-7.21 (m, 5H, *E* and *Z* isomer), 5.82-5.76 (m, 1H, *E* and *Z* isomer), 5.56-5.51 (m, 1H, *E* and *Z* isomer), 4.34-4.33 (m, 1H, *E* and *Z* isomer), 3.74 (t, *J* = 6.5 Hz, 1H, *E* isomer), 3.59 (t, *J* = 6.0 Hz, 1H, *Z* isomer), 3.36-3.34 (m, 2H, *E* and *Z* isomer), 2.45 (s, 3H, *Z* isomer), 2.44 (s, 3H, *E* isomer). ^{13}C NMR (125 MHz, $CDCl_3$) δ 143.8, 143.8, 138.4, 137.2, 137.0, 135.6, 134.0, 131.2, 130.8, 130.7, 130.6, 130.0, 129.9, 129.5, 129.5, 127.4, 127.0, 126.9, 126.4, 125.2, 125.1, 123.4, 123.4, 123.1, 121.3, 121.1, 119.8, 119.6, 114.0, 45.3, 40.3, 28.0, 23.3, 21.7. IR ν(cm^{-1}): 3315, 2973, 2875,

1627, 1455, 1379, 1088, 1045, 881. HRMS (ES): *m/z* 503.1230 $[C_{25}S_2O_4N_2H_{24} + Na]^+$, calcd. 503.1075.

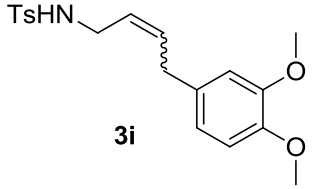
Synthesis of (E/Z)-4-methyl-N-(3-methoxyphenylbut-2-enyl)benezenesulfonamide 3h



3h

Following the general procedure, using 25 mg, 0.112 mmol of 1-tosyl-2-vinyl aziridine and 4-methoxyphenylboronic acid yielded the title compound as a clear oil (15.6 mg, 42%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.75 (d, *J* = 8.5 Hz, 2H, *E* and *Z* isomer), 7.30 (m, 2H, *E* and *Z* isomer), 7.19 (t, *J* = 8 Hz, 1H, *E* and *Z* isomer), 6.75-6.73 (dd, *J* = 8.5, 2.0 Hz, 1H, *E* and *Z* isomer), 6.67 (d, *J* = 7.5 Hz, *E* and *Z* isomer) 6.64 (s, 1H, *E* and *Z* isomer) 5.77-5.72 (m, 1H, *E* and *Z* isomer), 5.50-5.45 (m, 1H, *E* and *Z* isomer), 4.39 (br s, 1H, *E* and *Z* isomer), 3.83 (s, 3H, *Z* isomer), 3.82 (s, 3H, *E* isomer), 3.74 (t, *J* = 7.0 Hz, 1H, *E* isomer), 3.59 (t, *J* = 7.0 Hz, 1H, *Z* isomer), 3.32- 3.29 (m, 2H, *E* and *Z* isomer), 2.46 (s, 3H, *E* and *Z* isomer). ^{13}C NMR (125 MHz, $CDCl_3$) δ 160.2, 160.0, 143.6, 143.4, 141.4, 137.2, 137.1, 130.0, 129.9, 129.8, 129.6, 127.4, 127.3, 126.2, 125,2, 121.1, 120.8, 114.5, 114.2, 111.9, 55.4, 45.2, 40.1, 38.5, 33.4, 21.5. IR ν(cm^{-1}): 3257, 2919, 1600, 1490, 1454, 1325, 1259, 1159, 1094, 1045. HRMS (ES): *m/z* 354.1131 $[C_{18}S_1O_3N_1H_{21} + Na]^+$, calcd. 354.1140.

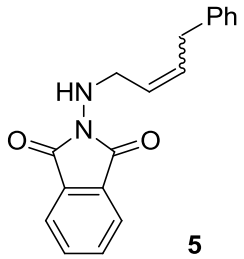
4.12 Synthesis of (E/Z)-4-methyl-N-(3.4-dimethoxyphenylbut-2-enyl)benezenesulfonamide 3i



3i

Following the general procedure, using 25 mg, 0.112 mmol of 1-tosyl-2-vinyl aziridine and 3,4-dimethoxyphenylboronic acid yielded the title compound as a clear oil (10.1 mg, 25%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.78-7.72 (m, 2H, *E* and *Z* isomer), 7.31-7.28 (m, 2H, *E* and *Z* isomer), 6.78-6.77 (m, 1H, *E* and *Z* isomer), 6.64- 6.62 (m, 2H, *E* and *Z* isomer), 5.73-5.68 (m, 1H, *E* and *Z* isomer), 5.45-5.40 (m, 1H, *E* and *Z* isomer), 4.36 (t, *J* = 5.5 Hz, 1H, *E* and *Z* isomer), 3.85 (s, 6H, *E* and *Z* isomer), 3.71 (t, *J* = 6.5 Hz, 1H, *E* isomer), 3.56 (t, *J* = 5.5 Hz, 1H, *Z* isomer), 3.27 (d, *J* = 7.0 Hz, 2H, *E* isomer), 3.23 (d, *J* = 6.5 Hz, *Z* isomer), 2.43 (s, 3H, *E* and *Z* isomer).^{13}C NMR (125 MHz, $CDCl_3$) δ 149.4, 149.3, 147.7, 143.8, 143.7, 137.1, 137.0, 134.0, 133.2, 132.6, 132.2, 130.0, 129.9, 127.4, 127.3, 125.8, 124.9, 120.6, 120.1, 111.9, 111.8, 111.5, 111.4, 56.2, 56.1, 45.5, 40.3, 38.4, 33.0, 21.6. IR ν(cm^{-1}): 3243, 2919, 1616, 1515, 1328, 1139. HRMS (ES): *m/z* 384.1238 $[C_{19}H_{23}N_1S_1O_4 + Na]^+$, calcd. 384.1245.

Synthesis of 2-((4-phenylbut-2-en-1-yl)amino)isoindoline-1,3-dione 5



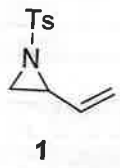
5

$Pd(OAc)_2$ (7.30 mg, 0.0327 mmol, 10 mol%), 1,10-phenanthroline (8.80 mg, 0.0490 mmol, 15 mol%), $AgSbF_6$ (10.9 mg, 0.0327 mmol, 10 mol%) were dissolved in anhydrous dichloroethane (2.8 ml). The resulting mixture was stirred for 30mins at room temperature. 2-(2-vinylaziridin-1-yl) isoindoline-1,3-dione (70 mg, 0.327 mmol, 1.0 eq), phenylboronic acid (59.8 mg, 0.490 mmol, 1.5 eq) and potassium acetate (11.1 mg, 0.117 mmol, 0.5 eq) were subsequently added. The mixture was then stirred at room temperature for 17hrs, and then diluted with ethyl acetate, passed through a short pad of silica gel (1 cm), evaporated and finally purified with silica gel chromatography (30% ethyl acetate/n-pentane) to give the yellow solids (67.2 mg, 70%). ^{1}H NMR (500Mz, $CDCl_3$) δ 7.83-7.81 (m, 2H), 7.73-7.72 (m, 2H), 7.16-7.13 (m, 2H), 7.10-7.08 (m, 1H), 7.05-7.01 (m, 2H), 5.83-5.78 (m, 1H), 5.74-5.68 (m, 1H), 4.68-4.65 (m, 1H), 3.86 (d, *J* = 6.0 Hz, 2H, *cis*), 3.66 (d, *J* = 5.5 Hz, 2H, *trans*), 3.38 (d, *J* = 7.5 Hz, 2H, *cis*), 3.30 (d, *J* = 6.5 Hz, *trans*). ^{13}C NMR (125 MHz, $CDCl_3$) δ 166.7, 166.7, 140.0, 139.7, 135.3, 135.1, 134.2, 133.7, 131.1, 130.2, 128.4, 128.4, 128.2, 128.0, 126.0, 126.0, 124.9, 124.5, 123.6, 123.5, 123.4, 53.2, 47.8, 38.8, 33.4. HRMS (ES): m/z 315.1109 $[C_{18}H_{16}N_2O_2+Na]^+$, calcd. 315.1101. IR ν($cm^{-1}$): 2989, 2362, 1773, 1717, 1560, 1507, 1457, 1376, 1192, 1127, 1084, 883, 712.

jxy140924_2_yjx_465_1_PROTON

exp1 PROTON

SAMPLE		PRESATURATION	
date	Sep 24 2014	satmode	n
solvent	cdcl3	wet	n
file	exp	SPECIAL	
ACQUISITION		temp	not used
sw	5367.0	gain	40
at	3.053	spin	not used
np	32768	hst	0.008
fb	3000	pw90	11.250
bs	16	alfa	10.000
d1	1.000	FLAGS	
nt	16	il	n
ct	16	in	n
TRANSMITTER		dp	y
tn	H1	hs	nn
sfrq	499.907	PROCESSING	
tof	24.9	lb	0.50
tpwr	60	fn	not used
pw	11.250	DISPLAY	
DECOUPLER		sp	-89.4
dn	C13	wp	4639.4
dof	0	rf1	173.9
dm	nnn	rfp	0
decwave	W40_autoxdb	rp	3.0
dpwr	37	lp	-70.6
dmf	32258	PLOT	
		wc	268
		sc	0
		vs	108
		th	150
		ai cdc ph	



1

jxy131121_2_yjx_255_1_PROTON

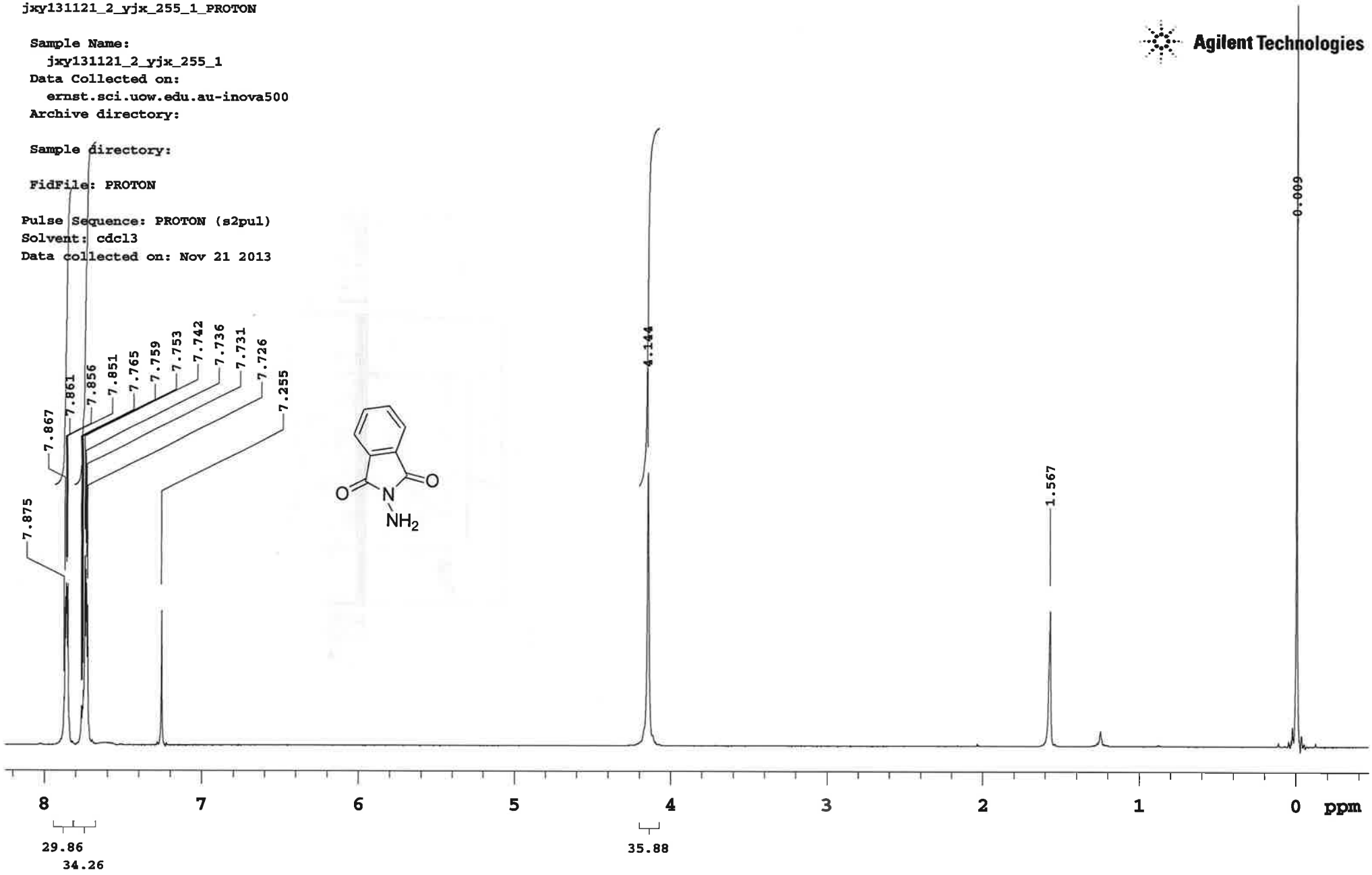
Sample Name:
jxy131121_2_yjx_255_1
Data Collected on:
ernst.sci.uow.edu.au-inova500
Archive directory:

