

**Reactions of Oxetan-3-*tert*-butylsulfinimine
for the Preparation of Substituted 3-Aminooxetanes**

Philip J. Hamzik and Jason D. Brubaker*

Organic Letters

Supporting Information

Index:

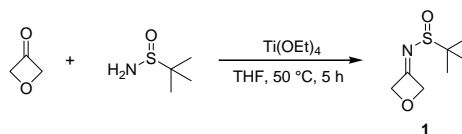
General Experimental Procedures	S1
Materials	S1
Instrumentation	S2
Synthesis of Oxetan-3- <i>tert</i> -butylsulfinimine (1)	S2
Synthesis of Sulfinyl Aziridine 6	S4
Aryllithium Additions Into Oxetan-3- <i>tert</i> -butylsulfinimine (1)	S5
Addition of Diverse Nucleophiles into Oxetan-3- <i>tert</i> -butylsulfinimine (1)	S13
Desilylation of TMS-Alkyne 5a and [2+3] Cyclization of the Deprotected Alkyne	S23
Ring-opening Reactions of the Sulfinyl Aziridine X	S24
Representative Deprotections of the <i>tert</i> -Butylsulfinyl Group	S27
¹ H and ¹³ C NMR Spectra	S31

General Experimental Procedures. All reactions were performed in oven-dried glassware fitted with rubber septa under a positive pressure of nitrogen, unless otherwise noted. Air- and moisture-sensitive liquids were transferred under a positive pressure of nitrogen via syringe. Organic solutions were concentrated by rotary evaporation (house vacuum, ca. 25-40 torr) at ambient temperature. Reactions were monitored using analytical thin-layer chromatography on pre-coated glass plates (silica gel 60 F254, 250 μm thickness), or by LC/MS (30 mm x 2 mm 2 micron column + guard; 2 μL injection; 3% to 98% MeCN/water + 0.05% TFA gradient over 2.3 minutes; 0.9 mL/min flow; APCI; positive ion mode; UV detection at 254 nM). Developed TLC plates were visualized by either exposure to ultraviolet light (UV) or iodine stain. Flash-column chromatography was performed on an automated purification system using pre-packed silica columns (Biotage SNAP Cartridge KP-Sil). Acetone cooling baths were cooled to the appropriate temperature by the addition of dry ice.

Materials. Commercial reagents and solvents were used. All solvents were purchased in septum-sealed bottles and stored under an inert atmosphere.

Instrumentation. Automated purifications were performed on a Biotage SP1. Analytical LC/MS runs were performed on an Agilent Technologies 1100 Series chromatograph with an Agilent Technologies LC/MSD SL mass spectrometer. Proton magnetic resonance (^1H NMR) spectra were recorded on Varian INOVA-600 (600 MHz) at 22 °C. Proton chemical shifts are expressed in parts per million (ppm, δ scale) and are referred to residual protium in the NMR solvent (CHCl_3 : δ 7.26, D_2HCO : δ 3.31). Data are represented as follows: chemical shift, multiplicity (s = singlet, d = doublet, dd = doublet of doublets, ddd = doublet of doublet of doublets, t = triplet, q = quartet, m = multiplet), integration, and coupling constant (J) in Hertz (Hz). Carbon nuclear magnetic resonance (^{13}C NMR) spectra were recorded on Varian INOVA-600 (150 MHz) NMR spectrometer at 22 °C. Carbon chemical shifts are expressed in parts per million (ppm, δ scale) and are referenced to the carbon resonance of the NMR solvent (CDCl_3 : δ 77.0, D_3COD : 44.9).

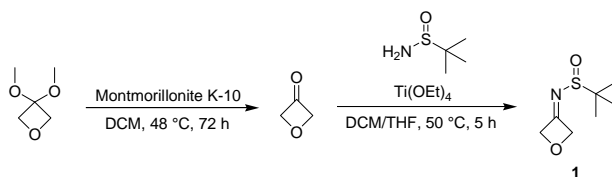
Synthesis of Oxetan-3-*tert*-butylsulfinimine (1).



2-Methyl-2-propane-sulfinamide (841 mg, 6.94 mmol, 1.00 equiv) and titanium (IV) ethoxide (3.14 mL, 13.9 mmol, 2.00 equiv) were added sequentially to a solution of oxetan-3-one (500 mg, 6.94 mmol, 1 equiv) in tetrahydrofuran (17.0 mL) at 22 °C. The mixture was stirred at 50 °C for 5 h before being poured over a stirring saturated aqueous sodium chloride solution (100 mL). The suspension was filtered through a pad of celite, washing with ethyl acetate. The filtrate was partitioned, and the aqueous layer was further extracted with ethyl acetate (100 mL). The organic layers were combined and dried with anhydrous sodium sulfate. The dried solution was filtered and the filtrate was concentrated, yielding a yellow oil. The crude product was purified by flash-column chromatography (30% ethyl acetate-hexanes, grading to 70% ethyl acetate-hexanes), affording oxetan-3-*tert*-butylsulfinimine (**1**, 551 mg, 3.14 mmol, 45.3%) as a yellow oil. Note: The product-containing fractions from the column were concentrated at \approx 20 torr, followed by brief (10–20 min) exposure to high vacuum (1.0 torr) due to the volatility of oxetan-3-*tert*-butylsulfinimine.

TLC (50% ethyl acetate-hexanes):	$R_f = 0.51$ (UV)
^1H NMR (600 MHz, CDCl_3) δ :	5.78 (ddd, $J = 2.0, 4.4, 15.5$ Hz, 1H), 5.65 (ddd, $J = 1.6, 4.4, 15.4$ Hz, 1H), 5.52 – 5.35 (m, 2H), 1.25 (s, 9H).
^{13}C NMR (150 MHz, CDCl_3) δ :	176.3, 86.2, 86.0, 58.0, 22.3.
HRMS:	Calcd for $(\text{C}_7\text{H}_{13}\text{NO}_2\text{S}+\text{H})^+$: 176.0740 Found: 176.0733

On large scale, rather than purchase oxetan-3-one, **1** was prepared in a two-step procedure from 3,3-dimethoxy oxetane, an intermediate easily prepared in large quantities.¹



Montmorillonite K-10 (18.8 g) was added to a solution of 3,3-dimethoxyoxetane (3.00 g, 25.4 mmol, 1 equiv) in dichloromethane (445 mL). A reflux condenser was fitted to the top of the round-bottomed flask, and the suspension was heated at reflux (bath temp 48 °C) for 48 h. ^1H NMR analysis of an aliquot from the reaction mixture indicated that 3,3-dimethoxyoxetane still remained, so an additional portion of Montmorillonite K-10 clay (18.8 g) was added and the mixture was stirred at 48 °C for an additional 24 h. After cooling to 22 °C, anhydrous sodium sulfate was added and the mixture was stirred for 1 min before the dried reaction mixture was filtered through a fritted funnel into a 1-liter round-bottomed flask. Tetrahydrofuran (100 mL) was added to the filtrate, followed sequentially by 2-methyl-2-propane-sulfinamide (4.62 g, 38.1 mmol, 1.50 equiv) and titanium(IV) ethoxide (20.1 mL, 89.0 mmol, 3.50 equiv). A reflux condenser was fitted to the top of the round-bottomed flask and the reaction was heated at reflux (bath temp 50 °C) for 5 h before being poured over stirring saturated sodium chloride solution (400

(1) Wuitschik, G. ; Rogers-Evans, M. ; Muller, K.; Fischer, H.; Wagner, B.; Schuler, F.; Polonchuk, L. ; Carreira, E. M. *Angew. Chem. Int. Ed.* **2006**, 45, 7736–7739.

mL). The suspension was filtered through a pad of celite, washing with dichloromethane. The filtrate was concentrated, ethyl acetate (300 mL) was added, and the mixture was partitioned. The aqueous layer was further extracted with ethyl acetate (300 mL), and the organic layers were combined and dried with anhydrous sodium sulfate. The dried solution was filtered and the filtrate was concentrated, yielding a yellow oil. The crude product was purified by flash-column chromatography (30% ethyl acetate-hexanes, grading to 70% ethyl acetate-hexanes), affording the desired oxetan-3-*tert*-butylsulfinimine (**1**, 1.42 g, 7.70 mmol, 30.0%) as a yellow oil. Note: The product-containing fractions were concentrated at \approx 20 torr, followed by brief (10–20 min) exposure to high vacuum (1 torr) due to the volatility of oxetan-3-*tert*-butylsulfinimine.

TLC (50% ethyl acetate-hexanes):	$R_f = 0.51$ (UV)
^1H NMR (600 MHz, CDCl_3) δ :	5.78 (ddd, $J = 2.0, 4.4, 15.5$ Hz, 1H), 5.65b (ddd, $J = 1.6, 4.4, 15.4$, 1H), 5.52 – 5.35 (m, 2H), 1.25 (s, 9H).
^{13}C NMR (150 MHz, CDCl_3) δ :	176.3, 86.2, 86.0, 58.0, 22.3.
HRMS:	Calcd for $(\text{C}_7\text{H}_{13}\text{NO}_2\text{S}+\text{H})^+$: 176.0740 Found: 176.0733

Synthesis of Sulfinyl Aziridine 6.

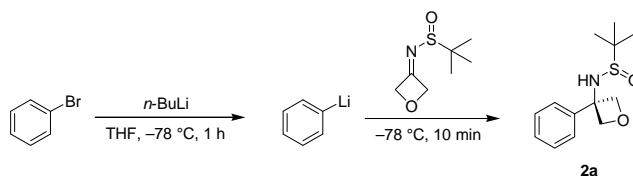


Sodium hydride (60 wt % in mineral oil, 75 mg, 1.9 mmol, 1.1 equiv) was added to a solution of trimethylsulfoxonium iodide (414 mg, 1.88 mmol, 1.10 equiv) in dimethyl sulfoxide (12.0 mL) at 22 °C. The mixture was stirred for 20 min at 22 °C, after which a solution of oxetan-3-*tert*-butylsulfinimine (**1**, 300 mg, 1.71 mmol, 1 equiv) in dimethyl sulfoxide (1.0 mL) was added. The resulting mixture was stirred for 1 h at 22 °C. Ethyl acetate (50 mL) and water (50 mL) were added and partitioned, and the aqueous layer was further extracted with ethyl acetate (50 mL). The organic layers were combined, washed with a saturated aqueous sodium chloride solution (50 mL), and the organic layer

was dried with anhydrous sodium sulfate. The dried solution was filtered, and the filtrate was concentrated to afford a colorless oil. The crude product was purified by flash-column chromatography (30% ethyl acetate-hexanes, grading to 80% ethyl acetate-hexanes) affording the sulfinyl aziridine **6** (270 mg, 1.4 mmol, 83%) as a white solid.

TLC (50% ethyl acetate-hexanes):	$R_f = 0.50$ (I_2)
^1H NMR (600 MHz, CDCl_3) δ :	5.05 (d, $J = 7.8$ Hz, 1H), 4.95 (s, 1H), 4.83 (d, $J = 7.8$ Hz, 1H), 4.79 (d, $J = 7.1$ Hz, 1H), 2.68 (s, 1H), 1.99 (s, 1H), 1.23 (s, 9H).
^{13}C NMR (150 MHz, CDCl_3) δ :	78.5, 75.8, 57.0, 41.6, 22.6.
HRMS:	Calcd for $(\text{C}_8\text{H}_{15}\text{NO}_2\text{S}+\text{H})^+$: 190.0896 Found: 190.0889

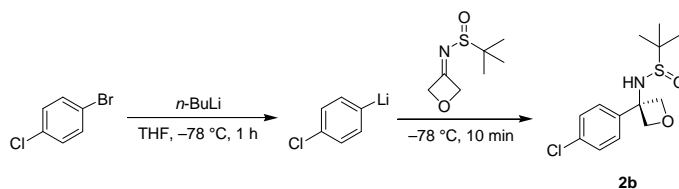
Aryllithium Additions Into Oxetan-3-*tert*-butylsulfinimine (**1**).



A solution of *n*-butyllithium in hexanes (2.5 M, 0.64 mL, 1.6 mmol, 1.4 equiv) was added dropwise to a solution of bromobenzene (269 mg, 1.71 mmol, 1.5 equiv) in tetrahydrofuran (10 mL) at -78 °C. The resulting mixture was stirred for 1.0 h at -78 °C before a solution of oxetan-3-*tert*-butylsulfinimine (**1**, 200 mg, 1.14 mmol, 1 equiv) in tetrahydrofuran (1.0 mL) was added dropwise at -78 °C. The reaction solution was stirred for an addition 10 min at -78 °C before being warmed to 22 °C and quenched with saturated aqueous ammonium chloride solution (3.0 mL). The crude reaction mixture was partitioned between water (20.0 mL) and ethyl acetate (40.0 mL). The aqueous layer was further extracted with ethyl acetate (40.0 mL), and the organic layers were combined. The combined layers were washed with saturated aqueous sodium chloride solution (40.0 mL), and the washed organic layer was dried with anhydrous sodium sulfate. The dried solution was filtered and the filtrate was concentrated, yielding a yellow oil. The crude product was purified by flash-column chromatography (50% ethyl acetate-hexanes,

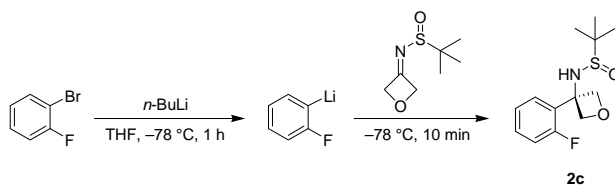
grading to 100% ethyl acetate), affording the addition product **2a** (262 mg, 1.03 mmol, 91.0%) as a colorless oil that solidified over time.

TLC (70 % ethyl acetate-hexanes):	$R_f = 0.28$ (UV)
^1H NMR (600 MHz, CDCl_3) δ :	7.45 – 7.39 (m, 2H), 7.36 (dd, $J = 7.1, 13.8$ Hz, 3H), 5.21 (d, $J = 6.9$ Hz, 1H), 5.07 (dd, $J = 3.3, 6.9$ Hz, 2H), 5.02 (d, $J = 6.9$ Hz, 1H), 4.03 (s, 1H), 1.21 (s, 9H).
^{13}C NMR (150 MHz, CDCl_3) δ :	141.1, 128.8, 128.2, 126.5, 83.8, 82.4, 63.0, 56.0, 22.5.
HRMS:	Calcd for $\text{C}_{13}\text{H}_{19}\text{NO}_2\text{S}$ $[\text{M}+\text{H}]^+$: 254.1209 Found: 254.1205



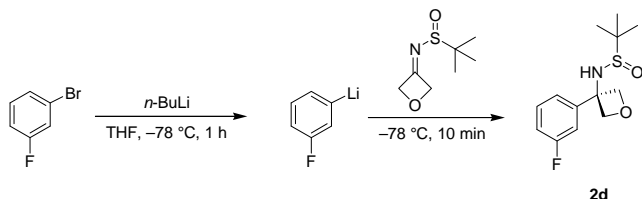
A solution of *n*-butyllithium in hexanes (1.6 M, 0.50 mL, 0.80 mmol, 1.4 equiv) was added dropwise to a solution of 4-chloro-bromobenzene (164 mg, 0.856 mmol, 1.50 equiv) in tetrahydrofuran (5.7 mL) at $-78\text{ }^{\circ}\text{C}$. The resulting mixture was stirred for 1.0 h at $-78\text{ }^{\circ}\text{C}$ before a solution of oxetan-3-*tert*-butylsulfinimine (**1**, 100 mg, 0.571 mmol, 1 equiv) in tetrahydrofuran (1.0 mL) was added dropwise at $-78\text{ }^{\circ}\text{C}$. The reaction solution was stirred for an addition 10 min at $-78\text{ }^{\circ}\text{C}$ before being warmed to $22\text{ }^{\circ}\text{C}$ and quenched with saturated aqueous ammonium chloride solution (3.0 mL). The crude reaction mixture was partitioned between water (20.0 mL) and ethyl acetate (20.0 mL). The aqueous layer was further extracted with ethyl acetate (20.0 mL), and the organic layers were combined. The combined layers were washed with saturated aqueous sodium chloride solution (20.0 mL), and the washed organic layer was dried with anhydrous sodium sulfate. The dried solution was filtered and the filtrate was concentrated, yielding a yellow oil. The crude product was purified by flash-column chromatography (50% ethyl acetate-hexanes, grading to 100% ethyl acetate-hexanes), affording the addition product **2b** (129 mg, 0.448 mmol, 79.0%) as a colorless oil that solidified over time.

TLC (70 % ethyl acetate-hexanes):	$R_f = 0.24$ (UV)
^1H NMR (600 MHz, CDCl_3) δ :	7.42 – 7.36 (m, 2H), 7.35 – 7.29 (m, 2H), 5.15 (d, $J = 7.0$ Hz, 1H), 5.05 (d, $J = 7.0$ Hz, 1H), 5.01 (d, $J = 6.9$ Hz, 1H), 4.93 (d, $J = 6.9$ Hz, 1H), 4.08 (s, 1H), 1.21 (s, 9H).
^{13}C NMR (150 MHz, CDCl_3) δ :	139.5, 134.2, 129.0, 128.0, 83.8, 82.2, 62.6, 56.1, 22.4.
HRMS:	Calcd for $(\text{C}_{13}\text{H}_{18}\text{ClNO}_2\text{S}+\text{H})^+$: 288.0820 Found: 288.0816



A solution of *n*-butyllithium in hexanes (1.6 M, 0.50 mL, 0.80 mmol, 1.4 equiv) was added dropwise to a solution of the aryl bromide (150 mg, 0.856 mmol, 1.50 equiv) in tetrahydrofuran (5.7 mL) at $-78\text{ }^{\circ}\text{C}$. The resulting mixture was stirred for 1.0 h at $-78\text{ }^{\circ}\text{C}$ before a solution of oxetan-3-*tert*-butylsulfinimine (**1**, 100 mg, 0.571 mmol, 1 equiv) in tetrahydrofuran (1.0 mL) was added dropwise at $-78\text{ }^{\circ}\text{C}$. The reaction solution was stirred for an addition 10 min at $-78\text{ }^{\circ}\text{C}$ before being warmed to $22\text{ }^{\circ}\text{C}$ and quenched with saturated aqueous ammonium chloride solution (3.0 mL). The crude reaction mixture was partitioned between water (20.0 mL) and ethyl acetate (20.0 mL). The aqueous layer was further extracted with ethyl acetate (20.0 mL), and the organic layers were combined. The combined layers were washed with saturated aqueous sodium chloride solution (20.0 mL), and the washed organic layer was dried with anhydrous sodium sulfate. The dried solution was filtered and the filtrate was concentrated, yielding a yellow oil. The crude product was purified by flash-column chromatography (50% ethyl acetate-hexanes, grading to 100% ethyl acetate, then flushing with 10% methanol-dichloromethane), affording the addition product **2c** (128 mg, 0.472 mmol, 83.0 %) as a colorless oil that solidified over time.

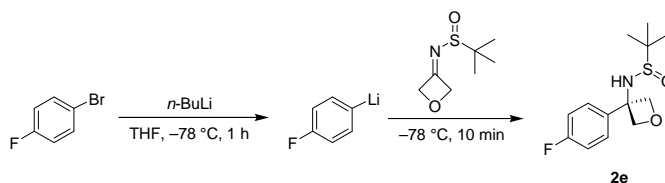
TLC (70% ethyl acetate-hexanes):	$R_f = 0.32$ (UV)
^1H NMR (600 MHz, CDCl_3) δ :	7.39 – 7.31 (m, 1H), 7.30 – 7.23 (m, 1H), 7.19 (t, $J = 7.5$ Hz, 1H), 7.13 – 7.02 (m, 1H), 5.21 (d, $J = 7.7$ Hz, 1H), 5.17 (d, $J = 7.2$ Hz, 1H), 5.00 (d, $J = 7.0$ Hz, 1H), 4.94 (d, $J = 7.2$ Hz, 1H), 4.12 (s, 1H), 1.18 (s, 9H).
^{13}C NMR (150 MHz, CDCl_3) δ :	161.2 (d, $J = 248.6$ Hz), 130.5, 128.4, 127.9, 124.3, 116.2 (d, $J = 21.0$ Hz), 82.5, 82.1, 61.1, 56.0, 22.3.
HRMS:	Calcd for $(\text{C}_{13}\text{H}_{18}\text{FNO}_2\text{S}+\text{H})^+$: 272.1115 Found: 272.1113



A solution of *n*-butyllithium in hexanes (1.6 M, 0.50 mL, 0.80 mmol, 1.4 equiv) was added dropwise to a solution of 3-fluoro-bromobenzene (150 mg, 0.856 mmol, 1.50 equiv) in tetrahydrofuran (5.7 mL) at -78 °C. The resulting mixture was stirred for 1.0 h at -78 °C before a solution of oxetan-3-*tert*-butylsulfinimine (**1**, 100 mg, 0.571 mmol, 1 equiv) in tetrahydrofuran (1.0 mL) was added dropwise at -78 °C. The reaction solution was stirred for an addition 10 min at -78 °C before being warmed to 22 °C and quenched with saturated aqueous ammonium chloride solution (3.0 mL). The crude reaction mixture was partitioned between water (20.0 mL) and ethyl acetate (20.0 mL). The aqueous layer was further extracted with ethyl acetate (20.0 mL), and the organic layers were combined. The combined layers were washed with saturated aqueous sodium chloride solution (20.0 mL), and the washed organic layer was dried with anhydrous sodium sulfate. The dried solution was filtered and the filtrate was concentrated, yielding a yellow oil. The crude product was purified by flash-column chromatography (50% ethyl acetate-hexanes, grading to 100% ethyl acetate, then flushing with 10% methanol-

dichloromethane), affording the addition product **2d** (140 mg, 0.516 mmol, 90.0%) as an amber oil.

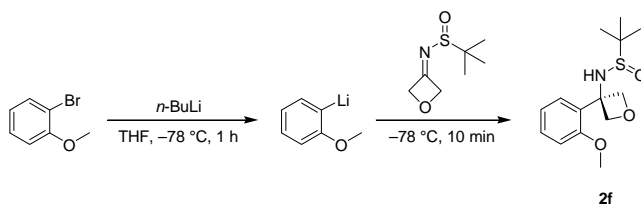
TLC (70% ethyl acetate-hexanes):	$R_f = 0.24$ (UV)
^1H NMR (600 MHz, CDCl_3) δ :	7.44 – 7.33 (m, 1H), 7.20 (d, $J = 7.8$ Hz, 1H), 7.09 (d, $J = 9.9$ Hz, 1H), 7.07 – 7.01 (m, 1H), 5.14 (d, $J = 7.0$ Hz, 1H), 5.04 (dd, $J = 6.9, 22.8$ Hz, 2H), 4.93 (d, $J = 6.9$ Hz, 1H), 4.09 (s, 1H), 1.22 (s, 9H).
^{13}C NMR (150 MHz, CDCl_3) δ :	163.8 (d, $J = 247.3$ Hz), 143.6, 130.5, 122.2, 115.3 (d, $J = 21.0$ Hz), 113.8 (d, $J = 22.2$ Hz), 83.7, 82.2, 62.7, 56.2, 22.4.
HRMS:	Calcd for $(\text{C}_{13}\text{H}_{18}\text{FNO}_2\text{S}+\text{H})^+$: 272.1115 Found: 272.1112



A solution of *n*-butyllithium in hexanes (1.6 M, 0.50 mL, 0.80 mmol, 1.4 equiv) was added dropwise to a solution of 4-fluoro-bromobenzene (100 mg, 0.856 mmol, 1.50 equiv) in tetrahydrofuran (5.7 mL) at -78 °C. The resulting mixture was stirred for 1.0 h at -78 °C before a solution of oxetan-3-*tert*-butylsulfinimine (**1**, 100 mg, 0.571 mmol, 1 equiv) in tetrahydrofuran (1.0 mL) was added dropwise at -78 °C. The reaction solution was stirred for an addition 10 min at -78 °C before being warmed to 22 °C and quenched with saturated aqueous ammonium chloride solution (3.0 mL). The crude reaction mixture was partitioned between water (20.0 mL) and ethyl acetate (20.0 mL). The aqueous layer was further extracted with ethyl acetate (20.0 mL), and the organic layers were combined. The combined layers were washed with saturated aqueous sodium chloride solution (20.0 mL), and the washed organic layer was dried with anhydrous sodium sulfate. The dried solution was filtered and the filtrate was concentrated, yielding a yellow oil. The crude product was purified by flash-column chromatography (50% ethyl acetate-hexanes, grading to 100 % ethyl acetate, then flushing with 10% methanol-

dichloromethane), affording the addition product **2e** (119 mg, 0.440 mmol, 77.0%) as a colorless oil that solidified over time.

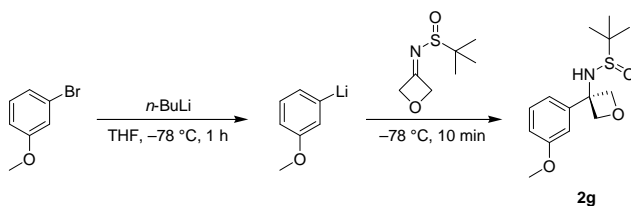
TLC (70 % ethyl acetate-hexanes):	$R_f = 0.22$ (UV)
^1H NMR (600 MHz, CDCl_3) δ :	7.40 – 7.29 (m, 2H), 7.08 (t, $J = 8.6$ Hz, 2H), 5.15 (d, $J = 6.9$ Hz, 1H), 5.02 (dd, $J = 6.9, 18.2$ Hz, 2H), 4.93 (d, $J = 6.8$ Hz, 1H), 4.18 (s, 1H), 1.19 (s, 9H).
^{13}C NMR (150 MHz, CDCl_3) δ :	163.1 (d, $J = 247.3$ Hz), 136.9, 128.4, 115.7, 115.6, 83.8, 82.4, 62.5, 56.0, 22.4.
HRMS:	Calcd for $(\text{C}_{13}\text{H}_{18}\text{FNO}_2\text{S}+\text{H})^+$: 272.1115 Found: 272.1111



A solution of *n*-butyllithium in hexanes (1.6 M, 0.50 mL, 0.80 mmol, 1.4 equiv) was added dropwise to a solution of 2-bromoanisole (160 mg, 0.856 mmol, 1.50 equiv) in tetrahydrofuran (5.7 mL) at -78 °C. The resulting mixture was stirred for 1.0 h at -78 °C before a solution of oxetan-3-*tert*-butylsulfinimine (**1**, 100 mg, 0.571 mmol, 1 equiv) in tetrahydrofuran (1.0 mL) was added dropwise at -78 °C. The reaction solution was stirred for an addition 10 min at -78 °C before being warmed to 22 °C and quenched with saturated aqueous ammonium chloride solution (3.0 mL). The crude reaction mixture was partitioned between water (10.0 mL) and ethyl acetate (20.0 mL). The aqueous layer was further extracted with ethyl acetate (20.0 mL), and the organic layers were combined. The combined layers were washed with saturated aqueous sodium chloride solution (20.0 mL), and the washed organic layer was dried with anhydrous sodium sulfate. The dried solution was filtered and the filtrate was concentrated, yielding a yellow oil. The crude product was purified by flash-column chromatography (50% ethyl acetate-hexanes, grading to 100% ethyl acetate, then flushing with 10% methanol-dichloromethane),

affording the addition product **2f** (84 mg, 0.30 mmol, 52%) as a colorless oil that solidified over time.

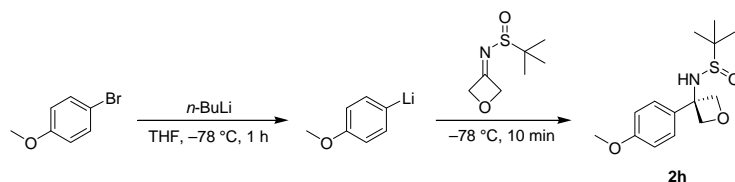
TLC (70% ethyl acetate-hexanes):	$R_f = 0.20$ (UV)
^1H NMR (600 MHz, CDCl_3) δ :	7.36 – 7.27 (m, 1H), 7.15 (dd, $J = 1.5, 7.5$ Hz, 1H), 6.99 (t, $J = 7.5$ Hz, 1H), 6.89 (d, $J = 8.2$ Hz, 1H), 5.18 (d, $J = 7.2$ Hz, 1H), 5.07 (d, $J = 7.1$ Hz, 1H), 4.88 (t, $J = 6.3$ Hz, 2H), 4.16 (s, 1H), 3.81 (s, 3H), 1.18 (s, 9H).
^{13}C NMR (150 MHz, CDCl_3) δ :	156.5, 129.7, 129.2, 127.1, 120.7, 111.0, 82.6, 81.4, 62.0, 55.8, 55.2, 22.3.
HRMS:	Calcd for $(\text{C}_{14}\text{H}_{21}\text{NO}_3\text{S}+\text{H})^+$: 284.1315 Found: 284.1312



A solution of *n*-butyllithium in hexanes (1.6 M, 0.50 mL, 0.80 mmol, 1.4 equiv) was added dropwise to a solution of 3-bromoanisole (160 mg, 0.856 mmol, 1.50 equiv) in tetrahydrofuran (5.7 mL) at $-78\text{ }^{\circ}\text{C}$. The resulting mixture was stirred for 1.0 h at $-78\text{ }^{\circ}\text{C}$ before a solution of oxetan-3-*tert*-butylsulfinimine (**1**, 100 mg, 0.571 mmol, 1 equiv) in tetrahydrofuran (1.0 mL) was added dropwise at $-78\text{ }^{\circ}\text{C}$. The reaction solution was stirred for an addition 10 min at $-78\text{ }^{\circ}\text{C}$ before being warmed to $22\text{ }^{\circ}\text{C}$ and quenched with saturated aqueous ammonium chloride solution (3.0 mL). The crude reaction mixture was partitioned between water (10.0 mL) and ethyl acetate (20.0 mL). The aqueous layer was further extracted with ethyl acetate (20.0 mL), and the organic layers were combined. The combined layers were washed with saturated aqueous sodium chloride solution (20.0 mL), and the washed organic layer was dried with anhydrous sodium sulfate. The dried solution was filtered and the filtrate was concentrated, yielding a yellow oil. The crude product was purified by flash-column chromatography (50% ethyl acetate-hexanes,

grading to 100% ethyl acetate, then flushing with 10% methanol-dichloromethane), affording the addition product **2g** (92 mg, 0.33 mmol, 57%) as a colorless oil that solidified over time.

