Cu(II)-Catalyzed [4 + 1] and [4 + 3] Annulation Reactions: A Modular Approach to N-Aryl/Alkyl Substituted 2,5-di-Amidopyrroles and Diazepines

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SUPPORTING INFORMATION

Table of Contents **Page**

General Experimental S2

Materials S2

Structure of primary amines **(1)**, ynamide-derived buta-1,3-diynes **(2)** and tentative reaction pathway S3

Optimization Reaction, Experimental Procedures, Spectral, Analytical data S4-S30

References S30

Spectra S31-S110

General Experimental

All the reactions were performed in an oven-dried Schlenk flask under an argon atmosphere. Unless otherwise noted, all the reagents and intermediates were obtained commercially and used without purification. Dichloromethane (CH_2Cl_2), acetonitrile, ethyl acetate, acetone and dichloroethane (DCE) were distilled over CaH_2. THF, toluene, 1,4-dioxane was freshly distilled over sodium/benzophenone ketyl under dry nitrogen. TMEDA was distilled over KOH. Column chromatography was performed using silica gel (100-200 Mesh) eluting with hexanes and ethyl acetate mixture. Flash column chromatography was performed using silica gel (230-400 Mesh) eluting with hexanes and ethyl acetate mixture. Thin layer chromatography (TLC) was performed on silica gel GF254 plates. Visualization of spots on TLC plate was accomplished with UV light (254 nm) and staining over I_2 chamber or an aqueous alkaline $KMnO_4$ solution followed by heating.

Proton and carbon nuclear magnetic resonance spectra (^{1}H NMR, ^{13}C NMR and ^{19}F NMR) were recorded on a Bruker Avance 400 (^{1}H NMR, 400 MHz; ^{13}C NMR, 101 MHz; ^{19}F NMR, 376 MHz) spectrometer or some cases on a Bruker Avance 500 (^{1}H NMR, 500 MHz; ^{13}C NMR, 125 MHz) spectrometer, having solvent resonance as internal standard (^{1}H NMR, $CDCl_3$ at 7.26 ppm; ^{13}C NMR, $CDCl_3$ at 77.0 ppm). Few cases tetramethylsilane (TMS) at 0.00 ppm was used as reference standard. All the catalysts used in this reaction were procured directly from commercial sources. Data for ^{1}H NMR are reported as follows: chemical shift (ppm), multiplicity (s = singlet; br s = broad singlet; d = doublet; br d = broad doublet, t = triplet; br t = broad triplet; q = quartet; m = multiplet), coupling constants, *J*, in (Hz), and integration. Data for ^{13}C NMR, ^{19}F NMR were reported in terms of chemical shift (ppm). IR spectra were recorded on FT/IR-5300 spectrometer and reported in cm^{-1}. High resolution mass spectra were obtained in ESI mode. Melting points were determined by electro-thermal heating and are uncorrected.

All primary amines (**1a-1z**) were purchased from commercial source and used as it is for this reaction. However, ynamide-derived buta-1,3-diynes (**2a-2e**) were prepared following literature procedures.[1]

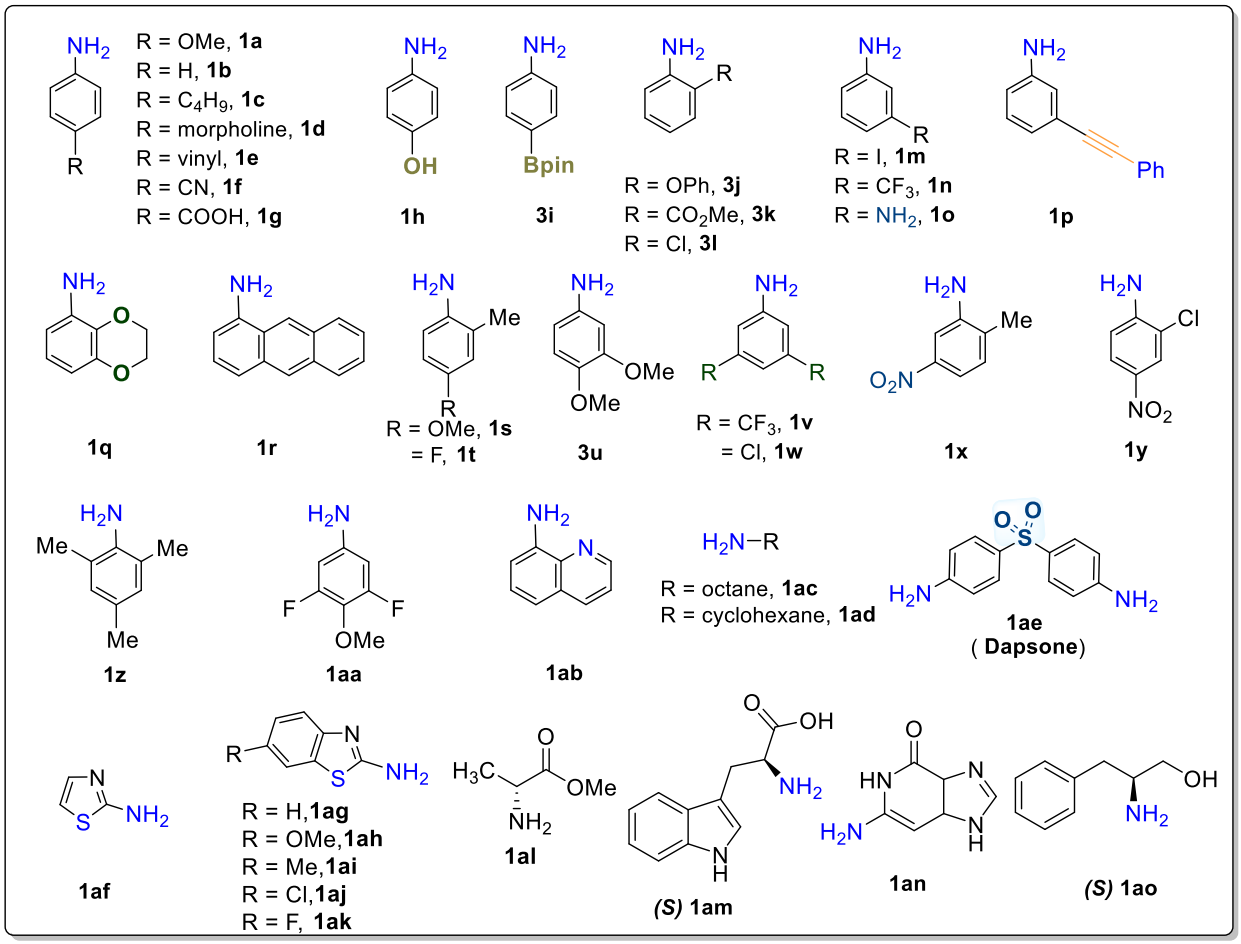


Figure S1. Different primary amines (**1**) employed in [4+1] and [4+3] annulation.

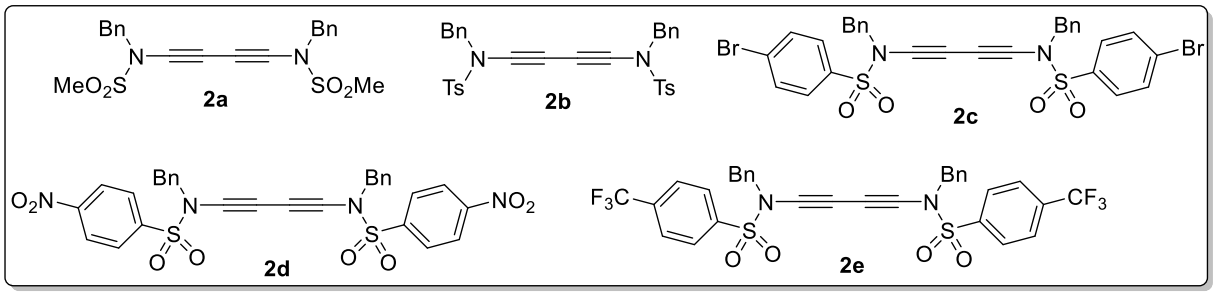
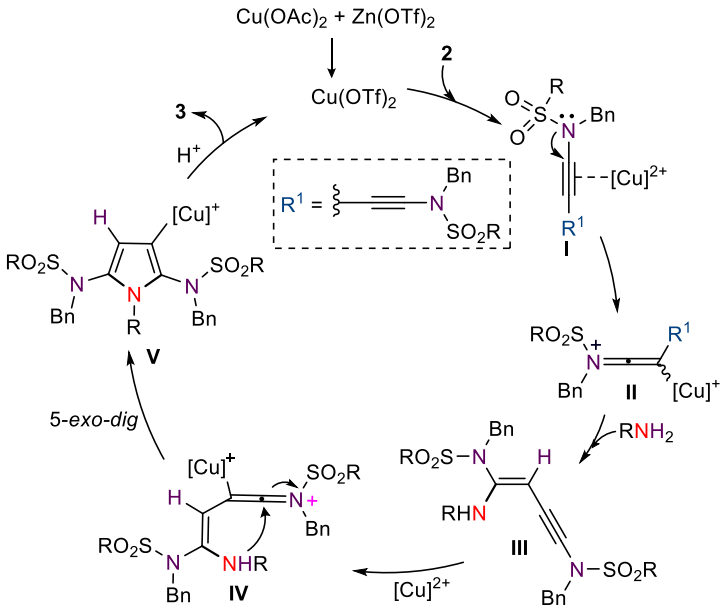
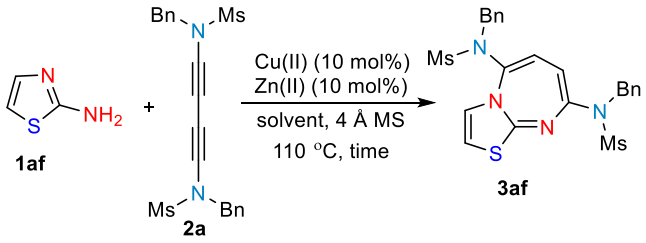


Figure S2. Different ynamide-derived buta-1,3-diynes (**2**) employed in [4+1] and [4+3] annulation.



Scheme S1. Tentative reaction pathway.

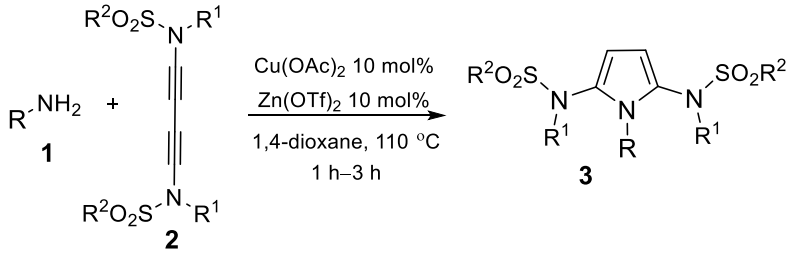
Table S1. Optimization of reaction conditions between 2-aminothiazole and **2a**[a]



Entry	Catalyst/Additive	Solvent/time (h)	Yield (%)[b]
1	$Cu(OTf)_2$ /$Zn(OTf)_2$	1,4-dioxane/3 h	20
2	$CuCl_2$ /$Zn(OTf)_2$	1,4-dioxane/3 h	Trace
3	$Cu(OAc)_2$/Zn Dust	1,4-dioxane/12 h	10
4	$Cu(OAc)_2$/$ZnCl_2$	1,4-dioxane/12 h	28
5	$Cu(OAc)_2$/$Zn(OAc)_2$	1,4-dioxane/12 h	26
6	$Cu(OAc)_2$ /$Zn(OTf)_2$	Toluene/3 h	15
7[c]	$Cu(OAc)_2$ /$Zn(OTf)_2$	THF/12 h	26
8	$Cu(OAc)_2$ /$Zn(OTf)_2$	1,2-DCE/12 h	25

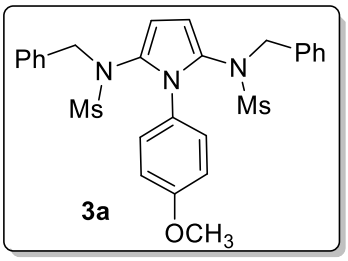
[a]Reaction conditions: **1af** (0.21 mmol), **2a** (0.19 mmol), solvent (2.0 mL), and 4 Å MS (15 mg) under argon. [b]^{1}H NMR yield of crude reaction mixture and 1,3,5-trimethoxybenzene as an internal standard. [c]Reaction temperature 80 °C.

General procedure for [4+1] annulation (GP-1):



Initially required reagents such as **1** (0.21 mmol), **2** (0.19 mmol), $Cu(OAc)_2$ (0.019 mmol), and $Zn(OTf)_2$ (0.019 mmol) were taken in an oven-dried 15 mL Schlenk flask along with 4 Å MS (15 mg) and anhydrous 1,4-dioxane (2.0 mL) was introduced into the flask under argon atmosphere at room temperature. Upon stirring the reaction mixture for 5 min at RT, the Schlenk flask was placed in an oil bath at 110 °C and stirring continued for 1 h–3 h. The progress of the reaction was routinely monitored by TLC. The reaction mixture was cooled to room temperature after completion of reaction. Next, the solution was diluted with dichloromethane (5.0 mL), and filtered through a small pad of Celite. The filtrate was concentrated and the residue was purified through silica gel column chromatography to obtain **3**.

***N*,*N*'-(1-(4-Methoxyphenyl)-1*H*-pyrrole-2,5-diyl)bis(*N*-benzylmethansulfonamide) (3a)**

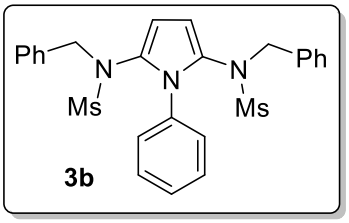


Following the general procedure GP-1, product **3a** (92 mg) was obtained in 88% yield as yellow solid; mp = 211–213 °C; R_f = 0.41 (7:3 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.32–7.23 (m, 8H), 7.09–7.07 (m, 4H), 6.70 (d, J = 7.2 Hz, 2H), 6.03 (s, 2H), 4.34 (s, 4H), 3.82 (s, 3H), 2.75 (s, 6H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 159.4, 135.0, 130.5, 129.7, 128.5, 128.2, 127.4, 127.3, 113.4, 106.0, 56.2, 55.3, 39.2; **IR (Neat)** ν_{max} 3402, 2926, 1330, 1153, 830 cm^{-1}; HRMS (ESI) for $C_{27}H_{30}N_3O_5S_2$ $(M+H)^+$: calcd 540.1627, found 540.1630.

In scale-up reaction, a mixture of **1a** (1.3 mmol, 162 mg), **2a** (1.2 mmol, 500 mg), $Zn(OTf)_2$ (0.12 mmol, 44 mg), $Cu(OAc)_2$ (0.12 mmol, 22 mg) and 4 Å MS (50 mg) was stirred in

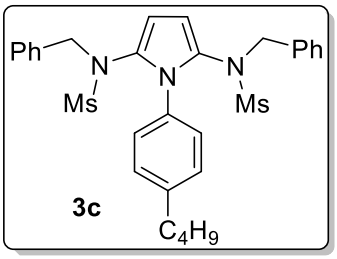
anhydrous 1,4-dioxane (7.0 mL). After completion, the crude residue was purified to obtain pure **3a** (587 mg) in 90% yield.

***N*,*N*'-(1-Phenyl-1*H*-pyrrole-2,5-diyl)bis(*N*-benzylmethanesulfonamide) (3b)**



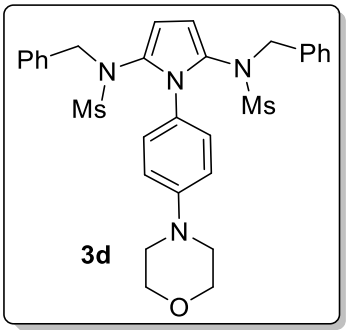
Following the general procedure GP-1, product **3b** (92 mg) was obtained in 94% yield as pale yellow solid; mp = 160–162 °C; R_f = 0.47 (3:2 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (500 MHz, $CDCl_3$)** δ 7.31– 7.18 (m, 10H), 7.05 (d, J = 7.0 Hz, 4H), 6.84 (s, 1H), 6.03 (s, 2H), 4.32 (s, 4H), 2.72 (s, 6H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 134.9, 134.8, 129.7, 129.4, 128.5, 128.4, 128.3, 127.2, 106.2, 56.3, 39.3; **IR (Neat)** ν_{max} 3394, 2931, 1335, 1154, 757 cm^{-1}; HRMS (ESI) for $C_{26}H_{28}N_3O_4S_2$ $(M+H)^+$: calcd 510.1521, found 510.1517.

***N*,*N*'-(1-(4-Butylphenyl)-1*H*-pyrrole-2,5-diyl)bis(*N*-benzylmethanesulfonamide) (3c)**



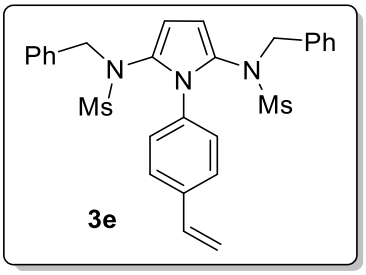
Following the general procedure GP-1, product **3c** (96 mg) was obtained in 89% yield as colorless solid ; mp = 177–179 °C; R_f = 0.46 (1:1 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (500 MHz, $CDCl_3$)** δ 7.30–7.22 (m, 7H), 7.07 (d, J = 7.0 Hz, 4H), 7.01 (d, J = 8.0 Hz, 2H), 6.77 (brs, 1H), 6.01 (s, 2H), 4.33 (s, 4H), 2.71 (s, 6H), 2.61 (t, J = 7.5 Hz, 2H), 1.64–1.58 (m, 2H), 1.41–1.35 (m, 2H), 0.96 (t, J = 7.5 Hz, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 143.3, 135.0, 132.3, 129.7, 129.1, 128.5, 128.3, 128.2, 127.1, 106.2, 56.2 , 39.4, 35.2, 33.4, 22.4, 13.9; **IR (Neat)** ν_{max} 3393, 2926, 1607, 1383, 1055, 767 cm^{-1}; HRMS (ESI) for $C_{30}H_{36}N_3O_4S_2$ $(M+H)^+$: calcd 566.2147, found 566.2146.

***N*,*N*'-(1-(4-Morpholinophenyl)-1*H*-pyrrole-2,5-diyl)bis(*N*-benzylmethanesulfonamide) (3d)**



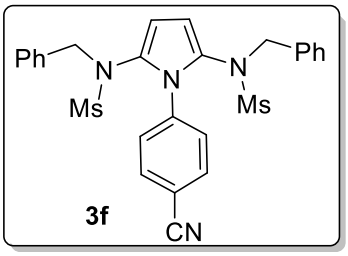
Following the general procedure GP-1, product **3d** (64 mg) was obtained in 65% yield as orange solid; mp = 184–186 °C; R_f = 0.41 (2:3 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.29–7.27 (m, 4H), 7.26–7.23 (m, 4H), 7.09 (dd, J = 1.6, 8.0 Hz, 4H), 6.70 (d, J = 8.4 Hz, 2H), 5.99 (s, 2H), 4.34 (s, 4H), 3.88 (t, J = 4.8 Hz, 4H), 3.19 (t, J = 4.8 Hz, 4H), 2.74 (s, 6H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 150.9, 135.1, 130.0, 129.7, 128.5, 128.2, 127.2, 126.3, 114.5, 106.0, 66.8, 56.1, 48.7, 39.5; **IR (Neat)** ν_{max} 3393, 2925, 1758, 1518, 1338, 1152, 1059, 758 cm^{-1}; HRMS (ESI) for $C_{30}H_{35}N_4O_5S_2$ $(M+H)^+$: calcd 595.2049, found 595.2048.

***N*,*N*'-(1-(4-Vinylphenyl)-1*H*-pyrrole-2,5-diyl)bis(*N*-benzylmethanesulfonamide) (3e)**



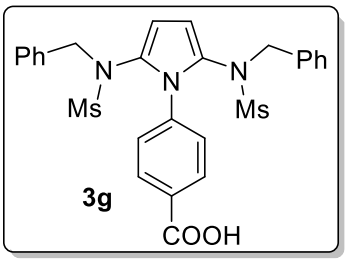
Following the general procedure GP-1, product **3e** (70 mg) was obtained in 68% yield as pale yellow solid; mp = 236–238 °C; R_f = 0.41 (7:3 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (500 MHz, $CDCl_3$)** δ 7.30–7.22 (m, 9H), 7.06 (d, J = 7.0 Hz, 4H), 6.81 (brs, 1H), 6.69 (dd, J = 7.0 Hz, 1H), 6.05 (s, 2H), 5.76 (d, J = 17.5, 1H), 5.31 (d, J = 10.5, 1H), 4.33 (s, 4H), 2.76 (s, 6H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 137.5, 136.0, 134.9, 134.1, 129.6, 129.4, 128.5, 128.2, 127.2, 126.0, 115.0, 106.1, 56.3, 39.3; **IR (Neat)** ν_{max} 3386, 2924, 1624, 1383, 1062, 693 cm^{-1}; HRMS (ESI) for $C_{28}H_{30}N_3O_4S_2$ $(M+H)^+$: calcd 536.1678, found 536.1668.

***N*,*N*'-(1-(4-Cyanophenyl)-1*H*-pyrrole-2,5-diyl)bis(*N*-benzylmethanesulfonamide) (3f)**



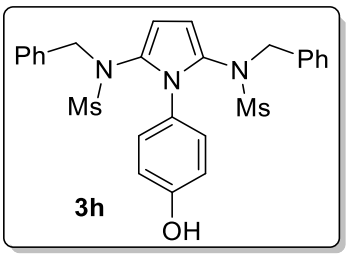
Following the general procedure GP-1, product **3f** (85 mg) was obtained in 83% yield as pale yellow solid; mp = 259–261 °C; R_f = 0.40 (EtOAc); [Silica, UV and I_2]; **^{1}H NMR (500 MHz, $CDCl_3$)** δ 7.29 (t, J = 9.5 Hz, 5H), 7.18 (t, J = 7.5 Hz, 4H), 6.93 (d, J = 7.0 Hz, 5H), 6.21 (s, 2H), 4.32 (brs, 4H), 2.91 (s, 6H); **^{13}C NMR (101 MHz, d_6-DMSO)** δ 139.0, 135.1, 131.6, 130.4, 129.7, 128.7, 128.3, 127.8, 118.9, 110.5, 106.4, 56.7, 39.4; **IR (Neat)** ν_{max} 3401, 2926, 1384, 1052, 765 cm^{-1}; HRMS (ESI) for $C_{27}H_{27}N_4O_4S_2$ $(M+H)^+$: calcd 535.1474, found 535.1466.

4-(2,5-Bis(*N*-benzylmethylsulfonamido)-1*H*-pyrrol-1-yl)benzoic acid (3g)



Following the general procedure GP-1, product **3g** (24 mg) was obtained in 92% yield as pale yellow solid; mp = 191–193 °C; R_f = 0.4 (EtOAc); [Silica, UV and I_2]; **^{1}H NMR (400 MHz, d_6-DMSO)** δ 13.01 (brs, 1H), 7.51 (d, J = 6.4 Hz, 2H), 7.27 (d, J = 7.2 Hz, 2H), 7.18 (t, J = 7.2 Hz, 5H), 6.88 (d, J = 7.2 Hz, 5H), 6.41 (s, 2H), 4.35 (brs, 4H), 3.01 (s, 6H); **^{13}C NMR (101 MHz, d_6-DMSO)** δ 167.3, 138.9, 135.3, 130.2, 129.8, 128.7, 128.6, 128.3, 127.6, 106.3, 56.4, 37.8; **IR (Neat)** ν_{max} 3388, 2923, 1617, 1063, 767 cm^{-1}; HRMS (ESI) for $C_{27}H_{31}N_4O_6S_2$ $(M+NH_4)^+$: calcd 571.1685, found 571.1686.

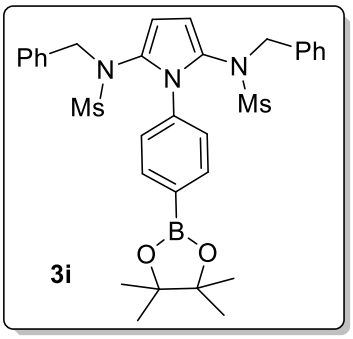
***N*,*N*'-(1-(4-Hydroxyphenyl)-1*H*-pyrrole-2,5-diyl)bis(*N*-benzylmethanesulfonamide) (3h)**



Following the general procedure GP-1, product **3h** (101 mg) was obtained in 82% yield as brown solid; mp = 135–137 °C; R_f = 0.41 (3:2 hexane/Acetone); [Silica, UV and I_2]; **^{1}H NMR (400 MHz, DMSO-d6)** δ 9.56 (s, 1H), 7.31–7.22 (m, 8H), 6.97 (d, J = 6.8 Hz, 4H), 6.46 (s,

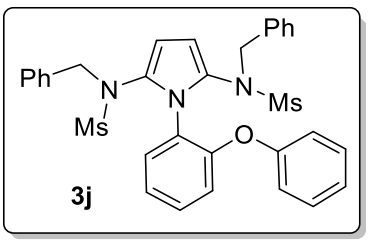
2H), 6.14 (s, 2H), 4.32 (s, 4H), 2.89 (s, 6H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 157.4, 135.7, 130.9, 129.8, 128.6, 128.3, 127.2, 126.2, 114.5, 106.1, 55.9, 38.5; **IR (Neat)** ν_{max} 3390, 2924, 1621, 1064, 768 cm^{-1}; HRMS (ESI) for $C_{26}H_{28}N_3O_5S_2$ $(M+H)^+$: calcd 526.1470, found 526.1472.

***N*,*N*'-(1-(4-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)-1*H*-pyrrole-2,5-diyl)bis(*N*-benzylmethanesulfonamide) (3i)**



Following the general procedure GP-1, product **3i** (75 mg) was obtained in 61% yield as yellow solid; mp = 114–116 °C; R_f = 0.44 (2:3 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (500 MHz, $CDCl_3$)** δ 7.66 (d, J = 7.5 Hz, 2H), 7.31–7.23 (m, 7H), 7.07 (q, J = 7.0 Hz, 5H), 6.03 (s, 2H), 4.32 (d, J = 6.5 Hz, 4H), 2.71 (s, 6H), 1.37 (s, 12H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 137.4, 134.9, 134.8, 129.7, 128.5, 128.4, 128.3, 128.2, 127.0, 106.4, 84.0, 56.1, 39.4, 24.9; **IR (Neat)** ν_{max} 3379, 2923, 1608, 1383, 1152, 1062, 757 cm^{-1}; HRMS (ESI) for $C_{32}H_{38}BN_3NaO_6S_2$ $(M+Na)^+$: calcd 658.2193, found 658.2193.

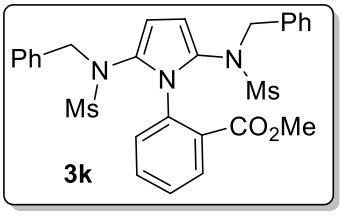
***N*,*N*'-(1-(2-Phenoxyphenyl)-1*H*-pyrrole-2,5-diyl)bis(*N*-benzylmethanesulfonamide) (3j)**



Following the general procedure GP-1, product **3j** (85 mg) was obtained in 73% yield as brown solid; mp = 200–202 °C; R_f = 0.49 (7:3 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (500 MHz, $CDCl_3$)** δ 7.34–7.25 (m, 14H), 7.14 (t, J = 7.5 Hz, 1H), 7.05 (d, J = 8.0 Hz, 3H), 6.81 (d, J = 8.0 Hz, 1H), 5.88 (s, 2H), 4.74 (d, J =14.4 Hz, 2H), 4.50 (d, J = 14.5 Hz, 2H), 2.61 (s, 6H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 154.8, 154.5, 135.7, 133.6, 130.5, 130.2, 129.9, 128.5, 128.3, 127.6, 125.0, 124.5, 122.1, 120.7, 115.3, 107.5, 55.6, 39.9; **IR (Neat)** ν_{max} 3391, 2924,

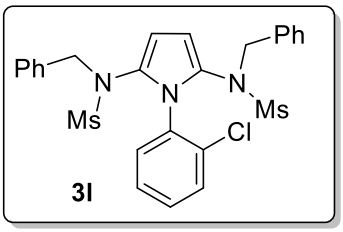
1623, 1384, 1063, 759 cm^{-1}; HRMS (ESI) for $C_{32}H_{32}N_3O_5S_2$ $(M+H)^+$: calcd 602.1783, found 602.1786.

Methyl 2-(2,5-bis(*N*-benzylmethylsulfonamido)-1*H*-pyrrol-1-yl) benzoate (3k)



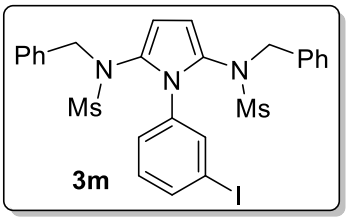
Following the general procedure GP-1, product **3k** (70 mg) was obtained in 63% yield as pale yellow solid; mp = 168–170 °C; R_f = 0.48 (7:3 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.96 (dd, J = 1.6, 7.6 Hz, 1H), 7.56–7.49 (m, 2H), 7.39 (s, 1H), 7.29–7.24 (m, 6H), 7.22–7.21 (m, 4H), 6.01 (s, 2H), 4.42 (q, J = 6.8Hz, 4H), 3.66 (s, 3H), 2.62 (s, 6H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 165.1, 135.6, 135.1, 133.5, 132.6, 130.9, 129.5, 129.3, 128.5, 128.1, 106.5, 55.7, 52.2, 39.6; **IR (Neat)** ν_{max} 3385, 2924, 1723, 1342, 1062, 762 cm^{-1}; HRMS (ESI) for $C_{28}H_{29}N_3NaO_6S_2$ $(M+Na)^+$: calcd 590.1395, found 590.1391.

N,N'-(1-(2-chlorophenyl)-1H-pyrrole-2,5-diyl)bis(N-benzylmethanesulfonamide) (3l)



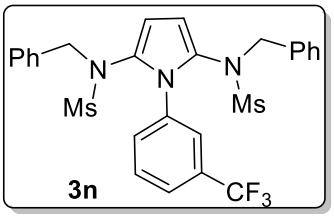
Following the general procedure GP-1, product **3l** (104 mg) was obtained in 71% yield as light brown solid; mp = 150–152 °C; R_f = 0.45 (7:3 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.46–7.45 (m, 1H), 7.45–7.44 (m, 1H), 7.42-7.35 (m, 2H), 7.33– 7.28 (m, 10 H), 5.95 (s, 2H), 4.64 (d, J = 14.7 Hz, 2H), 4.48 (d, J = 14.7 Hz, 2H), 2.57 (s, 6H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 135.6, 134.4, 133.5, 133.3, 130.9, 139.7, 129.5, 128.5, 128.3, 127.6, 127.5, 107.6, 55.4 (2C), 39.9 (2C); **IR (Neat)** ν_{max} 3402, 2926, 1829, 1135, 1028, 858 cm^{-1}; HRMS (ESI) for $C_{26}H_{27}ClN_3O_4S_2$ $(M+H)^+$: calcd 544.1132, found 544.1132.

***N,N*'-(1-(3-Iodophenyl)-1*H*-pyrrole-2,5-diyl)bis(*N*-benzylmethanesulfonamide) (3m)**



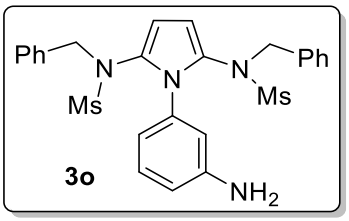
Following the general procedure GP-1, product **3m** (105 mg) was obtained in 86% yield as brown solid; mp = 224–226 °C; R_f = 0.48 (1:4 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.59 (dd, J = 1.6, 9.2 Hz, 1H), 7.32 (t, J = 7.2 Hz, 2H), 7.24 (t, J = 7.2 Hz, 5H), 6.98 (d, J = 7.2 Hz, 5H), 6.90 (t, J = 9.6 Hz, 1H), 6.13 (s, 2H), 4.33 (s, 4H), 2.81 (s, 6H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 137.3, 135.6, 134.3, 129.6, 129.5, 128.6, 128.5, 127.3, 106.1, 92.6, 56.6, 38.7; **IR (Neat)** ν_{max} 3396, 2926, 1330, 1154, 693 cm^{-1}; HRMS (ESI) for $C_{26}H_{27}IN_3O_4S_2$ $(M+H)^+$: calcd 636.0488, found 636.0480.

N,*N*'-(1-(3-(Trifluoromethyl)phenyl)-1*H*-pyrrole-2,5-diyl)bis(*N*-benzylmethanesulfonamide) (3n)



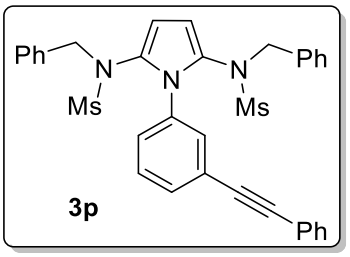
Following the general procedure GP-1, product **3n** (82 mg) was obtained in 73% yield as yellow solid; mp = 209–211 °C; R_f = 0.42 (7:3 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.50 (d, J = 8.0 Hz, 1H), 7.28–7.24 (m, 4H), 7.20–7.16 (m, 4H), 6.96–6.94 (m, 5H), 6.19 (s, 2H), 4.33 (s, 4H), 2.84 (s, 6H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 135.0, 134.2, 133.0, 130.5, 130.1, 129.5, 128.7, 128.6, 128.5, 127.5, 126.0, 124.9 (q, J = 3.0 Hz), 122.2, 106.1, 56.7, 38.5; **^{19}F NMR (376 MHz, $CDCl_3$)** δ – 62.1; **IR (Neat)** ν_{max} 3378, 2924, 1611, 1383, 1064, 771 cm^{-1}; HRMS (ESI) for $C_{27}H_{27}F_3N_3O_4S_2$ $(M+H)^+$: calcd 578.1395, found 578.1394.

N,*N*'-(1-(3-Aminophenyl)-1*H*-pyrrole-2,5-diyl)bis(*N*-benzylmethanesulfonamide) (3o)



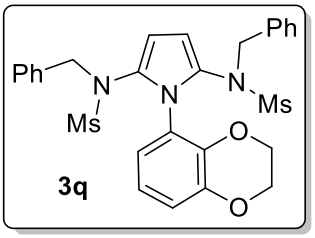
Following the general procedure GP-1, product **3o** (71 mg) was obtained in 71% yield as colorless solid; mp = 171–173 °C; R_f = 0.43 (EtOAc); [Silica, UV and I_2]; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.34–7.24 (m, 8H), 7.12–7.09 (m, 4H), 7.02 (t, J = 8.0 Hz, 1H), 6.61 (dd, J = 1.6, 7.6 Hz, 1H), 6.01 (s, 2H), 4.37 (s, 4H), 3.58 (s, 2H), 2.72 (s, 6H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 146.4, 135.7, 135.2, 129.7, 128.9, 128.5, 128.1, 126.9, 119.3, 116.0, 115.1, 106.3, 56.1, 39.5; **IR (Neat)** ν_{max} 3383, 2924, 1617, 1383, 1063, 765 cm^{-1}; HRMS (ESI) for $C_{26}H_{29}N_4O_4S_2$ $(M+H)^+$: calcd 525.1630, found 525.1627.

***N*,*N*'-(1-(3-(Phenylethynyl)phenyl)-1*H*-pyrrole-2,5-diyl)bis(*N*-benzylmethanesulfonamide) (3p)**



Following the general procedure GP-1, product **3p** (50 mg) was obtained in 43% yield as brown solid; mp = 179–181 °C; R_f = 0.39 (7:3 hexane/acetone); [Silica, UV and I_2]; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.58–7.55 (m, 2H), 7.45–7.42 (m, 2H), 7.40–7.36 (m, 3H), 7.30–7.27 (m, 4H), 7.25 (brs, 2H), 7.23–7.21 (m, 1H), 7.07–7.02 (m, 5H), 6.13 (s, 2H), 4.37 (s, 4H), 2.77 (s, 6H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 134.8, 134.6, 131.7, 131.6, 129.7, 129.5, 128.6, 128.5, 128.4, 128.3, 128.2, 128.1, 127.2, 123.2, 106.2, 90.2, 88.7, 56.5, 39.2, 38.8; **IR (Neat)** ν_{max} 3386, 2923, 1611, 1064, 760 cm^{-1}; HRMS (ESI) for $C_{34}H_{32}N_3O_4S_2$ $(M+H)^+$: calcd 610.1834, found 610.1832.

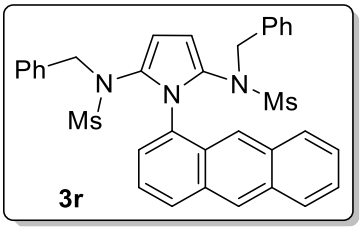
***N*,*N*'-(1-(2,3-Dihydrobenzo[b][1,4]dioxin-5-yl)-1*H*-pyrrole-2,5-diyl)bis(*N*-benzylmethanesulfonamide) (3q)**



Following the general procedure GP-1, product **3q** (81 mg) was obtained in 74% yield as colorless solid; mp = 157–159 °C; R_f = 0.48 (7:3 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR**

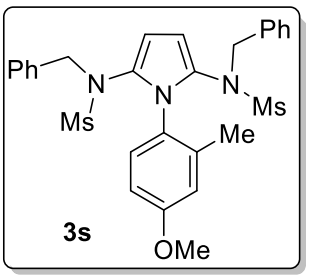
(500 MHz, $CDCl_3$) δ 7.29–7.25 (m, 7H), 7.09 (d, J = 6.5 Hz, 4H), 6.68 (d, J = 7.5 Hz, 1H), 6.41 (brs, 1H), 5.99 (s, 2H), 4.36 (s, 4H), 4.26 (d, J = 11.5 Hz, 4H), 2.76 (s, 6H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 143.8, 142.7, 135.0, 129.6, 128.5, 128.2, 127.8, 127.1, 116.5, 106.2, 64.3, 64.1, 56.2, 39.4; **IR (Neat)** ν_{max} 3378, 2926, 1598, 1338, 1153, 1063, 761 cm^{-1}; HRMS (ESI) for $C_{28}H_{30}N_3O_6S_2$ $(M+H)^+$: calcd 568.1576, found 568.1577.

***N*,*N*'-(1-(Anthracen-1-yl)-1*H*-pyrrole-2,5-diyl)bis(*N*-benzylmethanesulfonamide) (3r)**



Following the general procedure GP-1, product **3r** (77 mg) was obtained in 65% yield as brown solid; mp = 222–224 °C; R_f = 0.47 (1:4 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.41 (s, 1H), 8.22 (s, 1H), 8.03–7.99 (m, 2H), 7.82 (d, J = 9.2 Hz, 1H), 7.51–7.49 (m, 2H), 7.30–7.25 (m, 3H), 7.19–7.15 (m, 4H), 7.03–7.01 (m, 5H), 6.15 (s, 2H), 4.35 (s, 4H), 2.77 (s, 6H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 134.8, 132.2, 131.8, 131.7, 130.6, 130.5, 129.7, 128.5, 128.3, 128.2, 127.4, 127.3, 126.6, 126.0, 125.9, 125.7, 106.2, 56.4, 39.2; **IR (Neat)** ν_{max} 3390, 2924, 1614, 1384, 1153, 1062, 756 cm^{-1}; HRMS (ESI) for $C_{34}H_{32}N_3O_4S_2$ $(M+H)^+$: calcd 610.1834, found 610.1835.

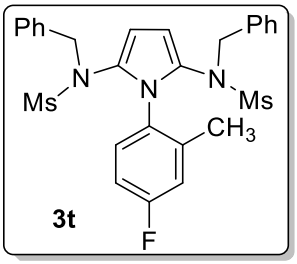
***N*,*N*'-(1-(4-Methoxy-2-methylphenyl)-1*H*-pyrrole-2,5-diyl)bis(*N*-benzylmethanesulfonamide) (3s)**



Following the general procedure GP-1, product **3s** (72 mg) was obtained in 67% yield as yellow solid; mp = 112–114 °C; R_f = 0.47 (3:2 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (500 MHz, $CDCl_3$)** δ 7.32–7.26 (m, 6H), 7.19 (dd, J = 1.5, 7.0 Hz, 4H), 7.05 (s, 1H), 6.72– 6.71 (m, 2H), 5.95 (s, 2H), 4.43 (q, J = 6.5 Hz, 4H), 3.84 (s, 3H), 2.60 (s, 6H), 1.87 (s, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 159.9, 135.4, 132.1, 129.5, 128.5, 128.2, 127.7, 126.9, 115.5,

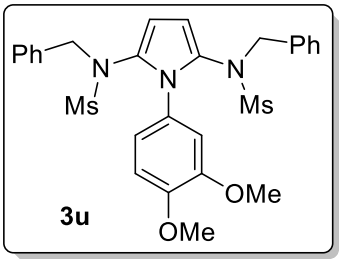
111.4, 106.8, 55.9, 55.4, 39.9, 18.1; **IR (Neat)** ν_{max} 3383, 2922, 1611, 1384, 1060, 767 cm^{-1}; HRMS (ESI) for $C_{28}H_{31}N_3NaO_5S_2$ $(M+Na)^+$: calcd 576.1603, found 576.1593.

_N,N'_-(1-(4-Fluoro-2-methylphenyl)-1_H_-pyrrole-2,5-diyl)bis(_N_-benzylmethanesulfonamide) (3t)



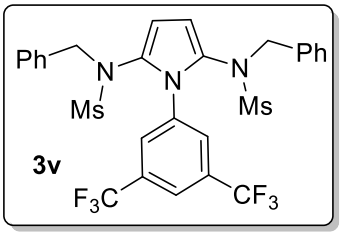
Following the general procedure GP-1, product **3t** (80 mg) was obtained in 77% yield as brown solid; mp = 147–149 °C; R_f = 0.43 (9:1 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.31–7.25 (m, 5H), 7.16 (dd, J = 1.6, 7.6 Hz, 4H), 7.01 (s, 1H), 6.86–6.84 (m, 3H), 6.02 (s, 2H), 4.43 (s, 4H), 2.64 (s, 6H), 1.78 (s, 3H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 162.6 (J = 246.5 Hz), 140.6 (J = 9.1 Hz), 135.2, 132.9 (J = 9.1 Hz), 130.0, 129.9, 129.4, 128.6, 128.4, 127.9, 116.9 (J = 22.2 Hz), 112.9 (J = 22.2 Hz), 106.8, 56.3, 39.5, 17.9; **^{19}F NMR (376 MHz, $CDCl_3$)** δ – 112.4; **IR (Neat)** ν_{max} 3393, 2927, 1501, 1342, 1154, 1038, 756 cm^{-1}; HRMS (ESI) for $C_{27}H_{29}FN_3O_4S_2$ $(M+H)^+$: calcd 542.1584, found 542.1575.

_N,N'_-(1-(3,4-Dimethoxyphenyl)-1_H_-pyrrole-2,5-diyl)bis(N-benzylmethanesulfonamide) (3u)



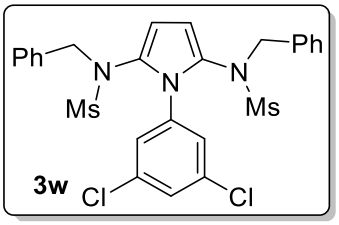
Following the general procedure GP-1, product **3u** (92 mg) was obtained in 84% yield as yellow solid; mp = 216–218 °C; R_f = 0.47 (1:1 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (500 MHz, $CDCl_3$)** δ 7.28–7.20 (m, 7H), 7.04 (d, J = 7.0 Hz, 4H), 6.75 (s, 1H), 6.68 (d, J = 8.0 Hz, 1H), 6.03 (s, 2H), 4.31 (s, 4H), 3.91 (s, 3H), 3.76 (s, 3H), 2.83 (s, 6H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 148.7, 148.3, 134.9, 129.5, 128.4, 128.2, 127.4, 127.3, 120.9, 113.0, 110.0, 105.7, 56.2, 56.1, 55.9, 39.6; **IR (Neat)** ν_{max} 3392, 2928, 1514, 1339, 1153, 757 cm^{-1}; HRMS (ESI) for $C_{28}H_{32}N_3O_6S_2$ $(M+H)^+$: calcd 570.1733, found 570.1722.

***N*,*N*'-(1-(3,5-Bis(trifluoromethyl)phenyl)-1*H*-pyrrole-2,5-diyl)bis(*N*-benzylmethanesulfonamide) (3v)**



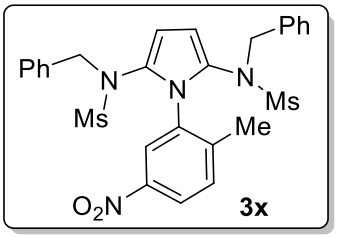
Following the general procedure GP-1, product **3v** (85 mg) was obtained in 68% yield as pale yellow solid; mp = 197–199 °C; R_f = 0.4 (1:1 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.65 (s, 1H), 7.21 (t, J = 7.6 Hz, 3H), 7.09 (t, J = 7.2 Hz, 5H), 6.82 (d, J = 6.4, 4H), 6.32 (s, 2H), 4.37 (brs, 4H), 2.97 (s, 6H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 135.6, 133.5, 131.1 (q, J = 27.3 Hz), 129.3, 128.7, 128.6, 127.7, 123.8, 121.7, 121.5, 106.1, 57.2, 37.8; **^{19}F NMR (376 MHz, $CDCl_3$)** δ – 62.4; **IR (Neat)** ν_{max} 3390, 2924, 1621, 1383, 1153, 1065, 697 cm^{-1}; HRMS (ESI) for $C_{28}H_{25}F_6N_3NaO_4S_2$ $(M+Na)^+$: calcd 668.1088, found 668.1089.

***N*,*N*'-(1-(3,5-Dichlorophenyl)-1*H*-pyrrole-2,5-diyl)bis(*N*-benzylmethanesulfonamide) (3w)**



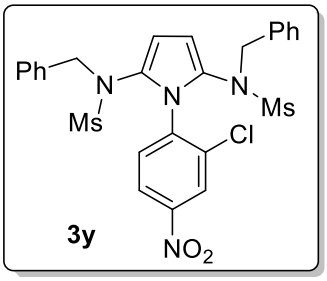
Following the general procedure GP-1, product **3w** (38 mg) was obtained in 34% yield as pale yellow solid; mp = 176–178 °C; R_f = 0.43 (7:3 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.34–7.28 (m, 4H), 7.20 (t, J = 8.0 Hz, 5H), 6.92 (d, J = 7.6 Hz, 4H), 6.22 (s, 2H), 4.35 (brs, 4H), 2.89 (s, 6H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 136.1, 134.0, 133.6, 129.5, 128.6, 128.4, 127.9, 127.4, 106.1, 56.9, 38.1; **IR (Neat)** ν_{max} 3385, 2923, 1622, 1383, 1156, 1060, 758 cm^{-1}; HRMS (ESI) for $C_{26}H_{26}Cl_2N_3O_4S_2$ $(M+H)^+$: calcd 578.0742, found 578.0735.

***N*,*N*'-(1-(2-Methyl-5-nitrophenyl)-1*H*-pyrrole-2,5-diyl)bis(*N*-benzylmethanesulfonamide) (3x)**



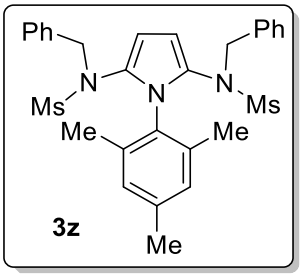
Following the general procedure GP-1, product **3x** (40 mg) was obtained in 36% yield as yellow solid; mp = 168–170 °C; R_f = 0.44 (1:4 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (500 MHz, $CDCl_3$)** δ 8.13 (dd, J = 2.0, 8.5 Hz, 1H), 7.77 (s, 1H), 7.32–7.25 (m, 7H), 7.13 (d, J = 7.0 Hz, 4H),, 6.14 (s, 2H), 4.45 (s, 4H), 2.71 (s, 6H), 1.84 (s, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 146.7, 145.6, 134.8, 134.7, 131.1, 129.3, 128.7, 128.6, 128.5, 126.8, 123.9, 107.0, 56.8, 38.8, 18.2; **IR (Neat)** ν_{max} 3391, 2923, 1622, 1384, 1063, 754 cm^{-1}; HRMS (ESI) for $C_{27}H_{29}N_4O_6S_2$ $(M+H)^+$: calcd 569.1529, found 569.1521.

***N*,*N*'-(1-(2-Chloro-4-nitrophenyl)-1*H*-pyrrole-2,5-diyl)bis(*N*-benzylmethanesulfonamide) (3y)**



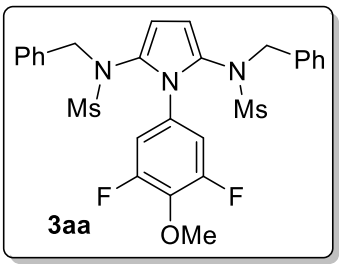
Following the general procedure GP-1, product **3y** (30 mg) was obtained in 26% yield as yellow solid; mp = 153–155 °C; R_f = 0.42 (3:2 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.17–8.12 (m, 2H), 7.35–7.25 (m, 12H), 6.09 (s, 2H), 4.64 (d, J=14.8 Hz, 2H), 4.50 (d, J= 14.8 Hz, 2H), 2.66 (s, 6H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 148.3, 138.9, 135.7, 135.2, 135.1, 129.7, 128.7, 128.6, 128.2, 124.4, 121.9, 107.7, 56.1, 39.0; **IR (Neat)** ν_{max} 3778, 3399, 2925, 2346, 1619, 1384, 1063, 758 cm^{-1}; HRMS (ESI) for $C_{26}H_{26}ClN_4O_6S_2$ $(M+H)^+$: calcd 589.0982, found 589.0972.