Sample directory:

FidFile: PROTON

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Nov 21 2013

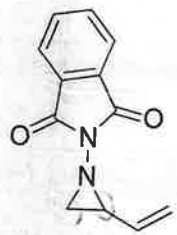
Agilent Technologies



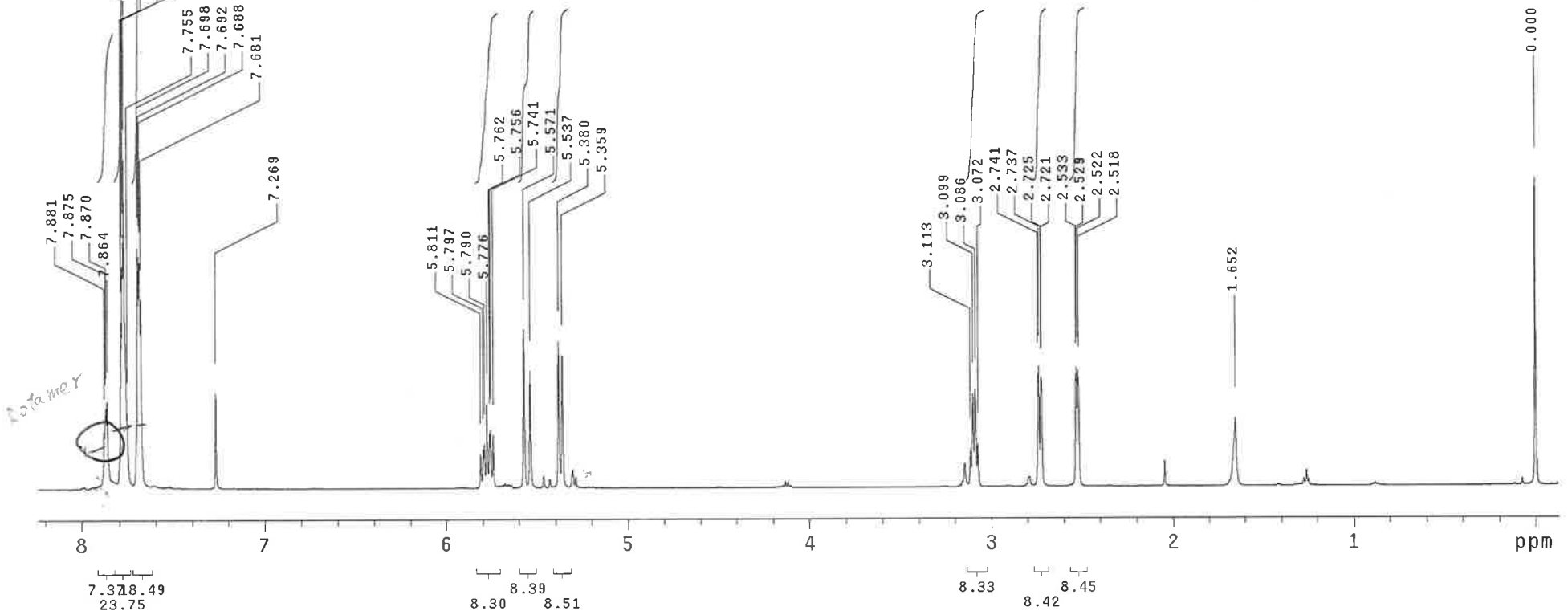
jxy131126_2_yjx_258_2_Proton

exp1 Proton

SAMPLE		SPECIAL	
date	Nov 26 2013	temp	25.0
solvent	cdcl3	gain	not used
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	4370.6	pw90	11.100
at	3.749	alfa	6.600
np	32768	**FLAGS**	
fb	4000	il	n
bs	16	in	n
d1	1.000	dp	y
nt	16	hs	nn
ct	16	**PROCESSING**	
TRANSMITTER		lb	0.50
tn	H1	fn	65536
sfrq	499.743	**DISPLAY**	
tof	-442.5	sp	-63.5
tpwr	62	wp	4180.7
pw	5.550	rfl	122.2
DECOUPLER		rfp	0
dn	C13	rp	-131.1
dof	0	lp	-20.5
dm	nnn	**PLOT**	
dmm	c	wc	250
dpwr	47	sc	0
dmf	22200	vs	712
		th	5
		ai cdc ph	



4



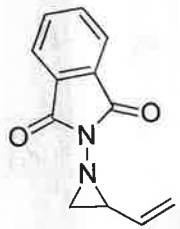
jxy131203_2_yjx_258_1_13c_Carbon

File: Carbon

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: uowvnmrs
VNMRS-500 "pyne06.domain.com"

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 0.537 sec
Width 30487.8 Hz
4456 repetitions
OBSERVE C13, 125.6599960 MHz
DECOUPLE H1, 499.7437041 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 17 min, 58 sec

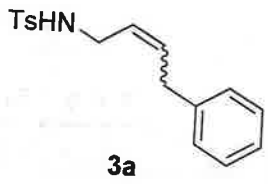


4

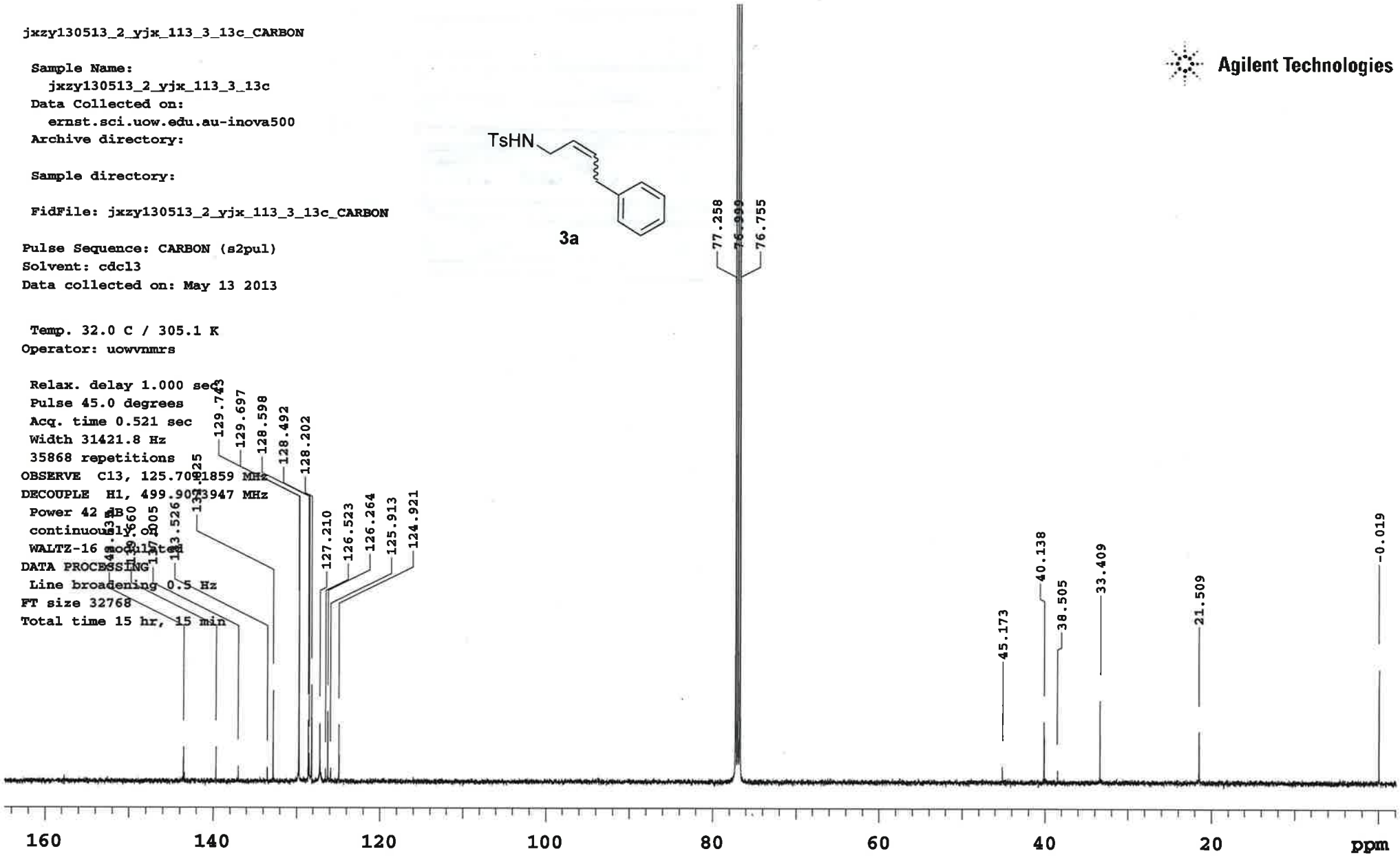
jxy130509_2_yjx_113_3_PROTON

exp4 PROTON

SAMPLE		PRESATURATION	
date	May 9 2013	satmode	n
solvent	cdcl3	wet	n
file	/home/uowvnmr~	SPECIAL	
s/bup_archive/bup_~		temp	25.0
1306xsent/jxy13050~		gain	not used
9_2_yjx_113_3_PROT~		spin	not used
	ON01.fid	hst	0.008
ACQUISITION		pw90	10.100
sw	7998.4	alfa	10.000
at	2.048	FLAGS	
np	32768	il	n
fb	4000	in	n
bs	16	dp	y
d1	1.000	hs	nn
nt	8	PROCESSING	
ct	8	lb	0.50
TRANSMITTER		fn	not used
tn	H1	DISPLAY	
sfrq	499.908	sp	-86.4
tof	499.9	wp	4762.7
tpwr	60	rfl	1018.4
pw	10.100	rfp	0
DECOUPLER		rp	-151.7
dn	C13	lp	-107.8
dof	0	PLOT	
dm	nnn	wc	268
decwave	W40_autoxdb	sc	0
dpwr	37	vs	50
dmf	32258	th	83
		ai cdc ph	



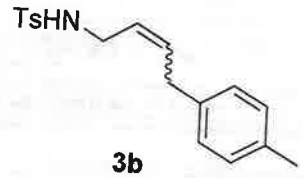
3a


jxzy130513_2_yjx_113_3_13c_CARBON
Sample Name:
jxzy130513_2_yjx_113_3_13c
Data Collected on:
ernst.sci.uow.edu.au-inova500
Archive directory:
Sample directory:
FidFile: jxzy130513_2_yjx_113_3_13c_CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: May 13 2013
Temp. 32.0 C / 305.1 K
Operator: uowvnmrs
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.521 sec
Width 31421.8 Hz
35868 repetitions
OBSERVE C13, 125.7091859 MHz
DECOUPLE H1, 499.9073947 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 32768
Total time 15 hr, 15 min
TsHN
3a
Agilent Technologies
129.743
129.697
128.598
128.492
128.202
127.210
126.523
126.264
125.913
124.921
77.258
76.999
76.755
45.173
40.138
38.505
33.409
21.509
-0.019
160
140
120
100
80
60
40
20
ppm

