Supporting Information

Gold(I)-Catalyzed Enantioselective Bromocyclization Reactions of Allenes

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General Information

Unless otherwise noted, all reagents were obtained commercially and used without purification. Gold(I) complexes were prepared according to the procedure of Toste[S1]. (*S*)-AgTRIP was prepared according to the procedure of Toste[S2]. Nitromethane ($MeNO_2$) (CHROMASOLV®, ≥96%) was obtained from Sigma-Aldrich. Toluene (99.98%) was obtained from EMD chemicals. Gold(I)-catalyzed reactions were performed in screw-cap vials under air without exclusion of moisture. Racemic samples were obtained using 5 mol % (±)-BINAP(AuPNB)$_2$ as catalyst.

Additional abbreviations. PNB = 4-nitrobenzoate; Ns = 4-nitrobenzenesulfonyl

Chromatography. Analytical thin layer chromatography was performed on EMD glass-backed TLC plates (silica gel 60 F254) and visualized by UV lamp (254 nm), anisaldehyde, or potassium permanganate. Column chromatography was carried out using Fisher 230-400 mesh, grade 60 silica gel. Purified compounds were further dried under high vacuum.

Nuclear magnetic resonance spectra. ^{1}H and ^{13}C spectra were recorded on Bruker AV-300, AVQ-400, or AVB-400 spectrometers. Chemical shifts (δ) are reported in ppm. ^{1}H NMR spectra were referenced to $CHCl_3$ (7.26 ppm) and ^{13}C NMR spectra were referenced to $CDCl_3$ (77.0 ppm). ^{13}C spectra were recorded with proton decoupling. Peak multiplicities are designated by the following abbreviations: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; br, broad; app, apparent. Coupling constants (*J*) are reported in units of Hz.

HPLC analyses. Chiral high performance liquid chromatography (HPLC) was performed on Shimadzu VP and Shimadzu prominence series instruments using the specified column (4.6 mm x 25 cm).

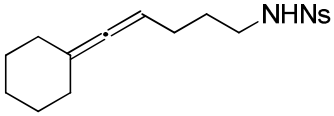
Mass spectrometry. Mass spectral data were obtained in the QB3/Chemistry Mass Spectrometry Facility, University of California, Berkeley.

X-ray crystallography. Data collection and analysis performed in the X-ray Crystallography Facility, College of Chemistry, University of California, Berkeley.

Synthesis of Allene Substrates

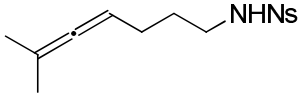
1a-**1e**, **1g**, **1h**, and **1m**-**1p** were synthesized according to the methods of Bäckvall[S3] starting from the corresponding propargyl alcohol. **1i** – **1k** were synthesized according to the method of Toste[S4] starting from the corresponding homoallenic alcohol. **1l** was synthesized according to the method of Toste[S4], using 4-nitrobenznesulfonyl chloride for the final protection step.

***N*-(5-cyclohexylidenepent-4-en-1-yl)-4-nitrobenzenesulfonamide** (**1a**)



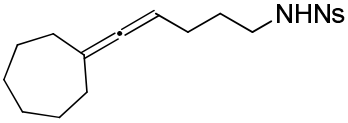
Purified by column chromatography on silica gel (hexanes:EtOAc 4:1) to afford **1a** (6.20 g, 77%) as a yellow solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.36 (d, *J* = 8.8, 2H), 8.05 (d, *J* = 8.8, 2H), 4.88-4.82 (m, 2H), 3.06 (q, *J* = 6.4, 2H), 2H), 2.02-1.92 (m, 6H), 1.62-1.49 (m, 8H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 198.4, 150.0, 146.1, 128.3, 124.4, 103.5, 87.1, 42.8, 31.6, 28.6, 27.4, 26.07,26.06; **HRMS** (ESI): found $[M+H]^+$ 351.1380 $C_{17}H_{23}N_2O_4{}^{32}S$ requires 351.1373

***N*-(6-methylhepta-4,5-dien-1-yl)-4-nitrobenzenesulfonamide** (**1b**)



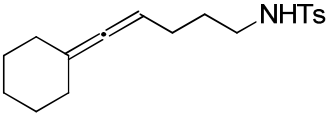
Purified by column chromatography on silica gel (hexanes:EtOAc 4:1) to afford **1b** (1.10 g, 77%) as a brown solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.36 (d, *J* = 8.8, 2H), 8.05 (d, *J* = 8.8, 2H), 4.90-4.82 (m, 2H), 3.05 (q, *J* = 6.4, 2H), 1.92 (q, *J* = 6.8, 2H), 1.62-1.54 (m, 8H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 201.8, 150.1, 146.1, 128.3, 124.3, 96.1, 87.2, 42.8, 28.7, 26.0, 20.6; **HRMS** (ESI): found $[M+H]^+$ 311.1060 $C_{14}H_{19}N_2O_4{}^{32}S$ requires 311.1060

***N*-(5-cycloheptylidenepent-4-en-1-yl)-4-nitrobenzenesulfonamide** (**1c**)



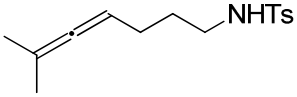
Purified by column chromatography on silica gel (hexanes:EtOAc 4:1) to afford **1c** (352 mg, 49%) as a yellow solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.36 (d, *J* = 9.2, 2H), 8.05 (d, *J* = 9.2, 2H), 4.89-4.84 (m, 1H), 4.75-4.71 (m, 1H), 3.07 (q, *J* = 6.8, 2H), 2.17-2.14 (m, 4H), 1.94 (q, *J* = 6.4, 2H), 1.62-1.51 (m, 10 H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 201.8, 150.0, 146.0, 128.3, 124.4, 105.4, 86.8, 42.8, 32.5, 29.3, 28.7, 28.4, 26.0; **HRMS** (ESI): found $[M+H]^+$ 365.1529 $C_{18}H_{25}N_2O_4{}^{32}S$ requires 365.1530

***N*-(5-cyclohexylidenepent-4-en-1-yl)-4-methylbenzenesulfonamide** (**1d**)



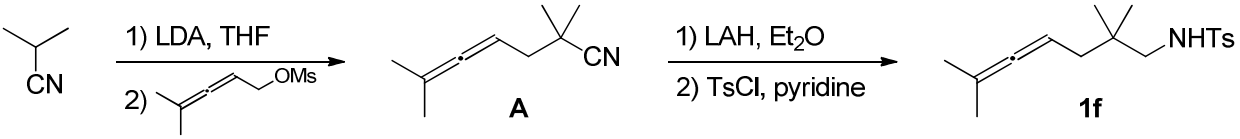
Purified by column chromatography on silica gel (hexanes:EtOAc 4:1) to afford **1d** (2.96 g, 48%) as a white solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.74 (d, *J* = 8.4, 2H), 7.30 (d, *J* = 8.0, 2H), 4.88-4.84 (m, 1H), 4.63-4.60 (m, 1H), 2.96 (q, *J* = 6.8, 2H), 2.42 (s, 3H), 2.02-1.90 (m, 6H), 1.59-1.48 (m, 10H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 198.3, 143.3, 137.0, 129.7,127.1, 103.3, 87.3, 42.6, 31.6, 28.6, 27.5, 26.2, 26.1, 21.6; **HRMS** (ESI): found $[M+H]^+$ 320.1684 $C_{18}H_{26}NO_2{}^{32}S$ requires 320.1679

4-methyl-*N*-(6-methylhepta-4,5-dien-1-yl)benzenesulfonamide (**1e**)



Purified by column chromatography on silica gel (hexanes:EtOAc 4:1) to afford **1e** (897 mg, 54%) as a yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.75 (d, *J* = 8.4, 2H), 7.30 (d, *J* = 8.4, 2H), 4.86-4.83 (m, 1H), 4.72-4.69 (m, 1H), 2.96 (q, *J* = 6.8, 2H), 2.42 (s, 3H), 1.91 (q, *J* = 6.8, 2H), 1.61 (d, *J* = 2.8, 6H), 1.54 (quintet, *J* = 7.2, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 201.7, 143.3, 136.9, 129.7, 127.1, 95.7, 87.4, 42.6, 28.7, 26.0, 21.5, 20.6; **HRMS** (ESI): found $[M+H]^+$ 280.1365 $C_{15}H_{22}NO_2{}^{32}S$ requires 280.1366

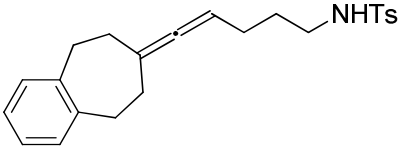
4-methyl-*N*-(2,2,6-trimethylhepta-4,5-dien-1-yl)benzenesulfonamide (**1f**)



Under a nitrogen atmosphere, a solution of *n*-butyllitium (7.1 mL, 13 mmol, 1.8 M in hexanes) was added dropwise to a solution of diisopropylamine (1.7 mL, 12 mmol) in dry THF (10 mL). The mixture was stirred for 15 min at 0°C and cooled to -78 °C. Isobutyronitrile (1.1 mL, 12 mmol) was added slowly and the mixture was stirred for 30 min at -78 °C. A solution of 4-methylpenta-2,3-dien-1-yl methanesulfonate[S5] (2.15 g, 12 mmol) in dry THF (10 mL) was added via cannula and the mixture was stirred for 14 h while gradually warming to r.t.. The mixture was quenched with sat. $NH_4Cl_{(aq)}$, extracted with ether, washed with brine, dried over $MgSO_4$, and concentrated *in vacuo*. The crude product purified by column chromatography on silica gel (hexanes:EtOAc 19:1) to afford **A** (310 mg, 17%) as a clear oil.

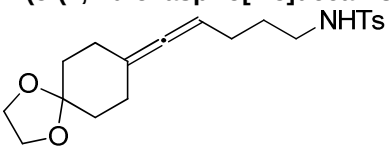
To a solution of **A** (268 mg, 1.80 mmol) in ether (3.7 mL) at 0 °C was added slightly crushed pellets of $LiAlH_4$ (82 mg, 2.2 mmol). The reaction was stirred for 2 h at r.t. under a nitrogen atmosphere after which the mixture was carefully quenched with 1 M $NaOH_{(aq)}$, the solids removed, dried over $MgSO_4$, and concentrated *in vacuo*. The crude product was dissolved in pyridine (1 mL) and cooled to 0 °C. 4-tolulenesulfonyl chloride (226 mg, 1.18 mmol) was added in one portion and the reaction was stirred for 14 h at r.t.. The mixture was extracted with ether, washed with $CuSO_{4(aq)}$, washed with brine, dried over $MgSO_4$, and concentrated *in vacuo*. The crude product was purified by column chromatography on silica gel (hexanes:EtOAc 4:1) to afford **1f** (147 mg, 61%) as a white solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.73 (d, *J* = 8.4, 2H), 7.29 (d, *J* = 8.0, 2H), 4.84-4.78 (m, 1H), 4.62-4.59 (m, 1H), 2.71 (d, *J* = 7.2, 2H), 2.42 (s, 3H), 1.83 (d, *J* = 8.0, 2H), 1.61 (d, *J* = 2.8, 6H), 0.86 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 203.3, 143.3, 137.1, 129.7, 127.1, 94.2, 84.2, 52.5, 39.8, 34.5, 24.8, 21.5, 20.5; **HRMS** (ESI): found $[M+H]^+$ 308.1676 $C_{17}H_{26}NO_2{}^{32}S$ requires 308.1679

***N*-(5-(8,9-dihydro-5H-benzo[7]annulen-7(6*H*)-ylidene)pent-4-en-1-yl)-4-methylbenzenesulfonamide** (**1g**)



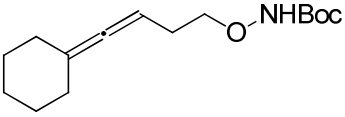
Purified by column chromatography on silica gel (hexanes:EtOAc 9:1) to afford **1g** (927 mg, 90%) as a white solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.76 (d, *J* = 8.4, 2H), 7.30 (d, *J* = 8.0, 2H), 7.13 (s, 4H), 4.93-4.92 (m, 1H), 4.66-4.63 (m, 1H), 2.98 (q, *J* = 6.8, 2H), 2.88-2.76 (m, 4H), 2.42 (s, 3H), 2.24-2.20 (m, 4H), 1.96 (q, *J* = 6.8, 2H), 1.56 (quintet, *J* = 7.2, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 200.8, 143.3, 142.3, 136.9, 129.7, 129.1, 127.1, 126.2, 106.2, 87.0, 42.6, 36.2, 33.6, 28.6, 26.1, 21.5; **HRMS** (ESI): found $[M+H]^+$ 382.1833 $C_{23}H_{28}NO_2{}^{32}S$ requires 382.1835

***N*-(5-(1,4-dioxaspiro[4.5]decan-8-ylidene)pent-4-en-1-yl)-4-methylbenzenesulfonamide** (**1h**)



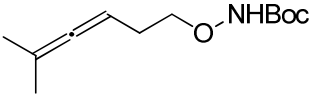
Purified by column chromatography on silica gel (hexanes:EtOAc 3:1) to afford **1h** (667 mg, 92%) as a colorless oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.73 (d, *J* = 8.4, 2H), 7.29 (d, *J* = 8.0, 2H), 4.92-4.89 (m, 1H), 4.67-4.64 (m, 1H), 3.94 (s, 4H), 2.95 (q, *J* = 6.8, 2H), 2.41 (s, 3H), 2.20-2.17 (m, 4H), 2.94 (q, *J* = 6.4, 2H), 1.70-1.66 (m, 4H), 1.54 (quintet, *J* = 7.2, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 198.6, 143.3, 137.0, 129.7, 127.1, 108.3, 100.7, 88.0, 64.3, 42.6, 35.4, 28.65, 28.60, 26.1, 21.5; **HRMS** (ESI): found $[M+H]^+$ 378.1730 $C_{21}H_{31}NNaO_4{}^{32}S$ requires 378.1734

***tert*-butyl (4-cyclohexylidenebut-3-en-1-yl)oxycarbamate** (**1i**)



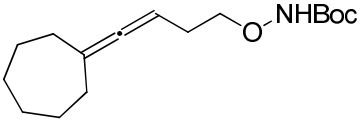
Purified by column chromatography on silica gel (hexanes:EtOAc 19:1) to afford **1i** (437 mg, 98%) as a white solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.14 (br s, 1H), 4.97-4.92 (m, 1H), 3.88 (t, *J* = 7.2, 2H), 2.29 (q, *J* = 6.8, 2H), 2.09-2.06 (m, 4H), 1.56-1.47 (m, 15H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 199.1, 156.9, 103.0, 84.4, 81.6, 76.1, 31.6, 28.2, 28.1, 27.4, 26.1; **HRMS** (ESI): found $[M+H]^+$ 268.1908 $C_{15}H_{26}NO_3$ requires 268.1907

***tert*-butyl (5-methylhexa-3,4-dien-1-yl)oxycarbamate** (**1j**)



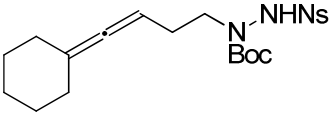
Purified by column chromatography on silica gel (hexanes:EtOAc 19:1) to afford **1j** (395 mg, 74%) as a colorless oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.21 (br s, 1H), 4.95-4.90 (m, 1H), 3.86 (t, *J* = 6.8, 2H), 2.26 (q, *J* = 7.2, 2H), 1.65 (d, *J* = 3.2, 6H), 1.46 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 202.4, 157.0, 95.6, 84.5, 81.6, 76.1, 28.2, 20.6; **HRMS** (ESI): found $[M+H]^+$ 228.1595 $C_{12}H_{22}NO_3$ requires 228.1594

***tert*-butyl (4-cycloheptylidenebut-3-en-1-yl)oxycarbamate** (**1k**)



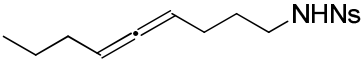
Purified by column chromatography on silica gel (hexanes:EtOAc 19:1) to afford **1k** (764 mg, 57%) as a yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.21 (br s, 1H), 4.94-4.91 (m, 1H), 3.87 (t, *J* = 7.2, 2H), 2.27 (q, *J* = 6.8, 2H), 2.21-2.18 (m, 4H), 1.58-1.46 (m, 17H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 202.4, 156.9, 104.9, 84.0, 81.5, 76.1, 32.5, 29.3, 28.4, 28.2, 27.9; **HRMS** (ESI): found $[M+H]^+$ 282.2064 $C_{16}H_{28}NO_3$ requires 282.2064

***tert*-butyl 1-(4-cyclohexylidenebut-3-en-1-yl)-2-((4-nitrophenyl)sulfonyl)hydrazinecarboxylate** (**1l**)



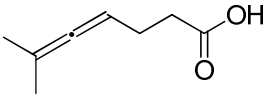
Purified by column chromatography on silica gel (hexanes:EtOAc 6:1) to afford **1l** (181 mg, 59%) as a white solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.32 (d, *J* = 8.4, 2H), 8.08 (d, *J* = 9.2, 2H), 6.94 (br s, 1H), 4.83-4.81 (m, 1H), 3.65 (br s, 2H), 2.28-2.24 (m, 2H), 2.05-2.03 (m, 4H), 1.55-1.49 (m, 6H), 1.18 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 199.3, 150.5, 130.1, 123.8, 103.1, 84.8, 82.9, 31.4, 27.7, 27.4, 26.1; **HRMS** (ESI): found $[M+Na]^+$ 474.1667 $C_{21}H_{29}N_3NaO_6{}^{32}S$ requires 474.1669

4-nitro-*N*-(nona-4,5-dien-1-yl)benzenesulfonamide (**1m**)



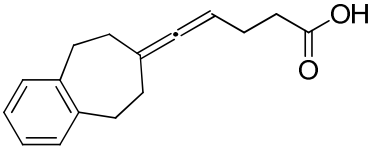
Purified by column chromatography on silica gel (hexanes:EtOAc 4:1) to afford **1m** (1.07 g, 36%) as a brown solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.36 (d, *J* = 8.4, 2H), 8.06(d, *J* = 8.8, 2H), 5.10-4.96 (m, 3H), 3.04 (q, *J* = 6.4, 2H), 1.98-1.86 (m, 4H), 1.59 (quintet, *J* = 6.8, 2H), 1.37 (sextet, *J* = 7.6, 2H), 1.86 (t, *J* = 7.6, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 203.9, 150.0, 145.9, 128.3, 124.4, 91.8, 89.2, 42.7, 30.9, 28.7, 25.6, 22.3, 13.6; **HRMS** (ESI): found $[M+H]^+$ 325.1221 $C_{15}H_{21}N_2O_4{}^{32}S$ requires 325.1217

6-methylhepta-4,5-dienoic acid (**1n**)



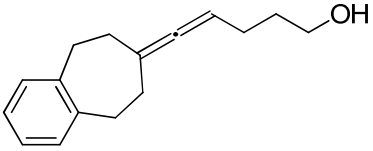
Purified by acid-base extraction to afford **1n** (3.83 g, 88%) as a yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 10.79 (br s, 1H), 5.04-4.98 (m, 1H), 2.44 (t, *J* = 7.2, 2H), 2.26 (q, *J* = 6.0, 2H), 1.65 (d, *J* = 2.8, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 201.5, 180.0, 97.2, 87.2, 33.0, 23.8, 20.5; **HRMS** (ESI): found $[M-H]^-$ 139.0764 $C_8H_{11}O_2$ requires 139.0765

5-(8,9-dihydro-5H-benzo[7]annulen-7(6*H*)-ylidene)pent-4-enoic acid (**1o**)



Purified by column chromatography on silica gel (hexanes:EtOAc 3:1) to afford **1o** (167 mg, 56%) as a white solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 10.62 (br s, 1H), 7.13 (s, 4H), 5.10-5.09 (m, 1H), 2.88-2.85 (m, 4H), 2.49 (t, *J* = 7.2, 2H), 2.34-2.26 (m, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 200.5, 179.6, 142.4, 129.1, 126.2, 107.5, 86.8, 36.1, 33.6, 33.0, 23.8; **HRMS** (ESI): found $[M-H]^-$ 241.1234 $C_{16}H_{17}O_2$ requires 241.1234

5-(8,9-dihydro-5H-benzo[7]annulen-7(6*H*)-ylidene)pent-4-en-1-ol (**1p**)



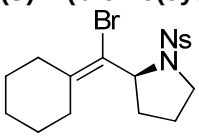
Purified by column chromatography on silica gel (hexanes:EtOAc 5:1) to afford **1p** (147 mg, 82%) as a white solid; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 7.14 (s, 4H), 5.10-4.90 (m, 1H), 3.70 (t, *J* = 8.8, 2H), 2.89-2.86 (m, 4H), 2.31-2.28 (m, 4H), 2.08 (q, *J* = 9.2, 2H), 1.69 3.70 (quart., *J* = 9.6, 2H), 1.41 (br s, 1H); **^{13}C NMR** (75.5 MHz, $CDCl_3$): δ 201.1, 142.7, 129.4, 126.5, 106.4, 88.0, 62.7, 36.6, 34.1, 32.2, 25.8; **HRMS** (ESI): found $[M+H]^+$ 229.1589 $C_{16}H_{21}O$ requires 229.1587

Gold(I)-Catalyzed Enantioselective Bromocyclizations

General Procedure **A:**
Unless otherwise noted, in a one-dram vial, to the specified dinuclear gold(I) catalyst (0.005 mmol) and **6a** (33 mg, 0.20 mmol) was added the specified allene (0.10 mmol) as a solution in $MeNO_2$ (0.5 mL). The mixture was stirred in the dark for the specified time at r.t. at which point the solvent was evaporated under a stream of nitrogen. The residue was purified by column chromatography on silica gel with the specified mobile phase to afford the corresponding vinyl bromide.

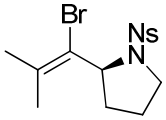
General Procedure **B:**
In a one-dram vial, to (*R*)-DM-BINAP(AuPNB)$_2$ (7.3 mg, 0.005 mmol) and **6c** (31 mg, 0.15 mmol) was added the specified allene (0.10 mmol) as a solution in $MeNO_2$ (0.5 mL). The mixture was stirred in the dark for 2 h at r.t. at which point the solvent was evaporated under a stream of nitrogen. The residue was purified by column chromatography on silica gel (pentanes:Et_2O 9:1 to 4:1) to afford the corresponding vinyl bromide.

(*S*)-2-(bromo(cyclohexylidene)methyl)-1-((4-nitrophenyl)sulfonyl)pyrrolidine (**3a**)



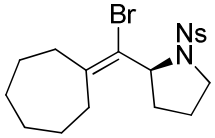
General procedure **A** was used. **L**(AuPNB)$_2$: (*R*)-DM-BINAP(AuPNB)$_2$ (7.3 mg, 0.005 mmol); allene: **1a** (35 mg, 0.10 mmol); time: 12 h; mobile phase (pentanes:DCM 1:1); afforded **3a** (38 mg, 88%) as a white solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.30 (d, *J* = 8.8, 2H), 7.95 (d, *J* = 8.8, 2H), 5.15 (app t, 1H), 3.86-3.81 (m, 1H), 3.42-3.35 (m, 1H), 2.54-2.48 (m, 1H), 2.40-1.96 (m, 6H), 1.87-1.82 (m,1H), 1.73-1.59 (m, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 149.7, 146.2, 142.4, 128.3, 123.9, 121.1, 60.2, 49.5, 35.7, 32.6, 32.1, 27.7, 27.1, 26.3, 25.5; **HRMS** (ESI): found $[M+H]^+$ 429.0476 $C_{17}H_{22}{}^{79}BrN_2O_4{}^{32}S$ requires 429.0478; **HPLC** Chiralpak IA column (98:2 hexanes:isopropanol, 1.0 mL/min, 250 nm); major enantiomer t_r = 15.24 min, minor enantiomer t_r = 17.07 min, 99% ee; absolute configuration assigned as (*S*) by analogy to **3b**.

(*S*)-2-(1-bromo-2-methylprop-1-en-1-yl)-1-((4-nitrophenyl)sulfonyl)pyrrolidine (**3b**)



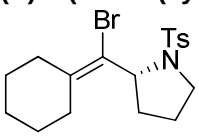
General procedure **A** was used. **L**(AuPNB)$_2$: (*R*)-Cl-MeO-BIPHEP(AuPNB)$_2$ (6.9 mg, 0.005 mmol); allene: **1b** (31 mg, 0.10 mmol); time: 13 h; mobile phase (pentanes:Et_2O 9:1 to 4:1); afforded **3b** (35 mg, 89%) as a white solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.30 (d, *J* = 8.8, 2H), 7.91 (d, *J* = 8.8, 2H), 5.05 (app t, 1H), 3.85-3.79 (m, 1H), 3.42-3.36 (m, 1H), 2.21-2.05 (m, 2H), 2.01-1.84 (m, 2H), 1.99 (s, 3H), 1.83 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 149.7, 146.1, 135.3, 128.3, 123.8, 123.4, 60.8, 49.4, 32.3, 25.8, 25.4, 21.1; **HRMS** (EI): found $[M]^+$ 388.0093 $C_{14}H_{17}{}^{79}BrN_2O_4{}^{32}S$ requires 388.0092; **HPLC** Chiralpak IA column (97:3 hexanes:isopropanol, 1.0 mL/min, 250 nm); major enantiomer t_r = 12.90 min, minor enantiomer t_r = 13.94 min, 98% ee; absolute configuration assigned as (*S*) by X-ray crystal structure analysis of **3b**; crystals obtained by slow evaporation of toluene solution or by MTBE/hexane vapor diffusion.

(*S*)-2-(bromo(cycloheptylidene)methyl)-1-((4-nitrophenyl)sulfonyl)pyrrolidine (**3c**)



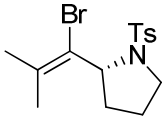
General procedure **A** was used. **L**(AuPNB)$_2$: (*R*)-Cl-MeO-BIPHEP(AuPNB)$_2$ (6.9 mg, 0.005 mmol); allene: **1c** (36 mg, 0.10 mmol); time: 13 h; mobile phase (pentanes:Et_2O 9:1 to 4:1); afforded **3c** (41 mg, 93%) as a white solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.29 (d, *J* = 8.8, 2H), 7.94 (d, *J* = 8.8, 2H), 5.08 (app t, 1H), 3.85-3.80 (m, 1H), 3.41-3.35 (m, 1H), 2.67-2.61 (m, 1H), 2.47-2.33 (m, 2H), 2.21-1.75 (m, 6H), 1.67-1.34 (m, 7H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 149.7, 146.1, 143.8, 128.3, 124.2, 123.8, 60.5, 49.5, 37.1, 32.8, 32.3, 28.6, 28.4, 27.8, 26.1, 25.5; **HRMS** (ESI): found $[M+H]^+$ 443.0633 $C_{18}H_{24}{}^{79}BrN_2O_4{}^{32}S$ requires 443.0635; **HPLC** Chiralpak IA column (98:2 hexanes:isopropanol, 1.0 mL/min, 250 nm); major enantiomer t_r = 14.69 min, minor enantiomer t_r = 17.72 min, 99% ee; absolute configuration assigned as (*S*) by analogy to **3b**.

(*R*)-2-(bromo(cyclohexylidene)methyl)-1-tosylpyrrolidine (**3d**)



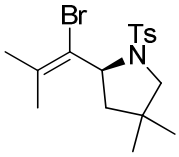
General procedure **A** was used. **L**(AuPNB)$_2$: (*S*)-DM-BINAP(AuPNB)$_2$ (7.3 mg, 0.005 mmol); allene: **1d** (32 mg, 0.10 mmol); time: 13 h; purified by preparatory TLC (hexanes:EtOAc 3:1); afforded **3d** (37 mg, 93%) as a white solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.70 (d, *J* = 8.4, 2H), 7.28 (d, *J* = 7.2, 2H), 4.94 (app t, 1H), 3.67-3.63 (m, 1H), 3.43-3.37 (m, 1H), 2.45-2.32 (m, 4H), 2.42 (s, 3H), 2.02-1.99 (m, 3H), 1.79-1.45 (m, 7H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 143.0, 140.8, 136.7, 129.3, 127.3, 122.2, 59.7, 49.4, 35.8, 32.6, 32.1, 27.6, 27.0, 26.4, 25.2, 21.5; **HRMS** (ESI): found $[M+H]^+$ 398.0784 $C_{18}H_{25}{}^{79}BrNO_2{}^{32}S$ requires 398.0784; **HPLC** Chiralpak IA column (98:2 hexanes:isopropanol, 1.0 mL/min, 250 nm); major enantiomer t_r = 17.50 min, minor enantiomer t_r = 15.99 min, 98% ee; absolute configuration assigned as (*R*) by analogy to **3b**.

(*R*)-2-(1-bromo-2-methylprop-1-en-1-yl)-1-tosylpyrrolidine (**3e**)



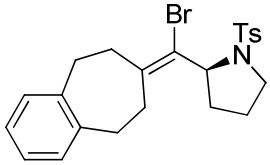
General procedure **A** was used. **L**(AuPNB)$_2$: (*S*)-DM-BINAP(AuPNB)$_2$ (7.3 mg, 0.005 mmol); allene: **1e** (28 mg, 0.10 mmol); time: 14 h; mobile phase (pentane:Et_2O 5:1); afforded **3e** (32 mg, 89%) as a white solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.68 (d, *J* = 8.4, 2H), 7.27 (d, *J* = 8.0, 2H), 4.86 (app t, 1H), 3.66-3.61 (m, 1H), 3.42-3.36 (m, 1H), 2.41 (s, 3H), 2.08-1.92 (m, 3H), 1.91 (s, 3H), 1.80 (s, 3H), 1.63-1.60 (m, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 143.1, 136.7, 133.6, 129.3, 127.4, 124.4, 60.5, 49.3, 32.3, 25.8, 25.2, 21.6, 21.0; **HRMS** (ESI): found $[M+H]^+$ 358.0467 $C_{15}H_{21}{}^{79}BrNO_2{}^{32}S$ requires 358.0471; **HPLC** Chiralpak IA column (98:2 hexanes:isopropanol, 1.0 mL/min, 250 nm); major enantiomer t_r = 21.12 min, minor enantiomer t_r = 15.85 min, 99% ee; absolute configuration assigned as (*R*) by analogy to **3b**.

(*S*)-2-(1-bromo-2-methylprop-1-en-1-yl)-4,4-dimethyl-1-tosylpyrrolidine (**3f**)



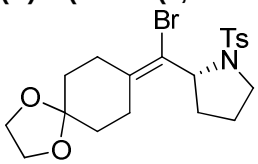
General procedure **A** was used except the amounts of **6a** (41 mg, 0.25 mmol) and $MeNO_2$ (0.625 mL) were increased proportionally to **1f**. **L**(AuPNB)$_2$: (*R*)-Cl-MeO-BIPHEP(AuPNB)$_2$ (8.6 mg, 0.0063 mmol); allene: **1f** (38 mg, 0.13 mmol); time: 14h; mobile phase (pentanes:Et_2O 9:1 to 4:1); afforded **3f** (42 mg, 87%) as a white solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.65 (d, *J* = 8.4, 2H), 7.24 (d, *J* = 8.0, 2H), 4.96 (dd, *J* = 8.8, 7.6, 1H), 3.47 (dd, *J* = 10.0, 1.2, 1H), 3.12 (d, *J* = 10, 1H), 2.40 (s, 3H), 1.92 (s, 3H), 1.90-1.86 (m, 1H), 1.76 (s, 3H), 1.74-1.71 (m, 1H), 1.07 (s, 3H), 0.94 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 142.8, 137.7, 133.9, 129.1, 127.2, 124.6, 60.7, 60.4, 45.7, 38.1, 26.3, 25.8, 25.3, 21.6, 21.0; **HRMS** (ESI): found $[M+H]^+$ 386.0780 $C_{17}H_{25}{}^{79}BrNO_2{}^{32}S$ requires 386.0784; **HPLC** Chiralpak IA column (98:2 hexanes:isopropanol, 1.0 mL/min, 250 nm); major enantiomer t_r = 13.92 min, minor enantiomer t_r = 11.72 min, 91% ee; absolute configuration assigned as (*S*) by analogy to **3b**.

(*S*)-2-(bromo(8,9-dihydro-5H-benzo[7]annulen-7(6*H*)-ylidene)methyl)-1-tosylpyrrolidine (**3g**)



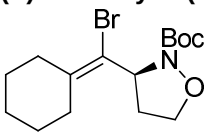
General procedure **A** was used. **L**(AuPNB)$_2$: (*R*)-Cl-MeO-BIPHEP(AuPNB)$_2$ (6.9 mg, 0.005 mmol); allene: **1g** (38 mg, 0.10 mmol); time: 14 h; mobile phase (pentanes:Et_2O 4:1); afforded **3g** (40 mg, 86%) as a white solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.69 (d, *J* = 8.4, 2H), 7.26 (d, *J* = 8.0, 2H), 7.14 (s, 4H), 5.00 (app t, 1H), 3.71-3.66 (m, 1H), 3.45-3.39 (m, 1H), 3.11-3.04 (m, 1H), 2.86-7.32 (m, 3H), 2.66-2.63 (m, 2H), 2.58-2.56 (m, 2H), 2.41 (s, 3H), 2.06-1.93 (m, 3H), 1.65-1.62 (m, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 143.2, 142.5, 141.6, 141.2, 136.8, 129.5, 129.42, 129.39, 127.4, 126.4, 126.3, 125.4, 60.1, 49.5, 37.5, 35.1, 34.2, 33.0, 32.9, 25.3, 21.6; **HRMS** (ESI): found $[M+H]^+$ 460.0938 $C_{23}H_{27}{}^{79}BrNO_2{}^{32}S$ requires 460.0940; **HPLC** Chiralpak AD-H column (95:5 hexanes:isopropanol, 1.0 mL/min, 250 nm); major enantiomer t_r = 18.11 min, minor enantiomer t_r = 15.98 min, 95% ee; absolute configuration assigned as (*S*) by analogy to **3b**.

(*R*)-2-(bromo(1,4-dioxaspiro[4.5]decan-8-ylidene)methyl)-1-tosylpyrrolidine (**3h**)



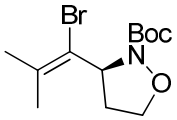
General procedure **A** was used except the amounts of **6a** (36 mg, 0.22 mmol) and $MeNO_2$ (0.54 mL) were increased proportionally to **1h**. **L**$(AuPNB)_2$: (*S*)-BINAP$(AuPNB)_2$ (7.3 mg, 0.0054 mmol); allene: **1h** (41 mg, 0.11 mmol); time: 14 h; mobile phase (pentanes:Et_2O 1:1); afforded **3h** (37 mg, 74%) as a white solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.68 (d, *J* = 8.0, 2H), 7.26 (d, *J* = 8.4, 2H), 4.92 (app t, 1H), 3.96 (s, 4H), 3.70-3.64 (m, 1H), 3.42-3.36 (m, 1H), 2.68-2.61 (m, 1H), 2.57-2.52 (m, 1H), 2.47-2.41 (m, 5H), 2.06-1.99 (m, 3H), 1.88-1.82 (m, 1H), 1.73-1.62 (m, 4H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 143.1, 138.4, 136.9, 129.4, 127.3, 123.4, 108.3, 64.45, 64.43, 60.0, 49.4, 35.1, 34.7, 32.7, 32.5, 28.3, 25.3, 21.6; **HRMS** (ESI): found $[M+H]^+$ 456.0836 $C_{20}H_{27}{}^{79}BrNO_4{}^{32}S$ requires 456.0839; **HPLC** Chiralpak AD-H column (90:10 hexanes:isopropanol, 1.0 mL/min, 250 nm); major enantiomer t_r = 19.40 min, minor enantiomer t_r = 20.57 min, 93% ee; absolute configuration assigned as (*R*) by analogy to **3b**.

(*S*)-tert-butyl 3-(bromo(cyclohexylidene)methyl)isoxazolidine-2-carboxylate (**3i**)



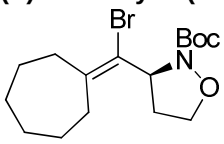
General procedure **B** was used. Allene: **1i** (27 mg, 0.10 mmol); afforded **3i** (26 mg, 75%) as a white solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 5.27 (app t, 1H), 4.21 (td, *J* = 7.6, 2.8, 1H), 3.72-3.66 (m, 1H), 2.50-2.35 (m, 6H), 1.65-1.52 (m, 15H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 156.8, 141.2, 119.6, 81.9, 69.2, 59.4, 35.8, 35.4, 31.8, 28.3, 27.9, 27.1, 26.3; **HRMS** (ESI): found $[M+H]^+$ 346.1010 $C_{15}H_{25}{}^{79}BrNO_3$ requires 346.1012; **HPLC** Regis Whelk-O1 column (98:2 hexanes:isopropanol, 1.0 mL/min, 210 nm); major enantiomer t_r = 25.92 min, minor enantiomer t_r = 21.05 min, 91% ee; absolute configuration assigned as (*S*) by analogy to **3b**.

(*S*)-tert-butyl 3-(1-bromo-2-methylprop-1-en-1-yl)isoxazolidine-2-carboxylate (**3j**)



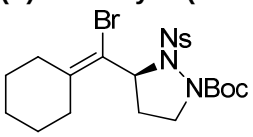
General procedure **B** was used. Allene: **1j** (23 mg, 0.10 mmol); afforded **3j** (26 mg, 83%) as a white solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 5.18 (dd, *J* = 8.8, 7.2, 1H), 4.21 (td *J* = 8.0, 2.4, 1H), 3.74-3.68 (m, 1H), 2.44-2.37 (m, 2H), 1.95 (s, 3H), 1.91 (s, 3H), 1.48 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 156.7, 133.9, 121.8, 81.9, 69.2, 60.0, 35.0, 28.3, 25.9, 20.8; **HRMS** (ESI): found $[M+Na]^+$ 328.0516 $C_{12}H_{20}{}^{79}BrNNaO_3$ requires 328.0519; **HPLC** Regis Whelk-O1 column (98:2 hexanes:isopropanol, 1.0 mL/min, 210 nm); major enantiomer t_r = 27.03 min, minor enantiomer t_r = 23.76 min, 88% ee; absolute configuration assigned as (*S*) by analogy to **3b**.

(*S*)-tert-butyl 3-(bromo(cycloheptylidene)methyl)isoxazolidine-2-carboxylate (**3k**)



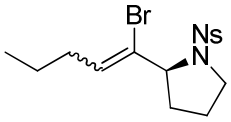
General procedure **B** was used. Allene: **1k** (28 mg, 0.10 mmol); afforded **3k** (32 mg, 88%) as a white solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 5.22 (app t, 1H), 4.21 (td, *J* = 8.0, 2.4, 1H), 3.73-3.67 (m, 1H), 2.67-2.54 (m, 2H), 2.44-2.33 (m, 4H), 1.83-1.72 (m, 1H), 1.63-1.34 (m, 16H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 156.7, 142.5, 122.7, 81.9, 69.2, 59.7, 37.2, 35.4, 32.1, 28.5, 28.2, 27.8, 26.1; **HRMS** (ESI): found $[M+H]^+$ 360.1165 $C_{16}H_{27}{}^{79}BrNO_3$ requires 360.1169; **HPLC** Regis Whelk-O1 column (98:2 hexanes:isopropanol, 1.0 mL/min, 210 nm); major enantiomer t_r = 23.90 min, minor enantiomer t_r = 19.45 min, 91% ee; absolute configuration assigned as (*S*) by analogy to **3b**.

(*S*)-tert-butyl 3-(bromo(cyclohexylidene)methyl)-2-((4-nitrophenyl)sulfonyl)pyrazolidine-1-carboxylate (**3l**)



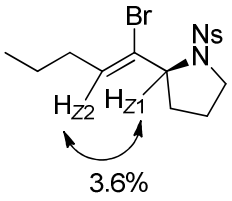
In a one-dram vial, to (*R*)-DM-BINAP(AuPNB)$_2$ (7.3 mg, 0.005 mmol) and **6b** (29 mg, 0.15 mmol) was added allene **1l** (45mg, 0.10 mmol) as a solution in $MeNO_2$ (0.5 mL). The mixture was stirred in the dark for 17 h at r.t. at which point the solvent was evaporated under a stream of nitrogen. The residue was purified by column chromatography on silica gel (pentanes:Et_2O 9:1 to 4:1) to afford **3l** (42 mg, 80%) as a white foam; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.35 (d, *J* = 8.8, 2H), 8.15 (d, *J* = 9.2, 2H), 5.56 (app t, 1H), 4.25-4.21 (m, 1H), 3.23-3.14 (m, 1H), 2.58-2.20 (m, 6H), 1.74-1.50 (m, 6H), 1.19 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 150.6, 143.9, 142.4, 130.9, 123.8, 117.3, 82.0, 47.9, 35.9, 32.2, 27.8, 27.2, 26.3; **HRMS** (ESI): found $[M+Na]^+$ 552.0773 $C_{21}H_{28}{}^{79}BrN_3NaO_6{}^{32}S$ requires 552.0774; **HPLC** Chiralpak IA column (98:2 hexanes:isopropanol, 1.0 mL/min, 232 nm); major enantiomer t_r = 16.30 min, minor enantiomer t_r = 15.58 min, 96% ee; absolute configuration assigned as (*S*) by analogy to **3b**.

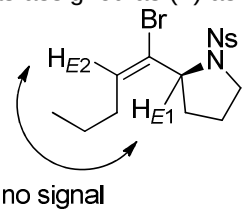
(*S*)-2-(1-bromopent-1-en-1-yl)-1-((4-nitrophenyl)sulfonyl)pyrrolidine (**3m**)



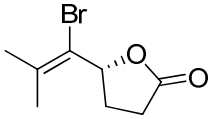
General procedure **A** was used. **L**(AuPNB)$_2$: (*R*)-DM-BINAP(AuPNB)$_2$ (7.3 mg, 0.005 mmol); allene: **1m** (32 mg, 0.10 mmol); time: 18 h; mobile phase (hexanes:Et_2O 4:1); afforded **3m** (30 mg, 74%) as a turbid oil; **^{1}H NMR** (400 MHz, $CDCl_3$): *Z* isomer: δ 8.37 (d, *J* = 8.8, 2H), 8.02 (d, *J* = 8.8, 2H), 6.07 (t, *J* = 6.8, 1H), 4.59-4.56 (m, 1H), 3.67-3.62 (m, 1H), 3.53-3.47 (m, 1H); *E* isomer: δ 8.37 (d, *J* = 8.8, 2H), 8.04 (d, *J* = 8.8, 2H), 5.96 (t, *J* = 7.6, 1H), 5.01-4.97 (m, 1H), 3.87-3.80 (m, 1H), 3.44-3.35 (m, 1H); Unassigned: 2.31-2.21 (m, 1H), 2.15-2.02 (m, 4H), 1.90-1.84 (m, 1H), 1.56-1.43 (m, 2H), 1.01-0.95 (m, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): *Z* isomer: δ 149.91, 145.2, 132.3, 128.5, 127.7, 124.1, 66.4, 49.5, 32.9, 32.2, 24.4, 21.5, 13.8; *E* isomer: δ 149.86, 145.8, 136.5, 128.4, 126.2, 124.0, 59.3, 49.4, 32.3, 31.7, 25.4, 22.4, 13.7.
HRMS (ESI): submitted; **HPLC** Chiralpak IA column (98:2 hexanes:isopropanol, 1.0 mL/min, 250 nm); major enantiomer (*E*) t_r = 14.08 min, minor enantiomer (*E*) t_r = 13.53 min, 91% ee; major enantiomer (*Z*) t_r = 18.92 min, minor enantiomer (*Z*) t_r = 16.35 min, 26% ee; absolute configuration assigned as (*S*) by analogy to **3b**.

NOE experiments:
The diastereomeric assignment of **3m** was performed by irradiation of the allylic proton (H_1), which only gave enhancement in one case; this was assigned as (*Z*) as the H_1-H_2 distance is smaller on average.



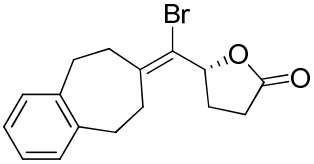


(*R*)-5-(1-bromo-2-methylprop-1-en-1-yl)dihydrofuran-2(3*H*)-one (**3n**)



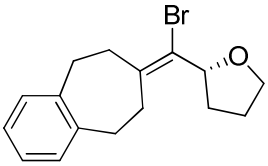
In a one-dram vial, a mixture of (*R*)-DTBM-BINAP(AuCl)$_2$ (4.2 mg, 0.0025 mmol), (*S*)-AgTRIP (4.3 mg, 0.005 mmol), and toluene (0.5 mL) was sonicated for 10 min at r.t. at which point **6b** (33 mg, 0.17 mmol) was added and the mixture cooled to -35 °C. A solution of **1n** (14 mg, 0.10 mmol) in toluene (0.5 mL) was added and the mixture stirred for 5 min at -35 °C. The mixture then remained in the dark at -35 °C for 72 h without stirring at which point it was warmed to r.t. and quenched with triethylamine (0.25 mL). The solvent was evaporated under a stream of nitrogen and the residue purified by column chromatography on silica gel (pentanes:Et_2O 2:1 to 1:1) to afford **3n** (20 mg, 90%) as a white solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 5.51 (dd, *J* = 7.6, 6.8, 1H), 2.79-2.70 (m, 1H), 2.59-2.50 (m, 1H), 2.40-2.26 (m, 2H), 1.94 (s, 3H), 1.92 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 176.7, 137.6, 120.4, 77.6, 28.5, 27.0, 25.9, 21.1; **HRMS** (EI): found $[M]^+$ 217.9938 $C_8H_{11}{}^{79}BrO_2$ requires 217.9942; **HPLC** Chiralpak IA column (96:4 hexanes:isopropanol, 1.0 mL/min, 210 nm); major enantiomer t_r = 14.89 min, minor enantiomer t_r = 13.91 min, 95% ee; absolute configuration assigned as (*R*) by analogy to (*S*)-5-(2-methylprop-1-en-1-yl)dihydrofuran-2(3*H*)-one listed in reference S2.

(*R*)-5-(bromo(8,9-dihydro-5H-benzo[7]annulen-7(6*H*)-ylidene)methyl)dihydrofuran-2(3*H*)-one (**3o**)



In a one-dram vial, a mixture of (*R*)-DTBM-BINAP(AuCl)$_2$ (4.2 mg, 0.0025 mmol), (*S*)-AgTRIP (4.3 mg, 0.005 mmol), and toluene (0.5 mL) was sonicated for 10 min at r.t. at which point **6b** (33 mg, 0.17 mmol) was added and the mixture cooled to -35 °C. A solution of **1o** (24 mg, 0.10 mmol) in toluene (0.5 mL) was added and the mixture stirred for 5 min at -35 °C. The mixture then remained in the dark at -35 °C for 72 h without stirring at which point it was warmed to r.t. and quenched with triethylamine (0.25 mL). The solvent was evaporated under a stream of nitrogen and the residue purified by column chromatography on silica gel (pentanes:Et_2O 2:1 to 1:1) to afford **3o** (28 mg, 86%) as a white solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.16-7.11 (m, 4H), 5.65 (dd, *J* = 8.0, 6.4, 1H), 2.93-2.53 (m, 10H), 2.38-2.35 (m, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 176.7, 146.5, 141.1, 140.5, 129.5, 129.4, 126.6, 126.4, 121.0, 76.98, 37.4, 35.2, 34.0, 33.2, 28.5, 27.2; **HRMS** (ESI): found $[M+H]^+$ 321.0484 $C_{16}H_{18}{}^{79}BrO_2$ requires 321.0485; **HPLC** Chiralpak IA column (95:5 hexanes:isopropanol, 1.0 mL/min, 210 nm); major enantiomer t_r = 17.52 min, minor enantiomer t_r = 16.50 min, 98% ee; absolute configuration assigned as (*R*) by analogy to (*S*)-5-(2-methylprop-1-en-1-yl)dihydrofuran-2(3*H*)-one listed in reference S2.