TLC (70% ethyl acetate-hexanes):	$R_f = 0.21$ (UV)
^1H NMR (600 MHz, CDCl_3) δ :	7.33 (t, $J = 7.9$ Hz, 1H), 6.96 (d, $J = 7.6$ Hz, 1H), 6.91 – 6.83 (m, 2H), 5.17 (d, $J = 6.9$ Hz, 1H), 5.04 (d, $J = 7.2$ Hz, 2H), 4.98 (d, $J = 6.9$ Hz, 1H), 4.04 (s, 1H), 3.82 (s, 3H), 1.22 (s, 9H).
^{13}C NMR (150 MHz, CDCl_3) δ :	159.9, 142.7, 129.9, 118.7, 113.3, 112.5, 83.8, 82.4, 63.0, 56.1, 55.3, 22.5.
HRMS:	Calcd for $(\text{C}_{14}\text{H}_{21}\text{NO}_3\text{S}+\text{H})^+$: 284.1315 Found: 284.1313

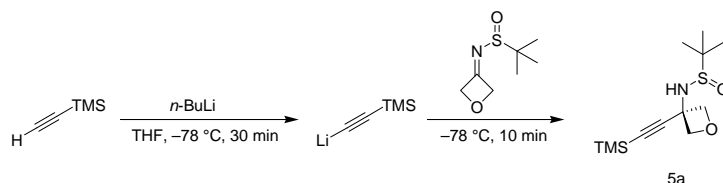


A solution of *n*-butyllithium in hexanes (1.6 M, 0.50 mL, 0.80 mmol, 1.4 equiv) was added dropwise to a solution of 4-bromoanisole (160 mg, 0.856 mmol, 1.50 equiv) in tetrahydrofuran (5.7 mL) at -78 °C. The resulting mixture was stirred for 1.0 h at -78 °C before a solution of oxetan-3-*tert*-butylsulfinimine (**1**, 100 mg, 0.571 mmol, 1 equiv) in tetrahydrofuran (1.0 mL) was added dropwise at -78 °C. The reaction solution was stirred for an addition 10 min at -78 °C before being warmed to 22 °C and quenched with saturated aqueous ammonium chloride solution (3.0 mL). The crude reaction mixture was partitioned between water (10.0 mL) and ethyl acetate (20.0 mL). The aqueous layer was further extracted with ethyl acetate (20.0 mL), and the organic layers were combined. The combined layers were washed with saturated aqueous sodium chloride solution (20.0 mL), and the washed organic layer was dried with anhydrous sodium sulfate. The dried solution was filtered and the filtrate was concentrated, yielding a yellow oil. The crude product was purified by flash-column chromatography (50% ethyl acetate-hexanes,

grading to 100% ethyl acetate, then flushing with 10% methanol-dichloromethane), affording the addition product **2h** (88.4 mg, 0.312 mmol, 54.7%) as a colorless oil that solidified over time.

TLC (70% ethyl acetate-hexanes):	$R_f = 0.20$ (UV)
^1H NMR (600 MHz, CDCl_3) δ :	7.31 – 7.26 (m, 2H), 6.97 – 6.89 (m, 2H), 5.18 (d, $J = 6.9$ Hz, 1H), 5.03 (t, $J = 12.0$ Hz, 2H), 4.98 (d, $J = 6.8$ Hz, 1H), 4.05 (s, 1H), 3.82 (s, 3H), 1.20 (s, 9H).
^{13}C NMR (150 MHz, CDCl_3) δ :	159.3, 133.1, 127.8, 114.1, 84.1, 82.7, 62.7, 55.9, 55.3, 22.5.
HRMS:	Calcd for $(\text{C}_{14}\text{H}_{21}\text{NO}_3\text{S}+\text{H})^+$: 284.1315 Found: 284.1313

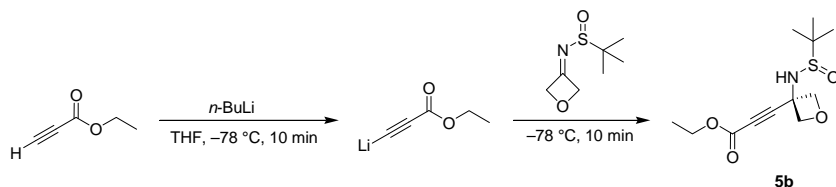
Addition of Diverse Nucleophiles into Oxetan-3-*tert*-butylsulfinimine (1).



A solution of *n*-butyllithium in hexanes (1.6 M, 0.50 mL, 0.80 mmol, 1.4 equiv) was added dropwise to a solution of trimethylsilylacetylene (84 mg, 0.86 mmol, 1.5 equiv) in tetrahydrofuran (5.7 mL) at -78 °C. The resulting mixture was stirred for 30 min at -78 °C before a solution of oxetan-3-*tert*-butylsulfinimine (**1**, 100 mg, 0.571 mmol, 1 equiv) in tetrahydrofuran (1.0 mL) was added dropwise at -78 °C. The reaction solution was stirred for an additional 10 min at -78 °C before being warmed to 22 °C, and then was quenched with saturated aqueous ammonium chloride solution (3.0 mL). The crude reaction mixture was then partitioned between water (10.0 mL) and ethyl acetate (20.0 mL). The aqueous layer was further extracted with ethyl acetate (20.0 mL) and the organic layers were combined. The combined layers were washed with saturated aqueous sodium chloride solution (20.0 mL), and the washed solution was dried with anhydrous sodium sulfate. The dried solution was filtered and the filtrate was

concentrated, yielding a yellow oil. The crude product was purified by flash-column chromatography (40% ethyl acetate-hexanes, grading to 100% ethyl acetate), affording the alkyne addition product **5a** (97 mg, 0.35 mmol, 62%) as a colorless oil that solidified over time.

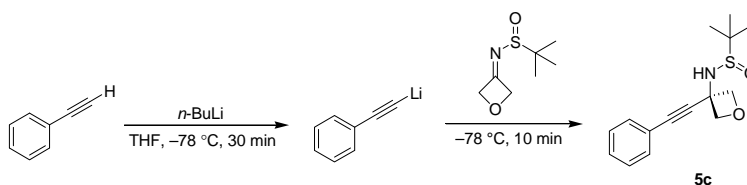
TLC (50% ethyl acetate-hexanes):	$R_f = 0.28$ (I_2)
^1H NMR (600 MHz, CDCl_3) δ :	4.95 – 4.82 (m, 2H), 4.73 (dd, $J = 6.5, 19.9$ Hz, 2H), 3.97 (s, 1H), 1.23 (s, 9H), 0.15 (s, 9H).
^{13}C NMR (150 MHz, CDCl_3) δ :	103.1, 92.6, 83.7, 83.4, 56.1, 54.0, 22.3, – 0.4.
HRMS:	Calcd for $(\text{C}_{12}\text{H}_{23}\text{NO}_2\text{SSi}+\text{H})^+$: 274.1292 Found: 274.1289



A solution of *n*-butyllithium in hexanes (1.6 M, 0.50 mL, 0.80 mmol, 1.4 equiv) was added dropwise to a solution of ethyl propiolate (84 mg, 0.86 mmol, 1.5 equiv) in tetrahydrofuran (5.7 mL) at $-78\text{ }^{\circ}\text{C}$. The resulting mixture was stirred for 10 min at $-78\text{ }^{\circ}\text{C}$ before a solution of oxetan-3-*tert*-butylsulfinimine (**1**, 100 mg, 0.571 mmol, 1 equiv) in tetrahydrofuran (1.0 mL) was added dropwise at $-78\text{ }^{\circ}\text{C}$. The reaction solution was stirred for an additional 10 min at $-78\text{ }^{\circ}\text{C}$ before being quenched at this temperature with a 1:4 acetic acid-hexanes mixture (1.0 mL). The crude reaction mixture was allowed to warm to $22\text{ }^{\circ}\text{C}$, and was then partitioned between water (10.0 mL), saturated aqueous ammonium chloride solution (5.0 mL) and ethyl acetate (20.0 mL). The aqueous layer was further extracted with ethyl acetate (20.0 mL) and the organic layers were combined. The combined layers were washed with saturated aqueous sodium chloride solution (10.0 mL), and the washed solution was dried with anhydrous sodium sulfate. The dried solution was filtered and the filtrate was concentrated, yielding a yellow oil. The crude

product was purified by flash-column chromatography (30% ethyl acetate-hexanes, grading to 100% ethyl acetate-hexanes), affording the alkyne addition product **5b** (128 mg, 0.468 mmol, 82.0%) as an amber oil.

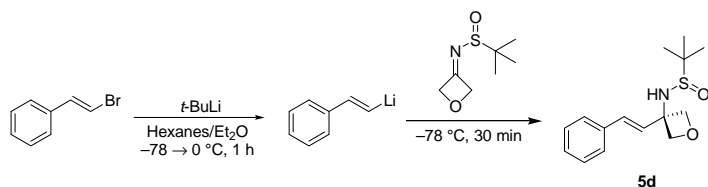
TLC (70% ethyl acetate-hexanes):	$R_f = 0.35$ (I_2)
^1H NMR (600 MHz, CDCl_3) δ :	4.95 (t, $J = 12.0$ Hz, 2H), 4.78 (dd, $J = 6.8$ Hz, 20.1, 2H), 4.25 (q, $J = 7.1$ Hz, 2H), 4.11 (s, 1H), 1.31 (t, $J = 7.1$ Hz, 3H), 1.25 (s, 9H).
^{13}C NMR (150 MHz, CDCl_3) δ :	152.9, 83.8, 82.5, 81.9, 78.6, 62.4, 56.6, 53.2, 22.3, 13.9.
HRMS:	Calcd for $(\text{C}_{12}\text{H}_{19}\text{NO}_4\text{S}+\text{H})^+$: 274.1108 Found: 274.1110



A solution of *n*-butyllithium in hexanes (2.5 M, 0.10 mL, 0.26 mmol, 1.4 equiv) was added dropwise to a solution of phenylacetylene (26.2 mg, 0.257 mmol, 1.5 equiv) in tetrahydrofuran (2.0 mL) at -78 °C. The resulting mixture was stirred for 30 min at -78 °C before a solution of oxetan-3-*tert*-butylsulfinimine (**1**, 30.0 mg, 0.171 mmol, 1 equiv) in tetrahydrofuran (1.0 mL) was added dropwise at -78 °C. The reaction solution was stirred for an additional 10 min at -78 °C before being warmed to 22 °C, and was then quenched with saturated aqueous ammonium chloride solution (3.0 mL). The crude reaction mixture was then partitioned between water (5.0 mL) and ethyl acetate (10.0 mL). The aqueous layer was further extracted with ethyl acetate (10.0 mL) and the organic layers were combined. The combined layers were washed with saturated aqueous sodium chloride solution (5.0 mL), and the washed solution was dried with anhydrous sodium sulfate. The dried solution was filtered and the filtrate was concentrated, yielding a yellow oil. The crude product was purified by flash-column chromatography (50%

ethyl acetate in hexanes, grading to 100% ethyl acetate), affording the phenyl acetylene addition product **5c** (38.2 mg, 0.138 mmol, 80.0%) as a colorless oil that solidified over time.

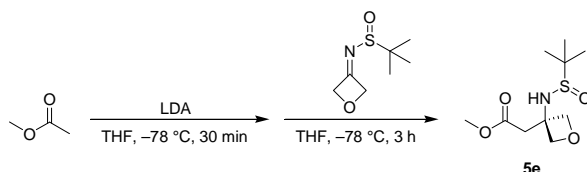
TLC (70% ethyl acetate-hexanes):	$R_f = 0.52$ (UV)
^1H NMR (600 MHz, CDCl_3) δ :	7.46 (d, $J = 6.6$ Hz, 2H), 7.37 – 7.28 (m, 3H), 5.01 (d, $J = 5.7$ Hz, 2H), 4.83 (dd, $J = 6.5, 20.8$ Hz, 2H), 4.06 (s, 1H), 1.28 (s, 9H).
^{13}C NMR (150 MHz, CDCl_3) δ :	131.7, 128.8, 128.3, 121.9, 87.6, 87.0, 84.0, 83.5, 56.2, 54.1, 22.4.
HRMS:	Calcd for $(\text{C}_{15}\text{H}_{19}\text{NO}_2\text{S}+\text{H})^+$: 278.1209 Found: 278.1207



A solution of *tert*-butyllithium in pentane (1.7 M, 0.42 mL, 0.714 mmol, 2.5 equiv) was added dropwise to a solution of β -bromostyrene (78 mg, 0.43 mmol, 1.5 equiv) in hexanes (1.5 mL) and diethyl ether (1.0 mL) at -78 °C. The reaction mixture was warmed to 22 °C and stirred for 1 h at this temperature. The resulting solution was then cooled to -78 °C before a solution of oxetan-3-*tert*-butylsulfinimine (**1**, 50 mg, 0.29 mmol, 1 equiv) in hexanes (0.6 mL) and diethyl ether (0.4 mL) was added dropwise at -78 °C. The reaction solution was stirred for an additional 30 min at -78 °C before being warmed to 22 °C, and then was quenched with saturated aqueous ammonium chloride solution (3.0 mL). The crude reaction mixture was then partitioned between water (5.0 mL) and ethyl acetate (10.0 mL). The aqueous layer was further extracted with ethyl acetate (10.0 mL) and the organic layers were combined. The combined layers were washed with saturated aqueous sodium chloride solution (5.0 mL), and the washed solution was dried with anhydrous sodium sulfate. The dried solution was filtered and the filtrate was concentrated, yielding a yellow oil. The crude product was purified by flash-

column chromatography (40% ethyl acetate-hexanes, grading to 100% ethyl acetate), affording the alkene addition product **5d** (53 mg, 0.19 mmol, 66%) as a colorless oil.

TLC (70% ethyl acetate-hexanes):	$R_f = 0.35$ (UV)
^1H NMR (600 MHz, CDCl_3) δ :	7.43 (d, $J = 7.5$ Hz, 2H), 7.35 (t, $J = 7.5$ Hz, 2H), 7.28 (t, $J = 7.2$ Hz, 1H), 6.80 (d, $J = 16.2$ Hz, 1H), 6.46 (d, $J = 16.2$ Hz, 1H), 4.98 – 4.82 (m, 3H), 4.77 (d, $J = 6.7$ Hz, 1H), 3.89 (s, 1H), 1.28 (s, 9H).
^{13}C NMR (150 MHz, CDCl_3) δ :	136.2, 132.1, 129.5, 128.9, 128.5, 126.9, 83.2, 82.3, 61.3, 56.4, 22.8.
HRMS:	Calcd for $(\text{C}_{15}\text{H}_{21}\text{NO}_2\text{S}+\text{H})^+$: 280.1366 Found: 280.1364



A solution of *n*-butyllithium in hexanes (1.6 M, 0.36 mL, 0.57 mmol, 2.0 equiv) was added to a solution of *N,N*-diisopropylamine (0.085 mL, 0.60 mmol, 2.1 equiv) in tetrahydrofuran (1.0 mL) at 0 °C. The resulting mixture was stirred at 0 °C for 30 min, then was cooled to –78 °C. A solution of methyl acetate (0.050 mL, 0.63 mmol, 2.2 equiv) in tetrahydrofuran (1.0 mL) was added dropwise at –78 °C, and the reaction mixture was stirred at –78 °C for an additional 30 min. A solution of oxetan-3-*tert*-butylsulfinimine (**1**, 50 mg, 0.29 mmol, 1 equiv) in tetrahydrofuran (1.0 mL) was added dropwise at –78 °C, and the resulting mixture was stirred at –78 °C for 3 h. After this time, the reaction was warmed to 22 °C and quenched with saturated aqueous ammonium chloride solution (3.0 mL). The crude reaction mixture was then partitioned between water (5.0 mL) and ethyl acetate (10.0 mL), and the aqueous layer was further extracted with ethyl acetate (10.0 mL). The organic layers were combined and washed with saturated aqueous sodium chloride solution (5.0 mL), and the washed solution was dried with anhydrous sodium sulfate. The dried solution was filtered and the filtrate was

concentrated, yielding a yellow oil. The crude product was purified by flash-column chromatography (100% hexanes, grading to 100% ethyl acetate, then flushing with 10% methanol-dichloromethane), affording the enolate addition product **5e** (65.0 mg, 0.26 mmol, 91 %) as a yellow oil.

TLC (70% ethyl acetate-hexanes):

$R_f = 0.21$ (I_2)

^1H NMR (600 MHz, CDCl_3) δ :

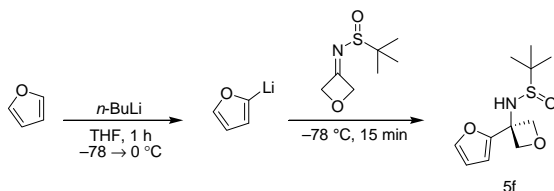
4.81 (d, $J = 6.9$ Hz, 1H), 4.74 (d, $J = 6.7$ Hz, 1H), 4.54 (d, $J = 6.8$ Hz, 1H), 4.51 – 4.39 (m, 2H), 3.69 (s, 3H), 3.17 (q, $J = 17.0$ Hz, 2H), 1.22 (s, 9H).

^{13}C NMR (150 MHz, CDCl_3) δ :

171.4, 82.0, 80.2, 57.3, 56.1, 51.9, 42.2, 22.4.

HRMS:

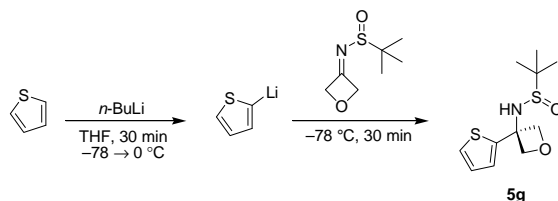
Calcd for $(\text{C}_{10}\text{H}_{19}\text{NO}_4\text{S}+\text{H})^+$: 250.1108
Found: 250.1105



A solution of *n*-butyllithium in hexanes (1.6 M, 0.50 mL, 0.80 mmol, 1.4 equiv) was added dropwise to a solution of furan (58.3 mg, 0.856 mmol, 1.50 equiv) in tetrahydrofuran (5.7 mL) at -78 °C. The reaction mixture was warmed to 0 °C and stirred for 1 h at this temperature. The resulting solution was then cooled to -78 °C before a solution of oxetan-3-*tert*-butylsulfinimine (**1**, 100 mg, 0.571 mmol, 1 equiv) in tetrahydrofuran (1.0 mL) was added dropwise at -78 °C. The reaction solution was stirred for an additional 15 min at -78 °C before being warmed to 22 °C, and then was quenched with saturated aqueous ammonium chloride solution (3.0 mL). The crude reaction mixture was then partitioned between water (10.0 mL) and ethyl acetate (20.0 mL). The aqueous layer was further extracted with ethyl acetate (20.0 mL) and the organic layers were combined. The combined layers were washed with saturated aqueous sodium chloride solution (10.0 mL), and the washed solution was dried with anhydrous sodium sulfate. The dried solution was filtered and the filtrate was concentrated, yielding

a yellow oil. The crude product was purified by flash-column chromatography (40% ethyl acetate-hexanes, grading to 100% ethyl acetate, then flushing with 10% methanol-dichloromethane), affording the furan addition product **5f** (106 mg, 0.434 mmol, 76.0%) as a colorless oil that solidified over time.

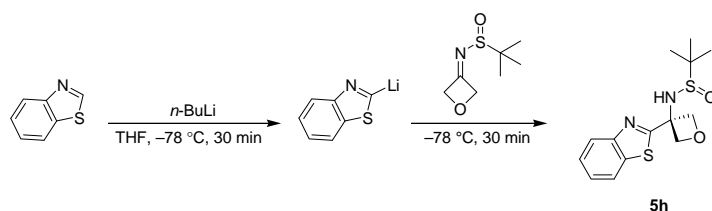
TLC (70% ethyl acetate-hexanes):	$R_f = 0.35$ (I_2)
^1H NMR (600 MHz, CDCl_3) δ :	7.43 (s, 1H), 6.47 (d, $J = 3.1$ Hz, 1H), 6.39 (dd, $J = 1.8, 3.3$ Hz, 1H), 5.03 (d, $J = 6.7$ Hz, 1H), 4.95 – 4.89 (m, 3H), 4.05 (s, 1H), 1.24 (s, 9H).
^{13}C NMR (150 MHz, CDCl_3) δ :	153.1, 142.9, 110.5, 108.3, 81.6, 81.5, 58.7, 56.2, 22.4.
HRMS:	Calcd for $(\text{C}_{11}\text{H}_{17}\text{NO}_3\text{S}+\text{H})^+$: 244.1002 Found: 244.0997



A solution of *n*-butyllithium in hexanes (1.6 M, 0.50 mL, 0.80 mmol, 1.4 equiv) was added dropwise to a solution of thiophene (72.0 mg, 0.856 mmol, 1.50 equiv) in tetrahydrofuran (5.7 mL) at -78 °C. The reaction mixture was warmed to 0 °C and stirred for 30 min at this temperature. The resulting solution was then cooled to -78 °C before a solution of oxetan-3-*tert*-butylsulfinimine (**1**, 100 mg, 0.571 mmol, 1 equiv) in tetrahydrofuran (1.0 mL) was added dropwise at -78 °C. The reaction solution was stirred for an additional 30 min at -78 °C before being warmed to 22 °C, and then was quenched with saturated aqueous ammonium chloride solution (3.0 mL). The crude reaction mixture was then partitioned between water (10.0 mL) and ethyl acetate (20.0 mL). The aqueous layer was further extracted with ethyl acetate (20.0 mL) and the organic layers were combined. The combined layers were washed with saturated aqueous sodium chloride solution (10.0 mL), and the washed solution was dried with anhydrous

sodium sulfate. The dried solution was filtered and the filtrate was concentrated, yielding a yellow oil. The crude product was purified by flash-column chromatography (40% ethyl acetate-hexanes, grading to 100% ethyl acetate), affording the thiophene addition product **5g** (122 mg, 0.470 mmol, 82.0%) as a colorless oil that solidified over time.

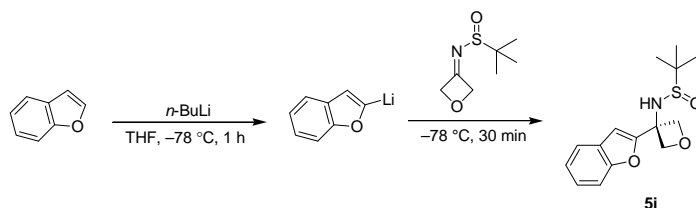
TLC (70% ethyl acetate-hexanes):	$R_f = 0.35$ (UV)
^1H NMR (600 MHz, CDCl_3) δ :	7.31 (d, $J = 5.0$ Hz, 1H), 7.20 (d, $J = 3.5$ Hz, 1H), 7.01 (dd, $J = 3.7, 5.0$ Hz, 1H), 5.12 (d, $J = 6.9$ Hz, 1H), 5.05 (d, $J = 6.9$ Hz, 1H), 5.00 (d, $J = 6.8$ Hz, 1H), 4.85 (d, $J = 6.8$ Hz, 1H), 4.22 (s, 1H), 1.24 (s, 9H).
^{13}C NMR (150 MHz, CDCl_3) δ :	145.2, 127.1, 126.1, 125.6, 84.7, 83.0, 60.8, 56.4, 22.5.
HRMS:	Calcd for $(\text{C}_{11}\text{H}_{17}\text{NO}_2\text{S}_2+\text{H})^+$: 260.0773 Found: 260.0771



A solution of *n*-butyllithium in hexanes (1.6 M, 0.50 mL, 0.80 mmol, 1.4 equiv) was added dropwise to a solution of benzothiazole (116 mg, 0.856 mmol, 1.50 equiv) in tetrahydrofuran (5.7 mL) at -78 °C. The resulting mixture was stirred for 30 min at -78 °C before a solution of oxetan-3-*tert*-butylsulfinimine (**1**, 100 mg, 0.571 mmol, 1 equiv) in tetrahydrofuran (1.0 mL) was added dropwise at -78 °C. The reaction solution was stirred for an additional 30 min at -78 °C before being warmed to 22 °C, and then was quenched with saturated aqueous ammonium chloride solution (3.0 mL). The crude reaction mixture was then partitioned between water (10.0 mL) and ethyl acetate (20.0 mL). The aqueous layer was further extracted with ethyl acetate (20.0 mL) and the organic layers were combined. The combined layers were washed with saturated aqueous sodium chloride solution (10.0 mL), and the washed solution was dried with anhydrous

sodium sulfate. The dried solution was filtered and the filtrate was concentrated, yielding a yellow oil. The crude product was purified by flash-column chromatography (40% ethyl acetate-hexanes, grading to 100% ethyl acetate, then flushing with 10% methanol-dichloromethane), affording the benzothiazole addition product **5h** (173 mg, 0.557 mmol, 98.0 %) as a colorless oil that solidified over time.

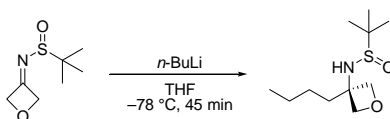
TLC (70% ethyl acetate-hexanes):	$R_f = 0.32$ (UV)
^1H NMR (600 MHz, CDCl_3) δ :	8.06 (d, $J = 8.2$ Hz, 1H), 7.89 (d, $J = 8.0$ Hz, 1H), 7.54 – 7.46 (m, 1H), 7.45 – 7.38 (m, 1H), 5.31 (d, $J = 6.8$ Hz, 1H), 5.20 (dd, $J = 6.9, 20.1$ Hz, 2H), 5.02 (d, $J = 7.0$ Hz, 1H), 4.82 (s, 1H), 1.32 (s, 9H).
^{13}C NMR (150 MHz, CDCl_3) δ :	171.3, 152.6, 135.5, 126.4, 125.7, 123.6, 121.8, 83.1, 81.6, 62.6, 57.0, 22.6.
HRMS:	Calcd for $(\text{C}_{14}\text{H}_{18}\text{N}_2\text{O}_2\text{S}_2+\text{H})^+$: 311.0882 Found: 311.0882



A solution of *n*-butyllithium in hexanes (1.6 M, 0.30 mL, 0.48 mmol, 1.4 equiv) was added dropwise to a solution of benzofuran (60.7 mg, 0.514 mmol, 1.50 equiv) in tetrahydrofuran (3.4 mL) at -78 °C. The resulting mixture was stirred for 1 h at -78 °C before a solution of oxetan-3-*tert*-butylsulfinimine (**1**, 60 mg, 0.34 mmol, 1 equiv) in tetrahydrofuran (1.0 mL) was added dropwise at -78 °C. The reaction solution was stirred for an additional 30 min at -78 °C before being warmed to 22 °C, and was then quenched with saturated aqueous ammonium chloride solution (3.0 mL). The crude reaction mixture was then partitioned between water (5.0 mL) and ethyl acetate (10.0 mL). The aqueous layer was further extracted with ethyl acetate (10.0 mL) and the organic layers were combined. The combined layers were washed with saturated aqueous sodium chloride solution (10.0 mL), and the washed solution was dried with anhydrous

sodium sulfate. The dried solution was filtered and the filtrate was concentrated, yielding a yellow oil. The crude product was purified by flash-column chromatography (40% ethyl acetate-hexanes, grading to 100% ethyl acetate), affording the benzofuran addition product **5i** (78 mg, 0.27 mmol, 78%) as a colorless oil that solidified over time.

TLC (70% ethyl acetate-hexanes):	$R_f = 0.32$ (UV)
^1H NMR (600 MHz, CDCl_3) δ :	7.57 (d, $J = 7.6$ Hz, 1H), 7.48 (d, $J = 8.2$ Hz, 1H), 7.35 – 7.28 (m, 1H), 7.27 – 7.21 (m, 1H), 6.91 (s, 1H), 5.16 (d, $J = 6.8$ Hz, 1H), 5.06 – 4.97 (m, 3H), 4.15 (s, 1H), 1.28 (s, 9H).
^{13}C NMR (150 MHz, CDCl_3) δ :	155.6, 155.2, 127.8, 124.8, 123.1, 121.4, 111.3, 105.3, 81.3, 81.2, 59.1, 56.5, 22.4.
HRMS:	Calcd for $(\text{C}_{15}\text{H}_{19}\text{NO}_3\text{S}+\text{H})^+$: 294.1158 Found: 294.1160

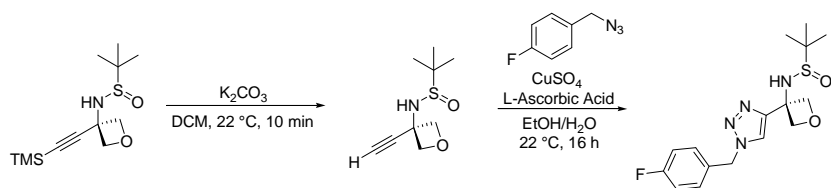


A solution of *n*-butyllithium in hexanes (1.6 M, 0.357 mL, 0.571 mmol, 2.0 equiv) was added dropwise to a solution of oxetan-3-*tert*-butylsulfinimine (**1**, 50.0 mg, 0.285 mmol, 1 equiv) in tetrahydrofuran at -78 °C. The reaction was stirred for 45 min at -78 °C before being quenched at this temperature with a 4:1 tetrahydrofuran-acetic acid mixture (1.0 mL). The quenched reaction was warmed to 22 °C and partitioned between water (5.0 mL) and ethyl acetate (10.0 mL). The aqueous layer was further extracted with ethyl acetate (10.0 mL), and the organic layers were combined. The combined layers were washed with saturated aqueous sodium chloride solution (5.0 mL), and the washed solution was dried with anhydrous sodium sulfate. The dried solution was filtered and the filtrate was concentrated, yielding a yellow oil. The crude product was purified by flash-column chromatography (50% ethyl acetate-hexanes grading to 100% ethyl acetate) to afford the butyl addition product (12.5 mg, 0.054 mmol, 18.8%) as a colorless oil.

