New Mechanistic Insights into the Copper Catalyzed Ring Expansion of Vinyl Aziridines: Evidence in Support of a Copper(I) Mediated Pathway.

Daniel J. Mack[§] and Jon T. Njardarson[§,†]

[†]Department of Chemistry and Biochemistry, The University of Arizona, 1306 E. University Blvd., Tucson, AZ 85721

[§]Department of Chemistry and Chemical Biology, Baker Laboratory, Cornell University, Ithaca, New York 14853

Supporting Information

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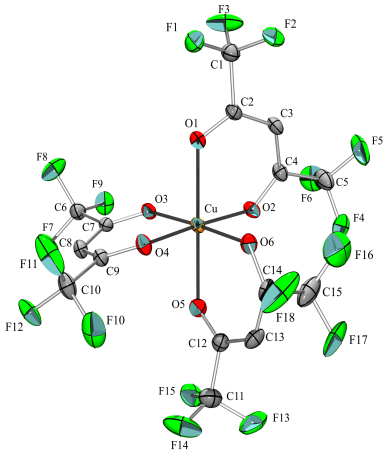
General Information: Commercial reagents were purchased and used without further purification. All glassware was flame dried under vacuum and reactions were performed under a nitrogen atmosphere, unless otherwise stated. Toluene, dichloromethane, diethyl ether, and THF were dried over a column of alumina. Benzene was distilled from sodium/benzophenone ketyl. Flash chromatography was done with Grace Davisil F60 40-63μm 60Å silica, and thin layer chromatography (TLC) was performed with EMD 250 μm silica gel 60-F_{254} plates. ^{1}H and ^{13}C NMR data was acquired on a Varian Inova 600, Bruker DRX 500, or Bruker DRX 600 and referenced to residual protic solvent ($CDCl_3$ ^{1}H [7.26 ppm], ^{13}C [77.00 ppm], C_6F_6 ^{19}F [-164.90 ppm]) or tetramethylsilane (0.00 ppm). Infrared spectrum were acquired on a Shimadzu Prestige FT-IR. Optical rotations were measured on a Rudolph Instruments Autopol IV polarimeter. High-resolution mass spectrometry was performed at The University of Arizona Mass Spectral Facility.

Synthesis of M(hfacac)$_3$NEt$_4$ Crystals:

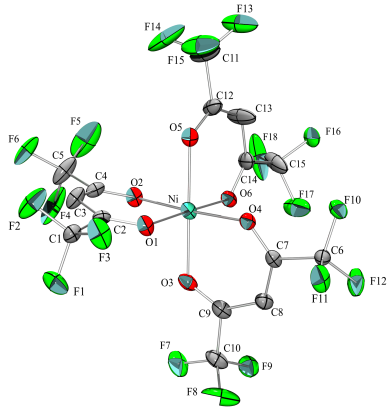
To M(hfacac)$_2$ species (10 mmol) was added dichloromethane (20 mL) and NEt$_4$hfacac[1,2] (10 mmol). The solution was filtered through a 0.45 µm PTFE syringe filter, and distributed in several 20 mL scintillation vials, which were placed in a crystallization dish filled with hexanes, and the crystallization dish was sealed with a glass cover. After slow evaporation, crystals suitable for x-ray diffraction were obtained, and data is below.

Synthesis of Cu(hfacac)$_2$-Aziridine 1 complex:

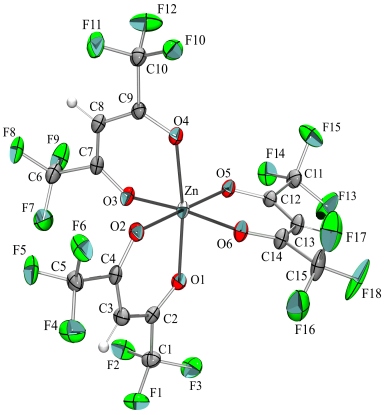
Anhydrous Cu(hfacac)$_2$ (1 mmol) was combined with vinyl aziridine **1** (2 mmol) in dichloromethane (10 mL). The solution was filtered through a 0.45 µm PTFE syringe filter, and X-Ray quality crystals were obtained by the slow evaporation of this solution. Structural data can be found on pages 28-74.



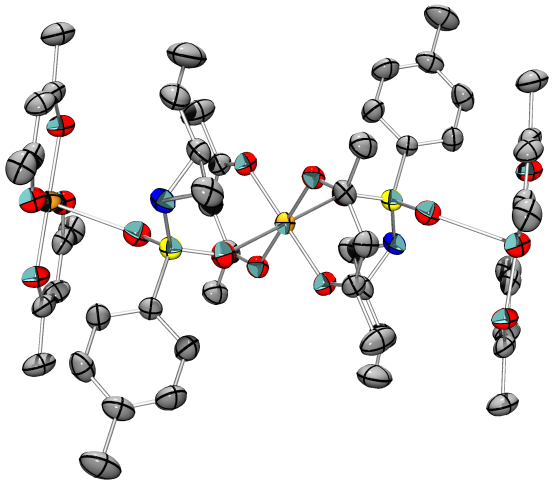
Cu(hfacac)$_3$NEt$_4$



Ni(hfacac)$_3$NEt$_4$



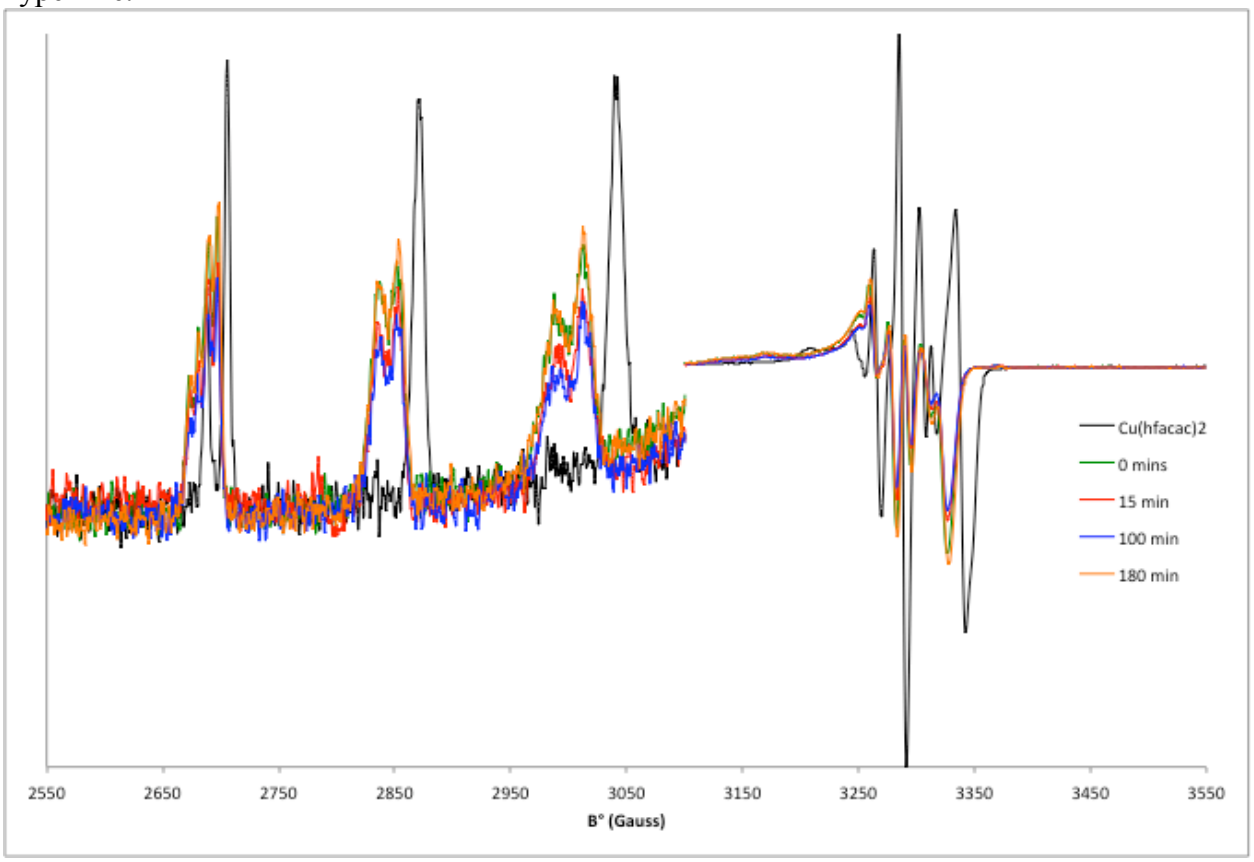
Zn(hfacac)$_3$NEt$_4$


Cu(hfacac)$_2$-Aziridine 1 complex

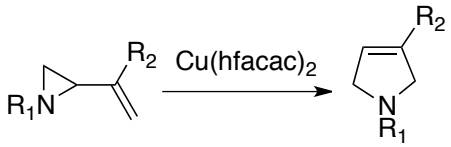
[1] Kitko, D.J.; Wiegers, K.E.; Smith, S.G.; Drago, R.S. *J. Am. Chem. Soc.* **1977**, *99*, 1410.
[2] In the case of M = Ni & Zn, the starting bis hexafluoroacetylacetonate is sparingly soluble in dichloromethane, however immediately after addition of the hfacac salt, the solution becomes fully soluble.

Electron Paramagnetic Resonance Experiments

Equal amounts of a 0.2 M solution of aziridine **1** in toluene and a 0.01 M solution of $Cu(hfacac)_2$ in toluene were added to a flame-dried quartz EPR tube and sealed using a torch. Several of these tubes were produced, and heated at 100°C in a silicon oil bath for various time points, at which they were immediately removed from the oil bath and frozen in liquid nitrogen, yielding a frozen glass suitable for Continuous Wave X-Band EPR analysis, monitoring copper(II) hyperfine.



Copper-catalyzed Rearrangement of Vinyl Aziridines

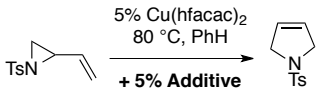


Without Additive

Following the procedure of Njardarson et al.[11], in a flame dried 8 mL threaded vial with stirrer, a 2.0 mL (0.20 M stock solution in benzene) of substrate was combined with 2.0 mL (0.01 M stock solution in benzene) of $Cu(hfacac)_2$ [3] (total substrate concentration 0.1 M, total catalyst concentration 0.005 M), and the vial was sealed with a Teflon-lined cap. The reaction was heated at the listed temperature for the listed time. The reaction was removed from heat, and after the reaction had completely cooled, it was filtered through neutral alumina (MP Bio Activity Grade I), and concentrated. Conversions were determined by ^1H integration.[4]

With Additive

Following the same procedure as above, metal additive was added as a solid after the substrate and copper solutions had been combined. The vial was sealed with a Teflon-lined cap. The reaction was heated at the listed temperature for the listed time. The reaction was removed from heat, and after the reaction had completely cooled, it was filtered through neutral alumina (MP Bio Activity Grade I), and concentrated. Conversions were determined by ^1H integration.

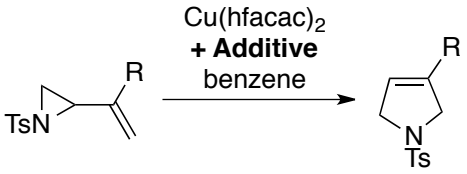


Additive	% Conversion
NONE	0
$Ni(hfacac)_2$	65
$Ni(tfacac)_2$	12
$Ni(acac)_2$	9
$Co(hfacac)_2$	66
$Co(tfacac)_2$	30
$Co(acac)_2$	11
$Zn(hfacac)_2$	91
$Zn(hfacac)_2$	69
$Zn(tfacac)_2$	82
$Zn(acac)_2$	76
$Mn(hfacac)_2$	21
$Pd(hfacac)_2$	28
$Fe(acac)_2$	0

Table 1: Metal Additive Effect on **1**.

[3] $Cu(hfacac)_2$-H_2O (teal powder) was placed under high vacuum until a complete color change had occurred to indicate anhydrous $Cu(hfacac)_2$ (violet powder).

[4] 90° pulse, delay 56 seconds, acquisition 4 seconds, to ensure complete relaxation between scans. Before integration, peaks were phased, and a baseline correction was applied.



Substituent (R)	Additive	Temperature (°C)	Time (hr)	Conversion (%)
Methyl (**SI 29**)	NONE	80	4	22
	5% $Zn(hfacac)_2$			72
Phenyl (**SI 17**)	NONE	80	1	25
	5% $Zn(hfacac)_2$			75
	10% $Zn(hfacac)_2$			90
OTBS (**SI 25**)	NONE	80	8	11
	5% $Zn(hfacac)_2$			92
CF_3 (**SI 27**)	NONE	100	10	27
	5% $Zn(hfacac)_2$			42
OTf (**SI 26**)	NONE	120	6	78
	5% $Zn(hfacac)_2$			58

Table 2: Exploring $Zn(hfacac)_2$ effect on olefin substituents.

Procedure For NMR Kinetics Experiments

Sample Preparation:

Following the general procedure for copper-catalyzed rearrangement of vinyl aziridines, in a flame dried NMR tube, with a needle inserted with a flow of dry nitrogen, a stock solution of vinyl aziridine (0.20 M) in d_6-benzene[5] was added to the tube. The substrate was frozen, followed by the addition of a stock solution of copper catalyst (0.01 M) . The catalyst portion was frozen, and the tube was sealed at this point (total concentration of substrate 0.10 M and total catalyst concentration 0.005 M).

For the $Zn(hfacac)_2$ experiments, $Zn(hfacac)_2$ was added (5 mol%) as a dispersion in d_6-benzene[6] (sonicated to maximize dispersion) after substrate and catalyst had been frozen, and the NMR tube was sealed with a torch.

For the cobaltocene experiments, cobaltocene (purchased new from Stem Chemicals Inc.) was pulverized into a fine powder in a nitrogen atmosphere glovebox, placed in a Schlenk vessel, and brought outside the glove box. The cobaltocene was added (25 mol%) as a dispersion (sonicated to maximize dispersion) in d_6-benzene to the frozen substrate and catalyst, and The NMR tube was sealed with a torch

For tetrakis(dimethylamino)ethylene and tin(II) 2-ethylhexanoate experiments, the reductants were added neat (TDAE 5 mol%, $Sn(2\text{-}EH)_2$ 25 mol%) via microliter syringe to the frozen sample and catalyst, and the NMR tube was sealed with a torch.

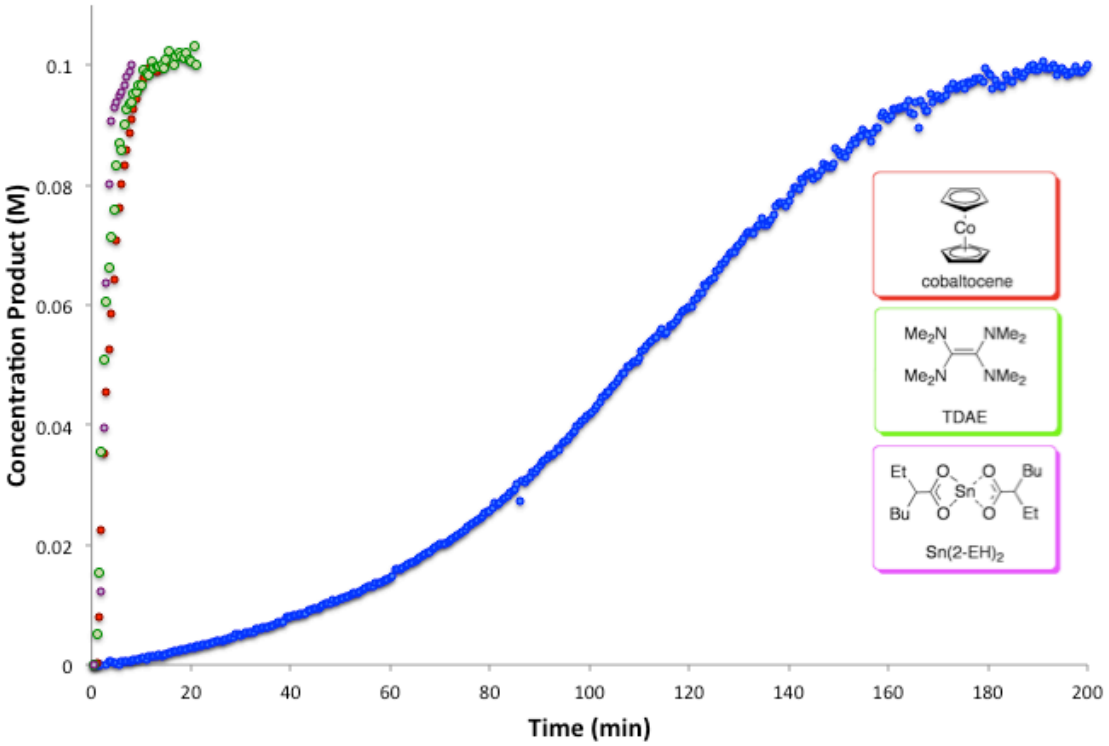
All (hfacac)Cu(COD) (Gelest) and (hfacac)Cu(BTMSA) (Aldrich) were purchased new and stored in an inert atmosphere glovebox. Copper(I) catalyst stock solutions were prepared in the glovebox in Schlenk vessels, and brought outside of the glovebox for NMR sample preparation.

The kinetics were collected on a Bruker DRX 500 system, with a BBO-Z axis gradient probe. Temperatures were calibrated using the shift difference method of 100% ethylene glycol.[7] The experiment was run in TopSpin 1.3 as a pseudo-2D experiment, with ns = 4, vd = 15, td = 8192, aq = 2.5. The sample was inserted frozen, and the acquisition was immediately started. The data was processed by phasing the last spectra of the kinetic run, integrating the peak, and using the T1/T2 relaxation delay module to extract the integrals for the entire run.

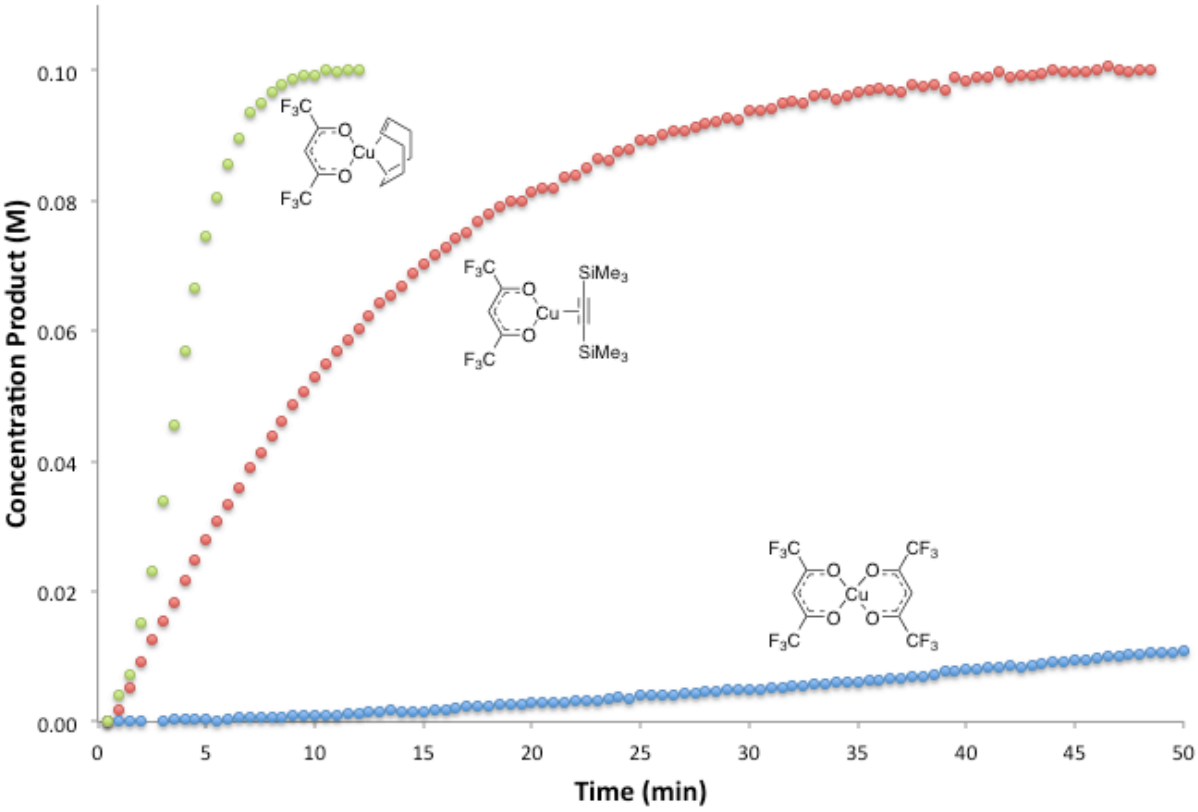
[5] d_6-benzene was refluxed over sodium for several hours, and subsequently degassed by four freeze-pump-thaw cycles, and stored in a Schlenk vessel over activated 4Å sieves.

[6] When additives were added as solutions in d_6-benzene, the molarities of the stock solutions of substrate and catalyst were adjusted for a total concentration of substrate of 0.10 M and total catalyst concentration of 0.005 M.

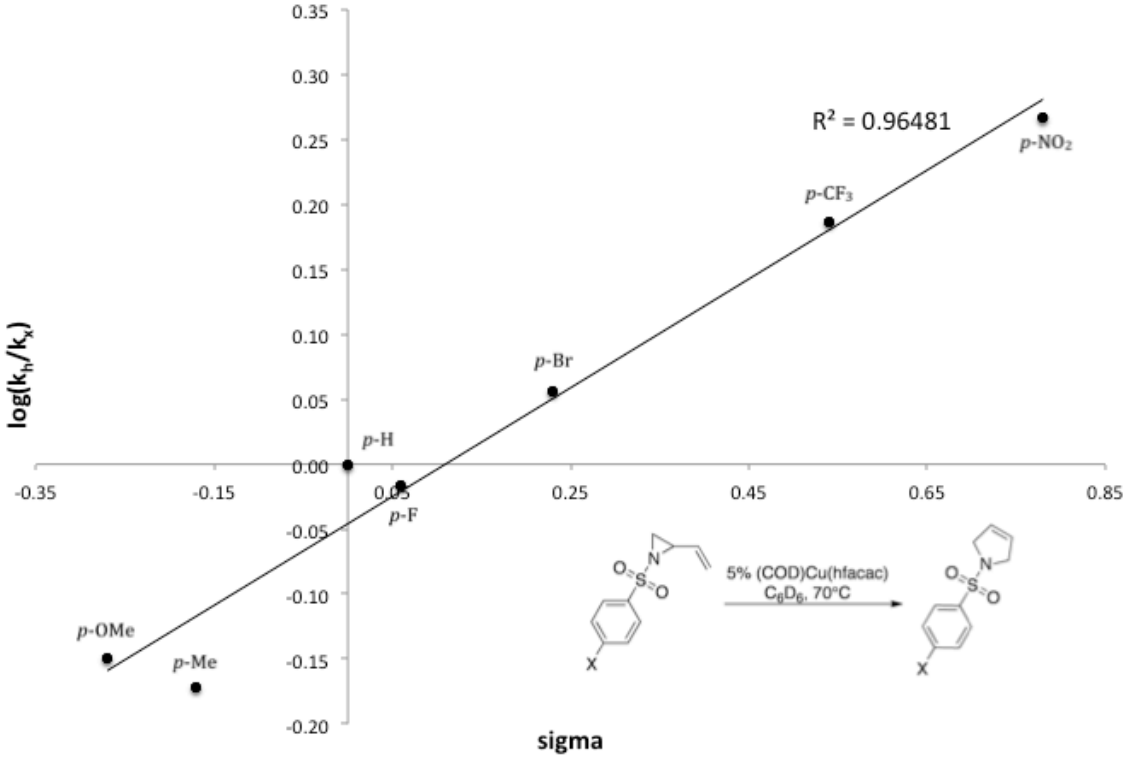
[7] Bruker Variable Temperature instruction manual.



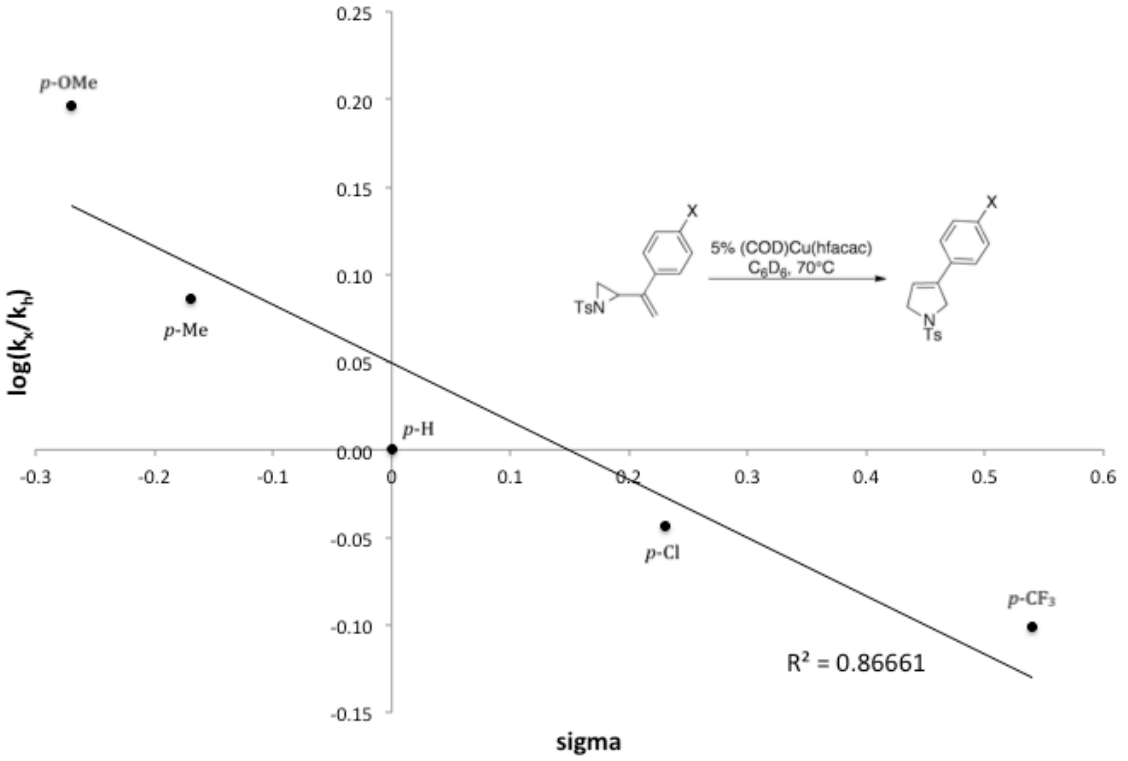
Scheme 1: Kinetic traces of rearrangement of **1** with and without added reductants.



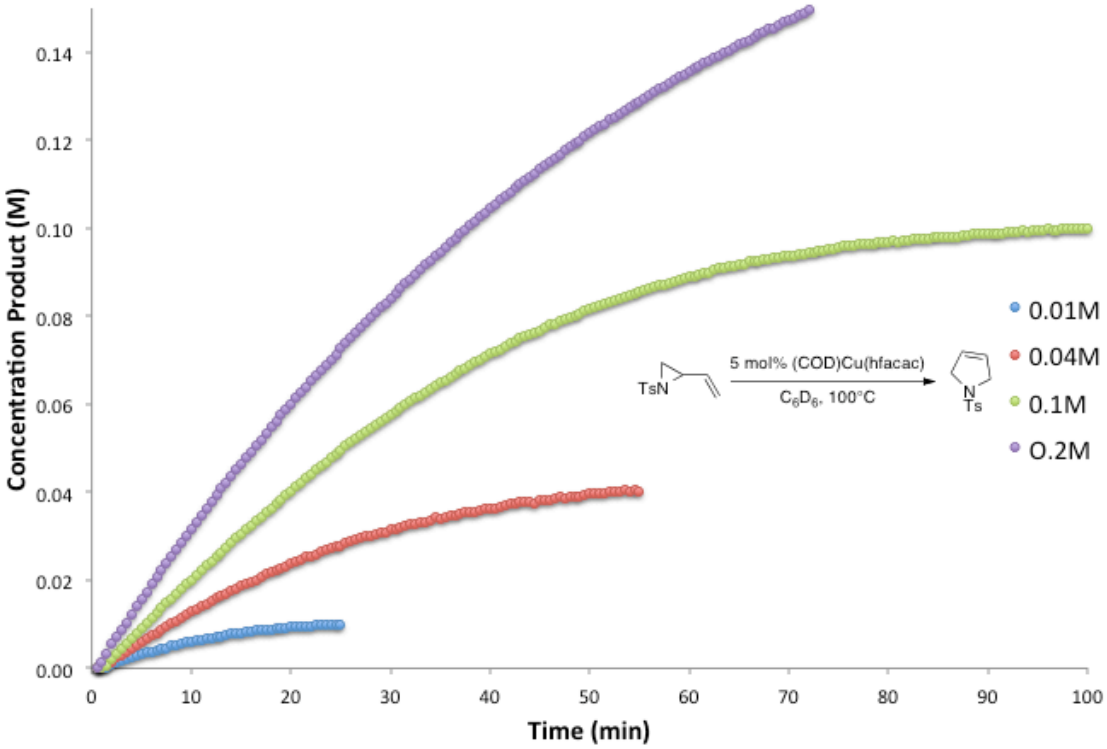
Scheme 2: Kinetic traces of rearrangement of **1** with different catalysts.



Scheme 3: Hammett plot of *para*-substituted sulfonamide substrates with (COD)Cu(hfacac).



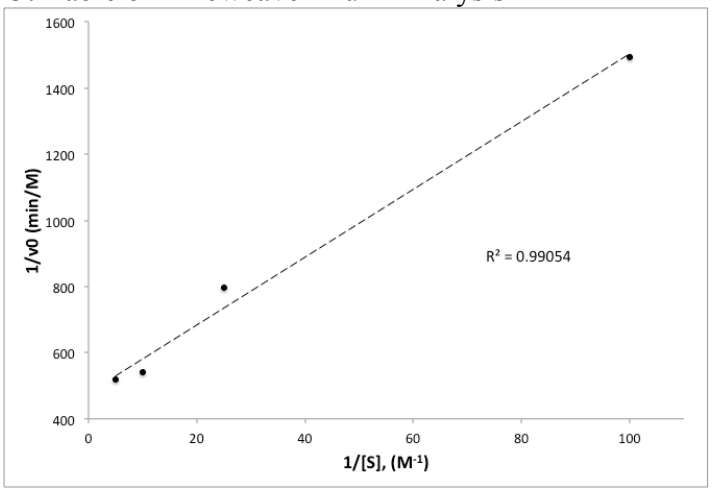
Scheme 4: Hammett plot of *para*-substituted aryl olefin substrates with (COD)Cu(hfacac).



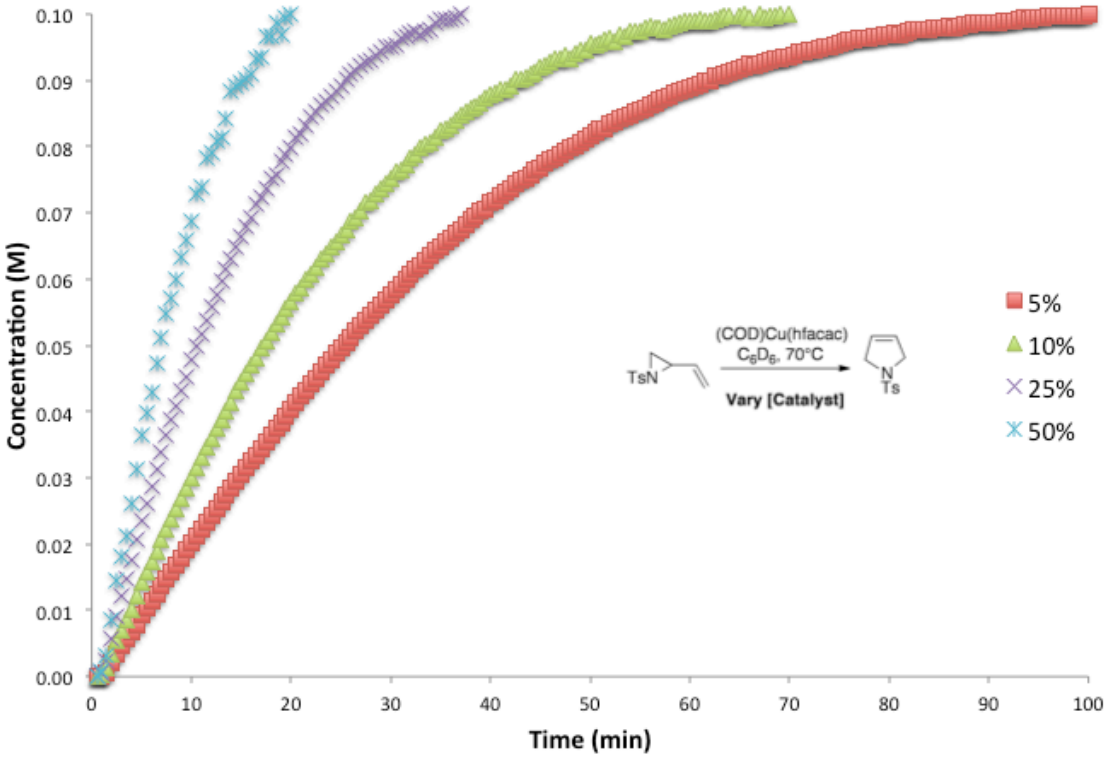
Scheme 5: Kinetic traces of varied substrate concentration **1**.

[sub] (M)	V^0 ($M\text{-}s^{-1}$)	range (min)	R^2	Lineweaver-Burk Parameters
0.01	$1.1x10^{-5}$	0.5-10	0.996	K_M (M) = $2.1x10^{-2}$
0.04	$2.1x10^{-5}$	0.5-20	0.997	V_{max} (Ms^{-1}) = $3.5x10^{-5}$
0.1	$3.1x10^{-5}$	0.5-40	0.996	R^2 = 0.995
0.2	$3.2x10^{-5}$	0.5-80	0.984	K_{cat}/K_M ($M^{-1}s^{-1}$) = 0.33

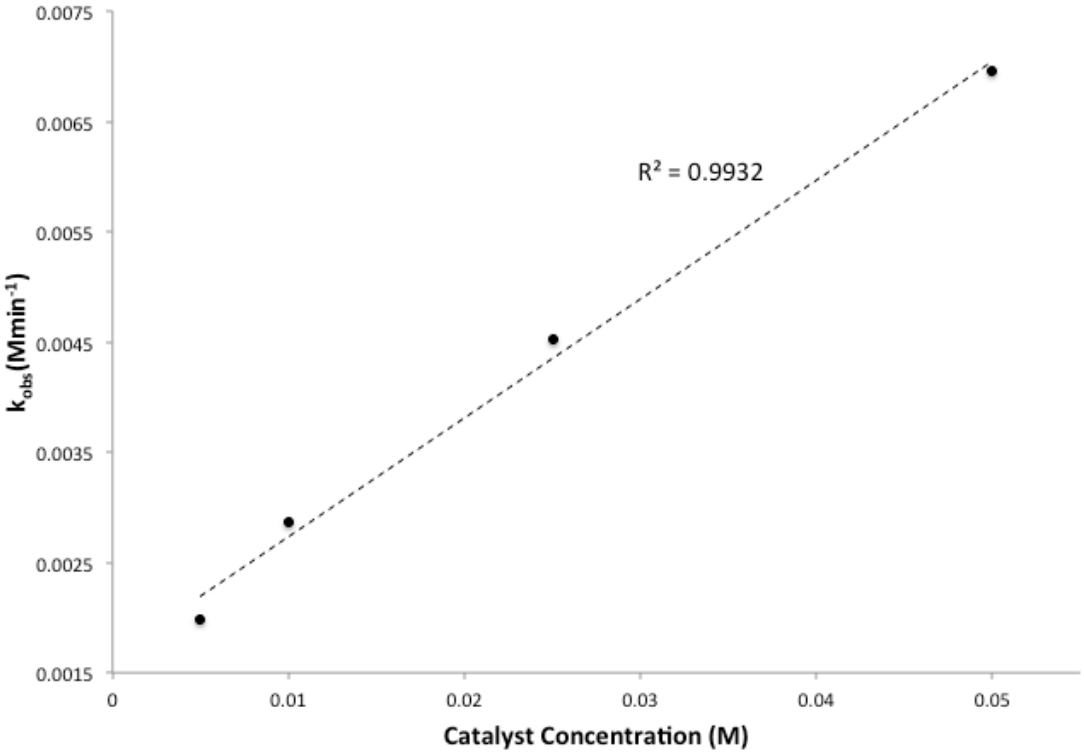
Table 3: Table of Lineweaver-Burk Analysis



Scheme 6: Lineweaver-Burk plot.

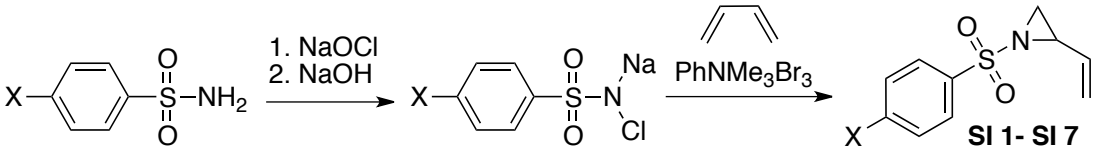


Scheme 6: Kinetic traces of varied catalyst concentration with **1**.



Scheme 7: Plot of k_{obs} vs. catalyst concentration, using method of initial rates.

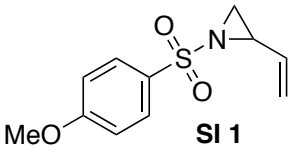
General Procedure for *para*-Substituted Sulfonyl Vinyl Aziridines SI 1-SI 7



Based on the method of Swern et al.[8], to a round bottom flask containing sulfonamide (5 mmol), a commercial sodium hypochlorite solution (6.25%, 16.7 mL, 14 mmol, 2.8 eq) was added. After stirring for 30 minutes, the solution was cooled to 0°C, and glacial acetic acid (1.72 mL, 30 mmol, 6 eq) was added dropwise. The precipitate was filtered, and washed with cold water. To a 10% solution of sodium hydroxide (10 mL, 25 mmol, 5 eq) heated to 80°C, the precipitate was added. This solution was cooled to 0°C, and left undisturbed until the chloramine salt crystallized. The crystals were collected buy filtration, and carried on to the next step without any purification.

The chloramine salt was placed under vacuum and heated at 80°C[9] until no observed mass change occurred. In a flame-dried heavy-walled flask, 1,3-butadiene was condensed (4 mL, 47.4 mmol), followed by acetonitrile (20 mL), the anhydrous chloramine salt, and phenyltrimethylammonium tribromide (188 mg, 0.5 mmol, 0.10 eq). The flask was sealed, and left to stir at room temperature for 16 hours. The contents of the flask were concentrated, and purified via column chromatography (EtOAc/hexane). Yields were based on sulfonamide.

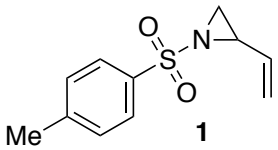
1-((4-methoxyphenyl)sulfonyl)-2-vinylaziridine (SI 1)



White solid, 70% yield. R_f = 0.25 (25% EtOAc:Hex)
^{1}H NMR (499 MHz, $CDCl_3$) δ 7.91 – 7.84 (m, 2H), 7.04 – 6.96 (m, 2H), 5.52 (ddd, *J* = 17.3, 10.0, 7.3 Hz, 1H), 5.42 (dd, *J* = 17.2, 1.2 Hz, 1H), 5.24 (ddd, *J* = 10.1, 1.4, 0.5 Hz, 1H), 3.88 (s, 3H), 3.25 (td, *J* = 7.2, 4.5 Hz, 1H), 2.76 (d, *J* = 7.1 Hz, 1H), 2.21 (d, *J* = 4.5 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 163.62, 133.00, 129.99, 129.57, 120.22, 114.27, 55.64, 40.90, 34.14. FTIR (thin film) 3088, 2995, 2949, 2841, 1595, 1578, 1497, 1460, 1420, 1327, 1302, 1261, 1153, 1094, 1022, 934, 835, 804, 741, 677, 627, 569 cm^{-1}. HRMS (ESI^+) *m/z* 240.0689 [calculated mass for $C_{11}H_{14}NO_3S$ $(M+H)^+$ 240.0689].

[8] Heintzelman, R.W.; Swern, D. *Synthesis* **1976**, 731.
[9] Sharpless and co-workers reported in one instance, this caused an explosion in a vacuum oven. We never observed this, however we always remained vigilant to this fact. *J. Am. Chem. Soc.***120**, 6844.

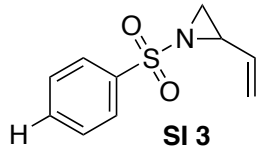
1-((4-methylphenyl)sulfonyl)-2-vinylaziridine (1)



1

White solid, 72% yield. R_f = 0.44 (25% EtOAc:Hex)
^{1}H NMR (499 MHz, $CDCl_3$) δ 7.85 – 7.79 (m, 2H), 7.36 – 7.30 (m, 2H), 5.51 (ddd, *J* = 17.2, 10.0, 7.3 Hz, 1H), 5.42 (dd, *J* = 17.2, 1.5 Hz, 1H), 5.23 (ddd, *J* = 10.3, 1.4, 0.5 Hz, 1H), 3.27 (ddd, *J* = 7.2, 4.8, 4.2 Hz, 1H), 2.78 (d, *J* = 7.1 Hz, 1H), 2.44 (s, 3H), 2.21 (d, *J* = 4.5 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 143.36, 134.19, 129.68, 127.30, 125.50, 125.22, 54.77, 54.19, 21.44. FTIR (thin film) 3090, 2997, 2926, 1597, 1456, 1323, 1159, 1092, 988, 932, 839, 816, 739, 677, 627, 561 cm^{-1}. HRMS (ESI$^+$) *m/z* 224.0741 [calculated mass for $C_{11}H_{14}NO_2S$ $(M+H)^+$ 224.0740]

1-((phenylsulfonyl)-2-vinylaziridine (SI 3)



SI 3

White solid, 68% yield. R_f = 0.37 (25% EtOAc:Hex)
^{1}H NMR (499 MHz, $CDCl_3$) δ 7.99 – 7.93 (m, 2H), 7.68 – 7.61 (m, 1H), 7.59 – 7.51 (m, 2H), 5.52 (ddd, *J* = 17.2, 10.0, 7.3 Hz, 1H), 5.43 (ddd, *J* = 17.1, 1.4, 0.4 Hz, 1H), 5.25 (ddd, *J* = 10.1, 1.4, 0.6 Hz, 1H), 3.30 (td, *J* = 7.2, 4.5 Hz, 1H), 2.82 (d, *J* = 7.1 Hz, 1H), 2.24 (d, *J* = 4.5 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 138.13, 133.55, 132.80, 129.06, 127.74, 120.42, 41.07, 34.27. FTIR (thin film) 3090, 3069, 3001, 1479, 1449, 1325, 1290, 1221, 1163, 1092, 988, 937, 839, 741, 689, 638, 596 cm^{-1}. HRMS (ESI$^+$) *m/z* 210.0583 [calculated mass for $C_{10}H_{12}NO_2S$ $(M+H)^+$ 210.0583]

1-((4-fluorophenyl)sulfonyl)-2-vinylaziridine (SI 4)

SI 4

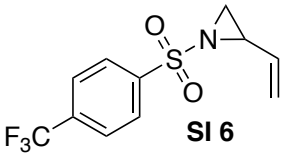
White Solid, 65% yield. R_f = 0.48 (25% EtOAc:Hex)
^{1}H NMR (499 MHz, $CDCl_3$) δ 8.01 – 7.94 (m, 2H), 7.26 – 7.19 (m, 2H), 5.52 (ddd, *J* = 12.6, 9.9, 7.2 Hz, 1H), 5.43 (dd, *J* = 17.2, 1.6 Hz, 1H), 5.26 (ddd, *J* = 9.9, 1.5, 0.5 Hz, 1H), 3.30 (td, *J* = 7.1, 4.6 Hz, 1H), 2.82 (d, *J* = 7.1 Hz, 1H), 2.25 (d, *J* = 4.5 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 165.65 (d, *J* = 255.9 Hz), 134.26 (d, *J* = 3.2 Hz), 132.66, 130.58 (d, *J* = 9.6 Hz), 120.53, 116.39 (d, *J* = 22.7 Hz), 41.22, 34.36. ^{19}F NMR (282 MHz, $CDCl_3$) δ -106.76. FTIR (thin film) 3107, 3080, 2997, 1593, 1495, 1406, 1325, 1292, 1236, 1196, 1169, 1155, 1092, 988, 928, 839, 806, 741, 687, 627, cm^{-1}. HRMS (ESI$^+$) *m/z* 228.0489 [calculated mass for $C_{10}H_{11}FNO_2S$ $(M+H)^+$ 228.0489]

1-((4-bromophenyl)sulfonyl)-2-vinylaziridine (SI 5)

SI 5

White solid, 68% yield. R_f = 0.50 (25% EtOAc:Hex)
^{1}H NMR (499 MHz, $CDCl_3$) δ 7.83 – 7.78 (m, 2H), 7.72 – 7.66 (m, 2H), 5.55 – 5.48 (m, 1H), 5.44 (dd, *J* = 17.2, 1.6 Hz, 1H), 5.27 (ddd, *J* = 9.9, 1.6, 0.5 Hz, 1H), 3.31 (td, *J* = 7.1, 4.6 Hz, 1H), 2.82 (d, *J* = 6.9 Hz, 1H), 2.26 (d, *J* = 4.6 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 137.27, 132.59, 132.41, 129.27, 128.79, 120.67, 41.33, 34.41. FTIR (thin film) 3092, 3022, 2999, 1645, 1574, 1472, 1391, 1325, 1277, 1221, 1161, 1090, 1069, 1011, 934, 839, 822, 795, 754 cm^{-1}. HRMS (ESI^+) *m/z* 287.9689 [calculated mass for $C_{10}H_{11}BrNO_2S$ $(M+H)^+$ 287.9688]

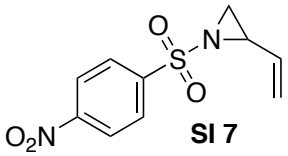
1-((4-(trifluoromethyl)phenyl)sulfonyl)-2-vinylaziridine (SI 6)



SI 6

Colorless oil, 60% yield. R_f = 0.50 (25% EtOAc:Hex)
^{1}H NMR (499 MHz, $CDCl_3$) δ 8.13 – 8.06 (m, 2H), 7.86 – 7.79 (m, 2H), 5.57 – 5.49 (m, 1H), 5.46 (dd, *J* = 17.1, 1.7 Hz, 1H), 5.29 (ddd, *J* = 9.8, 1.6, 0.5 Hz, 1H), 3.37 (td, *J* = 7.0, 4.6 Hz, 1H), 2.87 (d, *J* = 7.1 Hz, 1H), 2.29 (d, *J* = 4.6 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 141.86, 135.22 (q, *J* = 33.1 Hz), 132.42, 128.31, 126.24 (q, *J* = 3.7 Hz), 123.11 (q, *J* = 273.0 Hz), 120.89, 41.54, 34.59. ^{19}F NMR (282 MHz, $CDCl_3$) δ -66.39. FTIR (thin film) 3103, 3059, 3003, 1406, 1321, 1221, 1167, 1132, 1107, 1094, 1063, 1016, 988, 932, 841, 793, 745, 727, 716, 638, 613 cm^{-1}. HRMS (ESI^+) *m/z* 278.0457 [calculated mass for $C_{11}H_{11}F_3NO_2S$ $(M+H)^+$ 278.0457]

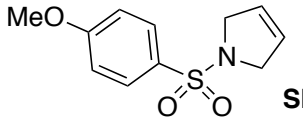
1-((4-nitrophenyl)sulfonyl)-2-vinylaziridine (SI 7)



SI 7

Tan solid, 57% yield. R_f = 0.37 (25% EtOAc:Hex)
^{1}H NMR (499 MHz, $CDCl_3$) δ 8.42 – 8.38 (m, 2H), 8.18 – 8.14 (m, 2H), 5.52 (ddd, *J* = 17.0, 9.4, 6.8 Hz, 1H), 5.46 (dd, *J* = 17.1, 1.9 Hz, 1H), 5.30 (ddd, *J* = 9.4, 1.8, 0.4 Hz, 1H), 3.39 (td, *J* = 7.1, 4.6 Hz, 1H), 2.91 (d, *J* = 7.1 Hz, 1H), 2.33 (d, *J* = 4.6 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 150.63, 144.04, 132.19, 129.08, 124.30, 121.15, 41.85, 34.78. FTIR (thin film) 3105, 3075, 3046, 1605, 1526, 1400, 1352, 1302, 1231, 1194, 1171, 1094, 1001, 976, 949, 935, 837, 762, 743, 669 cm^{-1}. HRMS (EI^+) *m/z* 254.0351 [calculated mass for $C_{10}H_{10}N_2O_4S$ (M) 254.0361]

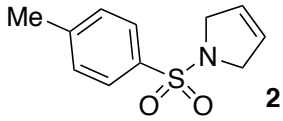
2,5-dihydro-((4-methoxyphenyl)sulfonyl)pyrrole SI 8



SI 8

White solid, R_f = 0.25 (25% EtOAc:Hex)
^{1}H NMR (499 MHz, $CDCl_3$) δ 7.81 – 7.75 (m, 2H), 7.02 – 6.96 (m, 2H), 5.68 – 5.62 (m, 1H), 4.14 – 4.08 (m, 4H), 3.87 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 162.88, 129.45, 128.99, 125.43, 114.24, 55.56, 54.80. FTIR (thin film) 3078, 2926, 2859, 1595, 1570, 1499, 1339, 1306, 1263, 1159, 1128, 1105, 1020, 814, 669, 559 cm^{-1}. HRMS (EI$^+$) *m/z* 239.0614 [calculated mass for $C_{11}H_{13}NO_3S$ (M) 239.0616]

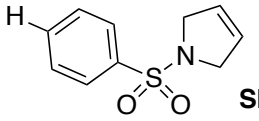
2,5-dihydro-((4-methylphenyl)sulfonyl)pyrrole (2)



2

White solid, R_f = 0.44 (25% EtOAc:Hex)
^{1}H NMR (499 MHz, $CDCl_3$) δ 7.74 – 7.67 (m, 2H), 7.34 – 7.28 (m, 2H), 5.65 – 5.63 (m, 2H), 4.14 – 4.07 (m, 4H), 2.41 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 143.38, 134.21, 129.69, 127.34, 125.38, 54.79, 21.47. FTIR (thin film) 2909, 2857, 1595, 1339, 1308, 1163, 1132, 1107, 947, 822, 667, 546 cm^{-1}. HRMS (ESI$^+$) *m/z* 224.0744 [calculated mass for $C_{11}H_{14}NO_2S$ $(M+H)^+$ 224.0740]

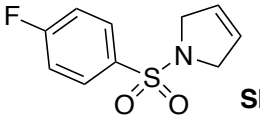
2,5-dihydro-(phenylsulfonyl)pyrrole (SI 10)



SI 10

White solid, R_f = 0.37 (25% EtOAc:Hex)
1H NMR (499 MHz, $CDCl_3$) δ 7.87 – 7.81 (m, 2H), 7.63 – 7.57 (m, 1H), 7.56 – 7.51 (m, 2H), 5.69 – 5.64 (m, 1H), 4.17 – 4.12 (m, 4H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 137.30, 132.63, 129.11, 127.30, 125.40, 54.83. FTIR (thin film) 3082, 2941, 2909, 2860, 1339, 1308, 1171, 1098, 1070, 764, 721, 698, 608, 573 cm^{-1}. HRMS (EI$^+$) *m/z* 209.0515 [calculated mass for $C_{10}H_{11}NO_2S$ (M) 209.0511]

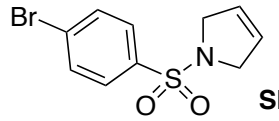
2,5-dihydro-((4-fluorophenyl)sulfonyl)pyrrole (SI 11)



SI 11

White Solid, R_f = 0.48 (25% EtOAc:Hex)
^{1}H NMR (499 MHz, $CDCl_3$) δ 7.87 (dd, *J* = 8.6, 5.1 Hz, 2H), 7.21 (t, *J* = 8.3 Hz, 2H), 5.70 – 5.65 (m, 1H), 4.19 – 4.10 (m, 4H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 165.12 (d, *J* = 254.6 Hz), 133.45, 129.95 (d, *J* = 9.2 Hz), 125.39, 116.36 (d, *J* = 22.4 Hz), 54.84. ^{19}F NMR (282 MHz, $CDCl_3$) δ -108.50. FTIR (thin film) 3103, 3076, 2957, 2920, 2864, 2851, 1591, 1493, 1474, 1339, 1294, 1231, 1163, 1101, 822, 712, 671, 546 cm^{-1}. HRMS (EI$^+$) *m/z* 227.0413 [calculated mass for $C_{10}H_{10}FNO_2S$ (M) 227.0416]

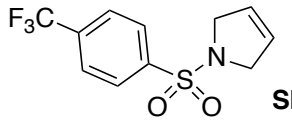
2,5-dihydro-((4-bromophenyl)sulfonyl)pyrrole (SI 12)



SI 12

White solid, R_f = 0.50 (25% EtOAc:Hex)
^{1}H NMR (499 MHz, $CDCl_3$) δ 7.74 – 7.64 (m, 4H), 5.71 – 5.65 (m, 1H), 4.16 – 4.09 (m, 4H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 136.38, 132.41, 128.81, 127.64, 125.38, 54.86. FTIR (thin film) 3088, 2951, 2911, 2857, 1391, 1341, 1167, 1098, 1069, 1009, 826, 808, 739, 619, 579 cm^{-1}. HRMS (EI$^+$) *m/z* 286.9605 [calculated mass for $C_{10}H_{10}BrNO_2S$ (M) 286.9616]

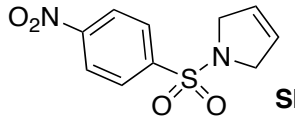
2,5-dihydro-((4-(trifluoromethyl)phenyl)sulfonyl)pyrrole (SI 13)



SI 13

White Solid, R_f = 0.50 (25% EtOAc:Hex)
^{1}H NMR (499 MHz, $CDCl_3$) δ 7.97 (dd, *J* = 8.8, 0.7 Hz, 2H), 7.84 – 7.77 (m, 2H), 5.72 – 5.67 (m, 2H), 4.17 – 4.14 (m, 4H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 141.03, 134.35 (q, *J* = 33.0 Hz), 127.74, 126.31 (q, *J* = 3.7 Hz), 125.36, 123.24 (q, *J* = 272.8 Hz), 54.90. ^{19}F NMR (282 MHz, $CDCl_3$) δ -66.27. FTIR (thin film) 3098, 3075, 3051, 2963, 2924, 2859, 1466, 1406, 1350, 1325, 1165, 1132, 1109, 1063, 1015, 814, 714, 691, 623, 602, 571 cm^{-1}. HRMS (EI$^+$) *m/z* 277.0394 [calculated mass for $C_{11}H_{10}F_3NO_2S$ (M) 277.0384]

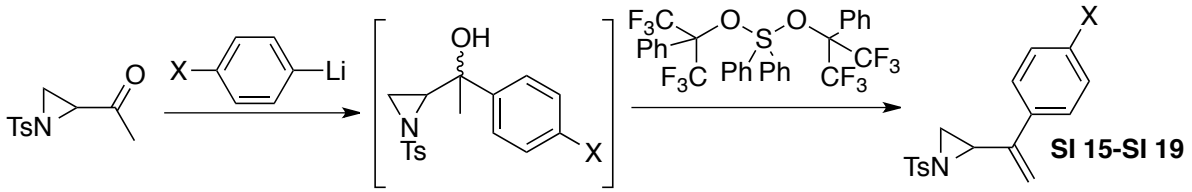
2,5-dihydro-((4-nitrophenyl)sulfonyl)pyrrole (SI 14)



SI 14

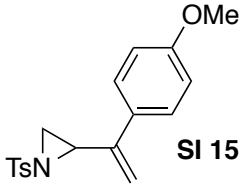
Tan solid, R_f = 0.37 (25% EtOAc:Hex)
^{1}H NMR (499 MHz, $CDCl_3$) δ 8.42 – 8.36 (m, 2H), 8.06 – 7.99 (m, 2H), 5.74 – 5.68 (m, 1H), 4.23 – 4.14 (m, 4H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 150.09, 143.25, 128.38, 125.32, 124.42, 54.95. FTIR (thin film) 3100, 3080, 3034, 2947, 2916, 2868, 1605, 1526, 1344, 1294, 1165, 1096, 1069, 947, 854, 735, 677, 579 cm^{-1}. HRMS (EI$^+$) *m/z* 254.0365 [calculated mass for $C_{10}H_{10}N_2O_4S$ (M) 254.0361]

General Procedure for the Synthesis of 2-Aryl Vinyl Aziridines SI 15-SI 19



To a round bottom flask was added aryl iodide (5 mmol) and diethyl ether (10 mL). After the aryl iodide completely dissolved, the solution was cooled to -78°C followed by the dropwise addition of ^{n}BuLi (2.0 mL, 5 mmol). The solution was stirred at -78°C for one hour, then the flask was moved into an ice water bath and left to stir for 2 additional hours.[10] In a separate round bottom flask, aziridine **8**[11] (240 mg, 1.0 mmol) dissolve in THF (2.0 mL) was cooled to -78°C, and aryl iodide (5.0 mL, 2.0 mmol, 2 eq) was added dropwise to this solution. The reaction was allowed to warm to 0°C, and subsequently quenched with saturated ammonium chloride. The layers were separated, and the aqueous layer was washed twice with EtOAc. The combined organic layers were washed once with water and once with brine, dried over magnesium sulfate, filtered through a plug of Celite® 545, and concentrated. The crude material was brought up in dichloromethane (5 mL) and cooled to 0°C, followed by the addition dropwise addition of Martin's Sulfurane (845 mg, 1.25 mmol, 1.25 eq) dissolved in dichloromethane (1.25 mL). The reaction was stirred for another hour at 0°C, then concentrated, and purified by column chromatography (EtOAc/hexane). Yields are based on aziridine **8**.

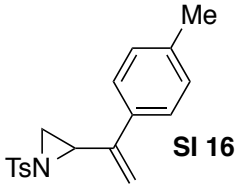
2-(1-(4-methoxyphenyl)vinyl)-1-tosylaziridine (SI 15)



White solid, 79% yield. R_f = 0.31 (25% EtOAc:Hex)
^{1}H NMR (499 MHz, $CDCl_3$) δ 7.89 – 7.84 (m, 2H), 7.38 – 7.31 (m, 5H), 6.87 – 6.82 (m, 2H), 5.35 (s, 1H), 5.19 (t, *J* = 1.0 Hz, 1H), 3.81 (s, 3H), 3.57 (ddd, *J* = 7.1, 4.5, 0.9 Hz, 1H), 2.89 (d, *J* = 7.1 Hz, 1H), 2.45 (s, 3H), 2.27 (d, *J* = 4.5 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 159.59, 144.62, 141.06, 134.94, 130.32, 129.70, 128.00, 127.14, 113.79, 113.08, 55.28, 41.23, 34.61, 21.66. FTIR (thin film) 3088, 2995, 1595, 1578, 1497, 1420, 1327, 1261, 1153, 1094, 1022, 934, 835, 804, 741, 677, 627, 569 cm^{-1}. HRMS (ESI^{+}) *m/z* 330.1155 [calculated mass for $C_{18}H_{20}NO_3S$ $(M+H)^{+}$ 330.1158]

[10] 4-(trifluoromethyl)phenyliodide was warmed to -40°C (dry ice/acetonitrile) and stirred for 2 hours. It was added to the subsequent reaction at this temperature.
[11] Smith, A.B., III; Kim, D.-S. *Org. Lett*. **2005**, *7*, 3247.