(*R*)-2-(bromo(8,9-dihydro-5H-benzo[7]annulen-7(6*H*)-ylidene)methyl)tetrahydrofuran (**3p**)

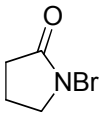


In a one-dram vial, a mixture of (*R*)-DTBM-BINAP(AuCl)$_2$ (8.3 mg, 0.005 mmol), (*S*)-AgTRIP (8.6 mg, 0.01 mmol), and toluene (0.5 mL) was sonicated for 10 min at r.t. at which point **6b** (33 mg, 0.17 mmol) was added and the mixture cooled to -35 °C. A solution of **1p** (23 mg, 0.10 mmol) in toluene (0.5 mL) was added and the mixture stirred for 5 min at -35 °C. The mixture then remained in the dark at -35 °C for 72 h without stirring at which point it was warmed to r.t. and quenched with triethylamine (0.25 mL). The solvent was evaporated under a stream of nitrogen and the residue purified by column chromatography on silica gel (pentanes:Et_2O 9:1 to 4:1) to afford **3p** (26 mg, 85%) as a white solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.15-7.11 (m, 4H), 4.97 (app t, 1H), 4.07-4.02 (m, 1H), 3.89-3.84 (m, 1H), 2.92-2.53 (m, 8H), 2.11-2.03 (m, 1H), 2.01-1.88 (m, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 143.4, 141.7, 141.1, 129.5, 129.4, 126.4, 126.2, 125.2, 69.1, 37.6, 35.6, 34.5, 33.2, 31.7, 26.7; **HRMS** (ESI): found $[M+H]^+$ 307.0692 $C_{16}H_{20}{}^{79}BrO$ requires 307.0692; **HPLC** Chiralpak IC column (98:2 hexanes:isopropanol, 1.0 mL/min, 210 nm); major enantiomer t_r = 9.46 min, minor enantiomer t_r = 10.55 min, 86% ee; absolute configuration assigned as (*R*) by analogy to (*S*)-5-(2-methylprop-1-en-1-yl)dihydrofuran-2(3*H*)-one listed in reference S2.

Synthesis of *N*-Bromolactams

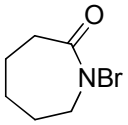
The syntheses of compounds **6** were developed using reference S6 as a guide. In a sealed vial at -30 °C and protected from ambient light, the lactams are indefinitely stable. Yellowing of the solid occurs at r.t. and in ambient light. 1-bromoazonan-2-one could be prepared in an analogous manner to **6c**. 1-bromopiperidin-2-one could not be prepared using the methods described below.

1-bromopyrrolidin-2-one (**6a**)



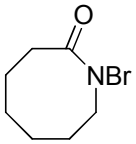
To a mixture of pyrrolidin-2-one (2.00 g, 23.5 mmol), sodium bromate (1.77 g, 11.8 mmol), and concentrated sulfuric acid (0.313 mL, 5.87 mmol) in water (7 mL) was added hydrobromic acid (1.78 mL, 15.7 mmol, 48% w/w in water) dropwise over a period of 5 min. The mixture was then capped and allowed to stir for 1 h at r.t. during which the mixture lightened from dark red to a pale yellow color. The mixture was then cooled to -78 °C for 5 min to induce precipitation and was warmed to r.t.. The solid was collected on a fritted funnel and washed twice with 20 mL of cold water and allowed to dry under a stream of nitrogen while pulling vacuum. The solid was then placed under high vacuum for 4 h to afford **6a** (1.96 g, 51%) as a pale yellow solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 3.58 (t, *J* = 6.8, 2H), 2.37 (t, *J* = 7.2, 2H), 2.22 (quintet, *J* = 7.6, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 174.4, 53.5, 26.6, 19.9; **HRMS** (EI): found $[M]^+$ 162.9631 $C_4H_6{}^{79}BrNO$ requires 162.9633

1-bromoazepan-2-one (**6b**)



To a mixture of azepan-2-one (2.66 g, 23.5 mmol), sodium bromate (1.77 g, 11.8 mmol), and concentrated sulfuric acid (0.313 mL, 5.87 mmol) in water (7 mL) was added hydrobromic acid (1.78 mL, 15.7 mmol, 48% w/w in water) dropwise over a period of 5 min. The biphasic mixture was then capped and was stirred vigorously for 1 h at r.t. after which it was cooled to -78 °C for 30 min during which the red bottom layer solidified and lightened in color. After warming to r.t., the solid bottom layer was pulverized by vigorous stirring for 2h. The solid was collected on a fritted funnel and washed twice with 20 mL of cold water and allowed to dry under a stream of nitrogen while pulling vacuum. The solid was then placed under high vacuum for 4 h to afford **6b** (2.18 g, 48%) as a pale yellow solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 3.82-3.80 (m, 2H), 2.67-2.64 (m, 2H), 1.72-1.61 (m, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 174.2, 59.5, 34.9, 29.2, 27.4, 22.9; **HRMS** (EI): found $[M]^+$ 190.9949 $C_6H_{10}{}^{79}BrNO$ requires 190.9946

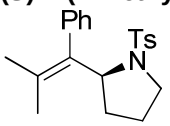
1-bromoazocan-2-one (**6c**):



To a mixture of azocan-2-one (2.50 g, 19.7 mmol), sodium bromate (1.48 g, 9.83 mmol), and concentrated sulfuric acid (0.262 mL, 4.91 mmol) in water (7 mL) was added hydrobromic acid (1.49 mL, 13.2 mmol, 48% w/w in water) dropwise over a period of 5 min. The biphasic mixture was then capped and was stirred vigorously for 1 h at r.t. after which it was cooled to -78 °C for 30 min during which the red bottom layer solidified and lightened in color. After warming to r.t., the solid bottom layer was pulverized by vigorous stirring for 2 h. The solid was collected on a fritted funnel and washed twice with 20 mL of cold water and allowed to dry under a stream of nitrogen while pulling vacuum. The solid was then placed under high vacuum for 4 h to afford **6c** (2.92 g, 72%) as a white solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 3.78 (app t, 2H), 2.63-2.60 (m, 2H), 1.77-1.68 (m, 4H), 1.54-1.50 (m, 2H), 1.41-1.36(m, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 174.0, 55.2, 33.4, 29.1, 28.8, 26.0, 22.9; **HRMS** (EI): found $[M]^+$ 205.0096 $C_7H_{12}{}^{79}BrNO$ requires 205.0102

Cyclization and Subsequent Suzuki Reaction of **1e**

(*S*)-2-(2-methyl-1-phenylprop-1-en-1-yl)-1-tosylpyrrolidine (**7e**)



In a one-dram vial, a mixture of (R)-BINAP(AuPNB)$_2$ (14 mg, 0.010 mmol), **1e** (56 mg, 0.20 mmol), and **6a** (66 mg, 0.40 mmol) was dissolved in $MeNO_2$ (1.0 mL) and allowed to stir for 16 h at r.t. at which point the solvent was evaporated. The residue was redissolved in 2:1 hexanes:EtOAc (1 mL) and passed through a pipette column packed with silica gel (0.65 g) eluting with additional 2:1 hexanes:EtOAc (6 mL) to remove unreacted **6a**. The solvent was evaporated under a stream of nitrogen and briefly placed under high vacuum to afford crude **3e** (42 mg, 59%) as a yellow solid (96% ee as determined by HPLC).

In a one-dram vial under a nitrogen atmosphere, to a mixture of tris(dibenzylideneacetone)dipalladium(0) (4.6 mg, 0.0050 mmol), 2-dicyclohexylphosphino-2′,6′-dimethoxybiphenyl (4.1 mg, 0.010 mmol), crude **3e** (36 mg, 0.10 mmol), phenylboronic acid (24 mg, 0.20 mmol), and finely ground K_3PO_4 (64 mg, 0.30 mmol) was added dry and degassed toluene (0.4 mL). The mixture was vigorously stirred for 3 h at 100 °C at at which point it was passed through a short silica gel plug and the solvent removed under a stream of nitrogen. The product was purified by column chromatography on silica gel (hexanes:EtOAc 9:1) to afford **7e** (19 mg, 52%) as a white solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.75 (d, *J* = 8.4, 2H), 7.33-7.10 (m, 7H), 4.82 (dd, *J* = 8.4, 5.2, 1H), 3.21-3.15 (m, 1H), 2.89-2.83 (m, 1H), 2.44 (s, 3H), 1.97(s, 3H), 1.82-1.73 (m, 1H), 1.69-1.61 (m, 1H), 1.45 (s, 3H), 1.16-1.03 (m, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 143.1, 140.0, 135.4, 135.2, 130.9, 130.1, 129.6, 127.7, 127.4, 126.2, 60.0, 49.5, 31.5, 24.4, 22.8, 21.5, 19.7; **HRMS** (ESI): found $[M+H]^+$ 356.1675 $C_{21}H_{26}NO_2{}^{32}S$ requires 356.1679; **HPLC** Chiralpak IA column (98:2 hexanes:isopropanol, 1.0 mL/min, 210 nm); major enantiomer t_r = 26.25 min, minor enantiomer t_r = 13.90 min, 93%ee; absolute configuration assigned as (*S*) by analogy to **3e**.

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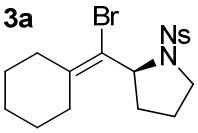
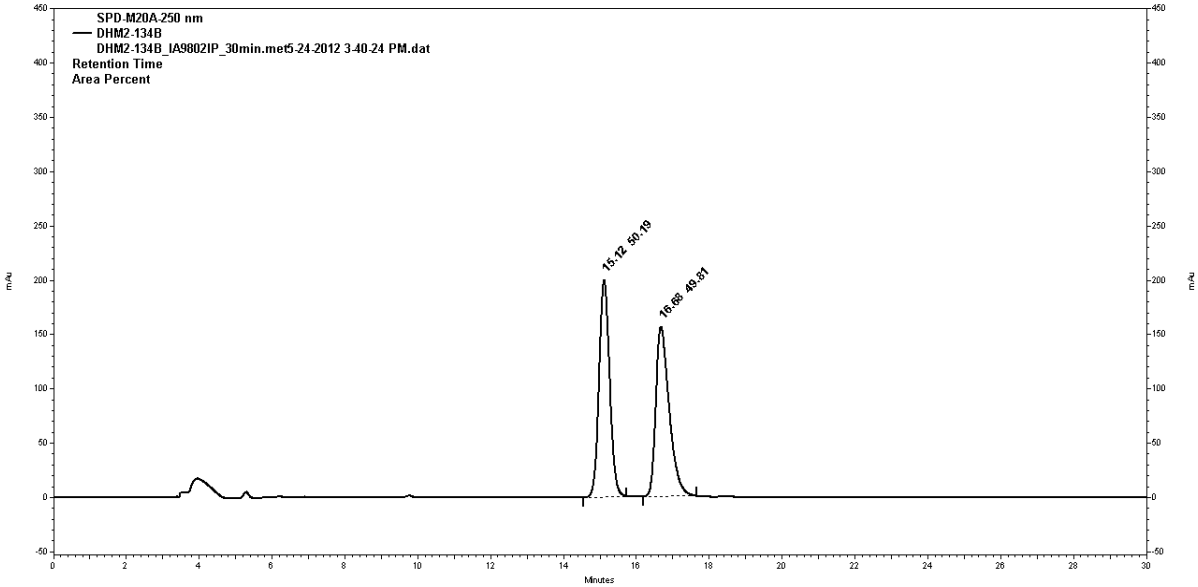
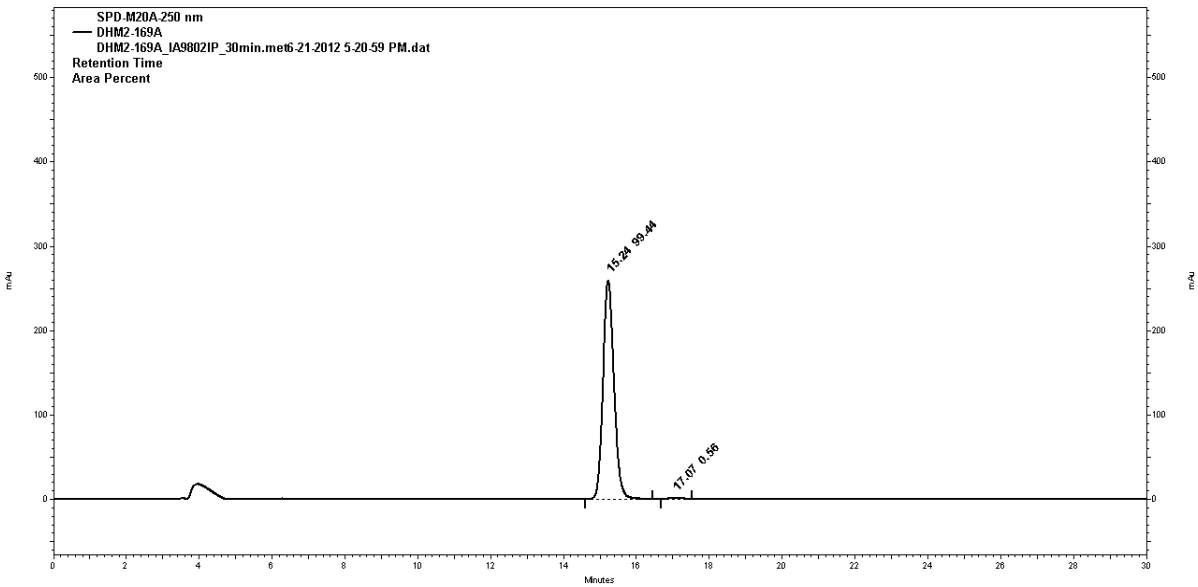
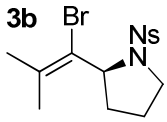


Racemic



Enantioenriched





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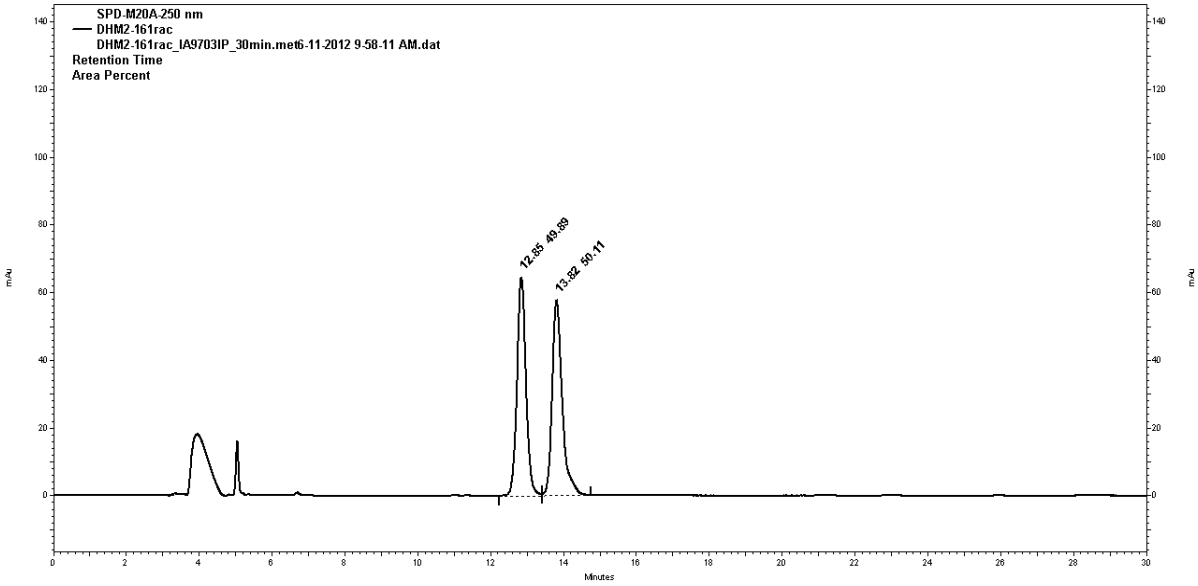
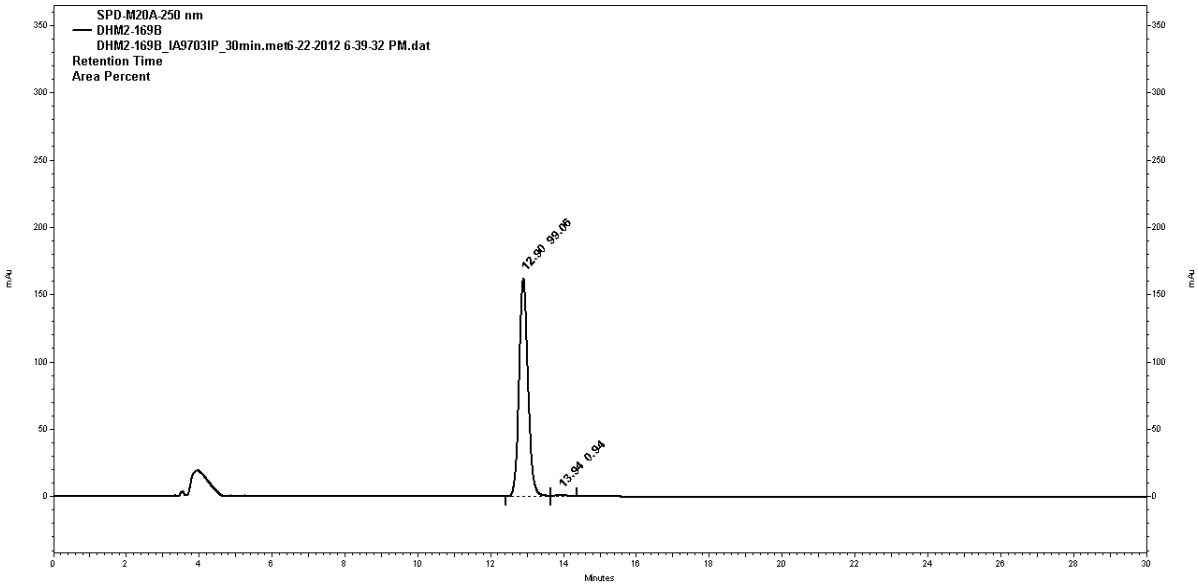
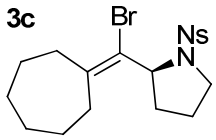


Enantioenriched





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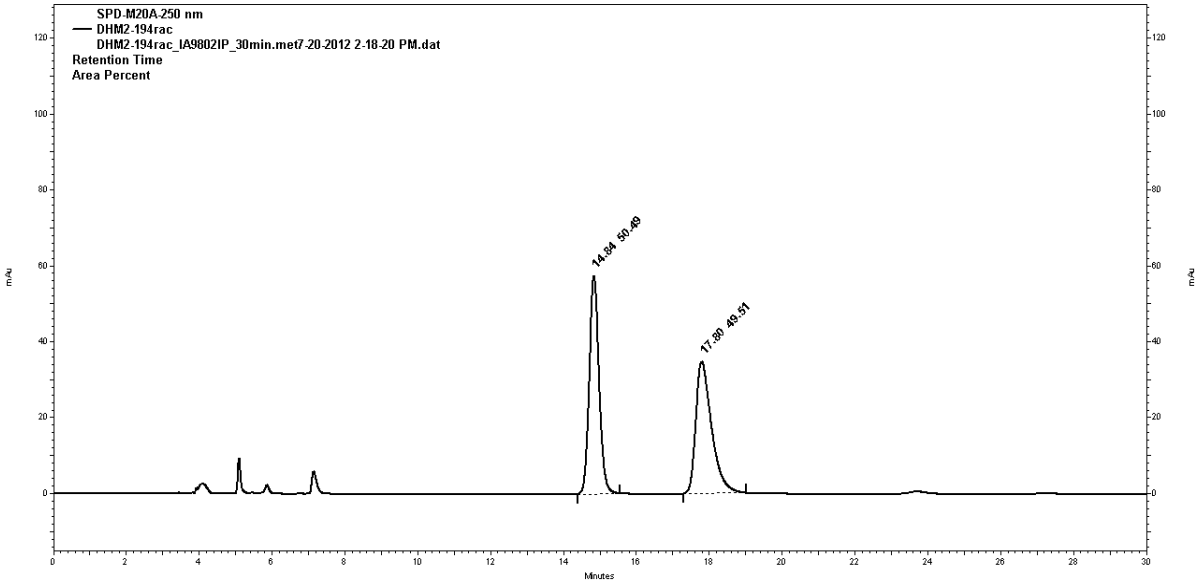

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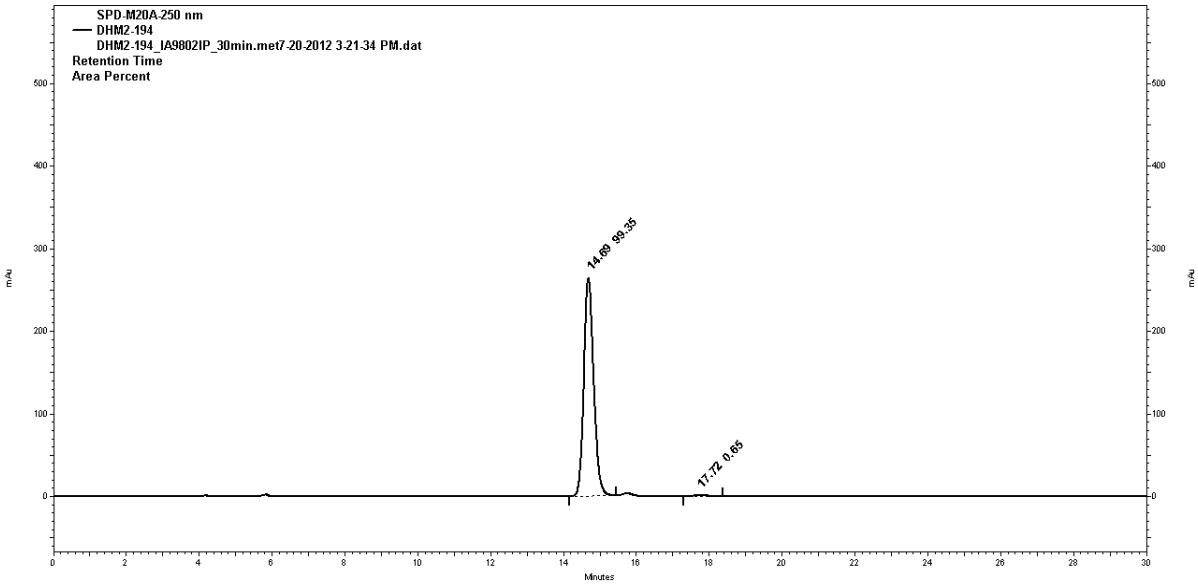

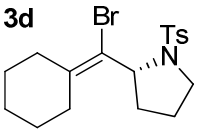

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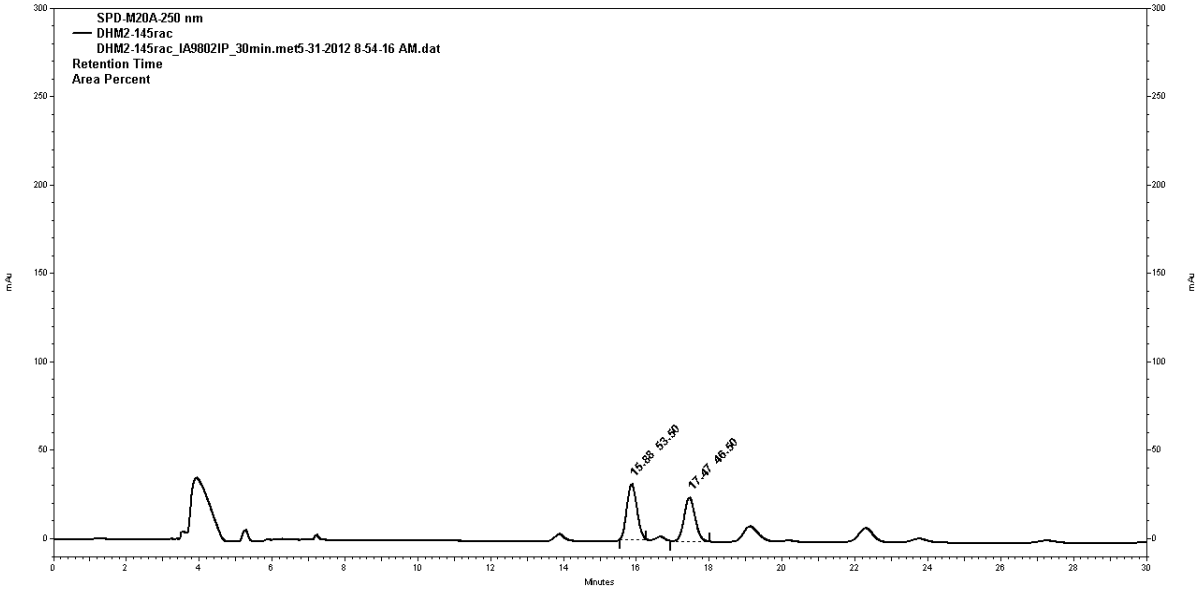

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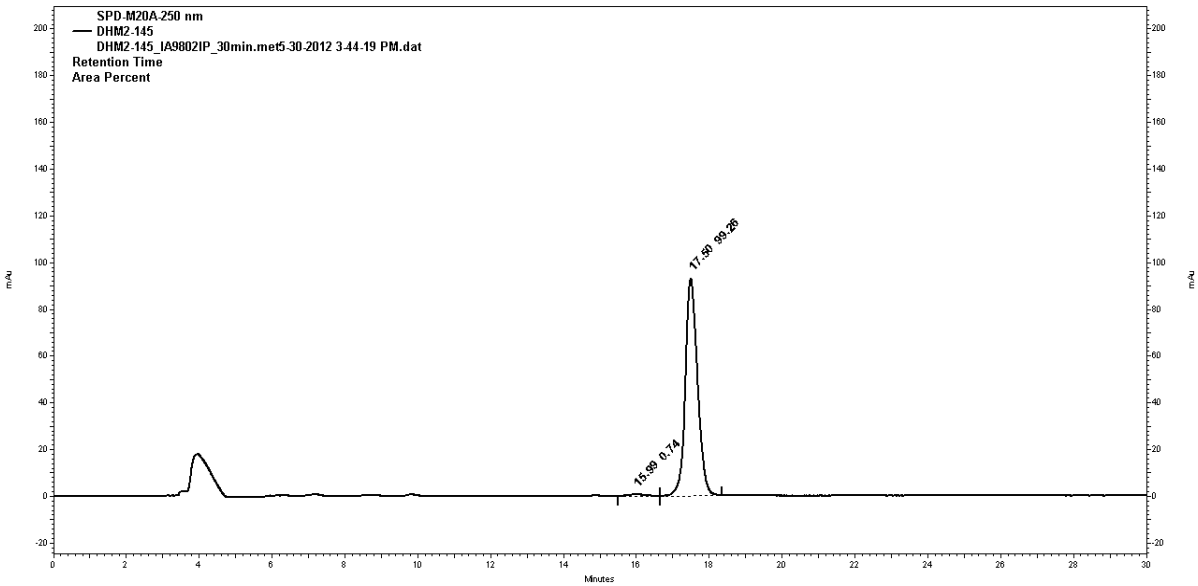

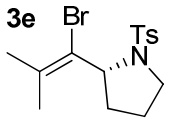

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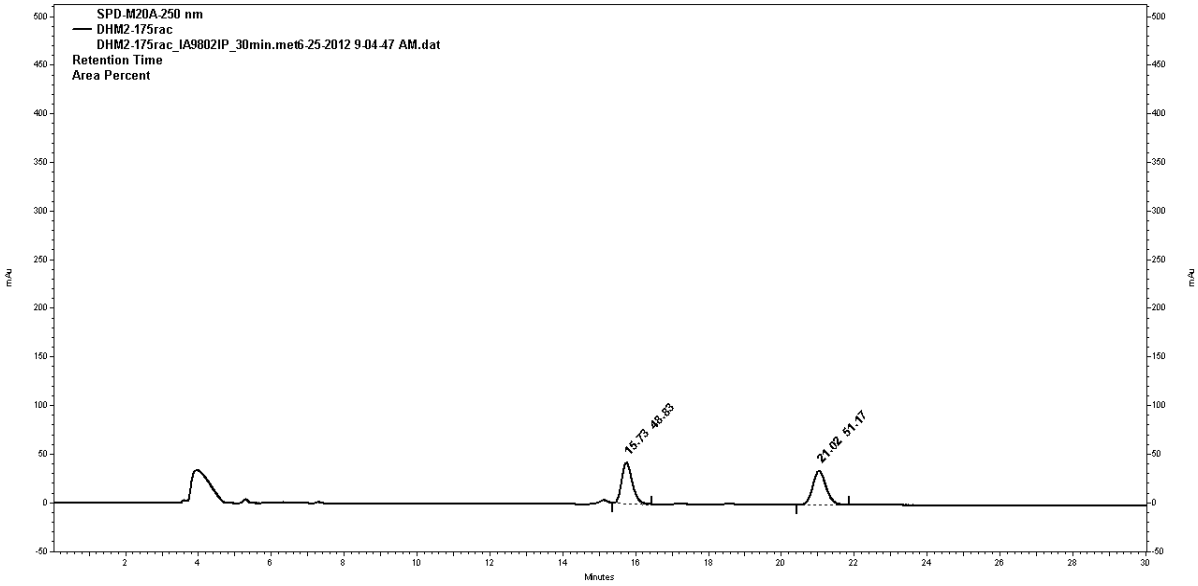

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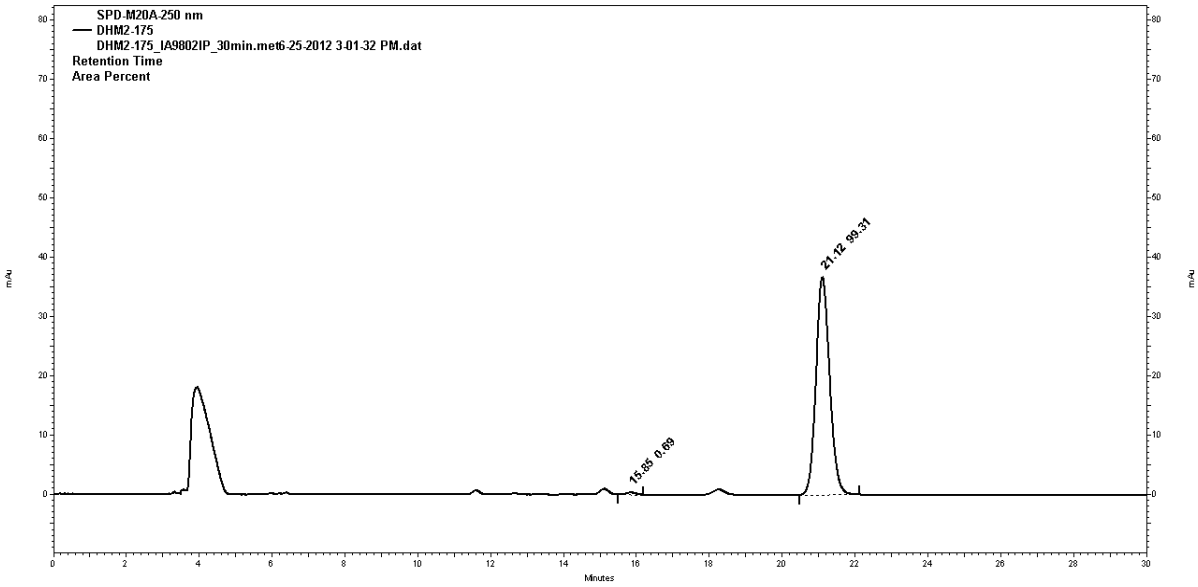

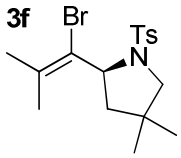

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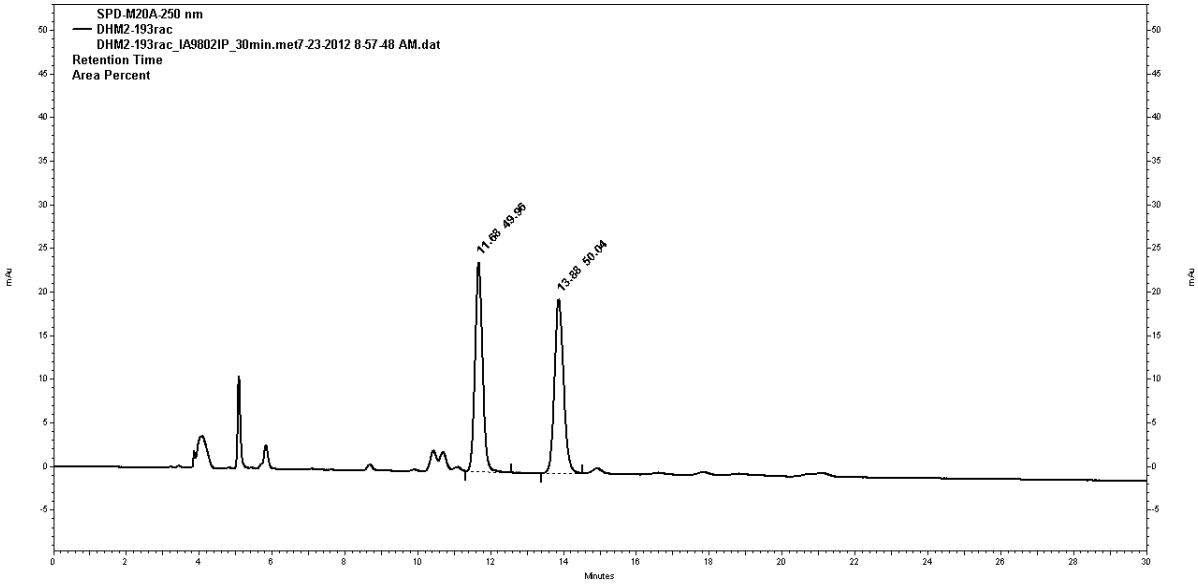

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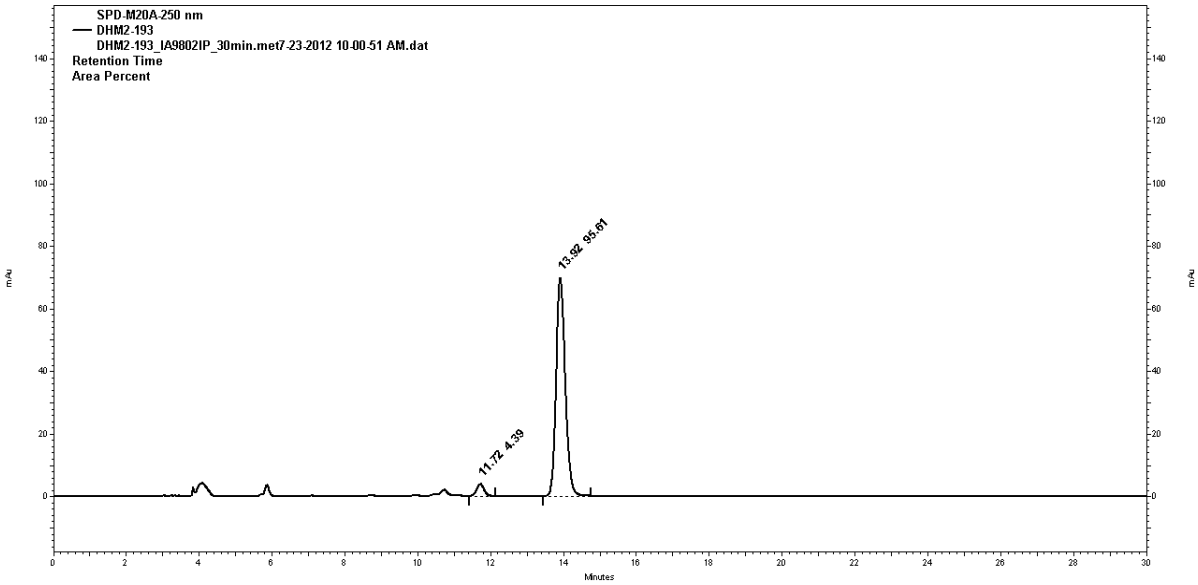

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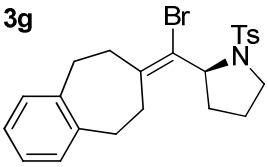

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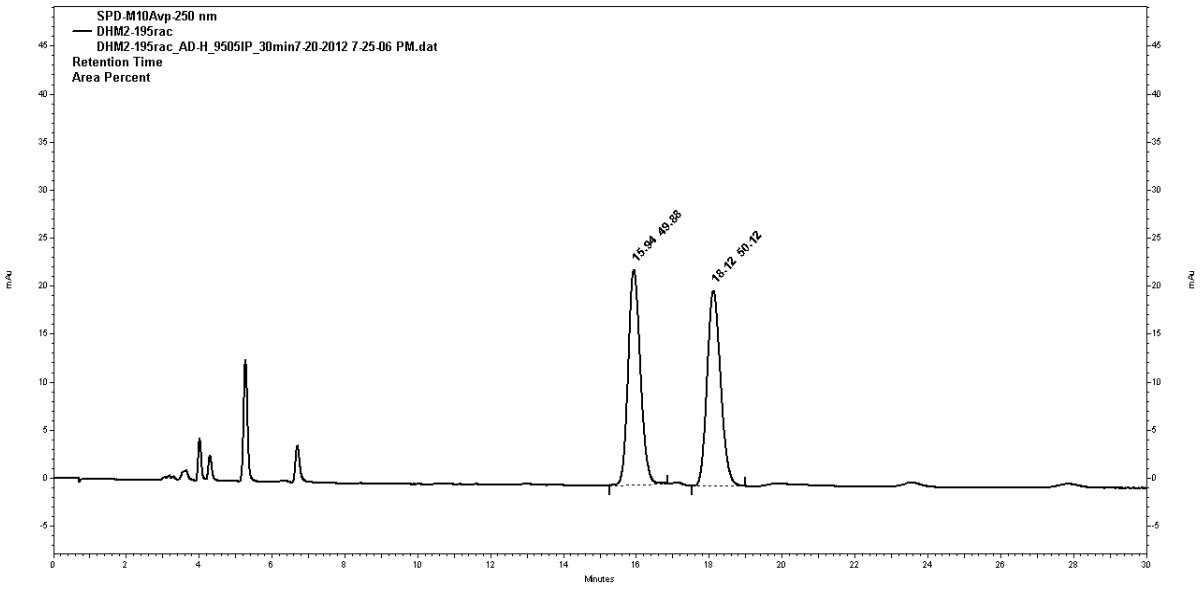

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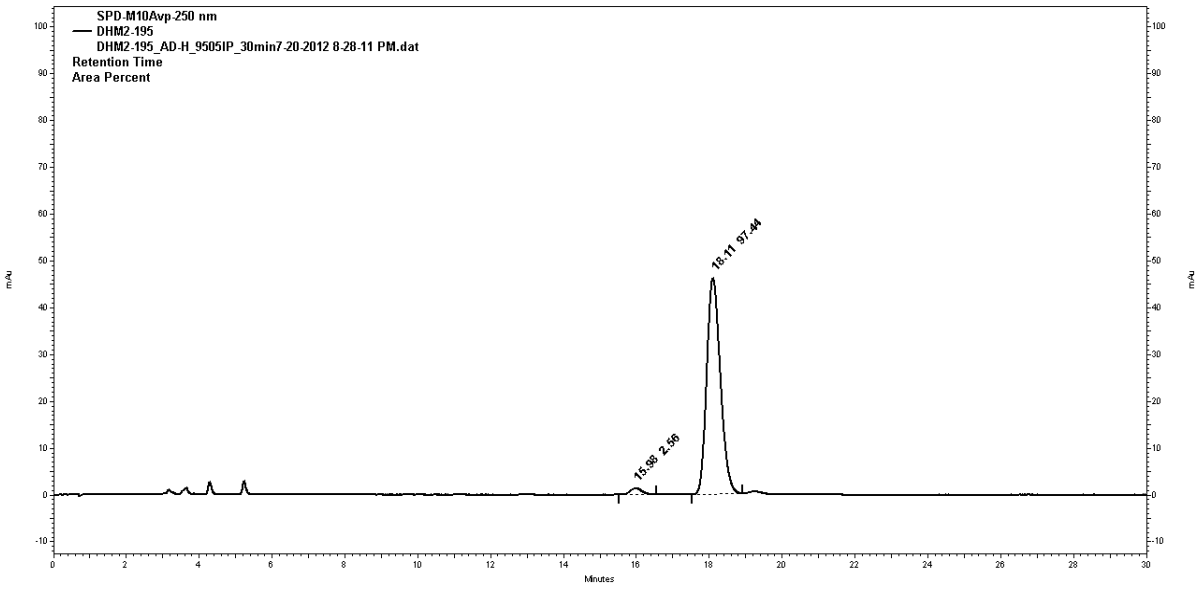

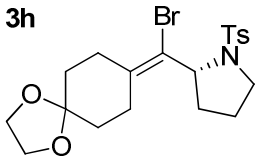

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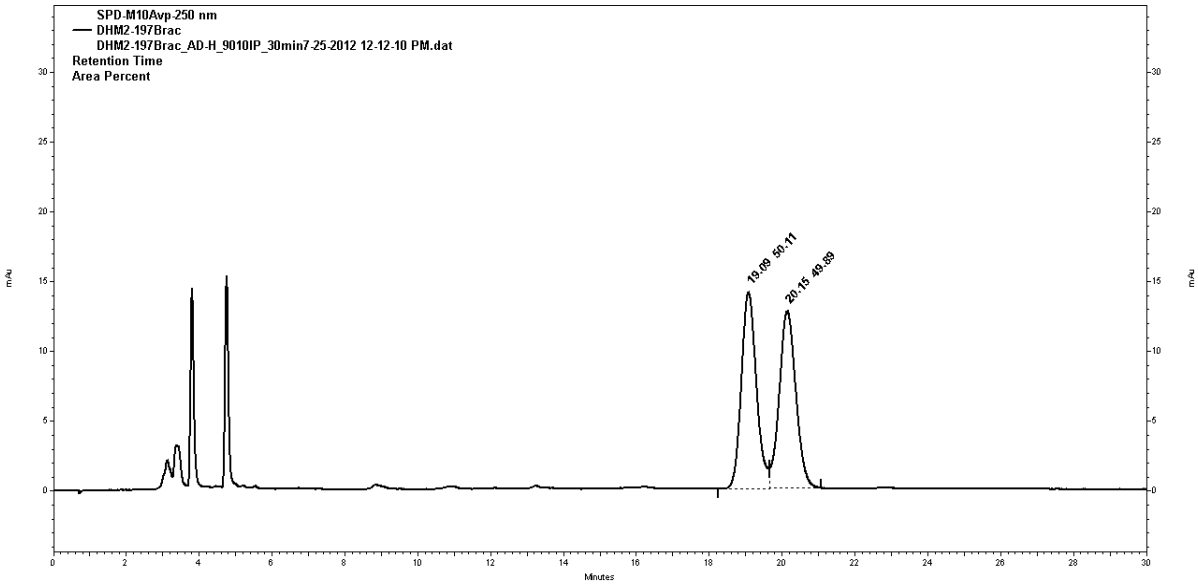

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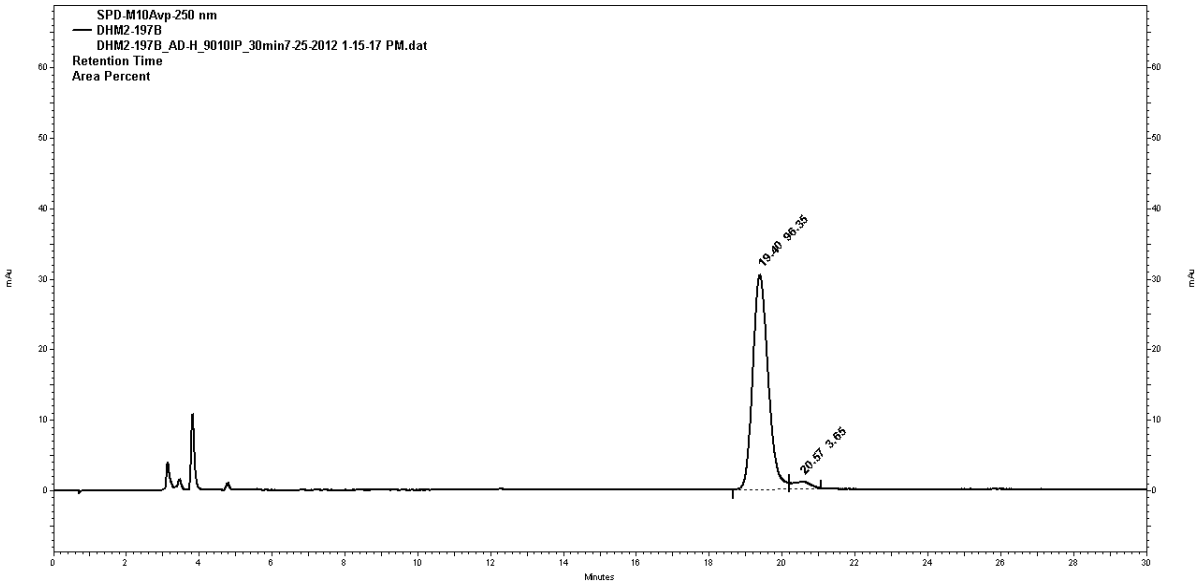

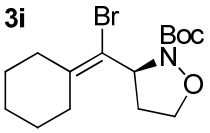

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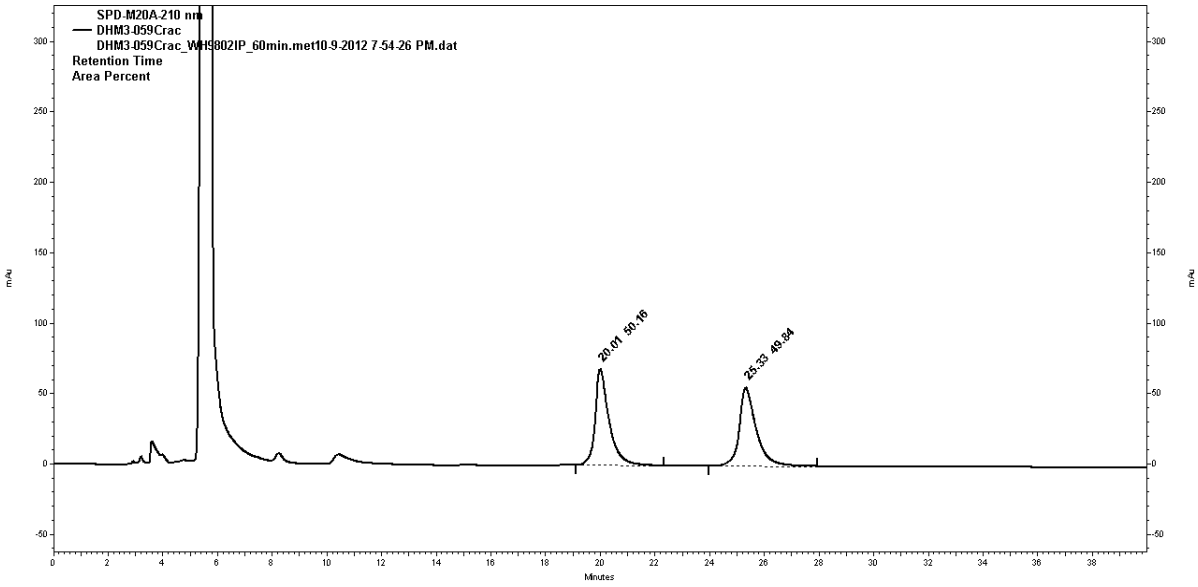