Percent yield was determined from ~90% pure sample, while an analytically pure sample for NMR analysis was attained after two additional purifications.

TLC (70% ethyl acetate-hexanes):	$R_f = 0.20$ (I_2)
^1H NMR (600 MHz, CDCl_3) δ :	4.72 (dd, $J = 3.1, 6.6$ Hz, 2H), 4.49 (dd, $J = 6.6, 33.3$ Hz, 2H), 3.55 (s, 1H), 2.13 – 1.88 (m, 2H), 1.46 – 1.28 (m, 4H), 1.24 (s, 9H), 0.93 (t, $J = 7.1$ Hz, 3H).
^{13}C NMR (150 MHz, CDCl_3) δ :	82.7, 82.5, 60.1, 56.1, 37.1, 25.7, 22.9, 22.7, 14.2.
HRMS:	Calcd for $(\text{C}_{11}\text{H}_{23}\text{NO}_2\text{S}+\text{H})^+$: 234.1522 Found: 234.1518

Desilylation of TMS-Alkyne 5a and [2+3] Cyclization of the Deprotected Alkyne.

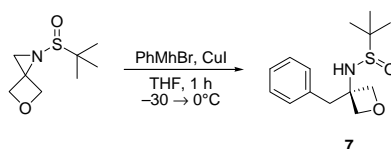


A saturated solution of potassium carbonate in methanol (1.0 mL) was added to a solution of the alkyne addition product **5a** (47.9 mg, 0.175 mmol) in dichloromethane (1.7 mL). The resulting mixture was stirred for 10 minutes before it was partitioned between water (10.0 mL) and dichloromethane (10.0 mL). The aqueous layer was further extracted with ethyl acetate (10.0 mL). The organic layers were combined and washed with saturated aqueous sodium chloride solution (10.0 mL), and then dried with anhydrous sodium sulfate. The dried solution was filtered and evaporated to dryness under reduced pressure, yielding the deprotected alkyne (33.0 mg, 0.164 mmol, 94.0%) as a colorless oil. The crude product was carried forward without purification. L-ascorbic acid (16.24 mg, 0.082 mmol, 0.50 equiv), 4-fluorobenzylazide (49.6 mg, 0.328 mmol, 2.00 equiv), and copper (II) sulfate (10.47 mg, 0.066 mmol, 0.400 equiv) were added sequentially to a solution of the deprotected alkyne in water (0.82 mL) and ethanol (0.82

mL). The mixture was stirred for 16 h at 22 °C before the ethanol was removed by rotary evaporation. The solution was then partitioned between water (10.0 mL) and ethyl acetate (10.0 mL), and the aqueous layer was further extracted with ethyl acetate (10.0 mL). The organic layers were combined and washed with saturated aqueous sodium chloride solution (5.0 mL), and then dried with anhydrous sodium sulfate. The dried solution was filtered and the filtrate was concentrated, yielding a yellow oil. The crude product was purified by flash-column chromatography (50% ethyl acetate-hexanes, grading to 100% ethyl acetate, then flushing with 10% methanol-dichloromethane) to afford the triazole (55.8 mg, 0.158 mmol, 90% over two steps from the alkyne addition product **5a**) as a colorless oil that solidified over time.

TLC (70% ethyl acetate-hexanes):	$R_f = 0.25$ (UV)
^1H NMR (600 MHz, CDCl_3) δ :	7.74 (s, 1H), 7.33 – 7.27 (m, 2H), 7.07 (t, $J = 8.6$ Hz, 2H), 5.50 (q, $J = 14.9$ Hz, 2H), 5.08 (d, $J = 6.5$ Hz, 2H), 4.90 (dd, $J = 6.5$ Hz, 14.4, 2H), 4.31 (s, 1H), 1.27 (s, 9H).
^{13}C NMR (150 MHz, CDCl_3) δ :	163.6 (d, $J = 248.4$ Hz), 149.8, 130.2, 130.0, 121.8, 116.2, 116.0, 82.9, 82.2, 57.6, 56.4, 53.5, 22.4.
HRMS:	Calcd for $(\text{C}_{16}\text{H}_{21}\text{FN}_4\text{O}_2\text{S}+\text{H})^+$: 353.1442 Found: 353.1446

Ring-opening Reactions of the Sulfinyl Aziridine **6**.



A solution of phenylmagnesium bromide (3.0 M, 0.26 mL, 0.79 mmol, 3.0 equiv) in diethyl ether and copper (I) iodide (5.03 mg, 0.026 mmol, 0.10 equiv) were added sequentially to tetrahydrofuran (2.6 mL). The mixture was cooled to -30 °C, and a solution of the sulfinyl aziridine **6** (50 mg, 0.26 mmol, 1 equiv) was added dropwise. The

resulting mixture was stirred at $-30\text{ }^{\circ}\text{C}$ for 10 min before being warmed to $0\text{ }^{\circ}\text{C}$ and stirred for 1 h. The reaction was quenched with saturated aqueous sodium chloride solution (2.0 mL) and let warm to $22\text{ }^{\circ}\text{C}$. The crude mixture was then partitioned between water (5.0 mL) and ethyl acetate (10.0 mL), and the aqueous layer was further extracted with ethyl acetate (10.0 mL). The organic layers were combined and washed with saturated aqueous sodium chloride solution (5.0 mL), and then dried with anhydrous sodium sulfate. The dried solution was filtered and the filtrate was concentrated, yielding a yellow oil. The crude product was purified by flash-column chromatography (100% dichloromethane, grading to 5% methanol-dichloromethane), affording the benzyl addition product **7** (68.9 mg, 0.258 mmol, 98.0%) as a colorless oil.

TLC (70% ethyl acetate-hexanes):

$R_f = 0.27$ (I_2)

^1H NMR (600 MHz, CDCl_3) δ :

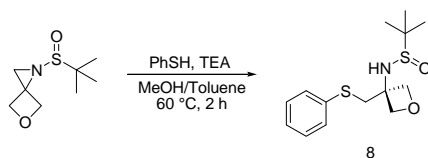
7.40 – 7.19 (m, 5H), 4.91 (d, $J = 6.7\text{ Hz}$, 1H), 4.68 (d, $J = 6.6\text{ Hz}$, 1H), 4.63 (s, 2H), 3.67 (s, 1H), 3.40 – 3.27 (m, 2H), 1.18 (s, 9H).

^{13}C NMR (150 MHz, CDCl_3) δ :

135.4, 130.1, 128.6, 127.1, 81.2, 81.0, 59.7, 56.2, 43.8, 22.4.

HRMS:

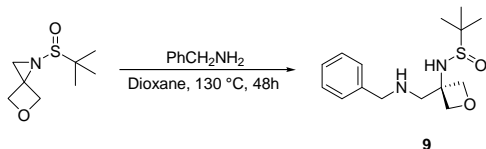
Calcd for $(\text{C}_{14}\text{H}_{21}\text{NO}_2\text{S}+\text{H})^+$: 268.1366
Found: 268.1362



A solution of thiophenol (0.0400 mL, 43.7 mg, 0.396 mmol, 1.50 equiv) in toluene (2.0 mL) and triethylamine (0.110 mL, 80.0 mg, 0.789 mmol, 3.00 equiv) were added sequentially to a solution of the sulfinyl aziridine **6** (50 mg, 0.26 mmol, 1 equiv) in methanol (2.0 mL). The resulting mixture was stirred at $60\text{ }^{\circ}\text{C}$ for 2 h before being cooled to $22\text{ }^{\circ}\text{C}$ and quenched with water (3.0 mL). The crude mixture was then partitioned between water (5.0 mL) and ethyl acetate (10.0 mL), and the aqueous layer was further extracted with ethyl acetate (10.0 mL). The organic layers were combined

and washed with saturated aqueous sodium chloride solution (5.0 mL), and then dried with anhydrous sodium sulfate. The dried solution was filtered and the filtrate was concentrated, yielding a colorless oil. The crude product was purified by flash-column chromatography (100% dichloromethane, grading to 5% methanol-dichloromethane), affording the thiol addition product **8** (76.2 mg, 0.254 mmol, 96.0%) as a colorless oil that solidified over time.

TLC (70% ethyl acetate-hexanes):	$R_f = 0.26$ (UV)
^1H NMR (600 MHz, CDCl_3) δ :	7.44 (d, $J = 7.5$ Hz, 2H), 7.30 (t, $J = 7.6$ Hz, 2H), 7.23 (t, $J = 7.4$ Hz, 1H), 4.85 (d, $J = 7.1$ Hz, 1H), 4.74 (d, $J = 6.8$ Hz, 1H), 4.44 (dd, $J = 6.9, 19.6$ Hz, 2H), 4.07 (s, 1H), 3.60 (s, 2H), 1.20 (s, 9H).
^{13}C NMR (150 MHz, CDCl_3) δ :	134.9, 130.8, 129.3, 127.2, 81.1, 81.0, 59.9, 56.2, 22.4.
HRMS:	Calcd for $(\text{C}_{14}\text{H}_{21}\text{NO}_2\text{S}_2+\text{H})^+$: 300.1086 Found: 300.1085



Benzylamine (0.346 mL, 340 mg, 3.17 mmol, 12.0 equiv) was added to the sulfinyl aziridine **6** (50 mg, 0.26 mmol, 1 equiv) in dioxane (2.6 mL). The resulting mixture was stirred at 130 °C for 48 h, and the cooled to 22 °C. The crude reaction was partitioned between water (5.0 mL), saturated aqueous ammonium chloride solution (5.0 mL), and ethyl acetate (10.0 mL), and the aqueous layer was further extracted with ethyl acetate (10.0 mL). The organic layers were combined and washed with saturated aqueous sodium chloride solution (5.0 mL), and then dried with anhydrous sodium sulfate. The dried solution was filtered and the filtrate was concentrated, yielding a colorless oil. The crude product was purified by flash-column chromatography (50% ethyl acetate-hexanes,

grading to 100% ethyl acetate), affording the benzylamine addition product **9** (55 mg, 0.19 mmol, 70%) as a colorless oil.

TLC (70% ethyl acetate-hexanes):

$R_f = 0.20$ (I_2)

^1H NMR (600 MHz, CDCl_3) δ :

7.38 – 7.29 (m, 4H), 7.26 (t, $J = 6.8$ Hz, 1H), 4.83 (d, $J = 6.8$ Hz, 1H), 4.65 (d, $J = 6.5$ Hz, 1H), 4.48 (d, $J = 6.7$ Hz, 1H), 4.41 – 4.30 (m, 2H), 3.84 (dd, $J = 13.4, 43.5$ Hz, 2H), 3.14 (dd, $J = 12.5, 130.6$ Hz, 2H), 2.10 (s, 1H), 1.25 (s, 9H).

^{13}C NMR (150 MHz, CDCl_3) δ :

139.5, 128.5, 128.1, 127.3, 81.1, 80.1, 59.0, 56.0, 54.9, 53.8, 22.5.

HRMS:

Calcd for $(\text{C}_{15}\text{H}_{24}\text{N}_2\text{O}_2\text{S}+\text{H})^+$: 297.1631
Found: 297.1627

Representative Deprotections of the *tert*-Butylsulfinyl Group.



A 4.0 M solution of hydrochloric acid (0.139 mL, 0.557 mmol, 1.50 equiv) in dioxane was added to a solution of the phenyl addition product **2a** (94 mg, 0.37 mmol, 1 equiv) in methanol (0.4 mL) at 0 °C. The mixture was stirred at 0 °C for 1 min before the solvents were removed under reduced pressure. The resulting white solid was triturated with diethyl ether (2.0 mL) and the mother liquor was removed using a Pasteur pipette. The solid was further washed with diethyl ether (2.0 mL) and dried under high vacuum to afford the amine hydrochloride salt (62.5 mg, 0.337 mmol, 91.0%) as a white solid.

^1H NMR (600 MHz, CD_3OD) δ :

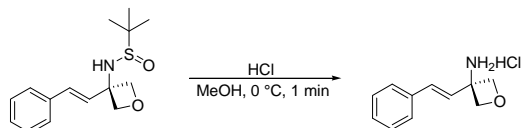
7.65 – 7.58 (m, 2H), 7.59 – 7.52 (m, 3H), 5.09 (dd, $J = 19.2, 38.5$ Hz, 4H).

^{13}C NMR (150 MHz, CD_3OD) δ :

137.8, 130.5, 126.8, 80.6.

HRMS:

Calcd for $(\text{C}_9\text{H}_{11}\text{NO}+\text{H})^+$: 150.0913
Found: 150.0905

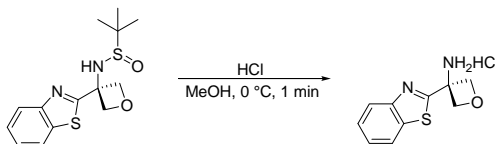


A 4.0 M solution of hydrochloric acid (0.10 mL, 0.37 mmol, 1.5 equiv) in dioxane was added to a solution of the vinyl addition product **5d** (69 mg, 0.25 mmol, 1 equiv) in methanol (0.5 mL) at 0 °C. The mixture was stirred at 0 °C for 1 min before the solvents were removed under reduced pressure. The resulting white solid was triturated with diethyl ether (2.0 mL) and the mother liquor was removed using a Pasteur pipette. The solid was further washed with diethyl ether (2.0 mL) and dried under high vacuum to afford the amine hydrochloride salt (50.0 mg, 0.236 mmol, 96.0%) as a white solid.

^1H NMR (600 MHz, CD_3OD) δ : 7.53 (d, $J = 7.4$ Hz, 2H), 7.38 (t, $J = 7.5$ Hz, 2H), 7.33 (t, $J = 7.3$ Hz, 1H), 6.85 (d, $J = 16.4$ Hz, 1H), 6.57 (d, $J = 16.4$ Hz, 1H), 4.93 (d, $J = 7.8$ Hz, 2H), 4.78 (d, $J = 7.8$ Hz, 2H).

^{13}C NMR (150 MHz, CD_3OD) δ : 136.6, 134.5, 130.0, 129.9, 127.9, 124.8, 79.7, 58.9.

HRMS: Calcd for $(\text{C}_{11}\text{H}_{13}\text{NO}+\text{H})^+$: 176.1070
Found: 176.1064



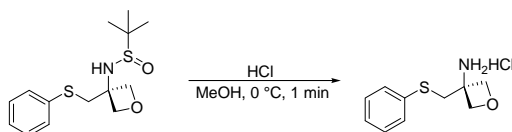
A 4.0 M solution of hydrochloric acid (0.266 mL, 1.06 mmol, 1.5 equiv) in dioxane was added to a solution of the **benzothiazole addition product #** (166 mg, 0.531 mmol, 1 equiv) in methanol (1.0 mL) at 0 °C. The mixture was stirred at 0 °C for 1 min before the solvents were removed under reduced pressure. The resulting white solid was triturated with diethyl ether (2.0 mL) and the mother liquor was removed using a Pasteur

pipette. The solid was further washed with diethyl ether (2.0 mL) and dried under high vacuum to afford the amine hydrochloride salt (125 mg, 0.511 mmol, 96.0%) as a beige solid.

^1H NMR (600 MHz, CD_3OD) δ : 8.13 (dd, $J = 8.1$ Hz, 20.9, 2H), 7.68 – 7.60 (m, 1H), 7.60 – 7.54 (m, 1H), 5.13 (s, 4H).

^{13}C NMR (150 MHz, CD_3OD) δ : 167.5, 153.5, 136.7, 128.2, 127.7, 124.6, 123.5, 80.8, 60.0.

HRMS: Calcd for $(\text{C}_{10}\text{H}_{10}\text{N}_2\text{OS}+\text{H})^+$: 207.0587
Found: 207.0581

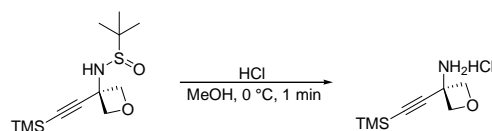


A 4.0 M solution of hydrochloric acid (0.065 mL, 0.26 mmol, 1.5 equiv) in dioxane was added to a solution of the thiol addition product **8** (52 mg, 0.17 mmol, 1 equiv) in methanol (0.4 mL) at 0 °C. The mixture was stirred at 0 °C for 1 min before the solvents were removed under reduced pressure. The resulting white solid was triturated with diethyl ether (2.0 mL) and the mother liquor was removed using a Pasteur pipette. The solid was further washed with diethyl ether (2.0 mL) and dried under high vacuum to afford the amine hydrochloride salt (38 mg, 0.164 mmol, 94.0%) as a white solid.

^1H NMR (600 MHz, CD_3OD) δ : 7.60 (d, $J = 7.6$ Hz, 2H), 7.46 – 7.28 (m, 3H), 4.59 (dd, $J = 7.8, 23.0$ Hz, 4H), 3.61 (s, 2H).

^{13}C NMR (150 MHz, CD_3OD) δ : 135.2, 132.2, 130.6, 128.9, 78.3, 58.8, 39.8.

HRMS: Calcd for $(\text{C}_{10}\text{H}_{13}\text{NOS}+\text{H})^+$: 196.0791
Found: 196.0783



A 4.0 M solution of hydrochloric acid (0.064 mL, 0.12 mmol, 1.5 equiv) in dioxane was added to a solution of the acetylene addition product **5a** (33.5 mg, 0.123 mmol, 1 equiv) in methanol (0.3 mL) at 0 °C. The mixture was stirred at 0 °C for 1 min before the solvents were removed under reduced pressure. The resulting white solid was triturated with diethyl ether (2.0 mL) and the mother liquor was removed using a Pasteur pipette. The solid was further washed with diethyl ether (2.0 mL) and dried under high vacuum to afford the amine hydrochloride salt (24 mg, 0.12 mmol, 95.0%) as a white solid.

^1H NMR (600 MHz, CD_3OD) δ : (d, $J = 8.0$ Hz, 2H), 4.72 (d, $J = 8.0$ Hz, 2H), 0.23 (s, 9H).

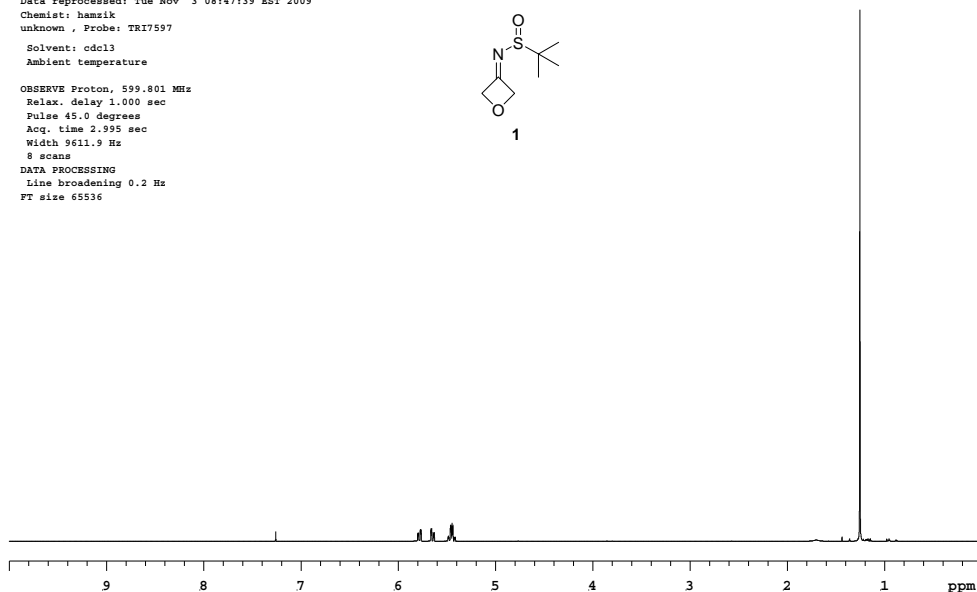
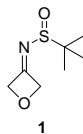
^{13}C NMR (150 MHz, CD_3OD) δ : 99.5, 96.3, 80.3, 51.0, -0.6 .

HRMS: Calcd for $(\text{C}_8\text{H}_{16}\text{ClNOSi}+\text{H})^+$: 170.0996
Found: 170.0988

1H and 13C NMR Spectra

Oxetane Imine SM

Sample name: Oxetane Imine SM
 Notebook: 0298608, Page: 0003
 Data acquired: 30-Oct-2009, 10:14:26
 Data file name: Proton01.fid
 Data reprocessed: Tue Nov 3 08:47:39 EST 2009
 Chemist: hanzik
 unknown, Probe: TRI7597
 Solvent: cdcl3
 Ambient temperature
 OBSERVE Proton, 599.801 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.995 sec
 Width 9611.9 Hz
 8 scans
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536



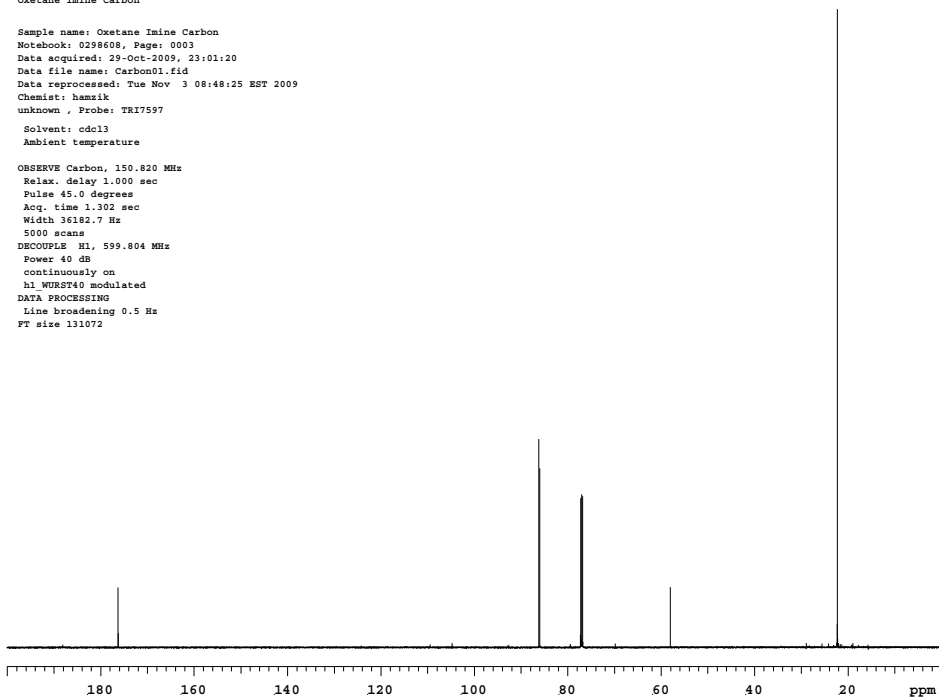
Chemist: hanzik; Notebook: 0298608; Page: 0003

Plotted: 11/03 08:47:39

Restricted Confidential, Limited Access

Oxetane Imine Carbon

Sample name: Oxetane Imine Carbon
 Notebook: 0298608, Page: 0003
 Data acquired: 29-Oct-2009, 23:01:20
 Data file name: Carbon01.fid
 Data reprocessed: Tue Nov 3 08:48:25 EST 2009
 Chemist: hanzik
 unknown, Probe: TRI7597
 Solvent: cdcl3
 Ambient temperature
 OBSERVE Carbon, 150.820 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.302 sec
 Width 36182.7 Hz
 5000 scans
 DECOUPLE H1, 599.804 MHz
 Power 40 dB
 continuously on
 h1_WURST40 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 131072



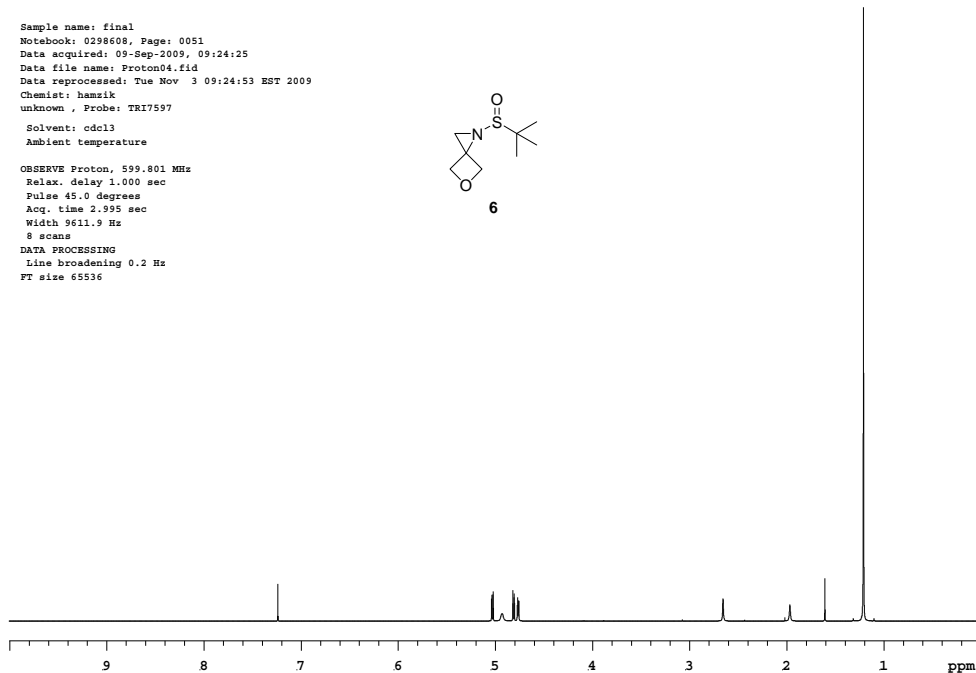
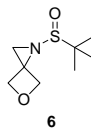
Chemist: hanzik; Notebook: 0298608; Page: 0003

Plotted: 11/03 08:48:25

Restricted Confidential, Limited Access

final

Sample name: final
 Notebook: 0298608, Page: 0051
 Data acquired: 09-Sep-2009, 09:24:25
 Data file name: Proton04.fid
 Data reprocessed: Tue Nov 3 09:24:53 EST 2009
 Chemist: hamzik
 unknown, Probe: TRI7597
 Solvent: cdcl3
 Ambient temperature
 OBSERVE Proton, 599.801 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.995 sec
 Width 9611.9 Hz
 8 scans
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536



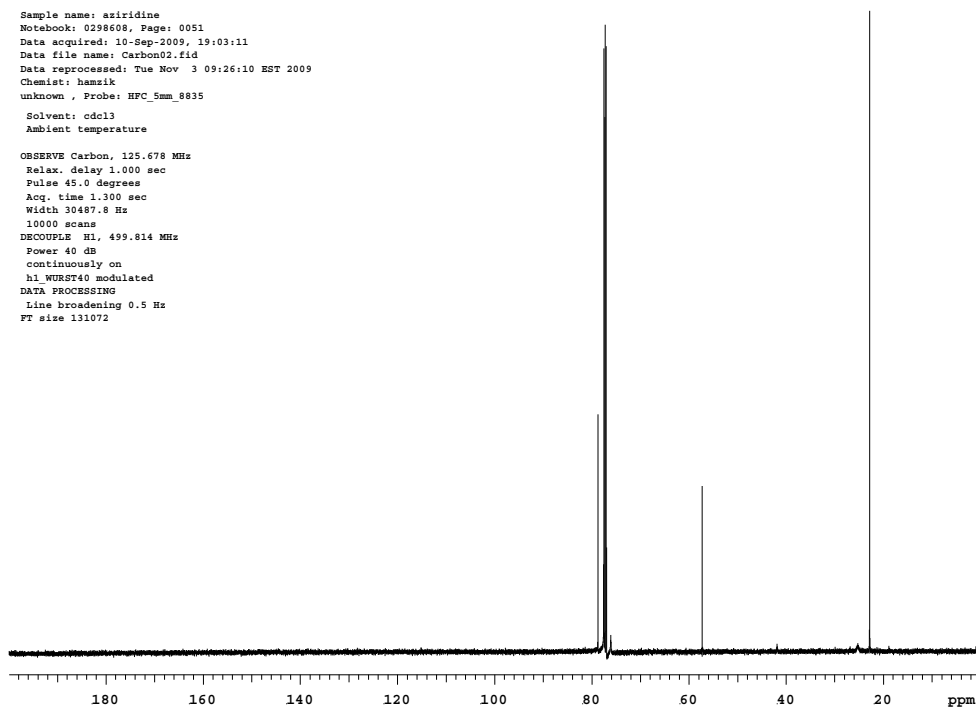
Chemist: hamzik; Notebook: 0298608; Page: 0051

Plotted: 11/03 09:24:53

Restricted Confidential, Limited Access

aziridine

Sample name: aziridine
 Notebook: 0298608, Page: 0051
 Data acquired: 10-Sep-2009, 19:03:11
 Data file name: Carbon02.fid
 Data reprocessed: Tue Nov 3 09:26:10 EST 2009
 Chemist: hamzik
 unknown, Probe: HFC_5mm_8835
 Solvent: cdcl3
 Ambient temperature
 OBSERVE Carbon, 125.678 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 30487.8 Hz
 10000 scans
 DECOUPLE H1, 499.814 MHz
 Power 40 dB
 continuously on
 h1 WURST40 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 131072