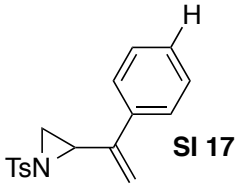
2-(1-(4-methylphenyl)vinyl)-1-tosylaziridine (SI 16)



SI 16

White solid, 83% yield. R_f = 0.50 (25% EtOAc:Hex)
^{1}H NMR (499 MHz, $CDCl_3$) δ 7.88 – 7.83 (m, 2H), 7.36 – 7.30 (m, 2H), 7.32 – 7.27 (m, 2H), 7.15 – 7.09 (m, 2H), 5.40 (s, 1H), 5.24 (s, 1H), 3.57 (ddd, *J* = 7.1, 4.5, 1.1 Hz, 1H), 2.90 (d, *J* = 7.1 Hz, 1H), 2.45 (s, 3H), 2.34 (s, 3H), 2.27 (d, *J* = 4.5 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 144.59, 141.53, 138.06, 134.95, 134.92, 129.69, 129.11, 127.99, 125.81, 113.85, 41.18, 34.75, 21.67, 21.14; FTIR (thin film) 3030, 2997, 2922, 2868, 1597, 1516, 1325, 1217, 1163, 1094, 937, 870, 824, 731, 681, 575, 561 cm^{-1}; HRMS (ESI^+) *m/z* 314.1215 [calculated mass for $C_{18}H_{19}NO_2S$ $(M+H)^+$ 314.1209]

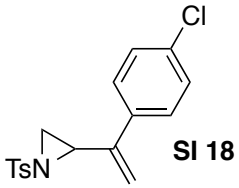
2-(1-(phenylvinyl)-1-tosylaziridine (SI 17)



SI 17

White solid, 82% yield. R_f = 0.47 (25% EtOAc:Hex)
^{1}H NMR (499 MHz, $CDCl_3$) δ 7.89 – 7.85 (m, 4H), 7.42 – 7.37 (m, 2H), 7.36 – 7.27 (m, 4H), 5.43 (s, 1H), 5.29 (t, *J* = 1.0 Hz, 1H), 3.58 (ddd, *J* = 7.1, 4.5, 0.9 Hz, 1H), 2.91 (d, *J* = 7.1 Hz, 1H), 2.45 (s, 3H), 2.28 (d, *J* = 4.5 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 144.64, 141.78, 137.82, 134.93, 129.72, 128.44, 128.16, 128.01, 126.00, 114.77, 41.11, 34.73, 21.67. FTIR (thin film) 3059, 3030, 2997, 2955, 2924, 1597, 1495, 1447, 1398, 1325, 1219, 1163, 1094, 937, 866, 816, 727, 702, 677, 561 cm^{-1}. HRMS (ESI^+) *m/z* 300.1053 [calculated mass for $C_{17}H_{18}NO_2S$ $(M+H)^+$ 300.1053]

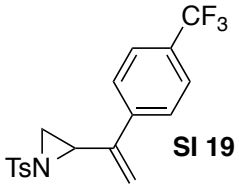
2-(1-(4-chlorophenyl)vinyl)-1-tosylaziridine (SI 18)



SI 18

White solid, 92% yield. R_f = 0.50 (25% EtOAc:Hex)
^{1}H NMR (499 MHz, $CDCl_3$) δ 7.87 – 7.82 (m, 2H), 7.36 – 7.29 (m, 4H), 7.29 – 7.24 (m, 2H), 5.42 (s, 1H), 5.31 (s, 1H), 3.54 (ddd, *J* = 6.8, 4.4, 1.0 Hz, 1H), 2.89 (d, *J* = 7.1 Hz, 1H), 2.45 (s, 3H), 2.27 (d, *J* = 4.5 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 144.76, 140.92, 136.26, 134.77, 134.06, 129.71, 128.54, 127.97, 127.38, 115.61, 40.88, 34.38, 21.66; FTIR (thin film) 3092, 2999, 2924, 1597, 1495, 1398, 1327, 1221, 1163, 1096, 1013, 937, 870, 835, 816, 773, 729, 673, 606, 561 cm^{-1}; HRMS (ESI^+) *m/z* 334.0668 [calculated mass for $C_{17}H_{16}NO_2SCl$ $(M+H)^+$ 334.0663]

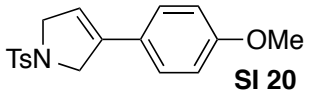
2-(1-(4-(trifluoromethyl)phenyl)vinyl)-1-tosylaziridine (SI 19)



SI 19

White solid, 86% yield. R_f = 0.50 (25% EtOAc:Hex)
^{1}H NMR (499 MHz, $CDCl_3$) δ 7.86 – 7.82 (m, 2H), 7.57 – 7.51 (m, 2H), 7.50 – 7.47 (m, 2H), 7.35 – 7.31 (m, 2H), 5.51 (s, 1H), 5.41 (s, 1H), 3.58 (ddd, *J* = 7.0, 4.5, 1.0 Hz, 1H), 2.91 (d, *J* = 7.0 Hz, 1H), 2.45 (s, 3H), 2.30 (d, *J* = 4.5 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 144.85, 141.35, 141.08, 134.72, 130.10 (q, *J* = 32.5 Hz), 129.74, 128.01, 126.48, 125.33 (q, *J* = 3.8 Hz), 123.98 (q, *J* = 271.9 Hz), 117.24, 40.79, 34.31, 21.64; ^{19}F NMR (282 MHz, $CDCl_3$) δ -65.84; FTIR (thin film) 3094, 3028, 2930, 1327, 1319, 1221, 1159, 1117, 1067, 1016, 934, 849, 814, 748, 704, 664, 554 cm^{-1}; HRMS (ESI$^+$) *m/z* 368.0931 [calculated mass for $C_{18}H_{16}NO_2SF_3$ $(M+H)^+$ 368.0927]

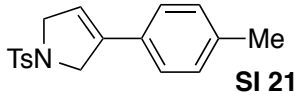
3-(4-methoxyphenyl)-2,5-dihydropyrrole (SI 20)



SI 20

White solid, R_f = 0.31 (25% EtOAc:Hex)
^{1}H NMR (499 MHz, $CDCl_3$) δ 7.82 – 7.66 (m, 1H), 7.34 – 7.29 (m, 1H), 7.24 – 7.19 (m, 1H), 6.99 – 6.73 (m, 1H), 5.85 (p, *J* = 2.1 Hz, 1H), 4.46 – 4.43 (m, 1H), 2.41 (s, 2H), 4.50 – 4.37 (m, 2H), 4.28 (td, *J* = 4.4, 2.3 Hz, 2H), 3.80 (s, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 159.66, 143.48, 136.71, 134.08, 129.78, 127.44, 126.64, 125.23, 116.61, 114.02, 55.63, 55.29, 54.98, 21.50; FTIR (thin film) 3030, 2997, 2922, 2868, 1597, 1516, 1325, 1217, 1163, 1094, 937, 870, 824, 731, 681, 575, 561 cm^{-1}; HRMS (ESI$^+$) *m/z* 330.1152 [calculated mass for $C_{18}H_{20}NO_3S$ $(M+H)^+$ 330.1158]

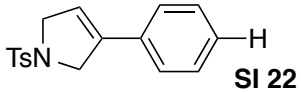
3-(4-methylphenyl)-2,5-dihydropyrrole (SI 21)



SI 21

White solid, R_f = 0.50 (25% EtOAc:Hex)
^{1}H NMR (499 MHz, $CDCl_3$) δ 7.78 – 7.74 (m, 2H), 7.34 – 7.28 (m, 2H), 7.20 – 7.14 (m, 2H), 7.15 – 7.09 (m, 2H), 5.93 (t, *J* = 2.1 Hz, 1H), 4.46 (td, *J* = 4.5, 2.0 Hz, 2H), 4.28 (td, *J* = 4.4, 2.2 Hz, 2H), 2.41 (s, 3H), 2.33 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 143.48, 138.39, 137.16, 134.07, 129.78, 129.67, 129.31, 127.43, 125.25, 117.79, 55.63, 54.94, 21.50, 21.21; FTIR (thin film) 3026, 2949, 2911, 2826, 1342, 1163, 1101, 816, 791, 667, 617, 586, 548 cm^{-1}; HRMS (ESI$^+$) *m/z* 314.1210 [calculated mass for $C_{18}H_{19}NO_2S$ $(M+H)^+$ 314.1209]

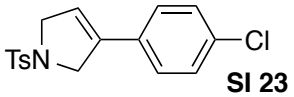
3-(phenyl)-2,5-dihydropyrrole (SI 22)



SI 22

White solid, R_f = 0.47 (25% EtOAc:Hex)
^{1}H NMR (499 MHz, $CDCl_3$) δ 7.79 – 7.74 (m, 2H), 7.35 – 7.25 (m, 7H), 6.00 (p, *J* = 2.1 Hz, 1H), 4.48 (ddd, *J* = 4.8, 3.9, 2.0 Hz, 2H), 4.30 (td, *J* = 4.5, 2.3 Hz, 2H), 2.41 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 143.55, 137.33, 134.07, 132.47, 129.82, 128.67, 128.43, 127.46, 125.37, 118.85, 55.66, 54.91, 21.51. FTIR (thin film) 3061, 3030, 2909, 2841, 1344, 1163, 1105, 1072, 831, 816, 750, 675, 664, 600, 550 cm^{-1}. HRMS (ESI$^+$) *m/z* 300.1054 [calculated mass for $C_{17}H_{18}NO_2S$ $(M+H)^+$ 300.1053]

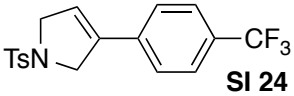
3-(4-chlorophenyl)-2,5-dihydropyrrole (SI 23)



SI 23

White solid, R_f = 0.50 (25% EtOAc:Hex)
^{1}H NMR (499 MHz, $CDCl_3$) δ 7.80 – 7.71 (m, 2H), 7.35 – 7.30 (m, 2H), 7.31 – 7.26 (m, 2H), 7.22 – 7.19 (m, 2H), 6.00 (p, *J* = 2.2 Hz, 1H), 4.44 (td, *J* = 4.5, 1.9 Hz, 2H), 4.29 (td, *J* = 4.5, 2.2 Hz, 2H), 2.42 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 143.62, 136.28, 134.19, 133.99, 130.92, 129.84, 128.84, 127.43, 126.61, 119.60, 55.61, 54.78, 21.52; FTIR (thin film) 3090, 3071, 2932, 2907, 2868, 2839, 1597, 1495, 1410, 1335, 1161, 1103, 1015, 995, 908, 799, 737, 667, 604, 550 cm^{-1}; HRMS (ESI$^+$) *m/z* 334.0666 [calculated mass for $C_{17}H_{16}NO_2SCl$ $(M+H)^+$ 334.0663]

3-(4-(trifluoromethyl)phenyl)-2,5-dihydropyrrole (SI 24)

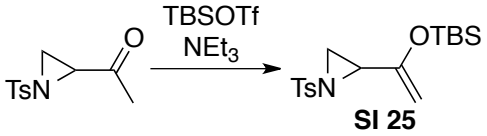


SI 24

White solid, R_f = 0.50 (25% EtOAc:Hex)
^{1}H NMR (499 MHz, $CDCl_3$) δ 7.80 – 7.74 (m, 2H), 7.60 – 7.55 (m, 2H), 7.41 – 7.35 (m, 2H), 7.36 – 7.30 (m, 2H), 6.14 (t, *J* = 2.2 Hz, 1H), 4.49 (td, *J* = 4.6, 2.0 Hz, 2H), 4.33 (td, *J* = 4.5, 2.2 Hz, 2H), 2.42 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 143.72, 136.28, 135.80, 133.94, 130.21(q, *J* = 32.8 Hz), 129.88, 127.44, 125.64 (q, *J* = 3.7 Hz), 125.63, 123.85(q, *J* = 272.1 Hz), 121.64, 55.64, 54.72, 21.52; ^{19}F NMR (376 MHz, $CDCl_3$) -65.90; FTIR (thin film) 3090, 2938, 2907, 2862, 2839, 1418, 1327, 1163, 1113, 1072, 1018 ,810, 667, 604, 548 cm^{-1}; HRMS (ESI$^+$) *m/z* 368.0928 [calculated mass for $C_{18}H_{16}NO_2SF_3$ $(M+H)^+$ 368.0927]

Synthesis of Vinyl Substituted Aziridines

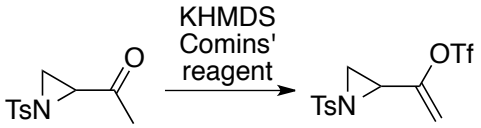
2-(^{t}butyldimethylsiloxyl)-vinyl aziridine (SI 25)



SI 25

Synthesized according to the procedure of Njardarson et. al.[12]

2-(trifluoromethylsulfonate)-vinyl aziridine (SI 26)

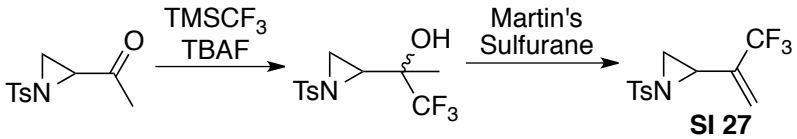


SI 26

A solution of potassium hexamethyldisilane (0.65 M in toluene, 6.75 mL, 4.4 mmol, 1.25 eq) in THF (18 mL) was cooled to -78°C. Aziridine ketone **8** (837 mg, 3.5 mmol, 1.0 eq) in THF (3.5 mL) was added to this solution[13], and after addition of ketone solution, the mixture was stirred for two hours at -78°C. Comins' reagent (2.06 g, 5.25 mmol, 1.5 eq) was added portionwise over 15 mins, and the reaction was stirred for one hour at -78°C, followed by warming to room temperature. The reaction was quenched with saturated ammonium chloride, the layers were separated, the aqueous layer was washed two times with EtOAc, and the combined organics were washed once with water and once with brine, dried over magnesium sulfate, filtered through Celite® 545, and concentrated. The crude was purified via column chromatography, yielding vinyl aziridine **33** (1.09 g, 84% yield) as an off-white solid.

R_f = 0.10 (25% EtOAc:Hex)
^{1}H NMR (499 MHz, $CDCl_3$) δ 7.86 – 7.81 (m, 2H), 7.39 – 7.33 (m, 2H), 5.30 (d, *J* = 3.9 Hz, 1H), 5.28 (d, *J* = 3.9 Hz, 1H), 3.36 (dd, *J* = 7.0, 4.1 Hz, 1H), 2.83 (d, *J* = 7.0 Hz, 1H), 2.45 (s, 3H), 2.43 (d, *J* = 4.1 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 149.30, 145.29, 134.04, 129.86, 128.07, 118.35 (q, *J* = 320.3 Hz), 108.07, 37.38, 32.92, 21.68. ^{19}F NMR (282 MHz, $CDCl_3$) δ -81.31. FTIR (thin film) 3134, 3040, 2928, 1423, 1333, 1213, 1165, 1140, 1094, 955, 932, 841, 818, 735, 681, 592 cm^{-1}. HRMS (ESI$^+$) *m/z* 372.0182 [calculated mass for $C_{12}H_{13}F_3NO_5S_2$ $(M+H)^+$ 372.0182]

2-(trifluoromethyl)-vinyl aziridine (SI 27)



SI 27

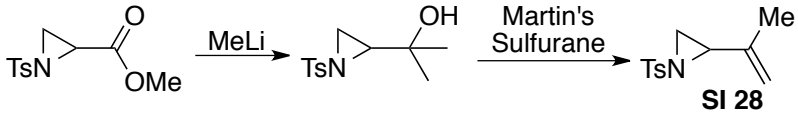
To a solution of aziridine ketone **8** (680 mg, 2.85 mmol, 1 eq) in THF (12 mL) was added trimethylsilyltrifluoromethyl (2.0 M in THF, 2.10 mL, 4.27 mmol, 1.5 eq) and the mixture was cooled to 0°C, and a tetrabutylammonium floride (1.0 M in THF, 3.10 mL, 3.14 mmol, 1.1 eq) was added drop wise over one hour. After addition, the reaction was allowed to stir for one

[12] Brichacek, M.; Lee, D.; Njardarson, J.T. *Org. Lett.* **2008**, *10*, 5023.
[13] The reaction fails if adding KHMDS to the solution of aziridine ketone.

additional hour, quenched with saturated ammonium chloride, the layers separated, the aqueous washed two times with EtOAc, the organic layers combined, and washed once with water and once with brine, dried over magnesium sulfate, filtered through Celite® 545, and concentrated. Alcohol **29** was brought up in dichloromethane (14 mL) and cooled to 0°C. Martin's Sulfurane (2.4 g, 3.56 mmol) in dichloromethane (3.6 mL) was added drop wise over 15 minutes, and the reaction was allowed to warm to room temperature and stir for one hour. The reaction was concentrated, and purified via column chromatography, yielding aziridine **35** (517 mg, 62% from **8**) as an off-white solid.

R_f = 0.50 (25% EtOAc:Hex)
^{1}H NMR (499 MHz, $CDCl_3$) δ 7.88 – 7.81 (m, 2H), 7.39 – 7.34 (m, 2H), 5.75 (q, *J* = 1.4 Hz, 1H), 5.57 (p, *J* = 1.2 Hz, 1H), 3.33 (dd, *J* = 6.4, 4.4 Hz, 1H), 2.85 (d, *J* = 7.1 Hz, 1H), 2.46 (s, 3H), 2.26 (d, *J* = 4.3 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 145.11, 134.27, 133.59 (q, *J* = 30.8 Hz), 129.84, 128.07, 122.41 (q, *J* = 273.3 Hz), 120.75 (q, *J* = 5.0 Hz), 35.85, 34.45, 21.69. ^{19}F NMR (282 MHz, $CDCl_3$) δ -70.64. FTIR (thin film) 3096, 3013, 2928, 2872, 1400, 1333, 1317, 1159, 1125, 1094, 1020, 941, 876, 814, 719, 683, 613, 557, 536 cm^{-1}. HRMS (ESI$^+$) *m/z* 292.0620 [calculated mass for $C_{12}H_{13}F_3NO_2S$ $(M+H)^+$ 292.0614]

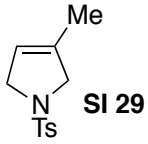
2-methyl-vinyl aziridine (SI 28)



SI 28

Synthesized according to the procedure of Smith et. al.[4]

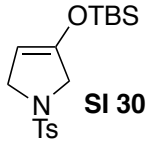
3-methyl-2,5-dihydropyrrole (SI 29)



SI 29

Matches previously reported characterization data.[14]

3-(^{t}butyldimethylsiloxy)-2,5-dihydrofuran (SI 30)

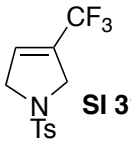


SI 30

Matches previously reported characterization data.[11]

[14] Cheng, H. Y.; Sun, C. S.; Hou, D. R. *J. Org. Chem*. **2007**, *72*, 2674.

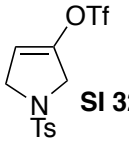
3-(trifluromethyl)-2,5-dihydrofuran (SI 31)



SI 31

White solid, R_f = 0.50 (25% EtOAc:Hex)
^{1}H NMR (499 MHz, $CDCl_3$) δ 7.76 – 7.71 (m, 2H), 7.38 – 7.33 (m, 2H), 6.22 – 6.17 (m, 1H), 4.26 (s, 4H), 2.45 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 144.12, 133.65, 130.02, 129.62 (q, J = 4.8 Hz), 129.19 (q, J = 35.5 Hz), 127.41, 120.62 (q, J = 269.3 Hz), 54.78, 52.10, 21.56. ^{19}F NMR (282 MHz, $CDCl_3$) δ -68.85. FTIR (thin film) 3092, 2924, 2862, 1379, 1350, 1308, 1267, 1163, 1121, 1070, 1043, 982, 816, 727, 669, 604, 548 cm^{-1}. HRMS (ESI$^+$) *m/z* 292.0616 [calculated mass for $C_{12}H_{13}F_3NO_2S$ $(M+H)^+$ 292.0614]

3-(trifluoromethanesulfonate)-2,5-dihydrofuran (SI 32)

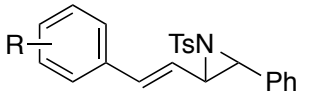


SI 32

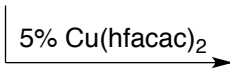
White solid, R_f = 0.10 (25% EtOAc:Hex)
^{1}H NMR (499 MHz, $CDCl_3$) δ 7.74 – 7.70 (m, 2H), 7.39 – 7.34 (m, 2H), 5.65 – 5.61 (m, 1H), 4.23 – 4.17 (m, 4H), 2.45 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 144.31, 142.04, 133.34, 130.07, 127.42, 118.34 (q, J = 320.9 Hz), 112.37, 51.76, 51.13, 21.56. ^{19}F NMR (282 MHz, $CDCl_3$) δ -76.26. FTIR (thin film) 3111, 2928, 2872, 1429, 1354, 1248, 1213, 1161, 1140, 1101, 1067, 908, 843, 816, 669, 610, 596, 550 cm^{-1}. HRMS (ESI$^+$) *m/z* 372.0185 [calculated mass for $C_{12}H_{13}F_3NO_5S_2$ $(M+H)^+$ 372.0182]

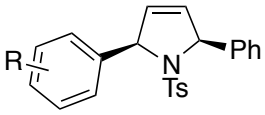
Rearrangement of 1,4-Diaryl Vinyl Aziridines

Following the general procedure for the copper-catalyzed rearrangement protocol, aziridines **SI 33-SI 37** were rearranged with $Cu(hfacac)_2$, and this substrate class displayed a temperature dependence, usually reserved for vinyl oxirane substrates, with the isomerization/hydride shift pathways becoming relevent. The following table illustrates this point.

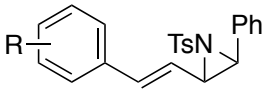


(*E*,*trans*)-vinyl aziridine

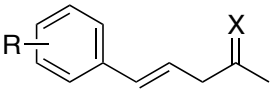




2,5-*cis*-pyrrole



(*E*,*cis*)-vinyl aziridine

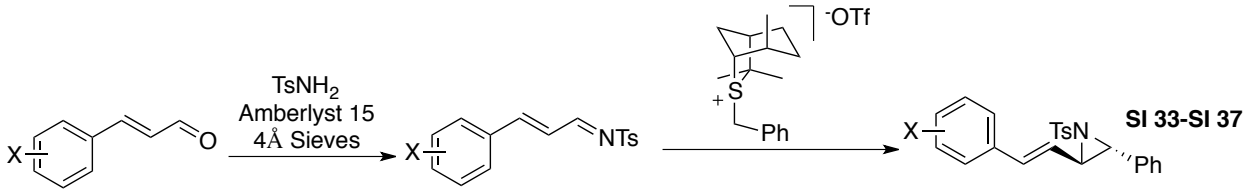


X = O + NTs

R	Temp (°C)	Time (min)					
4-^{t}Bu	60	120	2	:	1	:	0
	150	5	only prod.				
4-Cl	60	900	3	:	0	:	1
	150	5	only prod.				
3-CF_3	60	360	1	:	1	:	0
	150	5	only prod.				
3-CN	60	360	1	:	1.5	:	0
	150	5	only prod.				
4-NO_2	60	360					
	100	60	1	:	2.3	:	0
	150	5	only prod.				

Reactions run at 150°C are in PhMe, and in benzene at 60°C

General Procedure for the Synthesis of Diphenyl Vinyl Aziridines 14-18

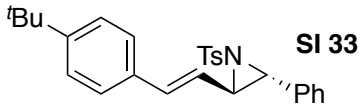


To a flame-dried heavy-walled flask was added substituted cinnamaldehyde[15] (1 mmol), para-toluenesulfonamide (171.2 mg, 1 mmol, 1 eq) Amberlyst® 15 (20 mg), and toluene (4 mL). The flask was sealed and heated at 150°C for 12 hours. The contents were filtered through Celite® 545, and concentrated, yielding a solid which was used without further purification in the next step.

Following the procedure of Aggarwal et al.[16], a flask containing imine and acetonitrile (8 mL) was cooled to 0°C, followed by the addition of sulfonium salt (410 mg, 1 mmol, 1 eq) and K_2CO_3 (276 mg, 2.0 mmol, 2 eq). The flask was allowed to warm to room temperature and was stirred for 12 hours. The reaction was concentrated, brought up in dichloromethane, washed with saturated $NaHSO_3$, 1M NaOH, and brine, dried over magnesium sulfate, filtered through Celite® 545, and concentrated. The vinyl aziridine could be purified by either dissolving in a minimum amount of dichloromethane, followed by dripping into a rapidly stirring solution of hexanes and collecting the precipitate, or via column chromatography.

Yield was based on substituted cinnamaldehyde. Diastereomeric ratio was determined by ^{1}H NMR of the crude aziridination reaction mixture. The enantiomeric ratio was determined by HPLC using a Chiralpak™ IB column, isocratic 98:2 Hexane:2-propanol mobile phase, and a flow rate of 1.5 mL/min. The absolute configuration of vinyl aziridines **14-18** were assigned based on the x-ray structure obtained from vinyl aziridine **15** (included below).

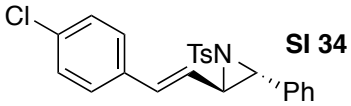
(2*R*,3*R*)-2-Phenyl-3-[(*E*)-2-(4-^{t}butylphenyl)-1-ethenyl]-1-tosylaziridine (SI 33)



Yellow solid, 66% yield, R_f = 0.55 (25% EtOAc: Hex), 19:1 (*trans:cis*), 97:3 (*e.r.*)
^{1}H NMR (499 MHz, $CDCl_3$) δ 7.86 – 7.82 (m, 2H), 7.30 – 7.20 (m, 11H), 6.77 (d, J = 15.8 Hz, 1H), 6.61 (dd, J = 15.7, 9.6 Hz, 1H), 4.15 (d, J = 4.1 Hz, 1H), 3.44 (dd, J = 9.6, 4.1 Hz, 1H), 2.39 (s, 3H), 1.33 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 151.53, 144.13, 137.24, 136.90, 135.13, 133.19, 129.54, 128.58, 128.19, 127.53, 126.46, 126.28, 125.56, 121.22, 77.25, 77.00, 76.75, 55.45, 48.66, 34.66, 31.25, 21.59. FTIR (thin film) 2961, 2903, 2868, 1331, 1159, 1092, 1028, 970, 887, 818, 694, 577 cm^{-1}. HRMS (ESI$^+$) *m/z* 432.2001 [calculated mass for $C_{27}H_{30}NO_2S$ $(M+H)^+$ 432.1992]. $[\alpha]_D^{20} = +15.6°$ (c = 1.16, $CHCl_3$).

[15] Liu, J.; Zhu, J.; Jiang, H.; Wang, W.; Li, J. *Chem. Comm.* **2010**, *46*, 415.
[16] Illa, O.; Arshad, M.; Ros, A.; McGarrigle, E.M.; Aggarwal, V.K. *J. Am. Chem. Soc*. **2010**, *132*, 1828.

(2*R*,3*R*)-2-Phenyl-3-[(*E*)-2-(4-chlorophenyl)-1-ethenyl]-1-tosylaziridine (SI 34)

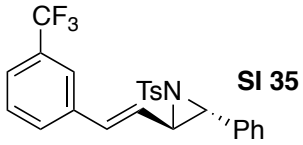


SI 34

White solid, 78% yield, R_f = 0.48 (25% EtOAc: Hex), ≥20:1 (*trans:cis*), 99:1 (*e.r.*)
^{1}H NMR (499 MHz, $CDCl_3$) δ 7.88 – 7.78 (m, 2H), 7.40 – 7.16 (m, 12H), 6.74 (d, *J* = 15.8 Hz, 1H), 6.64 (dd, *J* = 15.8, 9.5 Hz, 1H), 4.15 (d, *J* = 4.1 Hz, 1H), 3.43 (dd, *J* = 9.5, 4.1 Hz, 1H), 2.39 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 144.33, 136.82, 136.07, 134.93, 134.45, 134.05, 129.63, 128.89, 128.67, 128.34, 127.95, 127.54, 126.29, 122.92, 55.06, 48.88, 21.65. FTIR (thin film) 3065, 2922, 2886, 1319, 1153, 1086, 1011, 962, 893, 854, 810, 756, 673 cm^{-1}. HRMS (ESI$^+$) *m/z* 410.0981 [calculated mass for $C_{23}H_{21}ClNO_2S$ $(M+H)^+$ 410.0976]; $[\alpha]_D^{20}$ = + 33.0° (*c* = 1.33, $CHCl_3$).

Single crystal x-ray structure included below, which the absolute configuration was based upon.

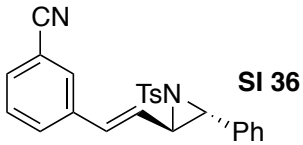
(2*R*,3*R*)-2-Phenyl-3-[(*E*)-2-(3-(trifluoromethyl)phenyl)-1-ethenyl]-1-tosylaziridine (SI 35)



SI 35

White solid, 65% yield, R_f = 0.48 (25% EtOAc: Hex), 19:1 (*trans:cis*), 96:4 (*e.r.*)
^{1}H NMR (499 MHz, $CDCl_3$) δ 7.87 – 7.82 (m, 2H), 7.63 (d, *J* = 8.7 Hz, 2H), 7.54 (d, *J* = 7.7 Hz, 2H), 7.47 (t, *J* = 7.6 Hz, 2H), 7.32 – 7.19 (m, 5H), 6.82 (d, *J* = 15.8 Hz, 1H), 6.73 (dd, *J* = 15.8, 9.4 Hz, 1H), 4.17 (d, *J* = 4.0 Hz, 1H), 3.46 (dd, *J* = 9.4, 4.0 Hz, 1H), 2.39 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 144.37, 136.70, 136.64, 135.82, 134.77, 131.11 (q, *J* = 32.2 Hz), 129.62, 129.62, 129.16, 128.65, 128.35, 127.51, 126.24, 124.84 (q, *J* = 3.8 Hz), 124.42 (q, *J* = 272.6 Hz), 124.29, 123.51 (q, *J* = 3.8 Hz), 54.71, 48.90, 21.60. ^{19}F NMR (282 MHz, $CDCl_3$) δ -65.93. FTIR (thin film) 3063, 3032, 1597, 1327, 1157, 1126, 1096, 1072, 887, 694 cm^{-1}. HRMS (ESI$^+$) *m/z* 444.1248 [calculated mass for $C_{24}H_{21}F_3NO_2S$ $(M+H)^+$ 444.1240]; $[\alpha]_D^{20}$ = + 34.9° (*c* = 1.60, $CHCl_3$).

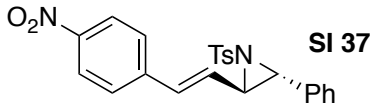
(2*R*,3*R*)-2-Phenyl-3-[(*E*)-2-(3-cyanophenyl)-1-ethenyl]-1-tosylaziridine (SI 36)



SI 36

White solid, 49% yield, R_f = 0.33 (25% EtOAc: Hex), 18:1 (*trans:cis*), 97:1 (*e.r.*)
^{1}H NMR (499 MHz, Chloroform-d) δ 7.86 – 7.81 (m, 2H), 7.70 – 7.65 (m, 2H), 7.59 – 7.53 (m, 1H), 7.49 – 7.42 (m, 1H), 7.32 – 7.24 (m, 5H), 7.23 – 7.18 (m, 2H), 6.79 (d, *J* = 15.8 Hz, 1H), 6.74 (dd, *J* = 15.8, 8.6 Hz, 1H), 4.16 (d, *J* = 4.0 Hz, 1H), 3.45 (ddd, *J* = 8.6, 4.0, 0.5 Hz, 1H), 2.40 (s, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 144.46, 137.14, 136.69, 134.84, 134.67, 131.55, 130.59, 130.29, 129.68, 129.55, 128.70, 128.44, 127.51, 126.24, 125.32, 118.56, 113.02, 54.54, 49.06, 21.66; FTIR (thin film) 3065, 3034, 2924, 2230, 1597, 1402, 1327, 1159, 1088, 966, 893, 851, 818, 735, 694, 586 cm^{-1}; HRMS (ESI$^+$) *m/z* 401.1315 [calculated mass for $C_{24}H_{21}N_2O_2S$ $(M+H)^+$ 401.1318]; $[\alpha]_D^{20}$ = + 26.5° (*c* = 2.90, $CHCl_3$).

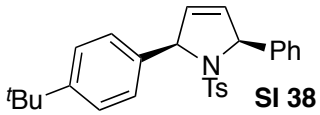
(2*R*,3*R*)-2-Phenyl-3-[(*E*)-2-(4-nitrophenyl)-1-ethenyl]-1-tosylaziridine (SI 37)



SI 37

Tan solid, 46% yield, R_f = 0.33 (25% EtOAc: Hex), 18:1 (*trans:cis*), 96:4 (*e.r.*)
^{1}H NMR (499 MHz, $CDCl_3$) δ 8.24 – 8.17 (m, 2H), 7.87 – 7.81 (m, 2H), 7.60 – 7.55 (m, 2H), 7.33 – 7.17 (m, 7H), 6.87 (s, 1H), 6.86 (d, *J* = 1.6 Hz, 1H), 4.18 (d, *J* = 4.0 Hz, 1H), 3.49 – 3.45 (m, 1H), 2.40 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 147.36, 144.52, 142.11, 136.56, 134.84, 134.54, 129.68, 128.70, 128.47, 127.49, 127.33, 127.26, 126.21, 124.10, 54.47, 49.15, 21.63. FTIR (thin film) 3063, 1597, 1520, 1342, 1157, 1111, 1088, 903, 864, 694 cm^{-1}. HRMS (ESI$^+$) *m/z* 421.1223 [calculated mass for $C_{23}H_{21}N_2O_4S$ $(M+H)^+$ 421.1217]; $[\alpha]_D^{20}$ = + 47.4° (*c* = 0.43, $CHCl_3$).

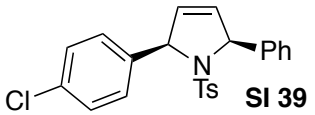
(2*R*,5*S*)-2-(4-(*t*butyl)phenyl)-5-phenyl-2,5-dihydropyrrole (SI 38)



SI 38

Yellow solid, R_f = 0.55 (25% EtOAc: Hex)
^{1}H NMR (499 MHz, $CDCl_3$) δ 7.41 – 7.37 (m, 2H), 7.35 – 7.20 (m, 9H), 7.00 (d, *J* = 7.9 Hz, 2H), 5.76 (t, *J* = 1.5 Hz, 2H), 5.74 (d, *J* = 2.9 Hz, 1H), 5.70 (d, *J* = 2.9 Hz, 1H), 2.31 (s, 3H), 1.31 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 150.76, 142.78, 140.23, 136.91, 136.26, 129.43, 129.06, 129.00, 128.36, 127.89, 127.78, 127.59, 127.38, 125.24, 70.65, 70.52, 34.53, 31.37, 21.42. FTIR (thin film) 3063, 3030, 2963, 2868, 1599, 1495, 1352, 1165, 1092, 1047, 822, 702, 664, 600, 542 cm^{-1}. HRMS (ESI$^+$) *m/z* 432.1994 [calculated mass for $C_{27}H_{30}NO_2S$ $(M+H)^+$ 432.1992]; $[\alpha]_D^{20}$ = -1.4° (*c* = 0.44, $CHCl_3$).

(2*R*,5*S*)-2-(4-chlorophenyl)-5-phenyl-2,5-dihydropyrrole (SI 39)

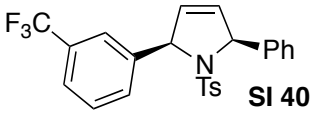


SI 39

White solid, R_f = 0.48 (25% EtOAc: Hex)
^{1}H NMR (499 MHz, $CDCl_3$) δ 7.38 – 7.23 (m, 11H), 7.10 (d, *J* = 7.9 Hz, 2H), 5.79 (dt, *J* = 6.1, 2.1 Hz, 1H), 5.74 (dt, *J* = 6.0, 2.1 Hz, 1H), 5.70 (dd, *J* = 4.6, 2.2 Hz, 1H), 5.68 (dd, *J* = 4.5, 2.2 Hz, 1H), 2.35 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 143.40, 139.94, 138.76, 135.71, 133.69, 129.79, 129.33, 129.14, 128.85, 128.58, 128.49, 127.96, 127.71, 127.49, 70.78, 70.07, 21.50. FTIR (thin film) 3088, 3063, 3030, 2922, 1597, 1489, 1456, 1404, 1344, 1163, 1090, 1042, 814, 735, 598, 548 cm^{-1}. HRMS (ESI$^+$) *m/z* 410.0973 [calculated mass for $C_{23}H_{21}ClNO_2S$ $(M+H)^+$ 410.0976]; $[\alpha]_D^{20}$ = + 10.0° (*c* = 0.84, $CHCl_3$).

Single crystal x-ray structure included below, which the absolute configuration was based upon.

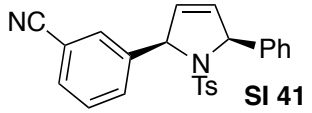
(2*R*,5*S*)-2-(4-(trifluoromethyl)phenyl)-5-phenyl-2,5-dihydropyrrole (SI 40)



SI 40

White solid, R_f = 0.48 (25% EtOAc: Hex)
^{1}H NMR (499 MHz, $CDCl_3$) δ 7.55 (d, *J* = 7.7 Hz, 1H), 7.49 (d, *J* = 8.6 Hz, 1H), 7.44 – 7.24 (m, 9H), 7.04 (d, *J* = 8.0 Hz, 2H), 5.83 (dt, *J* = 3.8, 2.2 Hz, 2H), 5.81 – 5.78 (m, 1H), 5.78 – 5.76 (m, 1H), 2.32 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 143.47, 141.13, 139.77, 135.84, 131.06, 130.73 (q, *J* = 32.3 Hz), 129.85, 129.27, 128.94, 128.54, 128.15, 127.89, 127.35, 126.45, 124.60 (q, *J* = 3.7 Hz), 124.38 (q, *J* = 3.7 Hz), 123.97 (q, *J* = 272.4 Hz), 70.71, 70.28, 21.42. ^{19}F NMR (282 MHz, $CDCl_3$) δ -65.88. FTIR (thin film) 2924, 2874, 1329, 1163, 1125, 1090, 1072, 812, 696, 665, 596, 544 cm^{-1}. HRMS (ESI^+) *m/z* 444.1233 [calculated mass for $C_{24}H_{21}F_3NO_2S$ $(M+H)^+$ 444.1240]; $[\alpha]_D^{20}$ = -1.5° (*c* = 1.04, $CHCl_3$).

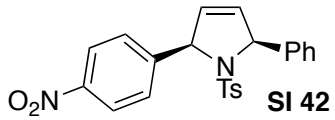
(2*R*,5*S*)-2-(4-cyanophenyl)-5-phenyl-2,5-dihydropyrrole (SI 41)



SI 41

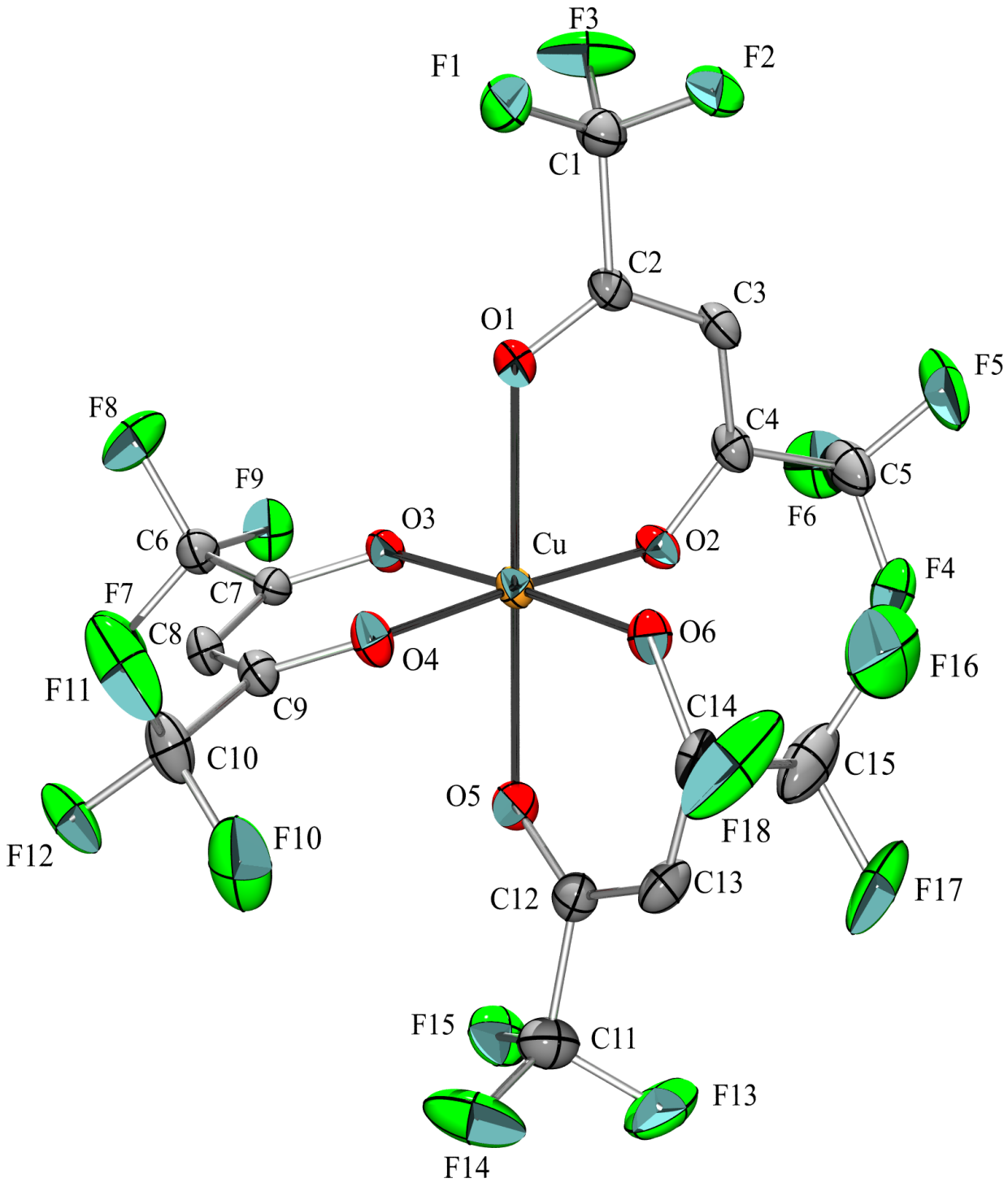
Yellow solid, R_f = 0.33 (25% EtOAc: Hex)
^{1}H NMR (499 MHz, $CDCl_3$) δ 7.64 – 7.58 (m, 2H), 7.56 – 7.50 (m, 2H), 7.44 – 7.37 (m, 2H), 7.37 – 7.29 (m, 2H), 7.14 – 7.08 (m, 1H), 5.87 – 5.69 (m, 4H), 2.36 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 143.74, 141.79, 139.59, 135.53, 132.11, 131.41, 131.15, 130.36, 129.42, 129.27, 128.63, 128.38, 128.23, 127.72, 127.46, 118.49, 112.55, 70.77, 69.93, 21.47. FTIR 3364, 2955, 2924, 2853, 2230, 1344, 1165, 1092, 1047, 1005, 841, 814, 762, 691, 667, 596, 544 cm^{-1}. HRMS (ESI^+) *m/z* 401.1318 [calculated mass for $C_{24}H_{21}N_2O_2S$ $(M+H)^+$ 401.1318]; $[\alpha]_D^{20}$ = 9.3° (*c* = 0.15, $CHCl_3$).

(2*R*,5*S*)-2-(4-nitrophenyl)-5-phenyl-2,5-dihydropyrrole (SI 42)



SI 42

Tan Solid, R_f = 0.33 (25% EtOAc: Hex)
^{1}H NMR (499 MHz, $CDCl_3$) δ 8.18 – 8.14 (m, 2H), 7.54 – 7.49 (m, 2H), 7.46 – 7.41 (m, 2H), 7.37 – 7.30 (m, 5H), 7.15 (d, *J* = 7.9 Hz, 2H), 5.85 (dt, *J* = 6.0, 2.1 Hz, 1H), 5.79 (dd, *J* = 4.0, 1.9 Hz, 1H), 5.77 (dt, *J* = 6.5, 2.1 Hz, 1H), 5.72 (dd, *J* = 4.3, 2.1 Hz, 1H), 2.37 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 147.44, 143.85, 139.53, 135.12, 130.41, 129.51, 129.01, 128.61, 128.29, 128.23, 127.71, 127.53, 124.33, 123.72, 70.89, 70.01, 21.50. FTIR (thin film) 3063, 2924, 2851, 1599, 1520, 1346, 1163, 1090, 829, 665, 598 cm^{-1}. HRMS (ESI^+) *m/z* 421.1221 [calculated mass for $C_{23}H_{21}NO_4S$ $(M+H)^+$ 421.1217]; $[\alpha]_D^{20}$ = + 21.9° (*c* = 0.32, $CHCl_3$).



$Cu(hfacac)_3NEt_4$ (tetraethylammonium salt and hydrogens omitted for clarity)

Table 1. Crystal data and structure refinement for $Cu(hfacac)_3NEt_4$.

Identification code	jtn101	
Chemical formula (moiety)	$C_8H_{20}N^+ \cdot C_{15}H_3CuF_{18}O_6^-$	
Chemical formula (total)	$C_{23}H_{23}CuF_{18}NO_6$	
Formula weight	814.96	
Temperature	200(2) K	
Radiation, wavelength	MoKα, 0.71073 Å	
Crystal system, space group	triclinic, $P\bar{1}$	
Unit cell parameters	a = 10.2817(7) Å	α = 88.673(3)°
	b = 10.9413(7) Å	β = 82.244(2)°
	c = 15.5048(11) Å	γ = 67.515(2)°
Cell volume	1596.10(19) $Å^3$	
Z	2	
Calculated density	1.696 g/cm^3	
Absorption coefficient μ	0.827 mm^{-1}	
F(000)	814	
Crystal colour and size	green, 0.98 × 0.25 × 0.18 mm^3	
Reflections for cell refinement	9893 (θ range 2.2 to 25.3°)	
Data collection method	Bruker Kappa APEXII DUO CCD diffractometer thin-slice ω scans	
θ range for data collection	2.0 to 25.4°	
Index ranges	h −12 to 12, k −13 to 13, l −18 to 18	
Completeness to θ = 25.4°	99.6 %	
Reflections collected	52748	
Independent reflections	5885 (R_{int} = 0.0272)	
Reflections with $F^2>2\sigma$	5088	
Absorption correction	numerical	
Min. and max. transmission	0.4994 and 0.8681	
Structure solution	direct methods	
Refinement method	Full-matrix least-squares on F^2	
Weighting parameters a, b	0.0559, 1.0159	
Data / restraints / parameters	5885 / 1243 / 620	
Final R indices [$F^2>2\sigma$]	R1 = 0.0356, wR2 = 0.0985	
R indices (all data)	R1 = 0.0433, wR2 = 0.1067	
Goodness-of-fit on F^2	1.043	
Largest and mean shift/su	0.001 and 0.000	
Largest diff. peak and hole	0.51 and −0.28 e $Å^{-3}$	

Table 2. Atomic coordinates and equivalent isotropic displacement parameters ($Å^2$) for $Cu(hfacac)_3NEt_4$. U_{eq} is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
Cu	0.86462(3)	0.72119(3)	0.725541(18)	0.03975(11)
C(6)	0.7657(3)	0.5992(3)	0.49066(18)	0.0531(6)
F(7)	0.6410(2)	0.6073(2)	0.47432(15)	0.0917(7)
F(8)	0.8254(3)	0.6383(2)	0.42061(12)	0.0963(7)
F(9)	0.8453(2)	0.47315(18)	0.49692(13)	0.0767(5)
C(1)	1.2797(3)	0.7507(3)	0.6293(2)	0.0606(7)
F(1)	1.2280(14)	0.8641(9)	0.5907(8)	0.092(3)
F(2)	1.3548(12)	0.7724(11)	0.6861(6)	0.085(2)
F(3)	1.3669(16)	0.6632(13)	0.5734(11)	0.124(4)
C(1')	1.2797(3)	0.7507(3)	0.6293(2)	0.0606(7)
F(1')	1.229(2)	0.8756(10)	0.6121(15)	0.100(4)
F(2')	1.3942(18)	0.724(3)	0.6635(15)	0.118(6)
F(3')	1.322(3)	0.689(2)	0.5518(9)	0.101(5)
C(5)	1.1996(4)	0.4200(4)	0.8267(3)	0.0777(10)
F(4)	1.1092(12)	0.4266(13)	0.8972(6)	0.100(3)
F(5)	1.3137(19)	0.416(2)	0.8542(16)	0.140(5)
F(6)	1.2185(15)	0.3066(9)	0.7868(9)	0.105(3)
C(5')	1.1996(4)	0.4200(4)	0.8267(3)	0.0777(10)
F(4')	1.151(3)	0.440(2)	0.9078(8)	0.163(7)
F(5')	1.3410(9)	0.3867(15)	0.8220(14)	0.097(4)
F(6')	1.1929(14)	0.3122(11)	0.7995(13)	0.113(4)
C(10)	0.5121(4)	1.0318(3)	0.6471(2)	0.0720(9)
F(10)	0.4468(17)	1.0579(17)	0.7307(5)	0.144(5)
F(11)	0.5407(15)	1.1302(11)	0.6194(14)	0.133(4)
F(12)	0.4075(14)	1.023(2)	0.6109(10)	0.083(4)
C(10')	0.5121(4)	1.0318(3)	0.6471(2)	0.0720(9)
F(10')	0.4876(15)	1.0944(13)	0.7197(8)	0.084(3)
F(11')	0.5677(17)	1.1081(12)	0.5899(7)	0.072(3)
F(12')	0.403(2)	1.043(3)	0.6067(13)	0.067(3)
C(11)	0.4947(4)	0.6876(4)	0.8954(3)	0.0839(10)
F(13)	0.501(2)	0.640(2)	0.9761(6)	0.130(4)
F(14)	0.3809(13)	0.7895(12)	0.8955(17)	0.153(5)
F(15)	0.4873(14)	0.5936(12)	0.8478(9)	0.093(3)
C(11')	0.4947(4)	0.6876(4)	0.8954(3)	0.0839(10)
F(13')	0.454(2)	0.701(3)	0.9791(8)	0.153(6)
F(14')	0.3882(14)	0.7875(15)	0.8638(15)	0.119(5)
F(15')	0.491(2)	0.5801(16)	0.8667(18)	0.125(5)
C(15)	0.8175(6)	0.8887(5)	0.9787(2)	0.1007(14)
F(16)	0.9461(12)	0.862(2)	0.9787(8)	0.194(6)
F(17)	0.7499(16)	0.8830(17)	1.0548(6)	0.120(4)
F(18)	0.756(2)	1.0226(8)	0.9602(5)	0.165(5)

C(15')	0.8175(6)	0.8887(5)	0.9787(2)	0.1007(14)
F(16')	0.9444(13)	0.7964(11)	1.0053(9)	0.118(4)
F(17')	0.728(2)	0.913(2)	1.0523(10)	0.116(5)
F(18')	0.8528(18)	0.9861(14)	0.9596(7)	0.111(4)
O(1)	1.04367(18)	0.76697(18)	0.65937(12)	0.0492(4)
O(2)	1.00473(18)	0.55542(17)	0.76367(13)	0.0496(4)
O(3)	0.85283(17)	0.63320(18)	0.61620(11)	0.0484(4)
O(4)	0.7199(2)	0.89097(18)	0.69585(13)	0.0534(5)
O(5)	0.6866(2)	0.6714(2)	0.79099(13)	0.0582(5)
O(6)	0.8669(2)	0.80831(19)	0.83675(13)	0.0550(5)
C(2)	1.1650(3)	0.7065(3)	0.67715(17)	0.0453(6)
C(3)	1.2127(3)	0.6023(3)	0.7326(2)	0.0524(6)
C(4)	1.1308(3)	0.5367(3)	0.77006(19)	0.0499(6)
C(7)	0.7564(2)	0.6831(3)	0.57050(16)	0.0418(5)
C(8)	0.6452(3)	0.8055(3)	0.58122(17)	0.0472(6)
C(9)	0.6365(3)	0.8981(3)	0.64239(18)	0.0465(6)
C(12)	0.6301(3)	0.7133(3)	0.86490(18)	0.0540(6)
C(13)	0.6715(4)	0.7810(3)	0.92357(19)	0.0636(8)
C(14)	0.7839(4)	0.8197(3)	0.90579(18)	0.0564(7)
N2	0.8632(2)	0.2235(2)	0.73108(14)	0.0473(5)
C162	0.856(3)	0.3242(18)	0.7994(10)	0.045(4)
C172	0.758(3)	0.318(3)	0.8811(15)	0.074(6)
C16'2	0.875(2)	0.3247(14)	0.7917(11)	0.086(4)
C17'2	0.804(4)	0.335(2)	0.8855(10)	0.118(6)
C182	0.7148(4)	0.2531(4)	0.7156(3)	0.0928(12)
C192	0.6423(4)	0.3866(4)	0.6729(3)	0.0916(12)
C202	0.962(2)	0.2425(18)	0.6561(8)	0.071(3)
C212	0.9855(17)	0.1654(10)	0.5707(5)	0.069(3)
C20'2	0.945(2)	0.2198(19)	0.6436(10)	0.086(4)
C21'2	0.925(2)	0.1252(19)	0.5837(11)	0.102(6)
C222	0.9175(4)	0.0861(3)	0.7667(2)	0.0758(9)
C232	1.0642(4)	0.0405(4)	0.7922(3)	0.0888(12)

Table 3. Bond lengths [Å] and angles [°] for $Cu(hfacac)_3NEt_4$.

Cu–O(1)	2.2056(18)	Cu–O(2)	1.9739(18)
Cu–O(3)	2.0089(18)	Cu–O(4)	1.9769(18)
Cu–O(5)	2.216(2)	Cu–O(6)	1.9965(19)
C(6)–F(7)	1.310(3)	C(6)–F(8)	1.319(3)
C(6)–F(9)	1.316(3)	C(6)–C(7)	1.529(4)
C(1)–F(1)	1.314(7)	C(1)–F(2)	1.328(7)
C(1)–F(3)	1.282(9)	C(1)–C(2)	1.536(4)
C(5)–F(4)	1.321(9)	C(5)–F(5)	1.285(10)
C(5)–F(6)	1.333(8)	C(5)–C(4)	1.525(4)
C(10)–F(10)	1.359(10)	C(10)–F(11)	1.271(9)
C(10)–F(12)	1.315(8)	C(10)–C(9)	1.527(4)
C(11)–F(13)	1.345(9)	C(11)–F(14)	1.270(10)
C(11)–F(15)	1.311(8)	C(11)–C(12)	1.535(5)
C(15)–F(16)	1.240(10)	C(15)–F(17)	1.299(9)
C(15)–F(18)	1.396(10)	C(15)–C(14)	1.521(5)
O(1)–C(2)	1.234(3)	O(2)–C(4)	1.250(3)
O(3)–C(7)	1.241(3)	O(4)–C(9)	1.251(3)
O(5)–C(12)	1.226(3)	O(6)–C(14)	1.252(4)
C(2)–C(3)	1.386(4)	C(3)–H(3)	0.950
C(3)–C(4)	1.369(4)	C(7)–C(8)	1.384(4)
C(8)–H(8)	0.950	C(8)–C(9)	1.373(4)
C(12)–C(13)	1.390(4)	C(13)–H(13)	0.950
C(13)–C(14)	1.369(5)	N2–C162	1.522(13)
N2–C16'2	1.520(9)	N2–C182	1.485(4)
N2–C202	1.506(13)	N2–C20'2	1.491(15)
N2–C222	1.510(4)	C162–H16A2	0.990
C162–H16B2	0.990	C162–C172	1.525(13)
C172–H17A2	0.980	C172–H17B2	0.980
C172–H17C2	0.980	C16'2–H16C2	0.990
C16'2–H16D2	0.990	C16'2–C17'2	1.522(11)
C17'2–H17D2	0.980	C17'2–H17E2	0.980
C17'2–H17F2	0.980	C182–H18A2	0.990
C182–H18B2	0.990	C182–C192	1.544(5)
C192–H19A2	0.980	C192–H19B2	0.980
C192–H19C2	0.980	C202–H20A2	0.990
C202–H20B2	0.990	C202–C212	1.527(10)
C212–H21A2	0.980	C212–H21B2	0.980
C212–H21C2	0.980	C20'2–H20C2	0.990
C20'2–H20D2	0.990	C20'2–C21'2	1.495(12)
C21'2–H21D2	0.980	C21'2–H21E2	0.980
C21'2–H21F2	0.980	C222–H22A2	0.990
C222–H22B2	0.990	C222–C232	1.503(5)
C232–H23A2	0.980	C232–H23B2	0.980
C232–H23C2	0.980		

O(1)–Cu–O(2)	88.24(7)	O(1)–Cu–O(3)	90.88(7)
O(1)–Cu–O(4)	93.57(8)	O(1)–Cu–O(5)	178.94(7)
O(1)–Cu–O(6)	91.97(8)	O(2)–Cu–O(3)	92.29(8)
O(2)–Cu–O(4)	176.08(8)	O(2)–Cu–O(5)	91.02(7)
O(2)–Cu–O(6)	88.39(8)	O(3)–Cu–O(4)	91.16(7)
O(3)–Cu–O(5)	88.38(7)	O(3)–Cu–O(6)	177.09(7)
O(4)–Cu–O(5)	87.20(8)	O(4)–Cu–O(6)	88.07(8)
O(5)–Cu–O(6)	88.78(8)	F(7)–C(6)–F(8)	106.9(3)
F(7)–C(6)–F(9)	107.9(2)	F(7)–C(6)–C(7)	112.8(2)
F(8)–C(6)–F(9)	105.3(2)	F(8)–C(6)–C(7)	110.4(2)
F(9)–C(6)–C(7)	113.1(2)	F(1)–C(1)–F(2)	104.2(6)
F(1)–C(1)–F(3)	109.0(9)	F(1)–C(1)–C(2)	113.7(6)
F(2)–C(1)–F(3)	107.4(7)	F(2)–C(1)–C(2)	109.7(5)
F(3)–C(1)–C(2)	112.4(8)	F(4)–C(5)–F(5)	105.7(10)
F(4)–C(5)–F(6)	104.0(7)	F(4)–C(5)–C(4)	108.7(6)
F(5)–C(5)–F(6)	111.9(10)	F(5)–C(5)–C(4)	115.5(8)
F(6)–C(5)–C(4)	110.3(7)	F(10)–C(10)–F(11)	109.3(6)
F(10)–C(10)–F(12)	98.7(11)	F(10)–C(10)–C(9)	108.9(5)
F(11)–C(10)–F(12)	111.3(11)	F(11)–C(10)–C(9)	116.6(8)
F(12)–C(10)–C(9)	110.5(9)	F(13)–C(11)–F(14)	109.2(9)
F(13)–C(11)–F(15)	104.5(8)	F(13)–C(11)–C(12)	108.9(7)
F(14)–C(11)–F(15)	108.7(10)	F(14)–C(11)–C(12)	113.8(9)
F(15)–C(11)–C(12)	111.3(7)	F(16)–C(15)–F(17)	113.9(9)
F(16)–C(15)–F(18)	107.0(7)	F(16)–C(15)–C(14)	114.2(7)
F(17)–C(15)–F(18)	102.7(9)	F(17)–C(15)–C(14)	114.1(7)
F(18)–C(15)–C(14)	103.4(6)	Cu–O(1)–C(2)	120.23(17)
Cu–O(2)–C(4)	124.61(17)	Cu–O(3)–C(7)	123.27(16)
Cu–O(4)–C(9)	122.56(17)	Cu–O(5)–C(12)	121.27(19)
Cu–O(6)–C(14)	125.96(19)	C(1)–C(2)–O(1)	115.4(2)
C(1)–C(2)–C(3)	115.6(2)	O(1)–C(2)–C(3)	129.0(2)
C(2)–C(3)–H(3)	118.2	C(2)–C(3)–C(4)	123.6(2)
H(3)–C(3)–C(4)	118.2	C(5)–C(4)–O(2)	112.6(2)
C(5)–C(4)–C(3)	117.7(2)	O(2)–C(4)–C(3)	129.6(3)
C(6)–C(7)–O(3)	115.1(2)	C(6)–C(7)–C(8)	116.5(2)
O(3)–C(7)–C(8)	128.4(2)	C(7)–C(8)–H(8)	118.9
C(7)–C(8)–C(9)	122.2(2)	H(8)–C(8)–C(9)	118.9
C(10)–C(9)–O(4)	112.7(2)	C(10)–C(9)–C(8)	118.2(2)
O(4)–C(9)–C(8)	129.1(2)	C(11)–C(12)–O(5)	114.7(3)
C(11)–C(12)–C(13)	116.5(3)	O(5)–C(12)–C(13)	128.8(3)
C(12)–C(13)–H(13)	117.7	C(12)–C(13)–C(14)	124.6(3)
H(13)–C(13)–C(14)	117.7	C(15)–C(14)–O(6)	112.2(3)
C(15)–C(14)–C(13)	117.7(3)	O(6)–C(14)–C(13)	130.1(3)

Table 4. Anisotropic displacement parameters ($Å^2$) for $Cu(hfacac)_3NEt_4$. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ...+ 2hka^*b^*U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Cu	0.03653(17)	0.04012(18)	0.04285(18)	−0.00317(12)	−0.00871(12)	−0.01359(12)
C(6)	0.0495(14)	0.0545(16)	0.0514(15)	−0.0046(12)	−0.0069(12)	−0.0152(12)
F(7)	0.0614(11)	0.1047(16)	0.1043(16)	−0.0502(13)	−0.0225(10)	−0.0200(11)
F(8)	0.145(2)	0.1030(16)	0.0473(10)	−0.0109(10)	0.0139(12)	−0.0626(15)
F(9)	0.0892(13)	0.0564(11)	0.0735(12)	−0.0140(9)	−0.0169(10)	−0.0130(9)
C(1)	0.0530(16)	0.0635(18)	0.0694(19)	0.0054(15)	−0.0073(14)	−0.0273(14)
F(1)	0.088(5)	0.111(5)	0.097(4)	0.056(4)	−0.032(4)	−0.055(4)
F(2)	0.072(4)	0.113(5)	0.102(4)	0.023(3)	−0.026(3)	−0.068(4)
F(3)	0.095(6)	0.105(5)	0.163(9)	−0.048(6)	0.071(6)	−0.054(4)
C(1')	0.0530(16)	0.0635(18)	0.0694(19)	0.0054(15)	−0.0073(14)	−0.0273(14)
F(1')	0.094(8)	0.054(4)	0.156(12)	−0.004(5)	0.020(6)	−0.044(4)
F(2')	0.055(6)	0.200(15)	0.123(10)	0.073(9)	−0.033(6)	−0.073(8)
F(3')	0.118(12)	0.121(9)	0.074(5)	−0.024(5)	0.036(5)	−0.073(8)
C(5)	0.063(2)	0.070(2)	0.110(3)	0.034(2)	−0.033(2)	−0.0314(18)
F(4)	0.106(5)	0.115(5)	0.076(4)	0.051(3)	−0.031(3)	−0.035(4)
F(5)	0.132(7)	0.157(12)	0.190(13)	0.116(8)	−0.117(8)	−0.096(8)
F(6)	0.103(6)	0.044(4)	0.142(6)	0.001(4)	−0.001(5)	−0.004(3)
C(5')	0.063(2)	0.070(2)	0.110(3)	0.034(2)	−0.033(2)	−0.0314(18)
F(4')	0.148(11)	0.187(12)	0.099(5)	0.062(5)	−0.037(6)	0.001(8)
F(5')	0.057(4)	0.079(5)	0.166(11)	0.055(6)	−0.062(4)	−0.026(3)
F(6')	0.072(5)	0.065(5)	0.229(12)	0.068(6)	−0.076(6)	−0.043(4)
C(10)	0.069(2)	0.0458(17)	0.098(3)	−0.0057(16)	−0.0401(18)	−0.0090(14)
F(10)	0.117(7)	0.122(8)	0.106(4)	−0.048(4)	−0.029(3)	0.056(5)
F(11)	0.108(6)	0.041(3)	0.272(12)	0.011(6)	−0.105(7)	−0.028(4)
F(12)	0.052(3)	0.042(5)	0.159(8)	0.008(3)	−0.044(4)	−0.011(3)
C(10')	0.069(2)	0.0458(17)	0.098(3)	−0.0057(16)	−0.0401(18)	−0.0090(14)
F(10')	0.075(5)	0.062(5)	0.099(5)	−0.026(4)	−0.023(4)	−0.002(4)
F(11')	0.097(6)	0.038(5)	0.093(6)	0.020(3)	−0.049(4)	−0.030(4)
F(12')	0.055(5)	0.041(8)	0.101(8)	0.002(4)	−0.035(4)	−0.006(4)
C(11)	0.071(2)	0.092(3)	0.086(3)	−0.013(2)	0.0074(19)	−0.033(2)
F(13)	0.159(10)	0.203(10)	0.079(4)	0.010(4)	0.014(4)	−0.135(9)
F(14)	0.070(4)	0.107(5)	0.246(15)	−0.036(6)	0.045(6)	−0.013(4)
F(15)	0.084(5)	0.108(5)	0.106(5)	−0.016(4)	−0.020(4)	−0.056(4)
C(11')	0.071(2)	0.092(3)	0.086(3)	−0.013(2)	0.0074(19)	−0.033(2)
F(13')	0.108(9)	0.276(19)	0.094(5)	−0.033(7)	0.028(5)	−0.105(11)
F(14')	0.051(5)	0.123(7)	0.172(11)	−0.025(6)	−0.032(7)	−0.014(4)
F(15')	0.115(10)	0.095(6)	0.172(13)	−0.015(7)	0.036(8)	−0.065(6)
C(15)	0.182(4)	0.108(3)	0.052(2)	0.004(2)	−0.019(2)	−0.099(3)
F(16)	0.231(8)	0.342(17)	0.114(7)	−0.017(10)	−0.055(6)	−0.213(9)

F(17)	0.246(10)	0.135(8)	0.039(3)	0.016(4)	−0.036(4)	−0.135(8)
F(18)	0.366(15)	0.112(4)	0.067(3)	−0.027(3)	0.026(6)	−0.163(7)
C(15')	0.182(4)	0.108(3)	0.052(2)	0.004(2)	−0.019(2)	−0.099(3)
F(16')	0.148(7)	0.130(7)	0.107(7)	0.017(4)	−0.084(6)	−0.066(5)
F(17')	0.188(10)	0.123(10)	0.054(6)	−0.033(6)	0.018(6)	−0.086(7)
F(18')	0.206(13)	0.118(7)	0.070(4)	0.020(5)	−0.042(7)	−0.125(8)
O(1)	0.0419(9)	0.0506(10)	0.0539(10)	0.0030(8)	−0.0116(8)	−0.0149(8)
O(2)	0.0408(9)	0.0446(10)	0.0683(12)	0.0046(8)	−0.0115(8)	−0.0206(8)
O(3)	0.0360(8)	0.0546(10)	0.0487(10)	−0.0047(8)	−0.0071(7)	−0.0100(8)
O(4)	0.0514(10)	0.0484(10)	0.0608(11)	−0.0061(8)	−0.0201(9)	−0.0148(8)
O(5)	0.0548(11)	0.0634(12)	0.0519(11)	−0.0137(9)	−0.0050(9)	−0.0177(9)
O(6)	0.0594(11)	0.0543(11)	0.0545(11)	−0.0003(9)	−0.0147(9)	−0.0230(9)
C(2)	0.0400(13)	0.0465(14)	0.0516(14)	−0.0065(11)	−0.0063(10)	−0.0184(11)
C(3)	0.0381(13)	0.0513(15)	0.0724(18)	0.0070(13)	−0.0189(12)	−0.0189(11)
C(4)	0.0474(14)	0.0435(14)	0.0632(16)	0.0028(12)	−0.0167(12)	−0.0191(11)
C(7)	0.0365(12)	0.0504(14)	0.0411(12)	0.0017(10)	−0.0042(10)	−0.0200(10)
C(8)	0.0444(13)	0.0482(14)	0.0508(14)	0.0022(11)	−0.0169(11)	−0.0164(11)
C(9)	0.0432(13)	0.0444(13)	0.0540(15)	0.0045(11)	−0.0121(11)	−0.0174(11)
C(12)	0.0562(15)	0.0478(15)	0.0516(16)	−0.0014(12)	−0.0055(12)	−0.0135(12)
C(13)	0.088(2)	0.0615(18)	0.0418(15)	−0.0047(13)	0.0029(14)	−0.0332(16)
C(14)	0.086(2)	0.0469(15)	0.0403(14)	0.0052(11)	−0.0182(14)	−0.0269(14)
N2	0.0497(12)	0.0427(12)	0.0561(13)	0.0040(10)	−0.0095(10)	−0.0243(10)
C162	0.046(7)	0.040(6)	0.048(7)	0.003(5)	−0.003(4)	−0.017(5)
C172	0.090(12)	0.074(8)	0.044(7)	0.005(6)	0.008(6)	−0.020(8)
C16'2	0.097(9)	0.069(5)	0.102(7)	−0.023(5)	−0.019(6)	−0.040(5)
C17'2	0.128(14)	0.109(9)	0.081(6)	−0.029(6)	−0.018(6)	−0.003(7)
C182	0.071(2)	0.084(3)	0.139(4)	0.000(2)	−0.036(2)	−0.039(2)
C192	0.068(2)	0.085(3)	0.100(3)	0.002(2)	−0.025(2)	−0.0019(19)
C202	0.087(6)	0.080(6)	0.044(4)	−0.011(4)	0.011(4)	−0.034(5)
C212	0.091(7)	0.062(5)	0.040(3)	−0.006(3)	−0.009(3)	−0.014(4)
C20'2	0.081(6)	0.087(9)	0.060(6)	0.021(5)	−0.011(5)	0.001(6)
C21'2	0.099(9)	0.091(9)	0.094(7)	−0.012(6)	−0.027(7)	−0.007(7)
C222	0.101(3)	0.0509(17)	0.081(2)	0.0107(16)	−0.021(2)	−0.0329(18)
C232	0.101(3)	0.067(2)	0.083(2)	−0.0061(18)	−0.045(2)	−0.004(2)

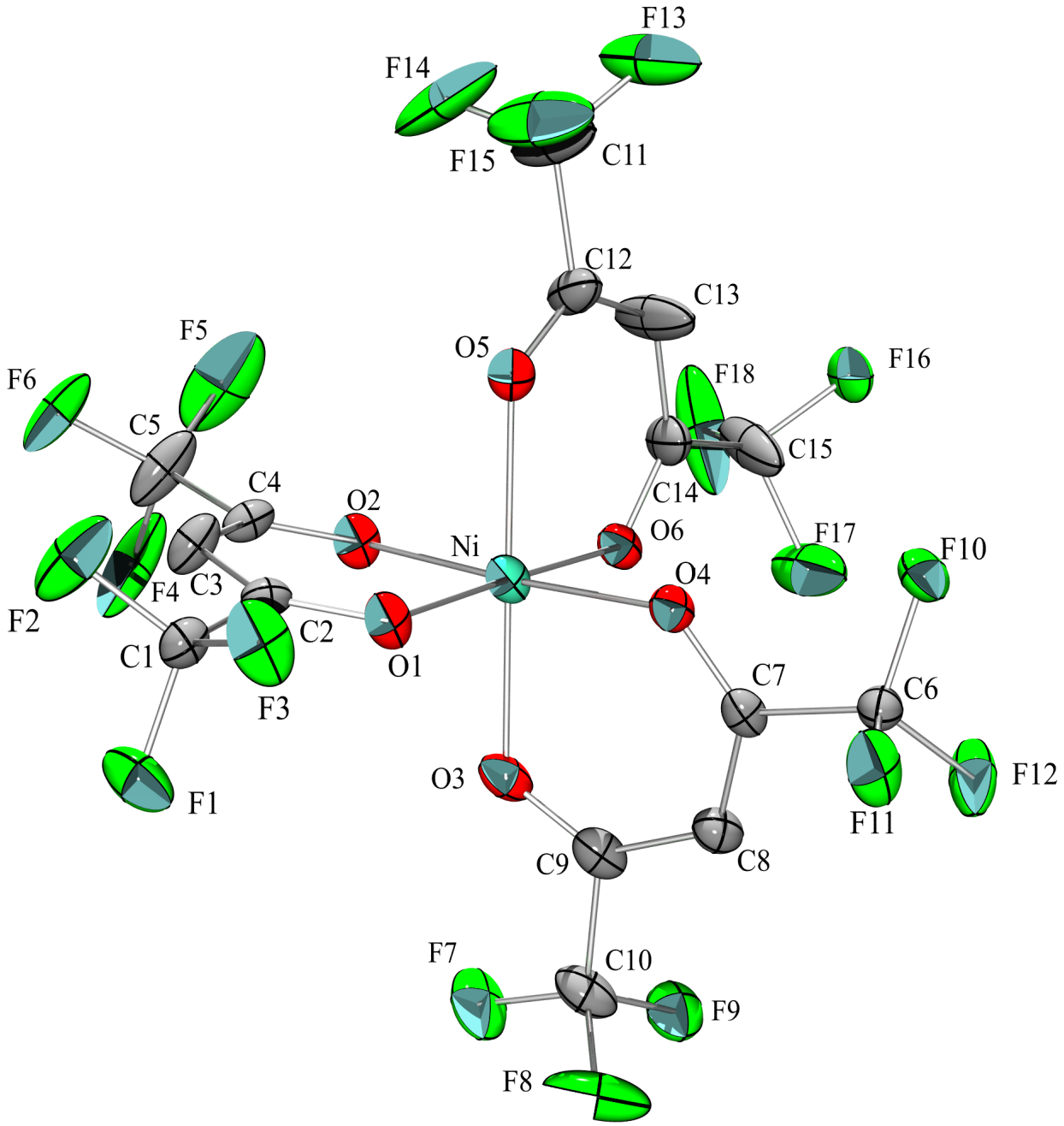
Table 5. Hydrogen coordinates and isotropic displacement parameters ($Å^2$) for $Cu(hfacac)_3NEt_4$.

	x	y	z	U
H(3)	1.3074	0.5749	0.7453	0.063
H(8)	0.5726	0.8262	0.5450	0.057
H(13)	0.6180	0.8018	0.9800	0.076
H16A2	0.8202	0.4142	0.7758	0.054
H16B2	0.9526	0.3053	0.8142	0.054
H17A2	0.7547	0.3833	0.9248	0.111
H17B2	0.7944	0.2295	0.9046	0.111
H17C2	0.6624	0.3388	0.8665	0.111
H16C2	0.8345	0.4129	0.7662	0.103
H16D2	0.9774	0.3046	0.7927	0.103
H17D2	0.8167	0.4050	0.9171	0.177
H17E2	0.8478	0.2501	0.9135	0.177
H17F2	0.7027	0.3549	0.8861	0.177
H18A2	0.6573	0.2523	0.7720	0.111
H18B2	0.7146	0.1814	0.6779	0.111
H19A2	0.5450	0.3984	0.6659	0.137
H19B2	0.6958	0.3874	0.6157	0.137
H19C2	0.6402	0.4589	0.7100	0.137
H20A2	1.0563	0.2195	0.6759	0.085
H20B2	0.9268	0.3379	0.6433	0.085
H21A2	1.0599	0.1797	0.5302	0.103
H21B2	0.8969	0.1963	0.5450	0.103
H21C2	1.0146	0.0709	0.5821	0.103
H20C2	1.0475	0.1918	0.6491	0.103
H20D2	0.9126	0.3093	0.6193	0.103
H21D2	0.9746	0.1277	0.5254	0.153
H21E2	0.8238	0.1503	0.5809	0.153
H21F2	0.9648	0.0354	0.6056	0.153
H22A2	0.8508	0.0832	0.8183	0.091
H22B2	0.9171	0.0228	0.7222	0.091
H23A2	1.0904	–0.0487	0.8150	0.133
H23B2	1.0658	0.1013	0.8372	0.133
H23C2	1.1322	0.0394	0.7411	0.133

Table 6. Torsion angles [°] for $Cu(hfacac)_3NEt_4$.