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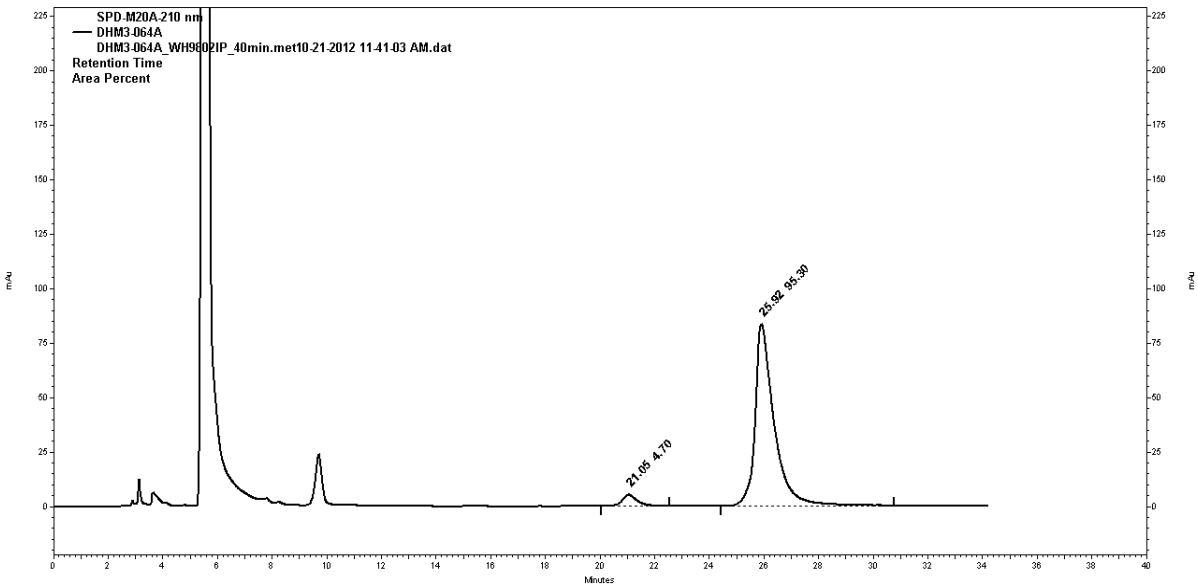

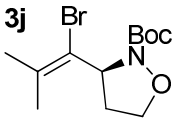

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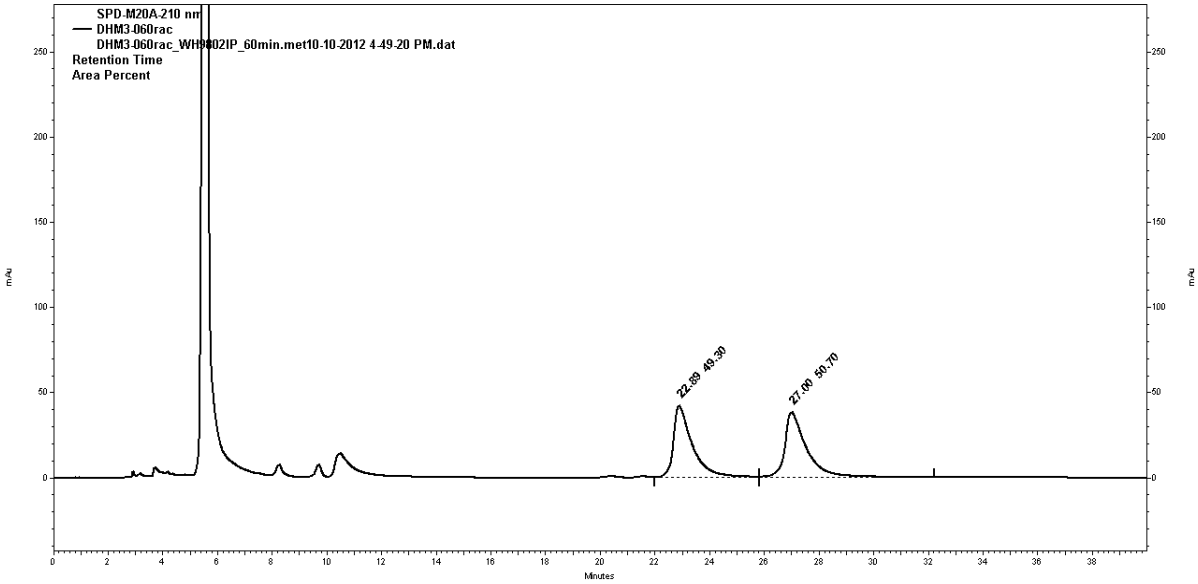

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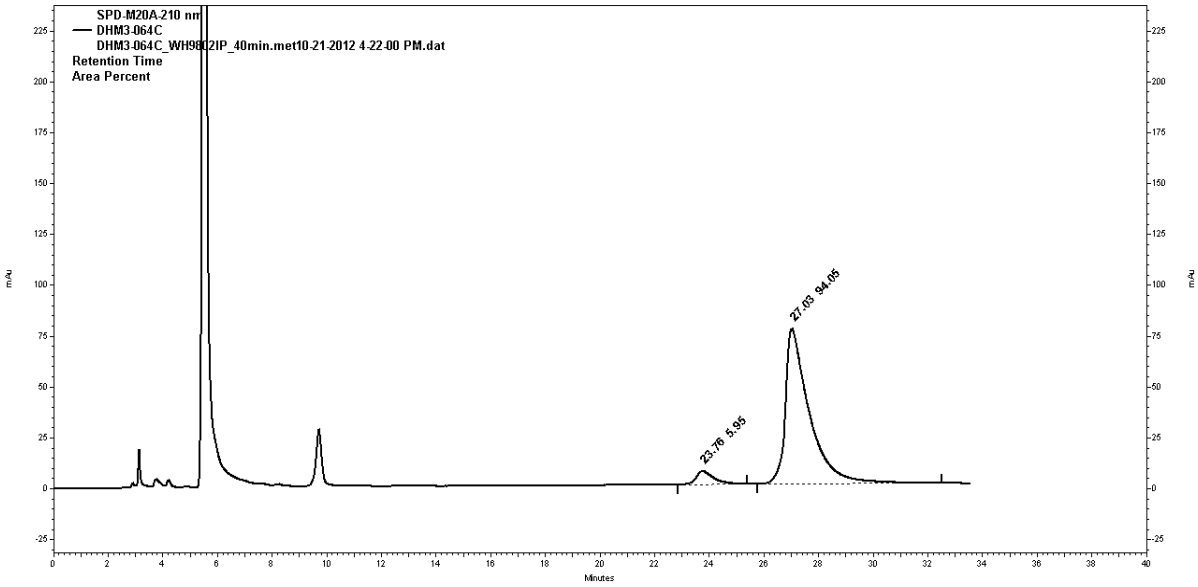

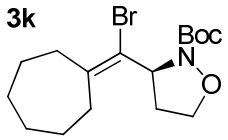

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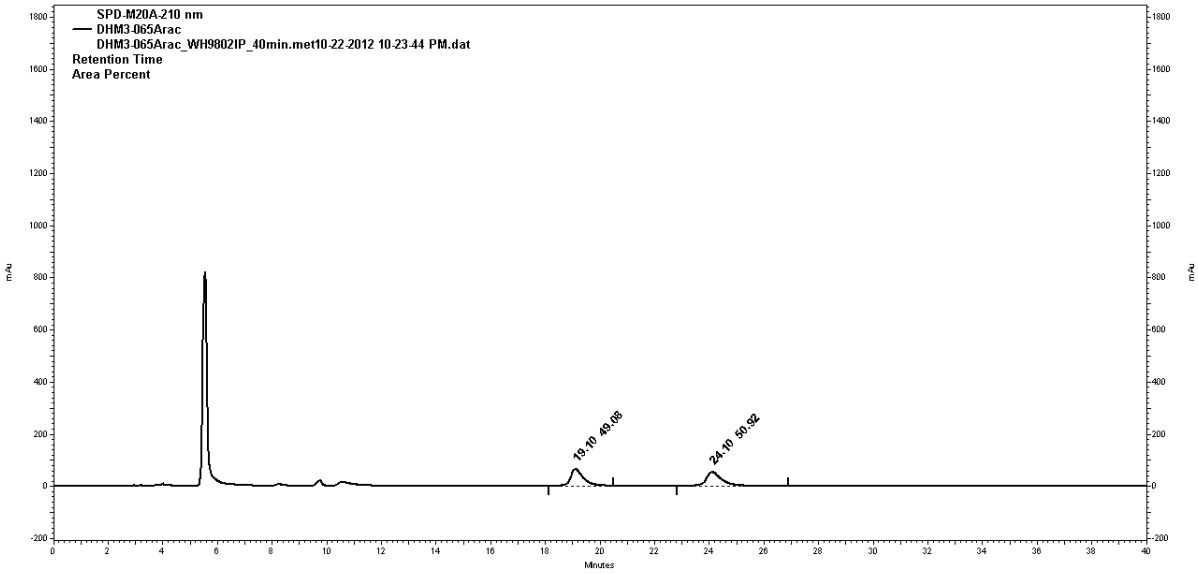

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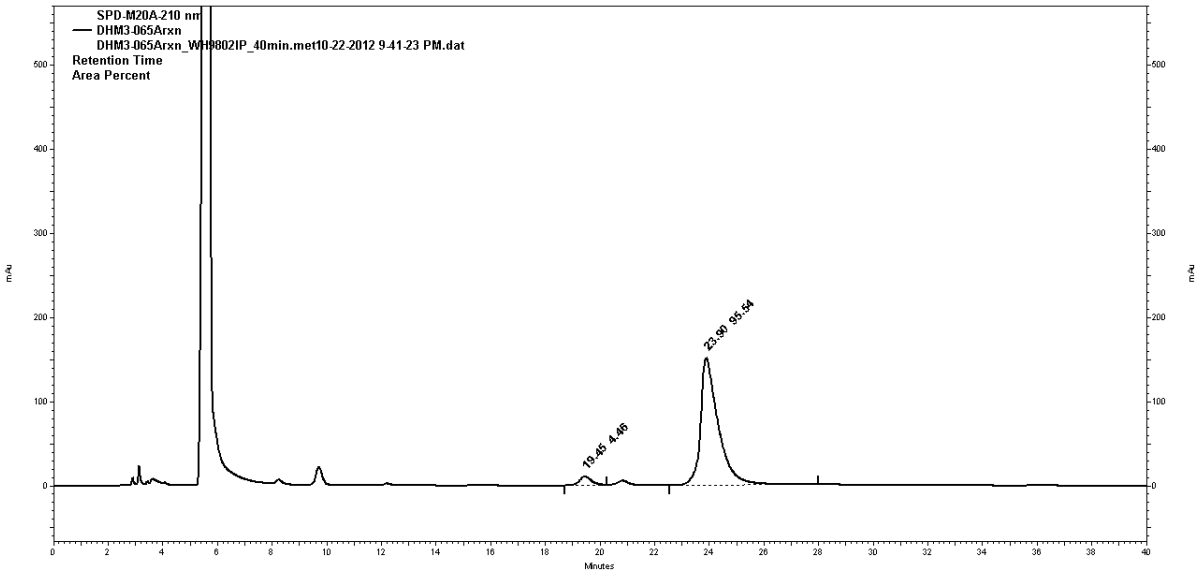

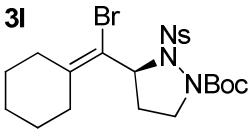

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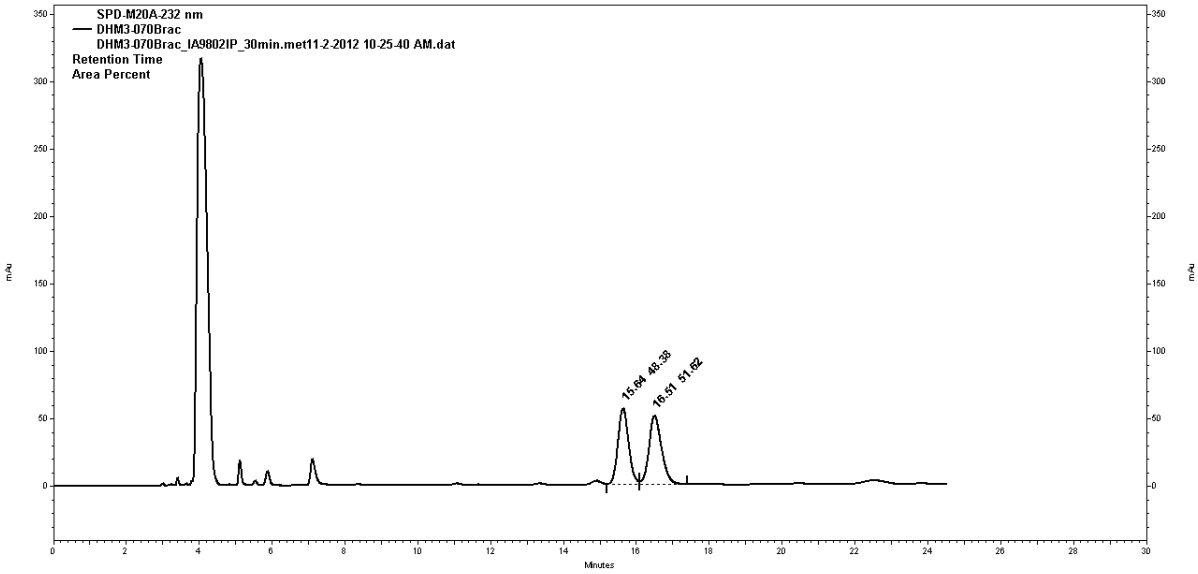

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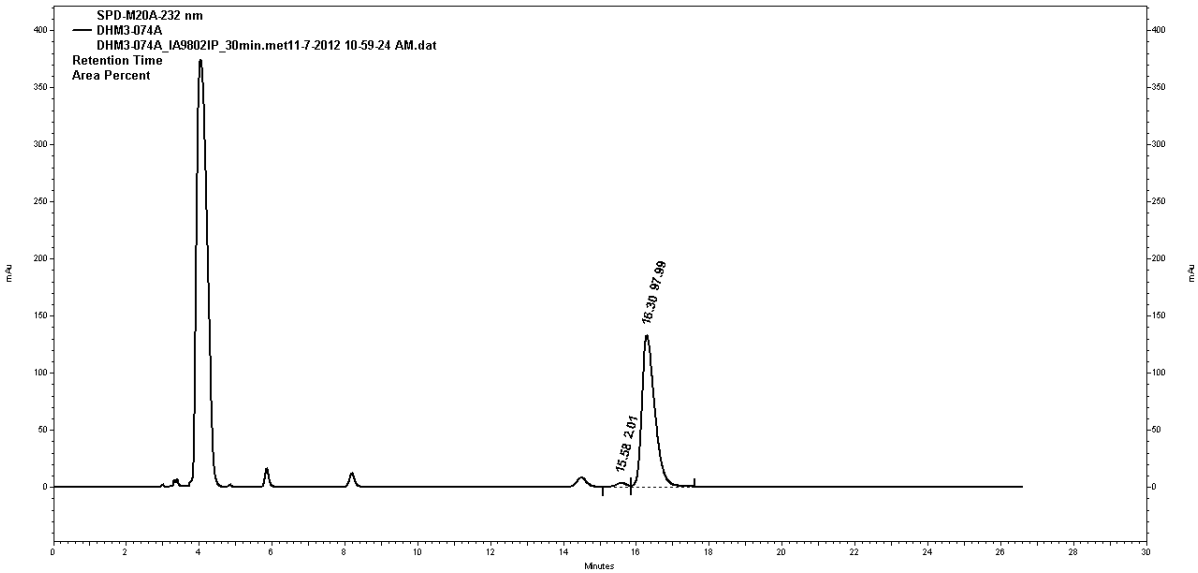

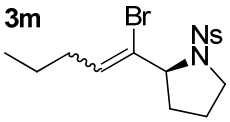

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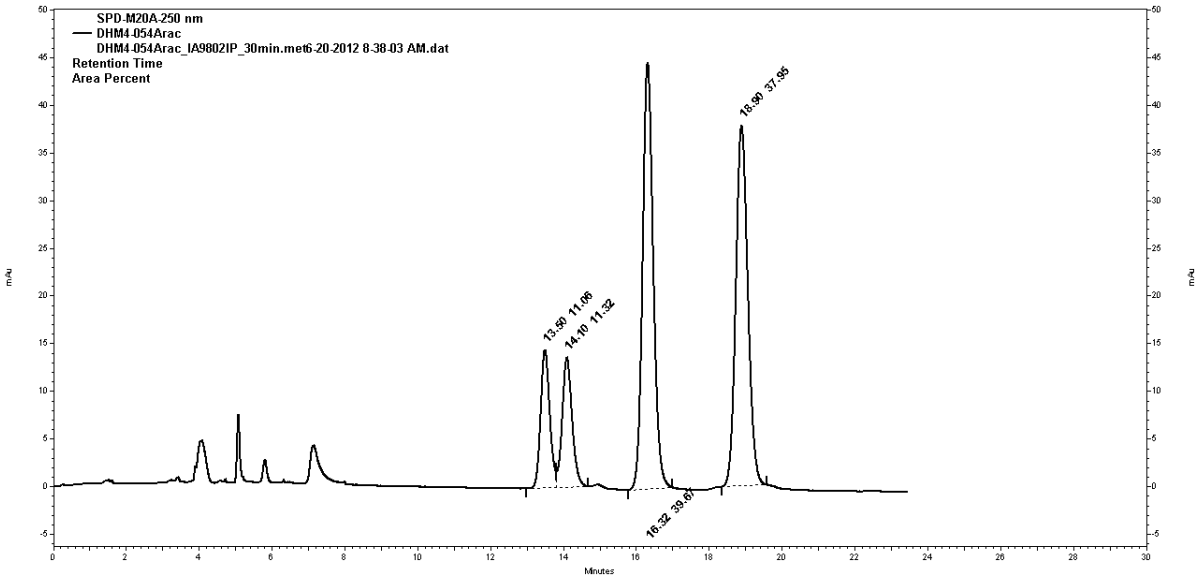

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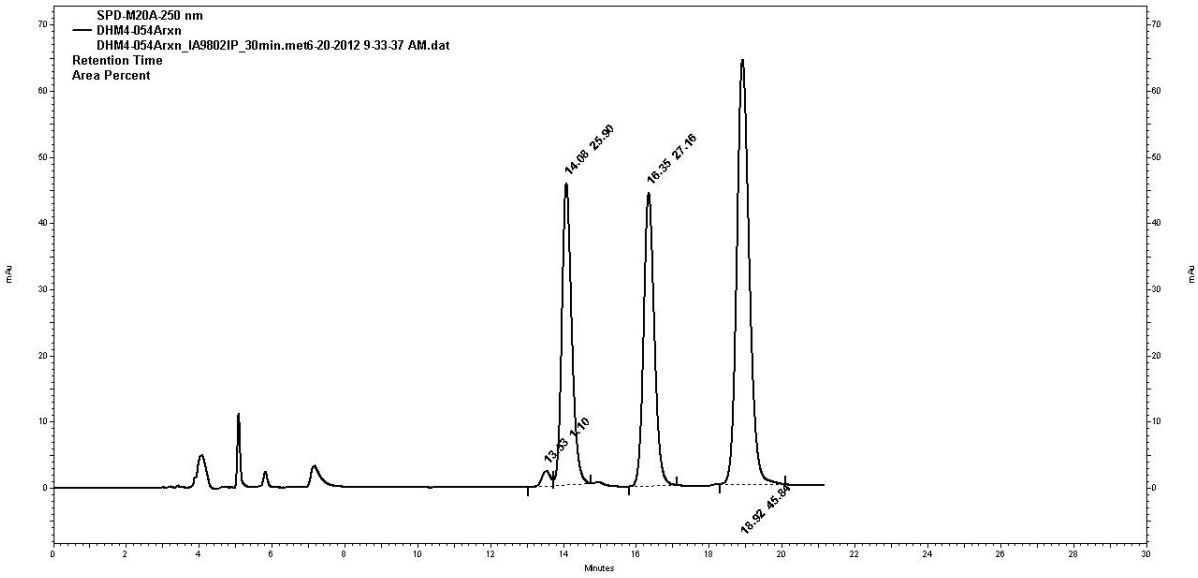

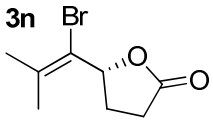

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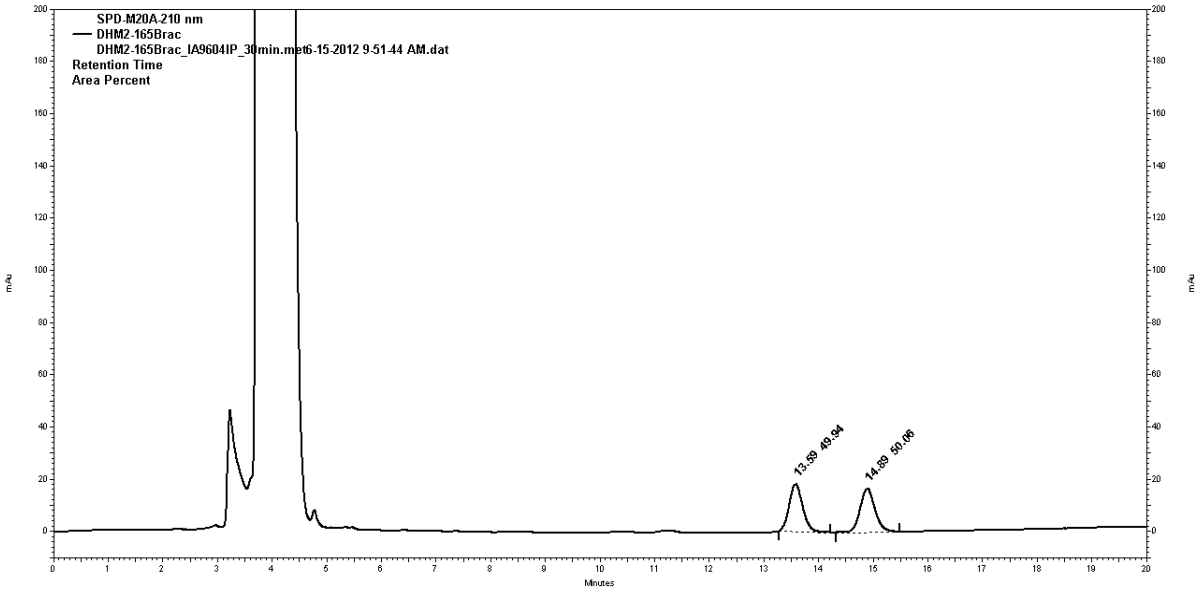

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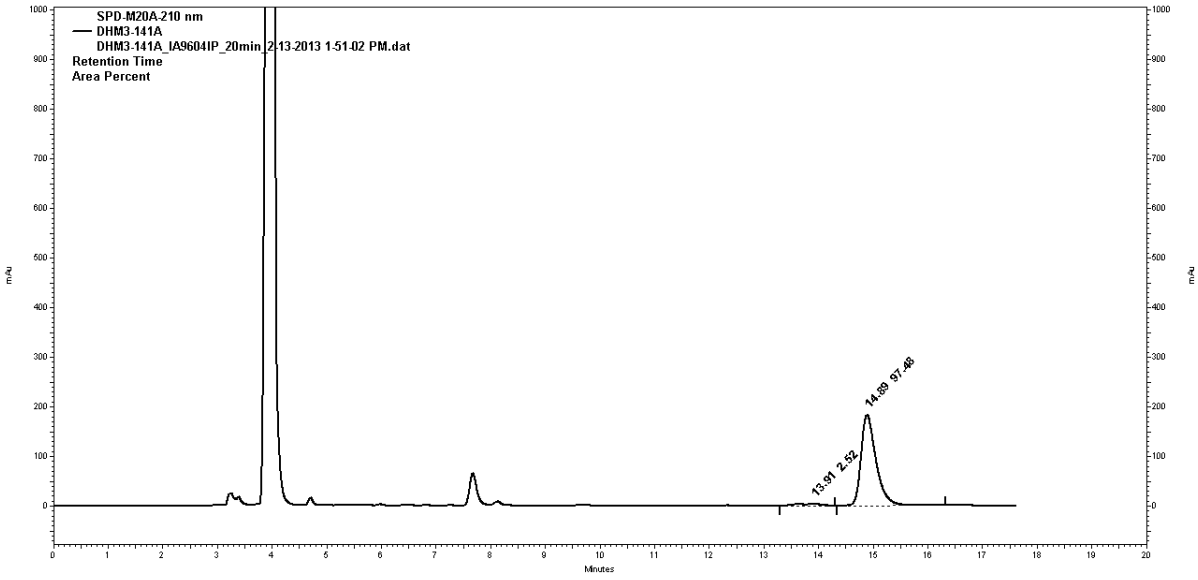

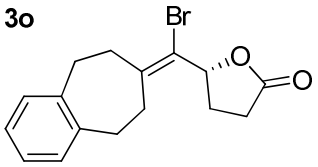

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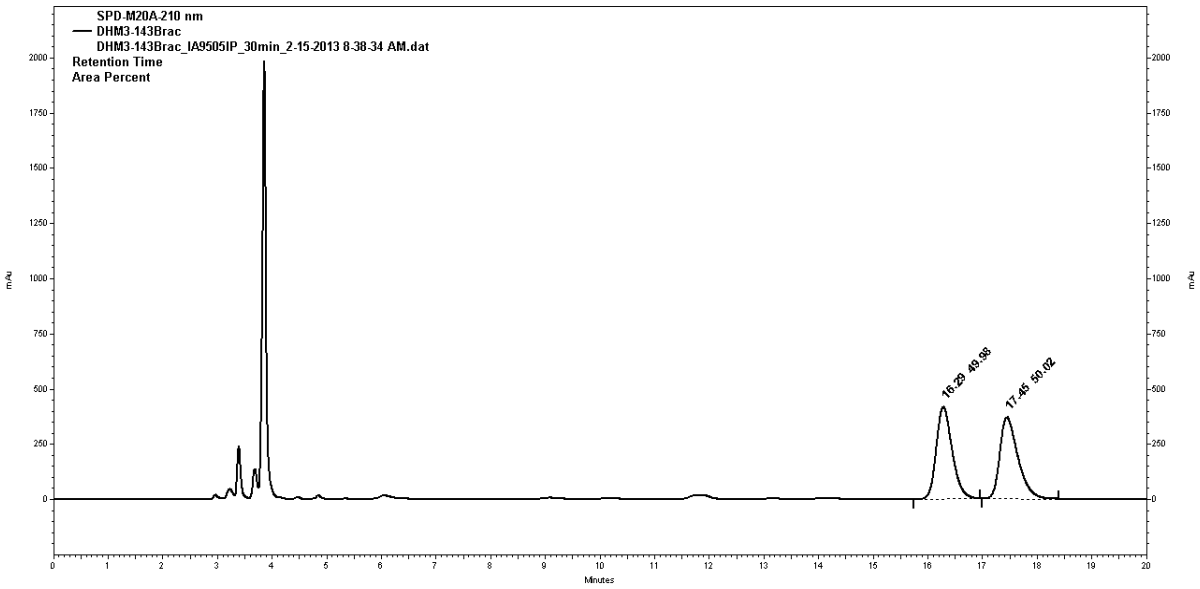

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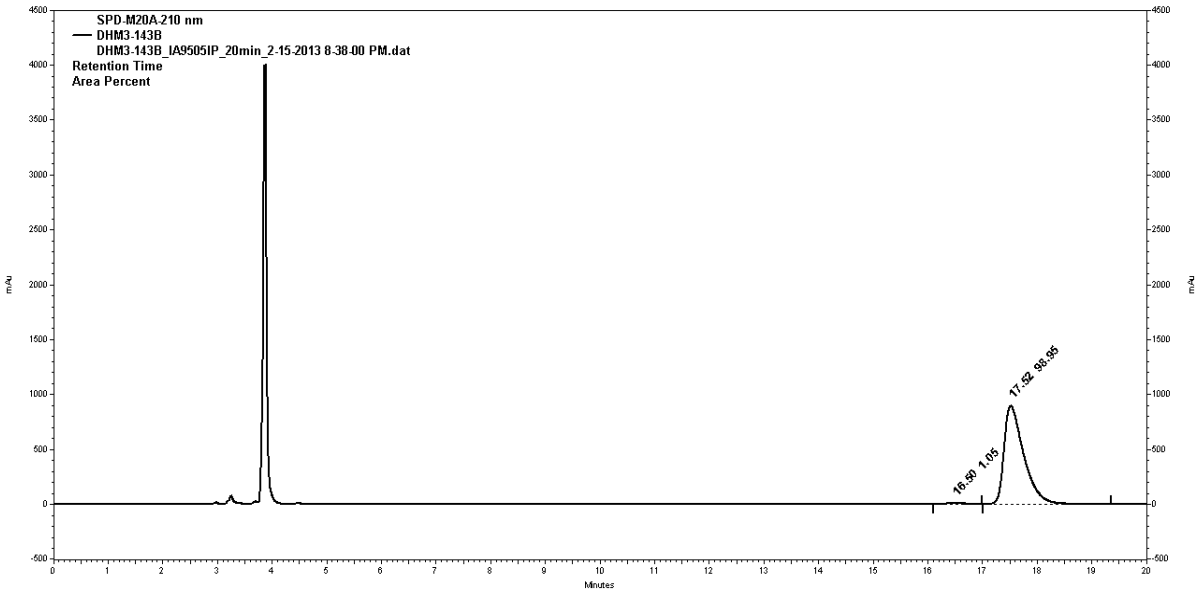

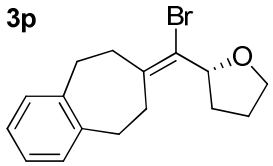

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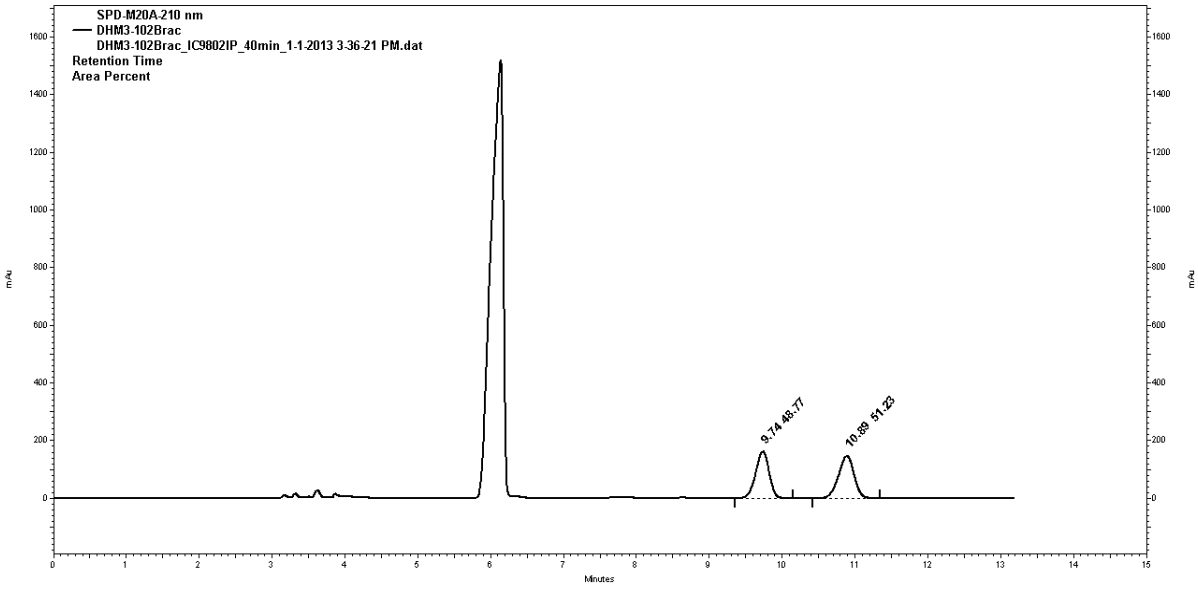

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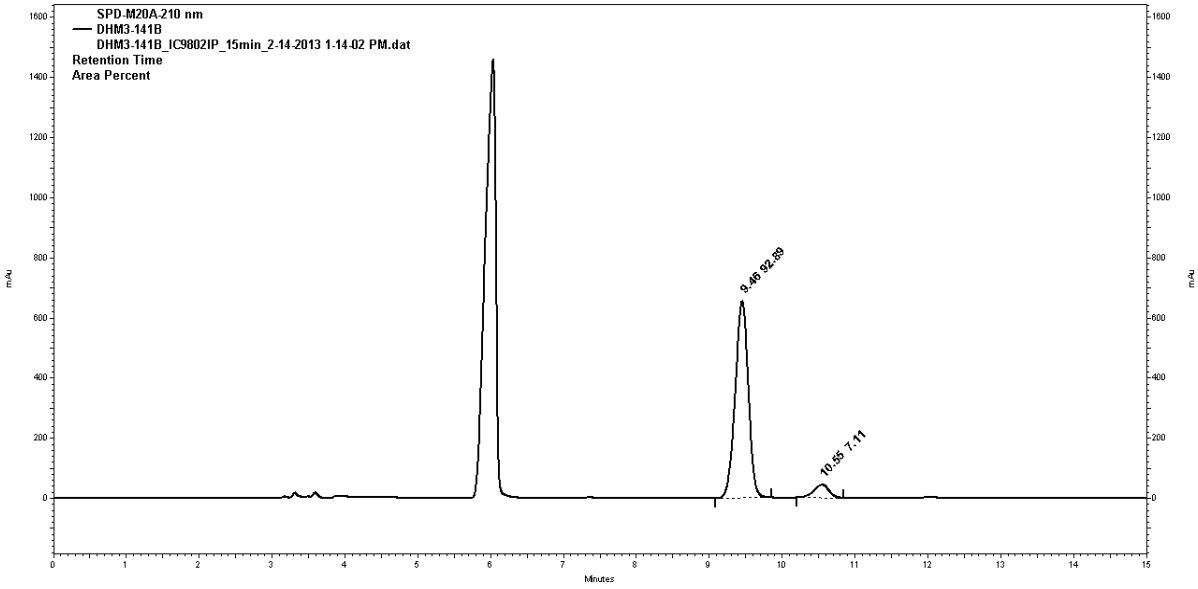

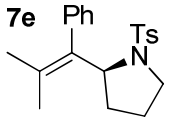

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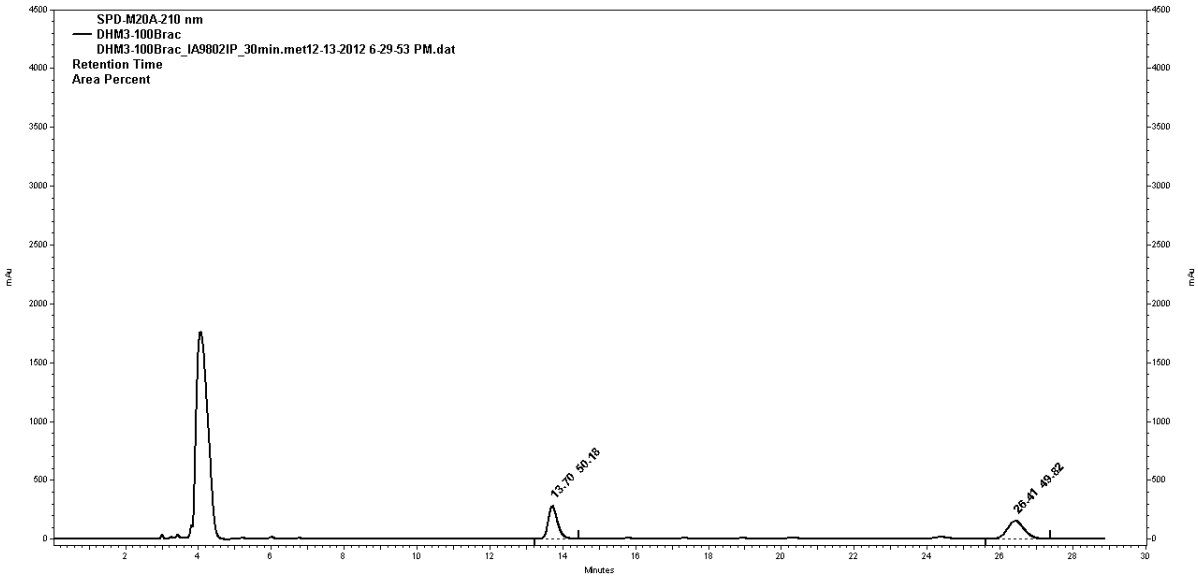

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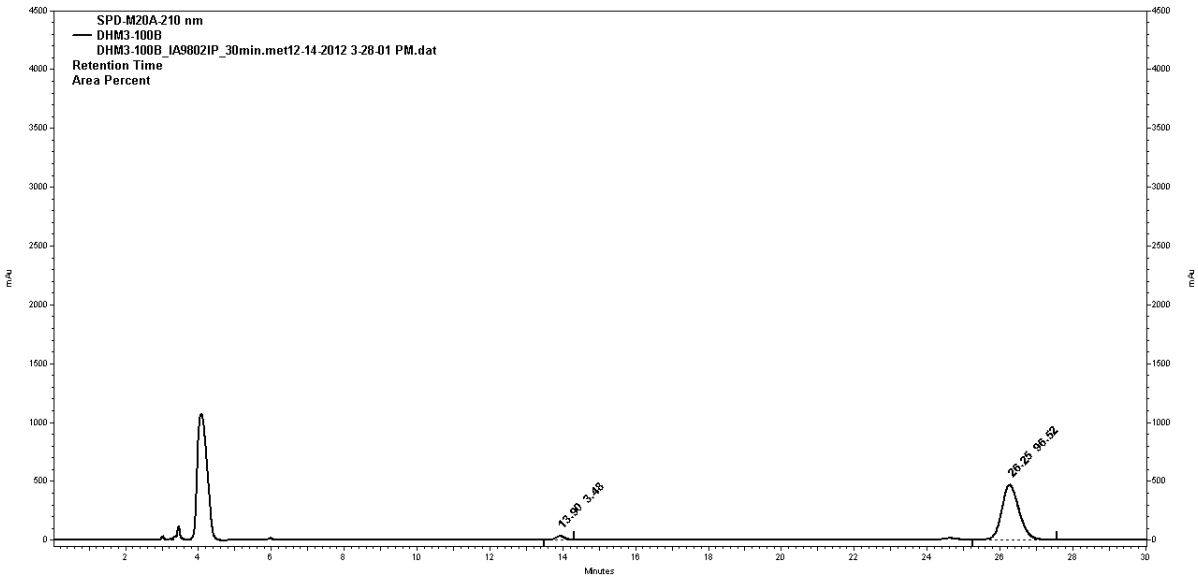

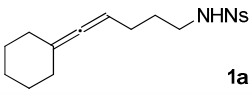
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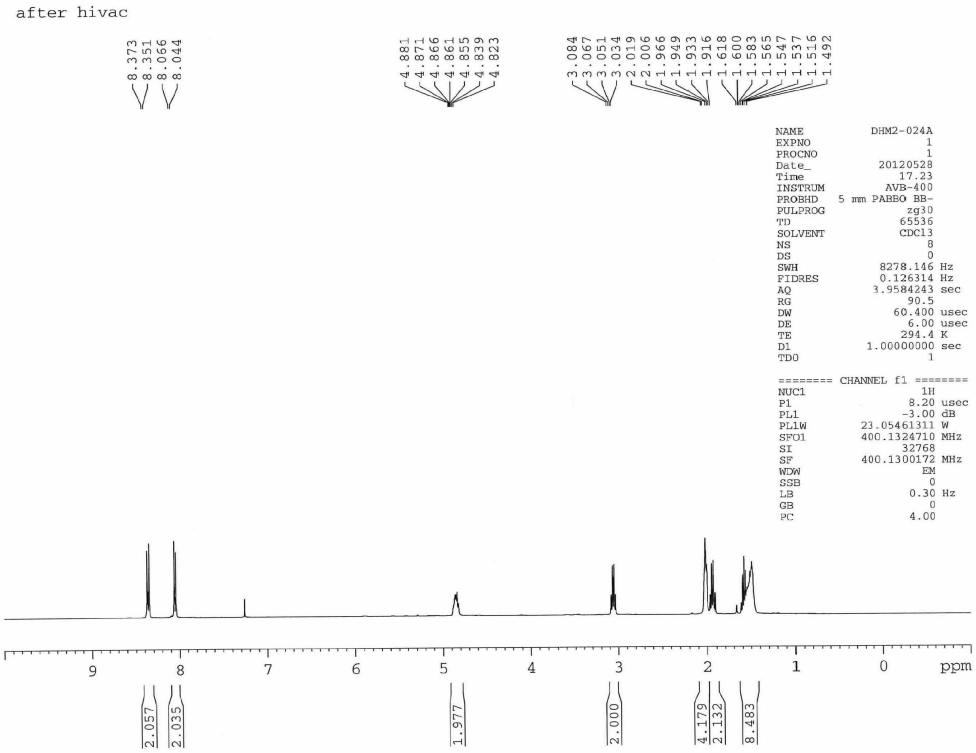
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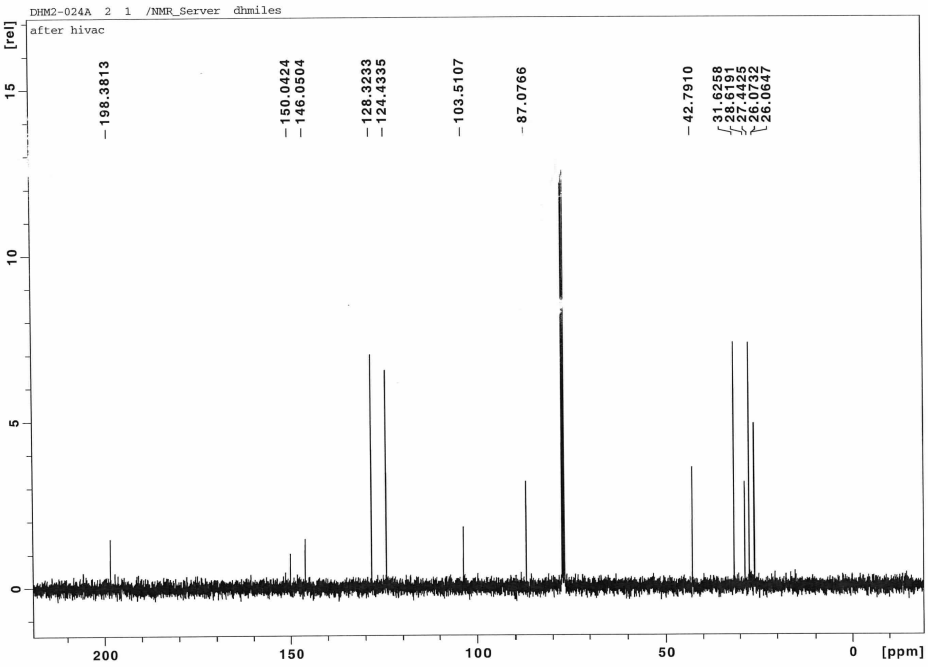
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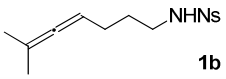
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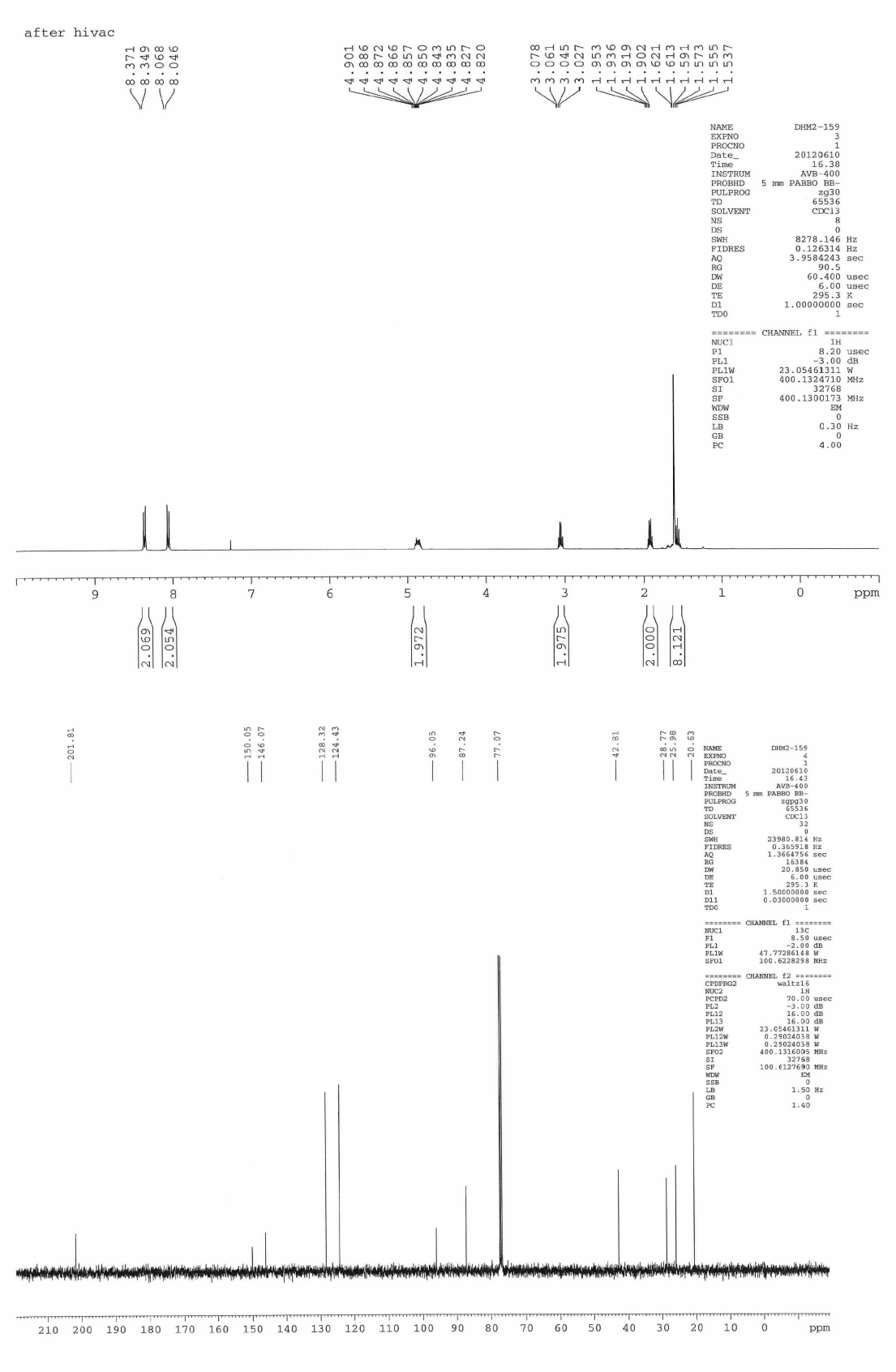
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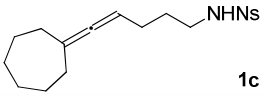
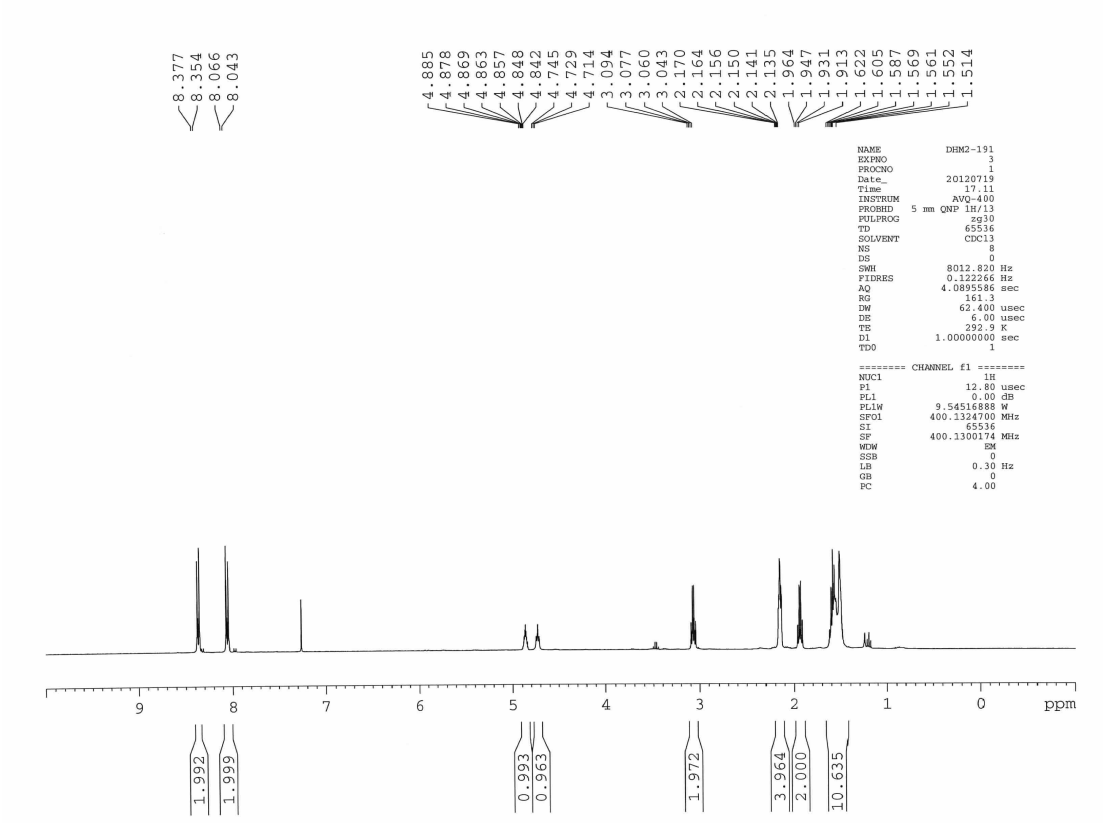
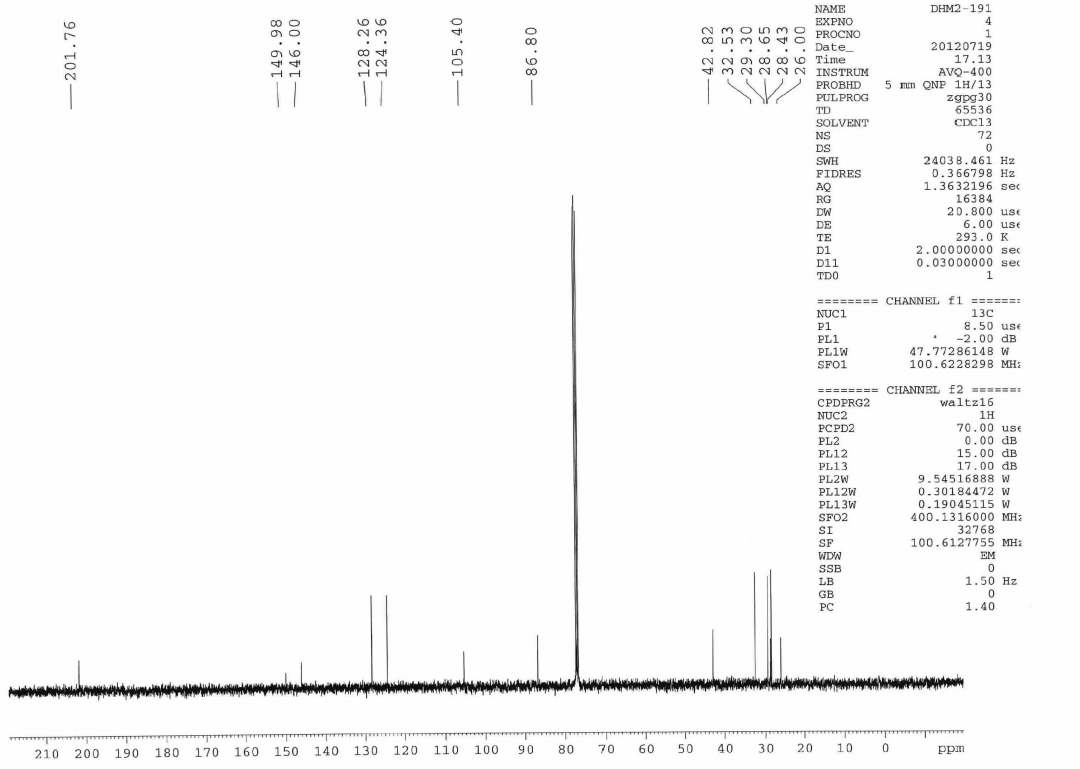
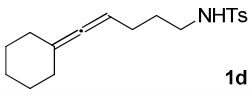
EXPNO 3
PROCNO 1
Date_ 20120610
Time 16.38
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 90.5
DW 60.400 usec
DE 6.00 usec
TE 295.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.20 usec
PL1 -3.00 dB
PL1W 23.05461311 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300173 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
9 8 7 6 5 4 3 2 1 0 ppm
2.069
2.054
1.972
1.975
2.000
8.121
201.81
150.05
146.07
128.32
124.43
96.05
87.24
77.07
42.81
28.77
25.98
20.63
NAME DHM2-159
EXPNO 4
PROCNO 1
Date_ 20120610
Time 16.43
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 32
DS 0
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 16384
DW 20.850 usec
DE 6.00 usec
TE 295.3 K
D1 1.50000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 47.77286148 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -3.00 dB
PL12 16.00 dB
PL13 16.00 dB
PL2W 23.05461311 W
PL12W 0.29024038 W
PL13W 0.29024038 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.50 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