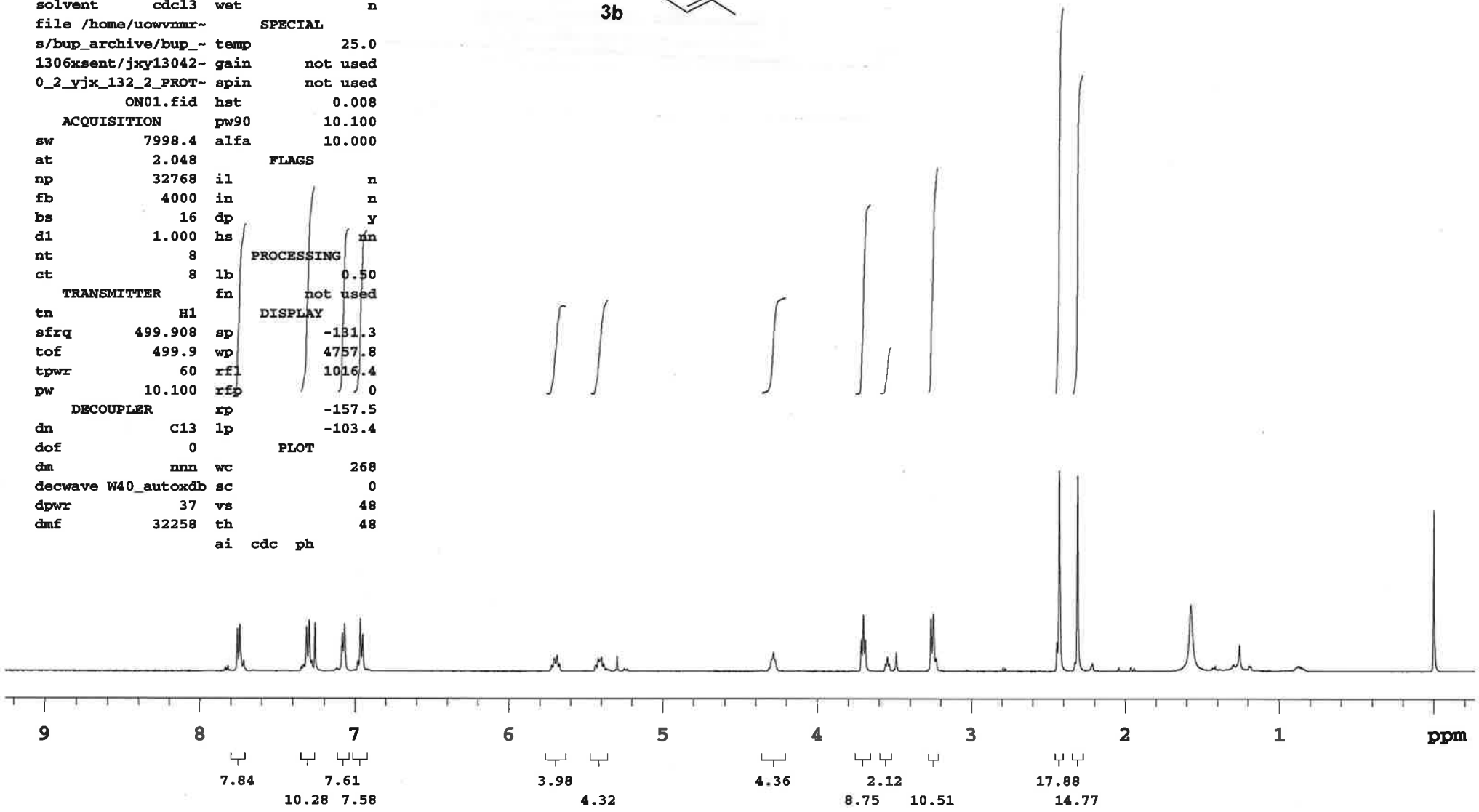
jxy130420_2_yjx_132_2_PROTON

exp6 PROTON

SAMPLE		PRESATURATION	
date	Apr 20 2013	satmode	n
solvent	cdcl3	wet	n
file	/home/uowvnmr~	SPECIAL	
	s/bup_archive/bup_~	temp	25.0
	1306xsent/jxy13042~	gain	not used
	0_2_yjx_132_2_PROT~	spin	not used
	ON01.fid	hst	0.008
ACQUISITION		pw90	10.100
sw	7998.4	alfa	10.000
at	2.048	FLAGS	
np	32768	il	n
fb	4000	in	n
bs	16	dp	y
d1	1.000	hs	nn
nt	8	PROCESSING	
ct	8	lb	0.50
TRANSMITTER		fn	not used
tn	H1	DISPLAY	
sfrq	499.908	sp	-131.3
tof	499.9	wp	4757.8
tpwr	60	rfl	1016.4
pw	10.100	rfp	0
DECOUPLER		rp	-157.5
dn	C13	lp	-103.4
dof	0	PLOT	
dm	nnn	wc	268
decwave	W40_autoxdb	sc	0
dpwr	37	vs	48
dmf	32258	th	48
		ai cdc ph	



3b



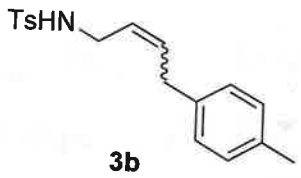
jxy130606_2_yjx_132_3_13c_Apt

File: Apt

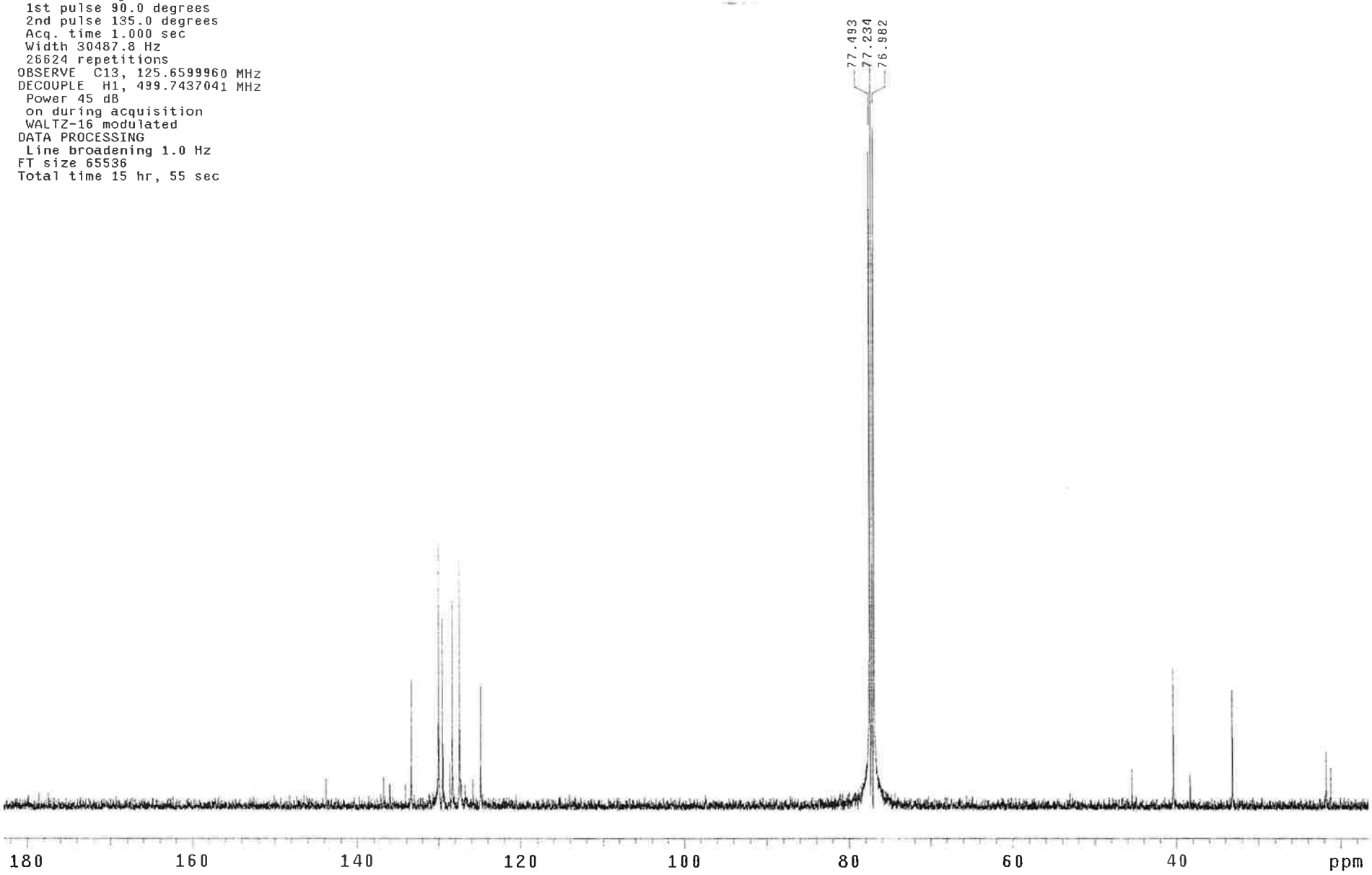
Pulse Sequence: APT

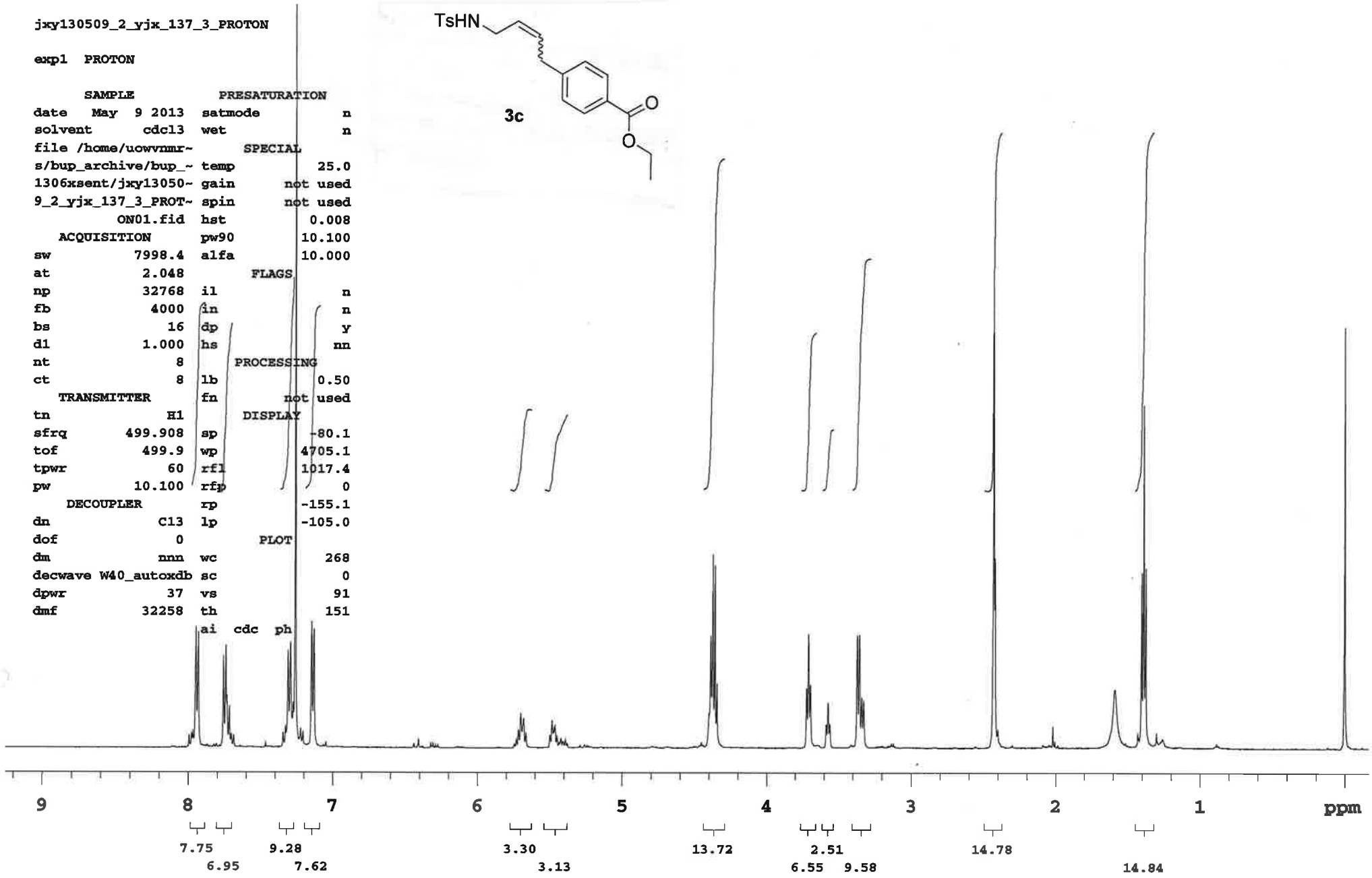
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: uowvnmrs
VNMRS-500 "pyne06.domain.com"