***N*,*N*'-(1-Mesityl-1*H*-pyrrole-2,5-diyl)bis(*N*-benzylmethanesulfonamide) (3z)**



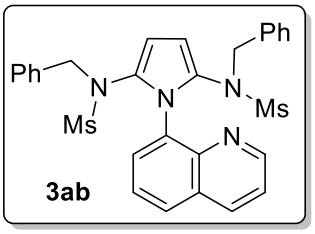
Following the general procedure GP-2, product **3z** (96 mg) was obtained in 90% yield as yellow solid; mp = 174–176 °C; R_f = 0.42 (3:2 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (500 MHz, $CDCl_3$)** δ 7.28–7.25 (m, 6H), 7.23– 7.22 (m, 4H), 6.96 (s, 2H), 5.97 (s, 2H), 4.44 (s, 4H), 2.49 (s, 6H), 2.36 (s, 3H), 2.13 (s, 6H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 139.2, 137.9, 135.6, 131.1, 129.5, 129.2, 128.5, 128.2, 127.9, 107.4, 55.5, 41.0, 21.1, 18.9; **IR (Neat)** ν_{max} 3387, 2925, 1615, 1342, 1154, 1060, 759 cm^{-1}; HRMS (ESI) for $C_{29}H_{34}N_3O_4S_2$ $(M+H)^+$: calcd 552.1991, found 552.1979.

_N,N'_-(1-(3,5-Difluoro-4-methoxyphenyl)-1_H_-pyrrole-2,5-diyl)bis(_N_-benzylmethanesulfonamide) (3aa)



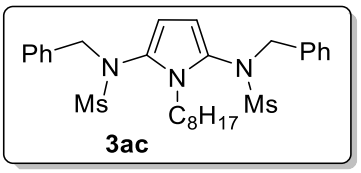
Following the general procedure GP-1, product **3aa** (76 mg) was obtained in 69% yield as brown solid; mp = 58–60 °C; R_f = 0.47 (7:3 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.31–7.28 (m, 3H), 7.21 (t, J = 7.7 Hz, 5H), 6.98 (d, J = 7.7 Hz, 4H), 6.16 (s, 2H), 4.36 (brs, 4H), 4.01 (s, 3H), 2.90 (s, 6H); **^{13}C NMR (101 MHz, d_6-DMSO)** δ 153.3 (d, J = 245.4 Hz), 153.2 (d, J = 245.4 Hz), 135.6, 135.5 (d, J = 28.5 Hz), 135.3, 129.6, 129.4 (d, J = 26.6 Hz), 129.3, 128.6, 128.2, 127.9, 114.1 (d, J = 18.2 Hz), 106.1, 62.1, 56.8, 37.4; **^{19}F NMR (376 MHz, $CDCl_3$)** δ –128.5; **IR (Neat)** ν_{max} 3391, 2923, 1622, 1384, 1063, 754 cm^{-1}; HRMS (ESI) for $C_{27}H_{27}F_2N_3NaO_5S_2$ $(M+Na)^+$: calcd 598.1258, found 598.1259.

_N,N'_-(1-(Quinolin-8-yl)-1_H_-pyrrole-2,5-diyl)bis(_N_-benzylmethanesulfonamide) (3ab)



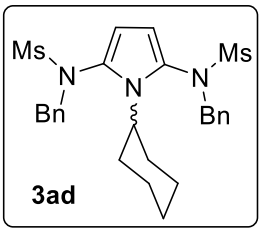
Following the general procedure GP-1, product **3ab** (63 mg) was obtained in 59% yield as pale-yellow solid; mp = 75–77 °C; R_f = 0.44 (7:3 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.77 (dd, J = 1.5, 4.1 Hz, 1H), 8.22 (dd, J = 1.5, 8.2 Hz, 1H), 7.97–7.95 (m, 1H), 7.67 (t, J =15.6 Hz, 1H), 7.39–7.36 (m, 1H), 7.26–7.21 (m, 11H), 6.02 (s, 2H), 4.29 (q, J = 13.0 Hz, 4H), 2.59 (s, 6H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 150.7, 145.4, 136.4, 135.9, 133.9, 132.8, 129.7, 129.6, 128.7, 128.6, 128.3, 128.0, 126.4, 121.7, 107.0, 55.2, 39.9; **IR (Neat)** ν_{max} 3386, 2924, 1620, 1384, 1153, 1062, 756 cm^{-1}; HRMS (ESI) for $C_{29}H_{29}N_4O_4S_2$ $(M+H)^+$: calcd 561.1630, found 561.1630.

***N*,*N*'-(1-Octyl-1*H*-pyrrole-2,5-diyl)bis(*N*-benzylmethanesulfonamide) (3ac)**



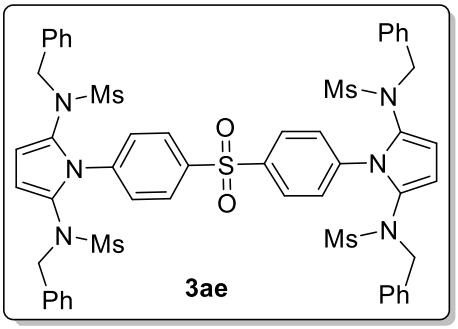
Following the general procedure GP-1, product **3ac** (89 mg) was obtained in 85% yield as yellow solid after 2 h; mp = 107–109 °C; R_f = 0.41 (7:3 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (500 MHz, $CDCl_3$)** δ 7.30–7.28 (m, 6H), 7.25–7.21 (m, 4H), 5.94 (s, 2H), 4.58 (s, 4H), 3.55 (s, 1H), 2.82 (s, 6H), 1.6 (s, 1H), 1.29–1.20 (m, 8H), 1.04 (brs, 4H), 0.89 (t, J = 7.0 Hz, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 135.2, 129.9, 128.6, 128.5, 126.4, 105.1, 56.8, 43.0, 31.8, 29.8, 29.2, 29.0, 27.3, 22.6, 14.1; **IR (Neat)** ν_{max} 3391, 2925, 1457, 1329, 1154, 1056, 698 cm^{-1}; HRMS (ESI) for $C_{28}H_{40}N_3O_4S_2$ $(M+H)^+$: calcd 546.2460, found 546.2460.

***N*,*N*'-(1-Cyclohexyl-1*H*-pyrrole-2,5-diyl)bis(*N*-benzylmethanesulfonamide) (3ad)**



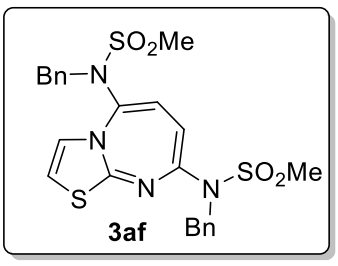
Following the general procedure GP-1, an inseparable isomeric mixture of product **3ad** (57 mg) was obtained in 58% yield as pale yellow solid after 2 h; mp = 154–156 °C; R_f = 0.49 (3:2 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.34–7.31 (m, 8H), 7.28–7.26 (m, 2H), 5.90 (d, J = 4.8 Hz, 2H), 4.66–4.59 (m, 4H), 3.93–3.88 (m, 1H), 2.80 (s, 3H), 2.76 (s, 3H), 1.95–1.88 (m, 1H), 1.73 (s, 1H), 1.66–1.63 (m, 2H), 1.32–1.29 (m, 2H), 1.12–1.08 (s, 3H), 0.93–0.87 (m, 1H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 135.0, 134.9, 130.3, 130.2, 128.7, 128.6, 128.5, 126.7, 126.2, 105.8, 105.6, 57.5, 57.3, 56.6, 56.5, 38.5, 38.0, 33.3, 33.1, 33.0, 30.9, 29.7, 27.0, 26.9, 25.5, 25.4; **IR (Neat)** ν_{max} 3381, 2928, 1338, 1153, 1062, 757 cm^{-1}; HRMS (ESI) for $C_{26}H_{34}N_3O_4S_2$ $(M+H)^+$: calcd 516.1991, found 516.1989.

***N,N'*-(1-(4-((4-(2,5-Bis(N-benzylmethylsulfonamido)-1*H*-pyrrol-1-yl)phenyl)sulfonyl)phenyl)-1*H*-pyrrole-2,5-diyl)bis(*N*-benzylmethanesulfonamide) (3ae)**



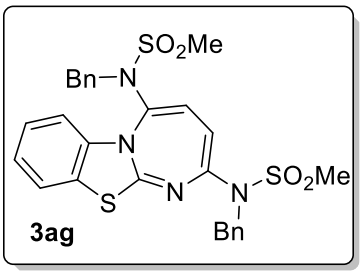
Following the general procedure GP-1, **1ae** (0.1, 24 mg) and **2a** (0.19 mmol, 80 mg) reacted to obtain product **3ae** (55 mg) in 53% yield as pale yellow solid; mp = 170–172 °C; R_f = 0.47 (1:4 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (400 MHz, d_6-DMSO)** δ 7.49 (s, 5H), 7.23 (t, J = 6.8 Hz, 5H), 7.05 (brs, 9H), 6.77 (brs, 9H), 6.50 (s, 4H), 4.32 (brs, 8H), 3.05 (s, 12H);**^{13}C NMR (101 MHz, d_6-DMSO)** δ 139.6, 139.5, 135.0, 130.2, 129.6, 128.7, 128.3, 127.8, 126.9, 106.5, 56.7, 37.4; **IR (Neat)** ν_{max} 3395, 2924, 1384, 1066, 765 cm^{-1}; HRMS (ESI) for $C_{52}H_{52}N_6NaO_{10}S_5$ $(M+Na)^+$: calcd 1103.2246, found 1103.2257.

***N,N'*-(Thiazolo[3,2-*a*][1,3]diazepine-5,8-diyl)bis(*N*-benzylmethanesulfonamide) (3af)**



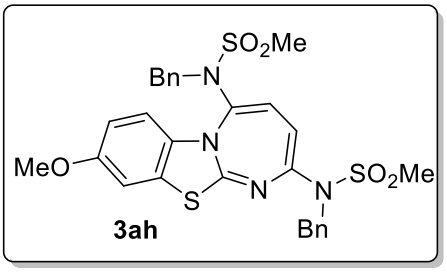
Following the general procedure GP-1, **1af** (0.48 mmol, 48 mg), **2a** (0.24 mmol, 100 mg), $Cu(OAc)_2$ (0.024 mmol, 4.3 mg), $Zn(OTf)_2$ (0.024 mmol, 8.7 mg) and 4 Å MS (15 mg) were used in 1,4-dioxane (3 mL) and product **3af** (33 mg) was obtained in 26% yield as brown solid; mp = 63–65 °C; R_f = 0.43 (2:3 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.68 (d, J = 14.0 Hz, 1H), 7.37–7.25 (m, 8H), 7.13 (d, J = 7.2 Hz, 2H), 6.84 (d, J = 4.4 Hz, 1H), 6.53 (d, J = 4.8 Hz, 1H), 5.13 (d, J = 14.0 Hz, 1H), 5.00 (d, J = 16.4 Hz, 1H), 4.65 (d, J = 14.0 Hz, 1H), 4.45 (d, J = 16.4 Hz, 1H), 4.32 (d, J = 14.0 Hz, 1H), 3.06 (s, 3H), 2.37 (s, 3H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 149.5, 140.6, 135.9, 135.3, 129.3, 129.0, 128.8, 128.5, 128.3, 127.8, 126.4, 117.7, 116.8, 111.6, 100.8, 54.9, 49.6, 39.9, 39.0; **IR (Neat)** ν_{max} 3618, 3016, 1452, 1205, 1024, 910 cm^{-1}; HRMS (ESI) for $C_{23}H_{25}N_4O_4S_3$ $(M+H)^+$: calcd 517.1038, found 517.1038.

N,*N*'-(Benzo[4,5]thiazolo[3,2-a][1,3]diazepine-2,5-diyl)bis(*N*-benzylmethanesulfonamide) (3ag)



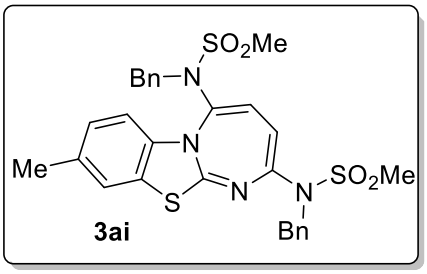
Following the general procedure GP-1, **1ag** (0.48 mmol, 72 mg), **2a** (0.24 mmol, 100 mg), $Cu(OAc)_2$ (0.024 mmol, 4.3 mg), $Zn(OTf)_2$ (0.024 mmol, 8.7 mg) and 4 Å MS (15 mg) were used in 1,4-dioxane (3 mL) and product **3ag** (39 mg) was obtained in 28% yield as brown solid; mp = 171–173 °C; R_f = 0.41 (2:3 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.73–7.67 (m, 2H), 7.60 (dd, J = 0.8, 8.0 Hz, 1H), 7.36–7.31 (m, 7H), 7.23 (d, J = 6.8 Hz, 1H), 7.20–7.16 (m, 4H), 5.00 (d, J = 16.4 Hz, 1H), 4.89 (d, J = 10.0 Hz, 1H), 4.86 (d, J = 9.6 Hz, 1H), 4.42 (d, J = 13.6 Hz, 1H), 4.17 (d, J = 16.4 Hz, 1H), 3.05 (s, 3H), 2.25 (s, 3H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 148.1, 141.2, 135.4, 135.3, 132.0, 129.9, 129.5, 129.0, 128.9, 128.7, 128.6, 127.7, 126.3, 126.2, 124.7, 123.8, 118.4, 113.6, 100.5, 55.3, 49.4, 40.1, 40.0; **IR (Neat)** ν_{max} 3348, 2970, 1466, 1408, 1305, 1160, 1128, 950 cm^{-1}; HRMS (ESI) for $C_{27}H_{27}N_4O_4S_3$ $(M+H)^+$: calcd 567.1194, found 567.1191.

N,*N*'-(9-Methoxybenzo[4,5]thiazolo[3,2-*a*][1,3]diazepine-2,5-diyl)bis(*N*-benzylmethanesulfonamide) (3ah)



Following the general procedure GP-1, **1ah** (0.48 mmol, 86 mg), **2a** (0.24 mmol, 100 mg), $Cu(OAc)_2$ (0.024 mmol, 4.3 mg), $Zn(OTf)_2$ (0.024 mmol, 8.7 mg) and 4 Å MS (15 mg) were used in 1,4-dioxane (3 mL) and product **3ah** (36 mg) was obtained in 25% yield as brown solid; mp = 172–174 °C; R_f = 0.41 (2:3 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.66 (d, J = 14.0 Hz, 1H), 7.55 (d, J = 8.8 Hz, 1H), 7.35–7.27 (m, 5H), 7.24–7.16 (m, 5H), 7.10 (d, J = 2.0 Hz, 1H), 6.88 (dd, J = 2.4, 8.8 Hz, 1H), 4.99 (d, J = 16.4 Hz, 1H), 4.88 (d, J = 14.0 Hz, 1H), 4.83 (d, J = 14.0 Hz, 1H), 4.43 (d, J = 14.0 Hz, 1H), 4.19 (d, J = 16.4 Hz, 1H), 3.82 (s, 3H), 3.05 (s, 3H), 2.25 (s, 3H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 156.9, 147.4, 140.5, 135.4, 130.8, 129.8, 129.0, 128.9, 128.6, 128.2, 127.7, 126.2, 114.2, 113.4, 108.3, 100.7, 55.9, 55.3, 49.4, 39.9; **IR (Neat)** ν_{max} 3343, 2970, 1408, 1304, 1160, 1127, 949 cm^{-1}; HRMS (ESI) for $C_{28}H_{29}N_4O_5S_3$ $(M+H)^+$: calcd 597.1300, found 597.1281.

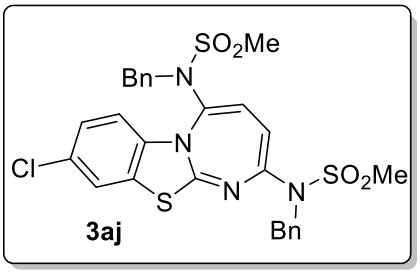
***N,N'*-(9-Methylbenzo[4,5]thiazolo[3,2-a][1,3]diazepine-2,5-diyl)bis(*N*-benzylmethanesulfonamide) (3ai)**



Following the general procedure GP-1, **1ai** (0.48 mmol, 79 mg), **2a** (0.24 mmol, 100 mg), $Cu(OAc)_2$ (0.024 mmol, 4.3 mg), $Zn(OTf)_2$ (0.024 mmol, 8.7 mg) and 4 Å MS (15 mg) were used in 1,4-dioxane (3 mL) and product **3ai** (58 mg) was obtained in 41% yield as brown solid; mp = 167–169 °C; R_f = 0.47 (1:4 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.69 (d, J = 14.0 Hz, 1H), 7.55 (d, J = 8.4 Hz, 1H), 7.39 (s, 1H), 7.35–7.28 (m, 5H), 7.23 (d, J = 6.8 Hz, 1H), 7.18 (t, J = 6.4 Hz, 4H), 7.13 (d, J = 8.4 Hz, 1H), 4.99 (d, J = 16.4 Hz, 1H), 4.88 (t, J = 14.0 Hz, 2H), 4.40 (d, J = 14.0 Hz, 1H), 4.16 (d, J = 16.4 Hz, 1H), 3.04 (s, 3H), 2.40 (s, 3H), 2.24 (s, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 147.9, 140.9, 135.4, 135.3, 134.8, 130.0, 129.9, 129.6, 129.0, 128.9, 128.6, 128.3, 127.7, 127.3, 126.2, 123.9, 118.3, 113.2,

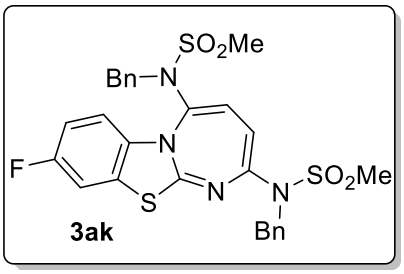
100.7, 55.3, 49.4, 40.0, 39.9, 21.3; **IR (Neat)** ν_{max} 3340, 2969, 1466, 1407, 1305, 1159, 1127, 1107, 949 cm^{-1}; HRMS (ESI) for $C_{28}H_{29}N_4O_4S_3$ $(M+H)^+$: calcd 581.1351, found 581.1350.

***N,N'*-(9-Chlorobenzo[4,5]thiazolo[3,2-a][1,3]diazepine-2,5-diyl)bis(*N*-benzylmethanesulfonamide) (3aj)**



Following the general procedure GP-1, **1aj** (0.48 mmol, 88 mg), **2a** (0.24 mmol, 100 mg), $Cu(OAc)_2$ (0.024 mmol, 4.3 mg), $Zn(OTf)_2$ (0.024 mmol, 8.7 mg) and 4 Å MS (15 mg) were used in 1,4-dioxane (3 mL) and product **3aj** (36 mg) was obtained in 25% yield as brown solid; mp = 184–186 °C; R_f = 0.41 (2:3 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.73 (d, J = 14.0 Hz, 1H), 7.56 (d, J = 2.0 Hz, 1H), 7.52 (d, J = 8.8 Hz, 1H), 7.34–7.20 (m, 9H), 7.14 (d, J = 8.0 Hz, 2H), 4.99 (d, J = 16.4 Hz, 1H), 4.92 (d, J = 14.0 Hz, 1H), 4.73 (d, J = 14.0 Hz, 1H), 4.47 (d, J = 14.0 Hz, 1H), 4.26 (d, J = 16.4 Hz, 1H), 3.07 (s, 3H), 2.27 (s, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 147.8, 141.2, 135.3, 135.1, 130.9, 130.5, 130.2, 129.8, 129.0, 128.9, 128.7, 127.8, 126.6, 126.2, 123.4, 118.5, 114.3, 100.2, 55.4, 49.5, 40.0, 39.8; **IR (Neat)** ν_{max} 3345, 2970, 1647, 1466, 1379, 1304, 1160, 1127, 1106, 949 cm^{-1}; HRMS (ESI) for $C_{27}H_{26}ClN_4O_4S_3$ $(M+H)^+$: calcd 601.0805, found 601.0803.

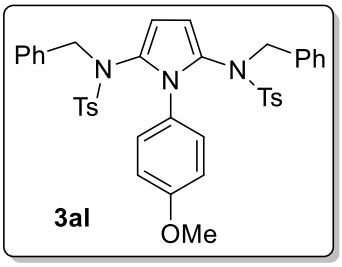
***N,N'*-(9-Fluorobenzo[4,5]thiazolo[3,2-a][1,3]diazepine-2,5-diyl)bis(*N*-benzylmethanesulfonamide) (3ak)**



Following the general procedure GP-1, **1ak** (0.48 mmol, 81 mg), **2a** (0.24 mmol, 100 mg), $Cu(OAc)_2$ (0.024 mmol, 4.3 mg), $Zn(OTf)_2$ (0.024 mmol, 8.7 mg) and 4 Å MS (15 mg) were used in 1,4-dioxane (3 mL) and, product **3ak** (30 mg) was obtained in 21% yield as brown solid; mp = 191–193 °C; R_f = 0.43 (2:3 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.65 (d, J = 14.0 Hz, 1H), 7.48 (dd, J = 4.4, 8.8 Hz, 1H), 7.33–7.23 (m, 3H),

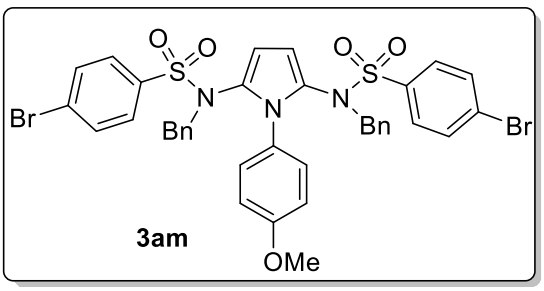
7.20 (d, J = 4.4 Hz, 2H), 7.19 (s, 1H), 7.15 (t, J = 6.8 Hz, 3H), 7.08 (d, J = 6.4 Hz, 2H), 6.95 (dt, J = 2.8, 8.8 Hz, 1H), 4.94 (d, J = 16.4 Hz, 1H), 4.85 (d, J = 14.0 Hz, 1H), 4.66 (d, J = 14.0 Hz, 1H), 4.41 (d, J = 14.0 Hz, 1H), 4.18 (d, J = 16.4 Hz, 1H), 3.00 (s, 3H), 2.20 (s, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 159.4 (d, J = 245 Hz), 147.7, 140.9, 135.3, 135.1, 130.8, 130.7, 129.7, 129.0, 128.9, 128.8, 128.7, 128.6, 128.5, 127.8, 127.7, 126.2, 114.4 (d, J = 8.7 Hz), 113.8 (d, J = 25.0 Hz), 110.7 (d, J = 27.5 Hz), 100.3, 55.3, 49.5, 39.9, 39.7; **^{19}F NMR (376 MHz, $CDCl_3$)** δ – 115.8; **IR (Neat)** ν_{max} 3345, 2970, 1767, 1466, 1408, 1304, 1160, 1128, 1107, 950 cm^{-1}; HRMS (ESI) for $C_{27}H_{26}FN_4O_4S_3$ $(M+H)^+$: calcd 585.1100, found 585.1102.

_N,N_'-(1-(4-Methoxyphenyl)-1_H_-pyrrole-2,5-diyl)bis(_N_-benzyl-4-methylbenzenesulfonamide) (3al)



Following the general procedure GP-1, product **3al** (131 mg) was obtained in 77% yield as pale yellow solid; mp = 185–187 °C; R_f = 0.54 (7:3 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.51 (d, J = 8.4 Hz, 4H), 7.25–7.12 (m, 12H), 6.82 (d, J =7.2 Hz, 4H), 6.51 (s, 2H), 5.63 (s, 2H), 4.21 (brs, 4H), 3.81 (s, 3H), 2.44 (s, 6H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 158.9, 143.7, 135.7, 134.8, 130.7, 129.7, 129.3, 128.4, 128.2, 127.8, 127.4, 126.7, 112.7, 105.9, 56.4, 55.3, 21.6; **IR (Neat)** ν_{max} 3392, 2924, 1384, 1162, 762 cm^{-1}; HRMS (ESI) for $C_{39}H_{38}N_3O_5S_2$ $(M+ H)^+$: calcd 692.2253, found 692.2244.

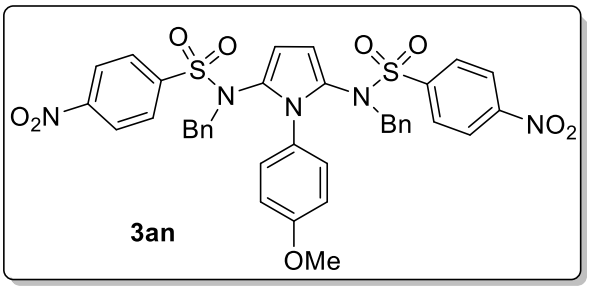
_N,N_'-(1-(4-methoxyphenyl)-1_H_-pyrrole-2,5-diyl)bis(_N_-benzyl-4-bromobenzenesulfonamide) (3am)



Following the general procedure GP-1, product **3am** (101 mg) was obtained in 65% yield as pale yellow solid; mp = 152–154 °C; R_f = 0.48 (4:1 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H**

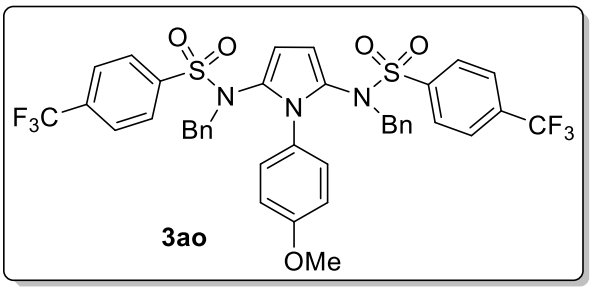
NMR (400 MHz, $CDCl_3$) δ 7.57 (d, J = 8.4 Hz, 4H), 7.44 (d, J = 8.4 Hz, 4H), 7.28–7.15 (m, 8H), 6.86 (d, J = 7.2 Hz, 4H), 6.53 (s, 2H), 5.64 (s, 2H), 4.24 (brs, 4H), 3.83 (s, 3H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 159.1, 139.1, 137.6, 135.9, 134.5, 132.4, 132.0, 129.8, 129.7, 128.8, 128.7, 128.3, 128.1, 128.0, 127.9, 127.7, 127.0, 126.6, 112.9, 106.1, 56.5, 55.4; **IR (Neat)** ν_{max} 3400, 2923, 1384, 1066, 741 cm^{-1}; HRMS (ESI) for $C_{37}H_{32}Br_2N_3O_5S_2$ $(M+H)^+$: calcd 820.0150, found 820.0146.

N,*N*'-(1-(4-Methoxyphenyl)-1*H*-pyrrole-2,5-diyl)bis(*N*-benzyl-4-nitrobenzenesulfonamide) (3an)



Following the general procedure GP-1, product **3an** (71 mg) was obtained in 49% yield as yellow solid; mp = 172–174 °C; R_f = 0.43 (7:3 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.31–8.25 (m, 4H), 7.74 (d, J = 8.4 Hz, 4H), 7.31–7.17 (m, 8H), 6.89 (d, J = 7.2 Hz, 4H), 6.58 (s, 2H), 5.64 (s, 2H), 4.29 (brs, 4H), 3.84 (s, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 159.5, 150.2, 144.4, 134.1, 129.7, 129.4, 128.8, 128.5, 128.4, 127.9, 126.7, 126.6, 124.3, 123.9, 113.2, 106.3, 56.8, 55.4; **IR (Neat)** ν_{max} 3379, 2923, 1526, 1382, 1165, 740 cm^{-1}; HRMS (ESI) for $C_{37}H_{32}N_5O_9S_2$ $(M+H)^+$: calcd 754.1641, found 754.1640.

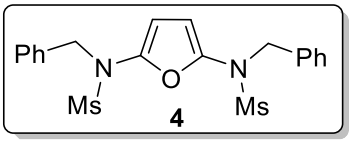
N,*N*'-(1-(4-Methoxyphenyl)-1*H*-pyrrole-2,5-diyl)bis(*N*-benzyl-4-(trifluoromethyl)benzenesulfonamide) (3ao)



Following the general procedure GP-1, compound **3ao** (92 mg) was obtained in 60% yield as yellow solid; mp = 155–157 °C; R_f = 0.44 (4:1 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.71 (t, J = 9.6 Hz, 8H), 7.29–7.25 (m, 3H), 7.20–7.14 (m, 4H), 6.87 (d, J = 7.2 Hz, 4H), 6.54 (s, 2H), 6.35 (s, 1H), 5.63 (s, 2H), 4.27 (brs, 4H), 3.83 (s, 3H); **^{13}C NMR**

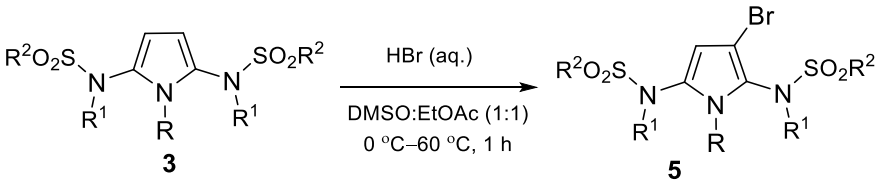
(125 MHz, $CDCl_3$) δ 159.3, 142.3, 134.7, 134.4, 134.3, 129.7, 128.7, 128.4, 128.2, 126.9, 126.6, 125.8 (q, J = 3.7 Hz), 124.3, 122.2, 113.0, 106.3, 56.6, 55.3; **^{19}F NMR (376 MHz, $CDCl_3$)** δ – 63.1; **IR (Neat)** ν_{max} 3403, 2926, 1384, 1062, 769 cm^{-1}; HRMS (ESI) for $C_{39}H_{32}F_6N_3O_5S_2$ $(M+H)^+$: calcd 800.1688, found 800.1684.

***N*,*N*'-(Furan-2,5-diyl)bis(*N*-benzylmethanesulfonamide) (4)**



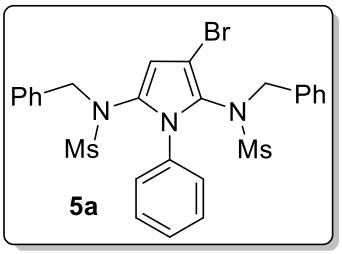
Following the general procedure of GP-1, compound **4** (76 mg) was obtained in 92% yield as yellow solid; mp = 67–69 °C; R_f = 0.47 (7:3 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.29–7.25 (m, 6H), 7.23–7.20 (m, 4H), 6.01 (s, 2H), 4.67 (s, 4H), 2.86 (s, 6H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 142.8, 135.2, 128.9, 128.6, 128.3, 108.7, 54.6, 39.3; **IR (Neat)** ν_{max} 3389, 2925, 1617, 1347, 1156, 763 cm^{-1}; HRMS (ESI) for $C_{20}H_{23}N_2O_5S_2$ $(M+H)^+$: calcd 435.1048, found 435.1044.

General procedure for bromination of 3 (GP-2):[1]



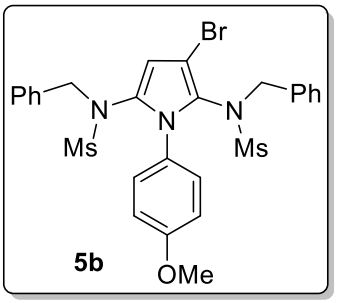
In a 15 mL Schlenk flask, substrate **3** (0.10 mmol) was dissolved in DMSO (2 mL) and EtOAc (2 mL) and subsequently the reaction flask was cooled at 0 °C. Next, HBr (aq.) (0.11 mmol) was introduced into the reaction flask dropwise and stirred for an additional 5 min at 0 °C. Later, the flask was heated at 60 °C for 1 h. Upon full conversion of starting material **3**, the reaction mixture was cooled to room temperature, diluted with dichloromethane (5 mL), and concentrated under reduced pressure. The crude residue was purified through silica gel column chromatography to obtain **5**.

***N*,*N*'-(3-Bromo-1-phenyl-1*H*-pyrrole-2,5-diyl)bis(*N*-benzylmethanesulfonamide) (5a)**



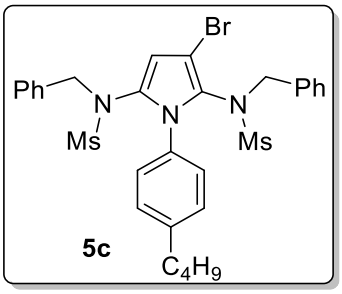
3b (55 mg), HBr (6 μL), and following the general procedure of GP-2, product **5a** (58 mg) was obtained in 92% yield as pale yellow solid; mp = 154–155 °C; R_f = 0.52 (7:3 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.32–7.21 (m, 10H), 7.07–7.02 (m, 5H), 6.13 (s, 1H), 4.44 (q, J = 13.6 Hz, 2H), 4.27 (s, 2H), 2.93 (s, 3H), 2.67 (s, 3H); **^{13}C NMR (125 MHz, CDCl$_3$)** δ 134.6, 134.5, 134.3, 130.1, 129.6, 128.8, 128.7, 128.6, 128.5, 128.4, 128.1, 127.5, 109.1, 95.0, 56.2, 54.4, 41.8, 39.5; **IR (Neat)** ν_{max} 3389, 2925, 1342, 1154, 1028, 761 cm^{-1}; HRMS (ESI) for $C_{26}H_{27}BrN_3O_4S_2$ $(M+H)^+$: calcd 588.0626, found 588.0614.

_N_,_N_'-(3-Bromo-1-(4-methoxyphenyl)-1_H_-pyrrole-2,5-diyl)bis(_N_-benzylmethanesulfonamide) (5b)



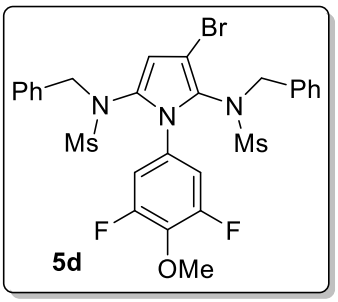
3a (55mg), HBr (6 μL) and following the general procedure of GP-2, product **5b** (46 mg) was obtained in 70% yield as pale yellow solid; mp = 57–59 °C; R_f = 0.52 (7:3 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.32–7.22 (m, 7H), 7.09–7.05 (m, 5H), 6.62 (brs, 2H), 6.11 (s, 1H), 4.45 (q, J = 14.0 Hz, 2H), 4.28 (s, 2H), 3.80 (s, 3H), 2.95 (s, 3H), 2.69 (s, 3H); **^{13}C NMR (125 MHz, CDCl$_3$)** δ 159.6, 134.7, 134.6, 130.1, 129.6, 128.7, 128.6, 128.4, 128.4, 127.6, 126.8, 125.2, 113.2, 108.9, 94.7, 56.2, 55.3, 54.4, 41.8, 39.6; **IR (Neat)** ν_{max} 3392, 2924, 1154, 1064, 768 cm^{-1}; HRMS (ESI) for $C_{27}H_{32}BrN_4O_5S_2$ $(M+NH_4)^+$: calcd 635.0997, found 635.0996.

_N_,N'-(3-Bromo-1-(4-butylphenyl)-1_H_-pyrrole-2,5-diyl)bis(_N_-benzylmethanesulfonamide) (5c)



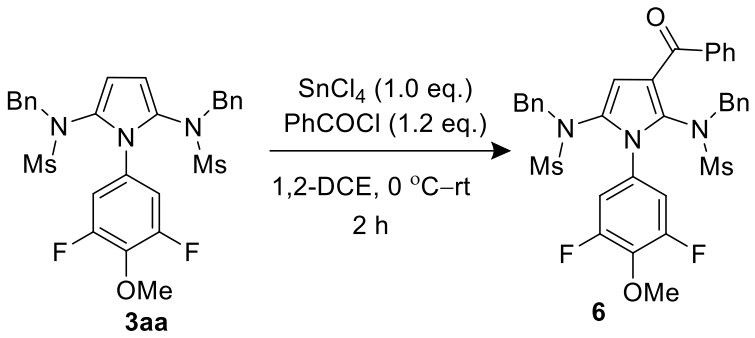
3c (60 mg), HBr (6 μL), and following the general procedure of GP-2, product **5c** (58 mg) was obtained in 85% yield as colorless solid; mp = 133–135 °C; R_f = 0.5 (7:3 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.32–7.20 (m, 7H), 7.08–7.03 (m, 5H), 6.94 (brs, 2H), 6.10 (s, 1H), 4.44 (q, J = 7.2 Hz, 2H), 4.27 (s, 2H), 2.91 (s, 3H), 2.65 (s, 3H), 2.59 (t, J = 7.6 Hz, 2H), 1.63–1.55 (m, 2H), 1.42–1.33 (m, 2H), 0.95 (t, J = 7.2 Hz, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 143.7, 134.7, 134.6, 131.8, 130.0, 129.9, 129.6, 128.7, 128.6, 128.5, 128.4, 128.3, 128.1, 127.4, 109.1, 94.9, 56.1, 54.4, 41.8, 39.6, 35.2, 33.3, 22.3, 13.9; **IR (Neat)** ν_{max} 3391, 2925, 1344, 1155, 767 cm^{-1}; HRMS (ESI) for $C_{30}H_{35}BrN_3O_4S_2$ $(M+H)^+$: calcd 644.1252, found 644.1240.

_N_,_N_'-(3-Bromo-1-(3,5-difluoro-4-methoxyphenyl)-1_H_-pyrrole-2,5-diyl)bis(_N_-benzylmethanesulfonamide) (5d)



3aa (60 mg), HBr (6 μL), and following the general procedure of GP-2, product **5d** (58 mg) was obtained in 85% yield as yellow solid; mp = 161–163 °C; R_f = 0.47 (7:3 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.32–7.18 (m, 8H), 6.99–6.95 (m, 4H), 6.23 (s, 1H), 4.54 (d, J = 13.6, 1H), 4.43–4.32 (m, 2H), 4.25 (d, J = 14.0, 1H), 4.00 (s, 3H), 3.10 (s, 3H), 2.85 (s, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 153 (d, J = 246.2 Hz), 136.8, 136.7 (d, J = 13.5), 134.2, 134.0, 129.8, 129.5, 128.7, 127.9, 127.7, 127.6 (d, J = 12.4), 124.9, 114.5, 108.7, 94.9, 61.7 (t, J = 3.5 Hz), 56.7, 54.1, 41.4, 38.4; **^{19}F NMR (376 MHz, $CDCl_3$)** δ –63.1; **IR (Neat)** ν_{max} 3391, 2926, 1514, 1343, 1155, 1035, 763 cm^{-1}; HRMS (ESI) for $C_{27}H_{26}BrF_2N_3NaO_5S_2$ $(M+Na)^+$: calcd 676.0363, found 676.0365.

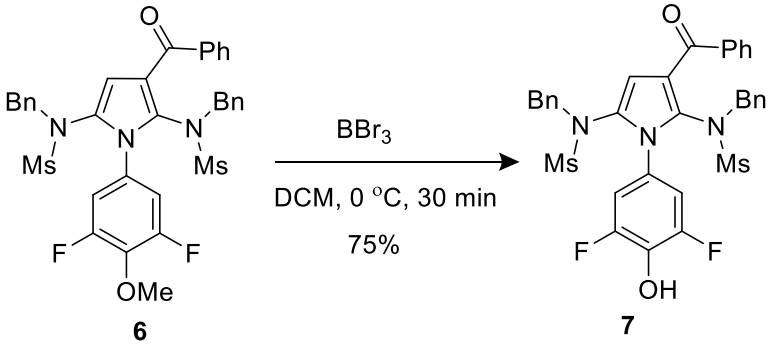
***N*,*N*'-(3-Benzoyl-1-(3,5-difluoro-4-methoxyphenyl)-1*H*-pyrrole-2,5-diyl)bis(*N*-benzylmethanesulfonamide) (6)**[2]



In an oven-dried 15 mL Schlenk flask, **3aa** (0.21 mmol, 120 mg) and $SnCl_4$ (0.21 mmol, 54 mg) were taken. The Schlenk flask was cooled at 0 °C upon addition of 1,2-dichloroethane (5 mL). Next, PhCOCl (0.25 mmol, 28 μL) was added dropwise at 0 °C and later, the reaction mixture was stirred at room temperature for 2 h. The progress of the reaction was monitored by TLC and upon full consumption of starting material, the reaction mixture was diluted with dichloromethane (5 mL), and filtered through a small pad of Celite. The filtrate was concentrated under reduced pressure and crude residue was purified through silica gel column chromatography to obtain **6** (115 mg).

81% yield as brown solid; mp = 206–208 °C; R_f = 0.46 (7:3 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.72 (d, J = 7.2 Hz, 2H), 7.62 (t, J = 7.2 Hz, 1H), 7.51 (t, J = 7.6 Hz, 3H), 7.36 (t, J = 7.2 Hz, 1H), 7.31–7.27 (m, 4H), 7.17 (t, J = 7.6 Hz, 2H), 7.01 (d, J = 7.2 Hz, 2H), 6.95 (d, J = 7.2 Hz, 2H), 6.22 (s, 1H), 4.70 (s, 2H), 4.23 (s, 2H), 4.04 (s, 3H), 3.08 (s, 3H), 2.79 (s. 3H); **^{13}C NMR (101 MHz, $CDCl_3$)** δ 191.1, 153.5 (d, J = 251.5 Hz), 139.1, 137.1, 137.0 (d, J = 26.9 Hz), 134.6, 134.1, 132.5, 130.2, 129.9, 129.7, 129.2, 128.8, 128.5, 127.2 (d, J = 24.9 Hz), 127.1, 126.6, 118.4, 114.1, 110.8, 61.7, 56.2, 40.3; **IR (Neat)** ν_{max} 3389, 2926, 1644, 1342, 1154, 1038, 758 cm^{-1}; **^{19}F NMR (376 MHz, $CDCl_3$)** δ –127.7, –127.8; HRMS (ESI) for $C_{34}H_{32}F_2N_3O_6S_2$ $(M+H)^+$: calcd 680.1701, found 680.1694.

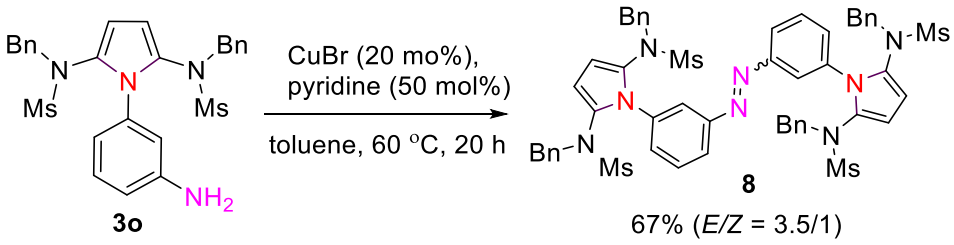
***N*,*N*'-(3-Benzoyl-1-(3,5-difluoro-4-hydroxyphenyl)-1*H*-pyrrole-2,5-diyl)bis(*N*-benzylmethanesulfonamide) (7)**



In an oven-dried 15 mL Schlenk flask, **6** (0.09 mmol, 60 mg) was dissolved in dichloromethane (2 mL) at 0 °C. Next, dropwise addition of BBr_3 (0.44 mmol, 42 µL) was performed at 0 °C into the flask and subsequently, the reaction mixture was stirred for an additional 30 min before quenching with H_2O (2 mL). Later, the reaction mixture was extracted with DCM (10 mL), dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified through silica gel column chromatography to obtain **7** (44 mg).

75% yield as pale yellow solid; mp = 214–216 °C; R_f = 0.5 (3:2 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.73 (s, 1H), 7.71 (s, 1H), 7.62 (t, J = 7.4 Hz, 1H), 7.51 (t, J = 7.7 Hz, 2H), 7.37 (t, J = 7.5 Hz, 1H), 7.32–7.26 (m, 5H), 7.19 (t, J = 7.5 Hz, 2H), 7.02 (d, J = 7.0 Hz, 2H), 6.96 (d, J = 7.1Hz, 2H), 6.22 (s, 1H), 5.72 (brs, 1H), 4.70 (s, 2H), 4.29 (s, 2H), 3.09 (s, 3H), 2.78 (s, 3H); **^{13}C NMR (101 MHz, DMSO-d_6)** δ 190.7, 150.5 (d, J = 241.7 Hz), 150.4 (d, J = 241.4 Hz), 139.1, 135.2, 135.1, 134.7 (t, J = 11.3 Hz), 132.9, 130.1, 129.9, 129.7, 129.6, 129.0, 128.8 (d, J = 8.7 Hz), 128.5 (d, J = 10.2 Hz), 127.4, 123.5, 118.4, 110.3, 79.6, 60.2, 55.9, 37.7; **^{19}F NMR (376 MHz, $CDCl_3$)** δ –134.3, –134.6; **IR (Neat)** ν_{max} 3385, 2923, 1635, 1384, 1064, 1028 cm^{-1}; HRMS (ESI) for $C_{33}H_{29}F_2KN_3O_6S_2$ $(M+K)^+$: calcd 704.1103, found 704.1073.

_N,N'_-(1-(3-((3-(2,5-Bis(_N_-benzylmethylsulfonamido)-1_H_-pyrrol-1-yl)phenyl)diazenyl)phenyl)-1_H_-pyrrole-2,5-diyl)bis(_N_-benzylmethanesulfonamide) (8)[3]



At first, **3o** (0.11 mmol, 60 mg) and CuBr (0.022 mmol, 3.1 mg) were taken in an oven-dried 15 mL Schlenk flask under inert atmosphere and subsequently freshly distilled toluene (1.0

mL) and pyridine (0.055 mmol, 4.0 μL) were introduced successively into the reaction mixture at room temperature. Next, the reaction mixture was stirred vigorously at 60 ℃ for 20 h. Later, the reaction mixture was cooled to room temperature, diluted with dichloromethane (10 mL), and filtered through a short pad of Celite. The filtrate was concentrated under reduced pressure and the crude residue was purified through silica gel column chromatography to obtain **8** in 67% (40 mg) yield.

yellow solid with isomeric ratio of E:Z (3.5:1); mp = 177–179 °C; R_f = 0.41 (3:7 hexane/EtOAc); [Silica, UV and I_2]; **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.86 (d, J = 8.4 Hz, 1H), 7.34–7.31 (m, 4H), 7.22–7.17 (m, 13H), 7.08 (d, J = 6.8 Hz, 3H), 7.03 (d, J = 6.8 Hz, 7H), 6.12 (s, 4H), 4.38 (s, 8H), 2.83 (s, 12H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 152.1, 135.6, 134.6, 134.5, 129.7, 129.6, 128.8, 128.7, 128.6, 128.3, 127.2, 106.5, 56.5, 39.0; **IR (Neat)** ν_{max} 3398, 2923, 1339, 1153, 1027, 756 cm^{-1}; HRMS (ESI) for $C_{52}H_{53}N_8O_8S_4$ $(M+H)^+$: calcd 1045.2869, found 1045.2852.

References:

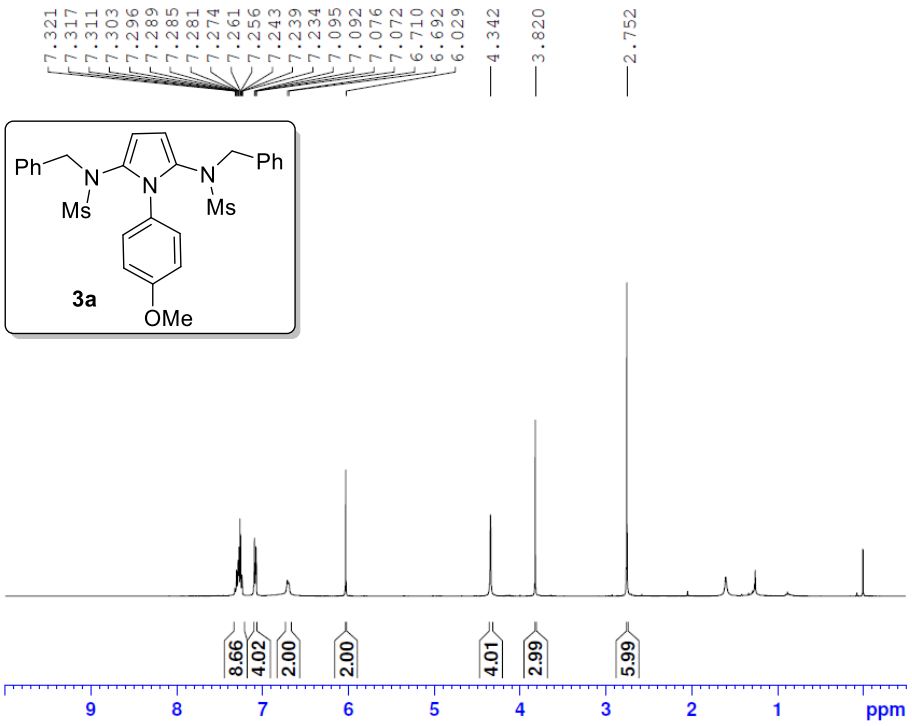
1. (a) Gayyur; Choudhary, S.; K, Ruchir.; Ghosh, N. Synergetic copper/zinc catalysis: synthesis of aryl/heteroaryl-fused 1H-pyrrolo[3,2-c]pyridines. *Chem. Commun.,* **2022**, *58*, 1974–1977. (b) Tian, X.; Song, L.; Rudolph, M.; Rominger, F.; Oeser, T.; Hashmi, A. S. K. Sulfilimines as Versatile Nitrene Transfer Reagents: Facile Access to Diverse Aza-Heterocycles. *Angew. Chem., Int. Ed.* **2019**, *58*, 3589–3593. (c) Kramer, S.; Madsen, J. L. H.; Rottländer, M.; Skrydstrup, T. Access to 2,5-Diamidopyrroles and 2,5-Diamidofurans by Au(I)-Catalyzed Double Hydroamination or Hydration of 1,3-Diynes. *Org. Lett.* **2010**, *12*, 2758–2761. (d) Martínez-Esperón, M. F.; Rodríguez, D.; Castedo, L.; Saá, C. Coupling and cycloaddition of ynamides: homo- and Negishi coupling of tosylynamides and intramolecular [4D2] cycloaddition of N-(o-ethynyl)phenyl ynamides and arylynamides. *Tetrahedron* **2006**, *62*, 3843–3855. (e) Rodríguez, D.; Castedo, L.; Saá, C. Homocoupling of 1-Alkynyl Tosylamides. *Synlett* **2004**, 377–379.