NHNs

1c

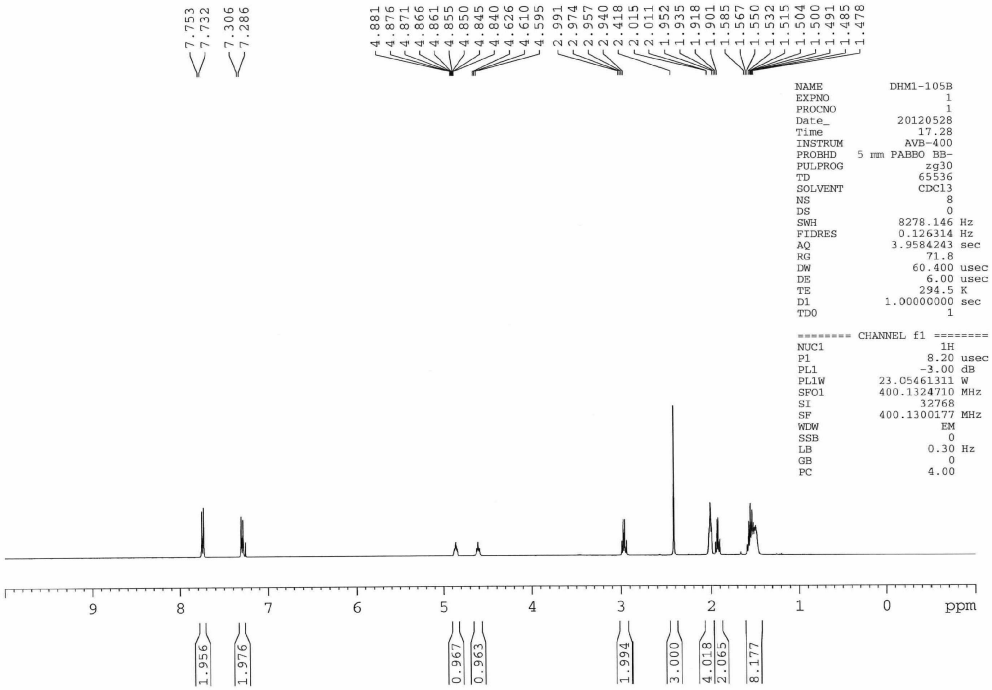

8.377
8.354
8.066
8.043
4.885
4.878
4.869
4.863
4.857
4.848
4.842
4.745
4.729
4.714
3.094
3.077
3.060
3.043
2.170
2.164
2.156
2.150
2.141
2.135
1.964
1.947
1.931
1.913
1.622
1.605
1.587
1.569
1.561
1.552
1.514
NAME DHM2-191
EXPNO 3
PROCNO 1
Date_ 20120719
Time 17.11
INSTRUM AVQ-400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0895586 sec
RG 161.3
DW 62.400 usec
DE 6.00 usec
TE 292.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.80 usec
PL1 0.00 dB
PL1W 9.54516888 W
SFO1 400.1324700 MHz
SI 65536
SF 400.1300174 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
9
8
7
6
5
4
3
2
1
0
ppm
1.992
1.999
0.993
0.963
1.972
3.964
2.000
10.635

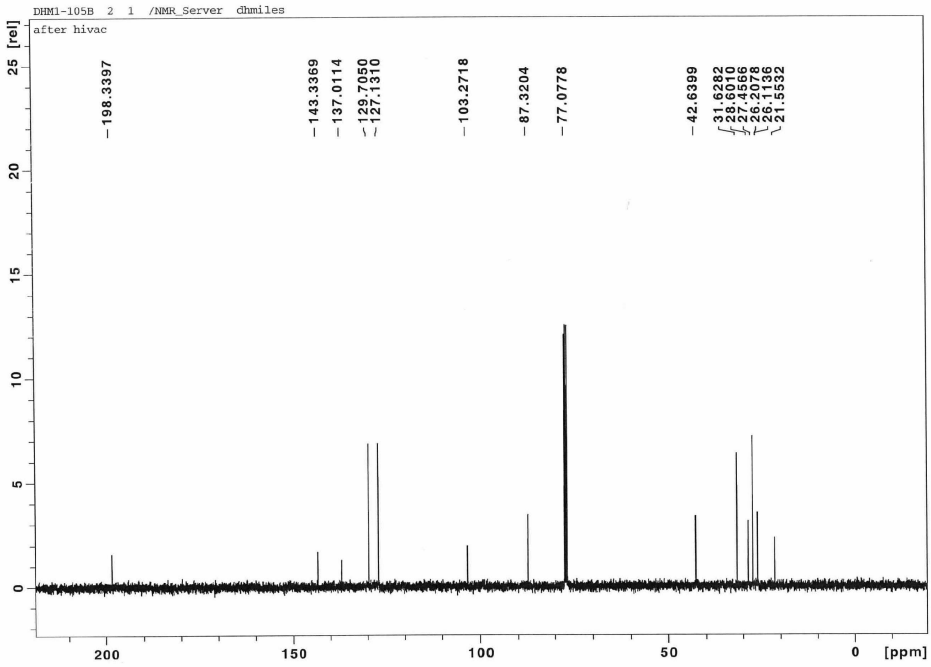

201.76
149.98
146.00
128.26
124.36
105.40
86.80
42.82
32.53
29.30
28.65
28.43
26.00
NAME DHM2-191
EXPNO 4
PROCNO 1
Date_ 20120719
Time 17.13
INSTRUM AVQ-400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 72
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3632196 sec
RG 16384
DW 20.800 use
DE 6.00 use
TE 293.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 =======
NUC1 13C
P1 8.50 use
PL1 -2.00 dB
PL1W 47.77286148 W
SFO1 100.6228298 MHz
======== CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 use
PL2 0.00 dB
PL12 15.00 dB
PL13 17.00 dB
PL2W 9.54516888 W
PL12W 0.30184472 W
PL13W 0.19045115 W
SFO2 400.1316000 MHz
SI 32768
SF 100.6127755 MHz
WDW EM
SSB 0
LB 1.50 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

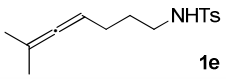


1d

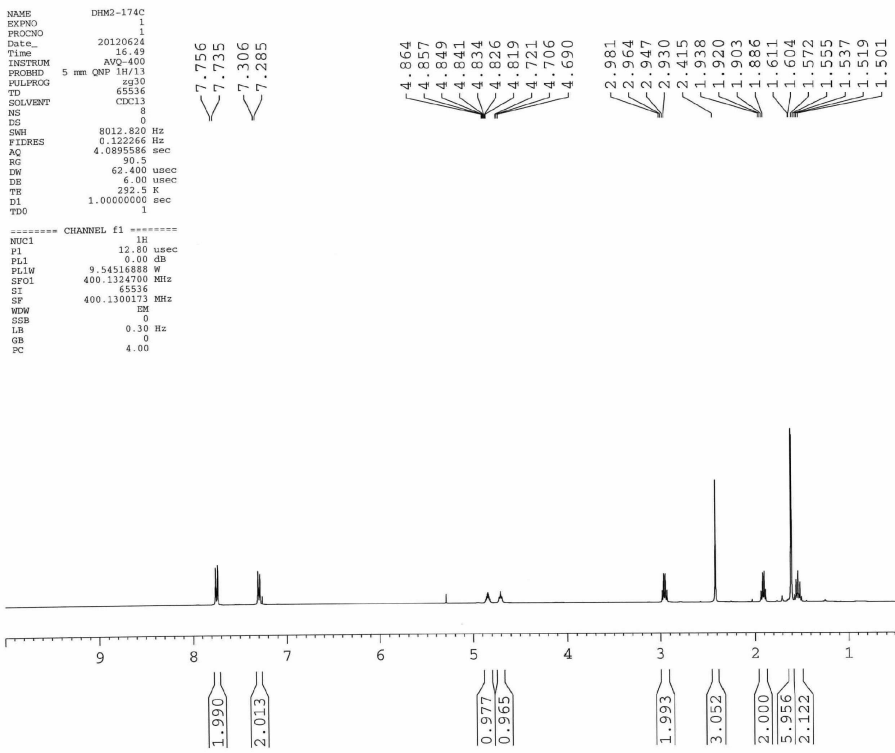
after hivac

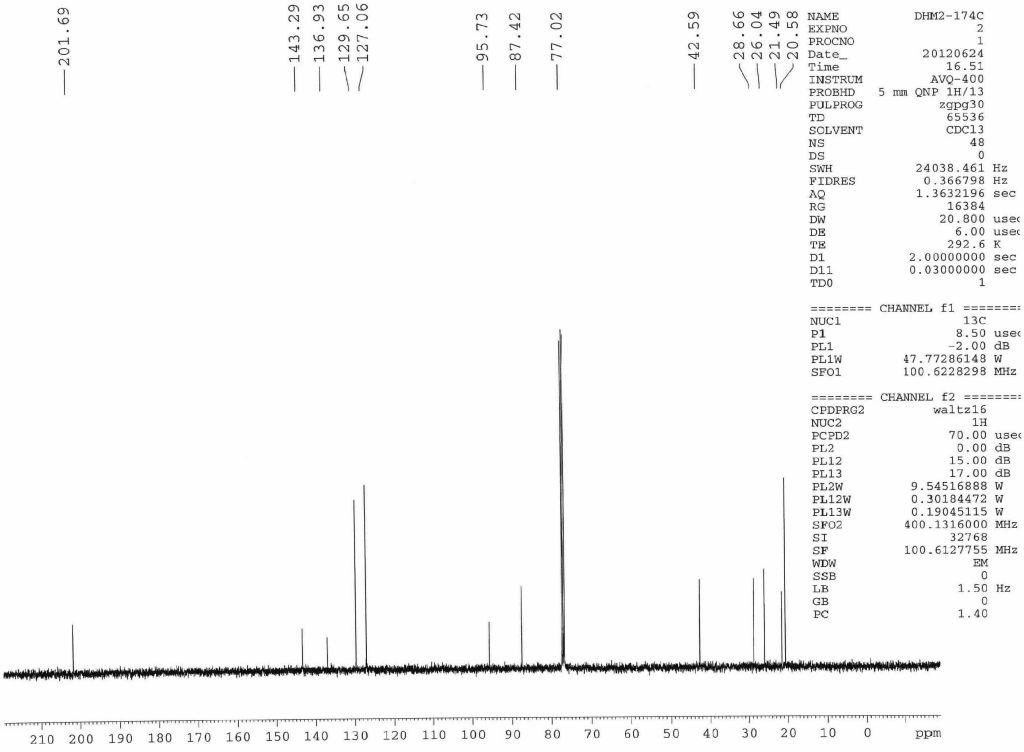


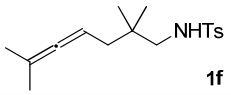



NHTs

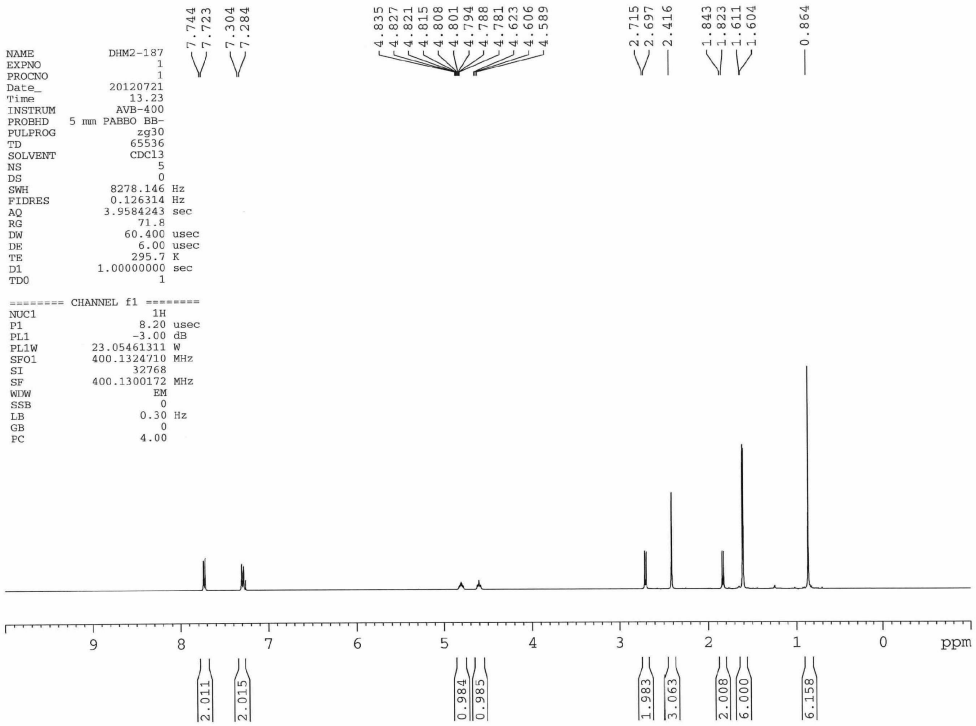
1e

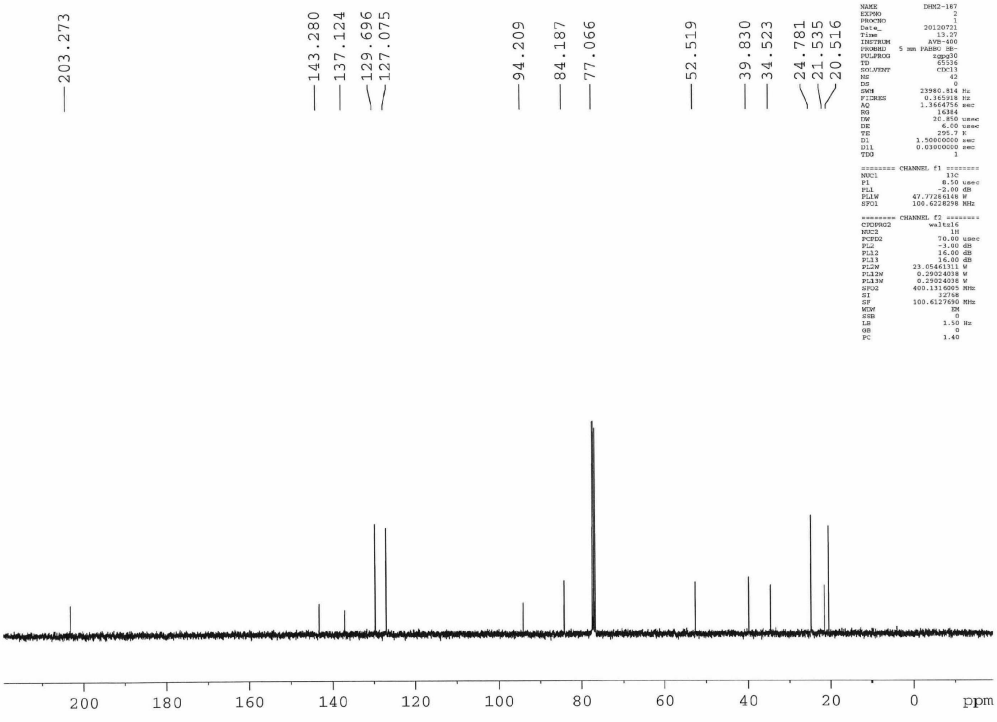

NAME DHM2-174C
EXPNO 1
PROCNO 1
Date_ 20120624
Time 16.49
INSTRUM AVQ-400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0895586 sec
RG 90.5
DW 62.400 usec
DE 6.00 usec
TE 292.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.80 usec
PL1 0.00 dB
PL1W 9.54516888 W
SFO1 400.1324700 MHz
SI 65536
SF 400.1300173 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
7.756
7.735
7.306
7.285
4.864
4.857
4.849
4.841
4.834
4.826
4.819
4.721
4.706
4.690
2.981
2.964
2.947
2.930
2.415
1.938
1.920
1.903
1.886
1.611
1.604
1.572
1.555
1.537
1.519
1.501
9
8
7
6
5
4
3
2
1
0
ppm
1.990
2.013
0.977
0.965
1.993
3.052
2.000
5.956
2.122

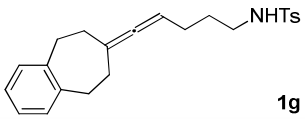

201.69
143.29
136.93
129.65
127.06
95.73
87.42
77.02
42.59
28.66
26.04
21.49
20.58
NAME DHM2-174C
EXPNO 2
PROCNO 1
Date_ 20120624
Time 16.51
INSTRUM AVQ-400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 48
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3632196 sec
RG 16384
DW 20.800 usec
DE 6.00 usec
TE 292.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 47.77286148 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 0.00 dB
PL12 15.00 dB
PL13 17.00 dB
PL2W 9.54516888 W
PL12W 0.30184472 W
PL13W 0.19045115 W
SFO2 400.1316000 MHz
SI 32768
SF 100.6127755 MHz
WDW EM
SSB 0
LB 1.50 Hz
GB 0
PC 1.40
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


NHTs

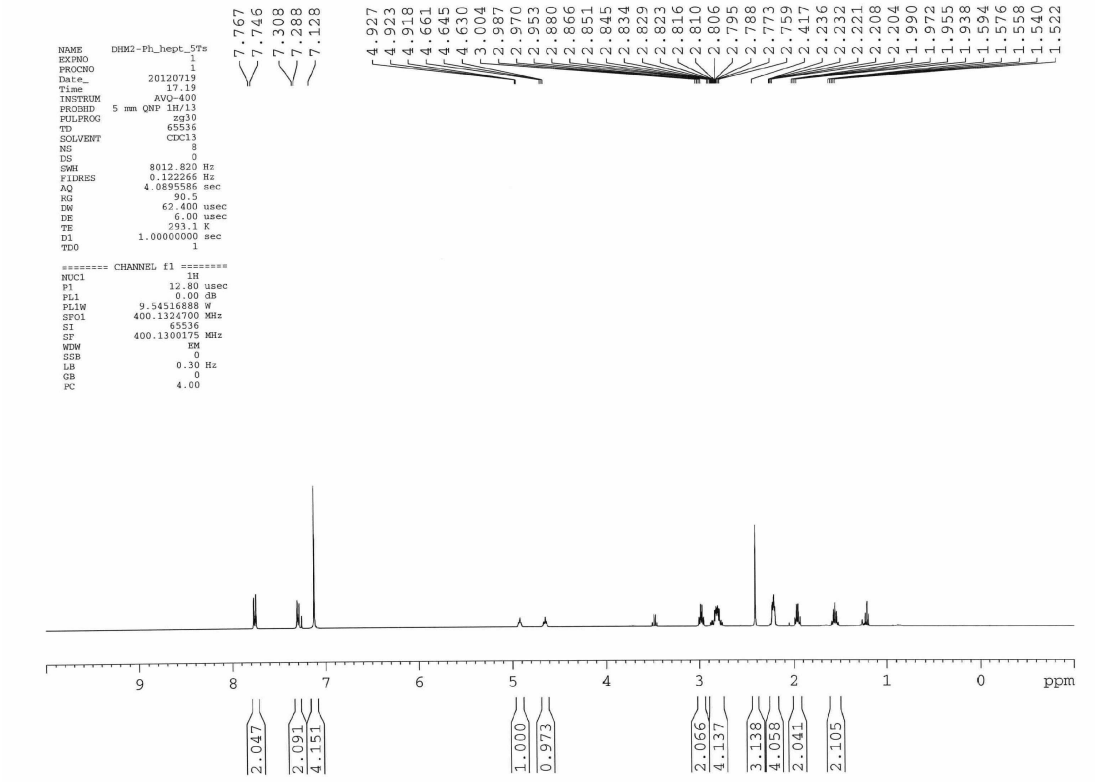
1f

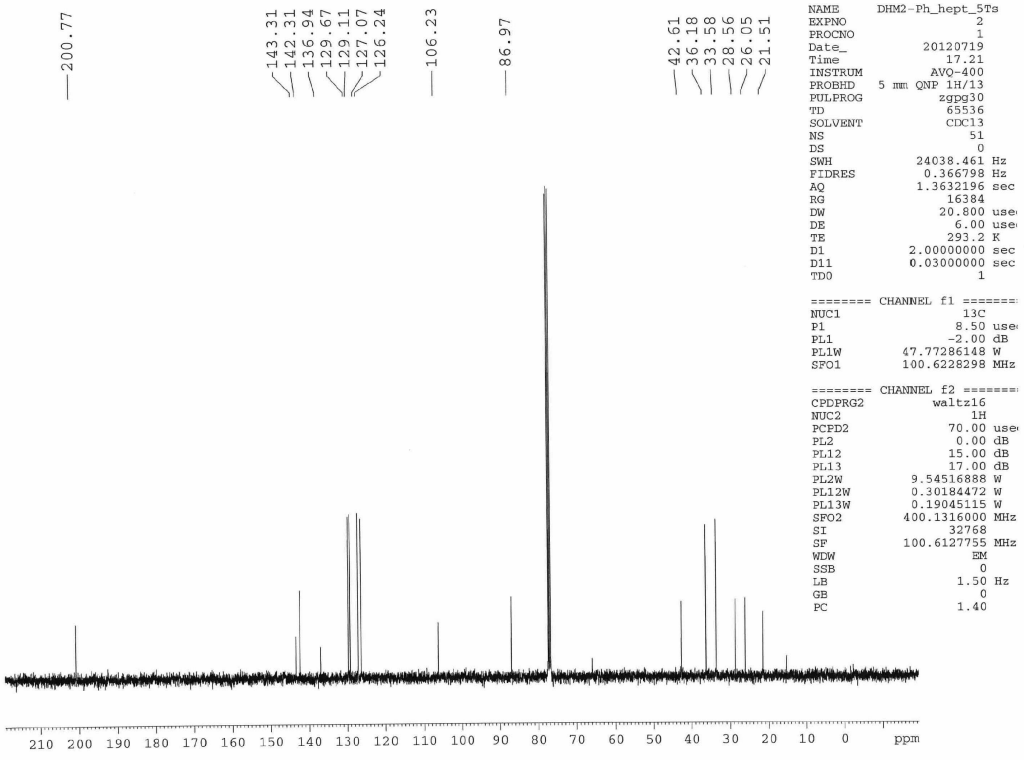

7.744
7.723
7.304
7.284
4.835
4.827
4.821
4.815
4.808
4.801
4.794
4.788
4.781
4.623
4.606
4.589
2.715
2.697
2.416
1.843
1.823
1.611
1.604
0.864
NAME DHM2-187
EXPNO 1
PROCNO 1
Date_ 20120721
Time 13.23
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 5
DS 0
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 71.8
DW 60.400 usec
DE 6.00 usec
TE 295.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.20 usec
PL1 -3.00 dB
PL1W 23.05461311 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300172 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
9
8
7
6
5
4
3
2
1
0
ppm
2.011
2.015
0.984
0.985
1.983
3.063
2.008
6.000
6.158

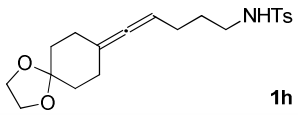

203.273
143.280
137.124
129.696
127.075
94.209
84.187
77.066
52.519
39.830
34.523
24.781
21.535
20.516
NAME DHM2-187
EXPNO 2
PROCNO 1
Date_ 20120721
Time 13.27
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 42
DS 0
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 16384
DW 20.850 usec
DE 6.00 usec
TE 295.7 K
D1 1.50000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 47.77286148 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -3.00 dB
PL12 16.00 dB
PL13 16.00 dB
PL2W 23.05461311 W
PL12W 0.29024038 W
PL13W 0.29024038 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.50 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm


NHTs

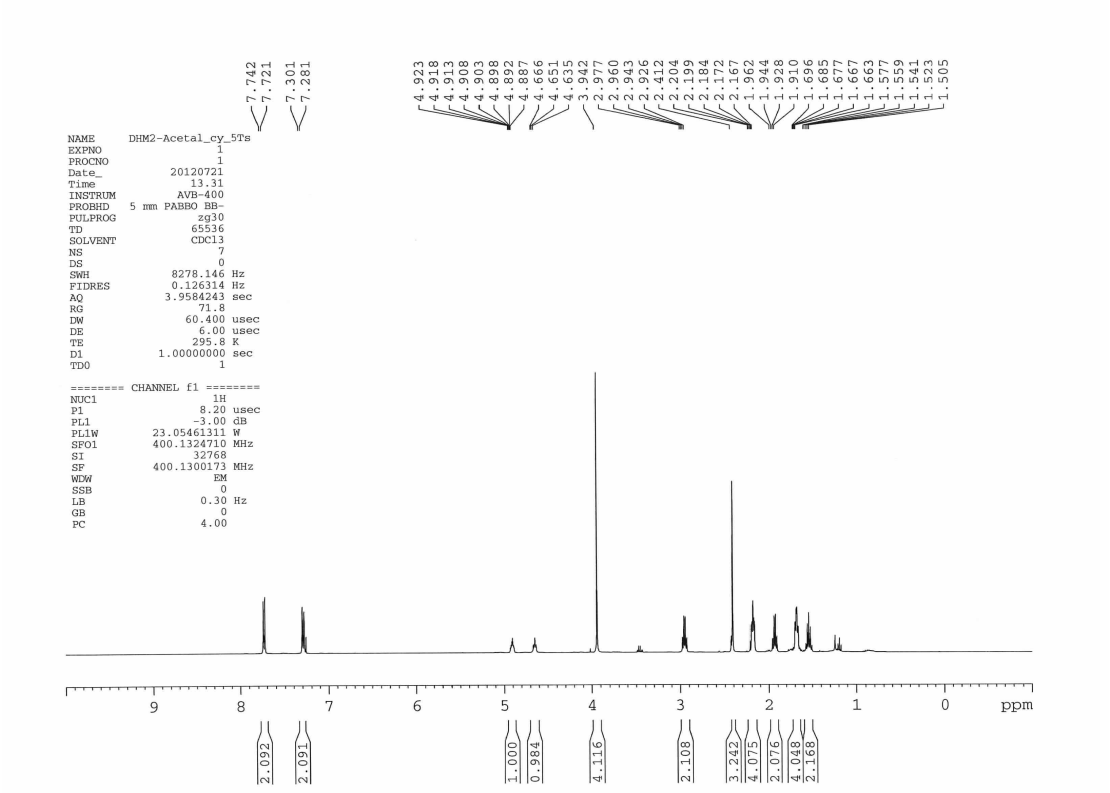
1g

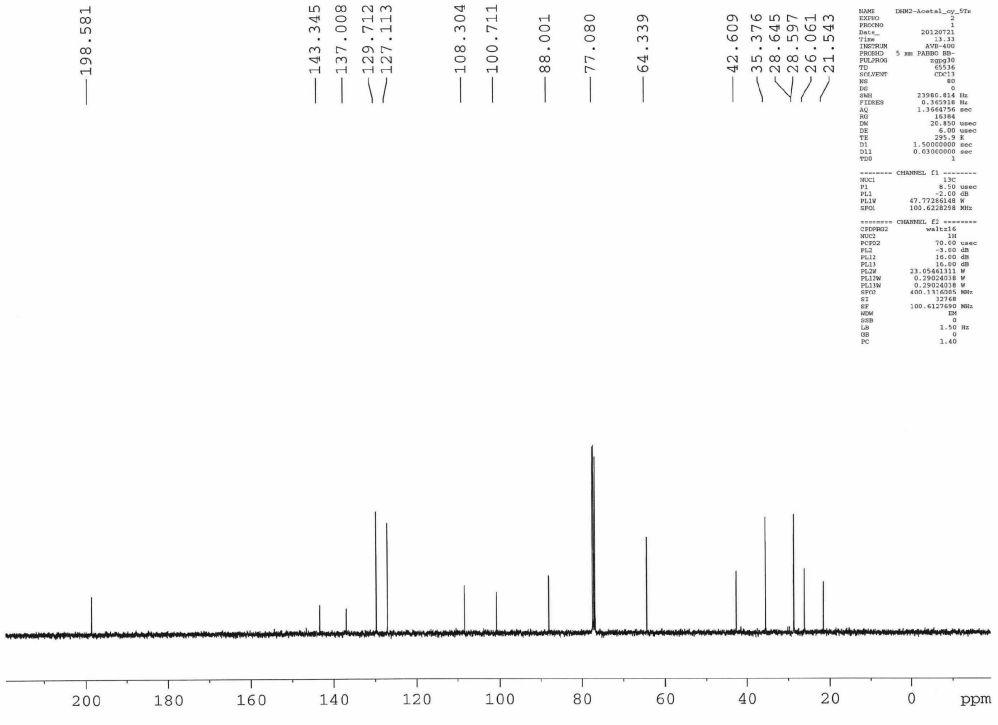

7.767
7.746
7.308
7.288
7.128
4.927
4.923
4.918
4.661
4.645
4.630
3.004
2.987
2.970
2.953
2.880
2.866
2.851
2.845
2.834
2.829
2.823
2.816
2.810
2.806
2.795
2.788
2.773
2.759
2.417
2.236
2.232
2.221
2.208
2.204
1.990
1.972
1.955
1.938
1.594
1.576
1.558
1.540
1.522
NAME DHM2-Ph_hept_5Ts
EXPNO 1
PROCNO 1
Date_ 20120719
Time 17.19
INSTRUM AVQ-400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0895586 sec
RG 90.5
DW 62.400 usec
DE 6.00 usec
TE 293.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.80 usec
PL1 0.00 dB
PL1W 9.54516888 W
SFO1 400.1324700 MHz
SI 65536
SF 400.1300175 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
9 8 7 6 5 4 3 2 1 0 ppm
2.047
2.091
4.151
1.000
0.973
2.066
4.137
3.138
4.058
2.041
2.105

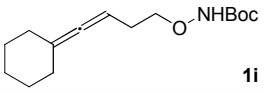

200.77
143.31
142.31
136.94
129.67
129.11
127.07
126.24
106.23
86.97
42.61
36.18
33.58
28.56
26.05
21.51
NAME DHM2-Ph_hept_5Ts
EXPNO 2
PROCNO 1
Date_ 20120719
Time 17.21
INSTRUM AVQ-400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 51
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3632196 sec
RG 16384
DW 20.800 use
DE 6.00 use
TE 293.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 use
PL1 -2.00 dB
PL1W 47.77286148 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 use
PL2 0.00 dB
PL12 15.00 dB
PL13 17.00 dB
PL2W 9.54516888 W
PL12W 0.30184472 W
PL13W 0.19045115 W
SFO2 400.1316000 MHz
SI 32768
SF 100.6127755 MHz
WDW EM
SSB 0
LB 1.50 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


NHTs
O
O

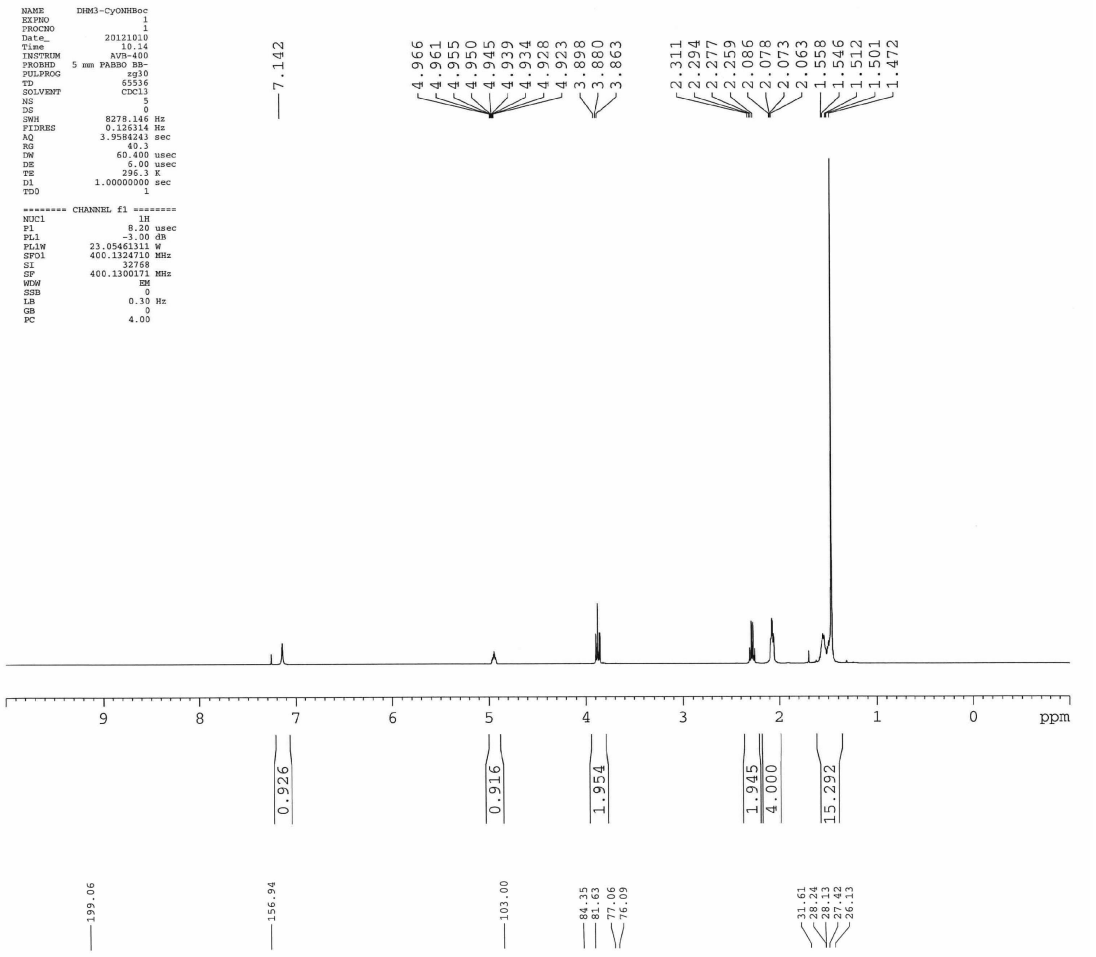
1h

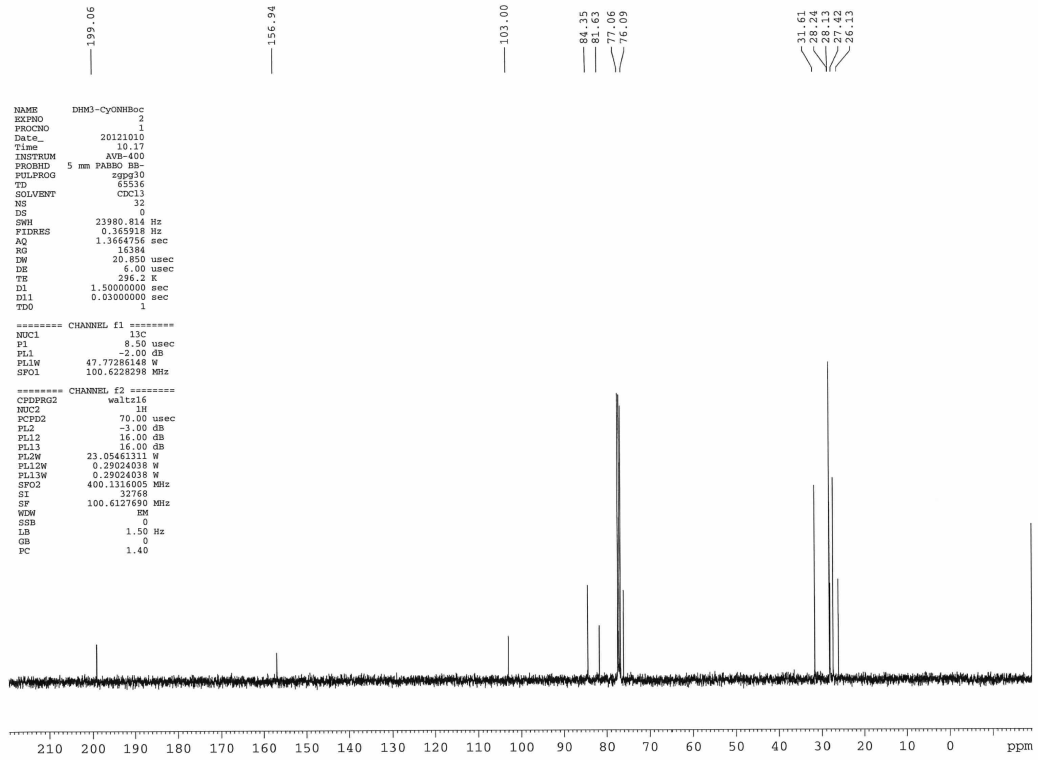

7.742
7.721
7.301
7.281
4.923
4.918
4.913
4.908
4.903
4.898
4.892
4.887
4.666
4.651
4.635
3.942
2.977
2.960
2.943
2.926
2.412
2.204
2.199
2.184
2.172
2.167
1.962
1.944
1.928
1.910
1.696
1.685
1.677
1.667
1.663
1.577
1.559
1.541
1.523
1.505
NAME DHM2-Acetal_cy_5Ts
EXPNO 1
PROCNO 1
Date_ 20120721
Time 13.31
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 7
DS 0
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 71.8
DW 60.400 usec
DE 6.00 usec
TE 295.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.20 usec
PL1 -3.00 dB
PL1W 23.05461311 W
SFO1 400.1324710 MHz
SI 32768
SP 400.1300173 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
9
8
7
6
5
4
3
2
1
0
ppm
2.092
2.091
1.000
0.984
4.116
2.108
3.242
4.075
2.076
4.048
2.168

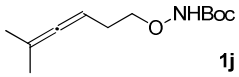

198.581
143.345
137.008
129.712
127.113
108.304
100.711
88.001
77.080
64.339
42.609
35.376
28.645
28.597
26.061
21.543
NAME DHM2-Acetal_cy_5Ts
EXPNO 2
PROCNO 1
Date_ 20120721
Time 13.33
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 80
DS 0
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 16384
DW 20.850 usec
DE 6.00 usec
TE 295.9 K
D1 1.50000000 sec
D11 0.03060000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 2.00 dB
PL1W 47.77286148 W
SFO1 100.6228258 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 10.60 usec
PL2 -3.00 dB
PL12 16.00 dB
PL13 16.00 dB
PL2W 23.05461311 W
PL12W 0.29024038 W
PL13W 0.29024038 W
SFO2 400.1316458 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.50 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm


NHBoc
O

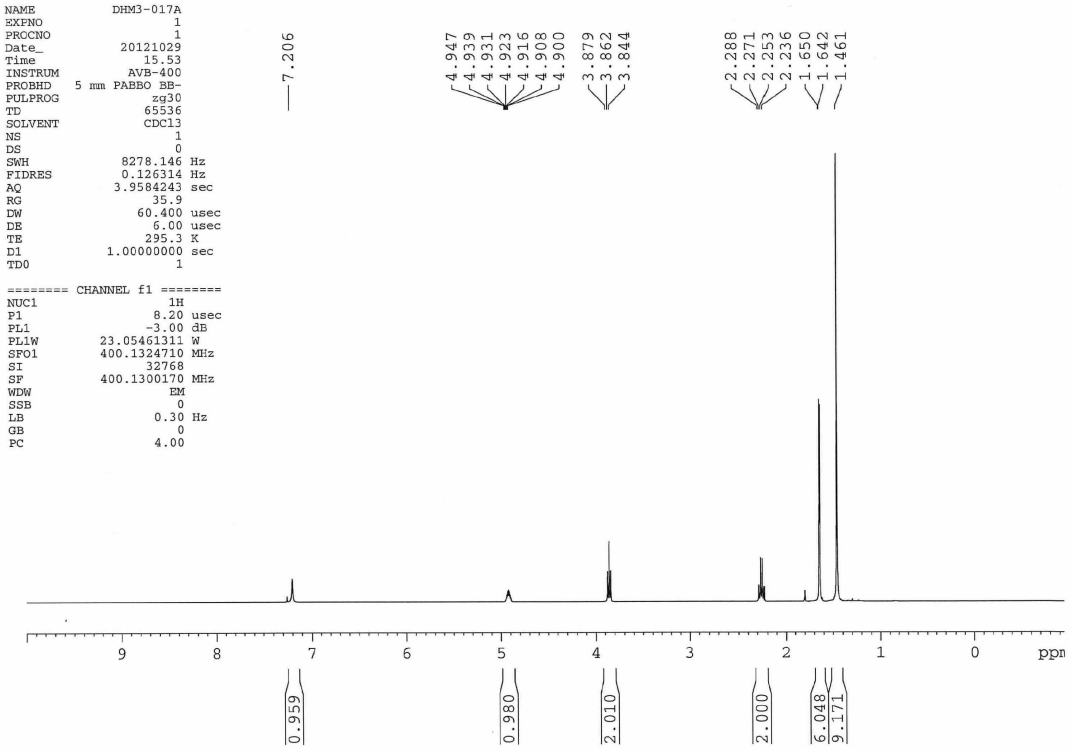
1i

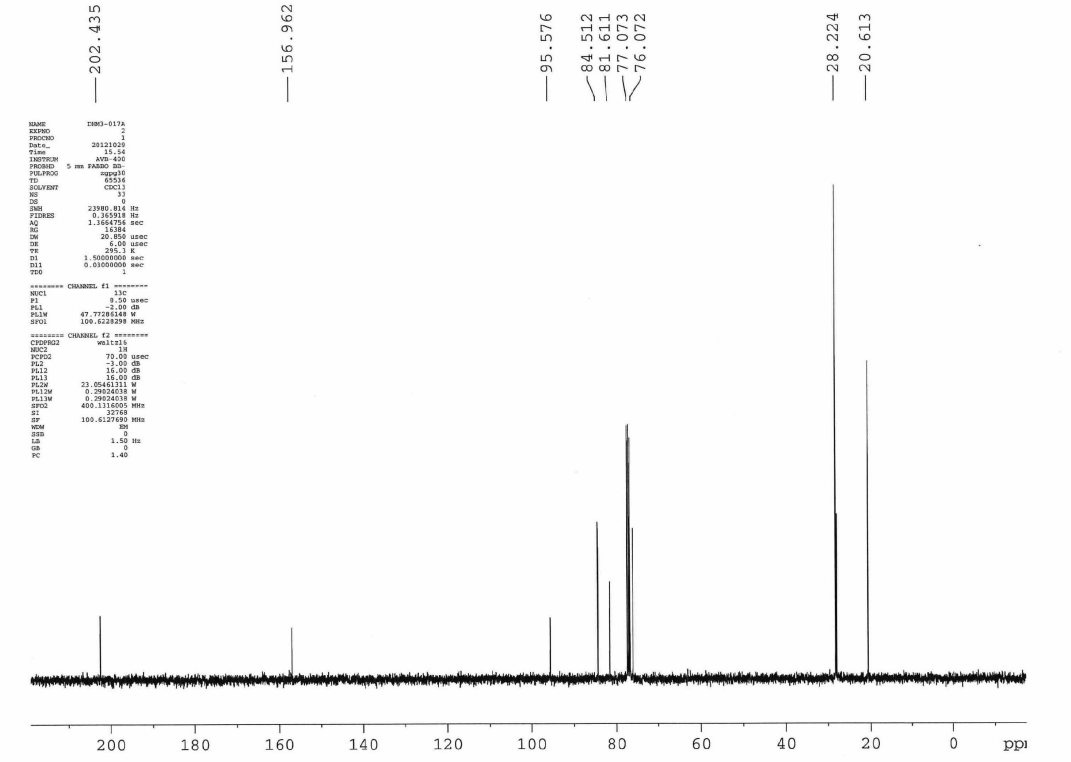

NAME DHM3-CyONHBoc
EXPNO 1
PROCNO 1
Date_ 20121010
Time 10.14
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 5
DS 0
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 40.3
DW 60.400 usec
DE 6.00 usec
TE 296.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.20 usec
PL1 -3.00 dB
PL1W 23.05461311 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300171 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
7.142
4.966
4.961
4.955
4.950
4.945
4.939
4.934
4.928
4.923
3.898
3.880
3.863
2.311
2.294
2.277
2.259
2.086
2.078
2.073
2.063
1.558
1.546
1.512
1.501
1.472
9
8
7
6
5
4
3
2
1
0
ppm
0.926
0.916
1.954
1.945
4.000
15.292