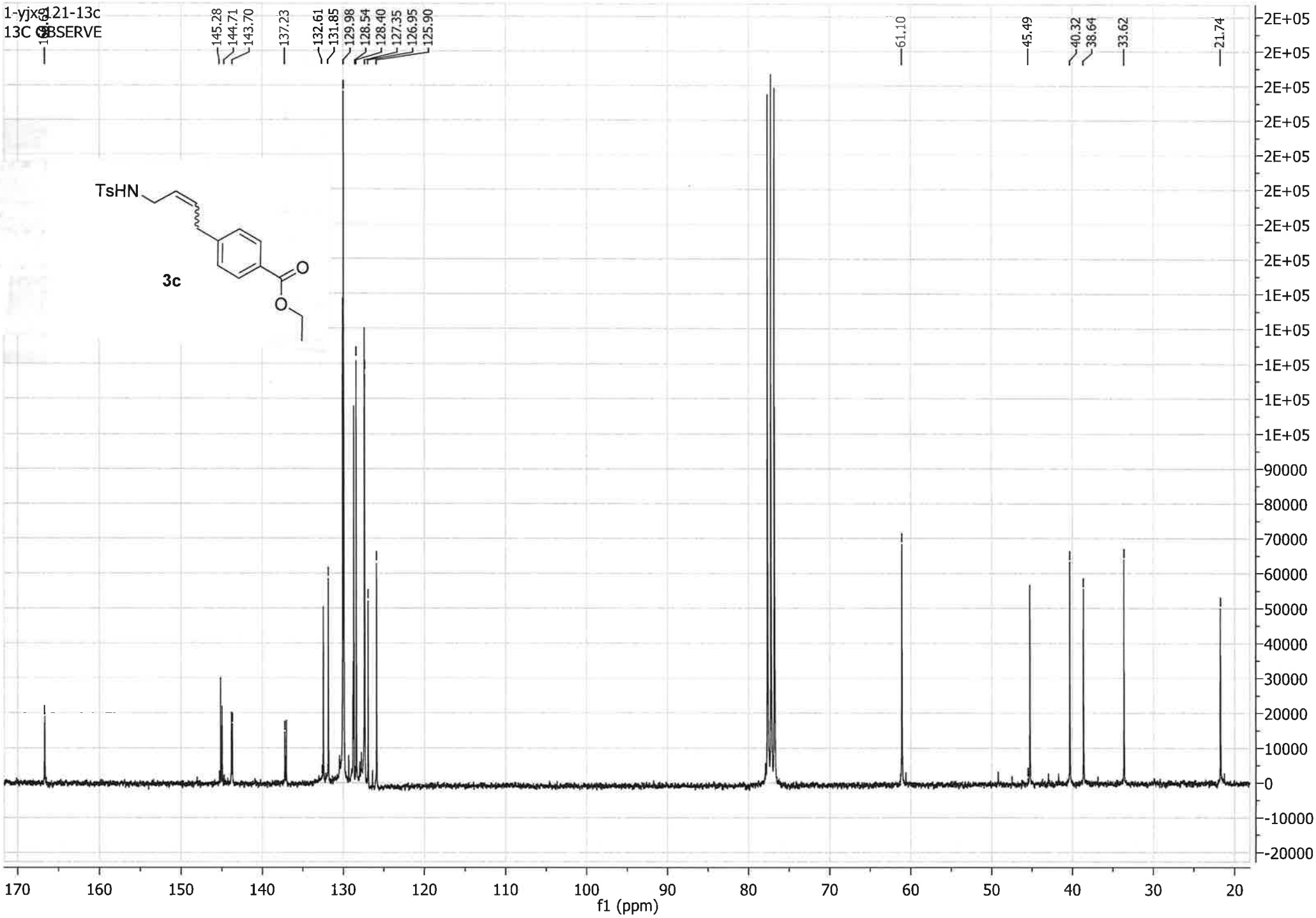
Relax. delay 1.000 sec
1st pulse 90.0 degrees
2nd pulse 135.0 degrees
Acq. time 1.000 sec
Width 30487.8 Hz
26624 repetitions
OBSERVE C13, 125.6599960 MHz
DECOUPLE H1, 499.7437041 MHz
Power 45 dB
on during acquisition
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 15 hr, 55 sec



3b



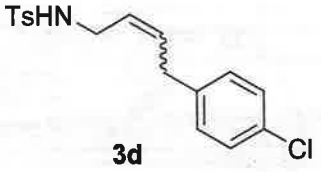

jxy130509_2_yjx_137_3_PROTON
exp1 PROTON
SAMPLE
date May 9 2013
solvent cdcl3
file /home/uowvnmr~
s/bup_archive/bup_~
1306xsent/jxy13050~
9_2_yjx_137_3_PROT~
ON01.fid
ACQUISITION
sw 7998.4
at 2.048
np 32768
fb 4000
bs 16
d1 1.000
nt 8
ct 8
TRANSMITTER
tn H1
sfrq 499.908
tof 499.9
tpwr 60
pw 10.100
DECOUPLER
dn C13
dof 0
dm nnn
decwave W40_autoxdb
dpwr 37
dmf 32258
PRESATURATION
satmode n
wet n
SPECIAL
temp 25.0
gain not used
spin not used
hst 0.008
pw90 10.100
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.50
fn not used
DISPLAY
sp -80.1
wp 4705.1
rfl 1017.4
rfp 0
rp -155.1
lp -105.0
PLOT
wc 268
sc 0
vs 91
th 151
ai cdc ph
TsHN
O
O
3c
9
8
7
6
5
4
3
2
1
ppm
7.75
6.95
9.28
7.62
3.30
3.13
13.72
2.51
6.55
9.58
14.78
14.84


1-yjx-121-13c
13C OBSERVE
145.28
144.71
143.70
137.23
132.61
131.85
129.98
128.54
128.40
127.35
126.95
125.90
61.10
45.49
40.32
38.64
33.62
21.74
TsHN
O
O
3c
f1 (ppm)

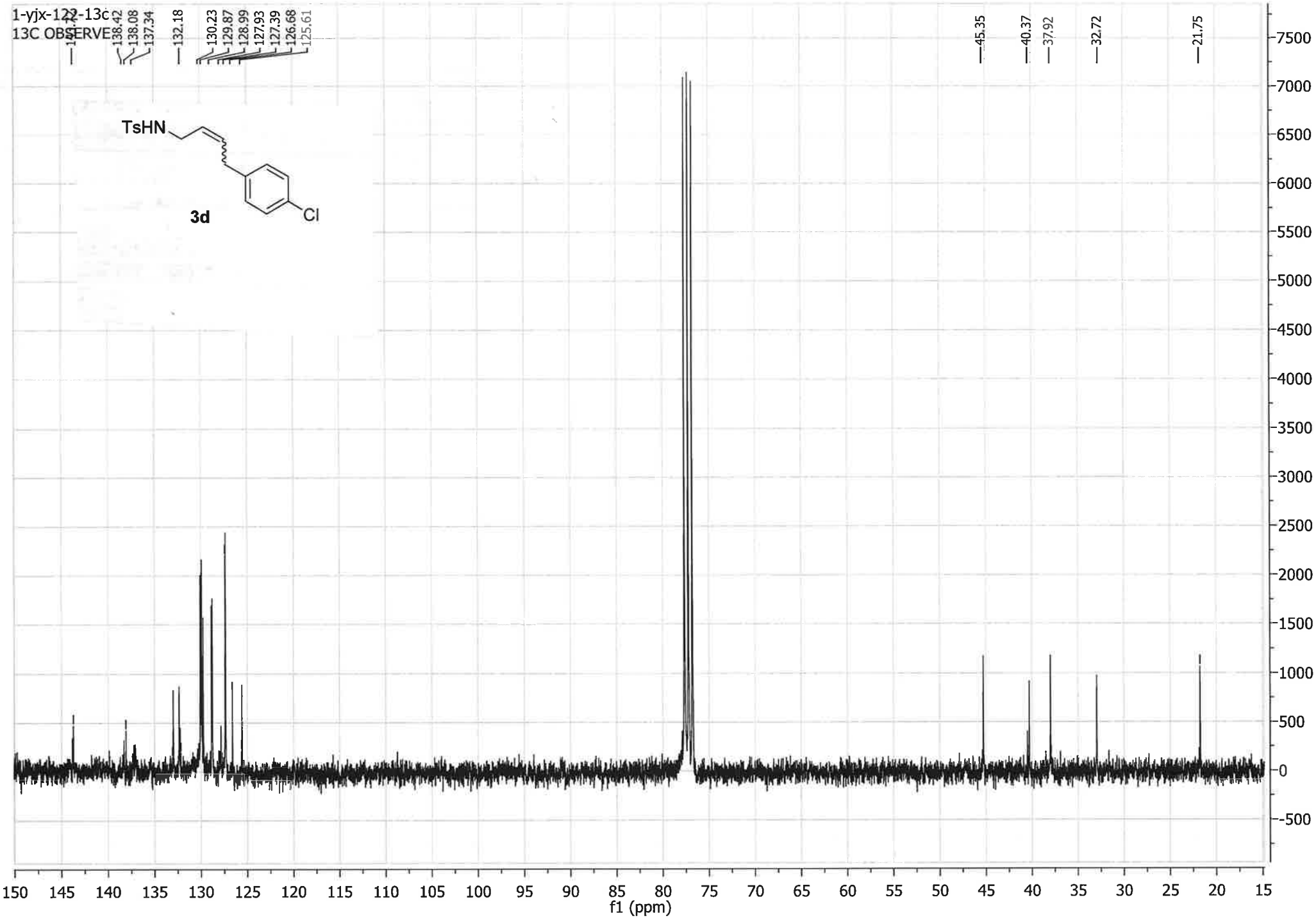
jxy130509_2_yjx_133_3_PROTON

exp4 PROTON

SAMPLE		PRESATURATION	
date	May 9 2013	satmode	n
solvent	cdcl3	wet	n
file	/home/uowvnmr~	SPECIAL	
s/bup_archive/bup_~		temp	25.0
1306xsent/jxy13050~		gain	not used
9_2_yjx_133_3_PROT~		spin	not used
	ON01.fid	hst	0.008
ACQUISITION		pw90	10.100
sw	7998.4	alfa	10.000
at	2.048	FLAGS	
np	32768	il	n
fb	4000	in	n
bs	16	dp	y
d1	1.000	hs	nn
nt	8	PROCESSING	
ct	8	lb	0.50
TRANSMITTER		fn	not used
tn	H1	DISPLAY	
sfrq	499.908	sp	-85.9
tof	499.9	wp	4689.5
tpwr	60	rf1	1017.9
pw	10.100	rfp	0
DECOUPLER		rp	-159.5
dn	C13	lp	-102.1
dof	0	PLOT	
dm	nnn	wc	268
decwave	W40_autoxdb	sc	0
dpwr	37	vs	157
dmf	32258	th	147
		ai cdc ph	