O(2)–Cu–O(1)–C(2)	−15.72(19)	O(3)–Cu–O(1)–C(2)	−107.98(19)
O(4)–Cu–O(1)–C(2)	160.80(19)	O(5)–Cu–O(1)–C(2)	−62(4)
O(6)–Cu–O(1)–C(2)	72.61(19)	O(1)–Cu–O(2)–C(4)	22.9(2)
O(3)–Cu–O(2)–C(4)	113.7(2)	O(4)–Cu–O(2)–C(4)	−94.8(11)
O(5)–Cu–O(2)–C(4)	−157.9(2)	O(6)–Cu–O(2)–C(4)	−69.1(2)
O(1)–Cu–O(3)–C(7)	−106.94(19)	O(2)–Cu–O(3)–C(7)	164.78(19)
O(4)–Cu–O(3)–C(7)	−13.3(2)	O(5)–Cu–O(3)–C(7)	73.8(2)
O(6)–Cu–O(3)–C(7)	61.3(15)	O(1)–Cu–O(4)–C(9)	110.3(2)
O(2)–Cu–O(4)–C(9)	−132.2(10)	O(3)–Cu–O(4)–C(9)	19.3(2)
O(5)–Cu–O(4)–C(9)	−69.0(2)	O(6)–Cu–O(4)–C(9)	−157.9(2)
O(1)–Cu–O(5)–C(12)	141(4)	O(2)–Cu–O(5)–C(12)	95.0(2)
O(3)–Cu–O(5)–C(12)	−172.8(2)	O(4)–Cu–O(5)–C(12)	−81.5(2)
O(6)–Cu–O(5)–C(12)	6.6(2)	O(1)–Cu–O(6)–C(14)	178.8(2)
O(2)–Cu–O(6)–C(14)	−93.0(2)	O(3)–Cu–O(6)–C(14)	10.6(16)
O(4)–Cu–O(6)–C(14)	85.3(2)	O(5)–Cu–O(6)–C(14)	−2.0(2)
Cu–O(1)–C(2)–C(1)	−177.85(17)	Cu–O(1)–C(2)–C(3)	3.9(4)
F(1)–C(1)–C(2)–O(1)	13.8(7)	F(1)–C(1)–C(2)–C(3)	−167.8(7)
F(2)–C(1)–C(2)–O(1)	130.0(6)	F(2)–C(1)–C(2)–C(3)	−51.6(7)
F(3)–C(1)–C(2)–O(1)	−110.6(11)	F(3)–C(1)–C(2)–C(3)	67.8(11)
C(1)–C(2)–C(3)–C(4)	−169.6(3)	O(1)–C(2)–C(3)–C(4)	8.6(5)
Cu–O(2)–C(4)–C(5)	162.0(2)	Cu–O(2)–C(4)–C(3)	−19.9(4)
C(2)–C(3)–C(4)–C(5)	177.4(3)	C(2)–C(3)–C(4)–O(2)	−0.6(5)
F(4)–C(5)–C(4)–O(2)	−44.3(7)	F(4)–C(5)–C(4)–C(3)	137.3(7)
F(5)–C(5)–C(4)–O(2)	−162.8(15)	F(5)–C(5)–C(4)–C(3)	18.8(15)
F(6)–C(5)–C(4)–O(2)	69.1(7)	F(6)–C(5)–C(4)–C(3)	−109.2(7)
Cu–O(3)–C(7)–C(6)	−179.12(16)	Cu–O(3)–C(7)–C(8)	3.0(4)
F(7)–C(6)–C(7)–O(3)	142.9(3)	F(7)–C(6)–C(7)–C(8)	−39.0(3)
F(8)–C(6)–C(7)–O(3)	−97.6(3)	F(8)–C(6)–C(7)–C(8)	80.5(3)
F(9)–C(6)–C(7)–O(3)	20.1(3)	F(9)–C(6)–C(7)–C(8)	−161.7(2)
C(6)–C(7)–C(8)–C(9)	−170.3(2)	O(3)–C(7)–C(8)–C(9)	7.6(4)
Cu–O(4)–C(9)–C(10)	165.5(2)	Cu–O(4)–C(9)–C(8)	−16.4(4)
C(7)–C(8)–C(9)–C(10)	178.1(3)	C(7)–C(8)–C(9)–O(4)	0.1(5)
F(10)–C(10)–C(9)–O(4)	−53.6(12)	F(10)–C(10)–C(9)–C(8)	128.1(12)
F(11)–C(10)–C(9)–O(4)	70.6(10)	F(11)–C(10)–C(9)–C(8)	−107.7(10)
F(12)–C(10)–C(9)–O(4)	−161.0(9)	F(12)–C(10)–C(9)–C(8)	20.7(9)
Cu–O(5)–C(12)–C(11)	171.1(2)	Cu–O(5)–C(12)–C(13)	−8.3(4)
F(13)–C(11)–C(12)–O(5)	133.3(10)	F(13)–C(11)–C(12)–C(13)	−47.2(10)
F(14)–C(11)–C(12)–O(5)	−104.7(13)	F(14)–C(11)–C(12)–C(13)	74.9(13)
F(15)–C(11)–C(12)–O(5)	18.6(8)	F(15)–C(11)–C(12)–C(13)	−161.9(8)
C(11)–C(12)–C(13)–C(14)	−175.8(3)	O(5)–C(12)–C(13)–C(14)	3.7(5)
Cu–O(6)–C(14)–C(15)	178.7(2)	Cu–O(6)–C(14)–C(13)	−1.8(5)
C(12)–C(13)–C(14)–C(15)	−177.9(3)	C(12)–C(13)–C(14)–O(6)	2.6(6)

F(16)–C(15)–C(14)–O(6)	−35.7(12)	F(16)–C(15)–C(14)–C(13)	144.7(12)
F(17)–C(15)–C(14)–O(6)	−169.1(9)	F(17)–C(15)–C(14)–C(13)	11.3(10)
F(18)–C(15)–C(14)–O(6)	80.2(8)	F(18)–C(15)–C(14)–C(13)	−99.4(8)
C182–N2–C162–C172	56.3(18)		



Ni(hfacac)$_3$NEt$_4$ (tetraethylammonium salt and hydrogens omitted for clarity)

Table 1. Crystal data and structure refinement for $Ni(hfacac)_3NEt_4$.

Identification code	jtn102	
Chemical formula (moiety)	$C_8H_{20}N^+ \cdot C_{15}H_3F_{18}NiO_6^- \cdot 0.5C_6H_{14}$	
Chemical formula (total)	$C_{26}H_{30}F_{18}NNiO_6$	
Formula weight	853.22	
Temperature	103(2) K	
Radiation, wavelength	MoKα, 0.71073 Å	
Crystal system, space group	triclinic, $P\bar{1}$	
Unit cell parameters	a = 9.9155(6) Å	α = 83.547(3)°
	b = 10.9991(6) Å	β = 90.332(3)°
	c = 15.6366(9) Å	γ = 70.468(3)°
Cell volume	1595.32(16) $Å^3$	
Z	2	
Calculated density	1.776 g/cm^3	
Absorption coefficient μ	0.756 mm^{-1}	
F(000)	862	
Crystal colour and size	colourless, 0.89 × 0.15 × 0.06 mm^3	
Reflections for cell refinement	9952 (θ range 2.2 to 26.8°)	
Data collection method	Bruker Kappa APEXII DUO CCD diffractometer	
	thin-slice ω scans	
θ range for data collection	2.0 to 28.3°	
Index ranges	h −13 to 13, k −14 to 14, l 0 to 20	
Completeness to θ = 28.3°	99.9 %	
Reflections collected	81970	
Independent reflections	7908 (R_{int} = 0.0425)	
Reflections with $F^2>2\sigma$	6539	
Absorption correction	numerical	
Min. and max. transmission	0.5520 and 0.9561	
Structure solution	direct methods	
Refinement method	Full-matrix least-squares on F^2	
Weighting parameters a, b	0.0247, 2.8243	
Data / restraints / parameters	7908 / 95 / 502	
Final R indices [$F^2>2\sigma$]	R1 = 0.0442, wR2 = 0.0977	
R indices (all data)	R1 = 0.0555, wR2 = 0.1028	
Goodness-of-fit on F^2	1.065	
Largest and mean shift/su	0.001 and 0.000	
Largest diff. peak and hole	0.85 and −0.55 e $Å^{-3}$	

Table 2. Atomic coordinates and equivalent isotropic displacement parameters ($Å^2$) for $Ni(hfacac)_3NEt_4$. U_{eq} is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
Ni	0.92953(3)	0.15884(3)	0.72160(2)	0.02194(8)
C(1)	0.7589(2)	0.0878(2)	0.48522(16)	0.0223(5)
F(1)	0.8222(2)	0.12992(17)	0.41896(10)	0.0401(4)
F(2)	0.62382(18)	0.11179(19)	0.45922(14)	0.0503(5)
F(3)	0.8218(2)	–0.03994(14)	0.49828(11)	0.0401(4)
F(4)	0.6008(2)	0.57963(16)	0.59508(19)	0.0693(8)
F(5)	0.5266(2)	0.5083(2)	0.71206(18)	0.0805(9)
F(6)	0.43878(16)	0.49119(16)	0.58977(14)	0.0462(5)
F(7)	1.2338(2)	0.34293(18)	0.58115(12)	0.0490(5)
F(8)	1.3909(3)	0.1553(2)	0.57283(14)	0.0613(7)
F(9)	1.39575(19)	0.25349(17)	0.68148(11)	0.0395(4)
F(10)	1.23961(17)	–0.21333(15)	0.86853(10)	0.0337(4)
F(11)	1.33857(19)	–0.25421(15)	0.75001(10)	0.0384(4)
F(12)	1.42750(19)	–0.16522(18)	0.83966(13)	0.0469(5)
C(11)	0.6342(4)	0.0686(4)	0.8962(2)	0.0519(9)
F(13)	0.6426(18)	0.035(2)	0.9813(4)	0.069(3)
F(14)	0.5135(9)	0.1667(13)	0.8744(9)	0.085(2)
F(15)	0.625(2)	–0.0325(15)	0.8609(11)	0.068(3)
C(11')	0.6342(4)	0.0686(4)	0.8962(2)	0.0519(9)
F(13')	0.607(3)	0.085(3)	0.9775(11)	0.058(4)
F(14')	0.5081(18)	0.141(2)	0.8565(18)	0.062(4)
F(15')	0.657(2)	–0.0550(13)	0.8819(17)	0.042(4)
C(15)	1.0051(4)	0.2558(5)	0.97172(19)	0.0585(11)
F(16)	1.0280(15)	0.1723(12)	1.0435(5)	0.0354(19)
F(17)	1.1524(6)	0.2280(9)	0.9503(2)	0.072(3)
F(18)	0.9466(11)	0.3648(6)	0.9812(5)	0.090(3)
C(15')	1.0051(4)	0.2558(5)	0.97172(19)	0.0585(11)
F(16')	1.0545(19)	0.1785(15)	1.0401(9)	0.048(4)
F(17')	1.0818(7)	0.3335(7)	0.9477(3)	0.0430(19)
F(18')	0.8856(7)	0.3615(6)	1.0084(4)	0.0398(14)
O(1)	0.87988(19)	0.09328(16)	0.61346(11)	0.0255(4)
O(2)	0.77537(19)	0.33407(16)	0.68867(11)	0.0252(4)
O(3)	1.0780(2)	0.22110(16)	0.65857(11)	0.0277(4)
O(4)	1.07895(18)	–0.01700(16)	0.76113(11)	0.0218(3)
O(5)	0.78057(18)	0.10058(16)	0.78679(11)	0.0235(3)
O(6)	0.98400(18)	0.22526(16)	0.82786(11)	0.0236(4)
C(2)	0.7735(2)	0.1535(2)	0.56489(15)	0.0190(4)
C(3)	0.6692(3)	0.2739(2)	0.57093(18)	0.0271(5)
C(4)	0.6794(2)	0.3522(2)	0.63261(16)	0.0218(5)
C(5)	0.5592(3)	0.4831(3)	0.6320(2)	0.0428(8)
C(6)	1.3053(2)	–0.1672(2)	0.80587(15)	0.0209(5)

C(7)	1.2103(3)	−0.0342(2)	0.76120(14)	0.0200(4)
C(8)	1.2804(3)	0.0471(2)	0.72340(16)	0.0239(5)
C(9)	1.2089(3)	0.1639(2)	0.67266(16)	0.0248(5)
C(10)	1.3070(3)	0.2302(3)	0.62628(17)	0.0323(6)
C(12)	0.7602(3)	0.1100(3)	0.86400(17)	0.0302(6)
C(13)	0.8338(4)	0.1538(5)	0.92238(19)	0.0576(11)
C(14)	0.9380(3)	0.2075(3)	0.89939(16)	0.0320(6)
N	0.9186(2)	0.6583(2)	0.71568(16)	0.0305(5)
C(16)	0.8348(3)	0.5873(3)	0.7690(2)	0.0400(7)
C(17)	0.8020(4)	0.6270(3)	0.8577(2)	0.0490(8)
C(18)	0.9389(3)	0.6031(3)	0.6300(2)	0.0367(7)
C(19)	1.0319(3)	0.6506(3)	0.5682(2)	0.0411(7)
C(20)	1.0613(3)	0.6398(3)	0.7601(2)	0.0411(7)
C(21)	1.1615(4)	0.5000(4)	0.7766(3)	0.0622(11)
C(22)	0.8402(3)	0.8045(2)	0.7033(2)	0.0333(6)
C(23)	0.6887(3)	0.8472(3)	0.6657(2)	0.0437(8)

Table 3. Bond lengths [Å] and angles [°] for $Ni(hfacac)_3NEt_4$.

Ni–O(1)	2.0306(17)	Ni–O(2)	2.0284(17)
Ni–O(3)	2.0366(18)	Ni–O(4)	2.0303(16)
Ni–O(5)	2.0334(18)	Ni–O(6)	2.0279(17)
C(1)–F(1)	1.332(3)	C(1)–F(2)	1.327(3)
C(1)–F(3)	1.324(3)	C(1)–C(2)	1.538(3)
F(4)–C(5)	1.337(4)	F(5)–C(5)	1.330(4)
F(6)–C(5)	1.333(3)	F(7)–C(10)	1.325(3)
F(8)–C(10)	1.332(3)	F(9)–C(10)	1.333(3)
F(10)–C(6)	1.326(3)	F(11)–C(6)	1.329(3)
F(12)–C(6)	1.327(3)	C(11)–F(13)	1.336(7)
C(11)–F(14)	1.327(8)	C(11)–F(15)	1.323(7)
C(11)–C(12)	1.536(4)	C(15)–F(16)	1.334(8)
C(15)–F(17)	1.439(6)	C(15)–F(18)	1.171(7)
C(15)–C(14)	1.538(4)	O(1)–C(2)	1.241(3)
O(2)–C(4)	1.240(3)	O(3)–C(9)	1.244(3)
O(4)–C(7)	1.252(3)	O(5)–C(12)	1.235(3)
O(6)–C(14)	1.230(3)	C(2)–C(3)	1.394(3)
C(3)–H(3)	0.950	C(3)–C(4)	1.388(3)
C(4)–C(5)	1.530(3)	C(6)–C(7)	1.535(3)
C(7)–C(8)	1.390(3)	C(8)–H(8)	0.950
C(8)–C(9)	1.393(3)	C(9)–C(10)	1.539(4)
C(12)–C(13)	1.387(4)	C(13)–H(13)	0.950
C(13)–C(14)	1.382(4)	N–C(16)	1.514(4)
N–C(18)	1.519(4)	N–C(20)	1.513(3)
N–C(22)	1.523(3)	C(16)–H(16A)	0.990
C(16)–H(16B)	0.990	C(16)–C(17)	1.505(5)
C(17)–H(17A)	0.980	C(17)–H(17B)	0.980
C(17)–H(17C)	0.980	C(18)–H(18A)	0.990
C(18)–H(18B)	0.990	C(18)–C(19)	1.509(4)
C(19)–H(19A)	0.980	C(19)–H(19B)	0.980
C(19)–H(19C)	0.980	C(20)–H(20A)	0.990
C(20)–H(20B)	0.990	C(20)–C(21)	1.517(4)
C(21)–H(21A)	0.980	C(21)–H(21B)	0.980
C(21)–H(21C)	0.980	C(22)–H(22A)	0.990
C(22)–H(22B)	0.990	C(22)–C(23)	1.509(4)
C(23)–H(23A)	0.980	C(23)–H(23B)	0.980
C(23)–H(23C)	0.980		
O(1)–Ni–O(2)	90.78(7)	O(1)–Ni–O(3)	90.81(7)
O(1)–Ni–O(4)	91.18(7)	O(1)–Ni–O(5)	90.63(7)
O(1)–Ni–O(6)	178.33(7)	O(2)–Ni–O(3)	91.13(7)
O(2)–Ni–O(4)	176.78(7)	O(2)–Ni–O(5)	88.11(7)
O(2)–Ni–O(6)	89.50(7)	O(3)–Ni–O(4)	91.39(7)
O(3)–Ni–O(5)	178.39(7)	O(3)–Ni–O(6)	87.54(7)
O(4)–Ni–O(5)	89.32(7)	O(4)–Ni–O(6)	88.61(7)

O(5)–Ni–O(6)	91.02(7)	F(1)–C(1)–F(2)	106.7(2)
F(1)–C(1)–F(3)	106.19(19)	F(1)–C(1)–C(2)	110.43(19)
F(2)–C(1)–F(3)	107.6(2)	F(2)–C(1)–C(2)	113.27(19)
F(3)–C(1)–C(2)	112.3(2)	F(13)–C(11)–F(14)	109.4(7)
F(13)–C(11)–F(15)	106.3(6)	F(13)–C(11)–C(12)	112.8(5)
F(14)–C(11)–F(15)	107.3(5)	F(14)–C(11)–C(12)	108.4(7)
F(15)–C(11)–C(12)	112.5(6)	F(16)–C(15)–F(17)	97.4(7)
F(16)–C(15)–F(18)	114.9(8)	F(16)–C(15)–C(14)	110.3(7)
F(17)–C(15)–F(18)	113.5(5)	F(17)–C(15)–C(14)	104.5(3)
F(18)–C(15)–C(14)	114.5(4)	Ni–O(1)–C(2)	123.61(15)
Ni–O(2)–C(4)	122.84(15)	Ni–O(3)–C(9)	121.99(16)
Ni–O(4)–C(7)	121.88(15)	Ni–O(5)–C(12)	123.61(17)
Ni–O(6)–C(14)	123.58(17)	C(1)–C(2)–O(1)	114.91(19)
C(1)–C(2)–C(3)	116.0(2)	O(1)–C(2)–C(3)	129.1(2)
C(2)–C(3)–H(3)	118.9	C(2)–C(3)–C(4)	122.3(2)
H(3)–C(3)–C(4)	118.9	O(2)–C(4)–C(3)	129.5(2)
O(2)–C(4)–C(5)	113.1(2)	C(3)–C(4)–C(5)	117.5(2)
F(4)–C(5)–F(5)	106.7(3)	F(4)–C(5)–F(6)	106.8(3)
F(4)–C(5)–C(4)	110.6(2)	F(5)–C(5)–F(6)	107.6(3)
F(5)–C(5)–C(4)	110.7(3)	F(6)–C(5)–C(4)	114.1(2)
F(10)–C(6)–F(11)	106.27(19)	F(10)–C(6)–F(12)	107.4(2)
F(10)–C(6)–C(7)	112.16(19)	F(11)–C(6)–F(12)	107.2(2)
F(11)–C(6)–C(7)	110.35(19)	F(12)–C(6)–C(7)	113.1(2)
O(4)–C(7)–C(6)	114.2(2)	O(4)–C(7)–C(8)	129.1(2)
C(6)–C(7)–C(8)	116.6(2)	C(7)–C(8)–H(8)	118.6
C(7)–C(8)–C(9)	122.8(2)	H(8)–C(8)–C(9)	118.6
O(3)–C(9)–C(8)	129.5(2)	O(3)–C(9)–C(10)	115.7(2)
C(8)–C(9)–C(10)	114.8(2)	F(7)–C(10)–F(8)	107.9(2)
F(7)–C(10)–F(9)	107.2(2)	F(7)–C(10)–C(9)	112.4(2)
F(8)–C(10)–F(9)	105.5(3)	F(8)–C(10)–C(9)	111.7(2)
F(9)–C(10)–C(9)	111.8(2)	C(11)–C(12)–O(5)	113.4(2)
C(11)–C(12)–C(13)	118.0(2)	O(5)–C(12)–C(13)	128.6(3)
C(12)–C(13)–H(13)	118.3	C(12)–C(13)–C(14)	123.5(3)
H(13)–C(13)–C(14)	118.3	C(15)–C(14)–O(6)	114.2(2)
C(15)–C(14)–C(13)	116.8(2)	O(6)–C(14)–C(13)	129.0(3)
C(16)–N–C(18)	106.1(2)	C(16)–N–C(20)	111.3(2)
C(16)–N–C(22)	111.5(2)	C(18)–N–C(20)	111.2(2)
C(18)–N–C(22)	111.3(2)	C(20)–N–C(22)	105.5(2)
N–C(16)–H(16A)	108.4	N–C(16)–H(16B)	108.4
N–C(16)–C(17)	115.4(2)	H(16A)–C(16)–H(16B)	107.5
H(16A)–C(16)–C(17)	108.4	H(16B)–C(16)–C(17)	108.4
C(16)–C(17)–H(17A)	109.5	C(16)–C(17)–H(17B)	109.5
C(16)–C(17)–H(17C)	109.5	H(17A)–C(17)–H(17B)	109.5
H(17A)–C(17)–H(17C)	109.5	H(17B)–C(17)–H(17C)	109.5
N–C(18)–H(18A)	108.3	N–C(18)–H(18B)	108.3
N–C(18)–C(19)	116.0(2)	H(18A)–C(18)–H(18B)	107.4

H(18A)–C(18)–C(19)	108.3	H(18B)–C(18)–C(19)	108.3
C(18)–C(19)–H(19A)	109.5	C(18)–C(19)–H(19B)	109.5
C(18)–C(19)–H(19C)	109.5	H(19A)–C(19)–H(19B)	109.5
H(19A)–C(19)–H(19C)	109.5	H(19B)–C(19)–H(19C)	109.5
N–C(20)–H(20A)	108.5	N–C(20)–H(20B)	108.5
N–C(20)–C(21)	115.0(2)	H(20A)–C(20)–H(20B)	107.5
H(20A)–C(20)–C(21)	108.5	H(20B)–C(20)–C(21)	108.5
C(20)–C(21)–H(21A)	109.5	C(20)–C(21)–H(21B)	109.5
C(20)–C(21)–H(21C)	109.5	H(21A)–C(21)–H(21B)	109.5
H(21A)–C(21)–H(21C)	109.5	H(21B)–C(21)–H(21C)	109.5
N–C(22)–H(22A)	108.4	N–C(22)–H(22B)	108.4
N–C(22)–C(23)	115.4(2)	H(22A)–C(22)–H(22B)	107.5
H(22A)–C(22)–C(23)	108.4	H(22B)–C(22)–C(23)	108.4
C(22)–C(23)–H(23A)	109.5	C(22)–C(23)–H(23B)	109.5
C(22)–C(23)–H(23C)	109.5	H(23A)–C(23)–H(23B)	109.5
H(23A)–C(23)–H(23C)	109.5	H(23B)–C(23)–H(23C)	109.5

Table 4. Anisotropic displacement parameters ($Å^2$) for $Ni(hfacac)_3NEt_4$. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ...+ 2hka^*b^*U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Ni	0.02764(16)	0.01527(15)	0.01944(15)	–0.00347(11)	0.00419(11)	–0.00218(12)
C(1)	0.0202(11)	0.0176(11)	0.0287(12)	–0.0051(9)	0.0021(9)	–0.0053(9)
F(1)	0.0635(12)	0.0442(10)	0.0238(8)	–0.0073(7)	0.0071(8)	–0.0317(9)
F(2)	0.0245(8)	0.0557(12)	0.0760(14)	–0.0448(11)	0.0002(8)	–0.0089(8)
F(3)	0.0642(12)	0.0162(7)	0.0344(9)	–0.0093(6)	–0.0004(8)	–0.0044(7)
F(4)	0.0366(10)	0.0129(8)	0.155(2)	–0.0039(10)	–0.0389(12)	–0.0064(7)
F(5)	0.0410(11)	0.0814(17)	0.111(2)	–0.0767(16)	0.0041(12)	0.0112(11)
F(6)	0.0169(7)	0.0259(8)	0.0954(15)	–0.0260(9)	–0.0079(8)	–0.0010(6)
F(7)	0.0595(12)	0.0419(10)	0.0497(11)	0.0209(8)	–0.0099(9)	–0.0312(9)
F(8)	0.0942(17)	0.0554(12)	0.0621(13)	–0.0341(10)	0.0548(12)	–0.0525(12)
F(9)	0.0444(10)	0.0387(9)	0.0399(9)	0.0023(7)	0.0004(8)	–0.0223(8)
F(10)	0.0344(8)	0.0281(8)	0.0324(8)	0.0090(6)	0.0064(7)	–0.0062(7)
F(11)	0.0496(10)	0.0211(8)	0.0319(8)	–0.0074(6)	0.0008(7)	0.0059(7)
F(12)	0.0390(10)	0.0473(11)	0.0570(12)	0.0205(9)	–0.0238(8)	–0.0270(9)
C(11)	0.0396(17)	0.097(3)	0.0421(17)	–0.0401(18)	0.0202(14)	–0.0433(19)
F(13)	0.082(5)	0.118(8)	0.046(2)	–0.034(3)	0.032(2)	–0.078(5)
F(14)	0.027(2)	0.139(5)	0.111(5)	–0.071(4)	0.025(2)	–0.039(3)
F(15)	0.066(6)	0.120(5)	0.062(6)	–0.050(4)	0.030(4)	–0.074(4)
C(11')	0.0396(17)	0.097(3)	0.0421(17)	–0.0401(18)	0.0202(14)	–0.0433(19)
F(13')	0.072(9)	0.102(11)	0.041(5)	–0.048(6)	0.041(5)	–0.069(8)
F(14')	0.022(4)	0.089(8)	0.089(9)	–0.042(6)	0.003(5)	–0.028(4)
F(15')	0.037(6)	0.060(7)	0.045(8)	–0.021(5)	0.006(5)	–0.031(5)
C(15)	0.073(3)	0.109(3)	0.0210(14)	0.0098(18)	–0.0123(15)	–0.073(3)
F(16)	0.036(4)	0.058(4)	0.016(2)	–0.002(2)	–0.0073(18)	–0.022(2)
F(17)	0.058(3)	0.157(8)	0.0310(19)	–0.008(3)	–0.0009(17)	–0.075(5)
F(18)	0.164(9)	0.041(3)	0.075(5)	0.015(3)	–0.080(5)	–0.054(4)
C(15')	0.073(3)	0.109(3)	0.0210(14)	0.0098(18)	–0.0123(15)	–0.073(3)
F(16')	0.043(7)	0.046(5)	0.053(6)	–0.007(3)	–0.025(4)	–0.009(4)
F(17')	0.060(4)	0.066(4)	0.028(2)	–0.015(2)	0.010(2)	–0.051(4)
F(18')	0.061(3)	0.034(3)	0.034(3)	–0.005(2)	–0.003(2)	–0.028(2)
O(1)	0.0317(9)	0.0174(8)	0.0207(8)	–0.0045(6)	0.0025(7)	0.0012(7)
O(2)	0.0312(9)	0.0157(8)	0.0255(9)	–0.0060(7)	0.0018(7)	–0.0026(7)
O(3)	0.0378(10)	0.0178(8)	0.0240(9)	–0.0008(7)	0.0108(7)	–0.0053(7)
O(4)	0.0237(8)	0.0172(8)	0.0224(8)	–0.0012(6)	0.0035(6)	–0.0046(6)
O(5)	0.0254(8)	0.0214(8)	0.0225(8)	–0.0071(7)	0.0014(7)	–0.0048(7)
O(6)	0.0270(9)	0.0217(8)	0.0212(8)	–0.0026(7)	0.0040(7)	–0.0070(7)
C(2)	0.0199(10)	0.0144(10)	0.0242(11)	–0.0035(8)	0.0070(9)	–0.0072(8)
C(3)	0.0192(11)	0.0186(11)	0.0434(15)	–0.0120(10)	–0.0021(10)	–0.0037(9)
C(4)	0.0172(10)	0.0150(10)	0.0347(13)	–0.0070(9)	0.0067(9)	–0.0062(8)

C(5)	0.0211(12)	0.0233(14)	0.084(2)	−0.0277(15)	−0.0044(14)	−0.0013(11)
C(6)	0.0204(11)	0.0211(11)	0.0221(11)	−0.0013(9)	0.0014(9)	−0.0084(9)
C(7)	0.0279(12)	0.0147(10)	0.0167(10)	−0.0052(8)	0.0029(9)	−0.0051(9)
C(8)	0.0280(12)	0.0198(11)	0.0254(12)	−0.0033(9)	0.0056(9)	−0.0097(10)
C(9)	0.0379(14)	0.0159(11)	0.0223(11)	−0.0070(9)	0.0073(10)	−0.0098(10)
C(10)	0.0525(17)	0.0244(13)	0.0268(13)	−0.0050(10)	0.0113(12)	−0.0210(12)
C(12)	0.0228(12)	0.0467(16)	0.0253(12)	−0.0120(11)	0.0056(10)	−0.0146(11)
C(13)	0.061(2)	0.123(4)	0.0204(14)	−0.0226(17)	0.0131(14)	−0.068(2)
C(14)	0.0291(13)	0.0508(17)	0.0200(12)	−0.0014(11)	−0.0033(10)	−0.0196(12)
N	0.0284(11)	0.0171(10)	0.0470(14)	−0.0002(9)	−0.0127(10)	−0.0103(9)
C(16)	0.0461(17)	0.0286(14)	0.0509(18)	0.0093(13)	−0.0190(14)	−0.0249(13)
C(17)	0.057(2)	0.0433(18)	0.052(2)	0.0125(15)	−0.0154(16)	−0.0293(16)
C(18)	0.0341(14)	0.0243(13)	0.0532(18)	−0.0083(12)	−0.0108(13)	−0.0110(11)
C(19)	0.0346(15)	0.0258(14)	0.060(2)	−0.0076(13)	−0.0031(14)	−0.0063(12)
C(20)	0.0306(14)	0.0295(15)	0.064(2)	−0.0088(14)	−0.0181(14)	−0.0105(12)
C(21)	0.046(2)	0.0407(19)	0.086(3)	−0.0046(19)	−0.033(2)	0.0016(16)
C(22)	0.0318(14)	0.0175(12)	0.0481(17)	0.0018(11)	0.0010(12)	−0.0067(10)
C(23)	0.0256(14)	0.0397(17)	0.0534(19)	0.0141(14)	0.0014(13)	−0.0012(12)

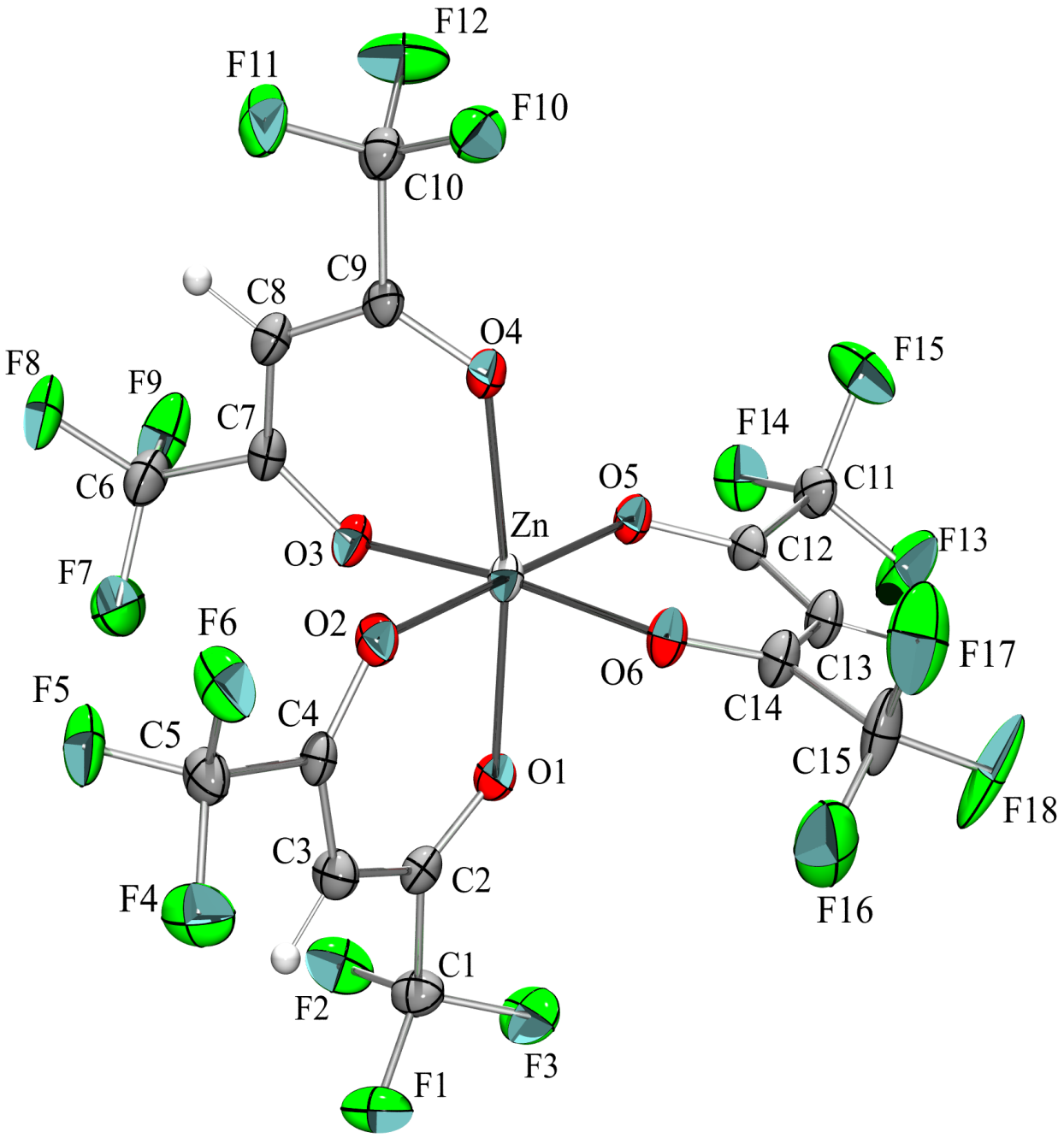
Table 5. Hydrogen coordinates and isotropic displacement parameters ($Å^2$) for $Ni(hfacac)_3NEt_4$.

	x	y	z	U
H(3)	0.5884	0.3032	0.5315	0.033
H(8)	1.3813	0.0218	0.7326	0.029
H(13)	0.8113	0.1466	0.9813	0.069
H(16A)	0.7432	0.6013	0.7372	0.048
H(16B)	0.8895	0.4930	0.7749	0.048
H(17A)	0.7459	0.5775	0.8866	0.073
H(17B)	0.7469	0.7201	0.8530	0.073
H(17C)	0.8917	0.6092	0.8912	0.073
H(18A)	0.9810	0.5072	0.6417	0.044
H(18B)	0.8433	0.6238	0.6012	0.044
H(19A)	1.0340	0.6127	0.5142	0.062
H(19B)	1.1294	0.6244	0.5938	0.062
H(19C)	0.9927	0.7456	0.5565	0.062
H(20A)	1.0433	0.6754	0.8160	0.049
H(20B)	1.1102	0.6911	0.7245	0.049
H(21A)	1.2522	0.4975	0.8033	0.093
H(21B)	1.1796	0.4631	0.7219	0.093
H(21C)	1.1173	0.4492	0.8153	0.093
H(22A)	0.8381	0.8374	0.7598	0.040
H(22B)	0.8959	0.8459	0.6651	0.040
H(23A)	0.6474	0.9422	0.6601	0.065
H(23B)	0.6312	0.8094	0.7038	0.065
H(23C)	0.6892	0.8177	0.6088	0.065

Table 6. Torsion angles [°] for $Ni(hfacac)_3NEt_4$.

O(2)–Ni–O(1)–C(2)	−9.22(19)	O(3)–Ni–O(1)–C(2)	−100.37(19)
O(4)–Ni–O(1)–C(2)	168.22(19)	O(5)–Ni–O(1)–C(2)	78.89(19)
O(6)–Ni–O(1)–C(2)	−109(2)	O(1)–Ni–O(2)–C(4)	15.07(19)
O(3)–Ni–O(2)–C(4)	105.89(19)	O(4)–Ni–O(2)–C(4)	−112.5(13)
O(5)–Ni–O(2)–C(4)	−75.53(19)	O(6)–Ni–O(2)–C(4)	−166.57(19)
O(1)–Ni–O(3)–C(9)	−107.20(19)	O(2)–Ni–O(3)–C(9)	162.01(19)
O(4)–Ni–O(3)–C(9)	−15.99(19)	O(5)–Ni–O(3)–C(9)	100(3)
O(6)–Ni–O(3)–C(9)	72.55(19)	O(1)–Ni–O(4)–C(7)	109.29(17)
O(2)–Ni–O(4)–C(7)	−123.1(13)	O(3)–Ni–O(4)–C(7)	18.45(18)
O(5)–Ni–O(4)–C(7)	−160.10(17)	O(6)–Ni–O(4)–C(7)	−69.05(17)
O(1)–Ni–O(5)–C(12)	177.1(2)	O(2)–Ni–O(5)–C(12)	−92.1(2)
O(3)–Ni–O(5)–C(12)	−30(3)	O(4)–Ni–O(5)–C(12)	86.0(2)
O(6)–Ni–O(5)–C(12)	−2.6(2)	O(1)–Ni–O(6)–C(14)	−165(2)
O(2)–Ni–O(6)–C(14)	95.5(2)	O(3)–Ni–O(6)–C(14)	−173.3(2)
O(4)–Ni–O(6)–C(14)	−81.9(2)	O(5)–Ni–O(6)–C(14)	7.4(2)
Ni–O(1)–C(2)–C(1)	178.27(14)	Ni–O(1)–C(2)–C(3)	1.0(4)
F(1)–C(1)–C(2)–O(1)	−91.8(2)	F(1)–C(1)–C(2)–C(3)	85.9(3)
F(2)–C(1)–C(2)–O(1)	148.6(2)	F(2)–C(1)–C(2)–C(3)	−33.7(3)
F(3)–C(1)–C(2)–O(1)	26.5(3)	F(3)–C(1)–C(2)–C(3)	−155.8(2)
C(1)–C(2)–C(3)–C(4)	−171.0(2)	O(1)–C(2)–C(3)–C(4)	6.3(4)
Ni–O(2)–C(4)–C(3)	−13.9(4)	Ni–O(2)–C(4)–C(5)	168.15(19)
C(2)–C(3)–C(4)–O(2)	1.3(4)	C(2)–C(3)–C(4)–C(5)	179.2(3)
O(2)–C(4)–C(5)–F(4)	76.6(3)	O(2)–C(4)–C(5)–F(5)	−41.4(3)
O(2)–C(4)–C(5)–F(6)	−163.0(3)	C(3)–C(4)–C(5)–F(4)	−101.6(3)
C(3)–C(4)–C(5)–F(5)	140.4(3)	C(3)–C(4)–C(5)–F(6)	18.8(4)
Ni–O(4)–C(7)–C(6)	171.16(14)	Ni–O(4)–C(7)–C(8)	−12.2(3)
F(10)–C(6)–C(7)–O(4)	−29.9(3)	F(10)–C(6)–C(7)–C(8)	153.0(2)
F(11)–C(6)–C(7)–O(4)	88.4(2)	F(11)–C(6)–C(7)–C(8)	−88.7(3)
F(12)–C(6)–C(7)–O(4)	−151.5(2)	F(12)–C(6)–C(7)–C(8)	31.4(3)
O(4)–C(7)–C(8)–C(9)	−3.9(4)	C(6)–C(7)–C(8)–C(9)	172.7(2)
Ni–O(3)–C(9)–C(8)	6.7(4)	Ni–O(3)–C(9)–C(10)	−174.42(15)
C(7)–C(8)–C(9)–O(3)	7.2(4)	C(7)–C(8)–C(9)–C(10)	−171.7(2)
O(3)–C(9)–C(10)–F(7)	3.0(3)	O(3)–C(9)–C(10)–F(8)	−118.4(3)
O(3)–C(9)–C(10)–F(9)	123.6(3)	C(8)–C(9)–C(10)–F(7)	−178.0(2)
C(8)–C(9)–C(10)–F(8)	60.6(3)	C(8)–C(9)–C(10)–F(9)	−57.4(3)
Ni–O(5)–C(12)–C(11)	175.7(2)	Ni–O(5)–C(12)–C(13)	−3.3(5)
F(13)–C(11)–C(12)–O(5)	157.9(11)	F(13)–C(11)–C(12)–C(13)	−23.0(12)
F(14)–C(11)–C(12)–O(5)	−80.8(9)	F(14)–C(11)–C(12)–C(13)	98.3(9)
F(15)–C(11)–C(12)–O(5)	37.6(12)	F(15)–C(11)–C(12)–C(13)	−143.3(12)
C(11)–C(12)–C(13)–C(14)	−172.4(4)	O(5)–C(12)–C(13)–C(14)	6.5(7)
Ni–O(6)–C(14)–C(15)	174.6(2)	Ni–O(6)–C(14)–C(13)	−7.1(5)
C(12)–C(13)–C(14)–C(15)	177.8(4)	C(12)–C(13)–C(14)–O(6)	−0.5(7)

F(16)–C(15)–C(14)–O(6)	−140.5(7)	F(16)–C(15)–C(14)–C(13)	40.9(8)
F(17)–C(15)–C(14)–O(6)	−36.7(5)	F(17)–C(15)–C(14)–C(13)	144.7(5)
F(18)–C(15)–C(14)–O(6)	88.0(7)	F(18)–C(15)–C(14)–C(13)	−90.5(7)
C(18)–N–C(16)–C(17)	−180.0(3)	C(20)–N–C(16)–C(17)	58.9(3)
C(22)–N–C(16)–C(17)	−58.6(3)	C(16)–N–C(18)–C(19)	−174.7(2)
C(20)–N–C(18)–C(19)	−53.4(3)	C(22)–N–C(18)–C(19)	63.8(3)
C(16)–N–C(20)–C(21)	61.0(4)	C(18)–N–C(20)–C(21)	−57.1(4)
C(22)–N–C(20)–C(21)	−177.9(3)	C(16)–N–C(22)–C(23)	−54.6(3)
C(18)–N–C(22)–C(23)	63.8(3)	C(20)–N–C(22)–C(23)	−175.5(3)



$Zn(hfacac)_3NEt_4$ (tetraethylammonium salt and hydrogens omitted for clarity)

Table 1. Crystal data and structure refinement for $Zn(hfacac)_3NEt_4$.

Identification code	jtn103	
Chemical formula (moiety)	$C_8H_{20}N^+ \cdot C_{15}H_3F_{18}O_6Zn^-$	
Chemical formula (total)	$C_{23}H_{23}F_{18}NO_6Zn$	
Formula weight	816.79	
Temperature	100(2) K	
Radiation, wavelength	MoKα, 0.71073 Å	
Crystal system, space group	triclinic, $P\bar{1}$	
Unit cell parameters	a = 10.3786(13) Å	α = 86.056(2)°
	b = 10.8425(14) Å	β = 80.156(2)°
	c = 15.1786(19) Å	γ = 67.947(2)°
Cell volume	1559.8(3) $Å^3$	
Z	2	
Calculated density	1.739 g/cm^3	
Absorption coefficient μ	0.934 mm^{-1}	
F(000)	816	
Crystal colour and size	colourless, 1.12 × 0.17 × 0.12 mm^3	
Reflections for cell refinement	9995 (θ range 2.3 to 31.8°)	
Data collection method	Bruker Kappa APEXII DUO CCD diffractometer ϕ and ω scans	
θ range for data collection	2.0 to 33.1°	
Index ranges	h −15 to 15, k −11 to 16, l −23 to 23	
Completeness to θ = 29.0°	99.4 %	
Reflections collected	33132	
Independent reflections	11353 (R_{int} = 0.0319)	
Reflections with $F^2>2\sigma$	8896	
Absorption correction	numerical	
Min. and max. transmission	0.4201 and 0.8962	
Structure solution	Patterson synthesis	
Refinement method	Full-matrix least-squares on F^2	
Weighting parameters a, b	0.0513, 0.3996	
Data / restraints / parameters	11353 / 18 / 483	
Final R indices [$F^2>2\sigma$]	R1 = 0.0390, wR2 = 0.0957	
R indices (all data)	R1 = 0.0549, wR2 = 0.1042	
Goodness-of-fit on F^2	1.030	
Largest and mean shift/su	0.001 and 0.000	
Largest diff. peak and hole	0.63 and −0.74 e $Å^{-3}$	

Table 2. Atomic coordinates and equivalent isotropic displacement parameters ($Å^2$) for $Zn(hfacac)_3NEt_4$. U_{eq} is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
Zn	0.623216(16)	0.780877(16)	0.269972(11)	0.01899(5)
F(1)	0.80611(13)	0.57505(11)	–0.06150(6)	0.0378(2)
F(2)	0.80551(13)	0.43593(10)	0.04682(7)	0.0386(2)
F(3)	0.60966(12)	0.57243(13)	0.01179(8)	0.0443(3)
F(4)	1.02291(12)	0.84717(13)	0.01850(7)	0.0416(3)
F(5)	1.10919(10)	0.73108(11)	0.12935(8)	0.0404(3)
F(6)	0.98121(11)	0.93818(11)	0.14692(8)	0.0377(3)
F(7)	1.01696(14)	0.41522(13)	0.27472(9)	0.0558(4)
F(8)	1.08441(10)	0.46663(10)	0.38880(8)	0.0362(2)
F(9)	0.93416(13)	0.37045(11)	0.40671(10)	0.0474(3)
F(10)	0.65318(11)	1.02880(10)	0.49949(7)	0.0336(2)
F(11)	0.86332(11)	0.89765(12)	0.51181(9)	0.0476(3)
F(12)	0.68714(16)	0.85740(12)	0.58432(7)	0.0492(3)
C(11)	0.21518(16)	0.75040(17)	0.37210(11)	0.0258(3)
F(13)	0.11108(12)	0.76043(14)	0.32856(8)	0.0506(3)
F(14)	0.26931(11)	0.62460(11)	0.39916(8)	0.0360(2)
F(15)	0.15938(13)	0.82358(12)	0.44608(8)	0.0445(3)
C(15)	0.2910(5)	1.0920(6)	0.1537(4)	0.0361(11)
F(16)	0.3733(2)	1.0896(4)	0.0786(2)	0.0656(13)
F(17)	0.2727(3)	1.2016(3)	0.1965(3)	0.0627(9)
F(18)	0.1677(4)	1.0992(4)	0.1378(3)	0.0660(13)
C(15')	0.2978(17)	1.0664(16)	0.1425(11)	0.039(4)
F(16')	0.334(2)	1.0245(10)	0.0600(5)	0.150(7)
F(17')	0.3265(13)	1.1773(11)	0.1413(11)	0.095(6)
F(18')	0.1592(11)	1.1230(15)	0.1542(11)	0.092(5)
O(1)	0.64118(11)	0.67852(11)	0.15420(7)	0.0220(2)
O(2)	0.79222(11)	0.83348(11)	0.20671(7)	0.0229(2)
O(3)	0.76213(11)	0.60136(11)	0.31159(8)	0.0246(2)
O(4)	0.63764(11)	0.86750(11)	0.38452(7)	0.0235(2)
O(5)	0.44953(11)	0.73916(11)	0.33258(7)	0.0220(2)
O(6)	0.48591(11)	0.95000(11)	0.21717(8)	0.0236(2)
C(1)	0.73936(18)	0.56180(17)	0.01964(11)	0.0276(3)
C(2)	0.73757(15)	0.65945(14)	0.08884(10)	0.0200(3)
C(3)	0.84618(16)	0.70921(16)	0.07224(10)	0.0236(3)
C(4)	0.86477(14)	0.78976(14)	0.13280(10)	0.0199(3)
C(5)	0.99514(16)	0.82775(16)	0.10627(11)	0.0256(3)
C(6)	0.97367(18)	0.46164(16)	0.35725(12)	0.0297(3)
C(7)	0.84954(15)	0.59601(15)	0.36104(10)	0.0217(3)
C(8)	0.84536(15)	0.69146(15)	0.41907(10)	0.0234(3)
C(9)	0.73604(15)	0.81662(15)	0.42891(10)	0.0207(3)
C(10)	0.73540(16)	0.90165(16)	0.50624(11)	0.0249(3)

C(12)	0.32695(14)	0.79698(15)	0.31552(10)	0.0205(3)
C(13)	0.27699(15)	0.90116(16)	0.25569(11)	0.0248(3)
C(14)	0.36083(16)	0.96853(15)	0.21195(11)	0.0238(3)
N	0.62180(13)	0.28169(12)	0.26632(8)	0.0220(2)
C(16)	0.60888(18)	0.17420(16)	0.21366(12)	0.0293(3)
C(17)	0.6633(2)	0.1705(2)	0.11503(14)	0.0447(5)
C(18)	0.77509(18)	0.25878(18)	0.26747(13)	0.0336(4)
C(19)	0.85456(19)	0.1352(2)	0.31765(14)	0.0381(4)
C(20)	0.54494(19)	0.27577(19)	0.35962(11)	0.0342(4)
C(21)	0.5565(3)	0.3678(2)	0.42650(14)	0.0531(6)
C(22)	0.55755(19)	0.41850(16)	0.22491(12)	0.0312(3)
C(23)	0.4066(2)	0.45843(19)	0.21065(13)	0.0383(4)

Table 3. Bond lengths [Å] and angles [°] for $Zn(hfacac)_3NEt_4$.

Zn–O(1)	2.0869(11)	Zn–O(2)	2.1033(10)
Zn–O(3)	2.0739(11)	Zn–O(4)	2.0848(11)
Zn–O(5)	2.0796(10)	Zn–O(6)	2.0644(11)
F(1)–C(1)	1.3327(19)	F(2)–C(1)	1.349(2)
F(3)–C(1)	1.3327(19)	F(4)–C(5)	1.3334(19)
F(5)–C(5)	1.3350(19)	F(6)–C(5)	1.3327(18)
F(7)–C(6)	1.323(2)	F(8)–C(6)	1.3385(18)
F(9)–C(6)	1.344(2)	F(10)–C(10)	1.3290(19)
F(11)–C(10)	1.3291(17)	F(12)–C(10)	1.3361(19)
C(11)–F(13)	1.3273(18)	C(11)–F(14)	1.3309(19)
C(11)–F(15)	1.335(2)	C(11)–C(12)	1.542(2)
C(15)–F(16)	1.298(6)	C(15)–F(17)	1.329(7)
C(15)–F(18)	1.315(5)	C(15)–C(14)	1.558(6)
C(15')–F(16')	1.306(14)	C(15')–F(17')	1.342(14)
C(15')–F(18')	1.320(14)	C(15')–C(14)	1.492(19)
O(1)–C(2)	1.2495(18)	O(2)–C(4)	1.2515(18)
O(3)–C(7)	1.2557(17)	O(4)–C(9)	1.2500(17)
O(5)–C(12)	1.2530(17)	O(6)–C(14)	1.2526(17)
C(1)–C(2)	1.535(2)	C(2)–C(3)	1.4022(19)
C(3)–H(3)	0.950	C(3)–C(4)	1.394(2)
C(4)–C(5)	1.540(2)	C(6)–C(7)	1.537(2)
C(7)–C(8)	1.388(2)	C(8)–H(8)	0.950
C(8)–C(9)	1.402(2)	C(9)–C(10)	1.539(2)
C(12)–C(13)	1.396(2)	C(13)–H(13)	0.950
C(13)–C(14)	1.394(2)	N–C(16)	1.5156(19)
N–C(18)	1.518(2)	N–C(20)	1.512(2)
N–C(22)	1.521(2)	C(16)–H(16A)	0.990
C(16)–H(16B)	0.990	C(16)–C(17)	1.505(3)
C(17)–H(17A)	0.980	C(17)–H(17B)	0.980
C(17)–H(17C)	0.980	C(18)–H(18A)	0.990
C(18)–H(18B)	0.990	C(18)–C(19)	1.517(3)
C(19)–H(19A)	0.980	C(19)–H(19B)	0.980
C(19)–H(19C)	0.980	C(20)–H(20A)	0.990
C(20)–H(20B)	0.990	C(20)–C(21)	1.521(3)
C(21)–H(21A)	0.980	C(21)–H(21B)	0.980
C(21)–H(21C)	0.980	C(22)–H(22A)	0.990
C(22)–H(22B)	0.990	C(22)–C(23)	1.510(3)
C(23)–H(23A)	0.980	C(23)–H(23B)	0.980
C(23)–H(23C)	0.980		
O(1)–Zn–O(2)	88.21(4)	O(1)–Zn–O(3)	85.29(4)
O(1)–Zn–O(4)	171.41(4)	O(1)–Zn–O(5)	93.05(4)
O(1)–Zn–O(6)	89.04(4)	O(2)–Zn–O(3)	90.72(4)
O(2)–Zn–O(4)	86.31(4)	O(2)–Zn–O(5)	177.06(4)
O(2)–Zn–O(6)	89.22(4)	O(3)–Zn–O(4)	88.17(4)

O(3)–Zn–O(5)	92.03(4)	O(3)–Zn–O(6)	174.33(4)
O(4)–Zn–O(5)	92.74(4)	O(4)–Zn–O(6)	97.49(4)
O(5)–Zn–O(6)	88.15(4)	F(13)–C(11)–F(14)	107.29(13)
F(13)–C(11)–F(15)	107.93(14)	F(13)–C(11)–C(12)	112.70(13)
F(14)–C(11)–F(15)	106.29(13)	F(14)–C(11)–C(12)	112.45(13)
F(15)–C(11)–C(12)	109.88(12)	F(16)–C(15)–F(17)	107.3(4)
F(16)–C(15)–F(18)	109.7(5)	F(16)–C(15)–C(14)	110.2(4)
F(17)–C(15)–F(18)	108.3(5)	F(17)–C(15)–C(14)	108.9(4)
F(18)–C(15)–C(14)	112.3(4)	F(16')–C(15')–F(17')	105.3(14)
F(16')–C(15')–F(18')	105.0(15)	F(16')–C(15')–C(14)	116.5(11)
F(17')–C(15')–F(18')	98.0(13)	F(17')–C(15')–C(14)	113.0(11)
F(18')–C(15')–C(14)	116.9(13)	Zn–O(1)–C(2)	125.03(9)
Zn–O(2)–C(4)	124.84(9)	Zn–O(3)–C(7)	122.02(10)
Zn–O(4)–C(9)	123.38(10)	Zn–O(5)–C(12)	124.54(9)
Zn–O(6)–C(14)	124.28(9)	F(1)–C(1)–F(2)	106.90(14)
F(1)–C(1)–F(3)	108.15(13)	F(1)–C(1)–C(2)	113.96(12)
F(2)–C(1)–F(3)	106.34(13)	F(2)–C(1)–C(2)	109.42(13)
F(3)–C(1)–C(2)	111.68(14)	O(1)–C(2)–C(1)	114.16(12)
O(1)–C(2)–C(3)	129.47(14)	C(1)–C(2)–C(3)	116.31(13)
C(2)–C(3)–H(3)	118.7	C(2)–C(3)–C(4)	122.62(14)
H(3)–C(3)–C(4)	118.7	O(2)–C(4)–C(3)	129.24(13)
O(2)–C(4)–C(5)	115.21(12)	C(3)–C(4)–C(5)	115.53(13)
F(4)–C(5)–F(5)	106.89(13)	F(4)–C(5)–F(6)	107.38(13)
F(4)–C(5)–C(4)	112.72(12)	F(5)–C(5)–F(6)	107.01(13)
F(5)–C(5)–C(4)	110.43(13)	F(6)–C(5)–C(4)	112.09(13)
F(7)–C(6)–F(8)	108.08(15)	F(7)–C(6)–F(9)	106.73(15)
F(7)–C(6)–C(7)	111.41(14)	F(8)–C(6)–F(9)	106.67(13)
F(8)–C(6)–C(7)	113.42(13)	F(9)–C(6)–C(7)	110.20(14)
O(3)–C(7)–C(6)	113.43(13)	O(3)–C(7)–C(8)	128.95(14)
C(6)–C(7)–C(8)	117.58(13)	C(7)–C(8)–H(8)	118.8
C(7)–C(8)–C(9)	122.41(13)	H(8)–C(8)–C(9)	118.8
O(4)–C(9)–C(8)	128.83(14)	O(4)–C(9)–C(10)	115.65(13)
C(8)–C(9)–C(10)	115.50(13)	F(10)–C(10)–F(11)	107.37(13)
F(10)–C(10)–F(12)	106.67(13)	F(10)–C(10)–C(9)	112.76(13)
F(11)–C(10)–F(12)	107.01(14)	F(11)–C(10)–C(9)	112.36(13)
F(12)–C(10)–C(9)	110.33(12)	C(11)–C(12)–O(5)	114.96(13)
C(11)–C(12)–C(13)	115.90(13)	O(5)–C(12)–C(13)	129.07(13)
C(12)–C(13)–H(13)	119.0	C(12)–C(13)–C(14)	121.94(13)
H(13)–C(13)–C(14)	119.0	C(15)–C(14)–O(6)	112.9(2)
C(15)–C(14)–C(13)	117.9(2)	C(15')–C(14)–O(6)	115.4(6)
C(15')–C(14)–C(13)	115.3(6)	O(6)–C(14)–C(13)	128.96(14)
C(16)–N–C(18)	111.06(13)	C(16)–N–C(20)	105.99(12)
C(16)–N–C(22)	111.53(12)	C(18)–N–C(20)	111.30(13)
C(18)–N–C(22)	106.41(12)	C(20)–N–C(22)	110.63(13)
N–C(16)–H(16A)	108.3	N–C(16)–H(16B)	108.3
N–C(16)–C(17)	116.08(14)	H(16A)–C(16)–H(16B)	107.4

H(16A)–C(16)–C(17)	108.3	H(16B)–C(16)–C(17)	108.3
C(16)–C(17)–H(17A)	109.5	C(16)–C(17)–H(17B)	109.5
C(16)–C(17)–H(17C)	109.5	H(17A)–C(17)–H(17B)	109.5
H(17A)–C(17)–H(17C)	109.5	H(17B)–C(17)–H(17C)	109.5
N–C(18)–H(18A)	108.5	N–C(18)–H(18B)	108.5
N–C(18)–C(19)	115.08(14)	H(18A)–C(18)–H(18B)	107.5
H(18A)–C(18)–C(19)	108.5	H(18B)–C(18)–C(19)	108.5
C(18)–C(19)–H(19A)	109.5	C(18)–C(19)–H(19B)	109.5
C(18)–C(19)–H(19C)	109.5	H(19A)–C(19)–H(19B)	109.5
H(19A)–C(19)–H(19C)	109.5	H(19B)–C(19)–H(19C)	109.5
N–C(20)–H(20A)	108.6	N–C(20)–H(20B)	108.6
N–C(20)–C(21)	114.66(16)	H(20A)–C(20)–H(20B)	107.6
H(20A)–C(20)–C(21)	108.6	H(20B)–C(20)–C(21)	108.6
C(20)–C(21)–H(21A)	109.5	C(20)–C(21)–H(21B)	109.5
C(20)–C(21)–H(21C)	109.5	H(21A)–C(21)–H(21B)	109.5
H(21A)–C(21)–H(21C)	109.5	H(21B)–C(21)–H(21C)	109.5
N–C(22)–H(22A)	108.4	N–C(22)–H(22B)	108.4
N–C(22)–C(23)	115.64(14)	H(22A)–C(22)–H(22B)	107.4
H(22A)–C(22)–C(23)	108.4	H(22B)–C(22)–C(23)	108.4
C(22)–C(23)–H(23A)	109.5	C(22)–C(23)–H(23B)	109.5
C(22)–C(23)–H(23C)	109.5	H(23A)–C(23)–H(23B)	109.5
H(23A)–C(23)–H(23C)	109.5	H(23B)–C(23)–H(23C)	109.5