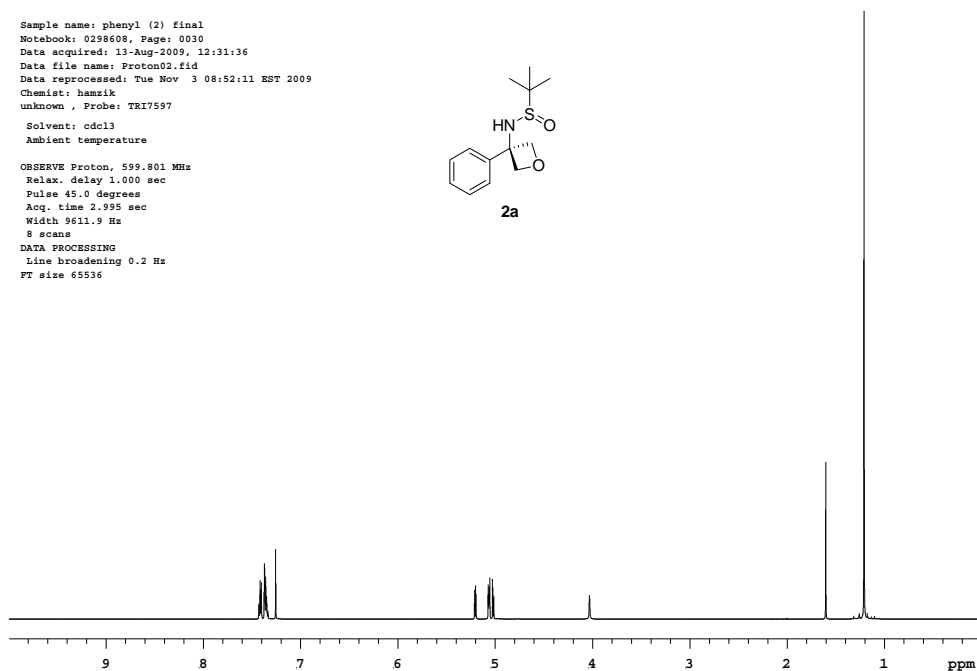
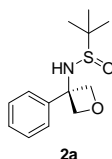
Chemist: hamzik; Notebook: 0298608; Page: 0051

Plotted: 11/03 09:26:10

Restricted Confidential, Limited Access

phenyl (2) final

Sample name: phenyl (2) final
 Notebook: 0298608, Page: 0030
 Data acquired: 13-Aug-2009, 12:31:36
 Data file name: Proton02.fid
 Data reprocessed: Tue Nov 3 08:52:11 EST 2009
 Chemist: hamzik
 unknown, Probe: TRI7597
 Solvent: cdcl3
 Ambient temperature
 OBSERVE Proton, 599.801 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.995 sec
 Width 9611.9 Hz
 8 scans
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536



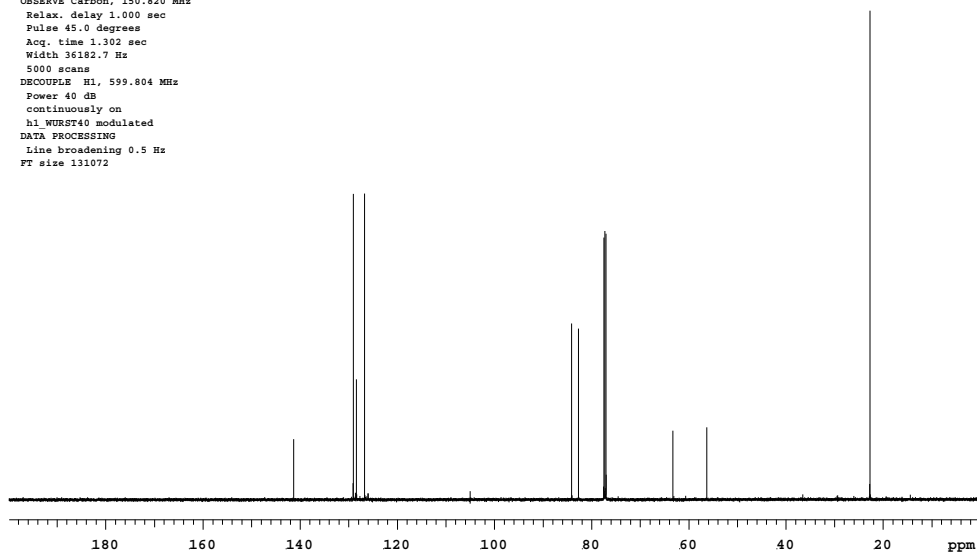
Chemist: hamzik; Notebook: 0298608; Page: 0030

Plotted: 11/03 08:52:12

Restricted Confidential, Limited Access

PhBr carbon

Sample name: PhBr carbon
 Notebook: 0298608, Page: 0097
 Data acquired: 21-Oct-2009, 00:29:04
 Data file name: Carbon01.fid
 Data reprocessed: Thu Oct 22 09:17:32 EDT 2009
 Chemist: hamzik
 unknown, Probe: TRI7597
 Solvent: cdcl3
 Ambient temperature
 OBSERVE Carbon, 150.820 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.302 sec
 Width 36182.7 Hz
 5000 scans
 DECOUPLE H1, 599.804 MHz
 Power 40 dB
 continuously on
 h1 WURST40 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 131072



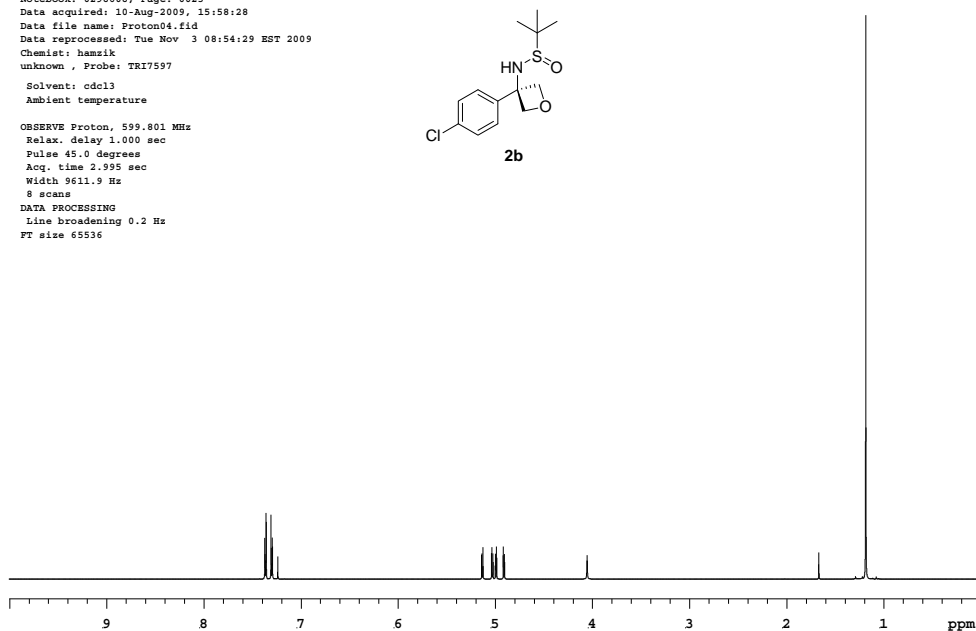
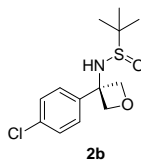
Chemist: hamzik; Notebook: 0298608; Page: 0097

Plotted: 10/22 09:17:32

Restricted Confidential, Limited Access

parachloro final (3)

Sample name: parachloro final (3)
 Notebook: 0298608, Page: 0023
 Data acquired: 10-Aug-2009, 15:58:28
 Data file name: Proton04.fid
 Data reprocessed: Tue Nov 3 08:54:29 EST 2009
 Chemist: hamzik
 unknown, Probe: TRI7597
 Solvent: cdcl3
 Ambient temperature
 OBSERVE Proton, 599.801 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.995 sec
 Width 9611.9 Hz
 8 scans
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536



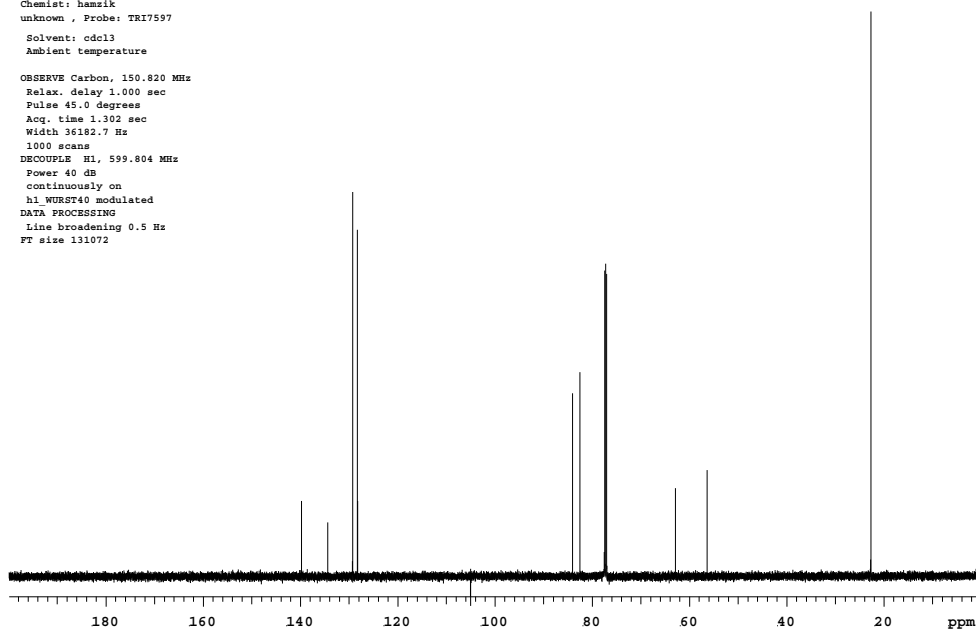
Chemist: hamzik; Notebook: 0298608; Page: 0023

Plotted: 11/03 08:54:29

Restricted Confidential, Limited Access

parachloro final carbon

Sample name: parachloro final carbon
 Notebook: 0298608, Page: 0023
 Data acquired: 11-Aug-2009, 19:02:44
 Data file name: Carbon01.fid
 Data reprocessed: Tue Nov 3 08:54:58 EST 2009
 Chemist: hamzik
 unknown, Probe: TRI7597
 Solvent: cdcl3
 Ambient temperature
 OBSERVE Carbon, 150.820 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.302 sec
 Width 36182.7 Hz
 1000 scans
 DECOUPLE H1, 599.804 MHz
 Power 40 dB
 continuously on
 h1 WURST40 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 131072



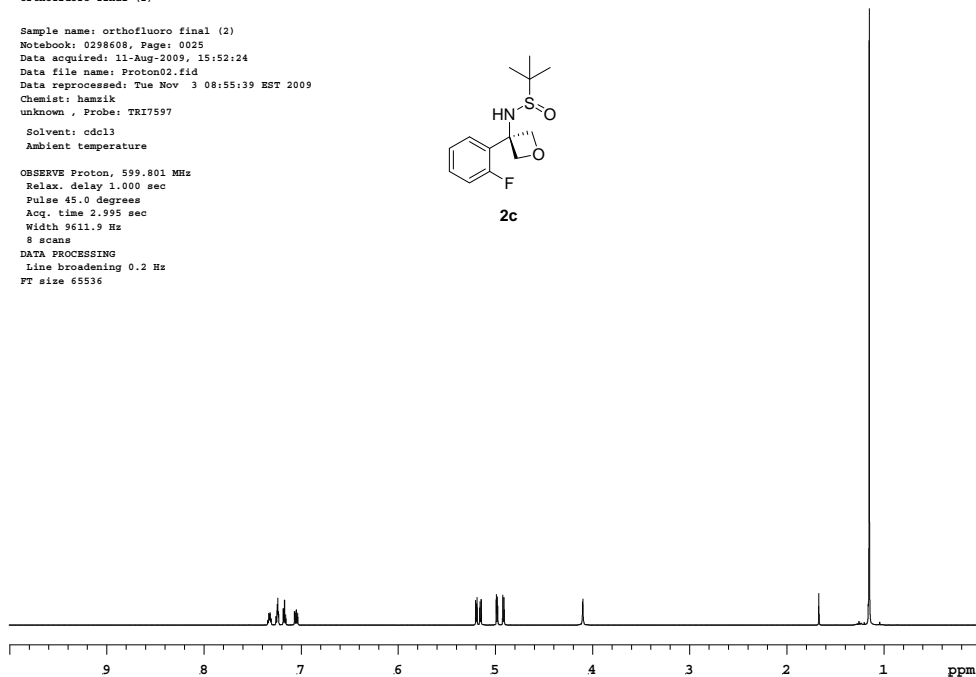
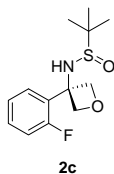
Chemist: hamzik; Notebook: 0298608; Page: 0023

Plotted: 11/03 08:54:58

Restricted Confidential, Limited Access

orthofluoro final (2)

Sample name: orthofluoro final (2)
Notebook: 0298608, Page: 0025
Data acquired: 11-Aug-2009, 15:52:24
Data file name: Proton02.fid
Data reprocessed: Tue Nov 3 08:55:39 EST 2009
Chemist: hanzik
unknown, Probe: TRI7597
Solvent: cdcl3
Ambient temperature
OBSERVE Proton, 599.801 MHz
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.995 sec
Width 9611.9 Hz
8 scans
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536



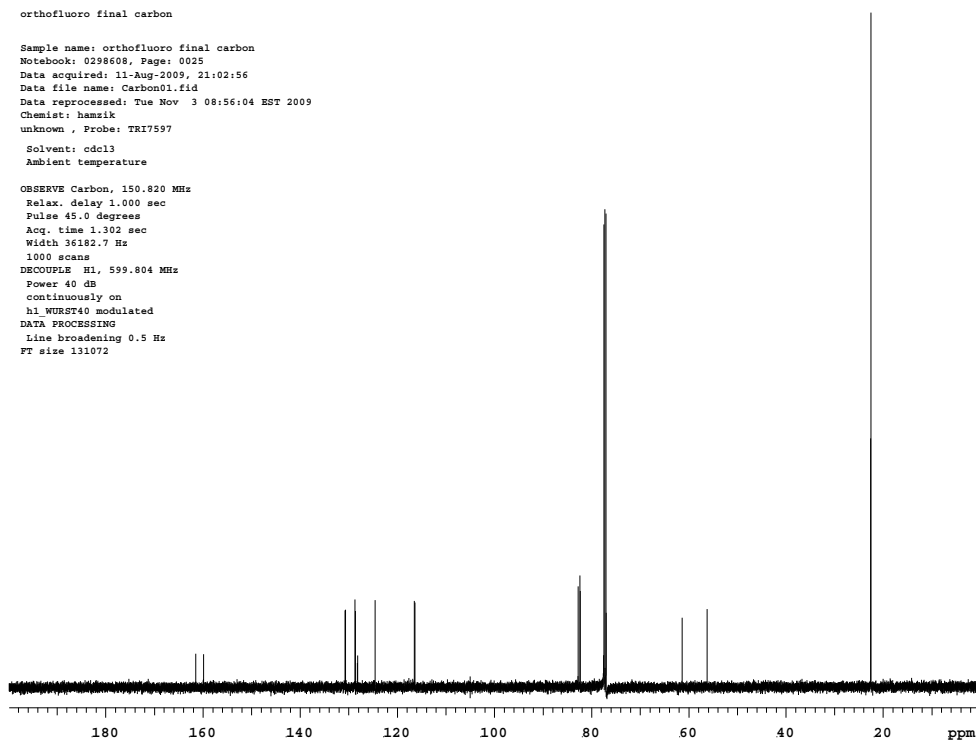
Chemist: hanzik; Notebook: 0298608; Page: 0025

Plotted: 11/03 08:55:39

Restricted Confidential, Limited Access

orthofluoro final carbon

Sample name: orthofluoro final carbon
Notebook: 0298608, Page: 0025
Data acquired: 11-Aug-2009, 21:02:56
Data file name: Carbon01.fid
Data reprocessed: Tue Nov 3 08:56:04 EST 2009
Chemist: hanzik
unknown, Probe: TRI7597
Solvent: cdcl3
Ambient temperature
OBSERVE Carbon, 150.820 MHz
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.302 sec
Width 36182.7 Hz
1000 scans
DECOUPLE H1, 599.804 MHz
Power 40 dB
continuously on
h1 WURST40 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072



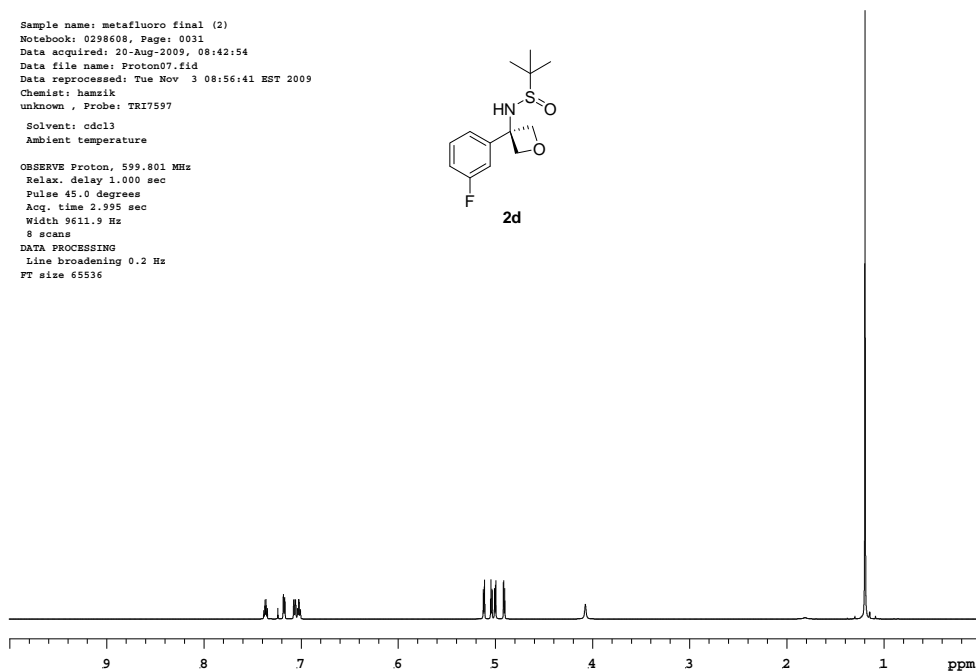
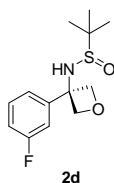
Chemist: hanzik; Notebook: 0298608; Page: 0025

Plotted: 11/03 08:56:05

Restricted Confidential, Limited Access

metafluoro final (2)

Sample name: metafluoro final (2)
Notebook: 0298608, Page: 0031
Data acquired: 20-Aug-2009, 08:42:54
Data file name: Proton07.fid
Data reprocessed: Tue Nov 3 08:56:41 EST 2009
Chemist: hamzik
unknown, Probe: TRI7597
Solvent: cdcl3
Ambient temperature
OBSERVE Proton, 599.801 MHz
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.995 sec
Width 9611.9 Hz
8 scans
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536



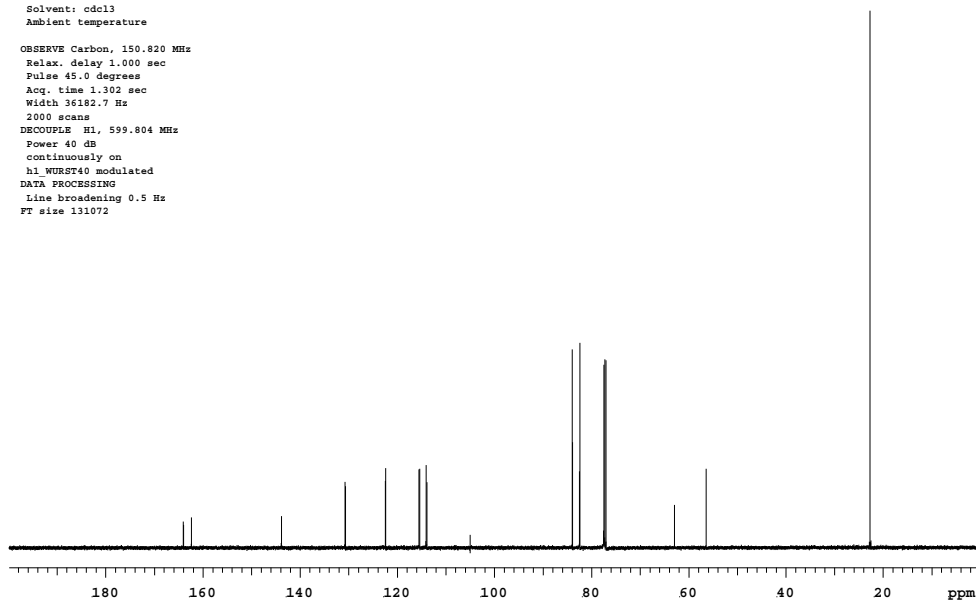
Chemist: hamzik; Notebook: 0298608; Page: 0031

Plotted: 11/03 08:56:41

Restricted Confidential, Limited Access

metafluoro carbon

Sample name: metafluoro carbon
Notebook: 0298608, Page: 0031
Data acquired: 20-Aug-2009, 19:02:18
Data file name: Carbon01.fid
Data reprocessed: Tue Nov 3 08:57:18 EST 2009
Chemist: hamzik
unknown, Probe: TRI7597
Solvent: cdcl3
Ambient temperature
OBSERVE Carbon, 150.820 MHz
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.302 sec
Width 36182.7 Hz
2000 scans
DECOUPLE H1, 599.804 MHz
Power 40 dB
continuously on
h1 WURST40 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072



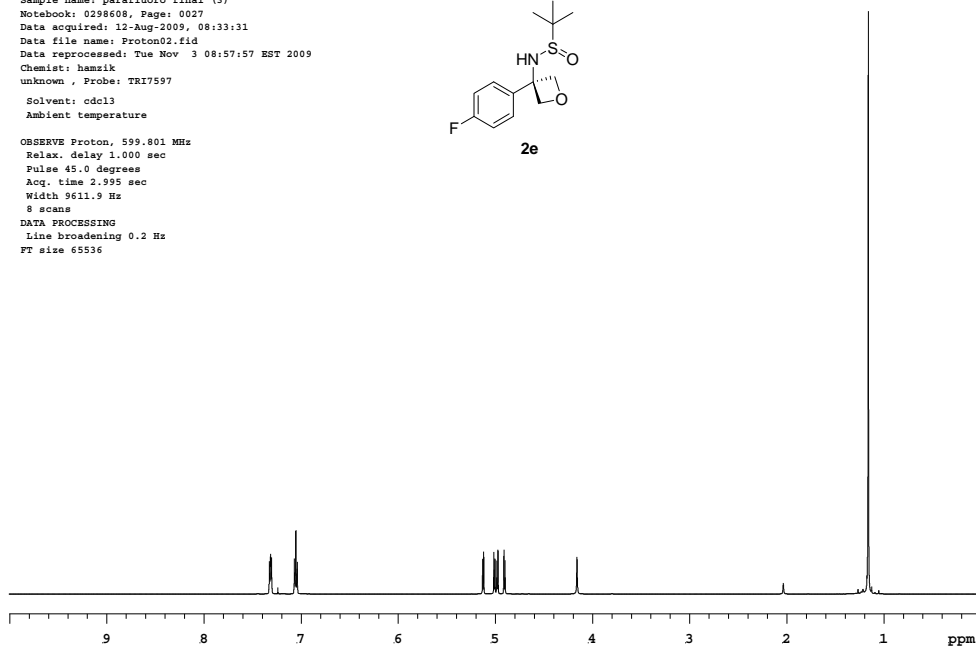
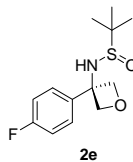
Chemist: hamzik; Notebook: 0298608; Page: 0031

Plotted: 11/03 08:57:19

Restricted Confidential, Limited Access

parafluoro final (3)

Sample name: parafluoro final (3)
 Notebook: 0298608, Page: 0027
 Data acquired: 12-Aug-2009, 08:33:31
 Data file name: Proton02.fid
 Data reprocessed: Tue Nov 3 08:57:57 EST 2009
 Chemist: hamzik
 unknown, Probe: TRI7597
 Solvent: cdcl3
 Ambient temperature
 OBSERVE Proton, 599.801 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.995 sec
 Width 9611.9 Hz
 8 scans
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536



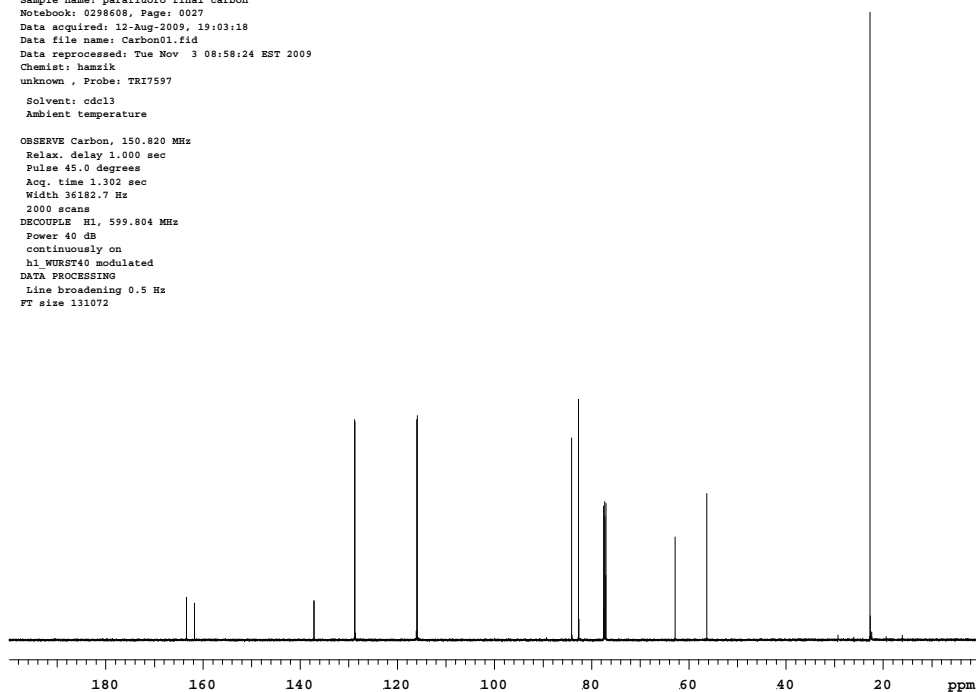
Chemist: hamzik; Notebook: 0298608; Page: 0027

Plotted: 11/03 08:57:57

Restricted Confidential, Limited Access

parafluoro final carbon

Sample name: parafluoro final carbon
 Notebook: 0298608, Page: 0027
 Data acquired: 12-Aug-2009, 19:03:18
 Data file name: Carbon01.fid
 Data reprocessed: Tue Nov 3 08:58:24 EST 2009
 Chemist: hamzik
 unknown, Probe: TRI7597
 Solvent: cdcl3
 Ambient temperature
 OBSERVE Carbon, 150.820 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.302 sec
 Width 36182.7 Hz
 2000 scans
 DECOUPLE H1, 599.804 MHz
 Power 40 dB
 continuously on
 h1 WURST40 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 131072



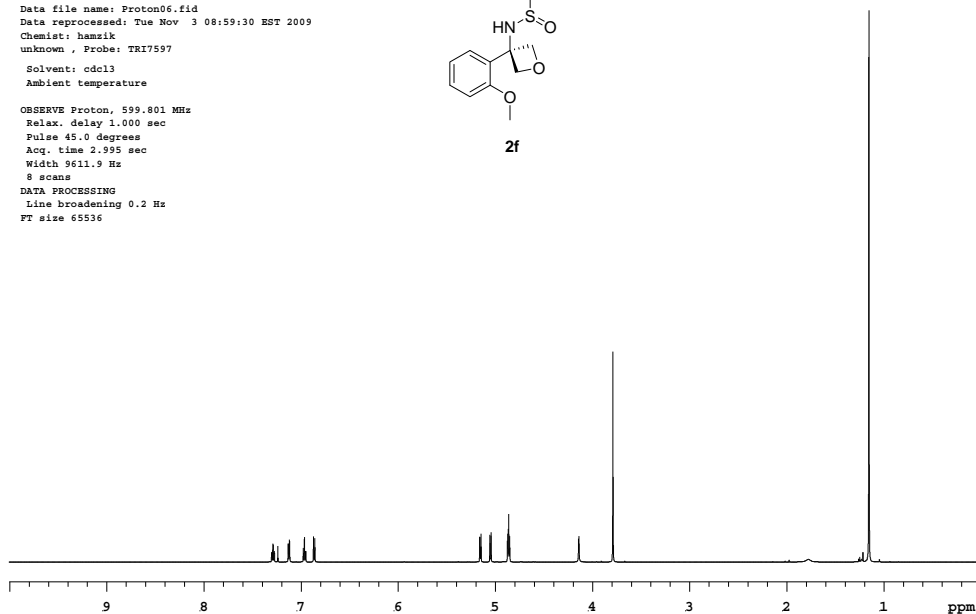
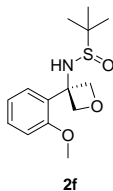
Chemist: hamzik; Notebook: 0298608; Page: 0027

Plotted: 11/03 08:58:24

Restricted Confidential, Limited Access

orthomethoxy FINAL

Sample name: orthomethoxy FINAL
Notebook: 0298608, Page: 0029
Data acquired: 14-Aug-2009, 14:44:12
Data file name: Proton06.fid
Data reprocessed: Tue Nov 3 08:59:30 EST 2009
Chemist: hamzik
unknown, Probe: TRI7597
Solvent: cdcl3
Ambient temperature
OBSERVE Proton, 599.801 MHz
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.995 sec
Width 9611.9 Hz
8 scans
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536