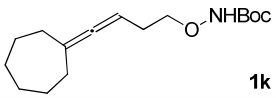

199.06
156.94
103.00
84.35
81.63
77.06
76.09
31.61
28.24
28.13
27.42
26.13
NAME DHM3-CyONHBoc
EXPNO 2
PROCNO 1
Date_ 20121010
Time 10.17
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 32
DS 0
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 16384
DW 20.850 usec
DE 6.00 usec
TE 296.2 K
D1 1.50000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 47.77286148 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -3.00 dB
PL12 16.00 dB
PL13 16.00 dB
PL2W 23.05461311 W
PL12W 0.29024038 W
PL13W 0.29024038 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.50 Hz
GB 0
PC 1.40
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


NHBoc
O

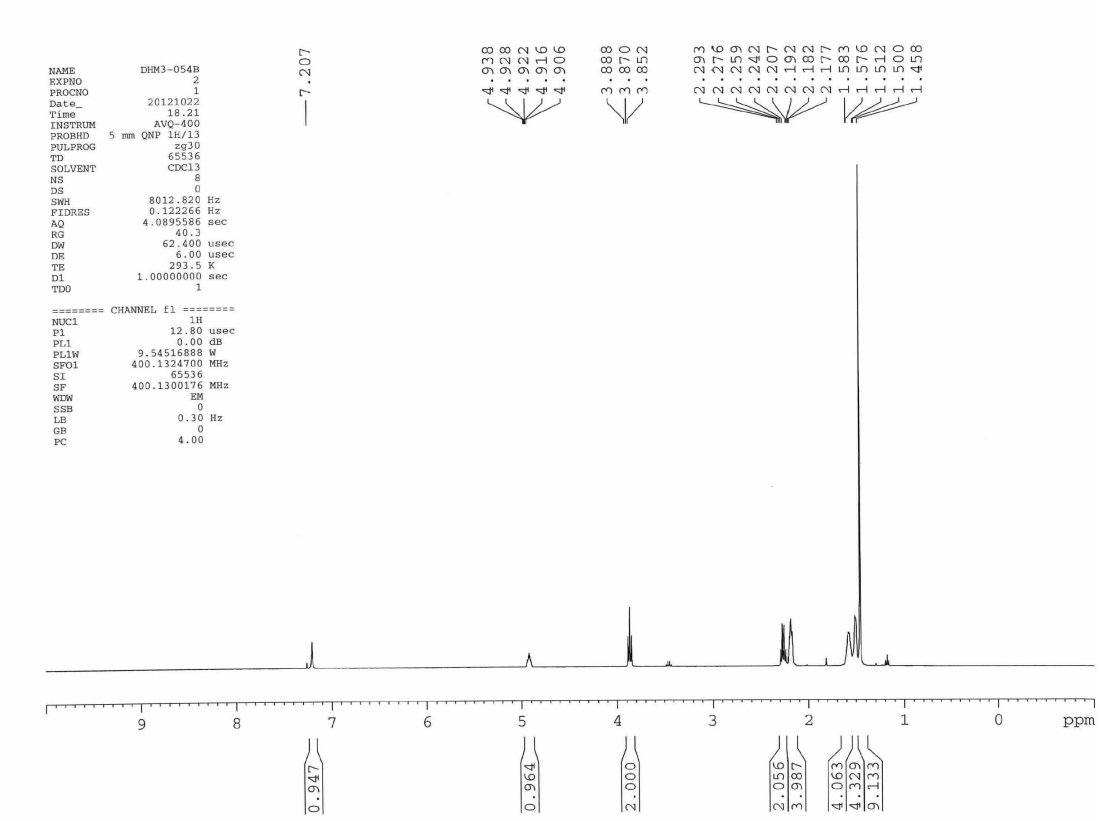
1j

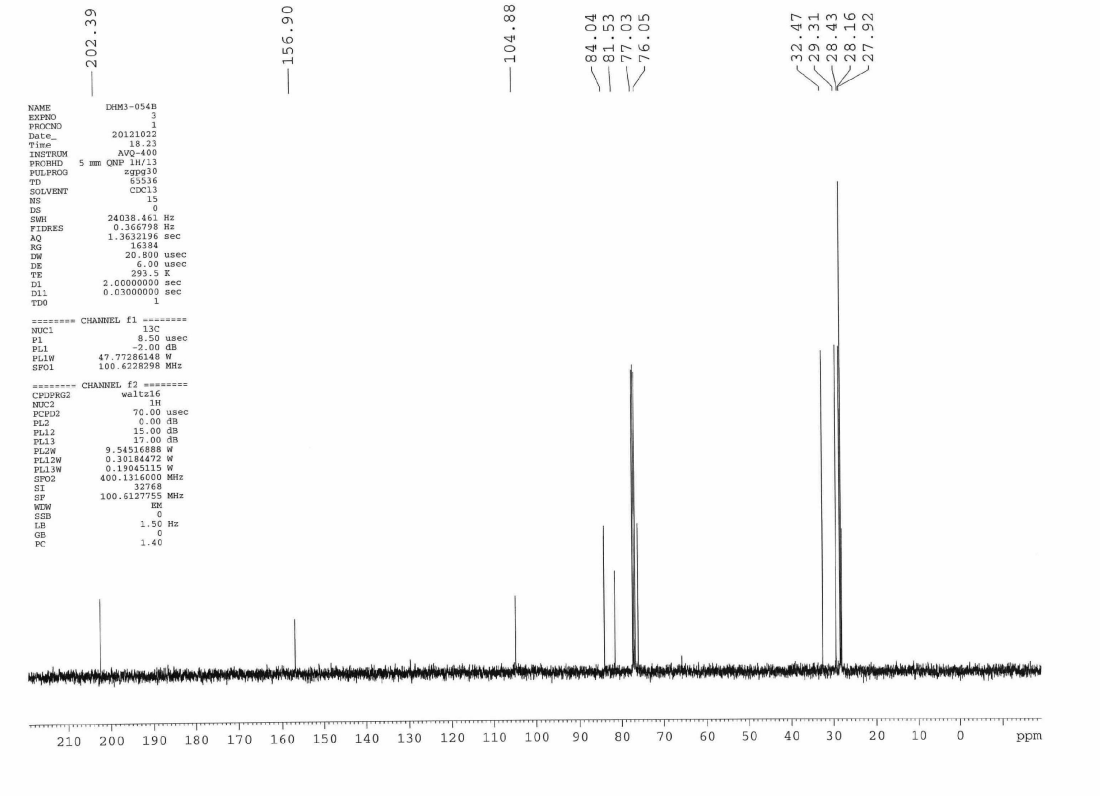

NAME DHM3-017A
EXPNO 1
PROCNO 1
Date_ 20121029
Time 15.53
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 35.9
DW 60.400 usec
DE 6.00 usec
TE 295.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.20 usec
PL1 -3.00 dB
PL1W 23.05461311 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300170 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
7.206
4.947
4.939
4.931
4.923
4.916
4.908
4.900
3.879
3.862
3.844
2.288
2.271
2.253
2.236
1.650
1.642
1.461
0.959
0.980
2.010
2.000
6.048
9.171
ppm

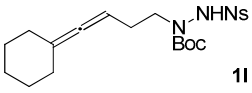

202.435
156.962
95.576
84.512
81.611
77.073
76.072
28.224
20.613
ppm


NHBoc
O

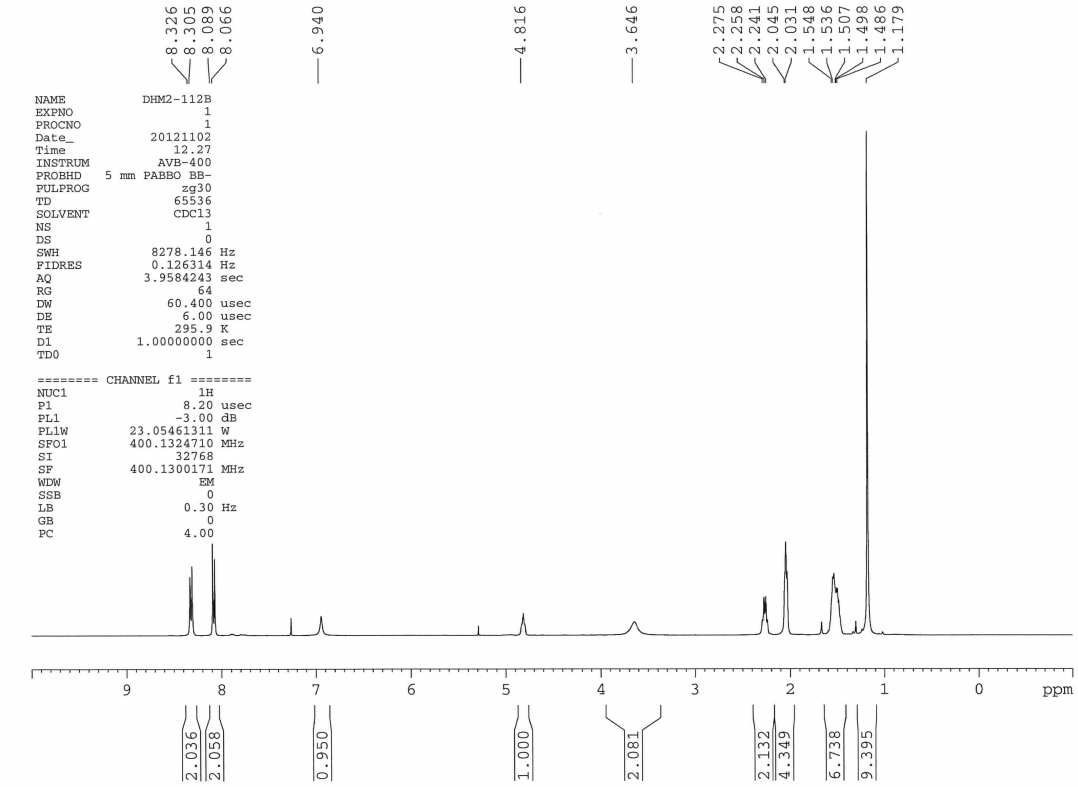
1k

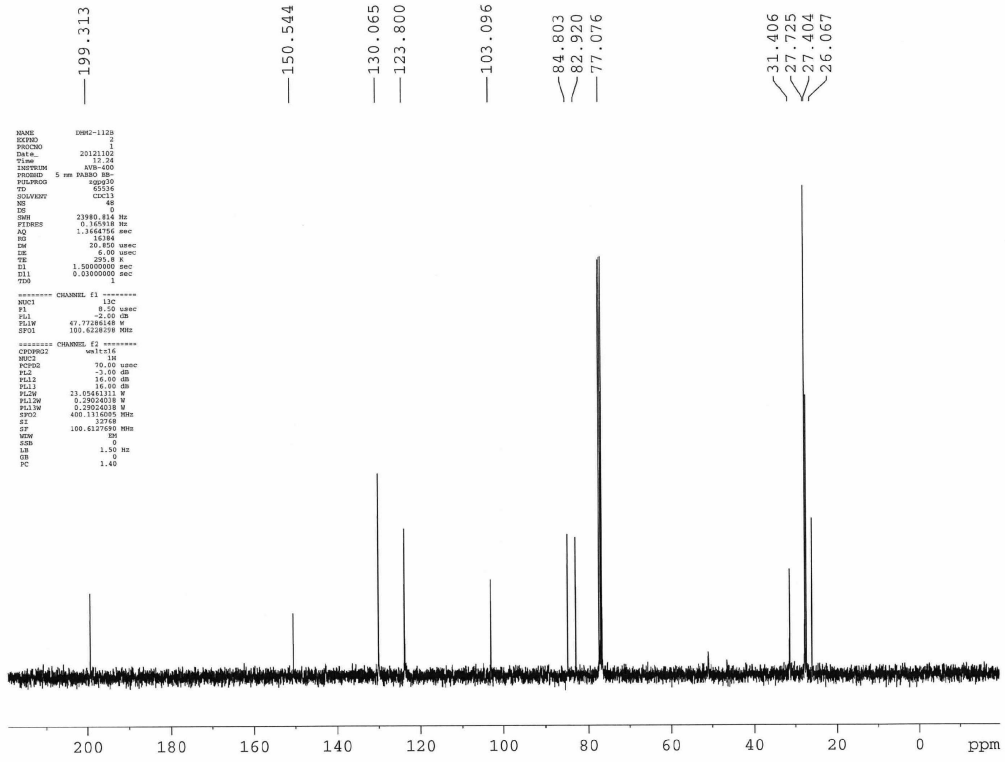

NAME DHM3-054B
EXPNO 2
PROCNO 1
Date_ 20121022
Time 18.21
INSTRUM AVQ-400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0895586 sec
RG 40.3
DW 62.400 usec
DE 6.00 usec
TE 293.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.80 usec
PL1 0.00 dB
PL1W 9.54516888 W
SFO1 400.1324700 MHz
SI 65536
SF 400.1300176 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
7.207
4.938
4.928
4.922
4.916
4.906
3.888
3.870
3.852
2.293
2.276
2.259
2.242
2.207
2.192
2.182
2.177
1.583
1.576
1.512
1.500
1.458
9 8 7 6 5 4 3 2 1 0 ppm
0.947
0.964
2.000
2.056
3.987
4.063
4.329
9.133

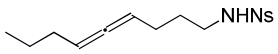

202.39
156.90
104.88
84.04
81.53
77.03
76.05
32.47
29.31
28.43
28.16
27.92
NAME DHM3-054B
EXPNO 3
PROCNO 1
Date_ 20121022
Time 18.23
INSTRUM AVQ-400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 15
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3632196 sec
RG 16384
DW 20.800 usec
DE 6.00 usec
TE 293.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 47.77286148 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 0.00 dB
PL12 15.00 dB
PL13 17.00 dB
PL2W 9.54516888 W
PL12W 0.30184472 W
PL13W 0.19045115 W
SFO2 400.1316000 MHz
SI 32768
SF 100.6127755 MHz
WDW EM
SSB 0
LB 1.50 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


NHNs
N
Boc

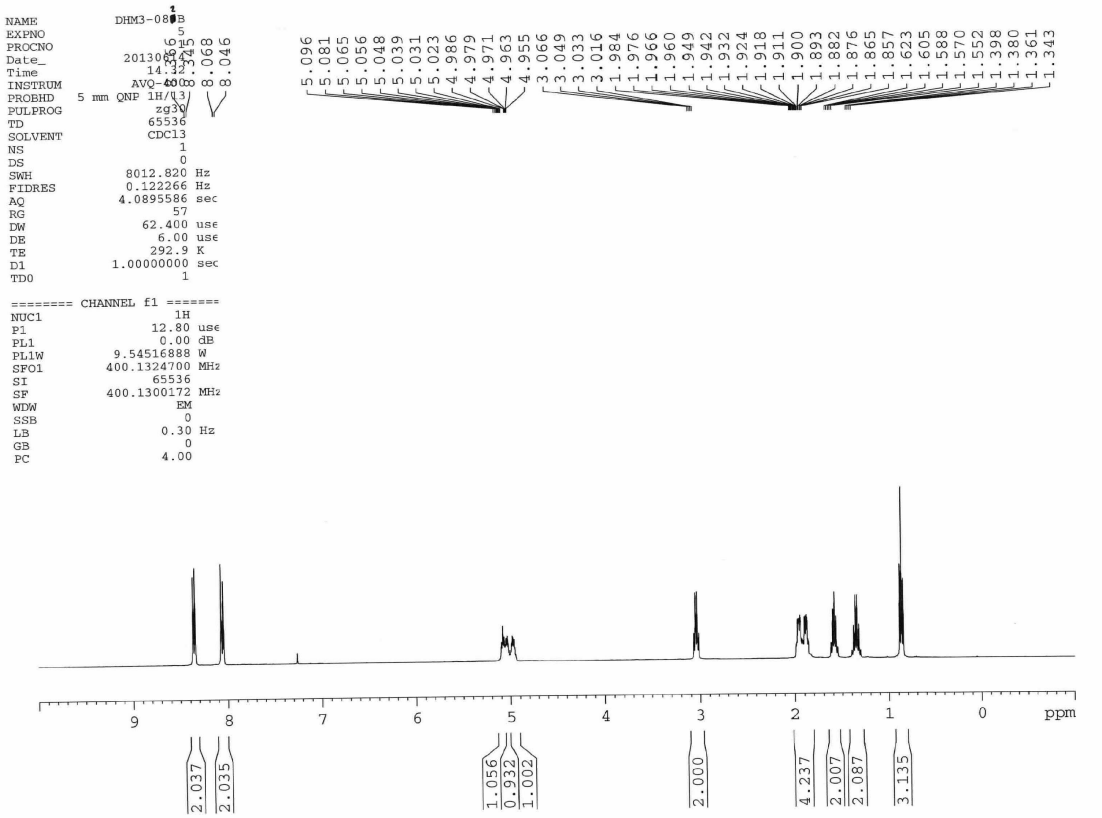
1l

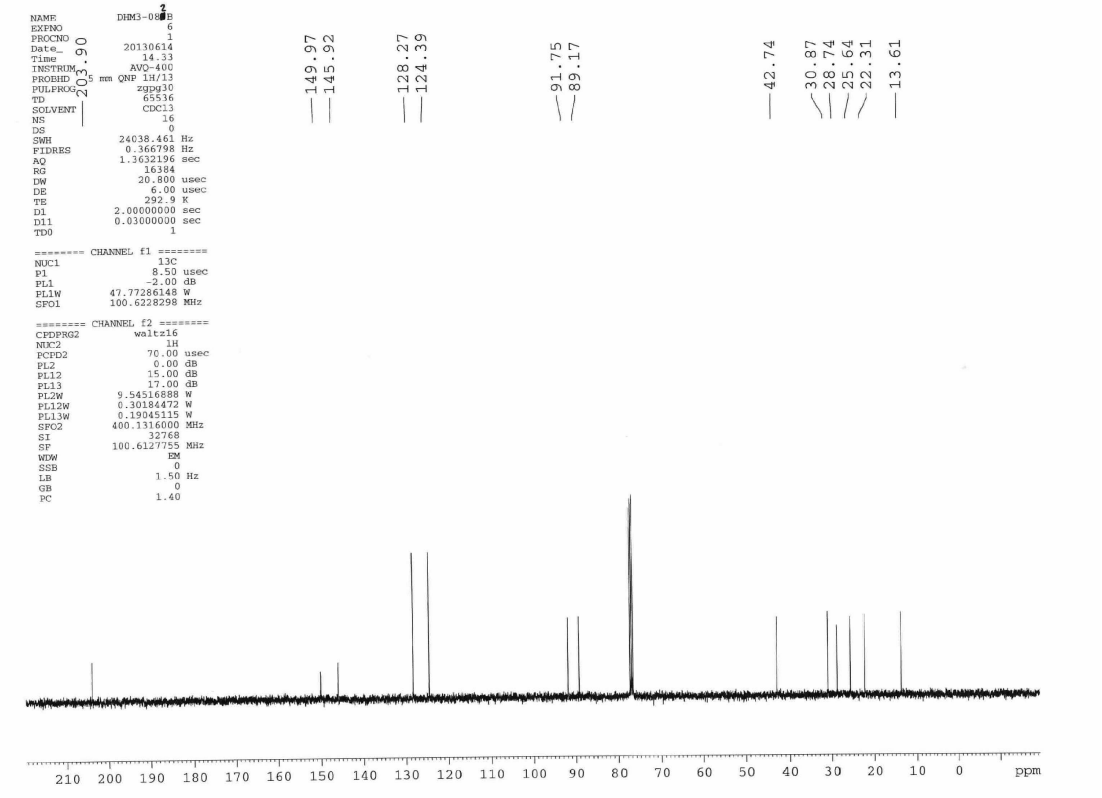

8.326
8.305
8.089
8.066
6.940
4.816
3.646
2.275
2.258
2.241
2.045
2.031
1.548
1.536
1.507
1.498
1.486
1.179
NAME DHM2-112B
EXPNO 1
PROCNO 1
Date_ 20121102
Time 12.27
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 64
DW 60.400 usec
DE 6.00 usec
TE 295.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.20 usec
PL1 -3.00 dB
PL1W 23.05461311 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300171 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
9
8
7
6
5
4
3
2
1
0
ppm
2.036
2.058
0.950
1.000
2.081
2.132
4.349
6.738
9.395

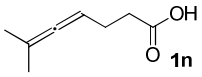

199.313
150.544
130.065
123.800
103.096
84.803
82.920
77.076
31.406
27.725
27.404
26.067
200
180
160
140
120
100
80
60
40
20
0
ppm


NHNs

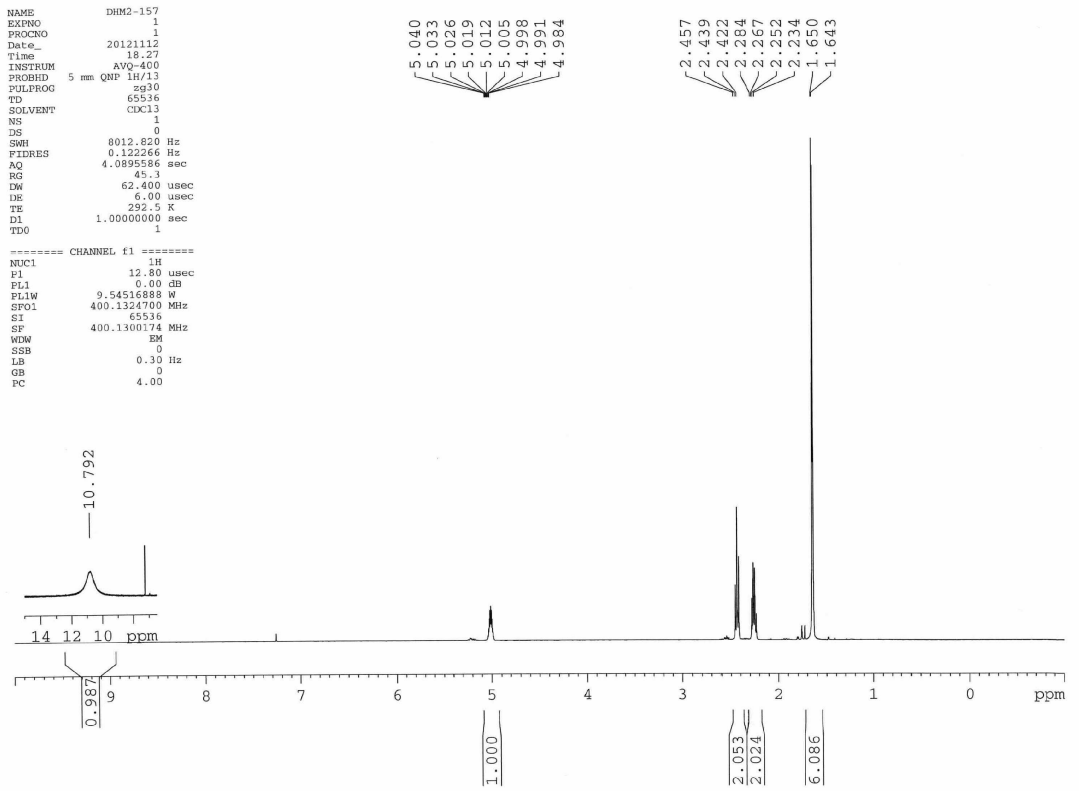
1m

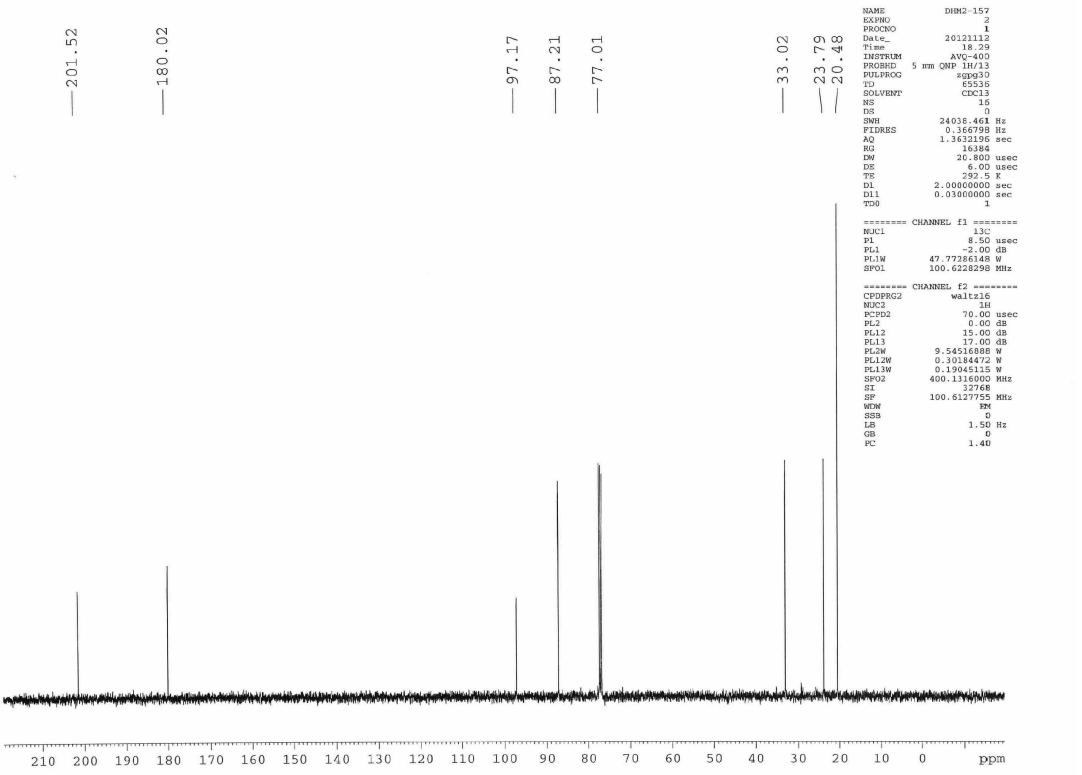

NAME DHM3-088B
EXPNO 5
PROCNO 1
Date_ 20130614
Time 14.32
INSTRUM AVQ-400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0895586 sec
RG 57
DW 62.400 use
DE 6.00 use
TE 292.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.80 use
PL1 0.00 dB
PL1W 9.54516888 W
SFO1 400.1324700 MHz
SI 65536
SF 400.1300172 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
7.334
8.068
8.046
5.096
5.081
5.065
5.056
5.048
5.039
5.031
5.023
4.986
4.979
4.971
4.963
4.955
3.066
3.049
3.033
3.016
1.984
1.976
1.966
1.960
1.949
1.942
1.932
1.924
1.918
1.911
1.900
1.893
1.882
1.876
1.865
1.857
1.623
1.605
1.588
1.570
1.552
1.398
1.380
1.361
1.343
9
8
7
6
5
4
3
2
1
0
ppm
2.037
2.035
1.056
0.932
1.002
2.000
4.237
2.007
2.087
3.135

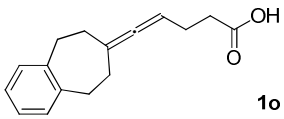

NAME DHM3-088B
EXPNO 6
PROCNO 1
Date_ 20130614
Time 14.33
INSTRUM AVQ-400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3632196 sec
RG 16384
DW 20.800 usec
DE 6.00 usec
TE 292.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 47.77286148 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 0.00 dB
PL12 15.00 dB
PL13 17.00 dB
PL2W 9.54516888 W
PL12W 0.30184472 W
PL13W 0.19045115 W
SFO2 400.1316000 MHz
SI 32768
SF 100.6127755 MHz
WDW EM
SSB 0
LB 1.50 Hz
GB 0
PC 1.40
203.90
149.97
145.92
128.27
124.39
91.75
89.17
42.74
30.87
28.74
25.64
22.31
13.61
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


OH
O

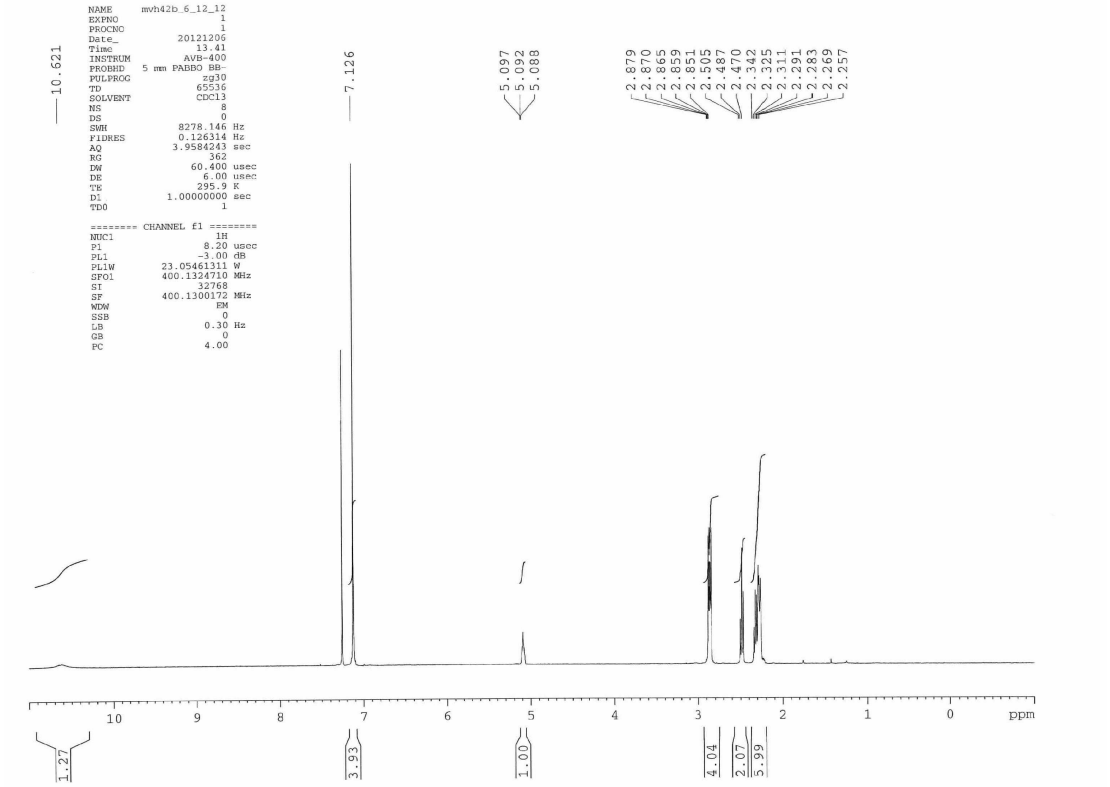
1n

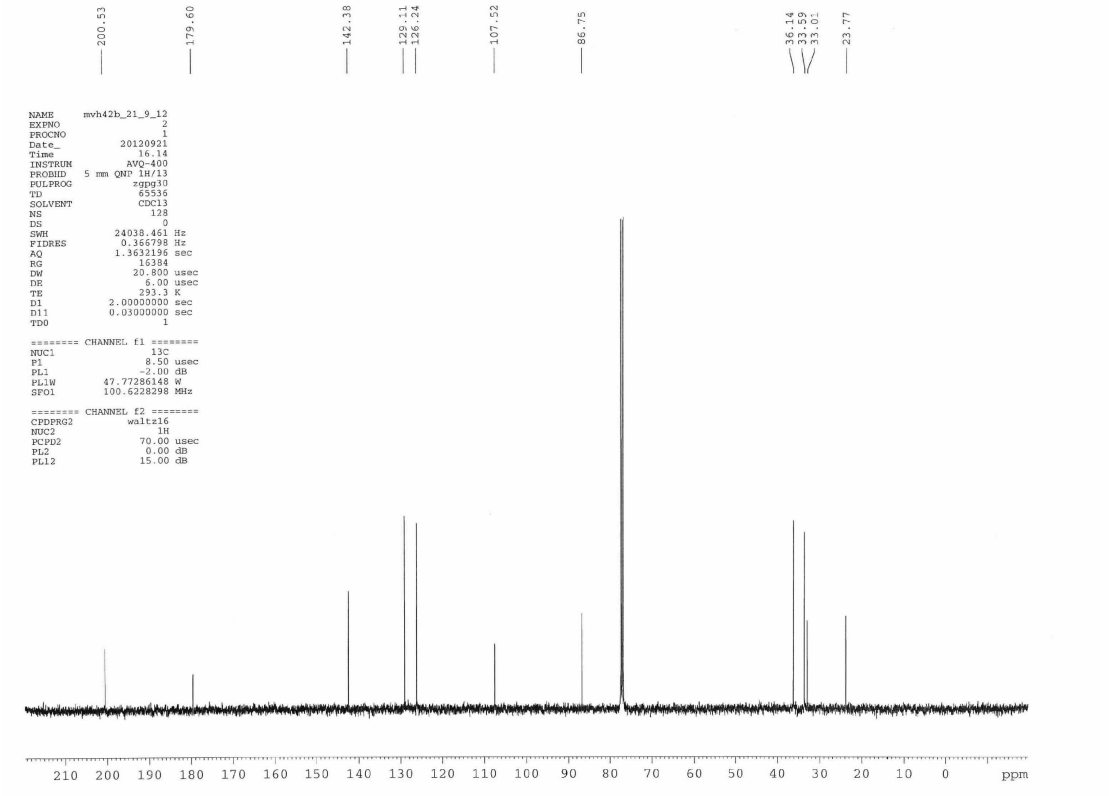

NAME DHM2-157
EXPNO 1
PROCNO 1
Date_ 20121112
Time 18.27
INSTRUM AVQ-400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0895586 sec
RG 45.3
DW 62.400 usec
DE 6.00 usec
TE 292.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.80 usec
PL1 0.00 dB
PL1W 9.54516888 W
SFO1 400.1324700 MHz
SI 65536
SF 400.1300174 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
5.040
5.033
5.026
5.019
5.012
5.005
4.998
4.991
4.984
2.457
2.439
2.422
2.284
2.267
2.252
2.234
1.650
1.643
10.792
14 12 10 ppm
0.987
1.000
2.053
2.024
6.086
ppm

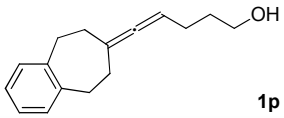

201.52
180.02
97.17
87.21
77.01
33.02
23.79
20.48
NAME DHM2-157
EXPNO 2
PROCNO 1
Date_ 20121112
Time 18.29
INSTRUM AVQ-400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3632196 sec
RG 16384
DW 20.800 usec
DE 6.00 usec
TE 292.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 47.77286148 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 0.00 dB
PL12 15.00 dB
PL13 17.00 dB
PL2W 9.54516888 W
PL12W 0.30184472 W
PL13W 0.19045115 W
SFO2 400.1316000 MHz
SI 32768
SF 100.6127755 MHz
WDW EM
SSB 0
LB 1.50 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


OH
O

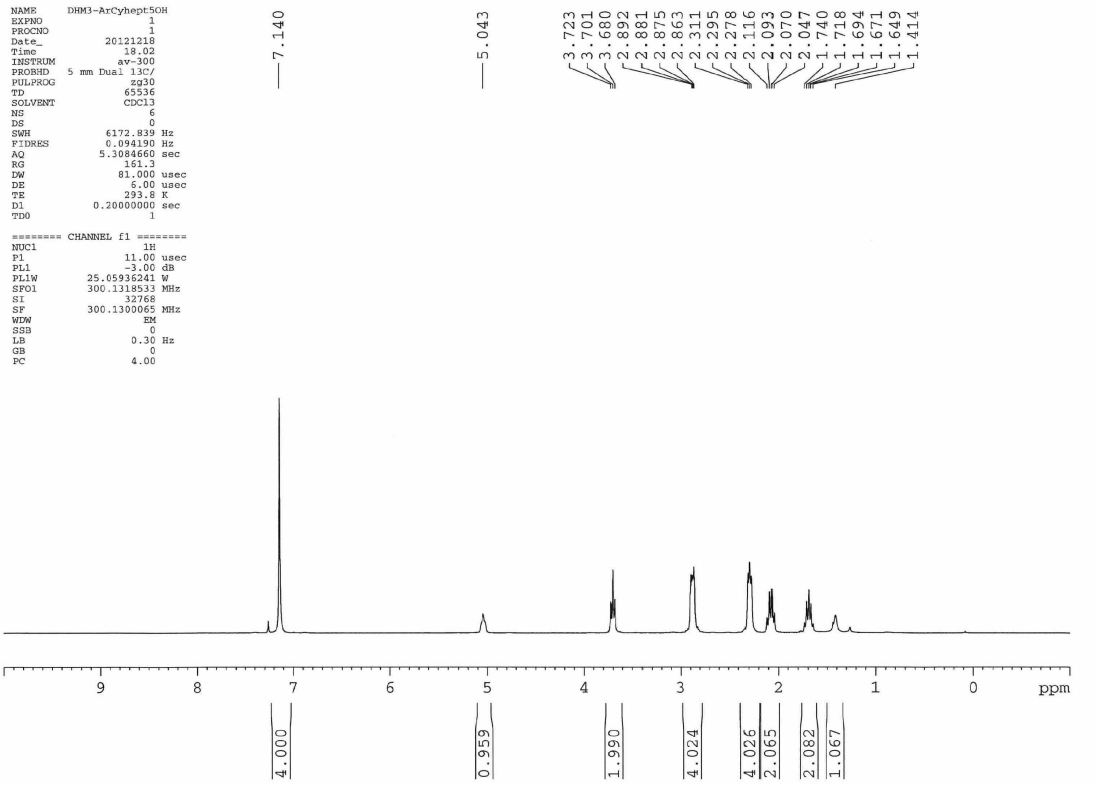
1o

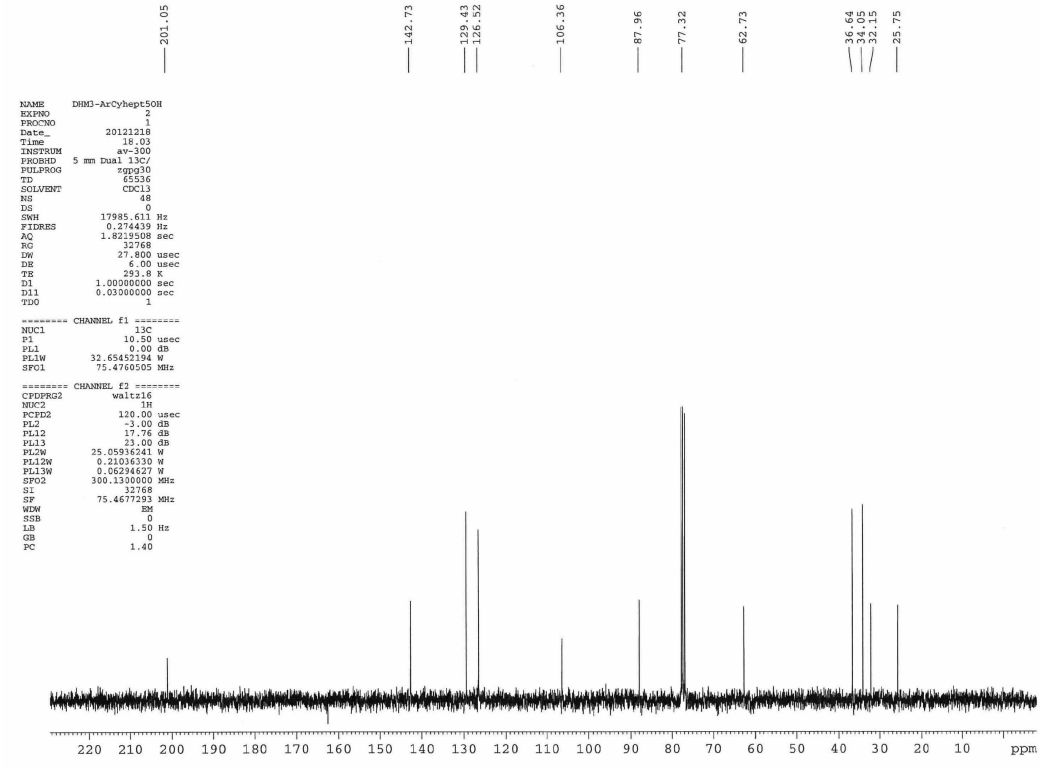

NAME mvh42b_6_12_12
EXPNO 1
PROCNO 1
Date_ 20121206
Time 13.41
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DW 60.400 usec
DE 6.00 usec
TE 295.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.20 usec
PL1 -3.00 dB
PL1W 23.05461311 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300172 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
10.621
7.126
5.097
5.092
5.088
2.879
2.870
2.865
2.859
2.851
2.505
2.487
2.470
2.342
2.325
2.311
2.291
2.283
2.269
2.257
ppm
1.27
3.93
1.00
4.04
2.07
5.99

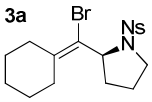

200.53
179.60
142.38
129.11
126.24
107.52
86.75
36.14
33.59
33.01
23.77
NAME mvh42b_21_9_12
EXPNO 2
PROCNO 1
Date_ 20120921
Time 16.14
INSTRUM AVQ-400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3632196 sec
RG 16384
DW 20.800 usec
DE 6.00 usec
TE 293.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 47.77286148 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 0.00 dB
PL12 15.00 dB
ppm

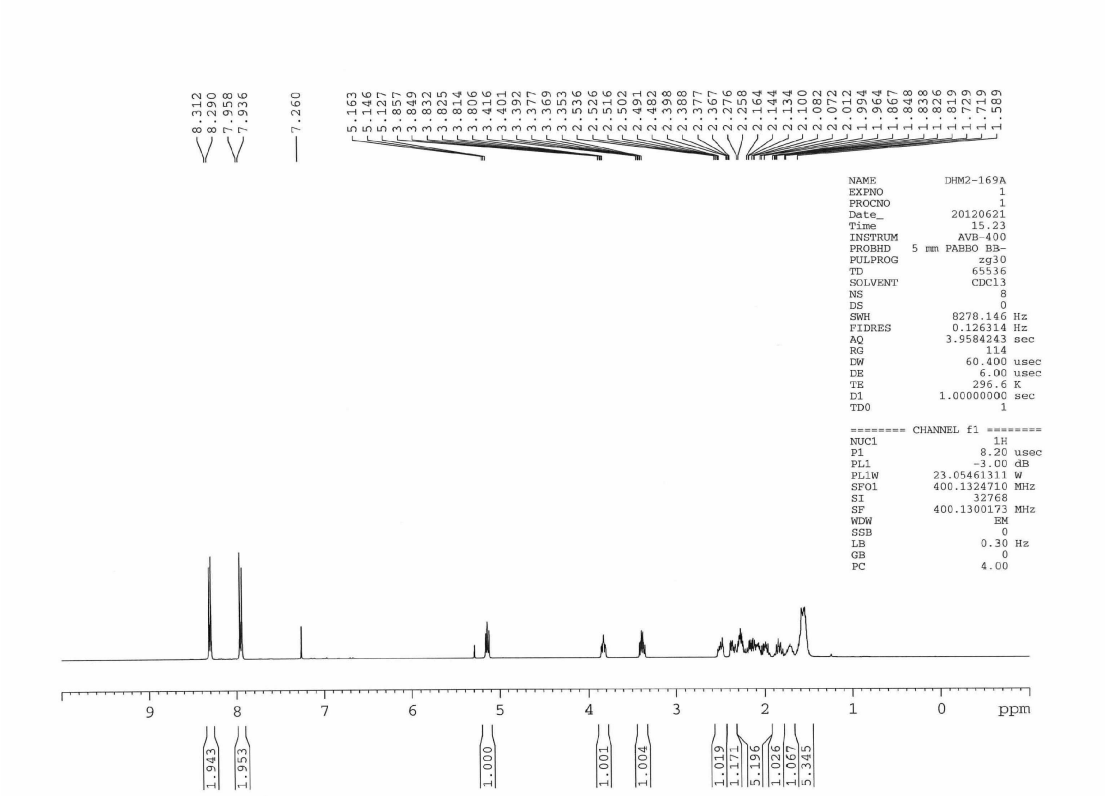

OH

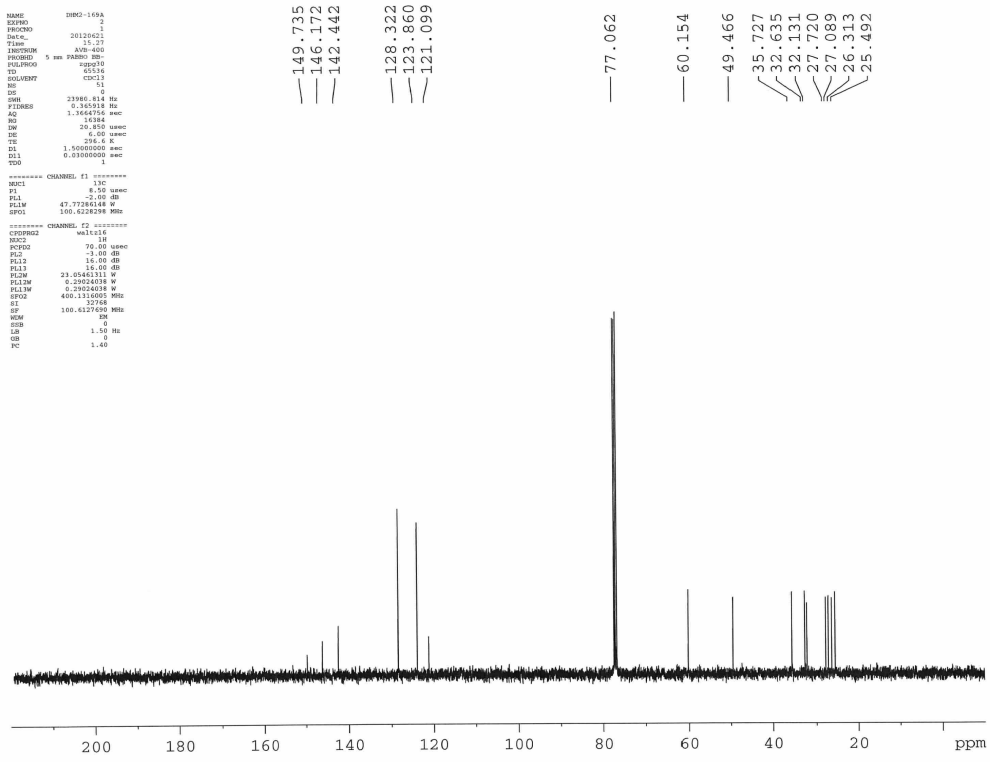
1p

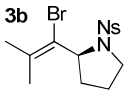

NAME DHM3-ArCyhept5OH
EXPNO 1
PROCNO 1
Date_ 20121218
Time 18.02
INSTRUM av-300
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 6
DS 0
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 161.3
DW 81.000 usec
DE 6.00 usec
TE 293.8 K
D1 0.20000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 -3.00 dB
PL1W 25.05936241 W
SFO1 300.1318533 MHz
SI 32768
SF 300.1300065 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
7.140
5.043
3.723
3.701
3.680
2.892
2.881
2.875
2.863
2.311
2.295
2.278
2.116
2.093
2.070
2.047
1.740
1.718
1.694
1.671
1.649
1.414
9
8
7
6
5
4
3
2
1
0
ppm
4.000
0.959
1.990
4.024
4.026
2.065
2.082
1.067

