Supporting Information For

Expedient Synthesis of Norbenzomorphan Library via Multicomponent Assembly Process Coupled with Ring-Closing Reactions

*James J. Sahn and Stephen F. Martin**

Department of Chemistry and Biochemistry, The Texas Institute for Drug and Diagnostic Development, The University of Texas at Austin, Austin, Texas 78712, USA.

sfmartin@mail.utexas.edu

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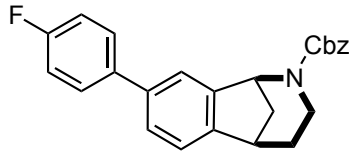
General. Tetrahydrofuran was dried by filtration through two columns of activated, neutral alumina according to the procedure described by Grubbs.[i] Acetonitrile (MeCN) was dried by filtration through two columns of activated molecular sieves, and toluene was dried by filtration through one column of activated, neutral alumina followed by one column of Q5 reactant. These solvents were determined to have less than 50 ppm H_2O by Karl Fischer coulometric moisture analysis. Methylene chloride (CH_2Cl_2), triethylamine (Et_3N) and diisopropylethylamine (*i*-Pr_2NEt) were distilled from calcium hydride immediately prior to use. Where required, solvents were degassed by sparging with argon prior to use. All reagents were reagent grade and used without purification unless otherwise noted, and air or moisture sensitive reagents were weighed in a glove box. All reactions involving air or moisture sensitive reagents or intermediates were performed under an inert atmosphere of nitrogen or argon in glassware that was flame or oven dried. Reaction temperatures refer to the temperature of the cooling/heating bath. Volatile solvents were removed under reduced pressure using a Büchi rotary evaporator at 25–30 °C (bath temperature). Thin layer chromatography was run on pre-coated plates of silica gel with a 0.25 mm thickness containing 60F-254 indicator (EMD Millipore). Chromatography was performed using forced flow (flash chromatography) and the indicated solvent system on 230-400 mesh silica gel (Silicycle flash F60) according to the method of Still,[ii] unless otherwise noted. Radial Preparative Liquid Chromatography (radial plc) was performed on a Chromatotron® using glass plates coated with Merck, TLC grade 7749 silica gel with gypsum binder and fluorescent indicator.

Infrared (IR) spectra were obtained either neat on sodium chloride or as solutions in the solvent indicated and reported as wavenumbers (cm^{-1}). Proton nuclear magnetic resonance (^1H NMR) and carbon nuclear magnetic resonance (^{13}C NMR) spectra were obtained at the indicated field as solutions in $CDCl_3$ unless otherwise indicated. Chemical shifts are referenced to the deuterated solvent (*e.g.,* for $CDCl_3$, δ = 7.26 ppm and 77.0 ppm for ^1H and ^{13}C NMR, respectively) and are reported in parts per million (ppm, δ) relative to tetramethylsilane (TMS, δ = 0.00 ppm). Coupling constants (*J*) are reported in Hz and the splitting abbreviations used are: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; comp, overlapping multiplets of magnetically nonequivalent protons; br, broad; app, apparent. Purity was determined using an LCMS system comprised of an Agilent 1200 Series HPLC and an Agilent 6130 single quadrupole mass spectrometer. Samples were injected onto a Phenomenex Gemini C18 column (5 micron, 2.1 x 50 mm) and eluted at 0.7 ml/min using a gradient of 10-90% acetonitrile, 0.1% formic acid (11 minute linear ramp). Positive mode electrospray ionization was used to verify the identity of the major component, and the purity was assessed via peak integration (AUC) of the UV chromatogram recorded at 214 nm.

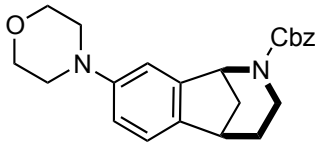
Representative procedure for Suzuki cross-coupling reactions with aryl chlorides 18{*1-3*}.



20{*2,10*}

Benzyl 8-(4-fluorophenyl)-4,5-dihydro-1*H*-1,5-methanobenzo[*c*]azepine-2(3*H*)-carboxylate (20{*2,10*}**).** A solution of carbamate **18**{*2*} (92 mg, 0.28 mmol), *p*-fluorophenylboronic acid (**19**{*10*}) (79 mg, 0.56 mmol), Cs_2CO_3 (183 mg, 0.56 mmol), palladium(bis)(*t*-butyl)$_3$ phosphine (7.2 mg, 0.014 mmol) in degassed 1,4-dioxane (0.85 mL) was stirred for 21 h at 100 °C. The reaction was cooled to room temperature and poured into water (2 mL). The mixture was extracted with CH_2Cl_2 (3 x 15 mL), and the combined organic layers were dried ($MgSO_4$) and concentrated under reduced pressure to provide the crude product, which was purified via radial plc (SiO_2) eluting with hexanes/EtOAc (100:0; 95:5; 90:10) to give 101 mg (94%) of **20**{*2,10*} as a colorless oil: ^{1}H NMR (400 MHz) δ 7.54-7.29 (comp, 10 H), 7.12 (t, *J* = 8.8 Hz, 2 H), 5.64-5.56 and 5.50-5.45 (rotomers, m, 1 H), 5.29-5.10 (m, 2 H), 3.95-3.79 (m, 1 H), 3.33 (d, *J* = 2.0 Hz, 1 H), 2.59-2.45 (m, 1 H), 2.27 (br s, 1 H), 2.03 (br s, 1 H), 1.93 (d, *J* = 11.2, 1 H), 1.71-1.58 (m, 1 H); ^{13}C NMR (75 MHz) (rotomers) δ 162.7 (d, $J_{C\text{-}F}$ = 245 Hz), 155.3, 145.7, 142.2, 139.9, 137.5, 137.1, 129.0, 128.8, 128.7, 128.2, 127.5, 123.3, 122.8, 115.9 (d, $J_{C\text{-}F}$ = 21.3 Hz), 67.3, 57.9, 57.6, 44.0, 39.7, 38.9, 30.5; IR (thin film, neat) 2951, 1695, 1421, 1234, 1101 cm^{-1}; mass spectrum (ESI) *m/z* 388.1709 [$C_{25}H_{23}FNO_2$ (M+1) requires 388.1713]; LCMS purity 99%.

Representative procedure for Buchwald-Hartwig reactions with aryl chlorides 18{*1-3*}.

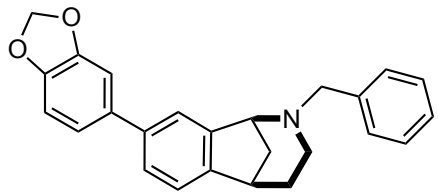


20{*2,2*}

Benzyl 8-morpholino-4,5-dihydro-1*H*-1,5-methanobenzo[*c*]azepine-2(3*H*)-carboxylate (20{*2,2*}**).** A solution of carbamate **18**{*2*} (130 mg, 0.396 mmol), NaO-*t*-Bu (53 mg, 0.55 mmol) and morpholine (45 mg, 0.515 mmol) in degassed toluene (0.75 mL) was stirred for 5 min. A freshly

prepared toluene solution of $Pd(OAc)_2$ and di-*tert*-butylphosphine biphenyl (JohnPhos®) (1:1, 0.1 mL, 0.08 M), that had been stirred for 20 min, was added to the reaction mixture via syringe. After heating at 100 °C for 4.75 h, the reaction was cooled to room temperature, poured into water (3 mL) and extracted with CH_2Cl_2 (3 x 15 mL). The combined organic extracts were dried (K_2CO_3), filtered and concentrated under reduced pressure to provide the crude product, which was purified via radial plc (SiO_2), eluting with hexanes/EtOAc (100:0; 90:10; 80:20) to give 146 mg (97%) of **20**{*2*,*2*} as a pale yellow oil: ^{1}H NMR (400 MHz) δ 7.43-7.27 (comp, 5 H), 7.13 (d, *J* = 8.2 Hz, 1 H), 6.96 and 6.78 (rotomers, s, 1 H), 6.80 (dd, *J* = 8.2, 2.4 Hz, 1 H), 5.47 and 5.35 (rotomers, br s, 1 H), 5.27-5.07 (m, 2 H), 3.85 (t, *J* = 4.8 Hz, 4 H), 3.84-3.65 (m, 1 H), 3.21 (br s, 1 H), 3.11 (app br d, *J* = 19.2 Hz, 4 H), 2.53-2.38 (m, 1 H), 2.30-2.12 (m, 1 H), 2.05-1.90 (m, 1 H), 1.85 (d, *J* = 10.4 Hz, 1 H), 1.64-1.50 (m, 1 H); ^{13}C NMR (75 MHz) (rotomers) δ 155.3, 155.1, 151.4, 142.6, 142.3, 138.1, 137.4, 137.2, 128.7, 128.2, 128.1, 123.4, 116.1, 115.8, 112.2, 112.0, 67.2, 67.1, 58.3, 58.0, 53.8, 50.2, 44.0, 39.2, 38.9, 30.7; IR (thin film, neat) 2958, 2851, 1695, 1615, 1495, 1421, 1234, 1121 cm^{-1}; mass spectrum (ESI) *m/z* 379.2016 [$C_{23}H_{27}N_2O_3$ (M+1) requires 379.2022]; LCMS purity 100%.

Representative procedure for TMSI promoted benzylation of carbamates 18 and 20.

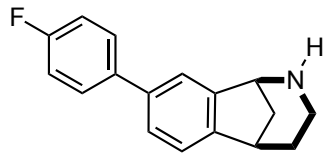


21{*2*,*11*}

8-(Benzo[*d*][1,3]dioxol-5-yl)-2-benzyl-2,3,4,5-tetrahydro-1*H*-1,5-methanobenzo[*c*]azepine (21{*2*,*11*}). *Reaction carried out in the dark.* A solution of carbamate **20**{*2*,*11*} (99 mg, 0.24 mmol) and TMSI (95 mg, 0.48 mmol) in CH_2Cl_2 (4.0 mL) was stirred for 3 h at 0 °C. MeOH (3 mL) and a saturated aqueous $NaHCO_3$ solution (3 mL) were added, and the mixture was stirred for 10 min. The MeOH was removed under reduced pressure, and the aqueous mixture was extracted with CH_2Cl_2 (3 x 10 mL). The combined organic extracts were dried (K_2CO_3), filtered and concentrated under reduced pressure, and the residue was purified via radial plc (SiO_2), eluting with hexanes/EtOAc (100:0; 95:5; 90:10) to give 70 mg (79 %) of benzylamine **21**{*2*,*11*} as a white solid: mp 142-143 °C; ^{1}H NMR (300 MHz) δ 7.46-7.27 (comp, 8 H), 7.14 (comp, 2 H), 6.95 (d, *J* = 7.5 Hz, 1 H), 6.05 (s, 2 H), 3.97 (d, *J* =

4.8 Hz, 1 H), 3.53 (d, *J* = 13.3 Hz, 1 H), 3.35 (d, *J* = 13.3 Hz, 1 H), 3.24-3.16 (m, 1 H), 2.63 (dd, *J* = 10.8, 5.7 Hz, 1 H), 2.30-2.20 (m, 1 H), 2.12-1.97 (comp, 2 H), 1.62-1.53 (comp, 2 H); ^{13}C NMR (75 MHz) δ 148.4, 147.1, 145.8, 140.0, 139.4, 136.4, 129.4, 128.6, 127.1, 126.6, 122.9, 120.9, 108.9, 108.1, 101.4, 63.0, 60.6, 47.3, 45.0, 39.9, 30.6; IR (thin film, neat) 3031, 2935, 2811, 1506, 1465, 1224, 1046 cm^{-1}; mass spectrum (ESI) *m/z* 370.1804 [$C_{25}H_{24}NO_2$ (M+1) requires 370.1802]; LCMS purity 100%.

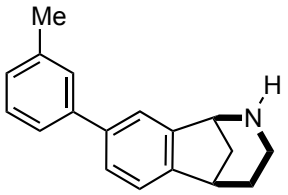
Representative procedure for Cbz deprotection of C-aryl norbenzomorphans[iii] **20**{*2,6*} – **20**{*3,11*}.



23{*2,10*}

8-(4-Fluorophenyl)-2,3,4,5-tetrahydro-1*H*-1,5-methanobenzo[*c*]azepine (23{*2,10*}**).** *Reaction carried out in the dark.* A solution of carbamate **20**{*2,10*} (244 mg, 0.63 mmol) and TMSI (250 mg, 1.25 mmol) in CH_2Cl_2 (7 mL) was stirred for 3 h at 0 °C. Methanolic HCl (2.5 mL, 1.8 M) was added and after stirring for 5 min, the reaction was concentrated under reduced pressure. Diethyl ether (5 mL) was added and the solution was stirred for 5 min. The solids were allowed to settle and the supernatant was removed via syringe. Aqueous NaOH (5 mL, 2.7 M) was added to the remaining solid and the mixture was stirred for 5 min. After the addition of CH_2Cl_2 (20 mL) and 5 more min of stirring, the layers were separated, and the aqueous layer was extracted with CH_2Cl_2 (3 x 10 mL). The combined organic layers were dried (K_2CO_3), filtered, and concentrated under reduced pressure and the residue was purified via radial plc (SiO_2), eluting with hexanes/EtOAc/Et_3N/MeOH (100:0:0:0; 51:49:2:0; 0:98:2:0; 0:88:2:10) to give 141 mg (87%) of **23**{*2,10*} as a light brown semi-solid: ^{1}H NMR (400 MHz) δ 7.55-7.52 (comp, 2 H), 7.42-7.39 (comp, 2 H), 7.24 (s, 1 H), 7.10 (t, *J* = 9.2 Hz, 2 H), 4.27 (d, *J* = 4.0 Hz, 1 H), 3.21 (s, 1 H), 2.73 (dd, *J* = 12.4, 6.0 Hz, 1 H), 2.35 (td, *J* = 12.4, 4.8 Hz, 1 H), 2.23-2.20 (m, 1 H), 2.11 (br s, 1 H), 2.08-1.92 (comp, 2 H), 1.56 (d, *J* = 11.2 Hz, 1 H); ^{13}C NMR (75 MHz) δ 164.2, 160.9, 145.7, 143.9, 139.6, 137.9, 128.9, 126.7, 122.9, 121.9, 115.8 (d, $J_{C\text{-}F}$ = 21.3 Hz), 59.1, 45.7, 40.2, 39.4, 31.5; IR (thin film, neat) 2941, 2852, 1526, 1224 cm^{-1}; mass spectrum (ESI) *m/z* 254.1344 [$C_{17}H_{17}FN$ (M+1) requires 254.1355]; LCMS purity 98%.

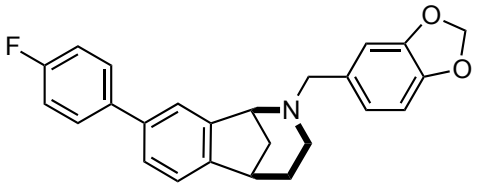
Representative procedure for hydrogenolysis of benzyl carbamates 20{*2,7-9*}**.**



23{*2,9*}

8-(*m*-Tolyl)-2,3,4,5-tetrahydro-1*H*-1,5-methanobenzo[*c*]azepine (23{*2,9*}**).** A solution of **20**{*2,9*} (224 mg, 0.58 mmol) in EtOH (15 mL) and 10% Pd/C (93 mg) was stirred under an atmosphere of H_2 for 1 h at room temperature followed by concentration under reduced pressure. The residue was filtered through a plug of Celite® with multiple portions of EtOAc and the filtrate was concentrated under reduced pressure to provide 112 mg (77%) of **23**{*2,9*} as a yellow oil that was of sufficient purity for use in subsequent reactions: ^{1}H NMR (400 MHz) δ 7.48-7.45 (comp, 2 H), 7.42-7.38 (comp, 2 H), 7.32 (t, *J* = 7.4 Hz, 1 H), 7.25 (d, *J* = 7.4 Hz, 1 H), 7.15 (br d, *J* = 7.4 Hz, 1 H), 4.27 (d, *J* = 4.0 Hz, 1 H), 3.25-3.20 (m, 1 H), 2.73 (dd, *J* = 12.2, 5.8 Hz, 1 H), 2.42 (s, 3 H), 2.37 (td, *J* = 12.2, 4.6 Hz, 1 H), 2.25-2.19 (m, 1 H), 2.03-1.96 (m, 1 H), 1.95 (d, *J* = 10.4 Hz, 1 H), 1.78 (br s, 1 H), 1.60-1.55 (m, 1 H); ^{13}C NMR (125 MHz) δ 145.3, 143.4, 141.5, 140.5, 138.2, 128.6, 127.9, 127.7, 126.7, 124.2, 122.5, 121.8, 58.9, 45.3, 40.0, 39.2, 31.2, 21.5; IR (thin film, neat) 3052, 2942, 2853, 1273 cm^{-1}; mass spectrum (ESI) *m/z* 250.1588 [$C_{18}H_{20}N$ (M+1) requires 250.1596]; LCMS purity 97%.

Representative procedure for reductive amination of norbenzomorphans 22 and 23.

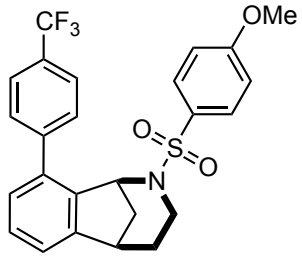


26{*2,10,3*}

2-(Benzo[*d*][1,3]dioxol-5-ylmethyl)-8-(4-fluorophenyl)-2,3,4,5-tetrahydro-1*H*-1,5-methanobenzo[*c*]azepine (26{*2,10,3*}**).** A solution of amine **23**{*2,10*} (25 mg, 0.098 mmol), $Na(OAc)_3BH$ (33 mg, 0.16 mmol), piperonal (**24**{*3*}) (30 mg, 0.20 mmol) and acetic acid (10 μL) in

1,2-dichloroethane (2 mL) was stirred for 24 h at room temperature. The reaction was quenched with an aqueous, saturated $NaHCO_3$ solution (2 mL) and the layers were separated. The aqueous layer was extracted with CH_2Cl_2 (3 x 3 mL), and the combined organic layers were dried (K_2CO_3), filtered and concentrated under reduced pressure. The crude product was purified via radial plc (SiO_2) eluting with hexanes/EtOAc (100:0; 90:10; 80:20) to give 27 mg (71%) of **26**{*2*,*10*,*3*} as a white solid: mp 106-108 °C; ^{1}H NMR (300 MHz) δ 7.63-7.58 (m, 2 H), 7.45 (d, *J* = 6.3 Hz, 1 H), 7.35 (s, 1 H), 7.31-7.29 (m, 1 H), 7.21-7.15 (m, 2 H), 6.99 (s, 1 H), 6.83-6.77 (m, 2 H), 5.97 (s, 2 H), 3.97 (d, *J* = 4.5 Hz, 1 H), 3.43 (d, *J* = 13.3 Hz, 1 H), 3.23 (d, *J* = 13.3, 1 H), 3.21-3.19 (m, 1 H), 2.62-2.57 (m, 1 H), 2.30-2.20 (m, 1 H), 2.11-2.05 (m, 1 H), 1.99 (d, *J* = 10.5 Hz, 1 H), 1.59-1.51 (m, 2 H); ^{13}C NMR (75 MHz) δ 162.6 (d, $J_{C\text{-}F}$ = 244 Hz), 147.9, 146.7, 146.1, 140.2, 138.7, 138.1, 133.3, 129.0, 128.9, 126.7, 123.0, 122.2, 115.8 (d, $J_{C\text{-}F}$ = 21.3 Hz), 109.7, 108.1, 101.1, 62.9, 60.3, 47.1, 44.9, 39.9, 30.5; IR (thin film, neat) 2942, 1492, 1238, 1039 cm^{-1}; mass spectrum (ESI) *m/z* 388.1711 [$C_{25}H_{23}FNO_2$ (M+1) requires 388.1707]; LCMS purity 100%.

Representative procedure for *N*-sulfonylation of norbenzomorphans 22 and 23.

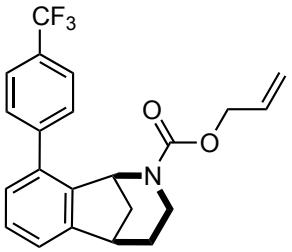


30{*3*,*7*,*30*}

2-((4-Methoxyphenyl)sulfonyl)-9-(4-(trifluoromethyl)phenyl)-2,3,4,5-tetrahydro-1*H*-1,5-methanobenzo[*c*]azepine (30{*3*,*7*,*30*}**).** *p*-Methoxybenzenesulfonyl chloride (**24**{*30*} (28 mg, 0.14 mmol) was added to a stirred solution of amine **23**{*3*,*7*} (21 mg, 0.07 mmol) and Et_3N (28 mg, 0.28 mmol) in CH_2Cl_2 (1 mL) at room temperature. The solution was stirred for 12 h, and the mixture was concentrated under reduced pressure and purified via radial plc (SiO_2), eluting with hexanes/EtOAc (100:0; 95:5; 90:10) to provide 28 mg (85%) of sulfonamide **30**{*3*,*7*,*30*} as a colorless oil: NMR (400 MHz) δ 7.71-7.64 (comp, 6 H), 7.38 (t, *J* = 7.6 Hz, 1 H), 7.30-7.24 (comp, 3 H), 6.92 (d, *J* = 8.8 Hz, 2 H), 5.27 (d, *J* = 4.0 Hz, 1 H), 3.88 (s, 3 H), 3.74 (dd, *J* = 14.4, 6.0 Hz, 1 H), 3.28-3.23 (m, 1 H), 2.84-2.75 (m, 1 H), 1.97-1.89 (m, 1 H), 1.88-1.82 (m, 1 H), 1.70 (d, *J* = 11.2 Hz, 1 H), 1.56-1.52 (m, 1 H);

^{13}C NMR (125 MHz) δ 162.7, 147.9, 143.3, 137.9, 137.2, 133.0, 129.6, 129.4, 129.1, 128.0, 125.5, 125.4, 125.3, 122.5, 114.2, 58.4, 55.6, 41.8, 40.5, 40.0, 29.9; IR (thin film, neat) 2949, 2262, 1733, 1602, 1499, 1327, 1259 cm^{-1}; mass spectrum (ESI) *m/z* 474.1342 [$C_{25}H_{23}NO_3F_3S$ (M+1) requires 474.1351]; LCMS purity 100%.

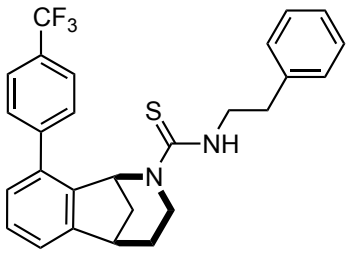
Representative procedure for acylation of norbenzomorphans 22 and 23.



32{*3*,*7*,*39*}

Allyl 9-(4-(trifluoromethyl)phenyl)-4,5-dihydro-1*H*-1,5-methanobenzo[*c*]azepine-2(3*H*)-carboxylate (32{*3*,*7*,*39*}**).** Allyl chloroformate (**24**{*39*}) (29 mg, 0.24 mmol) was added to a solution of amine **23**{*3*,*7*} (37 mg, 0.12 mmol) and Et_3N (37 mg, 0.37 mmol) in CH_2Cl_2 (0.75 mL) at room temperature, and the solution was stirred for 15 h. The mixture was concentrated under reduced pressure, and the residue was purified via radial plc (SiO_2) eluting with hexanes/EtOAc (100:0; 95:5) to give 34 mg (73%) of **32**{*3*,*7*,*39*} as a white solid: mp 76-77 °C; ^{1}H NMR (400 MHz) δ 7.64 (d, *J* = 8.2 Hz, 2 H), 7.46 (d, *J* = 8.2 Hz, 2 H), 7.38 (t, *J* = 7.6 Hz, 1 H), 7.31-7.24 (comp, 2 H), 6.01-5.92 and 5.83-5.73 (rotomers, m, 1 H), 5.42-5.25 (m, 1 H), 5.24-5.14 (m, 2 H), 4.70-4.18 (m, 2 H), 4.05-3.98 (m, 1 H), 3.38 (app br s, 1 H), 2.72 and 2.57 (rotomers, td, *J* = 13, 4.8 Hz, 1 H), 2.19-2.16 (m, 1 H), 2.10-1.99 (m, 1 H), 1.92-1.86 (m, 1 H), 1.68-1.63 (m, 1 H); ^{13}C NMR (75 MHz) δ (rotomers) 154.3, 154.1, 148.1, 147.8, 144.1, 143.7, 139.0, 138.8, 137.2, 133.5, 133.1, 129.8, 129.3, 127.9, 126.3, 125.5, 125.4, 122.7, 122.6, 117.9, 117.3, 66.3, 66.1, 56.7, 44.6, 44.3, 40.6, 39.0, 38.7, 31.1, 30.8; IR (thin film, neat) 3052, 2942, 2880, 1705, 1417, 1334, 1128 cm^{-1}; mass spectrum (ESI) *m/z* 388.1521 [$C_{22}H_{21}NO_2F_3$(M+1) requires 388.1524]; LCMS purity 100%.

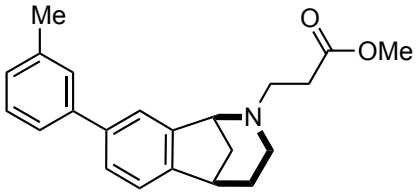
Representative procedure for thiourea synthesis from 22 and 23.



34{*3*,*7*,*35*}

***N*-Phenethyl-9-(4-(trifluoromethyl)phenyl)-4,5-dihydro-1*H*-1,5-methanobenzo[*c*]azepine-2(3*H*)-carbothioamide (34**{*3*,*7*,*35*}**).** Phenethylisothiocyanate **24**{*35*} (33 mg, 0.20 mmol) was added to a solution of amine **23**{*3*,*7*} (46 mg, 0.1 mmol) and Et_3N (10 mg, 0.1 mmol) in CH_2Cl_2 (0.75 mL) at room temperature and was stirred for 24 h. The reaction was concentrated, and the residue was purified via radial plc (SiO_2), eluting with hexanes/EtOAc (100:0; 90:10; 80:20) to give 41 mg (89%) of **34**{*3*,*7*,*35*} as a white solid: mp (DCM/CH_3CN 3:1) 165-166 °C; ^{1}H NMR (400 MHz) δ 7.65 (d, J = 8.0 Hz, 2 H), 7.41-7.38 (m, 3 H), 7.24 (comp, 4 H), 7.18 (comp, 3 H), 5.24 (t, J = 4.8 Hz, 1 H), 4.03-3.95 (m, 1 H), 3.81-3.75 (m, 1 H), 3.43-3.40 (m, 1 H), 2.98-2.91 (m, 1 H), 2.88-2.74 (comp, 3 H), 2.17-2.14 (m, 1 H), 2.11-2.03 (m, 1 H), 1.89 (d, J = 10.8 Hz, 1 H), 1.67 (d, J = 12.8 Hz, 1 H); ^{13}C (125 MHz) δ (rotomers) ^{13}C (125 MHz) δ 180.5, 147.8, 143.5, 138.8, 138.2, 137.0, 129.6 (q, J_{C-F} = 32.0 Hz), 129.5, 129.0, 128.7, 128.6, 127.7, 126.6, 125.6 (q, J_{C-F} = 3.75 Hz), 124.2 (q, J_{C-F} = 270 Hz), 122.4, 59.5, 46.8, 43.4, 40.8, 35.3, 30.9; IR (thin film, neat) 3397, 2973, 2925, 2863, 1542, 1399, 1330, 1269, 1180, 1139 cm^{-1}; mass spectrum (ESI) *m/z* 467.1764 [$C_{27}H_{26}N_2F_3S$ (M+1) requires 467.1691]; LCMS purity 100%.

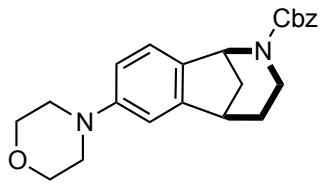
Representative procedure for conjugate additions of norbenzomorphans 22 and 23.



35{*2*,*9*,*40*}

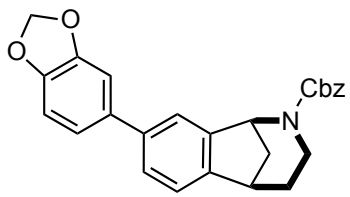
Methyl 3-(8-(*m*-tolyl)-4,5-dihydro-1*H*-1,5-methanobenzo[*c*]azepin-2(3*H*)-yl)propanoate (35{*2*,*9*,*40*}**).** Methyl acrylate **24**{*40*} (25 mg, 0.28 mmol) was added to a solution of amine **23**{*2*,*9*} (36 mg, 0.14 mmol) and Et_3N (14 mg, 0.14 mmol) in CH_2Cl_2 (3 mL). After stirring for 19 h at room temperature, the reaction was concentrated under reduced pressure, and the crude product purified via

radial plc (SiO_2), eluting with hexanes/EtOAc (100:0; 95:0; 90:10) to give 39 mg (83%) of **35**{*2*,*9*,*40*} as a white solid: mp 65-66°C; ^{1}H NMR (400 MHz) δ 7.46 (dd, *J* = 7.6, 2.4 Hz, 1 H), 7.42-7.38 (comp, 3 H), 7.33 (t, *J* = 7.8 Hz, 1 H), 7.26 (d, *J* = 7.6 Hz, 1 H), 7.18-7.14 (m, 1 H), 3.98 (d, *J* = 4.8 Hz, 1 H), 3.69 (s, 3 H), 3.18-3.14 (m, 1 H), 2.82-2.75 (m, 1 H), 2.64 (dd, *J* = 11.2, 5.2 Hz, 1 H), 2.58-2.53 (m, 2 H), 2.43 (s, 3 H), 2.45-2.38 (m, 1 H), 2.28-2.22 (m, 1 H), 2.00 (td, *J* = 15.2, 2.4 Hz, 1 H), 1.94 (d, *J* = 10.8 Hz, 1 H), 1.59-1.51 (m, 1 H), 1.50-1.44 (m, 1 H); ^{13}C NMR (75 MHz) δ 173.3, 145.6, 141.8, 139.9, 139.7, 138.5, 128.9, 128.3, 128.0, 126.9, 124.6, 123.1, 122.9, 64.2, 51.9, 51.7, 46.9, 44.9, 39.8, 33.3, 30.4, 21.8; IR (thin film, neat) 2941, 1732, 1478, 1272, 1203; mass spectrum (ESI) *m/z* 336.1963 [$C_{22}H_{26}NO_2$ (M+1) requires 336.1964]; LCMS purity 98%.



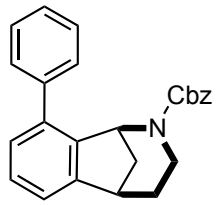
20{*1*,*2*}

Benzyl 7-morpholino-4,5-dihydro-1*H*-1,5-methanobenzo[*c*]azepine-2(3*H*)-carboxylate (20{*1*,*2*}**).** 88%, colorless oil. ^{1}H NMR (400 MHz) δ 7.42-7.28 (comp, 5 H), 7.23 and 7.12 (rotomers, d, *J* = 7.4 Hz, 1 H), 6.83 (d, *J* = 2.4 Hz, 1 H), 6.78-6.71 (m, 1 H), 5.47-5.44 and 5.36-5.34 (rotomers, m, 1 H), 5.21-5.10 (comp, 2 H), 3.86 (t, *J* = 4.9 Hz, 4 H), 3.86-3.75 (m, 1 H), 3.24-3.20 (m, 1 H), 3.17 and 3.16 (rotomers, t, *J* = 4.9 Hz, 4 H), 2.54-2.38 (m, 1 H), 2.26-2.12 (m, 1 H), 2.03-1.90 (m, 1 H), 1.86 (app d, *J* = 10 Hz, 1 H), 1.65-1.50 (m, 1 H); ^{13}C NMR (75 MHz) δ (rotomers) 155.1, 152.2, 148.0, 137.2, 132.9, 132.7, 128.7, 128.2, 128.1, 124.7, 124.6, 114.4, 110.8, 67.2, 67.1, 57.4, 57.1, 50.0, 44.3, 40.5, 38.8, 30.6; IR (thin film, neat) 2956, 2859, 1662, 1424, 1238, 1108 cm^{-1}; mass spectrum (ESI) *m/z* 379.2017 [$C_{23}H_{27}N_2O_3$(M+1) requires 379.2016]; LCMS purity 97%.



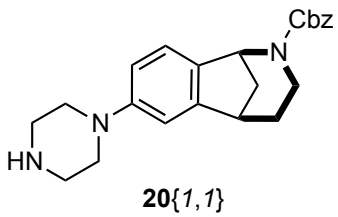
20{*2*,*11*}

Benzyl 8-(benzo[*d*][1,3]dioxol-5-yl)-4,5-dihydro-1*H*-1,5-methanobenzo[*c*]azepine-2(3*H*)-carboxylate (20{*2,11*}**).** 73%, colorless, viscous oil: ^{1}H NMR (400 MHz) δ 7.52-7.28 (comp, 7 H), 7.27 (d, *J* = 7.6 Hz, 1 H), 7.08-7.01 (comp, 2 H), 6.88 (d, *J* = 7.6 Hz, 1 H), 6.00 (s, 2 H), 5.93-5.89 and 5.47-5.44 (rotomers, m, 1 H), 5.29-5.14 (comp, 2 H), 3.95-3.80 (m, 1 H), 3.31 (app s, 1 H), 2.60-2.48 (m, 1 H), 2.31-2.18 (m, 1 H), 2.07-1.98 (m, 1 H), 1.92 (d, *J* = 11.2 Hz, 1 H), 1.72-1.58 (m, 1 H); ^{13}C NMR (100 MHz) δ (rotomers) 155.3, 155.1, 148.4, 147.2, 145.3, 142.1, 140.5, 137.3, 135.7, 128.7, 128.2, 127.3, 123.2, 122.6, 122.4, 120.8, 108.8, 107.9, 101.4, 67.3, 57.9, 57.6, 44.0, 39.7, 38.9, 30.5; IR (thin film, neat) 2945, 1696, 1475, 1235 cm^{-1}; mass spectrum (ESI) *m/z* 414.1706 [$C_{26}H_{24}NO_4$ (M+1) requires 414.1700]; LCMS purity 99%.



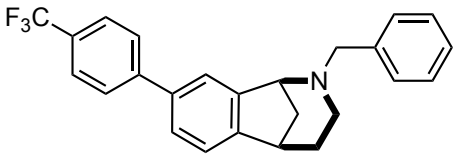
20{*3,6*}

Benzyl 9-phenyl-4,5-dihydro-1*H*-1,5-methanobenzo[*c*]azepine-2(3*H*)-carboxylate (20{*3,6*}**).** 82%, white solid: mp 97-98 °C; ^{1}H NMR (400 MHz) δ 7.39-7.23 (comp, 13 H), 5.43 (dd, *J* = 17.0, 3.8 Hz, 1 H), 5.27 and 5.00 (rotomers, d, *J* = 12.4 Hz, 1 H), 5.11 (dd, *J* = 12.4, 4.4 Hz, 1 H), 4.10-3.99 (m, 1 H), 3.37 (app s, 1 H), 2.75 and 2.64 (rotomers, td, *J* = 12.9, 4.8 Hz, 1 H), 2.21-2.13 (m, 1 H), 2.11-1.98 (m, 1 H), 1.92-1.85 (m, 1 H), 1.72-1.58 (m, 1 H); ^{13}C NMR (75 MHz) (rotomers) δ 154.7, 154.6, 147.8, 147.6, 140.5, 140.2, 138.8, 138.6, 137.5, 137.1, 129.1, 128.9, 128.7, 128.6, 128.5, 128.2, 128.1, 128.0, 127.9, 127.6, 127.4, 121.9, 121.7, 67.2, 67.1, 57.0, 56.9, 44.5, 40.7, 39.1, 38.9, 31.2, 30.9; IR (thin film, neat) 3066, 2935, 1698, 1437, 1314, 1259, 1197 cm^{-1}; mass spectrum (ESI) *m/z* 392.1629 [$C_{25}H_{23}NNaO_2$ (M+Na) requires 392.1626]; LCMS purity 98%.



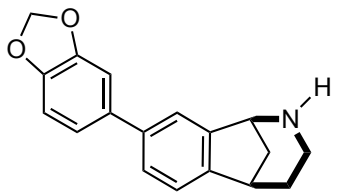
20{*1,1*}

Benzyl 7-(piperazin-1-yl)-4,5-dihydro-1*H*-1,5-methanobenzo[*c*]azepine-2(3*H*)-carboxylate (20{*1*,*1*}). 75%, colorless oil. ^{1}H NMR (400 MHz) δ 7.44-7.27 (comp, 5 H), 7.21 and 7.09 (rotomers, d, *J* = 7.6 Hz, 1 H), 6.83 (d, *J* = 2.4 Hz, 1 H), 6.78-6.70 (m, 1 H), 5.44-5.42 and 5.34-5.31 (rotomers, m, 1 H), 5.22-5.06 (comp, 2 H), 3.88-3.73 (m, 1 H), 3.22-3.00 (comp, 8 H), 2.80-2.68 (m, 1 H), 2.52-2.38 (m, 1 H), 2.24-2.12 (m, 1 H), 2.05-1.90 (m, 1 H), 1.84 (d, *J* = 10.4 Hz, 1 H), 1.62-1.50 (m, 1 H); ^{13}C NMR (75 MHz) (rotomers) δ 155.1, 152.7, 147.9, 137.4, 137.2, 132.6, 132.3, 128.7, 128.1, 128.0, 124.6, 124.4, 114.7, 111.2, 67.1, 57.4, 57.2, 51.0, 46.5, 44.3, 40.5, 38.8, 30.6; IR (thin film, neat) 2942, 2825, 1692, 1417, 1231, 1101 cm^{-1}; mass spectrum (ESI) *m/z* 378.21766 [$C_{23}H_{28}N_3O_2$ (M+1) requires 378.21815]; LCMS purity 99%.