2. Holtfrerich, A.; Hanekamp, W.; Lehr, M. (4-Phenoxyphenyl)tetrazolecarboxamides and related compounds as dual inhibitors of fatty acid amide hydrolase (FAAH) and monoacylglycerol lipase (MAGL). *Eur. J. Med. Chem.* **2013**, *63*, 64–75.

3. Xiao, Y.; Wu, X.; Wang, H.; Sun, S.; Yu, J.-T.; Cheng, J. Rhodium-Catalyzed Reaction of Azobenzenes and Nitrosoarenes toward Phenazines. *Org. Lett.* **2019**, *21*, 2565–2568.

SH-20-483

^{1}H NMR, $CDCl_3$, 400 MHz

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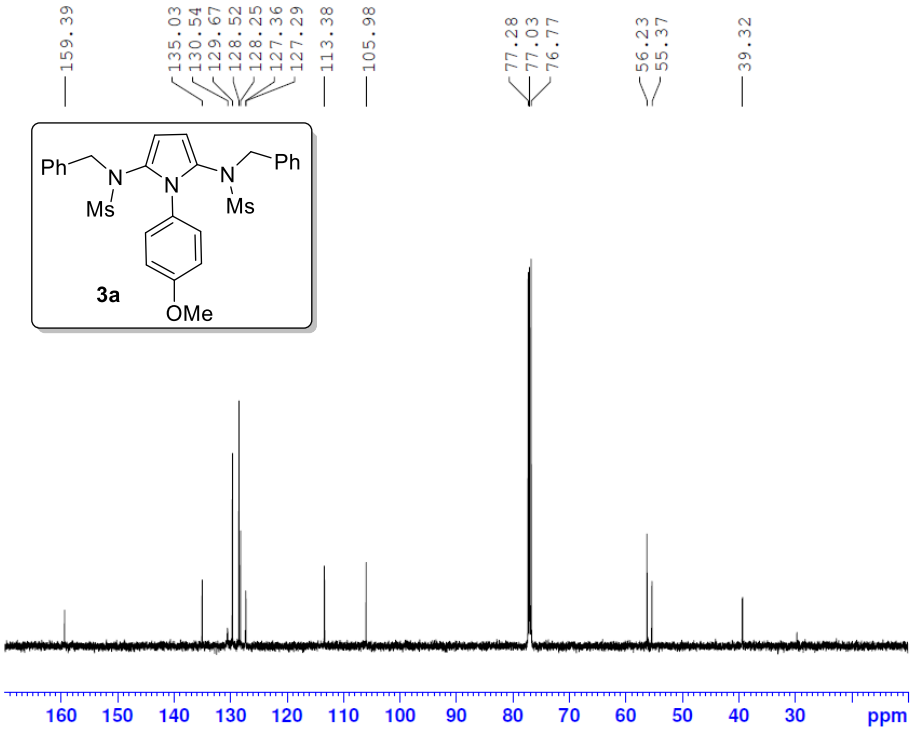
^{13}C NMR, $CDCl_3$, 125 MHz

SH-20-483

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EXPNO                 600
PROCNO                  1

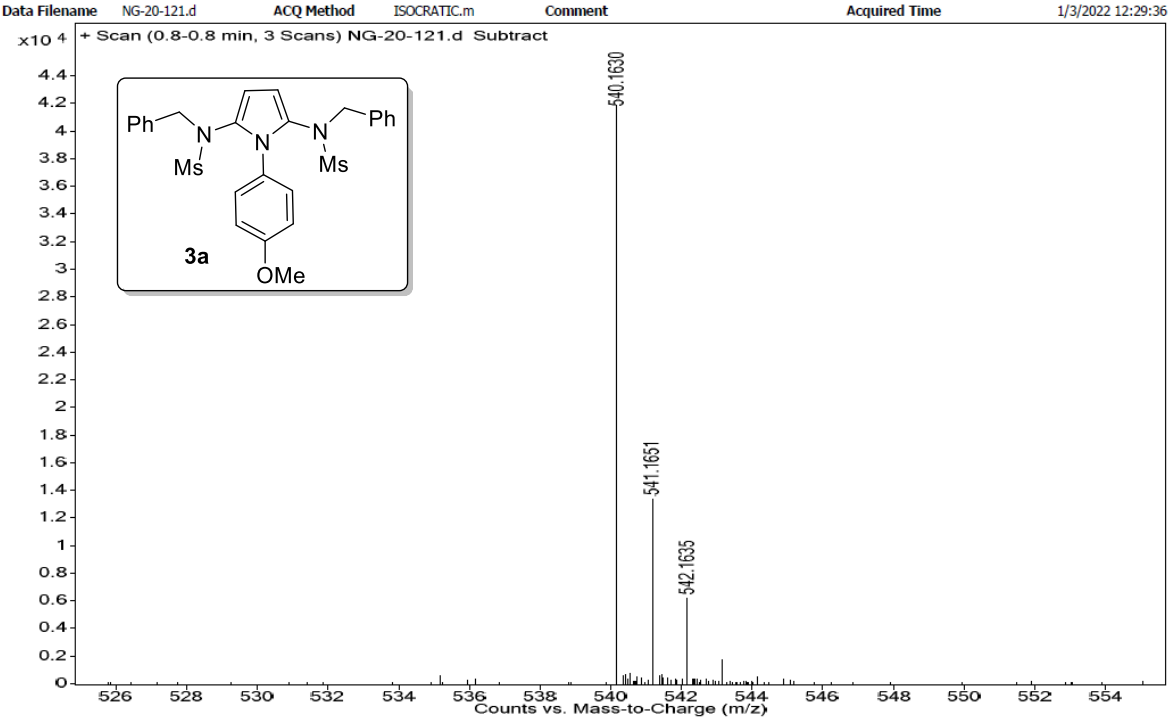
F2 - Acquisition Parameters
Date_            20211122
Time                13.29
INSTRUM             spect
PROBHD   5 mm PATXI 1H/
PULPROG           zgpg30
TD                 65536
SOLVENT             CDC13
NS                   1024
DS                      4
SWH             29761.904 Hz
FIDRES           0.454131 Hz
AQ               1.1010048 sec
RG                  56.22
DW                 16.800 usec
DE                  6.50 usec
TE                 303.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          125.9077573 MHz
NUC1                   13C
P1                  9.23 usec
PLW1        244.00000000 W

======== CHANNEL f2 ========
SFO2          500.6783527 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2          13.60000038 W
PLW12          0.08840500 W
PLW13          0.05657900 W

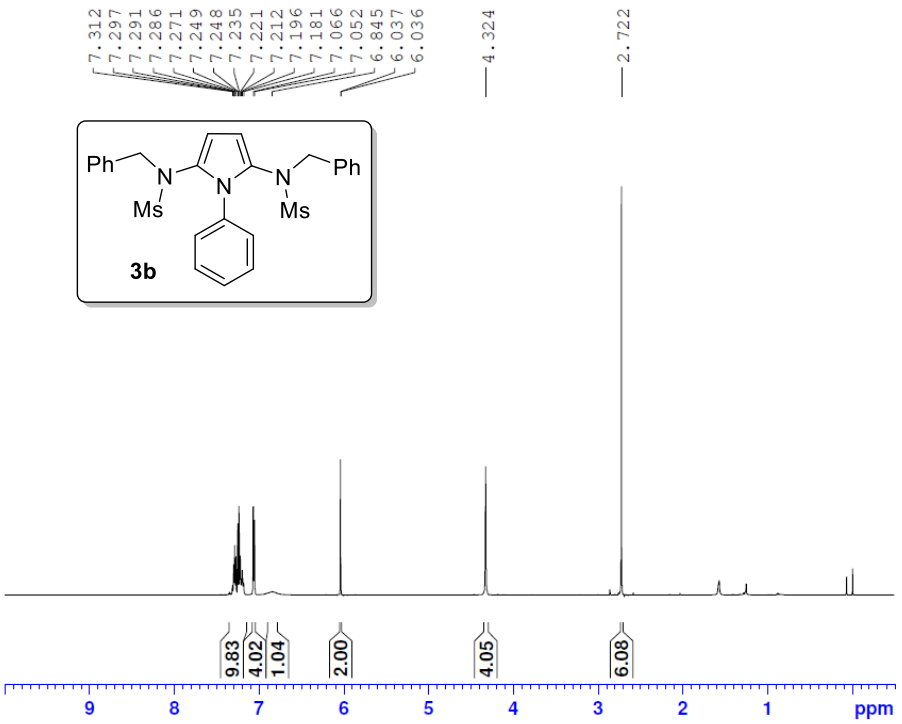
F2 - Processing parameters
SI                 32768
SF            125.8951680 MHz
WDW                   EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

Sample Name	HRMS22I03JAN14	**Position**	Vial 14	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	NG-20-121.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	1/3/2022 12:29:36 PM



^{1}H NMR, $CDCl_3$, 500 MHz

GR-21-53




```
Current Data Parameters
NAME            GR-21-53
EXPNO                490
PROCNO                 1

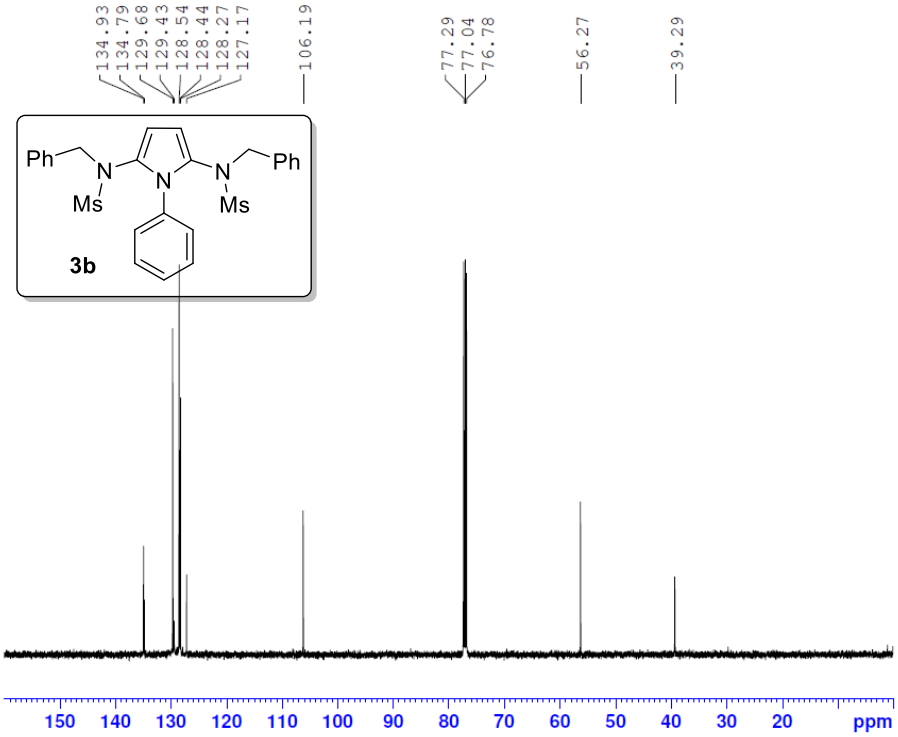
F2 - Acquisition Parameters
Date_           20211122
Time               11.53
INSTRUM            spect
PROBHD   5 mm PATXI 1H/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10026.738 Hz
FIDRES        0.152996 Hz
AQ            3.2680619 sec
RG                 87.95
DW                49.867 usec
DE                 6.50 usec
TE                303.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         500.6794419 MHz
NUC1                   1H
P1                 6.45 usec
PLW1        13.60000038 W

F2 - Processing parameters
SI                 65536
SF           500.6763678 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

GR-21-53

^{13}C NMR, $CDCl_3$, 125 MHz




```
Current Data Parameters
NAME                  Desktop
EXPNO                     500
PROCNO                      1

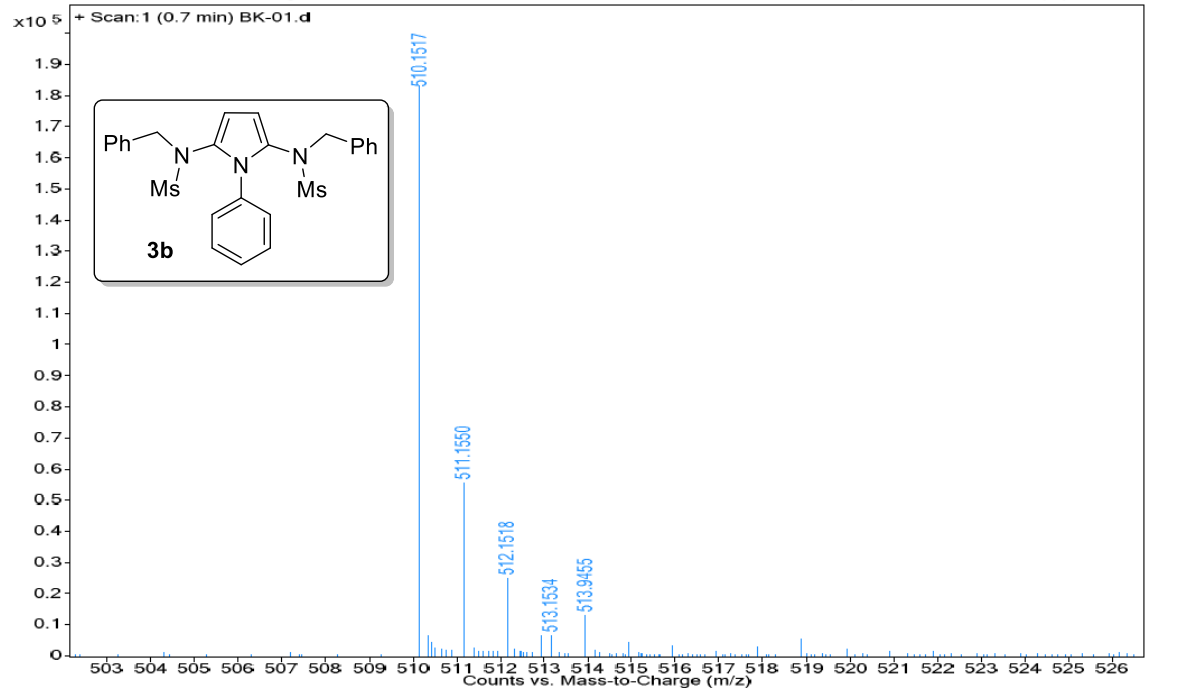
F2 - Acquisition Parameters
Date_               20211122
Time                   11.55
INSTRUM                spect
PROBHD    5 mm PATXI 1H/
PULPROG             zgpg30
TD                     65536
SOLVENT                CDCl3
NS                      2048
DS                         4
SWH                29761.904 Hz
FIDRES                 0.454131 Hz
AQ                     1.1010048 sec
RG                        56.22
DW                        16.800 usec
DE                         6.50 usec
TE                       303.0 K
D1                     2.00000000 sec
D11                    0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1                125.9077573 MHz
NUC1                       13C
P1                         9.23 usec
PLW1               244.00000000 W

======== CHANNEL f2 ========
SFO2                500.6783527 MHz
NUC2                       1H
CPDPRG[2                waltz16
PCPD2                     80.00 usec
PLW2               13.60000038 W
PLW12               0.08840500 W
PLW13               0.05657900 W

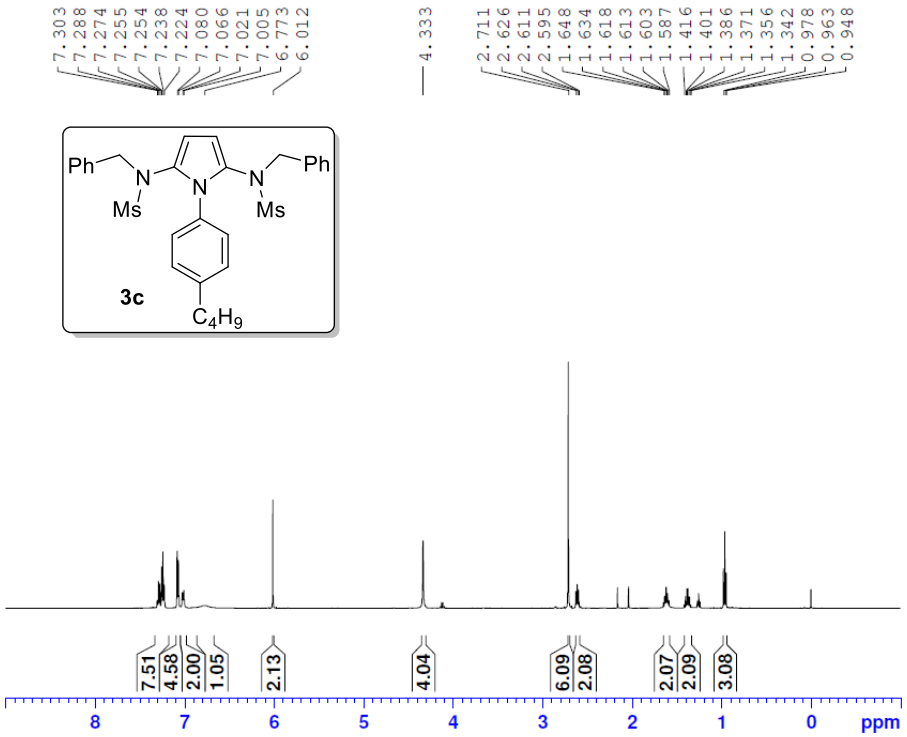
F2 - Processing parameters
SI                        32768
SF                 125.8951680 MHz
WDW                          EM
SSB          0
LB                         1.00 Hz
GB           0
PC                         1.40
```

Sample Name	HRMS22I19JAN29	**Position**	Vial 29	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	BK-01.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	1/19/2022 12:59:50 PM



GR-21-21

^{1}H NMR, $CDCl_3$, 500 MHz




```
Current Data Parameters
NAME                  GR-21-21
EXPNO                      350
PROCNO                       1

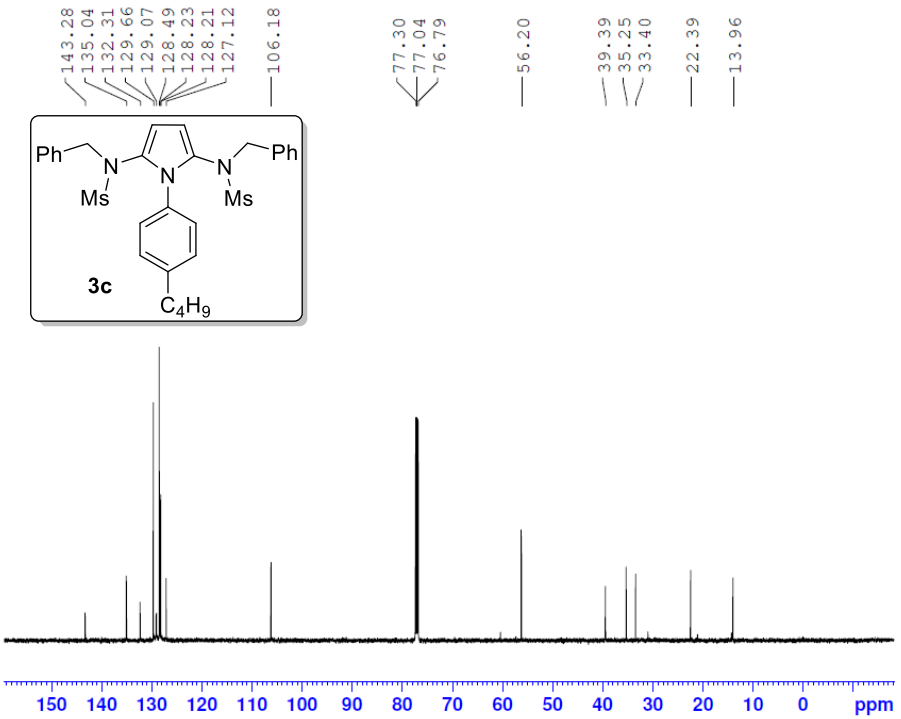
F2 - Acquisition Parameters
Date_               20211101
Time                   16.10
INSTRUM                spect
PROBHD    5 mm PATXI 1H/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH               10026.738 Hz
FIDRES                0.152996 Hz
AQ                3.2680619 sec
RG                     61.7
DW                   49.867 usec
DE                     6.50 usec
TE                    303.0 K
D1                1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1             500.6794419 MHz
NUC1                       1H
P1                       6.45 usec
PLW1             13.60000038 W

F2 - Processing parameters
SI                       65536
SF               500.6763637 MHz
WDW                         EM
SSB                      0
LB                       0.30 Hz
GB                       0
PC                       1.00
```

GR-21-21

^{13}C NMR, $CDCl_3$, 125 MHz




```
Current Data Parameters
NAME                  Desktop
EXPNO                     360
PROCNO                      1

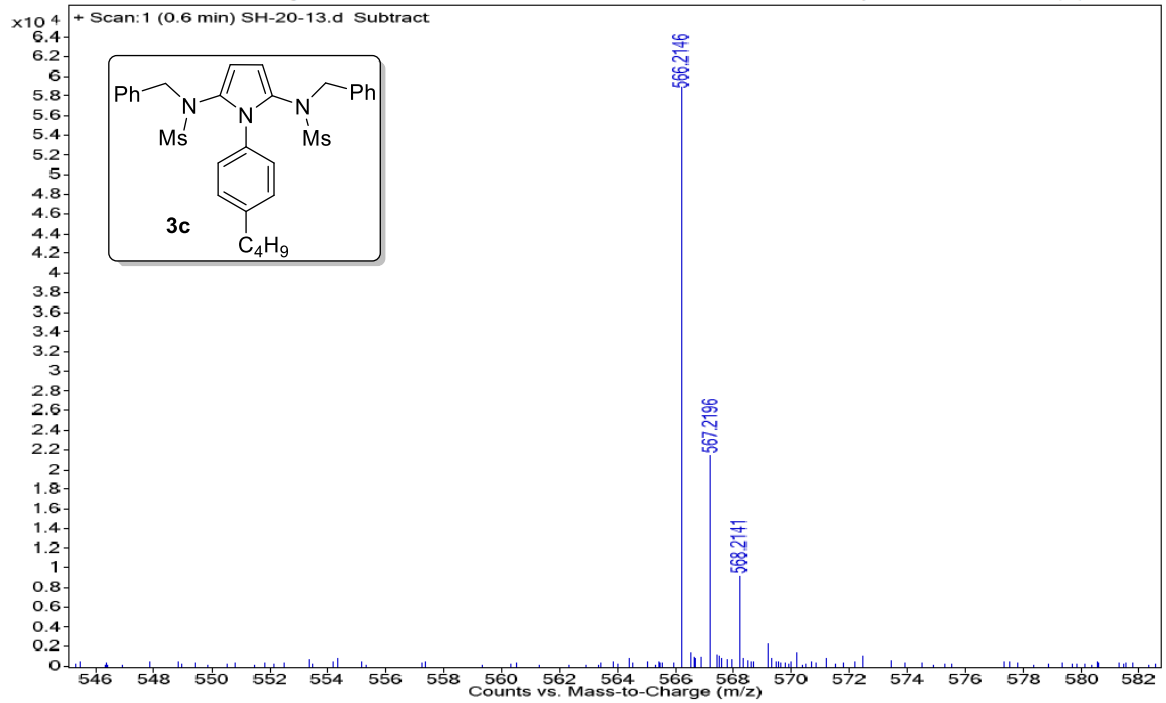
F2 - Acquisition Parameters
Date_               20211101
Time                   17.05
INSTRUM                spect
PROBHD    5 mm PATXI 1H/
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     1024
DS                        4
SWH               29761.904 Hz
FIDRES                0.454131 Hz
AQ                1.1010048 sec
RG                     56.22
DW                   16.800 usec
DE                     6.50 usec
TE                    303.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1             125.9077573 MHz
NUC1                       13C
P1                       9.23 usec
PLW1             244.00000000 W

======== CHANNEL f2 ========
SFO2             500.6783527 MHz
NUC2                       1H
CPDPRG[2              waltz16
PCPD2                  80.00 usec
PLW2             13.60000038 W
PLW12             0.08840500 W
PLW13             0.05657900 W

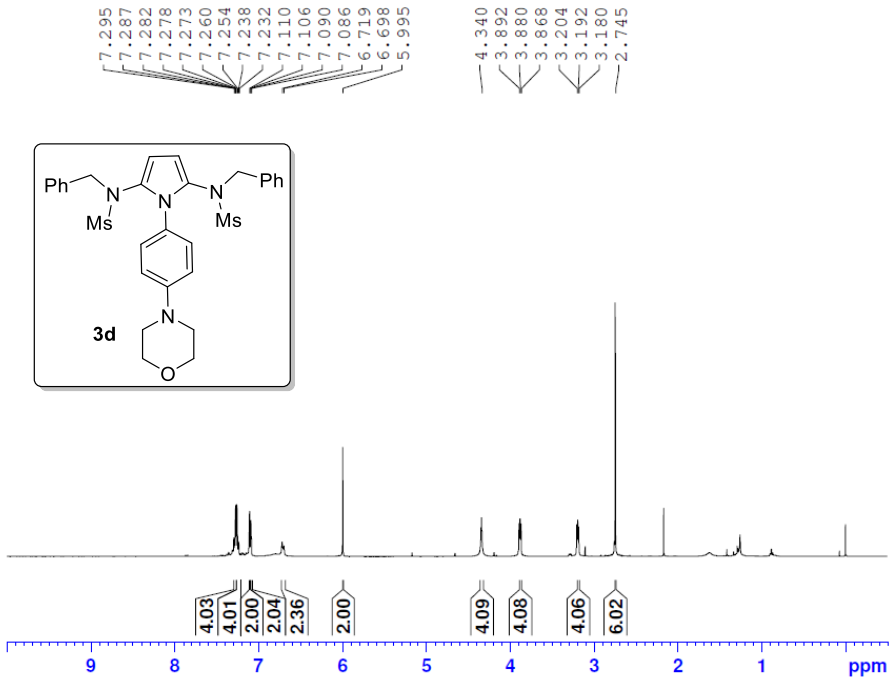
F2 - Processing parameters
SI                       32768
SF               125.8951680 MHz
WDW                         EM
SSB                      0
LB                       1.00 Hz
GB                       0
PC                       1.40
```

Sample Name	HRMS22I18JAN24	**Position**	Vial 24	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	SH-20-13.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	1/18/2022 12:53:21 PM



GR-21-10

^{1}H NMR, $CDCl_3$, 400 MHz




```
Current Data Parameters
NAME                  GR-21-10
EXPNO                      510
PROCNO                       1

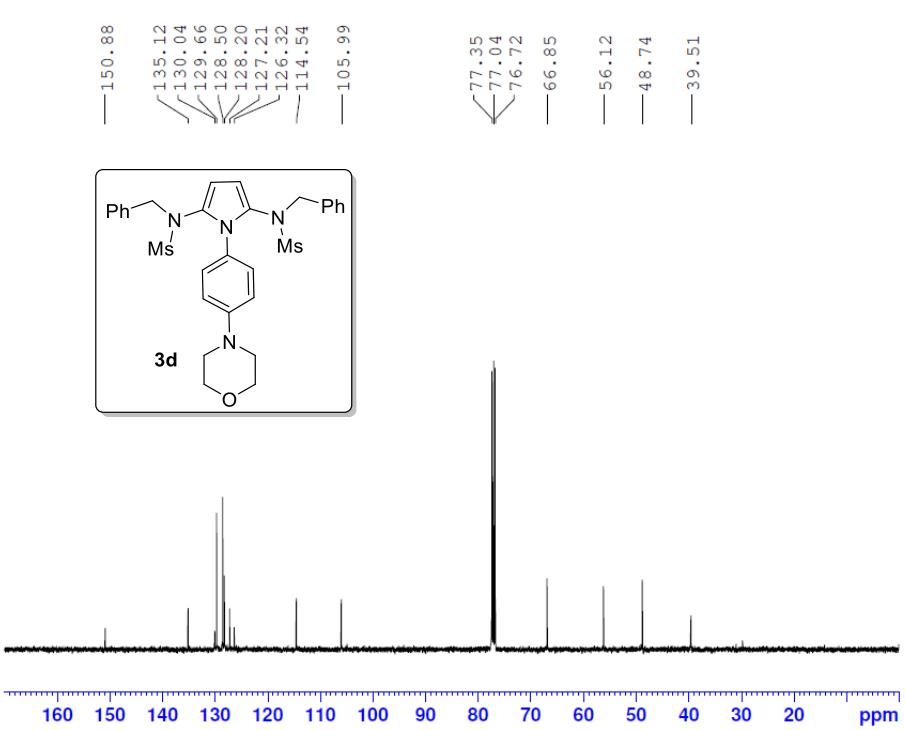
F2 - Acquisition Parameters
Date_                 20211214
Time                      5.43
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                           8
DS                           0
SWH                  9615.385 Hz
FIDRES               0.146719 Hz
AQ                   3.4078720 sec
RG                        89.7
DW                      52.000 usec
DE                       6.50 usec
TE                      300.0 K
D1                   1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                400.1629712 MHz
NUC1                        1H
P1                       13.20 usec
PLW1                13.00000000 W

F2 - Processing parameters
SI                      65536
SF                  400.1605095 MHz
WDW                        EM
SSB                  0
LB                       0.30 Hz
GB                   0
PC                       1.00
```

GR-21-10

^{13}C NMR, $CDCl_3$, 101 MHz




```
Current Data Parameters
NAME                  GR-21-10
EXPNO                      520
PROCNO                       1

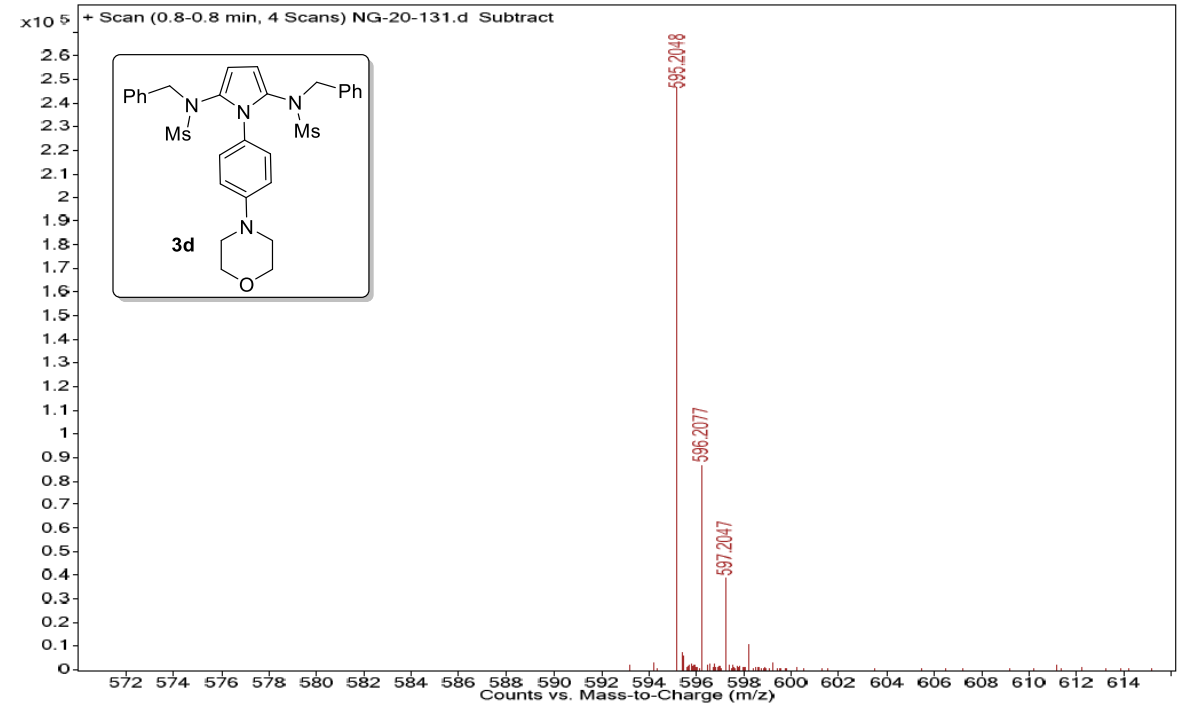
F2 - Acquisition Parameters
Date_                 20211214
Time                      6.04
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                         2048
DS                            0
SWH                24038.461 Hz
FIDRES              0.366798 Hz
AQ                  1.3631488 sec
RG                     201.48
DW                     20.800 usec
DE                      6.50 usec
TE                     300.0 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1               100.6304993 MHz
NUC1                       13C
P1                        9.90 usec
PLW1               53.00000000 W

======== CHANNEL f2 ========
SFO2               400.1621006 MHz
NUC2                       1H
CPDPRG[2                waltz16
PCPD2                    90.00 usec
PLW2               13.00000000 W
PLW12               0.27963999 W
PLW13               0.22651000 W

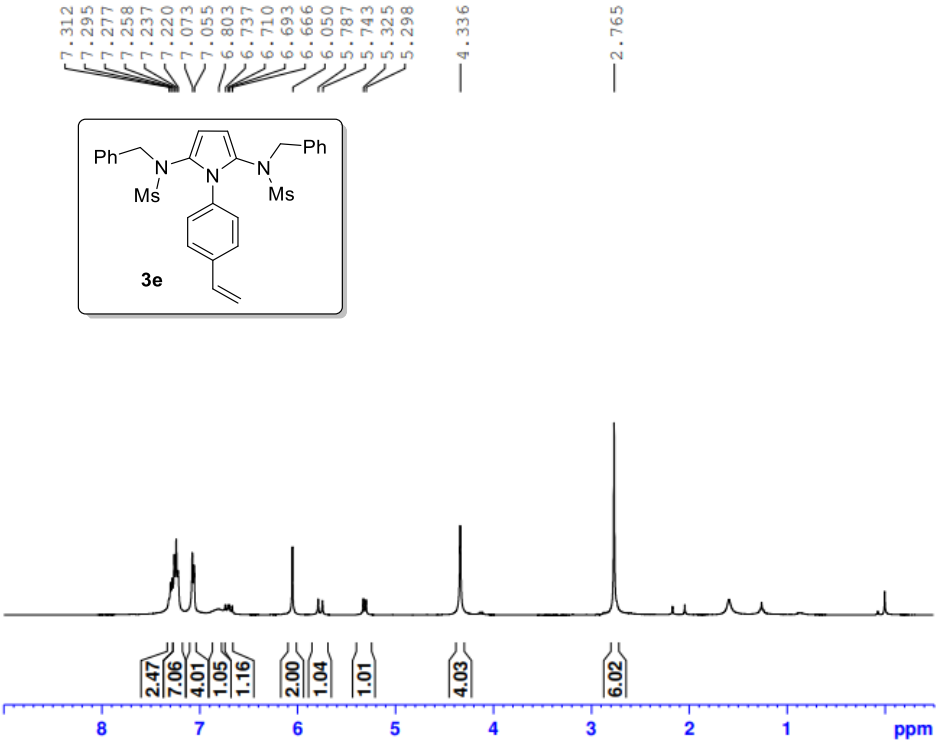
F2 - Processing parameters
SI                        32768
SF                 100.6204380 MHz
WDW                          EM
SSB           0
LB                        1.00 Hz
GB            0
PC                        1.40
```

Sample Name	HRMS22I03JAN13	**Position**	Vial 13	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	NG-20-131.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	1/3/2022 12:27:04 PM



GR-21-36

^{1}H NMR, $CDCl_3$, 400 MHz




```
Current Data Parameters
NAME                   1h
EXPNO                 580
PROCNO                  1

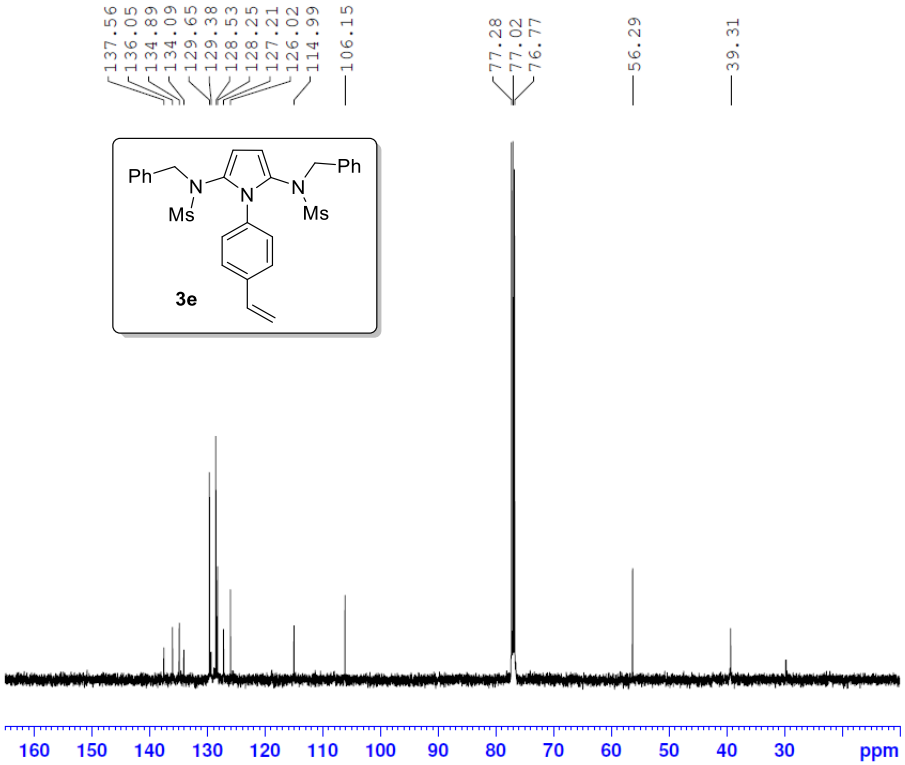
F2 - Acquisition Parameters
Date_           20220527
Time               13.10
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH             9615.385 Hz
FIDRES          0.146719 Hz
AQ              3.4078720 sec
RG                 114.26
DW                 52.000 usec
DE                  6.50 usec
TE                 300.0 K
D1              1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1629712 MHz
NUC1                   1H
P1                 13.20 usec
PLW1          13.00000000 W

F2 - Processing parameters
SI                 65536
SF            400.1605094 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

GR-21-36

^{13}C NMR, $CDCl_3$, 125 MHz




```
Current Data Parameters
NAME             GR-21-36
EXPNO                 380
PROCNO                  1

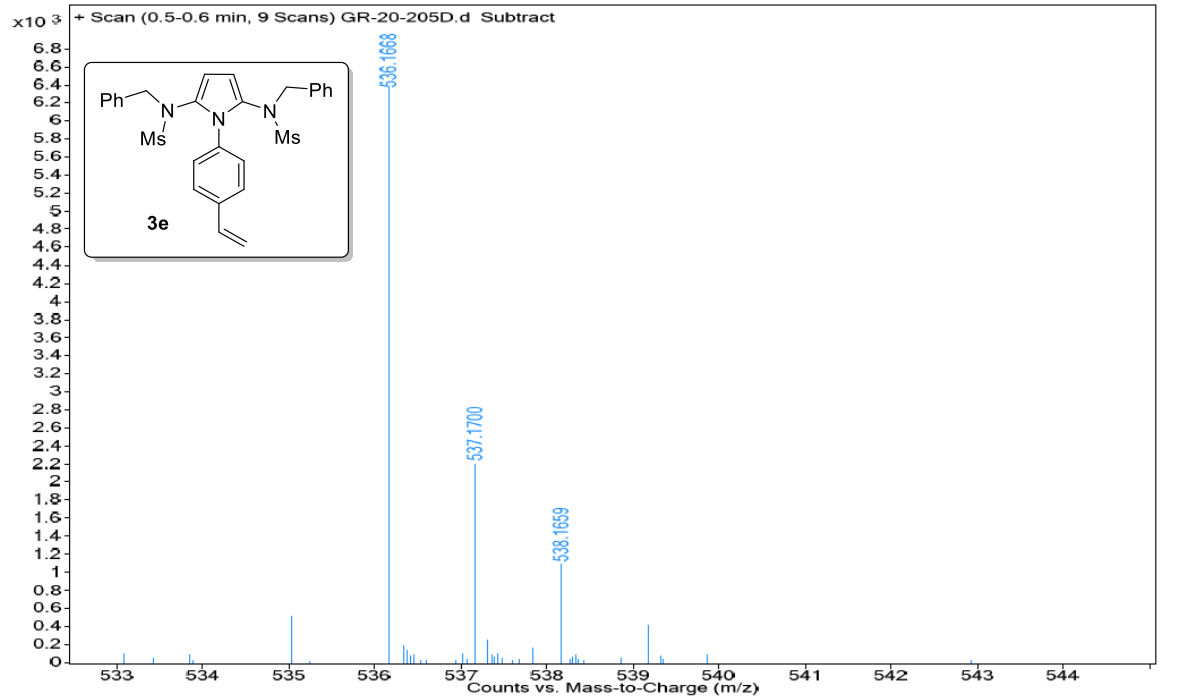
F2 - Acquisition Parameters
Date_           20211116
Time                8.25
INSTRUM            spect
PROBHD   5 mm PATXI 1H/
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            29761.904 Hz
FIDRES          0.454131 Hz
AQ              1.1010048 sec
RG                 56.22
DW                 16.800 usec
DE                  6.50 usec
TE                 303.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          125.9077573 MHz
NUC1                   13C
P1                  9.23 usec
PLW1        244.00000000 W

======== CHANNEL f2 ========
SFO2          500.6783527 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2          13.60000038 W
PLW12          0.08840500 W
PLW13          0.05657900 W

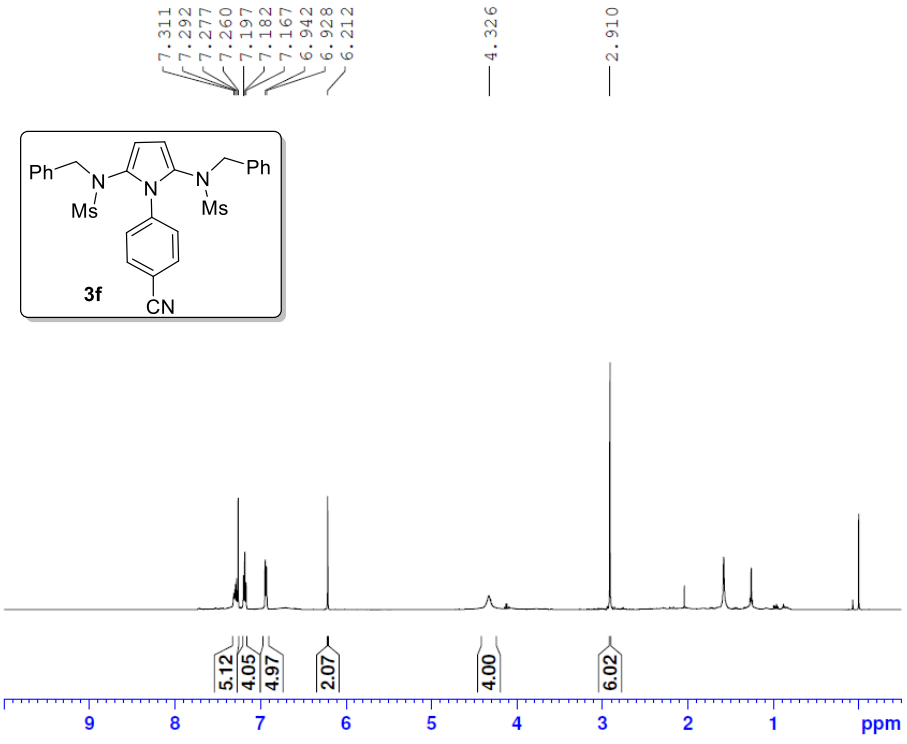
F2 - Processing parameters
SI                 32768
SF            125.8951680 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

Sample Name	HRMS21I09DEC22	**Position**	Vial 22	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	GR-20-205D.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	12/9/2021 1:09:38 PM



GR-21-50

^{1}H NMR, $CDCl_3$, 500 MHz




```
Current Data Parameters
NAME                  GR-21-50
EXPNO                      450
PROCNO                       1

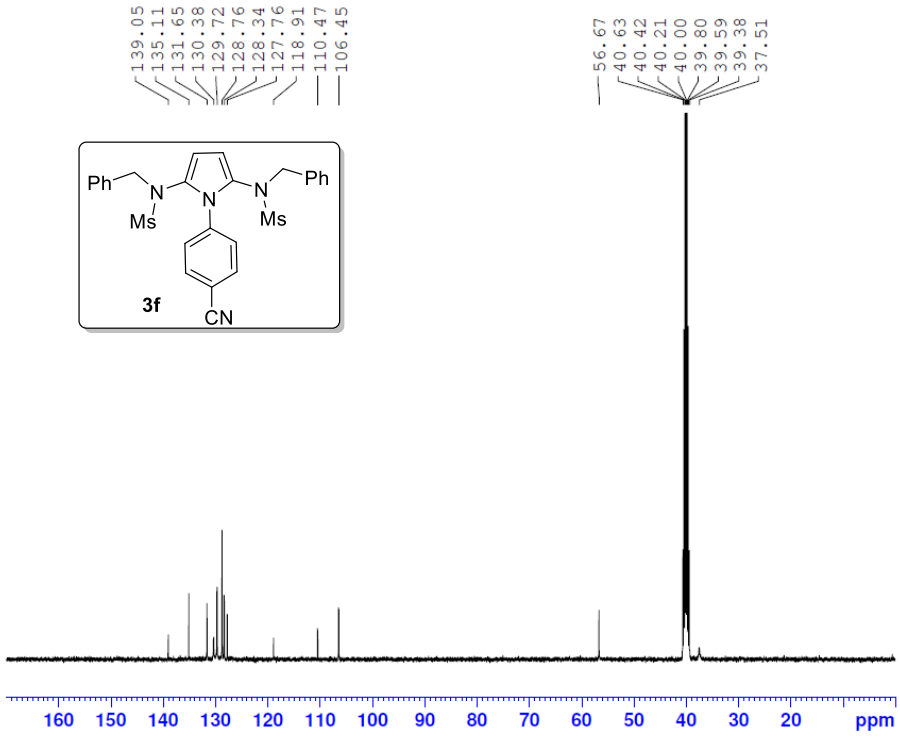
F2 - Acquisition Parameters
Date_                 20211122
Time                     10.34
INSTRUM                  spect
PROBHD    5 mm PATXI 1H/
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                         16
DS                          2
SWH                 10026.738 Hz
FIDRES                0.152996 Hz
AQ                    3.2680619 sec
RG                       181.18
DW                       49.867 usec
DE                        6.50 usec
TE                       303.0 K
D1                    1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1               500.6794419 MHz
NUC1                        1H
P1                        6.45 usec
PLW1               13.60000038 W