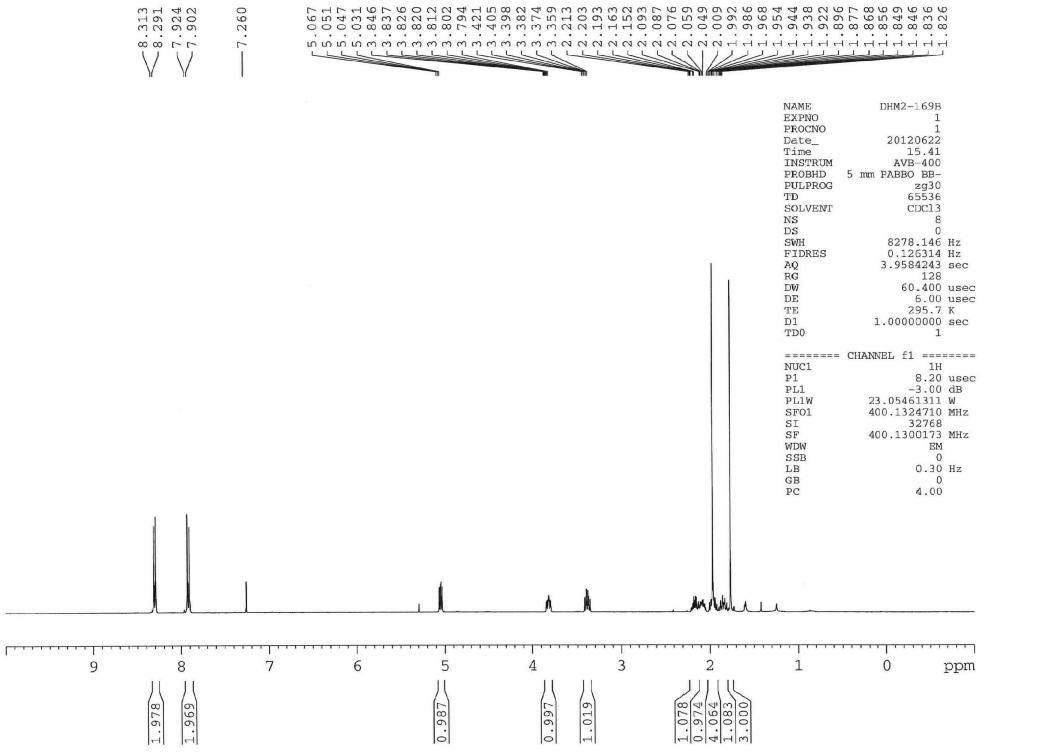

201.05
142.73
129.43
126.52
106.36
87.96
77.32
62.73
36.64
34.05
32.15
25.75
NAME DHM3-ArCyhept5OH
EXPNO 2
PROCNO 1
Date_ 20121218
Time 18.03
INSTRUM av-300
PROBHD 5 mm Dual 13C/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 48
DS 0
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 32768
DW 27.800 usec
DE 6.00 usec
TE 293.8 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 0.00 dB
PL1W 32.65452194 W
SFO1 75.4760505 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 120.00 usec
PL2 -3.00 dB
PL12 17.76 dB
PL13 23.00 dB
PL2W 25.05936241 W
PL12W 0.21036330 W
PL13W 0.06294627 W
SFO2 300.1300000 MHz
SI 32768
SF 75.4677293 MHz
WDW EM
SSB 0
LB 1.50 Hz
GB 0
PC 1.40
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

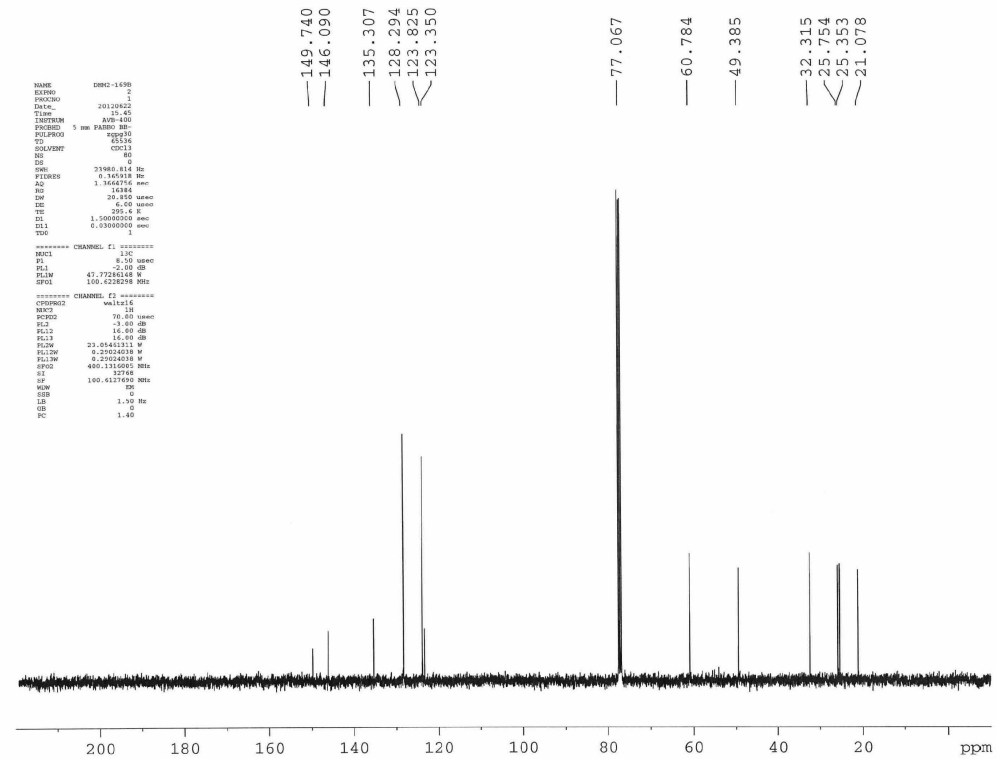

3a
Br
Ns
N

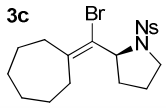

8.312
8.290
7.958
7.936
7.260
5.163
5.146
5.127
3.857
3.849
3.832
3.825
3.814
3.806
3.416
3.401
3.392
3.377
3.369
3.353
2.536
2.526
2.516
2.502
2.491
2.482
2.398
2.388
2.377
2.367
2.276
2.258
2.164
2.144
2.134
2.100
2.082
2.072
2.012
1.994
1.964
1.867
1.848
1.838
1.826
1.819
1.729
1.719
1.589
NAME DHM2-169A
EXPNO 1
PROCNO 1
Date_ 20120621
Time 15.23
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 114
DW 60.400 usec
DE 6.00 usec
TE 296.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.20 usec
PL1 -3.00 dB
PL1W 23.05461311 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300173 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
9
8
7
6
5
4
3
2
1
0
ppm
1.943
1.953
1.000
1.001
1.004
1.019
1.171
5.196
1.026
1.067
5.345

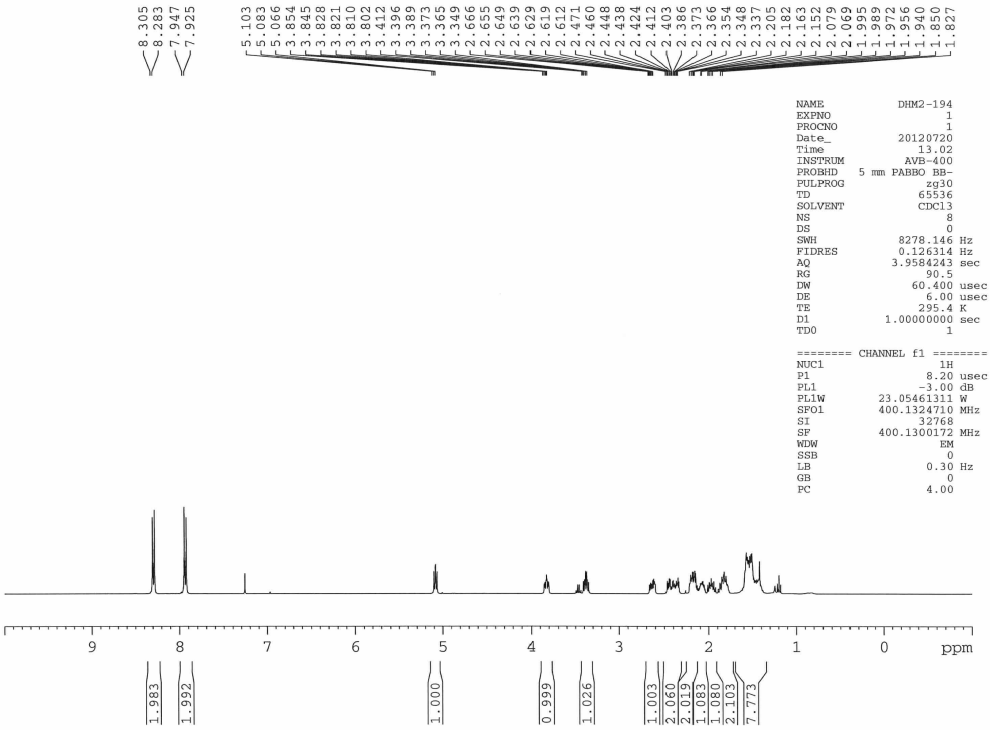

NAME DHM2-169A
EXPNO 2
PROCNO 1
Date_ 20120621
Time 15.27
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 51
DS 0
SWH 23986.614 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 16384
DW 20.850 usec
DE 6.00 usec
TE 296.6 K
D1 1.50000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 47.77286148 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -3.00 dB
PL12 16.00 dB
PL13 16.00 dB
PL2W 23.05461311 W
PL12W 0.29024038 W
PL13W 0.29024038 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.50 Hz
GB 0
PC 1.40
149.735
146.172
142.442
128.322
123.860
121.099
77.052
60.154
49.466
35.727
32.635
32.131
27.720
27.089
26.313
25.492
200
180
160
140
120
100
80
60
40
20
ppm

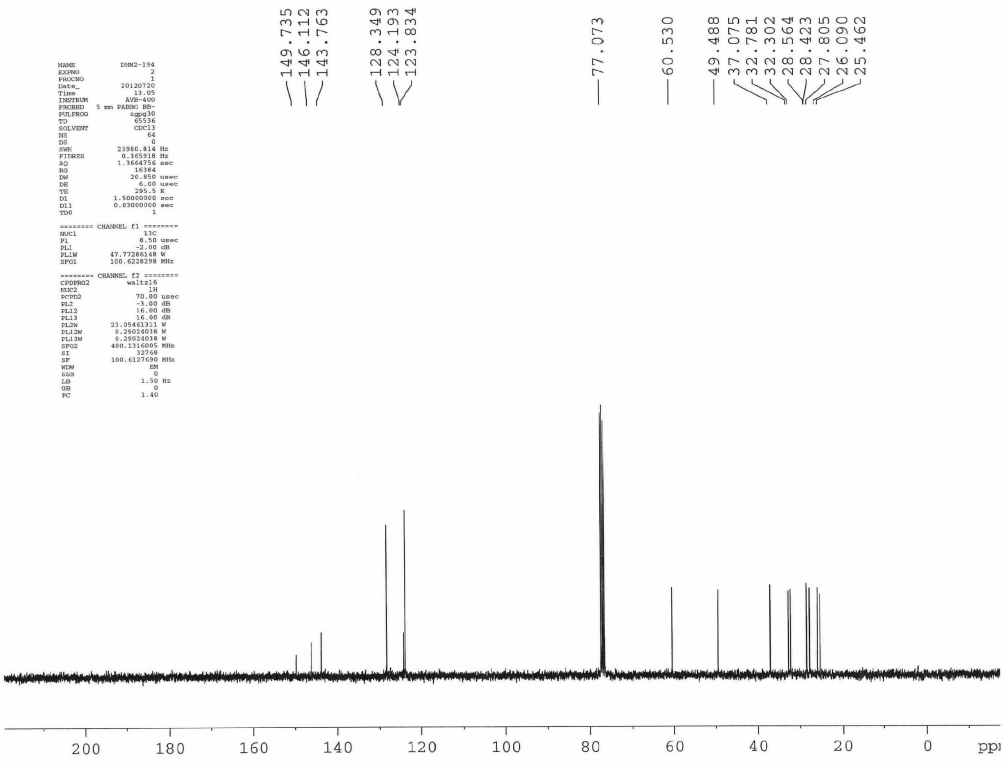




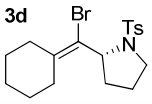



3c
Br
Ns
N

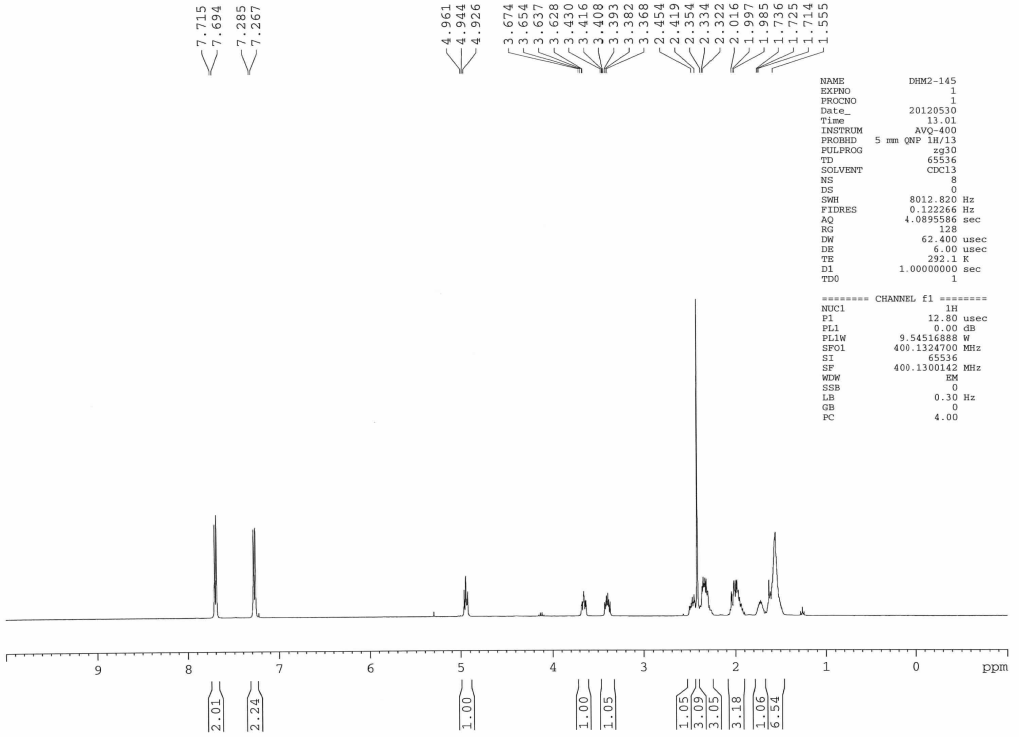

8.305
8.283
7.947
7.925
5.103
5.083
5.066
3.854
3.845
3.828
3.821
3.810
3.802
3.412
3.396
3.389
3.373
3.365
3.349
2.666
2.655
2.649
2.639
2.629
2.619
2.612
2.471
2.460
2.448
2.438
2.424
2.412
2.403
2.386
2.373
2.366
2.354
2.348
2.337
2.205
2.182
2.163
2.152
2.079
2.069
1.995
1.989
1.972
1.956
1.940
1.850
1.827
NAME DHM2-194
EXPNO 1
PROCNO 1
Date_ 20120720
Time 13.02
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 90.5
DW 60.400 usec
DE 6.00 usec
TE 295.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.20 usec
PL1 -3.00 dB
PL1W 23.05461311 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300172 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
9
8
7
6
5
4
3
2
1
0
ppm
1.983
1.992
1.000
0.999
1.026
1.003
2.060
2.019
1.083
1.080
2.103
7.773


149.735
146.112
143.763
128.349
124.193
123.834
77.073
60.530
49.488
37.075
32.781
32.302
28.564
28.423
27.005
26.090
25.462
NAME DHM2-194
EXPNO 2
PROCNO 1
Date_ 20120720
Time 13.05
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 0
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 16384
DW 20.850 usec
DE 6.00 usec
TE 295.5 K
D1 1.50000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 47.77226168 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -3.00 dB
PL12 16.00 dB
PL13 16.00 dB
PL2W 23.05461311 W
PL12W 0.29026038 W
PL13W 0.29026038 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.50 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

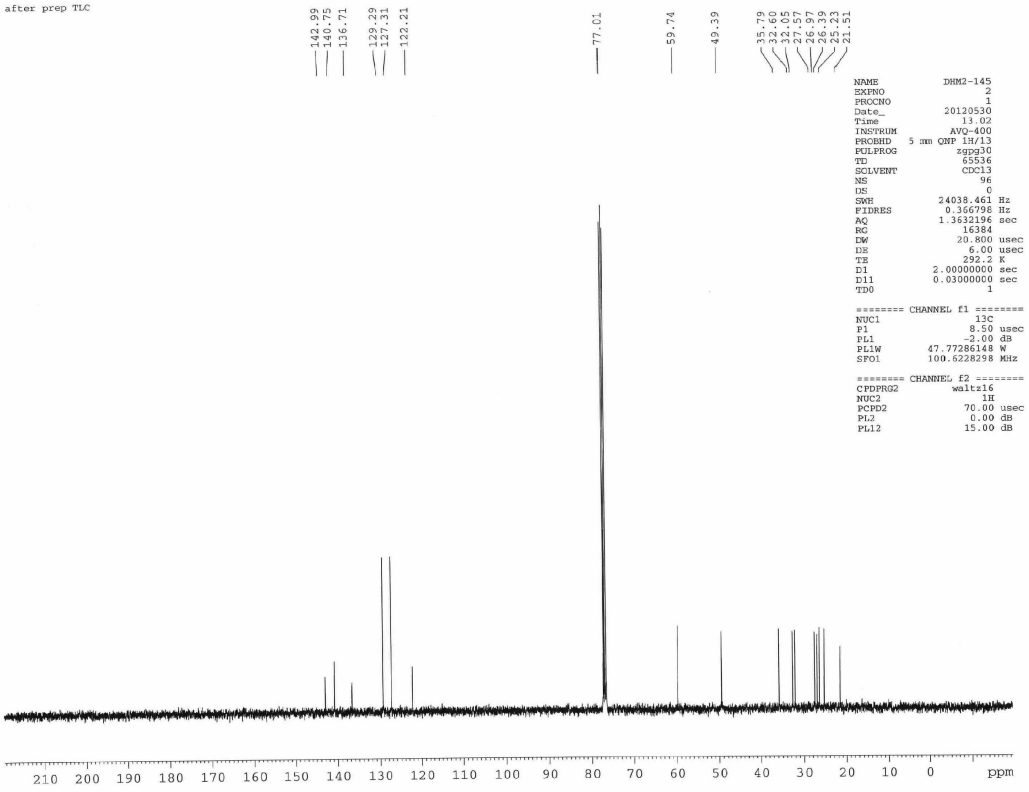
3d

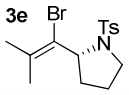


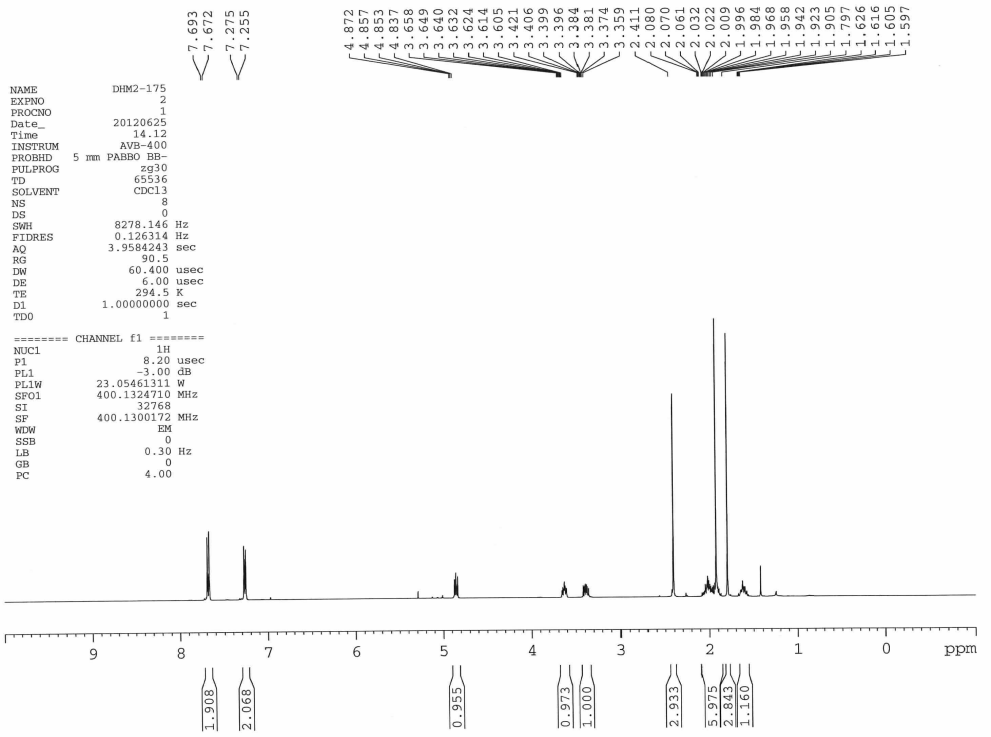
after prep TLC

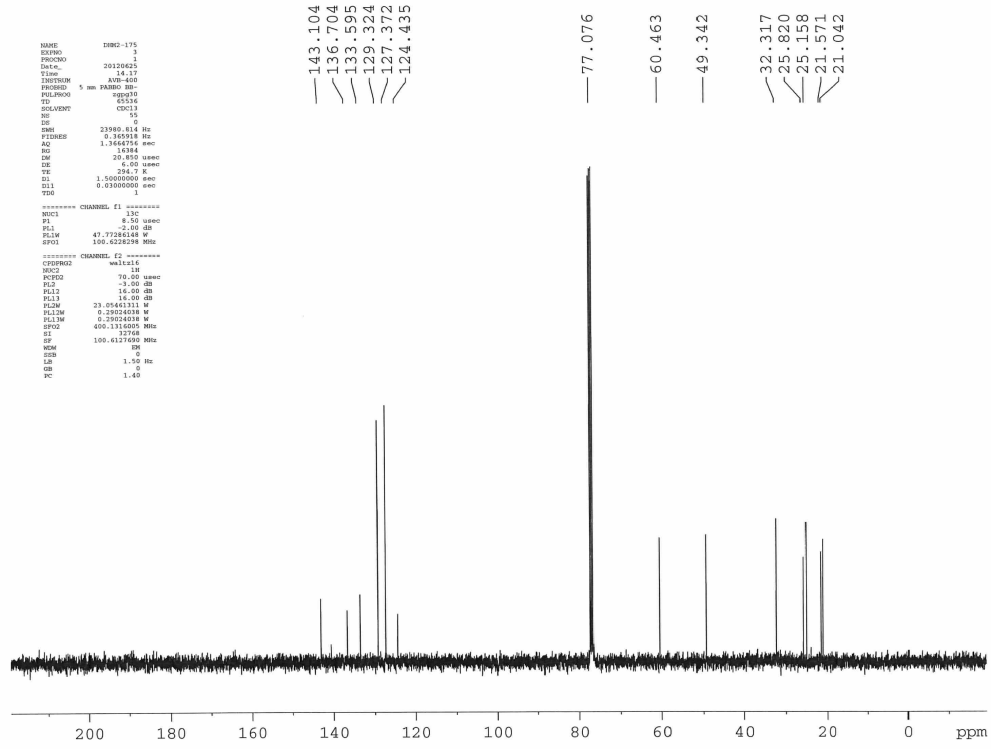


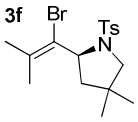
after prep TLC

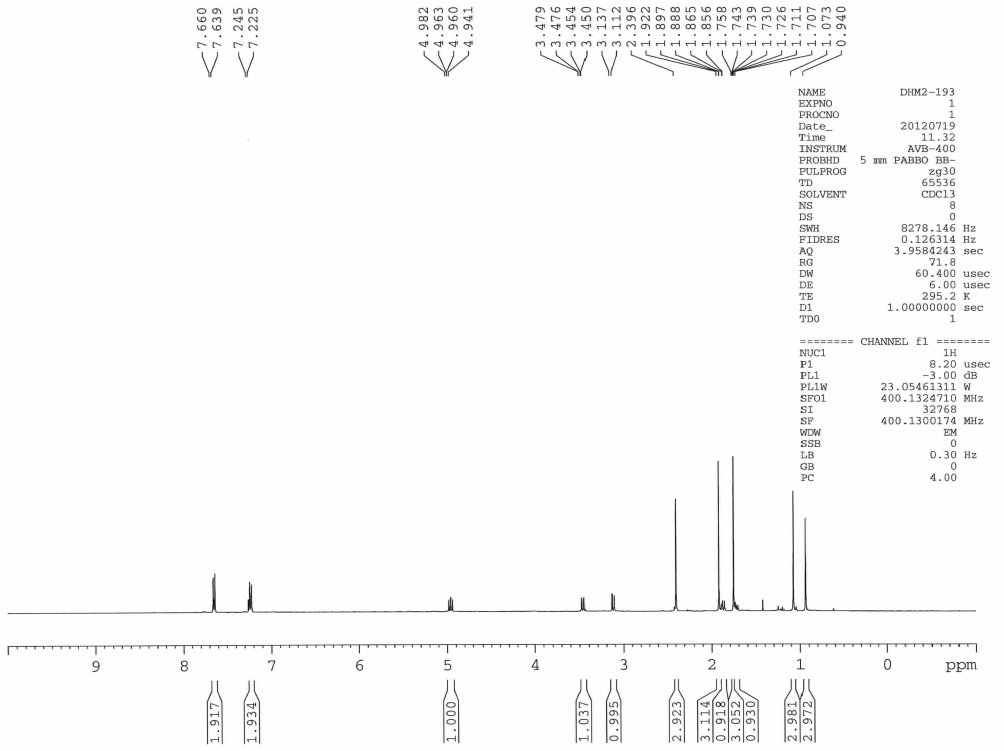


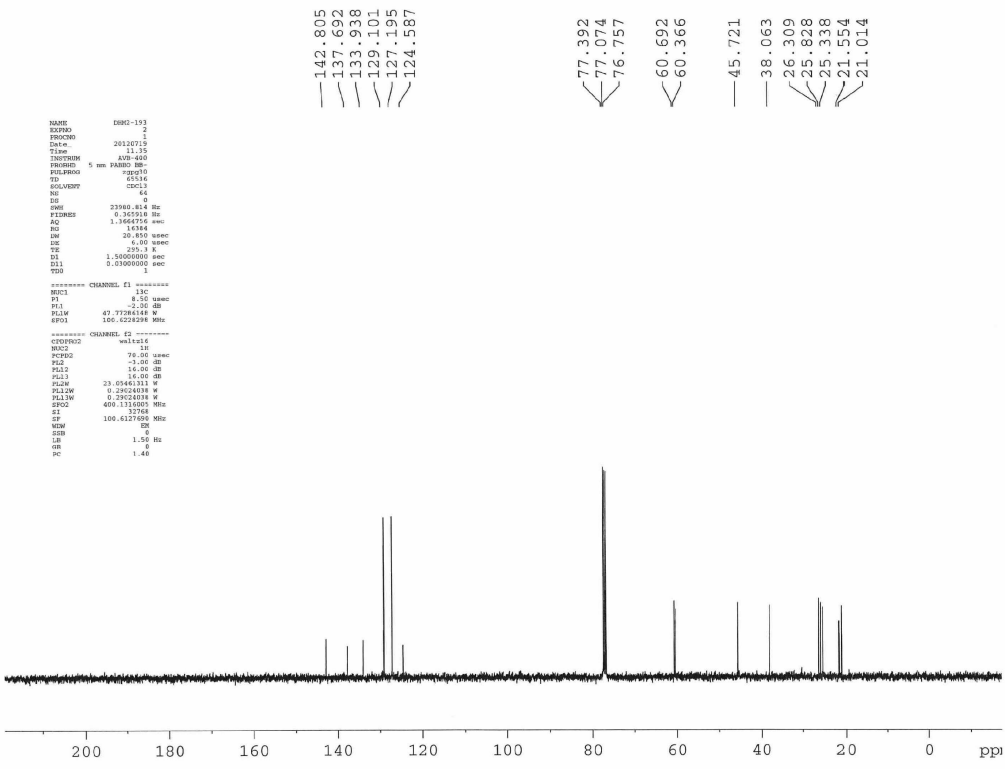

3e
Br
Ts
N

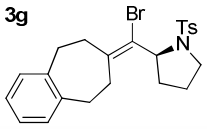

7.693
7.672
7.275
7.255
4.872
4.857
4.853
4.837
3.658
3.649
3.640
3.632
3.624
3.614
3.605
3.421
3.406
3.399
3.396
3.384
3.381
3.374
3.359
2.411
2.080
2.070
2.061
2.032
2.022
2.009
1.996
1.984
1.968
1.958
1.942
1.923
1.905
1.797
1.626
1.616
1.605
1.597
NAME DHM2-175
EXPNO 2
PROCNO 1
Date_ 20120625
Time 14.12
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 90.5
DW 60.400 usec
DE 6.00 usec
TE 294.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.20 usec
PL1 -3.00 dB
PL1W 23.05461311 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300172 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
9
8
7
6
5
4
3
2
1
0
ppm
1.908
2.068
0.955
0.973
1.000
2.933
5.975
2.843
1.160

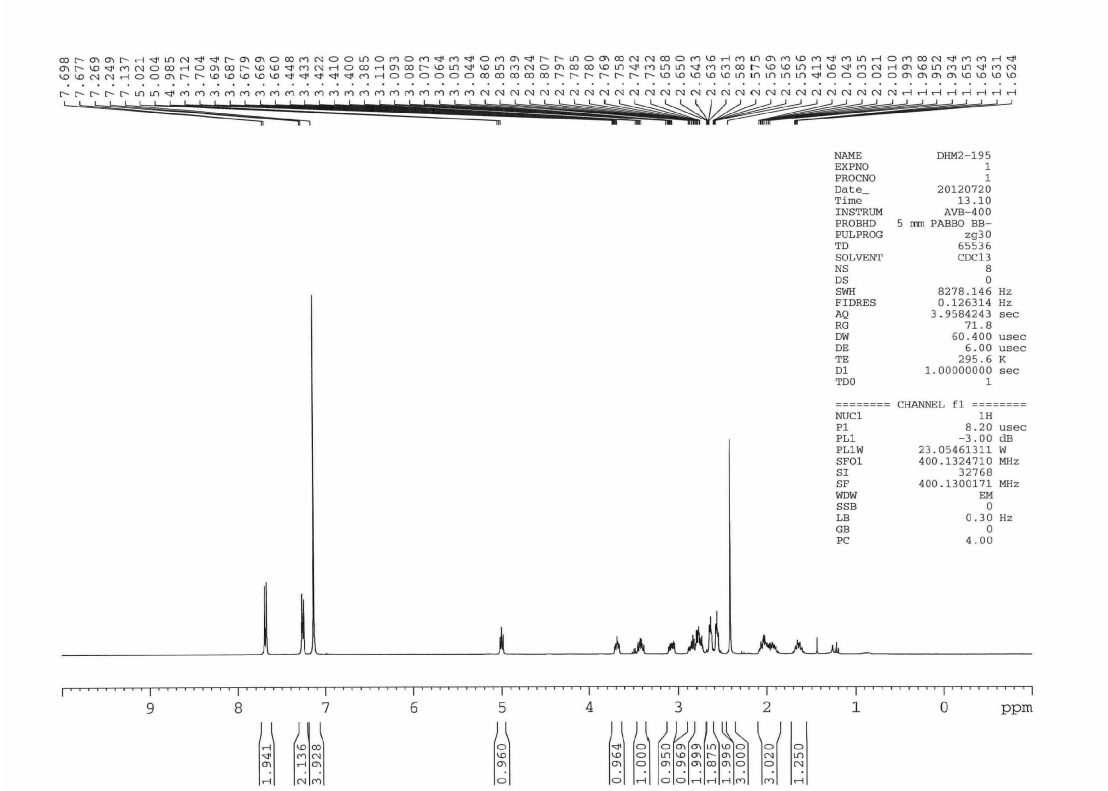

143.104
136.704
133.595
129.324
127.372
124.435
77.076
60.463
49.342
32.317
25.820
25.158
21.571
21.042
NAME DHM2-175
EXPNO 3
PROCNO 1
Date_ 20120625
Time 14.17
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 55
DS 0
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 16384
DW 20.850 usec
DE 6.00 usec
TE 294.7 K
D1 1.50000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 2.00 dB
PL1W 47.77286148 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -3.00 dB
PL12 16.00 dB
PL13 16.00 dB
PL2W 23.05461311 W
PL12W 0.29024038 W
PL13W 0.29024038 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.50 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

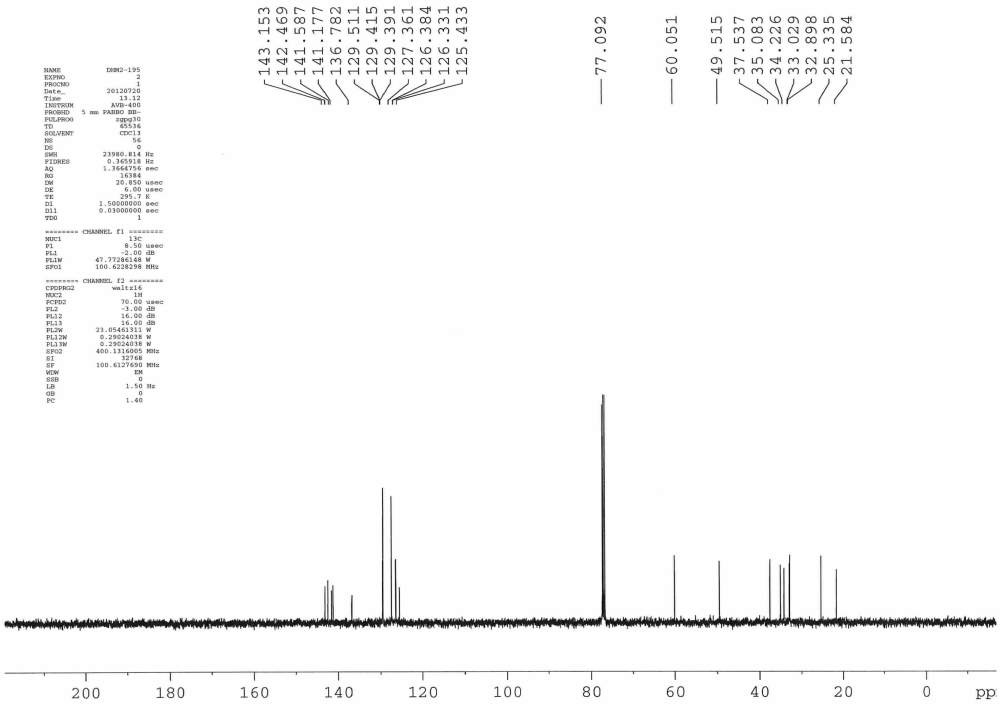

3f
Br
Ts
N

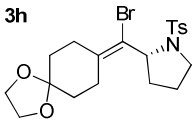

7.660
7.639
7.245
7.225
4.982
4.963
4.960
4.941
3.479
3.476
3.454
3.450
3.137
3.112
2.396
1.922
1.897
1.888
1.865
1.856
1.758
1.743
1.739
1.730
1.726
1.711
1.707
1.073
0.940
NAME DHM2-193
EXPNO 1
PROCNO 1
Date_ 20120719
Time 11.32
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 71.8
DW 60.400 usec
DE 6.00 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.20 usec
PL1 -3.00 dB
PL1W 23.05461311 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300174 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
9
8
7
6
5
4
3
2
1
0
ppm
1.917
1.934
1.000
1.037
0.995
2.923
3.114
0.918
3.052
0.930
2.981
2.972

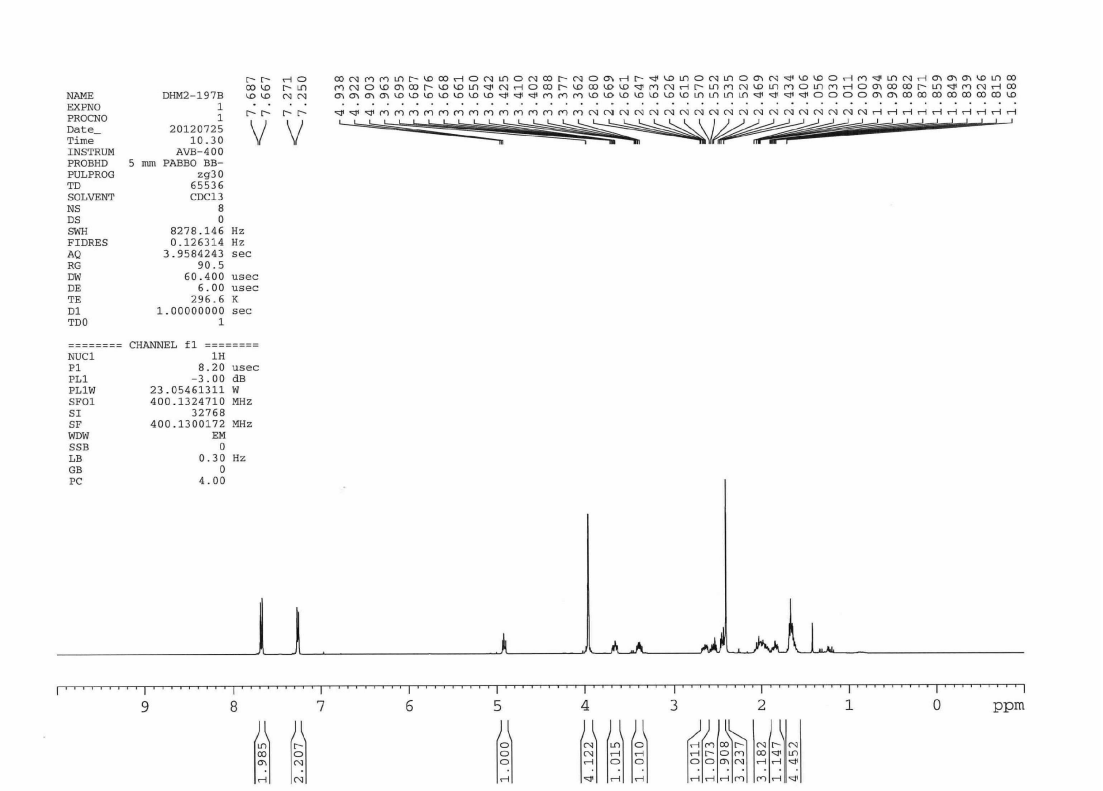

142.805
137.692
133.938
129.101
127.195
124.587
77.392
77.074
76.757
60.692
60.366
45.721
38.063
26.309
25.828
25.338
21.554
21.014
NAME DHM2-193
EXPNO 2
PROCNO 1
Date_ 20120719
Time 11.35
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 0
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 14384
DW 20.850 usec
DE 6.00 usec
TE 295.3 K
D1 1.50000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 47.77286148 W
SFO1 100.6229398 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -3.00 dB
PL12 16.00 dB
PL13 16.00 dB
PL2W 23.05461311 W
PL12W 0.29024038 W
PL13W 0.29024038 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127405 MHz
WDW EM
SSB 0
LB 1.50 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

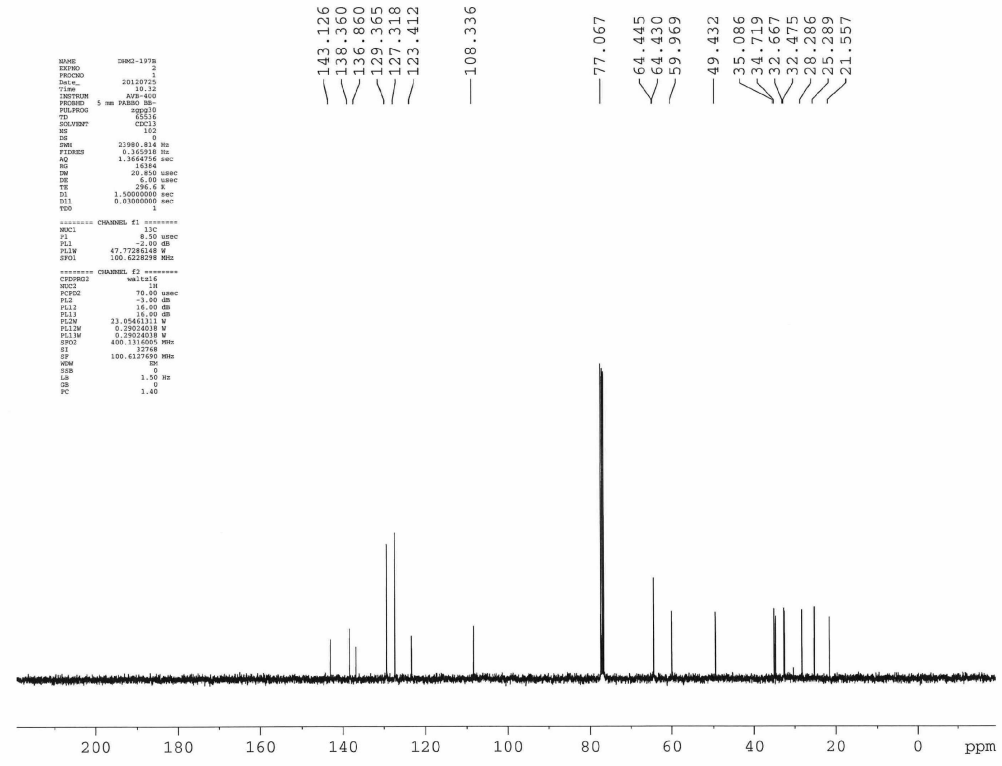

3g
Br
Ts
N

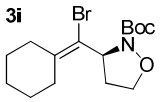

7.698 7.677 7.269 7.249 7.137 5.021 5.004 4.985 3.712 3.704 3.694 3.687 3.679 3.669 3.660 3.448 3.433 3.422 3.410 3.400 3.385 3.110 3.093 3.080 3.073 3.064 3.053 3.044 2.860 2.853 2.839 2.824 2.807 2.797 2.785 2.780 2.769 2.758 2.742 2.732 2.658 2.650 2.643 2.636 2.631 2.583 2.575 2.569 2.563 2.556 2.413 2.064 2.043 2.035 2.021 2.010 1.993 1.968 1.952 1.934 1.653 1.643 1.631 1.624
NAME DHM2-195
EXPNO 1
PROCNO 1
Date_ 20120720
Time 13.10
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 71.8
DW 60.400 usec
DE 6.00 usec
TE 295.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.20 usec
PL1 -3.00 dB
PL1W 23.05461311 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300171 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
9 8 7 6 5 4 3 2 1 0 ppm
1.941 2.136 3.928 0.960 0.964 1.000 0.950 0.969 1.999 1.875 1.996 3.000 3.020 1.250

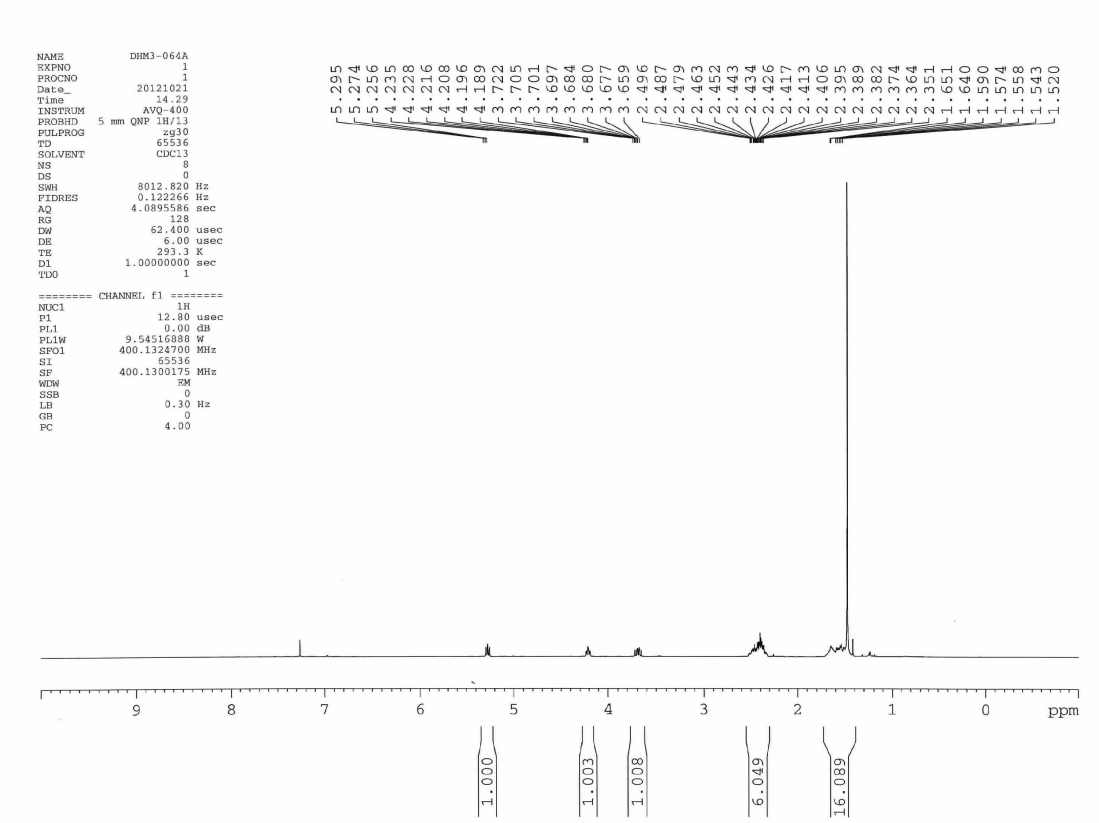

143.153 142.469 141.587 141.177 136.782 129.511 129.415 129.391 127.361 126.384 126.331 125.433
77.092
60.051
49.515
37.537 35.083 34.226 33.029 32.898 25.335 21.584
NAME DHM2-195
EXPNO 2
PROCNO 1
Date_ 20120720
Time 13.12
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 56
DS 0
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 16384
DW 20.850 usec
DE 6.00 usec
TE 295.7 K
D1 1.50000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 47.77286148 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -3.00 dB
PL12 16.00 dB
PL13 16.00 dB
PL2W 23.05461311 W
PL12W 0.29024038 W
PL13W 0.29024038 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.50 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm