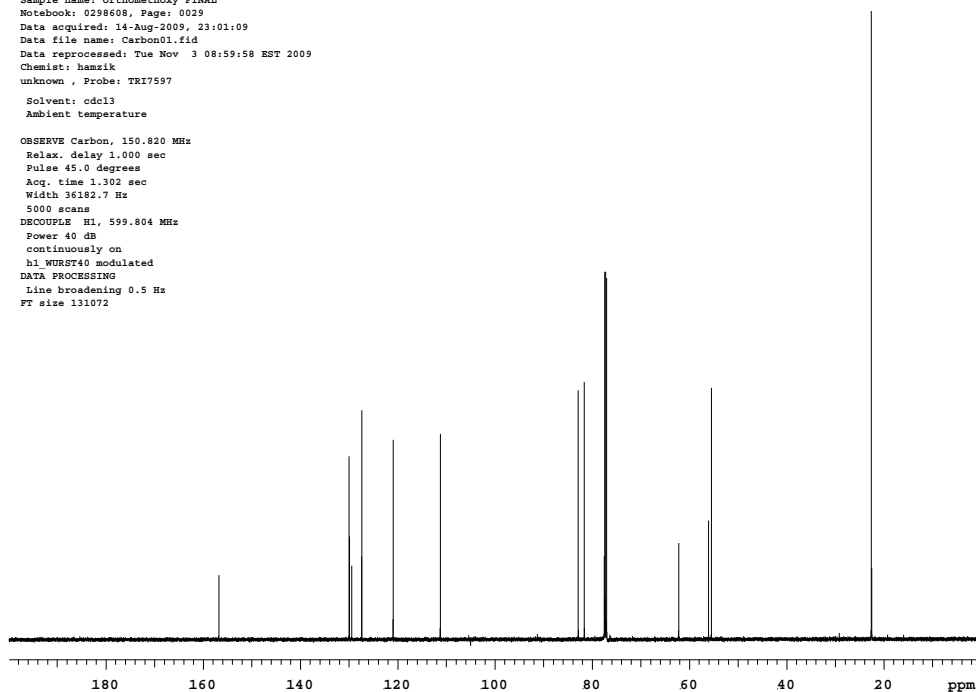
Chemist: hamzik; Notebook: 0298608; Page: 0029

Plotted: 11/03 08:59:30

Restricted Confidential, Limited Access

orthomethoxy FINAL

Sample name: orthomethoxy FINAL
Notebook: 0298608, Page: 0029
Data acquired: 14-Aug-2009, 23:01:09
Data file name: Carbon01.fid
Data reprocessed: Tue Nov 3 08:59:58 EST 2009
Chemist: hamzik
unknown, Probe: TRI7597
Solvent: cdcl3
Ambient temperature
OBSERVE Carbon, 150.820 MHz
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.302 sec
Width 36182.7 Hz
5000 scans
DECOUPLE H1, 599.804 MHz
Power 40 dB
continuously on
h1 WURST40 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072



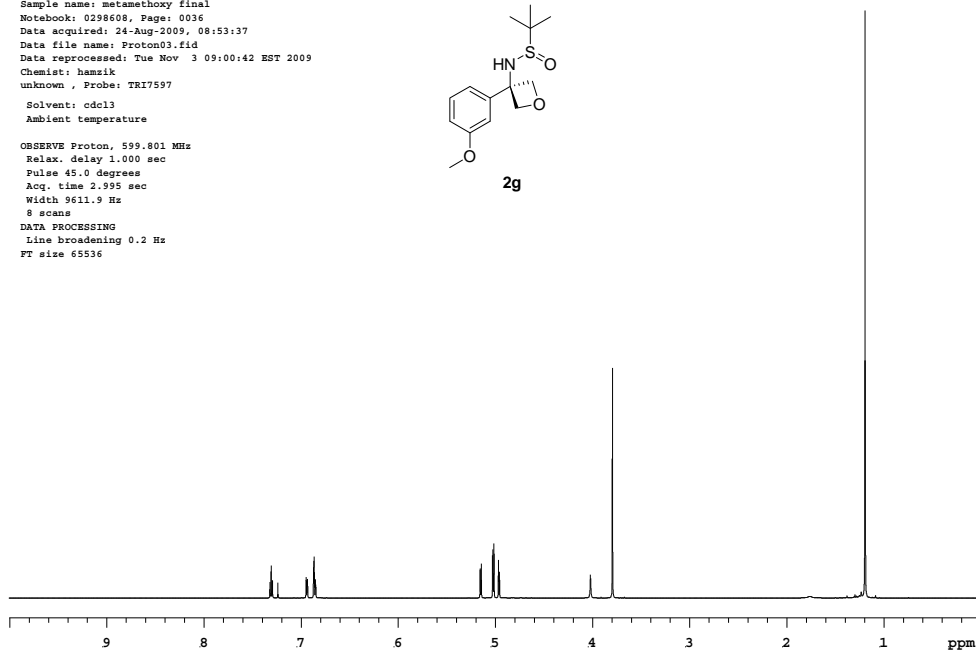
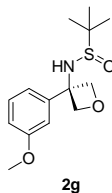
Chemist: hamzik; Notebook: 0298608; Page: 0029

Plotted: 11/03 08:59:58

Restricted Confidential, Limited Access

metamethoxy final

Sample name: metamethoxy final
Notebook: 0298608, Page: 0036
Data acquired: 24-Aug-2009, 08:53:37
Data file name: Proton03.fid
Data reprocessed: Tue Nov 3 09:00:42 EST 2009
Chemist: hamzik
unknown, Probe: TRI7597
Solvent: cdcl3
Ambient temperature
OBSERVE Proton, 599.801 MHz
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.995 sec
Width 9611.9 Hz
8 scans
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536



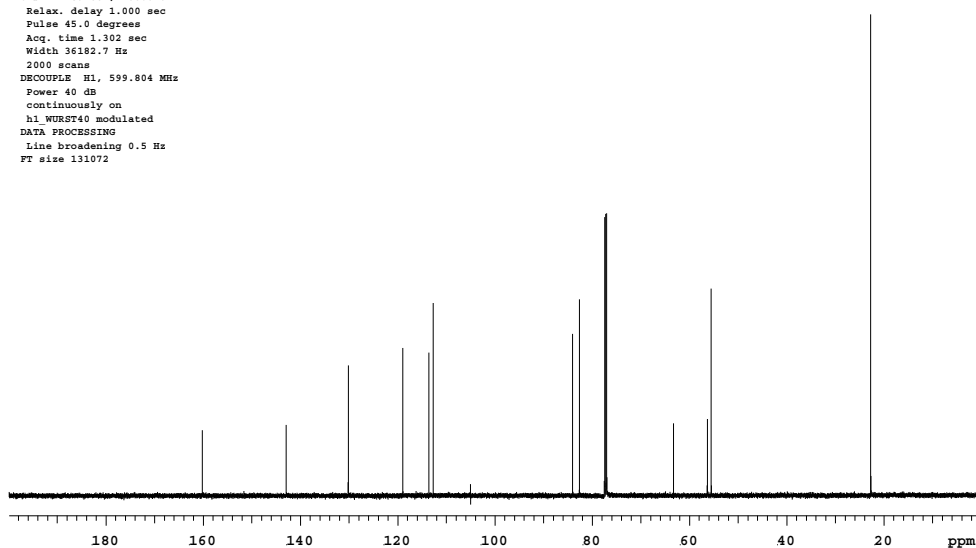
Chemist: hamzik; Notebook: 0298608; Page: 0036

Plotted: 11/03 09:00:43

Restricted Confidential, Limited Access

metamethoxy final carbon

Sample name: metamethoxy final carbon
Notebook: 0298608, Page: 0036
Data acquired: 24-Aug-2009, 19:02:55
Data file name: Carbon01.fid
Data reprocessed: Tue Nov 3 09:01:28 EST 2009
Chemist: hamzik
unknown, Probe: TRI7597
Solvent: cdcl3
Ambient temperature
OBSERVE Carbon, 150.820 MHz
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.302 sec
Width 36182.7 Hz
2000 scans
DECOUPLE H1, 599.804 MHz
Power 40 dB
continuously on
h1 WURST40 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072



Chemist: hamzik; Notebook: 0298608; Page: 0036

Plotted: 11/03 09:01:29

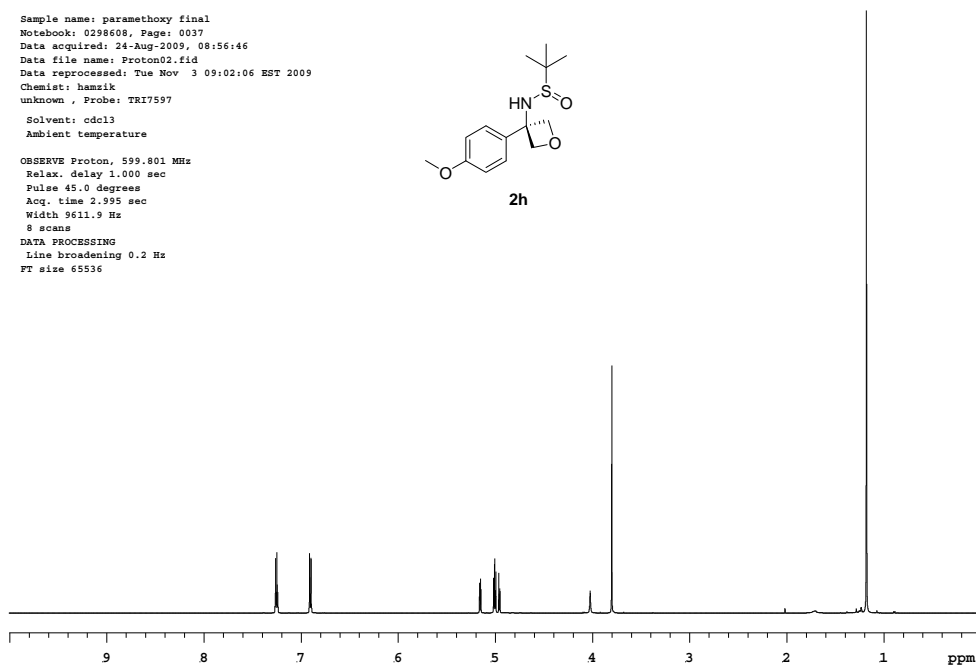
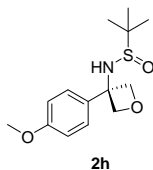
Restricted Confidential, Limited Access

paramethoxy final

Sample name: paramethoxy final
 Notebook: 0298608, Page: 0037
 Data acquired: 24-Aug-2009, 08:56:46
 Data file name: Proton02.fid
 Data reprocessed: Tue Nov 3 09:02:06 EST 2009
 Chemist: hamzik
 unknown, Probe: TRI7597
 Solvent: cdcl3
 Ambient temperature

OBSERVE Proton, 599.801 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.995 sec
 Width 9611.9 Hz
 8 scans

DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536



Chemist: hamzik; Notebook: 0298608; Page: 0037

Plotted: 11/03 09:02:07

Restricted Confidential, Limited Access

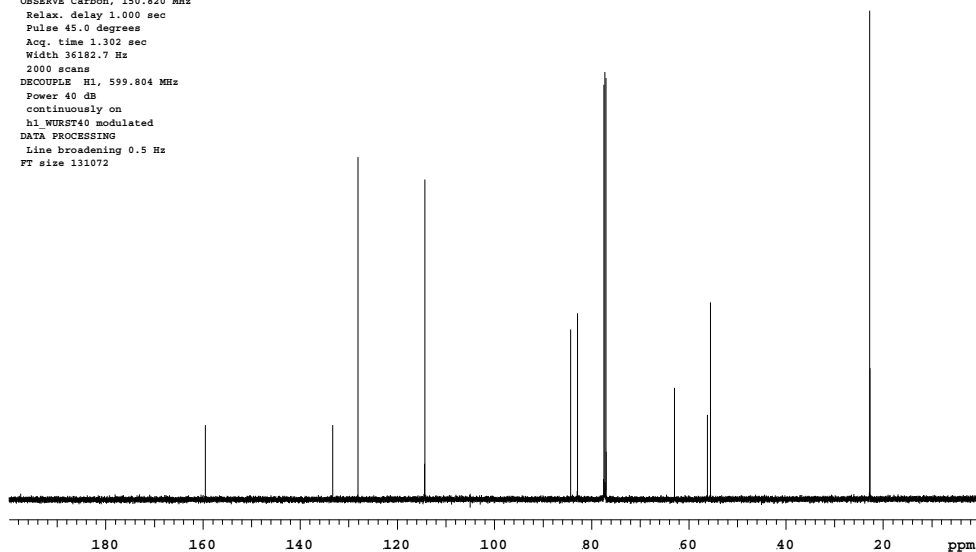
paramethoxy final carbon

Sample name: paramethoxy final carbon
 Notebook: 0298608, Page: 0037
 Data acquired: 24-Aug-2009, 20:22:02
 Data file name: Carbon01.fid
 Data reprocessed: Tue Nov 3 09:02:37 EST 2009
 Chemist: hamzik
 unknown, Probe: TRI7597
 Solvent: cdcl3
 Ambient temperature

OBSERVE Carbon, 150.820 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.302 sec
 Width 36182.7 Hz
 2000 scans

DECOUPLE H1, 599.804 MHz
 Power 40 dB
 continuously on
 h1 WURST40 modulated

DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 131072



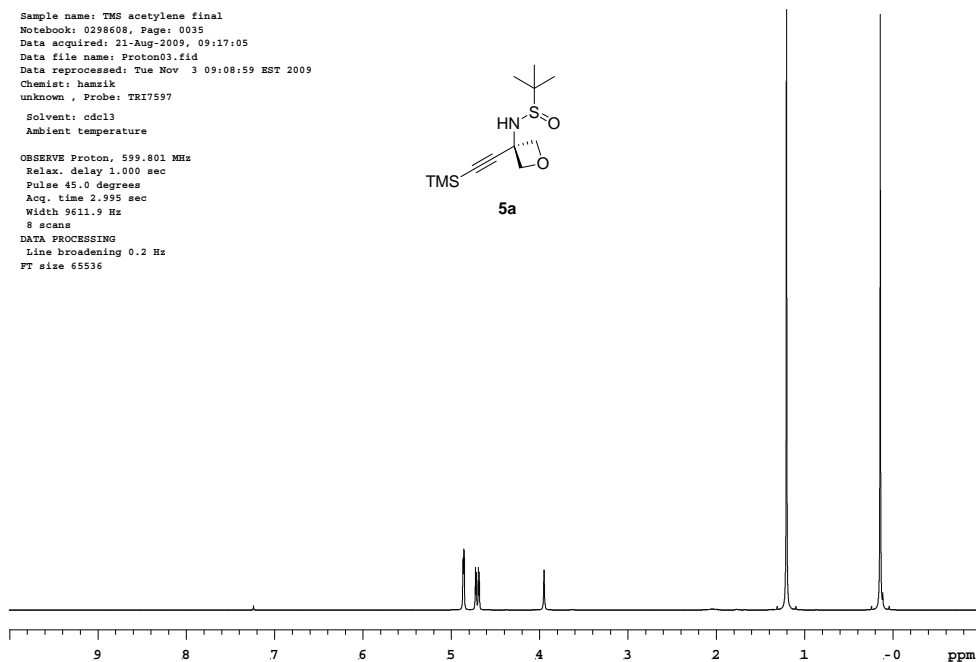
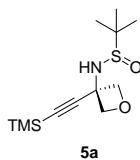
Chemist: hamzik; Notebook: 0298608; Page: 0037

Plotted: 11/03 09:02:37

Restricted Confidential, Limited Access

TMS acetylene final

Sample name: TMS acetylene final
Notebook: 0298608, Page: 0035
Data acquired: 21-Aug-2009, 09:17:05
Data file name: Proton03.fid
Data reprocessed: Tue Nov 3 09:08:59 EST 2009
Chemist: hamzik
unknown, Probe: TRI7597
Solvent: cdcl3
Ambient temperature
OBSERVE Proton, 599.801 MHz
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.995 sec
Width 9611.9 Hz
8 scans
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536



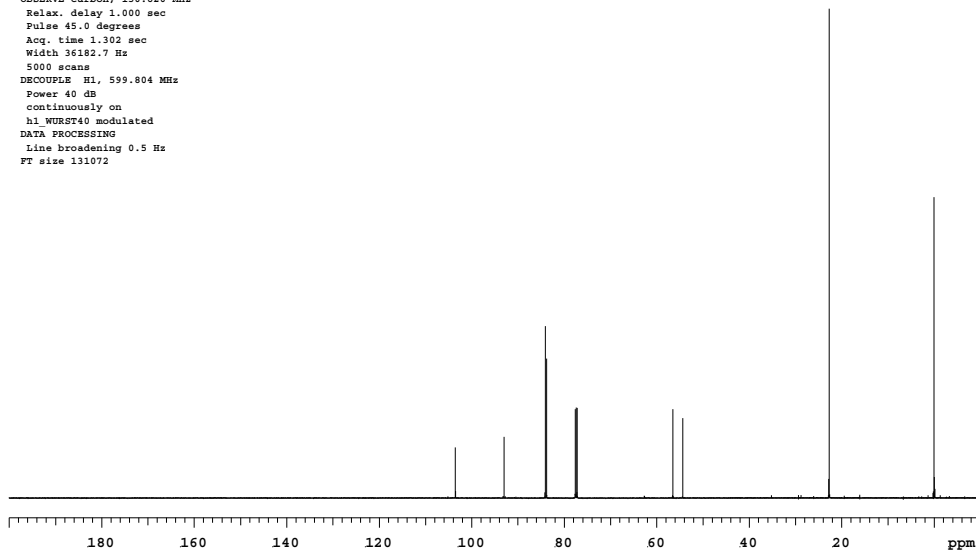
Chemist: hamzik; Notebook: 0298608; Page: 0035

Plotted: 11/03 09:08:59

Restricted Confidential, Limited Access

TMS acetylene carbon

Sample name: TMS acetylene carbon
Notebook: 0298608, Page: 0035
Data acquired: 21-Aug-2009, 19:02:45
Data file name: Carbon01.fid
Data reprocessed: Tue Nov 3 09:09:24 EST 2009
Chemist: hamzik
unknown, Probe: TRI7597
Solvent: cdcl3
Ambient temperature
OBSERVE Carbon, 150.820 MHz
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.302 sec
Width 36182.7 Hz
5000 scans
DECOUPLE H1, 599.804 MHz
Power 40 dB
continuously on
h1 WURST40 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072



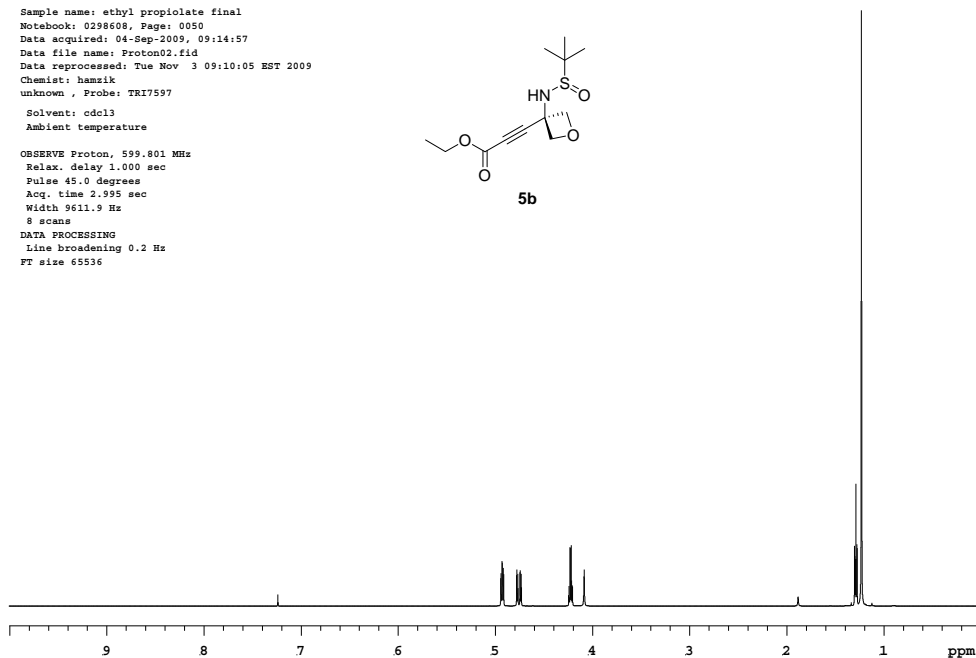
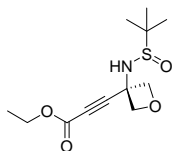
Chemist: hamzik; Notebook: 0298608; Page: 0035

Plotted: 11/03 09:09:24

Restricted Confidential, Limited Access

ethyl propiolate final

Sample name: ethyl propiolate final
 Notebook: 0298608, Page: 0050
 Data acquired: 04-Sep-2009, 09:14:57
 Data file name: Proton02.fid
 Data reprocessed: Tue Nov 3 09:10:05 EST 2009
 Chemist: hamzik
 unknown, Probe: TRI7597
 Solvent: cdcl3
 Ambient temperature
 OBSERVE Proton, 599.801 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.995 sec
 Width 9611.9 Hz
 8 scans
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536



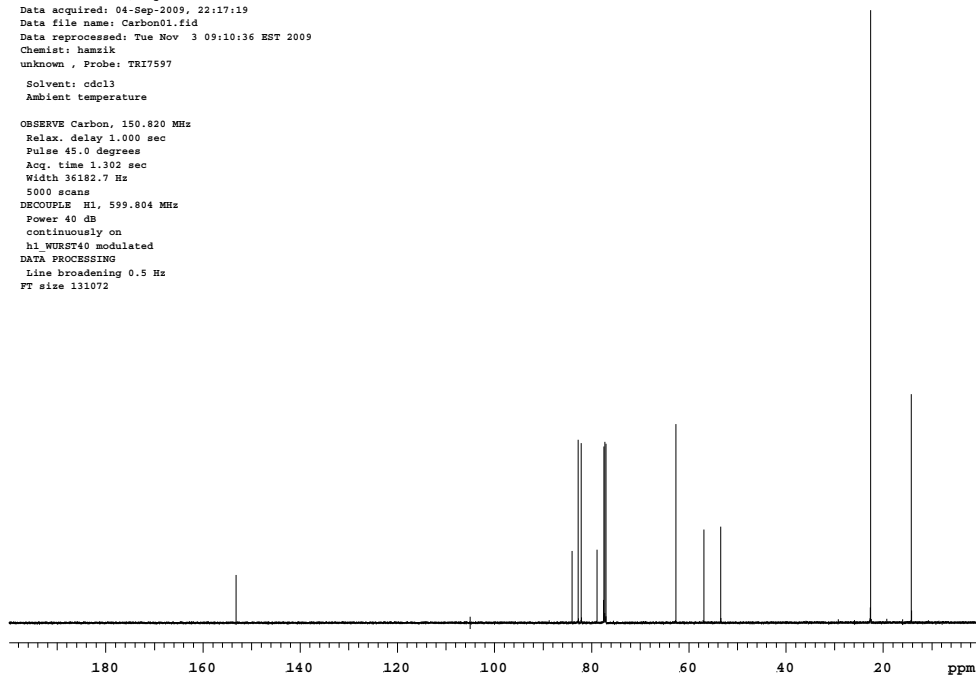
Chemist: hamzik; Notebook: 0298608; Page: 0050

Plotted: 11/03 09:10:05

Restricted Confidential, Limited Access

ethyl propiolate carbon

Sample name: ethyl propiolate carbon
 Notebook: 0298608, Page: 0050
 Data acquired: 04-Sep-2009, 22:17:19
 Data file name: Carbon01.fid
 Data reprocessed: Tue Nov 3 09:10:36 EST 2009
 Chemist: hamzik
 unknown, Probe: TRI7597
 Solvent: cdcl3
 Ambient temperature
 OBSERVE Carbon, 150.820 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.302 sec
 Width 36182.7 Hz
 5000 scans
 DECOUPLE H1, 599.804 MHz
 Power 40 dB
 continuously on
 h1 WURST40 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 131072



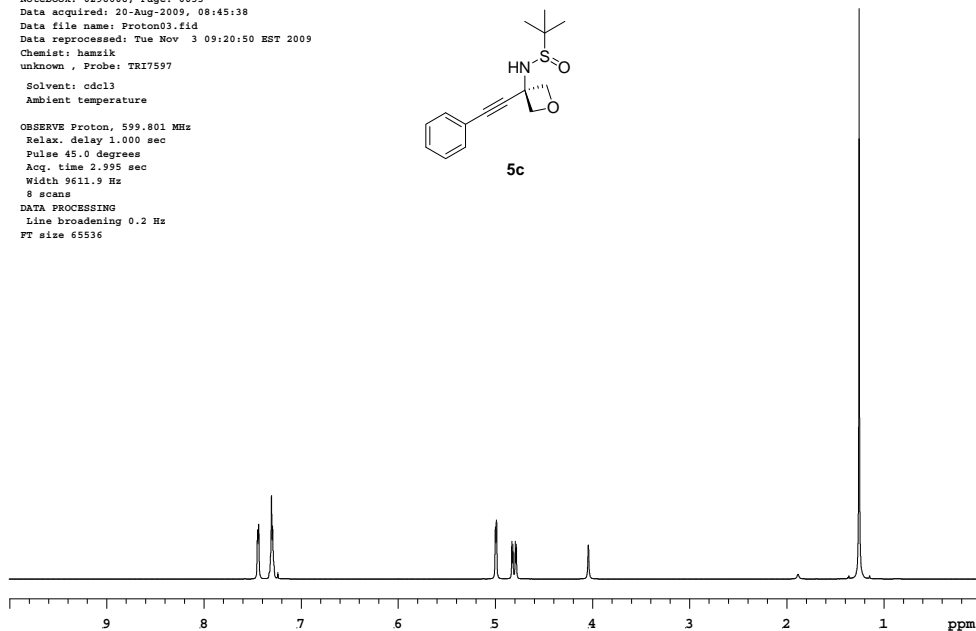
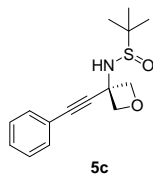
Chemist: hamzik; Notebook: 0298608; Page: 0050

Plotted: 11/03 09:10:36

Restricted Confidential, Limited Access

phenylacetylide final (2)

Sample name: phenylacetylide final (2)
 Notebook: 0298608, Page: 0033
 Data acquired: 20-Aug-2009, 08:45:38
 Data file name: Proton03.fid
 Data reprocessed: Tue Nov 3 09:20:50 EST 2009
 Chemist: hamzik
 unknown, Probe: TRI7597
 Solvent: cdcl3
 Ambient temperature
 OBSERVE Proton, 599.801 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.995 sec
 Width 9611.9 Hz
 8 scans
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536



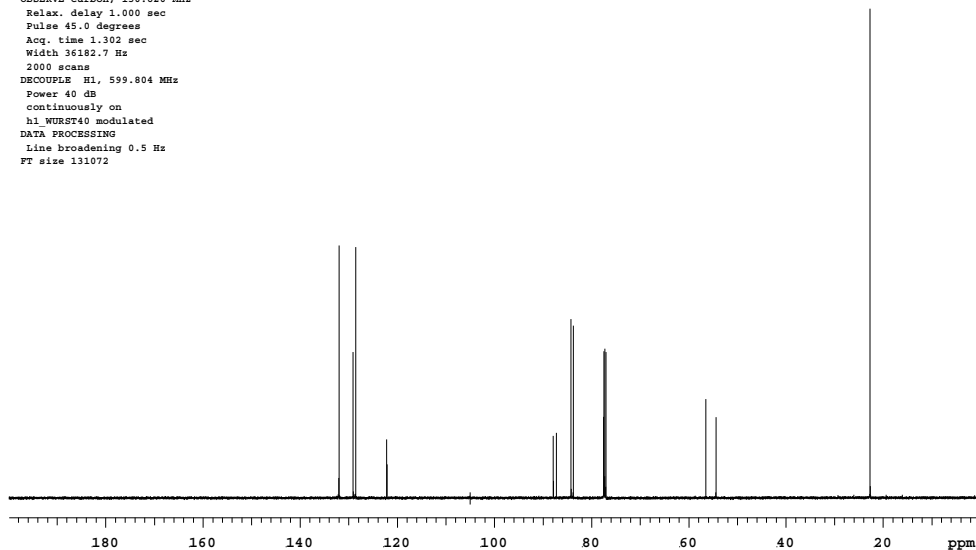
Chemist: hamzik; Notebook: 0298608; Page: 0033

Plotted: 11/03 09:20:51

Restricted Confidential, Limited Access

phenylacetylide carbon

Sample name: phenylacetylide carbon
 Notebook: 0298608, Page: 0033
 Data acquired: 20-Aug-2009, 20:21:26
 Data file name: Carbon01.fid
 Data reprocessed: Tue Nov 3 09:21:29 EST 2009
 Chemist: hamzik
 unknown, Probe: TRI7597
 Solvent: cdcl3
 Ambient temperature
 OBSERVE Carbon, 150.820 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.302 sec
 Width 36182.7 Hz
 2000 scans
 DECOUPLE H1, 599.804 MHz
 Power 40 dB
 continuously on
 h1 WURST40 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 131072



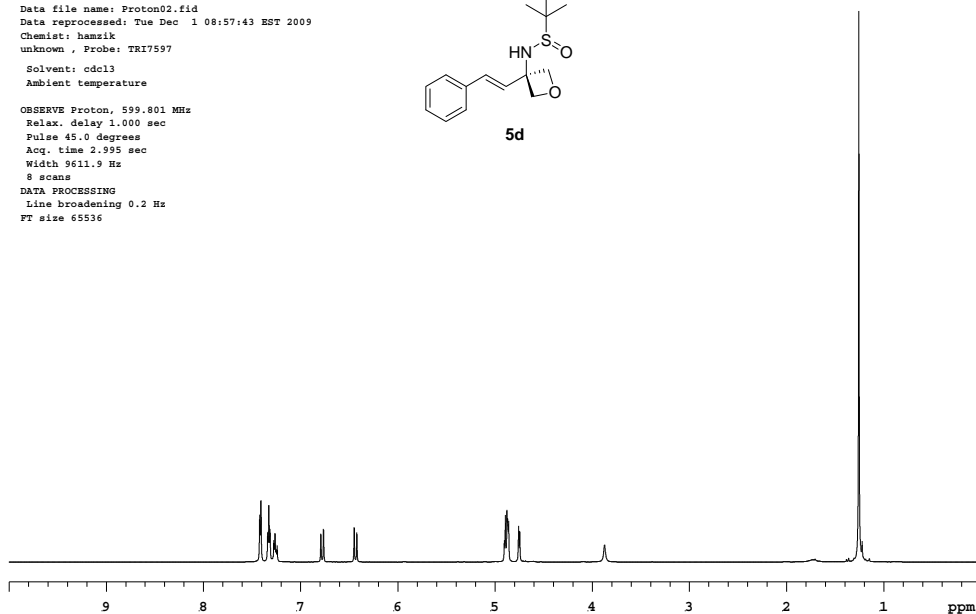
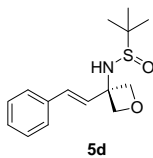
Chemist: hamzik; Notebook: 0298608; Page: 0033