F2 - Processing parameters
SI                      65536
SF                 500.6763619 MHz
WDW                        EM
SSB                 0
LB                        0.30 Hz
GB                  0
PC                        1.00
```

GR-21-50

^{13}C NMR, d_6-DMSO, 101 MHz



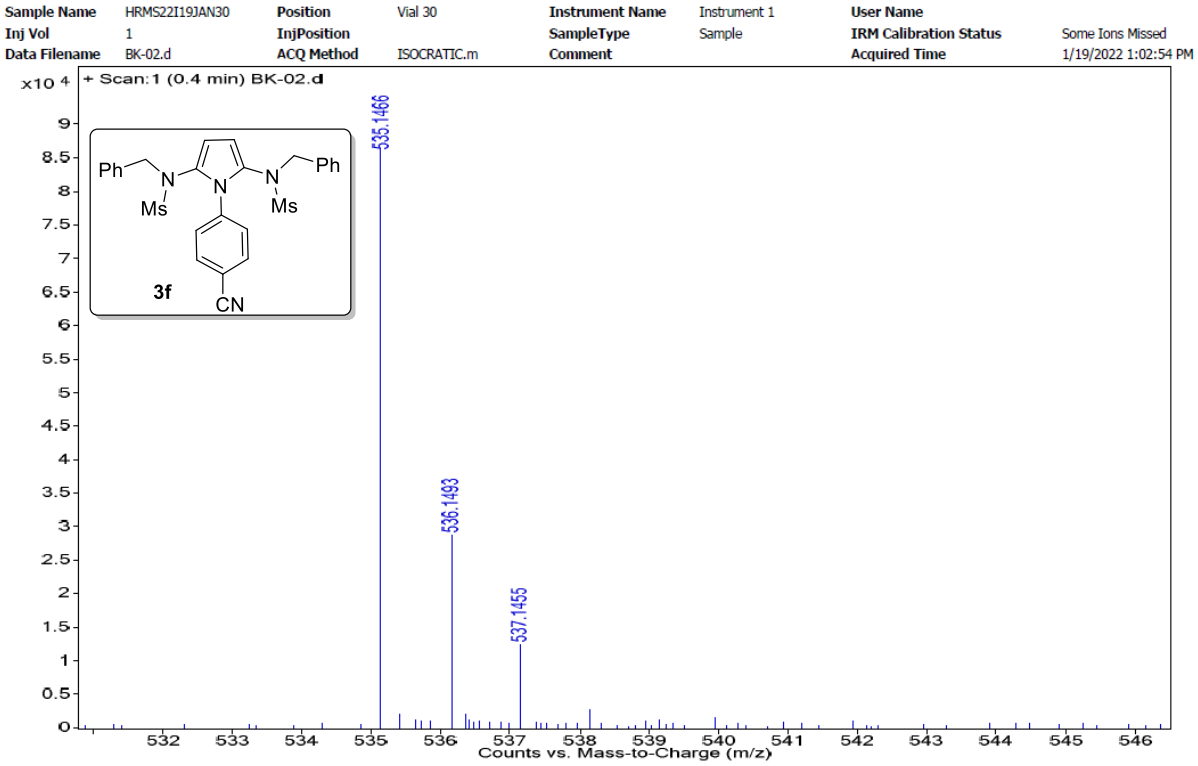

```
Current Data Parameters
NAME                  GR-21-50
EXPNO                      480
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20211124
Time                      4.42
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                zgpg30
TD                        65536
SOLVENT                   DMSO
NS                        2048
DS                           4
SWH                  24038.461 Hz
FIDRES                0.366798 Hz
AQ                    1.3631488 sec
RG                       201.48
DW                       20.800 usec
DE                        6.50 usec
TE                       300.0 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                100.6304993 MHz
NUC1                        13C
P1                         9.90 usec
PLW1                53.00000000 W

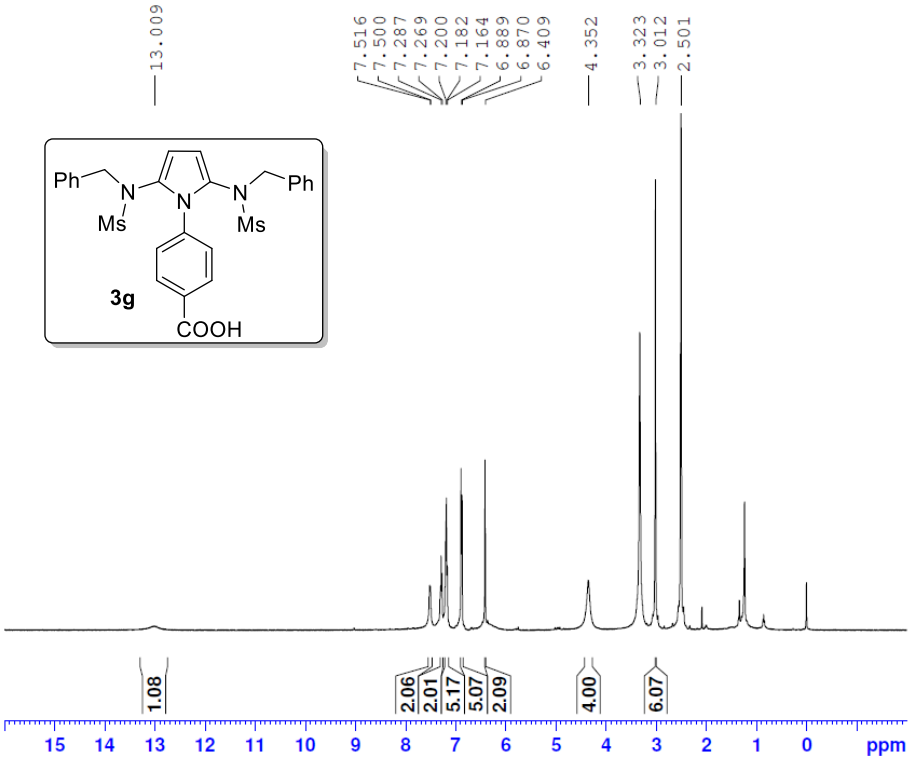
======== CHANNEL f2 ========
SFO2                400.1621006 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                     90.00 usec
PLW2                13.00000000 W
PLW12                0.27963999 W
PLW13                0.22651000 W

F2 - Processing parameters
SI                        32768
SF                  100.6204380 MHz
WDW                          EM
SSB                  0
LB                         1.00 Hz
GB                   0
PC                         1.40
```



SH-21-23

^{1}H NMR, d_6-DMSO, 400 MHz




```
Current Data Parameters
NAME                  sh-21-23
EXPNO                      380
PROCNO                       1

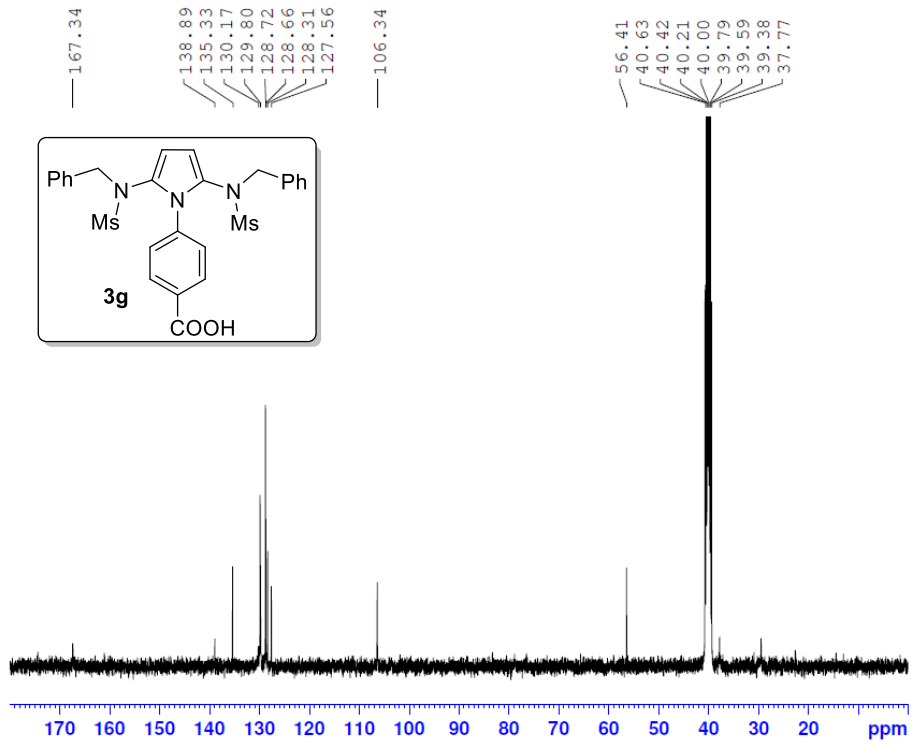
F2 - Acquisition Parameters
Date_                 20220208
Time                     12.28
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                      65536
SOLVENT                  DMSO
NS                          8
DS                          0
SWH                  9615.385 Hz
FIDRES               0.146719 Hz
AQ                   3.4078720 sec
RG                      114.26
DW                      52.000 usec
DE                       6.50 usec
TE                      300.0 K
D1                   1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                400.1629712 MHz
NUC1                        1H
P1                      13.20 usec
PLW1                13.00000000 W

F2 - Processing parameters
SI                          65536
SF                  400.1605021 MHz
WDW                            EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```

SH-21-23

^{13}C NMR, d_6-DMSO, 101 MHz




```
Current Data Parameters
NAME                  sh-21-23
EXPNO                      370
PROCNO                       1

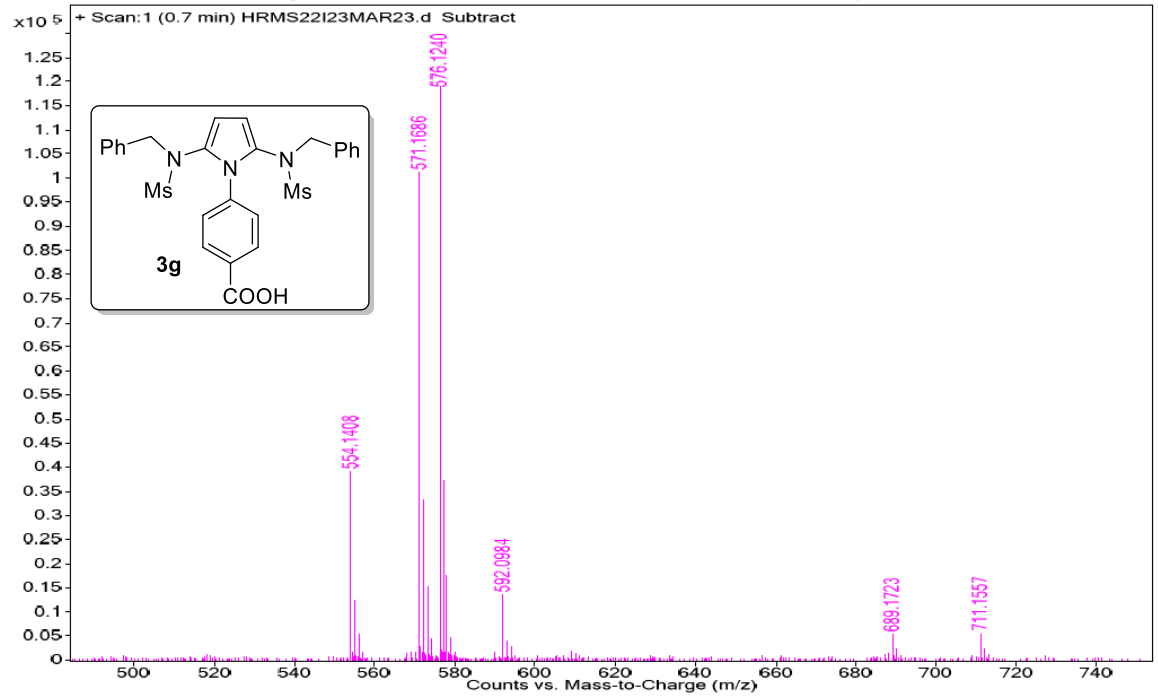
F2 - Acquisition Parameters
Date_                 20220210
Time                     10.25
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                zgpg30
TD                      65536
SOLVENT                  DMSO
NS                        2048
DS                          0
SWH                 24038.461 Hz
FIDRES               0.366798 Hz
AQ                   1.3631488 sec
RG                      201.48
DW                      20.800 usec
DE                       6.50 usec
TE                      300.1 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                100.6304993 MHz
NUC1                        13C
P1                       9.90 usec
PLW1                53.00000000 W

======== CHANNEL f2 ========
SFO2                400.1621006 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                   90.00 usec
PLW2                13.00000000 W
PLW12                0.27963999 W
PLW13                0.22651000 W

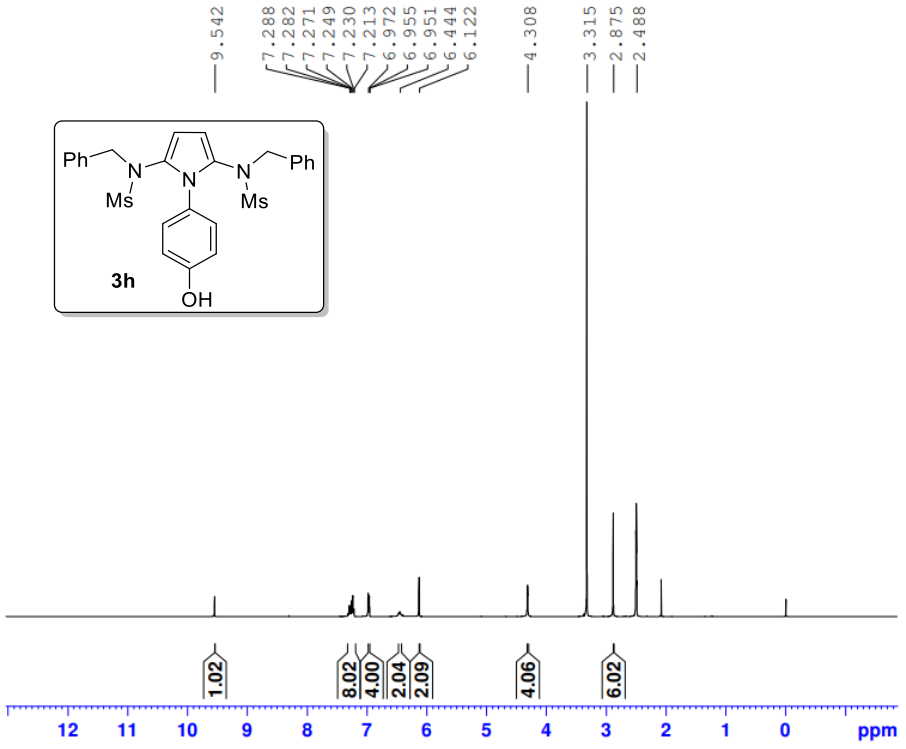
F2 - Processing parameters
SI                          32768
SF                  100.6204380 MHz
WDW                            EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40
```

Sample Name	GR-22-22	**Position**	Vial 23	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	HRMS22I23MAR23.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	3/23/2022 12:37:52 PM



^{1}H NMR, d_6-DMSO, 400 MHz

GR-21-67




```
Current Data Parameters
NAME                  1h
EXPNO                550
PROCNO                 1

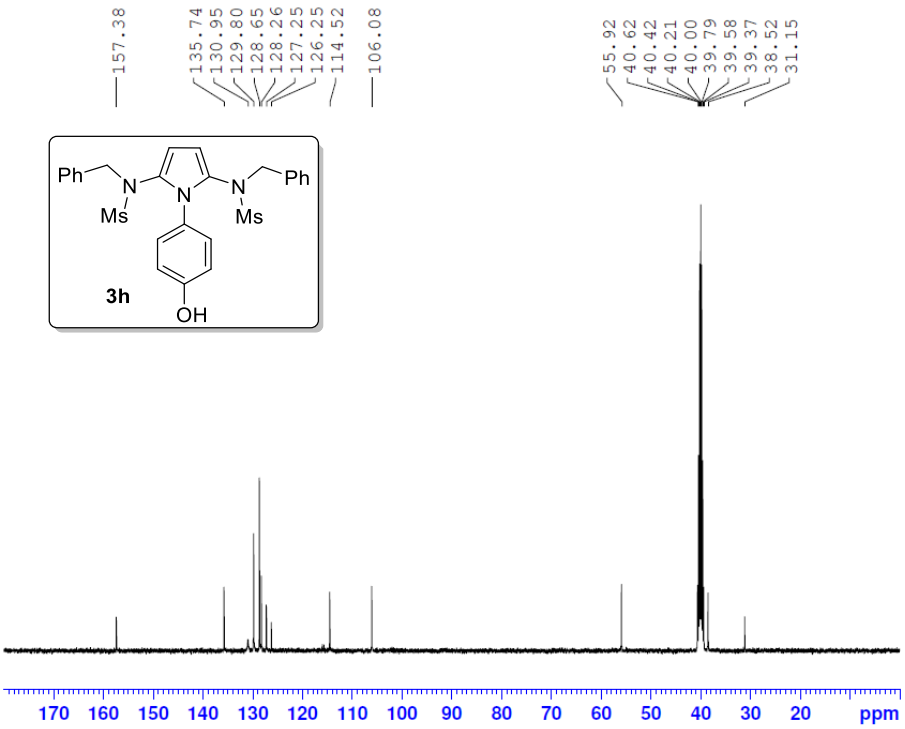
F2 - Acquisition Parameters
Date_           20220527
Time               12.55
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    0
SWH            9615.385 Hz
FIDRES        0.146719 Hz
AQ            3.4078720 sec
RG                114.26
DW                52.000 usec
DE                 6.50 usec
TE                300.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1629712 MHz
NUC1                   1H
P1                 13.20 usec
PLW1        13.00000000 W

F2 - Processing parameters
SI                    65536
SF           400.1624082 MHz
WDW                     EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```

GR-21-67

^{13}C NMR, d_6-DMSO, 101 MHz




```
Current Data Parameters
NAME                  GR-21-67
EXPNO                      360
PROCNO                       1

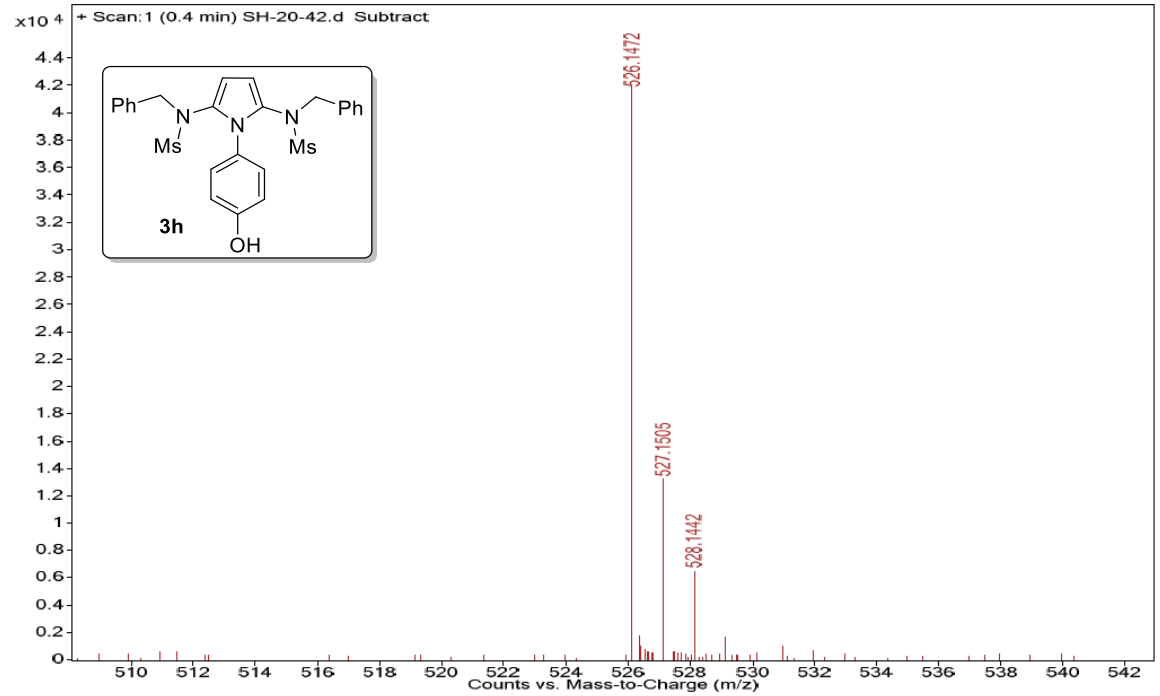
F2 - Acquisition Parameters
Date_                 20211214
Time                     15.57
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                zgpg30
TD                      65536
SOLVENT                   DMSO
NS                        2048
DS                           0
SWH                  24038.461 Hz
FIDRES                0.366798 Hz
AQ                    1.3631488 sec
RG                       201.48
DW                       20.800 usec
DE                        6.50 usec
TE                       300.0 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                100.6304993 MHz
NUC1                        13C
P1                         9.90 usec
PLW1                53.00000000 W

======== CHANNEL f2 ========
SFO2                400.1621006 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                     90.00 usec
PLW2                13.00000000 W
PLW12                0.27963999 W
PLW13                0.22651000 W

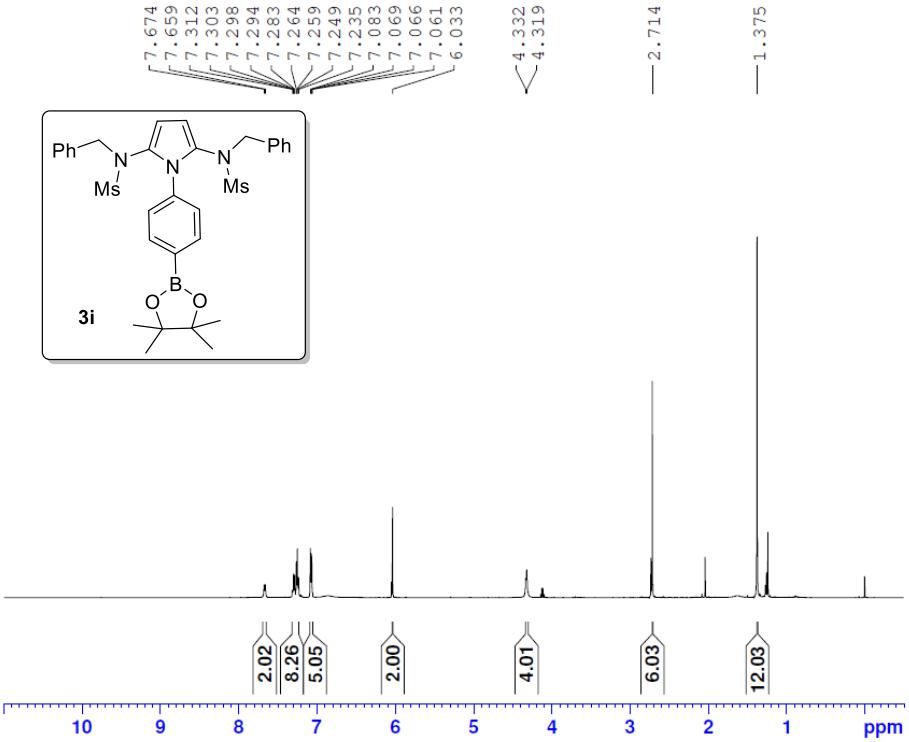
F2 - Processing parameters
SI                        32768
SF                  100.6204380 MHz
WDW                          EM
SSB                  0
LB                         1.00 Hz
GB                   0
PC                         1.40
```

Sample Name	HRMS22I20JAN22	**Position**	Vial 22	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	SH-20-42.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	1/20/2022 12:28:29 PM



GR-21-14

^{1}H NMR, $CDCl_3$, 500 MHz




```
Current Data Parameters
NAME            GR-21-14
EXPNO                390
PROCNO                 1

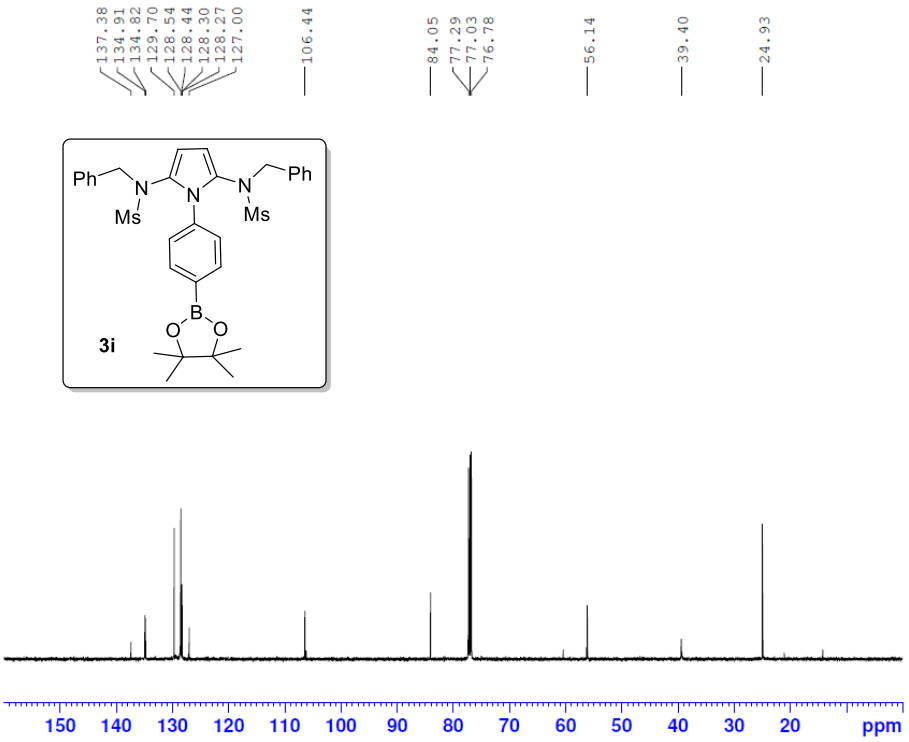
F2 - Acquisition Parameters
Date_           20211028
Time               14.37
INSTRUM            spect
PROBHD   5 mm PATXI 1H/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10026.738 Hz
FIDRES         0.152996 Hz
AQ             3.2680619 sec
RG                 69.83
DW                49.867 usec
DE                 6.50 usec
TE                303.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         500.6794419 MHz
NUC1                   1H
P1                  6.45 usec
PLW1        13.60000038 W

F2 - Processing parameters
SI                 65536
SF           500.6763622 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

GR-21-14

^{13}C NMR, $CDCl_3$, 125 MHz




```
Current Data Parameters
NAME             Desktop
EXPNO                400
PROCNO                 1

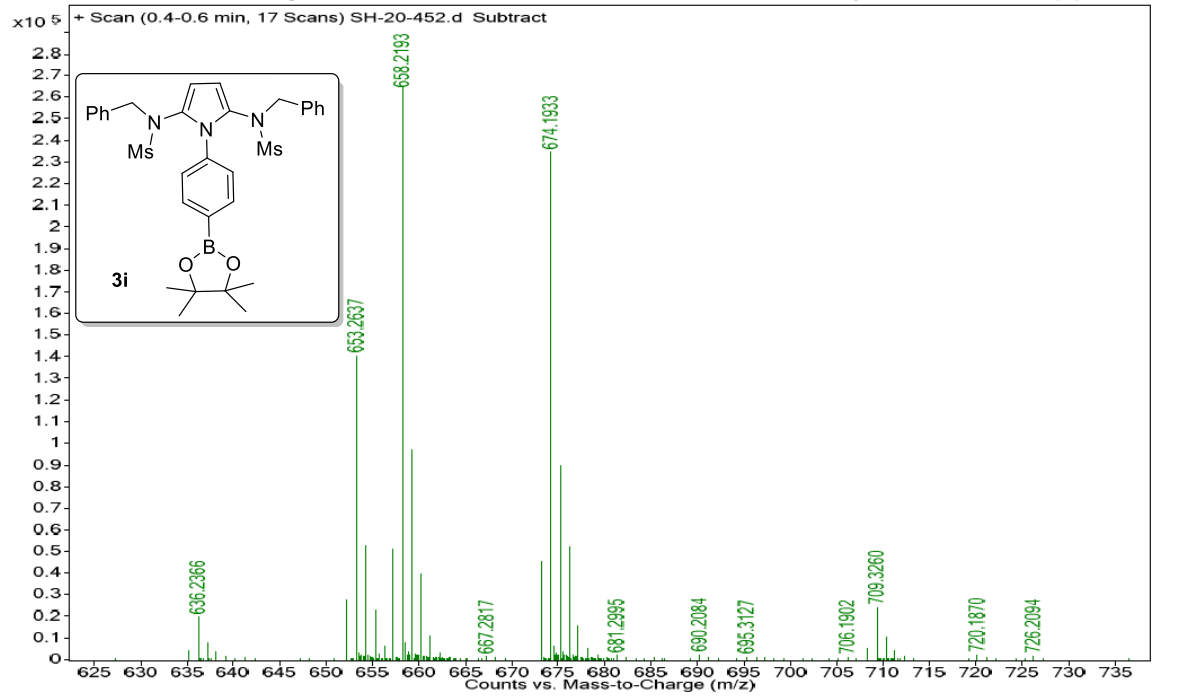
F2 - Acquisition Parameters
Date_           20211028
Time               15.43
INSTRUM            spect
PROBHD   5 mm PATXI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 2048
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ             1.1010048 sec
RG                 56.22
DW                16.800 usec
DE                 6.50 usec
TE                303.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         125.9077573 MHz
NUC1                   13C
P1                  9.23 usec
PLW1       244.00000000 W

======== CHANNEL f2 ========
SFO2         500.6783527 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2        13.60000038 W
PLW12        0.08840500 W
PLW13        0.05657900 W

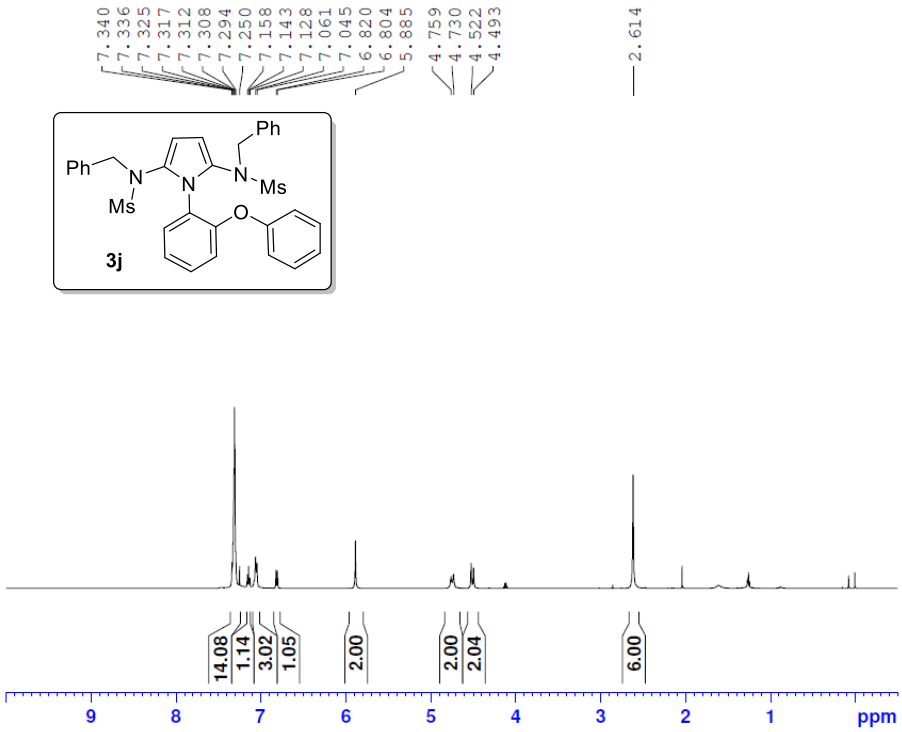
F2 - Processing parameters
SI                 32768
SF           125.8951680 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

Sample Name	HRMS21I13DEC13	**Position**	Vial 13	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	SH-20-452.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	12/13/2021 12:23:20 PM



GR-21-16

^{1}H NMR, $CDCl_3$, 500 MHz




```
Current Data Parameters
NAME                  Desktop
EXPNO                     510
PROCNO                      1

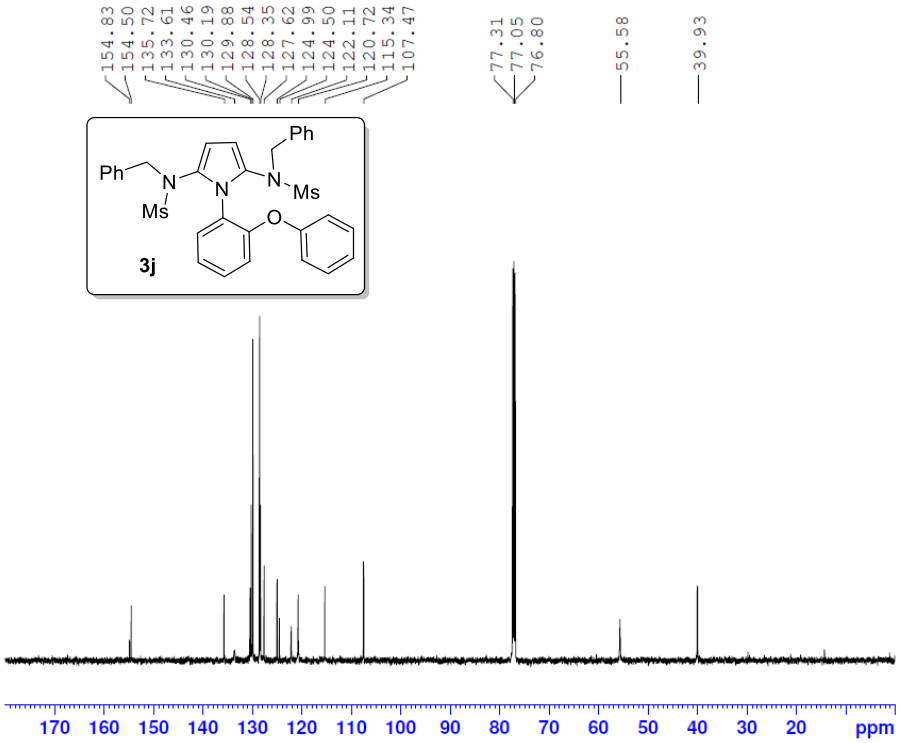
F2 - Acquisition Parameters
Date_               20211029
Time                   12.37
INSTRUM                spect
PROBHD    5 mm PATXI 1H/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH               10026.738 Hz
FIDRES             0.152996 Hz
AQ                 3.2680619 sec
RG                      61.7
DW                    49.867 usec
DE                     6.50 usec
TE                    303.0 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            500.6794419 MHz
NUC1                      1H
P1                     6.45 usec
PLW1           13.60000038 W

F2 - Processing parameters
SI                    65536
SF              500.6763669 MHz
WDW                      EM
SSB          0
LB                     0.30 Hz
GB           0
PC                     1.00
```

GR-21-16

^{13}C NMR, $CDCl_3$, 125 MHz




```
Current Data Parameters
NAME                  GR-21-16
EXPNO                      520
PROCNO                       1

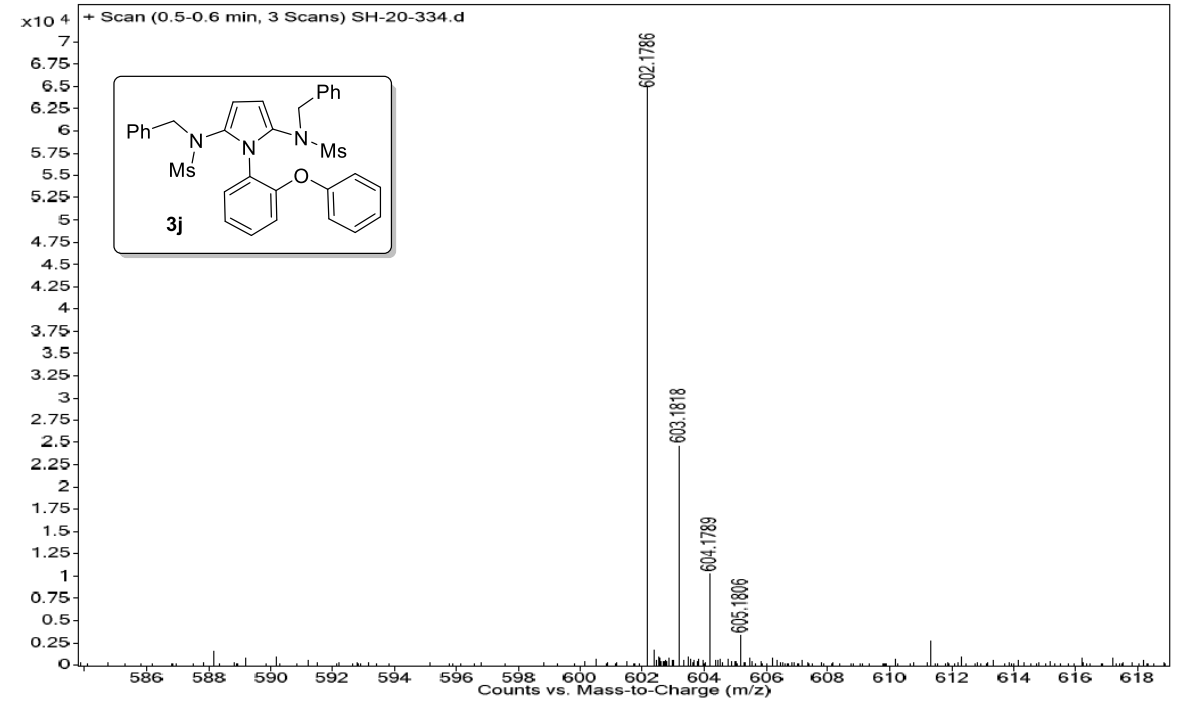
F2 - Acquisition Parameters
Date_                 20211029
Time                     12.47
INSTRUM                  spect
PROBHD    5 mm PATXI 1H/
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                       2048
DS                          4
SWH                29761.904 Hz
FIDRES                 0.454131 Hz
AQ                     1.1010048 sec
RG                        56.22
DW                       16.800 usec
DE                        6.50 usec
TE                       303.0 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                125.9077573 MHz
NUC1                       13C
P1                        9.23 usec
PLW1               244.00000000 W

======== CHANNEL f2 ========
SFO2                500.6783527 MHz
NUC2                       1H
CPDPRG[2                waltz16
PCPD2                    80.00 usec
PLW2                13.60000038 W
PLW12                0.08840500 W
PLW13                0.05657900 W

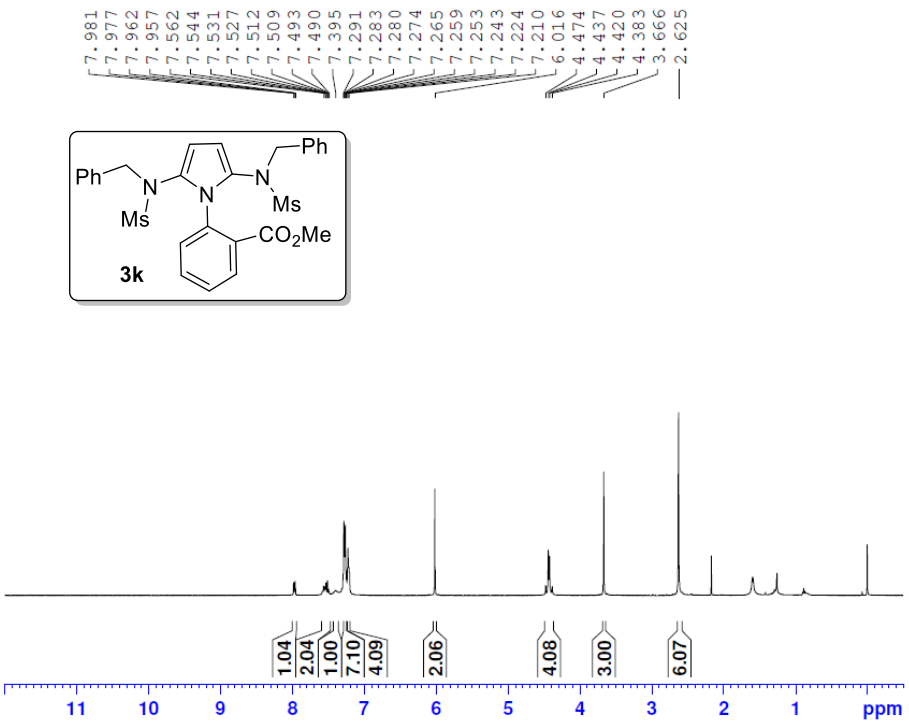
F2 - Processing parameters
SI                        32768
SF                  125.8951680 MHz
WDW                          EM
SSB                 0
LB                        2.00 Hz
GB                  0
PC                        1.40
```

Sample Name	HRMS22I04JAN21	**Position**	Vial 21	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	SH-20-334.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	1/4/2022 12:29:35 PM



GR-20-232

^{1}H NMR, $CDCl_3$, 400 MHz




```
Current Data Parameters
NAME           GR-20-232
EXPNO                400
PROCNO                 1

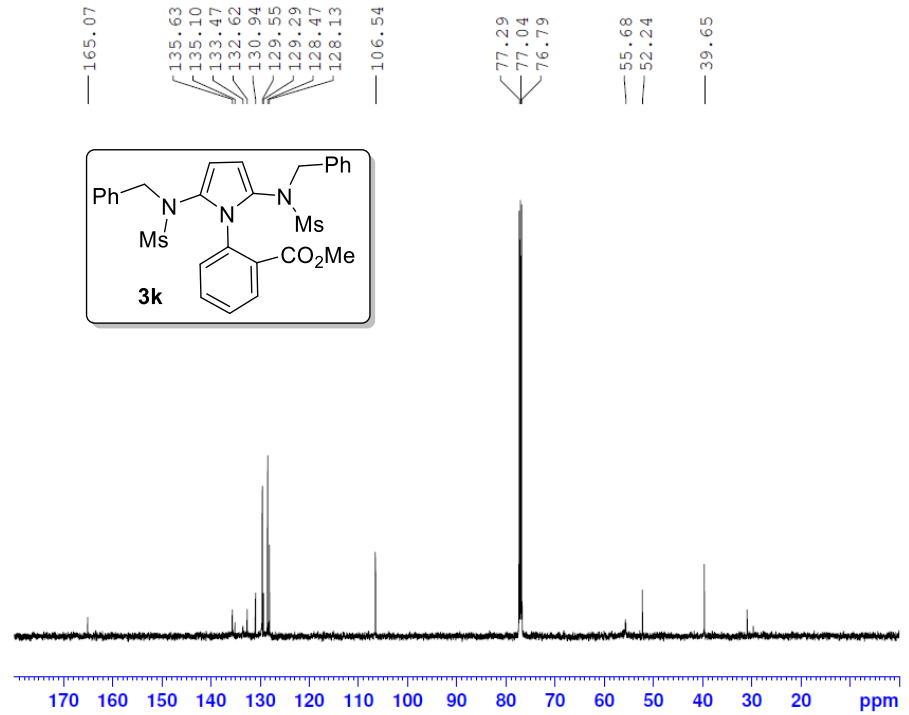
F2 - Acquisition Parameters
Date_           20211210
Time               12.33
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH             9615.385 Hz
FIDRES          0.146719 Hz
AQ              3.4078720 sec
RG                129.57
DW                52.000 usec
DE                 6.50 usec
TE                300.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1629712 MHz
NUC1                  1H
P1                 13.20 usec
PLW1          13.00000000 W

F2 - Processing parameters
SI                 65536
SF            400.1605097 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

GR-20-232

^{13}C NMR, $CDCl_3$, 125 MHz




```
Current Data Parameters
NAME           GR-20-232
EXPNO                370
PROCNO                 1

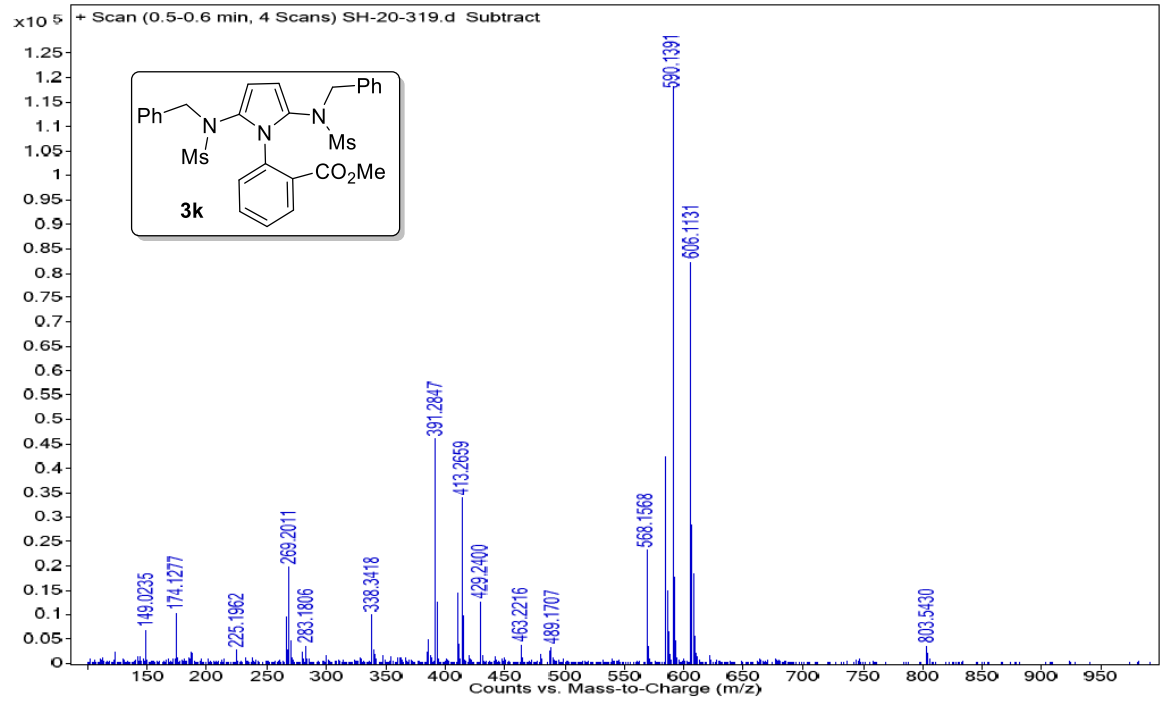
F2 - Acquisition Parameters
Date_           20211214
Time               8.44
INSTRUM            spect
PROBHD   5 mm PATXI 1H/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  2048
DS                     4
SWH            29761.904 Hz
FIDRES          0.454131 Hz
AQ              1.1010048 sec
RG                107.78
DW                16.800 usec
DE                 6.50 usec
TE                300.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          125.9077573 MHz
NUC1                  13C
P1                 9.23 usec
PLW1        244.00000000 W

======== CHANNEL f2 ========
SFO2          500.6783527 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2             80.00 usec
PLW2         13.60000038 W
PLW12         0.08840500 W
PLW13         0.05657900 W

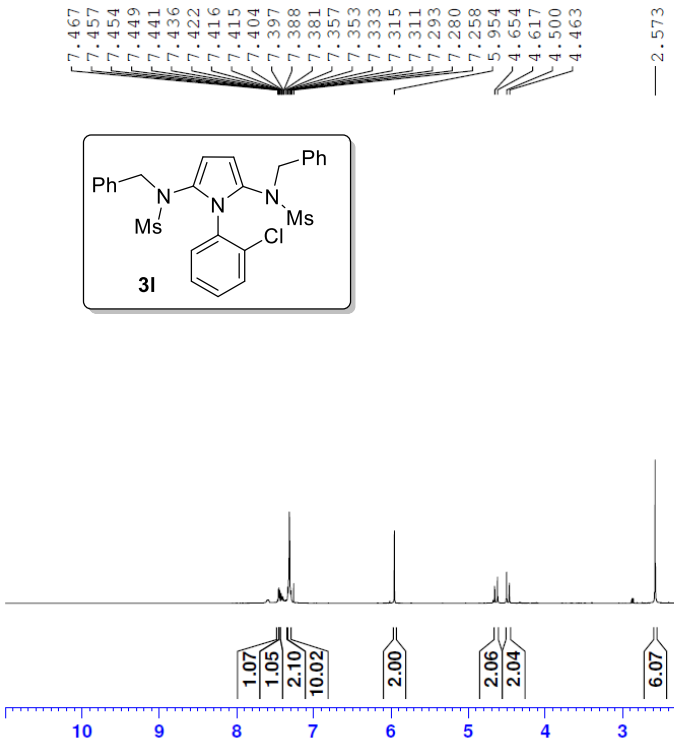
F2 - Processing parameters
SI                 32768
SF            125.8951680 MHz
WDW                   EM
SSB       0
LB                 2.00 Hz
GB        0
PC                 1.40
```

Sample Name	HRMS22I04JAN20	**Position**	Vial 20	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	SH-20-319.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	1/4/2022 12:27:02 PM



SH-21-72

^{1}H NMR, $CDCl_3$, 400 MHz




```
Current Data Parameters
NAME                  1h
EXPNO                420
PROCNO                 1

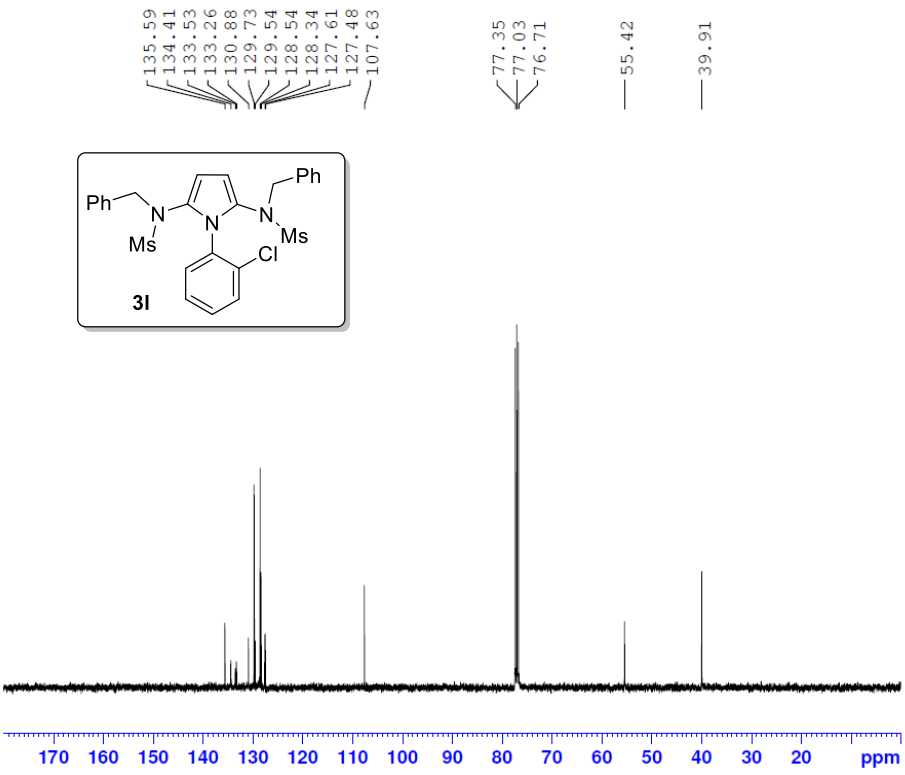
F2 - Acquisition Parameters
Date_           20220628
Time               14.19
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH             9615.385 Hz
FIDRES          0.146719 Hz
AQ              3.4078720 sec
RG                129.57
DW                52.000 usec
DE                 6.50 usec
TE                300.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1629712 MHz
NUC1                  1H
P1                 13.20 usec
PLW1          13.00000000 W

F2 - Processing parameters
SI                 65536
SF            400.1605100 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

SH-21-72

^{13}C NMR, $CDCl_3$, 101 MHz