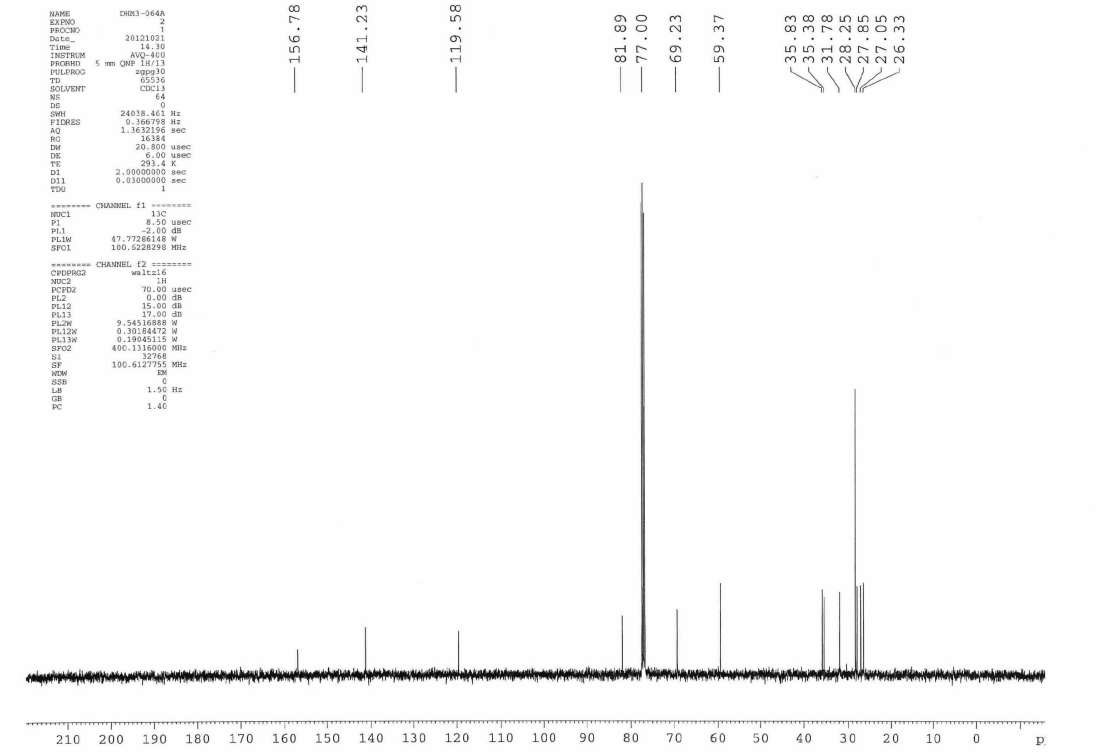

3h
Br
Ts
N
O
O

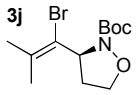

7.687
7.667
7.271
7.250
4.938
4.922
4.903
3.963
3.695
3.687
3.676
3.668
3.661
3.650
3.642
3.425
3.410
3.402
3.388
3.377
3.362
2.680
2.669
2.661
2.647
2.634
2.626
2.615
2.570
2.552
2.535
2.520
2.469
2.452
2.434
2.406
2.056
2.030
2.011
2.003
1.994
1.985
1.882
1.871
1.859
1.849
1.839
1.826
1.815
1.688
NAME DHM2-197B
EXPNO 1
PROCNO 1
Date_ 20120725
Time 10.30
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 90.5
DW 60.400 usec
DE 6.00 usec
TE 296.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.20 usec
PL1 -3.00 dB
PL1W 23.05461311 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300172 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
9
8
7
6
5
4
3
2
1
0
ppm
1.985
2.207
1.000
4.122
1.015
1.010
1.011
1.073
1.908
3.237
3.182
1.147
4.452

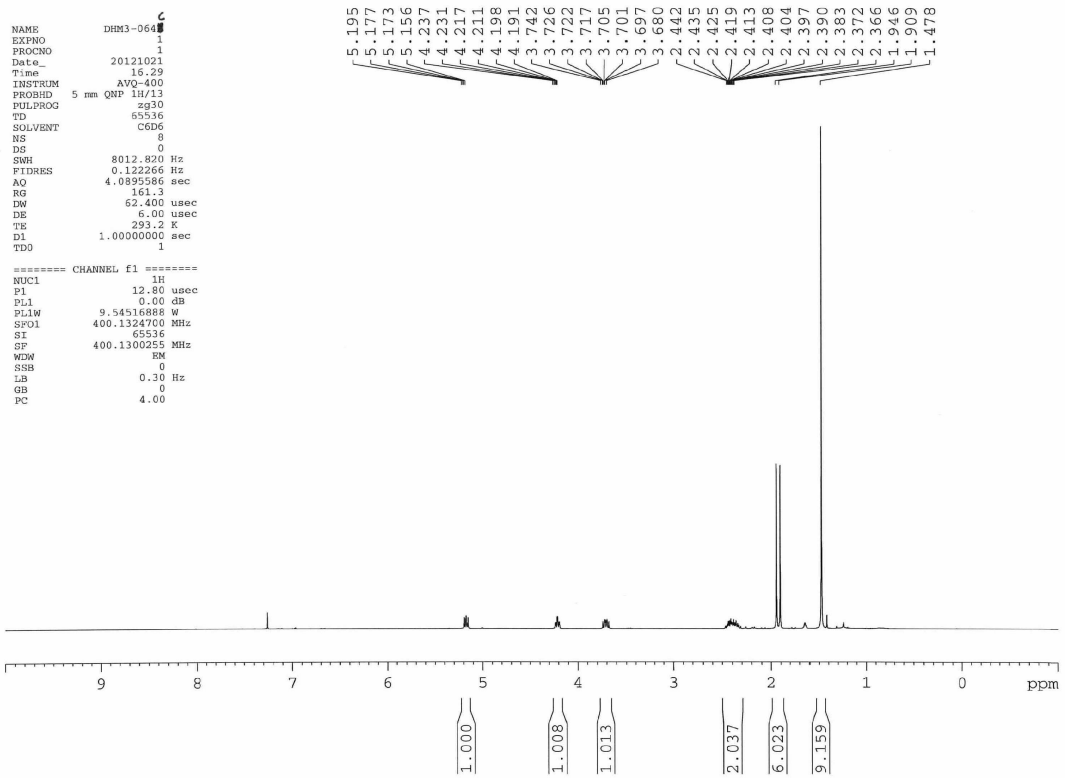

143.126
138.360
136.860
129.365
127.318
123.412
108.336
77.067
64.445
64.430
59.969
49.432
35.086
34.719
32.667
32.475
28.286
25.289
21.557
NAME DHM2-197B
EXPNO 2
PROCNO 1
Date_ 20120725
Time 10.32
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 162
DS 0
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 16384
DW 20.850 usec
DE 6.00 usec
TE 296.6 K
D1 1.50000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 47.77286148 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -3.00 dB
PL12 16.00 dB
PL13 16.00 dB
PL2W 23.05461311 W
PL12W 0.29024038 W
PL13W 0.29024038 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.50 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

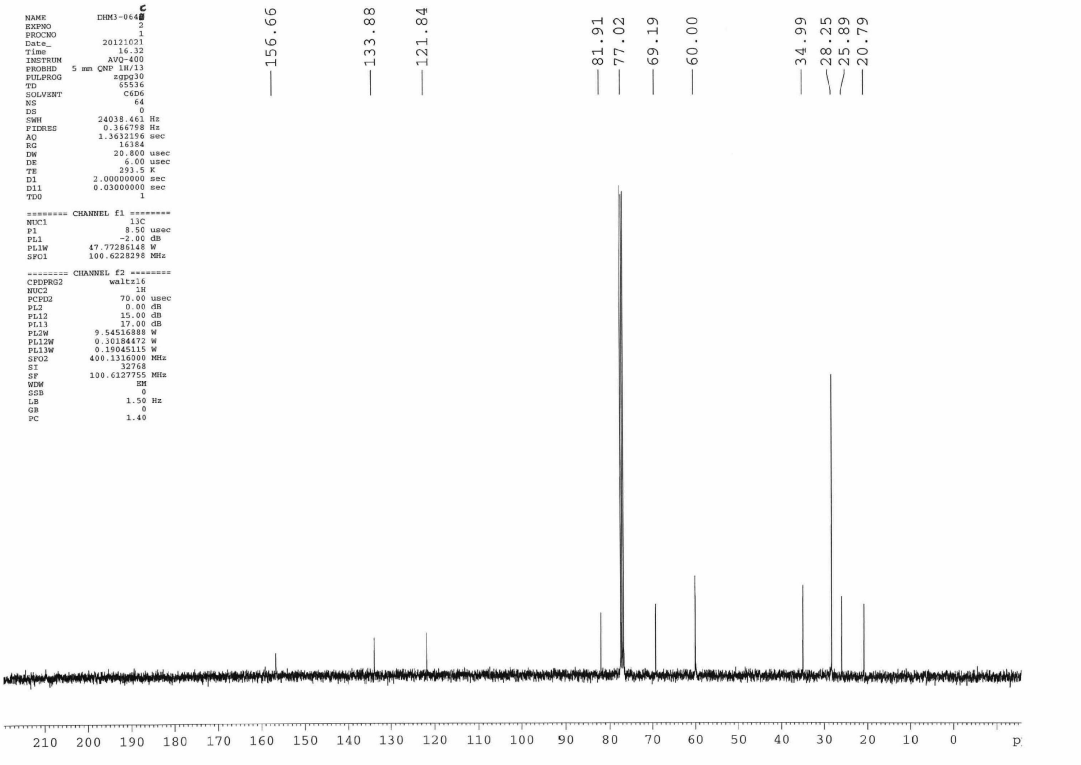

3i
Br
Boc
N
O

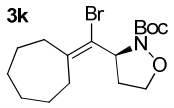

NAME DHM3-064A
EXPNO 1
PROCNO 1
Date_ 20121021
Time 14.29
INSTRUM AVQ-400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0895586 sec
RG 128
DW 62.400 usec
DE 6.00 usec
TE 293.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.80 usec
PL1 0.00 dB
PL1W 9.54516888 W
SFO1 400.1324700 MHz
SI 65536
SF 400.1300175 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
5.295
5.274
5.256
4.235
4.228
4.216
4.208
4.196
4.189
3.722
3.705
3.701
3.697
3.684
3.680
3.677
3.659
2.496
2.487
2.479
2.463
2.452
2.443
2.434
2.426
2.417
2.413
2.406
2.395
2.389
2.382
2.374
2.364
2.351
1.651
1.640
1.590
1.574
1.558
1.543
1.520
9
8
7
6
5
4
3
2
1
0
ppm
1.000
1.003
1.008
6.049
16.089

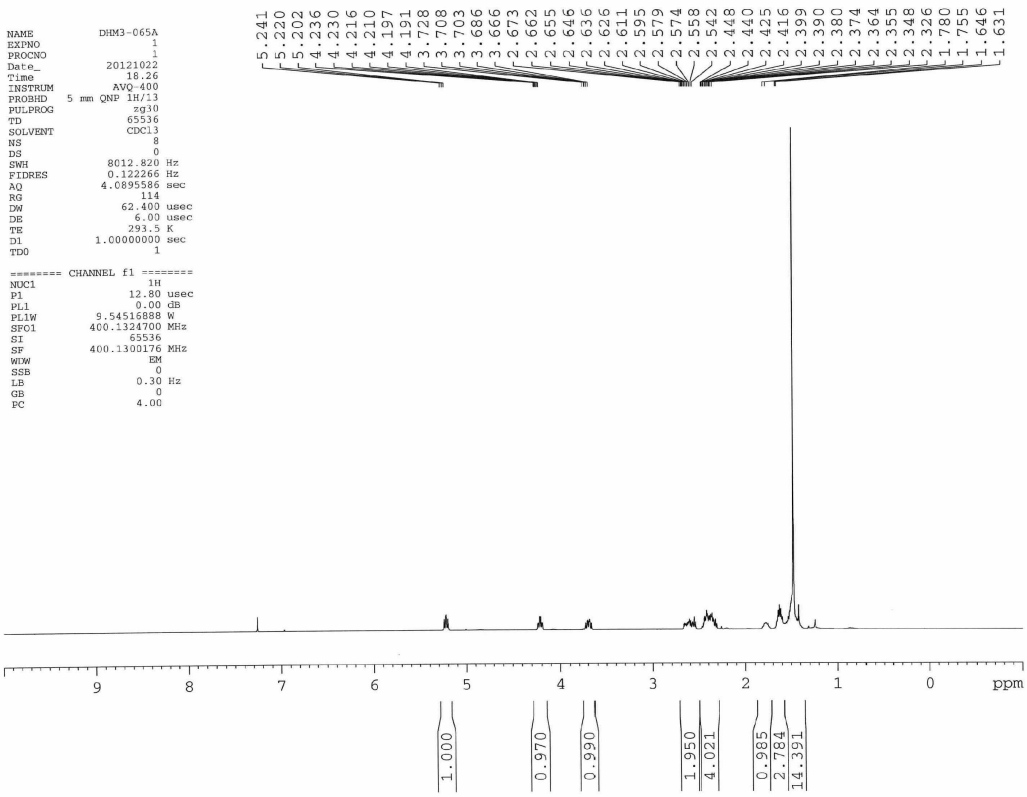

NAME DHM3-064A
EXPNO 2
PROCNO 1
Date_ 20121021
Time 14.30
INSTRUM AVQ-400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3632196 sec
RG 16384
DW 20.800 usec
DE 6.00 usec
TE 293.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 47.77286148 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 0.00 dB
PL12 15.00 dB
PL13 17.00 dB
PL2W 9.54516888 W
PL12W 0.30184472 W
PL13W 0.19045115 W
SFO2 400.1316000 MHz
SI 32768
SF 100.6127755 MHz
WDW EM
SSB 0
LB 1.50 Hz
GB 0
PC 1.40
156.78
141.23
119.58
81.89
77.00
69.23
59.37
35.83
35.38
31.78
28.25
27.85
27.05
26.33
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

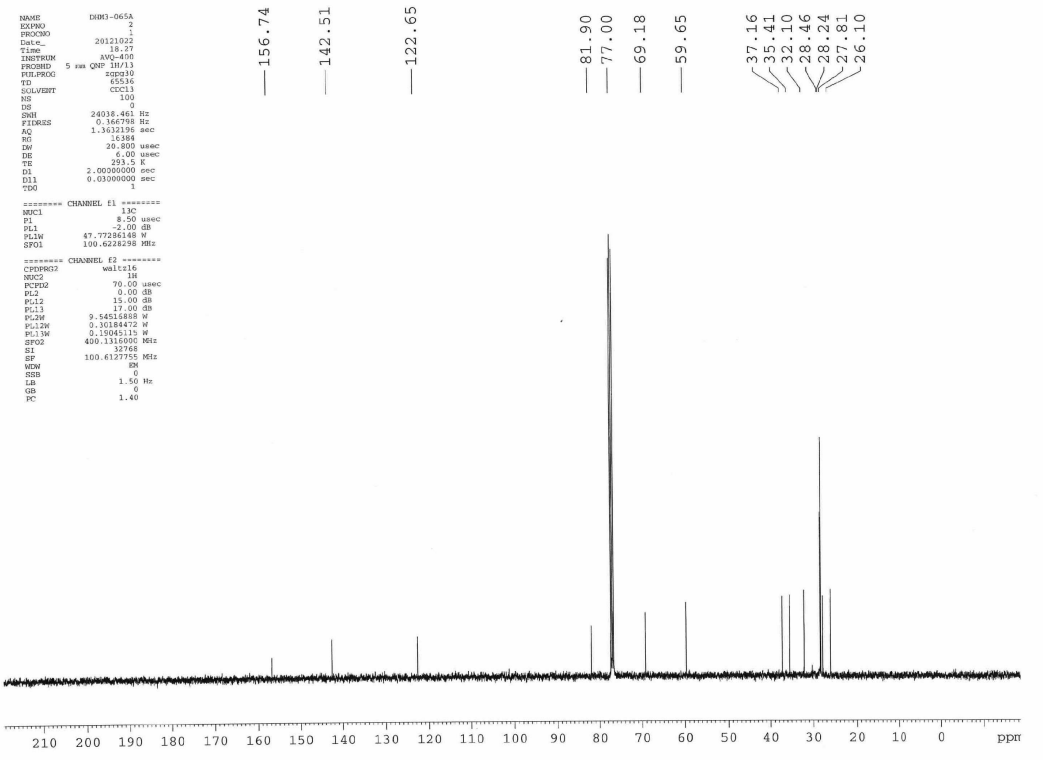

3j
Br
Boc
N
O

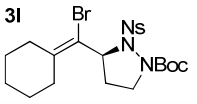

5.195
5.177
5.173
5.156
4.237
4.231
4.217
4.211
4.198
4.191
3.742
3.726
3.722
3.717
3.705
3.701
3.697
3.680
2.442
2.435
2.425
2.419
2.413
2.408
2.404
2.397
2.390
2.383
2.372
2.366
1.946
1.909
1.478
NAME DHM3-064
EXPNO 1
PROCNO 1
Date_ 20121021
Time 16.29
INSTRUM AVQ-400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0895586 sec
RG 161.3
DW 62.400 usec
DE 6.00 usec
TE 293.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.80 usec
PL1 0.00 dB
PL1W 9.54516888 W
SFO1 400.1324700 MHz
SI 65536
SF 400.1300255 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
9
8
7
6
5
4
3
2
1
0
ppm
1.000
1.008
1.013
2.037
6.023
9.159

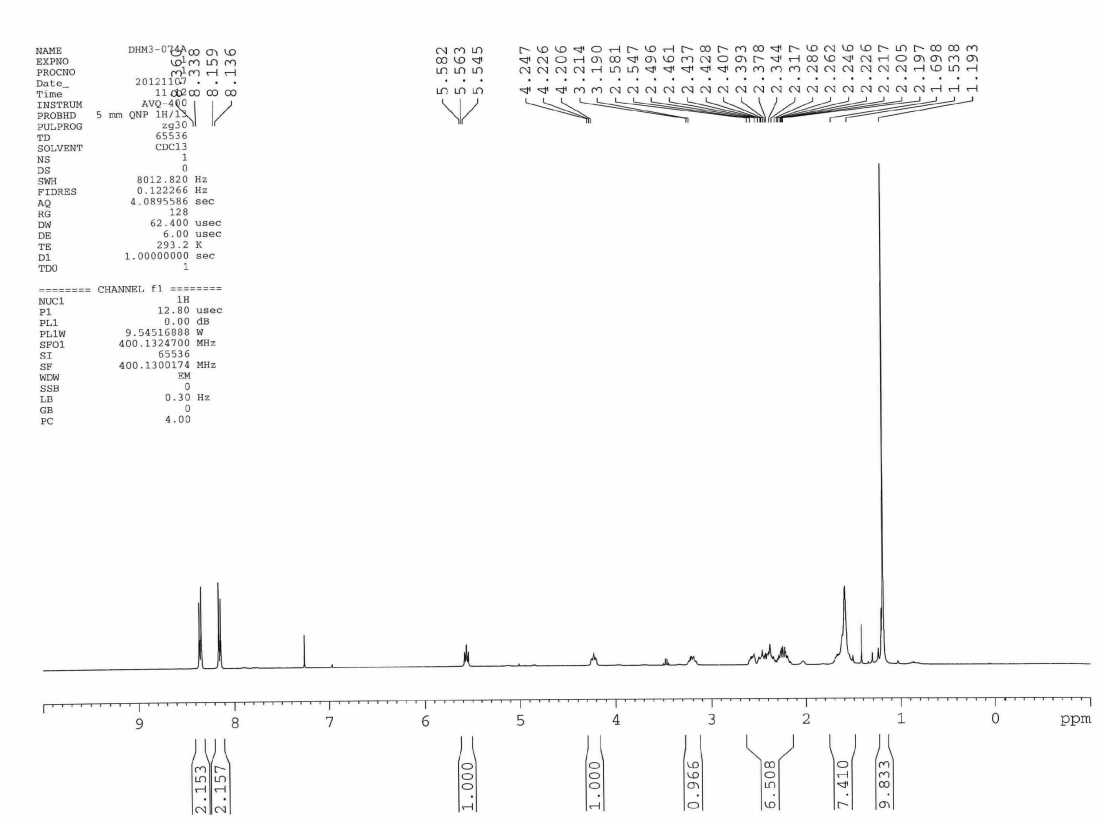

156.66
133.88
121.84
81.91
77.02
69.19
60.00
34.99
28.25
25.89
20.79
NAME DHM3-064
EXPNO 2
PROCNO 1
Date_ 20121021
Time 16.32
INSTRUM AVQ-400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT C6D6
NS 64
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3632196 sec
RG 16384
DW 20.800 usec
DE 6.00 usec
TE 293.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 47.77286148 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 0.00 dB
PL12 15.00 dB
PL13 17.00 dB
PL2W 9.54516888 W
PL12W 0.30184472 W
PL13W 0.19045115 W
SFO2 400.1316000 MHz
SI 32768
SF 100.6127755 MHz
WDW EM
SSB 0
LB 1.50 Hz
GB 0
PC 1.40
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

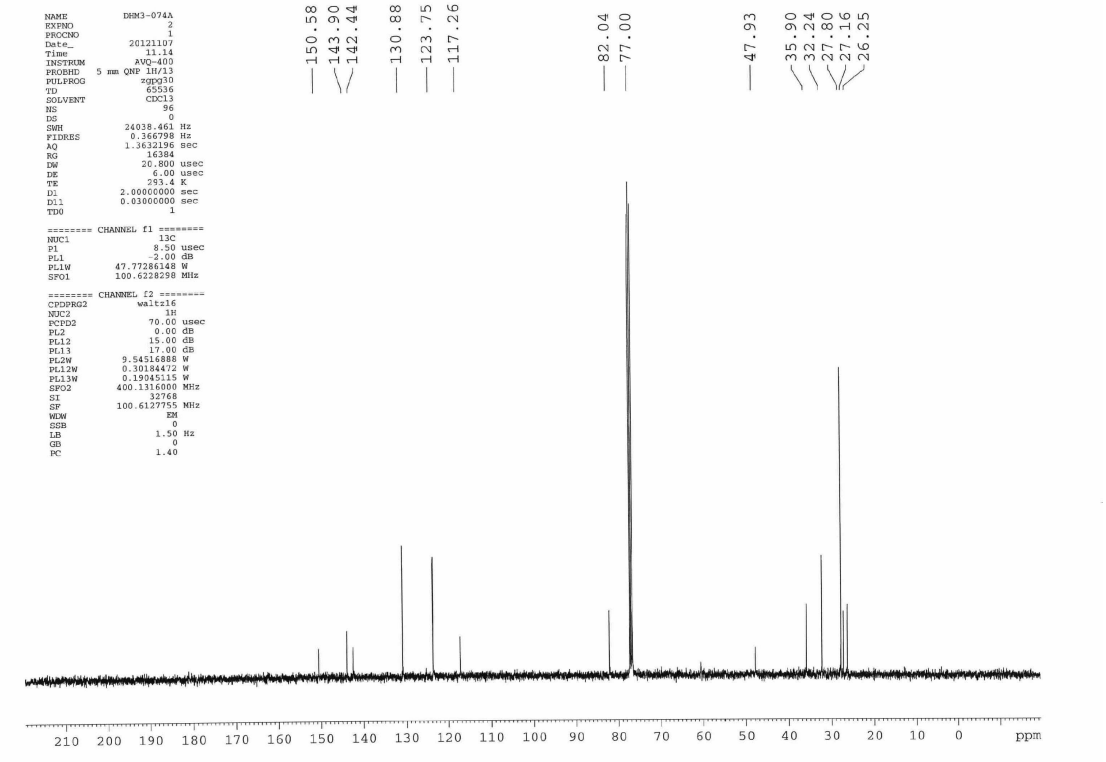

3k
Br
Boc
N
O


NAME DHM3-065A
EXPNO 1
PROCNO 1
Date_ 20121022
Time 18.26
INSTRUM AVQ-400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0895586 sec
RG 114
DW 62.400 usec
DE 6.00 usec
TE 293.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.80 usec
PL1 0.00 dB
PL1W 9.54516888 W
SFO1 400.1324700 MHz
SI 65536
SF 400.1300176 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
5.241 5.220 5.202 4.236 4.230 4.216 4.210 4.197 4.191 3.728 3.708 3.703 3.686 3.666 2.673 2.662 2.655 2.646 2.636 2.626 2.611 2.595 2.579 2.574 2.558 2.542 2.448 2.440 2.425 2.416 2.399 2.390 2.380 2.374 2.364 2.355 2.348 2.326 1.780 1.755 1.646 1.631
9 8 7 6 5 4 3 2 1 0 ppm
1.000 0.970 0.990 1.950 4.021 0.985 2.784 14.391


NAME DHM3-065A
EXPNO 2
PROCNO 1
Date_ 20121022
Time 18.27
INSTRUM AVQ-400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 100
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3632196 sec
RG 16384
DW 20.800 usec
DE 6.00 usec
TE 293.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 47.77286148 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 0.00 dB
PL12 15.00 dB
PL13 17.00 dB
PL2W 9.54516888 W
PL12W 0.36184472 W
PL13W 0.19045115 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127755 MHz
WDW EM
SSB 0
LB 1.50 Hz
GB 0
PC 1.40
156.74 142.51 122.65 81.90 77.00 69.18 59.65 37.16 35.41 32.10 28.46 28.24 27.81 26.10
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


3l
Br
Ns
N
NBoc


NAME DHM3-074A
EXPNO 2
PROCNO 1
Date_ 20121107
Time 11.08
INSTRUM AVQ-400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0895586 sec
RG 128
DW 62.400 usec
DE 6.00 usec
TE 293.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.80 usec
PL1 0.00 dB
PL1W 9.54516888 W
SFO1 400.1324700 MHz
SI 65536
SF 400.1300174 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
8.168 8.159 8.136 8.133
5.582 5.563 5.545
4.247 4.226 4.206 3.214 3.190 2.581 2.547 2.466 2.461 2.437 2.428 2.407 2.393 2.378 2.344 2.317 2.286 2.262 2.246 2.226 2.217 2.205 2.197 1.698 1.538 1.193
9 8 7 6 5 4 3 2 1 0 ppm
2.153 2.157 1.000 1.000 0.966 6.508 7.410 9.833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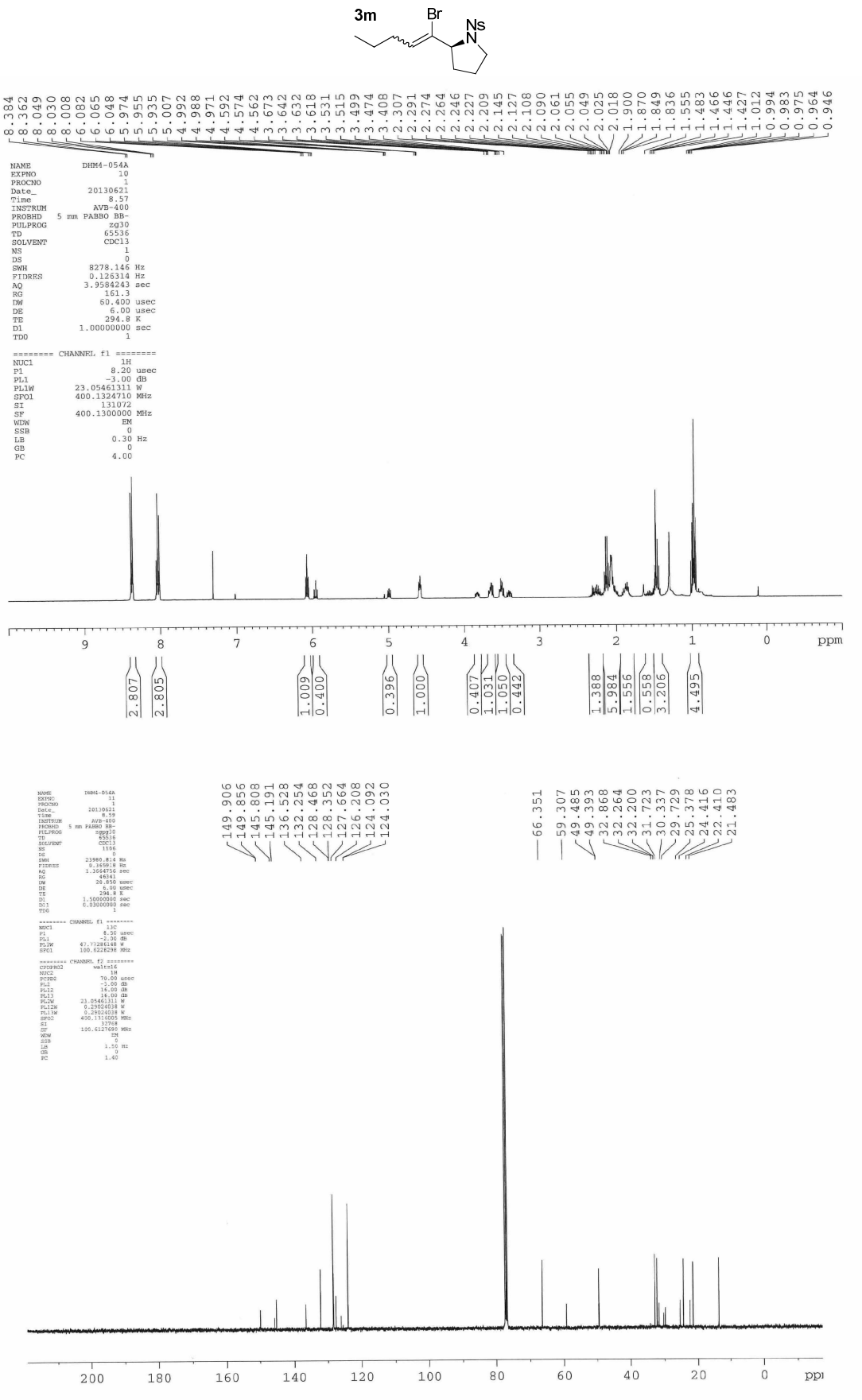
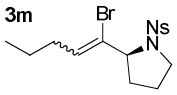
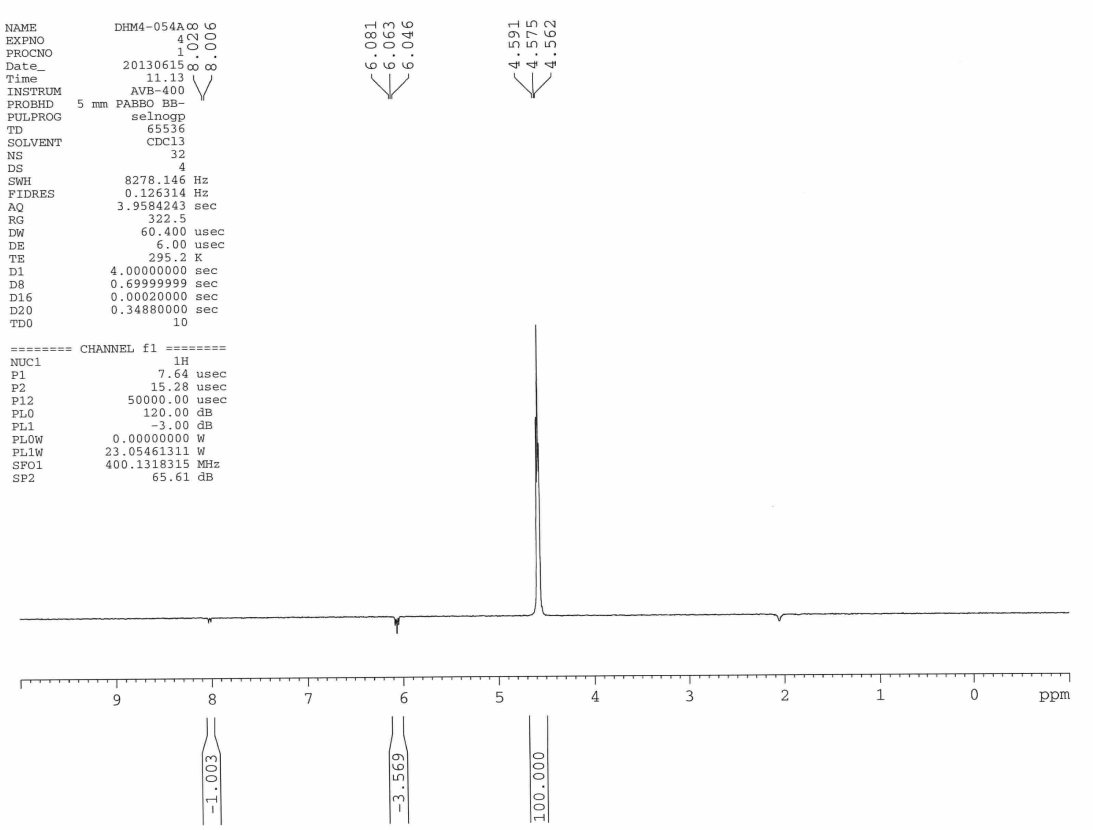
EXPNO 2
PROCNO 1
Date_ 20121107
Time 11.14
INSTRUM AVQ-400
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 96
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3632196 sec
RG 16384
DW 20.800 usec
DE 6.00 usec
TE 293.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 47.77286148 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 0.00 dB
PL12 15.00 dB
PL13 17.00 dB
PL2W 9.54516888 W
PL12W 0.30184472 W
PL13W 0.19045115 W
SFO2 400.1316000 MHz
SI 32768
SF 100.6127755 MHz
WDW EM
SSB 0
LB 1.50 Hz
GB 0
PC 1.40
150.58 143.90 142.44 130.88 123.75 117.26 82.04 77.00 47.93 35.90 32.24 27.80 27.16 26.25
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

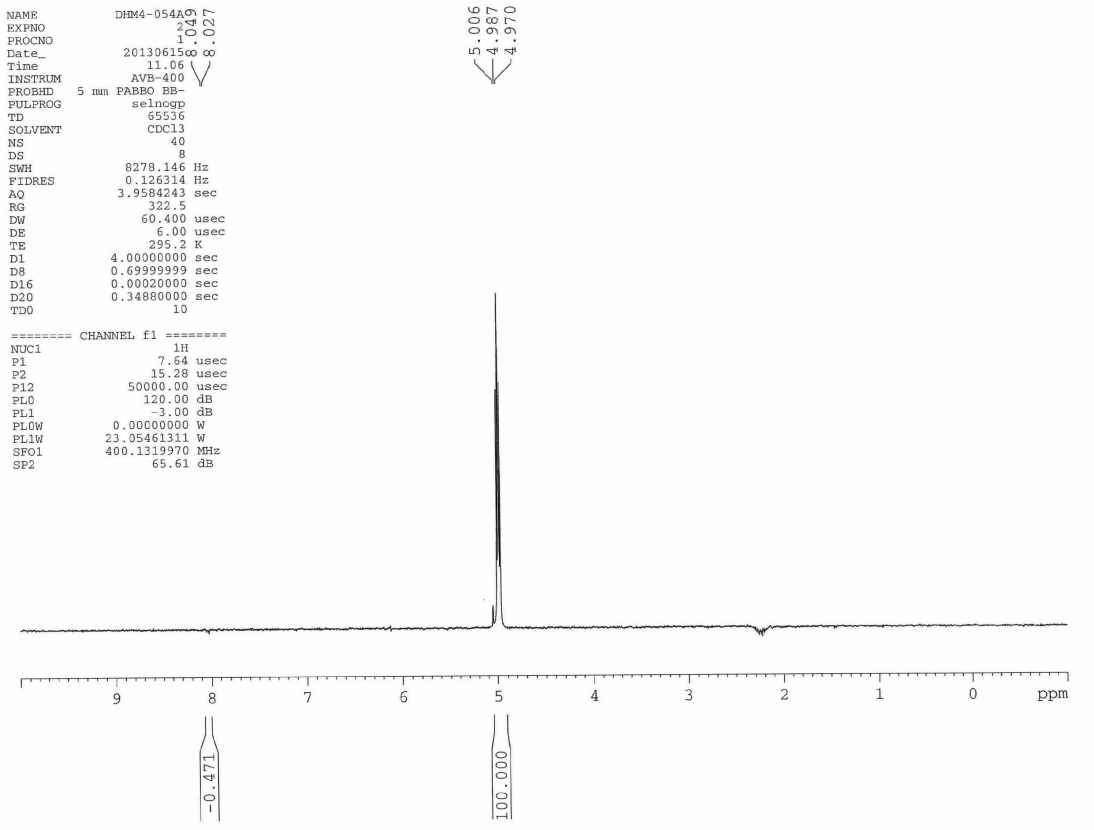

3m
Br
Ns
N
8.384
8.362
8.049
8.030
8.008
8.082
8.065
8.048
6.974
5.955
5.935
5.007
4.992
4.988
4.971
4.592
4.574
4.562
3.673
3.642
3.632
3.618
3.531
3.515
3.499
3.474
3.408
2.307
2.291
2.274
2.264
2.246
2.227
2.209
2.145
2.127
2.108
2.090
2.061
2.055
2.049
2.025
2.018
1.900
1.870
1.849
1.836
1.555
1.483
1.466
1.446
1.427
1.012
0.994
0.983
0.975
0.964
0.946
NAME DHM4-054A
EXPNO 10
PROCNO 1
Date_ 20130621
Time 8.57
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 161.3
DW 60.400 usec
DE 6.00 usec
TE 294.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.20 usec
PL1 -3.00 dB
PL1W 23.05461311 W
SFO1 400.1324710 MHz
SI 131072
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
9
8
7
6
5
4
3
2
1
0
ppm
2.807
2.805
1.009
0.400
0.396
1.000
0.407
1.031
1.050
0.442
1.388
5.984
1.556
0.558
3.206
4.495
149.906
149.856
145.808
145.191
136.528
132.254
128.468
128.352
127.664
126.208
124.092
124.030
66.351
59.307
49.485
49.393
32.868
32.264
32.200
31.723
30.337
29.729
25.378
24.416
22.410
21.483
200
180
160
140
120
100
80
60
40
20
0
ppm

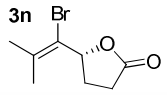
3m

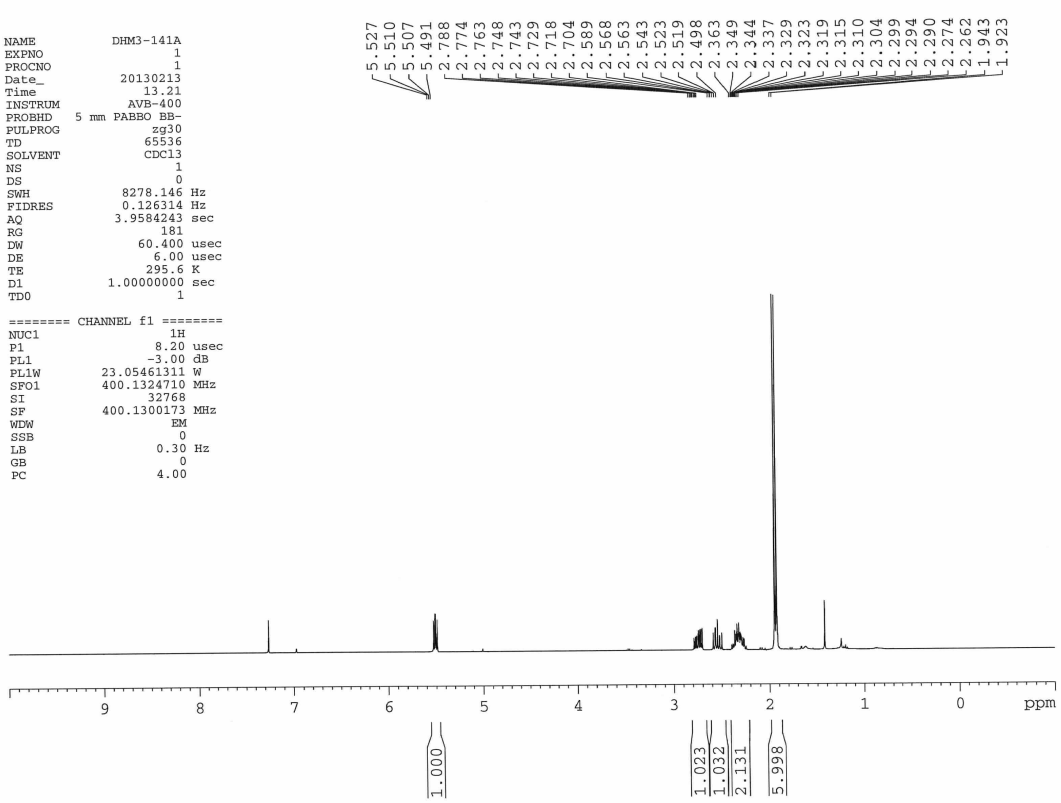


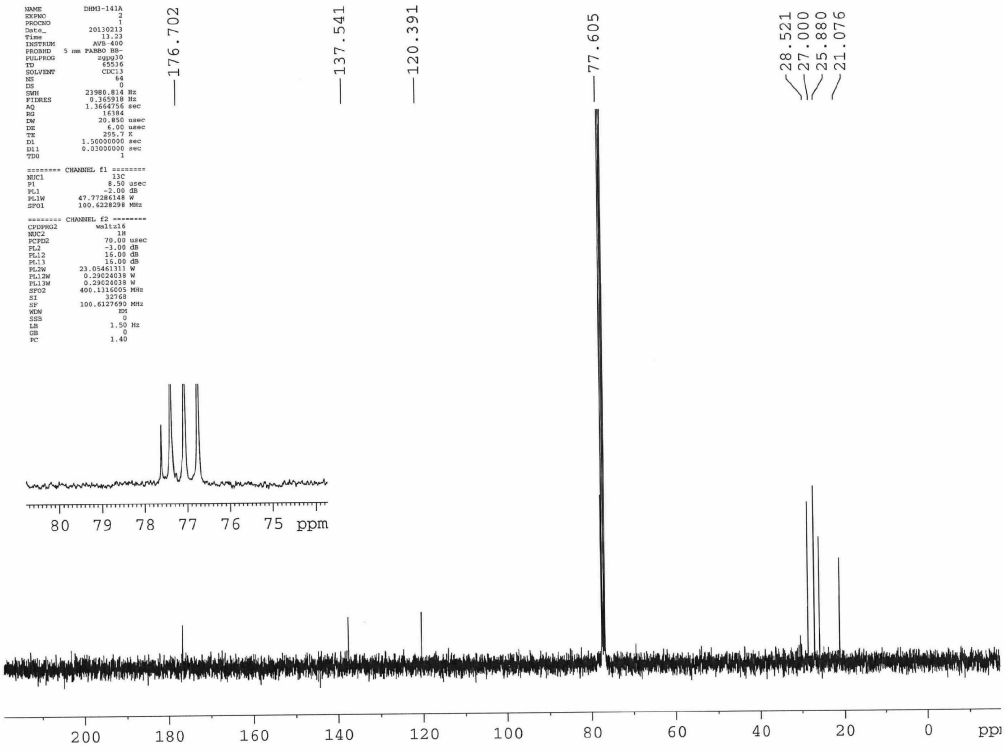
NOE spectra






3n
Br
O
O


NAME DHM3-141A
EXPNO 1
PROCNO 1
Date_ 20130213
Time 13.21
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 181
DW 60.400 usec
DE 6.00 usec
TE 295.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.20 usec
PL1 -3.00 dB
PL1W 23.05461311 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300173 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
5.527
5.510
5.507
5.491
2.788
2.774
2.763
2.748
2.743
2.729
2.718
2.704
2.589
2.568
2.563
2.543
2.523
2.519
2.498
2.363
2.349
2.344
2.337
2.329
2.323
2.319
2.315
2.310
2.304
2.299
2.294
2.290
2.274
2.262
1.943
1.923
9
8
7
6
5
4
3
2
1
0
ppm
1.000
1.023
1.032
2.131
5.998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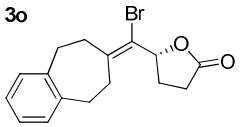
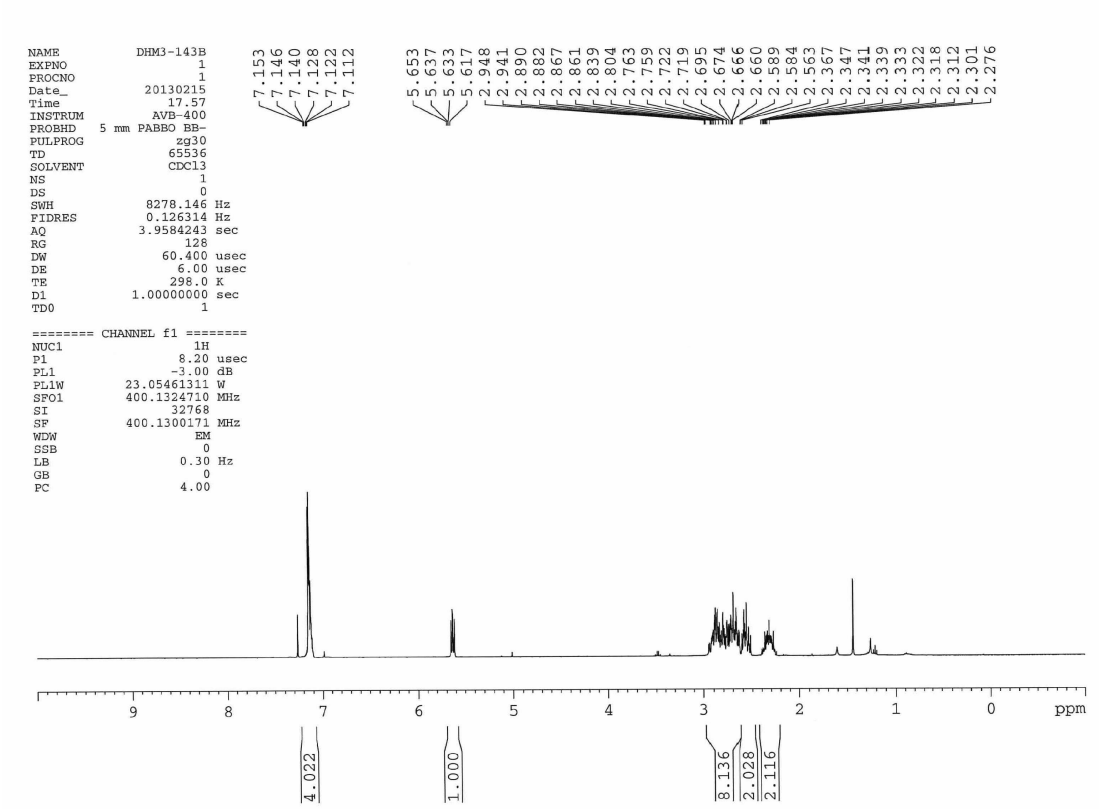
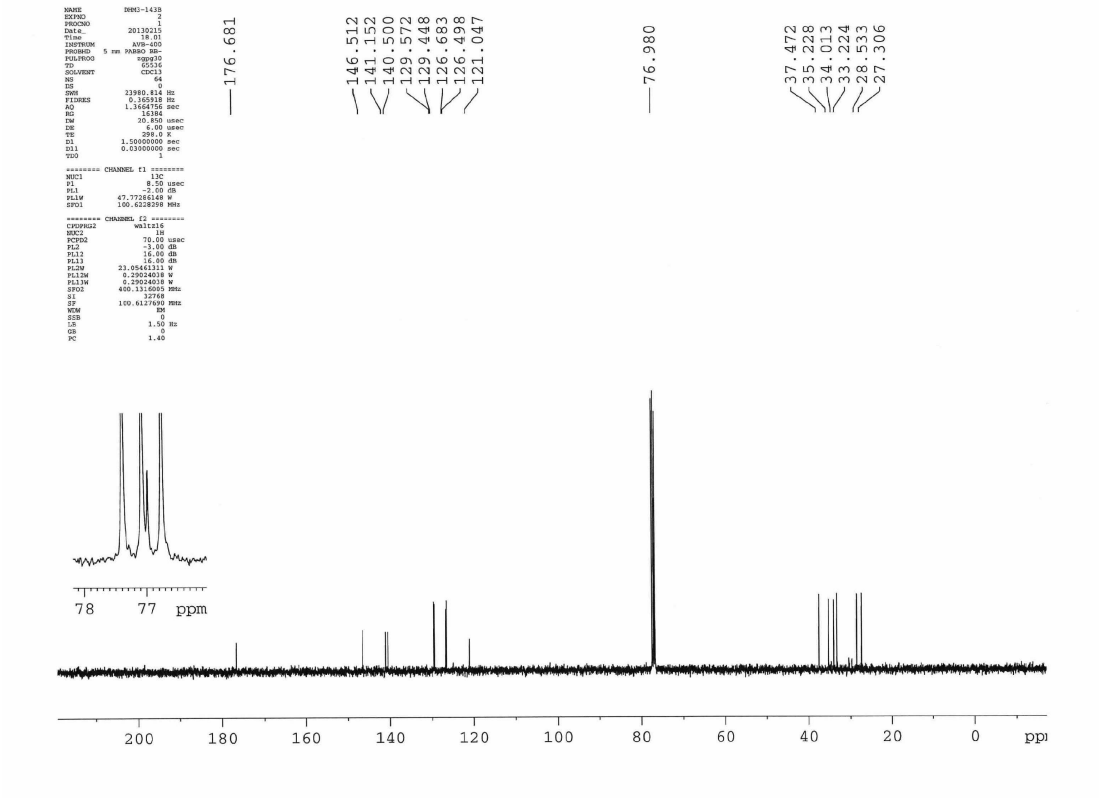
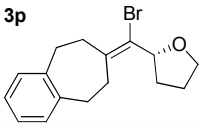
EXPNO 2
PROCNO 1
Date_ 20130213
Time 13.23
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 0
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 16384
DW 20.850 usec
DE 6.00 usec
TE 295.7 K
D1 1.50000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 47.77286148 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -3.00 dB
PL12 16.00 dB
PL13 16.00 dB
PL2W 23.05461311 W
PL12W 0.29024038 W
PL13W 0.29024038 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.50 Hz
GB 0
PC 1.40
176.702
137.541
120.391
77.605
28.521
27.000
25.880
21.076
80
79
78
77
76
75
ppm
200
180
160
140
120
100
80
60
40
20
0
ppm