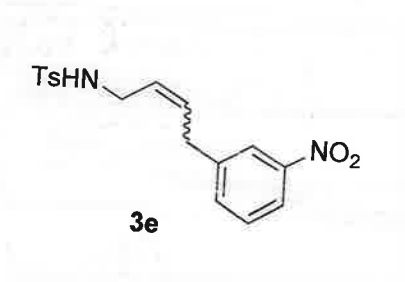
3d


1-yjx-122-13c
13C OBSERVE
143.42
138.42
138.08
137.34
132.18
130.23
129.87
128.99
127.93
127.39
126.68
125.61
45.35
40.37
37.92
32.72
21.75
TsHN
Cl
3d
f1 (ppm)

jxy130509_2_yjx_141_3_PROTON

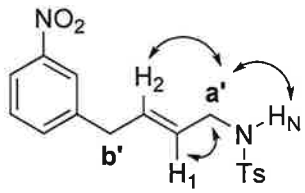
exp1 PROTON

SAMPLE		PRESATURATION	
date	May 9 2013	satmode	n
solvent	cdcl3	wet	n
file	/home/uowvnmr~	SPECIAL	
s/bup_archive/bup_~		temp	25.0
1306xsent/jxy13050~		gain	not used
9_2_yjx_141_3_PROT~		spin	not used
	ON01.fid	hst	0.008
ACQUISITION		pw90	10.100
sw	7998.4	alfa	10.000
at	2.048	FLAGS	
np	32768	il	n
fb	4000	in	n
bs	16	dp	y
d1	1.000	hs	nn
nt	8	PROCESSING	
ct	8	lb	0.50
TRANSMITTER		fn	not used
tn	H1	DISPLAY	
sfrq	499.908	sp	-140.6
tof	499.9	wp	4715.9
tpwr	60	rf1	1014.9
pw	10.100	rfp	0
DECOUPLER		rp	-148.2
dn	C13	lp	-120.7
dof	0	PLOT	
dm	nnn	wc	268
decwave	W40_autoxdb	sc	0
dpwr	37	vs	79
dmf	32258	th	134
		ai cdc ph	

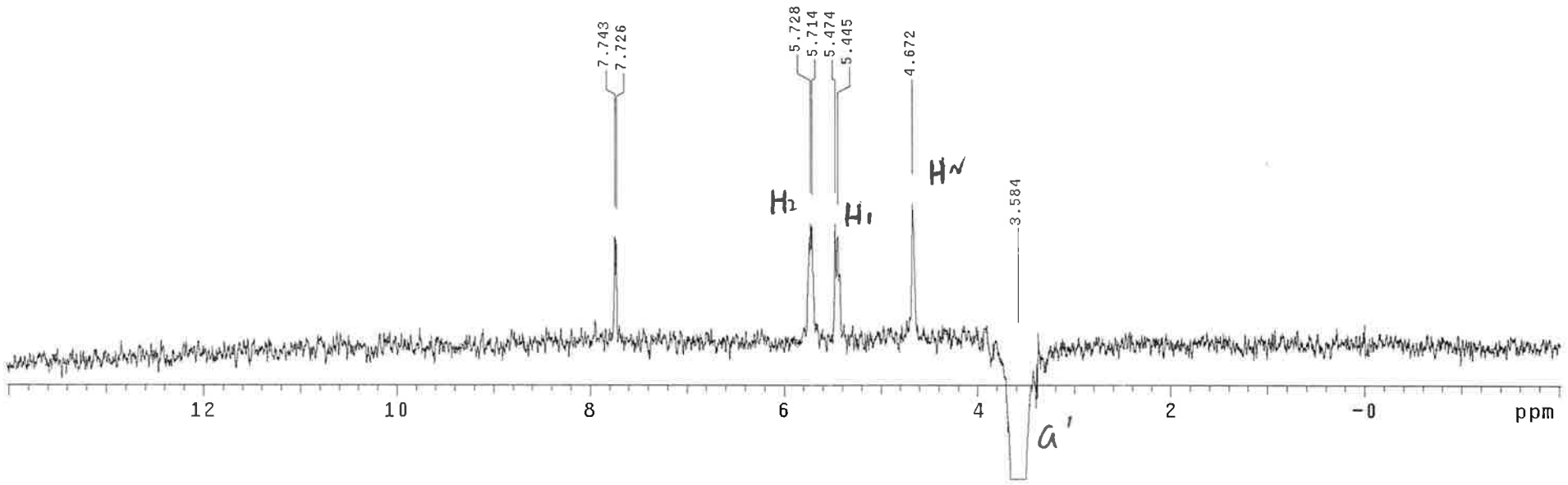


3e

```
jxy130927_2_yjx_141_noe_Noesy1d

 File: Noesy1d

Pulse Sequence: NOESY1D
 Solvent: cdcl3
 Temp. 25.0 C / 298.1 K
Operator: uowvnmrs
VNMRS-500  "pyne06.domain.com"

 Relax. delay 1.000 sec
 Pulse 90.0 degrees
 Mixing 0.500 sec
 Acq. time 1.998 sec
 Width 8012.8 Hz
 64 repetitions
OBSERVE   H1, 499.7412053 MHz
DATA PROCESSING
 Line broadening 3.0 Hz
FT size 32768
Total time 4 min, 4 sec
```



3d: *E*-4-methyl-N-(3-nitrophenylbut-2-enyl)benezenesulfonamide

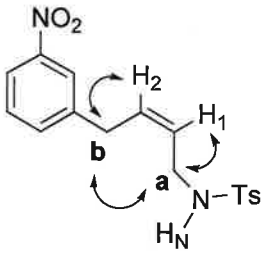


jxy130927_2_yjx_141_noe_NOESY1D_01

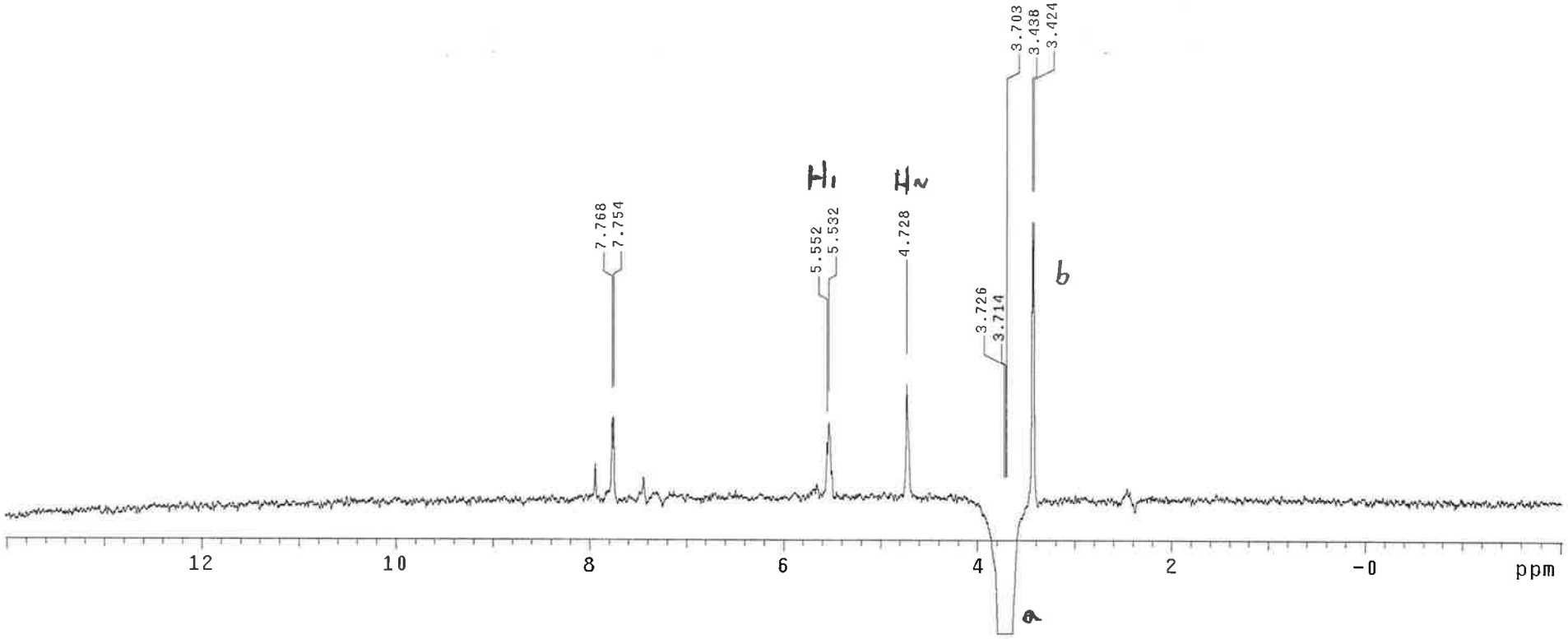
File: Noesy1d

Pulse Sequence: NOESY1D
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: uowvnmrs
VNMRS-500 "pyne06.domain.com"

Relax. delay 1.000 sec
Pulse 90.0 degrees
Mixing 0.500 sec
Acq. time 1.998 sec
Width 8012.8 Hz
64 repetitions
OBSERVE H1, 499.7412053 MHz
DATA PROCESSING
Line broadening 3.0 Hz
FT size 32768
Total time 4 min, 2 sec



3d: *Z*-4-methyl-N-(3-nitrophenylbut-2-enyl)benezenesulfonamide



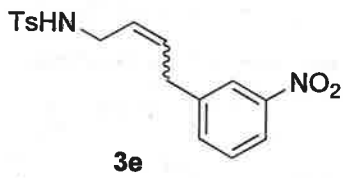
jxy130514_2_yjx_141_3_13c_Apt

File: Apt

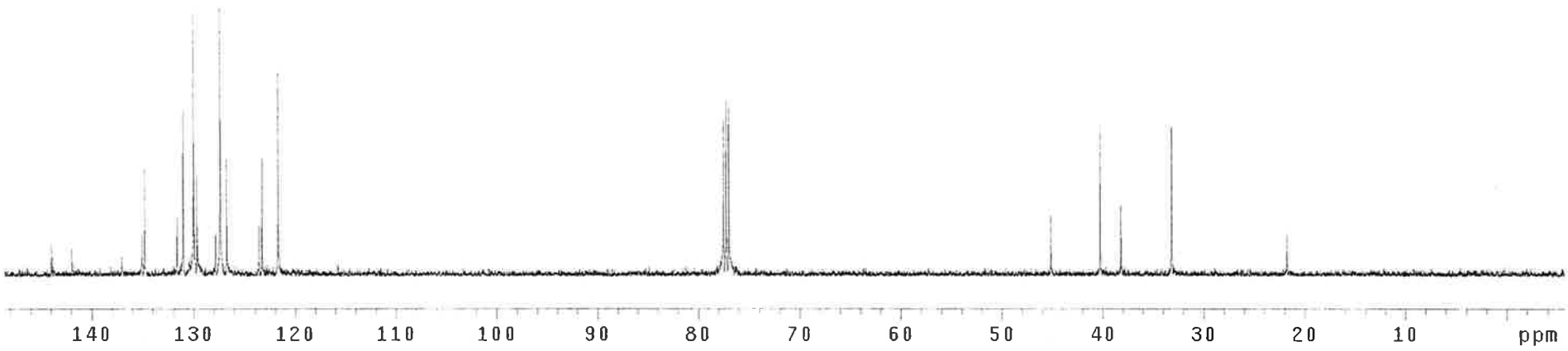
Pulse Sequence: APT

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: uowvnmrs
VNMRS-500 "pyne06.domain.com"

Relax. delay 1.000 sec
1st pulse 90.0 degrees
2nd pulse 135.0 degrees
Acq. time 1.000 sec
Width 30487.8 Hz
1760 repetitions
OBSERVE C13, 125.6599960 MHz
DECOUPLE H1, 499.7437041 MHz
Power 45 dB
on during acquisition
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 59 min, 55 sec