```
Current Data Parameters
NAME                  13c
EXPNO                 430
PROCNO                  1

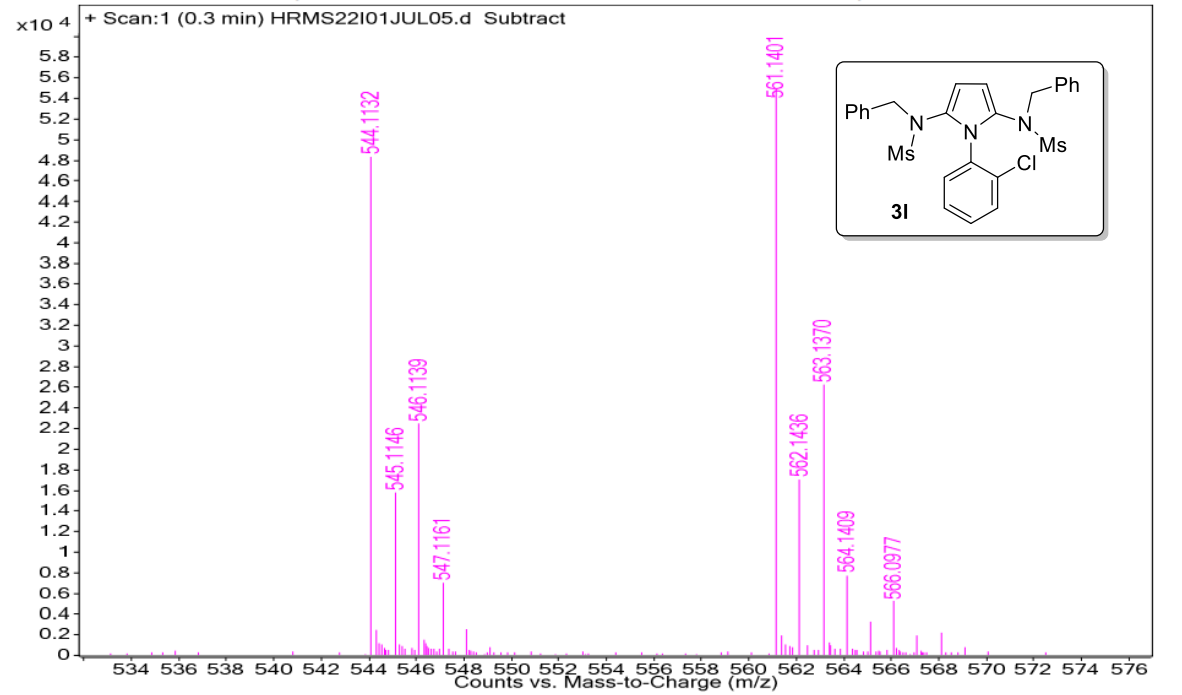
F2 - Acquisition Parameters
Date_              20220628
Time                 14.25
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  2048
DS                     0
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631488 sec
RG                201.48
DW                20.800 usec
DE                 6.50 usec
TE                300.0 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          100.6304993 MHz
NUC1                 13C
P1                  9.90 usec
PLW1         53.00000000 W

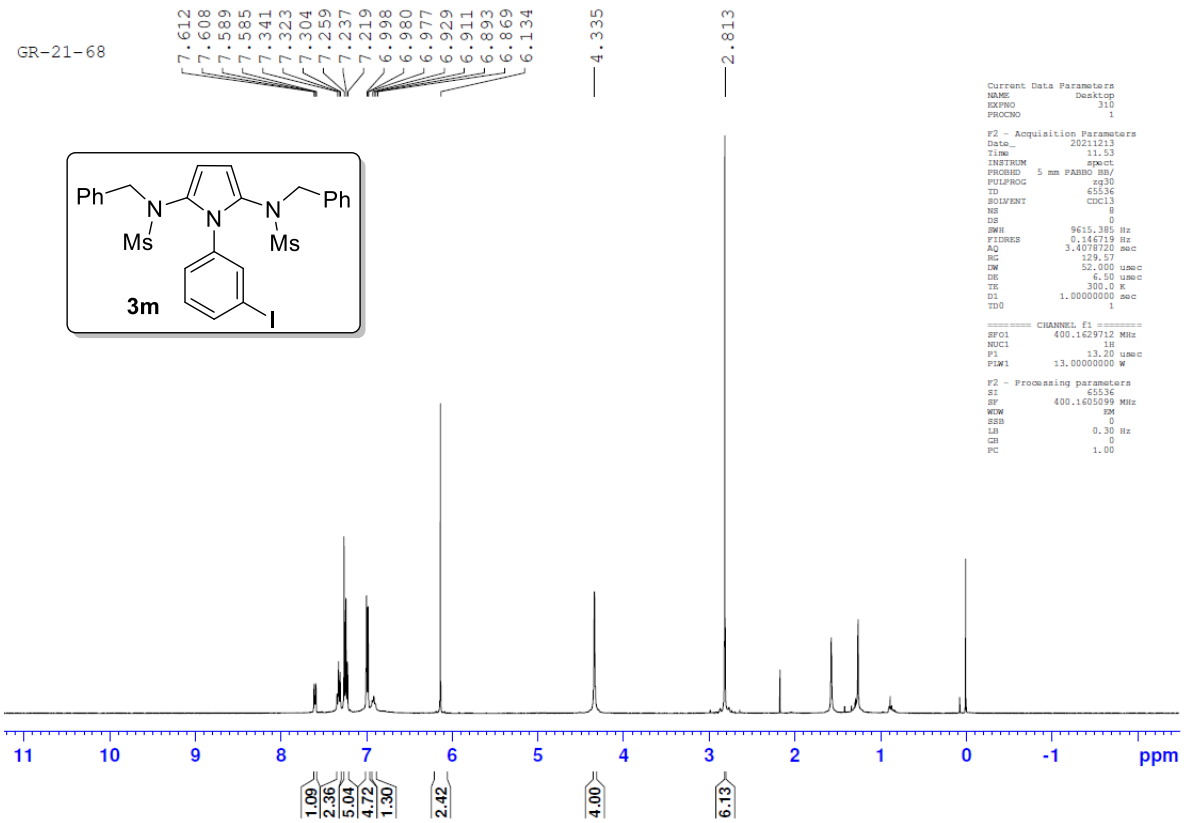
======== CHANNEL f2 ========
SFO2          400.1621006 MHz
NUC2                 1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2          13.00000000 W
PLW12          0.27963999 W
PLW13          0.22651000 W

F2 - Processing parameters
SI                  32768
SF            100.6204380 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

Sample Name	SH-21-09	**Position**	Vial 5	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	HRMS22I01JUL05.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	7/1/2022 12:19:51 PM



^{1}H NMR, $CDCl_3$, 400 MHz



Current Data Parameters
NAME Desktop
EXPNO 310
PROCNO 1

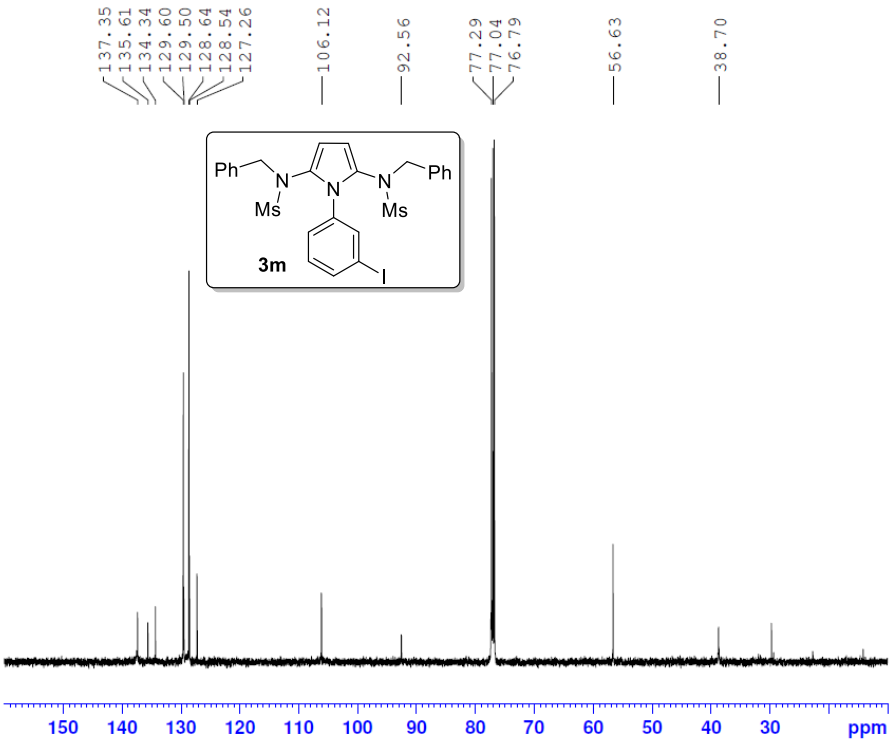
F2 - Acquisition Parameters
Date_ 20211213
Time 11.53
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 9615.385 Hz
FIDRES 0.146719 Hz
AQ 3.4078720 sec
RG 129.57
DW 52.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1629712 MHz
NUC1 1H
P1 13.20 usec
PLW1 13.00000000 W

F2 - Processing parameters
SI 65536
SF 400.1605099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

GR-21-68

^{13}C NMR, $CDCl_3$, 125 MHz



Current Data Parameters
NAME GR-21-68
EXPNO 340
PROCNO 1

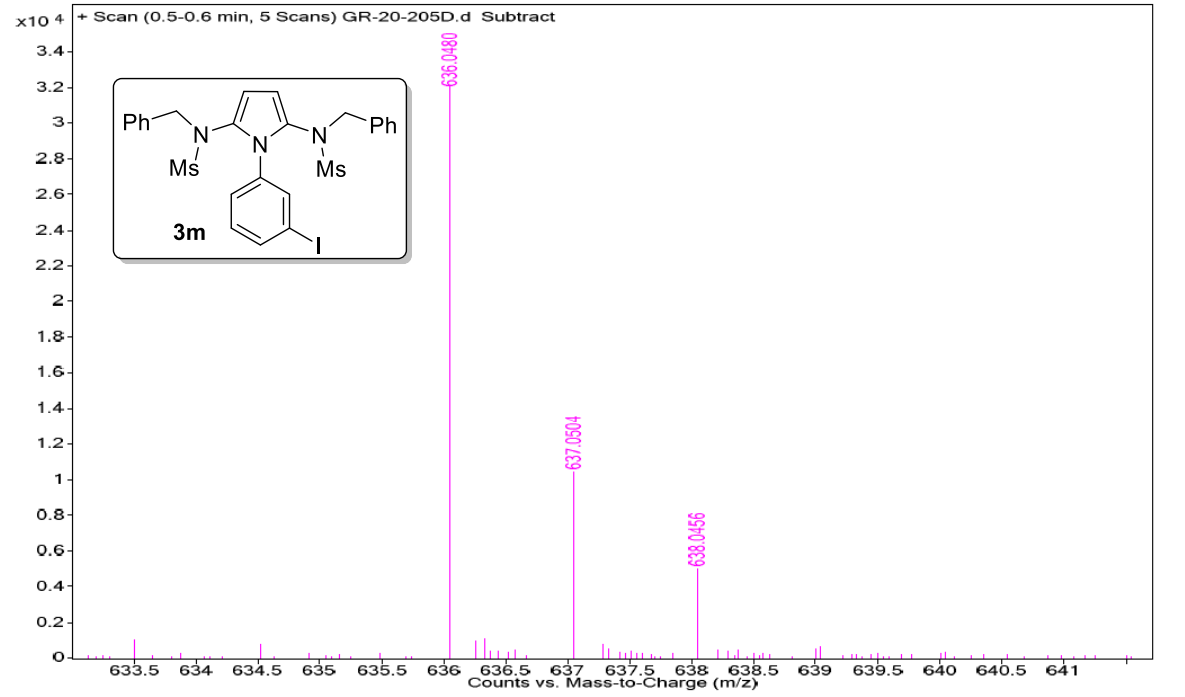
F2 - Acquisition Parameters
Date_ 20211213
Time 7.43
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 107.78
DW 16.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 125.9077573 MHz
NUC1 13C
P1 9.23 usec
PLW1 244.00000000 W

======== CHANNEL f2 ========
SFO2 500.6783527 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.60000038 W
PLW12 0.08840500 W
PLW13 0.05657900 W

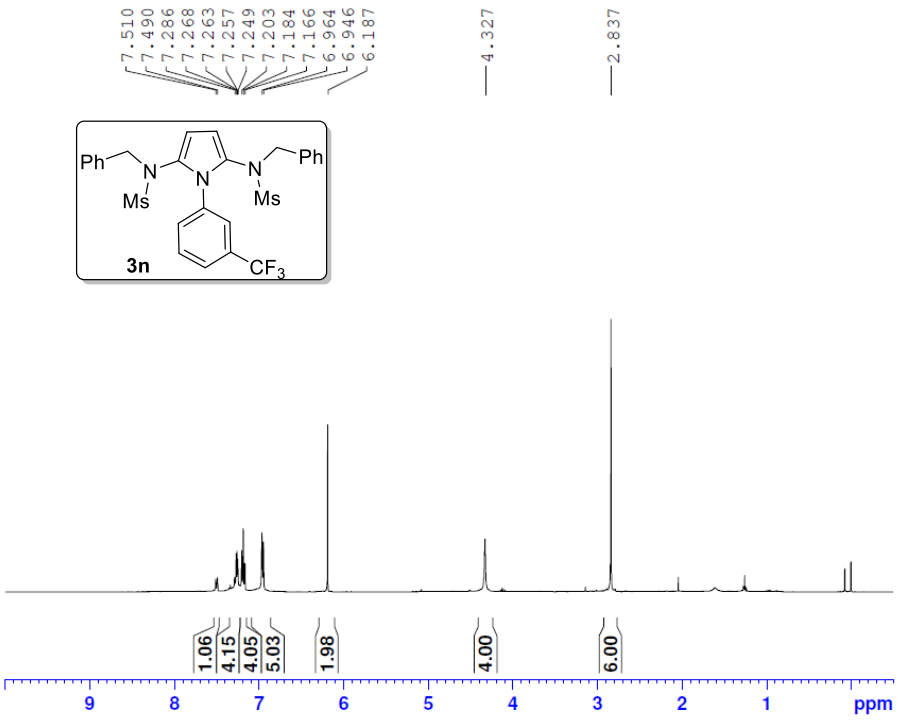
F2 - Processing parameters
SI 32768
SF 125.8951680 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Sample Name	HRMS21I15DEC22	**Position**	Vial 22	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	GR-20-205D.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	12/15/2021 1:40:37 PM



GR 21 09

^{1}H NMR, $CDCl_3$, 400 MHz




```
Current Data Parameters
NAME            GR-21-09
EXPNO                400
PROCNO                 1

F2 - Acquisition Parameters
Date_           20211102
Time               12.37
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDC13
NS                     8
DS                     0
SWH             9615.385 Hz
FIDRES          0.146719 Hz
AQ              3.4078720 sec
RG                 80.54
DW                 52.000 usec
DE                  6.50 usec
TE                 300.0 K
D1            1.00000000 sec
TD0                    1

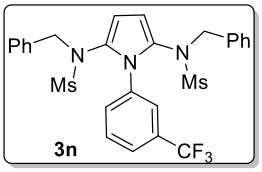
======== CHANNEL f1 ========
SFO1         400.1629712 MHz
NUC1                   1H
P1                 13.20 usec
PLW1         13.00000000 W

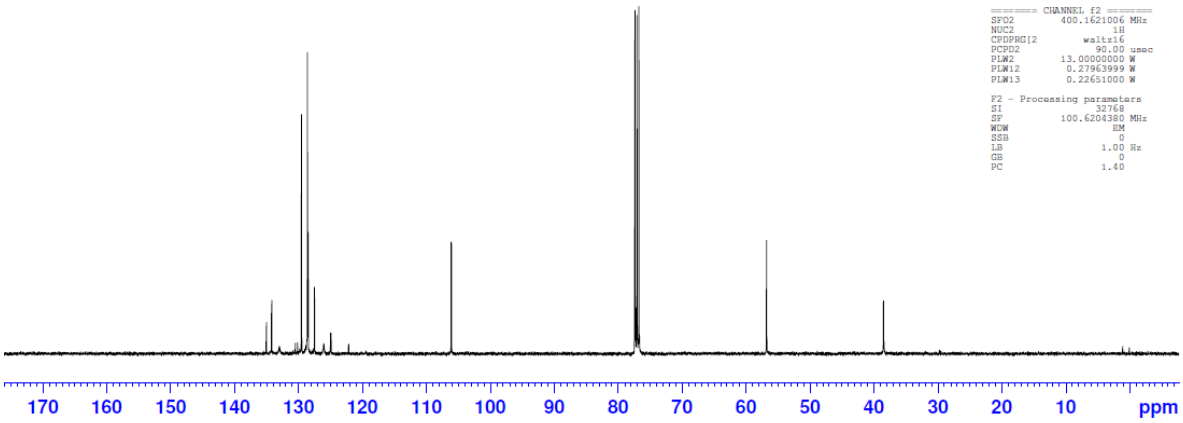
F2 - Processing parameters
SI                 65536
SF           400.1605107 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

GR 21 09

135.03
134.19
132.99
130.48
129.15
128.65
128.60
128.47
127.48
126.03
124.96
124.93
122.16
106.09
77.36
77.04
76.72
56.75
38.46

^{13}C NMR, $CDCl_3$, 101 MHz






```
Current Data Parameters
NAME                  Desktop
EXPNO                     580
PROCNO                      1

F2 - Acquisition Parameters
Date_               20211102
Time                   12.48
INSTRUM                spect
PROBHD   5 mm PABBO BB/
PULPROG             zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                       1200
DS                          0
SWH                24038.461 Hz
FIDRES             0.366798 Hz
AQ                 1.36314880 sec
RG                     201.48
DW                   20.800 usec
DE                    6.50 usec
TE                   300.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1               100.6304993 MHz
NUC1                       13C
P1                        9.90 usec
PLW1               53.00000000 W

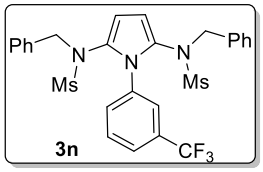
======== CHANNEL f2 ========
SFO2               400.1621006 MHz
NUC2                       1H
CPDPRG[2                waltz16
PCPD2                    90.00 usec
PLW2               13.00000000 W
PLW12               0.27963999 W
PLW13               0.22651000 W

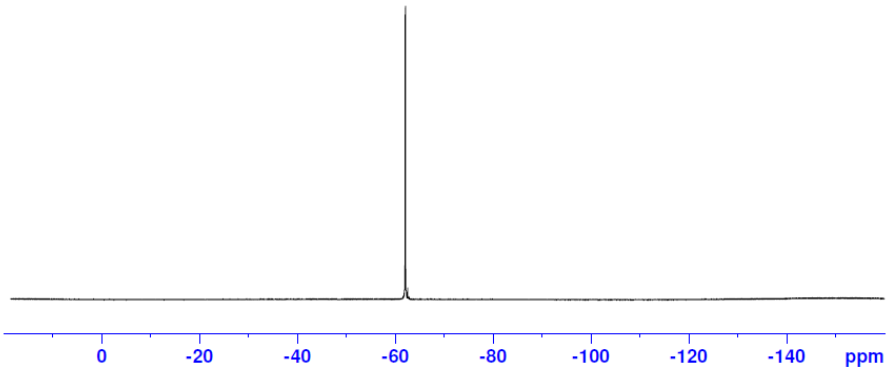
F2 - Processing parameters
SI                        32768
SF                 100.6204380 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40
```

GR 21 09

^{19}F NMR, $CDCl_3$, 376 MHz

−62.07






```
Current Data Parameters
NAME                  GR-21-09
EXPNO                      410
PROCNO                       1

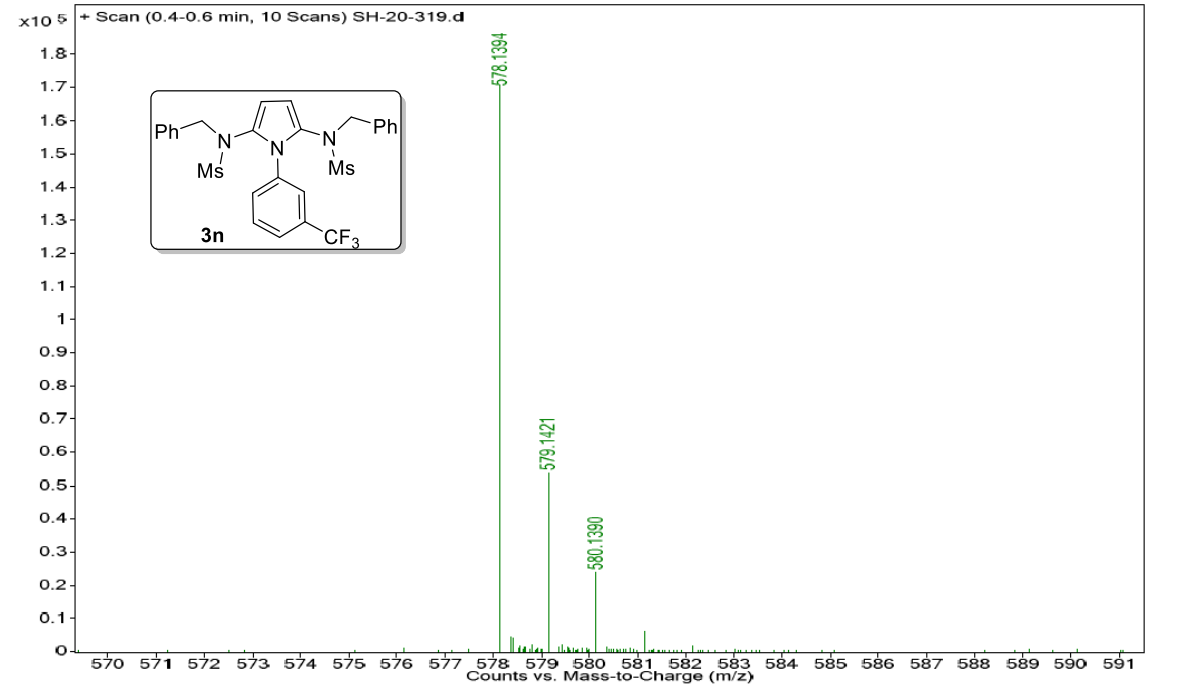
F2 - Acquisition Parameters
Date_               20211102
Time                   12.39
INSTRUM                spect
PROBHD   5 mm PABBO BB/
PULPROG             zgfhigqn.2
TD                      131072
SOLVENT                 CDCl3
NS                          16
DS                           4
SWH                89285.711 Hz
FIDRES             0.681196 Hz
AQ                 0.7340032 sec
RG                     201.48
DW                    5.600 usec
DE                    6.50 usec
TE                   300.0 K
D1                 1.00000000 sec
D11                0.03000000 sec
D12                0.00002000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1               376.4894122 MHz
NUC1                       19F
P1                       15.00 usec
PLW1               21.00000000 W

======== CHANNEL f2 ========
SFO2               400.1621006 MHz
NUC2                       1H
CPDPRG[2                waltz16
PCPD2                    90.00 usec
PLW2               13.00000000 W
PLW12               0.27963999 W

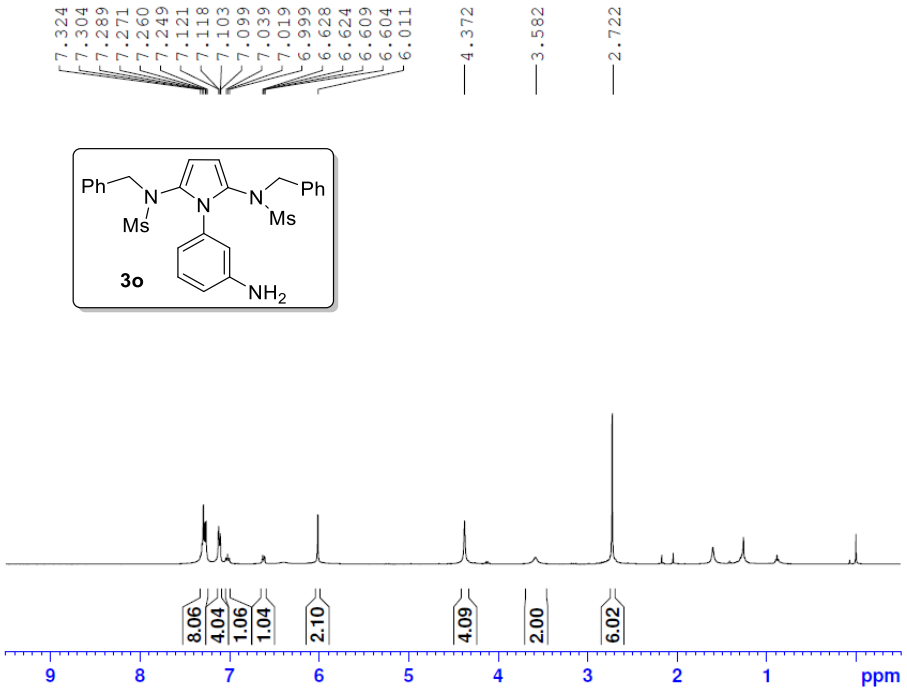
F2 - Processing parameters
SI                        65536
SF                 376.5270650 MHz
WDW                          EM
SSB                   0
LB                         0.30 Hz
GB                    0
PC                         1.00
```

Sample Name	HRMS21I27DEC20	**Position**	Vial 20	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	SH-20-319.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	12/28/2021 12:40:54 PM



GR-21-62A

^{1}H NMR, $CDCl_3$, 400 MHz




```
Current Data Parameters
NAME                  GR-21-62
EXPNO                      320
PROCNO                       1

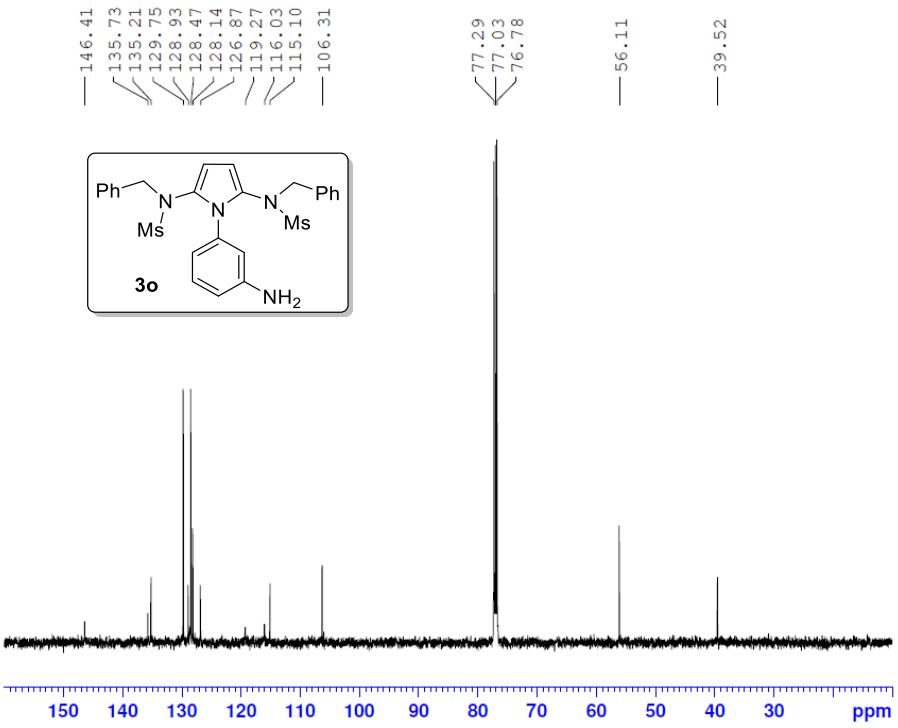
F2 - Acquisition Parameters
Date_                 20220115
Time                      2.53
INSTRUM                  spect
PROBHD     5 mm PABBO BB/
PULPROG                  zg30
TD                      65536
SOLVENT                 CDC13
NS                          8
DS                          0
SWH                  9615.385 Hz
FIDRES               0.146719 Hz
AQ                   3.4078720 sec
RG                      129.57
DW                      52.000 usec
DE                       6.50 usec
TE                      300.0 K
D1                   1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                400.1629712 MHz
NUC1                        1H
P1                      13.20 usec
PLW1                13.00000000 W

F2 - Processing parameters
SI                      65536
SF                  400.1605095 MHz
WDW                        EM
SSB                 0
LB                       0.30 Hz
GB                  0
PC                       1.00
```

GR-21-62

^{13}C NMR, $CDCl_3$, 125 MHz




```
Current Data Parameters
NAME                  GR-21-62
EXPNO                      370
PROCNO                       1

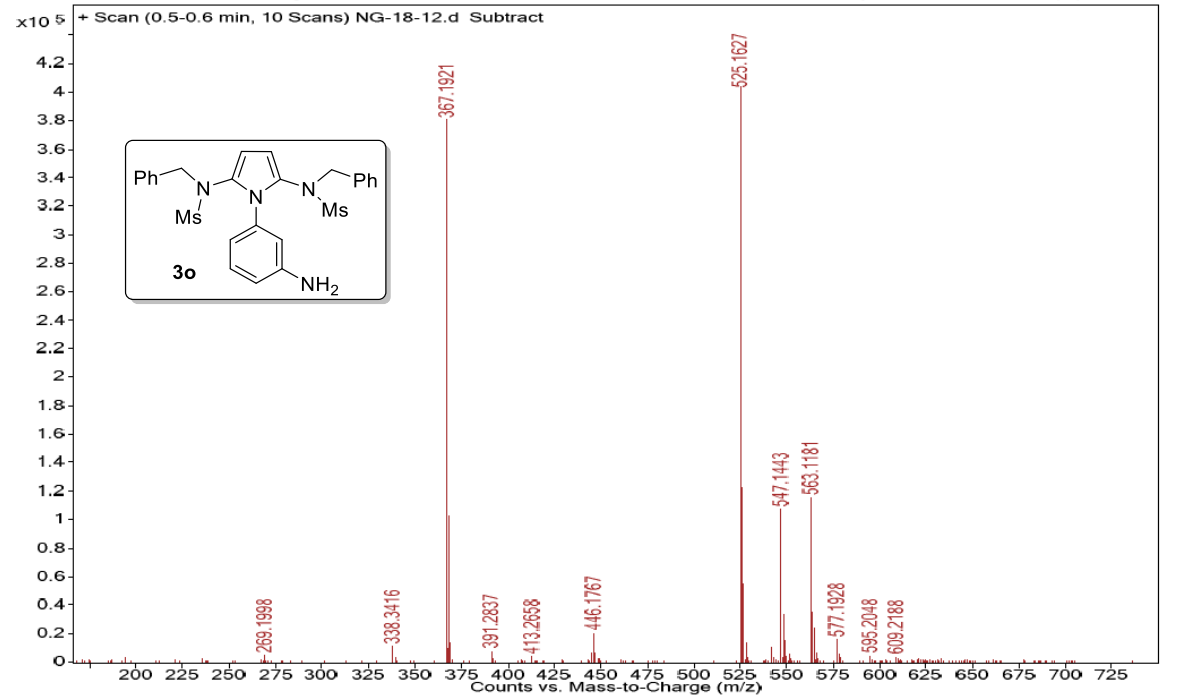
F2 - Acquisition Parameters
Date_                 20211203
Time                      8.47
INSTRUM                  spect
PROBHD    5 mm PATXI 1H/
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        2048
DS                           4
SWH                  29761.904 Hz
FIDRES                0.454131 Hz
AQ                    1.1010048 sec
RG                       56.22
DW                      16.800 usec
DE                       6.50 usec
TE                      303.0 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                125.9077573 MHz
NUC1                        13C
P1                         9.23 usec
PLW1               244.00000000 W

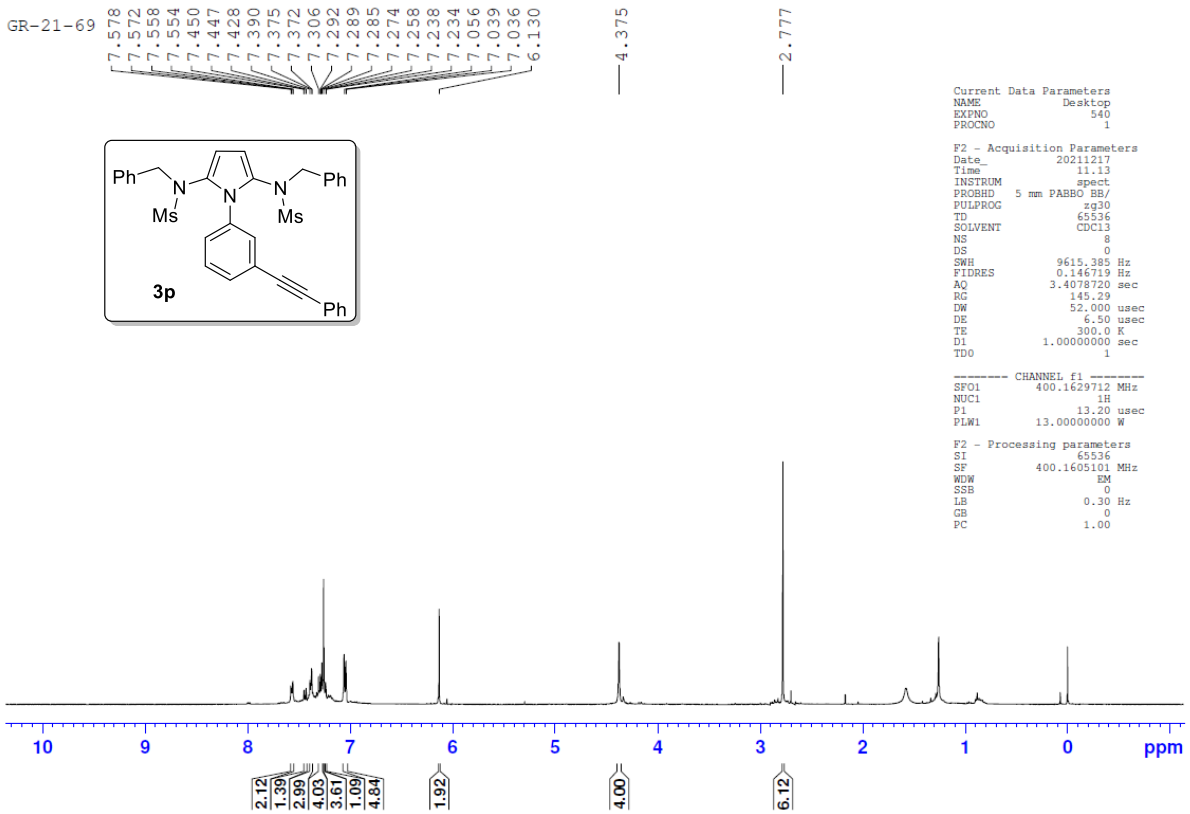
======== CHANNEL f2 ========
SFO2                500.6783527 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                    80.00 usec
PLW2                13.60000038 W
PLW12                0.08840500 W
PLW13                0.05657900 W

F2 - Processing parameters
SI                          32768
SF                  125.8951680 MHz
WDW                            EM
SSB                 0
LB                           2.00 Hz
GB                  0
PC                           1.40
```

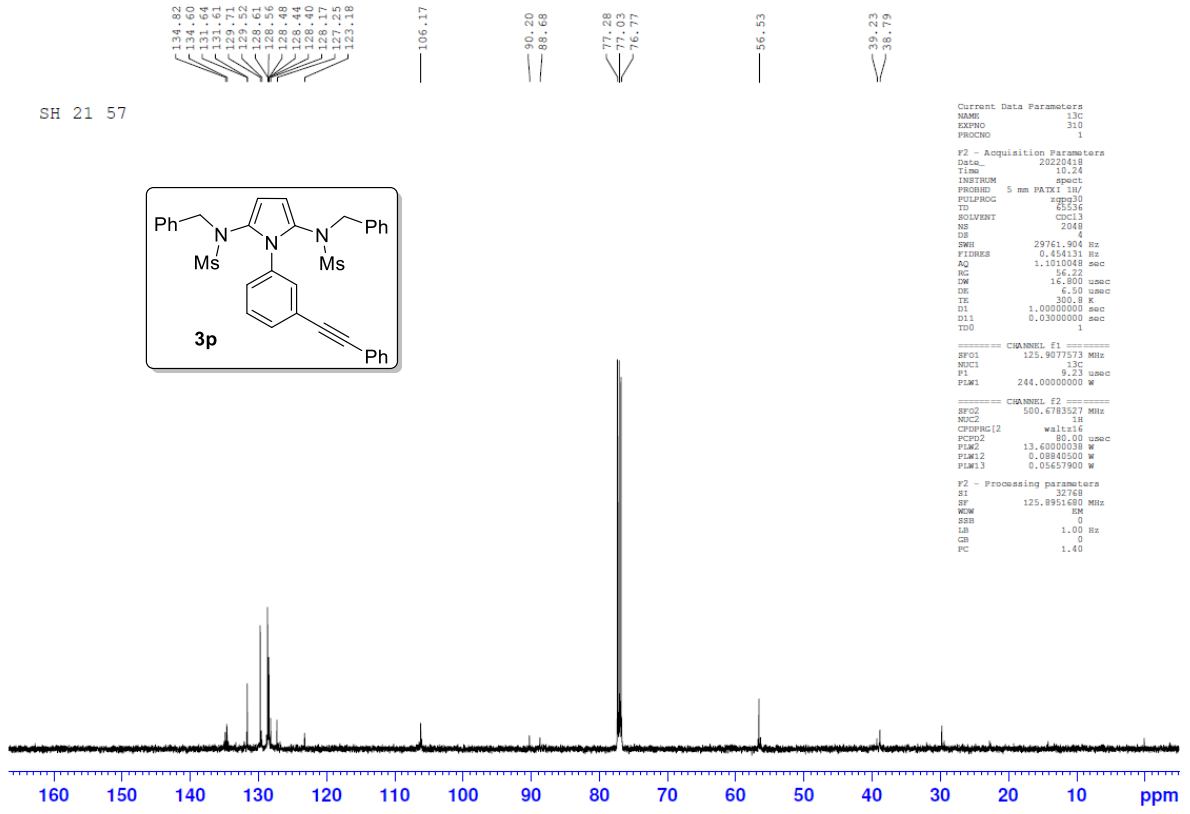
Sample Name	HRMS22I14JAN24	**Position**	Vial 24	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	NG-18-12.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	1/14/2022 12:54:26 PM



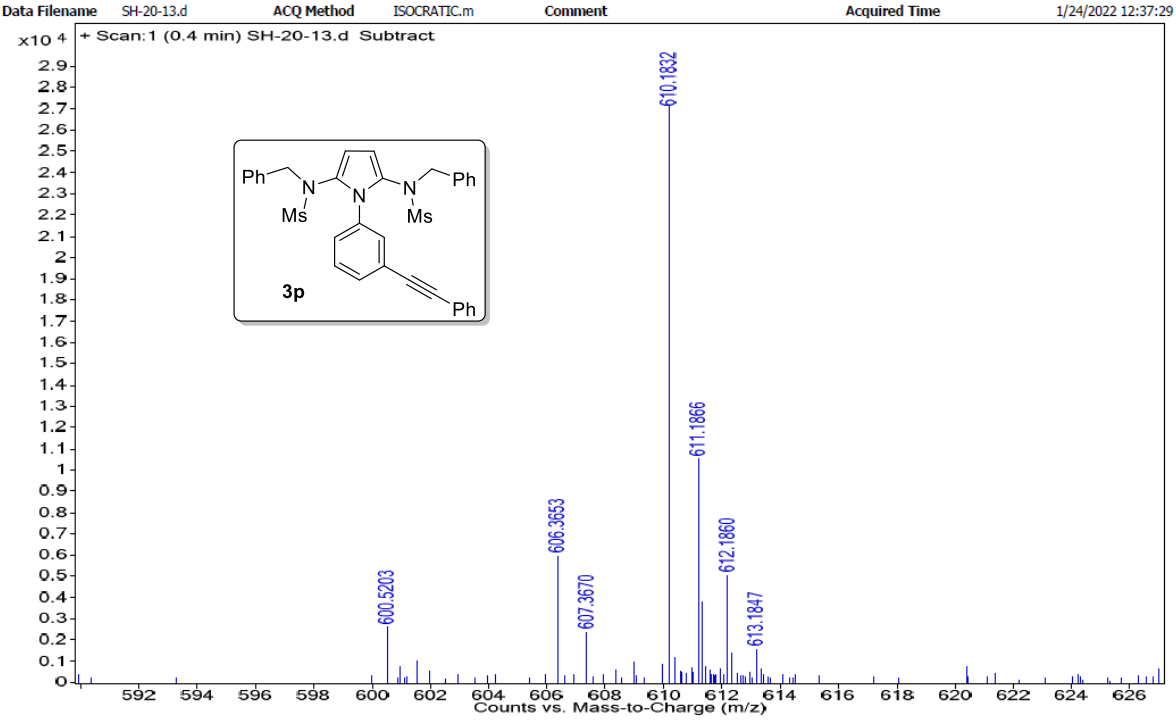
^{1}H NMR, $CDCl_3$, 400 MHz



^{13}C NMR, $CDCl_3$, 125 MHz

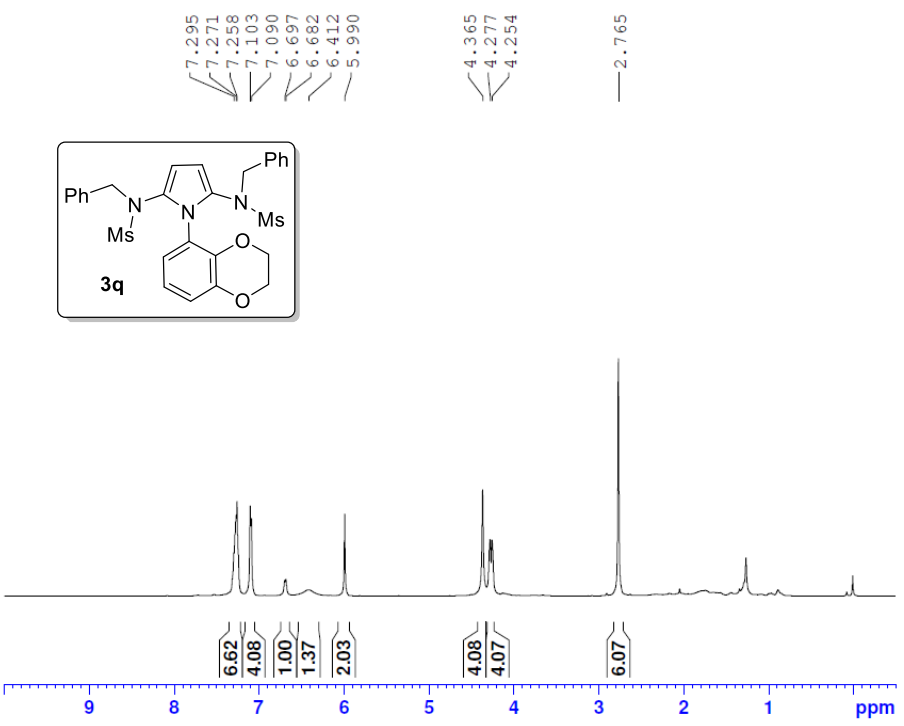


Sample Name	HRMS22I24JAN17	**Position**	Vial 17	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	SH-20-13.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	1/24/2022 12:37:29 PM



GR-21-15

^{1}H NMR, $CDCl_3$, 500 MHz




```
Current Data Parameters
NAME                  GR-21-15
EXPNO                      560
PROCNO                       1

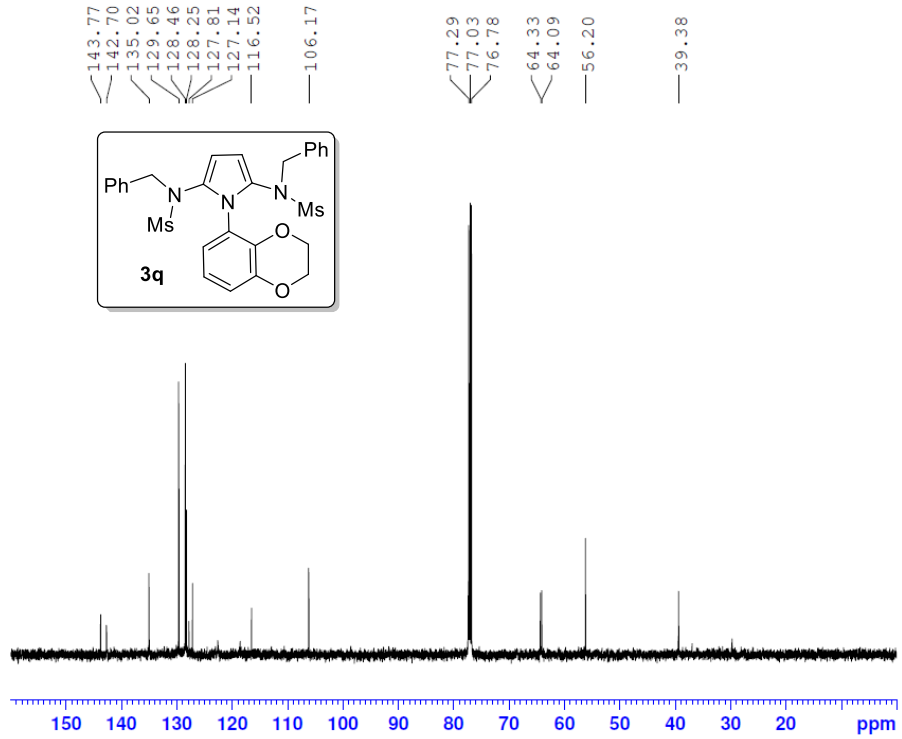
F2 - Acquisition Parameters
Date_                 20211101
Time                     11.31
INSTRUM                  spect
PROBHD     5 mm PATXI 1H/
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                         16
DS                          2
SWH                 10026.738 Hz
FIDRES                0.152996 Hz
AQ                    3.2680619 sec
RG                        87.95
DW                       49.867 usec
DE                        6.50 usec
TE                       303.0 K
D1                    1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                500.6794419 MHz
NUC1                        1H
P1                        6.45 usec
PLW1               13.60000038 W

F2 - Processing parameters
SI                          65536
SF                  500.6763602 MHz
WDW                            EM
SSB                 0
LB                            0.30 Hz
GB                  0
PC                            1.00
```

GR-21-15

^{13}C NMR, $CDCl_3$, 125 MHz




```
Current Data Parameters
NAME                  GR-21-15
EXPNO                      570
PROCNO                       1