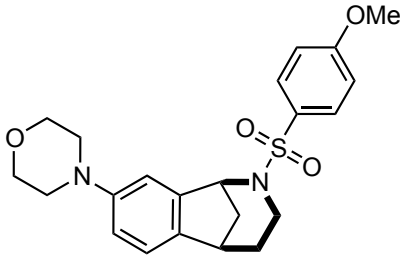
21{*2*,*7*}

2-Benzyl-8-(4-(trifluoromethyl)phenyl)-2,3,4,5-tetrahydro-1*H*-1,5-methanobenzo[*c*]azepine (21{*2*,*7*}). 91%, tan solid: mp 101-103 °C; 7.54-7.71 (comp, 4 H), 7.50 (d, *J* = 7.6 Hz, 1 H), 7.43-7.25 (comp, 7 H), 4.00 (d, *J* = 4.8 Hz, 1 H), 3.51 (d, *J* = 13.2 Hz, 1 H), 3.35 (d, *J* = 13.2 Hz, 1 H), 3.23-3.19 (m, 1 H), 2.64 (dd, *J* = 10.0, 5.6 Hz, 1 H), 2.27-2.20 (m, 1 H), 2.11-2.07 (m, 1 H), 2.04 (app d, *J* = 11.2 Hz, 1 H), 1.62-1.58 (m, 1 H), 1.57 (app d, *J* = 9.2 Hz, 1 H); ^{13}C NMR (75 MHz) δ 147.3, 145.4, 138.4, 129.5, 129.2, 128.7, 127.7, 127.4, 127.2, 126.4, 126.1, 126.0, 125.9, 123.3, 122.8, 63.0, 60.4, 47.3, 44.6, 39.8, 29.9; IR (thin film, neat) 2946, 2851, 1321, 1131 cm^{-1}; mass spectrum (ESI) *m/z* 394.17780 [$C_{25}H_{23}F_3N$ (M+1) requires 394.17771]; LCMS purity 96%.



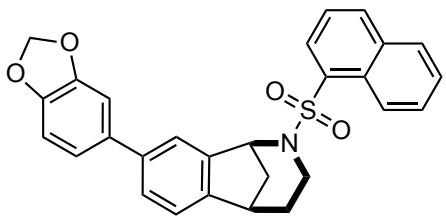
23{*2*,*11*}

8-(Benzo[*d*][1,3]dioxol-5-yl)-2,3,4,5-tetrahydro-1*H*-1,5-methanobenzo[*c*]azepine (23{*2,11*}**).** 93%, colorless oil: ^{1}H NMR (400 MHz) δ 7.33-7.28 (comp, 2 H), 7.14 (d, *J* = 7.8 Hz, 1H), 6.99-6.95 (comp, 2 H), 6.78 (d, *J* = 7.8 Hz, 1 H), 5.90 (s, 2 H), 4.18 (d, *J* = 4.0 Hz, 1 H), 3.15-3.09 (m, 1 H), 2.65 (dd, *J* = 12.0, 5.6 Hz, 1 H), 2.28 (td, *J* = 12.0, 4.7 Hz, 1 H), 2.17-2.11 (m, 1 H), 2.00 (app s, 1 H), 1.95-1.85 (m, 1 H), 1.86 (d, *J* = 10.4 Hz, 1 H), 1.52-1.44 (m, 1 H); IR (thin film, neat) 2951, 1509, 1482, 1228, 1041 cm^{-1}; mass spectrum (ESI) *m/z* 280.1337 [$C_{18}H_{18}NO_2$ (M+1) requires 280.1332]; LCMS purity 95%.



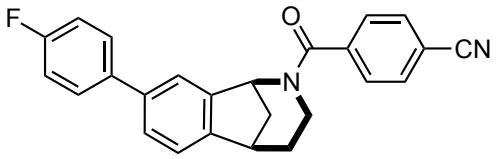
30{*2,2,30*}

4-(2-((4-Methoxyphenyl)sulfonyl)-2,3,4,5-tetrahydro-1*H*-1,5-methanobenzo[*c*]azepin-8-yl)morpholine (30{*2,2,30*}**).** 62%, white solid: mp (EtOH) 176-177 °C; ^{1}H NMR (400 MHz) δ 7.73 (dd, *J* = 6.8, 2.0 Hz, 2 H), 7.03 (d, *J* = 8.0 Hz, 1 H), 6.95 (dd, *J* = 6.8, 2.0 Hz, 2 H), 6.69 (dd, *J* = 8.0, 2.4 Hz, 1 H), 6.07 (d, *J* = 2.4 Hz, 1 H), 4.99 (d, *J* = 4.0 Hz, 1 H), 3.86 (s, 3 H), 3.78 (td, *J* = 4.8, 1.6 Hz, 4 H), 3.64 (dd, *J* = 11.9, 6.0 Hz, 1 H), 3.15 (br s, 1 H), 2.96-2.81 (m, 4 H), 2.29 (td, *J* = 11.9, 4.4 Hz, 1 H), 2.19-2.11 (m, 1 H), 2.10-2.01 (m, 1 H), 1.95 (d, *J* = 10.8 Hz, 1 H), 1.55 (br d, *J* = 12.4 Hz, 1 H); ^{13}C NMR (75 MHz) δ 162.9, 150.9, 139.6, 138.0, 132.3, 129.7, 123.0, 116.3, 114.4, 112.2, 67.1, 59.9, 55.8, 50.2, 44.7, 40.6, 39.0, 30.6; IR (thin film, neat) 2917, 1593, 1494, 1326, 1253, 1156 cm^{-1}; mass spectrum (ESI) *m/z* 415.1687 [$C_{22}H_{27}N_2O_4S$ (M+1) requires 415.1692]; LCMS purity 100%.



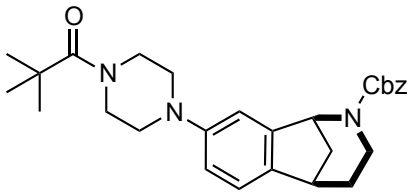
30{*2,11,24*}

8-(Benzo[*d*][1,3]dioxol-5-yl)-2-(naphthalen-1-ylsulfonyl)-2,3,4,5-tetrahydro-1*H*-1,5-methanobenzo[*c*]azepine (30{*2*,*11*,*24*}**).** 59%, white foam. ^{1}H NMR (400 MHz) δ 8.72 (d, *J* = 8.6 Hz, 1 H), 8.29 (dd, *J* = 7.5, 1.4 Hz, 1 H), 8.02 (d, *J* = 8.6 Hz, 1 H), 7.87 (d, *J* = 7.5 Hz, 1 H), 7.64-7.59 (m, 1 H), 7.57-7.52 (comp, 2 H), 7.27 (d, *J* = 8.4 Hz, 1 H), 7.14 (d, *J* = 7.6 Hz, 1 H), 6.82 (d, *J* = 8.4 Hz, 1 H), 6.71 (dd, *J* = 8.4, 1.7 Hz, 1 H), 6.66 (d, *J* = 1.7 Hz, 1 H), 6.62-6.61 (m, 1 H), 6.02 (s, 2 H), 5.19 (d, *J* = 4.0 Hz, 1 H), 3.65 (dd, *J* = 12.0, 6.0 Hz, 1 H), 3.26-3.22 (m, 1 H), 2.45 (td, *J* = 12.0, 4.8 Hz, 1 H), 2.23-2.16 (m, 1 H), 2.08-1.99 (m, 1 H), 1.97 (d, *J* = 11.2 Hz, 1 H), 1.63-1.56 (m, 1 H); ^{13}C NMR (125 MHz) δ 147.9, 146.9, 144.7, 140.2, 139.5, 135.2, 135.1, 134.4, 134.2, 130.1, 128.9, 128.8, 128.0, 127.2, 126.8, 125.2, 124.3, 122.6, 122.5, 120.5, 108.3, 107.7, 101.1, 58.9, 44.2, 40.2, 39.3, 30.3; IR (thin film, neat) 2969, 2873, 1479, 1341, 1224, 1163 cm^{-1}; mass spectrum (ESI) *m/z* 470.14196 [$C_{28}H_{24}NO_4S$ (M+1) requires 470.14206]; LCMS purity 100%.



28{*2*,*10*,*15*}

8-(4-Fluorophenyl)-2,3,4,5-tetrahydro-1*H*-1,5-methanobenzo[*c*]azepine-2-carbonyl)benzonitrile (28{*2*,*10*,*15*}**).** 82%, pale green gum. ^{1}H NMR (400 MHz) δ 7.80 (d, *J* = 8.2 Hz, 1 H), 7.66 (d, *J* = 8.2 Hz, 1 H) 7.65-7.47 (comp, 5 H), 7.46 (d, *J* = 7.6 Hz, 1 H), 7.32 (d, *J* = 7.6 Hz, 1 H), 7.20-7.09 (comp, 2 H), 6.01 and 4.86 (rotomers, d, *J* = 4.2 Hz, 1 H), 4.32 and 3.28 (rotomers, dd, *J* = 13.6, 6.4 Hz, 1 H), 3.45-3.39 (m, 1 H), 2.76 and 2.48 (rotomers, td, *J* = 12.8, 5.2 Hz, 1 H), 2.42-2.35 and 2.27-2.20 (rotomers, m, 1 H), 2.19-2.10 and 1.98-1.90 (rotomers, m, 1 H), 2.00 (d, *J* = 11.6 Hz, 1 H), 1.85-1.77 and 1.62-1.54 (rotomers, m, 1 H); ^{13}C NMR (75 MHz) (rotomers) δ 167.9, 163.3 (d, $J_{C\text{-}F}$ = 245 Hz), 146.0, 145.4, 144.4, 141.2, 140.8, 140.6, 140.2, 137.2, 133.0, 132.6, 128.9, 128.8, 128.4, 128.1, 127.9, 123.6, 123.4, 123.0, 122.2, 118.4, 116.0 (d, $J_{C\text{-}F}$ = 21.3 Hz), 113.6, 61.4, 55.9, 44.4, 43.0, 40.0, 37.5, 31.5, 30.3; IR (thin film, neat) 2949, 1637, 1437, 1231 cm^{-1}; mass spectrum (ESI) *m/z* 383.15611 [$C_{25}H_{20}FN_2O$ (M+1) requires 383.15542]; LCMS purity 100%.

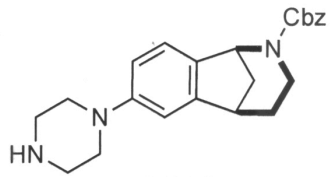


38{*2*,*1*,*9*}

Benzyl 8-(4-pivaloylpiperazin-1-yl)-4,5-dihydro-1*H*-1,5-methanobenzo[*c*]azepine-2(3*H*)-carboxylate (38{*2*,*1*,*9*}**).** 96%, tan semi-solid: ^{1}H NMR (400 MHz) δ 7.44-7.26 (m, 5 H), 7.12 (d, *J* = 8.0 Hz, 1 H), 6.80 (dd, *J* = 8.2, 2.2 Hz, 1 H) 6.96 and 6.75 (rotomers, br s, 1 H), 5.45 and 5.34 (rotomers, br s, 1 H), 5.26-5.07 (comp, 2 H), 3.80 (m, 1 H), 3.79 (t, *J* = 4.8 Hz, 4 H), 3.23-3.19 (m, 1 H), 3.15-3.06 (m, 4 H), 2.50-2.36 (m, 1 H), 2.24-2.12 (m, 1 H), 2.02-1.90 (m, 1 H), 1.85 (d, *J* = 10.8 Hz, 1 H), 1.62-1.50 (m, 1 H), 1.31 (s, 9 H); ^{13}C NMR (75 MHz) δ (rotomers) 176.6, 155.3, 155.2, 151.0, 142.6, 142.5, 138.6, 137.4, 137.3, 128.7, 128.2, 123.4, 116.8, 116.6, 112.9, 67.2, 58.1, 57.9, 54.0, 50.4, 45.3, 44.0, 39.2, 38.9, 30.7, 28.7; IR (thin film, neat) 2941, 2880, 2818, 1698, 1630, 1417, 1238, 1183 cm^{-1}; (ESI) *m/z* 462.2753 [$C_{28}H_{36}N_3O_3$ (M+1) requires 462.2751]; LCMS purity 100%.

Tabulated Lipinski Parameters

Compound #	Molecular Weight	C log P	H-Bond Donors	H-Bond Acceptors	Lipinski Rule of 5
18{*3*}	327.805	4.45	0	1	satisfied
20{*1,1*}	377.479	3.42	1	3	satisfied
20{*2,4*}	364.481	4.64	1	2	satisfied
20{*2,7*}	437.454	6.37	0	1	one violation
20{*2,9*}	399.482	5.33	0	2	one violation
20{*2,11*}	413.465	5.11	0	3	one violation
21{*2*}	283.795	4.60	0	1	satisfied
21{*2,3*}	332.482	4.96	0	2	satisfied
21{*2,8*}	355.472	5.49	0	2	one violation
23{*2,2*}	244.332	1.78	1	3	satisfied
23{*2,6*}	235.324	3.54	1	1	satisfied
23{*2,8*}	265.350	3.38	1	2	satisfied
23{*3,7*}	303.322	4.41	1	1	satisfied
25{*2,1*}	313.821	4.44	0	2	satisfied
26{*2,2,5*}	403.345	5.09	0	3	one violation
26{*2,7,8*}	317.348	4.80	0	1	satisfied
26{*2,9,1*}	369.499	6.00	0	2	one violation
26{*2,3,4*}	366.927	5.56	0	2	one violation
27{*2,10*}	303.826	4.22	0	1	satisfied
28{*2,3,19*}	415.355	5.52	0	2	one violation
28{*2,6,15*}	364.439	4.82	0	2	satisfied
28{*2,11,13*}	397.466	5.14	0	3	one violation
28{*2,11,18*}	459.535	6.27	0	3	one violation
28{*2,8,9*}	349.466	4.79	0	2	satisfied
28{*2,3,10*}	352.513	4.57	0	2	satisfied
28{*2,2,20*}	382.883	3.85	0	3	satisfied
28{*3,7,11*}	375.384	3.85	0	2	satisfied
29{*2,24*}	383.891	4.70	0	2	satisfied
30{*2,6,27*}	313.414	2.64	0	2	satisfied
30{*2,2,30*}	414.518	2.84	0	5	satisfied
31{*2,37*}	265.735	3.08	0	1	satisfied
32{*2,8,39*}	349.423	4.34	0	2	satisfied
33{*1,31*}	353.868	4.76	1	1	satisfied
33{*1,32*}	388.911	4.59	1	2	satisfied
34{*3,7,35*}	466.561	6.82	1	0	one violation
35{*2,8,41*}	318.412	3.53	0	3	satisfied
38{*1,1,21*}	481.585	4.88	0	3	satisfied
38{*2,1,9*}	461.596	4.82	0	3	satisfied
39{*2,1,30*}	547.665	4.48	0	5	one violation
40{*1,1,32*}	572.718	5.51	1	4	violated

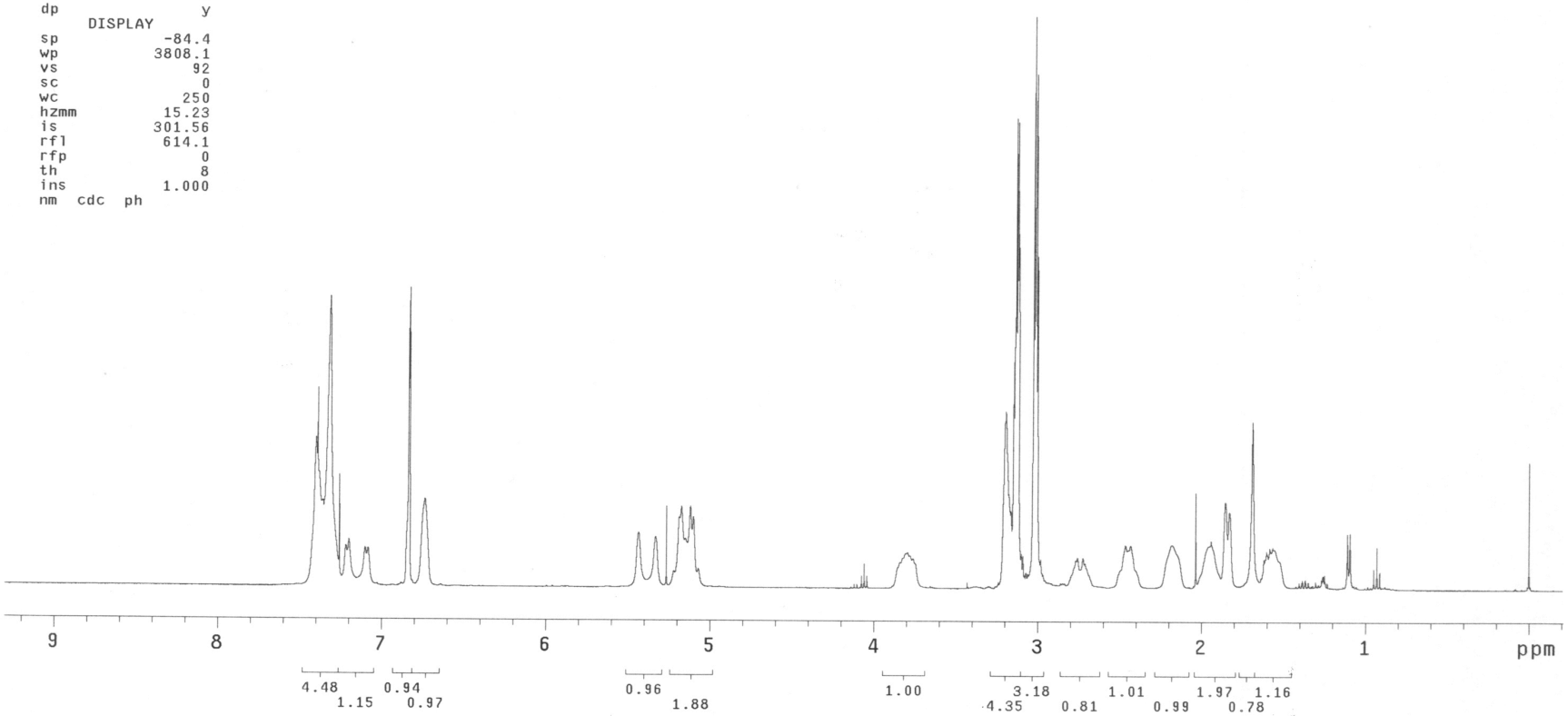


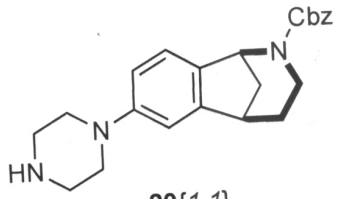
20{*1,1*}

STANDARD 1H OBSERVE

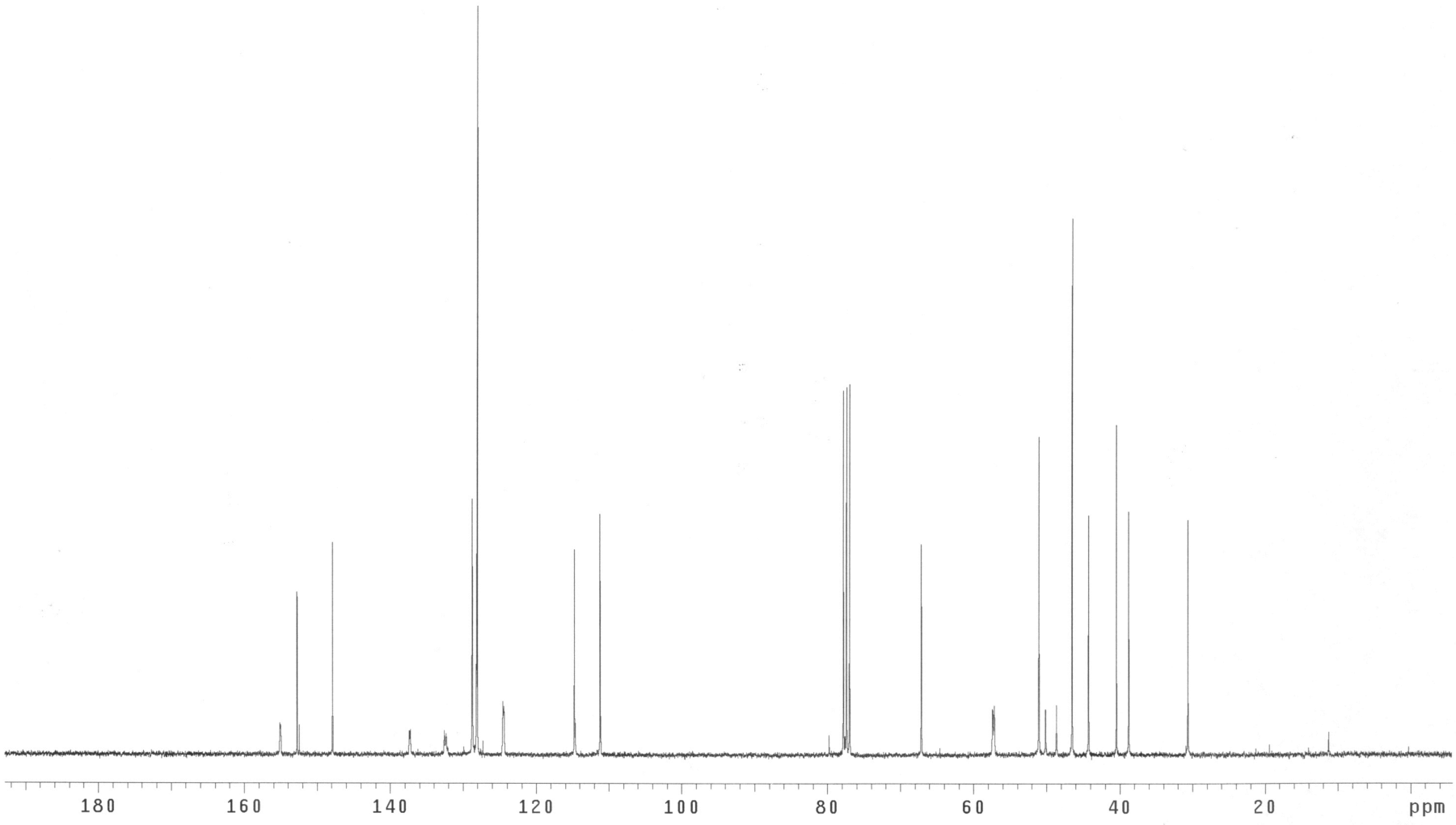
exp1 std1h

SAMPLE		DEC. & VT	
date	Aug 14 2010	dfrq	400.269
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	400.269	dm	nnn
tn	H1	dmm	c
at	2.856	dmf	200
np	32000	PROCESSING	
sw	5602.2	lb	0.10
fb	not used	wtfile	
bs	2	proc	ft
tpwr	58	fn	not used
pw	2.0		
d1	2.000	werr	
tof	169.9	wexp	
nt	100	wbs	
ct	78	wnt	
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-84.4		
wp	3808.1		
vs	92		
sc	0		
wc	250		
hzmm	15.23		
is	301.56		
rfl	614.1		
rfp	0		
th	8		
ins	1.000		
nm cdc ph			

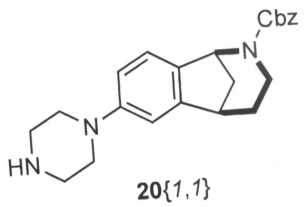




20{*1,1*}

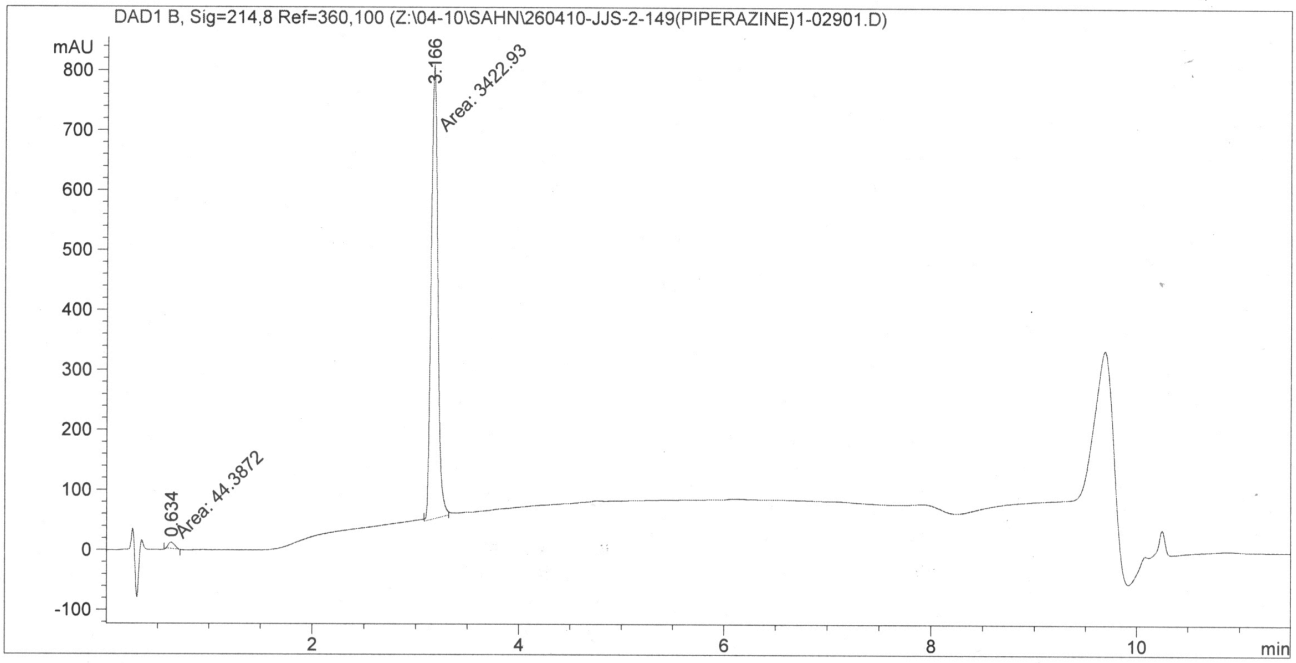


Data File Z:\04-10\SAHN\260410-JJS-2-149(PIPERAZINE)1-02901.D
Sample Name: jjs-2-149(piperazine)

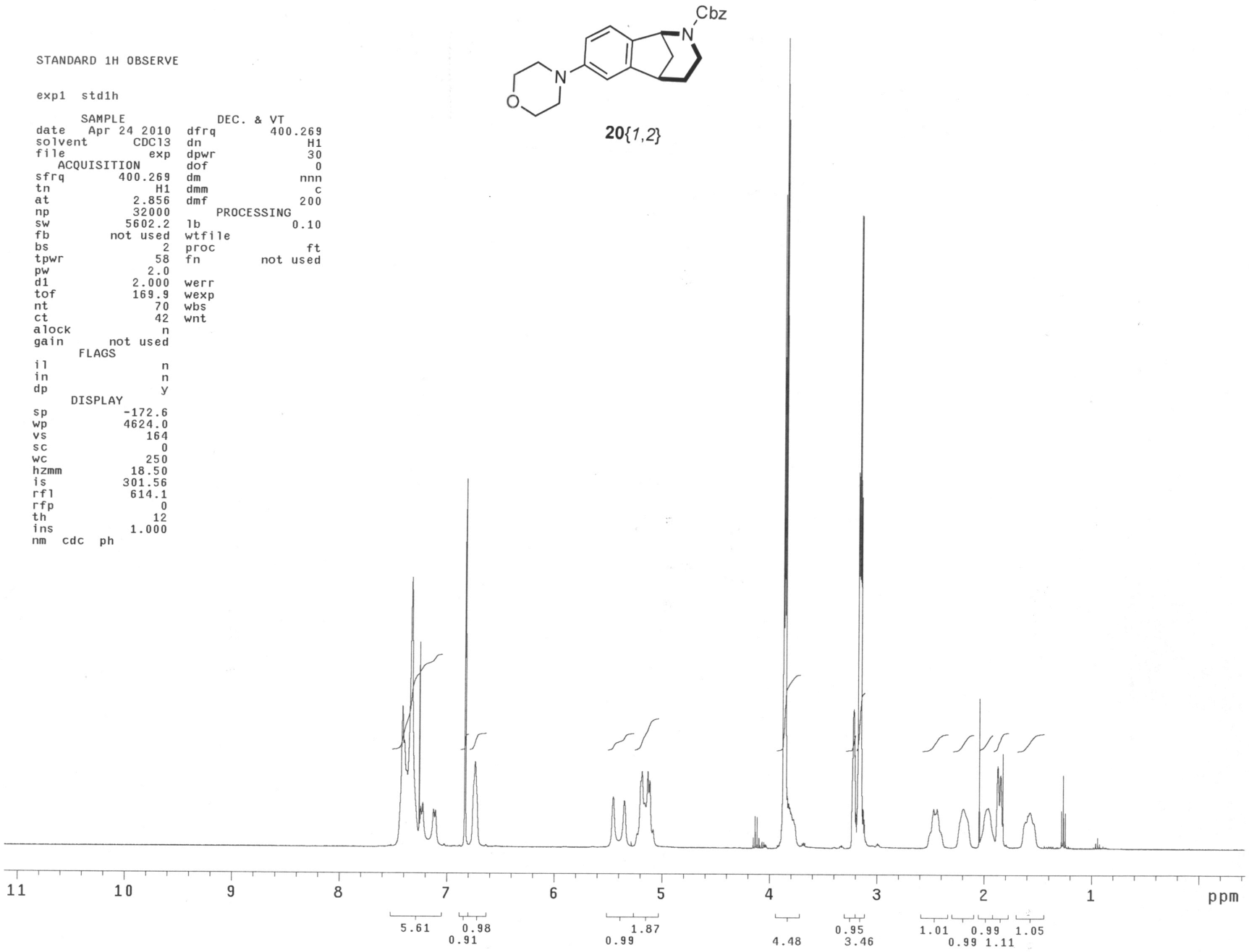


20{*1,1*}

```
==================================================================
Acq. Operator    : sahn@mail.utexas.edu
Acq. Instrument  : LCMS                          Location :
Injection Date   : 4/26/2010 3:03:15 PM
                                                 Inj Volume :
Acq. Method      : C:\CHEM32\1\METHODS\SP NIH.M
Last changed     : 4/26/2010 3:03:04 PM by sahn@mail.utexas.edu
                   (modified after loading)
Analysis Method  : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed     : 11/20/2006 4:14:44 AM
Sample Info      : Easy-Access Method: 'SP NIH'
```



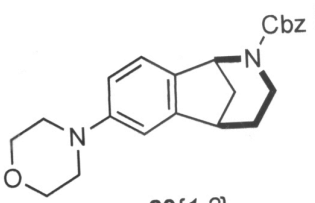

```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   0.634 MM    0.0707   44.38724   10.46296   1.2802
   2   3.166 MM    0.0751 3422.92969  759.33624  98.7198

Totals :                  3467.31693  769.79921
```


STANDARD 1H OBSERVE
exp1 std1h
SAMPLE
date Apr 24 2010
solvent CDCl3
file exp
ACQUISITION
sfrq 400.269
tn H1
at 2.856
np 32000
sw 5602.2
fb not used
bs 2
tpwr 58
pw 2.0
d1 2.000
tof 169.9
nt 70
ct 42
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -172.6
wp 4624.0
vs 164
sc 0
wc 250
hzmm 18.50
is 301.56
rfl 614.1
rfp 0
th 12
ins 1.000
nm cdc ph
DEC. & VT
dfrq 400.269
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
lb 0.10
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
Cbz
N
N
O
20{1,2}
11
10
9
8
7
6
5
4
3
2
1
ppm
5.61
0.98
0.91
1.87
0.99
4.48
0.95
3.46
1.01
0.99
1.11
1.05

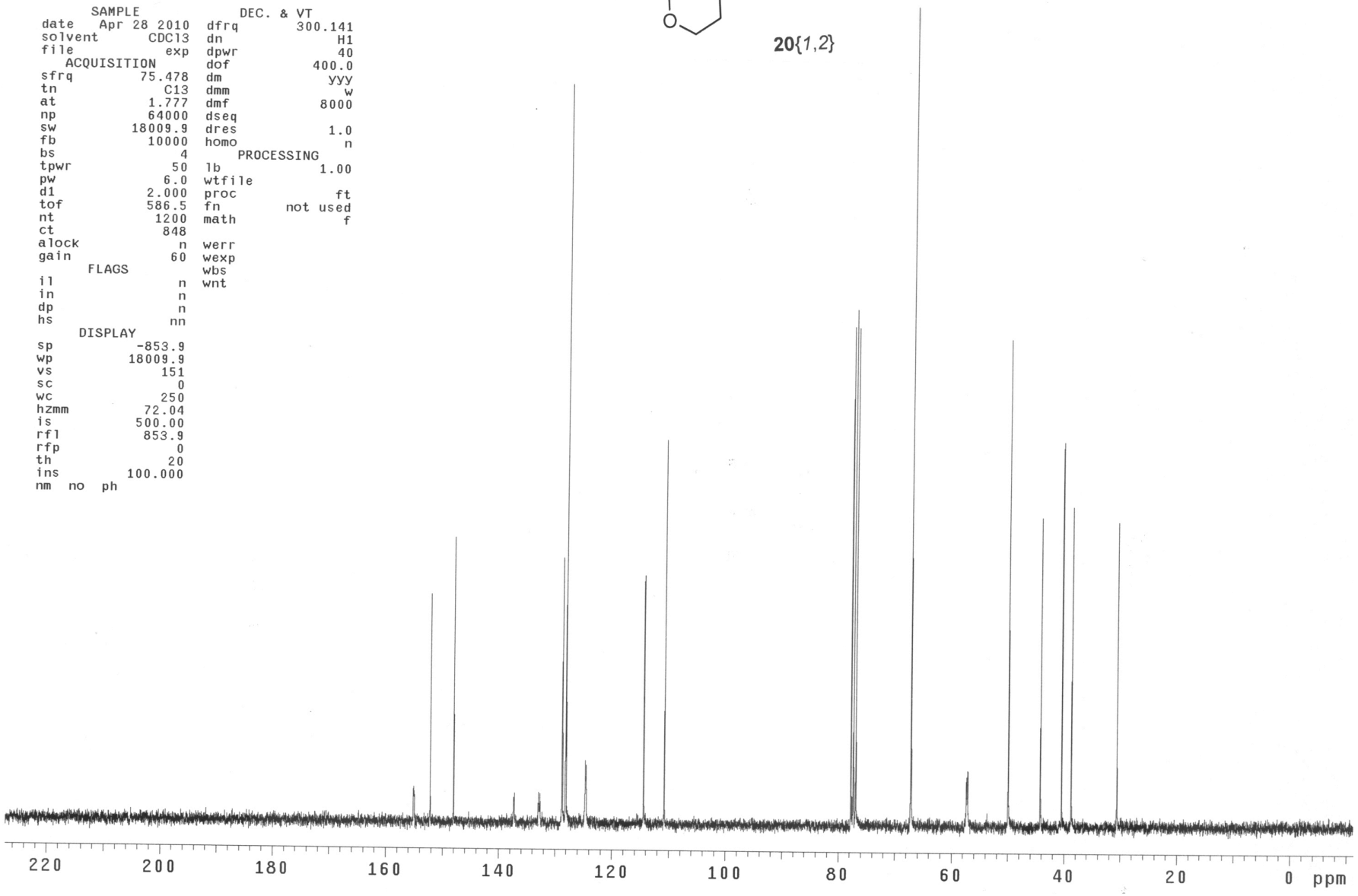
13C OBSERVE

exp1 std13c

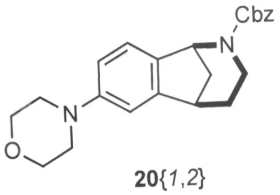
SAMPLE		DEC. & VT	
date	Apr 28 2010	dfrq	300.141
solvent	CDCl3	dn	H1
file	exp	dpwr	40
ACQUISITION		dof	400.0
sfrq	75.478	dm	yyy
tn	C13	dmm	w
at	1.777	dmf	8000
np	64000	dseq	
sw	18009.9	dres	1.0
fb	10000	homo	n
bs	4	PROCESSING	
tpwr	50	lb	1.00
pw	6.0	wtfile	
d1	2.000	proc	ft
tof	586.5	fn	not used
nt	1200	math	f
ct	848		
alock	n	werr	
gain	60	wexp	
FLAGS		wbs	
il	n	wnt	
in	n		
dp	n		
hs	nn		
DISPLAY			
sp	-853.9		
wp	18009.9		
vs	151		
sc	0		
wc	250		
hzmm	72.04		
is	500.00		
rfl	853.9		
rfp	0		
th	20		
ins	100.000		
nm no ph			