Table 4. Anisotropic displacement parameters ($Å^2$) for $Zn(hfacac)_3NEt_4$. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ...+ 2hka^*b^*U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Zn	0.01420(8)	0.02112(9)	0.02339(9)	−0.00023(6)	−0.00505(6)	−0.00760(6)
F(1)	0.0542(7)	0.0480(6)	0.0211(5)	−0.0033(4)	−0.0018(4)	−0.0314(6)
F(2)	0.0540(7)	0.0255(5)	0.0368(6)	−0.0054(4)	0.0012(5)	−0.0180(5)
F(3)	0.0381(6)	0.0683(8)	0.0406(6)	−0.0144(6)	−0.0105(5)	−0.0313(6)
F(4)	0.0461(7)	0.0602(7)	0.0323(5)	0.0010(5)	0.0022(5)	−0.0390(6)
F(5)	0.0176(4)	0.0427(6)	0.0612(7)	−0.0035(5)	−0.0096(5)	−0.0096(4)
F(6)	0.0305(5)	0.0377(6)	0.0522(7)	−0.0162(5)	0.0054(5)	−0.0231(5)
F(7)	0.0434(7)	0.0531(8)	0.0479(7)	−0.0184(6)	−0.0148(6)	0.0146(6)
F(8)	0.0211(5)	0.0288(5)	0.0603(7)	0.0019(5)	−0.0181(5)	−0.0064(4)
F(9)	0.0420(6)	0.0271(5)	0.0825(9)	0.0165(6)	−0.0328(6)	−0.0165(5)
F(10)	0.0365(5)	0.0269(5)	0.0354(5)	−0.0053(4)	−0.0108(4)	−0.0063(4)
F(11)	0.0241(5)	0.0518(7)	0.0716(8)	−0.0292(6)	−0.0144(5)	−0.0112(5)
F(12)	0.0857(10)	0.0495(7)	0.0227(5)	0.0007(5)	−0.0031(6)	−0.0391(7)
C(11)	0.0199(7)	0.0332(8)	0.0288(7)	0.0012(6)	−0.0043(6)	−0.0148(6)
F(13)	0.0366(6)	0.0859(10)	0.0514(7)	0.0270(7)	−0.0222(5)	−0.0458(7)
F(14)	0.0333(5)	0.0336(5)	0.0456(6)	0.0072(5)	−0.0025(5)	−0.0201(4)
F(15)	0.0426(6)	0.0462(7)	0.0427(6)	−0.0130(5)	0.0174(5)	−0.0222(5)
C(15)	0.0219(15)	0.028(2)	0.061(3)	0.0226(18)	−0.0188(15)	−0.0107(13)
F(16)	0.0457(12)	0.078(2)	0.0516(16)	0.0391(15)	−0.0054(10)	−0.0064(12)
F(17)	0.0520(14)	0.0269(10)	0.105(2)	0.0182(12)	−0.0276(15)	−0.0065(9)
F(18)	0.048(2)	0.073(2)	0.106(3)	0.062(2)	−0.061(2)	−0.0444(19)
C(15')	0.040(6)	0.024(6)	0.061(7)	0.005(4)	−0.016(5)	−0.020(5)
F(16')	0.290(15)	0.058(5)	0.038(3)	0.002(3)	−0.070(6)	0.027(7)
F(17')	0.091(9)	0.078(7)	0.173(14)	0.093(9)	−0.108(10)	−0.072(7)
F(18')	0.033(4)	0.090(7)	0.099(7)	0.048(5)	0.004(4)	0.024(4)
O(1)	0.0202(5)	0.0248(5)	0.0244(5)	−0.0015(4)	−0.0048(4)	−0.0114(4)
O(2)	0.0183(5)	0.0253(5)	0.0279(5)	−0.0035(4)	−0.0030(4)	−0.0109(4)
O(3)	0.0211(5)	0.0235(5)	0.0318(6)	−0.0003(4)	−0.0111(4)	−0.0080(4)
O(4)	0.0171(5)	0.0273(5)	0.0261(5)	−0.0029(4)	−0.0057(4)	−0.0069(4)
O(5)	0.0171(5)	0.0256(5)	0.0256(5)	0.0029(4)	−0.0061(4)	−0.0098(4)
O(6)	0.0177(5)	0.0233(5)	0.0330(6)	0.0038(4)	−0.0077(4)	−0.0102(4)
C(1)	0.0316(8)	0.0329(8)	0.0243(7)	−0.0021(6)	−0.0051(6)	−0.0182(7)
C(2)	0.0205(6)	0.0189(6)	0.0228(6)	0.0025(5)	−0.0088(5)	−0.0078(5)
C(3)	0.0224(7)	0.0269(7)	0.0245(7)	−0.0015(5)	−0.0024(5)	−0.0128(6)
C(4)	0.0151(6)	0.0197(6)	0.0262(7)	0.0018(5)	−0.0060(5)	−0.0073(5)
C(5)	0.0204(7)	0.0290(8)	0.0301(8)	−0.0045(6)	−0.0009(6)	−0.0128(6)
C(6)	0.0263(8)	0.0253(8)	0.0381(9)	−0.0003(6)	−0.0141(7)	−0.0063(6)
C(7)	0.0171(6)	0.0212(7)	0.0271(7)	0.0034(5)	−0.0061(5)	−0.0069(5)
C(8)	0.0202(6)	0.0249(7)	0.0276(7)	0.0015(5)	−0.0091(5)	−0.0092(5)

C(9)	0.0175(6)	0.0255(7)	0.0219(6)	0.0005(5)	−0.0033(5)	−0.0112(5)
C(10)	0.0223(7)	0.0276(7)	0.0274(7)	−0.0004(6)	−0.0063(6)	−0.0111(6)
C(12)	0.0166(6)	0.0253(7)	0.0233(6)	−0.0021(5)	−0.0034(5)	−0.0114(5)
C(13)	0.0167(6)	0.0267(7)	0.0340(8)	0.0037(6)	−0.0086(6)	−0.0102(5)
C(14)	0.0200(6)	0.0230(7)	0.0307(7)	0.0048(6)	−0.0091(6)	−0.0091(5)
N	0.0230(6)	0.0214(6)	0.0253(6)	0.0008(5)	−0.0056(5)	−0.0116(5)
C(16)	0.0327(8)	0.0243(7)	0.0367(9)	−0.0026(6)	−0.0112(7)	−0.0142(6)
C(17)	0.0472(12)	0.0422(11)	0.0361(10)	−0.0120(8)	−0.0091(9)	−0.0038(9)
C(18)	0.0252(8)	0.0357(9)	0.0467(10)	−0.0015(7)	−0.0082(7)	−0.0177(7)
C(19)	0.0271(8)	0.0395(10)	0.0440(10)	−0.0080(8)	−0.0109(7)	−0.0047(7)
C(20)	0.0311(8)	0.0398(10)	0.0256(8)	0.0071(7)	−0.0031(6)	−0.0082(7)
C(21)	0.0505(13)	0.0569(14)	0.0313(10)	−0.0122(9)	−0.0150(9)	0.0091(10)
C(22)	0.0391(9)	0.0219(7)	0.0357(9)	0.0054(6)	−0.0118(7)	−0.0133(7)
C(23)	0.0438(10)	0.0300(9)	0.0364(9)	−0.0014(7)	−0.0179(8)	−0.0031(8)

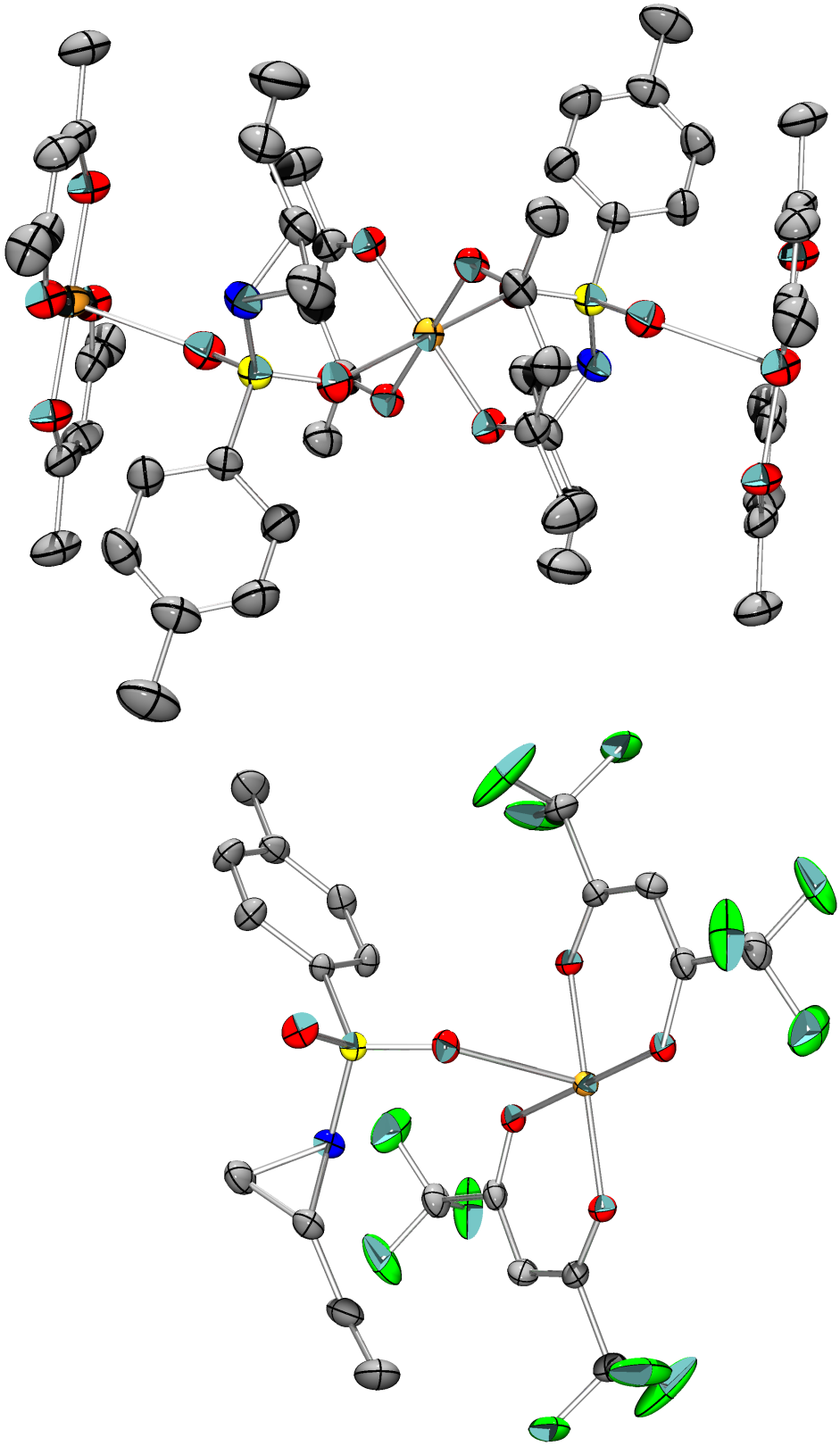
Table 5. Hydrogen coordinates and isotropic displacement parameters ($Å^2$) for $Zn(hfacac)_3NEt_4$.

	x	y	z	U
H(3)	0.9096	0.6871	0.0175	0.028
H(8)	0.9193	0.6712	0.4534	0.028
H(13)	0.1831	0.9269	0.2444	0.030
H(16A)	0.6600	0.0868	0.2398	0.035
H(16B)	0.5083	0.1858	0.2216	0.035
H(17A)	0.6097	0.2542	0.0872	0.067
H(17B)	0.6525	0.0962	0.0878	0.067
H(17C)	0.7630	0.1585	0.1058	0.067
H(18A)	0.8238	0.2527	0.2049	0.040
H(18B)	0.7793	0.3373	0.2947	0.040
H(19A)	0.8136	0.1441	0.3811	0.057
H(19B)	0.9537	0.1248	0.3111	0.057
H(19C)	0.8477	0.0570	0.2931	0.057
H(20A)	0.5824	0.1832	0.3821	0.041
H(20B)	0.4441	0.2985	0.3563	0.041
H(21A)	0.6557	0.3449	0.4316	0.080
H(21B)	0.5043	0.3578	0.4850	0.080
H(21C)	0.5169	0.4602	0.4060	0.080
H(22A)	0.5629	0.4855	0.2638	0.037
H(22B)	0.6157	0.4214	0.1664	0.037
H(23A)	0.3993	0.3928	0.1724	0.057
H(23B)	0.3761	0.5461	0.1818	0.057
H(23C)	0.3465	0.4623	0.2685	0.057

Table 6. Torsion angles [°] for $Zn(hfacac)_3NEt_4$.

O(2)–Zn–O(1)–C(2)	7.39(12)	O(3)–Zn–O(1)–C(2)	−83.48(12)
O(4)–Zn–O(1)–C(2)	−43.0(3)	O(5)–Zn–O(1)–C(2)	−175.27(12)
O(6)–Zn–O(1)–C(2)	96.64(12)	O(1)–Zn–O(2)–C(4)	−7.34(12)
O(3)–Zn–O(2)–C(4)	77.92(12)	O(4)–Zn–O(2)–C(4)	166.04(12)
O(5)–Zn–O(2)–C(4)	−122.6(8)	O(6)–Zn–O(2)–C(4)	−96.41(12)
O(1)–Zn–O(3)–C(7)	148.11(12)	O(2)–Zn–O(3)–C(7)	59.97(12)
O(4)–Zn–O(3)–C(7)	−26.31(12)	O(5)–Zn–O(3)–C(7)	−118.99(12)
O(6)–Zn–O(3)–C(7)	149.3(4)	O(1)–Zn–O(4)–C(9)	−21.2(3)
O(2)–Zn–O(4)–C(9)	−71.66(11)	O(3)–Zn–O(4)–C(9)	19.18(11)
O(5)–Zn–O(4)–C(9)	111.13(11)	O(6)–Zn–O(4)–C(9)	−160.38(11)
O(1)–Zn–O(5)–C(12)	−76.75(12)	O(2)–Zn–O(5)–C(12)	38.4(8)
O(3)–Zn–O(5)–C(12)	−162.14(12)	O(4)–Zn–O(5)–C(12)	109.60(12)
O(6)–Zn–O(5)–C(12)	12.19(12)	O(1)–Zn–O(6)–C(14)	74.60(13)
O(2)–Zn–O(6)–C(14)	162.81(13)	O(3)–Zn–O(6)–C(14)	73.4(4)
O(4)–Zn–O(6)–C(14)	−111.01(13)	O(5)–Zn–O(6)–C(14)	−18.48(13)
Zn–O(1)–C(2)–C(1)	170.27(10)	Zn–O(1)–C(2)–C(3)	−6.5(2)

F(1)–C(1)–C(2)–O(1)	158.09(14)	F(1)–C(1)–C(2)–C(3)	−24.7(2)
F(2)–C(1)–C(2)–O(1)	−82.31(16)	F(2)–C(1)–C(2)–C(3)	94.92(16)
F(3)–C(1)–C(2)–O(1)	35.16(19)	F(3)–C(1)–C(2)–C(3)	−147.61(14)
O(1)–C(2)–C(3)–C(4)	2.2(3)	C(1)–C(2)–C(3)–C(4)	−174.51(14)
Zn–O(2)–C(4)–C(3)	6.4(2)	Zn–O(2)–C(4)–C(5)	−171.96(10)
C(2)–C(3)–C(4)–O(2)	−2.2(3)	C(2)–C(3)–C(4)–C(5)	176.17(14)
O(2)–C(4)–C(5)–F(4)	−145.36(14)	O(2)–C(4)–C(5)–F(5)	95.16(16)
O(2)–C(4)–C(5)–F(6)	−24.05(19)	C(3)–C(4)–C(5)–F(4)	36.07(19)
C(3)–C(4)–C(5)–F(5)	−83.41(17)	C(3)–C(4)–C(5)–F(6)	157.37(14)
Zn–O(3)–C(7)–C(6)	−159.58(10)	Zn–O(3)–C(7)–C(8)	22.9(2)
F(7)–C(6)–C(7)–O(3)	40.22(19)	F(7)–C(6)–C(7)–C(8)	−141.97(15)
F(8)–C(6)–C(7)–O(3)	162.44(14)	F(8)–C(6)–C(7)–C(8)	−19.8(2)
F(9)–C(6)–C(7)–O(3)	−78.05(17)	F(9)–C(6)–C(7)–C(8)	99.76(16)
O(3)–C(7)–C(8)–C(9)	−1.6(3)	C(6)–C(7)–C(8)–C(9)	−179.02(14)
Zn–O(4)–C(9)–C(8)	−6.9(2)	Zn–O(4)–C(9)–C(10)	174.61(9)
C(7)–C(8)–C(9)–O(4)	−7.8(2)	C(7)–C(8)–C(9)–C(10)	170.77(13)
O(4)–C(9)–C(10)–F(10)	−16.79(18)	O(4)–C(9)–C(10)–F(11)	−138.30(14)
O(4)–C(9)–C(10)–F(12)	102.37(16)	C(8)–C(9)–C(10)–F(10)	164.48(13)
C(8)–C(9)–C(10)–F(11)	42.96(18)	C(8)–C(9)–C(10)–F(12)	−76.36(17)
Zn–O(5)–C(12)–C(11)	−178.90(9)	Zn–O(5)–C(12)–C(13)	−2.2(2)
F(13)–C(11)–C(12)–O(5)	−149.45(15)	F(13)–C(11)–C(12)–C(13)	33.4(2)
F(14)–C(11)–C(12)–O(5)	−28.02(19)	F(14)–C(11)–C(12)–C(13)	154.79(14)
F(15)–C(11)–C(12)–O(5)	90.15(16)	F(15)–C(11)–C(12)–C(13)	−87.03(17)
C(11)–C(12)–C(13)–C(14)	168.60(15)	O(5)–C(12)–C(13)–C(14)	−8.1(3)
Zn–O(6)–C(14)–C(15)	−169.3(3)	Zn–O(6)–C(14)–C(15')	−157.0(6)
Zn–O(6)–C(14)–C(13)	16.0(2)	C(12)–C(13)–C(14)–C(15)	−174.2(3)
C(12)–C(13)–C(14)–C(15')	173.2(6)	C(12)–C(13)–C(14)–O(6)	0.3(3)
F(16')–C(15')–C(14)–O(6)	77.7(14)	F(16')–C(15')–C(14)–C(13)	−96.2(14)
F(17')–C(15')–C(14)–O(6)	−44.4(14)	F(17')–C(15')–C(14)–C(13)	141.6(12)
F(18')–C(15')–C(14)–O(6)	−157.1(12)	F(18')–C(15')–C(14)–C(13)	28.9(15)
F(16)–C(15)–C(14)–O(6)	48.0(5)	F(16)–C(15)–C(14)–C(13)	−136.7(3)
F(17)–C(15)–C(14)–O(6)	−69.5(4)	F(17)–C(15)–C(14)–C(13)	105.9(3)
F(18)–C(15)–C(14)–O(6)	170.6(4)	F(18)–C(15)–C(14)–C(13)	−14.0(6)
C(18)–N–C(16)–C(17)	−65.76(19)	C(20)–N–C(16)–C(17)	173.23(15)
C(22)–N–C(16)–C(17)	52.76(19)	C(16)–N–C(18)–C(19)	−66.29(19)
C(20)–N–C(18)–C(19)	51.54(19)	C(22)–N–C(18)–C(19)	172.15(15)
C(16)–N–C(20)–C(21)	174.61(15)	C(18)–N–C(20)–C(21)	53.75(19)
C(22)–N–C(20)–C(21)	−64.34(18)	C(16)–N–C(22)–C(23)	54.38(19)
C(18)–N–C(22)–C(23)	175.64(15)	C(20)–N–C(22)–C(23)	−63.32(18)



Cu(hfacac)$_2$-Aziridine 1 Chain Representation and One Octahedral Unit

Table 1. Crystal data and structure refinement for **Cu(hfacac)$_2$-1**.

Identification code	jn201b	
Chemical formula	C21H15CuF12NO6S	
Formula weight	700.94	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal size	0.050 x 0.100 x 0.100 mm	
Crystal system	triclinic	
Space group	P -1	
Unit cell dimensions	a = 11.5841(3) Å	α = 63.8590(10)°
	b = 12.0774(4) Å	β = 71.4250(10)°
	c = 12.1591(3) Å	γ = 64.5010(10)°
Volume	1360.82(7) Å^3	
Z	2	
Density (calculated)	1.711 g/cm^3	
Absorption coefficient	0.999 mm^{-1}	
F(000)	698	
Theta range for data collection	1.89 to 25.29°	
Index ranges	-13<=h<=13, -14<=k<=14, -14<=l<=14	
Reflections collected	25131	
Independent reflections	4948 [R(int) = 0.0282]	
Coverage of independent reflections	99.9%	
Absorption correction	multi-scan	
Max. and min. transmission	0.9518 and 0.9067	
Structure solution technique	direct methods	
Structure solution program	SHELXS-97 (Sheldrick, 2008)	
Refinement method	Full-matrix least-squares on F^2	
Refinement program	SHELXL-97 (Sheldrick, 2008)	
Function minimized	$\Sigma\, w(F_o^2 - F_c^2)^2$	
Data / restraints / parameters	4948 / 0 / 467	
Goodness-of-fit on F^2	0.970	
Δ/σ_{max}	0.001	
Final R indices	4179 data; I>2σ(I)	R1 = 0.0290, wR2 = 0.0653
	all data	R1 = 0.0371, wR2 = 0.0696
Weighting scheme	$w=1/[\sigma^2(F_o^2)+(0.0224P)^2+1.2607P]$ where $P=(F_o^2+2F_c^2)/3$	
Absolute structure parameter	0.0(0)	

Largest diff. peak and hole	0.340 and -0.252 eÅ^{-3}
R.M.S. deviation from mean	0.044 eÅ^{-3}

Table 2. Atomic coordinates and equivalent isotropic displacement parameters ($Å^2$) for **Cu(hfacac)$_2$-1**. U_{eq} is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
Cu1	0.0	0.0	0.5	0.03182(10)
Cu2	0.0	0.5	0.0	0.03735(11)
S1	0.93134(5)	0.35263(5)	0.35550(5)	0.03438(13)
O2	0.87196(14)	0.02237(15)	0.41502(14)	0.0368(3)
O1	0.13232(14)	0.00704(15)	0.35332(14)	0.0371(4)
C1	0.1185(2)	0.0109(2)	0.2536(2)	0.0388(5)
C3	0.8944(2)	0.0264(2)	0.3057(2)	0.0371(5)
C2	0.0091(2)	0.0194(3)	0.2232(2)	0.0447(6)
C5	0.7748(2)	0.0457(3)	0.2620(2)	0.0508(6)
C4	0.2420(3)	0.0013(4)	0.1578(3)	0.0667(9)
F4	0.69690(16)	0.98955(19)	0.35145(16)	0.0753(5)
F5	0.70681(17)	0.17247(19)	0.21957(19)	0.0866(6)
F6	0.80319(17)	0.0031(2)	0.17081(19)	0.0974(7)
F3A	0.2226(12)	0.051(2)	0.0432(10)	0.121(4)
F1A	0.3155(10)	0.0517(16)	0.1617(7)	0.092(3)
F2A	0.3139(9)	0.8736(9)	0.1859(11)	0.120(3)
N1	0.05121(18)	0.38839(18)	0.36144(18)	0.0403(4)
C11	0.7149(2)	0.4512(3)	0.5022(2)	0.0484(6)
C10	0.7946(2)	0.4793(2)	0.3892(2)	0.0335(5)
C7	0.1718(2)	0.2715(2)	0.3965(2)	0.0439(6)
C15	0.7674(2)	0.6054(2)	0.3034(2)	0.0423(6)
C13	0.5766(2)	0.6774(3)	0.4443(3)	0.0510(6)
C12	0.6069(3)	0.5509(3)	0.5280(3)	0.0578(7)
C6	0.0880(3)	0.3373(3)	0.4847(3)	0.0568(7)
C14	0.6588(3)	0.7029(2)	0.3322(2)	0.0504(6)
C8	0.2942(3)	0.2905(3)	0.3260(3)	0.0609(7)
C16	0.4570(3)	0.7853(3)	0.4740(3)	0.0782(10)
C9	0.3908(3)	0.1998(4)	0.2912(4)	0.0931(12)
O3	0.01633(15)	0.61482(16)	0.06081(14)	0.0414(4)
C18	0.1061(3)	0.7088(3)	0.1290(3)	0.0548(7)
C17	0.1218(2)	0.6140(2)	0.0691(2)	0.0426(6)
O4	0.81485(15)	0.58862(17)	0.02555(15)	0.0429(4)

C19	0.7329(2)	0.5542(3)	0.0143(2)	0.0441(6)
C20	0.5927(3)	0.6277(4)	0.0609(3)	0.0630(8)
O6	0.92325(16)	0.23119(15)	0.45240(16)	0.0473(4)
O5	0.94705(16)	0.36216(16)	0.23052(15)	0.0453(4)
F7A	0.575(2)	0.721(4)	0.072(5)	0.136(11)
F9A	0.516(2)	0.656(3)	0.985(3)	0.109(8)
F8B	0.5650(6)	0.5765(13)	0.1817(6)	0.093(2)
F10A	0.1100(15)	0.8180(9)	0.0535(6)	0.100(4)
F11A	0.9913(8)	0.7328(11)	0.2048(11)	0.103(3)
F12A	0.1881(11)	0.6591(7)	0.2053(11)	0.106(4)
C21	0.2455(2)	0.5403(3)	0.0317(2)	0.0499(6)
F8A	0.562(3)	0.531(4)	0.170(3)	0.163(11)
F9B	0.5063(8)	0.6223(13)	0.0172(14)	0.099(3)
F7B	0.5717(9)	0.7515(9)	0.0397(9)	0.097(2)
F10B	0.0326(13)	0.8267(9)	0.0711(11)	0.096(4)
F12B	0.2195(6)	0.7266(14)	0.1115(17)	0.110(5)
F11B	0.064(2)	0.6754(12)	0.2374(6)	0.119(6)
F1B	0.262(2)	0.119(2)	0.129(2)	0.133(6)
F3B	0.229(2)	0.994(3)	0.0602(19)	0.100(7)
F2B	0.3394(16)	0.912(3)	0.1958(12)	0.142(10)

Table 3. Bond lengths [Å] and angles [°] for **Cu(hfacac)$_2$-1**.

Cu1-O2#1	1.9302(14)	Cu1-O2	1.9303(14)
Cu1-O1	1.9428(14)	Cu1-O1#1	1.9429(14)
Cu1-O6#1	2.3849(15)	Cu1-O6	2.3849(15)
Cu2-O3	1.9297(15)	Cu2-O3#2	1.9297(15)
Cu2-O4#2	1.9324(15)	Cu2-O4	1.9324(15)
S1-O5	1.4294(16)	S1-O6	1.4400(17)
S1-N1	1.6516(19)	S1-C10	1.753(2)
O2-C3	1.254(3)	O1-C1	1.252(3)
C1-C2	1.381(3)	C1-C4	1.527(3)
C3-C2	1.381(3)	C3-C5	1.528(3)
C5-F4	1.308(3)	C5-F6	1.314(3)
C5-F5	1.325(3)	N1-C6	1.475(3)
N1-C7	1.509(3)	C11-C12	1.379(4)
C11-C10	1.380(3)	C10-C15	1.380(3)
C7-C8	1.459(4)	C7-C6	1.466(4)
C15-C14	1.378(3)	C13-C12	1.374(4)
C13-C14	1.383(4)	C13-C16	1.513(3)
C8-C9	1.297(4)	O3-C17	1.252(3)
C18-F11B	1.195(8)	C18-F10A	1.242(7)
C18-F12A	1.304(5)	C18-F10B	1.305(9)
C18-F11A	1.351(7)	C18-F12B	1.356(6)
C18-C17	1.532(3)	C17-C21	1.377(3)
O4-C19	1.251(3)	C19-C21#2	1.379(4)
C19-C20	1.537(4)	C20-F7A	1.12(3)
C20-F8B	1.309(7)	C20-F9B	1.310(7)
C20-F9A	1.32(2)	C20-F7B	1.322(9)
C20-F8A	1.40(3)	C21-C19#2	1.379(4)
O2#1-Cu1-O2	180.0	O2#1-Cu1-O1	86.99(6)
O2-Cu1-O1	93.01(6)	O2#1-Cu1-O1#1	93.01(6)
O2-Cu1-O1#1	86.99(6)	O1-Cu1-O1#1	180.0
O2#1-Cu1-O6#1	89.86(6)	O2-Cu1-O6#1	90.13(6)
O1-Cu1-O6#1	86.58(6)	O1#1-Cu1-O6#1	93.42(6)
O2#1-Cu1-O6	90.14(6)	O2-Cu1-O6	89.87(6)
O1-Cu1-O6	93.42(6)	O1#1-Cu1-O6	86.58(6)
O6#1-Cu1-O6	180.0000(10)	O3-Cu2-O3#2	180.00(9)
O3-Cu2-O4#2	92.57(6)	O3#2-Cu2-O4#2	87.43(6)

O3-Cu2-O4	87.43(6)	O3#2-Cu2-O4	92.57(6)
O4#2-Cu2-O4	180.0000(10)	O5-S1-O6	117.19(10)
O5-S1-N1	106.19(10)	O6-S1-N1	111.61(10)
O5-S1-C10	111.23(10)	O6-S1-C10	107.33(10)
N1-S1-C10	102.32(10)	C3-O2-Cu1	124.29(14)
C1-O1-Cu1	124.10(14)	O1-C1-C2	128.2(2)
O1-C1-C4	113.4(2)	C2-C1-C4	118.3(2)
O2-C3-C2	128.4(2)	O2-C3-C5	113.0(2)
C2-C3-C5	118.5(2)	C3-C2-C1	121.6(2)
F4-C5-F6	108.0(2)	F4-C5-F5	106.4(2)
F6-C5-F5	106.4(2)	F4-C5-C3	112.7(2)
F6-C5-C3	113.3(2)	F5-C5-C3	109.6(2)
C6-N1-C7	58.83(15)	C6-N1-S1	116.36(17)
C7-N1-S1	114.50(15)	C12-C11-C10	119.0(2)
C15-C10-C11	120.7(2)	C15-C10-S1	120.08(17)
C11-C10-S1	119.19(18)	C8-C7-C6	123.1(2)
C8-C7-N1	115.7(2)	C6-C7-N1	59.42(16)
C14-C15-C10	118.8(2)	C12-C13-C14	118.0(2)
C12-C13-C16	120.9(3)	C14-C13-C16	121.1(3)
C13-C12-C11	121.7(2)	C7-C6-N1	61.75(16)
C15-C14-C13	121.8(2)	C9-C8-C7	122.4(3)
C17-O3-Cu2	124.57(16)	F10A-C18-F12A	110.1(5)
F11B-C18-F10B	110.0(8)	F12A-C18-F11A	101.8(5)
F10A-C18-F11A	105.7(6)	F11B-C18-F12B	109.3(6)
F10B-C18-F12B	102.2(5)	F10A-C18-C17	114.0(4)
F11B-C18-C17	113.3(4)	F10B-C18-C17	110.1(4)
F12A-C18-C17	112.7(3)	F12B-C18-C17	111.5(3)
F11A-C18-C17	111.6(4)	O3-C17-C18	113.6(2)
O3-C17-C21	128.4(2)	C19-O4-Cu2	124.58(16)
C21-C17-C18	118.0(2)	O4-C19-C20	113.4(2)
O4-C19-C21#2	128.1(2)	F8B-C20-F9B	105.4(6)

C21#2-C19-C20	118.5(2)	F9B-C20-F7B	109.4(7)
F7A-C20-F9A	107.1(18)	F7A-C20-F8A	114.5(16)
F8B-C20-F7B	103.9(7)	F8B-C20-C19	110.9(4)
F9A-C20-F8A	105.8(16)	F9A-C20-C19	110.0(10)
F7A-C20-C19	116.6(12)	F8A-C20-C19	102.3(10)
F7B-C20-C19	113.2(4)	C17-C21-C19#2	121.2(2)
S1-O6-Cu1	142.88(10)	F9B-C20-C19	113.4(5)

Table 4. Anisotropic displacement parameters ($Å^2$) for **Cu(hfacac)$_2$-1** The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ...+ 2hka^*b^*U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Cu1	0.0327(2)	0.0348(2)	0.0303(2)	-0.01311(16)	-0.00587(15)	-0.01128(16)
Cu2	0.0305(2)	0.0499(2)	0.0414(2)	-0.02519(19)	-0.00246(16)	-0.01531(18)
S1	0.0346(3)	0.0311(3)	0.0395(3)	-0.0160(2)	-0.0051(2)	-0.0100(2)
O2	0.0348(8)	0.0415(9)	0.0358(9)	-0.0163(7)	-0.0079(7)	-0.0103(7)
O1	0.0377(8)	0.0432(9)	0.0348(9)	-0.0166(7)	-0.0041(7)	-0.0159(7)
C1	0.0395(13)	0.0412(13)	0.0379(13)	-0.0185(10)	-0.0024(10)	-0.0138(10)
C3	0.0380(12)	0.0326(12)	0.0415(13)	-0.0141(10)	-0.0134(10)	-0.0067(10)
C2	0.0442(14)	0.0593(16)	0.0376(13)	-0.0247(12)	-0.0077(11)	-0.0151(12)
C5	0.0423(14)	0.0663(18)	0.0471(15)	-0.0238(13)	-0.0134(12)	-0.0130(13)
C4	0.0550(19)	0.118(3)	0.0507(18)	-0.0472(19)	0.0070(14)	-0.043(2)
F4	0.0648(11)	0.1078(14)	0.0675(11)	-0.0157(10)	-0.0202(9)	-0.0509(11)
F5	0.0571(11)	0.0760(13)	0.1055(15)	-0.0076(11)	-0.0446(10)	-0.0072(9)
F6	0.0627(11)	0.178(2)	0.0969(15)	-0.0941(16)	-0.0138(10)	-0.0334(13)
F3A	0.081(3)	0.249(12)	0.041(2)	-0.050(5)	0.012(2)	-0.080(6)
F1A	0.081(5)	0.168(7)	0.077(3)	-0.073(4)	0.033(3)	-0.087(5)
F2A	0.056(4)	0.143(5)	0.166(8)	-0.111(5)	0.031(4)	-0.015(3)
N1	0.0361(10)	0.0363(10)	0.0512(12)	-0.0165(9)	-0.0126(9)	-0.0097(8)
C11	0.0460(14)	0.0450(14)	0.0478(15)	-0.0163(12)	0.0023(11)	-0.0168(12)
C10	0.0329(11)	0.0348(12)	0.0362(12)	-0.0174(10)	-0.0047(9)	-0.0102(9)
C7	0.0407(13)	0.0393(13)	0.0509(15)	-0.0139(11)	-0.0165(11)	-0.0086(11)
C15	0.0480(14)	0.0385(13)	0.0371(13)	-0.0167(11)	-0.0038(10)	-0.0104(11)
C13	0.0378(14)	0.0584(17)	0.0621(17)	-0.0372(14)	-0.0103(12)	-0.0032(12)
C12	0.0439(15)	0.0705(19)	0.0540(16)	-0.0318(15)	0.0095(12)	-0.0173(14)
C6	0.0533(16)	0.0692(18)	0.0597(17)	-0.0340(15)	-0.0178(13)	-0.0135(14)
C14	0.0568(16)	0.0382(13)	0.0513(15)	-0.0193(12)	-0.0163(13)	-0.0031(12)
C8	0.0420(16)	0.0697(19)	0.0690(19)	-0.0163(16)	-0.0207(14)	-0.0175(14)
C16	0.0516(18)	0.084(2)	0.096(3)	-0.061(2)	-0.0133(17)	0.0097(16)
C9	0.0384(17)	0.123(3)	0.097(3)	-0.040(2)	-0.0123(17)	-0.0077(19)
O3	0.0405(9)	0.0525(10)	0.0409(9)	-0.0237(8)	-0.0035(7)	-0.0192(8)
C18	0.0664(19)	0.0618(18)	0.0542(18)	-0.0254(15)	-0.0124(15)	-0.0307(16)
C17	0.0521(15)	0.0517(14)	0.0344(12)	-0.0135(11)	-0.0072(11)	-0.0282(12)
O4	0.0327(8)	0.0586(10)	0.0464(10)	-0.0280(8)	-0.0011(7)	-0.0173(8)

C19	0.0371(13)	0.0606(16)	0.0358(13)	-0.0150(12)	-0.0008(10)	-0.0232(12)
C20	0.0379(16)	0.094(3)	0.064(2)	-0.038(2)	0.0058(14)	-0.0283(17)
O6	0.0484(10)	0.0306(8)	0.0570(11)	-0.0136(8)	-0.0047(8)	-0.0129(7)
O5	0.0451(10)	0.0508(10)	0.0455(10)	-0.0281(8)	-0.0069(7)	-0.0102(8)
F7A	0.034(7)	0.18(2)	0.27(3)	-0.19(2)	-0.016(12)	0.004(10)
F9A	0.026(6)	0.173(15)	0.079(9)	-0.035(10)	-0.013(5)	0.002(8)
F8B	0.062(3)	0.121(5)	0.065(3)	-0.033(3)	0.026(2)	-0.030(3)
F10A	0.205(12)	0.078(5)	0.051(3)	-0.014(3)	-0.010(5)	-0.093(7)
F11A	0.104(5)	0.148(8)	0.119(8)	-0.107(7)	0.032(3)	-0.070(4)
F12A	0.141(7)	0.102(5)	0.119(7)	-0.056(5)	-0.079(6)	-0.022(4)
C21	0.0407(14)	0.0691(18)	0.0553(16)	-0.0266(14)	-0.0038(12)	-0.0303(13)
F8A	0.136(14)	0.124(14)	0.144(17)	-0.052(12)	0.105(13)	-0.055(10)
F9B	0.040(2)	0.162(6)	0.144(8)	-0.101(6)	0.003(3)	-0.039(3)
F7B	0.046(3)	0.071(3)	0.149(4)	-0.046(4)	0.022(3)	-0.016(2)
F10B	0.133(7)	0.057(4)	0.119(8)	-0.048(5)	-0.071(7)	0.003(5)
F12B	0.069(3)	0.140(9)	0.198(13)	-0.123(10)	-0.003(5)	-0.051(4)
F11B	0.266(18)	0.113(8)	0.031(3)	-0.031(4)	0.017(6)	-0.132(11)
F1B	0.107(10)	0.112(10)	0.156(11)	-0.051(8)	0.069(7)	-0.078(9)
F3B	0.094(8)	0.218(17)	0.064(10)	-0.100(12)	0.044(7)	-0.107(11)
F2B	0.044(6)	0.24(2)	0.073(6)	-0.068(12)	-0.008(4)	0.021(11)

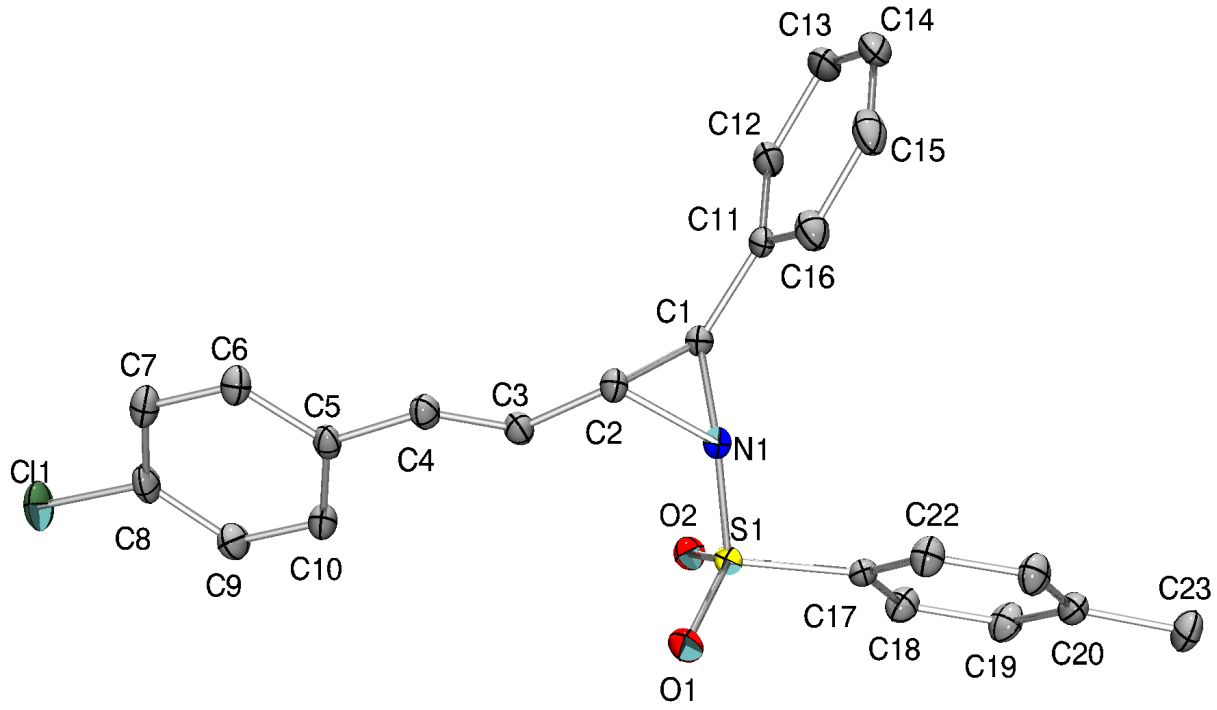
Table 5. Hydrogen coordinates and isotropic displacement parameters ($Å^2$) for **Cu(hfacac)$_2$-1**.

	x/a	**y/b**	**z/c**	**U(eq)**
H2	1.0128	0.0204	0.1436	0.054
H11	0.7343	0.3643	0.5613	0.058
H7	1.1664	0.1846	0.4144	0.053
H15	0.8226	0.6247	0.2259	0.051
H12	0.5520	0.5318	0.6058	0.069
H6A	1.1207	0.3903	0.5022	0.068
H6B	1.0352	0.2905	0.5568	0.068
H14	0.6397	0.7899	0.2735	0.06
H8	1.3034	0.3722	0.3048	0.073
H16A	0.4015	0.7465	0.5479	0.117
H16B	0.4093	0.8356	0.4037	0.117
H16C	0.4828	0.8438	0.4897	0.117
H9A	1.3834	0.1174	0.3116	0.112
H9B	1.4690	0.2158	0.2454	0.112
H21	1.3173	0.5547	0.0377	0.06

Table 6. Torsion angles [°] for **Cu(hfacac)$_2$-1**.

O1#1-Cu1-O2-C3	-175.16(17)	O1-Cu1-O2-C3	4.84(17)
O6-Cu1-O2-C3	98.25(17)	O6#1-Cu1-O2-C3	-81.75(17)
O2-Cu1-O1-C1	-6.19(18)	O2#1-Cu1-O1-C1	173.80(18)
O6#1-Cu1-O1-C1	83.76(18)	O6-Cu1-O1-C1	-96.24(18)
Cu1-O1-C1-C2	4.5(3)	Cu1-O1-C1-C4	-173.9(2)
Cu1-O2-C3-C2	-1.6(3)	Cu1-O2-C3-C5	-179.69(15)
O2-C3-C2-C1	-2.4(4)	C5-C3-C2-C1	175.6(2)
O1-C1-C2-C3	0.7(4)	C4-C1-C2-C3	179.1(3)
O2-C3-C5-F4	-33.3(3)	C2-C3-C5-F4	148.4(2)
O2-C3-C5-F6	-156.3(2)	C2-C3-C5-F6	25.4(4)
O2-C3-C5-F5	85.0(3)	C2-C3-C5-F5	-93.2(3)
O5-S1-N1-C6	-156.85(17)	O6-S1-N1-C6	-28.0(2)
C10-S1-N1-C6	86.44(18)	O5-S1-N1-C7	-91.00(17)
O6-S1-N1-C7	37.82(19)	C10-S1-N1-C7	152.30(16)
C12-C11-C10-C15	0.4(4)	C12-C11-C10-S1	-179.5(2)
O5-S1-C10-C15	-41.9(2)	O6-S1-C10-C15	-171.25(18)
N1-S1-C10-C15	71.2(2)	O5-S1-C10-C11	138.10(19)
O6-S1-C10-C11	8.7(2)	N1-S1-C10-C11	-108.9(2)
C6-N1-C7-C8	-114.8(3)	S1-N1-C7-C8	138.1(2)
S1-N1-C7-C6	-107.1(2)	C11-C10-C15-C14	-0.4(4)
S1-C10-C15-C14	179.58(19)	C14-C13-C12-C11	-0.5(4)
C16-C13-C12-C11	179.6(3)	C10-C11-C12-C13	0.0(4)
C8-C7-C6-N1	102.5(3)	S1-N1-C6-C7	103.95(18)
C10-C15-C14-C13	-0.2(4)	C12-C13-C14-C15	0.6(4)
C16-C13-C14-C15	-179.5(3)	C6-C7-C8-C9	150.1(3)
N1-C7-C8-C9	-141.0(3)	O4-Cu2-O3-C17	-178.76(19)
O4#2-Cu2-O3-C17	1.24(19)	Cu2-O3-C17-C18	-175.90(16)
Cu2-O3-C17-C21	4.1(4)	F10A-C18-C17-O3	-94.2(8)
F11B-C18-C17-O3	69.9(12)	F10B-C18-C17-O3	-53.7(8)
F12A-C18-C17-O3	139.3(8)	F12B-C18-C17-O3	-166.4(9)
F11A-C18-C17-O3	25.5(7)	F10A-C18-C17-C21	85.8(8)
F11B-C18-C17-C21	-110.2(12)	F10B-C18-C17-C21	126.2(8)
F12A-C18-C17-C21	-40.7(8)	F12B-C18-C17-C21	13.6(10)
F11A-C18-C17-C21	-154.6(7)	O3#2-Cu2-O4-C19	7.07(19)
O3-Cu2-O4-C19	-172.93(19)	Cu2-O4-C19-C21#2	-8.5(4)
Cu2-O4-C19-C20	170.38(18)	O4-C19-C20-F7A	18.(3)

C21#2-C19-C20-F7A	-163.(3)	O4-C19-C20-F8B	-80.9(7)
C21#2-C19-C20-F8B	98.1(7)	O4-C19-C20-F9B	160.8(8)
C21#2-C19-C20-F9B	-20.2(9)	O4-C19-C20-F9A	140.1(18)
C21#2-C19-C20-F9A	-40.9(18)	O4-C19-C20-F7B	35.5(6)
C21#2-C19-C20-F7B	-145.6(6)	O4-C19-C20-F8A	-108.(3)
C21#2-C19-C20-F8A	71.(3)	O5-S1-O6-Cu1	33.3(2)
N1-S1-O6-Cu1	-89.45(18)	C10-S1-O6-Cu1	159.21(16)
O2#1-Cu1-O6-S1	98.98(18)	O2-Cu1-O6-S1	-81.02(18)
O1-Cu1-O6-S1	11.98(18)	O1#1-Cu1-O6-S1	-168.02(18)
O6#1-Cu1-O6-S1	-120.(30)	O3-C17-C21-C19#2	-5.0(4)
C18-C17-C21-C19#2	175.0(2)		



The asymmetric unit of Aziridine **SI 34**, with displacement ellipsoids at the 50% probability level.

Table 1. Crystal data and structure refinement for **SI 34**.

Identification code	jn202	
Chemical formula	$C_{23}H_{20}ClNO_2S$	
Formula weight	409.91	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal size	0.200 x 0.300 x 0.400 mm	
Crystal habit	clear colorless rod	
Crystal system	monoclinic	
Space group	P 2_1	
Unit cell dimensions	a = 10.3431(4) Å	α = 90°
	b = 9.0814(4) Å	β = 102.940(2)°
	c = 10.9394(4) Å	γ = 90°
Volume	1001.44(7) $Å^3$	
Z	2	
Density (calculated)	1.359 g/cm^3	
Absorption coefficient	0.314 mm^{-1}	
F(000)	428	
Theta range for data collection	1.91 to 29.94°	
Index ranges	-14<=h<=14, -12<=k<=12, -15<=l<=15	
Reflections collected	24912	
Independent reflections	5721 [R(int) = 0.0242]	
Coverage of independent reflections	99.5%	
Absorption correction	multi-scan	
Max. and min. transmission	0.9399 and 0.8847	
Structure solution technique	direct methods	
Structure solution program	SHELXS-97 (Sheldrick, 2008)	
Refinement method	Full-matrix least-squares on F2	
Refinement program	SHELXL-97 (Sheldrick, 2008)	
Function minimized	Σ w(Fo2 - Fc2)2	
Data / restraints / parameters	5721 / 1 / 254	
Goodness-of-fit on F2	0.975	
Δ/σmax	0.001	
Final R indices	5561 data; I>2σ(I)	R1 = 0.0268, wR2 = 0.0625
	all data	R1 = 0.0279, wR2 = 0.0631
Weighting scheme	$w=1/[\sigma^2(F_o^2)+(0.0273P)^2+0.3328P]$ where $P=(F_o^2+2F_c^2)/3$	
Absolute structure parameter	-0.003(32)	
Largest diff. peak and hole	0.340 and -0.253 $eÅ^{-3}$	
R.M.S. deviation from mean	0.040 $eÅ^{-3}$	

Table 2. Atomic coordinates and equivalent isotropic displacement parameters ($Å^2$) for **SI 34**. U_{eq} is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
S1	0.52734(3)	0.51568(3)	0.29450(2)	0.01539(6)
Cl1	0.06847(3)	0.34121(4)	0.08968(3)	0.02894(8)
O1	0.61442(10)	0.64120(11)	0.31262(9)	0.0236(2)
O2	0.58640(9)	0.37286(11)	0.32726(8)	0.01953(19)
N1	0.40804(10)	0.55462(12)	0.36803(9)	0.01593(19)
C17	0.43614(10)	0.50763(16)	0.13783(10)	0.0154(2)
C22	0.34485(13)	0.61700(15)	0.08925(13)	0.0207(2)
C11	0.19940(12)	0.43470(15)	0.40560(11)	0.0174(2)
C18	0.46432(13)	0.39466(15)	0.06269(12)	0.0206(2)
C5	0.72157(11)	0.51111(16)	0.79554(10)	0.0174(2)
C8	0.93701(12)	0.41018(15)	0.97699(12)	0.0208(2)
C1	0.34675(11)	0.43457(14)	0.42661(11)	0.0157(2)
C10	0.82769(13)	0.44013(16)	0.76105(12)	0.0206(2)
C3	0.56038(11)	0.50144(15)	0.58919(10)	0.0181(2)
C6	0.72831(12)	0.53470(15)	0.92311(11)	0.0202(2)
C21	0.28142(13)	0.61123(16)	0.96314(13)	0.0222(3)
C9	0.93502(13)	0.38852(16)	0.85120(12)	0.0230(3)
C4	0.60213(12)	0.55795(14)	0.70365(11)	0.0183(2)
C7	0.83546(13)	0.48461(15)	0.01436(12)	0.0222(3)
C20	0.30951(12)	0.49973(16)	0.88524(11)	0.0196(2)
C14	0.92452(14)	0.4280(2)	0.37413(13)	0.0337(4)
C2	0.43252(11)	0.54657(14)	0.50786(11)	0.0166(2)
C19	0.40042(14)	0.39187(16)	0.93617(12)	0.0230(3)
C16	0.12511(13)	0.56013(17)	0.36745(13)	0.0237(3)
C12	0.13635(13)	0.30531(17)	0.42754(12)	0.0238(3)
C23	0.24286(14)	0.49886(19)	0.74734(11)	0.0275(3)
C13	0.99858(14)	0.3026(2)	0.41132(13)	0.0333(4)
C15	0.98742(13)	0.55698(19)	0.35210(13)	0.0303(3)

Table 3. Bond lengths [Å] and angles [°] for **SI 34**.

S1-O1	1.4387(10)	S1-O2	1.4443(10)
S1-N1	1.6550(10)	S1-C17	1.7631(11)
Cl1-C8	1.7350(13)	N1-C1	1.4775(16)
N1-C2	1.4952(15)	C17-C18	1.3857(18)
C17-C22	1.3913(18)	C22-C21	1.3896(18)
C11-C16	1.3851(19)	C11-C12	1.3906(19)
C11-C1	1.4895(16)	C18-C19	1.3941(18)
C5-C10	1.3958(18)	C5-C6	1.3981(16)
C5-C4	1.4702(16)	C8-C9	1.3857(19)
C8-C7	1.3854(19)	C1-C2	1.5028(17)
C10-C9	1.3908(17)	C3-C4	1.3325(17)
C3-C2	1.4770(16)	C6-C7	1.3914(17)
C21-C20	1.3953(19)	C20-C19	1.3854(19)
C20-C23	1.5116(16)	C14-C13	1.382(3)
C14-C15	1.387(3)	C16-C15	1.3965(18)
C12-C13	1.3961(18)	O1-S1-O2	117.41(6)
O2-S1-N1	113.03(5)	O1-S1-N1	106.00(6)
O2-S1-C17	107.96(6)	O1-S1-C17	110.42(6)
C1-N1-C2	60.73(8)	N1-S1-C17	100.75(5)
C2-N1-S1	120.86(8)	C1-N1-S1	119.48(8)
C18-C17-S1	118.00(10)	C18-C17-C22	121.12(11)
C17-C22-C21	118.78(12)	C22-C17-S1	120.75(10)
C16-C11-C1	121.77(12)	C16-C11-C12	119.80(11)
C17-C18-C19	119.11(12)	C12-C11-C1	118.42(12)
C10-C5-C4	122.69(11)	C10-C5-C6	118.31(11)
C9-C8-C7	121.24(12)	C6-C5-C4	118.99(11)
C7-C8-Cl1	119.46(10)	C9-C8-Cl1	119.29(11)
N1-C1-C2	60.22(7)	N1-C1-C11	117.13(10)
C9-C10-C5	121.00(12)	C11-C1-C2	122.61(11)
C7-C6-C5	121.31(12)	C4-C3-C2	121.15(12)
C8-C9-C10	119.23(13)	C20-C21-C22	121.14(12)
C8-C7-C6	118.85(12)	C3-C4-C5	125.24(12)
C19-C20-C23	120.96(12)	C19-C20-C21	118.87(11)
C13-C14-C15	119.76(13)	C21-C20-C23	120.16(12)
C3-C2-C1	119.92(11)	C3-C2-N1	123.61(10)
C20-C19-C18	120.97(12)	N1-C2-C1	59.05(7)

C11-C12-C13	119.85(14)	C11-C16-C15	120.10(14)
C14-C15-C16	120.11(14)	C14-C13-C12	120.38(15)

Table 4. Anisotropic displacement parameters ($Å^2$) for **SI 34.** The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ...+ 2hka^*b^*U^{12}]$

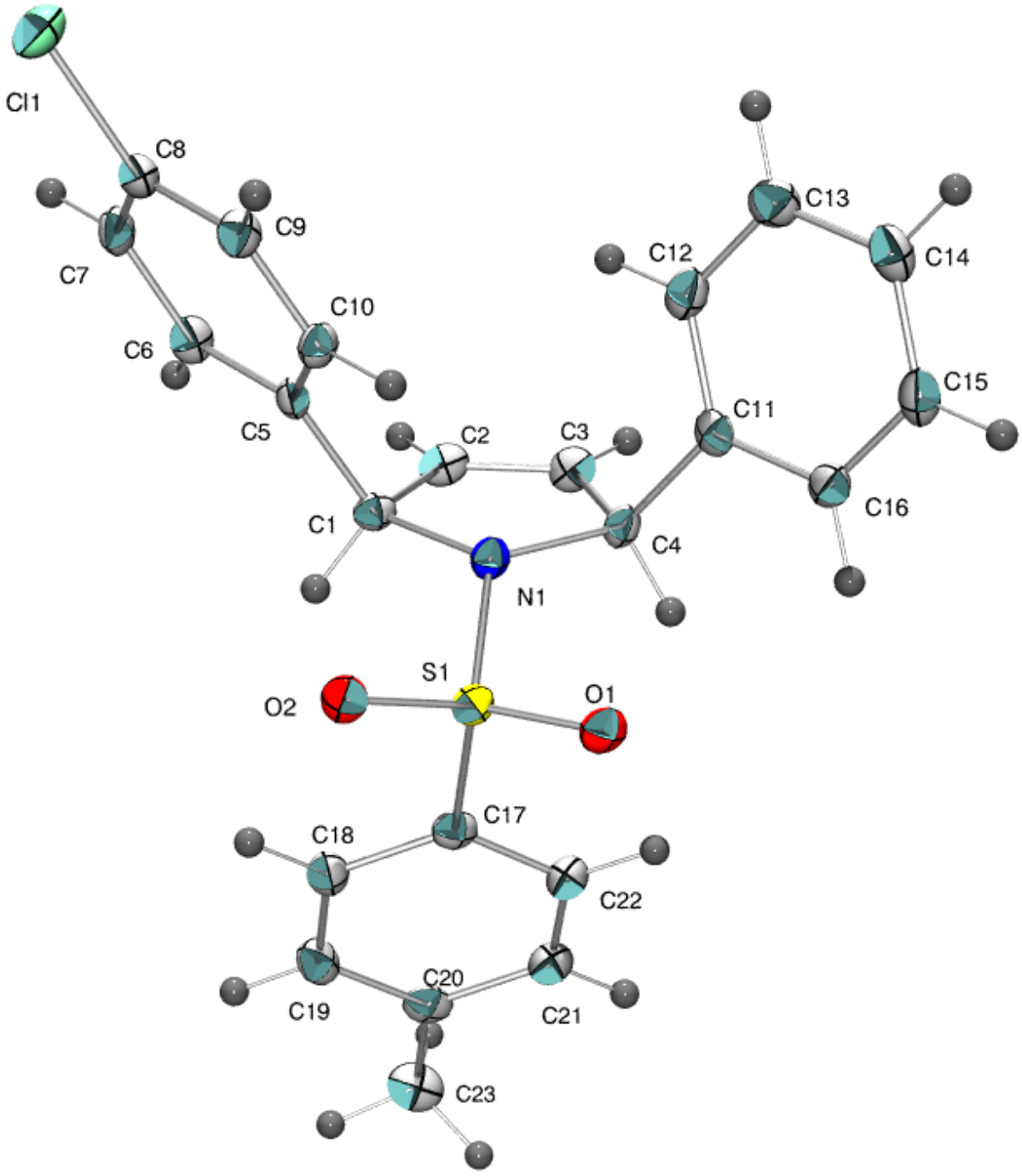
	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
S1	0.01256(11)	0.01852(13)	0.01501(11)	0.00138(11)	0.00291(9)	-0.00162(11)
Cl1	0.02600(15)	0.03296(19)	0.02249(14)	0.00287(13)	-0.00596(11)	0.00565(14)
O1	0.0206(4)	0.0260(5)	0.0232(5)	0.0016(4)	0.0027(4)	-0.0089(4)
O2	0.0158(4)	0.0223(5)	0.0204(4)	0.0033(3)	0.0036(3)	0.0032(3)
N1	0.0151(4)	0.0188(5)	0.0136(4)	-0.0002(4)	0.0026(3)	-0.0011(4)
C17	0.0148(4)	0.0182(5)	0.0137(4)	0.0019(5)	0.0041(4)	-0.0022(5)
C22	0.0239(6)	0.0186(6)	0.0193(6)	-0.0014(4)	0.0044(5)	0.0038(5)
C11	0.0142(5)	0.0260(6)	0.0116(5)	-0.0022(4)	0.0022(4)	-0.0018(5)
C18	0.0220(6)	0.0214(6)	0.0192(6)	0.0014(5)	0.0064(5)	0.0049(5)
C5	0.0184(5)	0.0159(5)	0.0162(5)	0.0011(5)	0.0002(4)	-0.0042(5)
C8	0.0195(6)	0.0198(6)	0.0194(6)	0.0035(5)	-0.0034(4)	-0.0019(5)
C1	0.0143(5)	0.0183(6)	0.0143(5)	0.0010(4)	0.0029(4)	-0.0006(4)
C10	0.0203(6)	0.0246(7)	0.0156(5)	0.0011(5)	0.0013(4)	-0.0017(5)
C3	0.0164(5)	0.0192(6)	0.0179(5)	0.0007(5)	0.0020(4)	-0.0019(5)
C6	0.0227(5)	0.0195(7)	0.0172(5)	-0.0015(5)	0.0019(4)	0.0003(5)
C21	0.0242(6)	0.0219(6)	0.0197(6)	0.0019(5)	0.0029(5)	0.0050(5)
C9	0.0189(6)	0.0271(7)	0.0222(6)	0.0027(5)	0.0027(5)	0.0017(5)
C4	0.0177(5)	0.0180(6)	0.0180(5)	0.0021(4)	0.0016(4)	-0.0006(4)
C7	0.0271(6)	0.0220(7)	0.0155(5)	-0.0004(5)	0.0004(4)	-0.0001(5)
C20	0.0197(5)	0.0239(6)	0.0159(5)	-0.0005(5)	0.0050(4)	-0.0030(5)
C14	0.0141(6)	0.0653(12)	0.0212(6)	-0.0004(7)	0.0029(5)	-0.0015(6)
C2	0.0174(5)	0.0182(6)	0.0138(5)	-0.0008(4)	0.0025(4)	-0.0007(4)
C19	0.0282(6)	0.0242(7)	0.0181(6)	-0.0030(5)	0.0080(5)	0.0039(5)
C16	0.0196(6)	0.0275(7)	0.0230(6)	-0.0039(5)	0.0027(5)	0.0026(5)
C12	0.0192(6)	0.0306(8)	0.0196(6)	0.0049(5)	0.0004(4)	-0.0057(5)
C23	0.0302(6)	0.0367(8)	0.0148(5)	-0.0001(6)	0.0039(4)	0.0002(6)
C13	0.0197(6)	0.0543(11)	0.0239(7)	0.0089(6)	0.0006(5)	-0.0142(6)
C15	0.0193(6)	0.0457(10)	0.0239(6)	-0.0071(6)	0.0006(5)	0.0096(6)

Table 5. Hydrogen coordinates and isotropic displacement parameters ($Å^2$) for **SI 34**.

	x/a	**y/b**	**z/c**	**U(eq)**
H22	0.3262	0.6942	0.1413	0.025
H18	0.5263	0.3201	0.0969	0.025
H1	0.3881	0.3354	0.4242	0.019
H10	0.8266	0.4269	0.6747	0.025
H3	0.6138	0.4309	0.5594	0.022
H6	0.6583	0.5859	0.9480	0.024
H21	0.2178	0.6845	-0.0706	0.027
H9	1.0062	0.3390	0.8268	0.028
H4	0.5506	0.6347	0.7278	0.022
H7	0.8389	0.5012	1.1008	0.027
H14	-0.1691	0.4258	0.3637	0.04
H2	0.3824	0.6209	0.5463	0.02
H19	0.4195	0.3149	-0.1159	0.028
H16	0.1679	0.6484	0.3517	0.028
H12	0.1869	0.2190	0.4535	0.029
H23A	0.2672	0.4089	-0.2915	0.041
H23B	0.2717	0.5850	-0.2935	0.041
H23C	0.1464	0.5022	-0.2623	0.041
H13	-0.0445	0.2141	0.4259	0.04
H15	-0.0633	0.6433	0.3265	0.036

Table 6. Torsion angles [°] for **SI 34**.

O1-S1-N1-C1	-147.45(9)	O2-S1-N1-C1	-17.49(10)
C17-S1-N1-C1	97.46(9)	O1-S1-N1-C2	-75.90(10)
O2-S1-N1-C2	54.06(10)	C17-S1-N1-C2	169.01(9)
O1-S1-C17-C18	110.97(11)	O2-S1-C17-C18	-18.61(11)
N1-S1-C17-C18	-137.31(10)	O1-S1-C17-C22	-64.95(11)
O2-S1-C17-C22	165.47(10)	N1-S1-C17-C22	46.77(11)
C18-C17-C22-C21	0.15(19)	S1-C17-C22-C21	175.93(10)
C22-C17-C18-C19	0.46(19)	S1-C17-C18-C19	-175.44(10)
C2-N1-C1-C11	113.85(12)	S1-N1-C1-C11	-135.14(10)
S1-N1-C1-C2	111.01(9)	C16-C11-C1-N1	-19.17(17)
C12-C11-C1-N1	162.06(11)	C16-C11-C1-C2	51.28(16)
C12-C11-C1-C2	-127.49(13)	C6-C5-C10-C9	2.7(2)
C4-C5-C10-C9	-175.58(13)	C10-C5-C6-C7	-2.2(2)
C4-C5-C6-C7	176.09(12)	C17-C22-C21-C20	-1.0(2)
C7-C8-C9-C10	-1.3(2)	Cl1-C8-C9-C10	178.14(11)
C5-C10-C9-C8	-1.0(2)	C2-C3-C4-C5	174.39(11)
C10-C5-C4-C3	22.6(2)	C6-C5-C4-C3	-155.59(13)
C9-C8-C7-C6	1.7(2)	Cl1-C8-C7-C6	-177.71(10)
C5-C6-C7-C8	0.1(2)	C22-C21-C20-C19	1.3(2)
C22-C21-C20-C23	-177.80(13)	C4-C3-C2-N1	147.50(12)
C4-C3-C2-C1	-141.76(13)	C1-N1-C2-C3	107.45(13)
S1-N1-C2-C3	-1.36(16)	S1-N1-C2-C1	-108.81(10)
N1-C1-C2-C3	-113.55(12)	C11-C1-C2-C3	141.52(12)
C11-C1-C2-N1	-104.93(12)	C21-C20-C19-C18	-0.7(2)
C23-C20-C19-C18	178.42(13)	C17-C18-C19-C20	-0.2(2)
C12-C11-C16-C15	0.53(19)	C1-C11-C16-C15	-178.22(12)
C16-C11-C12-C13	-0.2(2)	C1-C11-C12-C13	178.64(12)
C15-C14-C13-C12	0.4(2)	C11-C12-C13-C14	-0.3(2)
C13-C14-C15-C16	0.0(2)	C11-C16-C15-C14	-0.5(2)



The asymmetric unit of Pyrrole **SI 39**, with displacement ellipsoids at the 50% probability level.