Plotted: 11/03 09:21:30

Restricted Confidential, Limited Access

bromostyrene final

Sample name: bromostyrene final
 Notebook: 0298608, Page: 0079
 Data acquired: 08-Oct-2009, 08:38:33
 Data file name: Proton02.fid
 Data reprocessed: Tue Dec 1 08:57:43 EST 2009
 Chemist: hamzik
 unknown, Probe: TRI7597
 Solvent: cdcl3
 Ambient temperature
 OBSERVE Proton, 599.801 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.995 sec
 Width 9611.9 Hz
 8 scans
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536



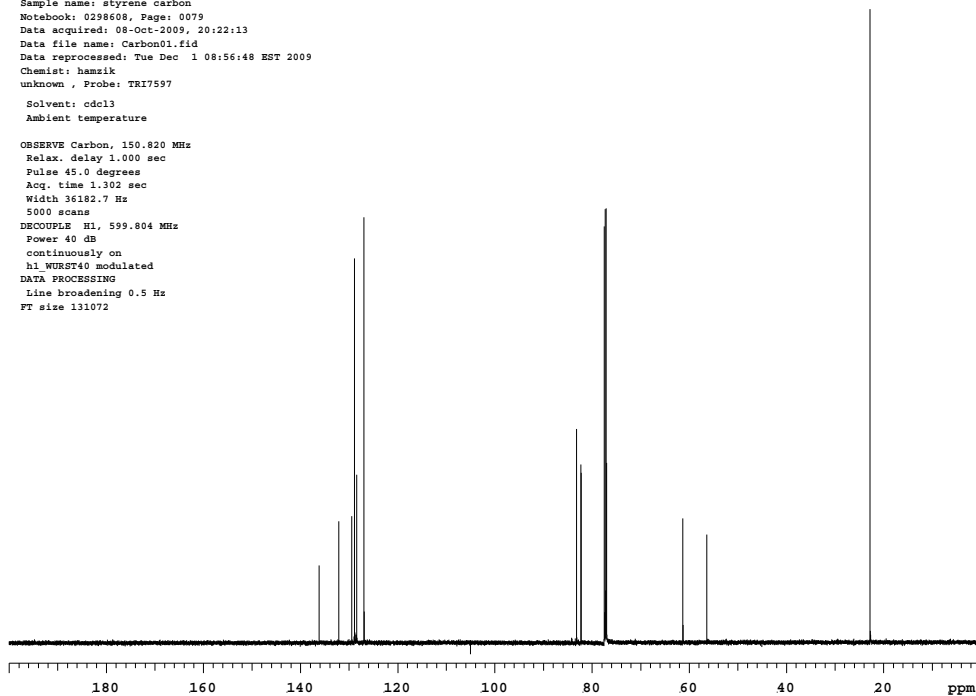
Chemist: hamzik; Notebook: 0298608; Page: 0079

Plotted: 12/01 08:57:44

Restricted Confidential, Limited Access

styrene carbon

Sample name: styrene carbon
 Notebook: 0298608, Page: 0079
 Data acquired: 08-Oct-2009, 20:22:13
 Data file name: Carbon01.fid
 Data reprocessed: Tue Dec 1 08:56:48 EST 2009
 Chemist: hamzik
 unknown, Probe: TRI7597
 Solvent: cdcl3
 Ambient temperature
 OBSERVE Carbon, 150.820 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.302 sec
 Width 36182.7 Hz
 5000 scans
 DECOUPLE H1, 599.804 MHz
 Power 40 dB
 continuously on
 h1_WURST40 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 131072



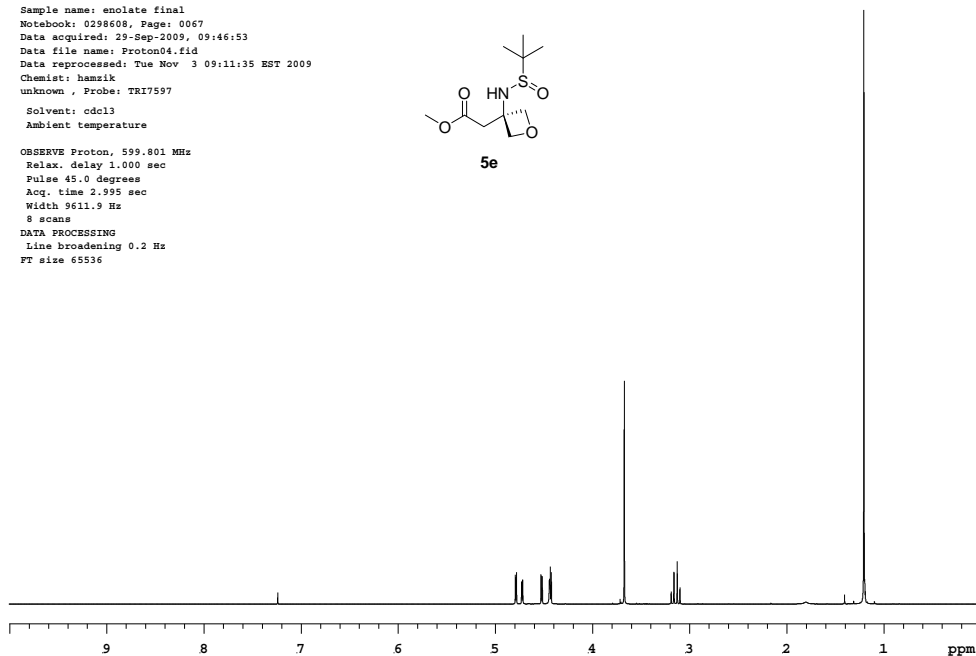
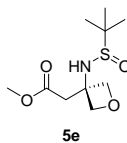
Chemist: hamzik; Notebook: 0298608; Page: 0079

Plotted: 12/01 08:56:49

Restricted Confidential, Limited Access

enolate final

Sample name: enolate final
 Notebook: 0298608, Page: 0067
 Data acquired: 29-Sep-2009, 09:46:53
 Data file name: Proton04.fid
 Data reprocessed: Tue Nov 3 09:11:35 EST 2009
 Chemist: hamzik
 unknown, Probe: TRI7597
 Solvent: cdcl3
 Ambient temperature
 OBSERVE Proton, 599.801 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.995 sec
 Width 9611.9 Hz
 8 scans
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536



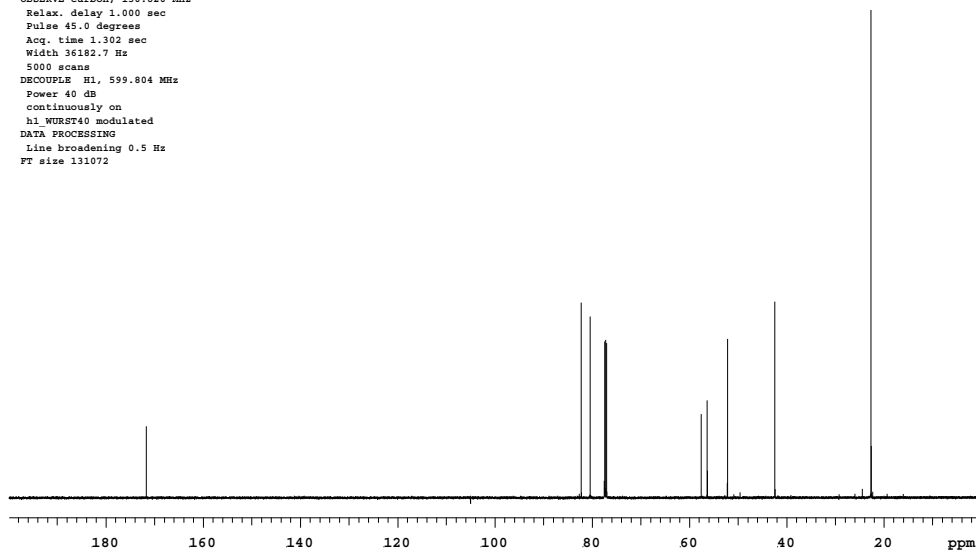
Chemist: hamzik; Notebook: 0298608; Page: 0067

Plotted: 11/03 09:11:36

Restricted Confidential, Limited Access

enolate addition carbon

Sample name: enolate addition carbon
 Notebook: 0298608, Page: 0067
 Data acquired: 29-Sep-2009, 21:41:45
 Data file name: Carbon01.fid
 Data reprocessed: Tue Nov 3 09:12:12 EST 2009
 Chemist: hamzik
 unknown, Probe: TRI7597
 Solvent: cdcl3
 Ambient temperature
 OBSERVE Carbon, 150.820 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.302 sec
 Width 36182.7 Hz
 5000 scans
 DECOUPLE H1, 599.804 MHz
 Power 40 dB
 continuously on
 h1 WURST40 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 131072



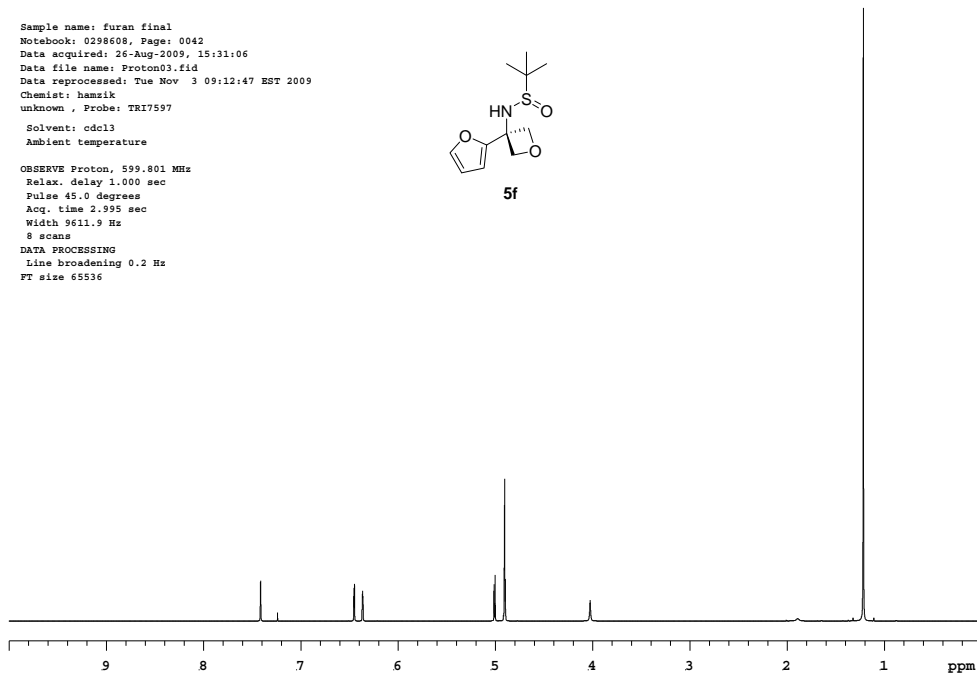
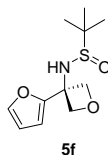
Chemist: hamzik; Notebook: 0298608; Page: 0067

Plotted: 11/03 09:12:12

Restricted Confidential, Limited Access

uran final

Sample name: uran final
 Notebook: 0298608, Page: 0042
 Data acquired: 26-Aug-2009, 15:31:06
 Data file name: Proton03.fid
 Data reprocessed: Tue Nov 3 09:12:47 EST 2009
 Chemist: hamzik
 unknown, Probe: TRI7597
 Solvent: cdcl3
 Ambient temperature
 OBSERVE Proton, 599.801 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.995 sec
 Width 9611.9 Hz
 8 scans
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536



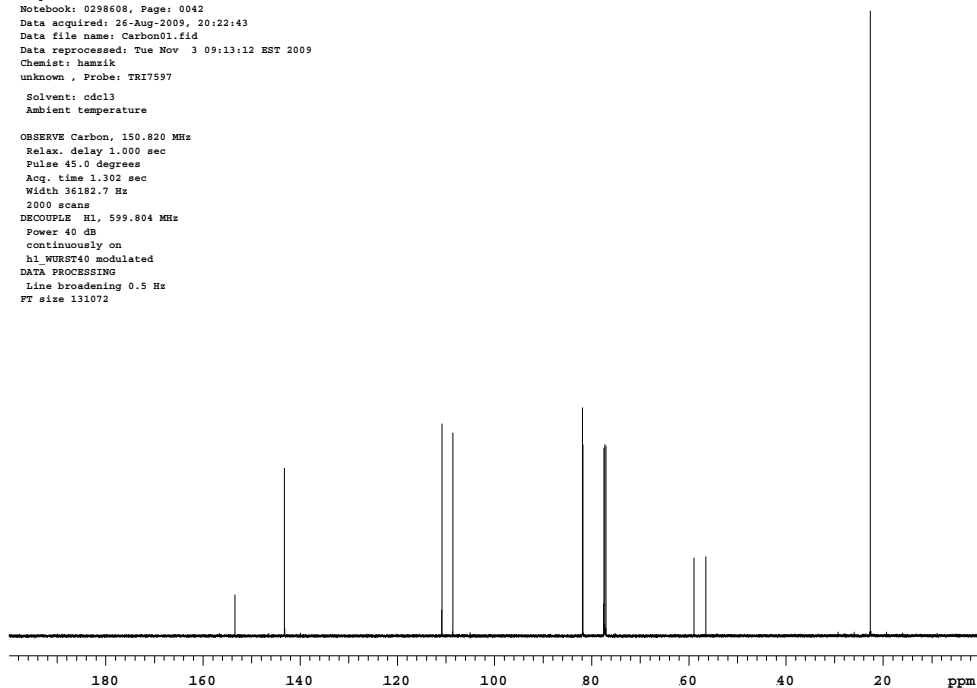
Chemist: hamzik; Notebook: 0298608; Page: 0042

Plotted: 11/03 09:12:48

Restricted Confidential, Limited Access

uran carbon

Sample name: uran carbon
 Notebook: 0298608, Page: 0042
 Data acquired: 26-Aug-2009, 20:22:43
 Data file name: Carbon01.fid
 Data reprocessed: Tue Nov 3 09:13:12 EST 2009
 Chemist: hamzik
 unknown, Probe: TRI7597
 Solvent: cdcl3
 Ambient temperature
 OBSERVE Carbon, 150.820 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.302 sec
 Width 36182.7 Hz
 2000 scans
 DECOUPLE H1, 599.804 MHz
 Power 40 dB
 continuously on
 h1 WURST40 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 131072



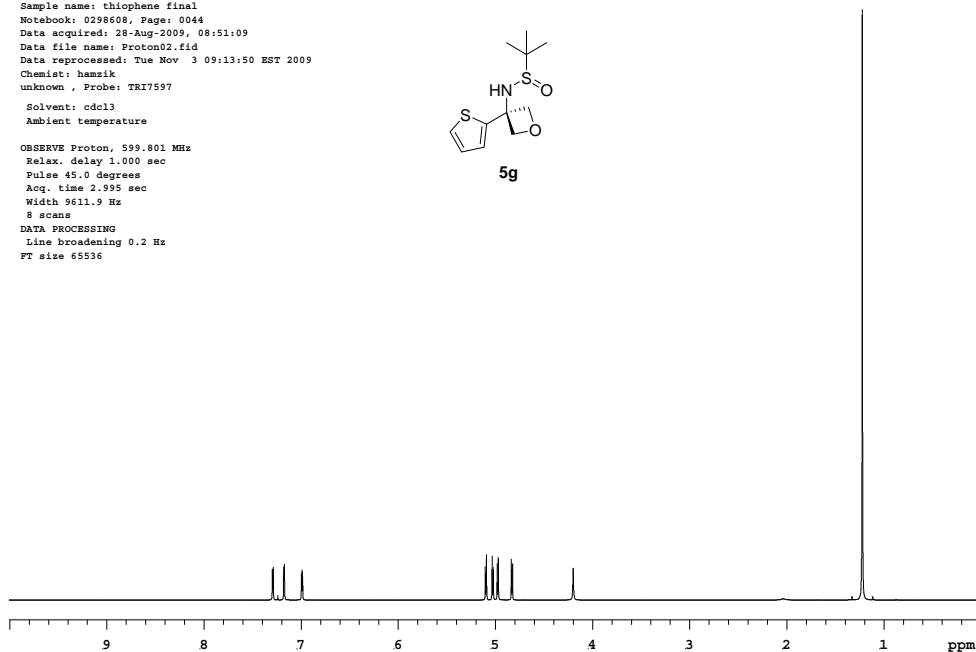
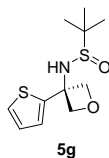
Chemist: hamzik; Notebook: 0298608; Page: 0042

Plotted: 11/03 09:13:12

Restricted Confidential, Limited Access

thiophene final

Sample name: thiophene final
Notebook: 0298608, Page: 0044
Data acquired: 28-Aug-2009, 08:51:09
Data file name: Proton02.fid
Data reprocessed: Tue Nov 3 09:13:50 EST 2009
Chemist: hamzik
unknown, Probe: TRI7597
Solvent: cdcl3
Ambient temperature
OBSERVE Proton, 599.801 MHz
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.995 sec
Width 9611.9 Hz
8 scans
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536



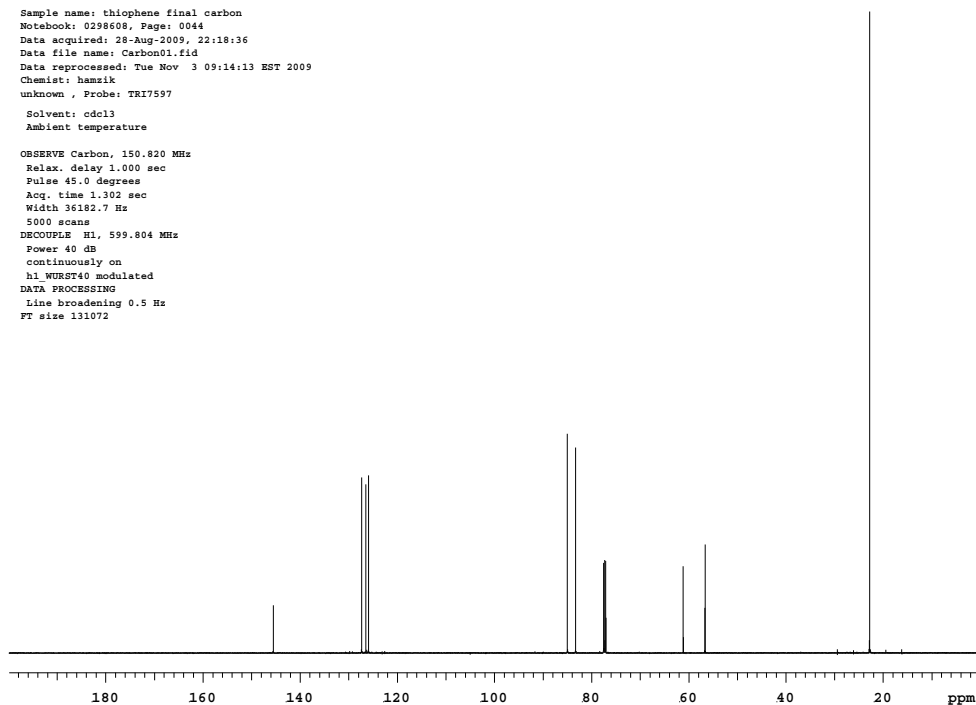
Chemist: hamzik; Notebook: 0298608; Page: 0044

Plotted: 11/03 09:13:51

Restricted Confidential, Limited Access

thiophene final carbon

Sample name: thiophene final carbon
Notebook: 0298608, Page: 0044
Data acquired: 28-Aug-2009, 22:18:36
Data file name: Carbon01.fid
Data reprocessed: Tue Nov 3 09:14:13 EST 2009
Chemist: hamzik
unknown, Probe: TRI7597
Solvent: cdcl3
Ambient temperature
OBSERVE Carbon, 150.820 MHz
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.302 sec
Width 36182.7 Hz
5000 scans
DECOUPLE H1, 599.804 MHz
Power 40 dB
continuously on
h1 WURST40 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072



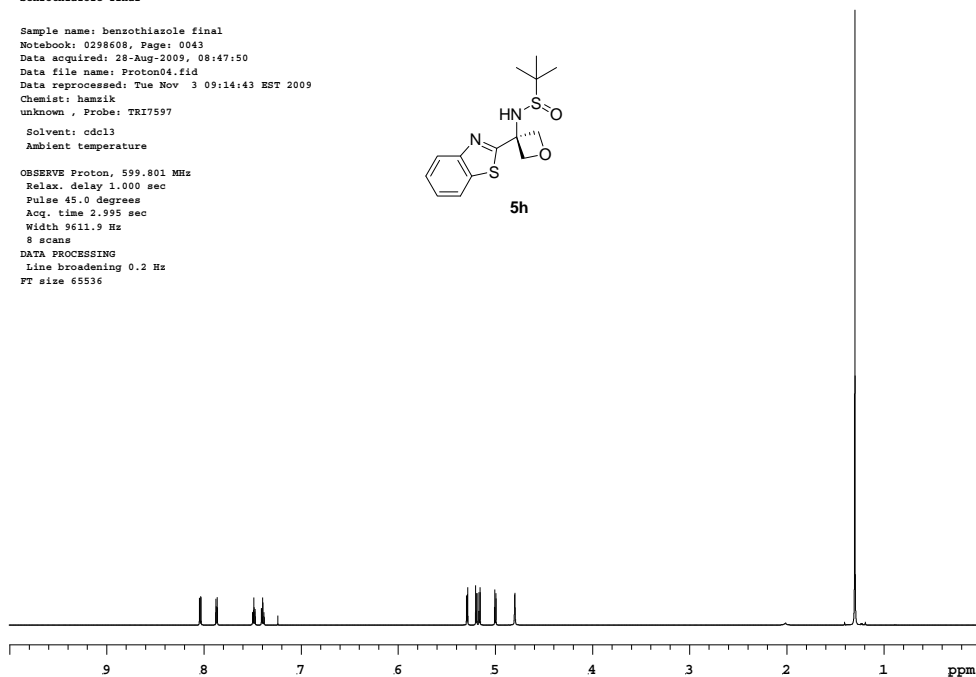
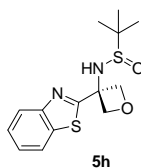
Chemist: hamzik; Notebook: 0298608; Page: 0044

Plotted: 11/03 09:14:13

Restricted Confidential, Limited Access

benzothiazole final

Sample name: benzothiazole final
 Notebook: 0298608, Page: 0043
 Data acquired: 28-Aug-2009, 08:47:50
 Data file name: Proton04.fid
 Data reprocessed: Tue Nov 3 09:14:43 EST 2009
 Chemist: hamzik
 unknown, Probe: TRI7597
 Solvent: cdcl3
 Ambient temperature
 OBSERVE Proton, 599.801 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.995 sec
 Width 9611.9 Hz
 8 scans
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536



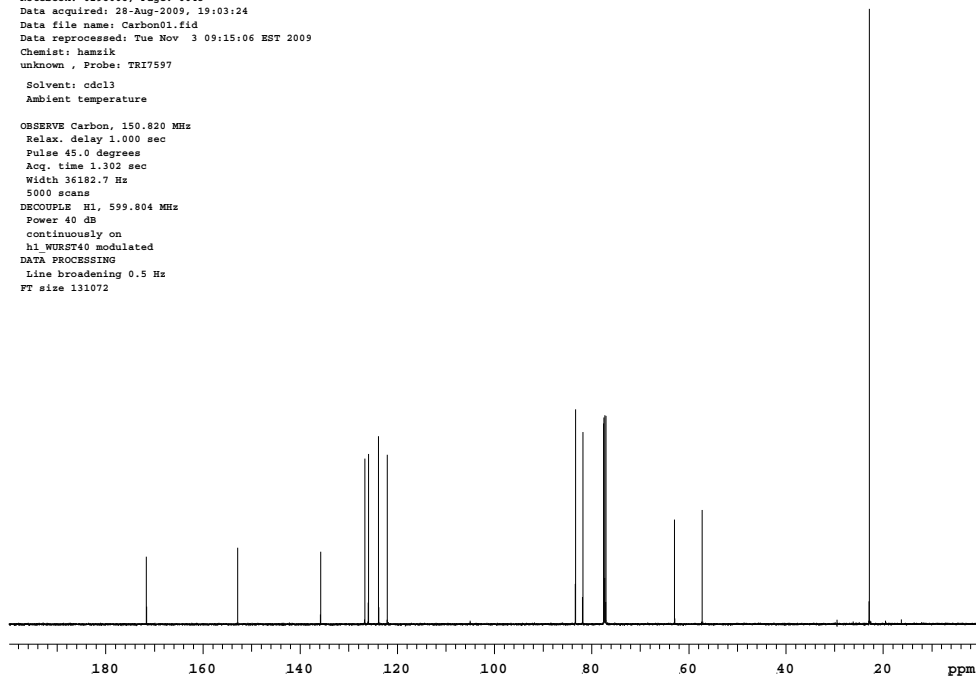
Chemist: hamzik; Notebook: 0298608; Page: 0043

Plotted: 11/03 09:14:44

Restricted Confidential, Limited Access

benzothiazole final carbon

Sample name: benzothiazole final carbon
 Notebook: 0298608, Page: 0043
 Data acquired: 28-Aug-2009, 19:03:24
 Data file name: Carbon01.fid
 Data reprocessed: Tue Nov 3 09:15:06 EST 2009
 Chemist: hamzik
 unknown, Probe: TRI7597
 Solvent: cdcl3
 Ambient temperature
 OBSERVE Carbon, 150.820 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.302 sec
 Width 36182.7 Hz
 5000 scans
 DECOUPLE H1, 599.804 MHz
 Power 40 dB
 continuously on
 h1_WURST40 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 131072



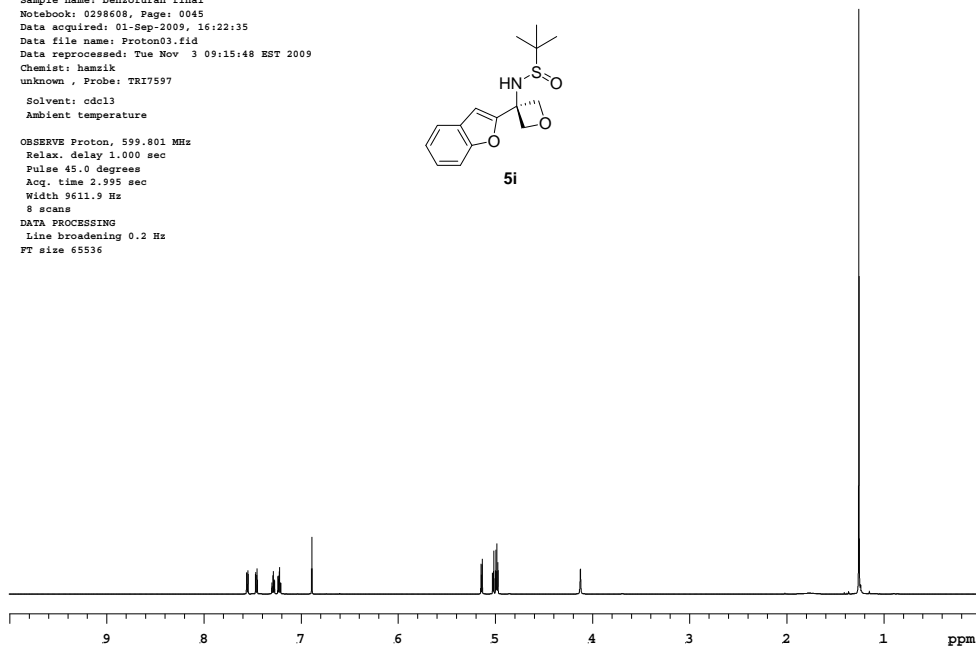
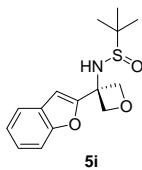
Chemist: hamzik; Notebook: 0298608; Page: 0043

Plotted: 11/03 09:15:07

Restricted Confidential, Limited Access

benzofuran final

Sample name: benzofuran final
 Notebook: 0298608, Page: 0045
 Data acquired: 01-Sep-2009, 16:22:35
 Data file name: Proton03.fid
 Data reprocessed: Tue Nov 3 09:15:48 EST 2009
 Chemist: hamzik
 unknown, Probe: TRI7597
 Solvent: cdcl3
 Ambient temperature
 OBSERVE Proton, 599.801 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.995 sec
 Width 9611.9 Hz
 8 scans
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536



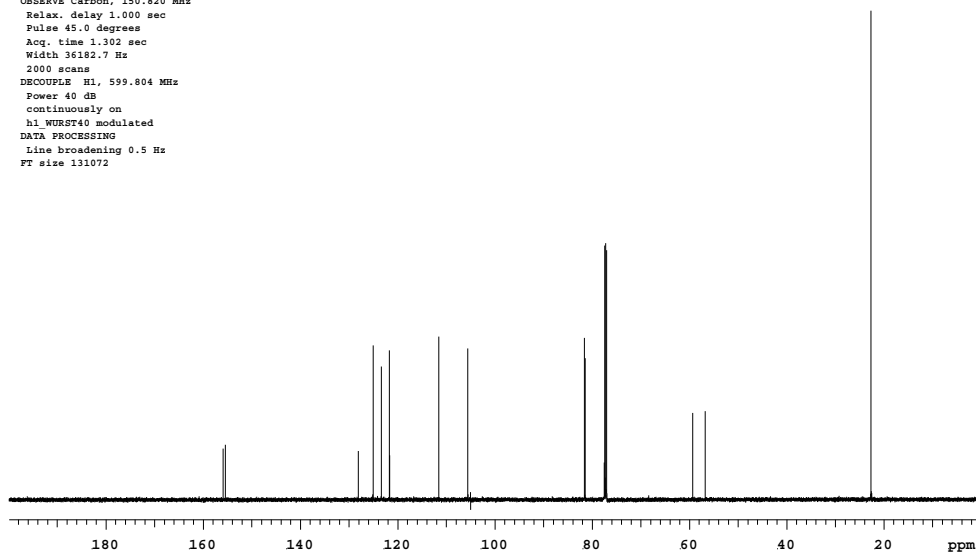
Chemist: hamzik; Notebook: 0298608; Page: 0045