20{*1,2*}

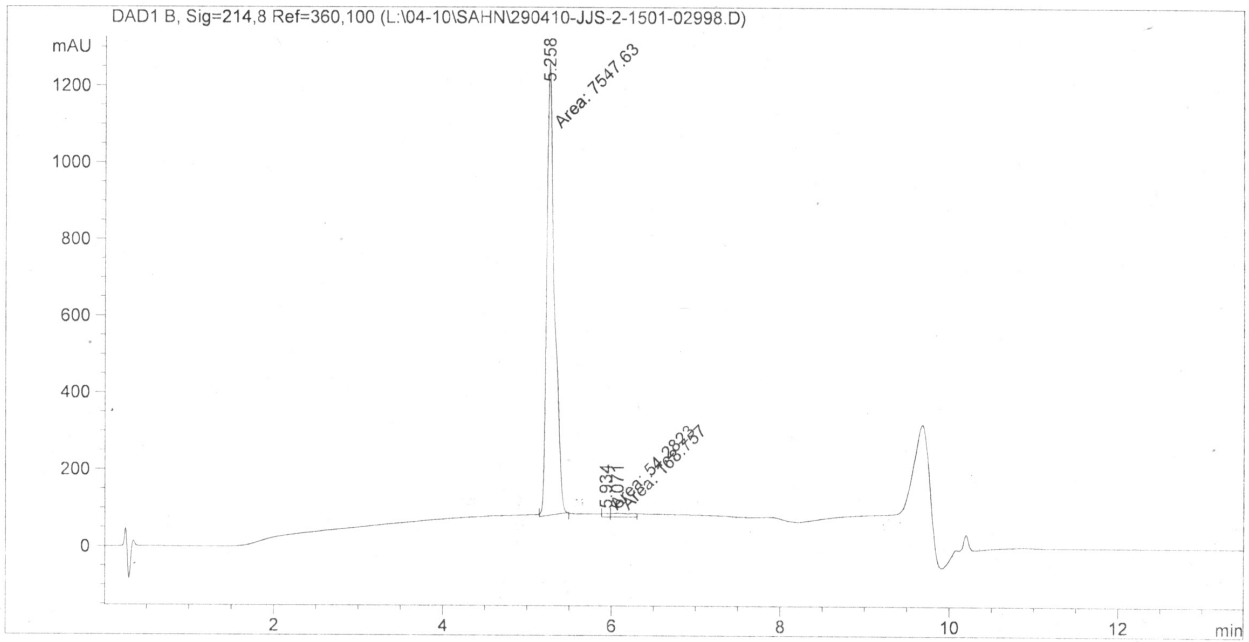


Data File L:\04-10\SAHN\290410-JJS-2-1501-02998.D
Sample Name: jjs-2-150

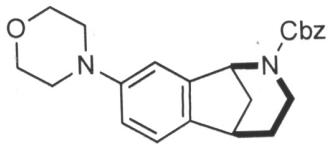


20{*1,2*}

```
Acq. Operator    : sahn@mail.utexas.edu
Acq. Instrument  : LCMS                          Location : Vial 35
Injection Date   : 4/29/2010 9:34:05 PM
                                                 Inj Volume : 1.0 µl
Acq. Method      : C:\CHEM32\1\METHODS\SP NIH.M
Last changed     : 4/29/2010 9:33:45 PM by sahn@mail.utexas.edu
                   (modified after loading)
Analysis Method  : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed     : 11/20/2006 4:14:44 AM
Sample Info      : Easy-Access Method: 'SP NIH'
```




```
                          Area Percent Report

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]      %
----|-------|----|-------|----------|----------|--------|
   1   5.258 MM    0.1065 7547.62891 1181.65027  97.1297
   2   5.934 MM    0.1015   54.28227    8.91613   0.6986
   3   6.071 MM    0.2922  168.75677    9.62579   2.1717

Totals :                  7770.66795 1200.19219
```



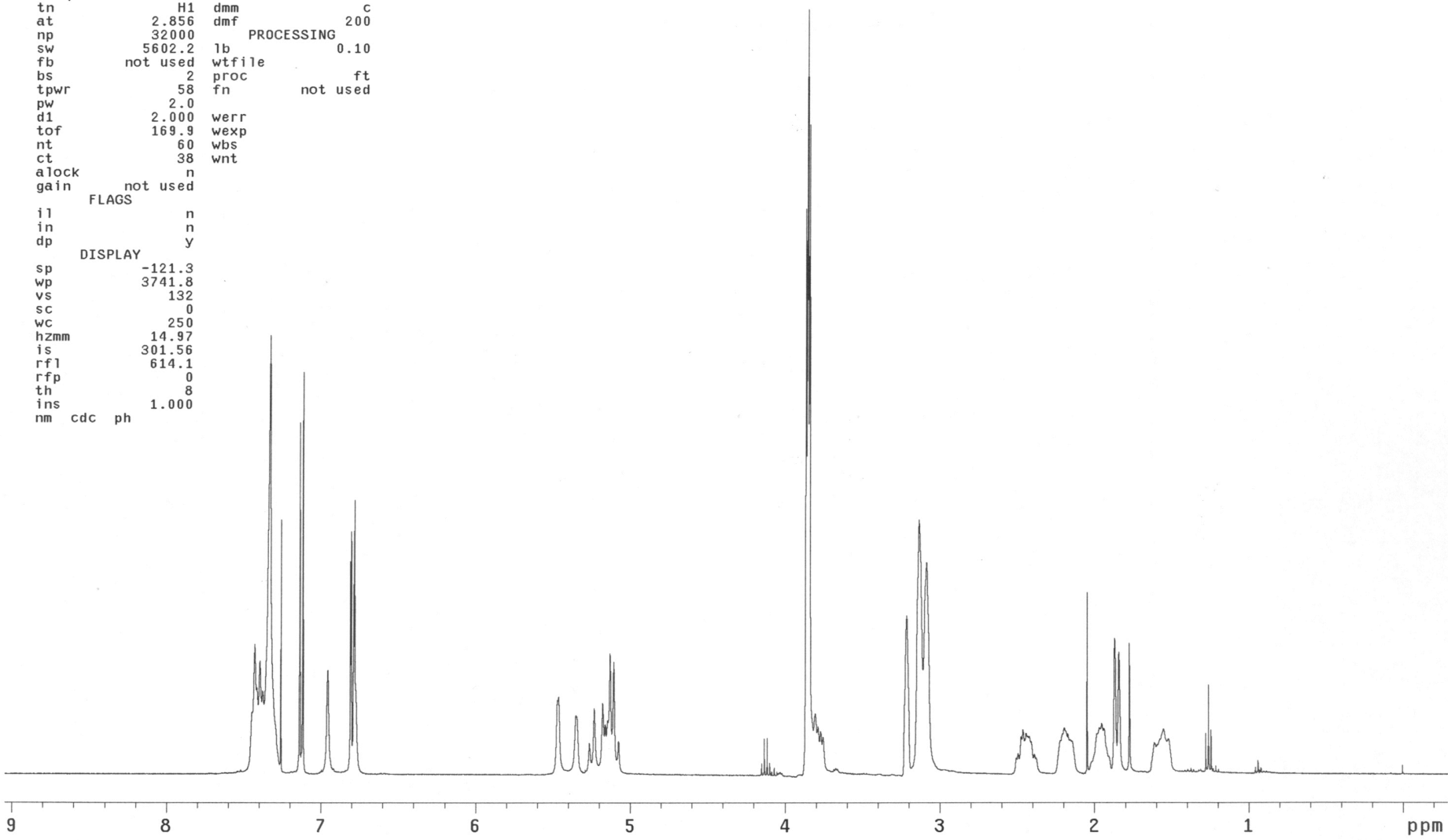
20{2,2}

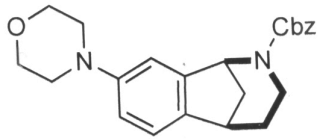
STANDARD 1H OBSERVE

exp1 std1h

SAMPLE		DEC. & VT	
date	Apr 22 2009	dfrq	400.269
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	400.269	dm	nnn
tn	H1	dmm	c
at	2.856	dmf	200
np	32000	PROCESSING	
sw	5602.2	lb	0.10
fb	not used	wtfile	
bs	2	proc	ft
tpwr	58	fn	not used
pw	2.0		
d1	2.000	werr	
tof	169.9	wexp	
nt	60	wbs	
ct	38	wnt	
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-121.3		
wp	3741.8		
vs	132		
sc	0		
wc	250		
hzmm	14.97		
is	301.56		
rfl	614.1		
rfp	0		
th	8		
ins	1.000		

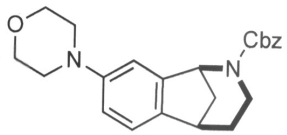
nm cdc ph





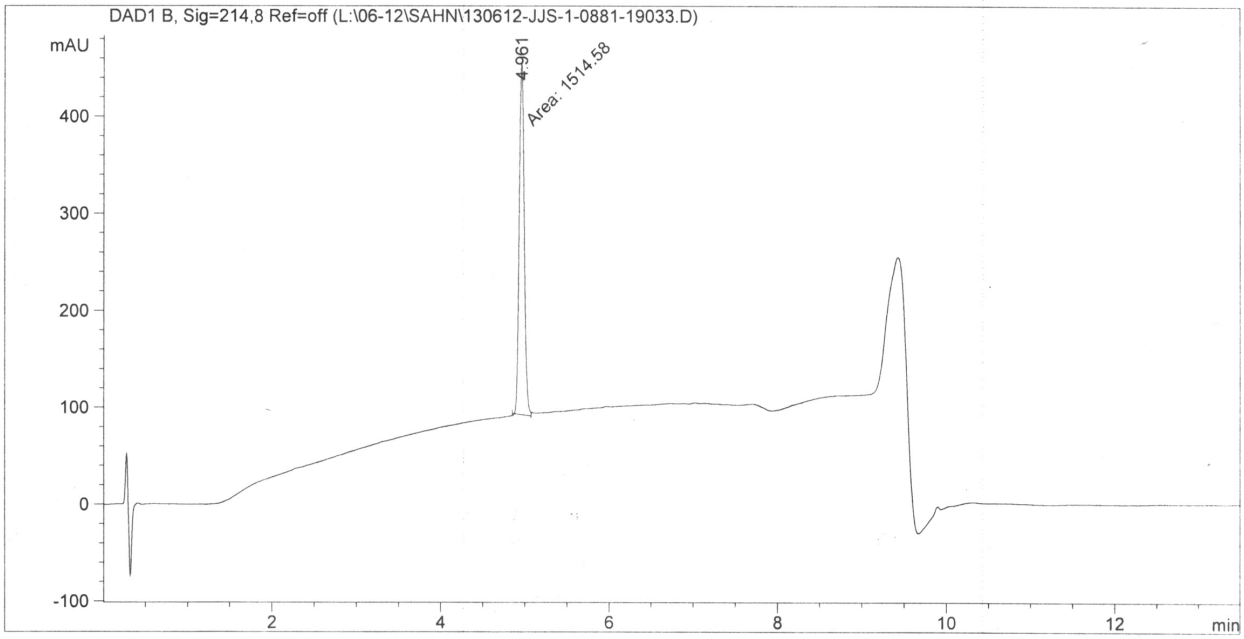
20{2,2}

Data File L:\06-12\SAHN\130612-JJS-1-0881-19033.D
Sample Name: jjs-1-088

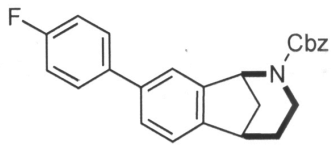


20{*2,2*}

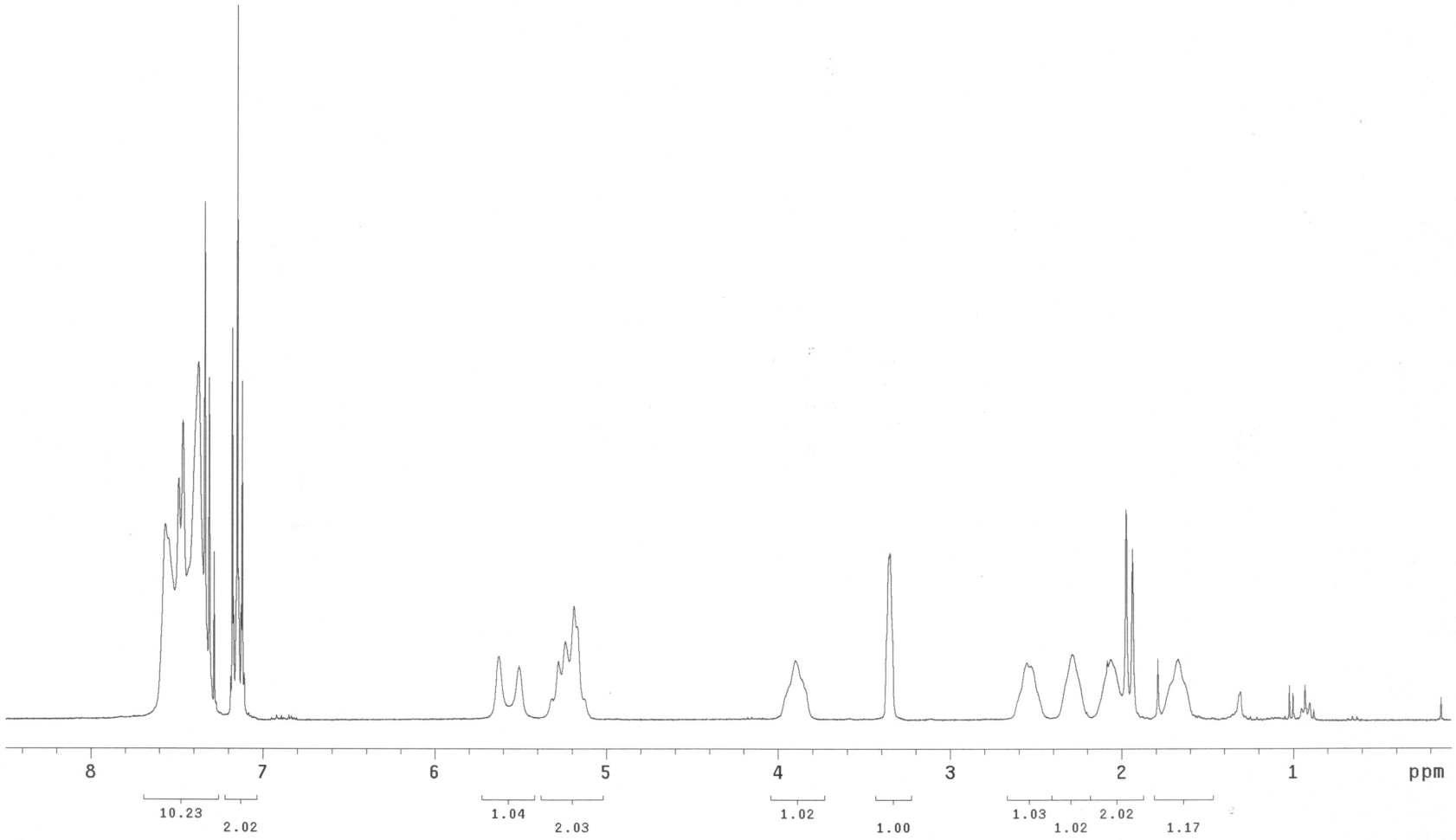
```
Acq. Operator    : sahn@mail.utexas.edu
Acq. Instrument  : LCMS                          Location : Vial 42
Injection Date   : 6/13/2012 8:08:33 PM
                                                 Inj Volume : 1.0 µl
Acq. Method      : C:\CHEM32\1\METHODS\SP NIH.M
Last changed     : 6/13/2012 8:08:18 PM by sahn@mail.utexas.edu
                   (modified after loading)
Analysis Method  : C:\CHEM32\1\METHODS\DEEF_LC.M
Last changed     : 6/13/2012 3:34:08 PM
                   (modified after loading)
Sample Info      : Easy-Access Method: 'SP NIH'
```

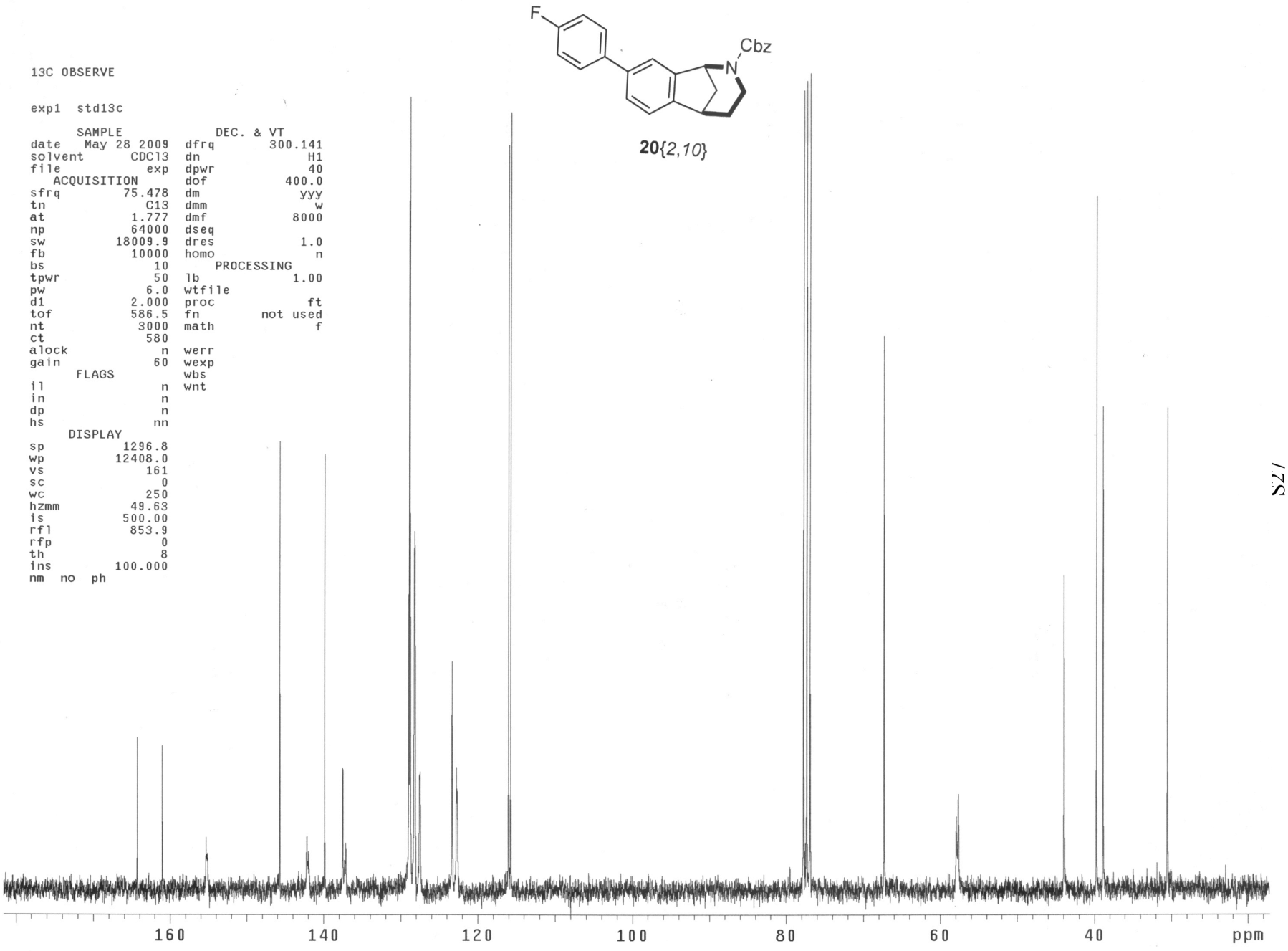



```
                        Area Percent Report

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,8 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   4.961 MM    0.0687 1514.57874  367.32224 100.0000

Totals :                  1514.57874  367.32224
```

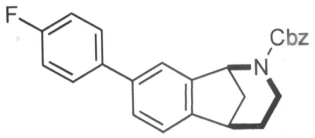


20{*2,10*}



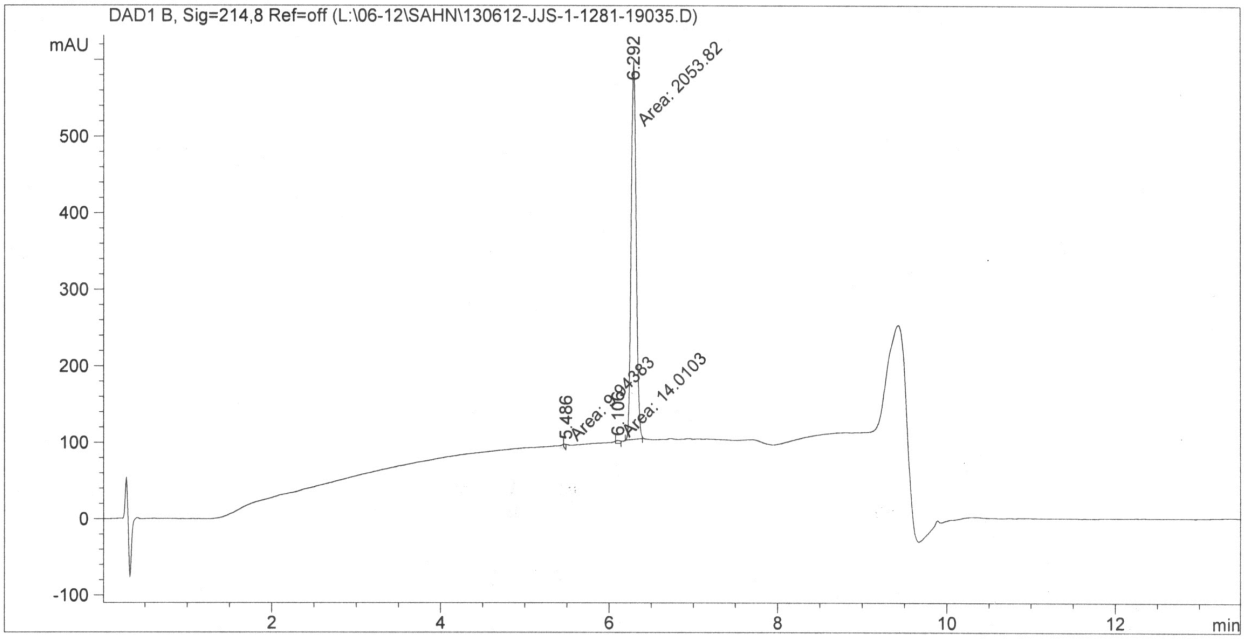

F
Cbz
N
20{2,10}
13C OBSERVE
exp1 std13c
SAMPLE
date May 28 2009
solvent CDCl3
file exp
ACQUISITION
sfrq 75.478
tn C13
at 1.777
np 64000
sw 18009.9
fb 10000
bs 10
tpwr 50
pw 6.0
d1 2.000
tof 586.5
nt 3000
ct 580
alock n
gain 60
FLAGS
il n
in n
dp n
hs nn
DISPLAY
sp 1296.8
wp 12408.0
vs 161
sc 0
wc 250
hzmm 49.63
is 500.00
rfl 853.9
rfp 0
th 8
ins 100.000
nm no ph
DEC. & VT
dfrq 300.141
dn H1
dpwr 40
dof 400.0
dm yyy
dmm w
dmf 8000
dseq
dres 1.0
homo n
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
160
140
120
100
80
60
40
ppm

Data File L:\06-12\SAHN\130612-JJS-1-1281-19035.D
Sample Name: jjs-1-128

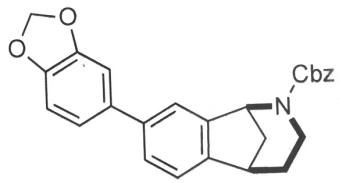


20{*2,10*}

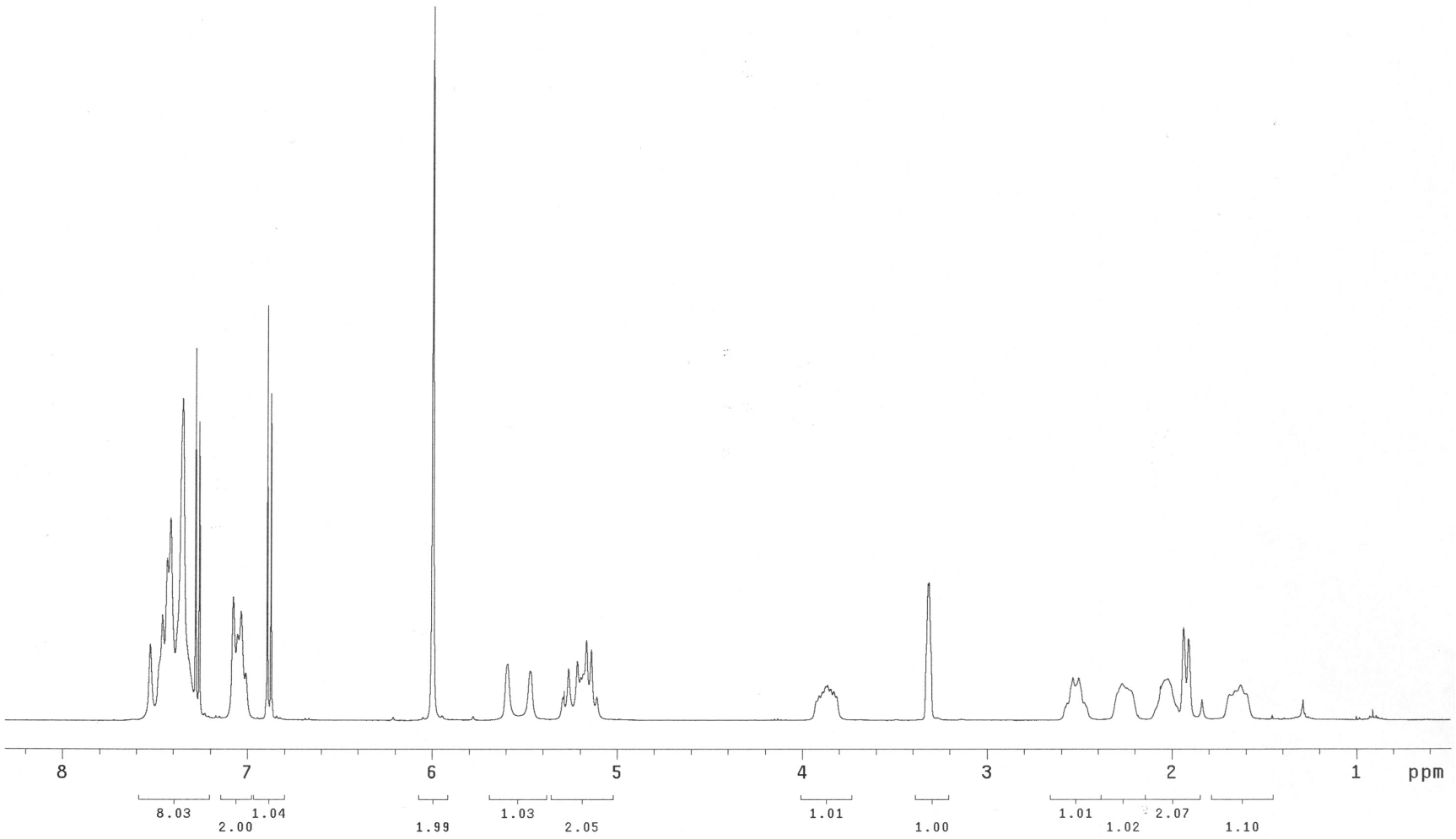
```
==========================================================================
Acq. Operator    : sahn@mail.utexas.edu
Acq. Instrument : LCMS                          Location : Vial 44
Injection Date   : 6/13/2012 8:38:34 PM
                                                Inj Volume : 1.0 µl
Acq. Method      : C:\CHEM32\1\METHODS\SP NIH.M
Last changed     : 6/13/2012 8:38:19 PM by sahn@mail.utexas.edu
                   (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEEF_LC.M
Last changed     : 9/28/2011 3:20:36 PM
Sample Info      : Easy-Access Method: 'SP NIH'
```

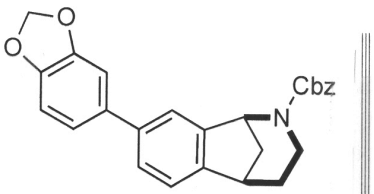



```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,8 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]      %
----|-------|----|-------|----------|----------|--------|
   1   5.486 MM    0.0236    9.94383    7.01881   0.4786
   2   6.106 MM    0.0586   14.01030    3.98387   0.6743
   3   6.292 MM    0.0690 2053.81860  496.00705  98.8471

Totals :                  2077.77274  507.00973
```


O
O
Cbz
N

20{*2,11*}


8
7
6
5
4
3
2
1
ppm
8.03
2.00
1.04
1.99
1.03
2.05
1.01
1.00
1.01
1.02
2.07
1.10

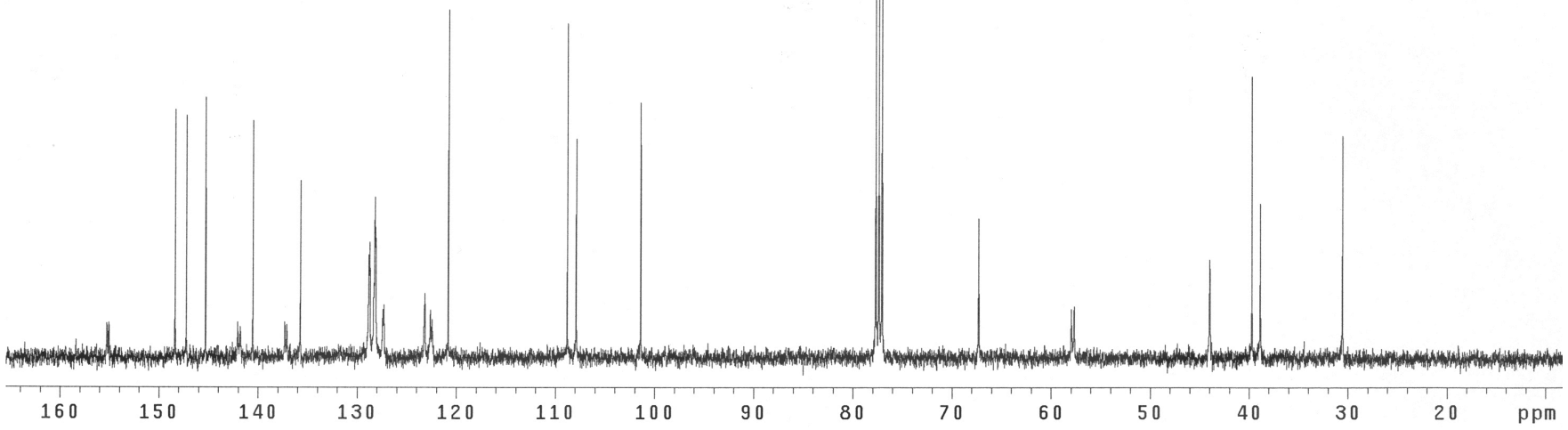


20{*2,11*}

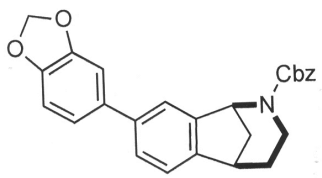
13C OBSERVE

exp1 std13c

SAMPLE		DEC. & VT	
date	May 27 2009	dfrq	400.269
solvent	CDCl3	dn	H1
file	exp	dpwr	38
ACQUISITION		dof	0
sfrq	100.658	dm	yyy
tn	C13	dmm	w
at	1.280	dmf	9300
np	64484	PROCESSING	
sw	25188.9	lb	1.00
fb	13800	wtfile	
bs	10	proc	ft
tpwr	60	fn	not used
pw	3.0		
d1	2.000	werr	
tof	1539.5	wexp	
nt	3000	wbs	
ct	610	wnt	
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	820.7		
wp	15833.0		
vs	198		
sc	0		
wc	250		
hzmm	63.33		
is	500.00		
rfl	1527.0		
rfp	0		
th	7		
ins	100.000		
nm cdc ph			

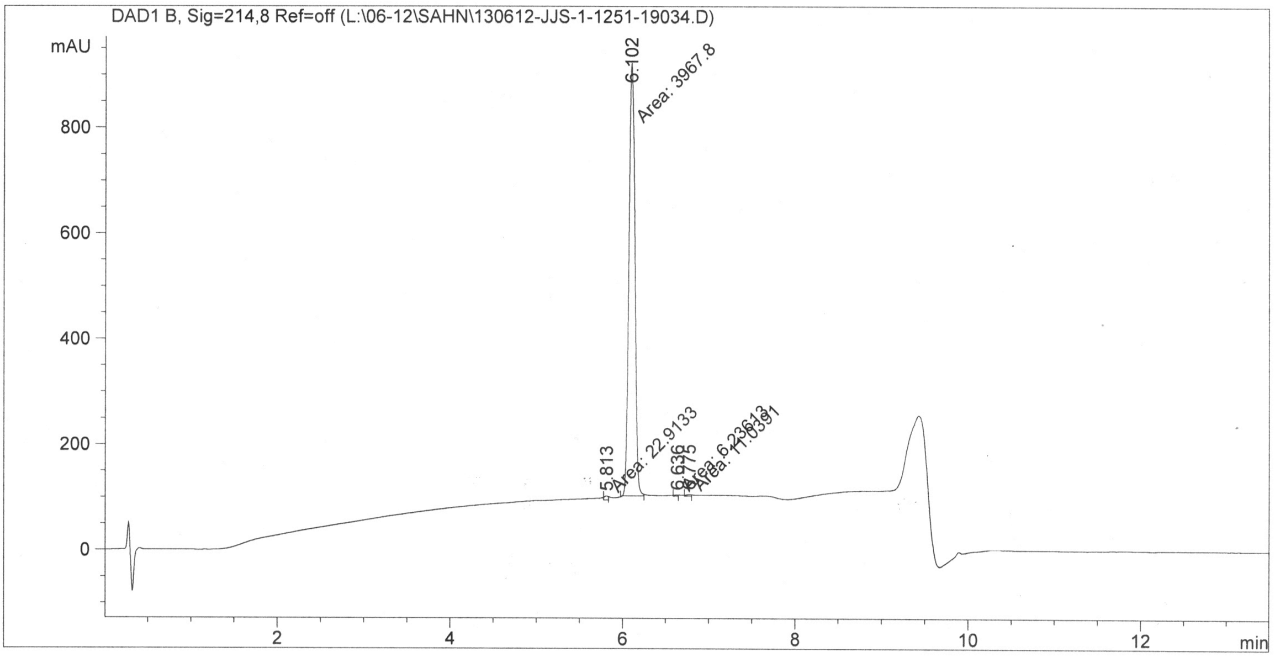


Data File L:\06-12\SAHN\130612-JJS-1-1251-19034.D
Sample Name: jjs-1-125

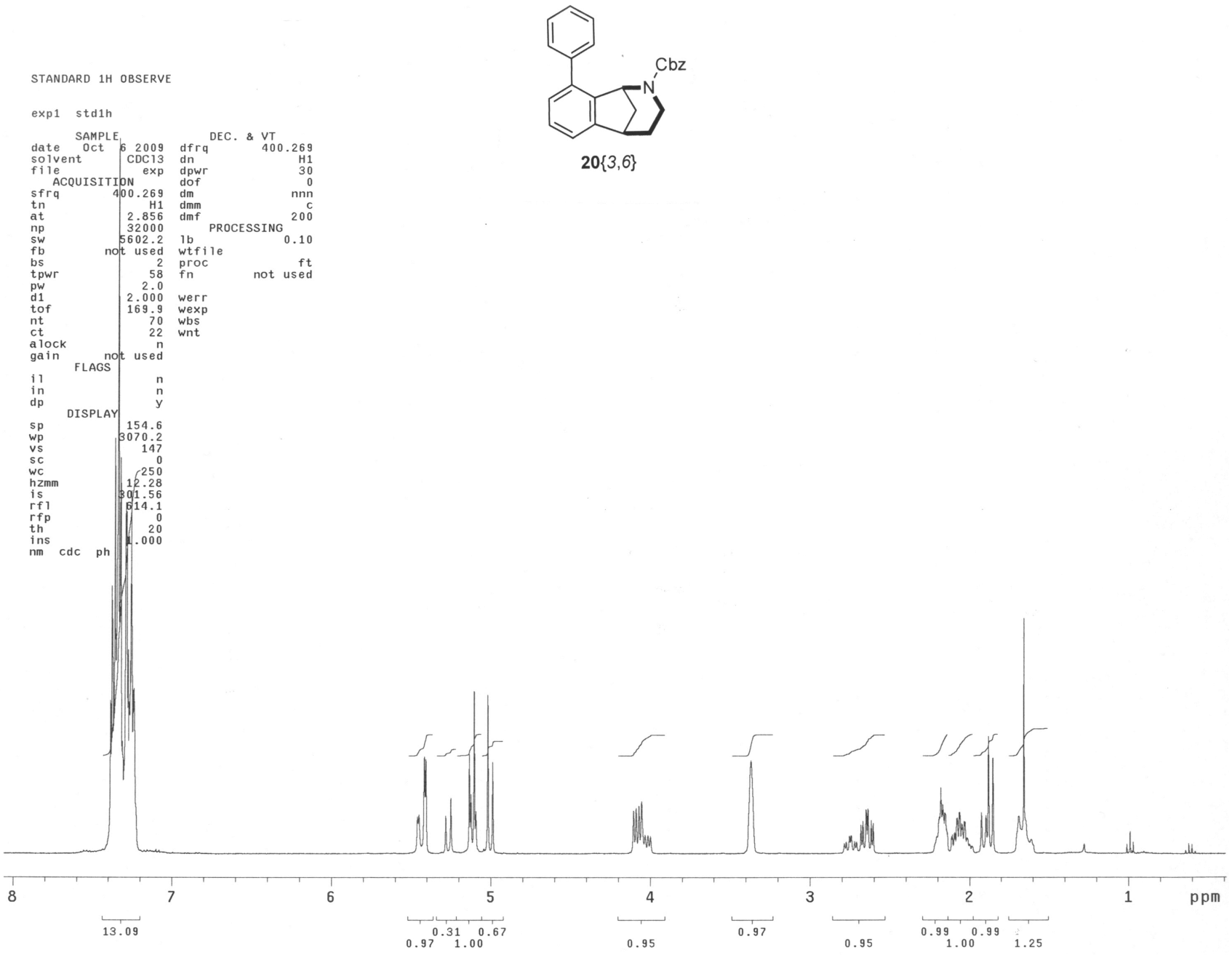


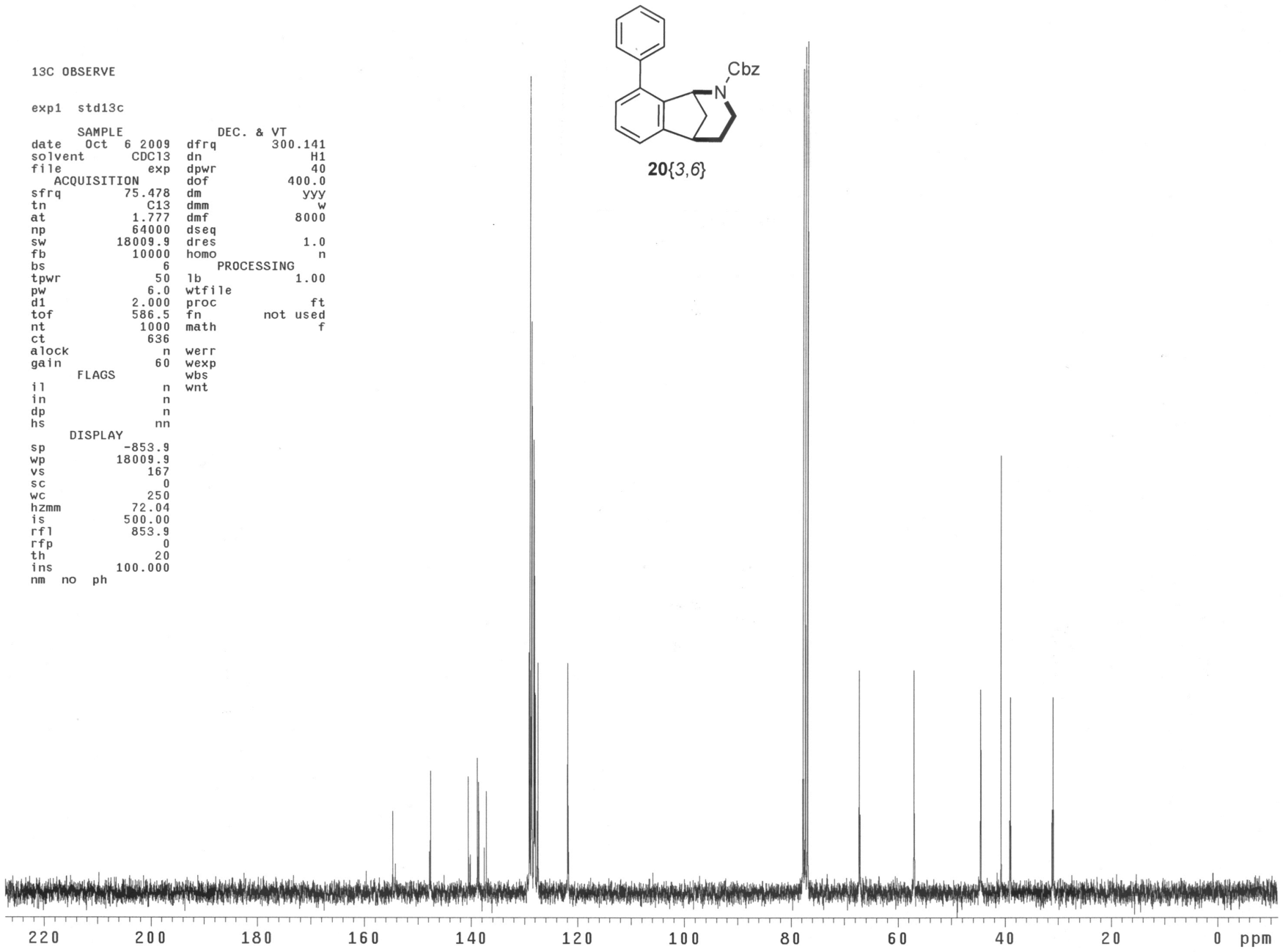
20{*2,11*}

```
================================================
Acq. Operator    : sahn@mail.utexas.edu
Acq. Instrument  : LCMS
Injection Date   : 6/13/2012 8:23:32 PM

Acq. Method      : C:\CHEM32\1\METHODS\SP NIH.M
Last changed     : 6/13/2012 8:23:18 PM by sahn@mail.utexas.edu
                   (modified after loading)
Analysis Method  : C:\CHEM32\1\METHODS\DEEF_LC.M
Last changed     : 9/28/2011 3:20:36 PM
Sample Info      : Easy-Access Method: 'SP NIH'
```