Table 1. Crystal data and structure refinement fo **SI 39**.

Identification code	jn203b	
Chemical formula	$C_{23}H_{20}ClNO_2S$	
Formula weight	409.91	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal size	0.05 x 0.30 x 0.30 mm	
Crystal system	monoclinic	
Space group	P 1 21 1	
Unit cell dimensions	a = 11.9219(4) Å	α = 90°
	b = 12.6553(4) Å	β = 106.461(2)°
	c = 13.5310(4) Å	γ = 90°
Volume	1957.82(11) $Å^3$	
Z	4	
Density (calculated)	1.391 Mg/cm^3	
Absorption coefficient	0.321 mm^{-1}	
F(000)	856	
Theta range for data collection	1.57 to 24.36°	
Index ranges	-13<=h<=13, -14<=k<=14, -15<=l<=15	
Reflections collected	33148	
Independent reflections	6414 [R(int) = 0.0501]	
Coverage of independent reflections	99.9%	
Absorption correction	multi-scan	
Structure solution technique	direct methods	
Structure solution program	SHELXS-97 (Sheldrick, 2008)	
Refinement method	Full-matrix least-squares on F^2	
Refinement program	SHELXL-97 (Sheldrick, 2008)	
Function minimized	$\Sigma\, w(F_o^2 - F_c^2)^2$	
Data / restraints / parameters	6414 / 1 / 507	
Goodness-of-fit on F^2	1.021	
Final R indices	5697 data; I>2σ(I)	R1 = 0.0343, wR2 = 0.0778
	all data	R1 = 0.0426, wR2 = 0.0819
Weighting scheme	$w=1/[\sigma^2(F_o^2)+(0.0378P)^2+0.8444P]$ where $P=(F_o^2+2F_c^2)/3$	
Absolute structure parameter	0.01(4)	
Largest diff. peak and hole	0.431 and -0.316 $eÅ^{-3}$	
R.M.S. deviation from mean	0.045 $eÅ^{-3}$	

Table 2. Atomic coordinates and equivalent isotropic displacement parameters ($Å^2$) for **SI 39**. U_{eq} is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x/a	y/b	z/c	U(eq)
S2	0.99969(6)	0.53607(5)	0.96651(5)	0.01687(16)
S1	0.49008(6)	0.05413(5)	0.02576(5)	0.01746(17)
Cl1	0.33366(7)	0.99295(6)	0.53419(5)	0.02946(19)
Cl2	0.55967(7)	0.21803(7)	0.55948(6)	0.0389(2)
O1	0.60832(16)	0.05716(16)	0.02046(14)	0.0216(5)
O4	0.88249(16)	0.52302(16)	0.97375(14)	0.0202(5)
O3	0.03498(17)	0.63518(15)	0.93383(15)	0.0225(5)
O2	0.44408(17)	0.95932(15)	0.05698(15)	0.0212(5)
N2	0.0184(2)	0.44673(18)	0.88584(17)	0.0160(5)
N1	0.4805(2)	0.14558(19)	0.10722(17)	0.0168(5)
C40	0.0948(2)	0.5065(2)	0.0892(2)	0.0160(6)
C1	0.3647(2)	0.1609(2)	0.1262(2)	0.0169(6)
C18	0.2868(2)	0.0471(3)	0.8686(2)	0.0204(6)
C22	0.4371(3)	0.1630(2)	0.8435(2)	0.0219(7)
C41	0.0552(3)	0.4425(2)	0.1564(2)	0.0192(7)
C10	0.4499(2)	0.0594(2)	0.2925(2)	0.0186(6)
C11	0.6579(3)	0.2564(2)	0.1886(2)	0.0191(7)
C19	0.2136(3)	0.0796(2)	0.7741(2)	0.0230(7)
C39	0.2217(2)	0.4894(2)	0.7184(2)	0.0209(7)
C38	0.2166(3)	0.5021(2)	0.6156(2)	0.0232(7)
C42	0.1318(3)	0.4150(2)	0.2505(2)	0.0225(7)
C34	0.1206(2)	0.4679(2)	0.7473(2)	0.0165(6)
C44	0.2839(2)	0.5160(2)	0.2109(2)	0.0209(7)
C20	0.2494(3)	0.1551(2)	0.7146(2)	0.0215(7)
C17	0.3980(2)	0.0902(2)	0.9033(2)	0.0186(7)
C43	0.2479(3)	0.4507(2)	0.2789(2)	0.0217(7)
C16	0.7618(3)	0.2442(2)	0.1628(2)	0.0213(7)
C37	0.1102(2)	0.4941(2)	0.5397(2)	0.0230(7)
C24	0.1326(2)	0.4470(2)	0.8601(2)	0.0182(6)
C21	0.3631(3)	0.1948(3)	0.7506(2)	0.0241(7)
C25	0.1753(3)	0.3356(2)	0.8882(2)	0.0206(7)
C46	0.3309(3)	0.4160(3)	0.3785(2)	0.0346(9)
C6	0.2574(2)	0.1324(2)	0.2591(2)	0.0204(7)
C29	0.7669(2)	0.3288(2)	0.8246(2)	0.0202(7)
C7	0.2496(2)	0.0962(2)	0.3529(2)	0.0208(7)

	x/a	y/b	z/c	U(eq)
C28	0.8782(2)	0.2995(2)	0.8204(2)	0.0170(6)
C12	0.6622(3)	0.2733(2)	0.2906(2)	0.0234(7)
C32	0.7914(3)	0.2171(3)	0.6556(2)	0.0259(7)
C2	0.3550(3)	0.2791(2)	0.1262(2)	0.0202(7)
C26	0.0955(2)	0.2764(2)	0.9108(2)	0.0206(7)
C31	0.6827(3)	0.2491(2)	0.6613(2)	0.0254(7)
C9	0.4417(2)	0.0210(2)	0.3857(2)	0.0202(7)
C3	0.4503(3)	0.3260(2)	0.1179(2)	0.0210(7)
C30	0.6692(3)	0.3034(2)	0.7453(2)	0.0222(7)
C14	0.8723(3)	0.2665(2)	0.3399(2)	0.0256(7)
C15	0.8686(3)	0.2484(2)	0.2382(2)	0.0250(7)
C33	0.8889(3)	0.2422(2)	0.7362(2)	0.0213(6)
C35	0.0150(2)	0.4611(2)	0.6712(2)	0.0197(6)
C23	0.1680(3)	0.1958(3)	0.6154(2)	0.0285(8)
C8	0.3423(2)	0.0407(2)	0.4159(2)	0.0212(6)
C36	0.0096(3)	0.4737(2)	0.5684(2)	0.0228(7)
C13	0.7690(3)	0.2793(2)	0.3660(2)	0.0254(7)
C4	0.5416(2)	0.2497(2)	0.1059(2)	0.0177(6)
C5	0.3580(2)	0.1152(2)	0.2276(2)	0.0163(6)
C27	0.9849(2)	0.3348(2)	0.9053(2)	0.0176(6)
C45	0.2094(2)	0.5447(2)	0.1172(2)	0.0190(6)

Table 3. Bond lengths [Å] and angles [°] for **SI 39.**

S2-O3	1.432(2)	S2-O4	1.4376(19)
S2-N2	1.630(2)	S2-C40	1.765(3)
S1-O2	1.432(2)	S1-O1	1.4321(19)
S1-N1	1.624(2)	S1-C17	1.769(3)
Cl1-C8	1.741(3)	Cl2-C31	1.749(3)
N2-C24	1.497(3)	N2-C27	1.515(4)
N1-C1	1.487(3)	N1-C4	1.508(4)
C40-C41	1.396(4)	C40-C45	1.396(4)
C1-C2	1.500(4)	C1-C5	1.512(4)
C18-C17	1.387(4)	C18-C19	1.390(4)
C22-C21	1.376(4)	C22-C17	1.391(4)
C41-C42	1.385(4)	C10-C9	1.381(4)
C10-C5	1.387(4)	C11-C12	1.383(4)
C11-C16	1.388(4)	C11-C4	1.517(4)
C19-C20	1.392(4)	C39-C38	1.384(4)
C39-C34	1.396(4)	C38-C37	1.392(4)
C42-C43	1.402(4)	C34-C35	1.385(4)
C34-C24	1.514(4)	C44-C45	1.376(4)
C44-C43	1.391(4)	C20-C21	1.397(4)
C20-C23	1.507(4)	C43-C46	1.494(4)
C16-C15	1.389(4)	C37-C36	1.386(4)
C24-C25	1.511(4)	C25-C26	1.312(4)
C6-C7	1.377(4)	C6-C5	1.398(4)
C29-C30	1.380(4)	C29-C28	1.395(4)
C7-C8	1.380(4)	C28-C33	1.386(4)
C28-C27	1.519(4)	C12-C13	1.390(4)
C32-C31	1.380(4)	C32-C33	1.387(4)
C2-C3	1.315(4)	C26-C27	1.495(4)
C31-C30	1.376(4)	C9-C8	1.381(4)
C3-C4	1.499(4)	C14-C13	1.383(4)
C14-C15	1.384(4)	C35-C36	1.384(4)
O3-S2-O4	119.85(12)	O3-S2-N2	107.02(12)
O4-S2-N2	106.63(12)	O3-S2-C40	108.02(13)
O4-S2-C40	106.83(12)	N2-S2-C40	108.01(13)
O2-S1-O1	120.13(12)	O2-S1-N1	106.88(12)
O1-S1-N1	105.93(12)	O2-S1-C17	107.75(13)
O1-S1-C17	107.73(12)	N1-S1-C17	107.89(13)

C24-N2-C27	110.2(2)	C24-N2-S2	117.40(19)
C27-N2-S2	116.01(18)	C1-N1-C4	111.3(2)
C1-N1-S1	117.23(19)	C4-N1-S1	119.17(18)
C41-C40-C45	120.7(3)	C41-C40-S2	119.3(2)
C45-C40-S2	120.0(2)	N1-C1-C2	101.9(2)
N1-C1-C5	113.4(2)	C2-C1-C5	111.0(2)
C17-C18-C19	119.1(3)	C21-C22-C17	119.4(3)
C42-C41-C40	119.2(3)	C9-C10-C5	120.7(3)
C12-C11-C16	119.0(3)	C12-C11-C4	120.8(3)
C16-C11-C4	120.1(3)	C18-C19-C20	121.3(3)
C38-C39-C34	120.6(3)	C39-C38-C37	120.3(3)
C41-C42-C43	120.9(3)	C35-C34-C39	118.6(3)
C35-C34-C24	123.0(3)	C39-C34-C24	118.3(2)
C45-C44-C43	121.8(3)	C19-C20-C21	118.2(3)
C19-C20-C23	121.6(3)	C21-C20-C23	120.2(3)
C18-C17-C22	120.6(3)	C18-C17-S1	119.7(2)
C22-C17-S1	119.6(2)	C44-C43-C42	118.4(3)
C44-C43-C46	121.4(3)	C42-C43-C46	120.1(3)
C11-C16-C15	120.5(3)	C36-C37-C38	119.1(3)
N2-C24-C25	101.7(2)	N2-C24-C34	113.7(2)
C25-C24-C34	110.2(2)	C22-C21-C20	121.4(3)
C26-C25-C24	112.1(3)	C7-C6-C5	121.1(3)
C30-C29-C28	120.7(3)	C6-C7-C8	119.2(3)
C33-C28-C29	118.8(3)	C33-C28-C27	121.6(3)
C29-C28-C27	119.5(3)	C11-C12-C13	120.5(3)
C31-C32-C33	118.7(3)	C3-C2-C1	112.5(3)
C25-C26-C27	113.2(3)	C30-C31-C32	121.6(3)
C30-C31-Cl2	119.6(2)	C32-C31-Cl2	118.8(2)
C10-C9-C8	119.7(3)	C2-C3-C4	113.0(3)
C31-C30-C29	119.2(3)	C13-C14-C15	119.5(3)
C14-C15-C16	120.1(3)	C28-C33-C32	121.0(3)
C36-C35-C34	120.9(3)	C7-C8-C9	120.9(3)
C7-C8-Cl1	119.9(2)	C9-C8-Cl1	119.2(2)
C35-C36-C37	120.4(3)	C14-C13-C12	120.2(3)
C3-C4-N1	101.2(2)	C3-C4-C11	115.1(2)
N1-C4-C11	112.5(2)	C10-C5-C6	118.5(3)
C10-C5-C1	122.3(2)	C6-C5-C1	119.2(2)
C26-C27-N2	101.4(2)	C26-C27-C28	115.4(2)

N2-C27-C28	110.5(2)	C44-C45-C40	119.0(3)

Table 4. Anisotropic displacement parameters ($Å^2$) The anisotropic displacement factor exponent for **SI 39** takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ...+ 2hka^*b^*U^{12}]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
S2	0.0154(3)	0.0163(4)	0.0204(4)	-0.0006(3)	0.0074(3)	0.0001(3)
S1	0.0163(4)	0.0184(4)	0.0191(4)	-0.0017(3)	0.0073(3)	0.0018(3)
Cl1	0.0317(4)	0.0351(5)	0.0254(4)	0.0057(3)	0.0143(3)	-0.0001(4)
Cl2	0.0340(5)	0.0421(5)	0.0321(4)	-0.0007(4)	-0.0041(4)	-0.0158(4)
O1	0.0164(10)	0.0272(12)	0.0229(10)	-0.0035(10)	0.0080(8)	0.0036(9)
O4	0.0160(10)	0.0208(12)	0.0259(11)	-0.0006(9)	0.0096(9)	-0.0009(9)
O3	0.0261(12)	0.0152(11)	0.0284(12)	0.0016(9)	0.0113(10)	-0.0031(9)
O2	0.0230(11)	0.0172(11)	0.0250(11)	-0.0011(9)	0.0095(9)	0.0007(9)
N2	0.0159(12)	0.0144(13)	0.0204(13)	-0.0031(10)	0.0096(10)	-0.0017(10)
N1	0.0154(13)	0.0167(13)	0.0193(13)	-0.0001(10)	0.0067(10)	0.0011(10)
C40	0.0139(14)	0.0146(15)	0.0212(15)	-0.0060(13)	0.0077(12)	-0.0009(12)
C1	0.0169(15)	0.0182(16)	0.0162(14)	-0.0007(12)	0.0055(12)	0.0022(12)
C18	0.0192(15)	0.0185(15)	0.0253(15)	-0.0039(14)	0.0092(12)	-0.0005(14)
C22	0.0206(16)	0.0248(18)	0.0223(16)	-0.0020(14)	0.0094(13)	-0.0022(14)
C41	0.0165(15)	0.0213(17)	0.0210(16)	-0.0044(13)	0.0075(13)	-0.0021(13)
C10	0.0141(14)	0.0225(17)	0.0208(14)	-0.0001(13)	0.0077(11)	0.0009(12)
C11	0.0232(16)	0.0106(15)	0.0243(16)	-0.0007(12)	0.0081(13)	-0.0043(12)
C19	0.0151(15)	0.0258(18)	0.0278(17)	-0.0064(14)	0.0055(13)	-0.0003(13)
C39	0.0147(15)	0.0234(17)	0.0259(16)	-0.0029(14)	0.0077(12)	-0.0035(13)
C38	0.0206(15)	0.0262(17)	0.0266(16)	-0.0005(14)	0.0126(13)	-0.0007(14)
C42	0.0241(17)	0.0234(18)	0.0211(16)	-0.0023(13)	0.0082(13)	-0.0028(14)
C34	0.0205(15)	0.0100(15)	0.0194(14)	-0.0018(12)	0.0062(12)	0.0025(12)
C44	0.0149(14)	0.0225(18)	0.0261(16)	-0.0107(13)	0.0068(12)	-0.0009(13)
C20	0.0242(17)	0.0191(17)	0.0227(16)	-0.0064(13)	0.0090(13)	0.0045(13)
C17	0.0157(15)	0.0227(17)	0.0176(15)	-0.0025(13)	0.0050(12)	0.0037(13)
C43	0.0193(16)	0.0234(17)	0.0219(16)	-0.0082(13)	0.0049(13)	0.0014(14)
C16	0.0252(17)	0.0172(16)	0.0247(15)	-0.0048(13)	0.0122(13)	-0.0047(13)
C37	0.0242(16)	0.0255(17)	0.0212(15)	-0.0003(14)	0.0094(13)	0.0021(14)
C24	0.0105(14)	0.0223(16)	0.0218(15)	-0.0027(13)	0.0044(12)	-0.0023(12)
C21	0.0250(17)	0.0264(18)	0.0237(16)	-0.0036(13)	0.0117(14)	-0.0024(14)
C25	0.0164(15)	0.0286(18)	0.0174(15)	0.0014(13)	0.0060(13)	0.0065(14)
C46	0.0239(18)	0.051(2)	0.0256(18)	0.0007(17)	0.0015(15)	-0.0047(16)
C6	0.0172(16)	0.0188(16)	0.0254(16)	-0.0011(13)	0.0065(13)	0.0025(13)
C29	0.0195(16)	0.0151(16)	0.0266(17)	0.0005(13)	0.0076(13)	-0.0020(13)
C7	0.0136(15)	0.0235(17)	0.0272(17)	-0.0034(13)	0.0087(13)	-0.0029(13)

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C28	0.0188(15)	0.0119(15)	0.0210(15)	0.0034(12)	0.0068(12)	-0.0026(12)
C12	0.0253(17)	0.0195(17)	0.0292(17)	0.0001(13)	0.0136(14)	-0.0018(13)
C32	0.0347(18)	0.0226(17)	0.0213(16)	-0.0011(14)	0.0092(14)	-0.0052(15)
C2	0.0224(16)	0.0198(17)	0.0187(15)	0.0028(13)	0.0064(13)	0.0073(13)
C26	0.0209(16)	0.0219(17)	0.0198(15)	0.0039(13)	0.0069(13)	0.0073(13)
C31	0.0243(17)	0.0210(17)	0.0254(17)	0.0048(14)	-0.0017(13)	-0.0100(13)
C9	0.0175(14)	0.0214(17)	0.0212(15)	0.0023(12)	0.0046(12)	0.0007(12)
C3	0.0263(17)	0.0151(15)	0.0234(16)	0.0018(13)	0.0099(14)	0.0032(13)
C30	0.0210(16)	0.0170(16)	0.0291(18)	0.0048(14)	0.0078(14)	-0.0028(13)
C14	0.0214(16)	0.0204(17)	0.0337(18)	-0.0039(14)	0.0058(14)	-0.0036(13)
C15	0.0196(16)	0.0214(17)	0.0365(18)	-0.0046(14)	0.0122(14)	-0.0032(13)
C33	0.0220(16)	0.0178(16)	0.0259(16)	0.0008(13)	0.0094(13)	-0.0042(12)
C35	0.0161(15)	0.0165(16)	0.0274(16)	-0.0004(13)	0.0075(13)	-0.0020(12)
C23	0.0274(18)	0.0275(19)	0.0278(17)	-0.0018(14)	0.0031(14)	0.0062(15)
C8	0.0232(15)	0.0215(16)	0.0193(14)	-0.0025(13)	0.0068(12)	-0.0067(14)
C36	0.0207(16)	0.0213(17)	0.0245(16)	0.0021(13)	0.0033(13)	-0.0004(13)
C13	0.0296(18)	0.0218(17)	0.0253(16)	-0.0038(13)	0.0087(14)	-0.0014(14)
C4	0.0203(15)	0.0145(16)	0.0210(15)	0.0004(12)	0.0104(12)	-0.0017(12)
C5	0.0132(14)	0.0163(16)	0.0204(15)	-0.0042(12)	0.0065(12)	-0.0037(12)
C27	0.0178(15)	0.0170(16)	0.0184(15)	0.0024(13)	0.0060(12)	-0.0005(12)
C45	0.0222(15)	0.0150(15)	0.0226(15)	-0.0049(13)	0.0108(12)	-0.0009(14)

Table 5. Hydrogen coordinates and isotropic displacement parameters ($Å^2$) for **SI 39**.

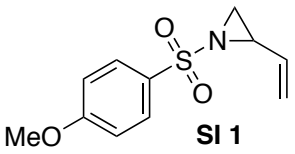
	x/a	y/b	z/c	U(eq)
H1	0.3014	0.1304	0.0681	0.02
H18	0.2610	-0.0039	-0.0912	0.024
H22	0.5143	0.1905	-0.1334	0.026
H41	-0.0234	0.4181	1.1377	0.023
H10	0.5191	0.0476	0.2727	0.022
H19	0.1376	0.0498	-0.2503	0.028
H39	0.2946	0.4955	0.7697	0.025
H38	0.2861	0.5164	0.5969	0.028
H42	0.1053	0.3714	1.2965	0.027
H44	0.3621	0.5414	1.2298	0.025
H16	0.7599	0.2329	0.0929	0.026
H37	0.1066	0.5024	0.4691	0.028
H24	0.1876	0.4991	0.9043	0.022
H21	0.3898	0.2448	-0.2900	0.029
H25	0.2510	0.3112	0.8894	0.025
H46A	0.3755	0.3548	1.3664	0.052
H46B	0.2868	0.3965	1.4269	0.052
H46C	0.3847	0.4739	1.4076	0.052
H6	0.1934	0.1696	0.2150	0.024
H29	-0.2418	0.3666	0.8827	0.024
H7	0.1813	0.1091	0.3739	0.025
H12	0.5916	0.2810	0.3093	0.028
H32	-0.2008	0.1785	0.5977	0.031
H2	0.2875	0.3159	0.1315	0.024
H26	0.1069	0.2039	0.9288	0.025
H9	0.5041	-0.0188	0.4289	0.024
H3	0.4602	0.4006	0.1195	0.025
H30	-0.4065	0.3232	0.7486	0.027
H14	0.9453	0.2700	0.3915	0.031
H15	0.9392	0.2389	0.2199	0.03
H33	-0.0359	0.2199	0.7337	0.026
H35	-0.0548	0.4476	0.6898	0.024
H23A	0.1012	0.1478	-0.4078	0.043
H23B	0.2098	0.1998	-0.4373	0.043
H23C	0.1401	0.2664	-0.3733	0.043

	x/a	y/b	z/c	U(eq)
H36	-0.0635	0.4684	0.5171	0.027
H13	0.7711	0.2922	0.4357	0.03
H4	0.5548	0.2600	0.0368	0.021
H27	-0.0322	0.3311	0.9735	0.021
H45	0.2354	0.5899	1.0721	0.023

Table 6. Torsion angles [°] for **SI 39**.

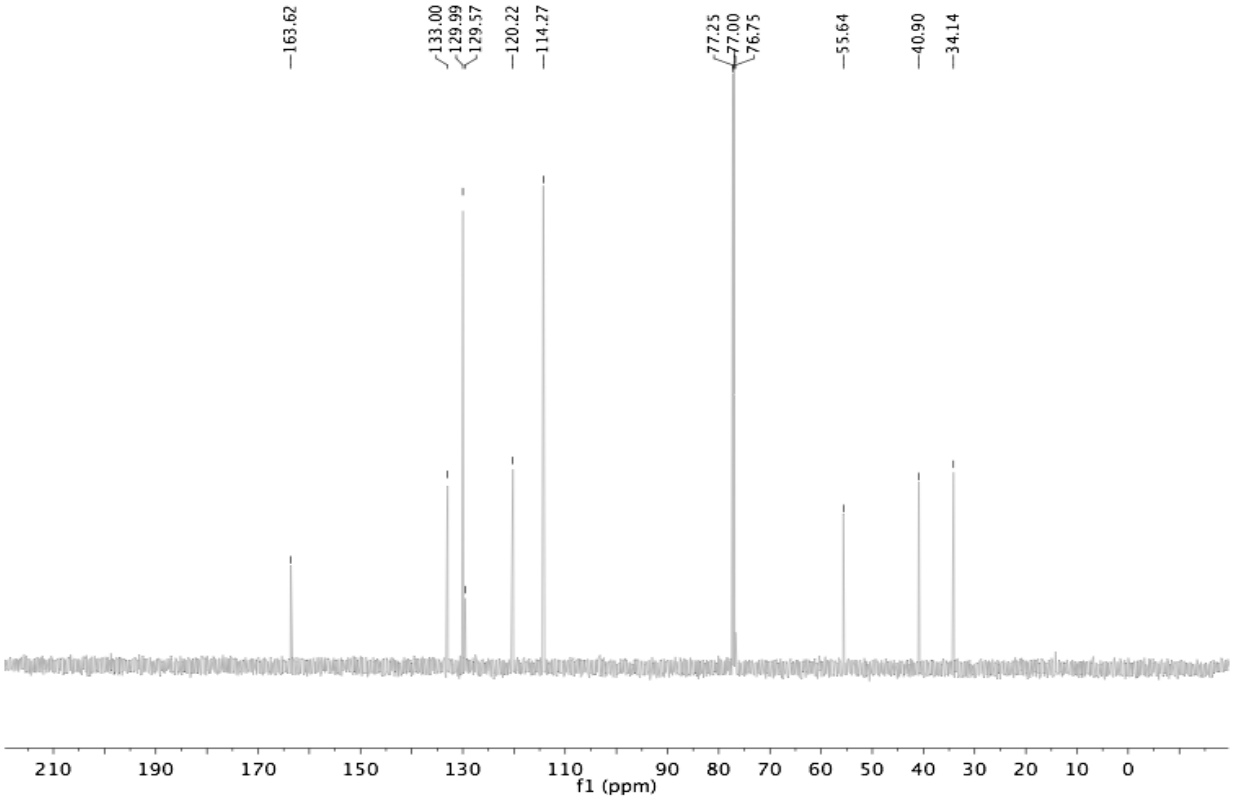
O3-S2-N2-C24	47.4(2)	O4-S2-N2-C24	176.81(19)
C40-S2-N2-C24	-68.7(2)	O3-S2-N2-C27	-179.30(19)
O4-S2-N2-C27	-49.9(2)	C40-S2-N2-C27	64.6(2)
O2-S1-N1-C1	-51.8(2)	O1-S1-N1-C1	179.01(19)
C17-S1-N1-C1	63.9(2)	O2-S1-N1-C4	169.19(19)
O1-S1-N1-C4	40.0(2)	C17-S1-N1-C4	-75.2(2)
O3-S2-C40-C41	153.3(2)	O4-S2-C40-C41	23.1(3)
N2-S2-C40-C41	-91.3(2)	O3-S2-C40-C45	-28.3(3)
O4-S2-C40-C45	-158.5(2)	N2-S2-C40-C45	87.1(3)
C4-N1-C1-C2	5.1(3)	S1-N1-C1-C2	-137.0(2)
C4-N1-C1-C5	-114.2(3)	S1-N1-C1-C5	103.7(2)
C45-C40-C41-C42	-1.6(4)	S2-C40-C41-C42	176.8(2)
C17-C18-C19-C20	-0.6(4)	C34-C39-C38-C37	0.3(5)
C40-C41-C42-C43	0.1(4)	C38-C39-C34-C35	-1.0(4)
C38-C39-C34-C24	175.7(3)	C18-C19-C20-C21	2.3(4)
C18-C19-C20-C23	-176.1(3)	C19-C18-C17-C22	-1.6(4)
C19-C18-C17-S1	177.3(2)	C21-C22-C17-C18	2.1(4)
C21-C22-C17-S1	-176.8(2)	O2-S1-C17-C18	17.2(3)
O1-S1-C17-C18	148.2(2)	N1-S1-C17-C18	-97.9(3)
O2-S1-C17-C22	-163.9(2)	O1-S1-C17-C22	-32.9(3)
N1-S1-C17-C22	81.0(3)	C45-C44-C43-C42	-0.8(4)
C45-C44-C43-C46	177.2(3)	C41-C42-C43-C44	1.1(4)
C41-C42-C43-C46	-177.0(3)	C12-C11-C16-C15	0.4(4)
C4-C11-C16-C15	-178.9(3)	C39-C38-C37-C36	0.3(5)
C27-N2-C24-C25	-11.9(3)	S2-N2-C24-C25	124.0(2)
C27-N2-C24-C34	106.6(3)	S2-N2-C24-C34	-117.6(2)
C35-C34-C24-N2	-15.3(4)	C39-C34-C24-N2	168.2(3)
C35-C34-C24-C25	98.1(3)	C39-C34-C24-C25	-78.4(3)
C17-C22-C21-C20	-0.4(4)	C19-C20-C21-C22	-1.8(4)
C23-C20-C21-C22	176.6(3)	N2-C24-C25-C26	8.2(3)
C34-C24-C25-C26	-112.7(3)	C5-C6-C7-C8	-0.9(4)
C30-C29-C28-C33	-1.5(4)	C30-C29-C28-C27	175.8(3)
C16-C11-C12-C13	0.7(4)	C4-C11-C12-C13	180.0(3)
N1-C1-C2-C3	-4.7(3)	C5-C1-C2-C3	116.3(3)
C24-C25-C26-C27	-1.4(3)	C33-C32-C31-C30	-1.1(4)
C33-C32-C31-Cl2	179.7(2)	C5-C10-C9-C8	-1.8(4)
C1-C2-C3-C4	2.6(4)	C32-C31-C30-C29	1.7(4)

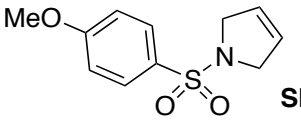
Cl2-C31-C30-C29	-179.2(2)	C28-C29-C30-C31	-0.3(4)
C13-C14-C15-C16	0.6(5)	C11-C16-C15-C14	-1.0(5)
C29-C28-C33-C32	2.0(4)	C27-C28-C33-C32	-175.2(3)
C31-C32-C33-C28	-0.8(4)	C39-C34-C35-C36	1.0(4)
C24-C34-C35-C36	-175.5(3)	C6-C7-C8-C9	-0.3(4)
C6-C7-C8-Cl1	-179.3(2)	C10-C9-C8-C7	1.6(4)
C10-C9-C8-Cl1	-179.4(2)	C34-C35-C36-C37	-0.4(4)
C38-C37-C36-C35	-0.3(5)	C15-C14-C13-C12	0.4(5)
C11-C12-C13-C14	-1.1(5)	C2-C3-C4-N1	0.7(3)
C2-C3-C4-C11	-120.9(3)	C1-N1-C4-C3	-3.8(3)
S1-N1-C4-C3	137.5(2)	C1-N1-C4-C11	119.6(2)
S1-N1-C4-C11	-99.1(2)	C12-C11-C4-C3	39.1(4)
C16-C11-C4-C3	-141.6(3)	C12-C11-C4-N1	-76.1(3)
C16-C11-C4-N1	103.2(3)	C9-C10-C5-C6	0.5(4)
C9-C10-C5-C1	178.4(3)	C7-C6-C5-C10	0.8(4)
C7-C6-C5-C1	-177.1(3)	N1-C1-C5-C10	-3.5(4)
C2-C1-C5-C10	-117.4(3)	N1-C1-C5-C6	174.4(2)
C2-C1-C5-C6	60.5(3)	C25-C26-C27-N2	-6.0(3)
C25-C26-C27-C28	113.4(3)	C24-N2-C27-C26	11.2(3)
S2-N2-C27-C26	-125.3(2)	C24-N2-C27-C28	-111.7(3)
S2-N2-C27-C28	111.8(2)	C33-C28-C27-C26	-19.1(4)
C29-C28-C27-C26	163.7(3)	C33-C28-C27-N2	95.2(3)
C29-C28-C27-N2	-82.1(3)	C43-C44-C45-C40	-0.6(4)
C41-C40-C45-C44	1.8(4)	S2-C40-C45-C44	-176.5(2)


O
N
S
O
MeO

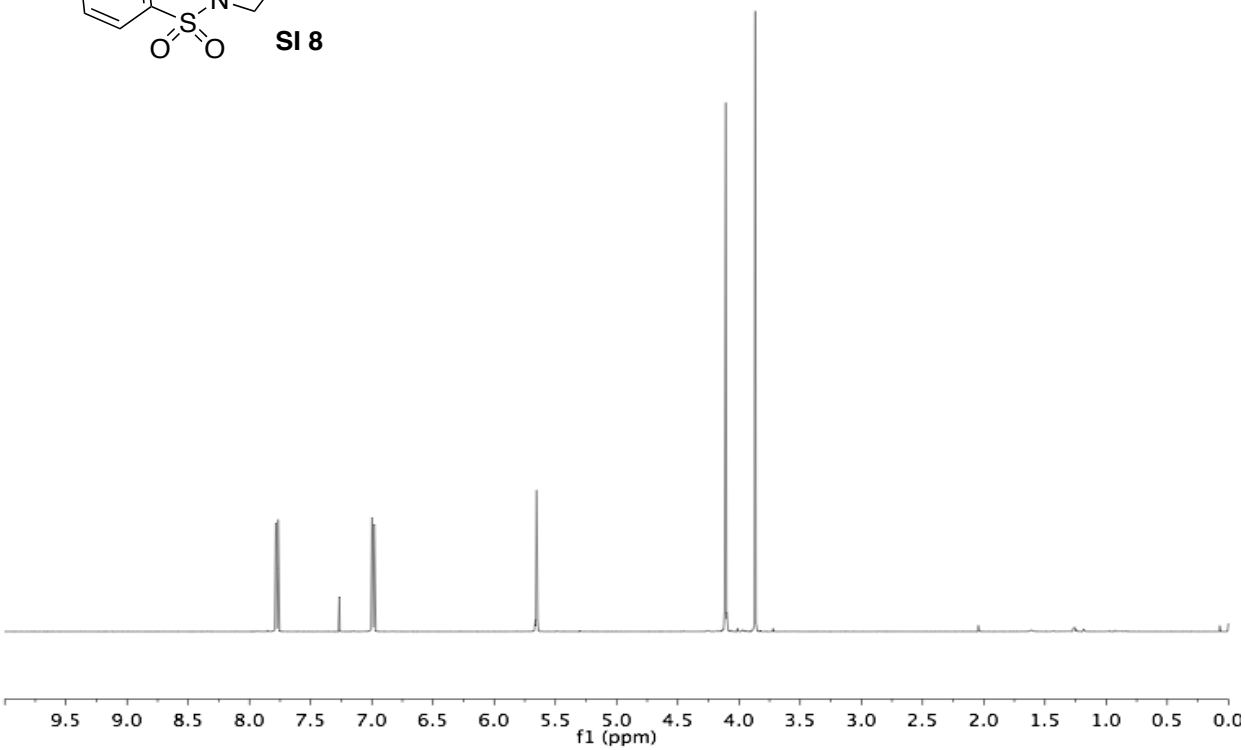
SI 1

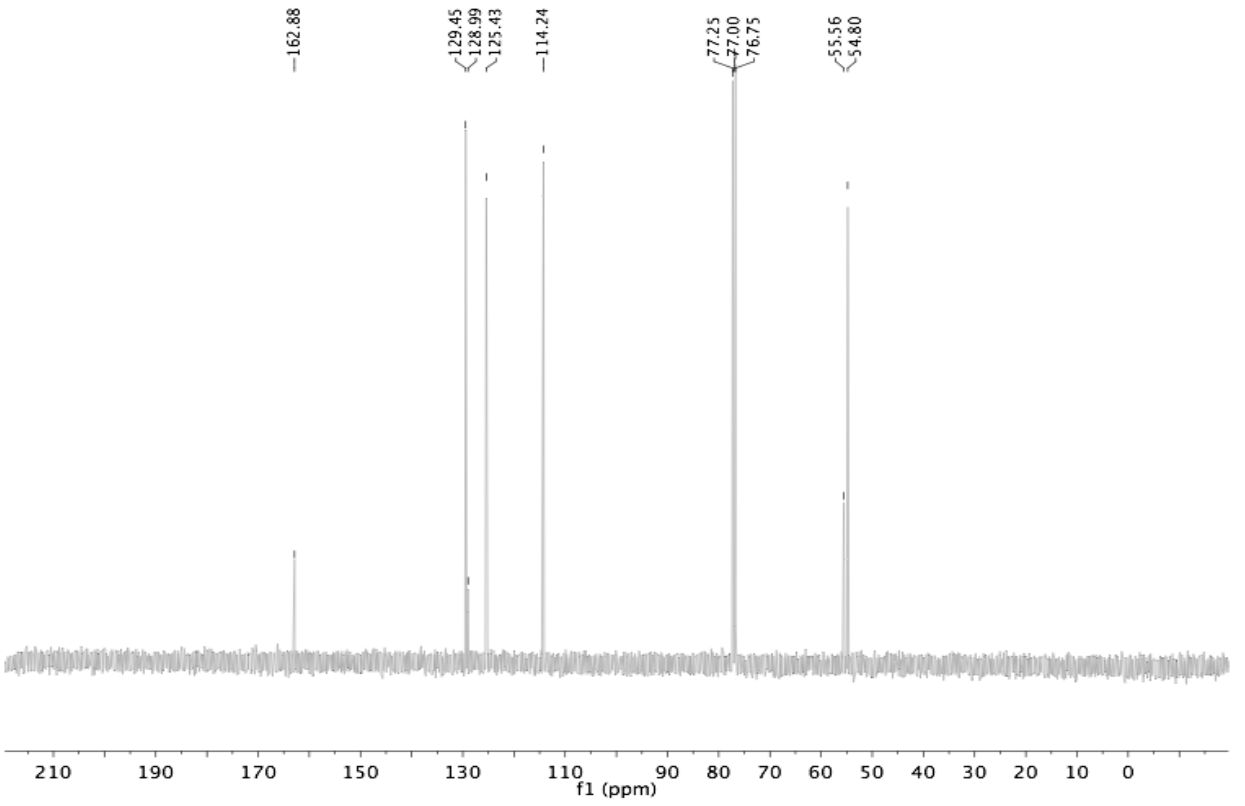
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

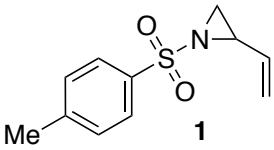

163.62
133.00
129.99
129.57
120.22
114.27
77.25
77.00
76.75
55.64
40.90
34.14
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


MeO
N
S
O
O

SI 8

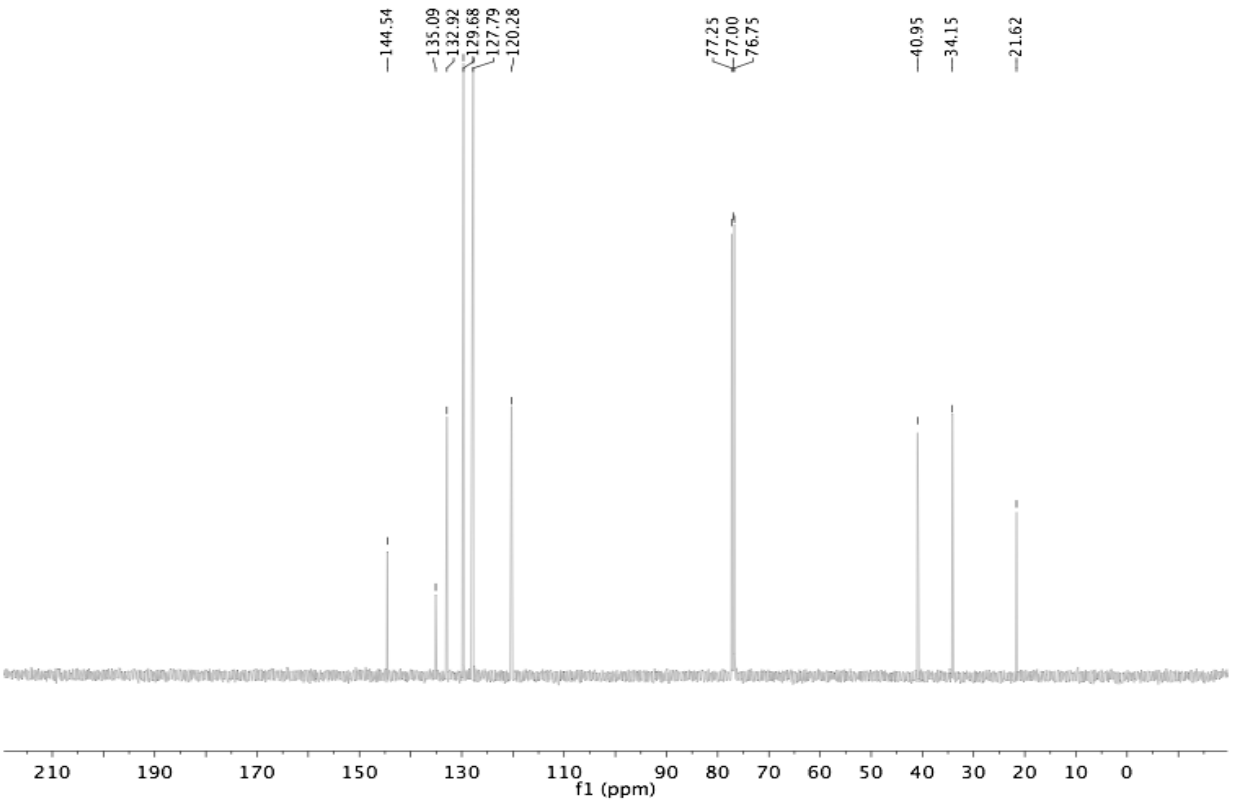

9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

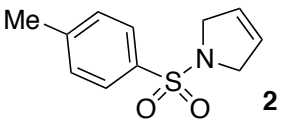

162.88
129.45
128.99
125.43
114.24
77.25
77.00
76.75
55.56
54.80
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
S
N
O
Me

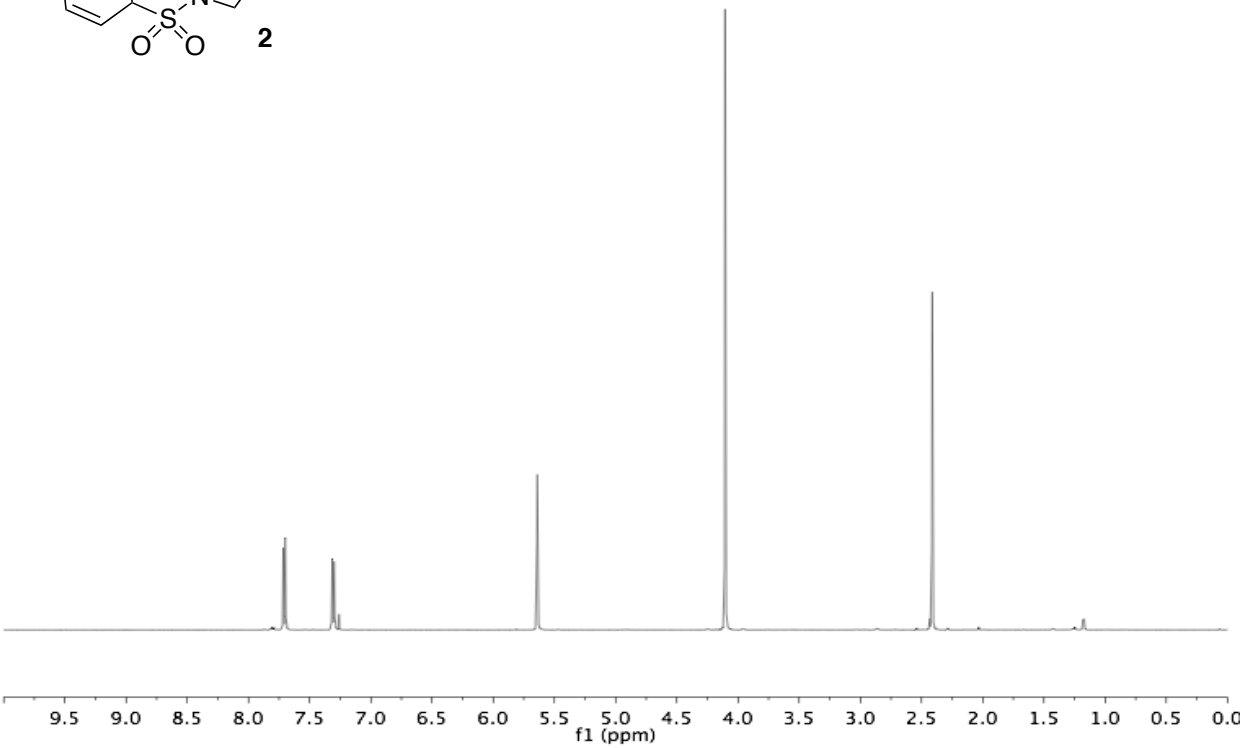
1

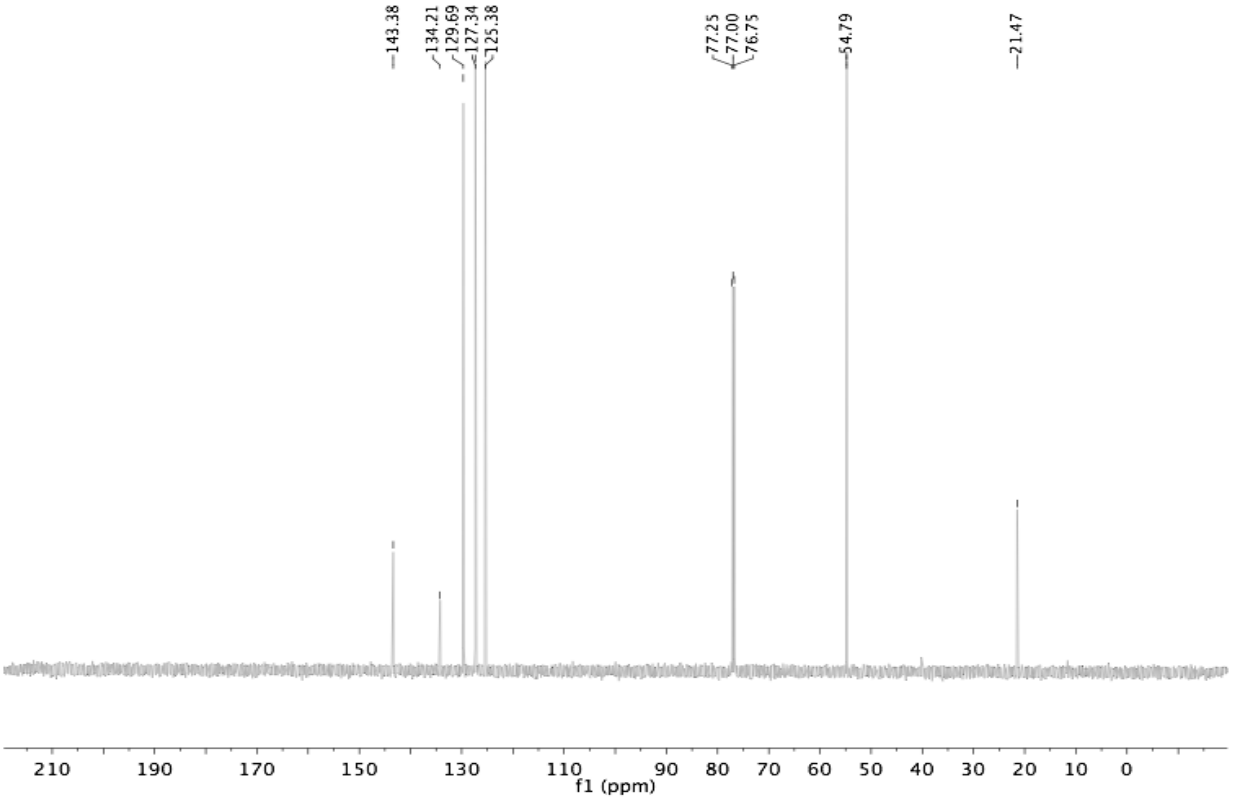
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

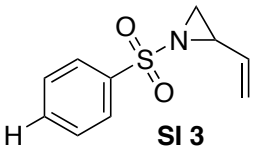

144.54
135.09
132.92
129.68
127.79
120.28
77.25
77.00
76.75
40.95
34.15
21.62
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


Me
N
S
O
O

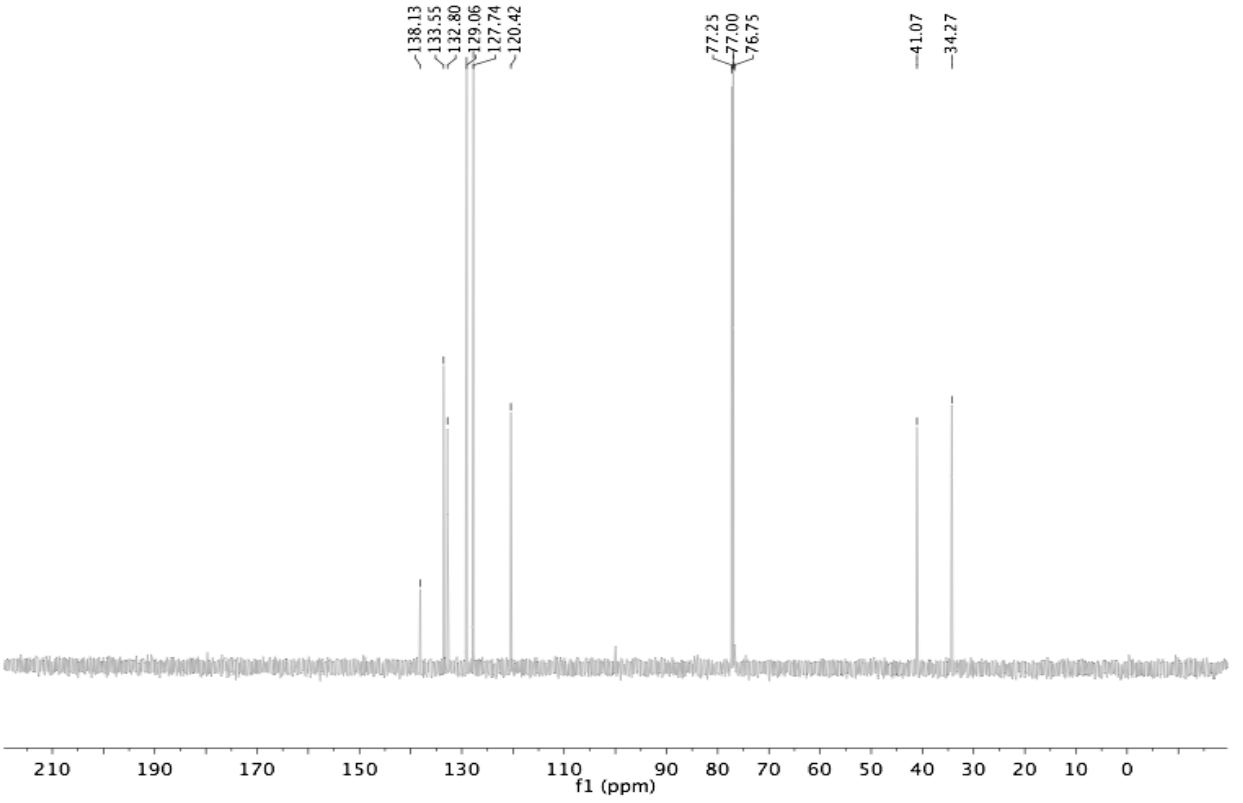
2

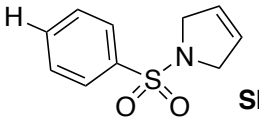

9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


143.38
134.21
129.69
127.34
125.38
77.25
77.00
76.75
54.79
21.47
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