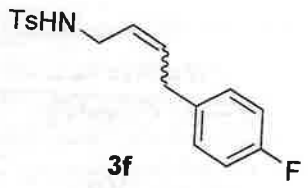
3e



jxy130830_2_yjx_189_3_2ndcolumn_PROTON

exp8 PROTON

SAMPLE		PRESATURATION	
date	Aug 30 2013	satmode	n
solvent	cdcl3	wet	n
file	/home/uowvnmr~	SPECIAL	
s/bup_archive/bup_~		temp	25.0
1309xsent/jxy13083~		gain	not used
0_2_yjx_189_3_2ndc~		spin	not used
olumn_PROTON01.fid		hst	0.008
ACQUISITION		pw90	10.100
sw	7998.4	alfa	10.000
at	2.048	FLAGS	
np	32768	il	n
fb	4000	in	n
bs	16	dp	y
d1	1.000	hs	nn
nt	14	PROCESSING	
ct	14	fn	not used
TRANSMITTER		DISPLAY	
tn	H1	sp	-159.1
sfrq	499.908	wp	4715.9
tof	499.9	rfl	1017.9
tpwr	60	rfp	0
pw	10.100	rp	+150.4
DECOUPLER		lp	-105.8
dn	C13	PLOT	
dof	0	wc	268
dm	nnn	sc	0
decwave	W40_autoxdb	vs	97
dpwr	37	th	149
dmf	32258	ai cdc ph	



3f

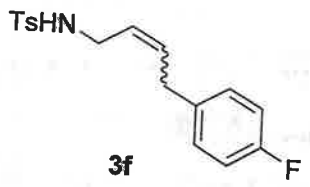
jxy130830_2_yjx_189_3_13c_3_Carbon

File: Carbon

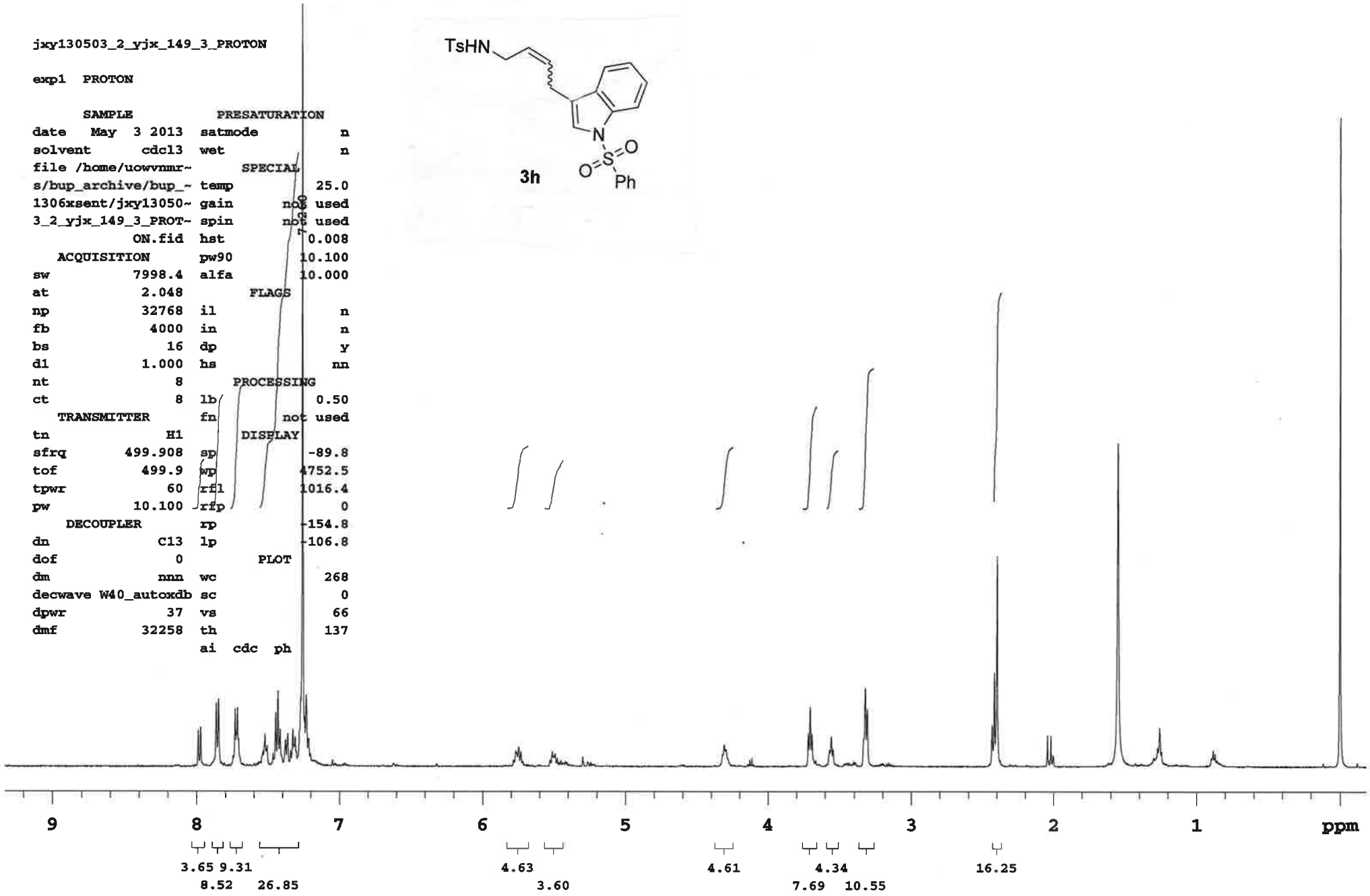
Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: uowvnmrs
VNMRS-500 "pyne06.domain.com"

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 0.537 sec
Width 30487.8 Hz
56756 repetitions
OBSERVE C13, 125.6599960 MHz
DECOUPLE H1, 499.7437041 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 16 hr, 29 min, 56 sec



3f


jxy130503_2_yjx_149_3_PROTON
exp1 PROTON
SAMPLE
date May 3 2013
solvent cdcl3
file /home/uowvnmr~
s/bup_archive/bup_~
1306xsent/jxy13050~
3_2_yjx_149_3_PROT~
ON.fid
ACQUISITION
sw 7998.4
at 2.048
np 32768
fb 4000
bs 16
d1 1.000
nt 8
ct 8
TRANSMITTER
tn H1
sfrq 499.908
tof 499.9
tpwr 60
pw 10.100
DECOUPLER
dn C13
dof 0
dm nnn
decwave W40_autoxdb
dpwr 37
dmf 32258
PRESATURATION
satmode n
wet n
SPECIAL
temp 25.0
gain not used
spin not used
hst 0.008
pw90 10.100
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.50
fn not used
DISPLAY
sp -89.8
wp 4752.5
rfl 1016.4
rfp 0
rp -154.8
lp -106.8
PLOT
wc 268
sc 0
vs 66
th 137
ai cdc ph
7.2260
TsHN
N
O=S=O
Ph
3h
9
8
7
6
5
4
3
2
1
ppm
3.65 9.31
8.52 26.85
4.63
3.60
4.61
4.34
7.69 10.55
16.25