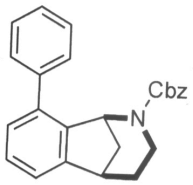



```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,8 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.813 MM    0.0534   22.91327    7.15006   0.5717
   2   6.102 MM    0.0804 3967.79663  822.65356  98.9973
   3   6.636 MM    0.0487    6.23613    2.13406   0.1556
   4   6.775 MM    0.0690   11.03907    2.66528   0.2754

Totals :                  4007.98510  834.60297
```


Cbz
N
20{3,6}
STANDARD 1H OBSERVE
exp1 std1h
SAMPLE
date Oct 6 2009
solvent CDCl3
file exp
ACQUISITION
sfrq 400.269
tn H1
at 2.856
np 32000
sw 5602.2
fb not used
bs 2
tpwr 58
pw 2.0
d1 2.000
tof 169.9
nt 70
ct 22
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp 154.6
wp 3070.2
vs 147
sc 0
wc 250
hzmm 12.28
is 301.56
rfl 614.1
rfp 0
th 20
ins 1.000
nm cdc ph
DEC. & VT
dfrq 400.269
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
lb 0.10
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
8
7
6
5
4
3
2
1
ppm
13.09
0.97
0.31
1.00
0.67
0.95
0.97
0.95
0.99
1.00
0.99
1.25

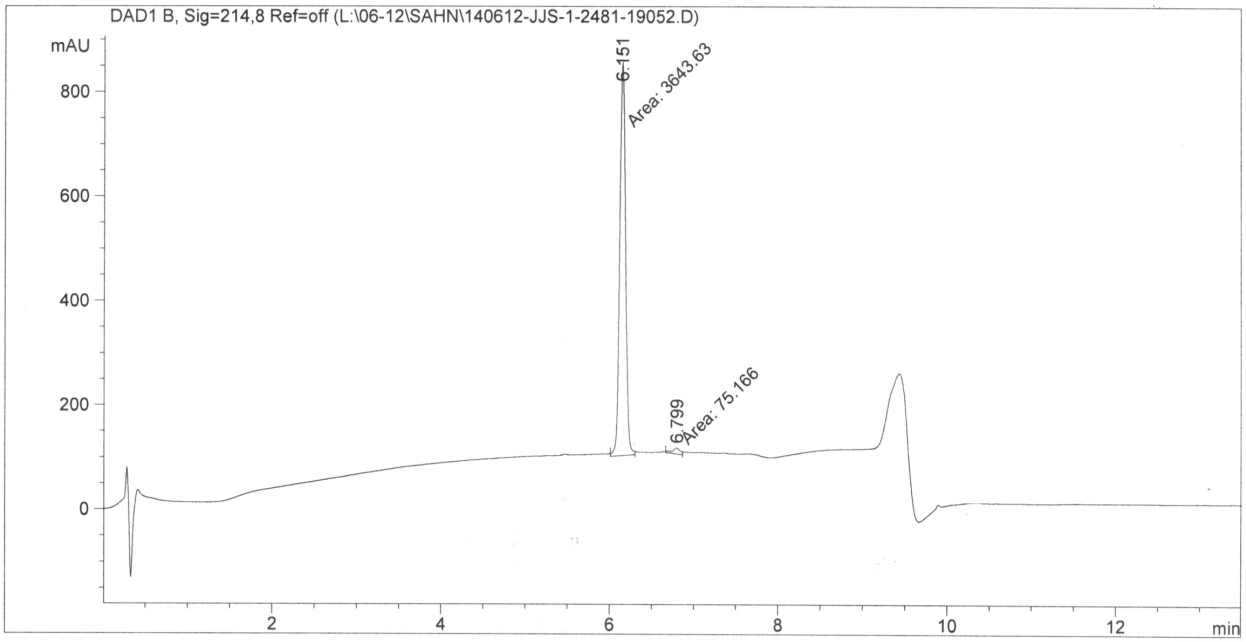

13C OBSERVE
exp1 std13c
SAMPLE
date Oct 6 2009
solvent CDCl3
file exp
ACQUISITION
sfrq 75.478
tn C13
at 1.777
np 64000
sw 18009.9
fb 10000
bs 6
tpwr 50
pw 6.0
d1 2.000
tof 586.5
nt 1000
ct 636
alock n
gain 60
FLAGS
il n
in n
dp n
hs nn
DISPLAY
sp -853.9
wp 18009.9
vs 167
sc 0
wc 250
hzmm 72.04
is 500.00
rfl 853.9
rfp 0
th 20
ins 100.000
nm no ph
DEC. & VT
dfrq 300.141
dn H1
dpwr 40
dof 400.0
dm yyy
dmm w
dmf 8000
dseq
dres 1.0
homo n
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
Cbz
N
20{3,6}
220
200
180
160
140
120
100
80
60
40
20
0
ppm

Data File L:\06-12\SAHN\140612-JJS-1-2481-19052.D
Sample Name: jjs-1-248

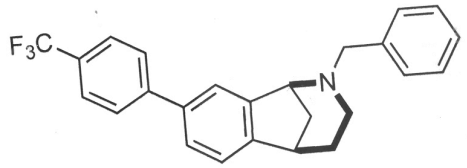


20{*3,6*}

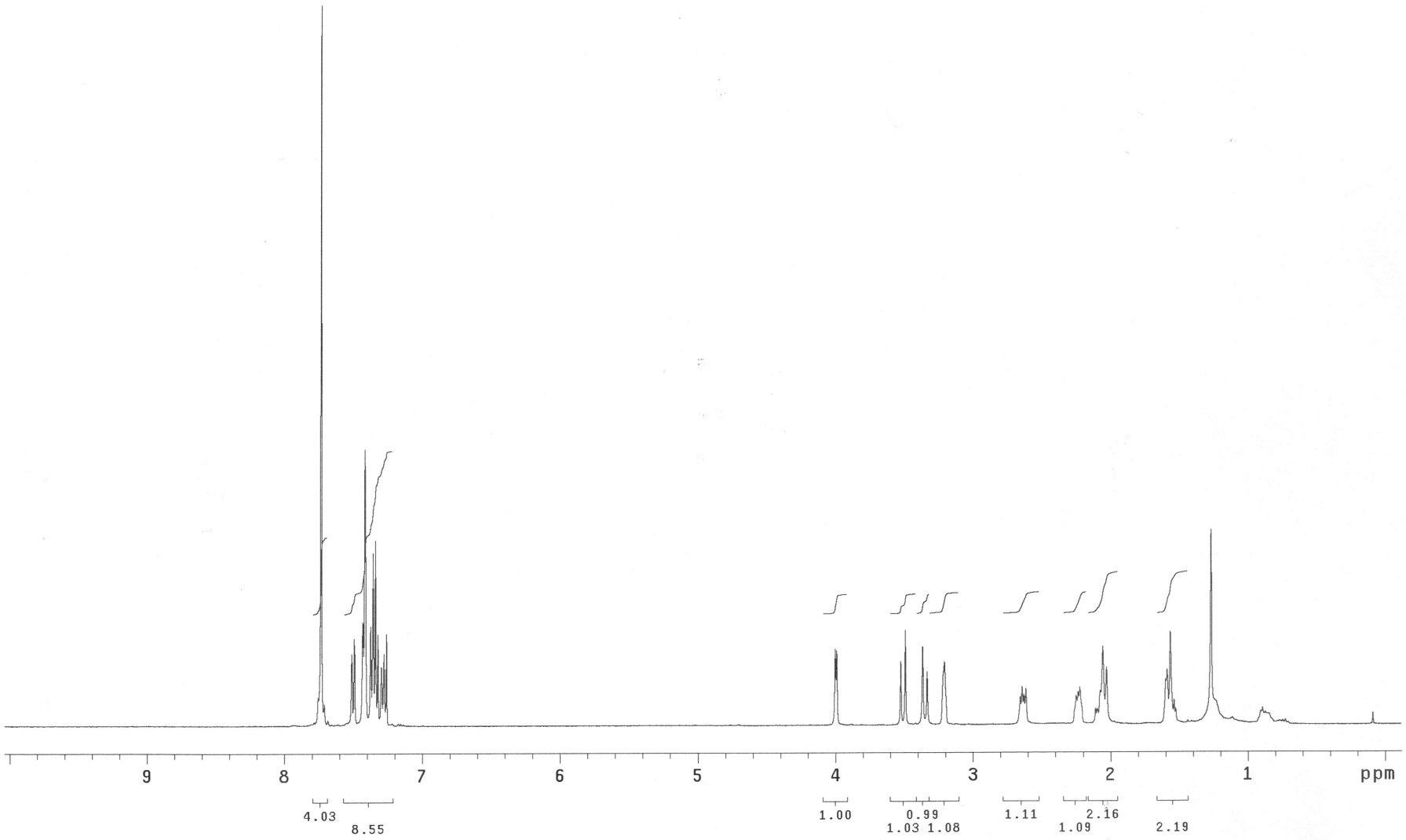
```
==========================================================================
Acq. Operator    : sahn@mail.utexas.edu
Acq. Instrument  : LCMS                              Location : Vial 8
Injection Date   : 6/14/2012 11:18:40 AM
                                                   Inj Volume : 1.0 µl
Acq. Method      : C:\CHEM32\1\METHODS\SP NIH.M
Last changed     : 6/14/2012 11:18:25 AM by sahn@mail.utexas.edu
                   (modified after loading)
Analysis Method  : C:\CHEM32\1\METHODS\DEEF_LC.M
Last changed     : 9/28/2011 3:20:36 PM
Sample Info      : Easy-Access Method: 'SP NIH'
```

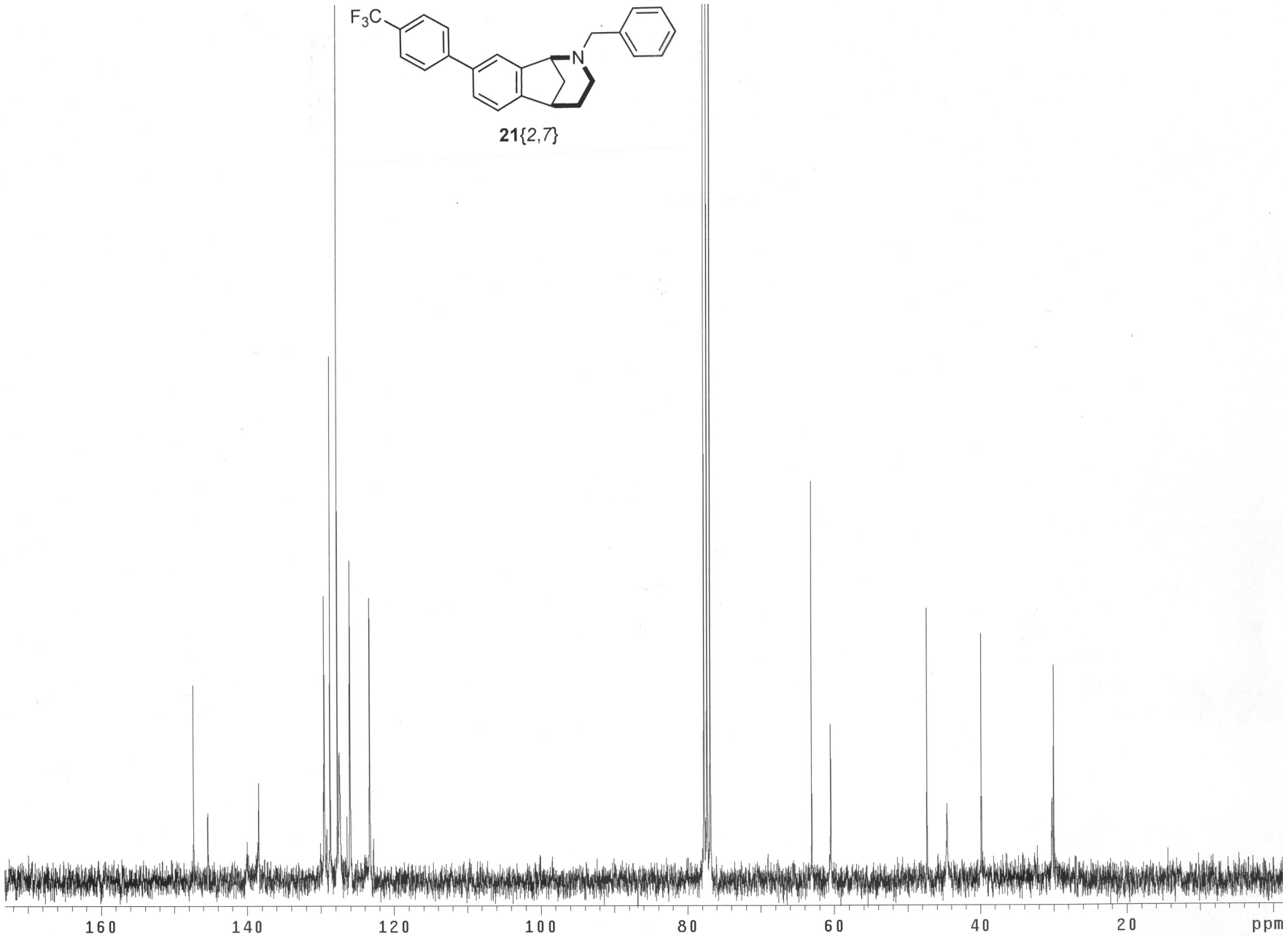



```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,8 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.151 MM    0.0804 3643.62866  755.43994  97.9788
   2   6.799 MM    0.1112   75.16598   11.26356   2.0212

Totals :                  3718.79464  766.70350
```

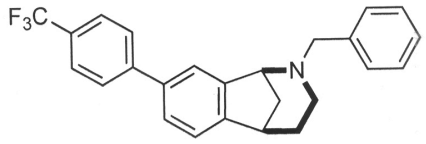


21{2,7}



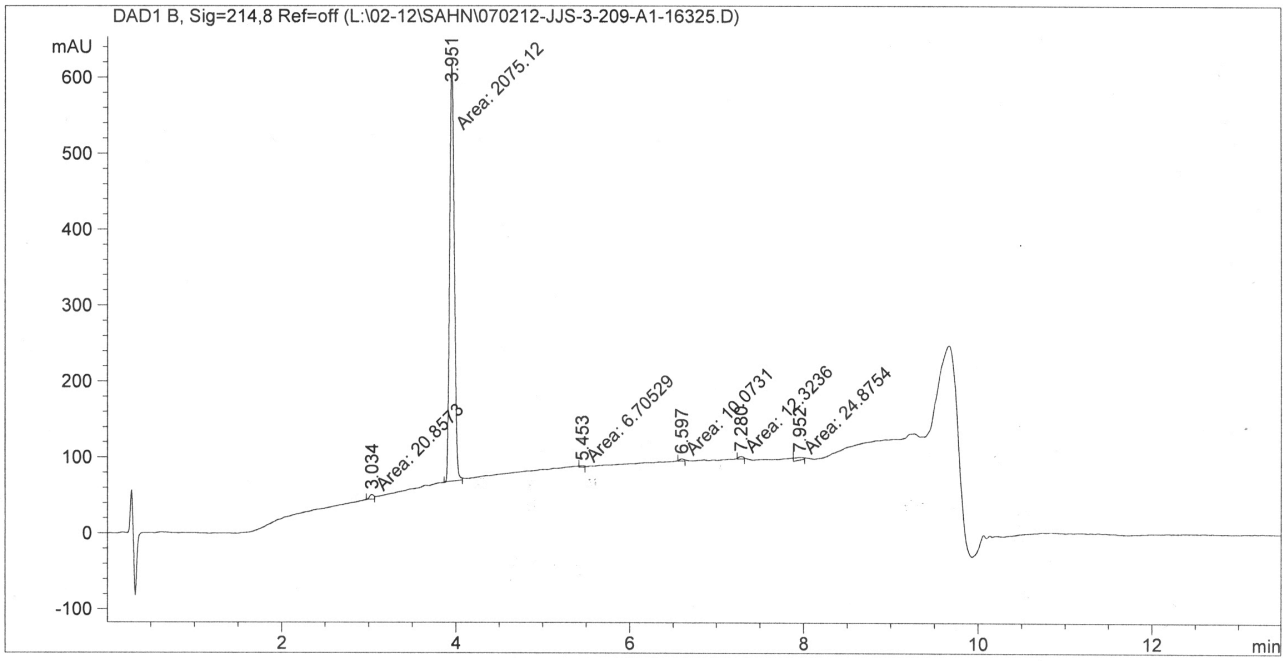

F3C
N
21{2,7}
160
140
120
100
80
60
40
20
ppm

Data File L:\02-12\SAHN\070212-JJS-3-209-A1-16325.D
Sample Name: JJS-3-209-A



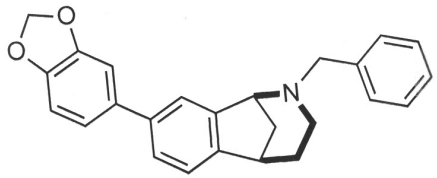
21{2,7}

```
==================================================
Acq. Operator    : sahn@mail.utexas.edu
Acq. Instrument  : LCMS
Injection Date   : 2/7/2012 3:50:35 PM

Acq. Method      : C:\CHEM32\1\METHODS\SP NIH.M
Last changed     : 2/7/2012 3:50:20 PM by sahn@mail.utexas.edu
                   (modified after loading)
Analysis Method  : C:\CHEM32\1\METHODS\DEEF_LC.M
Last changed     : 9/28/2011 3:20:36 PM
Sample Info      : Easy-Access Method: 'SP NIH'
```

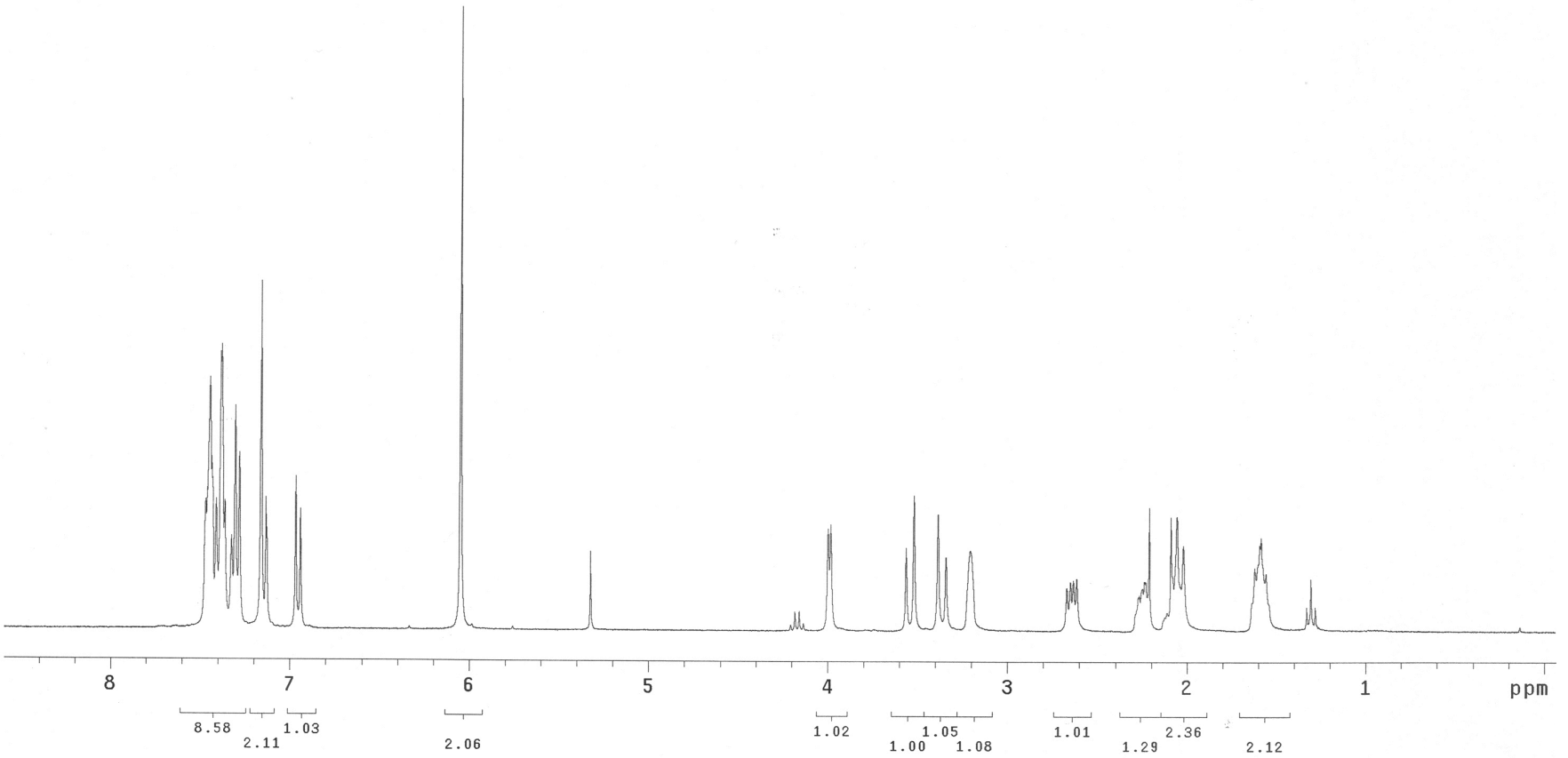


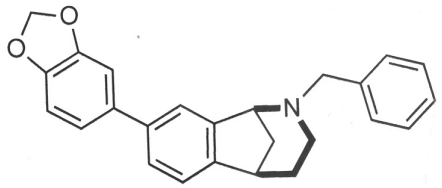

```
======================================================================
                        Area Percent Report
======================================================================

Sorted By                     Signal
Multiplier:                    :      1.0000
Dilution:                      :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,8 Ref=off
```

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.034	MM	0.0545	20.85730	6.37260	0.9701
2	3.951	MM	0.0623	2075.12036	555.51154	96.5192
3	5.453	MM	0.0610	6.70529	1.83278	0.3119
4	6.597	MM	0.0593	10.07314	2.83326	0.4685
5	7.280	MM	0.0615	12.32358	3.33878	0.5732
6	7.952	MM	0.0939	24.87543	3.55356	1.1570



21{2,11}





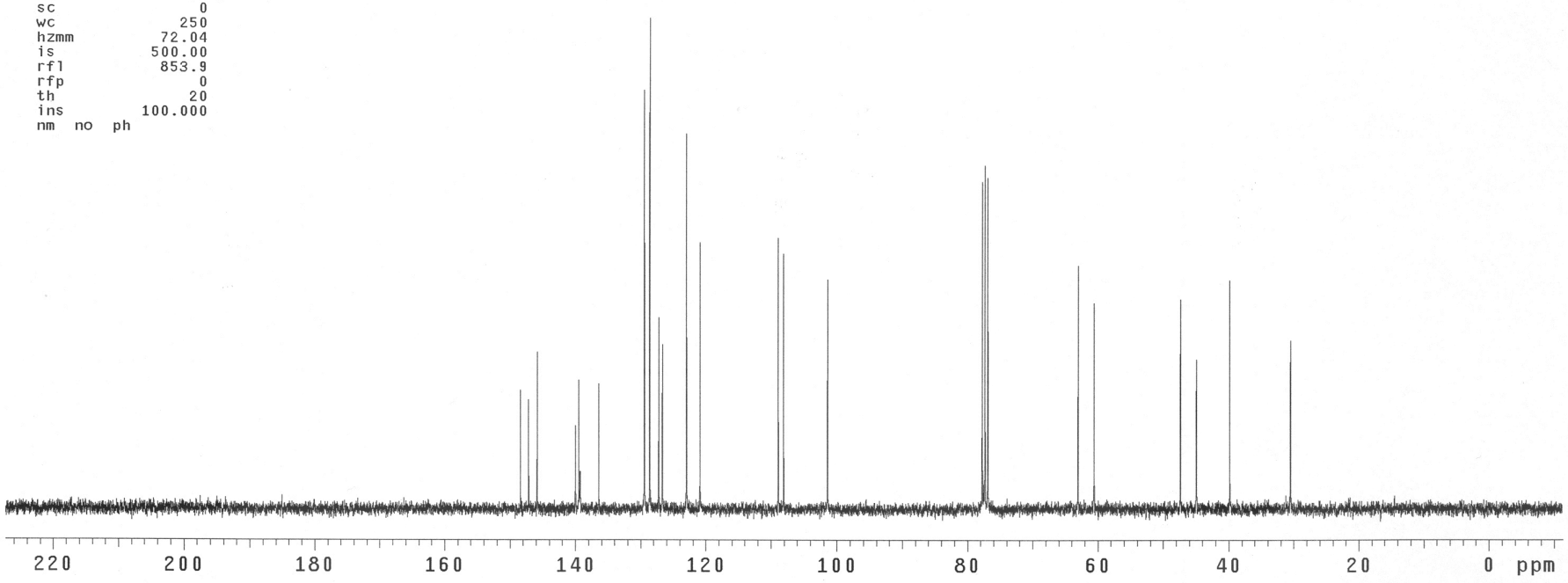
21{*2,11*}

13C OBSERVE

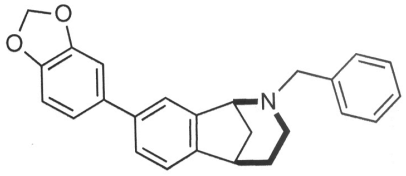
exp1 std13c

SAMPLE		DEC. & VT	
date	Jun 5 2009	dfrq	300.141
solvent	CDCl3	dn	H1
file	exp	dpwr	40
ACQUISITION		dof	400.0
sfrq	75.478	dm	yyy
tn	C13	dmm	w
at	1.777	dmf	8000
np	64000	dseq	
sw	18009.9	dres	1.0
fb	10000	homo	n
bs	16	PROCESSING	
tpwr	50	lb	1.00
pw	6.0	wtfile	
d1	2.000	proc	ft
tof	586.5	fn	not used
nt	1000	math	f
ct	448		
alock	n	werr	
gain	60	wexp	
FLAGS		wbs	
il	n	wnt	
in	n		
dp	n		
hs	nn		
DISPLAY			
sp	-853.9		
wp	18009.9		
vs	79		
sc	0		
wc	250		
hzmm	72.04		
is	500.00		
rfl	853.9		
rfp	0		
th	20		
ins	100.000		

nm no ph

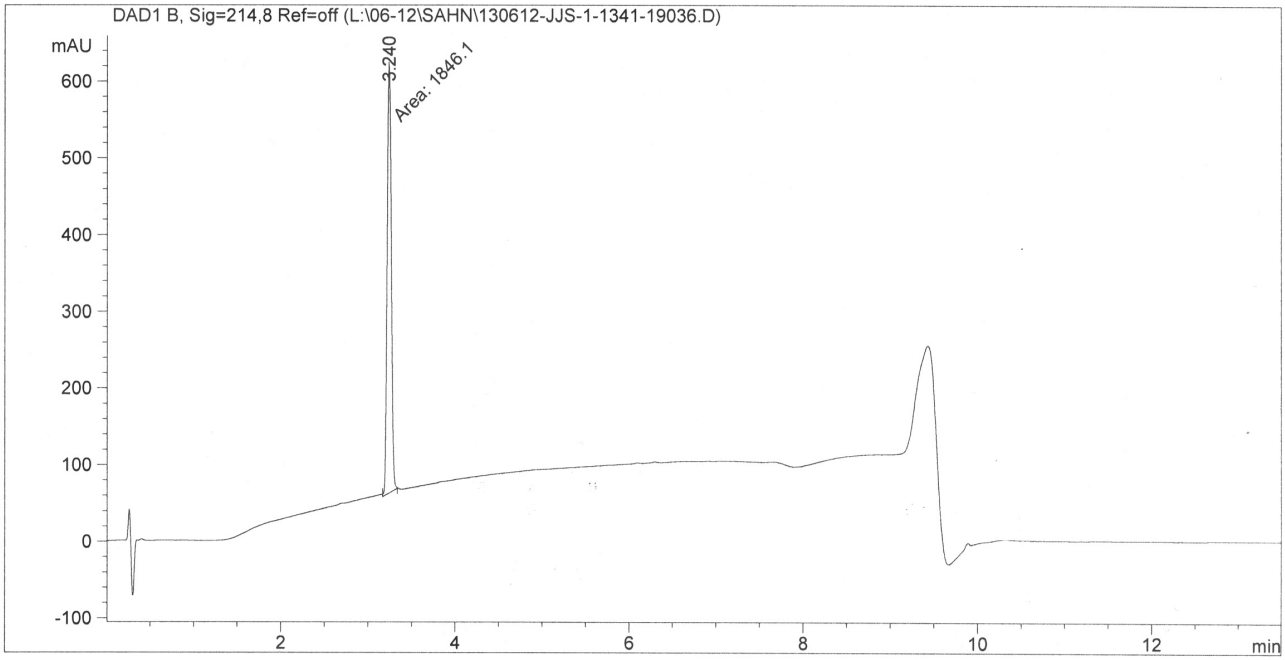


Data File L:\06-12\SAHN\130612-JJS-1-1341-19036.D
Sample Name: jjs-1-134

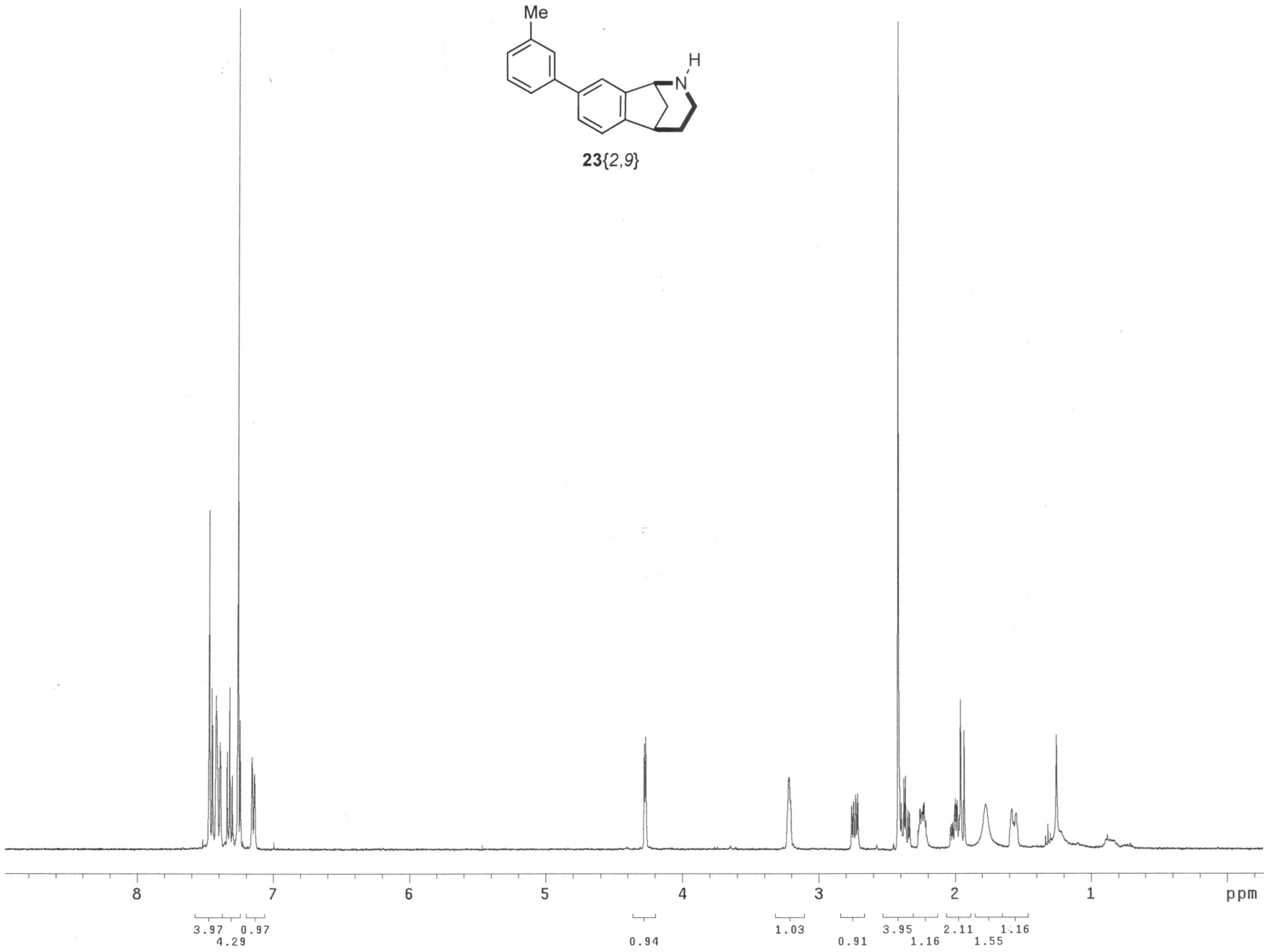


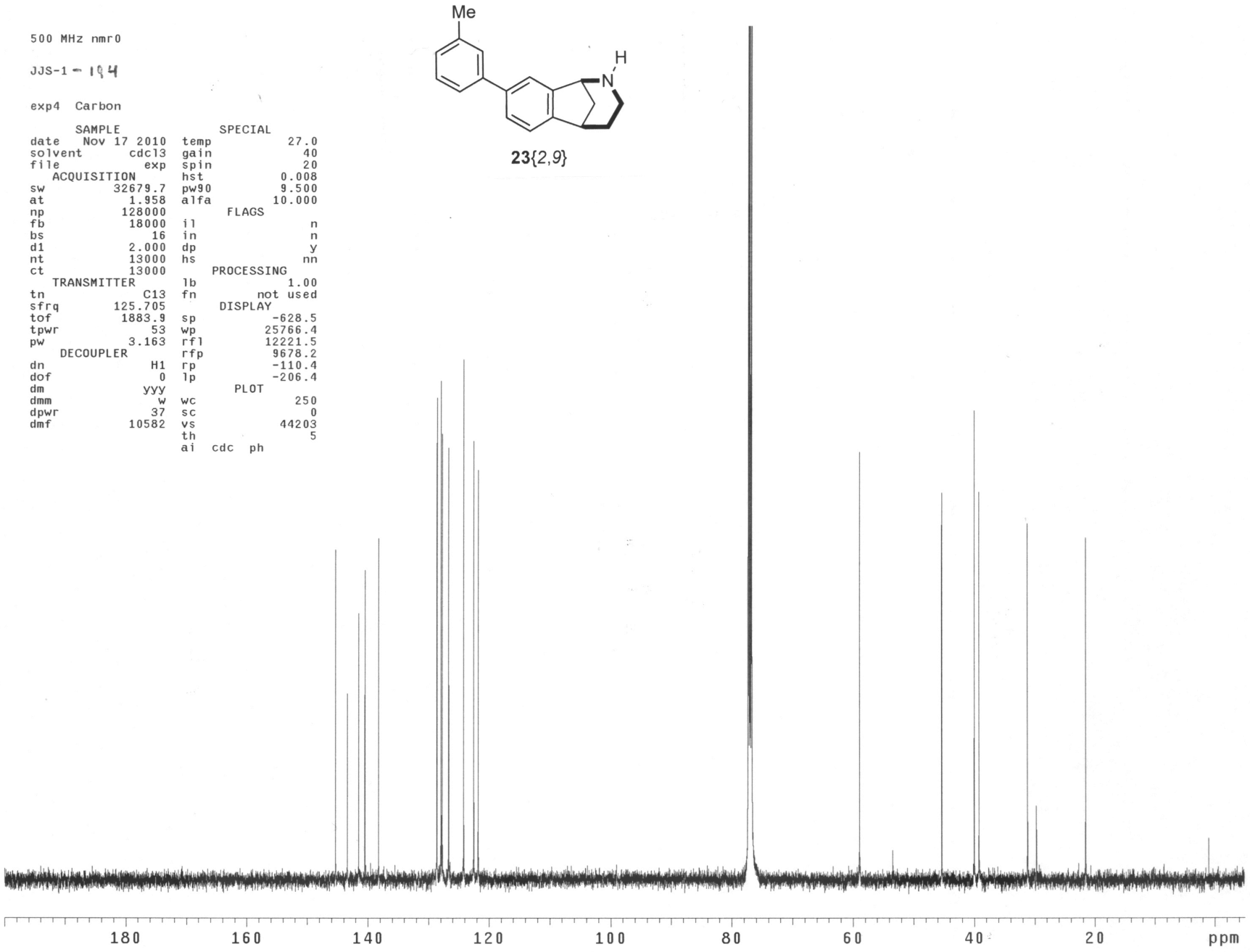
21{*2,11*}

```
==========================================================
Acq. Operator    : sahn@mail.utexas.edu
Acq. Instrument  : LCMS                          Location
Injection Date   : 6/13/2012 8:53:34 PM
                                                 Inj Volume
Acq. Method      : C:\CHEM32\1\METHODS\SP NIH.M
Last changed     : 6/13/2012 8:53:19 PM by sahn@mail.utexas
                   (modified after loading)
Analysis Method  : C:\CHEM32\1\METHODS\DEEF_LC.M
Last changed     : 9/28/2011 3:20:36 PM
Sample Info      : Easy-Access Method: 'SP NIH'
```