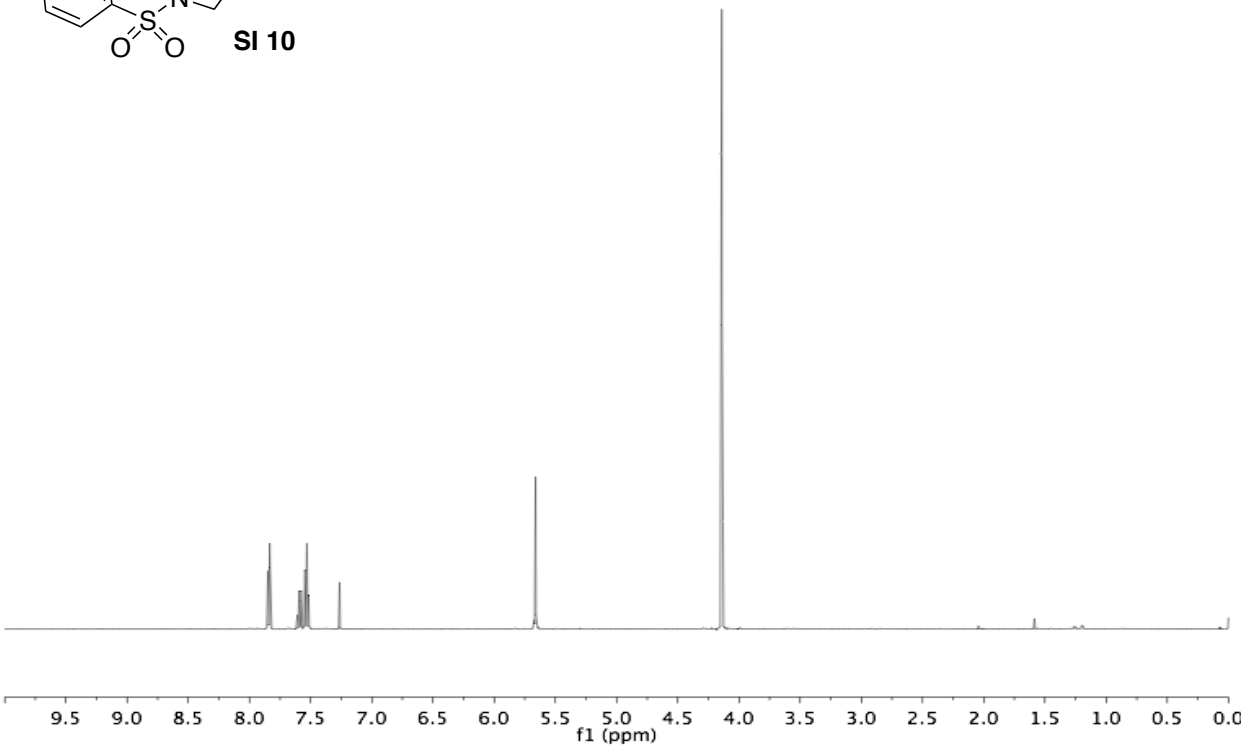


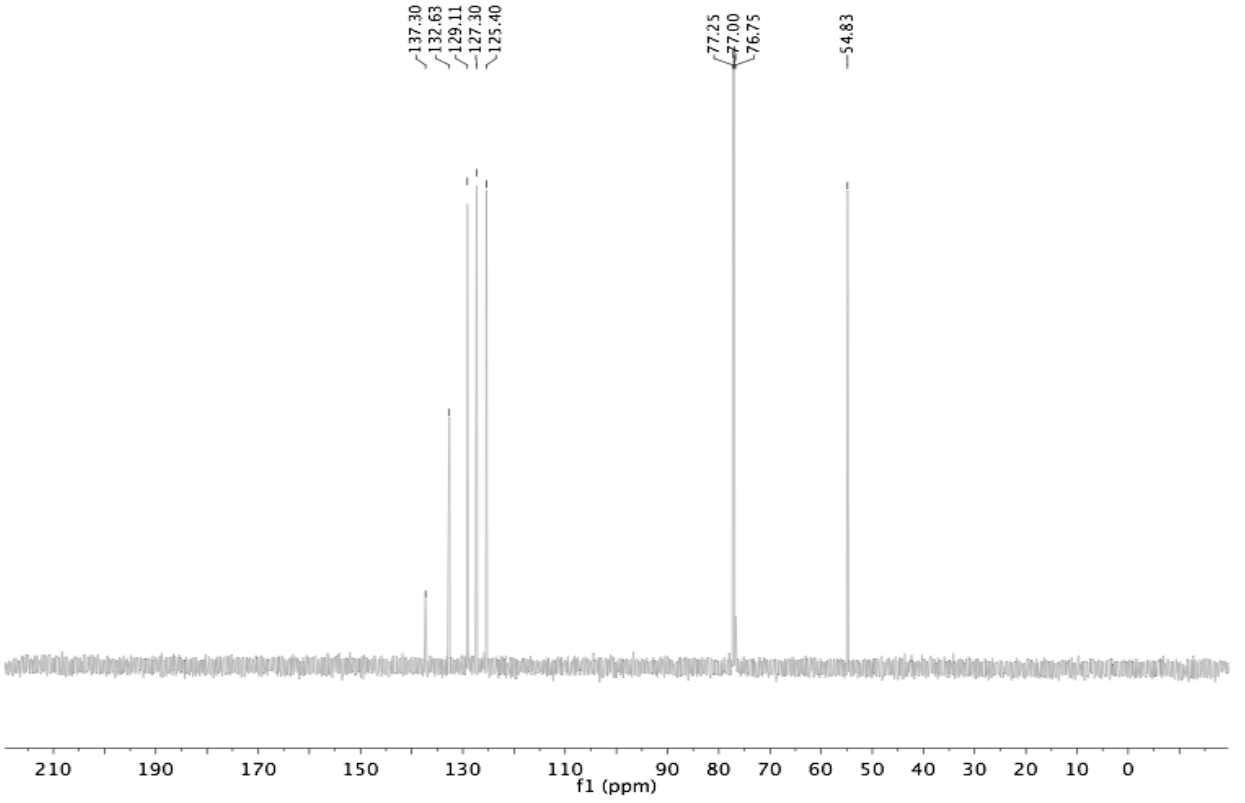
SI 3

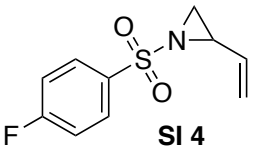




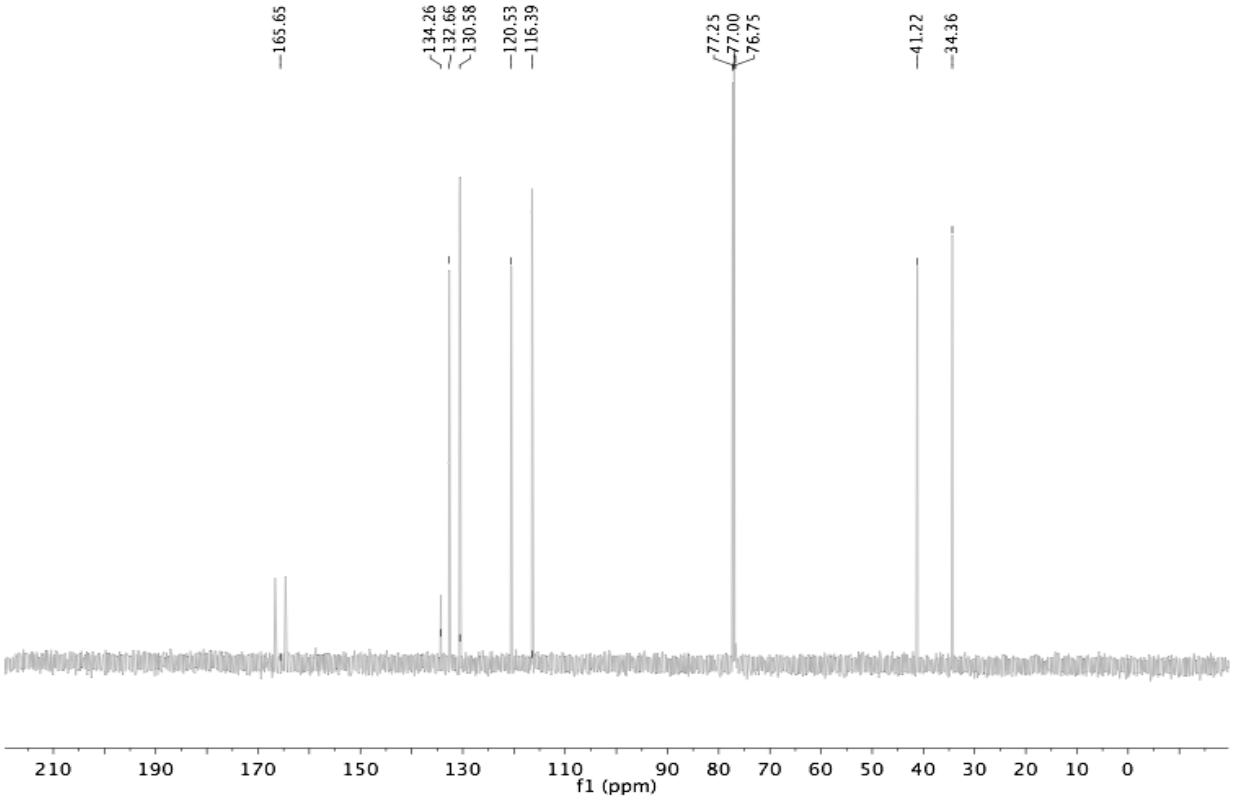
SI 10

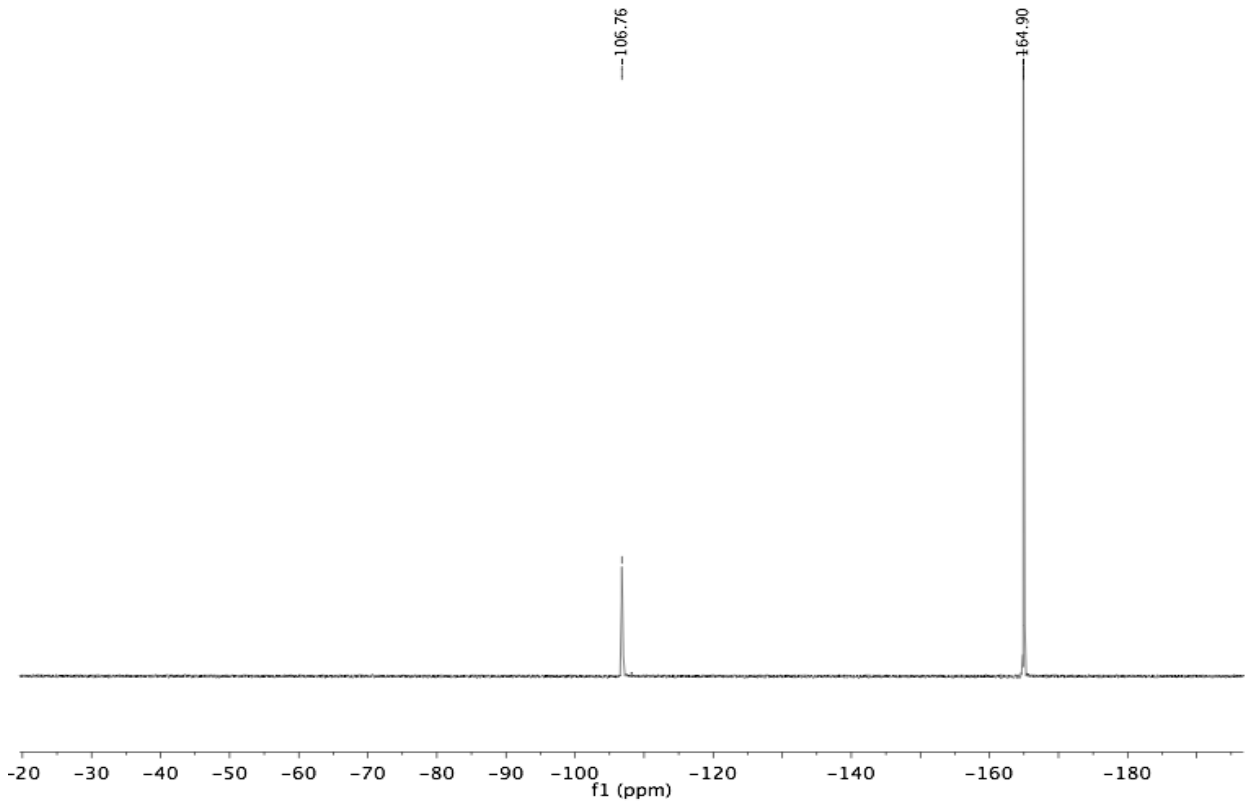


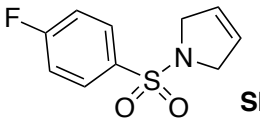




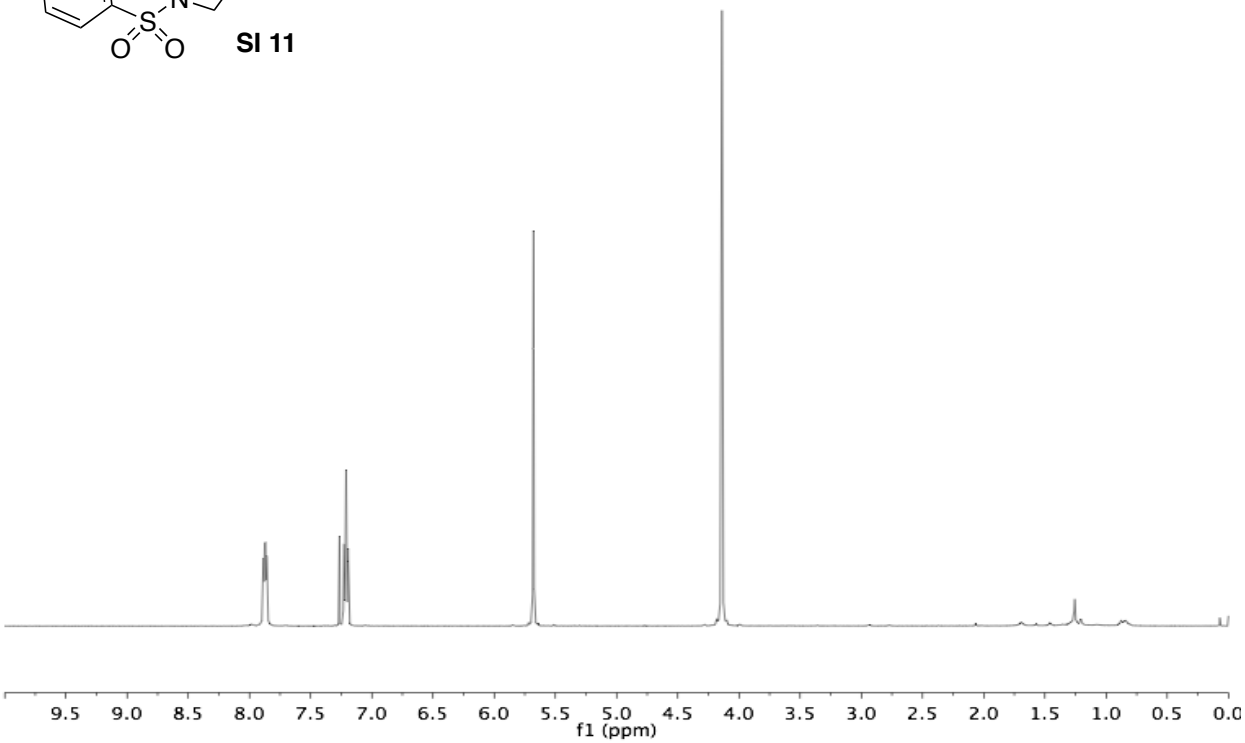
SI 4

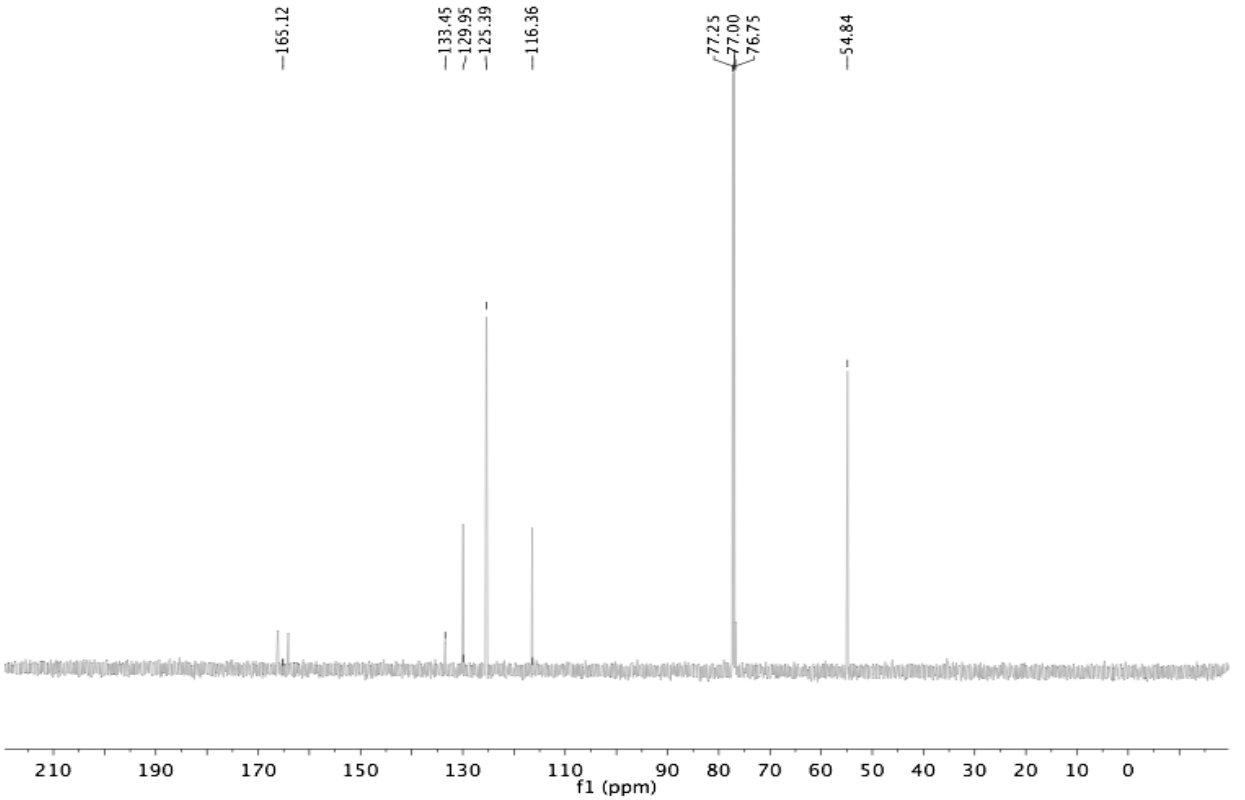


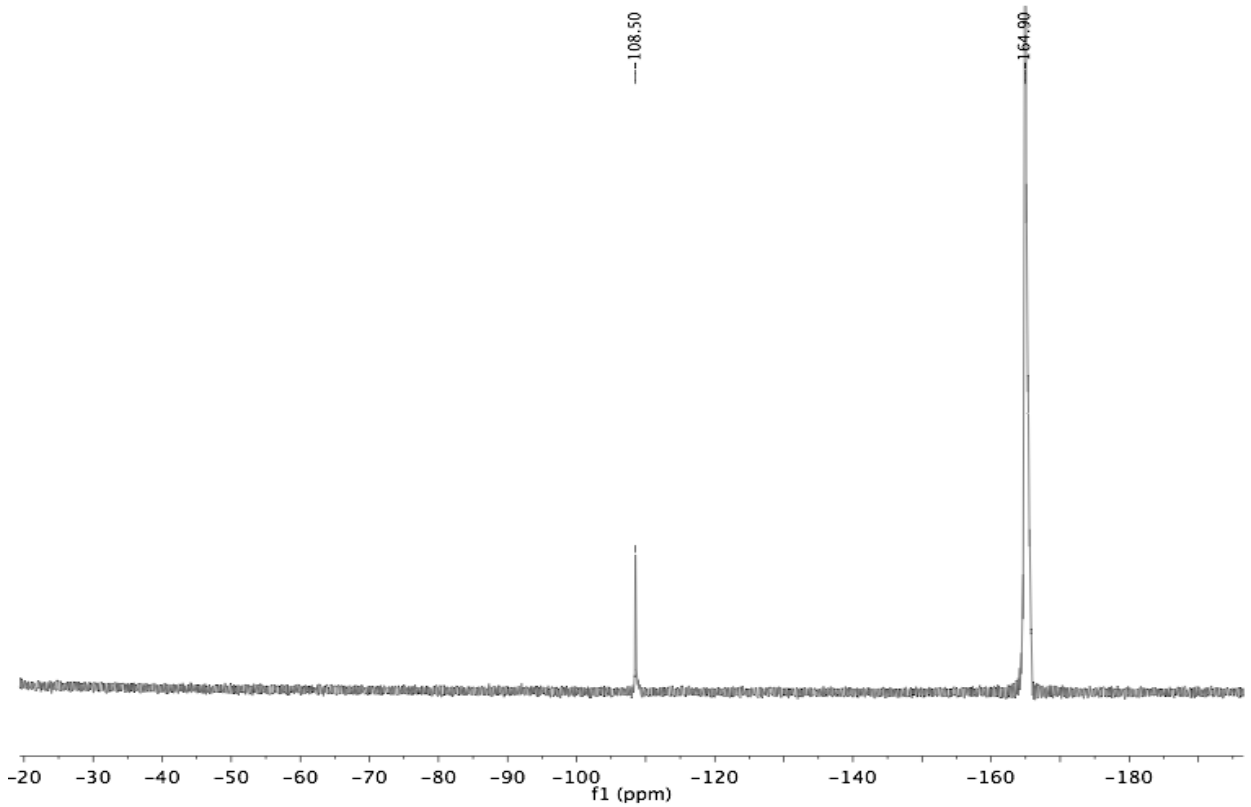



F
N
S
O
O

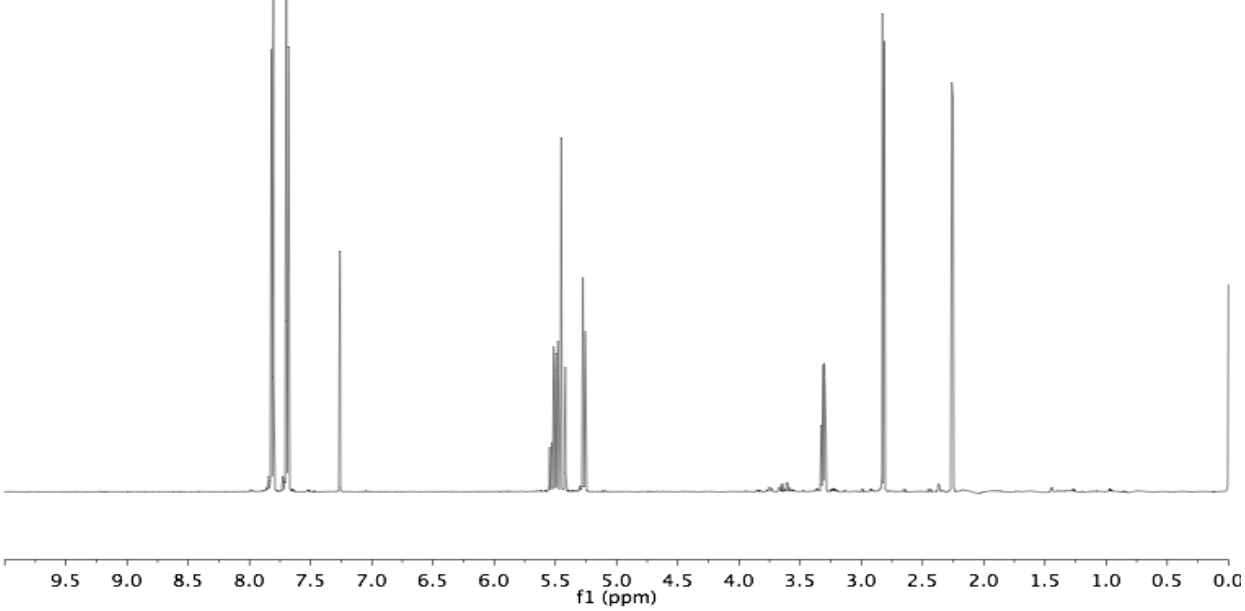
SI 11

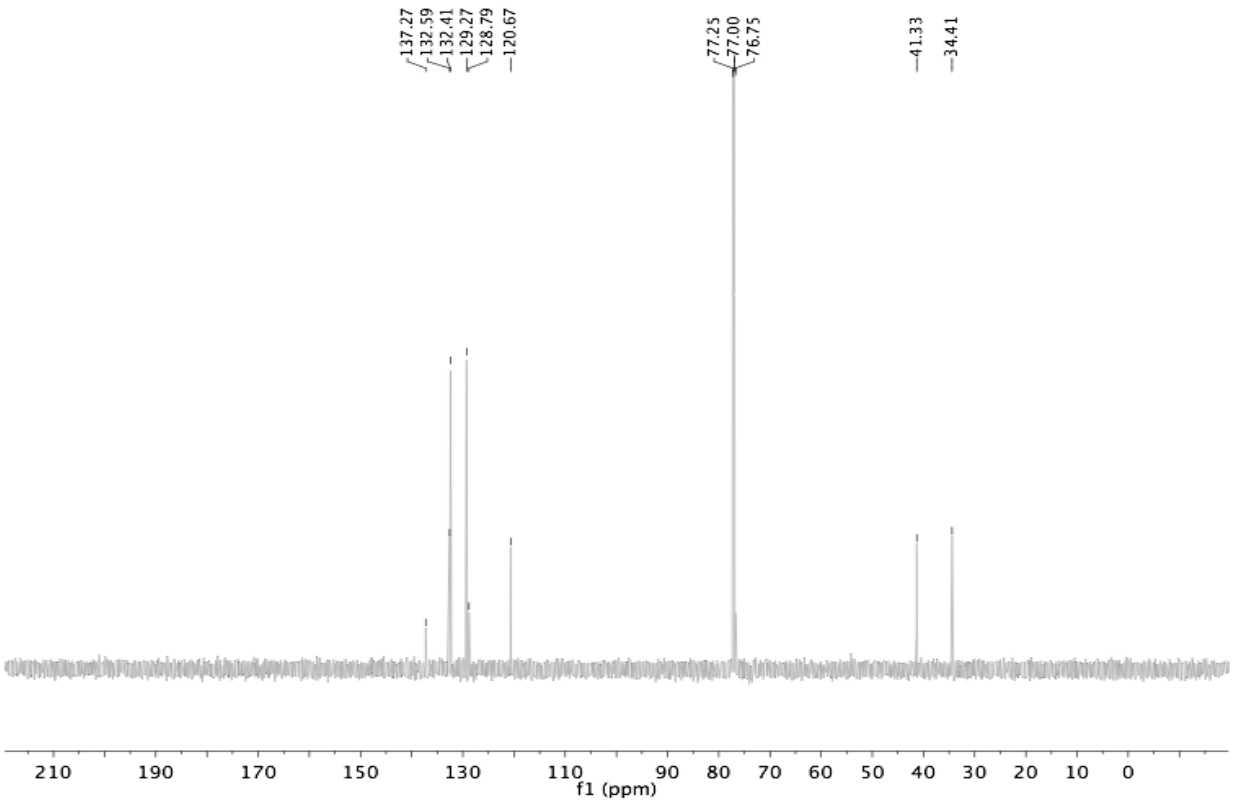

9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

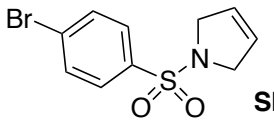

165.12
133.45
129.95
125.39
116.36
77.25
77.00
76.75
54.84
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



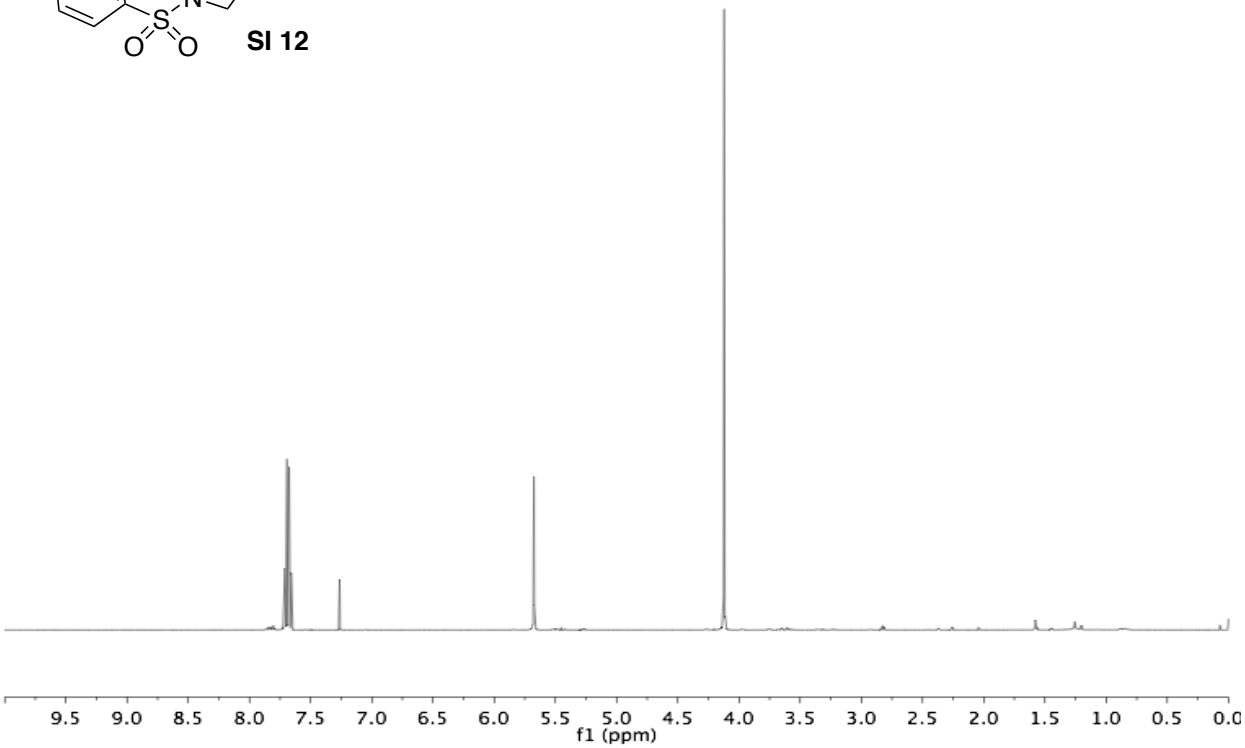
SI 5

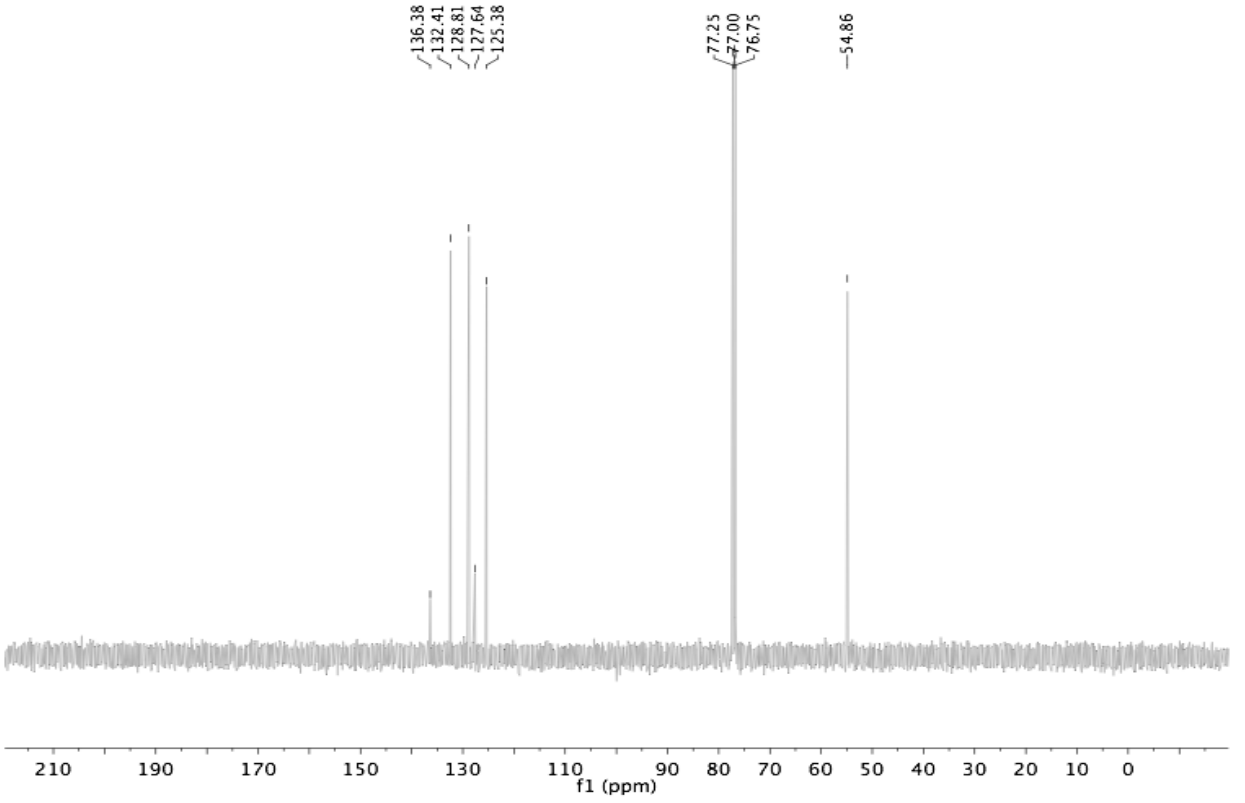


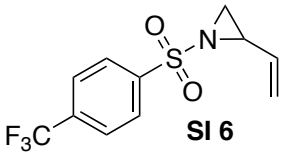



Br
N
S
O
O

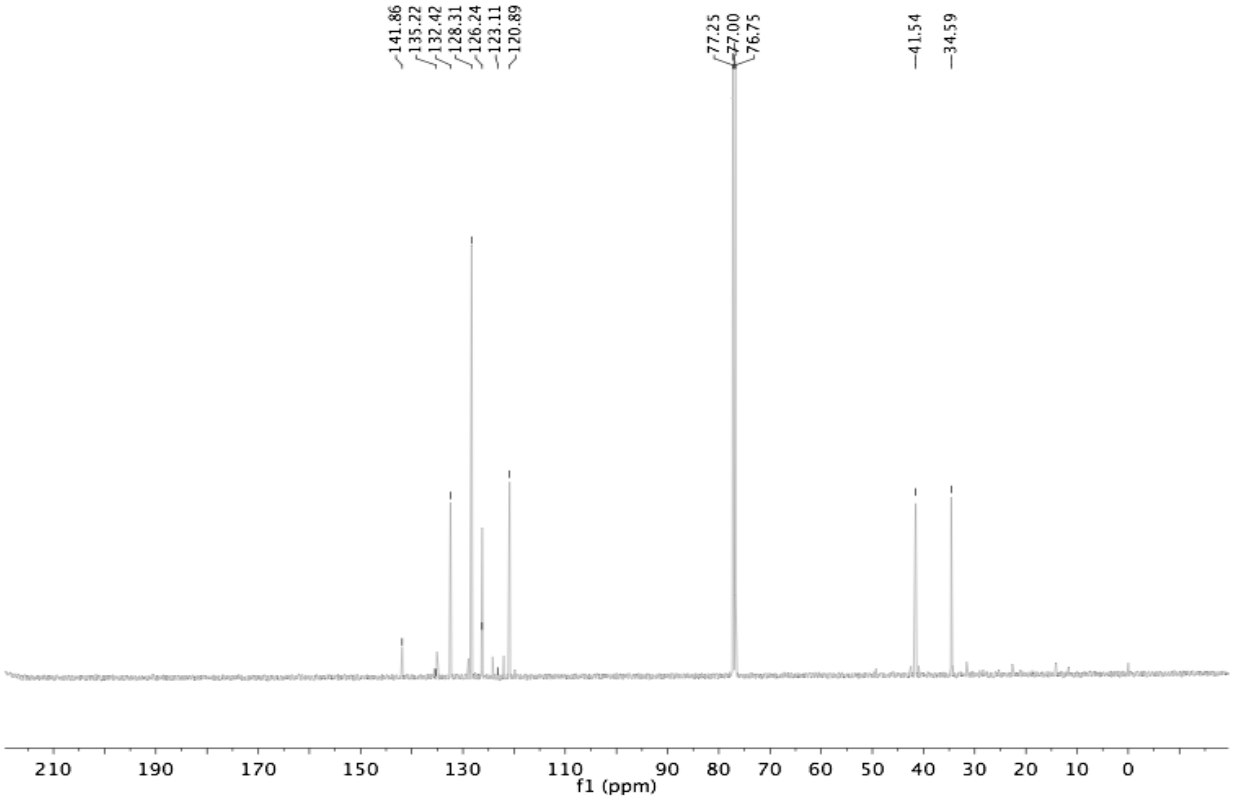
SI 12

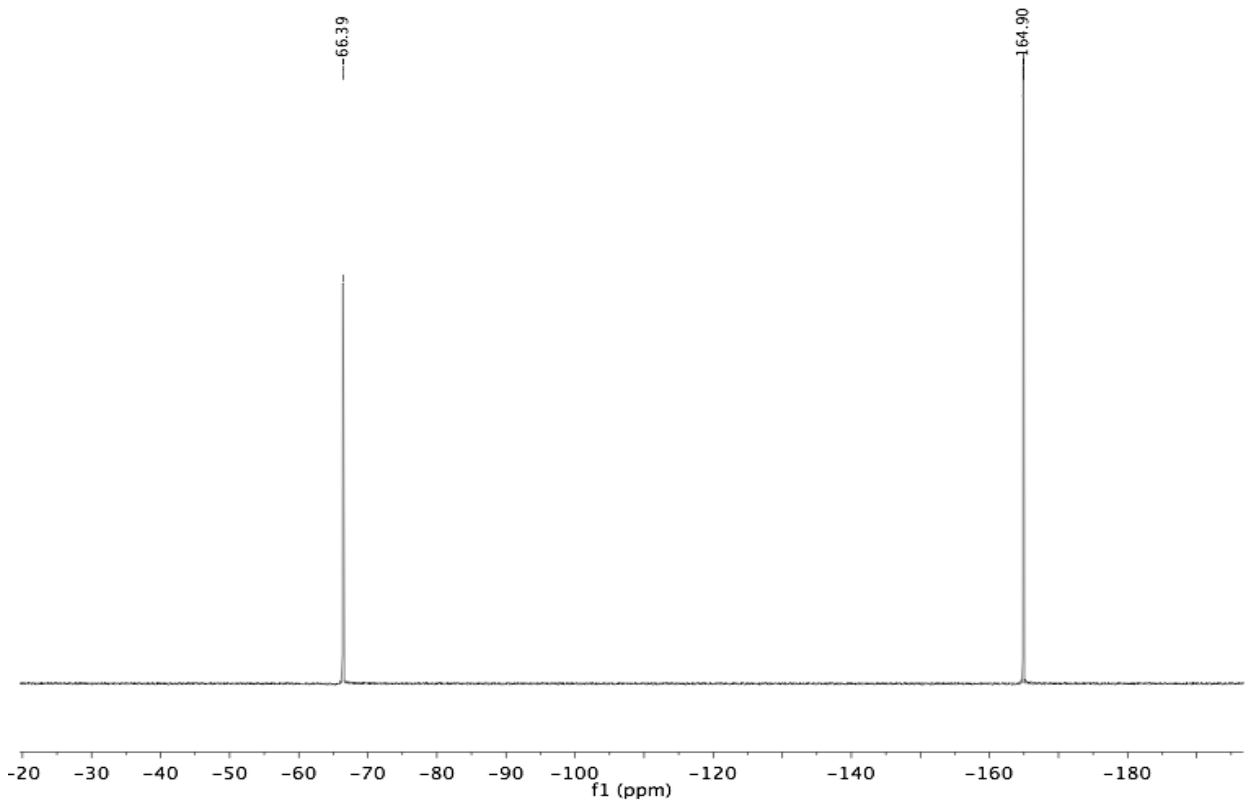

9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

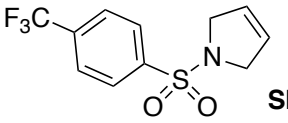

136.38
132.41
128.81
127.64
125.38
77.25
77.00
76.75
54.86
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



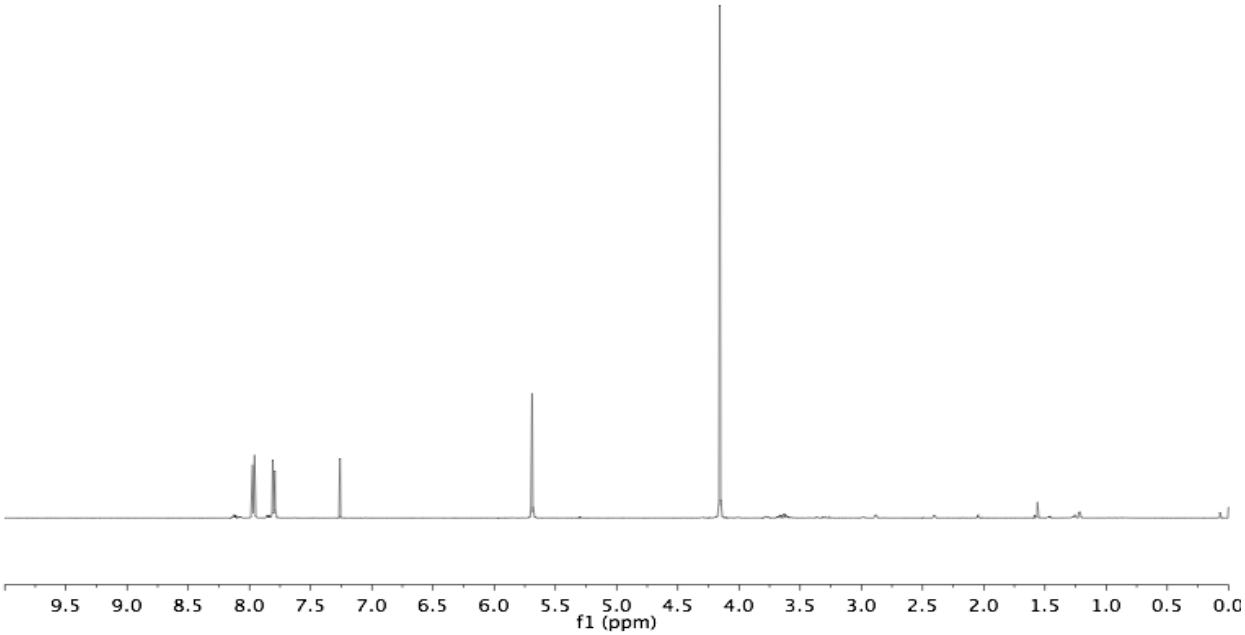
SI 6

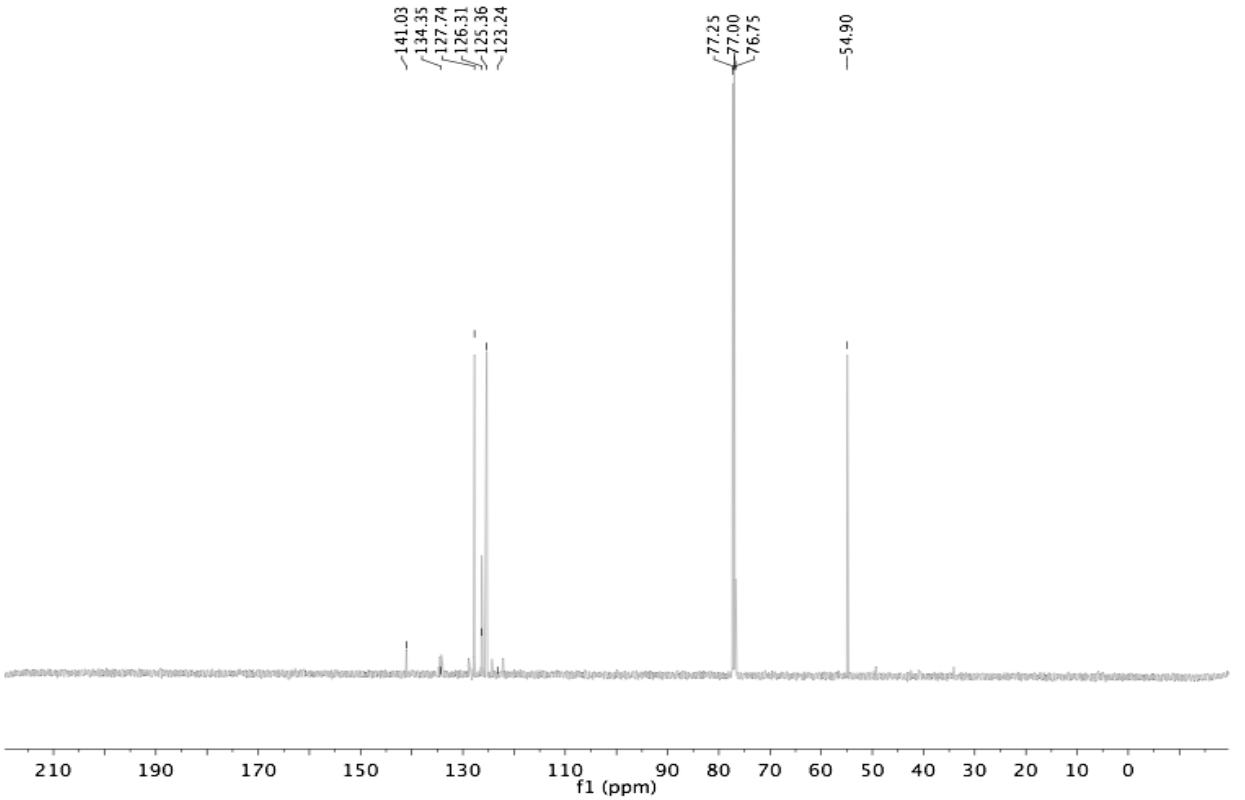


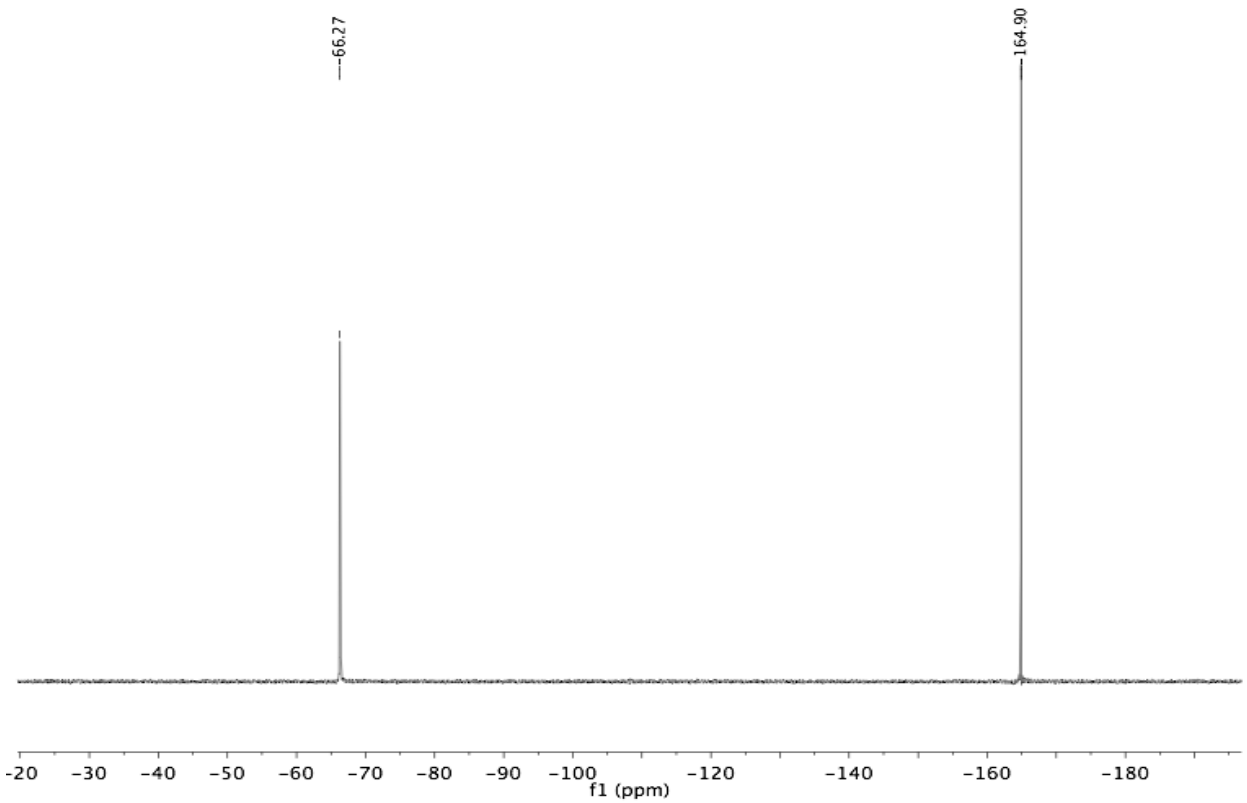


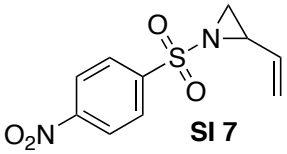


SI 13


9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

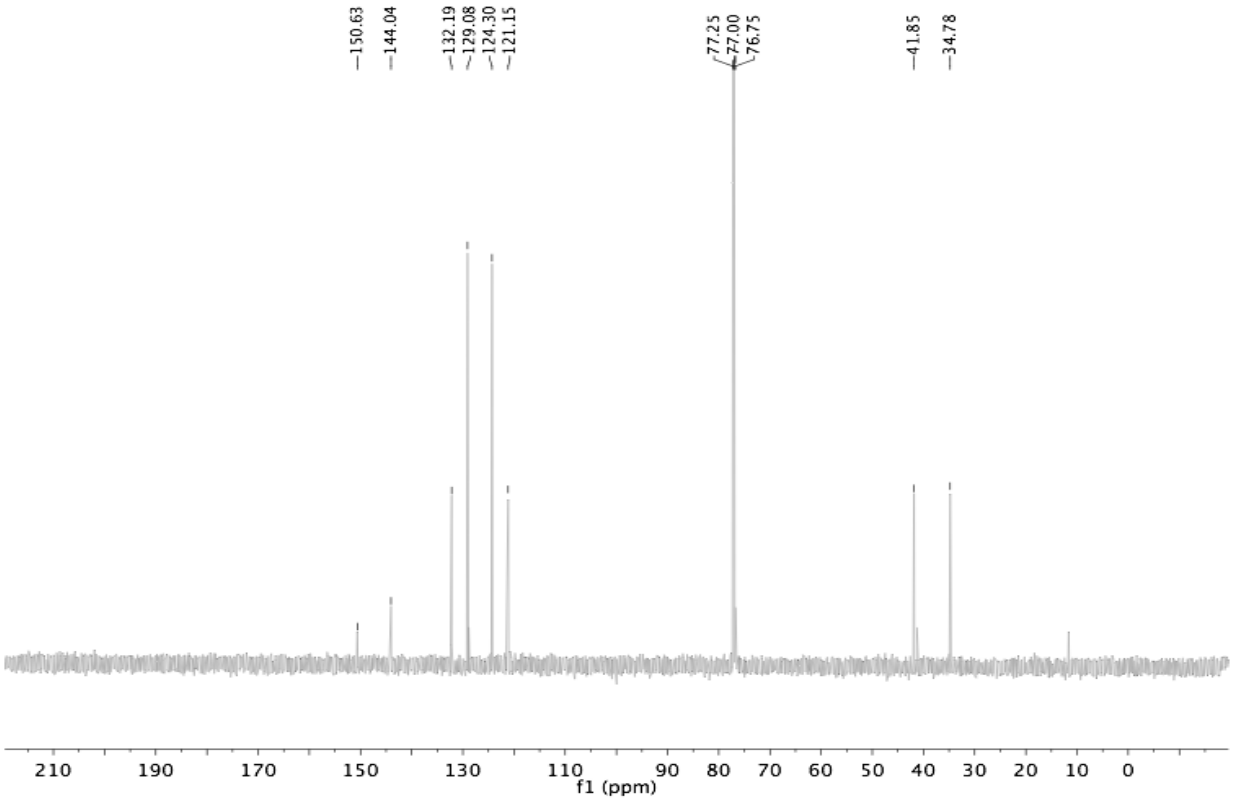

141.03
134.35
127.74
126.31
125.36
123.24
77.25
77.00
76.75
54.90
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

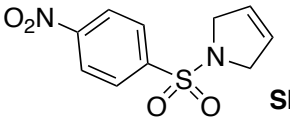



O
N
S
O
O2N

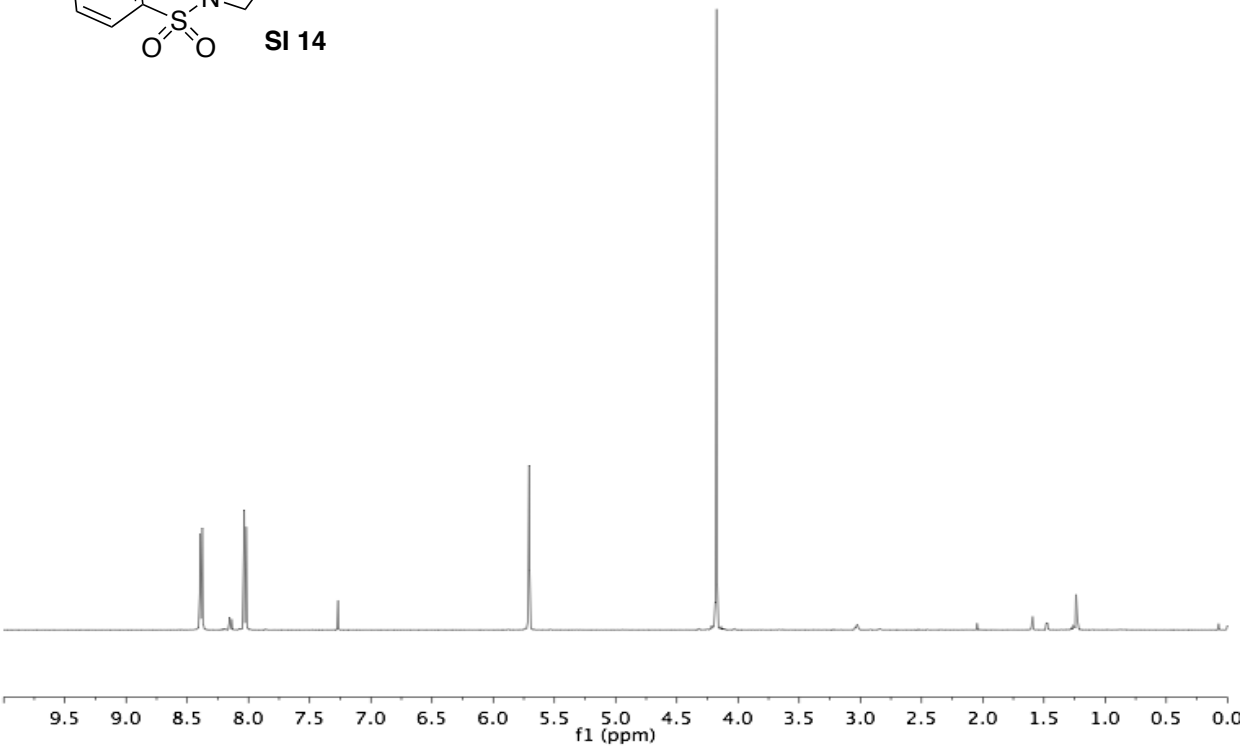
SI 7

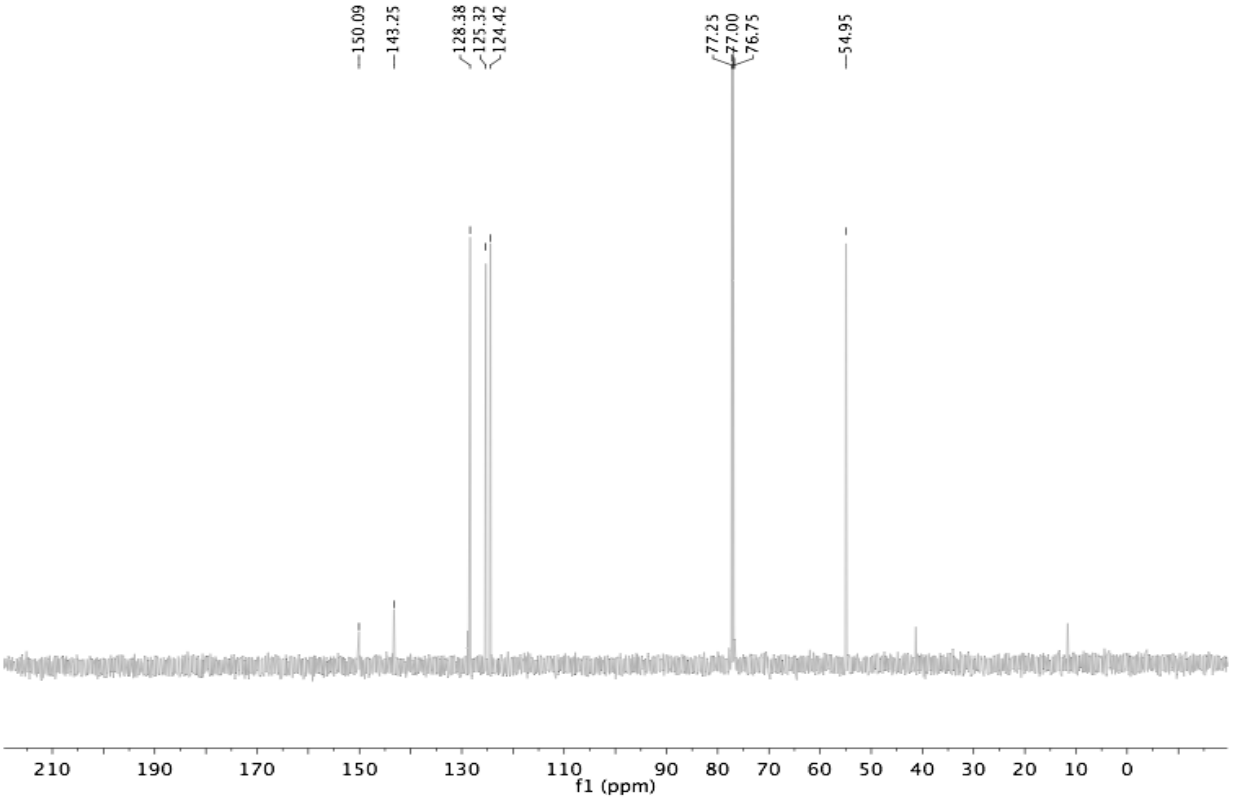
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

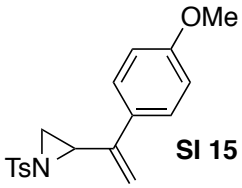

150.63
144.04
132.19
129.08
124.30
121.15
77.25
77.00
76.75
41.85
34.78
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



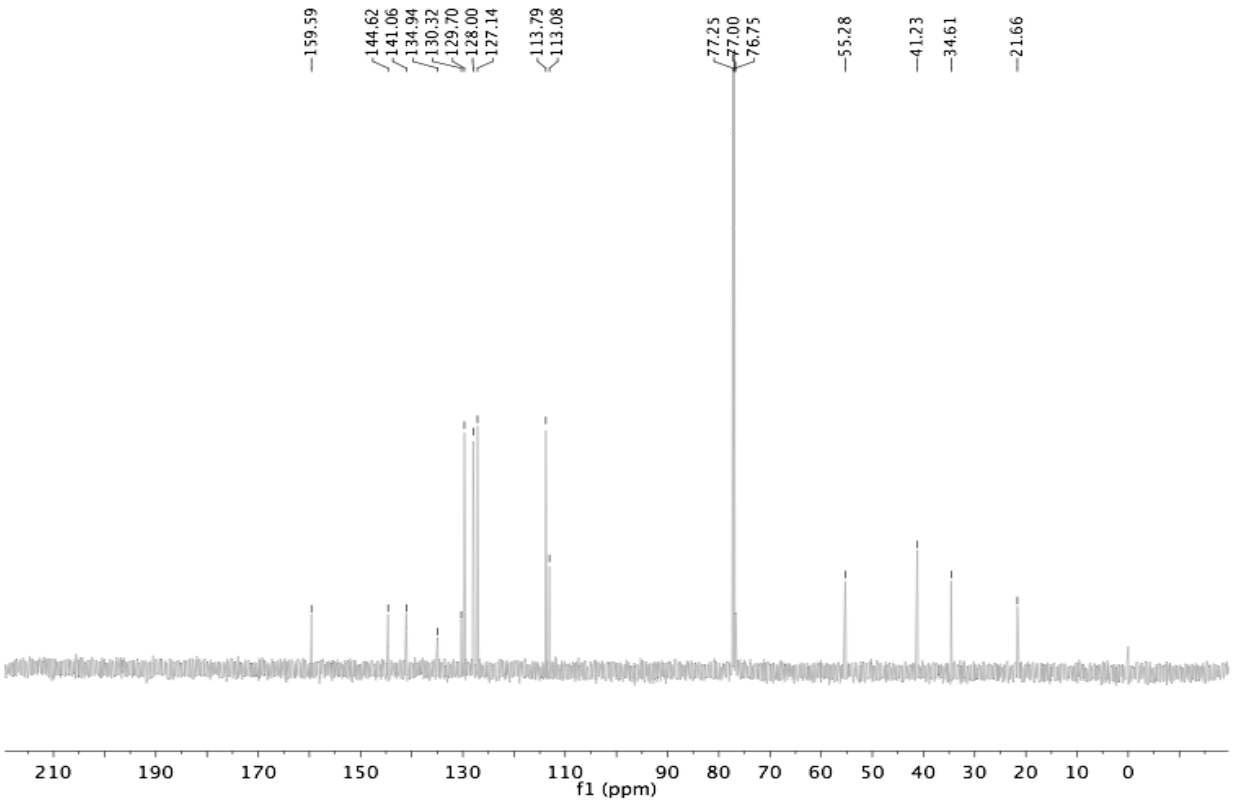
SI 14

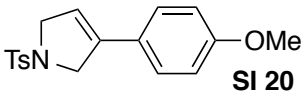




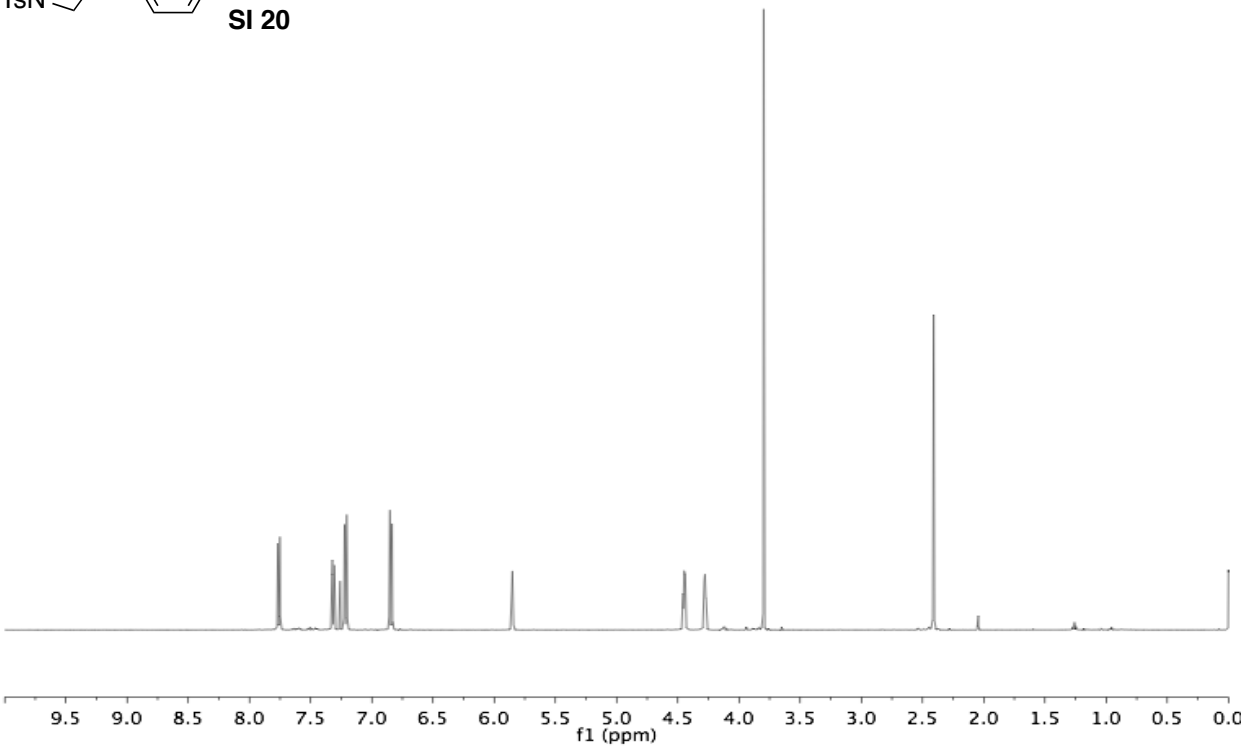


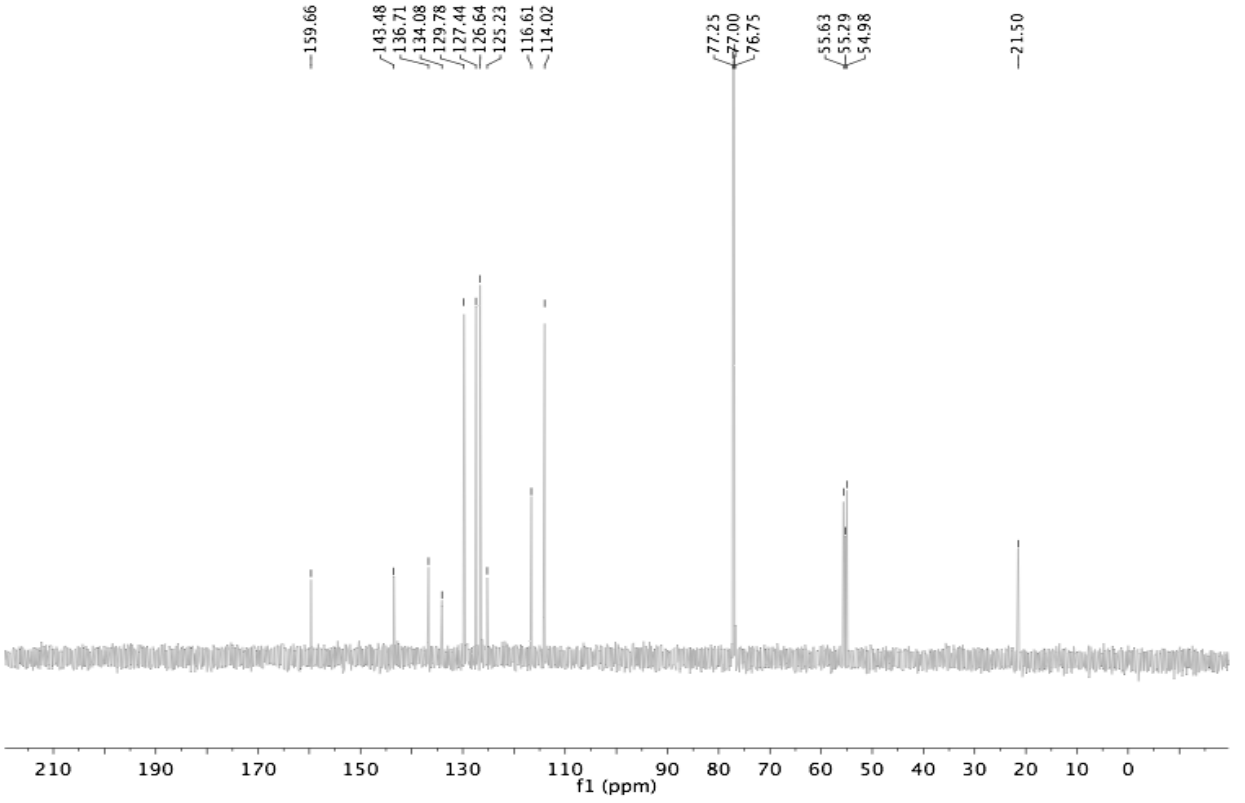
SI 15

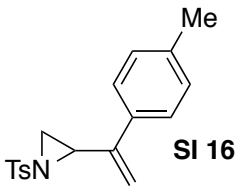



TsN
OMe

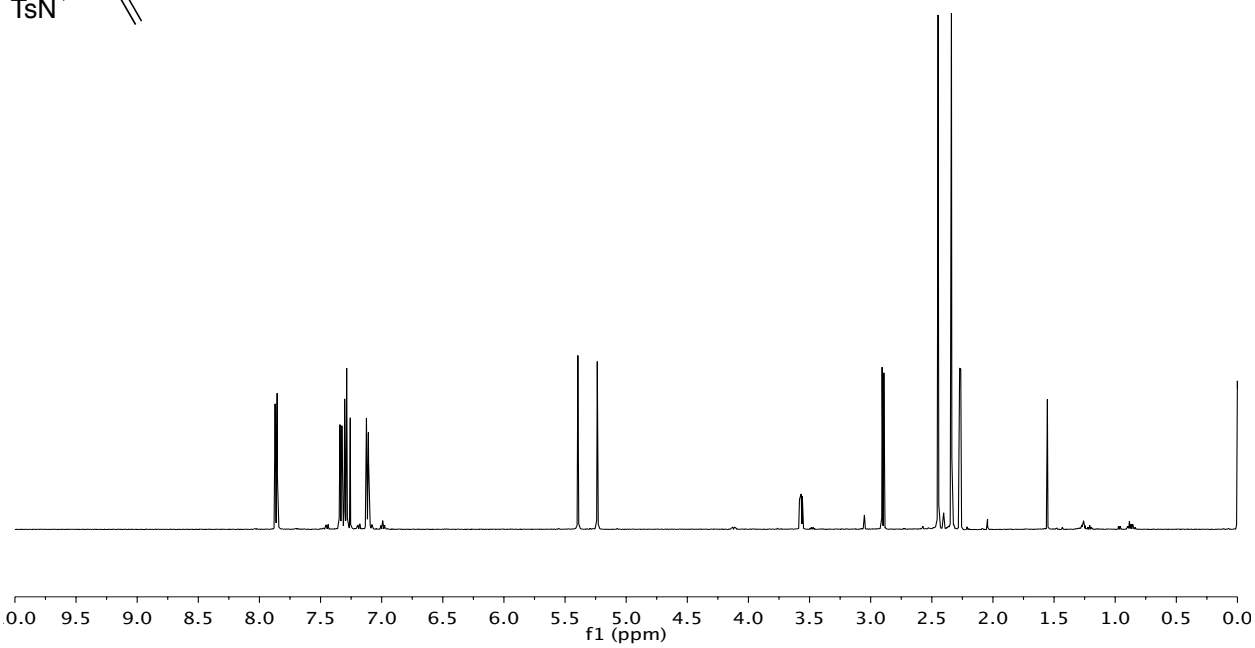
SI 20

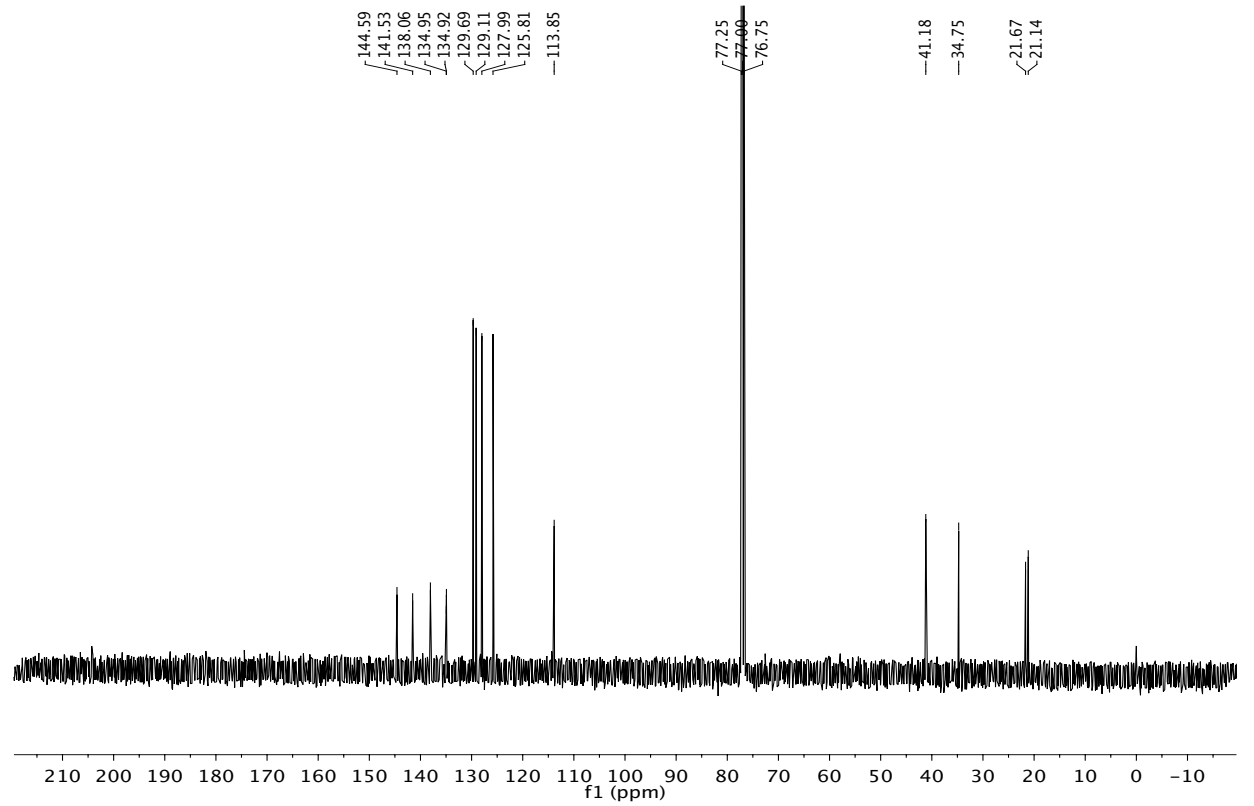

9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

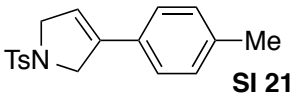

159.66
143.48
136.71
134.08
129.78
127.44
126.64
125.23
116.61
114.02
77.25
77.00
76.75
55.63
55.29
54.98
21.50
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



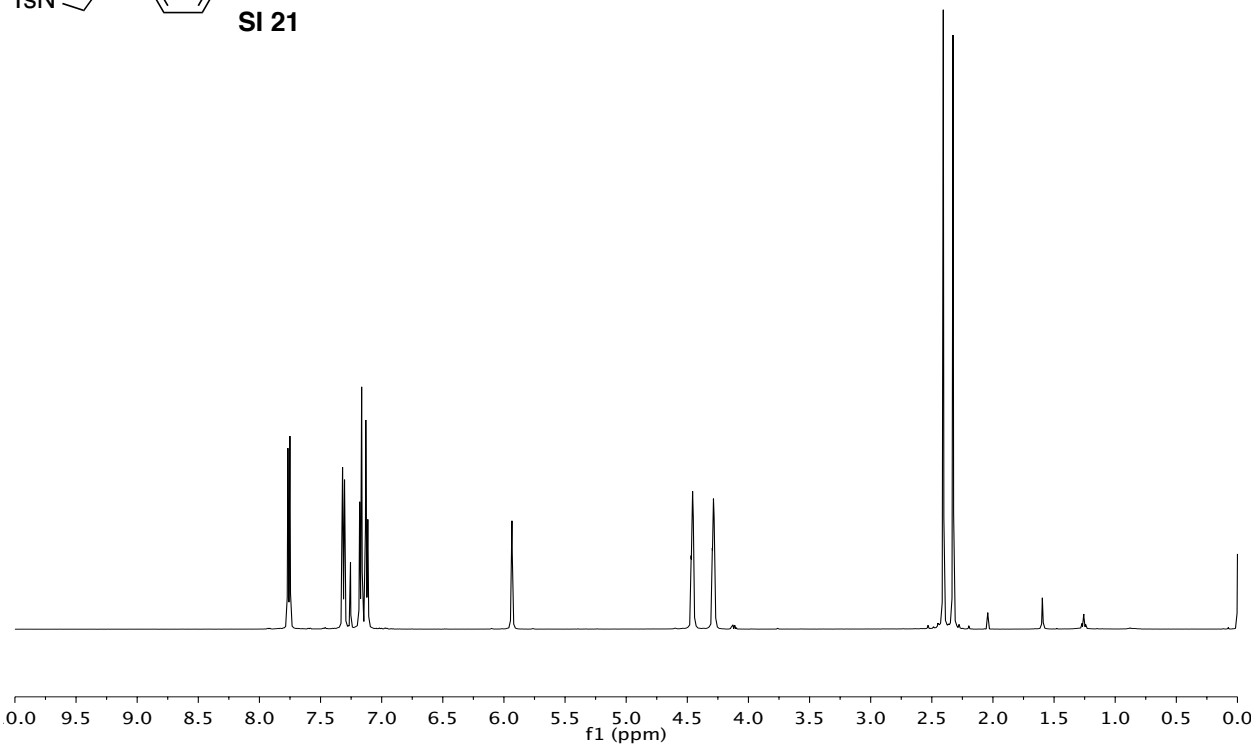
SI 16

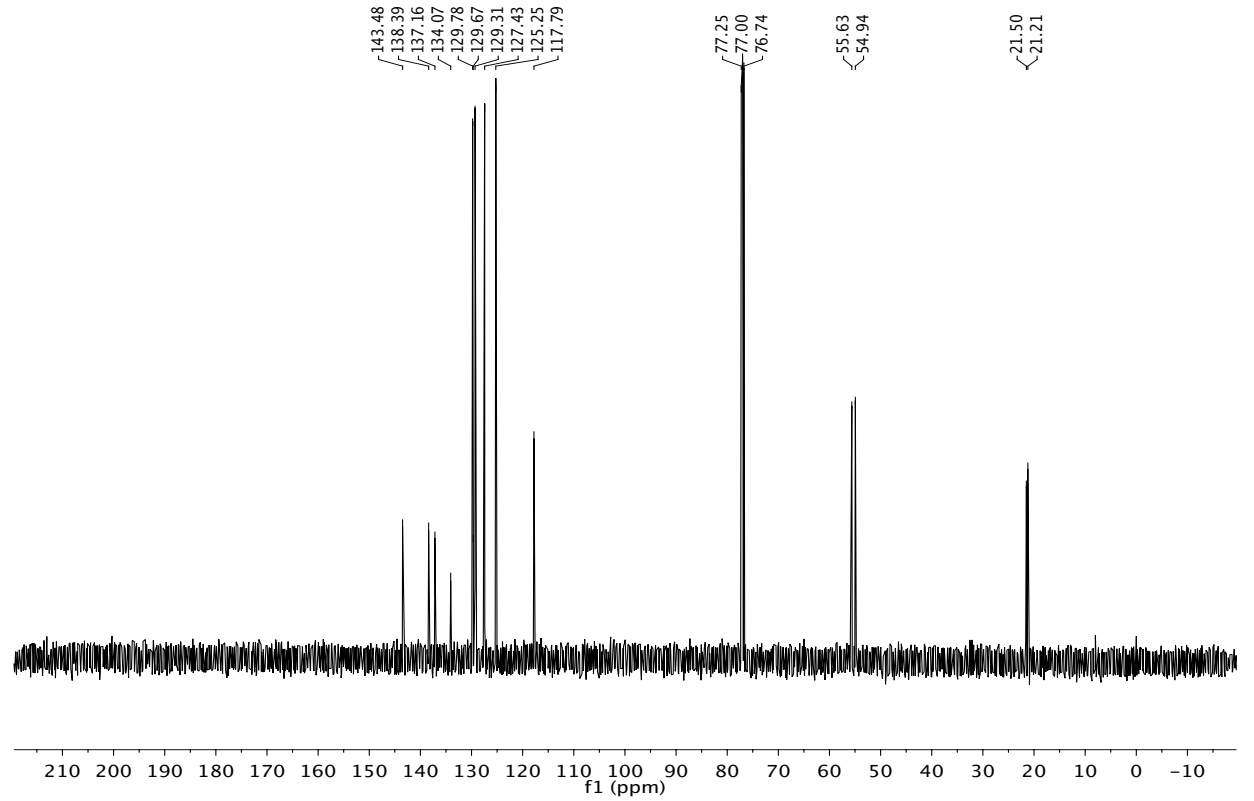


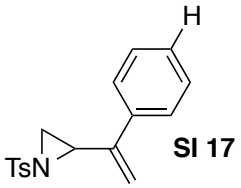



TsN
Me

SI 21

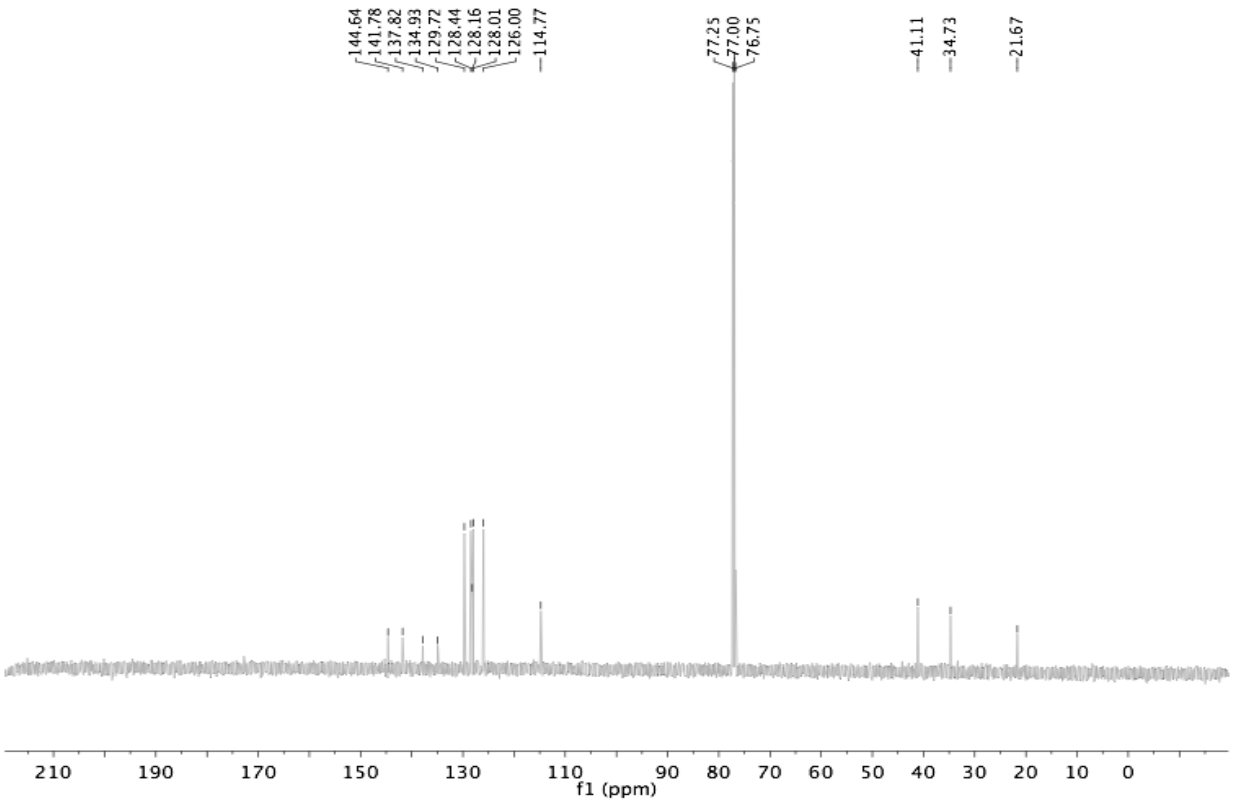

0.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

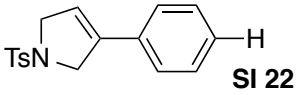

143.48
138.39
137.16
134.07
129.78
129.67
129.31
127.43
125.25
117.79
77.25
77.00
76.74
55.63
54.94
21.50
21.21
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