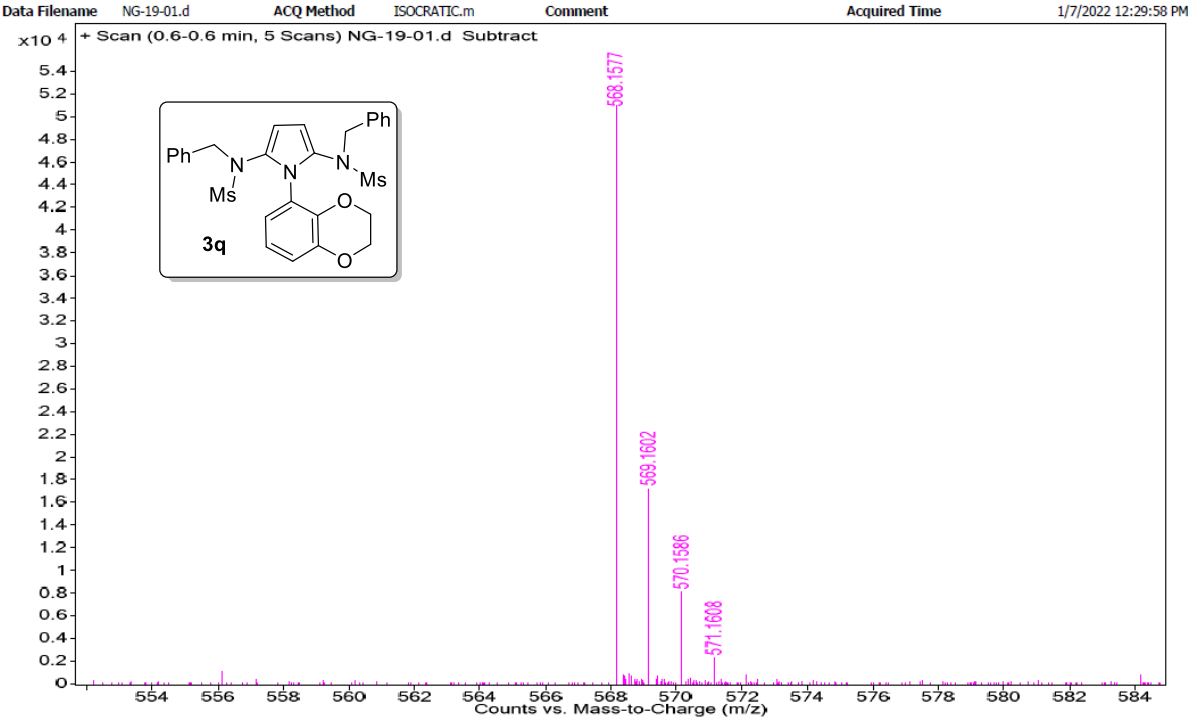
F2 - Acquisition Parameters
Date_                 20211101
Time                     11.39
INSTRUM                  spect
PROBHD    5 mm PATXI 1H/
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                       1024
DS                          4
SWH                 29761.904 Hz
FIDRES                0.454131 Hz
AQ                    1.1010048 sec
RG                        56.22
DW                       16.800 usec
DE                        6.50 usec
TE                       303.0 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                125.9077573 MHz
NUC1                        13C
P1                         9.23 usec
PLW1               244.00000000 W

======== CHANNEL f2 ========
SFO2                500.6783527 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                     80.00 usec
PLW2                13.60000038 W
PLW12                0.08840500 W
PLW13                0.05657900 W

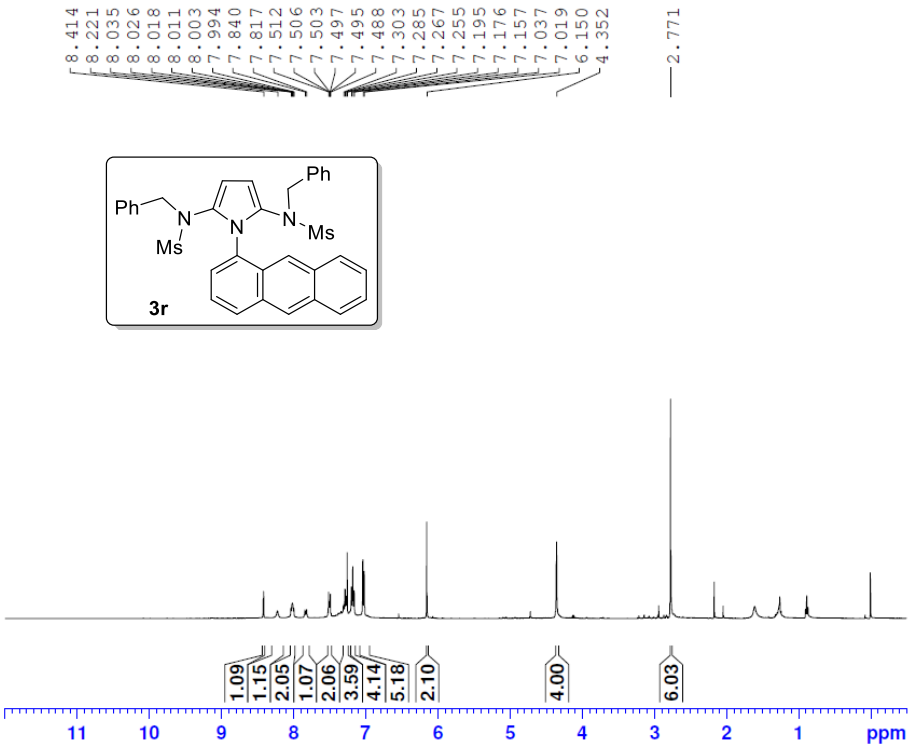
F2 - Processing parameters
SI                          32768
SF                  125.8951680 MHz
WDW                            EM
SSB                 0
LB                         1.00 Hz
GB                  0
PC                         1.40
```

Sample Name	HRMS22I07JAN20	**Position**	Vial 20	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	NG-19-01.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	1/7/2022 12:29:58 PM



GR-21-39

^{1}H NMR, $CDCl_3$, 400 MHz




```
Current Data Parameters
NAME            GR-21-39
EXPNO                440
PROCNO                 1

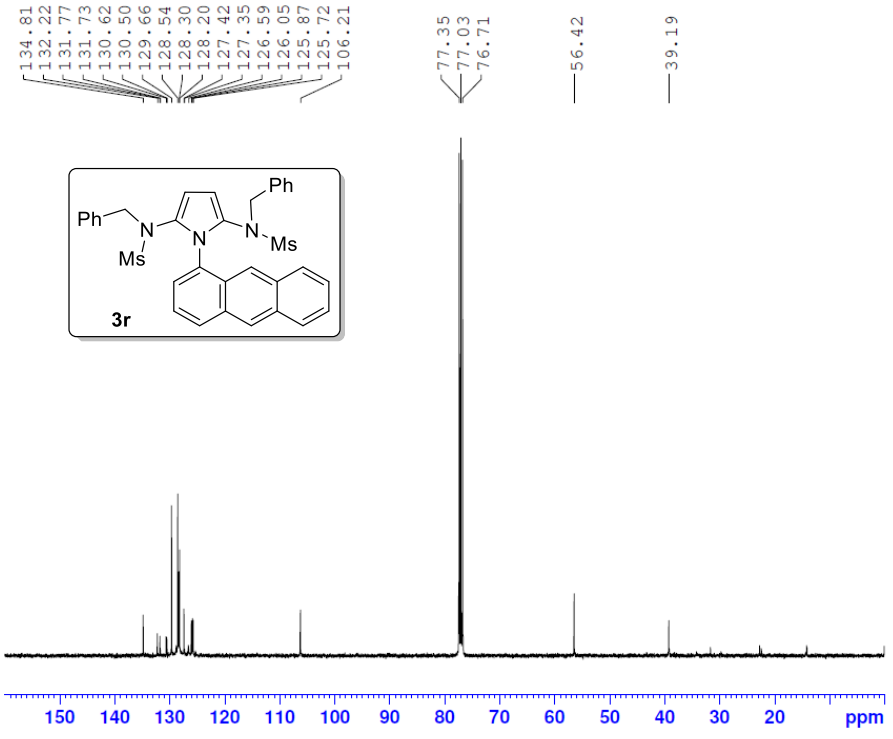
F2 - Acquisition Parameters
Date_           20211119
Time               17.03
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    0
SWH             9615.385 Hz
FIDRES          0.146719 Hz
AQ              3.4078720 sec
RG                114.26
DW                52.000 usec
DE                 6.50 usec
TE                300.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1629712 MHz
NUC1                  1H
P1                 13.20 usec
PLW1         13.00000000 W

F2 - Processing parameters
SI                 65536
SF            400.1605115 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

GR-21-39

^{13}C NMR, $CDCl_3$, 101 MHz




```
Current Data Parameters
NAME            Desktop
EXPNO                450
PROCNO                 1

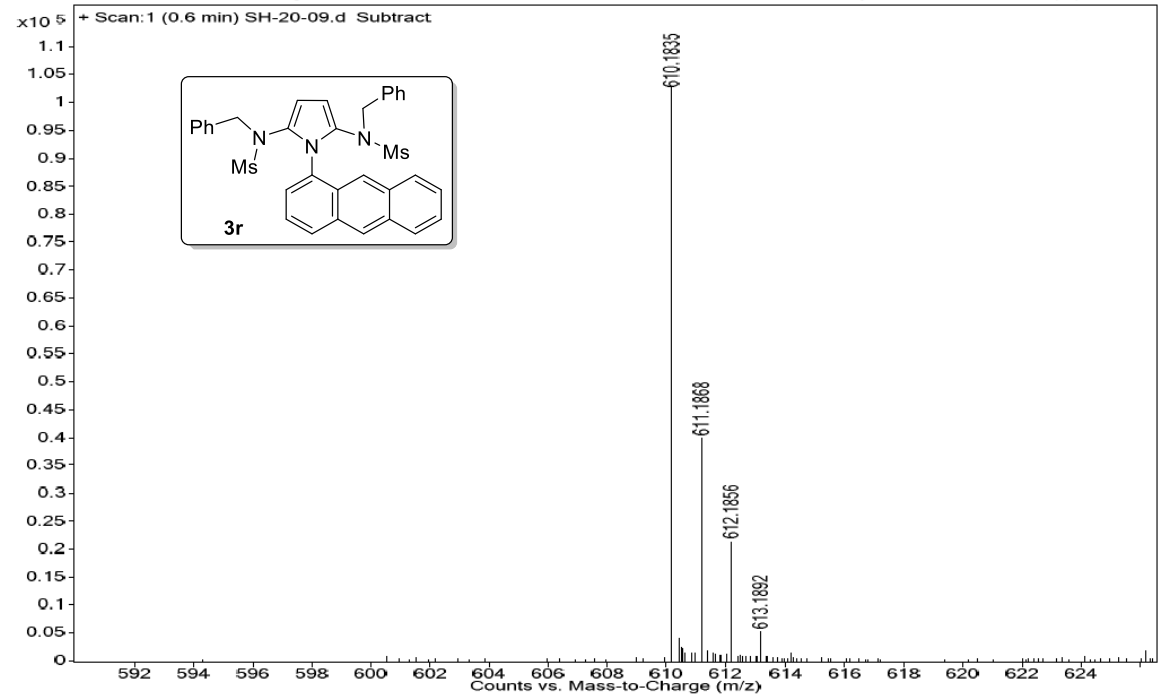
F2 - Acquisition Parameters
Date_           20211119
Time               19.01
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 2048
DS                    4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631488 sec
RG                201.48
DW                20.800 usec
DE                 6.50 usec
TE                300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          100.6304993 MHz
NUC1                  13C
P1                 9.90 usec
PLW1         53.00000000 W

======== CHANNEL f2 ========
SFO2          400.1621006 MHz
NUC2                  1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         13.00000000 W
PLW12         0.27963999 W
PLW13         0.22651000 W

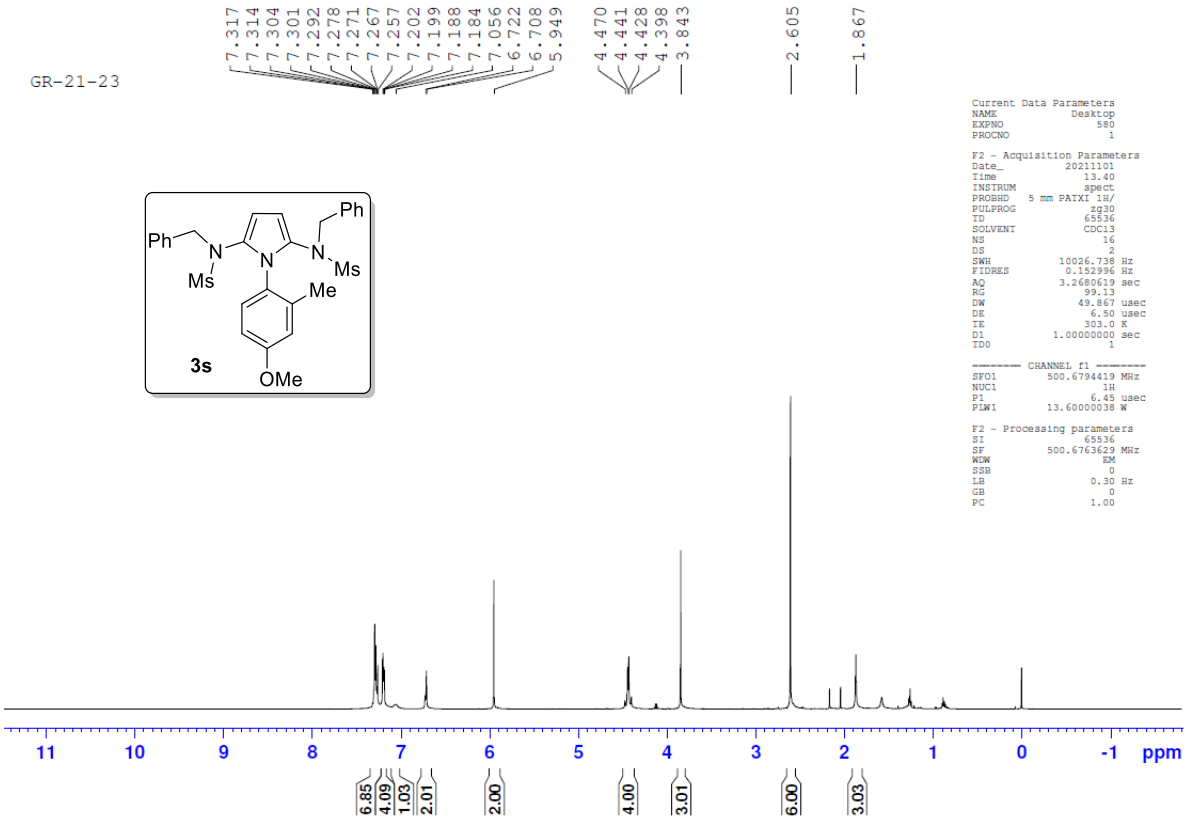
F2 - Processing parameters
SI                 32768
SF            100.6204380 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

Sample Name	HRMS22I24JAN16	**Position**	Vial 16	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	SH-20-09.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	1/24/2022 12:34:23 PM



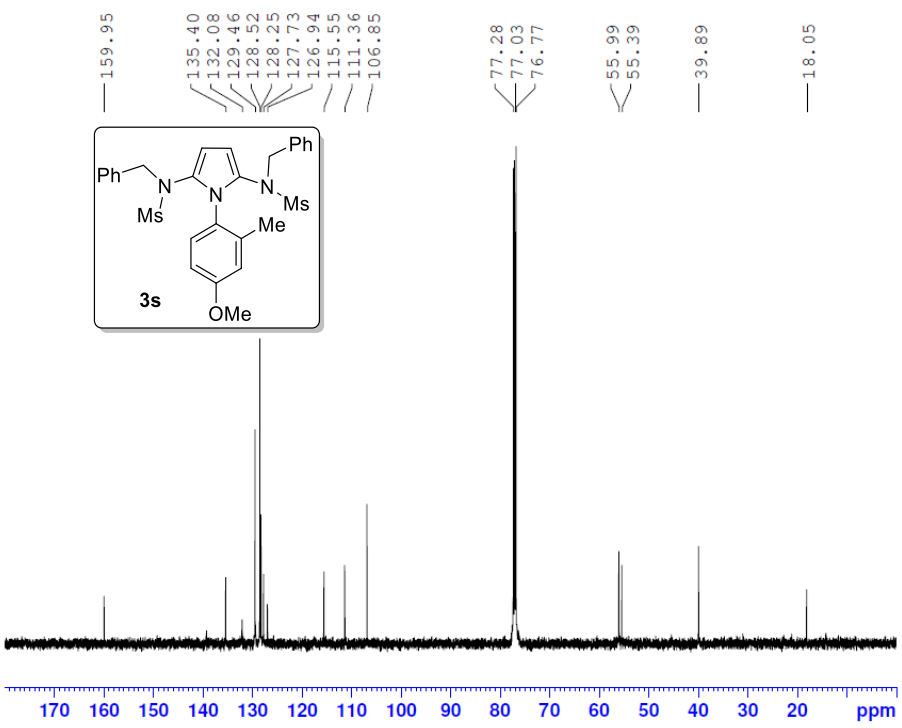
^{1}H NMR, $CDCl_3$, 500 MHz

GR-21-23



GR-21-23

^{13}C NMR, $CDCl_3$, 125 MHz




```
Current Data Parameters
NAME                  GR-21-23
EXPNO                      590
PROCNO                       1

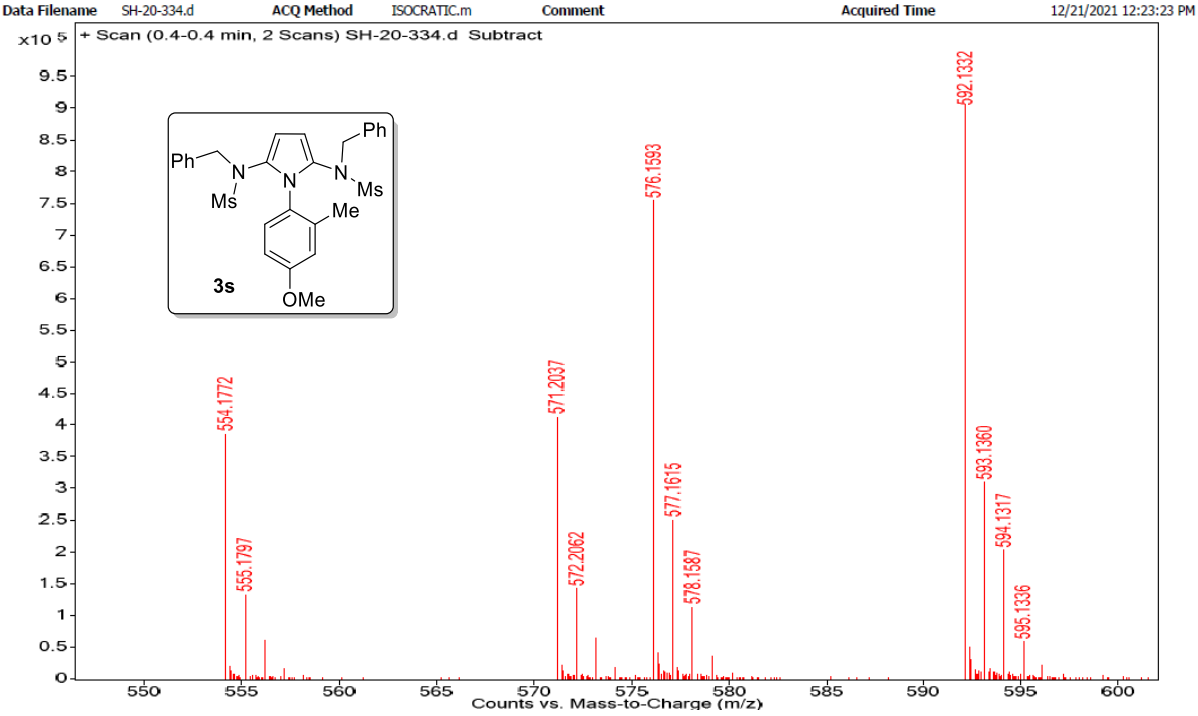
F2 - Acquisition Parameters
Date_                 20211101
Time                     14.36
INSTRUM                  spect
PROBHD    5 mm PATXI 1H/
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                       1024
DS                          4
SWH                 29761.904 Hz
FIDRES                0.454131 Hz
AQ                    1.1010048 sec
RG                        56.22
DW                       16.800 usec
DE                        6.50 usec
TE                       303.0 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                125.9077573 MHz
NUC1                        13C
P1                         9.23 usec
PLW1               244.00000000 W

======== CHANNEL f2 ========
SFO2                500.6783527 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                     80.00 usec
PLW2               13.60000038 W
PLW12               0.08840500 W
PLW13               0.05657900 W

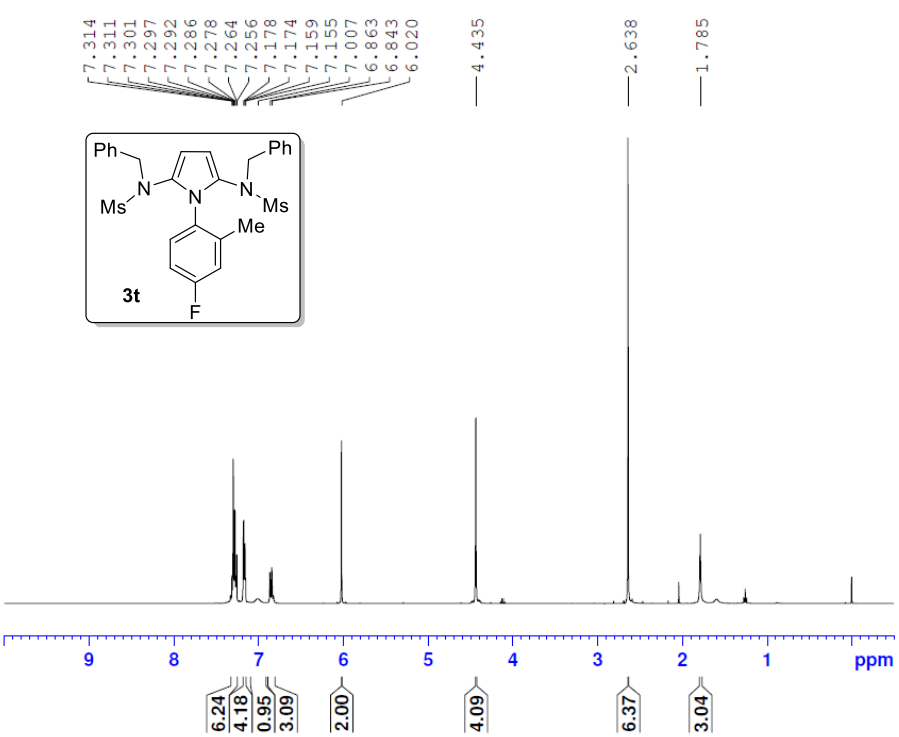
F2 - Processing parameters
SI                         32768
SF                  125.8951680 MHz
WDW                           EM
SSB                 0
LB                         1.00 Hz
GB                  0
PC                         1.40
```

Sample Name	HRMS21I21DEC16	**Position**	Vial 16	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	SH-20-334.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	12/21/2021 12:23:23 PM



GR-21-22

^{1}H NMR, $CDCl_3$, 400 MHz



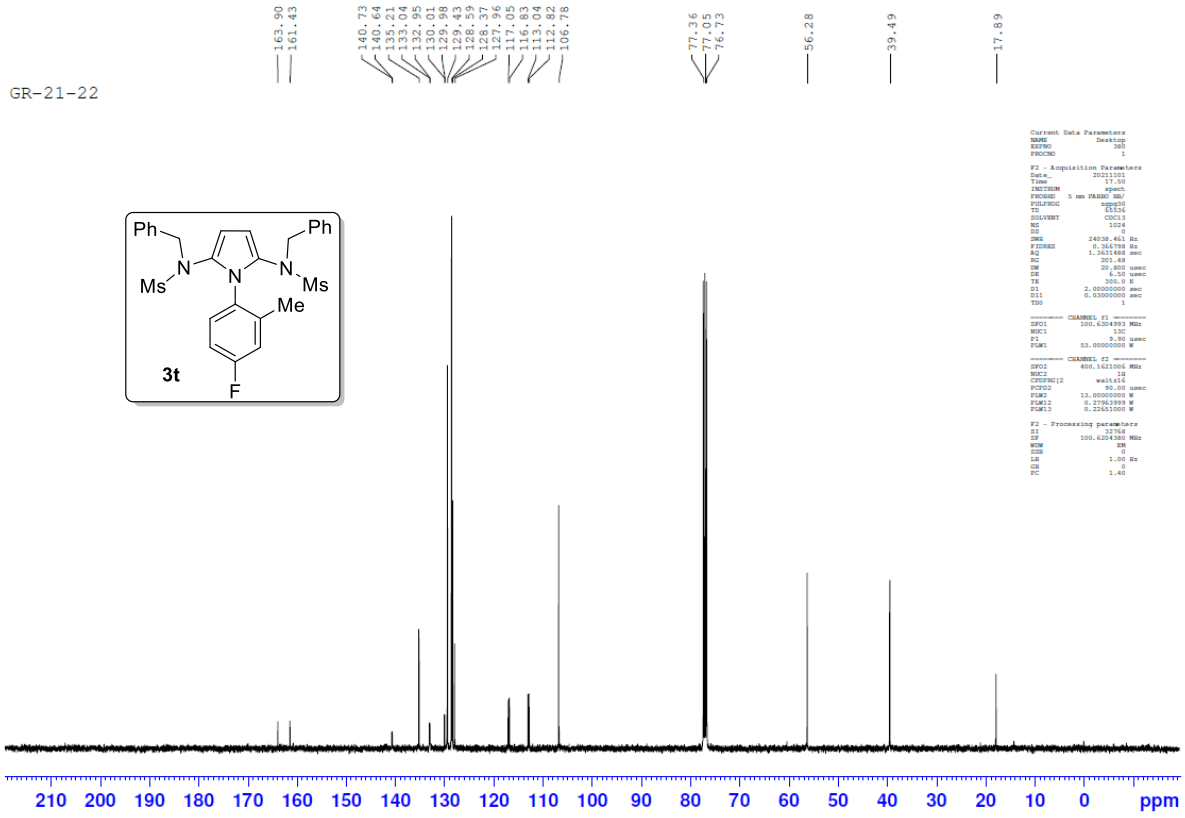

```
Current Data Parameters
NAME                  GR-21-22
EXPNO                      370
PROCNO                       1

F2 - Acquisition Parameters
Date_               20211101
Time                   17.09
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                         8
DS                         0
SWH                 9615.385 Hz
FIDRES              0.146719 Hz
AQ                  3.4078720 sec
RG                     80.54
DW                    52.000 usec
DE                     6.50 usec
TE                    300.0 K
D1                 1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1              400.1629712 MHz
NUC1                      1H
P1                     13.20 usec
PLW1              13.00000000 W

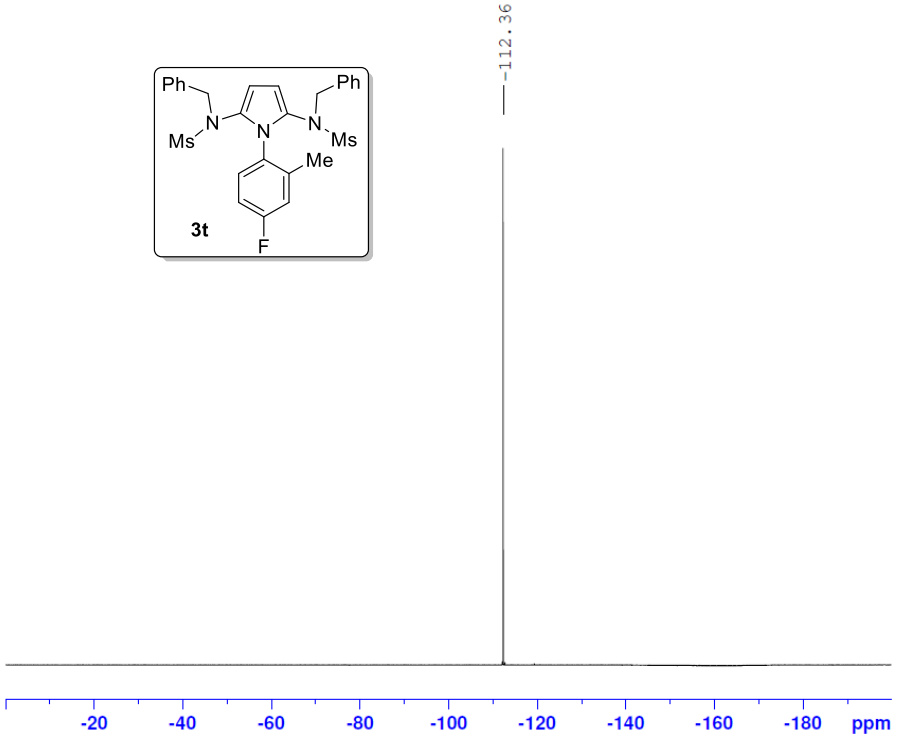
F2 - Processing parameters
SI                     65536
SF                400.1605113 MHz
WDW                       EM
SSB          0
LB                     0.30 Hz
GB           0
PC                     1.00
```

^{13}C NMR, $CDCl_3$, 101 MHz



^{19}F NMR, $CDCl_3$, 376 MHz

GR-21-22




```
Current Data Parameters
NAME                  GR-21-22
EXPNO                      390
PROCNO                       1

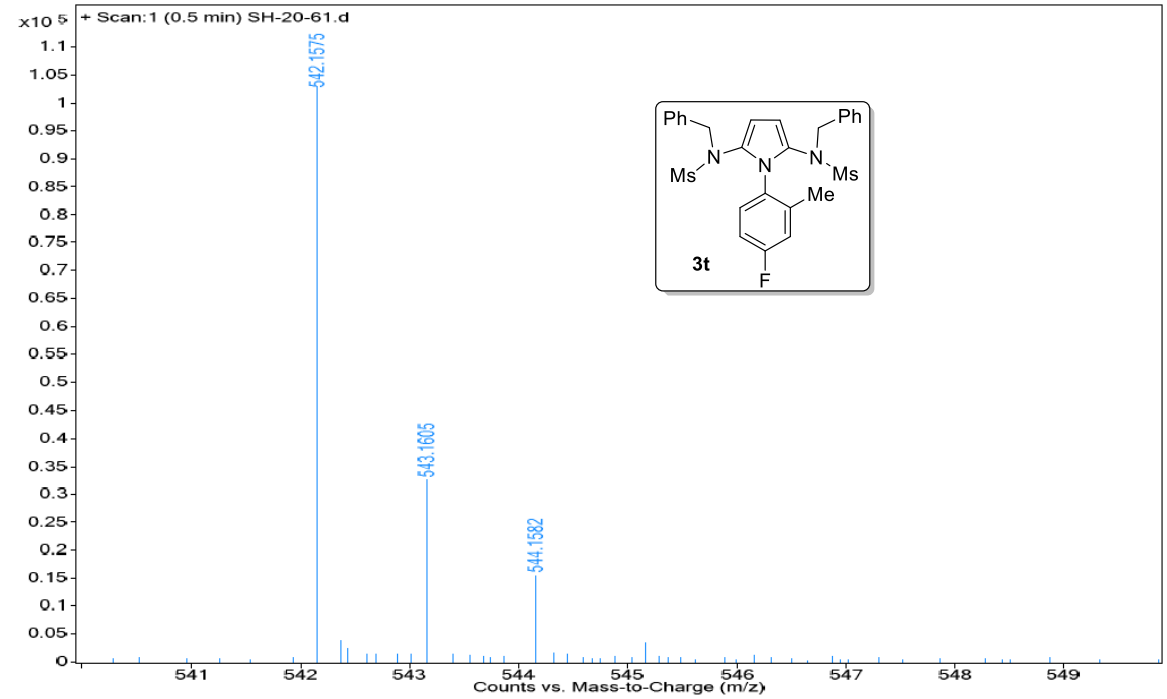
F2 - Acquisition Parameters
Date_                 20211101
Time                     17.52
INSTRUM                  spect
PROBHD     5 mm PABBO BB/
PULPROG           zgfhigqn.2
TD                      131072
SOLVENT                  CDCl3
NS                          16
DS                           4
SWH                  89285.711 Hz
FIDRES               0.681196 Hz
AQ                   0.7340032 sec
RG                      201.48
DW                       5.600 usec
DE                       6.50 usec
TE                      300.0 K
D1                   1.00000000 sec
D11                  0.03000000 sec
D12                  0.00002000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                376.4894122 MHz
NUC1                        19F
P1                       15.00 usec
PLW1                21.00000000 W

======== CHANNEL f2 ========
SFO2                400.1621006 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                    90.00 usec
PLW2                13.00000000 W
PLW12                0.27963999 W

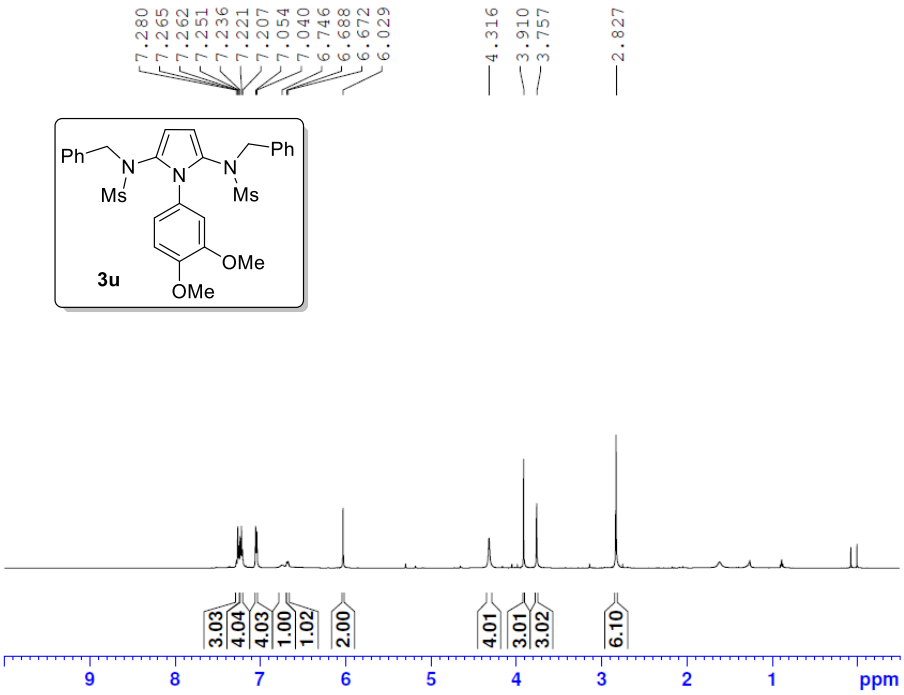
F2 - Processing parameters
SI                       65536
SF                  376.5270650 MHz
WDW                         EM
SSB          0
LB                       0.30 Hz
GB           0
PC                       1.00
```

Sample Name	HRMS22I19JAN21	**Position**	Vial 21	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	SH-20-61.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	1/19/2022 12:34:50 PM



GR-21-12

^{1}H NMR, $CDCl_3$, 500 MHz




```
Current Data Parameters
NAME            GR-21-12
EXPNO                320
PROCNO                 1

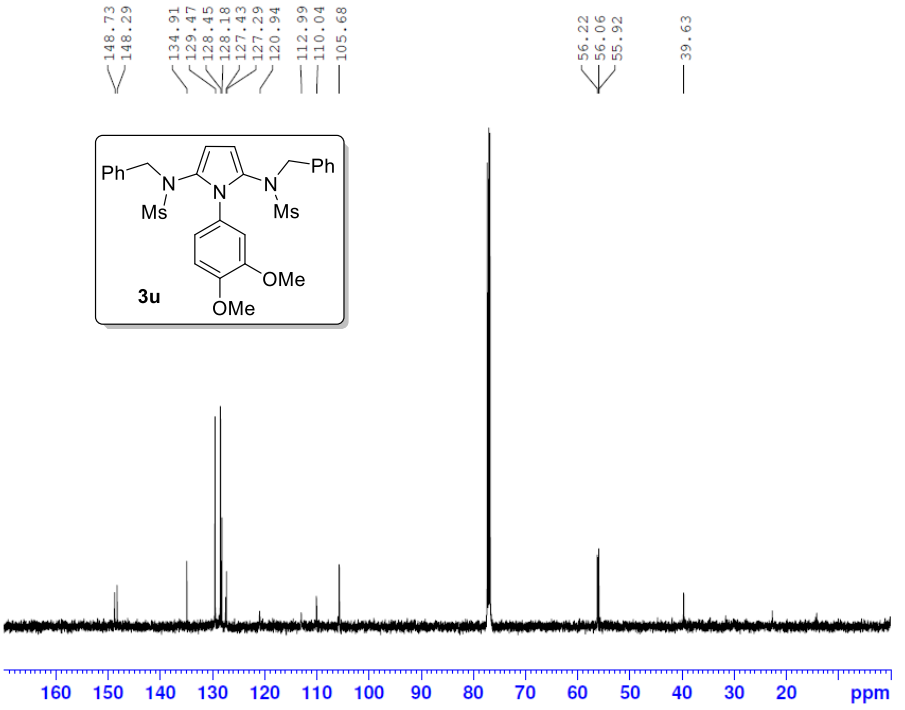
F2 - Acquisition Parameters
Date_           20211214
Time                7.04
INSTRUM            spect
PROBHD   5 mm PATXI 1H/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            10026.738 Hz
FIDRES         0.152996 Hz
AQ             3.2680619 sec
RG                 99.13
DW                49.867 usec
DE                 6.50 usec
TE                300.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         500.6794419 MHz
NUC1                   1H
P1                  6.45 usec
PLW1         13.60000038 W

F2 - Processing parameters
SI                 65536
SF           500.6763606 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

GR-21-12

^{13}C NMR, $CDCl_3$, 125 MHz




```
Current Data Parameters
NAME            GR-21-12
EXPNO                330
PROCNO                 1

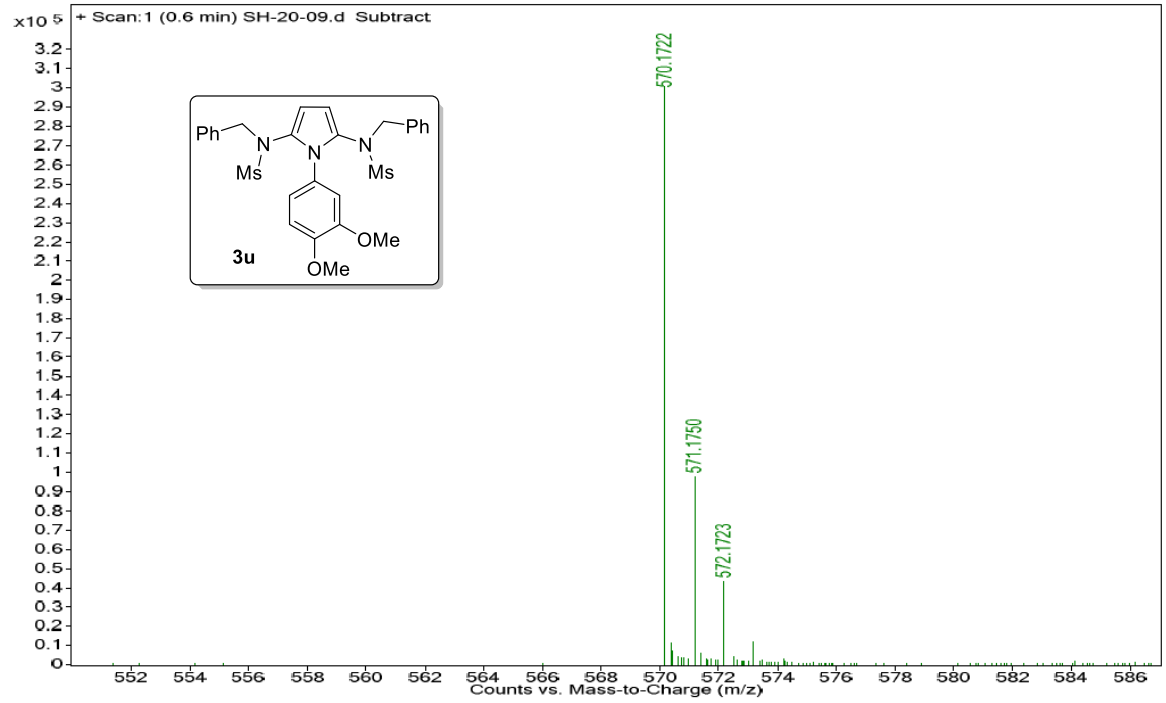
F2 - Acquisition Parameters
Date_           20211214
Time                7.23
INSTRUM            spect
PROBHD   5 mm PATXI 1H/
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  2048
DS                     4
SWH            29761.904 Hz
FIDRES         0.454131 Hz
AQ             1.1010048 sec
RG                107.78
DW                16.800 usec
DE                 6.50 usec
TE                300.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         125.9077573 MHz
NUC1                  13C
P1                  9.23 usec
PLW1        244.00000000 W

======== CHANNEL f2 ========
SFO2         500.6783527 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2         13.60000038 W
PLW12         0.08840500 W
PLW13         0.05657900 W

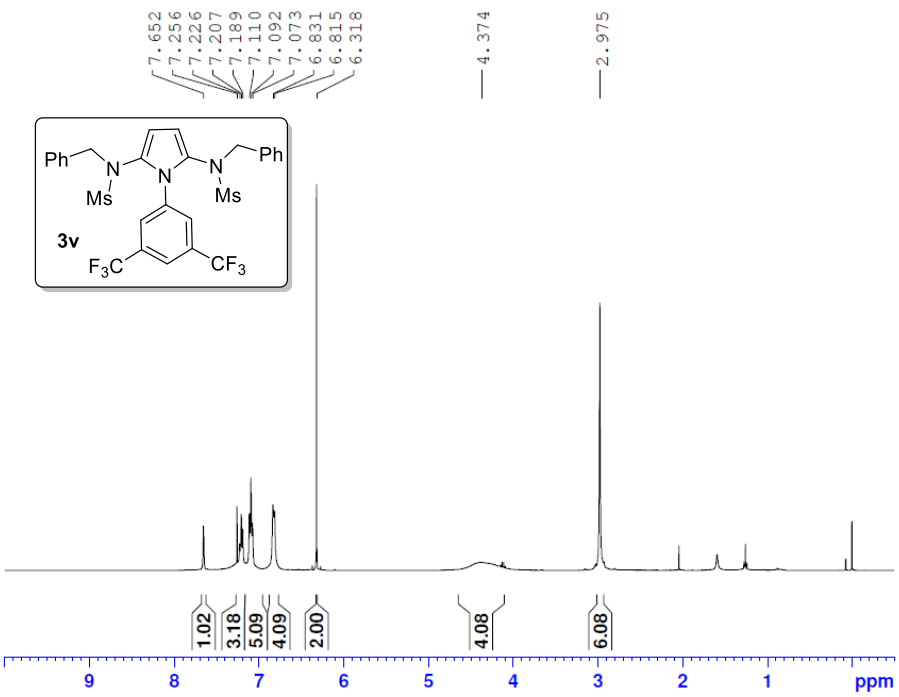
F2 - Processing parameters
SI                 32768
SF           125.8951680 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

Sample Name	HRMS22I18JAN23	**Position**	Vial 23	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	SH-20-09.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	1/18/2022 12:50:19 PM



GR-21-06

^{1}H NMR, $CDCl_3$, 400 MHz




```
Current Data Parameters
NAME            GR-21-06
EXPNO                580
PROCNO                 1

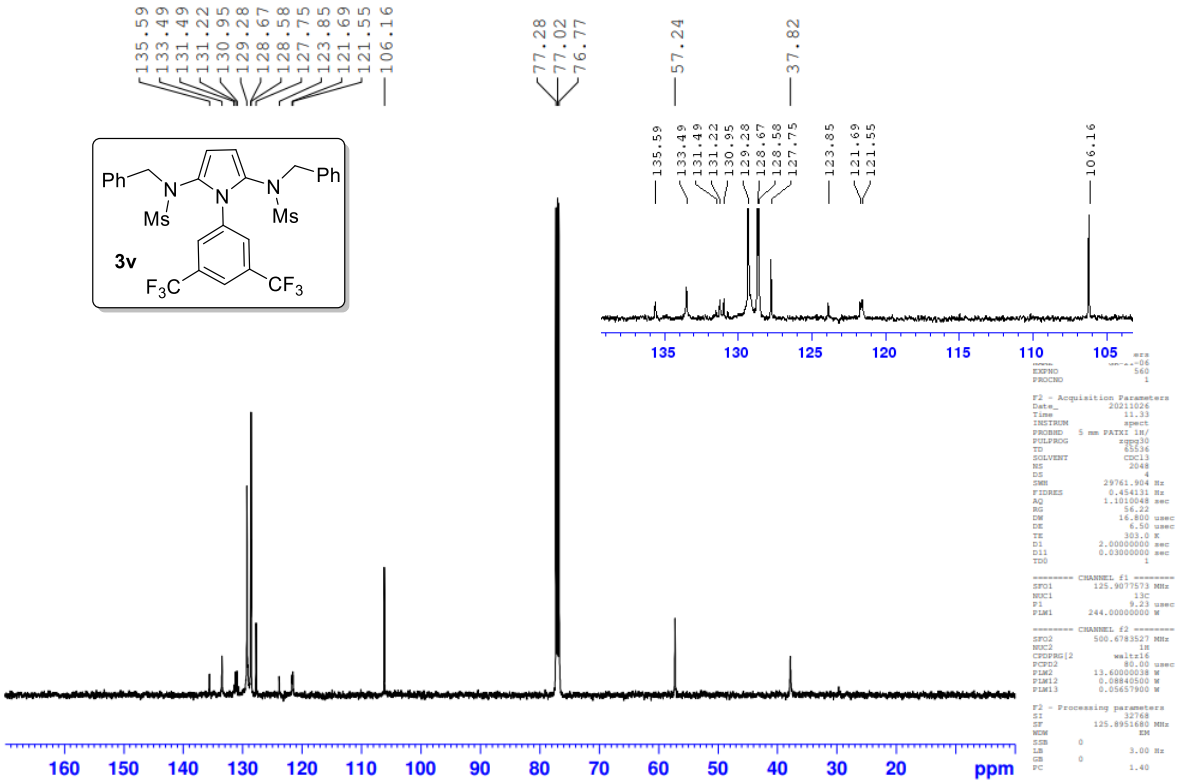
F2 - Acquisition Parameters
Date_           20211024
Time                5.24
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH             9615.385 Hz
FIDRES          0.146719 Hz
AQ              3.4078720 sec
RG                 80.54
DW                52.000 usec
DE                 6.50 usec
TE                300.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1629712 MHz
NUC1                   1H
P1                  13.20 usec
PLW1        13.00000000 W

F2 - Processing parameters
SI                 65536
SF          400.1605110 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

GR 21 06

^{13}C NMR, $CDCl_3$, 125 MHz



EXPNO 560
PROCNO 1

F2 - Acquisition Parameters
Date_ 20211026
Time 11.33
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 56.22
DW 16.800 usec
DE 6.50 usec
TE 303.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 125.9077573 MHz
NUC1 13C
P1 9.23 usec
PLW1 244.00000000 W

======== CHANNEL f2 ========
SFO2 500.6783527 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 13.60000038 W
PLW12 0.08840500 W
PLW13 0.05657900 W

F2 - Processing parameters
SI 32768
SF 125.8951680 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

GR-21-06

^{19}F NMR, $CDCl_3$, 376 MHz

Current Data Parameters
NAME GR-21-06
EXPNO 590
PROCNO 1

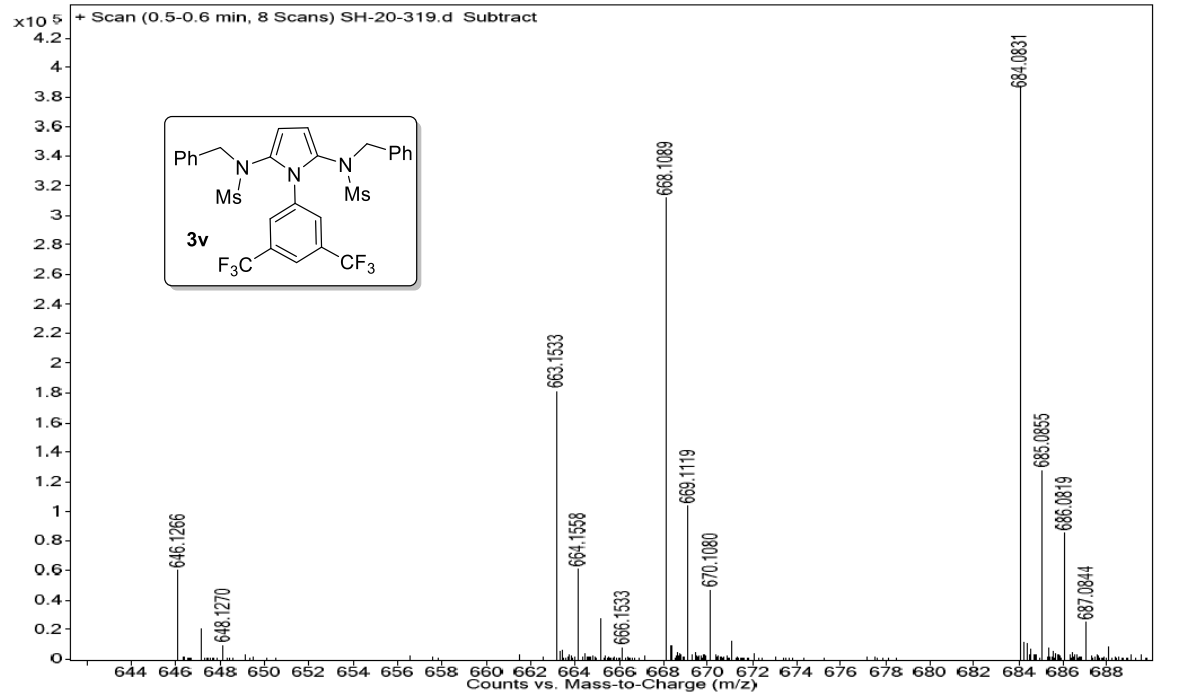
F2 - Acquisition Parameters
Date_ 20211024
Time 5.26
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgfhigqn.2
TD 131072
SOLVENT CDCl3
NS 16
DS 4
SWH 89285.711 Hz
FIDRES 0.681196 Hz
AQ 0.7340032 sec
RG 201.48
DW 5.600 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 376.4894122 MHz
NUC1 19F
P1 15.00 usec
PLW1 21.00000000 W