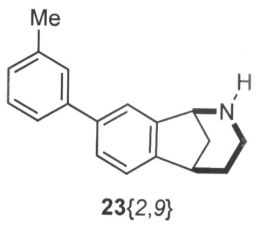



```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,8 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]    [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   3.240 MM    0.0542 1846.09924  567.89185 100.0000

Totals :                  1846.09924  567.89185

==========================================================================
```


Me
H
N
23{2,9}
8
7
6
5
4
3
2
1
ppm
3.97
0.97
4.29
0.94
1.03
0.91
3.95
1.16
2.11
1.55
1.16

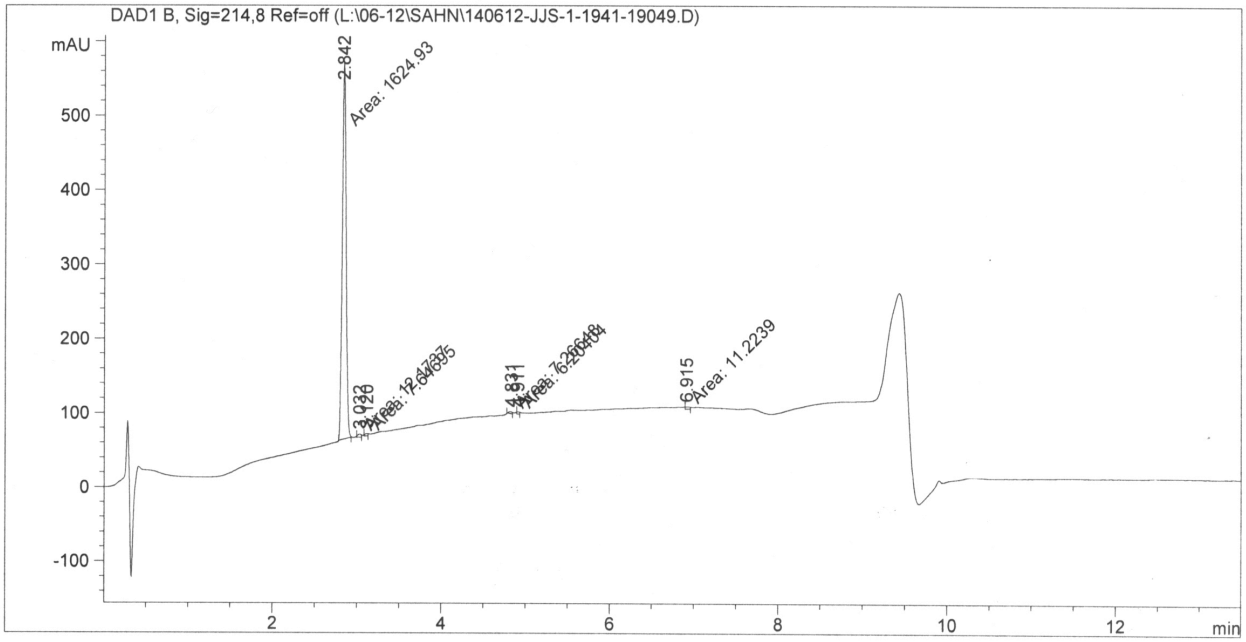

500 MHz nmr0
JJS-1-184
exp4 Carbon
SAMPLE
date Nov 17 2010
solvent cdcl3
file exp
ACQUISITION
sw 32679.7
at 1.958
np 128000
fb 18000
bs 16
d1 2.000
nt 13000
ct 13000
TRANSMITTER
tn C13
sfrq 125.705
tof 1883.9
tpwr 53
pw 3.163
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 37
dmf 10582
SPECIAL
temp 27.0
gain 40
spin 20
hst 0.008
pw90 9.500
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -628.5
wp 25766.4
rfl 12221.5
rfp 9678.2
rp -110.4
lp -206.4
PLOT
wc 250
sc 0
vs 44203
th 5
ai cdc ph
Me
H
N
23{2,9}
180
160
140
120
100
80
60
40
20
ppm

Data File L:\06-12\SAHN\140612-JJS-1-1941-19049.D
Sample Name: jjs-1-194

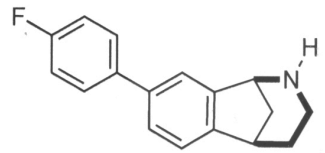


23{*2,9*}

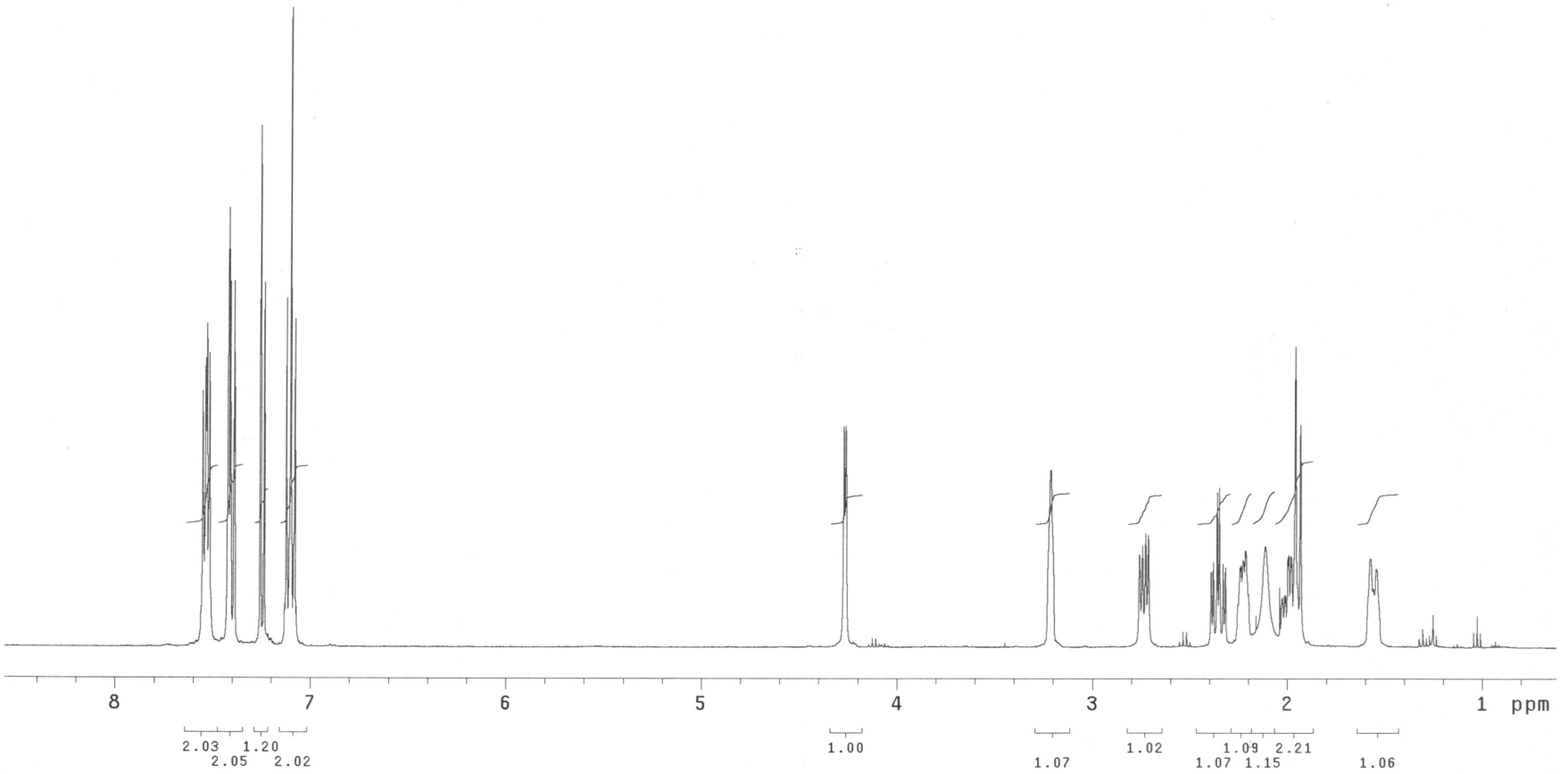
```
==========================================================================
Acq. Operator    : sahn@mail.utexas.edu
Acq. Instrument : LCMS                              Location : Vial 5
Injection Date   : 6/14/2012 10:28:59 AM
                                                   Inj Volume : 1.0 µl
Acq. Method      : C:\CHEM32\1\METHODS\SP NIH.M
Last changed     : 6/14/2012 10:28:44 AM by sahn@mail.utexas.edu
                   (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEEF_LC.M
Last changed     : 9/28/2011 3:20:36 PM
Sample Info      : Easy-Access Method: 'SP NIH'
```

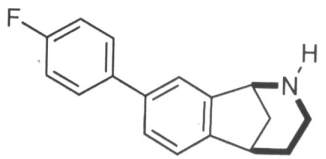



```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,8 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]      %
----|-------|----|-------|----------|----------|--------|
   1   2.842 MM    0.0528 1624.93042  512.66553  97.3335
   2   3.032 MM    0.0465   12.17374    4.36758   0.7292
   3   3.120 MM    0.0394    7.64695    3.23548   0.4581
   4   4.831 MM    0.0454    7.26648    2.66598   0.4353
   5   4.911 MM    0.0327    6.20404    3.16062   0.3716
   6   6.915 MM    0.0615   11.22390    3.04189   0.6723
```


F
H
N

23{*2,10*}


8
7
6
5
4
3
2
1 ppm
2.03
2.05
1.20
2.02
1.00
1.07
1.02
1.09
2.21
1.07
1.15
1.06



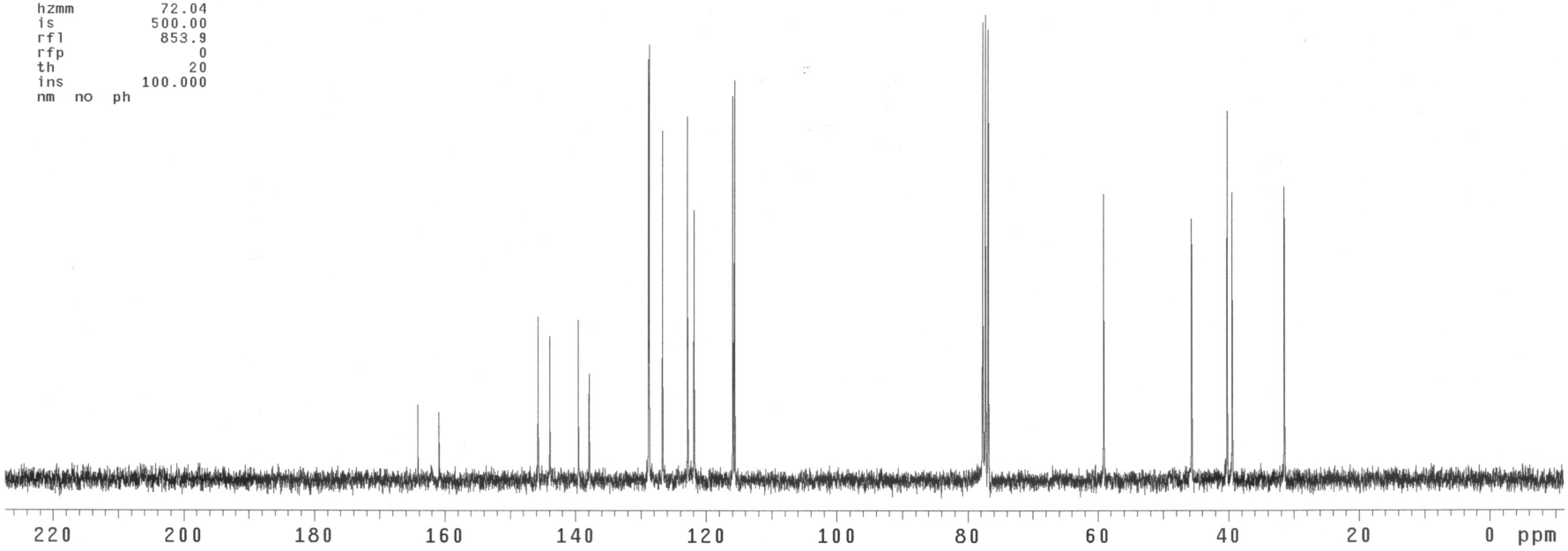
23{*2,10*}

13C OBSERVE

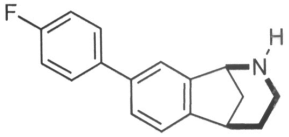
exp1 std13c

SAMPLE		DEC. & VT	
date	Jun 10 2009	dfrq	300.141
solvent	CDCl3	dn	H1
file	exp	dpwr	40
ACQUISITION		dof	400.0
sfrq	75.478	dm	yyy
tn	C13	dmm	w
at	1.777	dmf	8000
np	64000	dseq	
sw	18009.9	dres	1.0
fb	10000	homo	n
bs	16	PROCESSING	
tpwr	50	lb	1.00
pw	6.0	wtfile	
d1	2.000	proc	ft
tof	586.5	fn	not used
nt	1000	math	f
ct	672		
alock	n	werr	
gain	60	wexp	
FLAGS		wbs	
il	n	wnt	
in	n		
dp	n		
hs	nn		
DISPLAY			
sp	-853.9		
wp	18009.9		
vs	74		
sc	0		
wc	250		
hzmm	72.04		
is	500.00		
rfl	853.9		
rfp	0		
th	20		
ins	100.000		

nm no ph

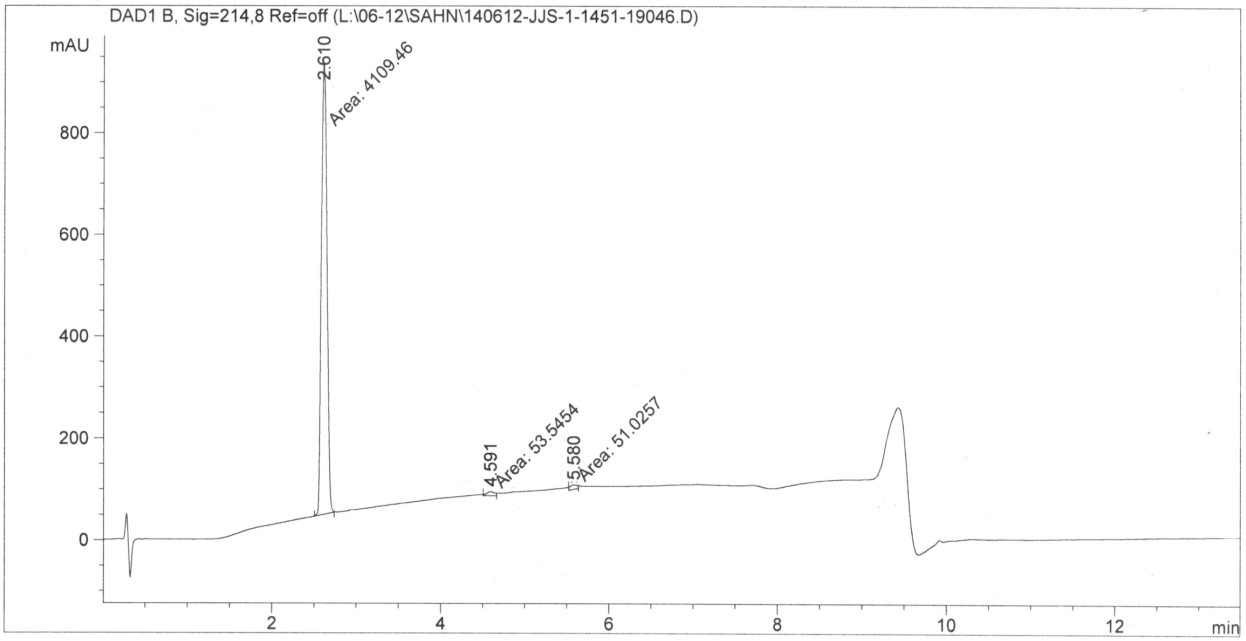


Data File L:\06-12\SAHN\140612-JJS-1-1451-19046.D
Sample Name: jjs-1-145

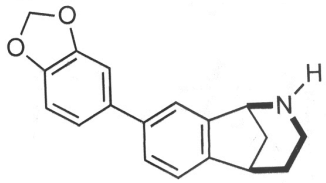


23{*2,10*}

```
Acq. Operator    : sahn@mail.utexas.edu
Acq. Instrument  : LCMS                          Location : Vial 2
Injection Date   : 6/14/2012 9:39:07 AM
                                                 Inj Volume : 1.0 µl
Acq. Method      : C:\CHEM32\1\METHODS\SP NIH.M
Last changed     : 6/14/2012 9:38:46 AM by sahn@mail.utexas.edu
                   (modified after loading)
Analysis Method  : C:\CHEM32\1\METHODS\DEEF_LC.M
Last changed     : 9/28/2011 3:20:36 PM
Sample Info      : Easy-Access Method: 'SP NIH'
```




```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,8 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   2.610 MM    0.0767 4109.46436  892.62555  97.5185
   2   4.591 MM    0.1078   53.54543    8.27777   1.2706
   3   5.580 MM    0.0966   51.02567    8.80549   1.2109

Totals :                  4214.03545  909.70882
```

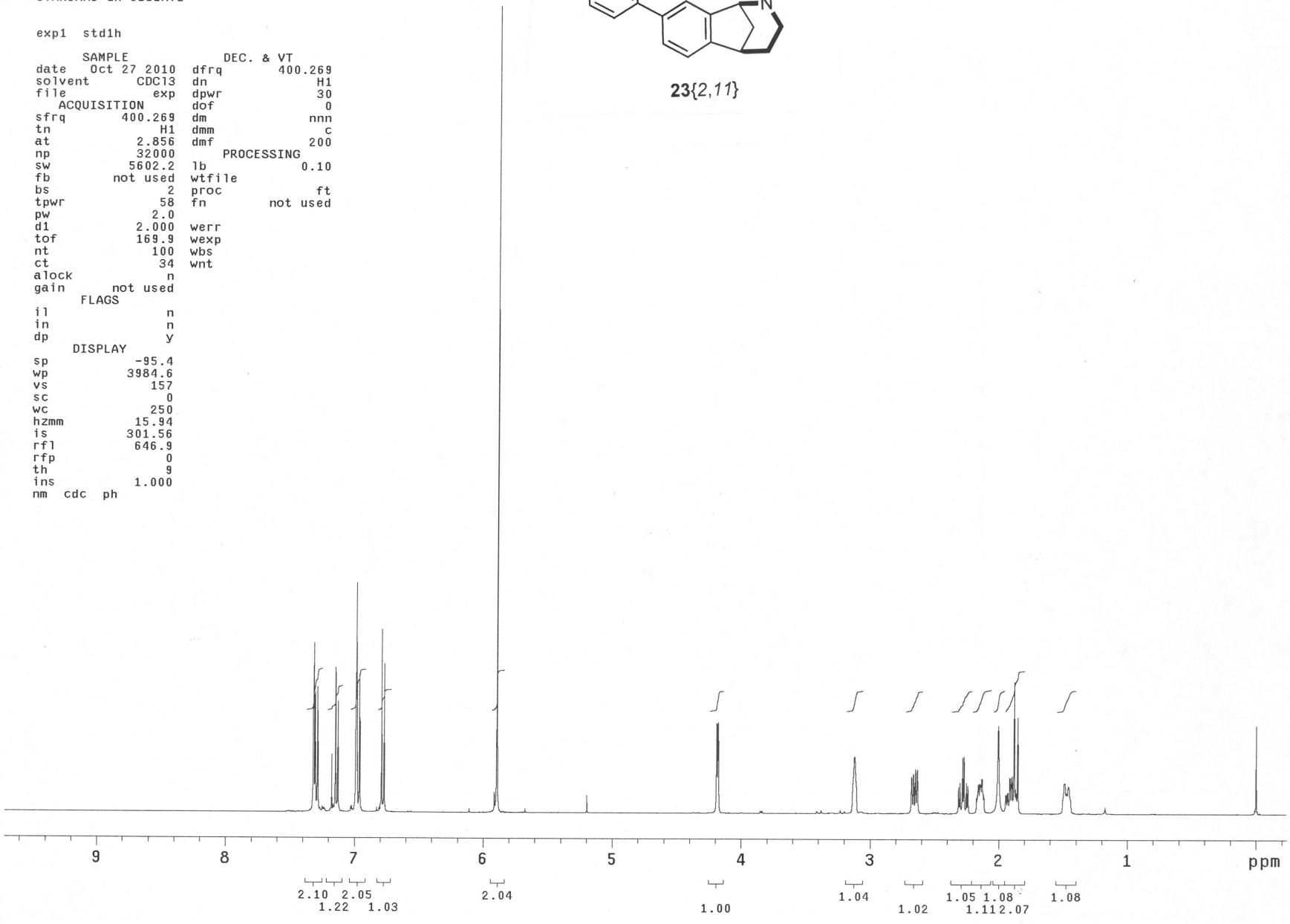


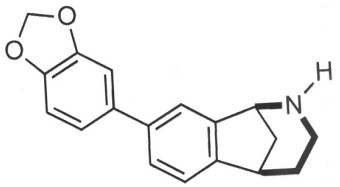
23{*2,11*}

STANDARD 1H OBSERVE

exp1 std1h

SAMPLE		DEC. & VT	
date	Oct 27 2010	dfrq	400.269
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	400.269	dm	nnn
tn	H1	dmm	c
at	2.856	dmf	200
np	32000	PROCESSING	
sw	5602.2	lb	0.10
fb	not used	wtfile	
bs	2	proc	ft
tpwr	58	fn	not used
pw	2.0		
d1	2.000	werr	
tof	169.9	wexp	
nt	100	wbs	
ct	34	wnt	
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-95.4		
wp	3984.6		
vs	157		
sc	0		
wc	250		
hzmm	15.94		
is	301.56		
rfl	646.9		
rfp	0		
th	9		
ins	1.000		
nm cdc ph			





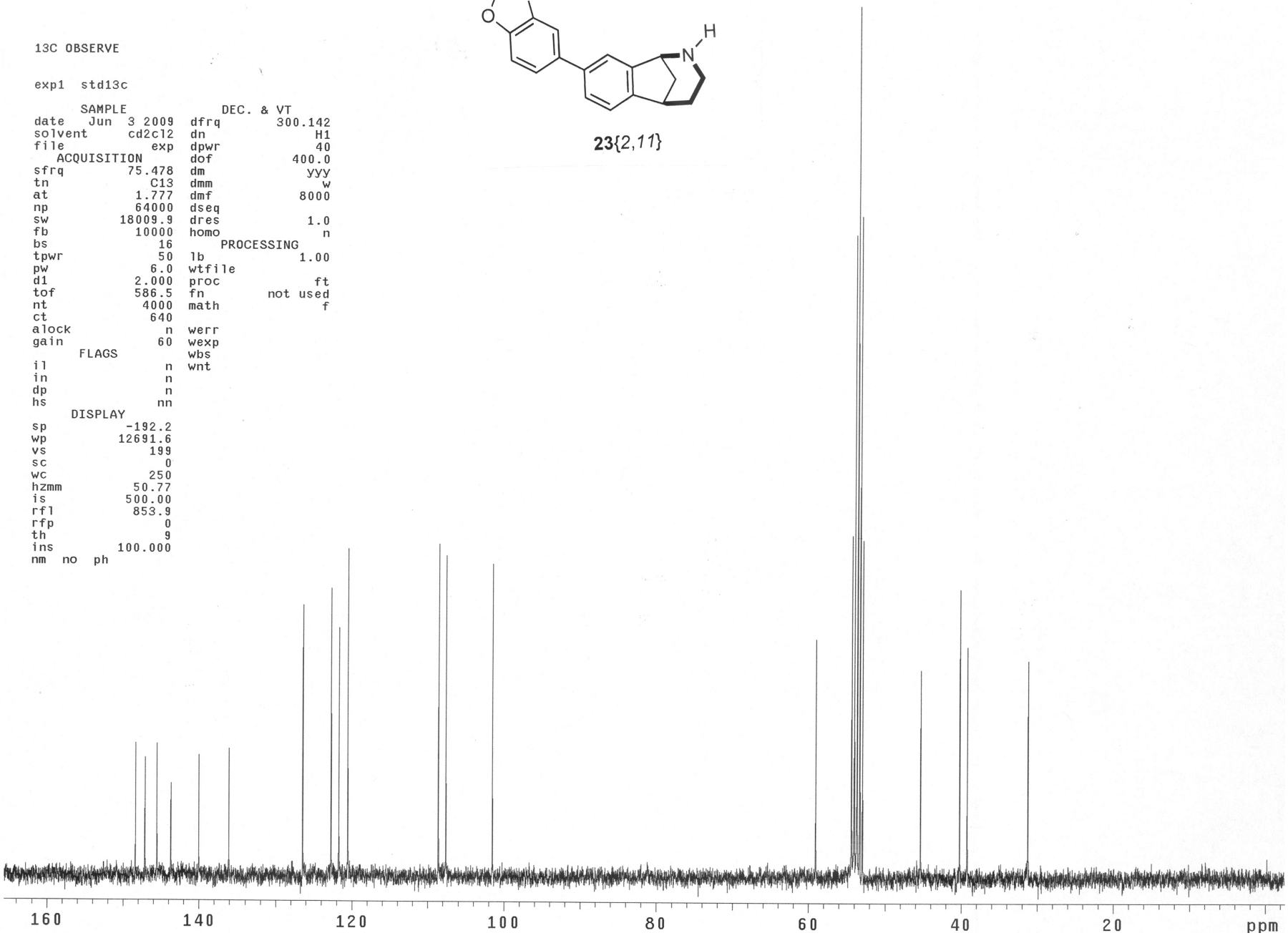
23{*2,11*}

13C OBSERVE

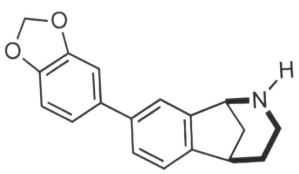
exp1 std13c

SAMPLE		DEC. & VT	
date	Jun 3 2009	dfrq	300.142
solvent	cd2cl2	dn	H1
file	exp	dpwr	40
ACQUISITION		dof	400.0
sfrq	75.478	dm	yyy
tn	C13	dmm	w
at	1.777	dmf	8000
np	64000	dseq	
sw	18009.9	dres	1.0
fb	10000	homo	n
bs	16	PROCESSING	
tpwr	50	lb	1.00
pw	6.0	wtfile	
d1	2.000	proc	ft
tof	586.5	fn	not used
nt	4000	math	f
ct	640		
alock	n	werr	
gain	60	wexp	
FLAGS		wbs	
il	n	wnt	
in	n		
dp	n		
hs	nn		
DISPLAY			
sp	-192.2		
wp	12691.6		
vs	199		
sc	0		
wc	250		
hzmm	50.77		
is	500.00		
rfl	853.9		
rfp	0		
th	9		
ins	100.000		

nm no ph

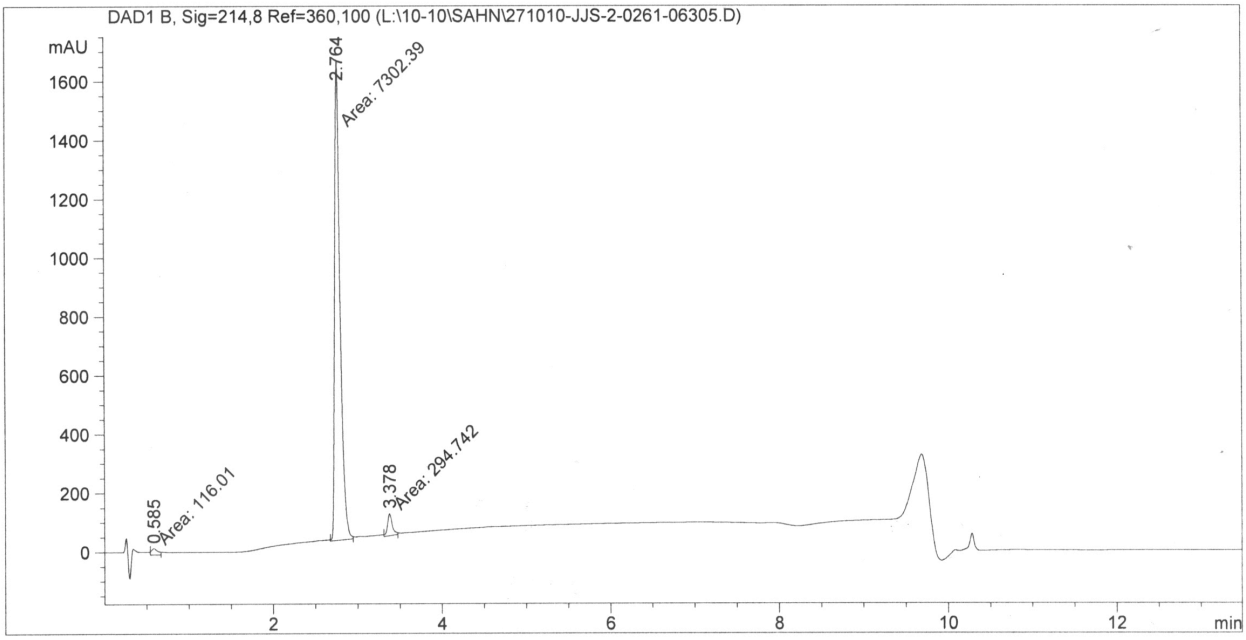


Data File L:\10-10\SAHN\271010-JJS-2-0261-06305.D
Sample Name: jjs-2-026

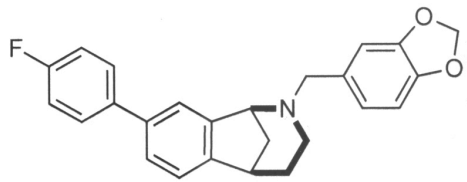


23{*2,11*}

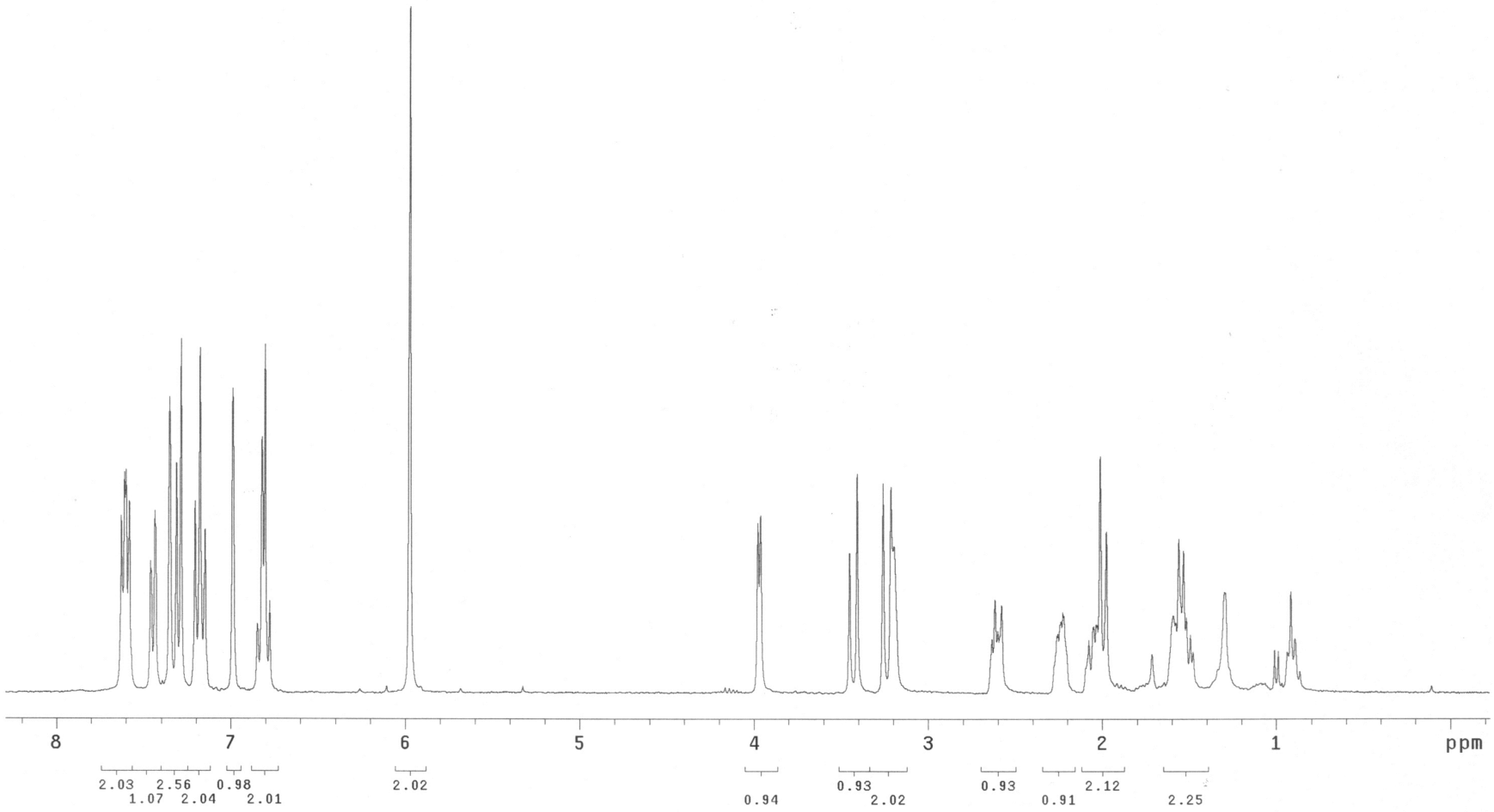
```
================================================
Acq. Operator    : sahn@mail.utexas.edu
Acq. Instrument  : LCMS
Injection Date   : 10/27/2010 4:31:56 PM

Acq. Method      : C:\CHEM32\1\METHODS\SP NIH.M
Last changed     : 10/27/2010 4:31:40 PM by sahn@mail.utexas.edu
                   (modified after loading)
Analysis Method  : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed     : 11/20/2006 4:14:44 AM
Sample Info      : Easy-Access Method: 'SP NIH'
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   0.585 MM    0.0895  116.00992   21.60857   1.5041
   2   2.764 MM    0.0741 7302.39258 1641.52917  94.6747
   3   3.378 MM    0.0652  294.74188   75.31119   3.8213

Totals :                  7713.14438 1738.44893
```