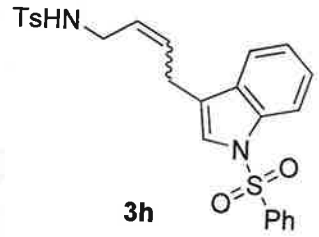
jxy130915_2_yjx_202_2_13c_Carbon

File: Carbon

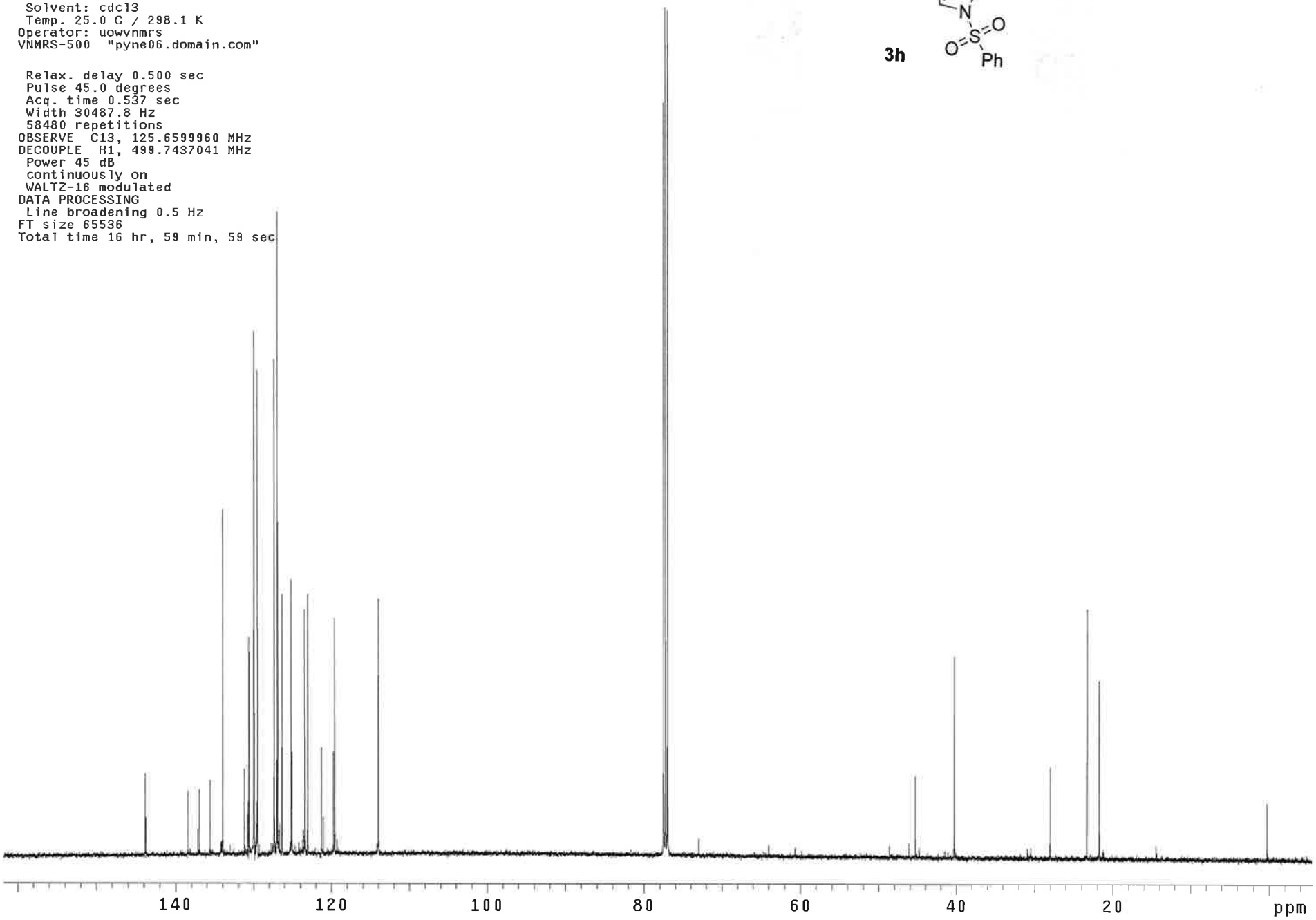
Pulse Sequence: s2pul

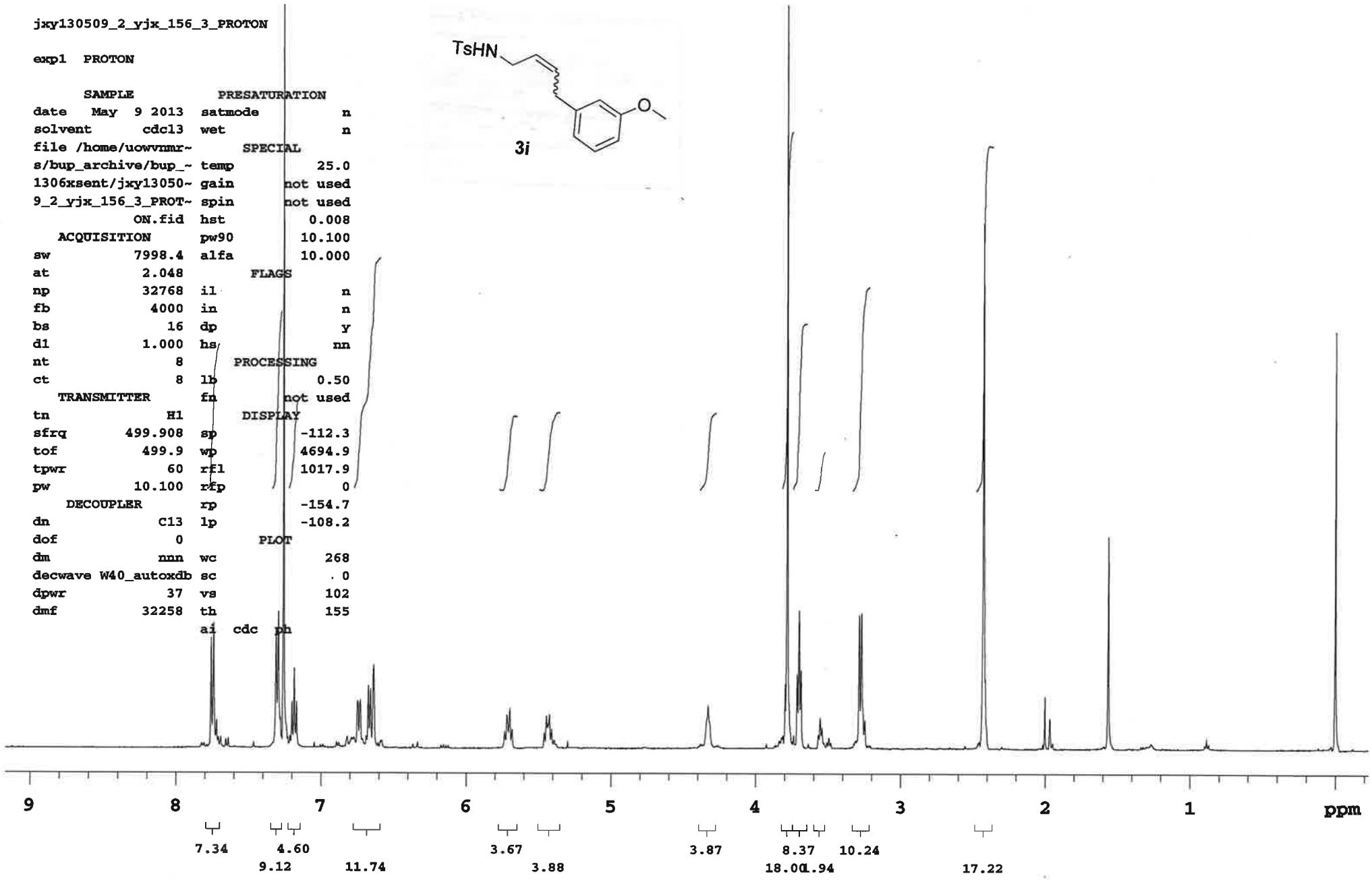
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: uowvnmrs
VNMRS-500 "pyne06.domain.com"

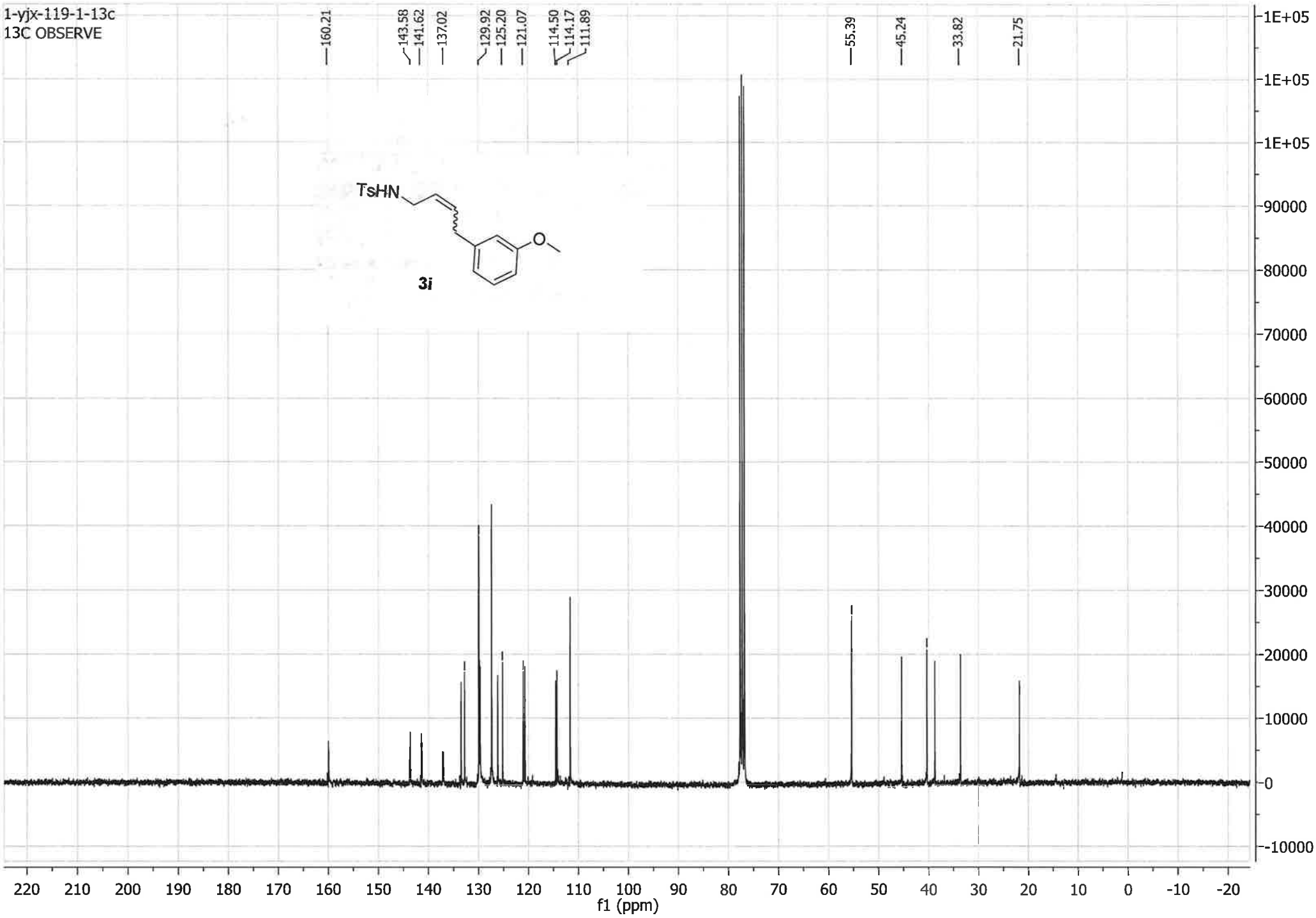
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 0.537 sec
Width 30487.8 Hz
58480 repetitions
OBSERVE C13, 125.6599960 MHz
DECOUPLE H1, 499.7437041 MHz
Power 45 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 16 hr, 59 min, 59 sec



3h



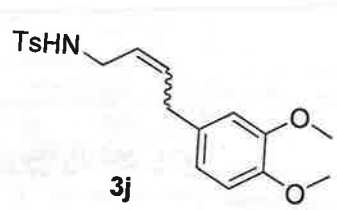

jxy130509_2_yjx_156_3_PROTON
exp1 PROTON
SAMPLE
date May 9 2013
solvent cdcl3
file /home/uowvnmr~
s/bup_archive/bup_~
1306xsent/jxy13050~
9_2_yjx_156_3_PROT~
ON.fid
ACQUISITION
sw 7998.4
at 2.048
np 32768
fb 4000
bs 16
d1 1.000
nt 8
ct 8
TRANSMITTER
tn H1
sfrq 499.908
tof 499.9
tpwr 60
pw 10.100
DECOUPLER
dn C13
dof 0
dm nnn
decwave W40_autoxdb
dpwr 37
dmf 32258
PRESATURATION
satmode n
wet n
SPECIAL
temp 25.0
gain not used
spin not used
hst 0.008
pw90 10.100
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.50
fn not used
DISPLAY
sp -112.3
wp 4694.9
rfl 1017.9
rfp 0
rp -154.7
lp -108.2
PLOT
wc 268
sc 0
vs 102
th 155
ai cdc ph
TsHN
O
3i
9
8
7
6
5
4
3
2
1
ppm
7.34
4.60
9.12
11.74
3.67
3.88
3.87
8.37
18.00
1.94
10.24
17.22


1-yjx-119-1-13c
13C OBSERVE
160.21
143.58
141.62
137.02
129.92
125.20
121.07
114.50
114.17
111.89
55.39
45.24
33.82
21.75
TsHN
O
3i
1E+05
1E+05
1E+05
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

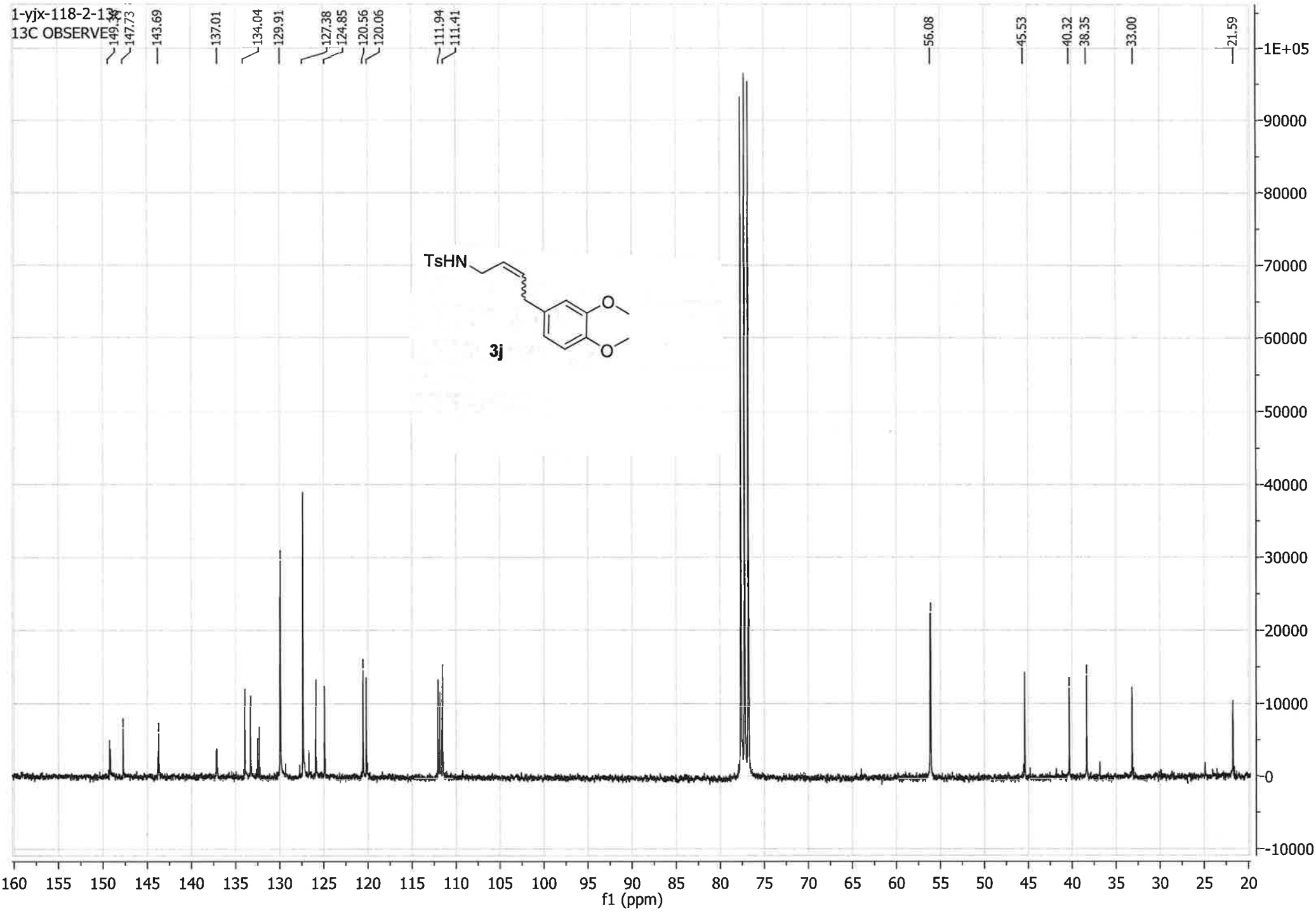
jxy130503_2_yjx_151_1_PROTON

exp1 PROTON

SAMPLE		PRESATURATION	
date	May 3 2013	satmode	n
solvent	cdcl3	wet	n
file	/home/uowvnmr~	SPECIAL	
s/bup_archive/bup_~		temp	25.0
1306xsent/jxy13050~		gain	not used
3_2_yjx_151_1_PROT~		spin	not used
	ON.fid	hst	0.008
ACQUISITION		pw90	10.100
sw	7998.4	alfa	10.000
at	2.048	FLAGS	
np	32768	il	n
fb	4000	in	n
bs	16	dp	y
d1	1.000	hs	nn
nt	8	PROCESSING	
ct	8	lb	0.50
TRANSMITTER		fn	not used
tn	H1	DISPLAY	
sfrq	499.908	sp	-84.0
tof	499.9	wp	4658.2
tpwr	60	rf1	1015.9
pw	10.100	rfp	0
DECOUPLER		rp	-153.0
dn	C13	lp	-115.1
dof	0	PLOT	
dm	nnn	wc	268
decwave	W40_autoxdb	sc	0
dpwr	37	vs	60
dmf	32258	th	150
		ai cdc ph	