======== CHANNEL f2 ========
SFO2 400.1621006 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.27963999 W

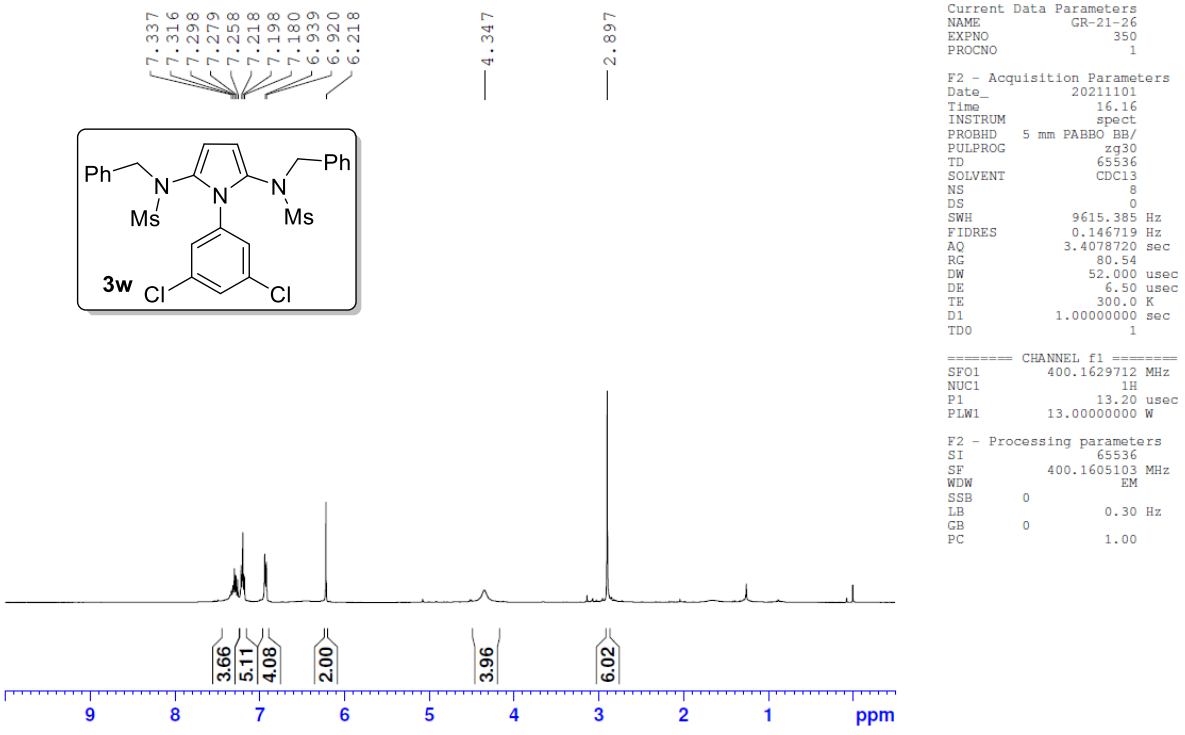
F2 - Processing parameters
SI 65536
SF 376.5270650 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Sample Name	HRMS21I21DEC15	**Position**	Vial 15	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	SH-20-319.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	12/21/2021 12:19:49 PM



GR-21-26

^{1}H NMR, $CDCl_3$, 400 MHz




```
Current Data Parameters
NAME                  GR-21-26
EXPNO                      350
PROCNO                       1

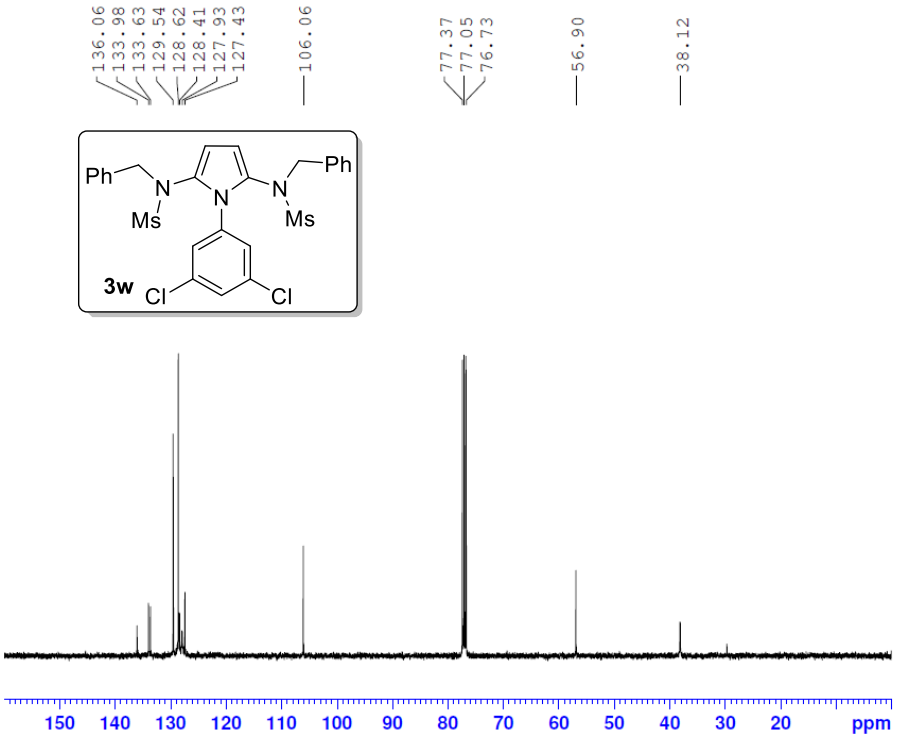
F2 - Acquisition Parameters
Date_                 20211101
Time                     16.16
INSTRUM                  spect
PROBHD     5 mm PABBO BB/
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                           8
DS                           0
SWH                 9615.385 Hz
FIDRES              0.146719 Hz
AQ                  3.4078720 sec
RG                       80.54
DW                      52.000 usec
DE                       6.50 usec
TE                      300.0 K
D1                  1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                400.1629712 MHz
NUC1                        1H
P1                       13.20 usec
PLW1               13.00000000 W

F2 - Processing parameters
SI                      65536
SF                  400.1605103 MHz
WDW                        EM
SSB                 0
LB                       0.30 Hz
GB                  0
PC                       1.00
```

GR-21-26

^{13}C NMR, $CDCl_3$, 101 MHz




```
Current Data Parameters
NAME                  GR-21-26
EXPNO                      360
PROCNO                       1

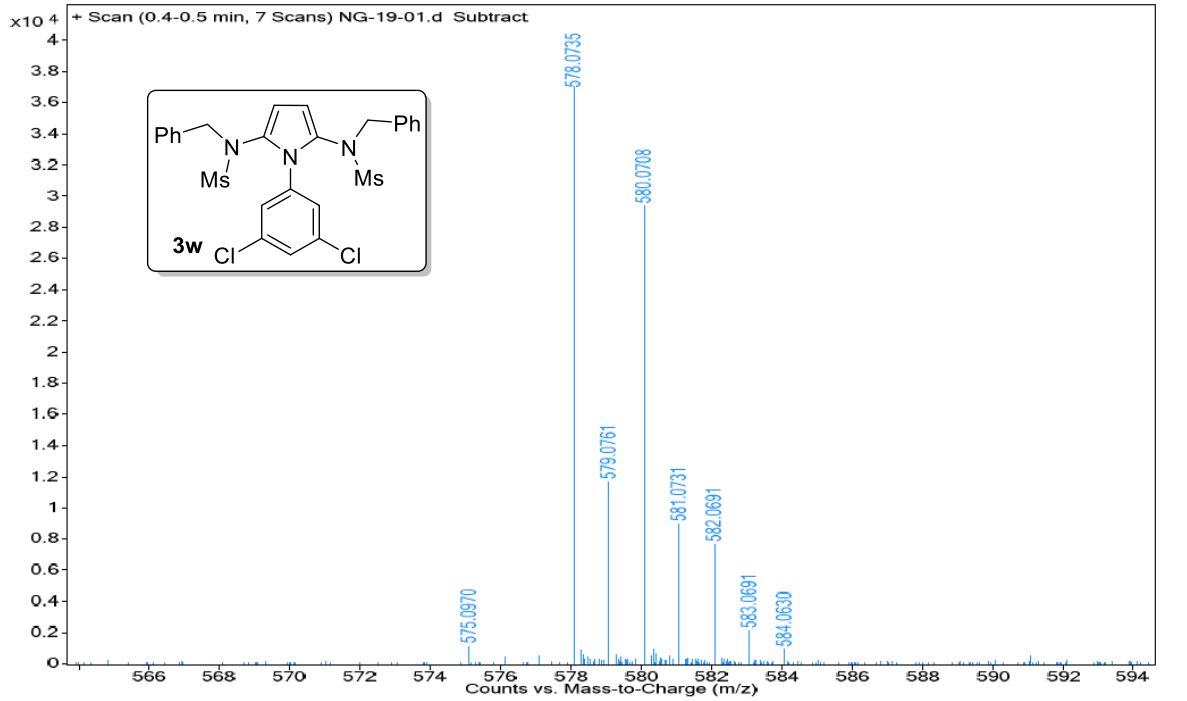
F2 - Acquisition Parameters
Date_                 20211101
Time                     16.33
INSTRUM                  spect
PROBHD     5 mm PABBO BB/
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                       2048
DS                          0
SWH                24038.461 Hz
FIDRES              0.366798 Hz
AQ                  1.3631488 sec
RG                     201.48
DW                     20.800 usec
DE                      6.50 usec
TE                     300.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1               100.6304993 MHz
NUC1                       13C
P1                        9.90 usec
PLW1               53.00000000 W

======== CHANNEL f2 ========
SFO2               400.1621006 MHz
NUC2                       1H
CPDPRG[2                waltz16
PCPD2                    90.00 usec
PLW2               13.00000000 W
PLW12               0.27963999 W
PLW13               0.22651000 W

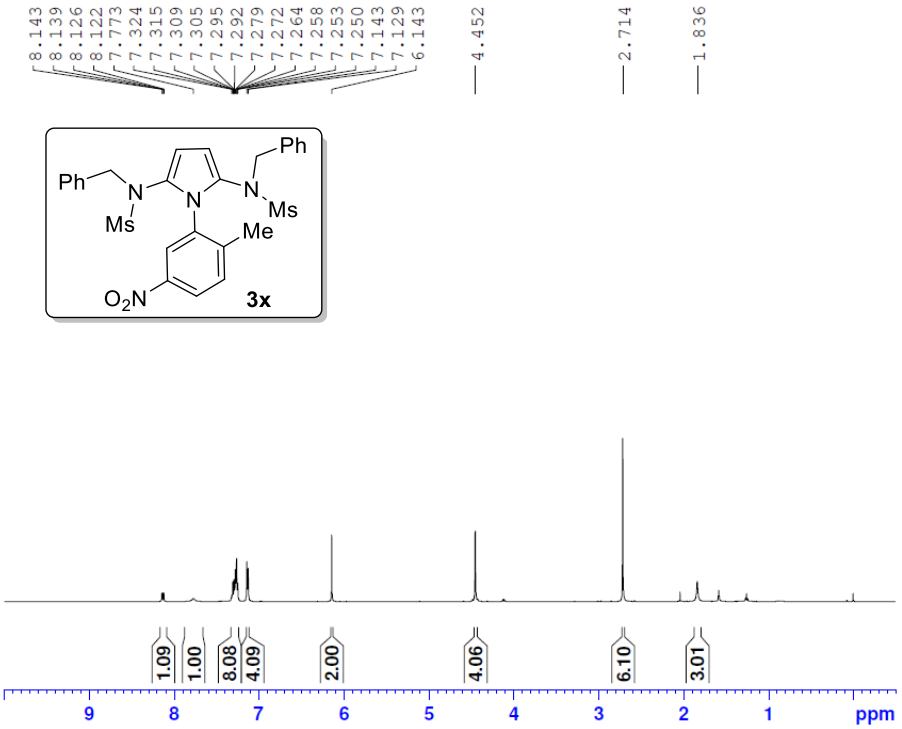
F2 - Processing parameters
SI                        32768
SF                 100.6204380 MHz
WDW                          EM
SSB                 0
LB                        1.00 Hz
GB                  0
PC                        1.40
```

Sample Name	HRMS22I13JAN11	**Position**	Vial 11	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	NG-19-01.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	1/13/2022 12:13:25 PM



GR-21-20

^{1}H NMR, $CDCl_3$, 500 MHz




```
Current Data Parameters
NAME                  GR-21-20
EXPNO                      530
PROCNO                       1

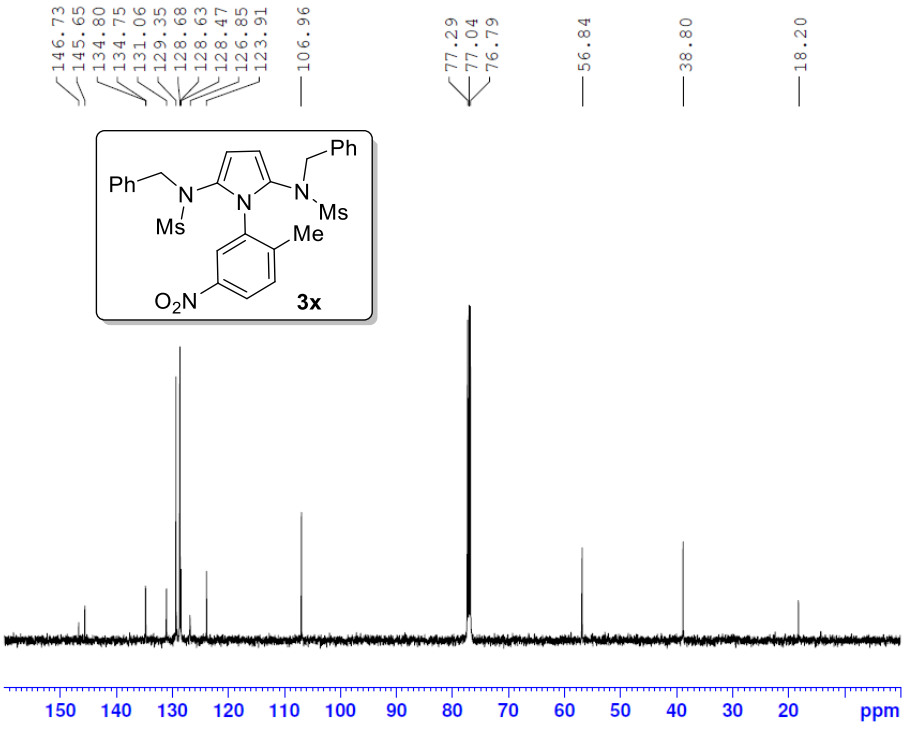
F2 - Acquisition Parameters
Date_                 20211029
Time                     11.37
INSTRUM                  spect
PROBHD    5 mm PATXI 1H/
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                         16
DS                          2
SWH                 10026.738 Hz
FIDRES                0.152996 Hz
AQ                    3.2680619 sec
RG                        87.95
DW                       49.867 usec
DE                        6.50 usec
TE                       303.0 K
D1                  1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                500.6794419 MHz
NUC1                        1H
P1                        6.45 usec
PLW1               13.60000038 W

F2 - Processing parameters
SI                      65536
SF                  500.6763626 MHz
WDW                        EM
SSB                 0
LB                        0.30 Hz
GB                  0
PC                        1.00
```

GR-21-20

^{13}C NMR, $CDCl_3$, 125 MHz




```
Current Data Parameters
NAME                  GR-21-20
EXPNO                      540
PROCNO                       1

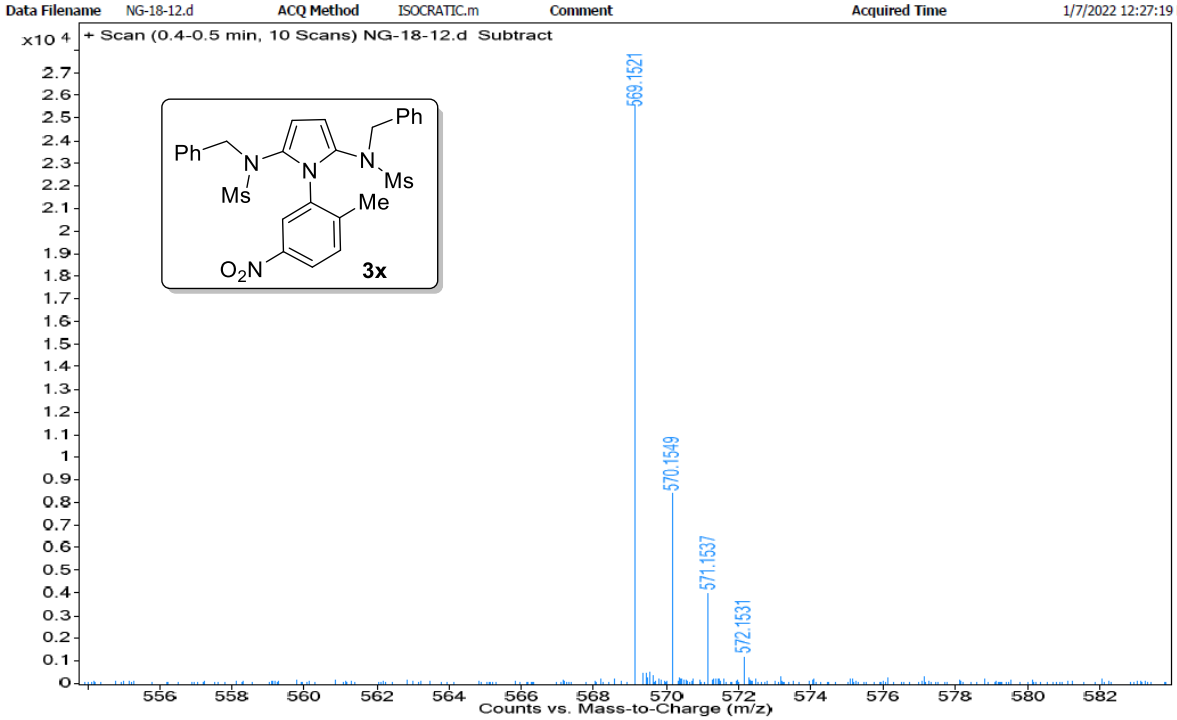
F2 - Acquisition Parameters
Date_                 20211029
Time                     11.39
INSTRUM                  spect
PROBHD    5 mm PATXI 1H/
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                       2048
DS                          4
SWH                 29761.904 Hz
FIDRES                0.454131 Hz
AQ                    1.1010048 sec
RG                        56.22
DW                       16.800 usec
DE                        6.50 usec
TE                       303.0 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                125.9077573 MHz
NUC1                        13C
P1                        9.23 usec
PLW1               244.00000000 W

======== CHANNEL f2 ========
SFO2                500.6783527 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                    80.00 usec
PLW2               13.60000038 W
PLW12               0.08840500 W
PLW13               0.05657900 W

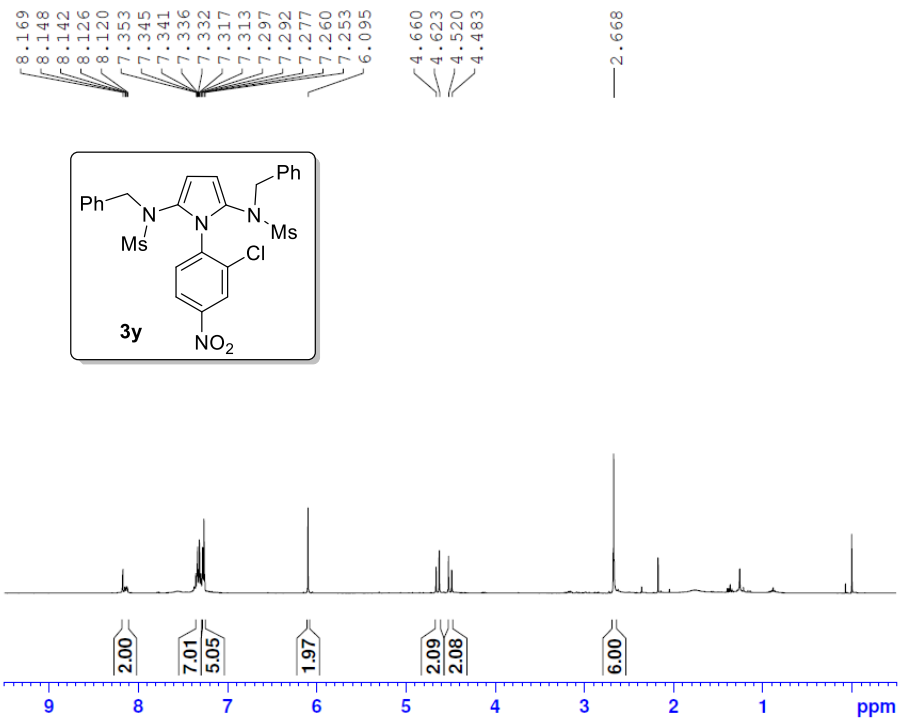
F2 - Processing parameters
SI                      32768
SF                  125.8951680 MHz
WDW                        EM
SSB                 0
LB                        2.00 Hz
GB                  0
PC                        1.40
```

Sample Name	HRMS22I07JAN19	**Position**	Vial 19	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	NG-18-12.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	1/7/2022 12:27:19 PM



GR 21 24 D

^{1}H NMR, $CDCl_3$, 400 MHz




```
Current Data Parameters
NAME                  Desktop
EXPNO                     410
PROCNO                      1

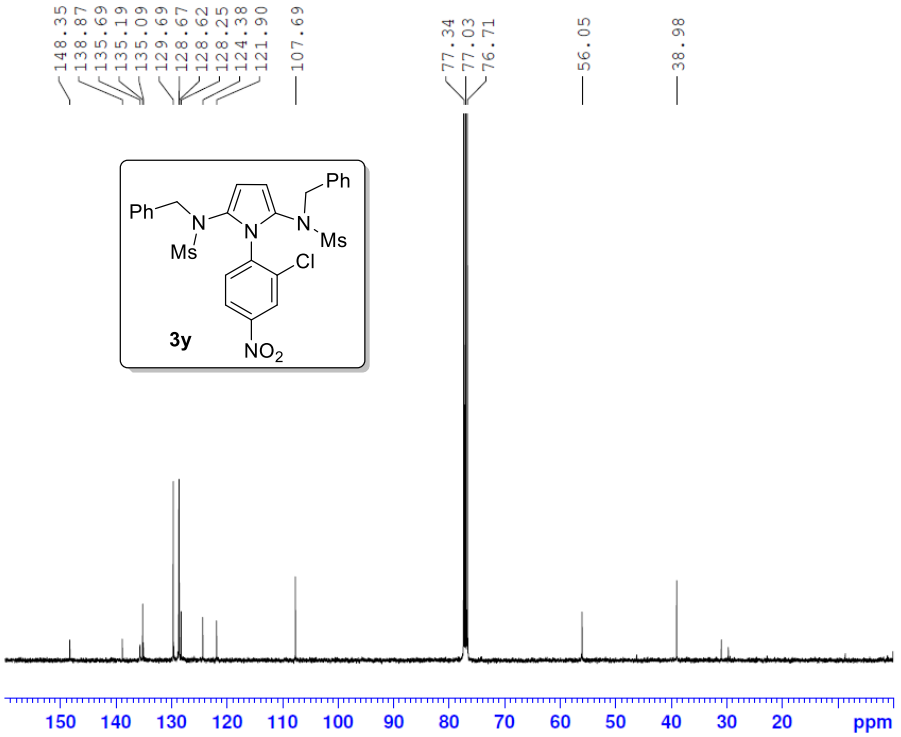
F2 - Acquisition Parameters
Date_              20211101
Time                  12.52
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        8
DS                        0
SWH                9615.385 Hz
FIDRES             0.146719 Hz
AQ                 3.4078720 sec
RG                    129.57
DW                    52.000 usec
DE                     6.50 usec
TE                    300.0 K
D1                 1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1             400.1629712 MHz
NUC1                       1H
P1                     13.20 usec
PLW1            13.00000000 W

F2 - Processing parameters
SI                        65536
SF               400.1605091 MHz
WDW                         EM
SSB          0
LB                      0.30 Hz
GB           0
PC                      1.00
```

GR-21-24D

^{13}C NMR, $CDCl_3$, 101 MHz




```
Current Data Parameters
NAME                  Desktop
EXPNO                     590
PROCNO                      1

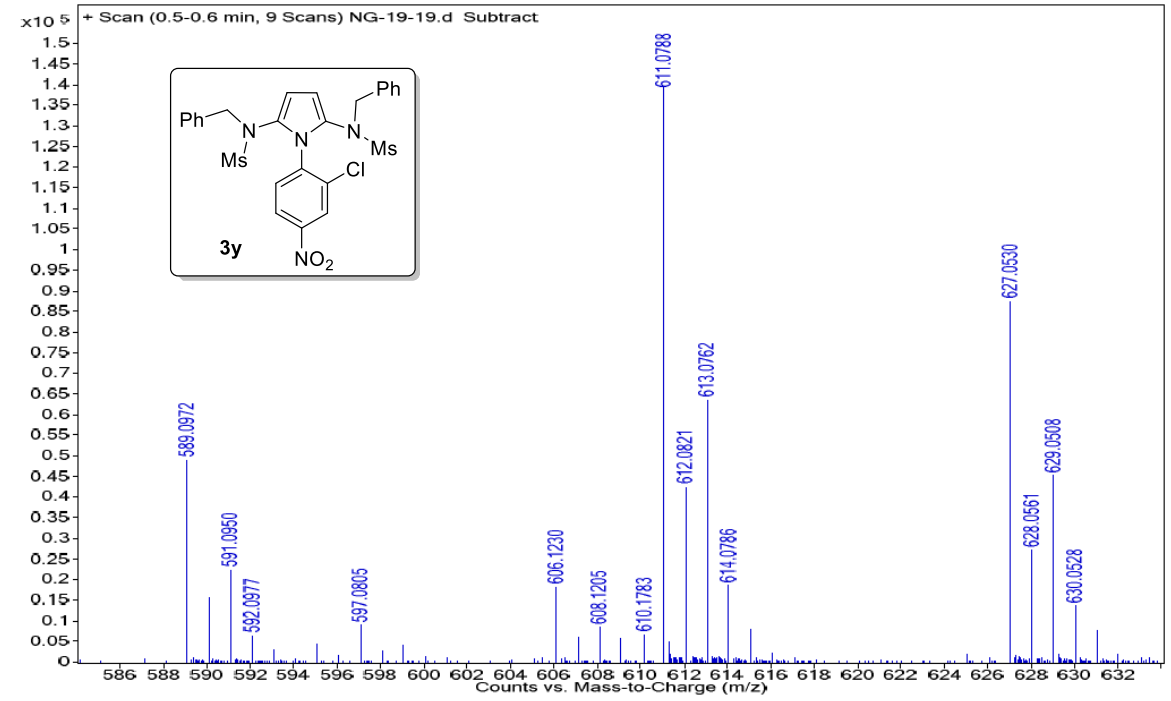
F2 - Acquisition Parameters
Date_              20211120
Time                  19.37
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG               zgpg30
TD                     65536
SOLVENT                CDCl3
NS                      2048
DS                         4
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                 1.3631488 sec
RG                    201.48
DW                    20.800 usec
DE                     6.50 usec
TE                    300.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1              100.6304993 MHz
NUC1                      13C
P1                       9.90 usec
PLW1              53.00000000 W

======== CHANNEL f2 ========
SFO2              400.1621006 MHz
NUC2                      1H
CPDPRG[2              waltz16
PCPD2                 90.00 usec
PLW2              13.00000000 W
PLW12              0.27963999 W
PLW13              0.22651000 W

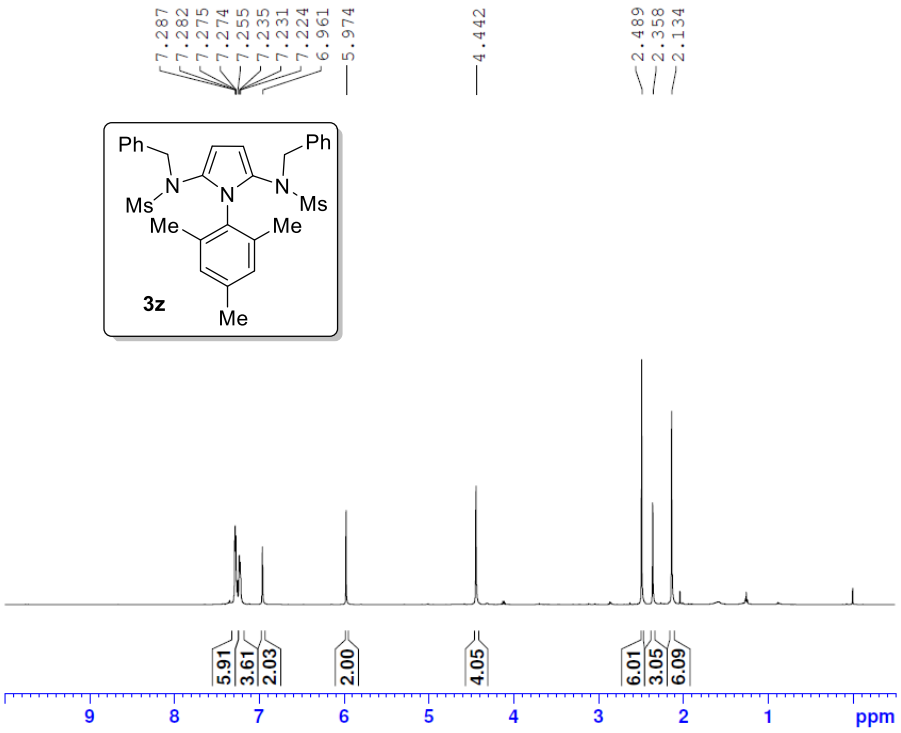
F2 - Processing parameters
SI                        32768
SF                100.6204380 MHz
WDW                        EM
SSB          0
LB                     1.00 Hz
GB           0
PC                     1.40
```

Sample Name	HRMS22I10JAN16	**Position**	Vial 16	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	NG-19-19.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	1/10/2022 12:02:33 PM



gr-21-17

^{1}H NMR, $CDCl_3$, 400 MHz




```
Current Data Parameters
NAME            GR-21-17
EXPNO                370
PROCNO                 1

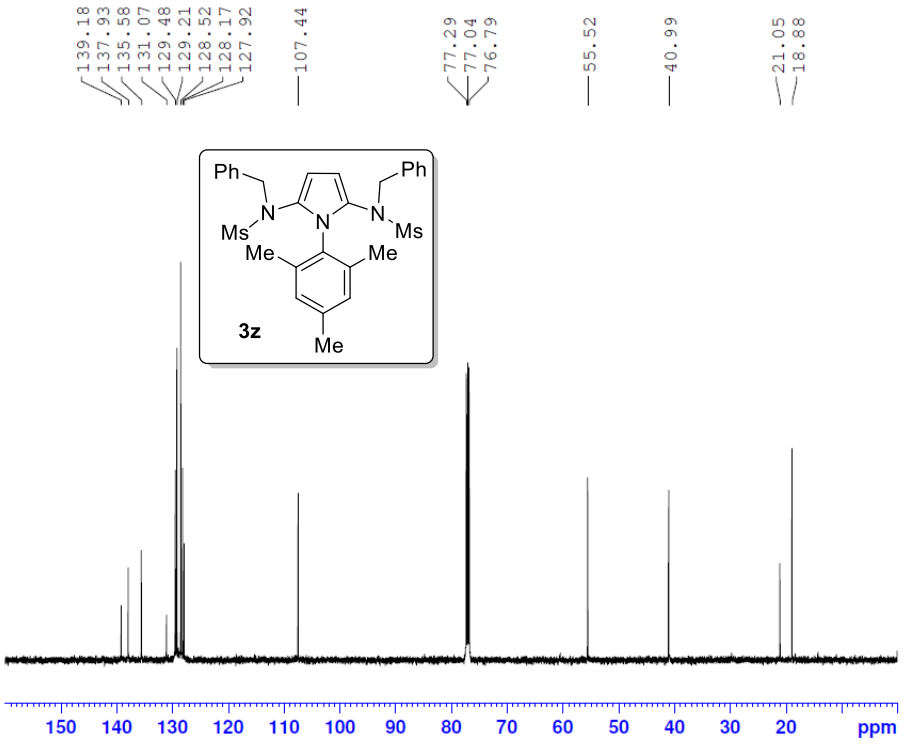
F2 - Acquisition Parameters
Date_          20211101
Time               15.08
INSTRUM            spect
PROBHD    5 mm PATXI 1H/
PULPROG            zg30
TD                65536
SOLVENT           CDC13
NS                   16
DS                    2
SWH           10026.738 Hz
FIDRES            0.152996 Hz
AQ             3.2680619 sec
RG                 61.7
DW                49.867 usec
DE                 6.50 usec
TE                303.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          500.6794419 MHz
NUC1                  1H
P1                 6.45 usec
PLW1          13.60000038 W

F2 - Processing parameters
SI                 65536
SF            500.6763641 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

gr-21-17

^{13}C NMR, $CDCl_3$, 125 MHz




```
Current Data Parameters
NAME            GR-21-17
EXPNO                380
PROCNO                 1

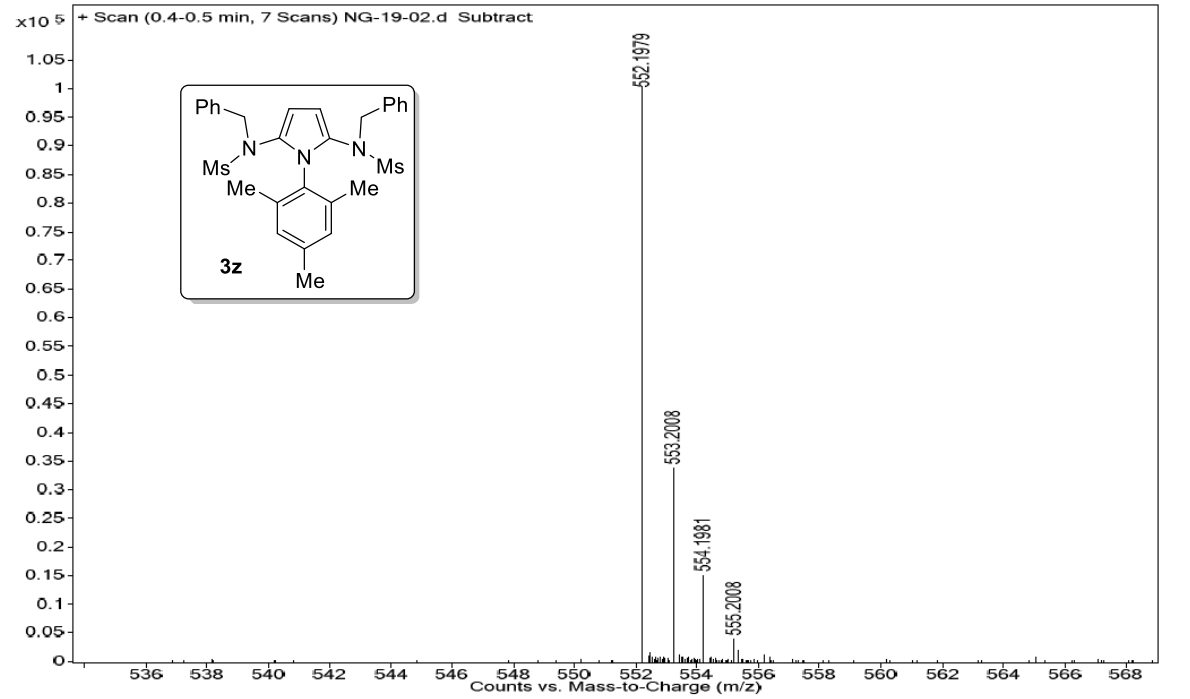
F2 - Acquisition Parameters
Date_          20211101
Time               16.04
INSTRUM            spect
PROBHD    5 mm PATXI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDC13
NS                 1024
DS                    4
SWH           29761.904 Hz
FIDRES            0.454131 Hz
AQ             1.1010048 sec
RG                 56.22
DW                16.800 usec
DE                 6.50 usec
TE                303.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          125.9077573 MHz
NUC1                  13C
P1                 9.23 usec
PLW1          244.00000000 W

======== CHANNEL f2 ========
SFO2          500.6783527 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2          13.60000038 W
PLW12          0.08840500 W
PLW13          0.05657900 W

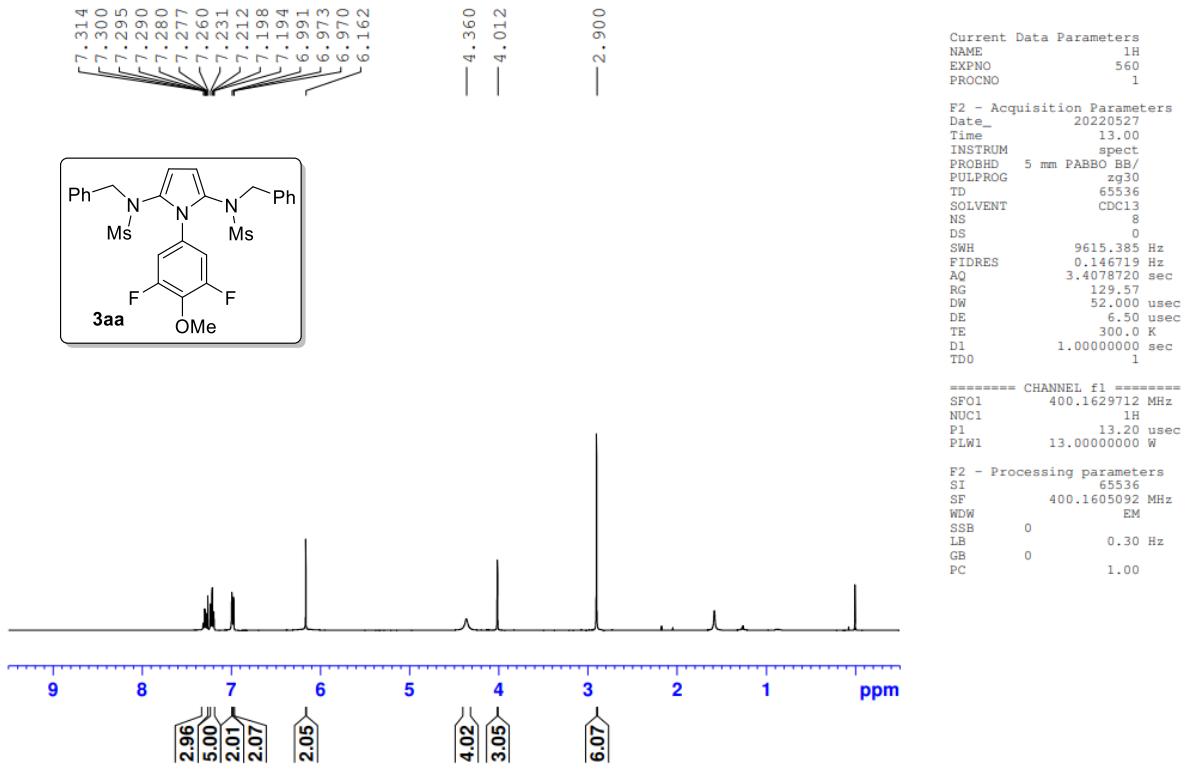
F2 - Processing parameters
SI                 32768
SF            125.8951680 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

Sample Name	HRMS22I13JAN12	**Position**	Vial 12	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	NG-19-02.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	1/13/2022 12:16:27 PM



GR-21-57A

^{1}H NMR, $CDCl_3$, 400 MHz




```
Current Data Parameters
NAME                   1H
EXPNO                 560
PROCNO                  1

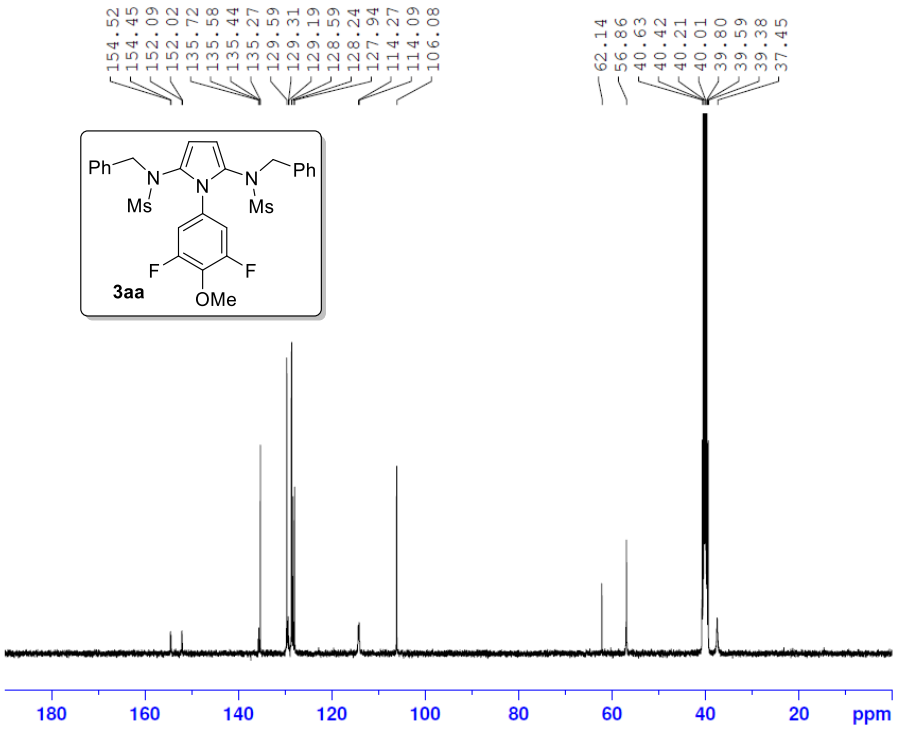
F2 - Acquisition Parameters
Date_           20220527
Time               13.00
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH             9615.385 Hz
FIDRES          0.146719 Hz
AQ              3.4078720 sec
RG                129.57
DW                52.000 usec
DE                 6.50 usec
TE                300.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1629712 MHz
NUC1                   1H
P1                 13.20 usec
PLW1          13.00000000 W

F2 - Processing parameters
SI                 65536
SF            400.1605092 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

GR 21 57L

^{13}C NMR, d_6-DMSO, 101 MHz




```
Current Data Parameters
NAME            GR-21-57A
EXPNO                 350
PROCNO                  1

F2 - Acquisition Parameters
Date_           20220212
Time                5.18
INSTRUM            spect
PROBHD    5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT            DMSO
NS                 2048
DS                    0
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631488 sec
RG               201.48
DW               20.800 usec
DE                6.50 usec
TE               300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

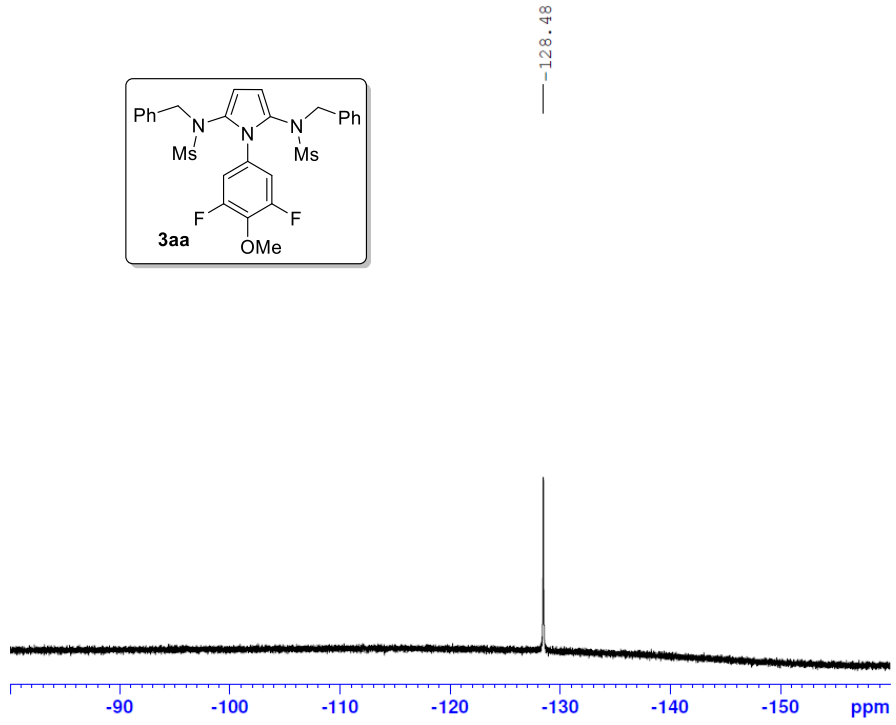
======== CHANNEL f1 ========
SFO1         100.6304993 MHz
NUC1                13C
P1                 9.90 usec
PLW1         53.00000000 W

======== CHANNEL f2 ========
SFO2         400.1621006 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         13.00000000 W
PLW12         0.27963999 W
PLW13         0.22651000 W

F2 - Processing parameters
SI                 32768
SF           100.6204380 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

gr-21-57

^{19}F NMR, $CDCl_3$, 376 MHz




```
Current Data Parameters
NAME            GR-21-57A
EXPNO                 340
PROCNO                  1

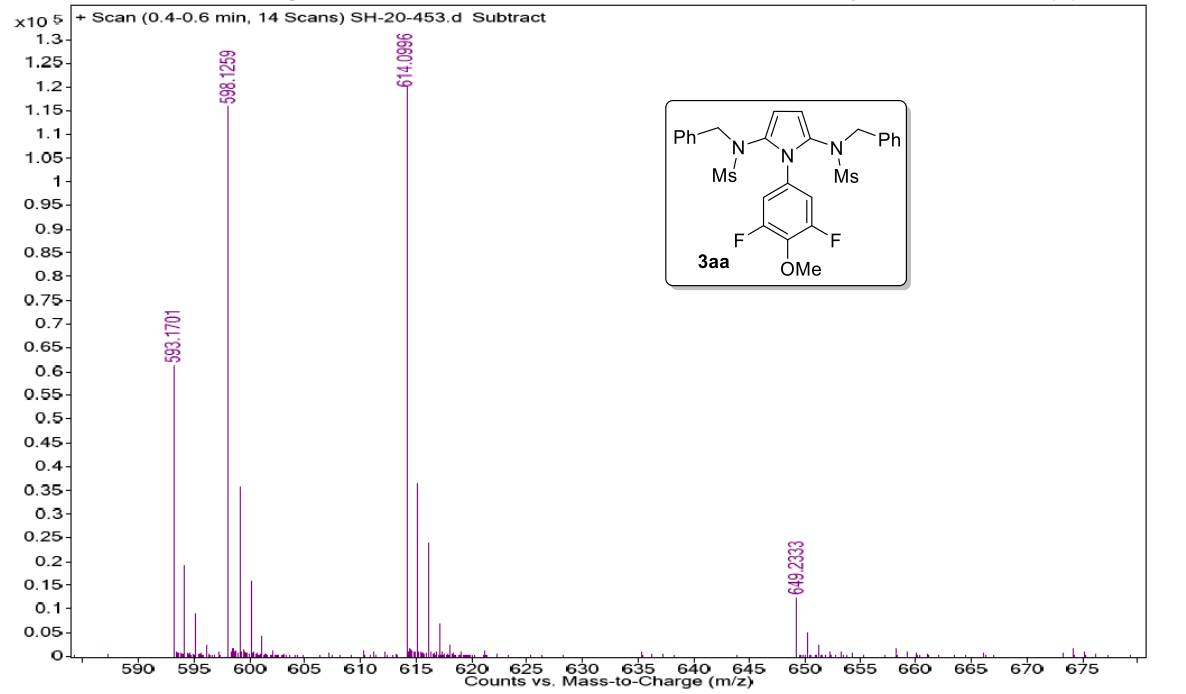
F2 - Acquisition Parameters
Date_           20211208
Time               12.33
INSTRUM            spect
PROBHD    5 mm PABBO BB/
PULPROG       zgfhigqn.2
TD                131072
SOLVENT             CDC13
NS                    16
DS                     4
SWH           89285.711 Hz
FIDRES        0.681196 Hz
AQ            0.7340032 sec
RG               201.48
DW                5.600 usec
DE                6.50 usec
TE               300.1 K
D1            1.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         376.4894122 MHz
NUC1                19F
P1                15.00 usec
PLW1         21.00000000 W

======== CHANNEL f2 ========
SFO2         400.1621006 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         13.00000000 W
PLW12         0.27963999 W

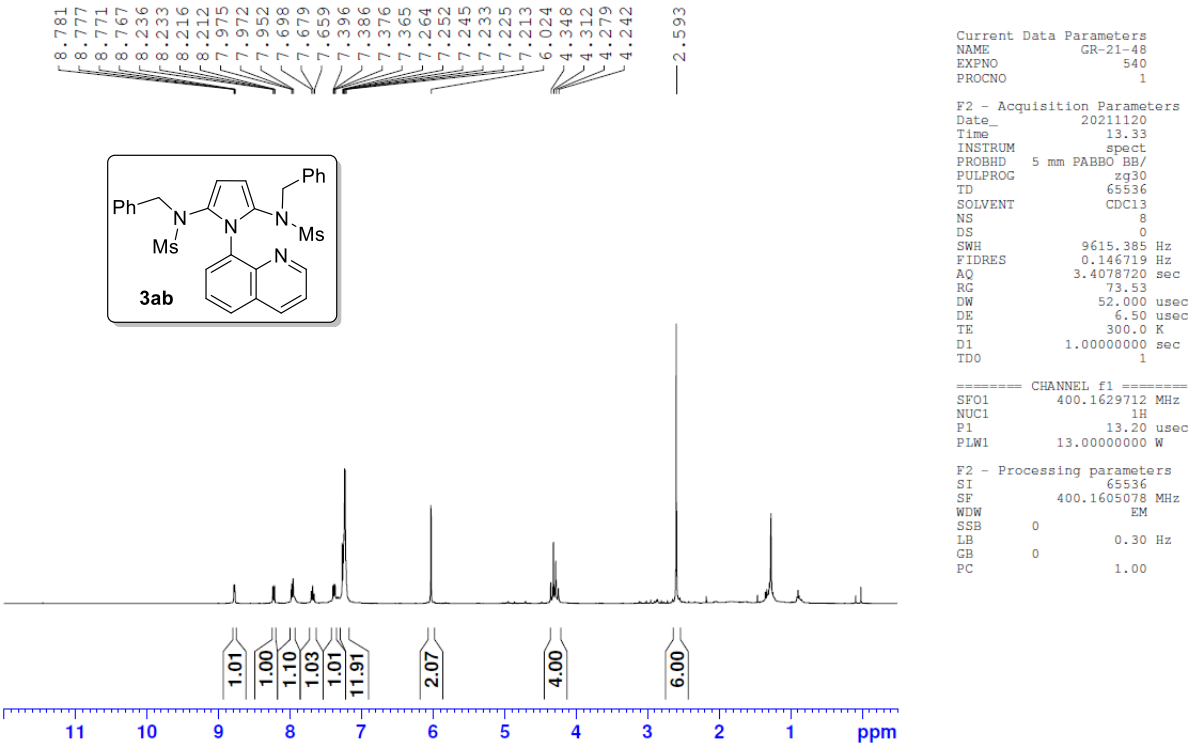
F2 - Processing parameters
SI                 65536
SF           376.5270650 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

Sample Name	HRMS21I13DEC14	**Position**	Vial 14	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	SH-20-453.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	12/13/2021 12:26:54 PM



GR-21-48

^{1}H NMR, $CDCl_3$, 400 MHz