F
N
O
O

26{*2,10,3*}

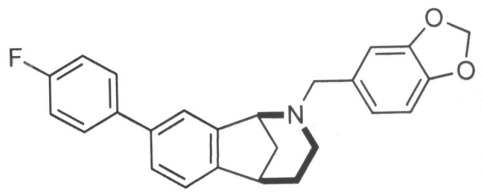

8
7
6
5
4
3
2
1
ppm
2.03
2.56
0.98
1.07
2.04
2.01
2.02
0.94
0.93
2.02
0.93
0.91
2.12
2.25

13C OBSERVE

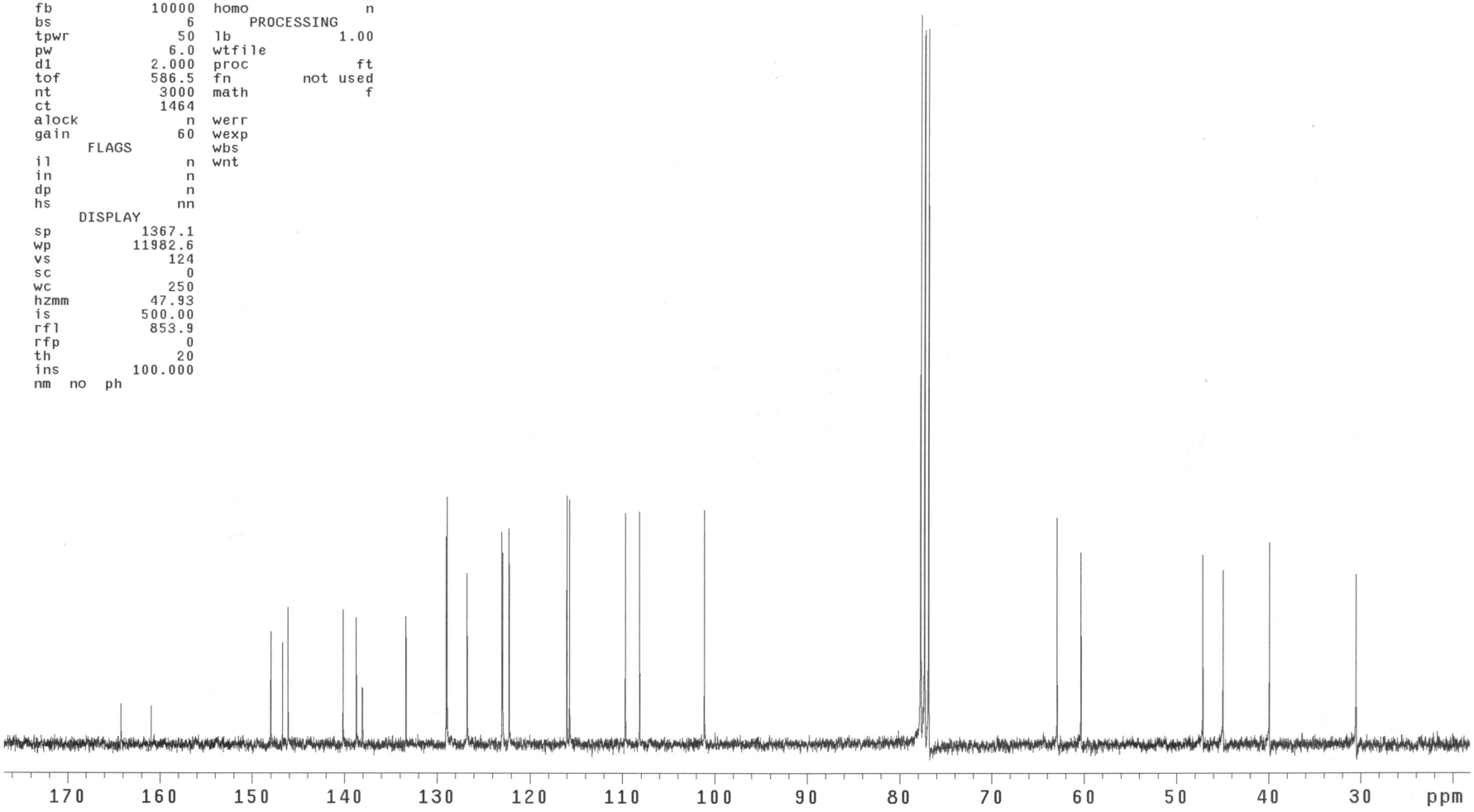
exp1 std13c

SAMPLE		DEC. & VT	
date	Jul 19 2009	dfrq	300.141
solvent	CDCl3	dn	H1
file	exp	dpwr	40
ACQUISITION		dof	400.0
sfrq	75.478	dm	yyy
tn	C13	dmm	w
at	1.777	dmf	8000
np	64000	dseq	
sw	18009.9	dres	1.0
fb	10000	homo	n
bs	6	PROCESSING	
tpwr	50	lb	1.00
pw	6.0	wtfile	
d1	2.000	proc	ft
tof	586.5	fn	not used
nt	3000	math	f
ct	1464		
alock	n	werr	
gain	60	wexp	
FLAGS		wbs	
il	n	wnt	
in	n		
dp	n		
hs	nn		
DISPLAY			
sp	1367.1		
wp	11982.6		
vs	124		
sc	0		
wc	250		
hzmm	47.93		
is	500.00		
rfl	853.9		
rfp	0		
th	20		
ins	100.000		

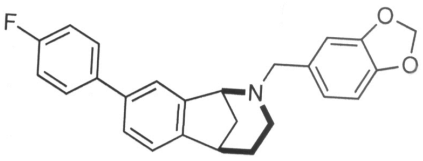
nm no ph



26{*2,10,3*}

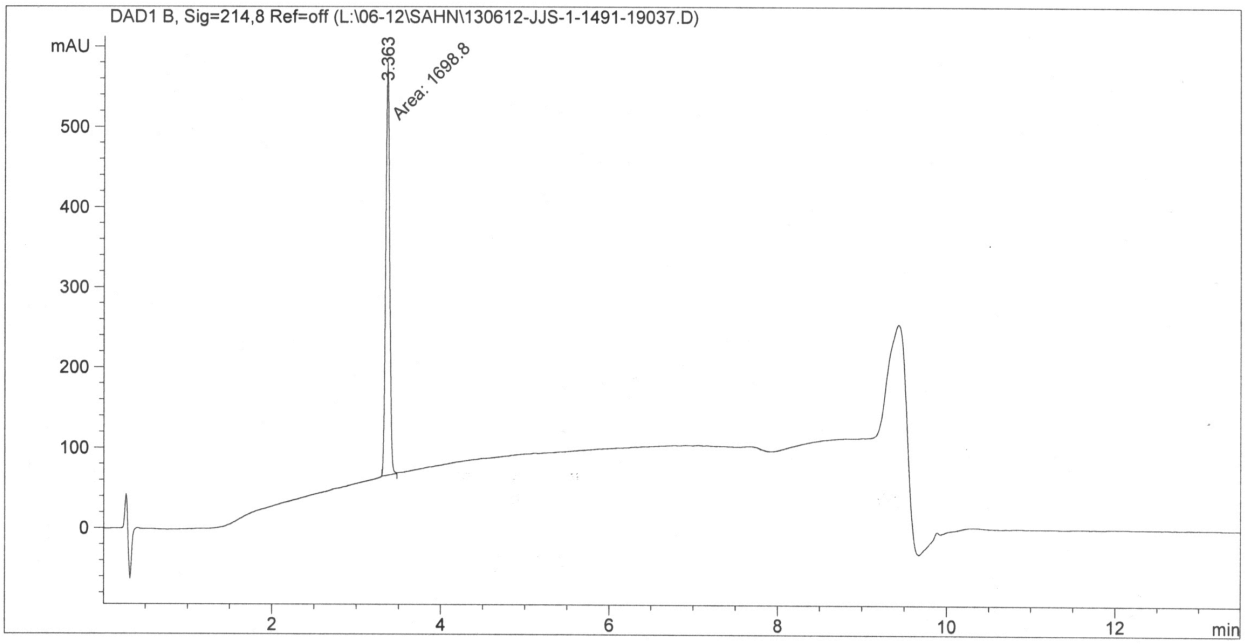


Data File L:\06-12\SAHN\130612-JJS-1-1491-19037.D
Sample Name: jjs-1-149



26{*2,10,3*}

```
==========================================================
Acq. Operator    : sahn@mail.utexas.edu
Acq. Instrument  : LCMS                              Locatio
Injection Date   : 6/13/2012 9:08:33 PM
                                                     Inj Volum
Acq. Method      : C:\CHEM32\1\METHODS\SP NIH.M
Last changed     : 6/13/2012 9:08:19 PM by sahn@mail.
                   (modified after loading)
Analysis Method  : C:\CHEM32\1\METHODS\DEEF_LC.M
Last changed     : 6/13/2012 3:34:08 PM
                   (modified after loading)
Sample Info      : Easy-Access Method: 'SP NIH'
```

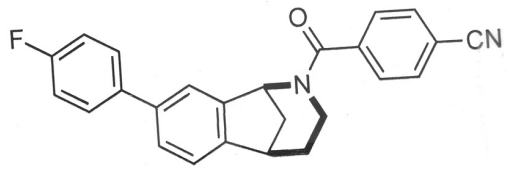



```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,8 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   3.363 MM    0.0547 1698.80042  517.54065 100.0000

Totals :                  1698.80042  517.54065
```

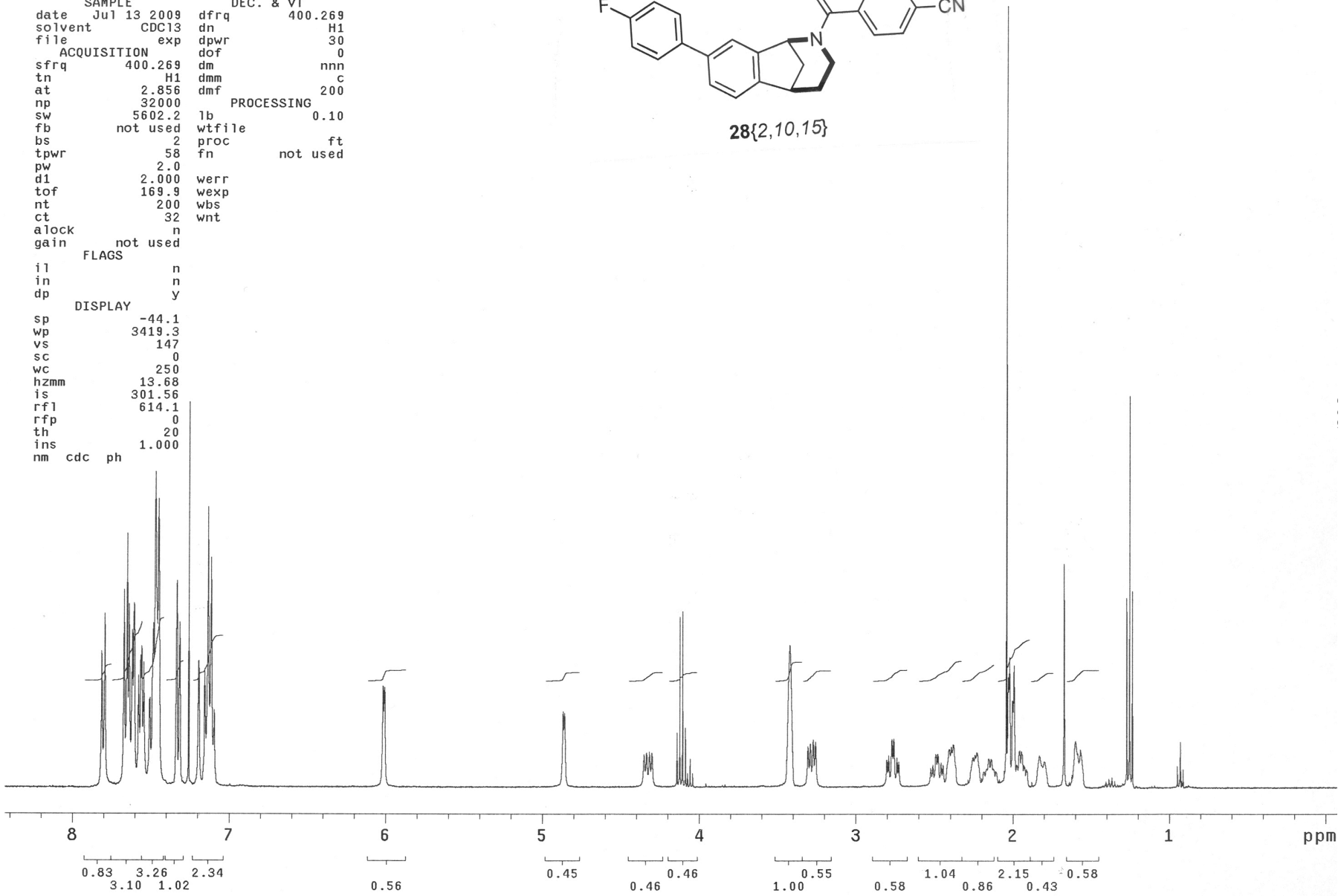
STANDARD 1H OBSERVE

exp1 std1h

SAMPLE		DEC. & VT	
date	Jul 13 2009	dfrq	400.269
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	400.269	dm	nnn
tn	H1	dmm	c
at	2.856	dmf	200
np	32000	PROCESSING	
sw	5602.2	lb	0.10
fb	not used	wtfile	
bs	2	proc	ft
tpwr	58	fn	not used
pw	2.0		
d1	2.000	werr	
tof	169.9	wexp	
nt	200	wbs	
ct	32	wnt	
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-44.1		
wp	3419.3		
vs	147		
sc	0		
wc	250		
hzmm	13.68		
is	301.56		
rfl	614.1		
rfp	0		
th	20		
ins	1.000		
nm cdc ph			



28{2,10,15}

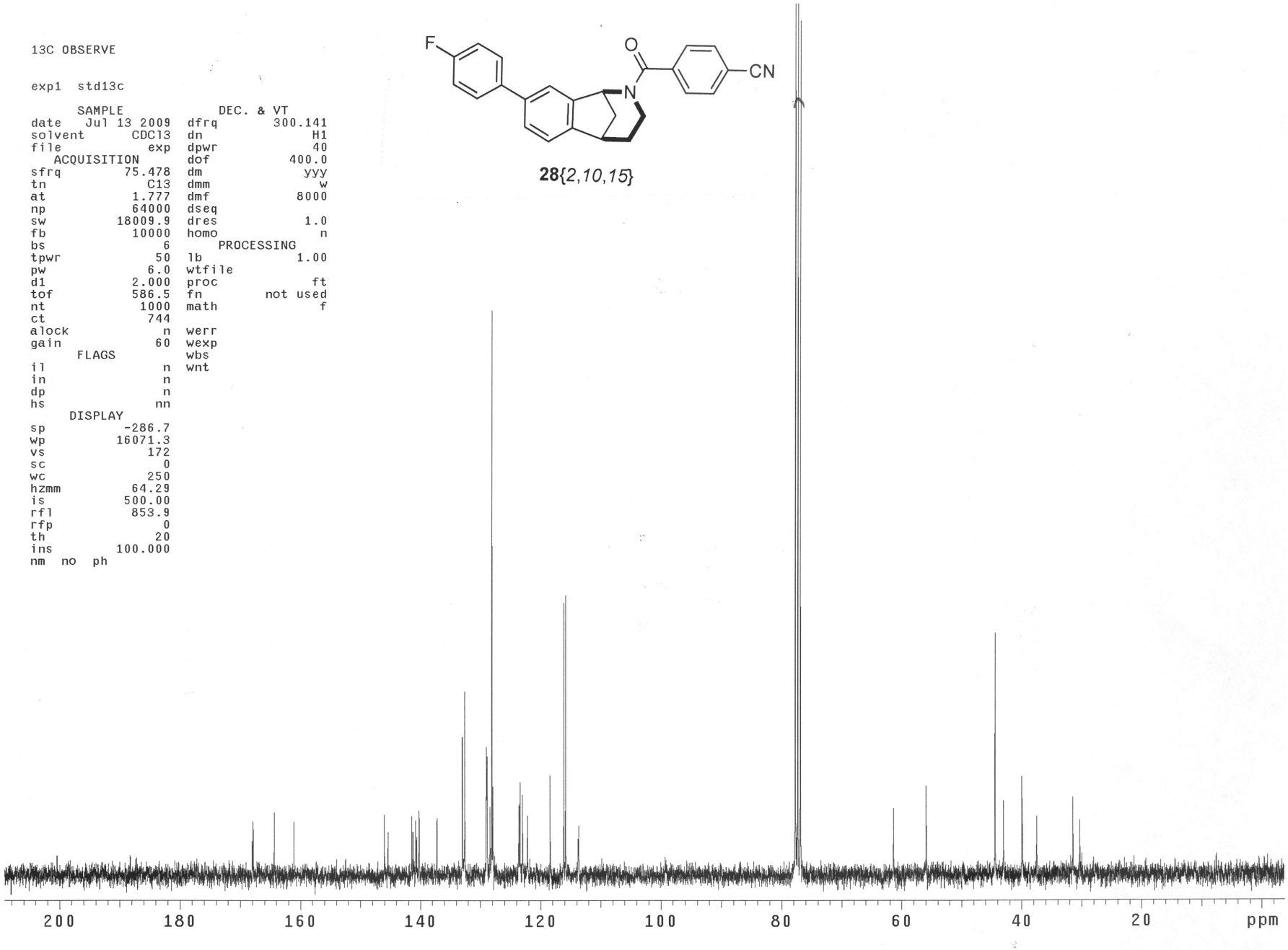


13C OBSERVE

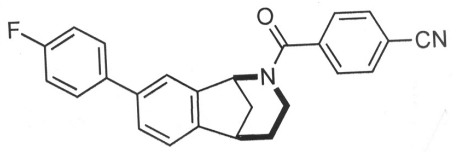
exp1 std13c

SAMPLE		DEC. & VT	
date	Jul 13 2009	dfrq	300.141
solvent	CDCl3	dn	H1
file	exp	dpwr	40
ACQUISITION		dof	400.0
sfrq	75.478	dm	yyy
tn	C13	dmm	w
at	1.777	dmf	8000
np	64000	dseq	
sw	18009.9	dres	1.0
fb	10000	homo	n
bs	6	PROCESSING	
tpwr	50	lb	1.00
pw	6.0	wtfile	
d1	2.000	proc	ft
tof	586.5	fn	not used
nt	1000	math	f
ct	744		
alock	n	werr	
gain	60	wexp	
FLAGS		wbs	
il	n	wnt	
in	n		
dp	n		
hs	nn		
DISPLAY			
sp	-286.7		
wp	16071.3		
vs	172		
sc	0		
wc	250		
hzmm	64.29		
is	500.00		
rfl	853.9		
rfp	0		
th	20		
ins	100.000		
nm no ph			

28{*2,10,15*}

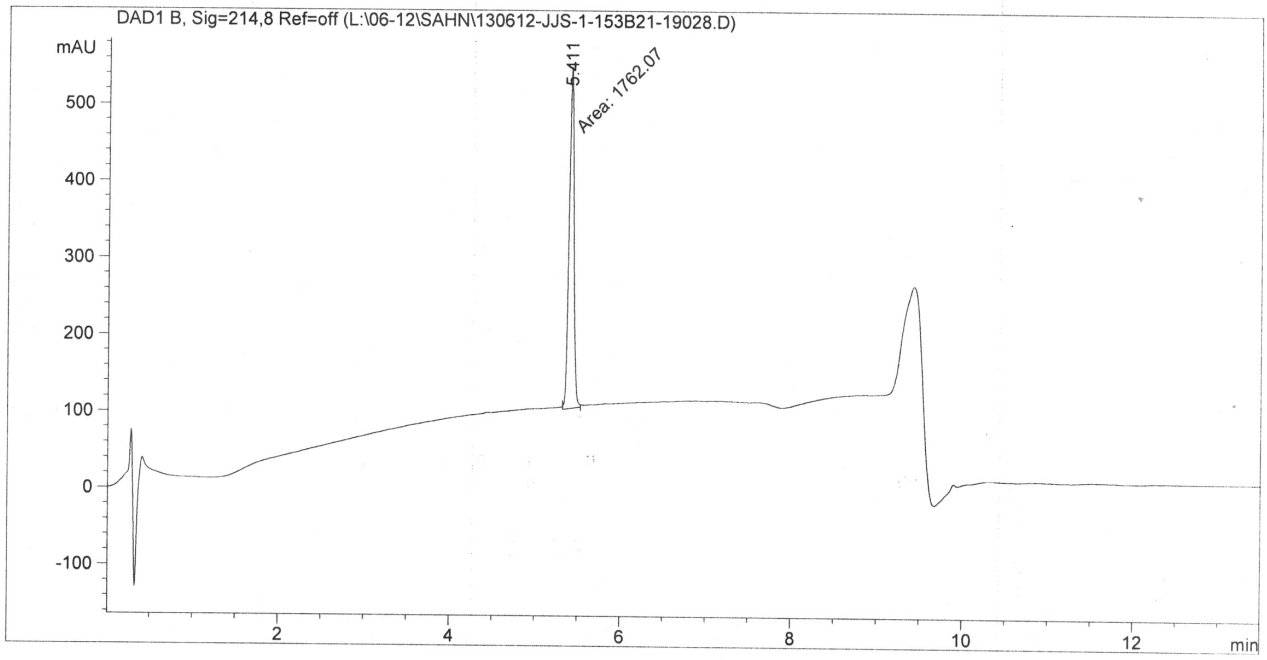


Data File L:\06-12\SAHN\130612-JJS-1-153B21-19028.D
Sample Name: jjs-1-153B2

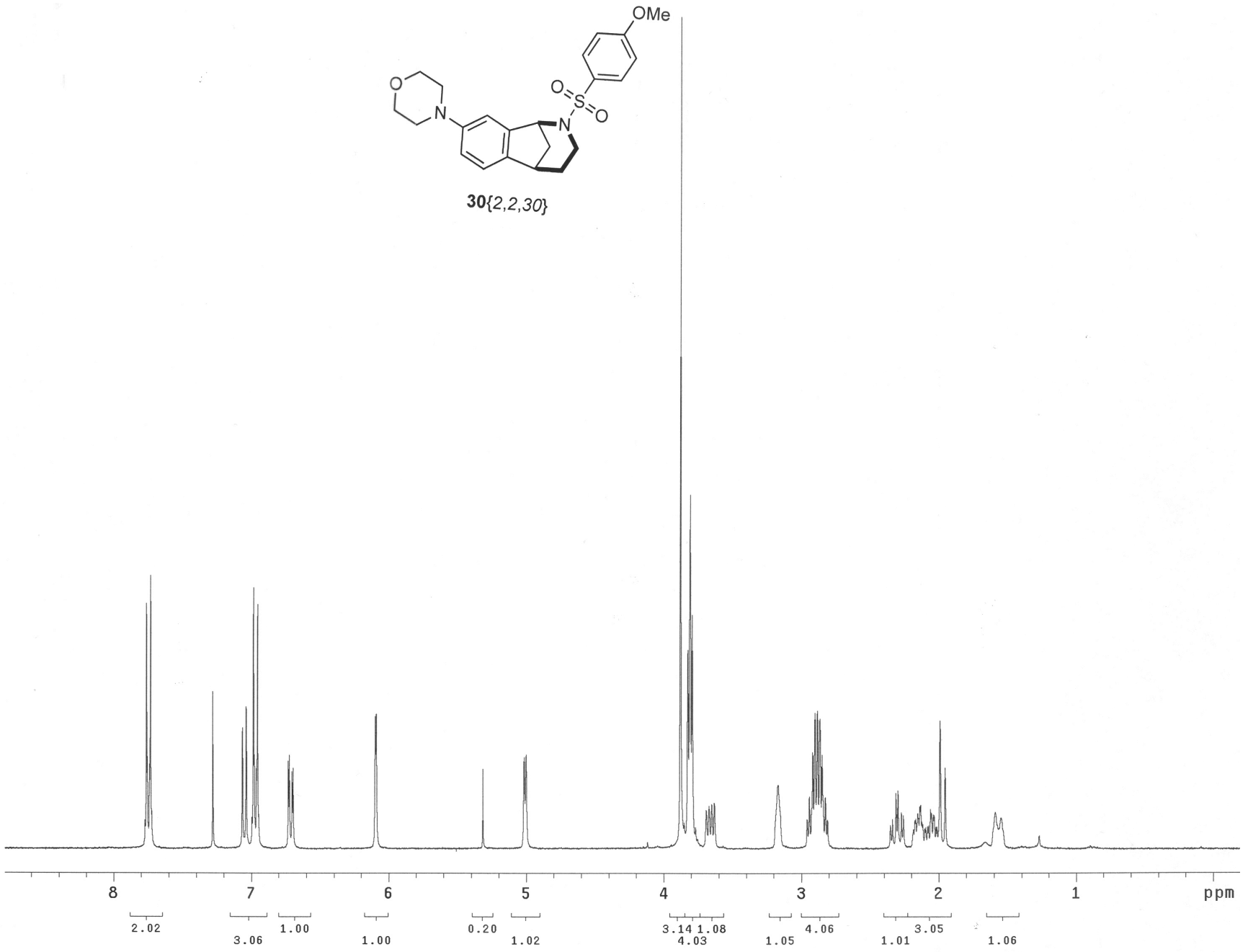


28{*2,10,15*}

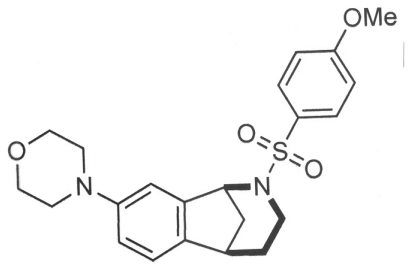
```
==================================================
Acq. Operator    : sahn@mail.utexas.edu
Acq. Instrument  : LCMS
Injection Date   : 6/13/2012 4:13:07 PM

Acq. Method      : C:\CHEM32\1\METHODS\SP NIH.M
Last changed     : 6/13/2012 4:12:46 PM by sahn@mail.utexas.edu
                   (modified after loading)
Analysis Method  : C:\CHEM32\1\METHODS\DEEF_LC.M
Last changed     : 6/13/2012 3:34:08 PM
                   (modified after loading)
Sample Info      : Easy-Access Method: 'SP NIH'
```




```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,8 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]    [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.411 MM    0.0659 1762.06799  445.93961 100.0000

Totals :                  1762.06799  445.93961
```

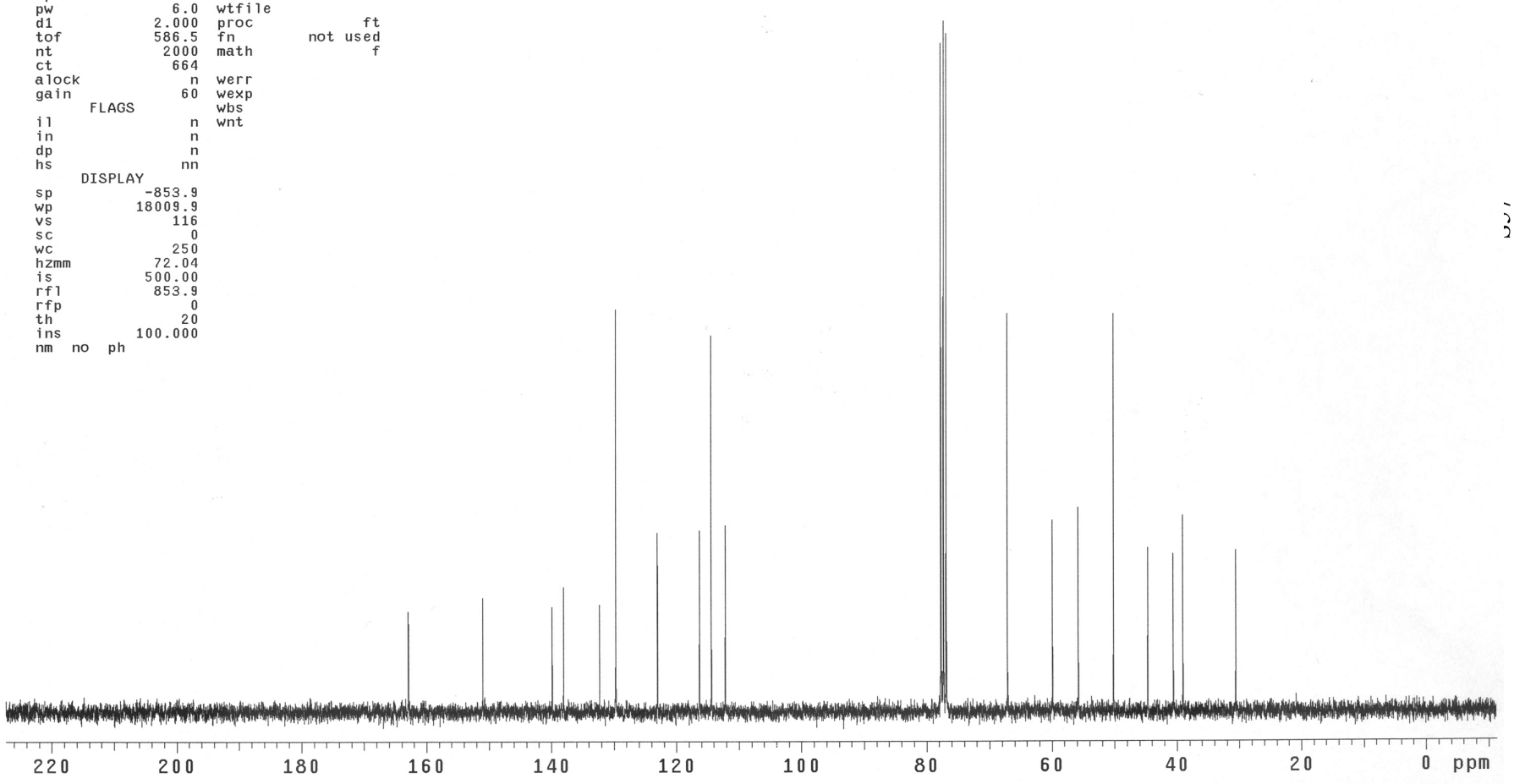

OMe
O
N
O=S=O
N
8
7
6
5
4
3
2
1
ppm
2.02
3.06
1.00
1.00
0.20
1.02
3.14
1.08
4.03
1.05
4.06
1.01
3.05
1.06

30{*2,2,30*}

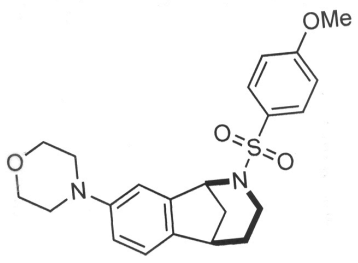


30{*2,2,30*}

```
13C OBSERVE

exp1  std13c

      SAMPLE
date   Jul 21 2009
solvent      CDCl3
file           exp
   ACQUISITION
sfrq        75.478
tn             C13
at             1.777
np             64000
sw          18009.9
fb             10000
bs                 8
tpwr              50
pw               6.0
d1             2.000
tof            586.5
nt              2000
ct               664
alock              n
gain              60
      FLAGS
il                 n
in                 n
dp                 n
hs                nn
     DISPLAY
sp           -853.9
wp          18009.9
vs              116
sc                0
wc              250
hzmm          72.04
is           500.00
rfl           853.9
rfp               0
th               20
ins          100.000
nm  no  ph

      DEC. & VT
dfrq        300.141
dn               H1
dpwr             40
dof           400.0
dm               yyy
dmm                w
dmf             8000
dseq
dres              1.0
homo                n
    PROCESSING
lb               1.00
wtfile
proc               ft
fn          not used
math                f

werr
wexp
wbs
wnt
```