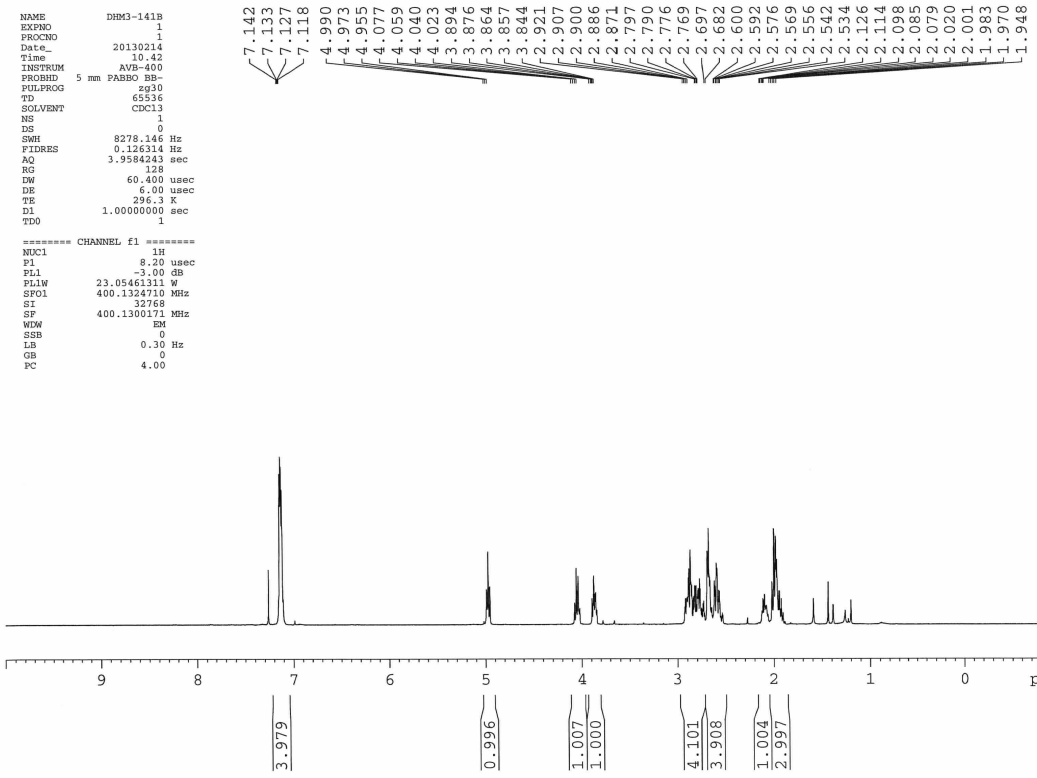

3o
Br
O
O


NAME DHM3-143B
EXPNO 1
PROCNO 1
Date_ 20130215
Time 17.57
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 128
DW 60.400 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.20 usec
PL1 -3.00 dB
PL1W 23.05461311 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300171 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
7.153
7.146
7.140
7.128
7.122
7.112
5.653
5.637
5.633
5.617
2.948
2.941
2.890
2.882
2.867
2.861
2.839
2.804
2.763
2.759
2.722
2.719
2.695
2.674
2.666
2.660
2.589
2.584
2.563
2.367
2.347
2.341
2.339
2.333
2.322
2.318
2.312
2.301
2.276
9
8
7
6
5
4
3
2
1
0
ppm
4.022
1.000
8.136
2.028
2.116

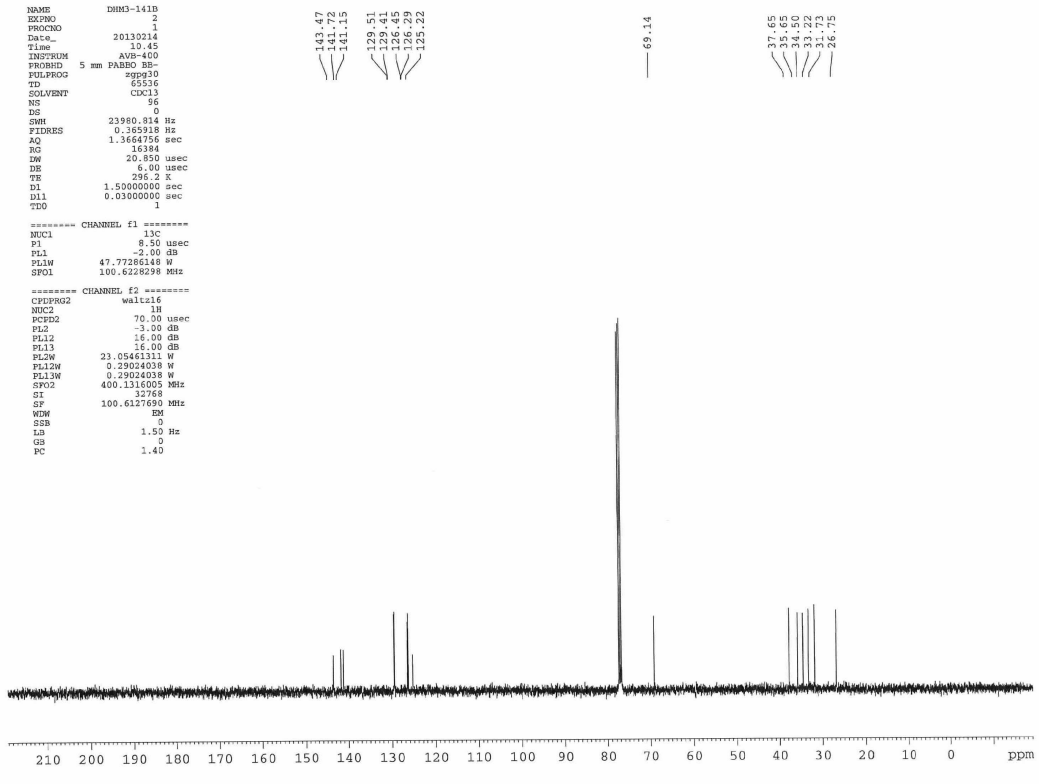

NAME DHM3-143B
EXPNO 2
PROCNO 1
Date_ 20130215
Time 18.01
INSTRUM AVB-400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 0
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 16384
DW 20.850 usec
DE 6.00 usec
TE 298.0 K
D1 1.50000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 47.77286148 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -3.00 dB
PL12 16.00 dB
PL13 16.00 dB
PL2W 23.05461311 W
PL12W 0.29024038 W
PL13W 0.29024038 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.50 Hz
GB 0
PC 1.40
176.681
146.512
141.152
140.500
129.572
129.448
126.683
126.498
121.047
76.980
37.472
35.228
34.013
33.224
28.533
27.306
78
77
ppm
200
180
160
140
120
100
80
60
40
20
0
ppm

3p

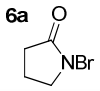


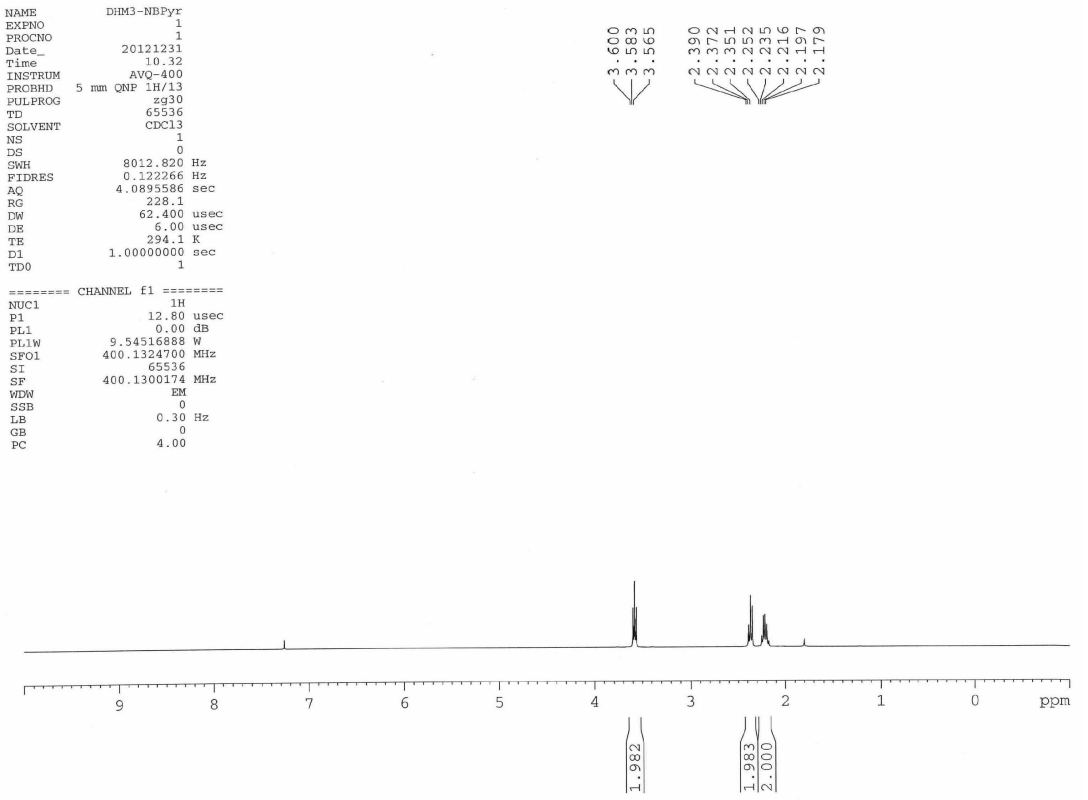


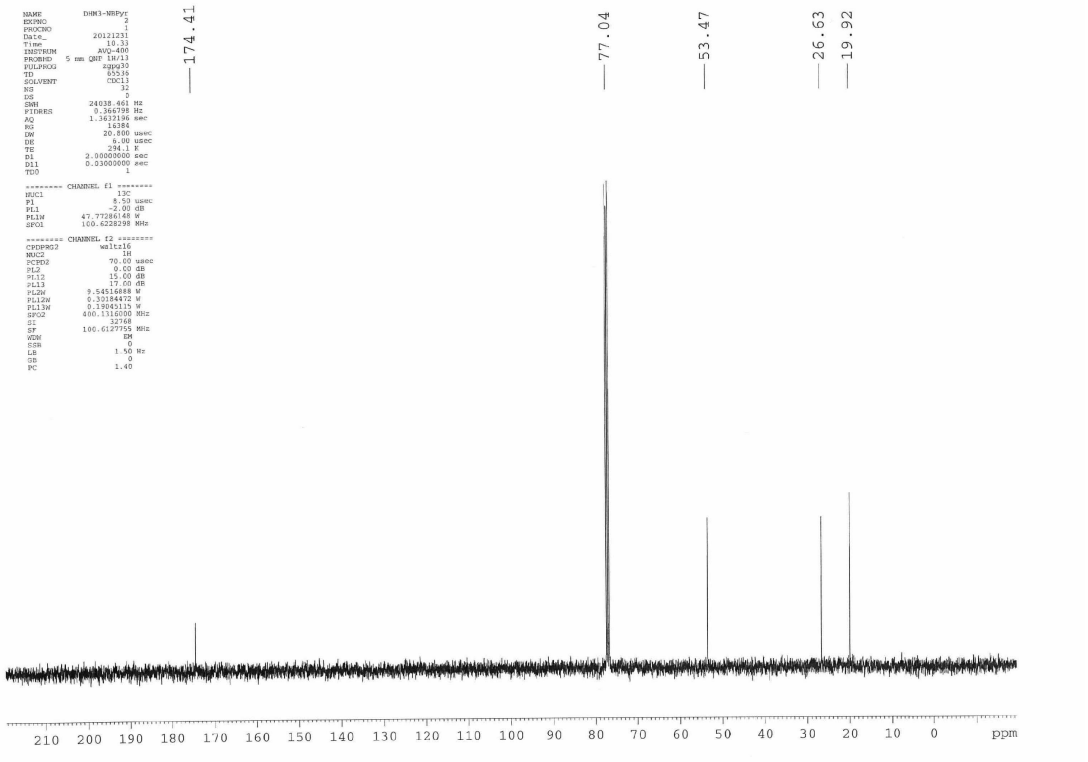

```
NAME            DHM3-141B
EXPNO                   1
PROCNO                  1
Date_            20130214
Time                10.42
INSTRUM           AVB-400
PROBHD   5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      1
DS                      0
SWH              8278.146 Hz
FIDRES           0.126314 Hz
AQ               3.9584243 sec
RG                    128
DW                 60.400 usec
DE                  6.00 usec
TE                 296.3 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                   8.20 usec
PL1                 -3.00 dB
PL1W          23.05461311 W
SFO1         400.1324710 MHz
SI                   32768
SF           400.1300171 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   4.00
```



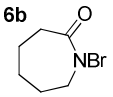

```
NAME            DHM3-141B
EXPNO                   2
PROCNO                  1
Date_            20130214
Time                10.45
INSTRUM           AVB-400
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                      96
DS                      0
SWH              23980.814 Hz
FIDRES           0.365918 Hz
AQ               1.3664756 sec
RG                    16384
DW                 20.850 usec
DE                  6.00 usec
TE                 296.2 K
D1              1.50000000 sec
D11             0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   13C
P1                   8.50 usec
PL1                 -2.00 dB
PL1W          47.77286148 W
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                   1H
PCPD2                70.00 usec
PL2                 -3.00 dB
PL12                16.00 dB
PL13                16.00 dB
PL2W          23.05461311 W
PL12W          0.29024038 W
PL13W          0.29024038 W
SFO2         400.1316005 MHz
SI                   32768
SF           100.6127690 MHz
WDW                    EM
SSB                     0
LB                   1.50 Hz
GB                      0
PC                   1.40
```

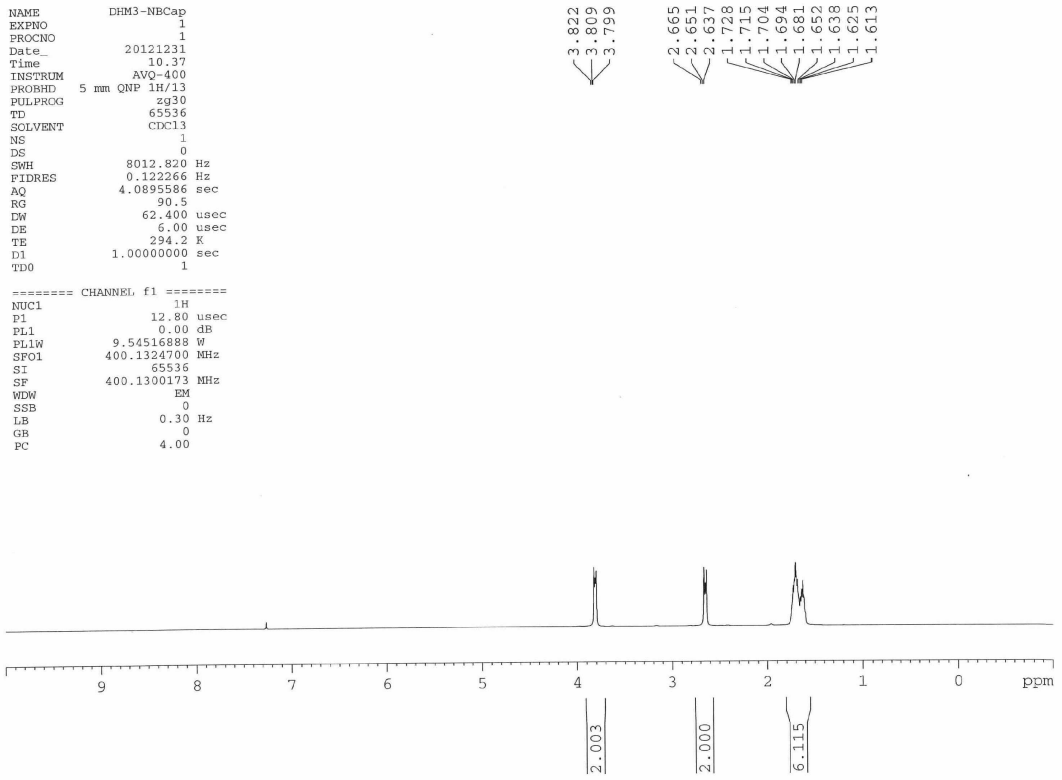

6a
O
NBr

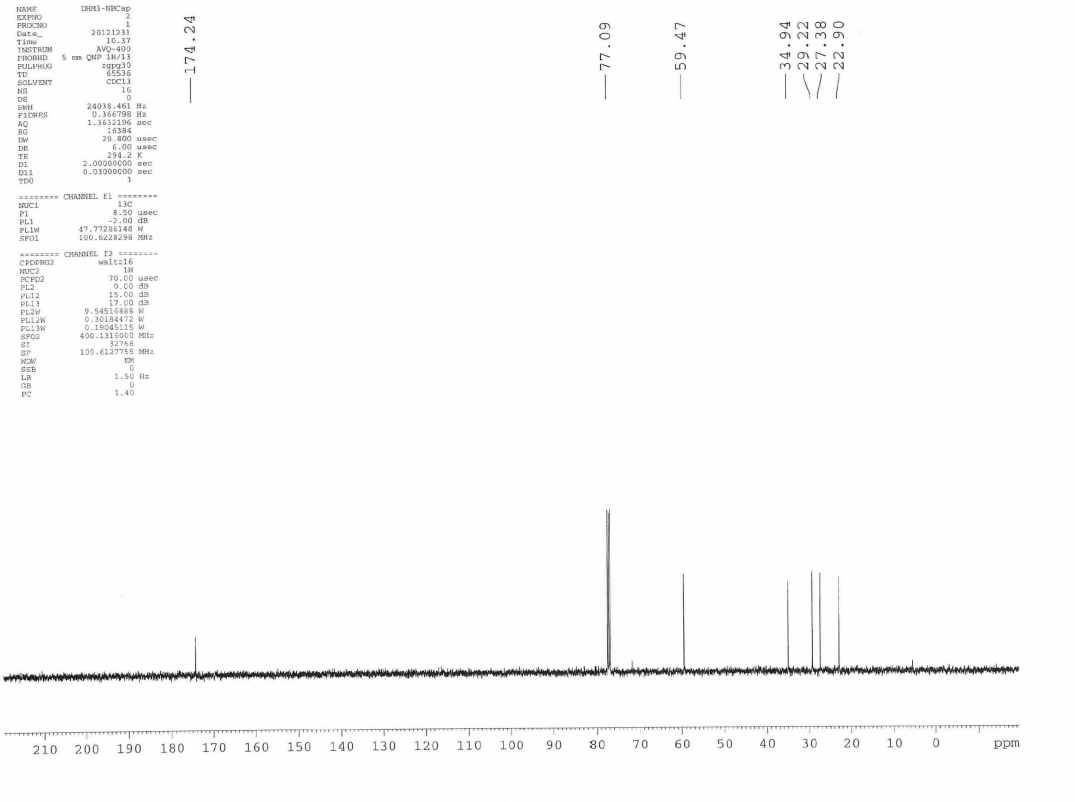

NAME DHM3-NBPyr
EXPNO 1
PROCNO 1
Date_ 20121231
Time 10.32
INSTRUM AVQ-400
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0895586 sec
RG 228.1
DW 62.400 usec
DE 6.00 usec
TE 294.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.80 usec
PL1 0.00 dB
PL1W 9.54516888 W
SFO1 400.1324700 MHz
SI 65536
SF 400.1300174 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
3.600
3.583
3.565
2.390
2.372
2.351
2.252
2.235
2.216
2.197
2.179
9
8
7
6
5
4
3
2
1
0
ppm
1.982
1.983
2.000

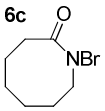

NAME DHM3-NBPyr
EXPNO 2
PROCNO 1
Date_ 20121231
Time 10.33
INSTRUM AVQ-400
PROBHD 5 mm QNP 1H/13
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3632196 sec
RG 16384
DW 20.800 usec
DE 6.00 usec
TE 294.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
PL1W 47.77286148 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 0.00 dB
PL12 15.60 dB
PL13 17.00 dB
PL2W 9.54516888 W
PL12W 0.30184472 W
PL13W 0.18045115 W
SFO2 400.1316000 MHz
SI 32768
SF 100.6127755 MHz
WDW EM
SSB 0
LB 1.50 Hz
GB 0
PC 1.40
174.41
77.04
53.47
26.63
19.92
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

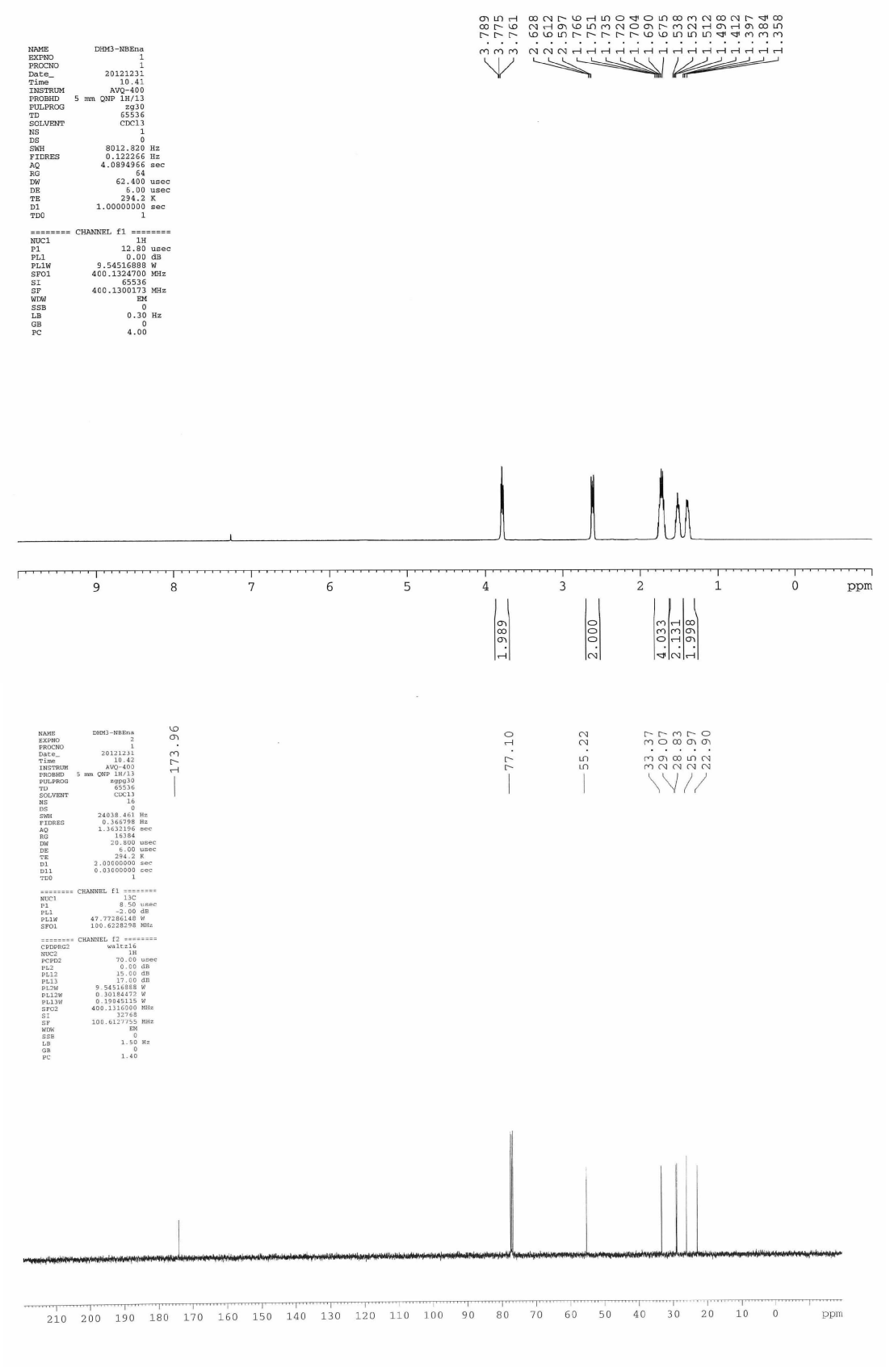
6b








6c
O
NBr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SOLVENT CDCl3
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D1 1.00000000 sec
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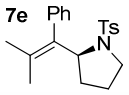
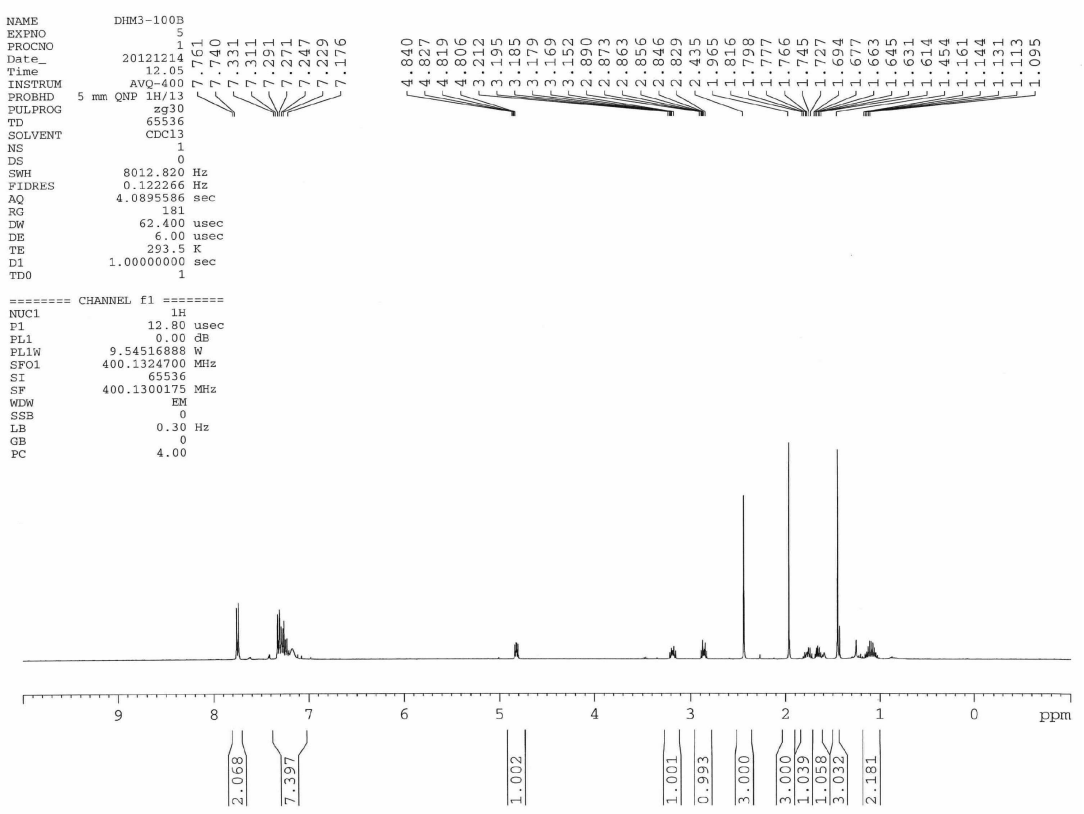
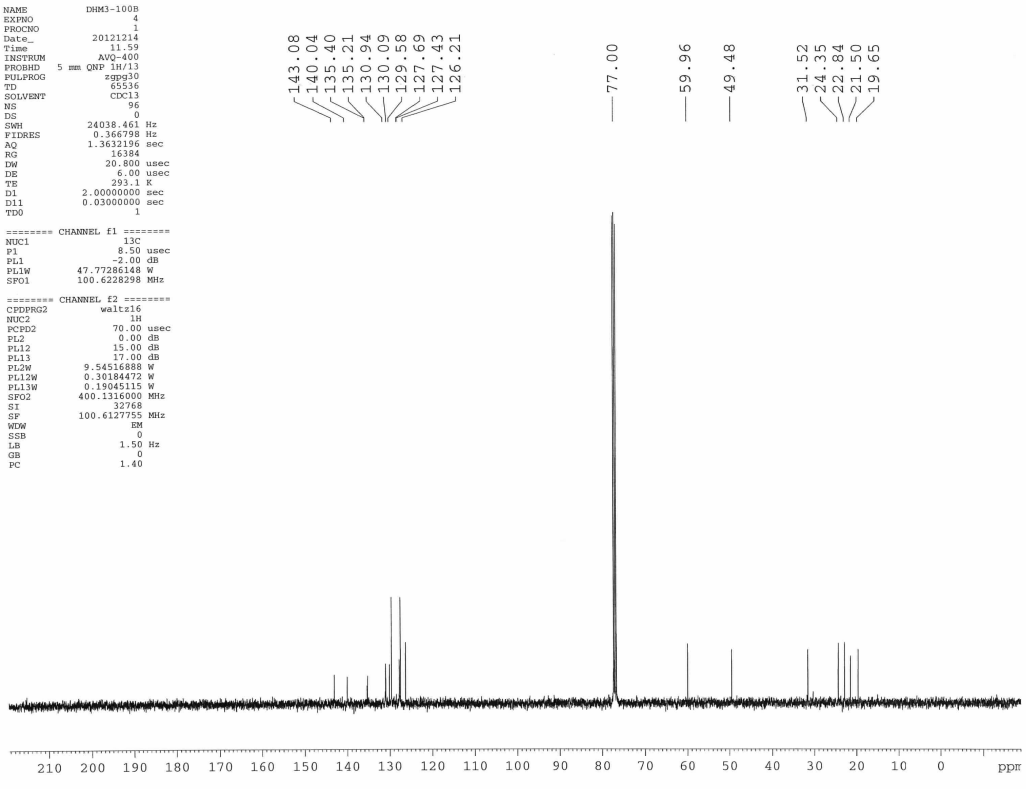
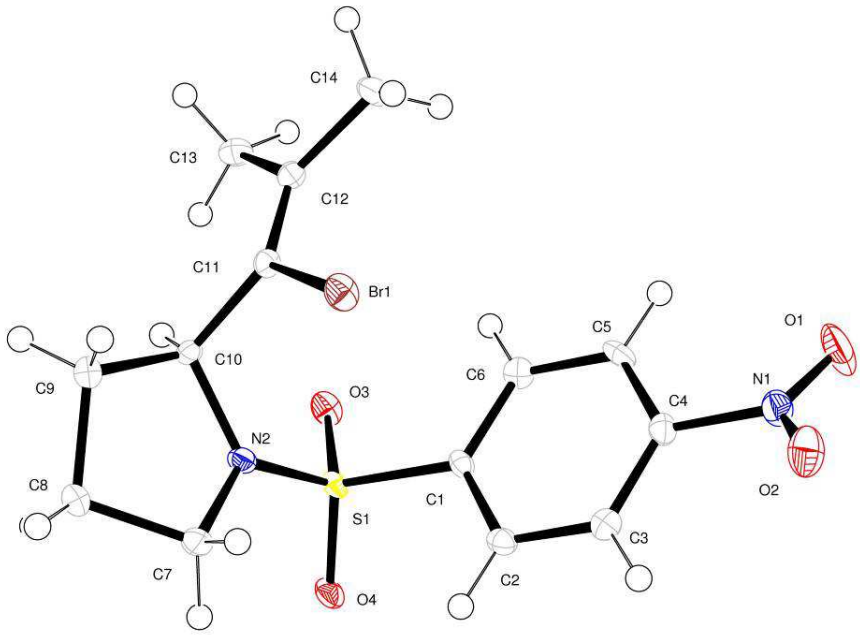






Crystal Structure of **3b**

ORTEP Representation:



Experimental Details:

A colorless block 0.120 x 0.100 x 0.100 mm in size was mounted on a Cryoloop with Paratone oil. Data were collected in a nitrogen gas stream at 100(2) K using phi and omega scans. Crystal-to-detector distance was 60 mm and exposure time was 5 seconds per frame using a scan width of 1.0°. Data collection was 99.9% complete to 67.000° in θ. A total of 33759 reflections were collected covering the indices, -9<=*h*<=9, -16<=*k*<=16, -16<=*l*<=17. 2848 reflections were found to be symmetry independent, with an R_{int} of 0.0251. Indexing and unit cell refinement indicated a primitive, orthorhombic lattice. The space group was found to be P 21 21 21 (No. 19). The data were integrated using the Bruker SAINT software program and scaled using the SADABS software program. Solution by charge flipping methods (SUPERFLIP) produced a complete heavy-atom phasing model consistent with the proposed structure. All non-hydrogen atoms were refined anisotropically by full-matrix least-squares (SHELXL-2012). All hydrogen atoms were placed using a riding model. Their positions were constrained relative to their parent atom using the appropriate HFIX command in SHELXL-2012. Absolute stereochemistry was unambiguously determined to be *S* at C10.

Table S1. Crystal data and structure refinement.

Empirical formula	C14 H17 Br N2 O4 S	
Formula weight	389.26	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	Orthorhombic	
Space group	P 21 21 21	
Unit cell dimensions	a = 7.6678(10) Å	α= 90°.
	b = 13.5683(18) Å	β= 90°.
	c = 14.958(2) Å	γ = 90°.
Volume	1556.3(4) $Å^3$	
Z	4	
Density (calculated)	1.661 Mg/m^3	

Absorption coefficient	5.038 mm^{-1}
F(000)	792
Crystal size	0.120 x 0.100 x 0.100 mm^3
Crystal color/habit	colorless block
Theta range for data collection	4.399 to 68.291°.
Index ranges	-9<=h<=9, -16<=k<=16, -16<=l<=17
Reflections collected	33759
Independent reflections	2848 [R(int) = 0.0251]
Completeness to theta = 67.000°	99.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.753 and 0.669
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2848 / 0 / 202
Goodness-of-fit on F^2	1.183
Final R indices [I>2sigma(I)]	R1 = 0.0159, wR2 = 0.0411
R indices (all data)	R1 = 0.0159, wR2 = 0.0411
Absolute structure parameter	-0.007(4)
Extinction coefficient	0.00218(12)
Largest diff. peak and hole	0.270 and -0.252 e.$Å^{-3}$

Table S2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3). U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	2069(3)	12457(2)	1873(2)	12(1)
C(2)	3838(4)	12526(2)	1669(2)	14(1)
C(3)	5031(4)	12627(2)	2357(2)	14(1)
C(4)	4408(3)	12671(2)	3225(2)	14(1)
C(5)	2649(4)	12612(2)	3440(2)	15(1)
C(6)	1468(3)	12499(2)	2752(2)	14(1)
C(7)	2021(4)	10818(2)	-14(2)	15(1)
C(8)	1047(4)	9947(2)	-423(2)	20(1)
C(9)	91(4)	9482(2)	370(2)	18(1)
C(10)	-381(3)	10350(2)	995(2)	12(1)
C(11)	-90(3)	10156(2)	1976(2)	12(1)
C(12)	-1262(3)	10169(2)	2629(2)	14(1)
C(13)	-3180(3)	10333(2)	2442(2)	17(1)
C(14)	-859(4)	10024(2)	3599(2)	20(1)
N(1)	5682(3)	12771(2)	3961(2)	20(1)
N(2)	739(3)	11159(2)	650(1)	13(1)
O(1)	5170(3)	13128(2)	4663(1)	34(1)
O(2)	7174(3)	12480(2)	3831(1)	26(1)
O(3)	-1150(2)	12388(1)	1365(1)	17(1)
O(4)	1116(3)	12906(1)	268(1)	17(1)
S(1)	553(1)	12280(1)	984(1)	12(1)
Br(1)	2318(1)	9887(1)	2245(1)	17(1)

Table 3. Bond lengths [Å] and angles [°].

C(1)-C(2)	1.393(4)	C(5)-C(6)	1.379(4)
C(1)-C(6)	1.395(4)	C(5)-H(5)	0.9500
C(1)-S(1)	1.782(3)	C(6)-H(6)	0.9500
C(2)-C(3)	1.383(4)	C(7)-N(2)	1.472(3)
C(2)-H(2)	0.9500	C(7)-C(8)	1.527(4)
C(3)-C(4)	1.385(4)	C(7)-H(7A)	0.9900
C(3)-H(3)	0.9500	C(7)-H(7B)	0.9900
C(4)-C(5)	1.389(4)	C(8)-C(9)	1.529(4)
C(4)-N(1)	1.479(3)	C(8)-H(8A)	0.9900

C(8)-H(8B)	0.9900
C(9)-C(10)	1.547(4)
C(9)-H(9A)	0.9900
C(9)-H(9B)	0.9900
C(10)-N(2)	1.485(3)
C(10)-C(11)	1.508(3)
C(10)-H(10)	1.0000
C(11)-C(12)	1.327(3)
C(11)-Br(1)	1.925(2)
C(12)-C(14)	1.497(4)
C(12)-C(13)	1.513(3)
C(13)-H(13A)	0.9800
C(13)-H(13B)	0.9800
C(13)-H(13C)	0.9800
C(14)-H(14A)	0.9800
C(14)-H(14B)	0.9800
C(14)-H(14C)	0.9800
N(1)-O(1)	1.221(3)
N(1)-O(2)	1.226(3)
N(2)-S(1)	1.608(2)
O(3)-S(1)	1.4324(19)
O(4)-S(1)	1.4334(19)

C(2)-C(1)-C(6)	121.7(2)
C(2)-C(1)-S(1)	118.70(19)
C(6)-C(1)-S(1)	119.58(19)
C(3)-C(2)-C(1)	119.2(2)
C(3)-C(2)-H(2)	120.4
C(1)-C(2)-H(2)	120.4
C(2)-C(3)-C(4)	118.2(3)
C(2)-C(3)-H(3)	120.9
C(4)-C(3)-H(3)	120.9
C(3)-C(4)-C(5)	123.3(2)
C(3)-C(4)-N(1)	118.3(2)
C(5)-C(4)-N(1)	118.3(2)
C(6)-C(5)-C(4)	118.2(2)
C(6)-C(5)-H(5)	120.9
C(4)-C(5)-H(5)	120.9
C(5)-C(6)-C(1)	119.4(2)
C(5)-C(6)-H(6)	120.3
C(1)-C(6)-H(6)	120.3
N(2)-C(7)-C(8)	100.7(2)
N(2)-C(7)-H(7A)	111.6
C(8)-C(7)-H(7A)	111.6
N(2)-C(7)-H(7B)	111.6
C(8)-C(7)-H(7B)	111.6
H(7A)-C(7)-H(7B)	109.4
C(7)-C(8)-C(9)	104.1(2)
C(7)-C(8)-H(8A)	110.9
C(9)-C(8)-H(8A)	110.9
C(7)-C(8)-H(8B)	110.9
C(9)-C(8)-H(8B)	110.9
H(8A)-C(8)-H(8B)	109.0
C(8)-C(9)-C(10)	105.5(2)
C(8)-C(9)-H(9A)	110.6
C(10)-C(9)-H(9A)	110.6
C(8)-C(9)-H(9B)	110.6
C(10)-C(9)-H(9B)	110.6
H(9A)-C(9)-H(9B)	108.8
N(2)-C(10)-C(11)	112.4(2)
N(2)-C(10)-C(9)	102.56(19)
C(11)-C(10)-C(9)	114.9(2)
N(2)-C(10)-H(10)	108.9
C(11)-C(10)-H(10)	108.9
C(9)-C(10)-H(10)	108.9
C(12)-C(11)-C(10)	127.8(2)
C(12)-C(11)-Br(1)	119.91(18)
C(10)-C(11)-Br(1)	112.24(16)
C(11)-C(12)-C(14)	124.8(2)
C(11)-C(12)-C(13)	121.6(2)
C(14)-C(12)-C(13)	113.5(2)
C(12)-C(13)-H(13A)	109.5
C(12)-C(13)-H(13B)	109.5
H(13A)-C(13)-H(13B)	109.5
C(12)-C(13)-H(13C)	109.5
H(13A)-C(13)-H(13C)	109.5
H(13B)-C(13)-H(13C)	109.5
C(12)-C(14)-H(14A)	109.5
C(12)-C(14)-H(14B)	109.5
H(14A)-C(14)-H(14B)	109.5
C(12)-C(14)-H(14C)	109.5
H(14A)-C(14)-H(14C)	109.5
H(14B)-C(14)-H(14C)	109.5
O(1)-N(1)-O(2)	124.4(2)
O(1)-N(1)-C(4)	117.7(2)
O(2)-N(1)-C(4)	118.0(2)
C(7)-N(2)-C(10)	112.86(19)
C(7)-N(2)-S(1)	124.43(18)
C(10)-N(2)-S(1)	122.71(17)
O(3)-S(1)-O(4)	120.74(12)
O(3)-S(1)-N(2)	107.49(12)
O(4)-S(1)-N(2)	107.60(11)
O(3)-S(1)-C(1)	106.52(12)
O(4)-S(1)-C(1)	106.36(11)
N(2)-S(1)-C(1)	107.51(12)

Symmetry transformations used to generate equivalent atoms:
-none-

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$). The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$.

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	15(1)	11(1)	9(1)	0(1)	-2(1)	2(1)
C(2)	18(1)	11(1)	12(1)	0(1)	2(1)	0(1)
C(3)	14(1)	12(1)	16(1)	0(1)	0(1)	1(1)

C(4)	18(1)	11(1)	14(1)	1(1)	-4(1)	0(1)
C(5)	22(1)	15(1)	8(1)	0(1)	3(1)	3(1)
C(6)	12(1)	15(1)	16(1)	-2(1)	1(1)	3(1)
C(7)	15(1)	16(1)	14(1)	-2(1)	5(1)	-1(1)
C(8)	26(1)	21(1)	13(1)	-5(1)	4(1)	-7(1)
C(9)	21(1)	18(1)	14(1)	-2(1)	0(1)	-5(1)
C(10)	10(1)	14(1)	12(1)	2(1)	-1(1)	-4(1)
C(11)	10(1)	13(1)	13(1)	2(1)	-4(1)	0(1)
C(12)	17(1)	10(1)	14(1)	1(1)	0(1)	-1(1)
C(13)	16(1)	18(1)	18(1)	2(1)	4(1)	-1(1)
C(14)	26(1)	22(1)	12(1)	3(1)	2(1)	-1(1)
N(1)	24(1)	18(1)	16(1)	2(1)	-6(1)	-4(1)
N(2)	14(1)	14(1)	11(1)	0(1)	2(1)	0(1)
O(1)	35(1)	52(1)	14(1)	-10(1)	-6(1)	1(1)
O(2)	20(1)	29(1)	29(1)	-1(1)	-11(1)	3(1)
O(3)	14(1)	22(1)	15(1)	-1(1)	-2(1)	5(1)
O(4)	26(1)	15(1)	10(1)	2(1)	-3(1)	0(1)
S(1)	15(1)	13(1)	9(1)	0(1)	-2(1)	2(1)
Br(1)	14(1)	22(1)	16(1)	1(1)	-4(1)	3(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3).

	x	y	z	U(eq)
H(2)	4220	12506	1065	16
H(3)	6246	12664	2237	17
H(5)	2270	12649	4043	18
H(6)	256	12449	2876	17
H(7A)	3124	10608	273	18
H(7B)	2273	11333	-465	18
H(8A)	1869	9472	-696	24
H(8B)	211	10171	-884	24
H(9A)	852	9004	681	21
H(9B)	-976	9137	166	21
H(10)	-1631	10531	899	15
H(13A)	-3324	10597	1836	26
H(13B)	-3654	10803	2876	26
H(13C)	-3805	9706	2492	26
H(14A)	270	9693	3660	30
H(14B)	-1770	9617	3873	30
H(14C)	-814	10665	3899	30