Plotted: 11/03 09:15:49

Restricted Confidential, Limited Access

benzofuran carbon

Sample name: benzofuran carbon
 Notebook: 0298608, Page: 0045
 Data acquired: 01-Sep-2009, 19:02:58
 Data file name: Carbon01.fid
 Data reprocessed: Tue Nov 3 09:18:52 EST 2009
 Chemist: hamzik
 unknown, Probe: TRI7597
 Solvent: cdcl3
 Ambient temperature
 OBSERVE Carbon, 150.820 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.302 sec
 Width 36182.7 Hz
 2000 scans
 DECOUPLE H1, 599.804 MHz
 Power 40 dB
 continuously on
 h1 WURST40 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 131072



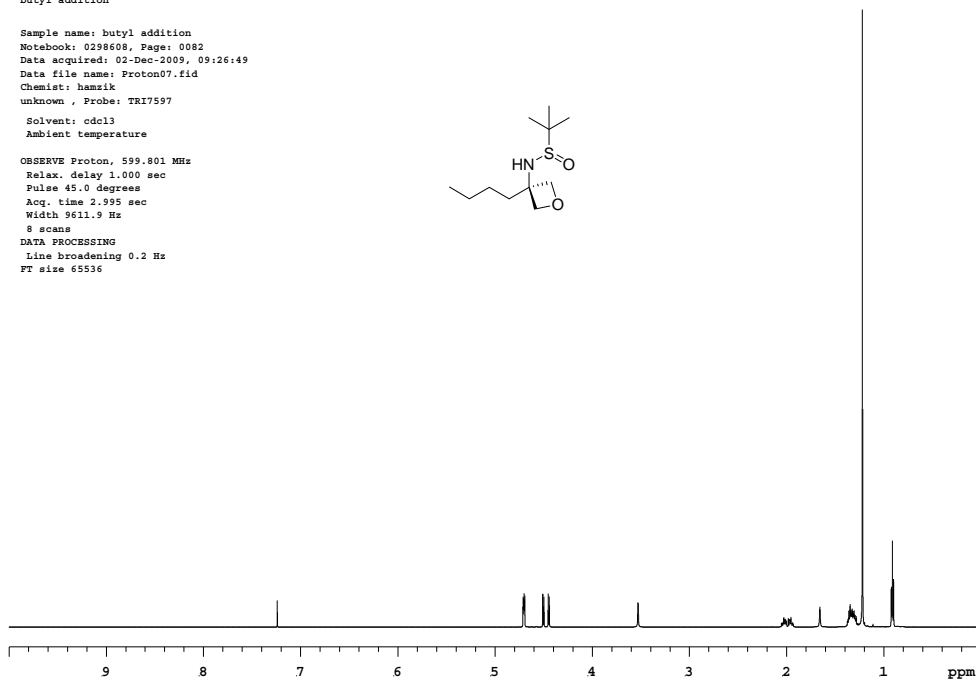
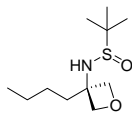
Chemist: hamzik; Notebook: 0298608; Page: 0045

Plotted: 11/03 09:18:52

Restricted Confidential, Limited Access

butyl addition

Sample name: butyl addition
Notebook: 0298608, Page: 0082
Data acquired: 02-Dec-2009, 09:26:49
Data file name: Proton07.fid
Chemist: hamzik
unknown, Probe: TRI7597
Solvent: cdcl3
Ambient temperature
OBSERVE Proton, 599.801 MHz
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.995 sec
Width 9611.9 Hz
8 scans
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536



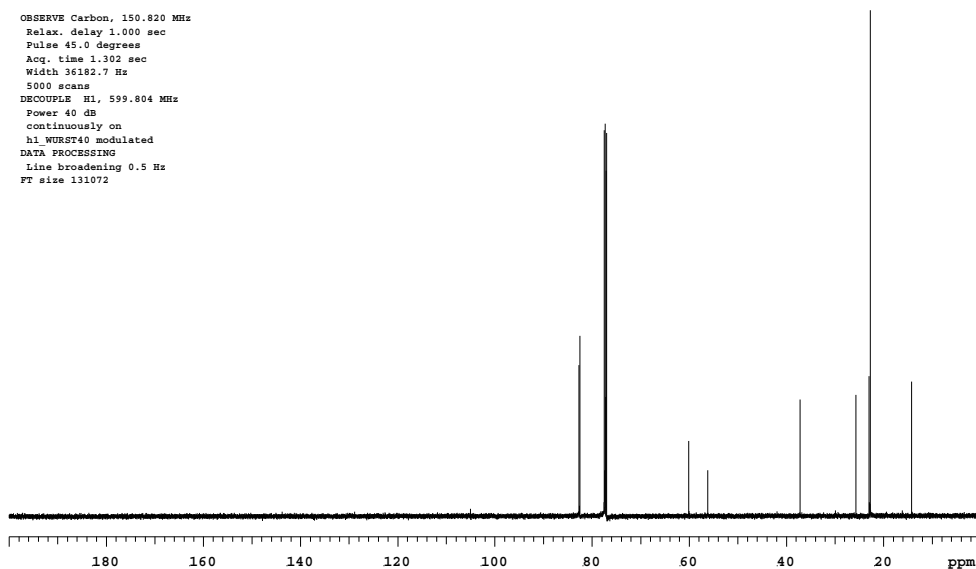
Chemist: hamzik; Notebook: 0298608; Page: 0082

Plotted: 12/02 09:28:44

Restricted Confidential, Limited Access

butyl addition carbon

Sample name: butyl addition carbon
Notebook: 0298608, Page: 0082
Data acquired: 01-Dec-2009, 19:03:19
Data file name: Carbon02.fid
Data reprocessed: Wed Dec 2 09:18:32 EST 2009
Chemist: hamzik
unknown, Probe: TRI7597
Solvent: cdcl3
Ambient temperature
OBSERVE Carbon, 150.820 MHz
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.302 sec
Width 36182.7 Hz
5000 scans
DECOUPLE H1, 599.804 MHz
Power 40 dB
continuously on
h1 WURST40 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072



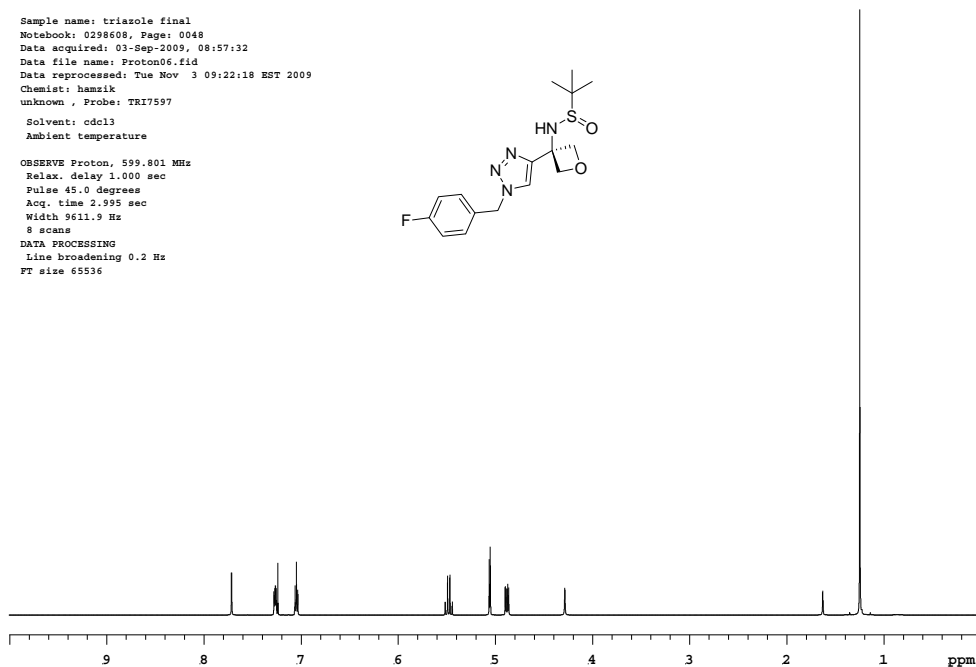
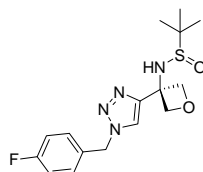
Chemist: hamzik; Notebook: 0298608; Page: 0082

Plotted: 12/02 09:18:33

Restricted Confidential, Limited Access

triazole final

Sample name: triazole final
 Notebook: 0298608, Page: 0048
 Data acquired: 03-Sep-2009, 08:57:32
 Data file name: Proton06.fid
 Data reprocessed: Tue Nov 3 09:22:18 EST 2009
 Chemist: hamzik
 unknown, Probe: TRI7597
 Solvent: cdcl3
 Ambient temperature
 OBSERVE Proton, 599.801 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.995 sec
 Width 9611.9 Hz
 8 scans
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536



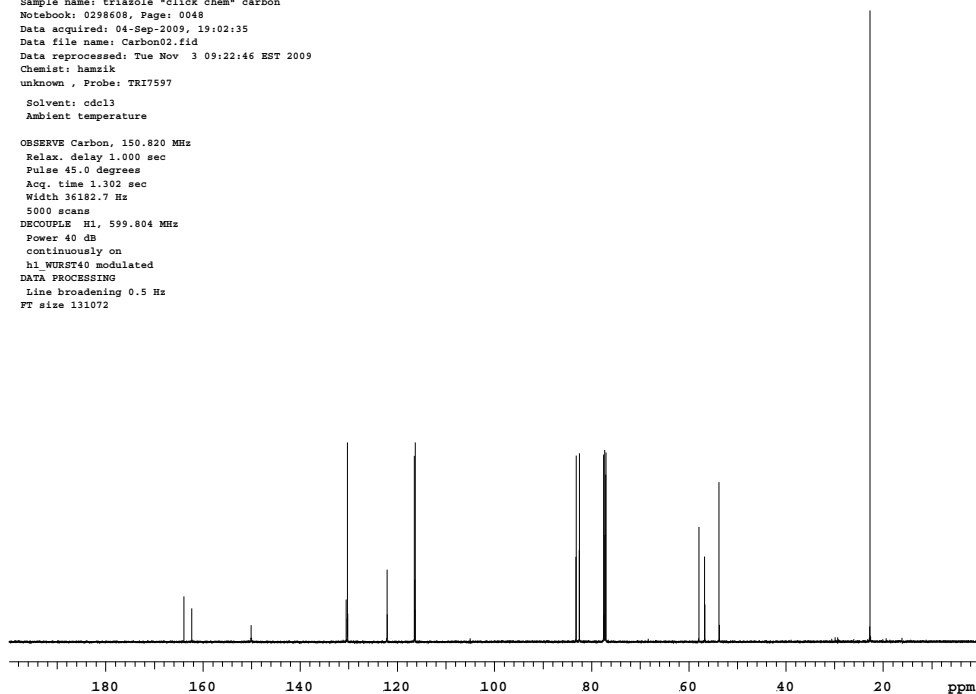
Chemist: hamzik; Notebook: 0298608; Page: 0048

Plotted: 11/03 09:22:18

Restricted Confidential, Limited Access

triazole "click chem" carbon

Sample name: triazole "click chem" carbon
 Notebook: 0298608, Page: 0048
 Data acquired: 04-Sep-2009, 19:02:35
 Data file name: Carbon02.fid
 Data reprocessed: Tue Nov 3 09:22:46 EST 2009
 Chemist: hamzik
 unknown, Probe: TRI7597
 Solvent: cdcl3
 Ambient temperature
 OBSERVE Carbon, 150.820 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.302 sec
 Width 36182.7 Hz
 5000 scans
 DECOUPLE H1, 599.804 MHz
 Power 40 dB
 continuously on
 h1 WURST40 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 131072



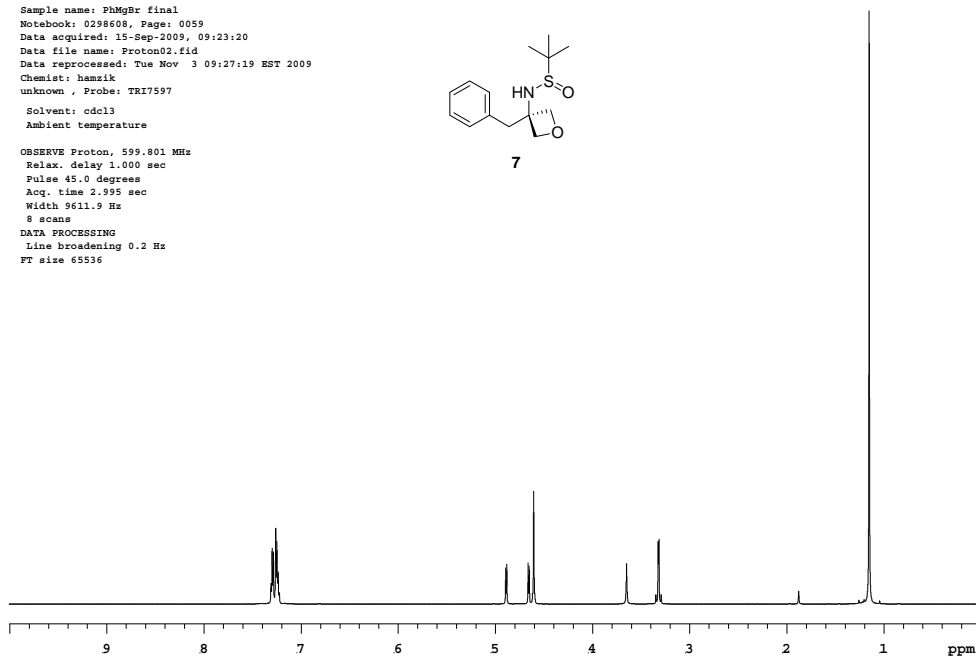
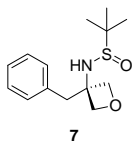
Chemist: hamzik; Notebook: 0298608; Page: 0048

Plotted: 11/03 09:22:46

Restricted Confidential, Limited Access

PhMgBr final

Sample name: PhMgBr final
Notebook: 0298608, Page: 0059
Data acquired: 15-Sep-2009, 09:23:20
Data file name: Proton02.fid
Data reprocessed: Tue Nov 3 09:27:19 EST 2009
Chemist: hamzik
unknown, Probe: TRI7597
Solvent: cdcl3
Ambient temperature
OBSERVE Proton, 599.801 MHz
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.995 sec
Width 9611.9 Hz
8 scans
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536



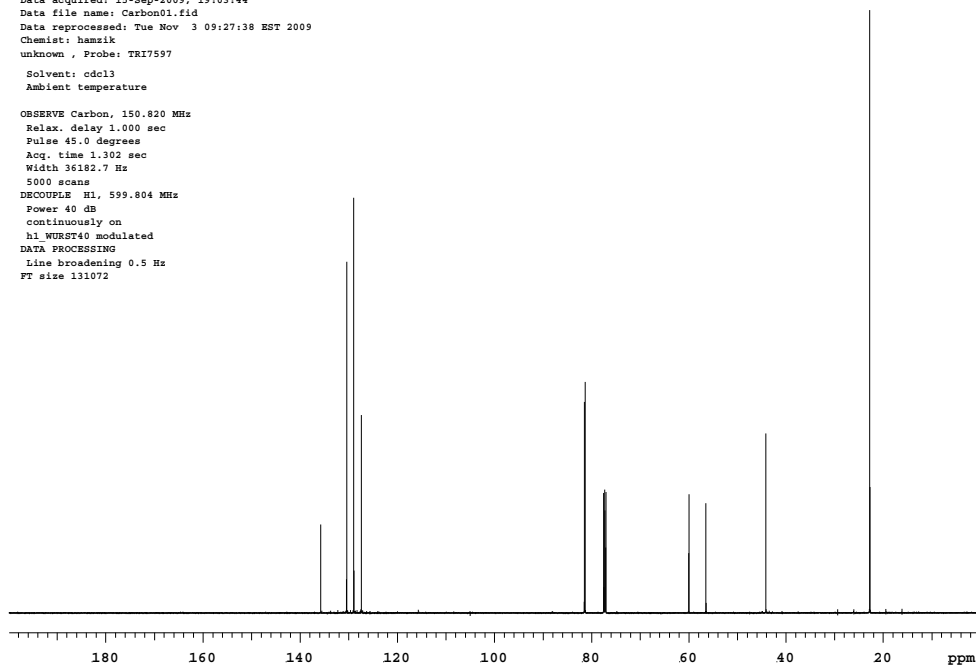
Chemist: hamzik; Notebook: 0298608; Page: 0059

Plotted: 11/03 09:27:19

Restricted Confidential, Limited Access

PhMgBr carbon

Sample name: PhMgBr carbon
Notebook: 0298608, Page: 0059
Data acquired: 15-Sep-2009, 19:03:44
Data file name: Carbon01.fid
Data reprocessed: Tue Nov 3 09:27:38 EST 2009
Chemist: hamzik
unknown, Probe: TRI7597
Solvent: cdcl3
Ambient temperature
OBSERVE Carbon, 150.820 MHz
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.302 sec
Width 36182.7 Hz
5000 scans
DECOUPLE H1, 599.804 MHz
Power 40 dB
continuously on
h1 WURST40 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072



Chemist: hamzik; Notebook: 0298608; Page: 0059

Plotted: 11/03 09:27:38

Restricted Confidential, Limited Access

0298608-0059

Sample name: 0298608-0059
 Notebook: 0298608, Page: 0059
 Data acquired: 08-Dec-2009, 04:54:23
 Data file name: Hsqcad01.fid
 Chemist: hamzik
 inova-600 bloch2, Probe: ID7552
 Solvent: cdcl3
 Ambient temperature

OBSERVE Hsqcad, 599.550 MHz
 Relax. delay 1.000 sec
 Acq. time 0.186 sec
 Width 4456.3 Hz
 2D Width 25624.6 Hz
 F1: C13

2 scans

2 x 128 increments

DECOUPLE C13, 150.768 MHz

Power 38 dB

on during acquisition

off during delay

W40 ID7552 modulated

DATA PROCESSING

Gauss apodization 0.186 sec

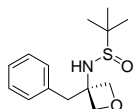
F1 DATA PROCESSING

Gauss apodization 0.002 sec

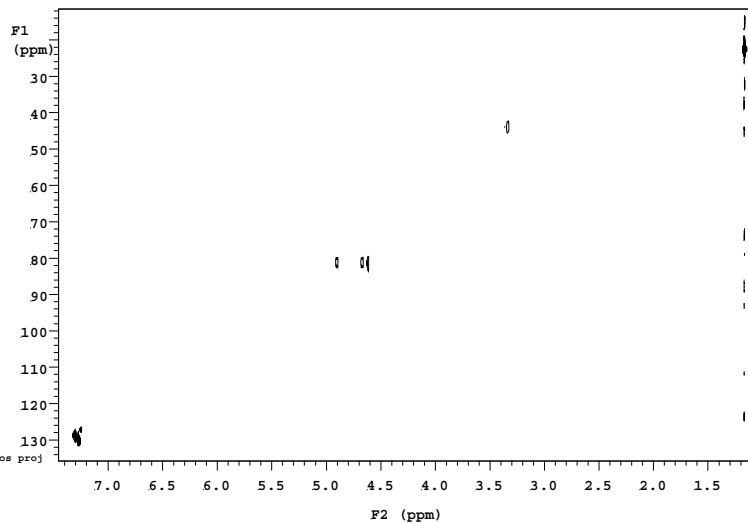
FT size 4096 x 2048

neg proj

pos proj



7
HSQC



Chemist: hamzik; Notebook: 0298608; Page: 0059

Plotted: 12/08 05:32:39

Restricted Confidential, Limited Access

0298608-0059

Sample name: 0298608-0059
 Notebook: 0298608, Page: 0059
 Data acquired: 08-Dec-2009, 04:54:28
 Data file name: Ghmbc01.fid
 Chemist: hamzik
 inova-600 bloch2, Probe: ID7552
 Solvent: cdcl3
 Ambient temperature

OBSERVE Ghmbc, 599.550 MHz

juxh = 8

Relax. delay 1.000 sec

Acq. time 0.300 sec

Width 4456.3 Hz

2D Width 30154.4 Hz

F1: C13

8 scans

200 increments

DATA PROCESSING

Gauss apodization 0.150 sec

F1 DATA PROCESSING

Sine bell 0.010 sec

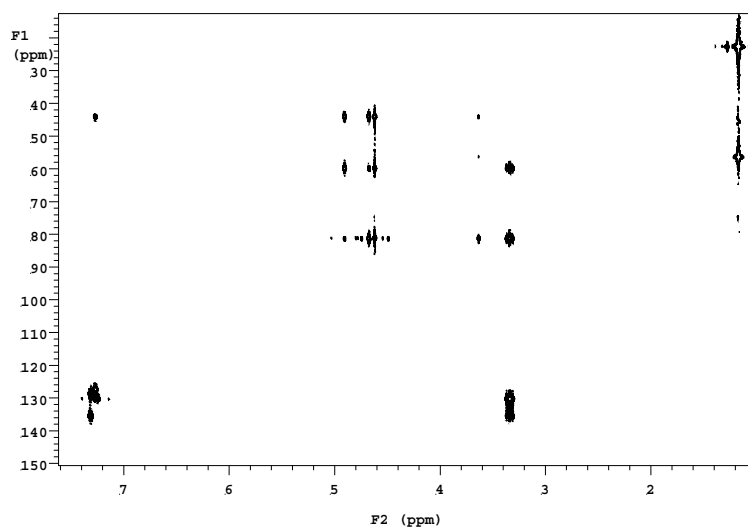
Shifted by -0.007 ppm

FT size 4096 X 2048

neg proj

pos proj

HMBC



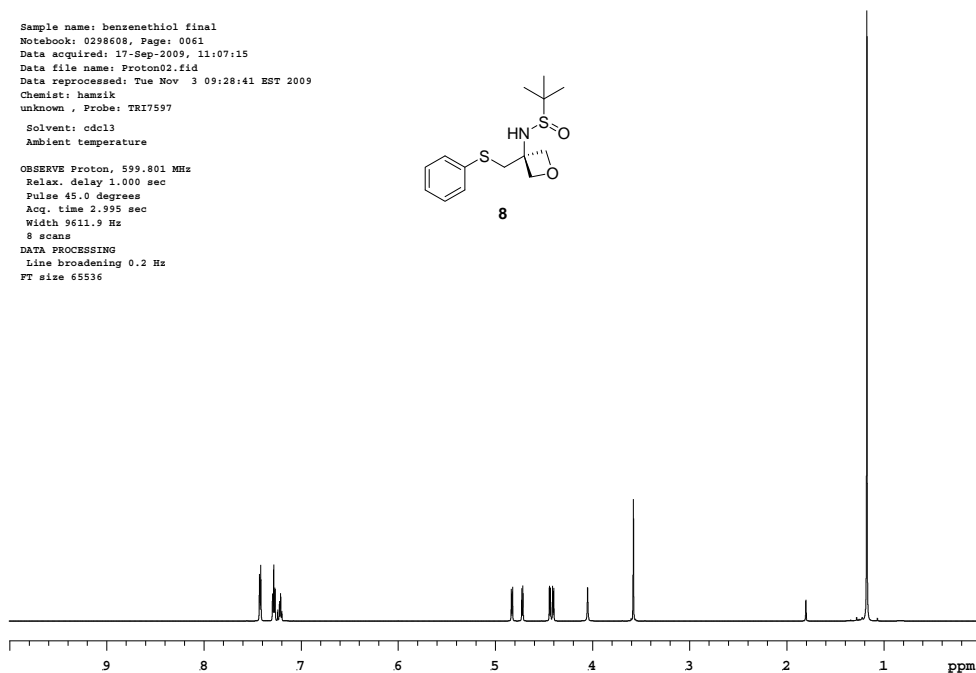
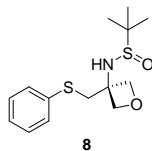
Chemist: hamzik; Notebook: 0298608; Page: 0059

Plotted: 12/08 06:05:38

Restricted Confidential, Limited Access

benzenethiol final

Sample name: benzenethiol final
 Notebook: 0298608, Page: 0061
 Data acquired: 17-Sep-2009, 11:07:15
 Data file name: Proton02.fid
 Data reprocessed: Tue Nov 3 09:28:41 EST 2009
 Chemist: hamzik
 unknown, Probe: TRI7597
 Solvent: cdcl3
 Ambient temperature
 OBSERVE Proton, 599.801 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.995 sec
 Width 9611.9 Hz
 8 scans
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536



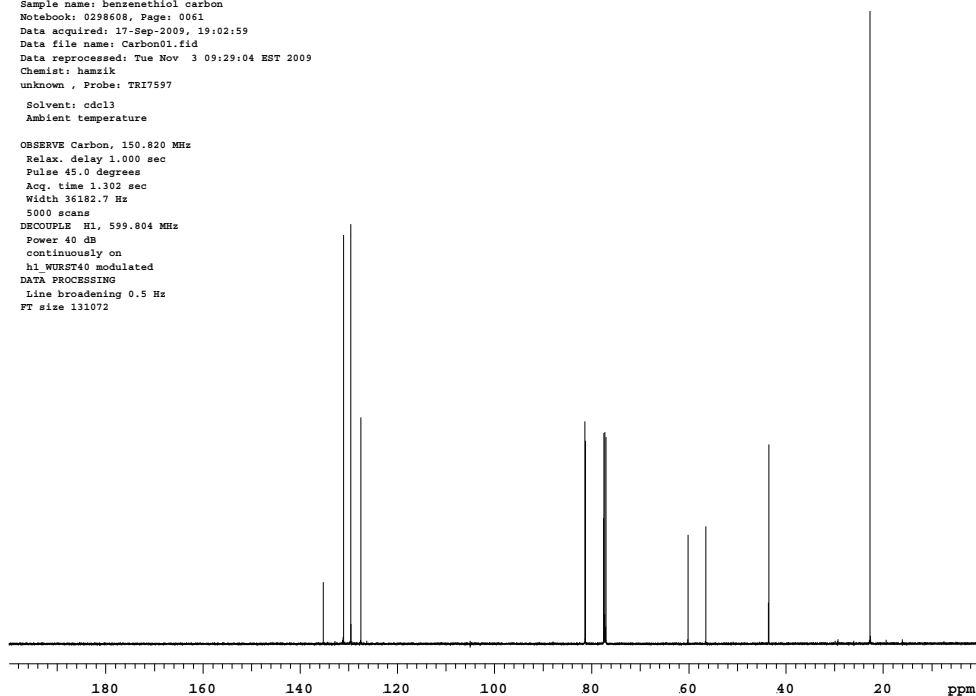
Chemist: hamzik; Notebook: 0298608; Page: 0061

Plotted: 11/03 09:28:41

Restricted Confidential, Limited Access

benzenethiol carbon

Sample name: benzenethiol carbon
 Notebook: 0298608, Page: 0061
 Data acquired: 17-Sep-2009, 19:02:59
 Data file name: Carbon01.fid
 Data reprocessed: Tue Nov 3 09:29:04 EST 2009
 Chemist: hamzik
 unknown, Probe: TRI7597
 Solvent: cdcl3
 Ambient temperature
 OBSERVE Carbon, 150.820 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.302 sec
 Width 36182.7 Hz
 5000 scans
 DECOUPLE H1, 599.804 MHz
 Power 40 dB
 continuously on
 h1 WURST40 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 131072



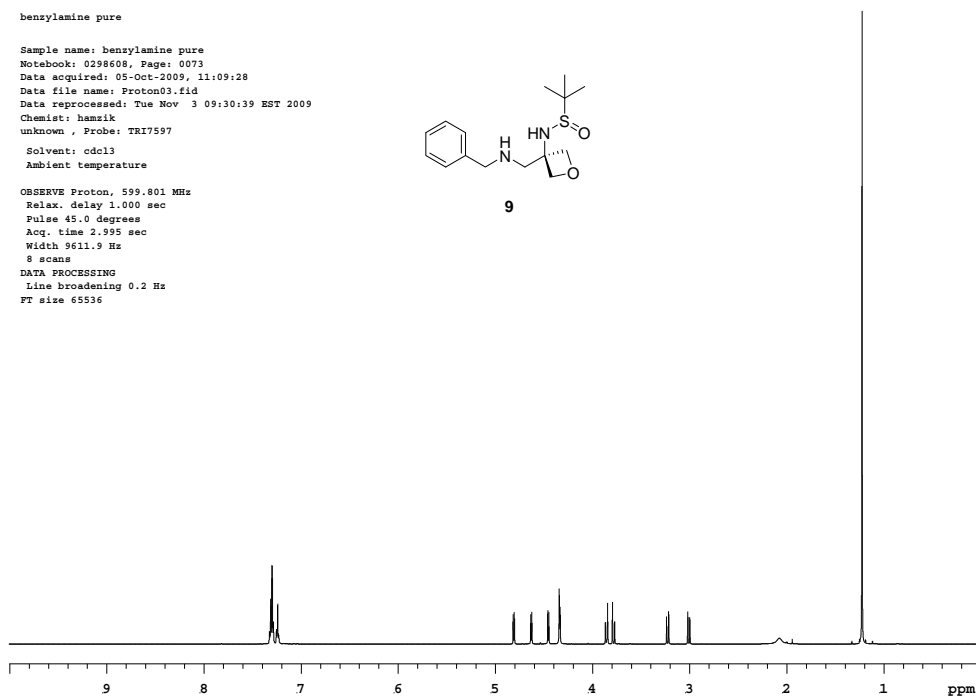
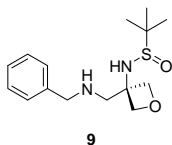
Chemist: hamzik; Notebook: 0298608; Page: 0061