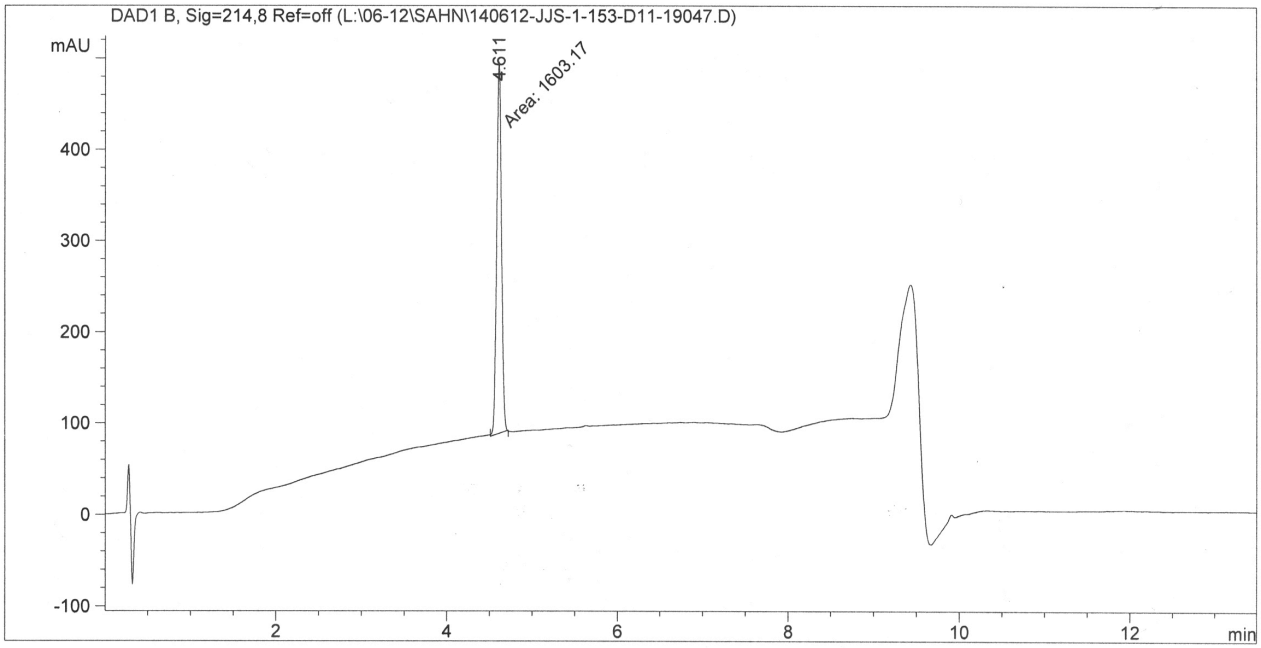


Data File L:\06-12\SAHN\140612-JJS-1-153-D11-19047.D
Sample Name: jjs-1-153-D1

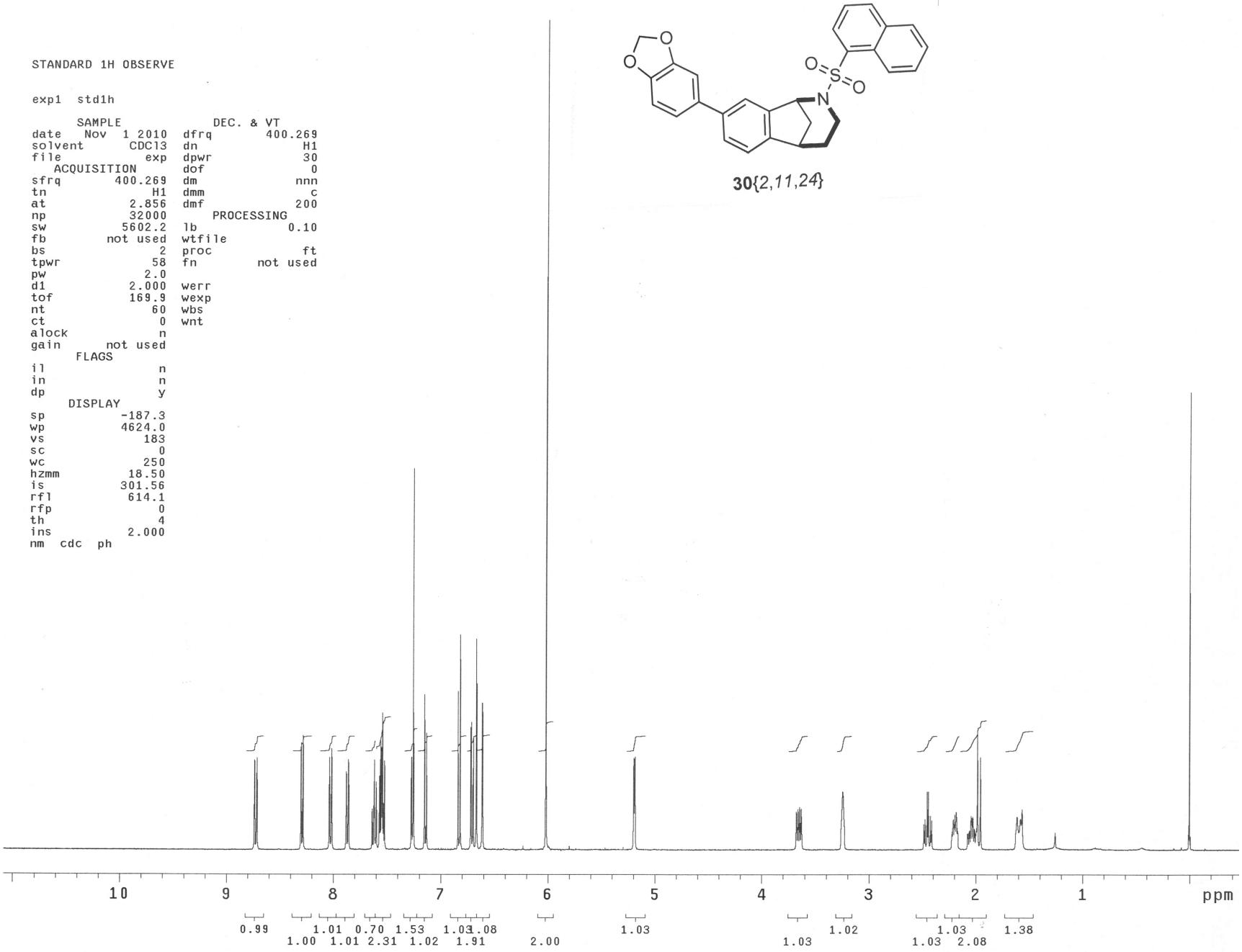


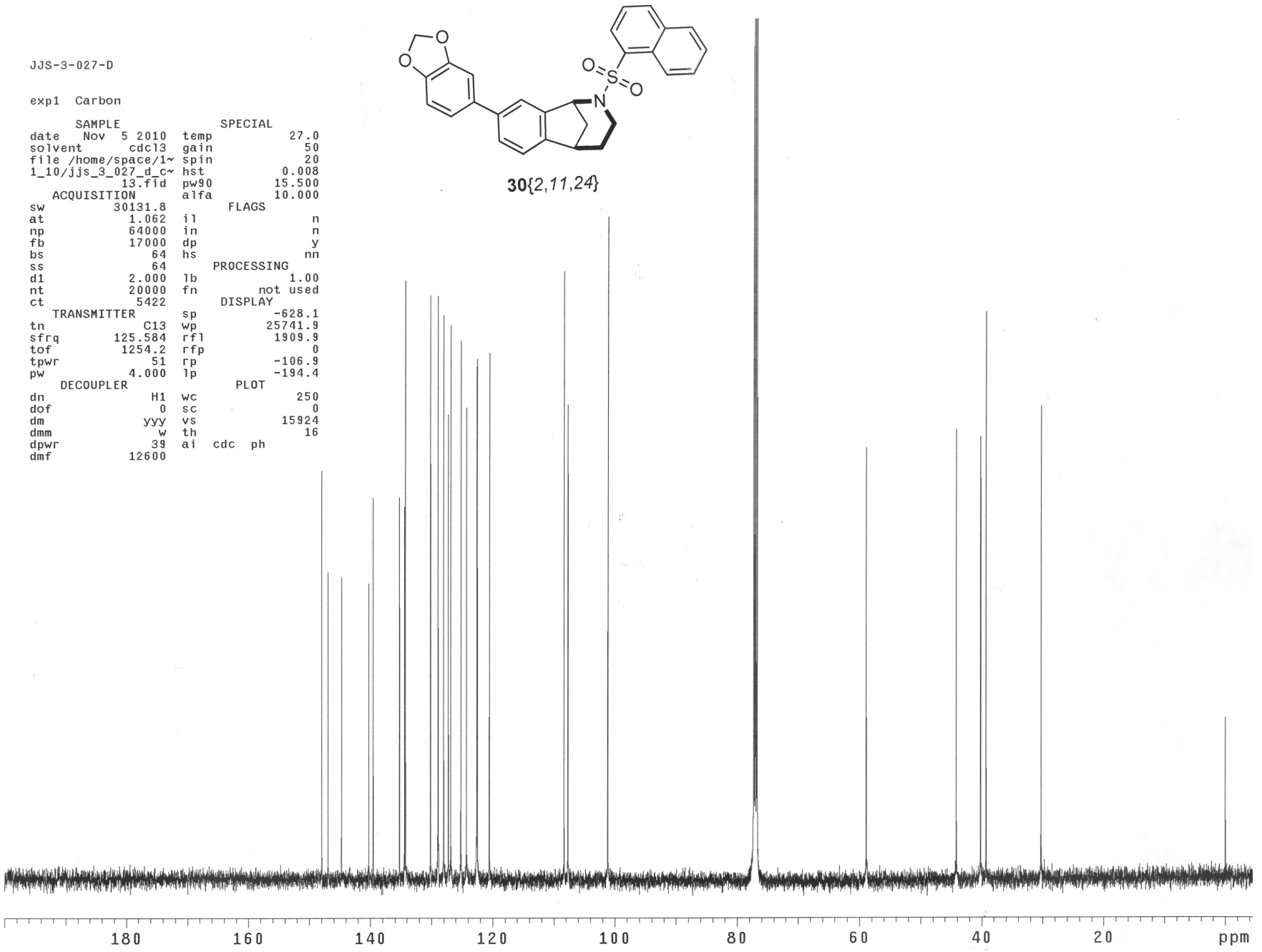
30{2,2,30}

```
    ========================================================
    Acq. Operator    : sahn@mail.utexas.edu
    Acq. Instrument  : LCMS                              Loca
    Injection Date   : 6/14/2012 9:54:05 AM
                                                        Inj Vo
    Acq. Method      : C:\CHEM32\1\METHODS\SP NIH.M
    Last changed     : 6/14/2012 9:53:50 AM by sahn@mail.ut
                       (modified after loading)
    Analysis Method  : C:\CHEM32\1\METHODS\DEEF_LC.M
    Last changed     : 9/28/2011 3:20:36 PM
    Sample Info      : Easy-Access Method: 'SP NIH'
```

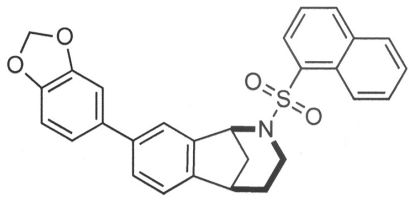



```
    ========================================================================
                              Area Percent Report
    ========================================================================

    Sorted By             :      Signal
    Multiplier:                   :      1.0000
    Dilution:                     :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: DAD1 B, Sig=214,8 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]    [mAU*s]    [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   4.611 MM    0.0650 1603.17493  411.30872 100.0000

    Totals :                   1603.17493  411.30872


    ========================================================================

Instrument 1 6/14/2012 11:09:14 AM                          Page   1 of 1
```


STANDARD 1H OBSERVE
exp1 std1h
SAMPLE
date Nov 1 2010
solvent CDCl3
file exp
ACQUISITION
sfrq 400.269
tn H1
at 2.856
np 32000
sw 5602.2
fb not used
bs 2
tpwr 58
pw 2.0
d1 2.000
tof 169.9
nt 60
ct 0
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -187.3
wp 4624.0
vs 183
sc 0
wc 250
hzmm 18.50
is 301.56
rfl 614.1
rfp 0
th 4
ins 2.000
nm cdc ph
DEC. & VT
dfrq 400.269
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
lb 0.10
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
O
O
O=S=O
N
30{2,11,24}
10
9
8
7
6
5
4
3
2
1
ppm
0.99
1.01
0.70
1.53
1.03
1.08
2.00
1.03
1.03
1.02
1.03
1.38
1.00
1.01
2.31
1.02
1.91
1.03
2.08

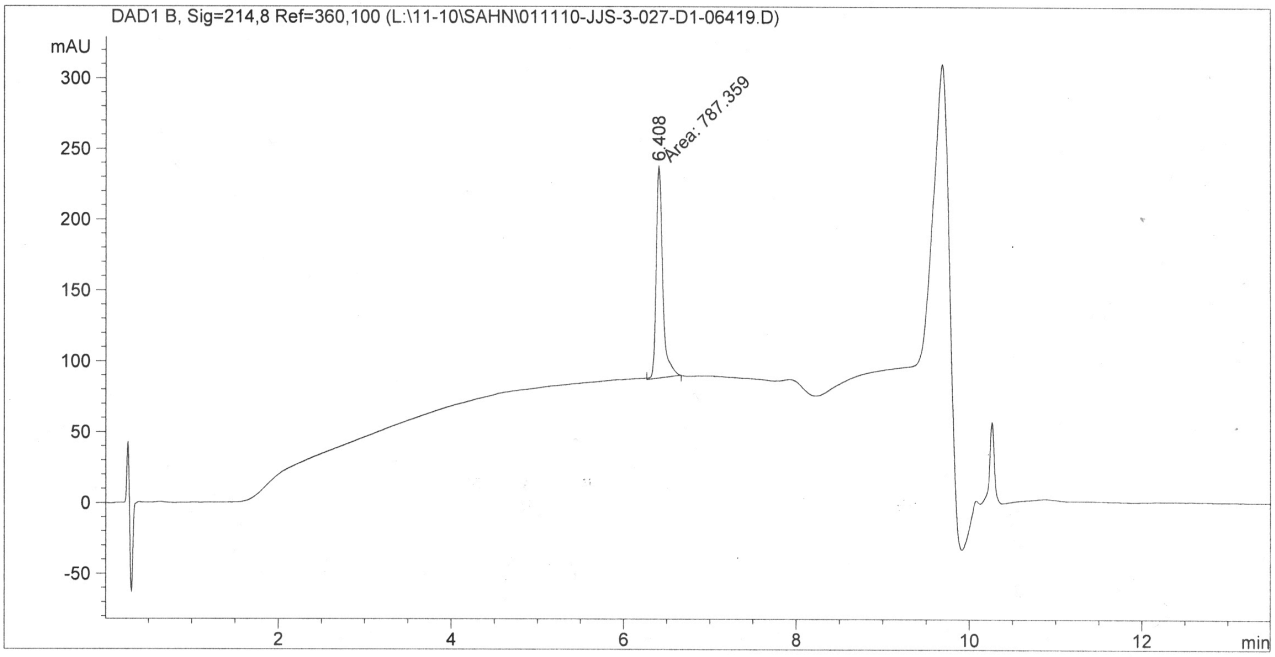

JJS-3-027-D
exp1 Carbon
SAMPLE
date Nov 5 2010
solvent cdcl3
file /home/space/1~
1_10/jjs_3_027_d_c~
13.fid
ACQUISITION
sw 30131.8
at 1.062
np 64000
fb 17000
bs 64
ss 64
d1 2.000
nt 20000
ct 5422
TRANSMITTER
tn C13
sfrq 125.584
tof 1254.2
tpwr 51
pw 4.000
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 39
dmf 12600
SPECIAL
temp 27.0
gain 50
spin 20
hst 0.008
pw90 15.500
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -628.1
wp 25741.9
rfl 1909.9
rfp 0
rp -106.9
lp -194.4
PLOT
wc 250
sc 0
vs 15924
th 16
ai cdc ph
O
O
O=S=O
N
30{2,11,24}
180
160
140
120
100
80
60
40
20
ppm

Data File L:\11-10\SAHN\011110-JJS-3-027-D1-06419.D
Sample Name: jjs-3-027-D

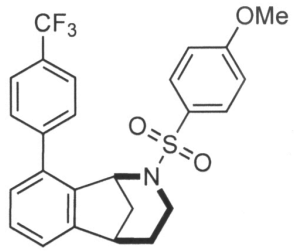


30{*2,11,24*}

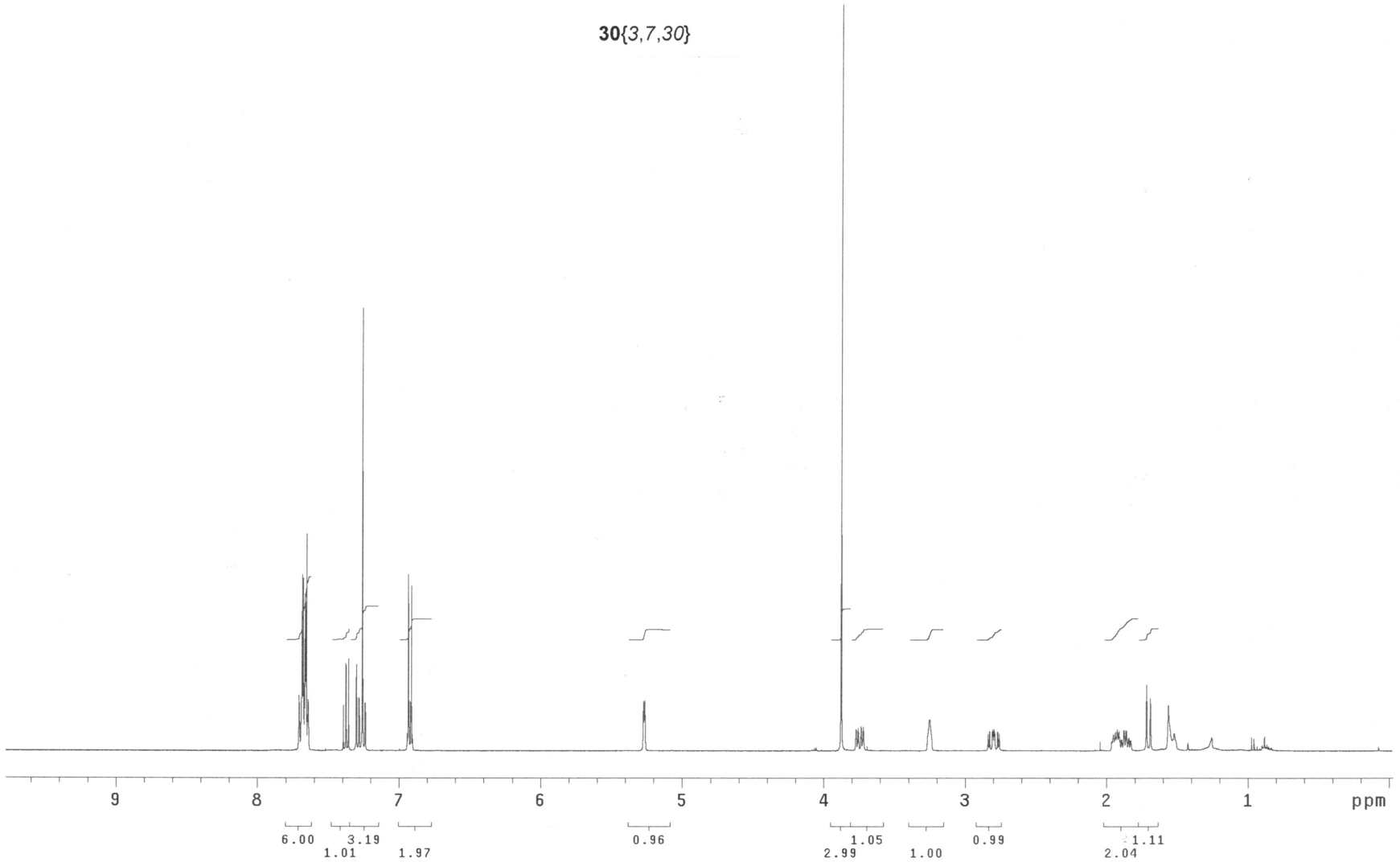
```
==================================================================
Acq. Operator    : sahn@mail.utexas.edu
Acq. Instrument  : LCMS                          Location :
Injection Date   : 11/1/2010 5:23:26 PM
                                                 Inj Volume :
Acq. Method      : C:\CHEM32\1\METHODS\SP NIH.M
Last changed     : 11/1/2010 5:23:11 PM by sahn@mail.utexas.edu
                   (modified after loading)
Analysis Method  : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed     : 11/20/2006 4:14:44 AM
Sample Info      : Easy-Access Method: 'SP NIH'
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]      %
----|-------|----|-------|----------|----------|--------|
   1   6.408 MM    0.0874  787.35876  150.15947 100.0000

Totals :                   787.35876  150.15947

==================================================================
```


CF3
OMe
O
S
O
N

30{*3*,*7*,*30*}

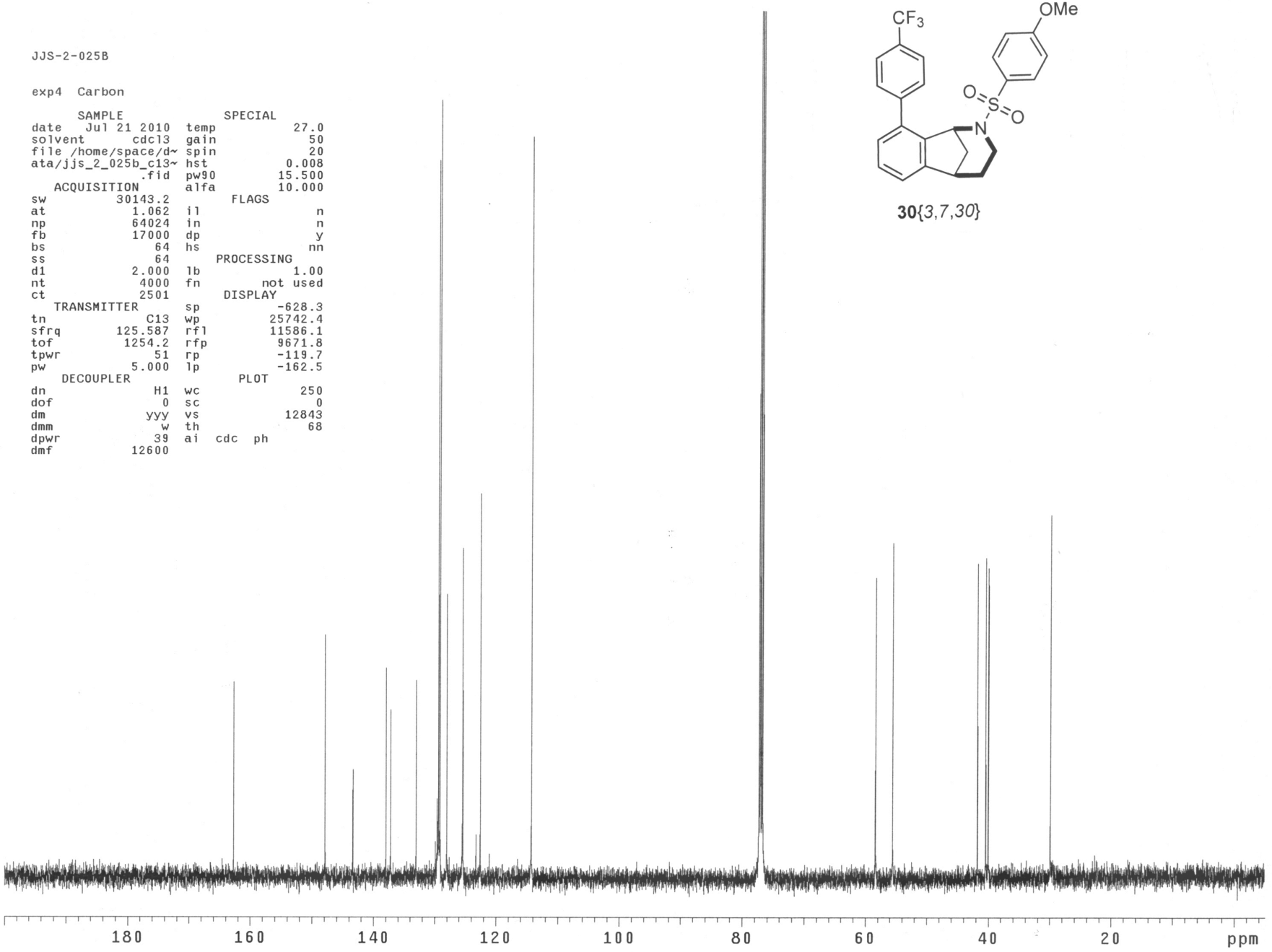

9
8
7
6
5
4
3
2
1
ppm
6.00
3.19
1.01
1.97
0.96
1.05
2.99
1.00
0.99
1.11
2.04

JJS-2-025B

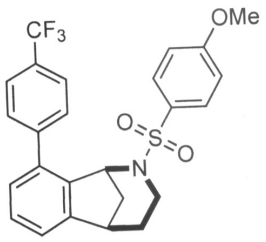
exp4 Carbon

SAMPLE		SPECIAL	
date	Jul 21 2010	temp	27.0
solvent	cdcl3	gain	50
file	/home/space/d~	spin	20
ata/jjs_2_025b_c13~		hst	0.008
	.fid	pw90	15.500
ACQUISITION		alfa	10.000
sw	30143.2	FLAGS	
at	1.062	il	n
np	64024	in	n
fb	17000	dp	y
bs	64	hs	nn
ss	64	PROCESSING	
d1	2.000	lb	1.00
nt	4000	fn	not used
ct	2501	DISPLAY	
TRANSMITTER		sp	-628.3
tn	C13	wp	25742.4
sfrq	125.587	rfl	11586.1
tof	1254.2	rfp	9671.8
tpwr	51	rp	-119.7
pw	5.000	lp	-162.5
DECOUPLER		PLOT	
dn	H1	wc	250
dof	0	sc	0
dm	yyy	vs	12843
dmm	w	th	68
dpwr	39	ai cdc ph	
dmf	12600		

30{*3,7,30*}

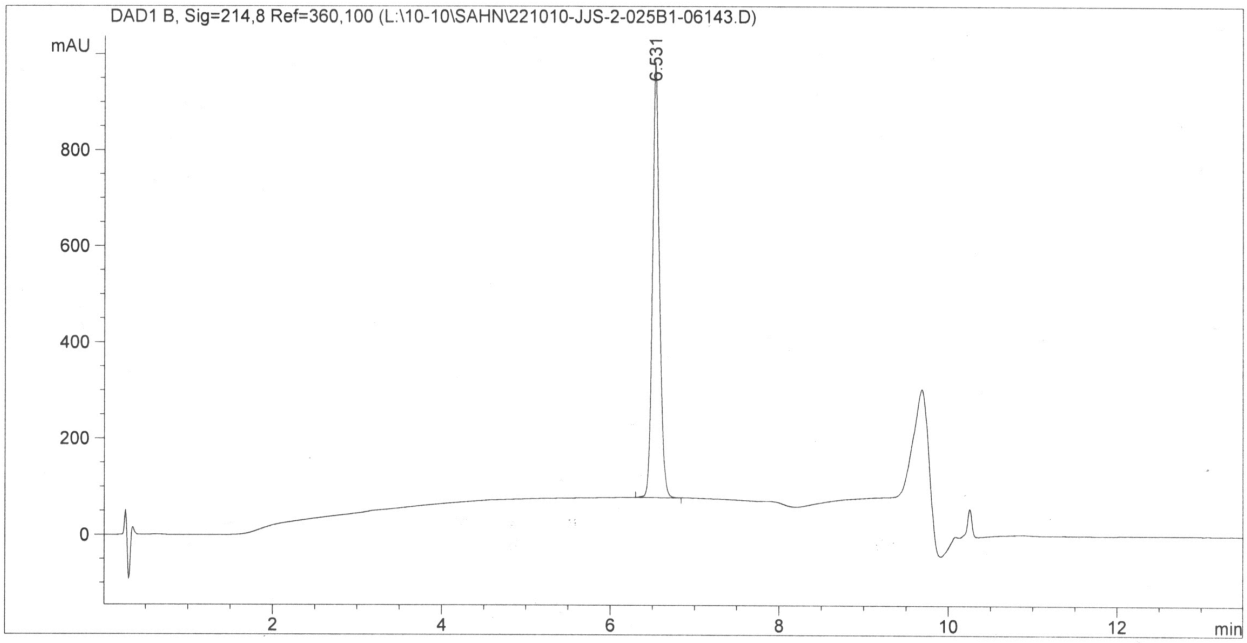


Data File L:\10-10\SAHN\221010-JJS-2-025B1-06143.D
Sample Name: jjs-2-025B

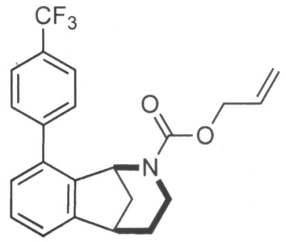


30{*3,7,30*}

```
Acq. Operator    : sahn@mail.utexas.edu
Acq. Instrument  : LCMS                              Location : Vial 37
Injection Date   : 10/22/2010 7:49:14 PM
                                                     Inj Volume : 1.0 µl
Acq. Method      : C:\CHEM32\1\METHODS\SP NIH.M
Last changed     : 10/22/2010 7:48:59 PM by sahn@mail.utexas.edu
                   (modified after loading)
Analysis Method  : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed     : 11/20/2006 4:14:44 AM
Sample Info      : Easy-Access Method: 'SP NIH'
```




```
                          Area Percent Report

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]      %
----|-------|----|-------|----------|----------|--------|
   1   6.531 BB    0.0852 5222.66846  906.70734 100.0000

Totals :                  5222.66846  906.70734
```

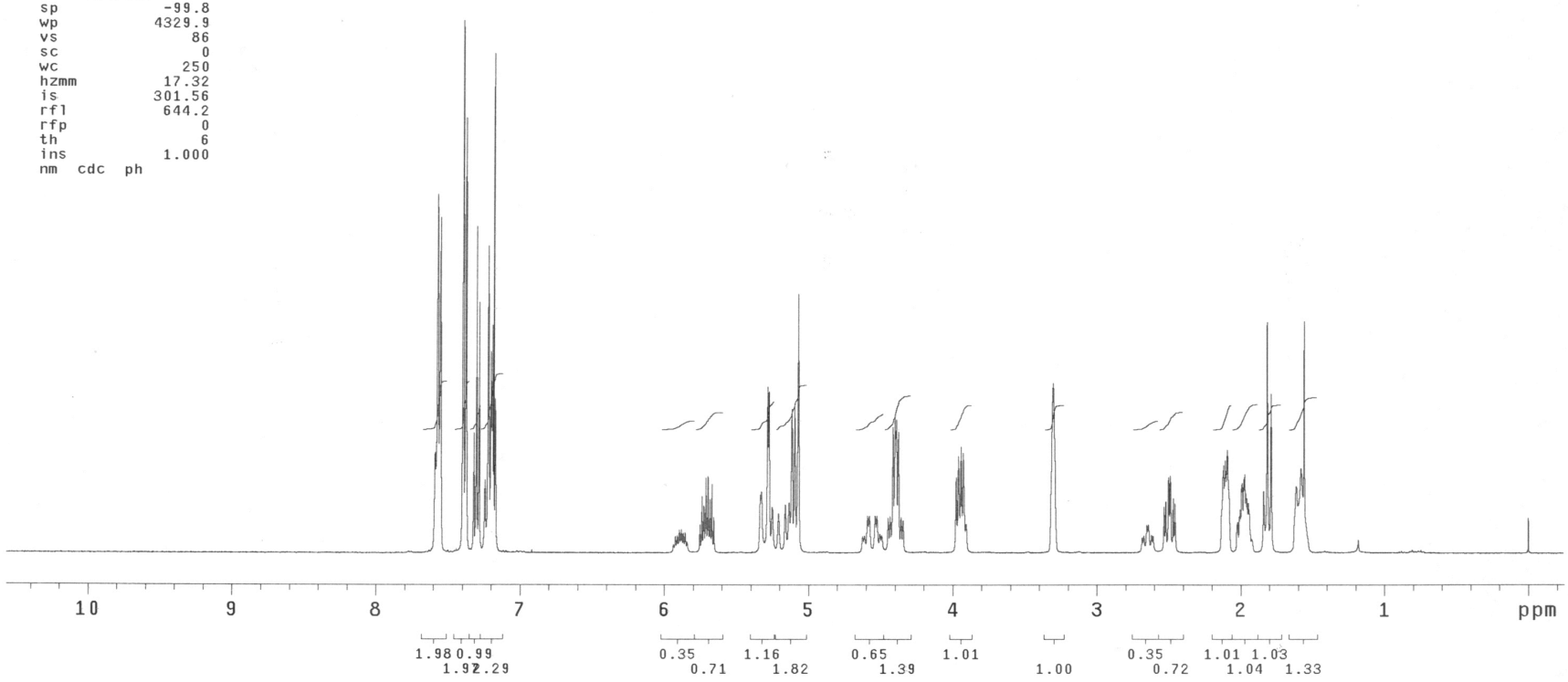


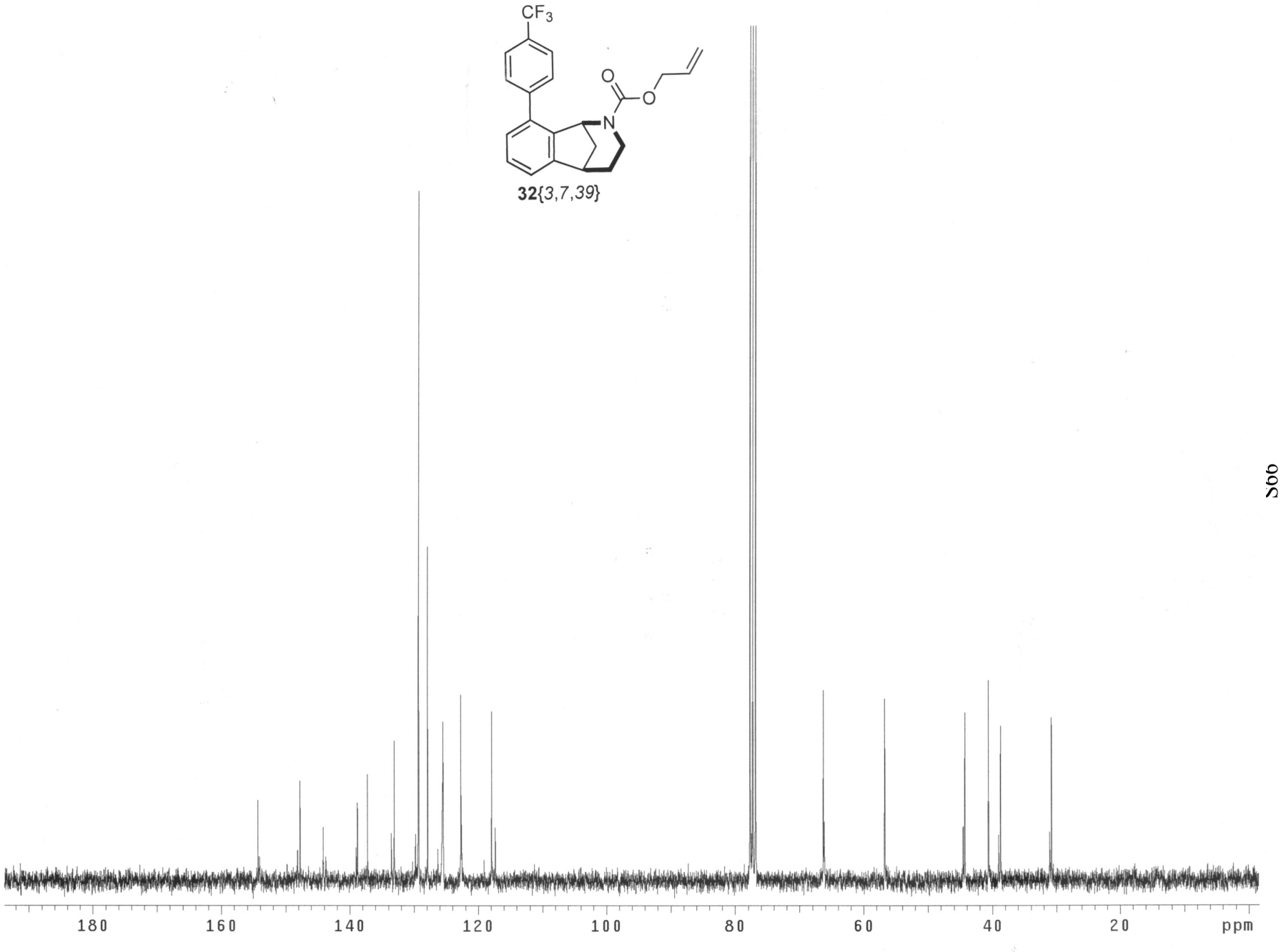
32{*3,7,39*}

STANDARD 1H OBSERVE

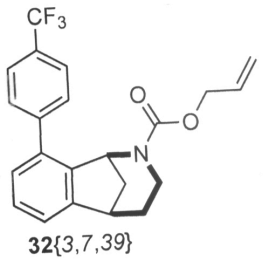
exp1 std1h

SAMPLE		DEC. & VT	
date	Jun 23 2010	dfrq	400.269
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	400.269	dm	nnn
tn	H1	dmm	c
at	2.856	dmf	200
np	32000	PROCESSING	
sw	5602.2	lb	0.10
fb	not used	wtfile	
bs	2	proc	ft
tpwr	58	fn	not used
pw	2.0		
d1	2.000	werr	
tof	169.9	wexp	
nt	100	wbs	
ct	72	wnt	
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-99.8		
wp	4329.9		
vs	86		
sc	0		
wc	250		
hzmm	17.32		
is	301.56		
rfl	644.2		
rfp	0		
th	6		
ins	1.000		
nm cdc ph			