H
TsN

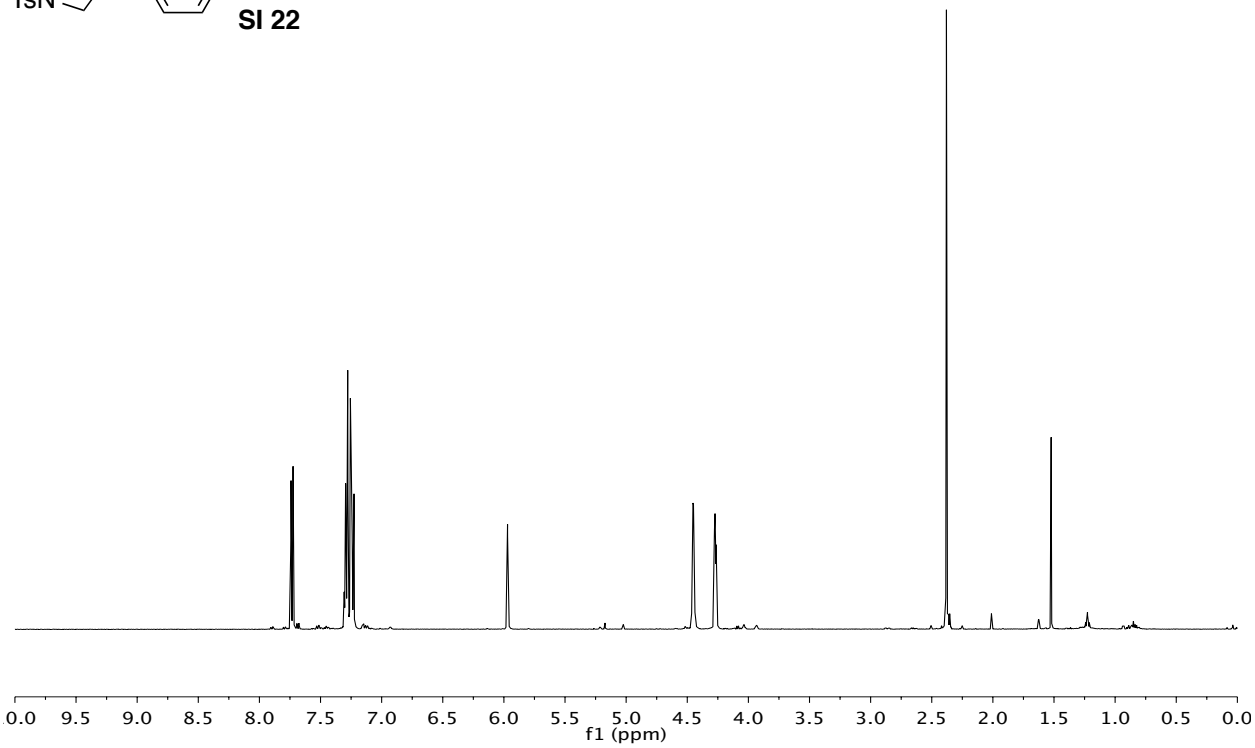
SI 17

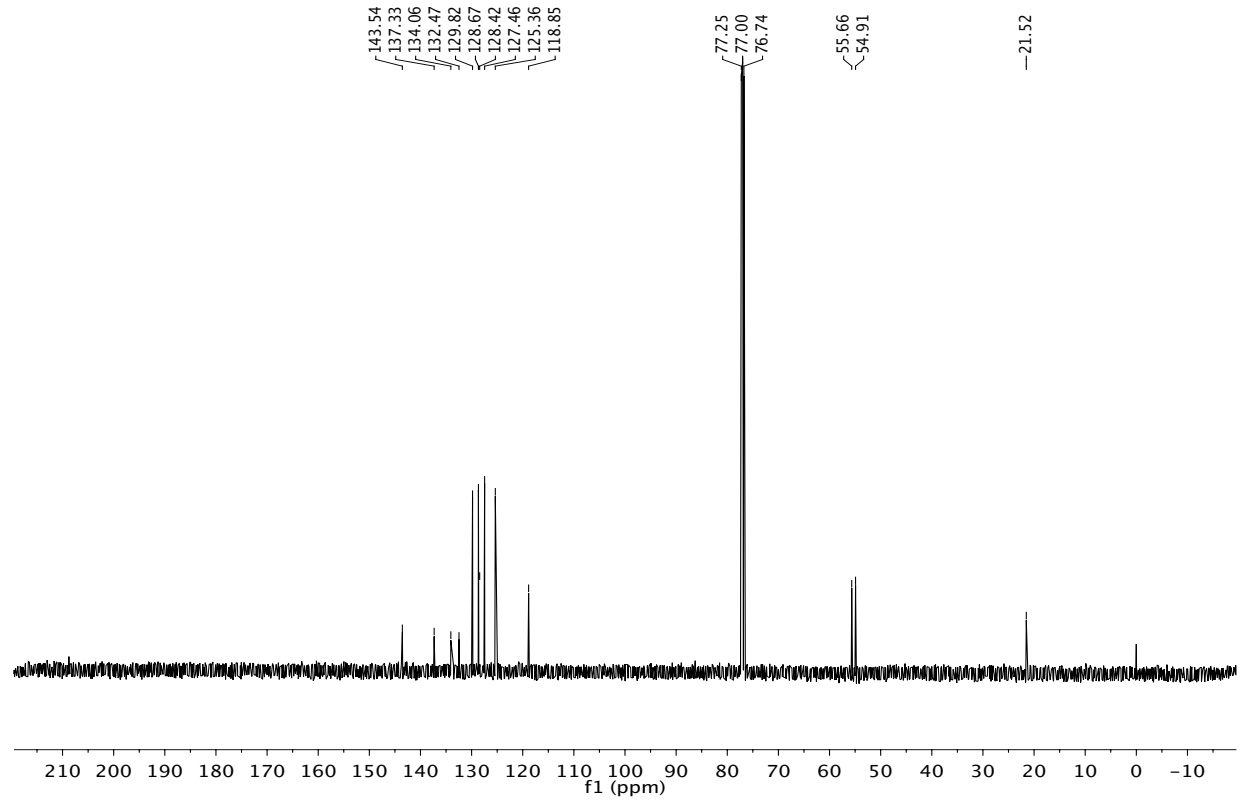
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

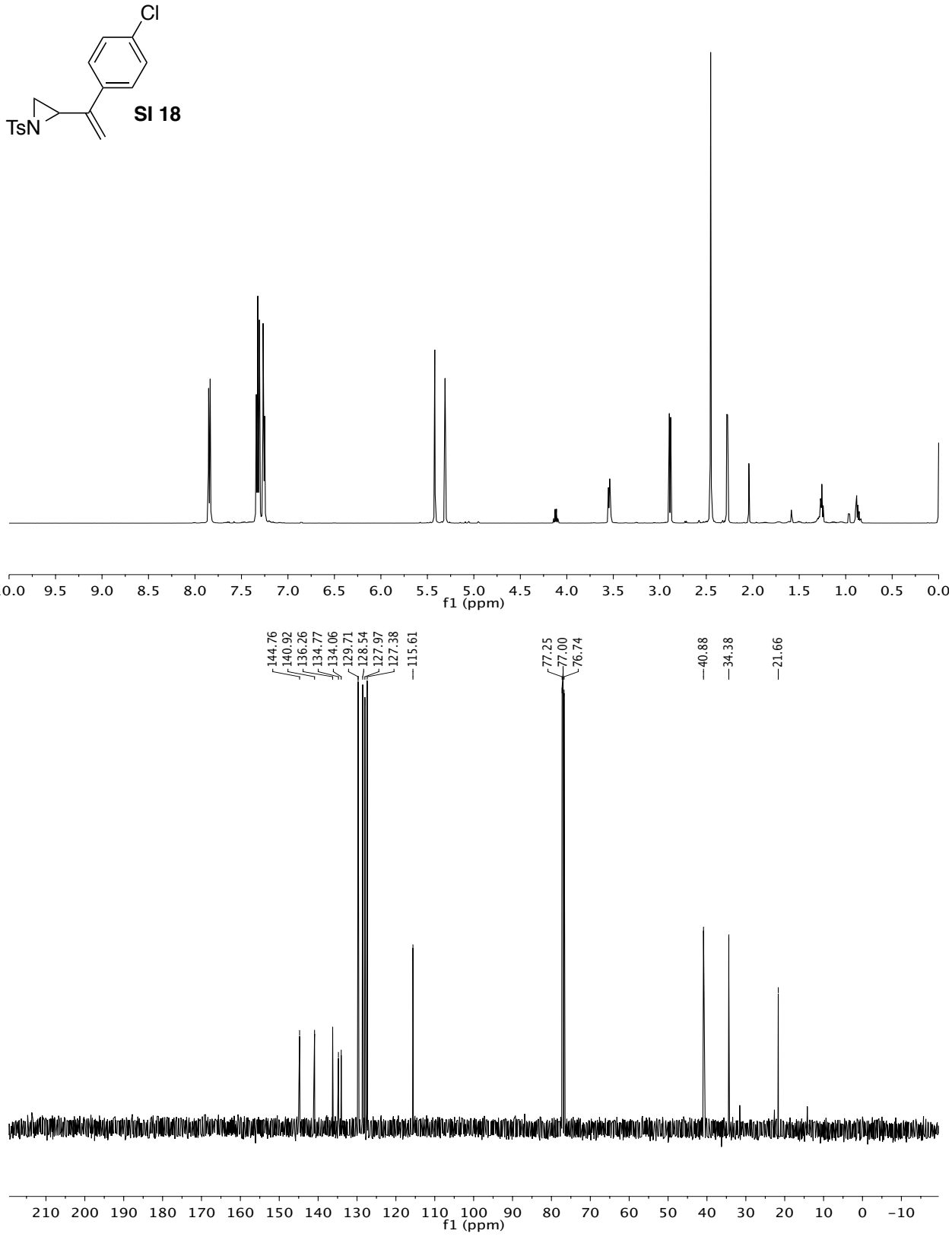

144.64
141.78
137.82
134.93
129.72
128.44
128.16
128.01
126.00
114.77
77.25
77.00
76.75
41.11
34.73
21.67
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

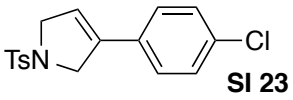

TsN
H

SI 22

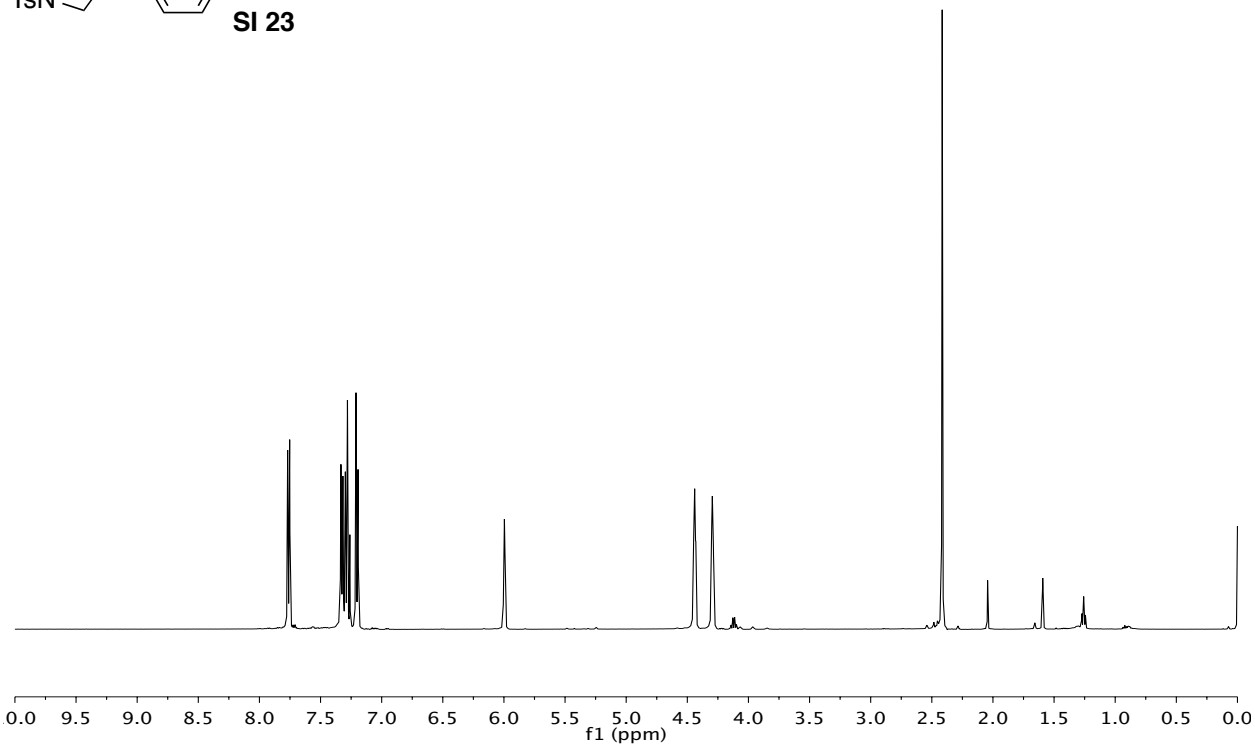

0.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

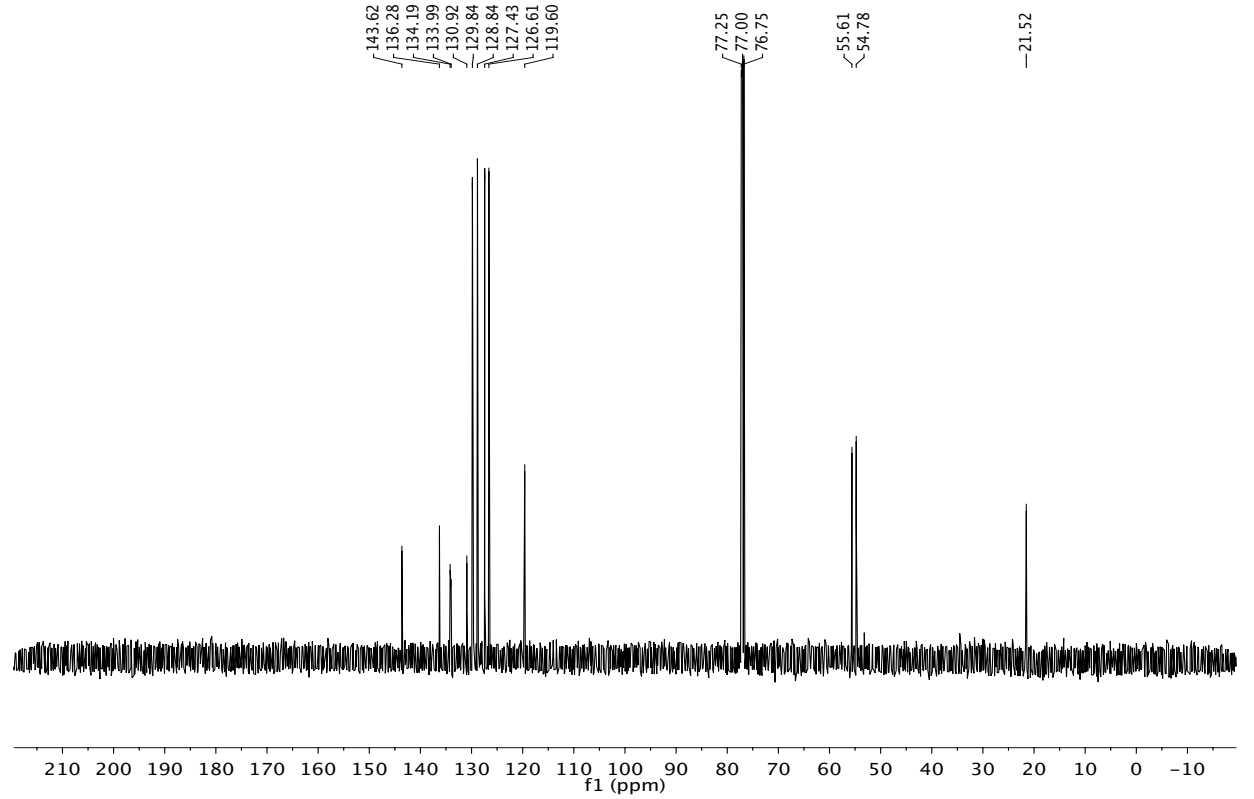

143.54
137.33
134.06
132.47
129.82
128.67
128.42
127.46
125.36
118.85
77.25
77.00
76.74
55.66
54.91
21.52
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

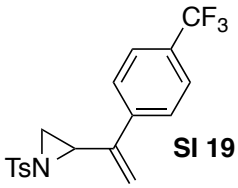




SI 23

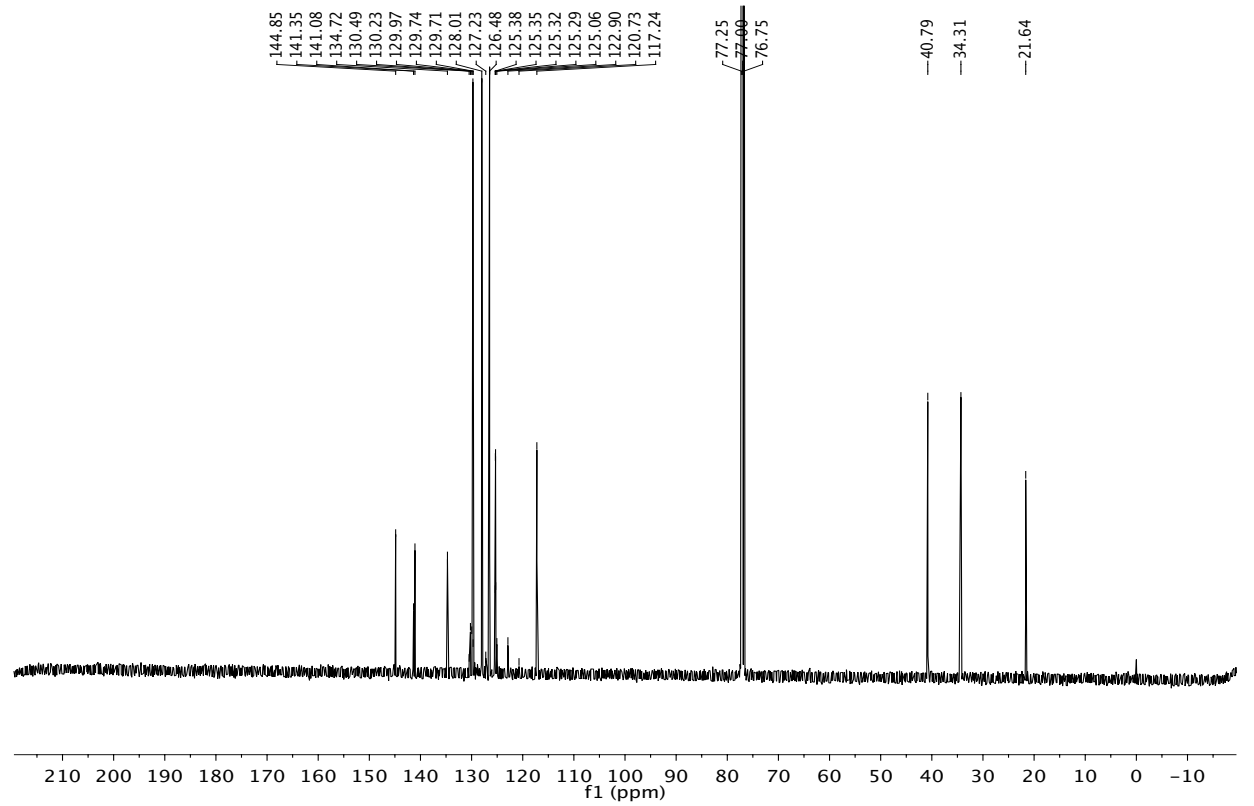


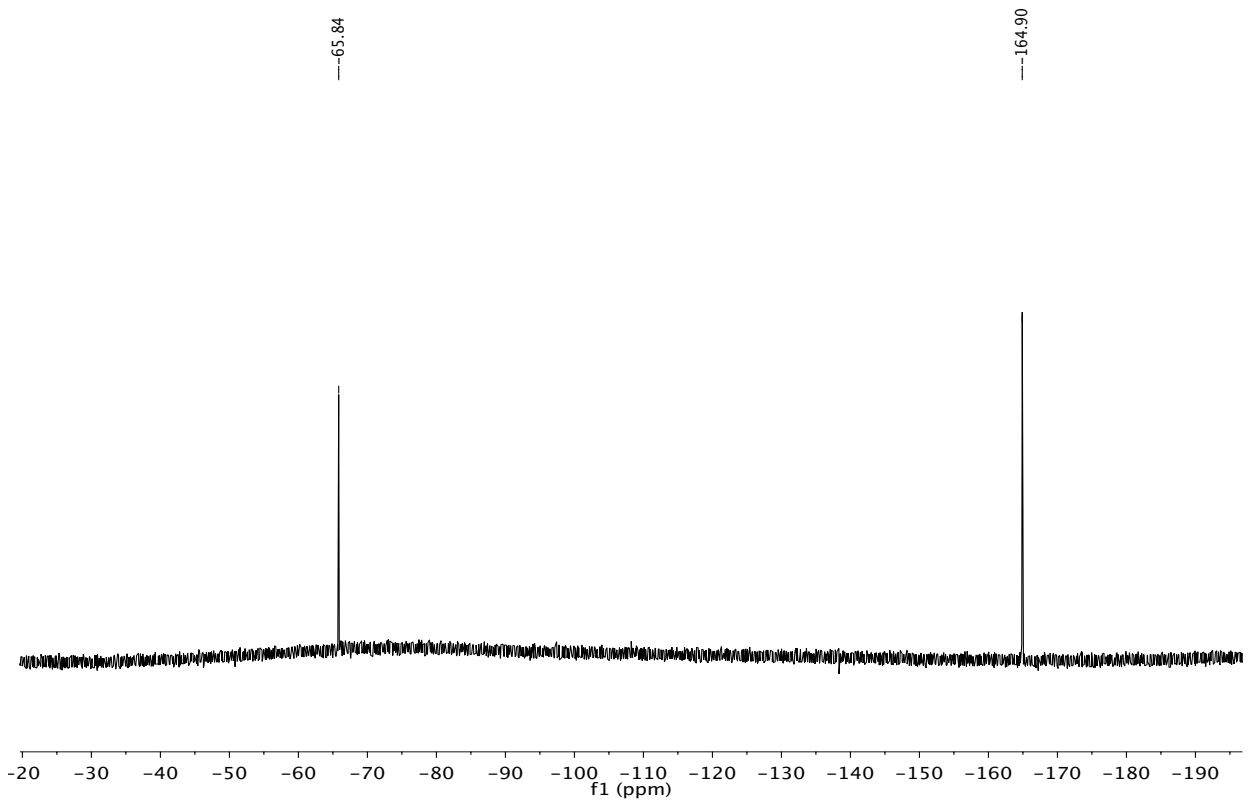


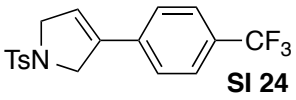

CF3
TsN

SI 19

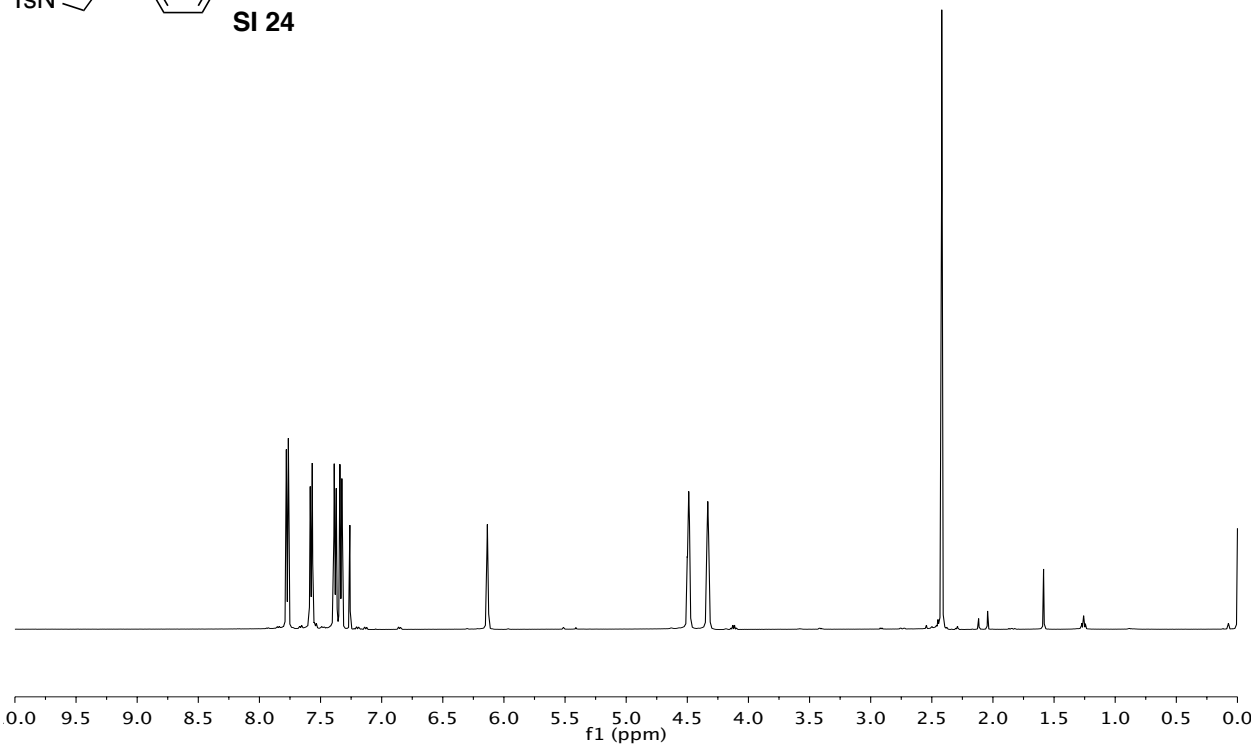
0.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

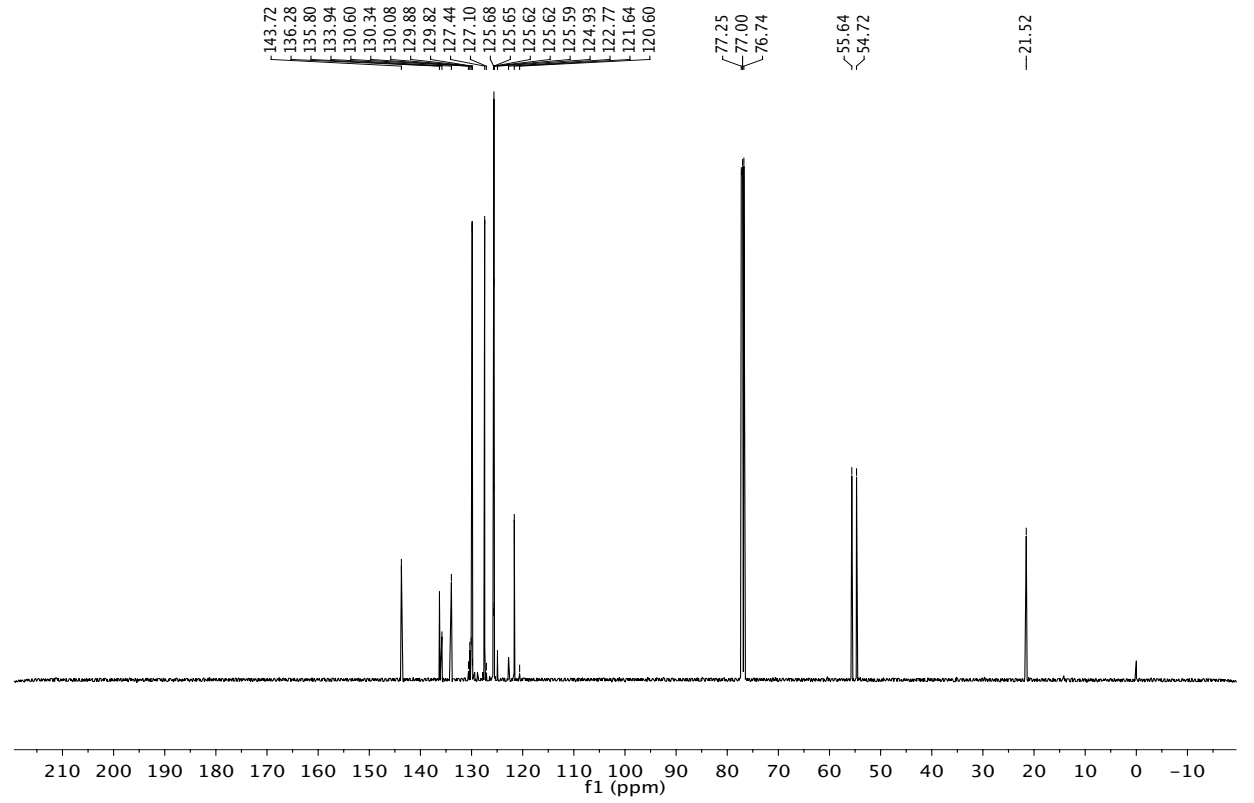

144.85
141.35
141.08
134.72
130.49
130.23
129.97
129.74
129.71
128.01
127.23
126.48
125.38
125.35
125.32
125.29
125.06
122.90
120.73
117.24
77.25
77.00
76.75
40.79
34.31
21.64
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

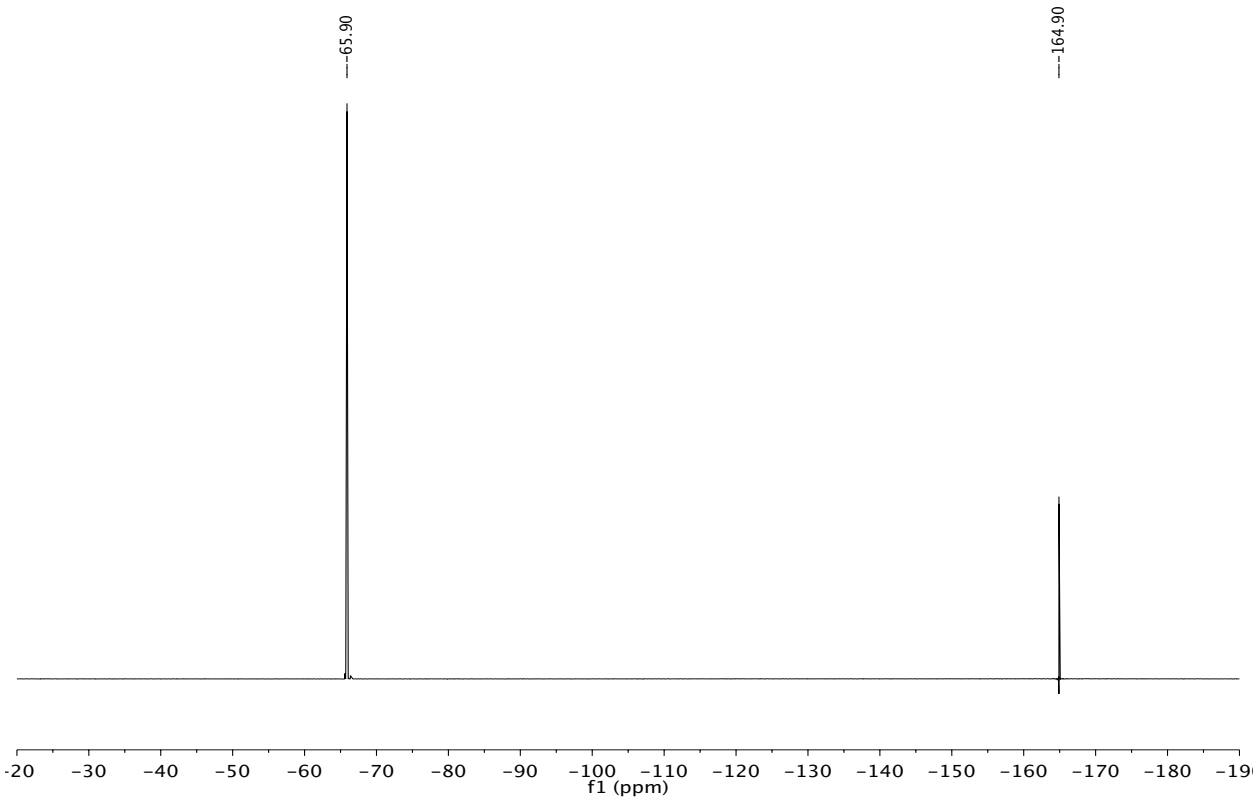


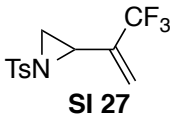

TsN
CF3

SI 24

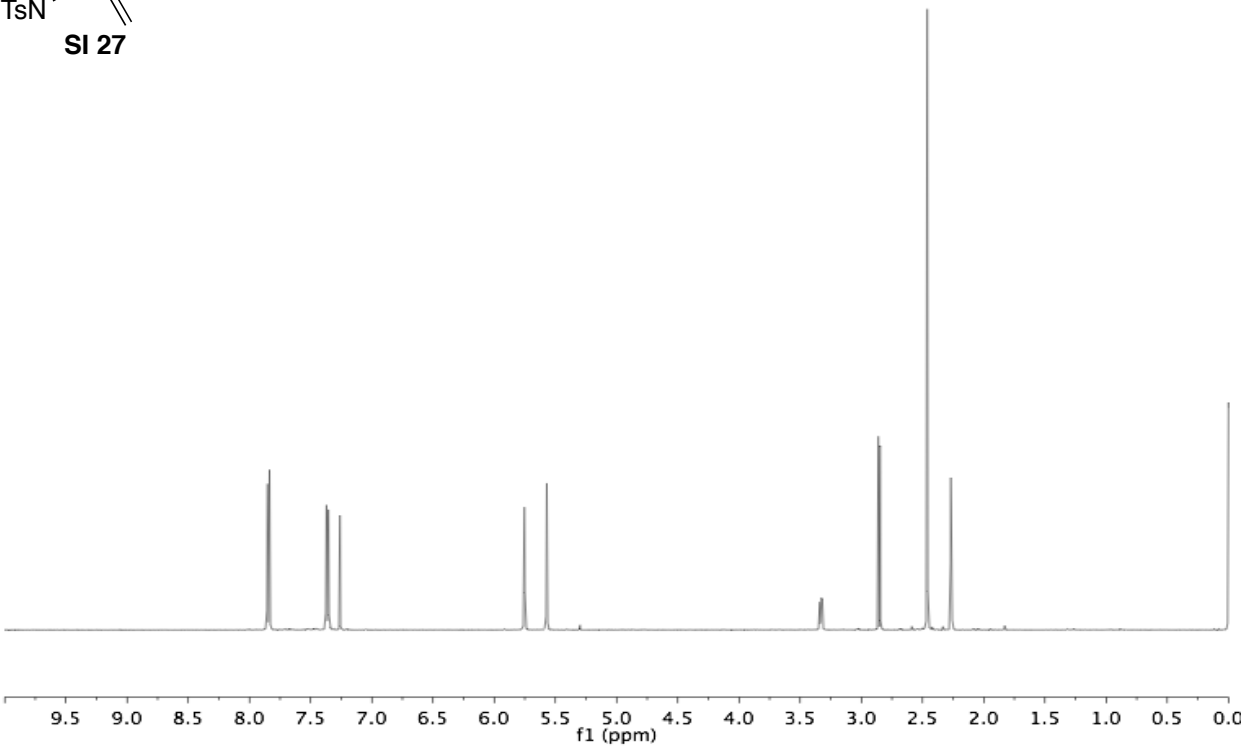

0.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

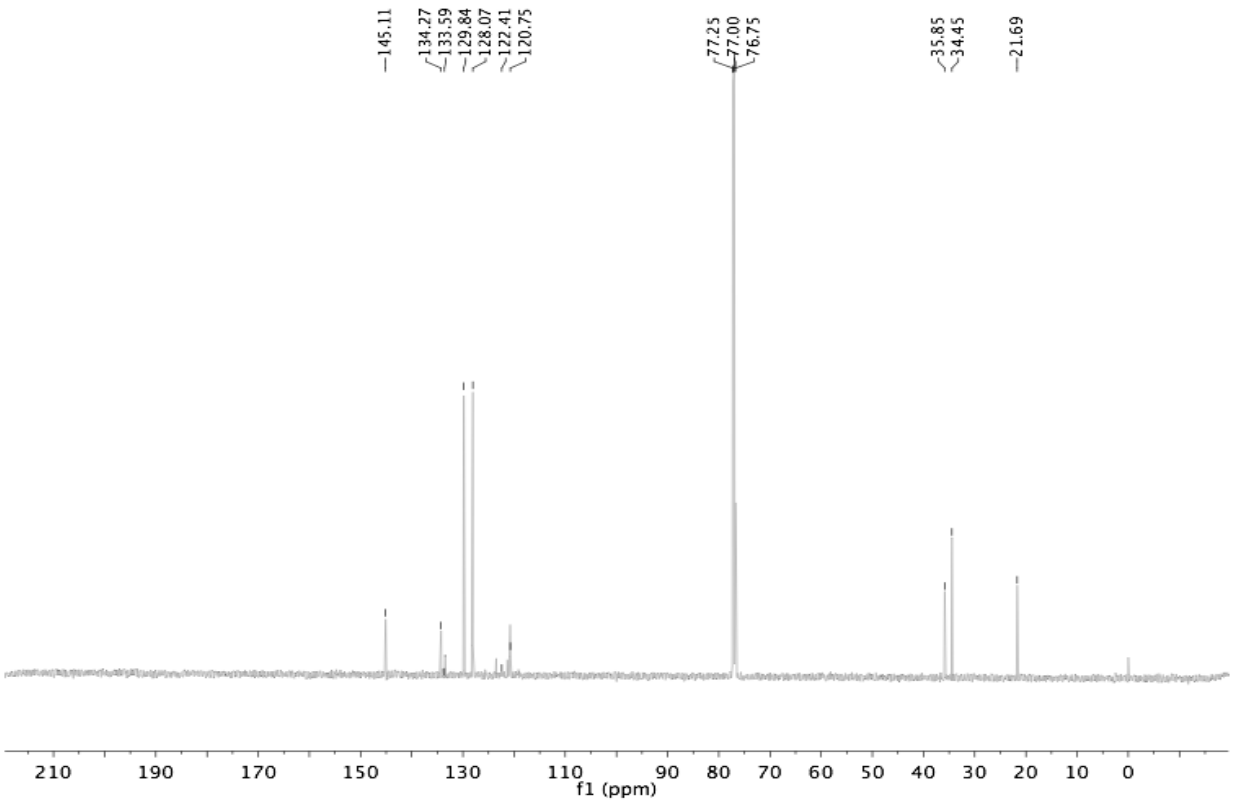

143.72
136.28
135.80
133.94
130.60
130.34
130.08
129.88
129.82
127.44
127.10
125.68
125.65
125.62
125.62
125.59
124.93
122.77
121.64
120.60
77.25
77.00
76.74
55.64
54.72
21.52
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

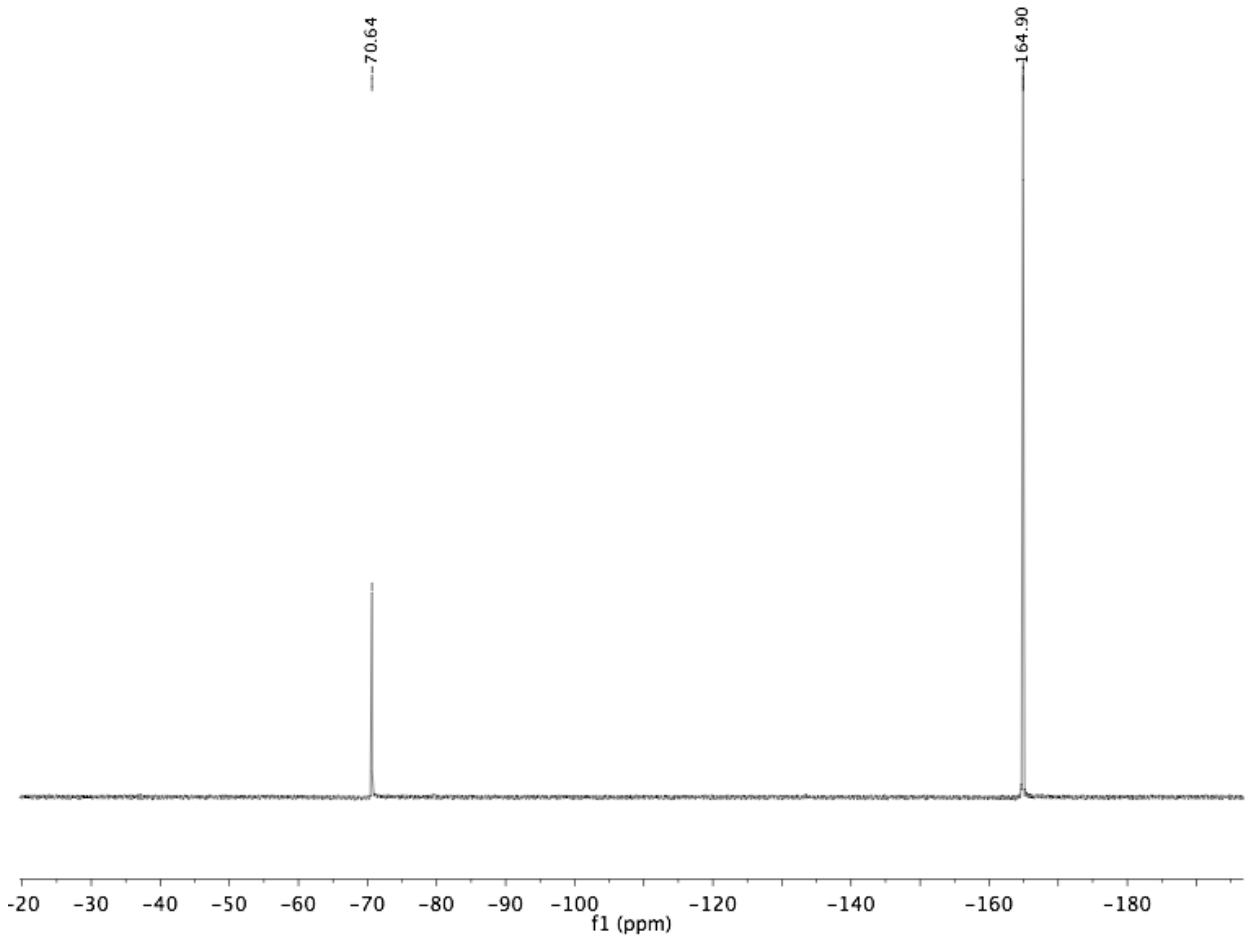


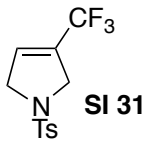

CF3
TsN

SI 27


9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

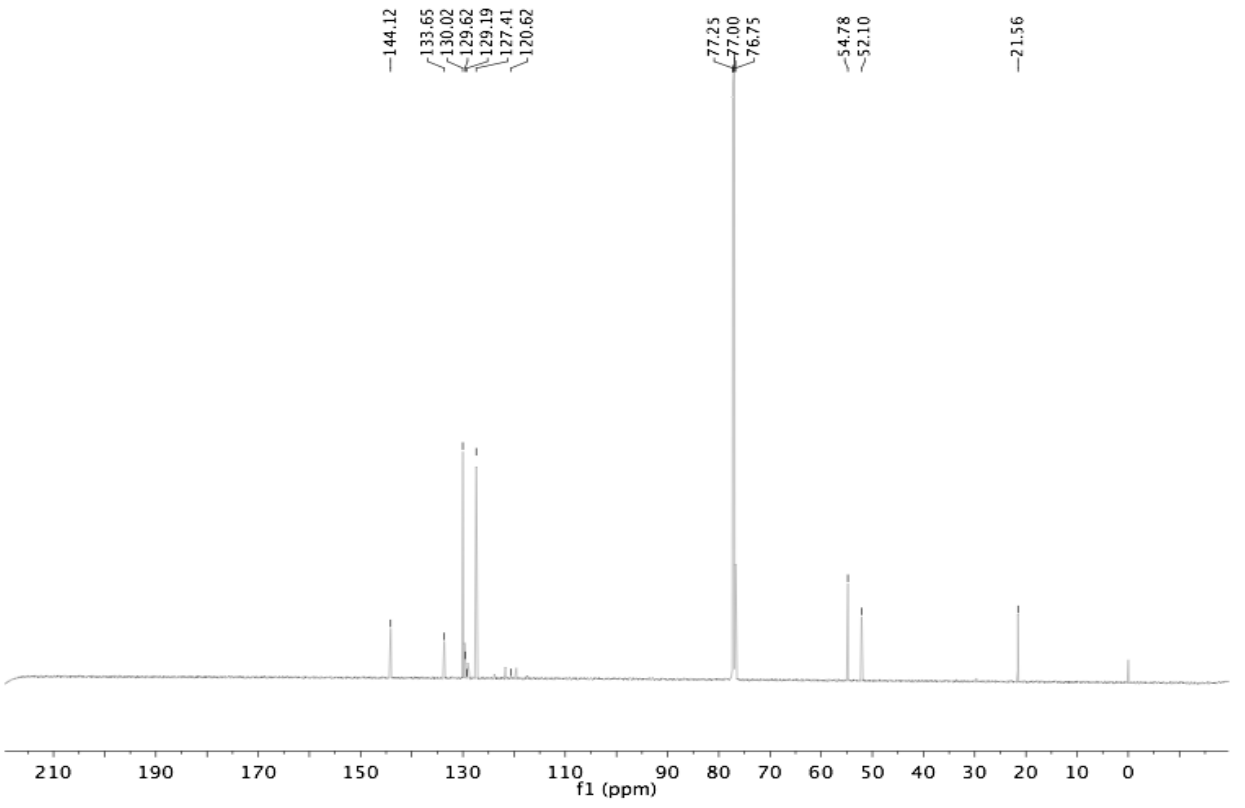

145.11
134.27
133.59
129.84
128.07
122.41
120.75
77.25
77.00
76.75
35.85
34.45
21.69
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

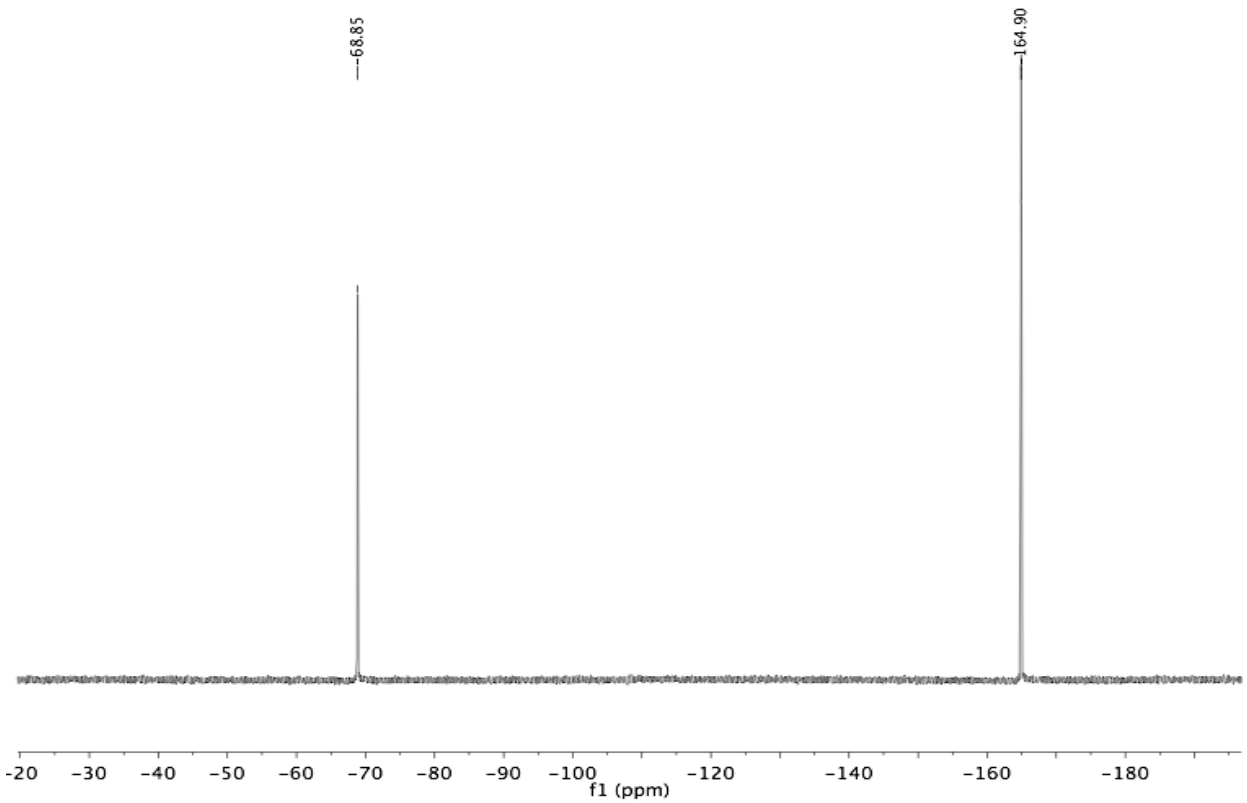


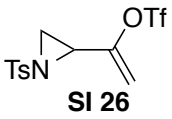

CF3
N
Ts

SI 31

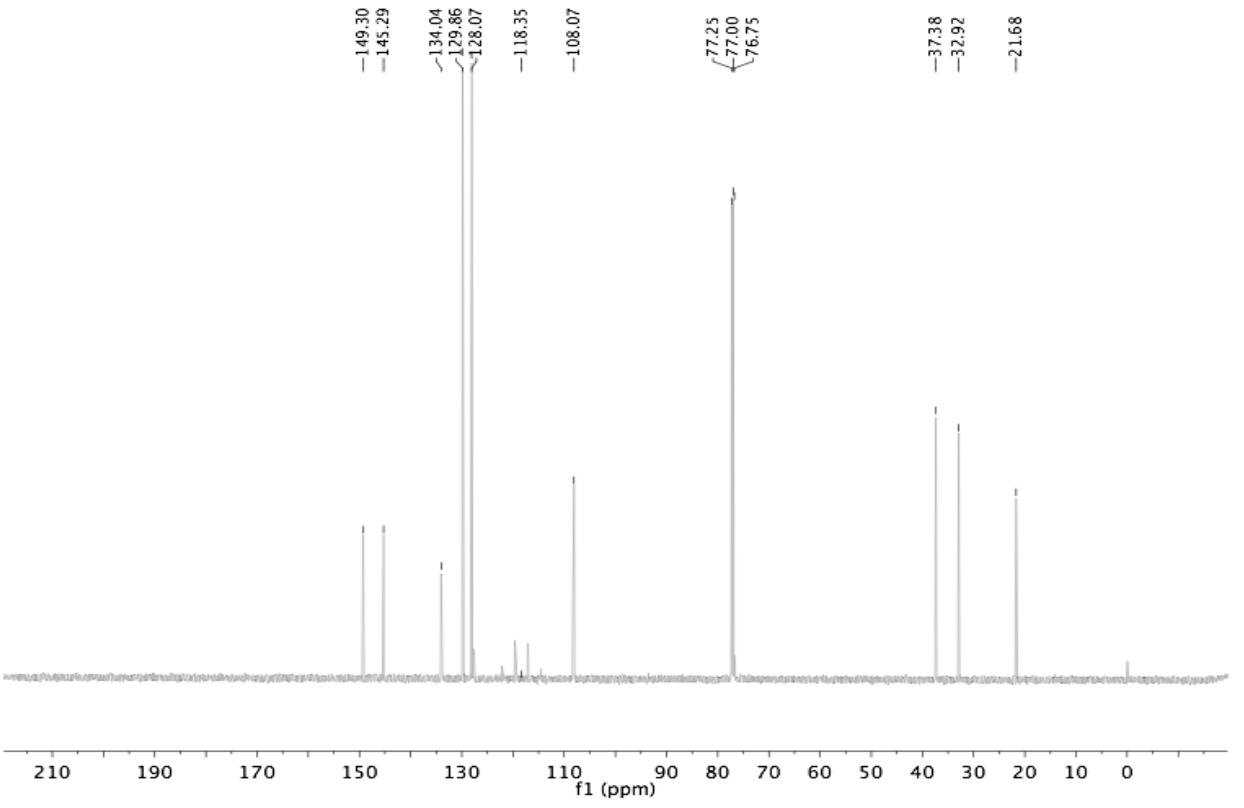
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

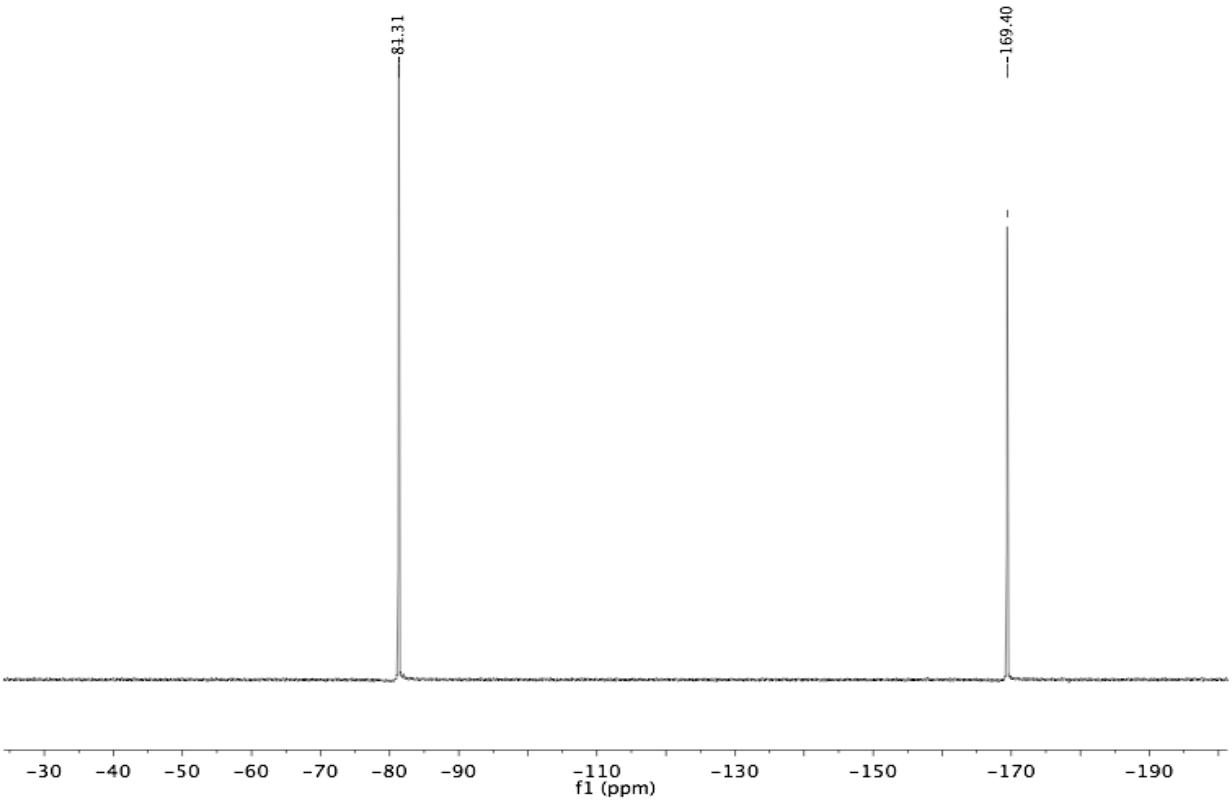

144.12
133.65
130.02
129.62
129.19
127.41
120.62
77.25
77.00
76.75
54.78
52.10
21.56
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

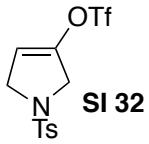




SI 26

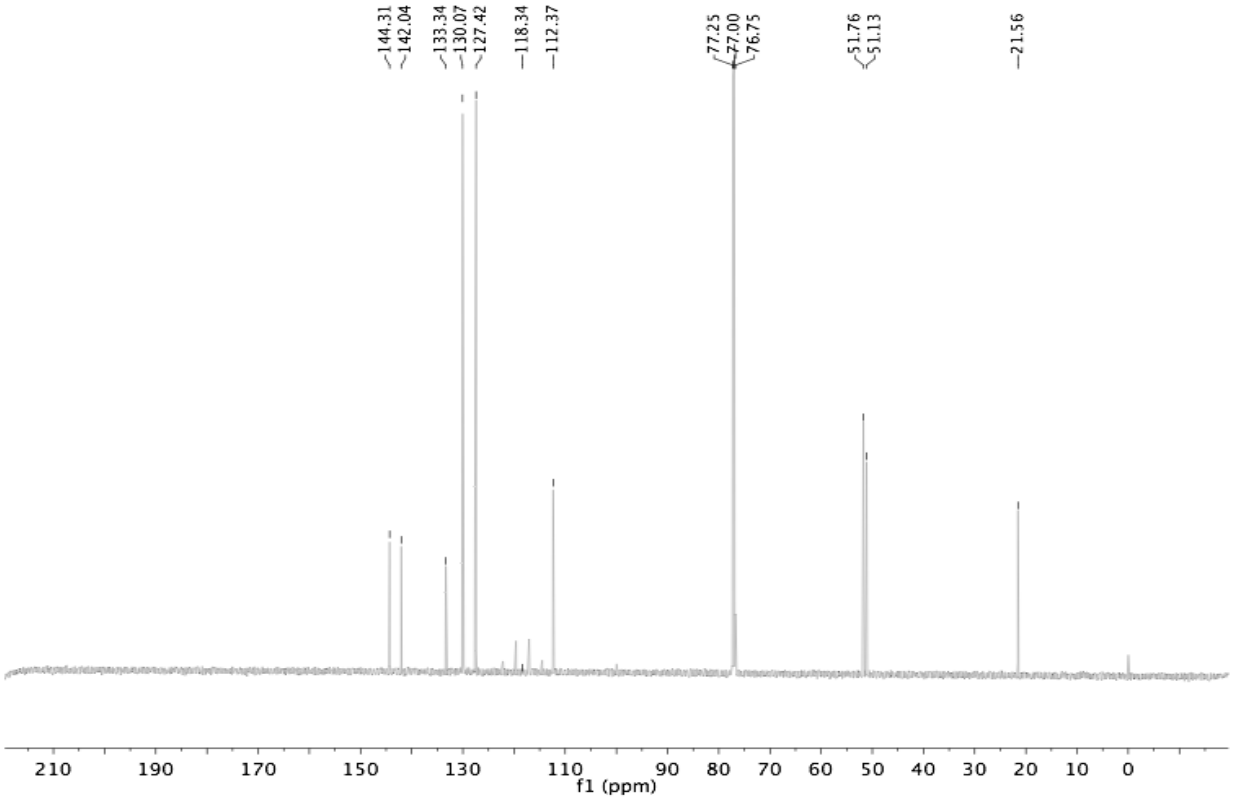


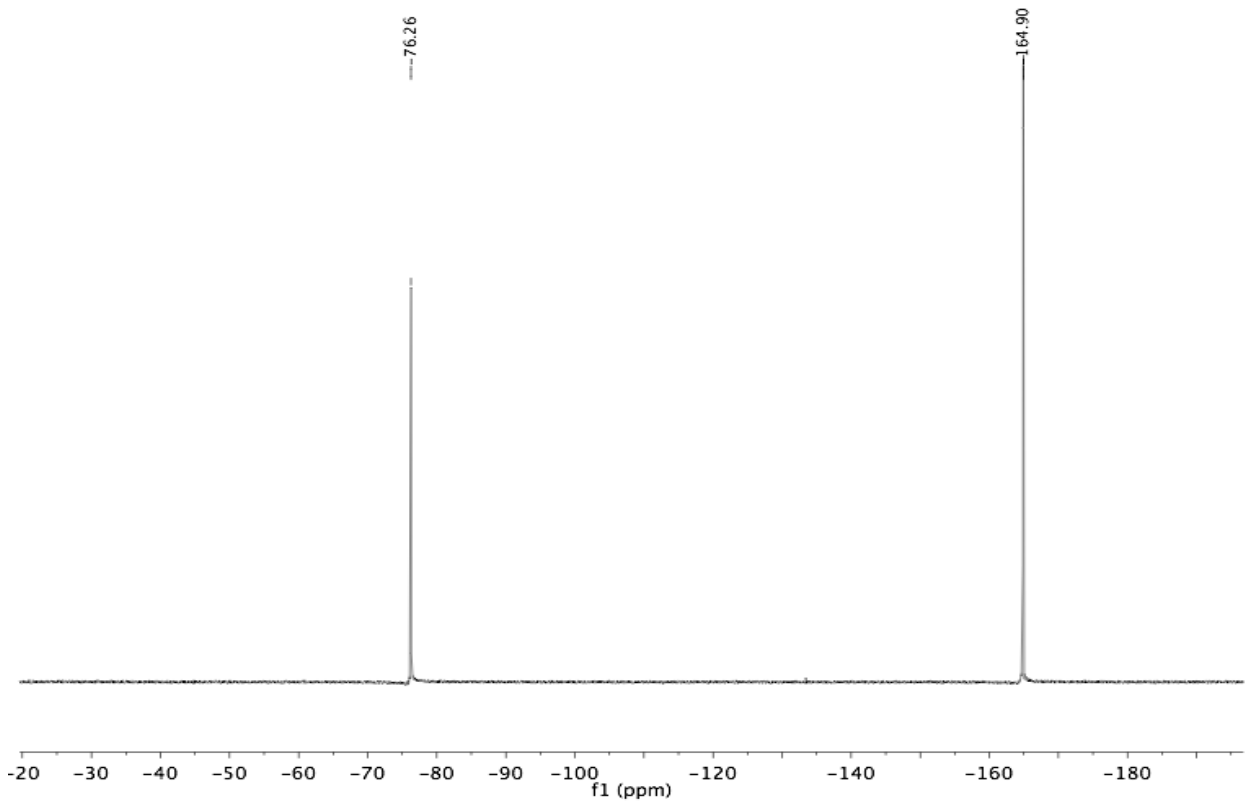


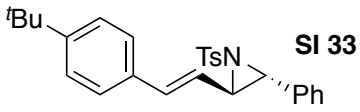

OTf
N
Ts

SI 32

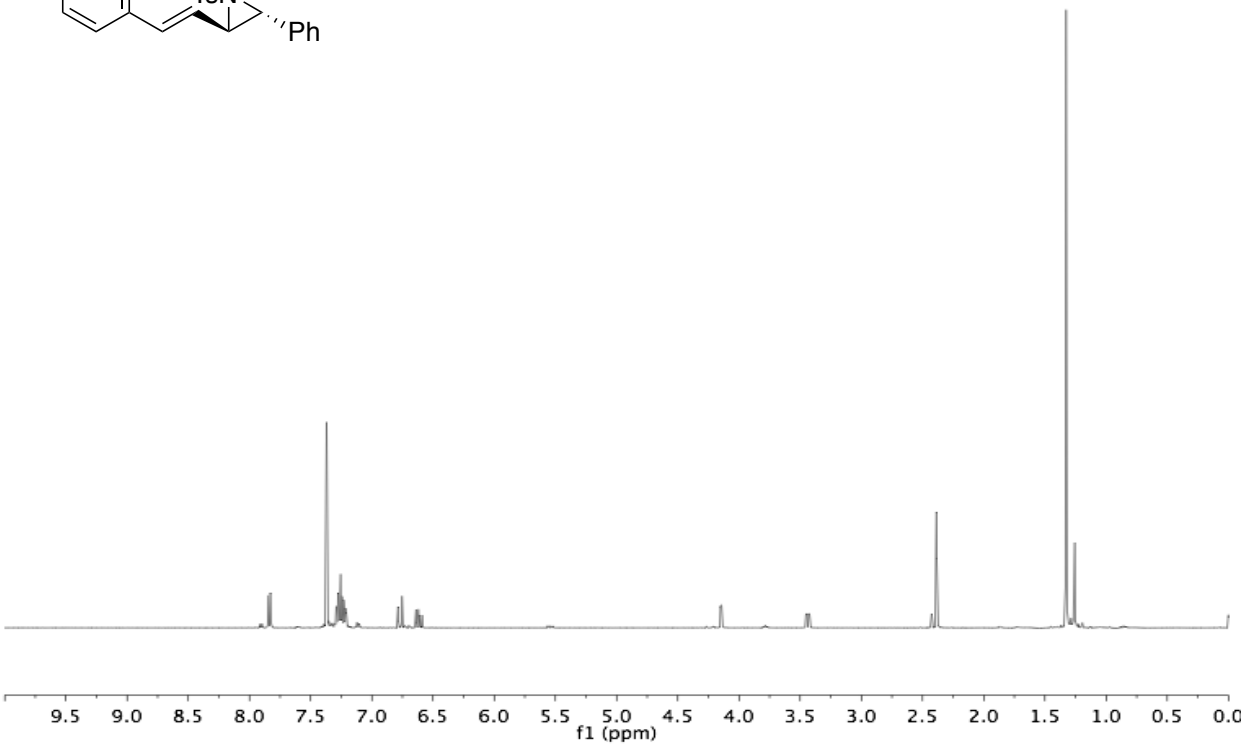
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