Plotted: 11/03 09:29:04

Restricted Confidential, Limited Access

benzylamine pure

Sample name: benzylamine pure
 Notebook: 0298608, Page: 0073
 Data acquired: 05-Oct-2009, 11:09:28
 Data file name: Proton03.fid
 Data reprocessed: Tue Nov 3 09:30:39 EST 2009
 Chemist: hamzik
 unknown, Probe: TRI7597
 Solvent: cdcl3
 Ambient temperature
 OBSERVE Proton, 599.801 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.995 sec
 Width 9611.9 Hz
 8 scans
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536



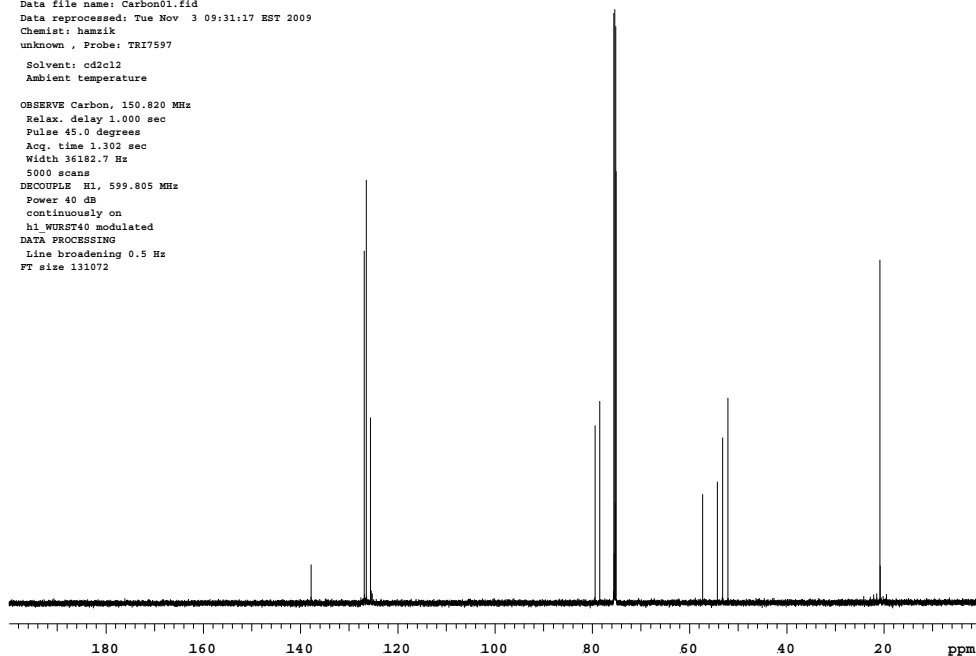
Chemist: hamzik; Notebook: 0298608; Page: 0073

Plotted: 11/03 09:30:40

Restricted Confidential, Limited Access

benzylamine carbon

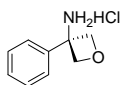
Sample name: benzylamine carbon
 Notebook: 0298608, Page: 0072
 Data acquired: 05-Oct-2009, 21:42:17
 Data file name: Carbon01.fid
 Data reprocessed: Tue Nov 3 09:31:17 EST 2009
 Chemist: hamzik
 unknown, Probe: TRI7597
 Solvent: cd2cl2
 Ambient temperature
 OBSERVE Carbon, 150.820 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.302 sec
 Width 36182.7 Hz
 5000 scans
 DECOUPLE H1, 599.805 MHz
 Power 40 dB
 continuously on
 h1 WURST40 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 131072



Chemist: hamzik; Notebook: 0298608; Page: 0072

Plotted: 11/03 09:31:17

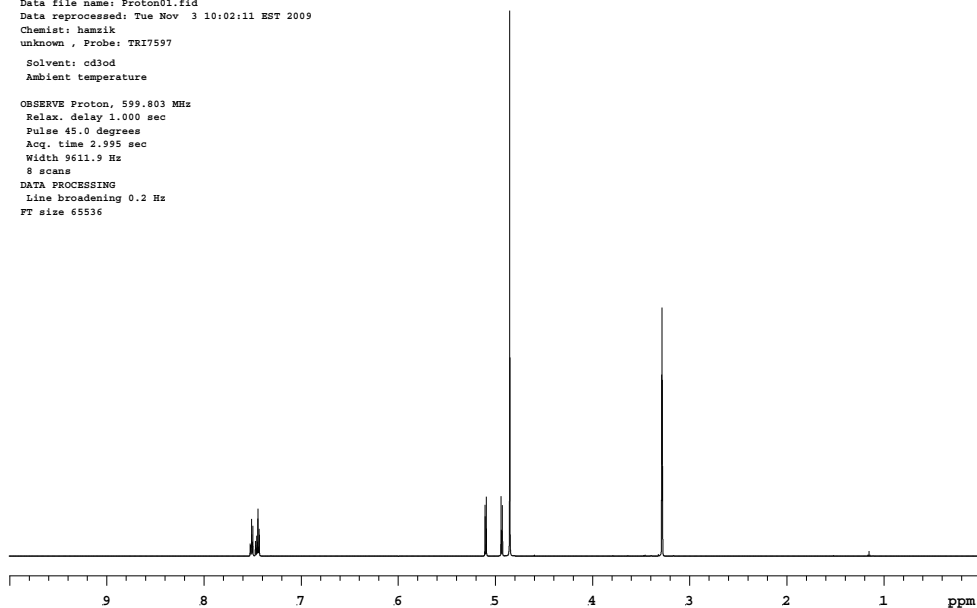
Restricted Confidential, Limited Access



3

phenyl deprotection final

Sample name: phenyl deprotection final
 Notebook: 0298608, Page: 0102
 Data acquired: 21-Oct-2009, 16:43:21
 Data file name: Proton01.fid
 Data reprocessed: Tue Nov 3 10:02:11 EST 2009
 Chemist: hamzik
 unknown, Probe: TRI7597
 Solvent: cd3od
 Ambient temperature
 OBSERVE Proton, 599.803 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.995 sec
 Width 9611.9 Hz
 8 scans
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536



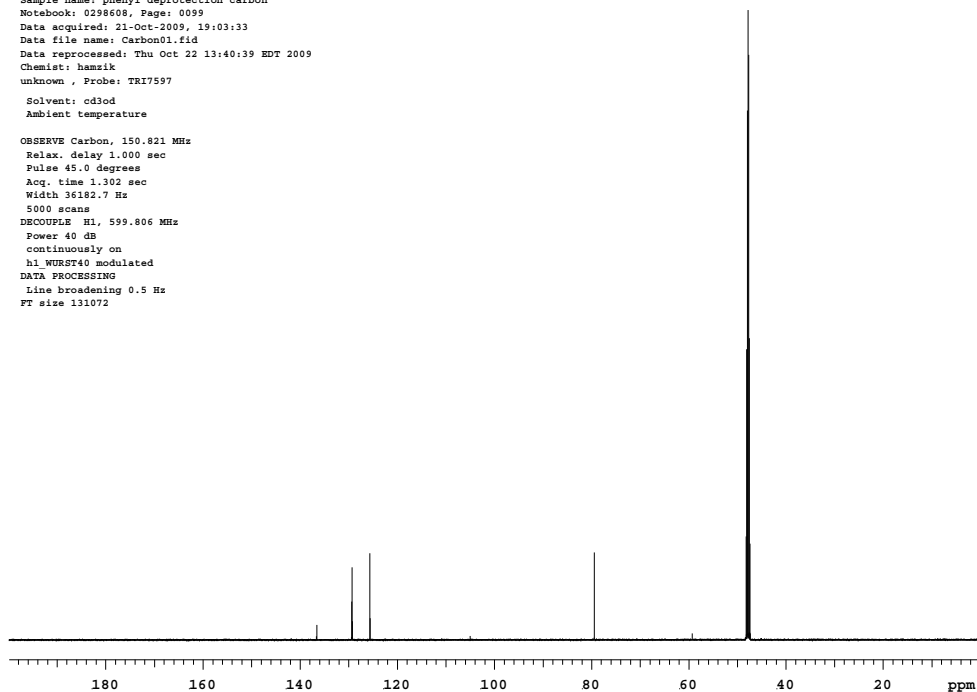
Chemist: hamzik; Notebook: 0298608; Page: 0102

Plotted: 11/03 10:02:12

Restricted Confidential, Limited Access

phenyl deprotection carbon

Sample name: phenyl deprotection carbon
 Notebook: 0298608, Page: 0099
 Data acquired: 21-Oct-2009, 19:03:33
 Data file name: Carbon01.fid
 Data reprocessed: Thu Oct 22 13:40:39 EDT 2009
 Chemist: hamzik
 unknown, Probe: TRI7597
 Solvent: cd3od
 Ambient temperature
 OBSERVE Carbon, 150.821 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.302 sec
 Width 36182.7 Hz
 5000 scans
 DECOUPLE H1, 599.806 MHz
 Power 40 dB
 continuously on
 h1 WURST40 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 131072



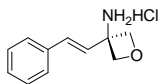
Chemist: hamzik; Notebook: 0298608; Page: 0099

Plotted: 10/22 13:40:40

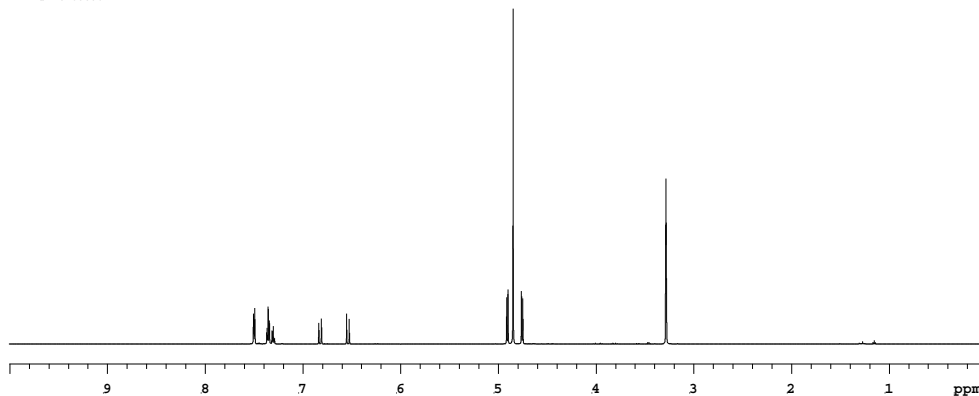
Restricted Confidential, Limited Access

alkene final

Sample name: alkene final
 Notebook: 0298608, Page: 0110
 Data acquired: 02-Nov-2009, 11:16:04
 Data file name: Proton04.fid
 Data reprocessed: Tue Nov 3 10:06:32 EST 2009
 Chemist: hamzik
 unknown, Probe: TRI7597
 Solvent: cd3od
 Ambient temperature



OBSERVE Proton, 599.803 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.995 sec
 Width 9611.9 Hz
 8 scans
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536



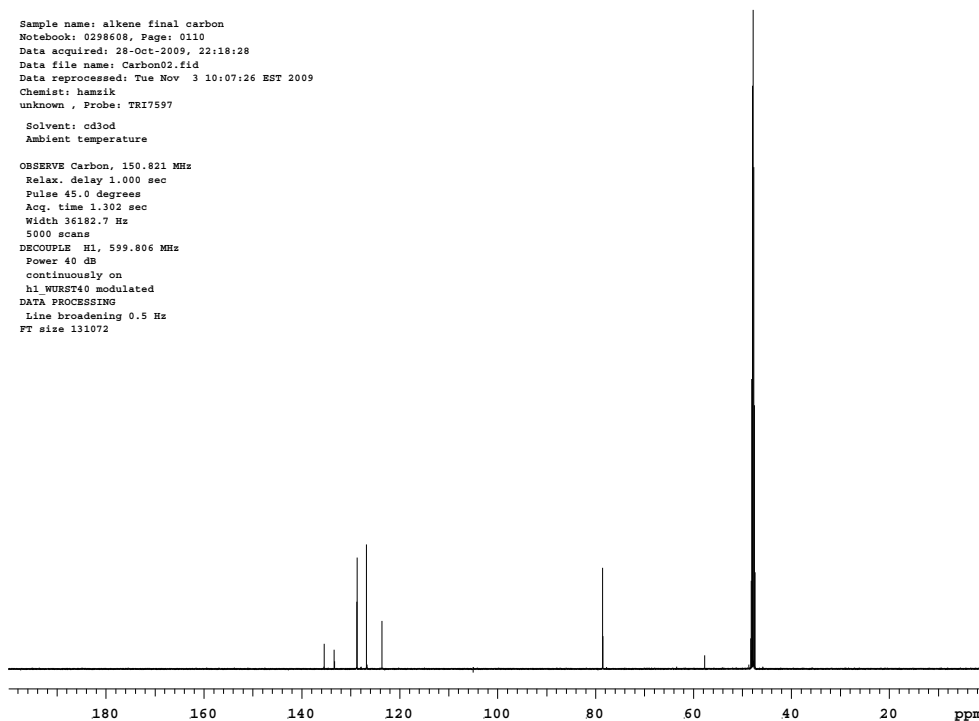
Chemist: hamzik; Notebook: 0298608; Page: 0110

Plotted: 11/03 10:06:32

Restricted Confidential, Limited Access

alkene final carbon

Sample name: alkene final carbon
 Notebook: 0298608, Page: 0110
 Data acquired: 28-Oct-2009, 22:18:28
 Data file name: Carbon02.fid
 Data reprocessed: Tue Nov 3 10:07:26 EST 2009
 Chemist: hamzik
 unknown, Probe: TRI7597
 Solvent: cd3od
 Ambient temperature
 OBSERVE Carbon, 150.821 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.302 sec
 Width 36182.7 Hz
 5000 scans
 DECOUPLE H1, 599.806 MHz
 Power 40 dB
 continuously on
 h1 WURST40 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 131072



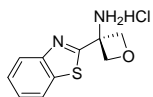
Chemist: hamzik; Notebook: 0298608; Page: 0110

Plotted: 11/03 10:07:26

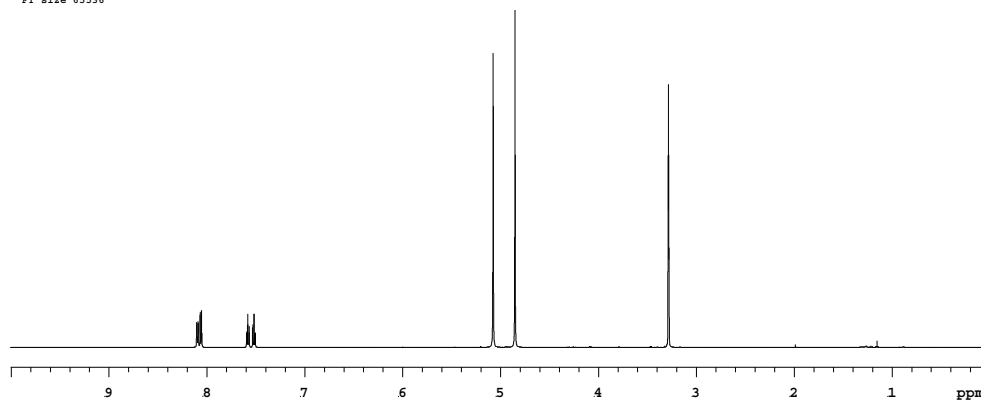
Restricted Confidential, Limited Access

benzothiazole dioxane?

Sample name: benzothiazole dioxane?
 Notebook: 0298608, Page: 0095
 Data acquired: 20-Oct-2009, 14:12:29
 Data file name: Proton04.fid
 Data reprocessed: Tue Nov 3 10:10:46 EST 2009
 Chemist: hamzik
 unknown, Probe: TRI7597
 Solvent: cd3od
 Ambient temperature



OBSERVE Proton, 599.803 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.995 sec
 Width 9611.9 Hz
 8 scans
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536



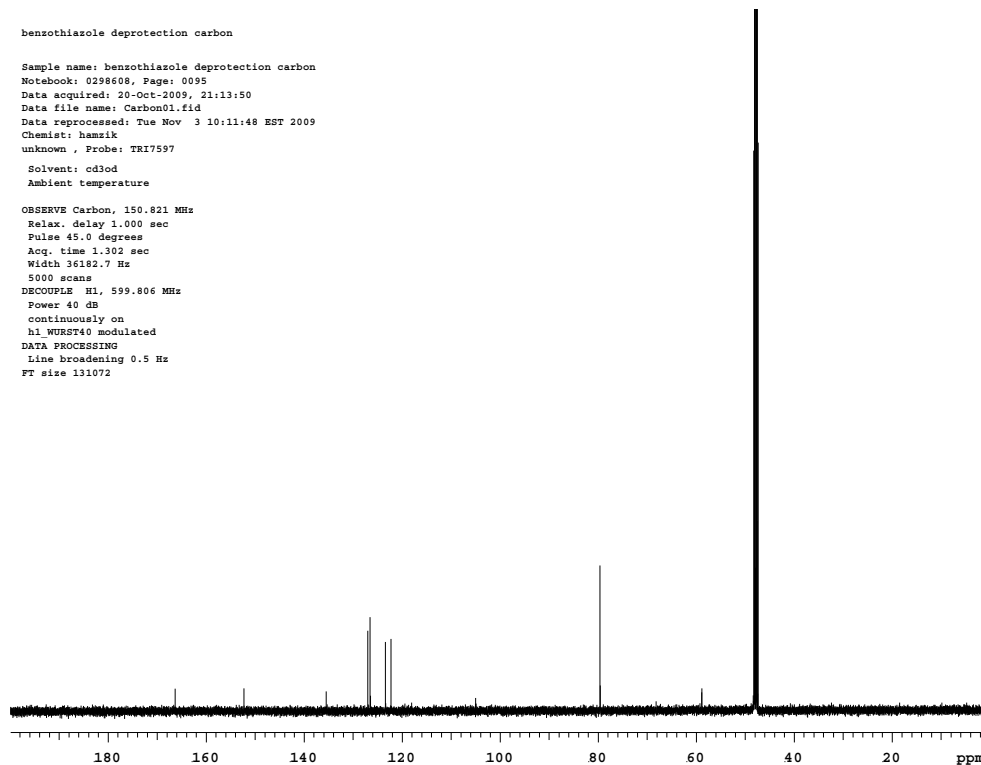
Chemist: hamzik; Notebook: 0298608; Page: 0095

Plotted: 11/03 10:10:46

Restricted Confidential, Limited Access

benzothiazole deprotection carbon

Sample name: benzothiazole deprotection carbon
 Notebook: 0298608, Page: 0095
 Data acquired: 20-Oct-2009, 21:13:50
 Data file name: Carbon01.fid
 Data reprocessed: Tue Nov 3 10:11:48 EST 2009
 Chemist: hamzik
 unknown, Probe: TRI7597
 Solvent: cd3od
 Ambient temperature
 OBSERVE Carbon, 150.821 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.302 sec
 Width 36182.7 Hz
 5000 scans
 DECOUPLE H1, 599.806 MHz
 Power 40 dB
 continuously on
 h1 WURST40 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 131072



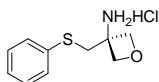
Chemist: hamzik; Notebook: 0298608; Page: 0095

Plotted: 11/03 10:11:48

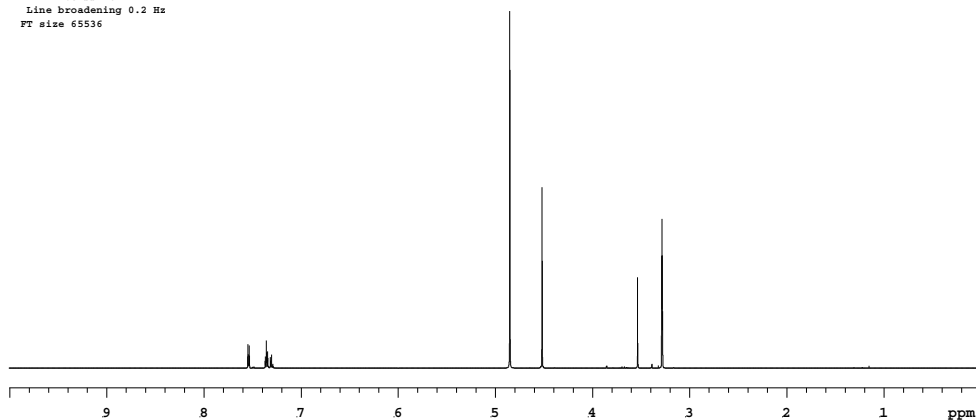
Restricted Confidential, Limited Access

thiol deprotection

Sample name: thiol deprotection
 Notebook: 0298608, Page: 0104
 Data acquired: 22-Oct-2009, 12:43:40
 Data file name: Proton01.fid
 Data reprocessed: Tue Nov 3 10:09:36 EST 2009
 Chemist: hamzik
 unknown, Probe: TRI7597
 Solvent: cd3od
 Ambient temperature



OBSERVE Proton, 599.803 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.995 sec
 Width 9611.9 Hz
 8 scans
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536



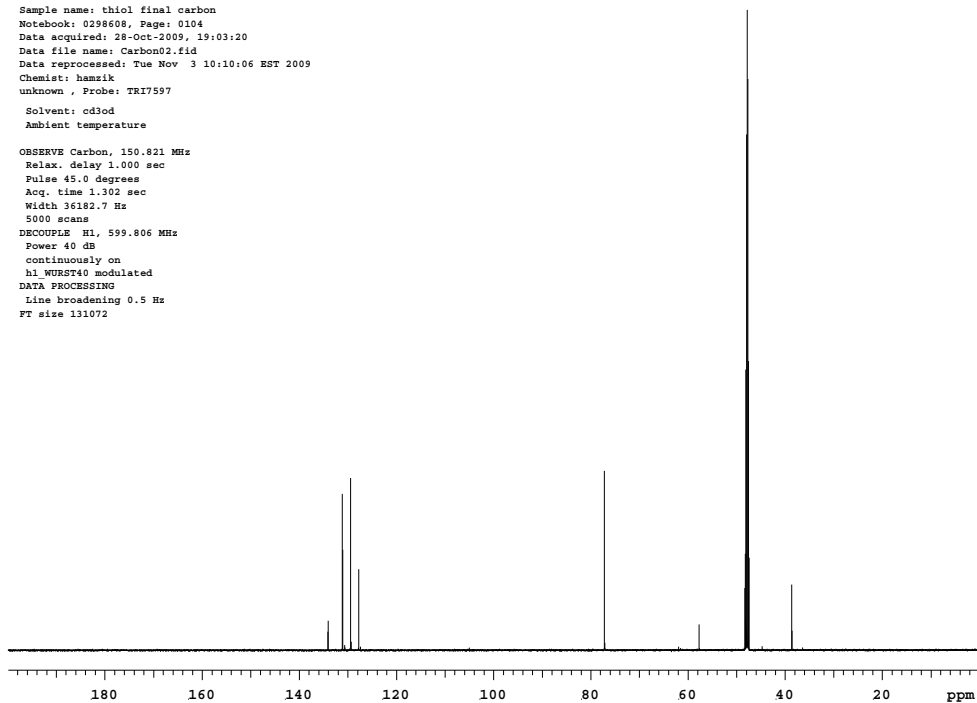
Chemist: hamzik; Notebook: 0298608; Page: 0104

Plotted: 11/03 10:09:36

Restricted Confidential, Limited Access

thiol final carbon

Sample name: thiol final carbon
 Notebook: 0298608, Page: 0104
 Data acquired: 28-Oct-2009, 19:03:20
 Data file name: Carbon02.fid
 Data reprocessed: Tue Nov 3 10:10:06 EST 2009
 Chemist: hamzik
 unknown, Probe: TRI7597
 Solvent: cd3od
 Ambient temperature
 OBSERVE Carbon, 150.821 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.302 sec
 Width 36182.7 Hz
 5000 scans
 DECOUPLE H1, 599.806 MHz
 Power 40 dB
 continuously on
 h1_WURST40 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 131072



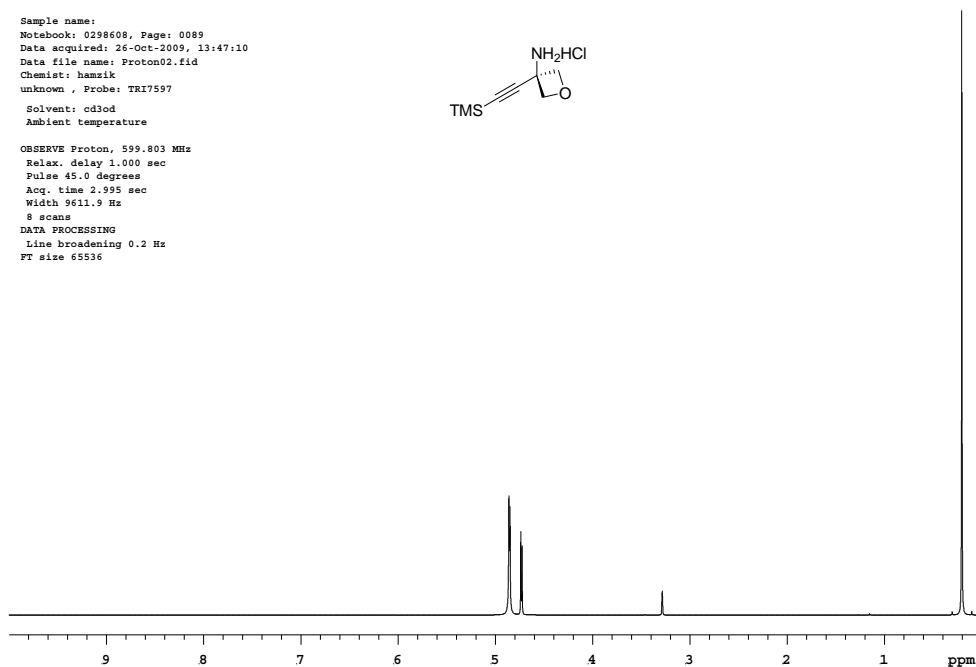
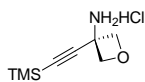
Chemist: hamzik; Notebook: 0298608; Page: 0104

Plotted: 11/03 10:10:06

Restricted Confidential, Limited Access

Sample name:
 Notebook: 0298608, Page: 0089
 Data acquired: 26-Oct-2009, 13:47:10
 Data file name: Proton02.fid
 Chemist: hamzik
 unknown, Probe: TRI7597
 Solvent: cd3od
 Ambient temperature

OBSERVE Proton, 599.803 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.995 sec
 Width 9611.9 Hz
 8 scans
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536



Chemist: hamzik; Notebook: 0298608; Page: 0089

Plotted: 10/26 13:50:45

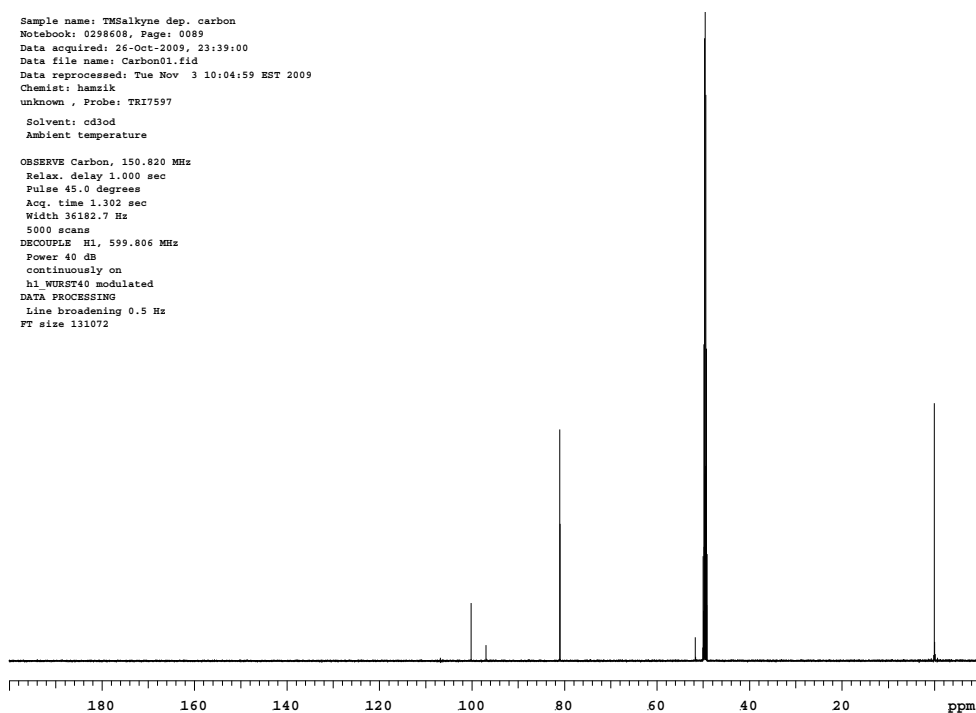
Restricted Confidential, Limited Access

TMS alkyne dep. carbon

Sample name: TMSalkyne dep. carbon
 Notebook: 0298608, Page: 0089
 Data acquired: 26-Oct-2009, 23:39:00
 Data file name: Carbon01.fid
 Data reprocessed: Tue Nov 3 10:04:59 EST 2009
 Chemist: hamzik
 unknown, Probe: TRI7597

Solvent: cd3od
 Ambient temperature

OBSERVE Carbon, 150.820 MHz
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.302 sec
 Width 36182.7 Hz
 5000 scans
 DECOUPLE H1, 599.806 MHz
 Power 40 dB
 continuously on
 h1 WURST40 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 131072



Chemist: hamzik; Notebook: 0298608; Page: 0089

Plotted: 11/03 10:04:59

Restricted Confidential, Limited Access