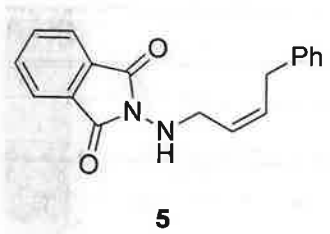
3j


1-yjx-118-2-13
13C OBSERVE
149.38
147.73
143.69
137.01
134.04
129.91
127.38
124.85
120.56
120.06
111.94
111.41
56.08
45.53
40.32
38.35
33.00
21.59
TsHN
O
O
3j
1E+05
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20
f1 (ppm)

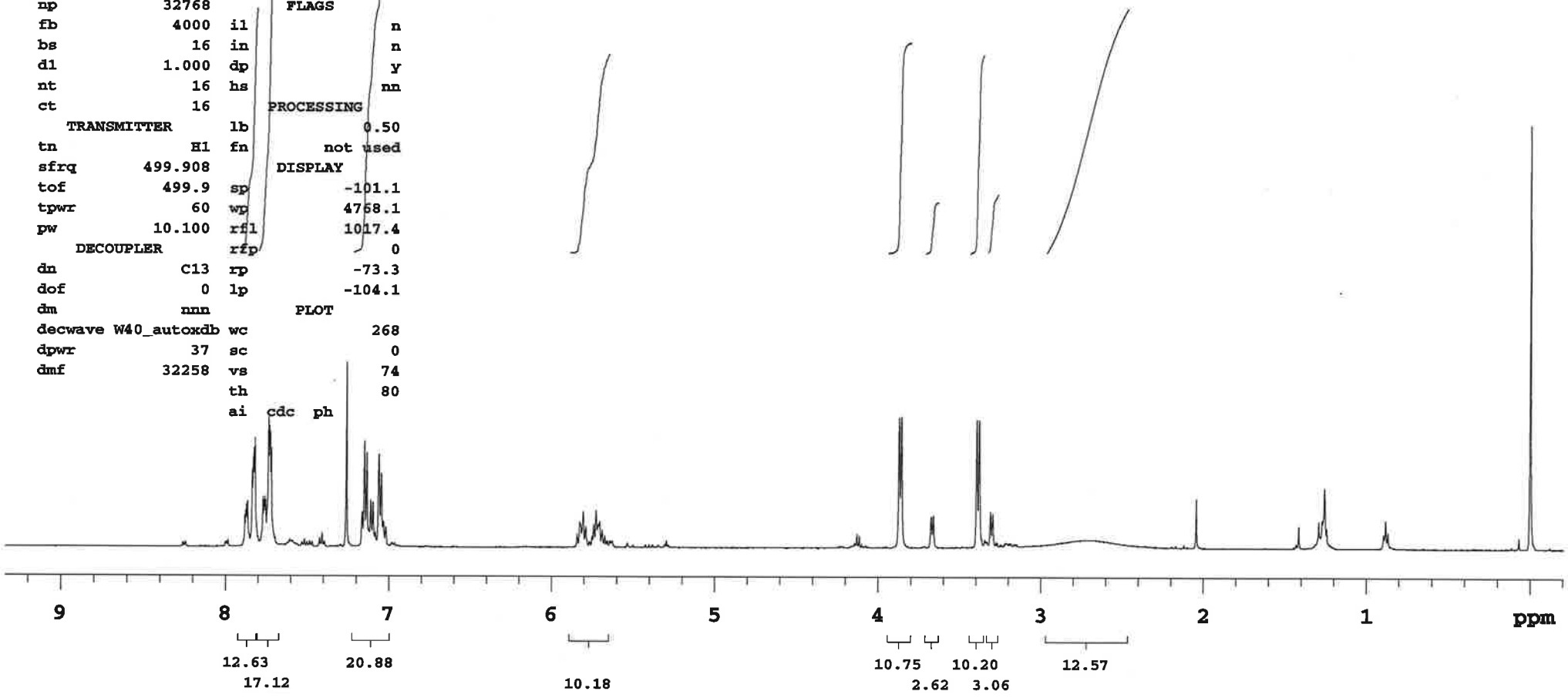
jxy140710_2_yjx_403_cp_PROTON

exp1 PROTON

SAMPLE		PRESATURATION	
date	Jul 10 2014	satmode	n
solvent	cdcl3	wet	n
file	/home/uowvnmr~	SPECIAL	
s/jxy140710_2_yjx_~		temp	25.0
403_cp_PROTON02.fi~		gain	not used
	d	spin	not used
ACQUISITION		hst	0.008
sw	7998.4	pw90	10.100
at	2.048	alfa	10.000
np	32768	FLAGS	
fb	4000	il	n
bs	16	in	n
d1	1.000	dp	y
nt	16	hs	nn
ct	16	PROCESSING	
TRANSMITTER		lb	0.50
tn	H1	fn	not used
sfrq	499.908	DISPLAY	
tof	499.9	sp	-101.1
tpwr	60	wp	4768.1
pw	10.100	rfl	1017.4
DECOUPLER		rfp	0
dn	C13	rp	-73.3
dof	0	lp	-104.1
dm	nnn	PLOT	
decwave	W40_autoxdb	wc	268
dpwr	37	sc	0
dmf	32258	vs	74
		th	80
		ai cdc ph	



5



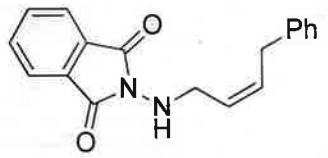
jxy131129_2_yjx_260_3_13c_CARBON

Sample Name:
jxy131129_2_yjx_260_3_13c
Data Collected on:
ernst.sci.uow.edu.au-inova500
Archive directory:

Sample directory:

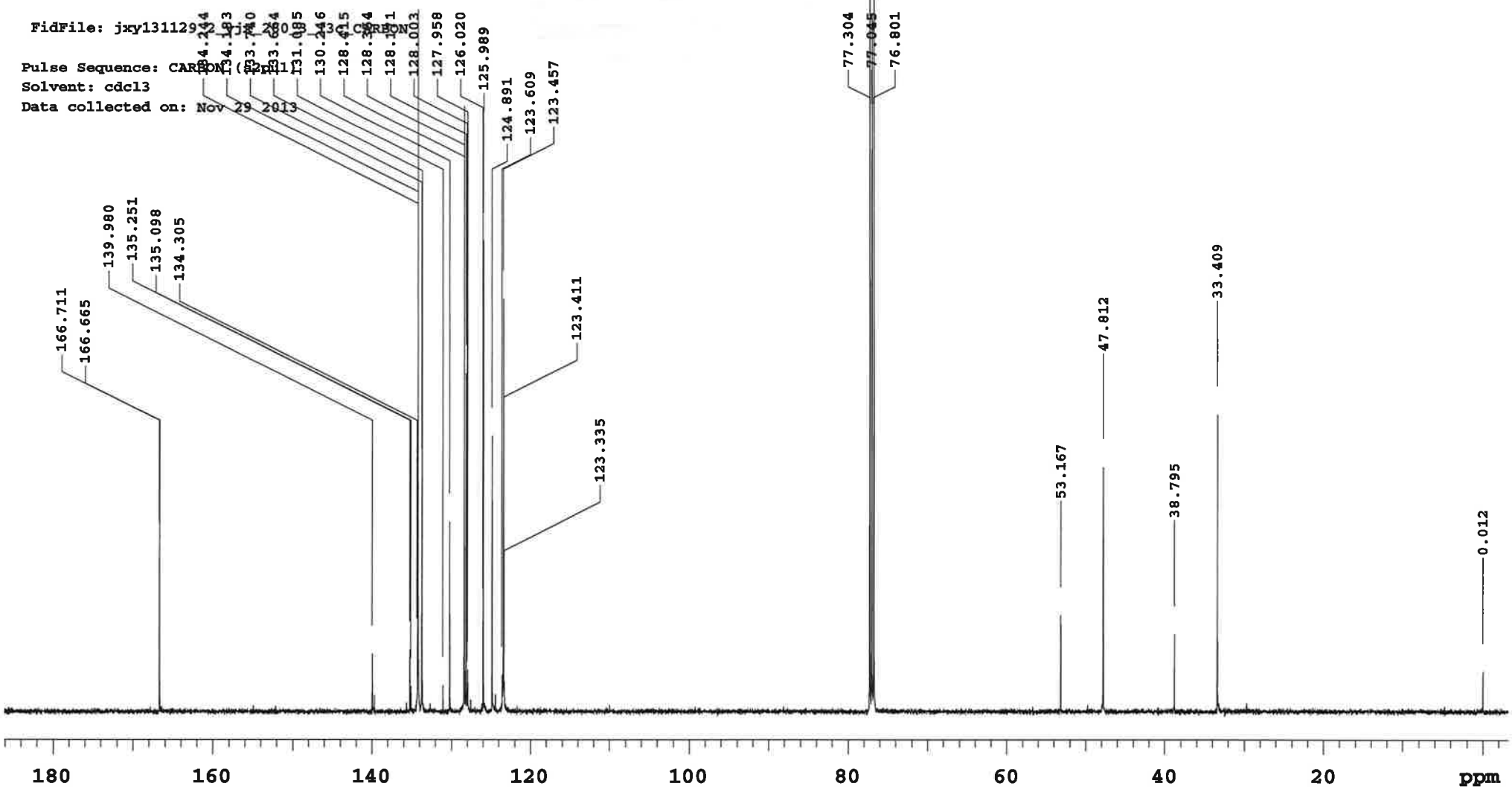
FidFile: jxy131129_2_yjx_260_3_13c_CARBON

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Nov 29 2013



5

Agilent Technologies



ac140818_5m2_NOESY1D

Sample Name:
ac140818_5m2
Data Collected on:
ernst.sci.uow.edu.au-inova500
Archive directory:

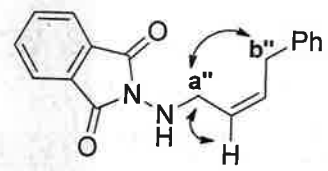
Sample directory:

FidFile: NOESY1D

Pulse Sequence: NOESY1D
Solvent: cdcl3
Data collected on: Aug 18 2014

Temp. 25.0 C / 298.1 K
Operator: uowvnmrs

Relax. delay 1.000 sec
Pulse 90.0 degrees
Acq. time 2.048 sec
Width 7998.4 Hz
64 repetitions
OBSERVE H1, 499.9048952 MHz
DATA PROCESSING
FT size 32768
Total time 4 min 8 sec



5

Agilent Technologies