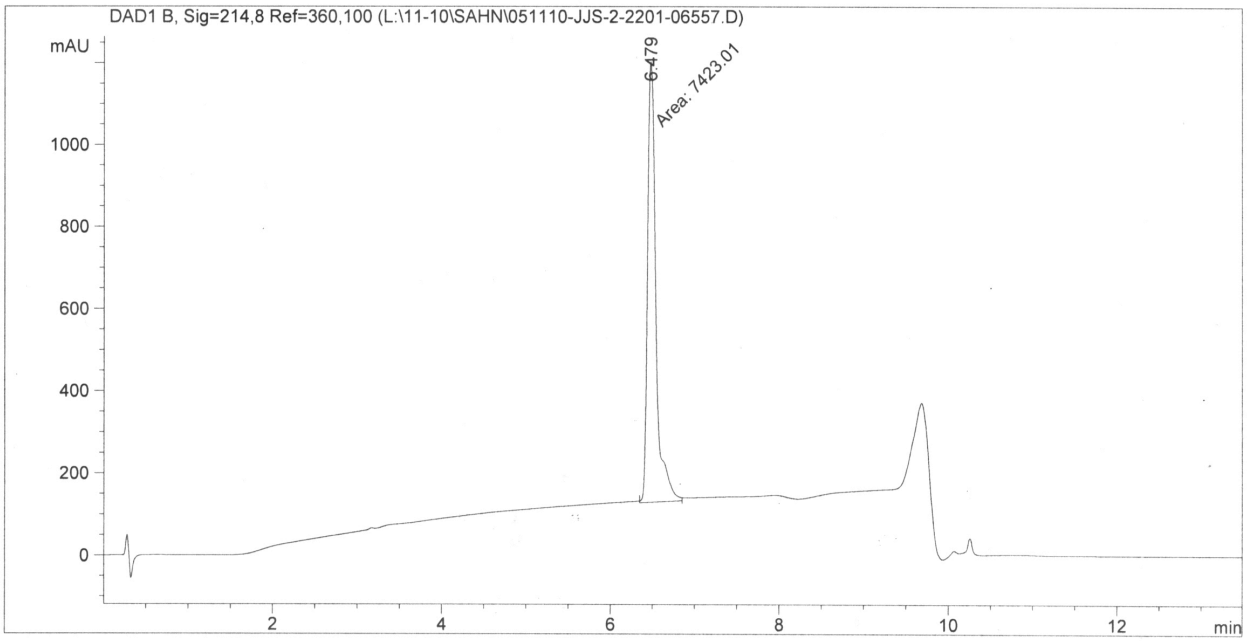

CF3
O
O
N
32{3,7,39}
180
160
140
120
100
80
60
40
20
ppm

Data File L:\11-10\SAHN\051110-JJS-2-2201-06557.D
Sample Name: jjs-2-220

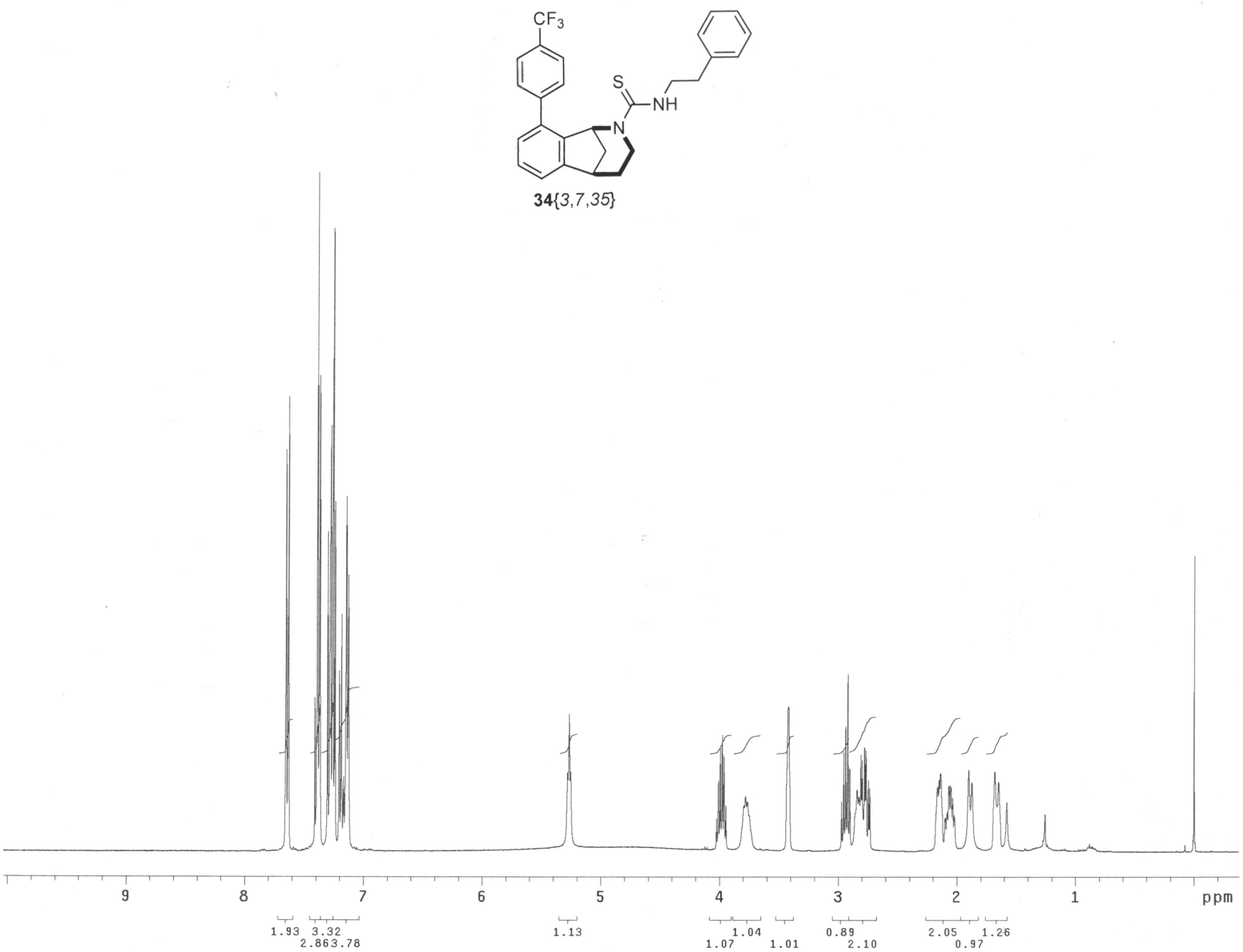


32{3,7,39}

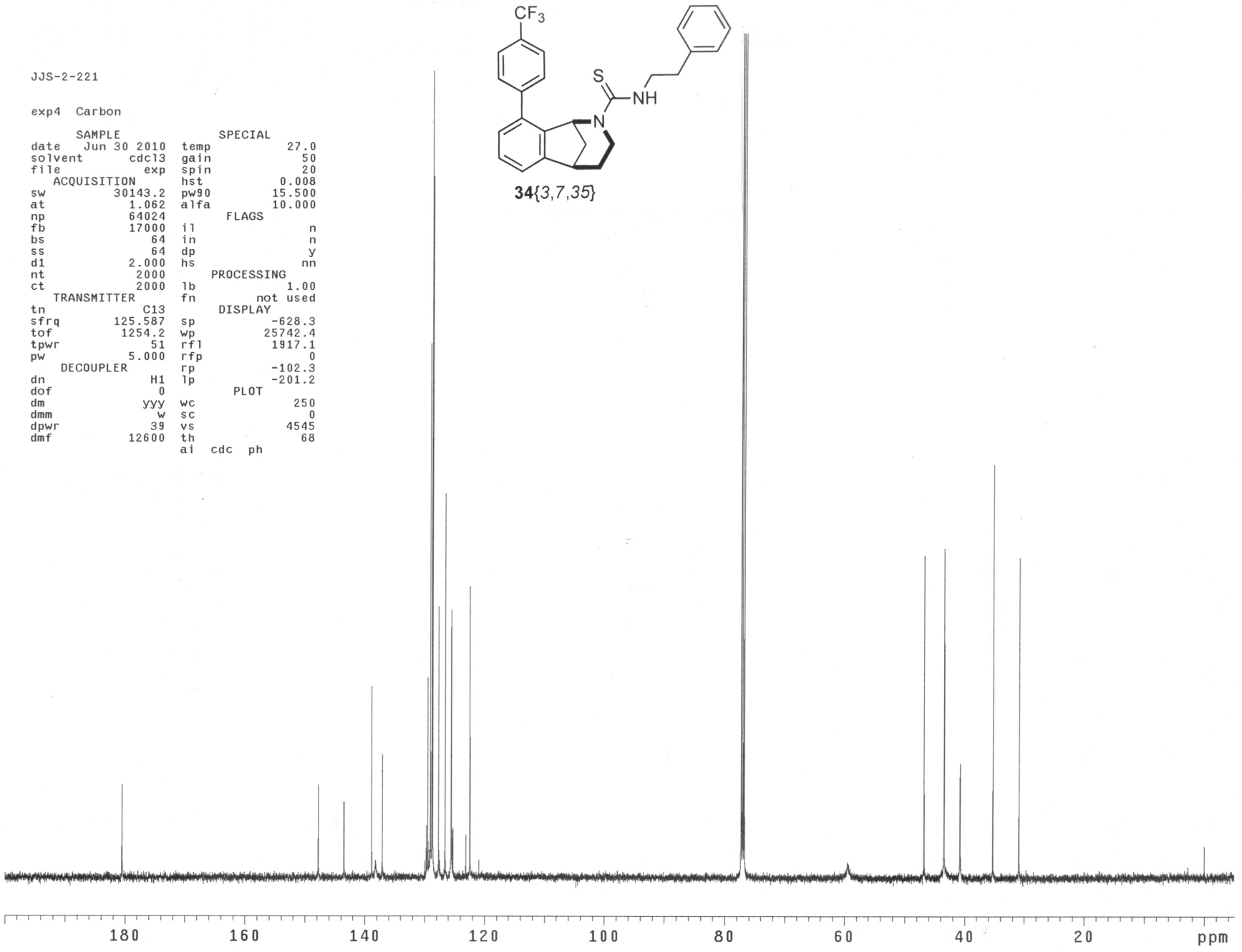
```
Acq. Operator    : sahn@mail.utexas.edu
Acq. Instrument  : LCMS                          Location : Vial 64
Injection Date   : 11/5/2010 7:40:54 PM
                                                 Inj Volume : 1.0 µl
Acq. Method      : C:\CHEM32\1\METHODS\SP NIH.M
Last changed     : 11/5/2010 7:40:39 PM by sahn@mail.utexas.edu
                   (modified after loading)
Analysis Method  : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed     : 11/20/2006 4:14:44 AM
Sample Info      : Easy-Access Method: 'SP NIH'
```




```
                          Area Percent Report

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.479 MM    0.1152 7423.00830 1074.12939 100.0000

Totals :                  7423.00830 1074.12939
```

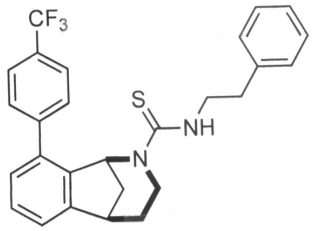

CF3
S
NH
N
9
8
7
6
5
4
3
2
1
ppm
1.93
3.32
2.86
3.78
1.13
1.07
1.04
1.01
0.89
2.10
2.05
0.97
1.26

34{*3,7,35*}

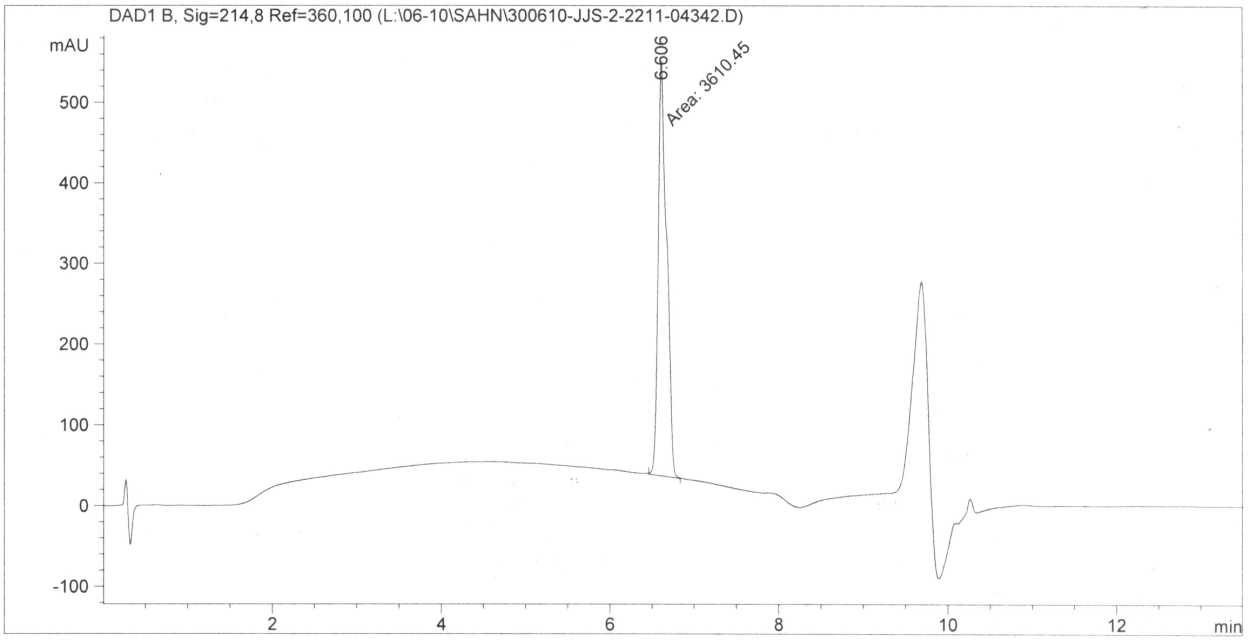

JJS-2-221
exp4 Carbon
SAMPLE
date Jun 30 2010
solvent cdcl3
file exp
ACQUISITION
sw 30143.2
at 1.062
np 64024
fb 17000
bs 64
ss 64
d1 2.000
nt 2000
ct 2000
TRANSMITTER
tn C13
sfrq 125.587
tof 1254.2
tpwr 51
pw 5.000
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 39
dmf 12600
SPECIAL
temp 27.0
gain 50
spin 20
hst 0.008
pw90 15.500
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -628.3
wp 25742.4
rfl 1917.1
rfp 0
rp -102.3
lp -201.2
PLOT
wc 250
sc 0
vs 4545
th 68
ai cdc ph
CF3
S
NH
N
34{3,7,35}
180
160
140
120
100
80
60
40
20
ppm

Data File L:\06-10\SAHN\300610-JJS-2-2211-04342.D
Sample Name: jjs-2-221

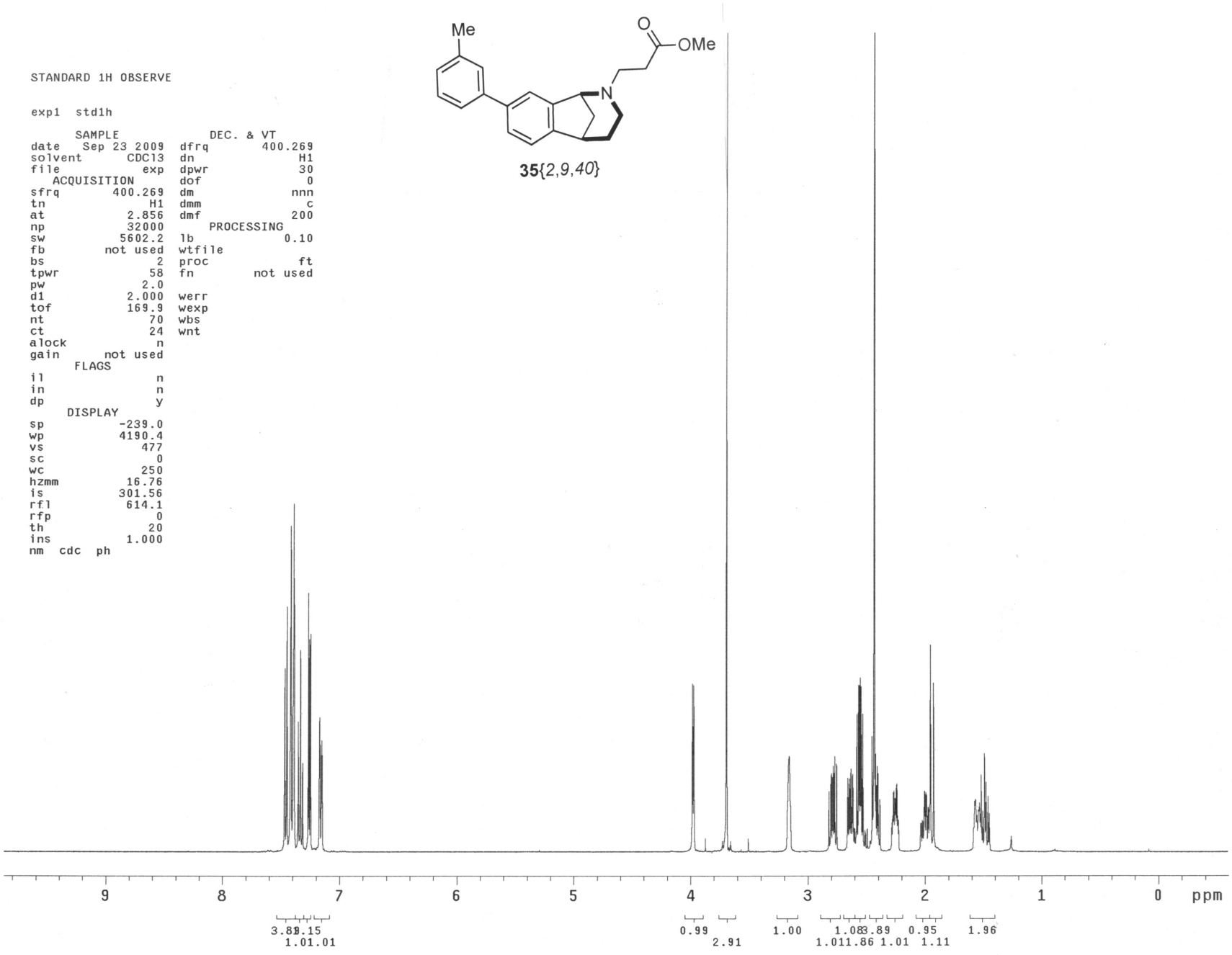
```
==========================================================================
Acq. Operator    : sahn@mail.utexas.edu
Acq. Instrument : LCMS                              Location : Vial 17
Injection Date   : 6/30/2010 6:22:42 PM
                                                   Inj Volume : 1.0 µl
Acq. Method      : C:\CHEM32\1\METHODS\SP NIH.M
Last changed     : 6/30/2010 6:22:27 PM by sahn@mail.utexas.edu
                   (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed     : 11/20/2006 4:14:44 AM
Sample Info      : Easy-Access Method: 'SP NIH'
```

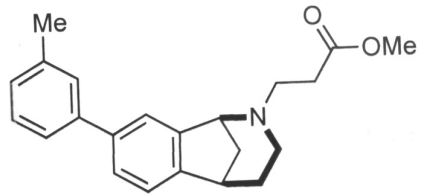


34{*3,7,35*}

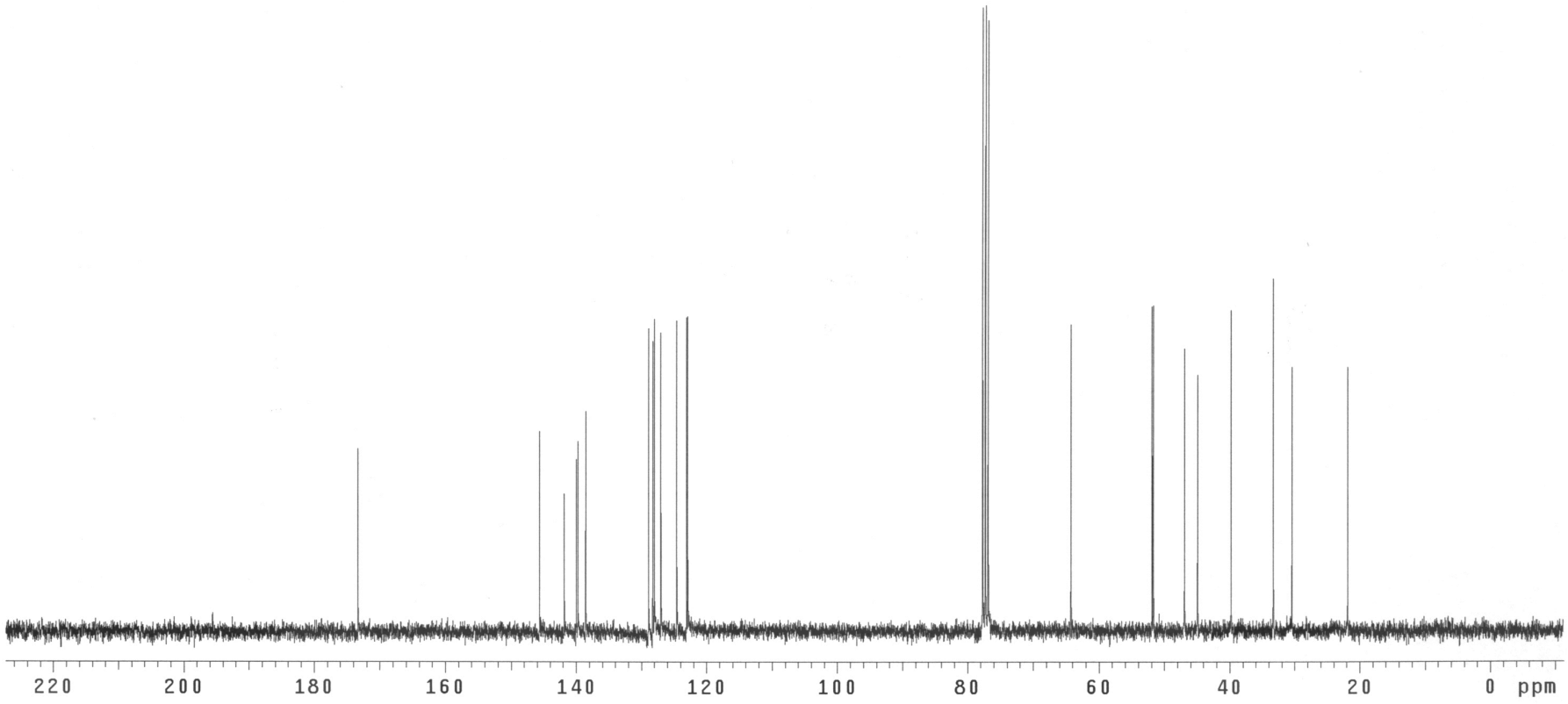



```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.606 MM    0.1164 3610.44775  517.13934 100.0000

Totals :                  3610.44775  517.13934

==========================================================================
```

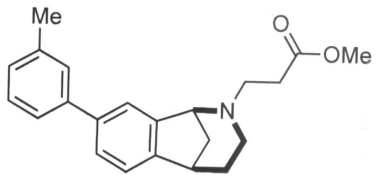

STANDARD 1H OBSERVE
exp1 std1h
SAMPLE
date Sep 23 2009
solvent CDCl3
file exp
ACQUISITION
sfrq 400.269
tn H1
at 2.856
np 32000
sw 5602.2
fb not used
bs 2
tpwr 58
pw 2.0
d1 2.000
tof 169.9
nt 70
ct 24
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -239.0
wp 4190.4
vs 477
sc 0
wc 250
hzmm 16.76
is 301.56
rfl 614.1
rfp 0
th 20
ins 1.000
nm cdc ph
DEC. & VT
dfrq 400.269
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
lb 0.10
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
Me
O
OMe
N
35{2,9,40}
9
8
7
6
5
4
3
2
1
0
ppm
3.81
3.15
1.01
1.01
0.99
2.91
1.00
1.01
1.08
1.86
3.89
1.01
0.95
1.11
1.96



35{*2,9,40*}

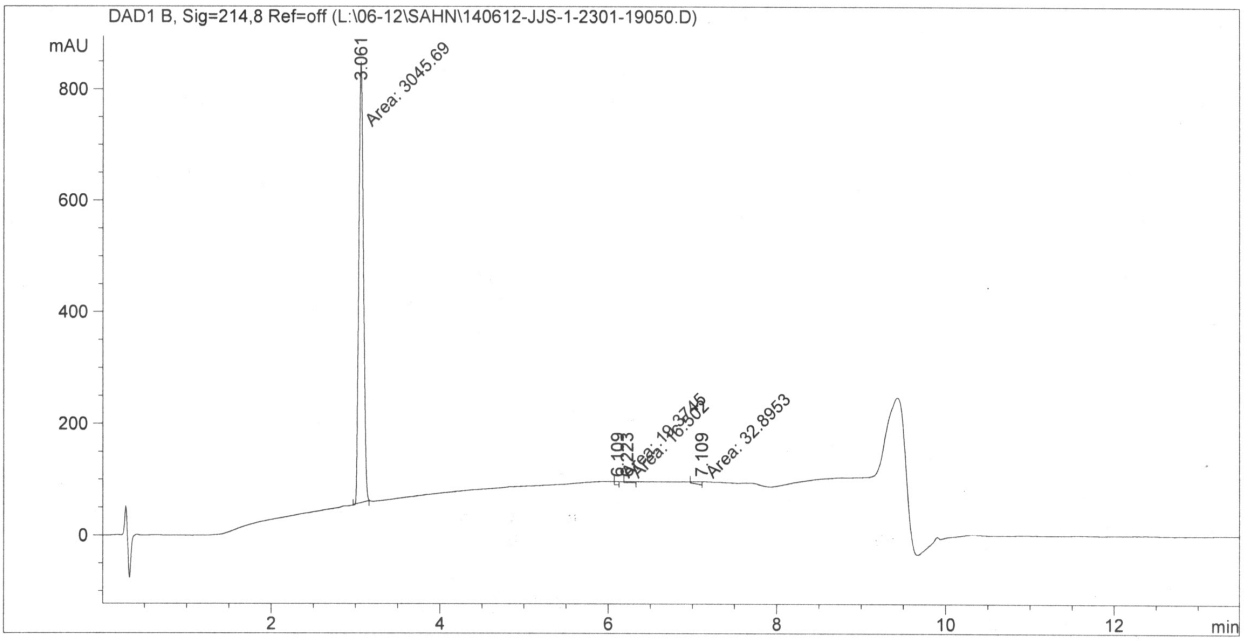


Data File L:\06-12\SAHN\140612-JJS-1-2301-19050.D
Sample Name: jjs-1-230



35{*2,9,40*}

```
============================================================
Acq. Operator    : sahn@mail.utexas.edu
Acq. Instrument  : LCMS                          Location :
Injection Date   : 6/14/2012 10:44:01 AM
                                                 Inj Volume :
Acq. Method      : C:\CHEM32\1\METHODS\SP NIH.M
Last changed     : 6/14/2012 10:43:46 AM by sahn@mail.utexas.
                   (modified after loading)
Analysis Method  : C:\CHEM32\1\METHODS\DEEF_LC.M
Last changed     : 9/28/2011 3:20:36 PM
Sample Info      : Easy-Access Method: 'SP NIH'
```



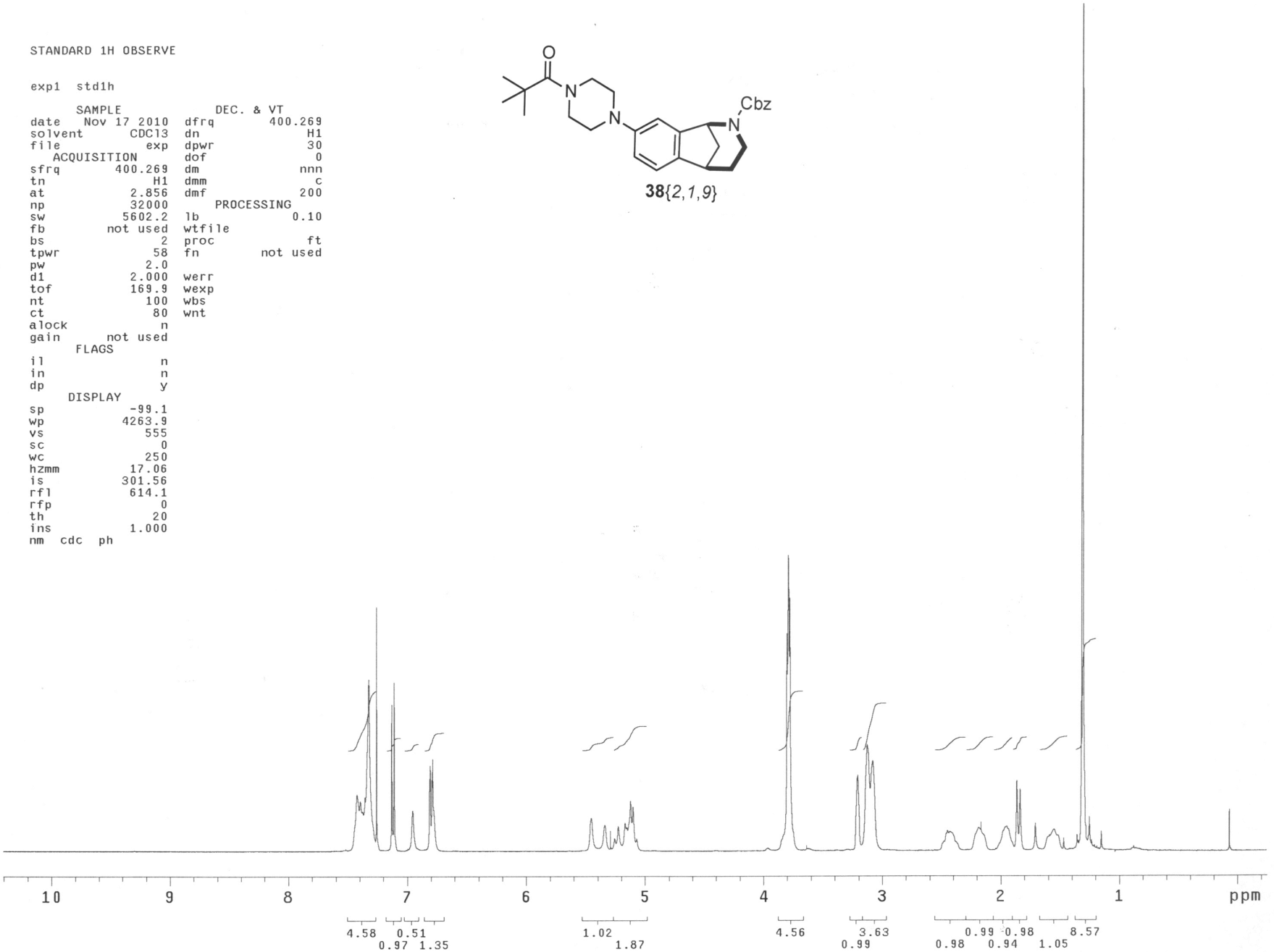

```
============================================================================
                          Area Percent Report
============================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,8 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   3.061 MM    0.0639 3045.68555  794.70520  97.7919
   2   6.109 MM    0.0581   19.37449    5.55921   0.6221
   3   6.223 MM    0.1369   16.50199    2.00963   0.5299
   4   7.109 MM    0.1018   32.89531    5.38541   1.0562

Totals :                  3114.45734  807.65945
```

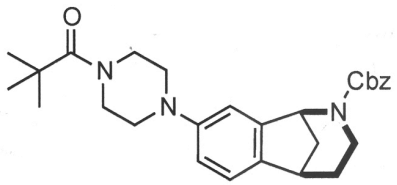
STANDARD 1H OBSERVE

exp1 std1h

SAMPLE		DEC. & VT	
date	Nov 17 2010	dfrq	400.269
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	400.269	dm	nnn
tn	H1	dmm	c
at	2.856	dmf	200
np	32000	PROCESSING	
sw	5602.2	lb	0.10
fb	not used	wtfile	
bs	2	proc	ft
tpwr	58	fn	not used
pw	2.0		
d1	2.000	werr	
tof	169.9	wexp	
nt	100	wbs	
ct	80	wnt	
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-99.1		
wp	4263.9		
vs	555		
sc	0		
wc	250		
hzmm	17.06		
is	301.56		
rfl	614.1		
rfp	0		
th	20		
ins	1.000		
nm cdc ph			

38{*2,1,9*}



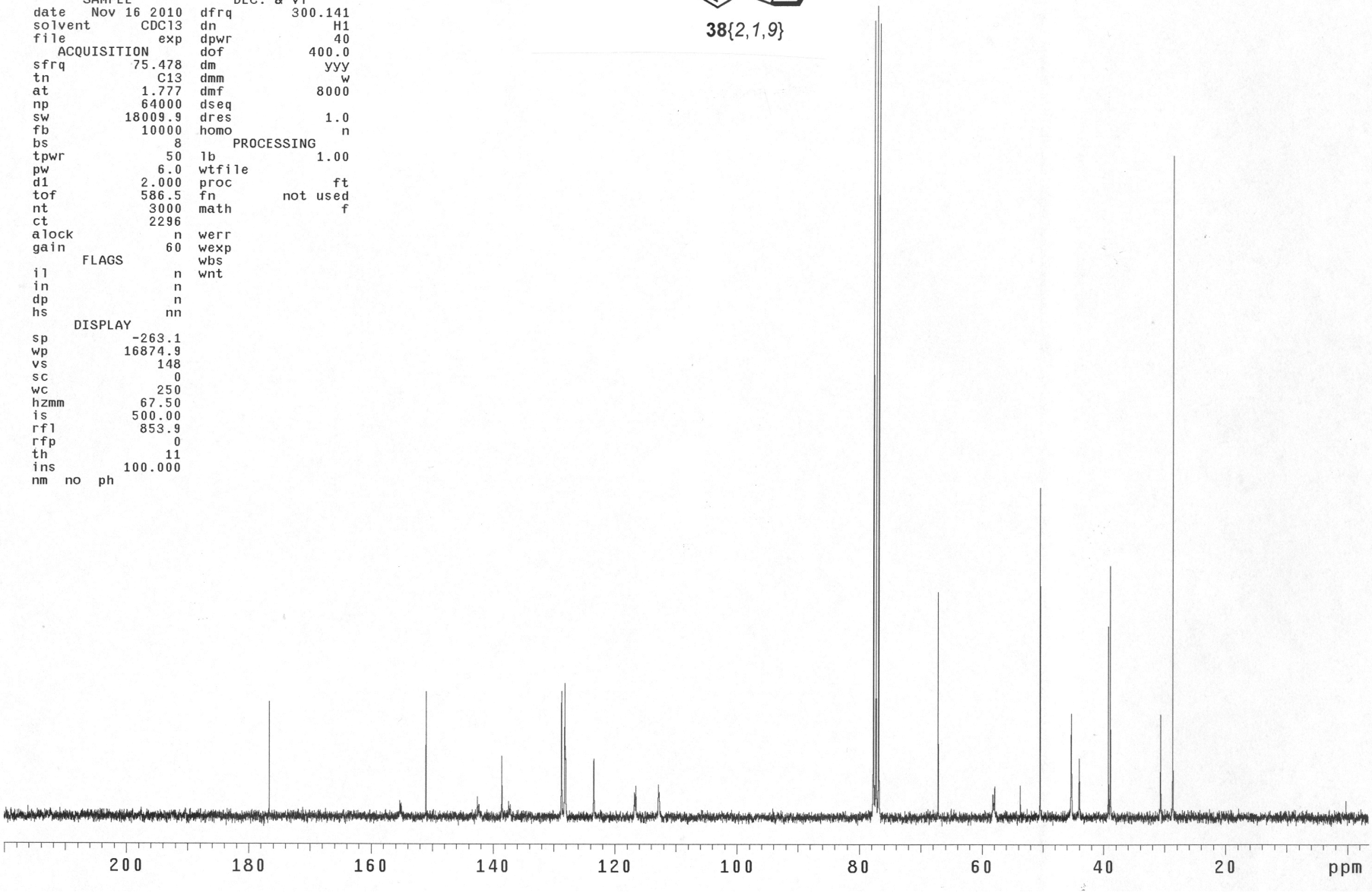


38{*2,1,9*}

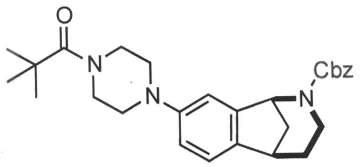
13C OBSERVE

exp1 std13c

SAMPLE		DEC. & VT	
date	Nov 16 2010	dfrq	300.141
solvent	CDCl3	dn	H1
file	exp	dpwr	40
ACQUISITION		dof	400.0
sfrq	75.478	dm	yyy
tn	C13	dmm	w
at	1.777	dmf	8000
np	64000	dseq	
sw	18009.9	dres	1.0
fb	10000	homo	n
bs	8	PROCESSING	
tpwr	50	lb	1.00
pw	6.0	wtfile	
d1	2.000	proc	ft
tof	586.5	fn	not used
nt	3000	math	f
ct	2296		
alock	n	werr	
gain	60	wexp	
FLAGS		wbs	
il	n	wnt	
in	n		
dp	n		
hs	nn		
DISPLAY			
sp	-263.1		
wp	16874.9		
vs	148		
sc	0		
wc	250		
hzmm	67.50		
is	500.00		
rfl	853.9		
rfp	0		
th	11		
ins	100.000		
nm no ph			

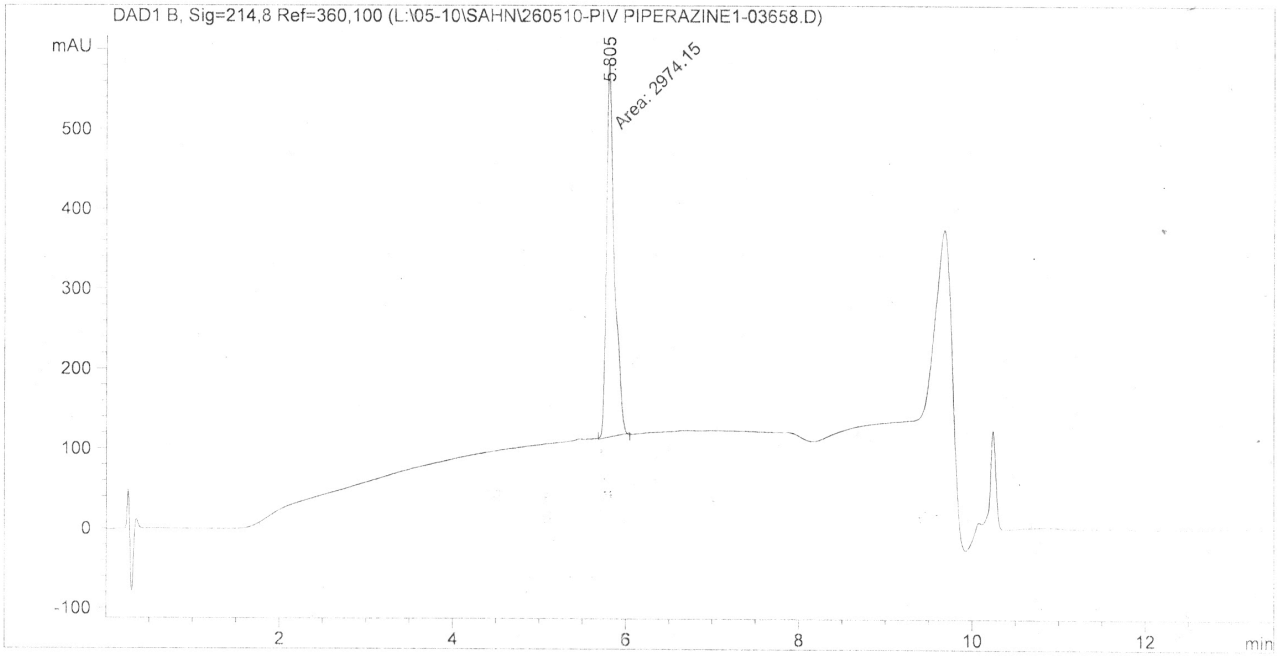


Data File L:\05-10\SAHN\260510-PIV PIPERAZINE1-03658.
Sample Name: piv piperazine



38{2,1,9}

```
==================================================
Acq. Operator    : sahn@mail.utexas.edu
Acq. Instrument  : LCMS
Injection Date   : 5/26/2010 11:59:35 PM

Acq. Method      : C:\CHEM32\1\METHODS\SP NIH.M
Last changed     : 5/26/2010 11:59:20 PM by sahn@mail.utexas.edu
                   (modified after loading)
Analysis Method  : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed     : 11/20/2006 4:14:44 AM
Sample Info      : Easy-Access Method: 'SP NIH'
```




```
================================================================================
                          Area Percent Report
================================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=214,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.805 MM    0.1055 2974.14893  469.95639 100.0000

Totals :                  2974.14893  469.95639

================================================================================
```

References:

[i] Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. "Safe and Convenient Procedure for Solvent Purification" *Organometallics* **1996**, *15*, 1518-1520.

ii. Still, W. C.; Kahn, M.; Mitra, A. "Rapid Chromatographic Technique for Preparative Separations with Moderate Resolution" *J. Org. Chem.* **1978**, *43*, 2923-2925.

[iii] For a representative procedure for Cbz removal of an anilino-norbenzomorphan, see text, reference 10.