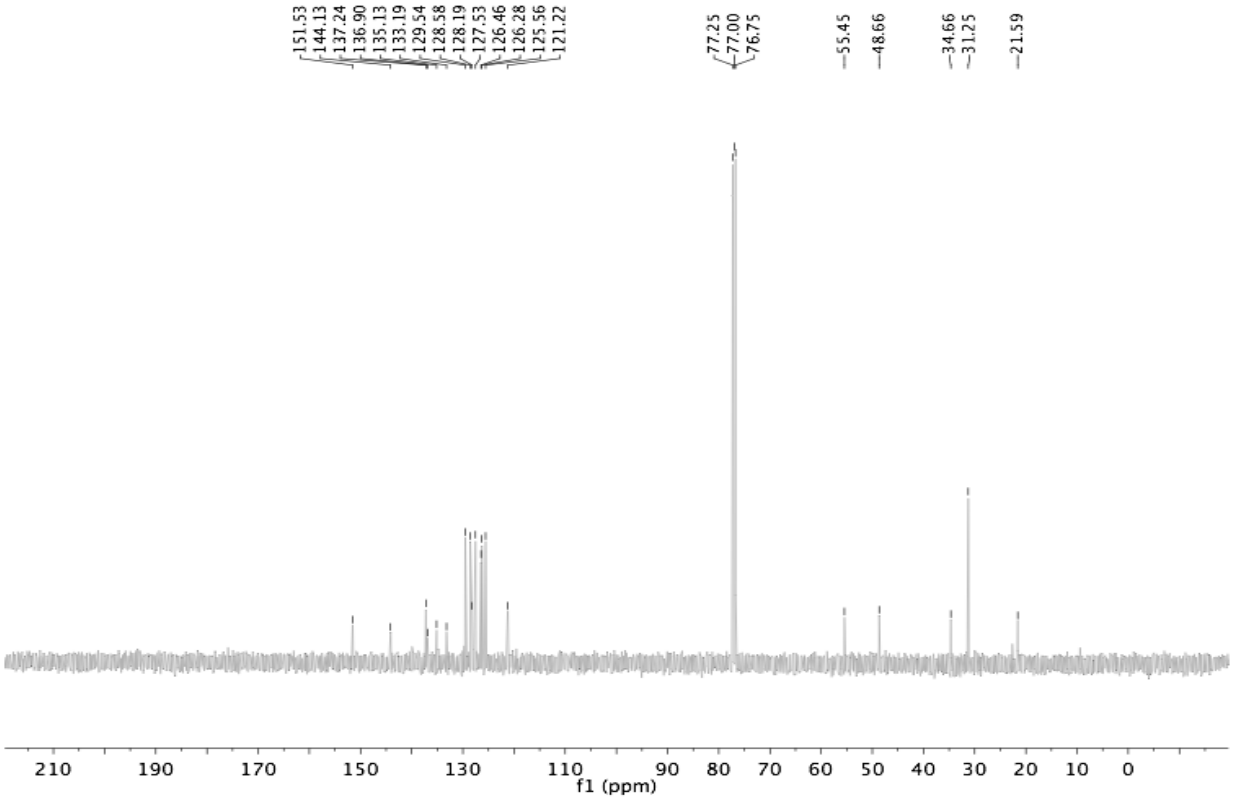

144.31
142.04
133.34
130.07
127.42
118.34
112.37
77.25
77.00
76.75
51.76
51.13
21.56
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

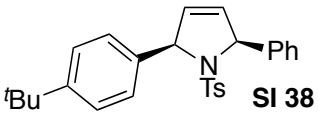




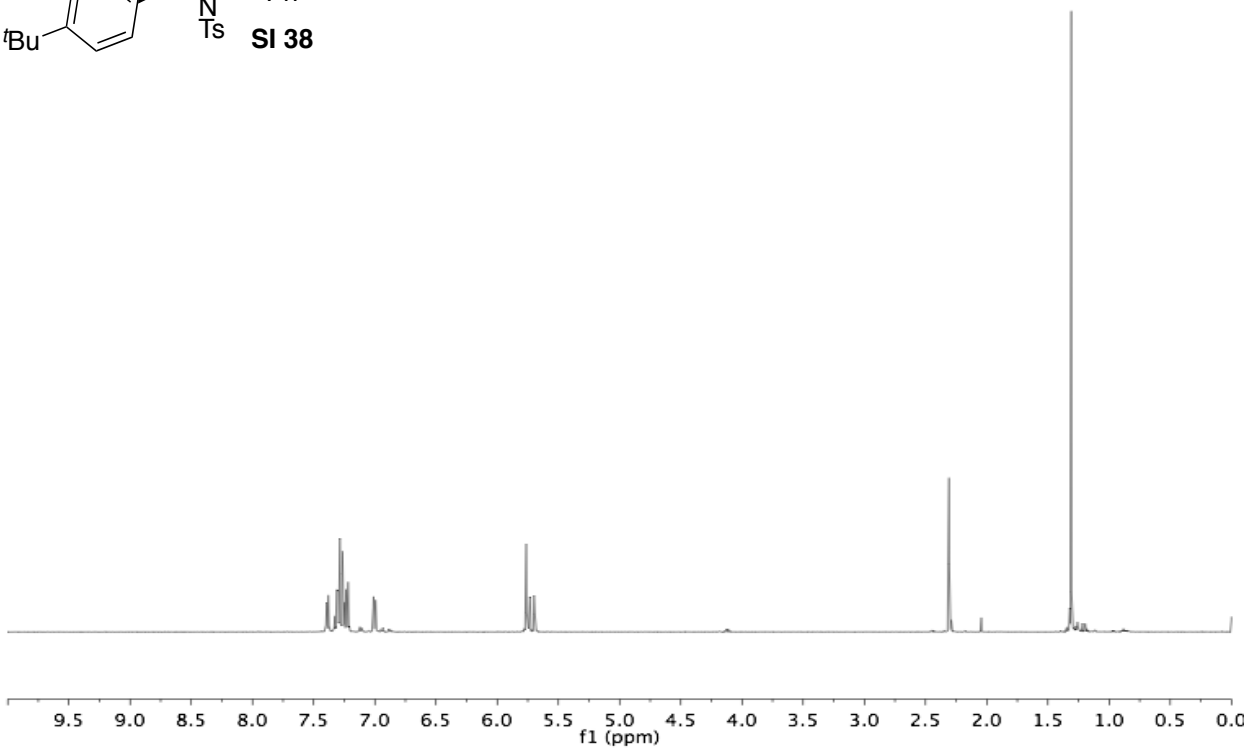
SI 33

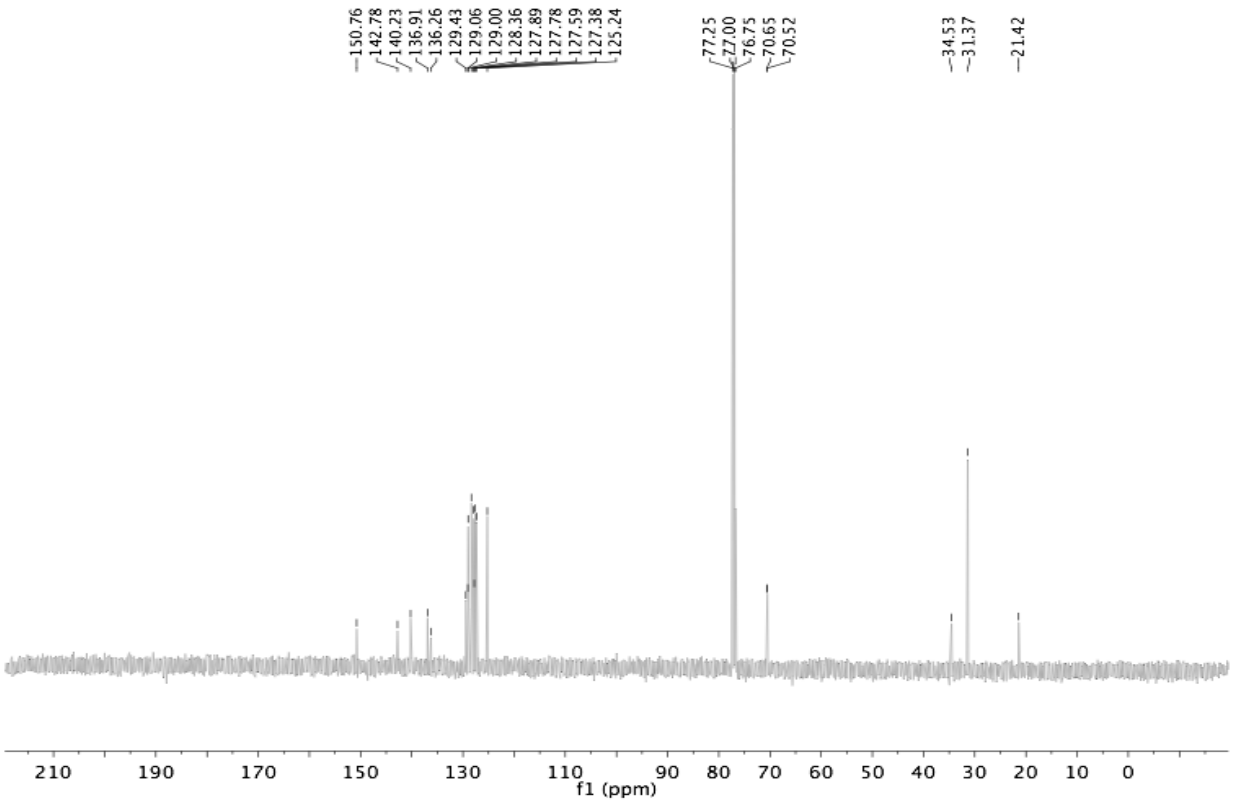


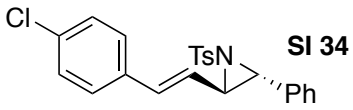



Ph
N
Ts
tBu

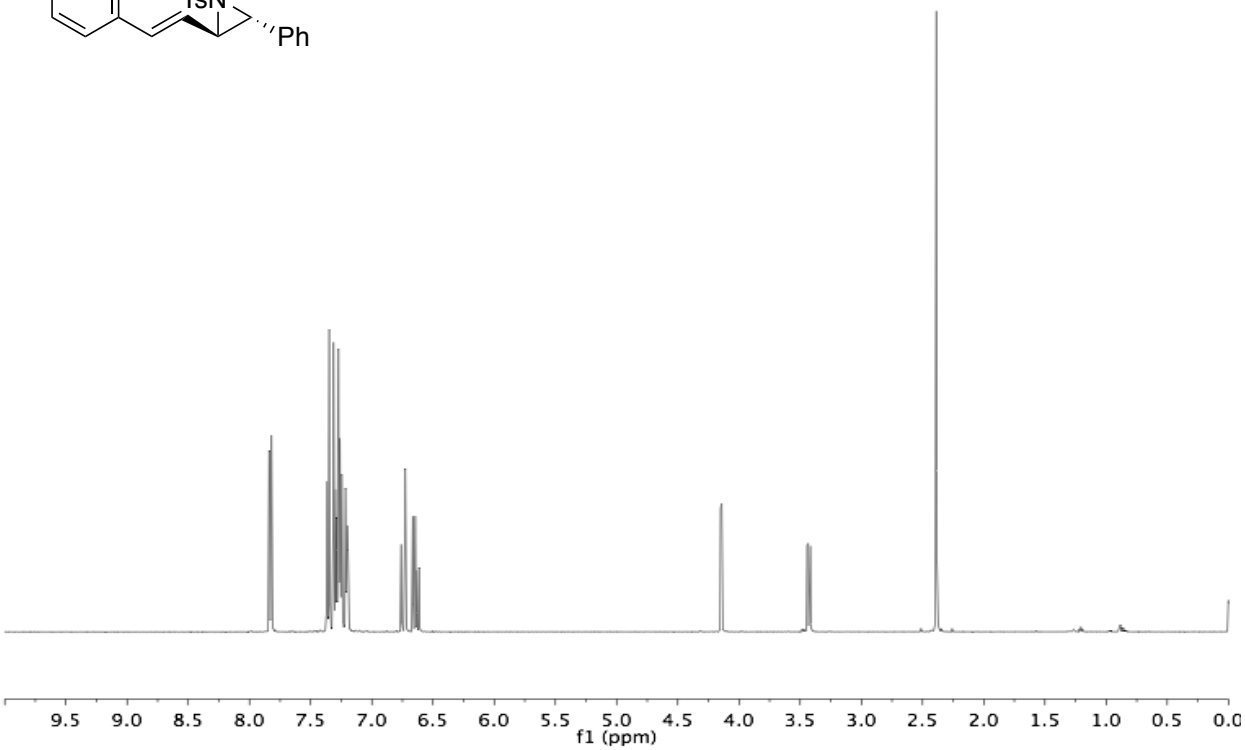
SI 38

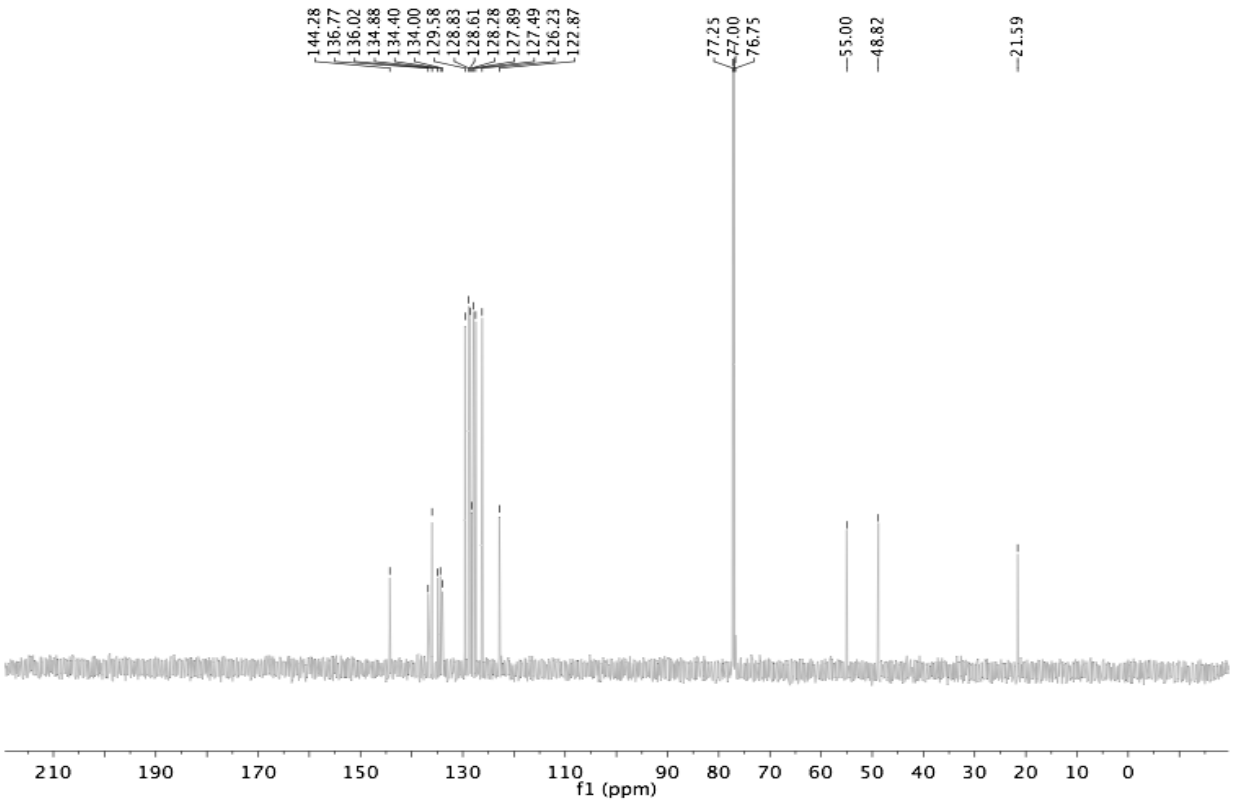

9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

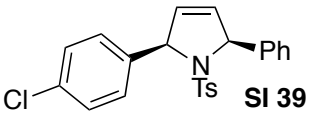

150.76
142.78
140.23
136.91
136.26
129.43
129.06
129.00
128.36
127.89
127.78
127.59
127.38
125.24
77.25
77.00
76.75
70.65
70.52
34.53
31.37
21.42
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



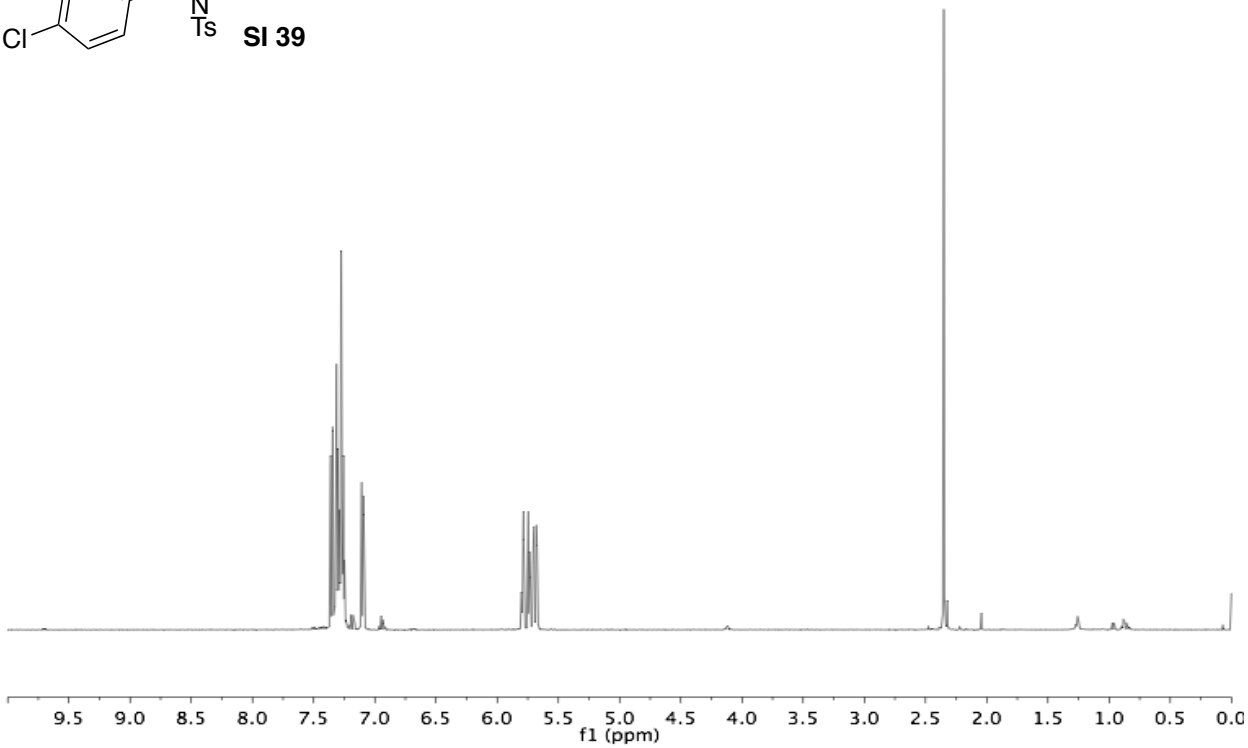
SI 34

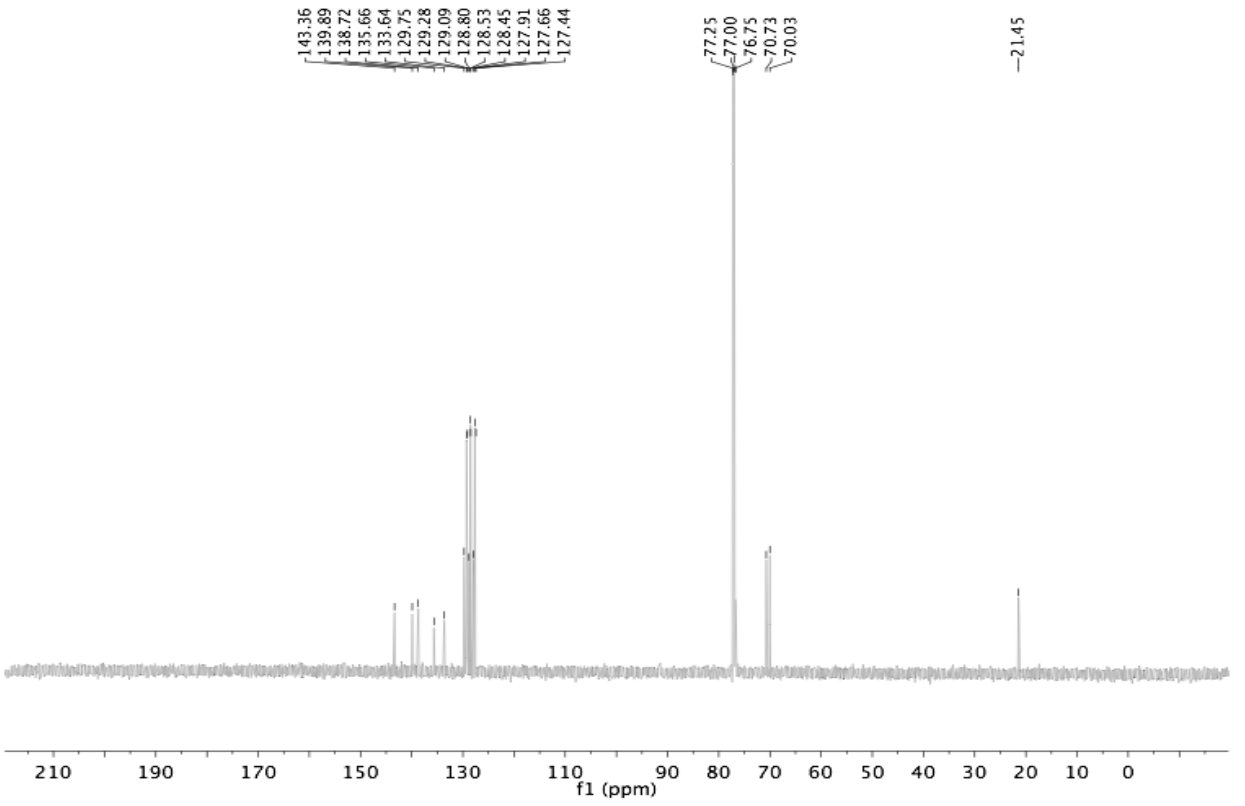


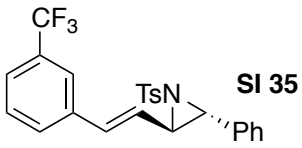



Cl
N
Ts
Ph

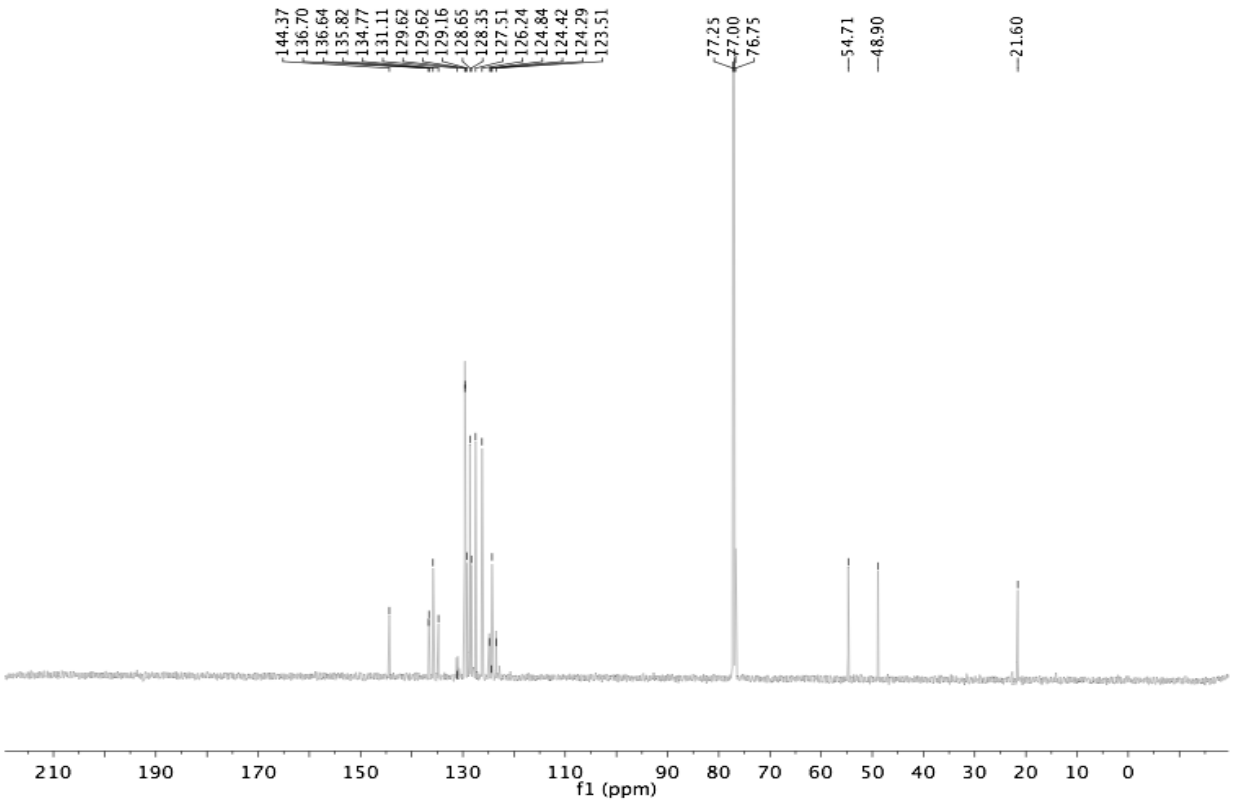
SI 39

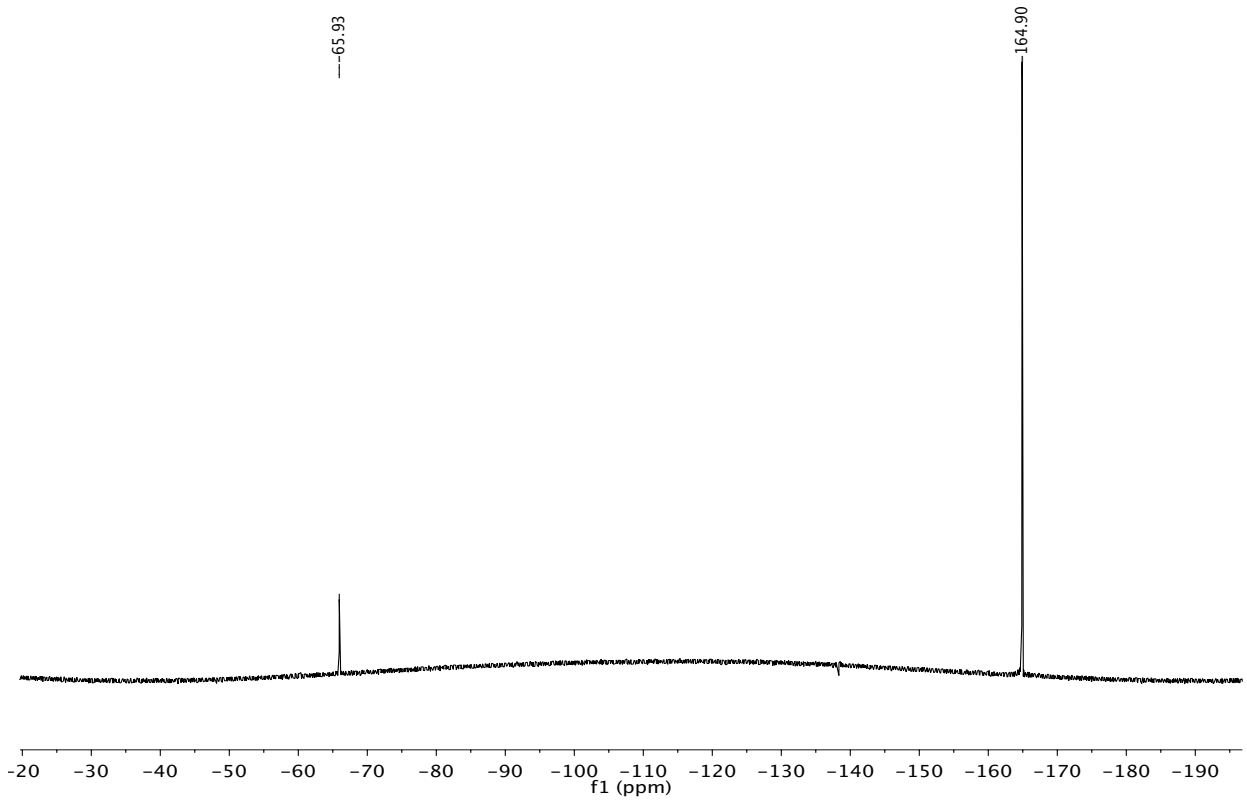

9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

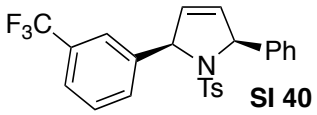

143.36
139.89
138.72
135.66
133.64
129.75
129.28
129.09
128.80
128.53
128.45
127.91
127.66
127.44
77.25
77.00
76.75
70.73
70.03
21.45
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



SI 35

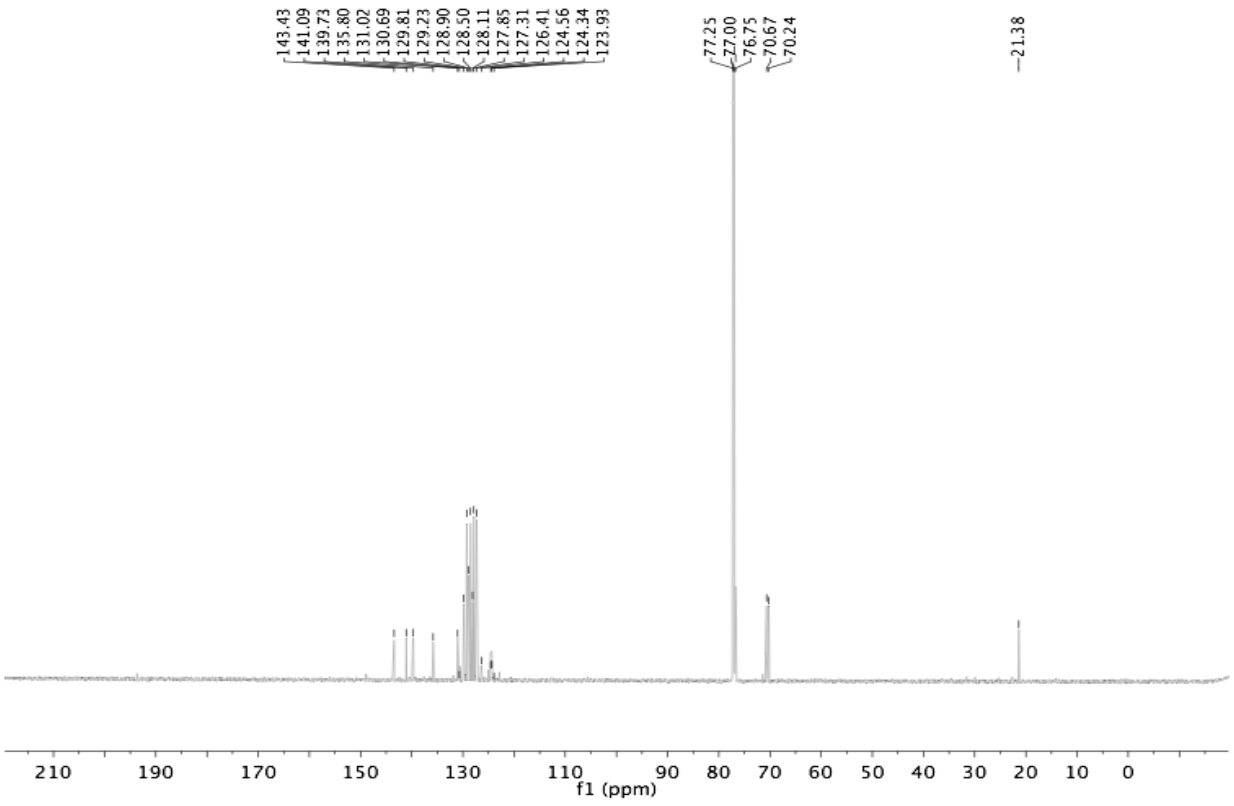


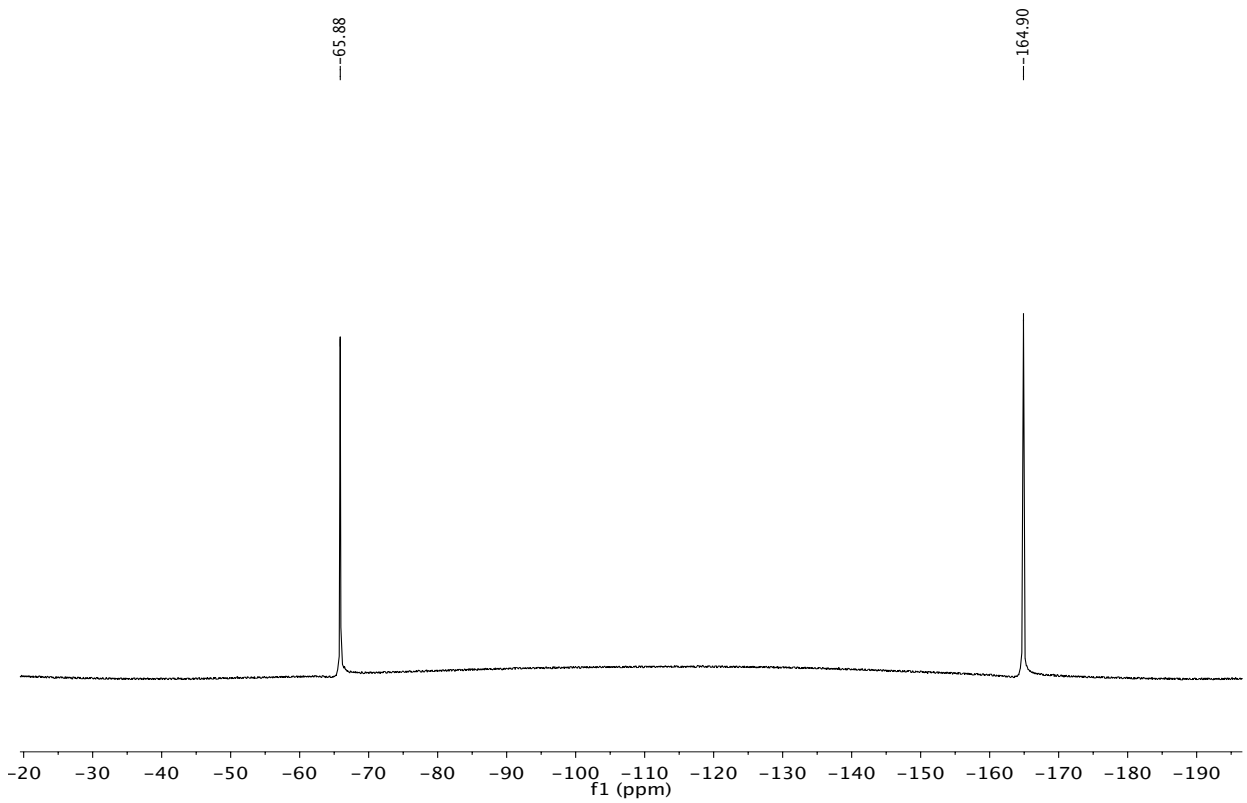


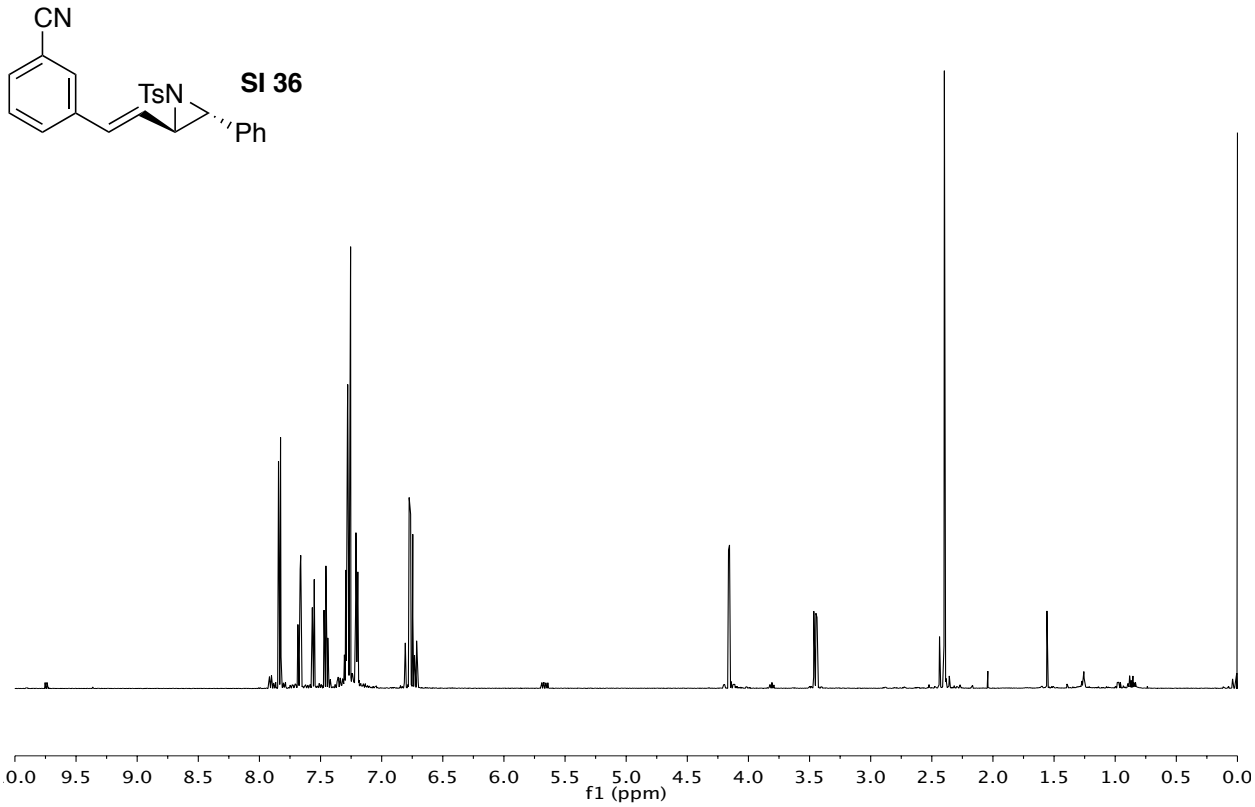

F3C
Ph
N
Ts

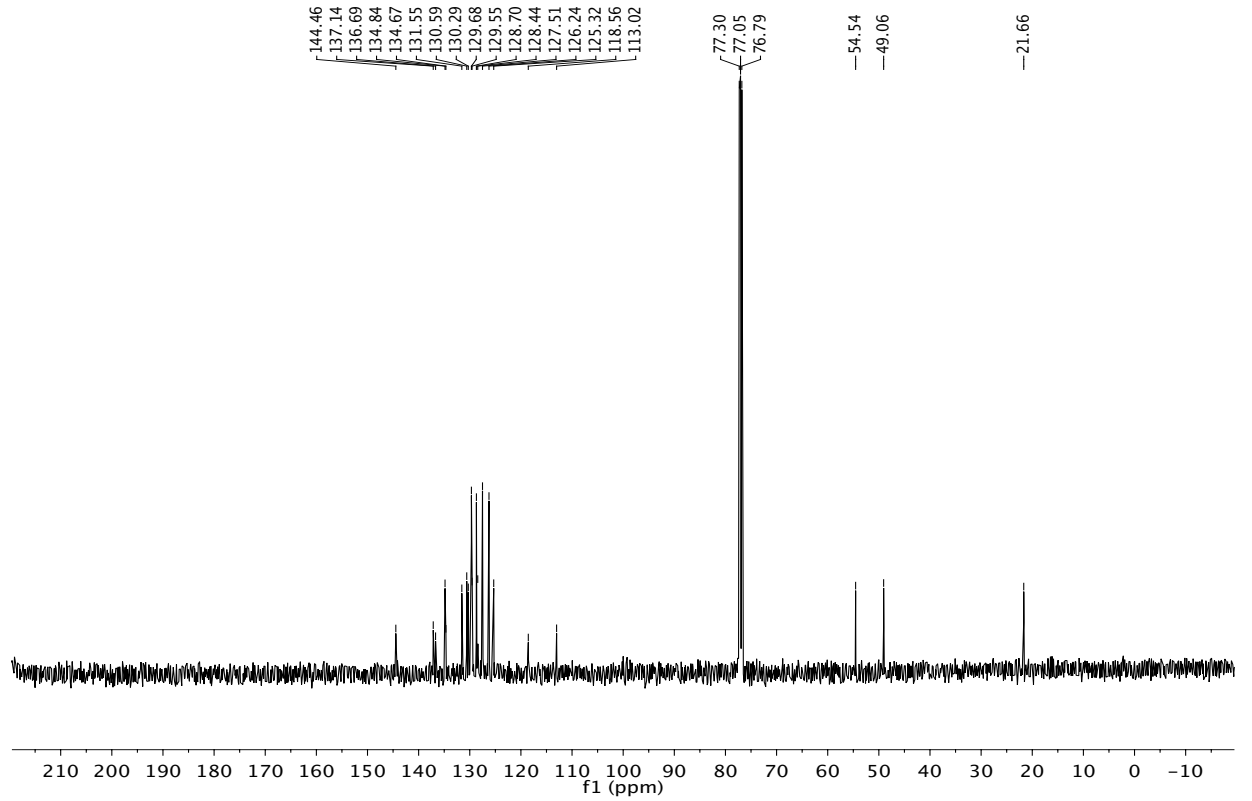
SI 40

9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)


143.43
141.09
139.73
135.80
131.02
130.69
129.81
129.23
128.90
128.50
128.11
127.85
127.31
126.41
124.56
124.34
123.93
77.25
77.00
76.75
70.67
70.24
21.38
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)




CN
TsN
Ph
SI 36
f1 (ppm)


144.46
137.14
136.69
134.84
134.67
131.55
130.59
130.29
129.68
129.55
128.70
128.44
127.51
126.24
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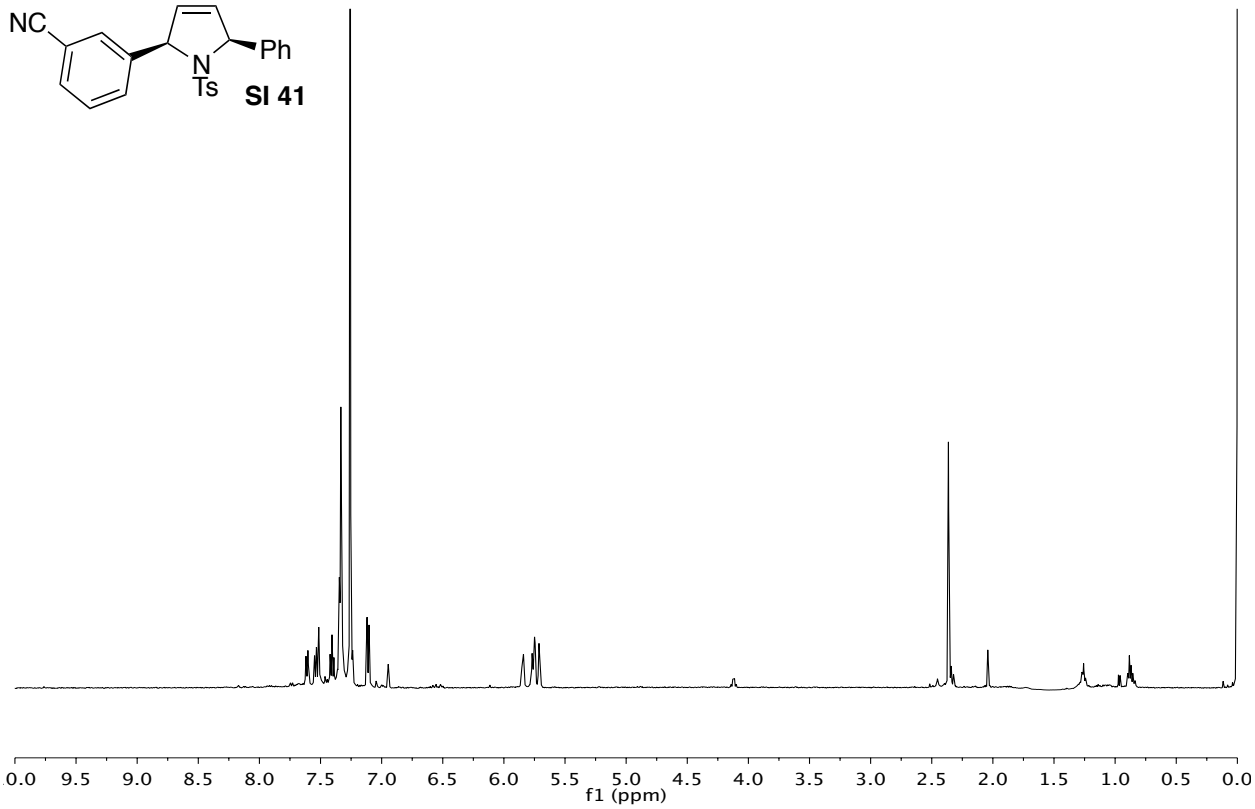
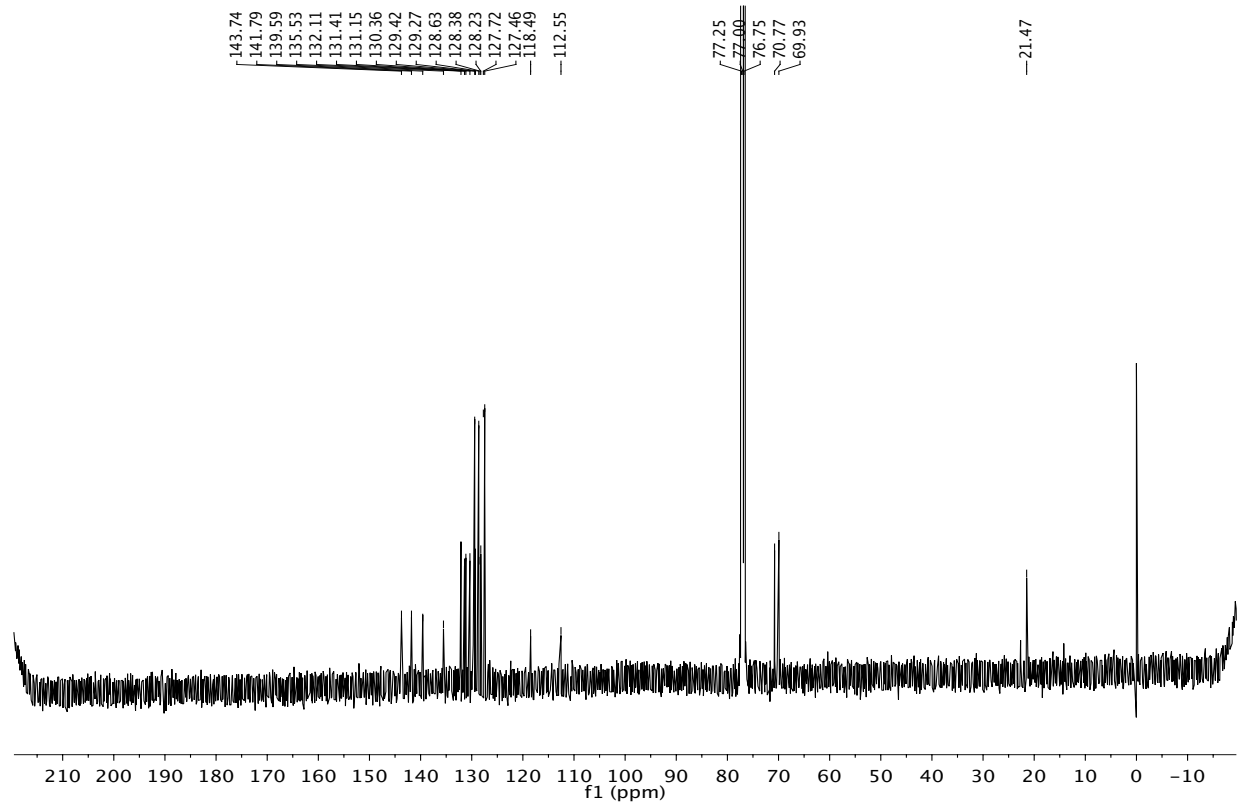
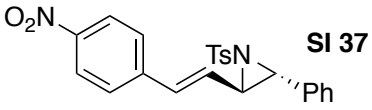
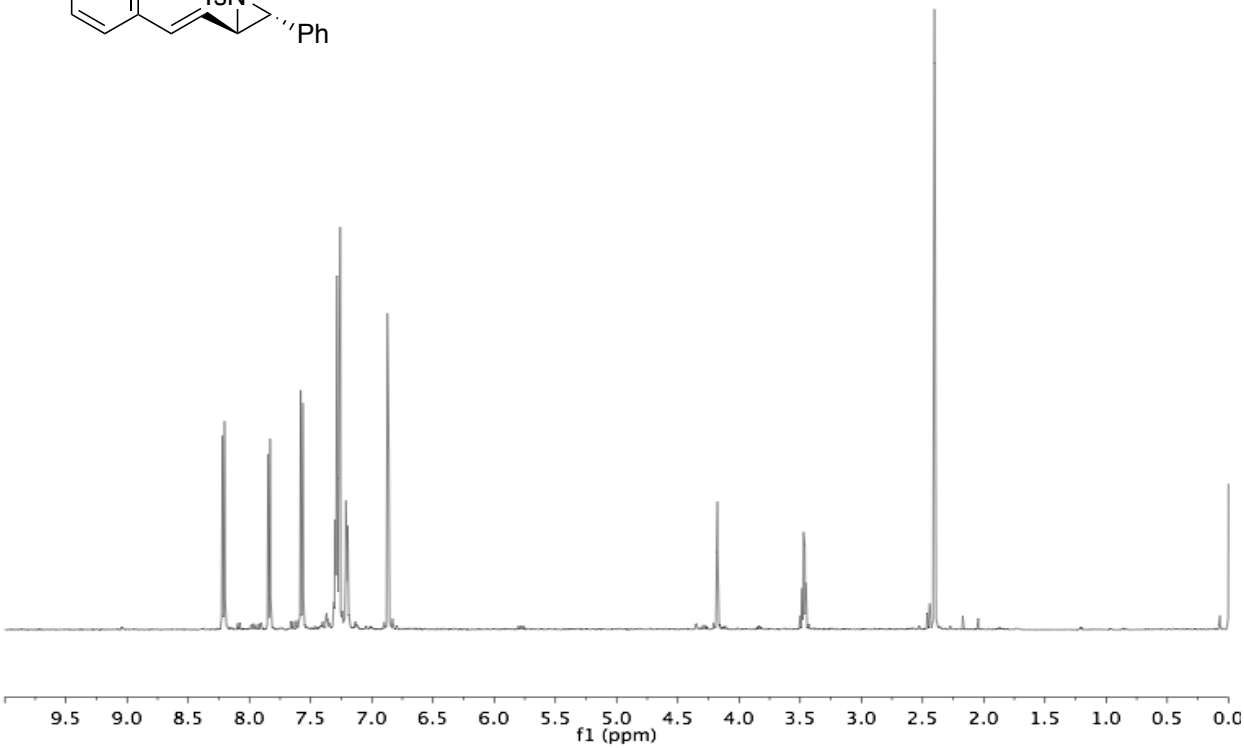
118.56
113.02
77.30
77.05
76.79
54.54
49.06
21.66
f1 (ppm)

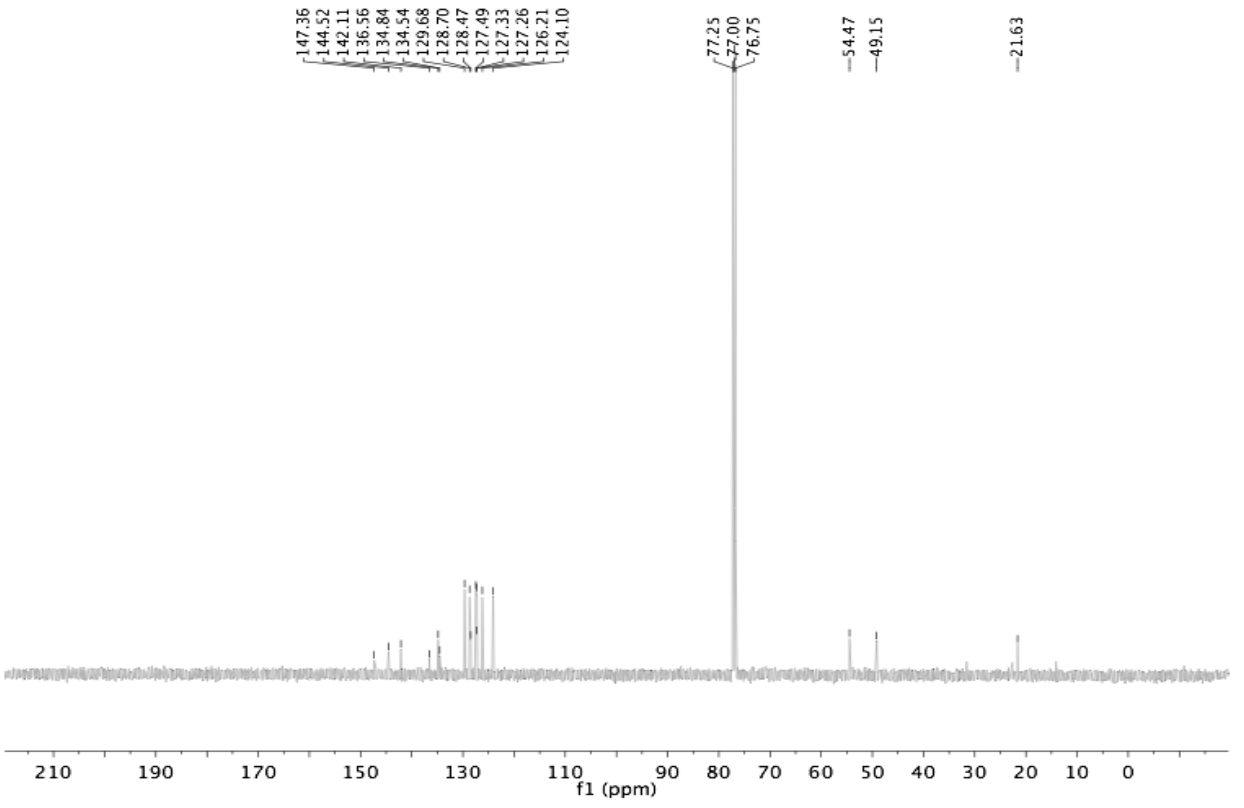


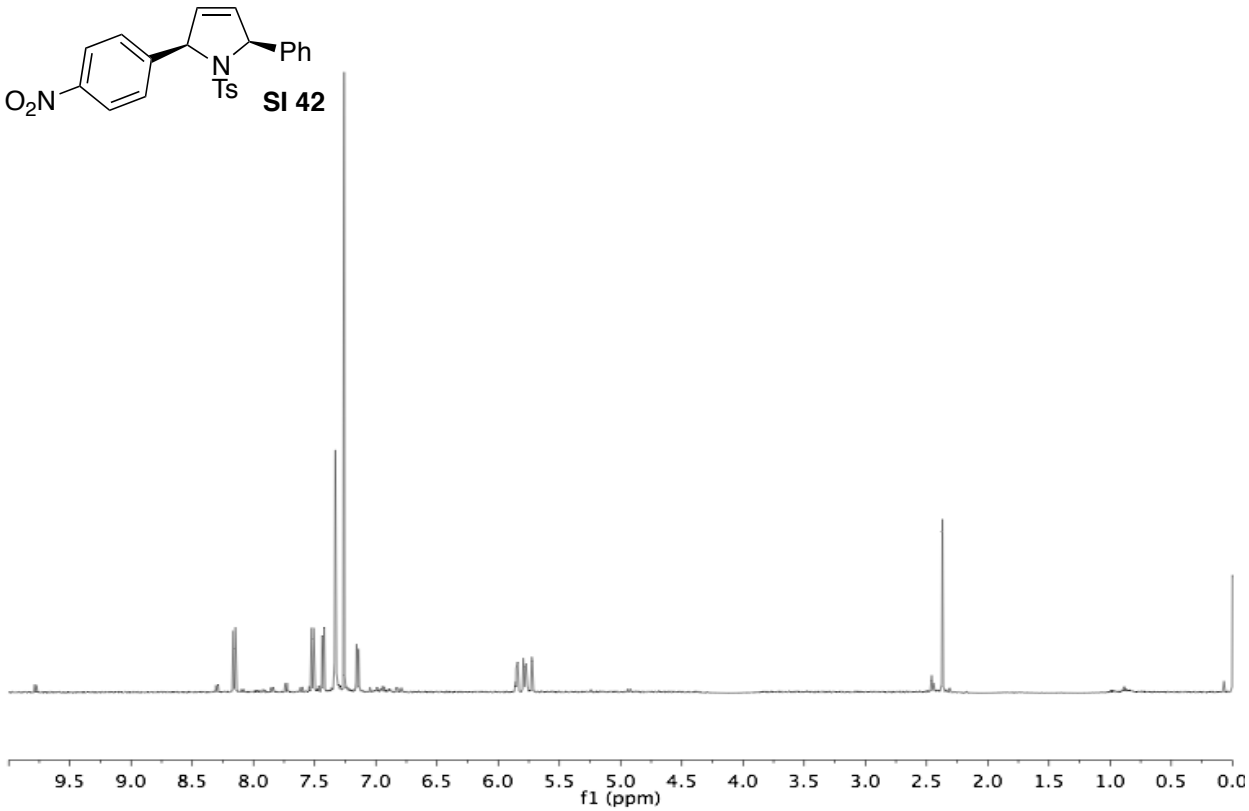


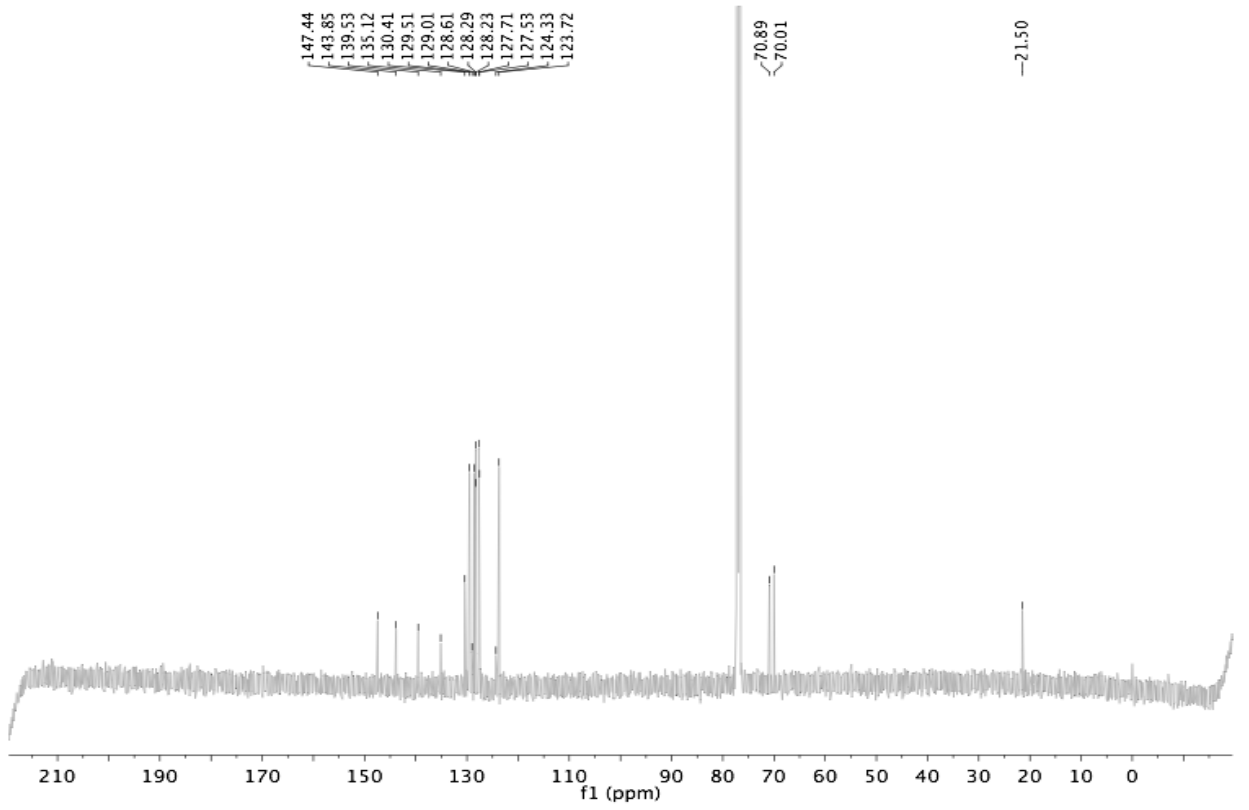


SI 37






O2N
N
Ts
Ph
SI 42
f1 (ppm)


147.44
143.85
139.53
135.12
130.41
129.51
129.01
128.61
128.29
128.23
127.71
127.53
124.33
123.72
70.89
70.01
21.50
f1 (ppm)