```
Current Data Parameters
NAME                  GR-21-48
EXPNO                      540
PROCNO                       1

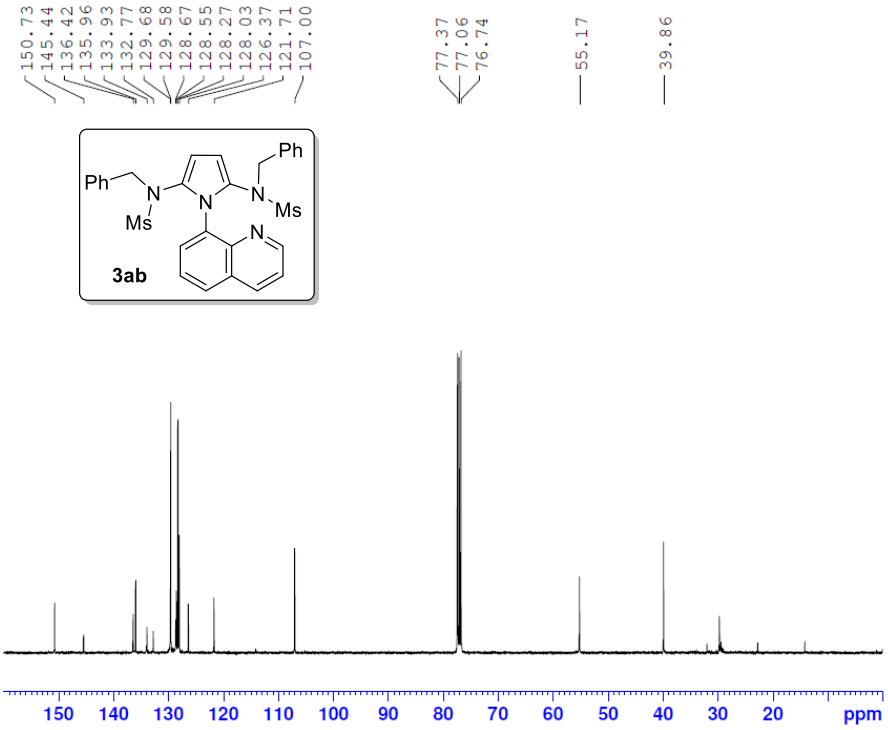
F2 - Acquisition Parameters
Date_                 20211120
Time                     13.33
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                      65536
SOLVENT                  CDCl3
NS                           8
DS                           0
SWH                   9615.385 Hz
FIDRES                0.146719 Hz
AQ                    3.4078720 sec
RG                       73.53
DW                       52.000 usec
DE                        6.50 usec
TE                       300.0 K
D1                    1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                400.1629712 MHz
NUC1                        1H
P1                       13.20 usec
PLW1                13.00000000 W

F2 - Processing parameters
SI                       65536
SF                  400.1605078 MHz
WDW                         EM
SSB                  0
LB                       0.30 Hz
GB                   0
PC                       1.00
```

GR-21-48

^{13}C NMR, $CDCl_3$, 101 MHz




```
Current Data Parameters
NAME                  Desktop
EXPNO                     550
PROCNO                      1

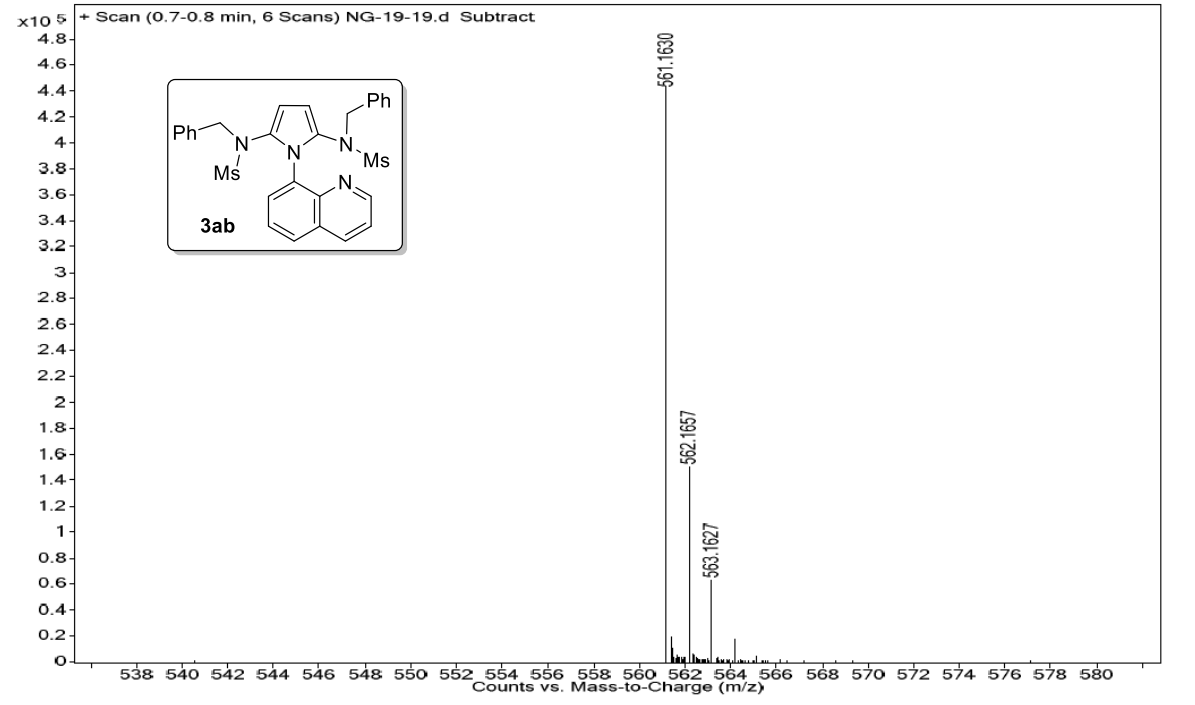
F2 - Acquisition Parameters
Date_              20211120
Time                  15.31
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG               zgpg30
TD                     65536
SOLVENT                CDCl3
NS                      2048
DS                         4
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                 1.3631488 sec
RG                    201.48
DW                   20.800 usec
DE                    6.50 usec
TE                   300.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1             100.6304993 MHz
NUC1                      13C
P1                       9.90 usec
PLW1             53.00000000 W

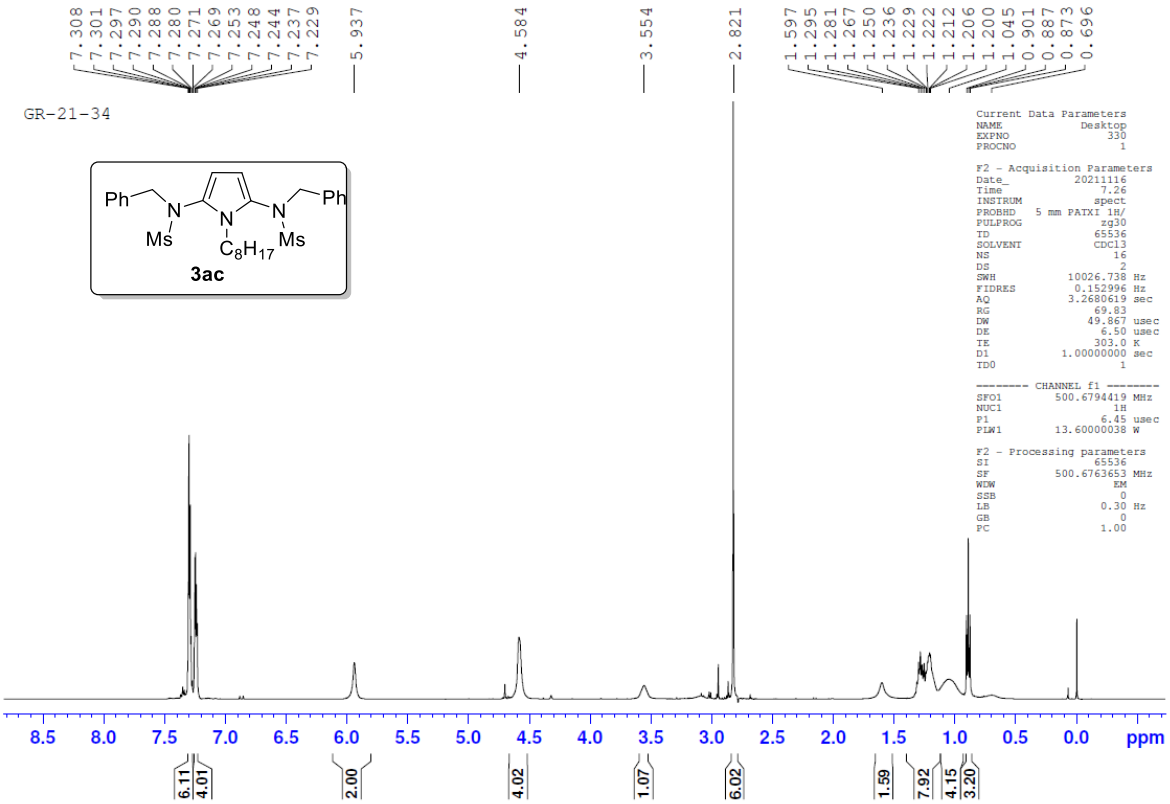
======== CHANNEL f2 ========
SFO2             400.1621006 MHz
NUC2                      1H
CPDPRG[2              waltz16
PCPD2                 90.00 usec
PLW2             13.00000000 W
PLW12             0.27963999 W
PLW13             0.22651000 W

F2 - Processing parameters
SI                        32768
SF               100.6204380 MHz
WDW                          EM
SSB          0
LB                     1.00 Hz
GB           0
PC                     1.40
```

Sample Name	HRMS22I14JAN25	**Position**	Vial 25	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	NG-19-19.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	1/14/2022 12:57:30 PM

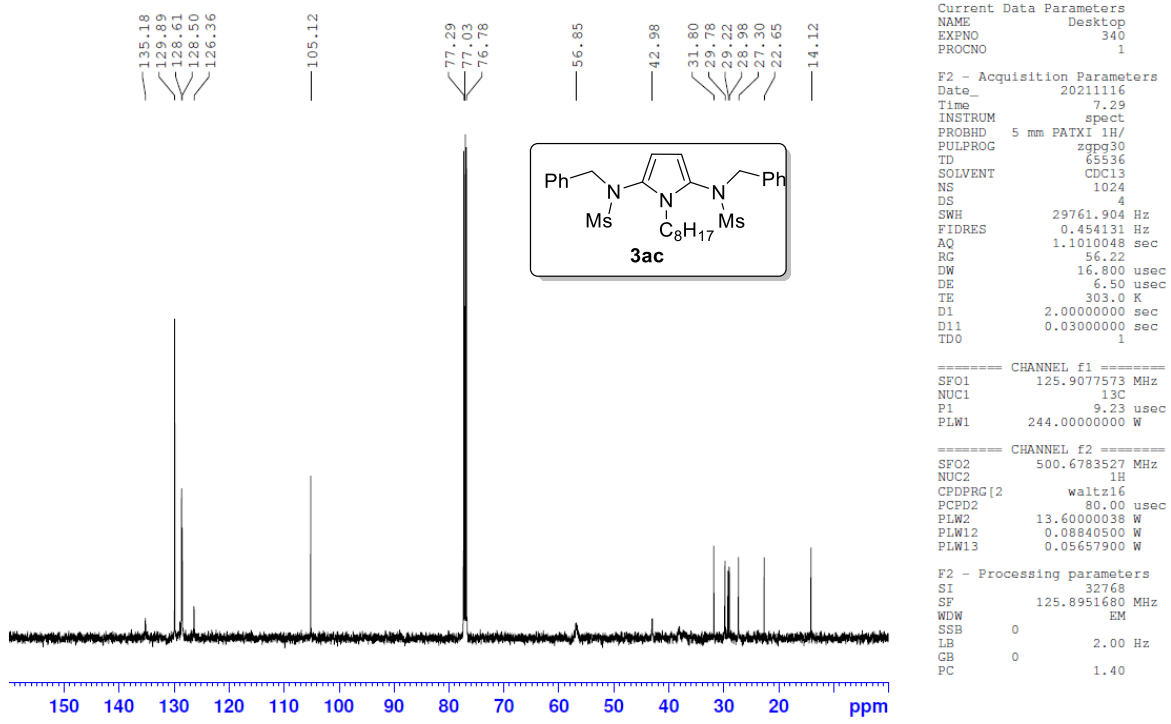


^{1}H NMR, $CDCl_3$, 500 MHz

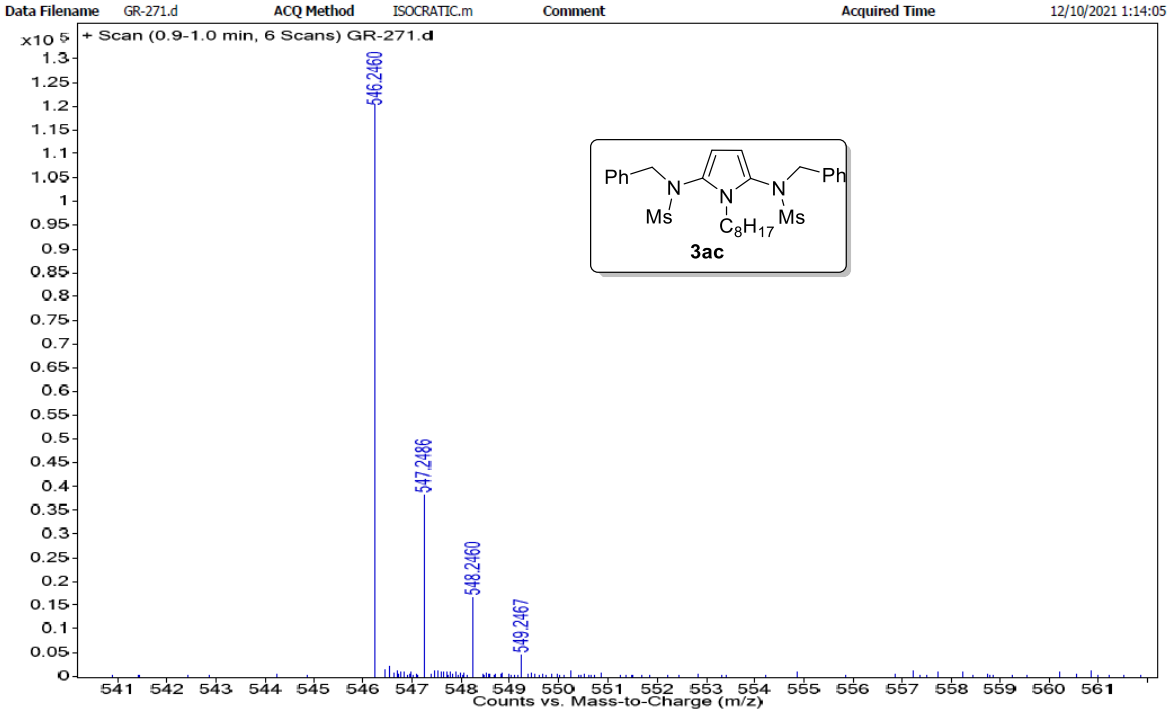


GR-21-34

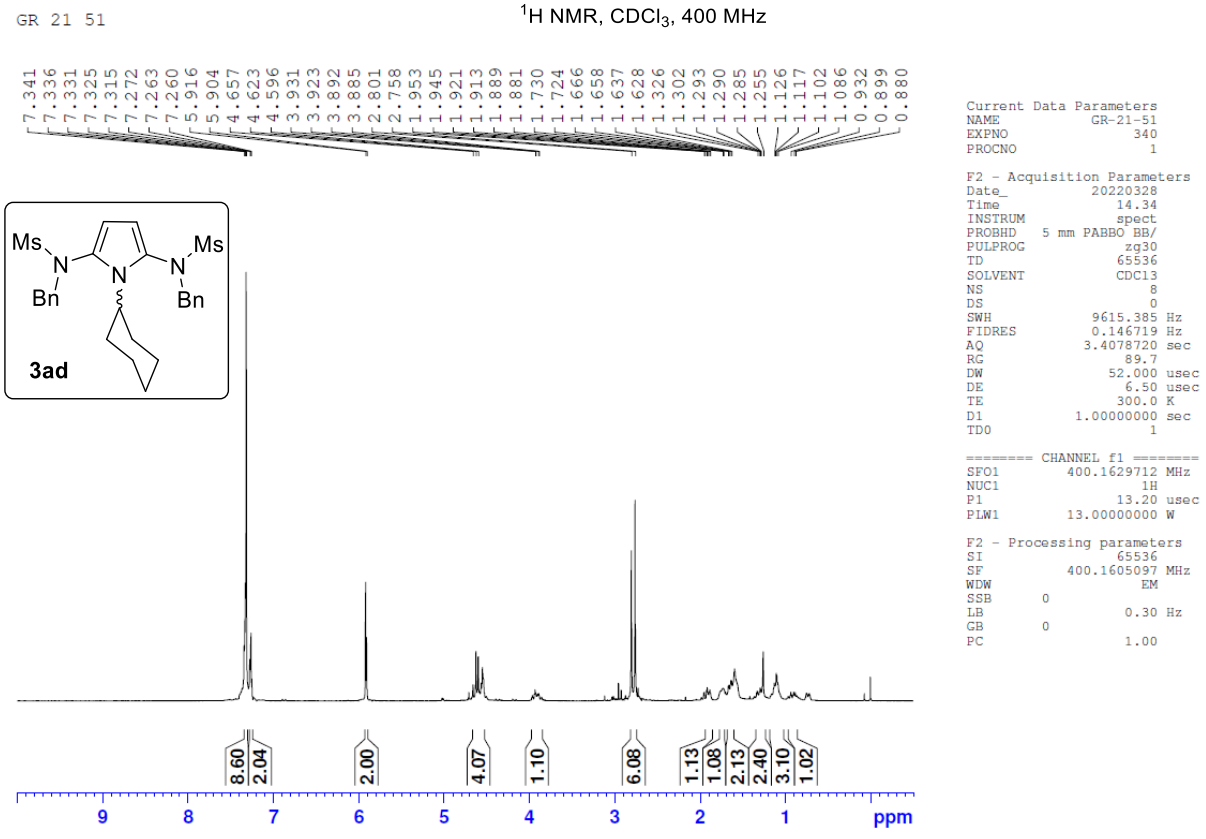
^{13}C NMR, $CDCl_3$, 125 MHz



Sample Name	HRMS21I10DEC21	**Position**	Vial 21	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	10	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	GR-271.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	12/10/2021 1:14:05 PM



^{1}H NMR, $CDCl_3$, 400 MHz



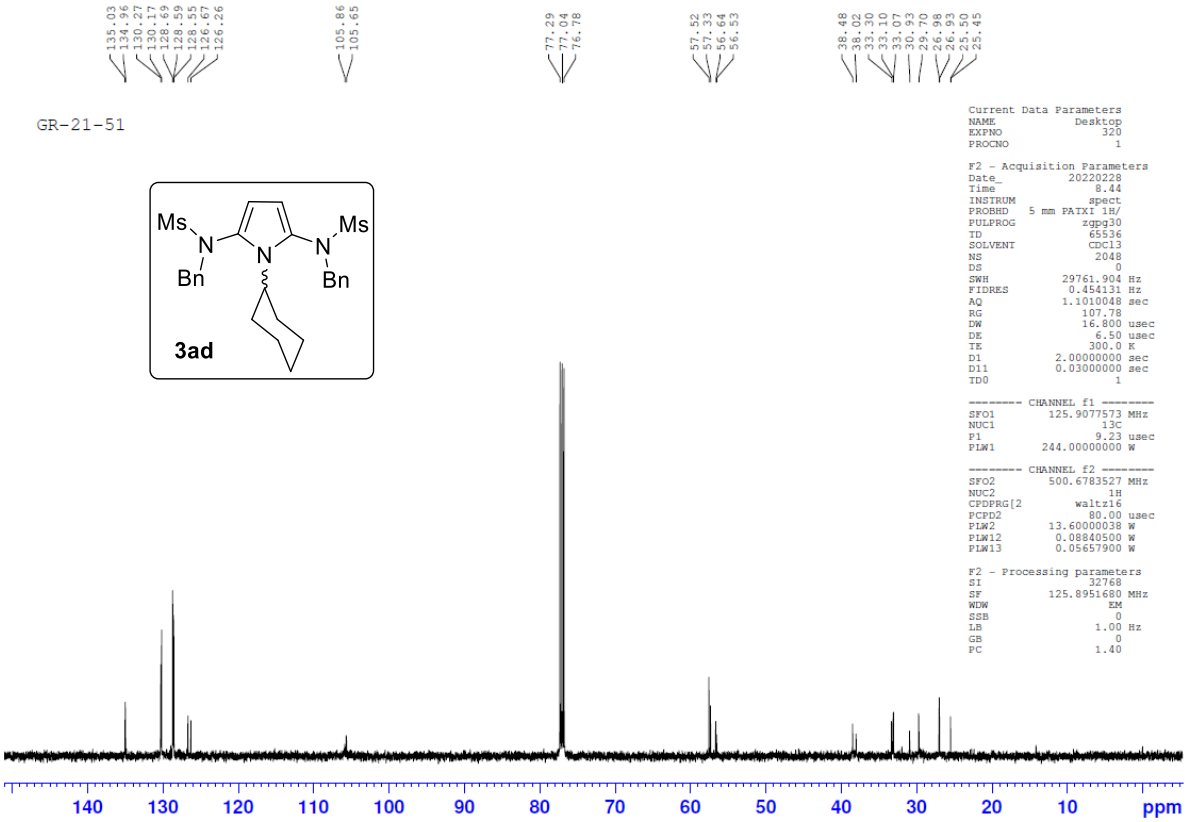

```
Current Data Parameters
NAME                  GR-21-51
EXPNO                      340
PROCNO                       1

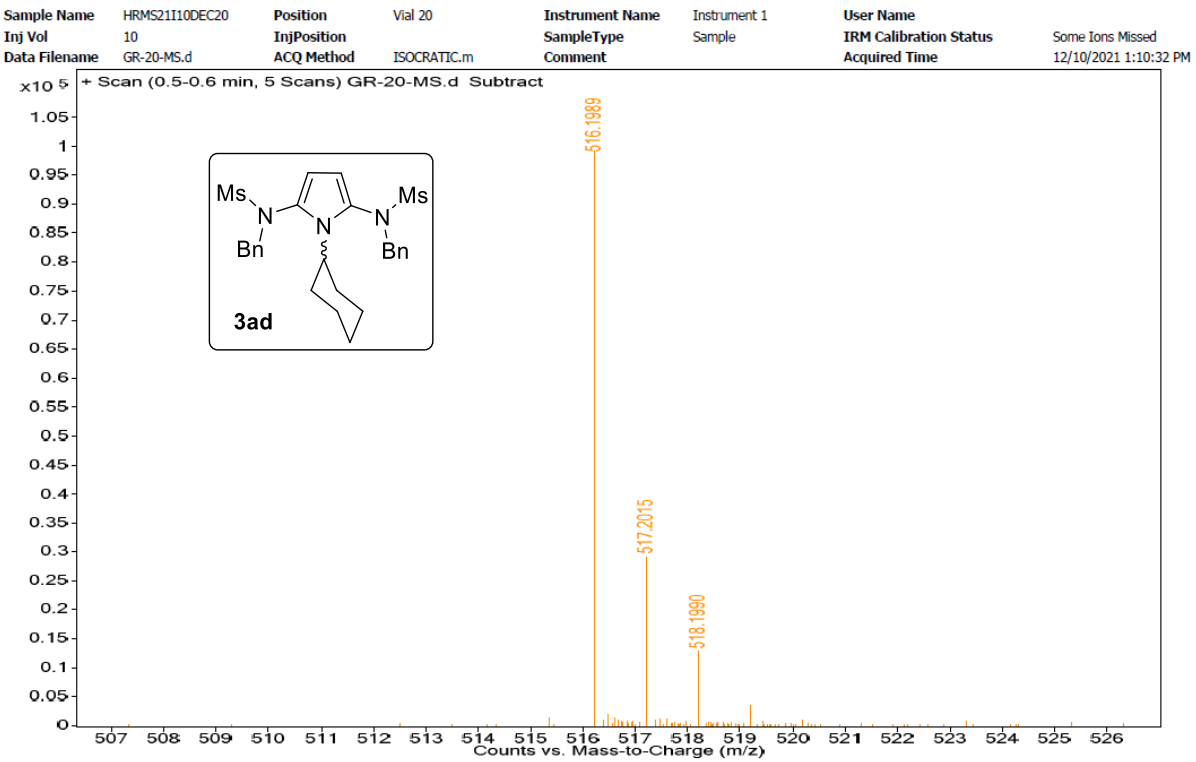
F2 - Acquisition Parameters
Date_                 20220328
Time                     14.34
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                          8
DS                          0
SWH                  9615.385 Hz
FIDRES               0.146719 Hz
AQ                   3.4078720 sec
RG                        89.7
DW                      52.000 usec
DE                       6.50 usec
TE                      300.0 K
D1                 1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1               400.1629712 MHz
NUC1                       1H
P1                      13.20 usec
PLW1              13.00000000 W

F2 - Processing parameters
SI                      65536
SF                 400.1605097 MHz
WDW                        EM
SSB          0
LB                       0.30 Hz
GB           0
PC                       1.00
```

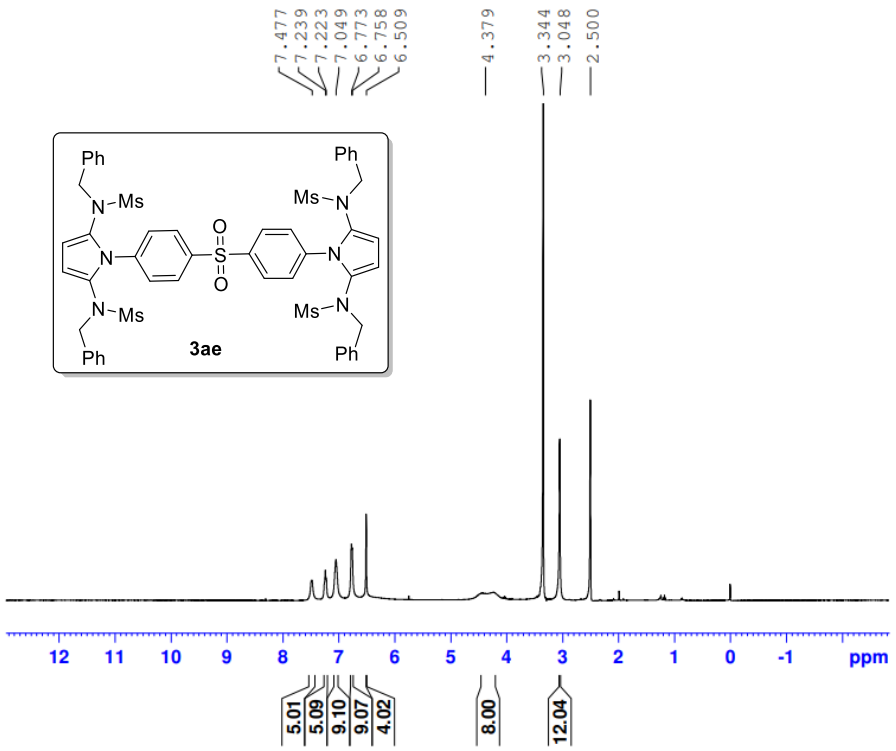
^{13}C NMR, $CDCl_3$, 125 MHz





GR-21-28

^{1}H NMR, d_6-DMSO, 400 MHz




```
Current Data Parameters
NAME                   1H
EXPNO                 340
PROCNO                  1

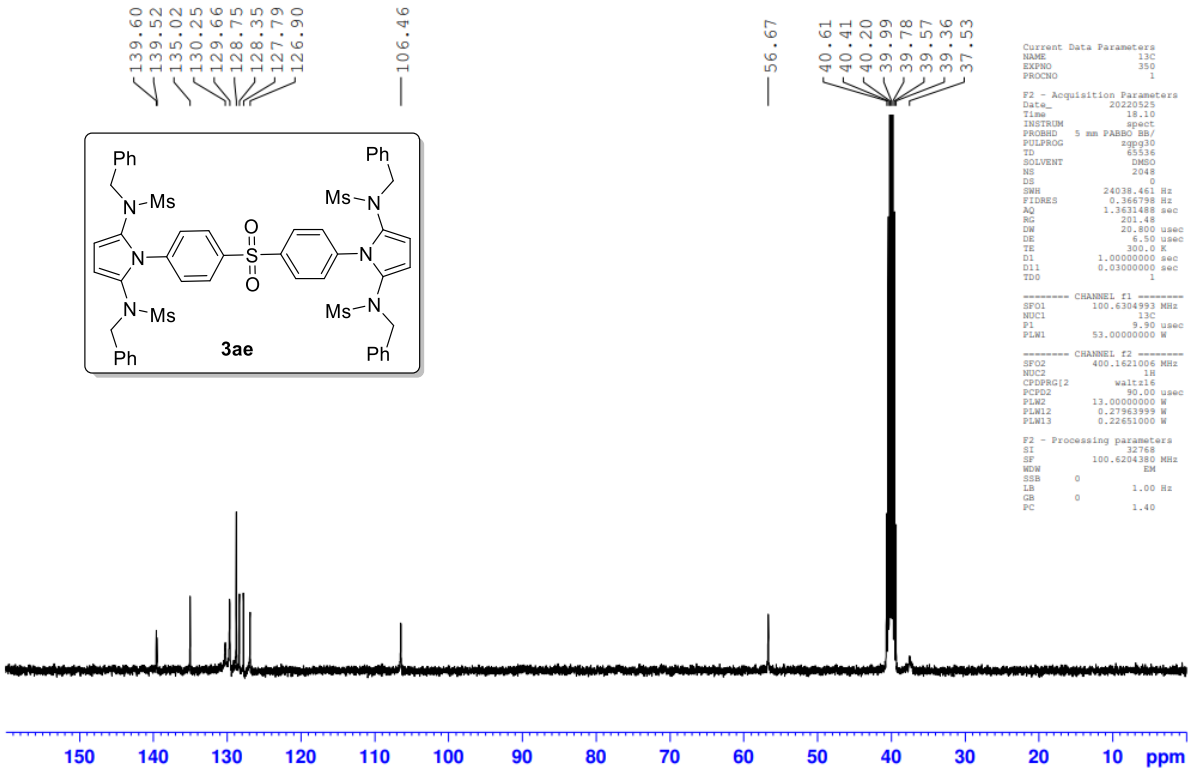
F2 - Acquisition Parameters
Date_           20220525
Time               17.44
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                     8
DS                     0
SWH             9615.385 Hz
FIDRES          0.146719 Hz
AQ              3.4078720 sec
RG                 80.54
DW                52.000 usec
DE                 6.50 usec
TE                300.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1629712 MHz
NUC1                  1H
P1                 13.20 usec
PLW1          13.00000000 W

F2 - Processing parameters
SI                 65536
SF            400.1605028 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

^{13}C NMR, d_6-DMSO, 101 MHz

GR-21-28




```
Current Data Parameters
NAME                   13C
EXPNO                  350
PROCNO                   1

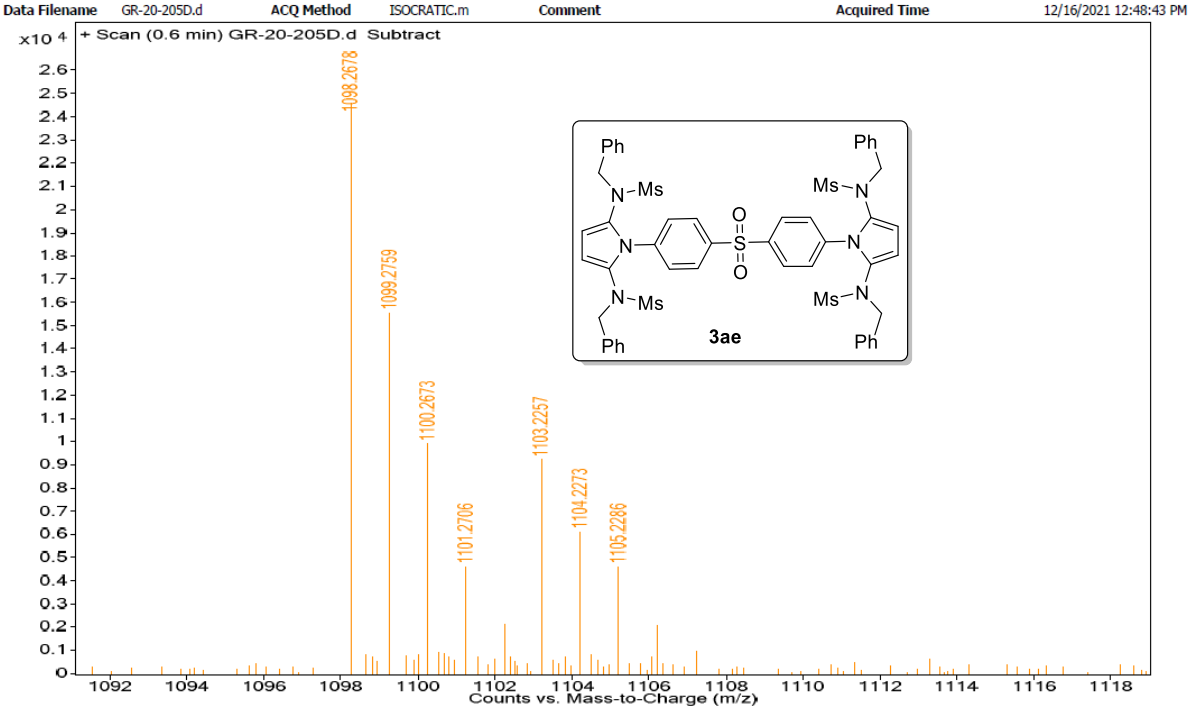
F2 - Acquisition Parameters
Date_           20220525
Time               18.10
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 65536
SOLVENT             DMSO
NS                  2048
DS                     0
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631488 sec
RG                201.48
DW                20.800 usec
DE                 6.50 usec
TE                300.0 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          100.6304993 MHz
NUC1                  13C
P1                  9.90 usec
PLW1          53.00000000 W

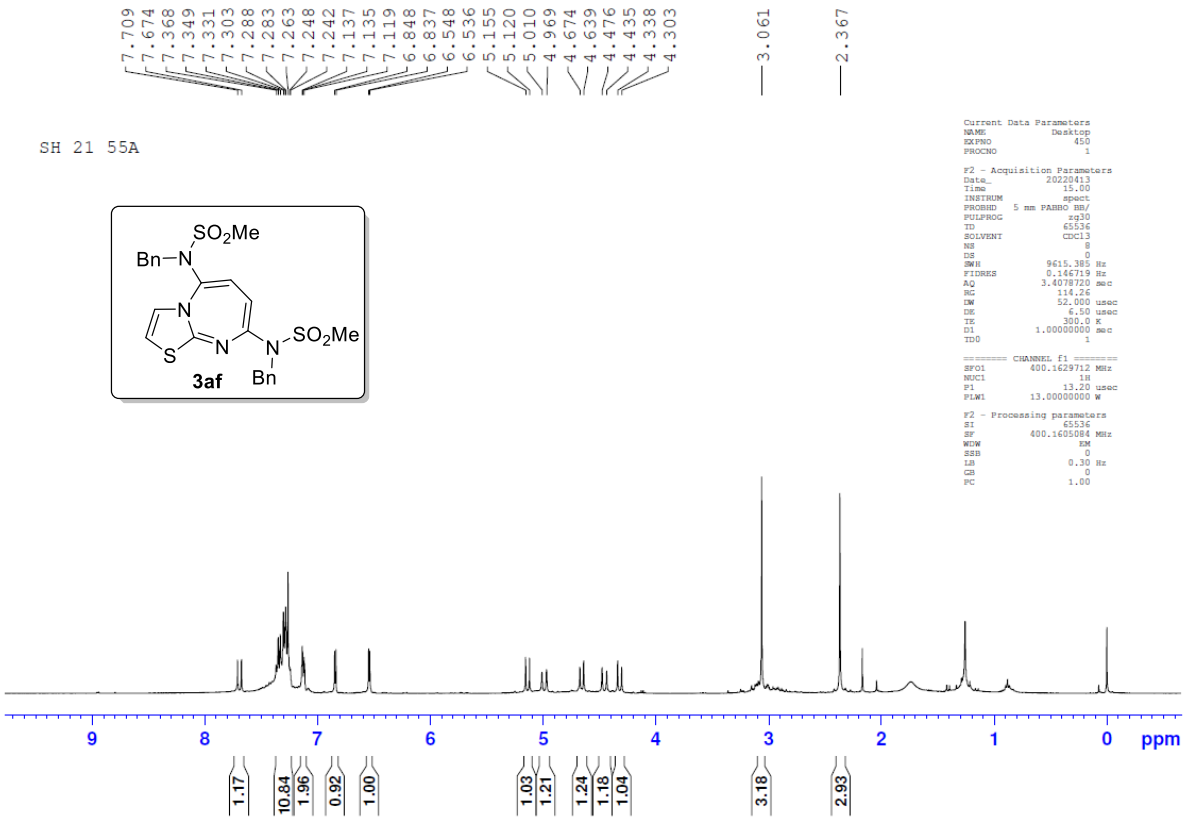
======== CHANNEL f2 ========
SFO2          400.1621006 MHz
NUC2                  1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2          13.00000000 W
PLW12          0.27963999 W
PLW13          0.22651000 W

F2 - Processing parameters
SI                 32768
SF            100.6204380 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

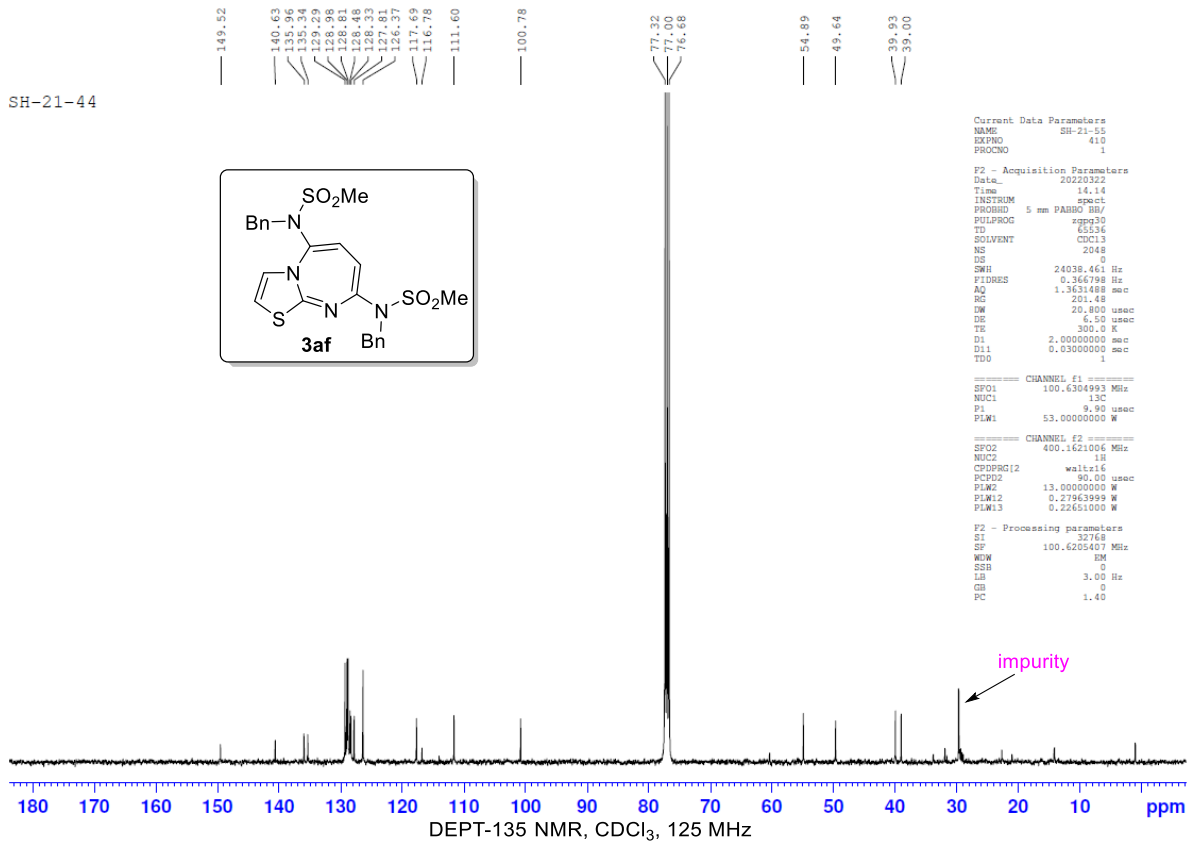
Sample Name	HRMS21I16DEC22	**Position**	Vial 22	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	GR-20-205D.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	12/16/2021 12:48:43 PM



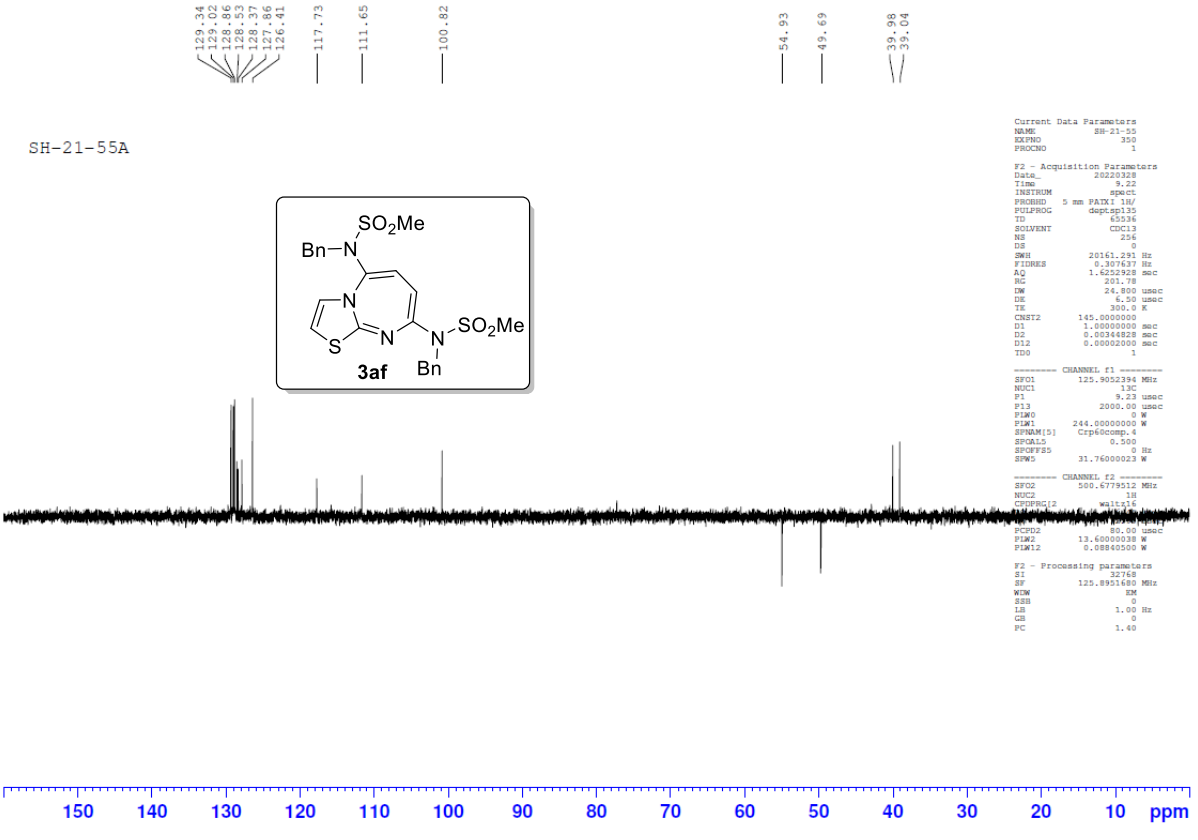
^{1}H NMR, $CDCl_3$, 400 MHz



^{13}C NMR, $CDCl_3$, 101 MHz

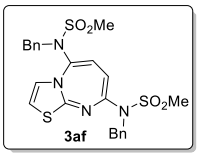


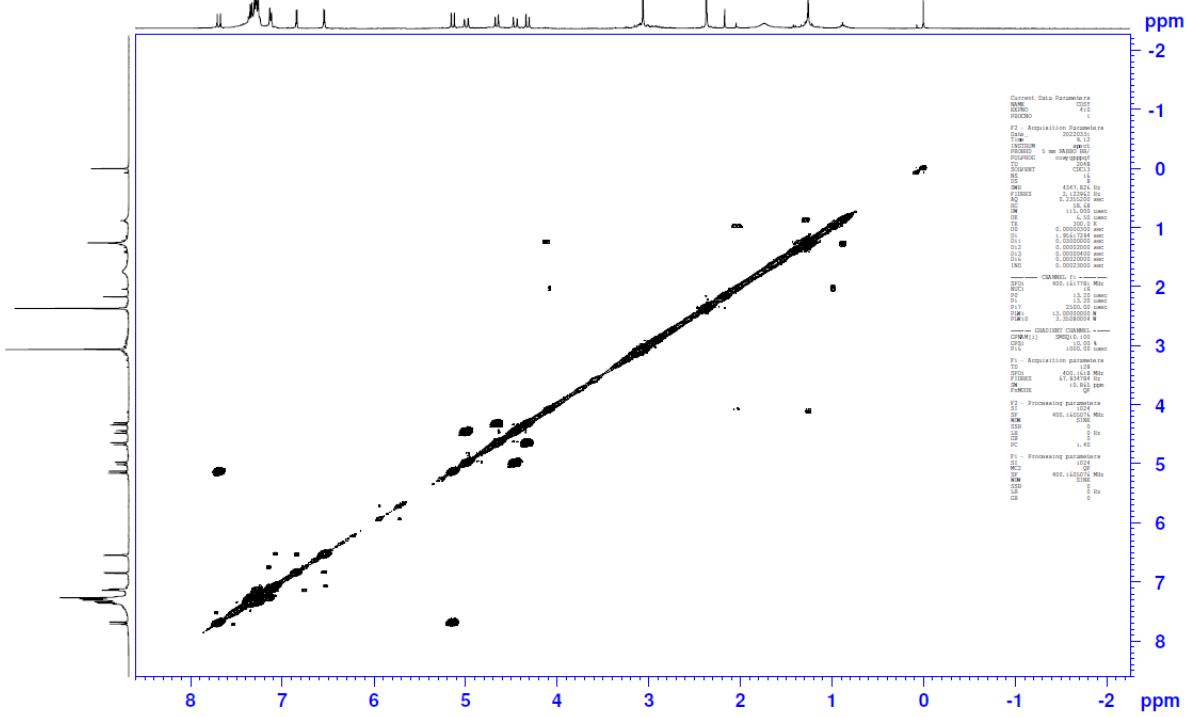
DEPT-135 NMR, $CDCl_3$, 125 MHz



SH-21-55

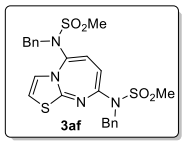
COSY NMR, $CDCl_3$, 400 MHz

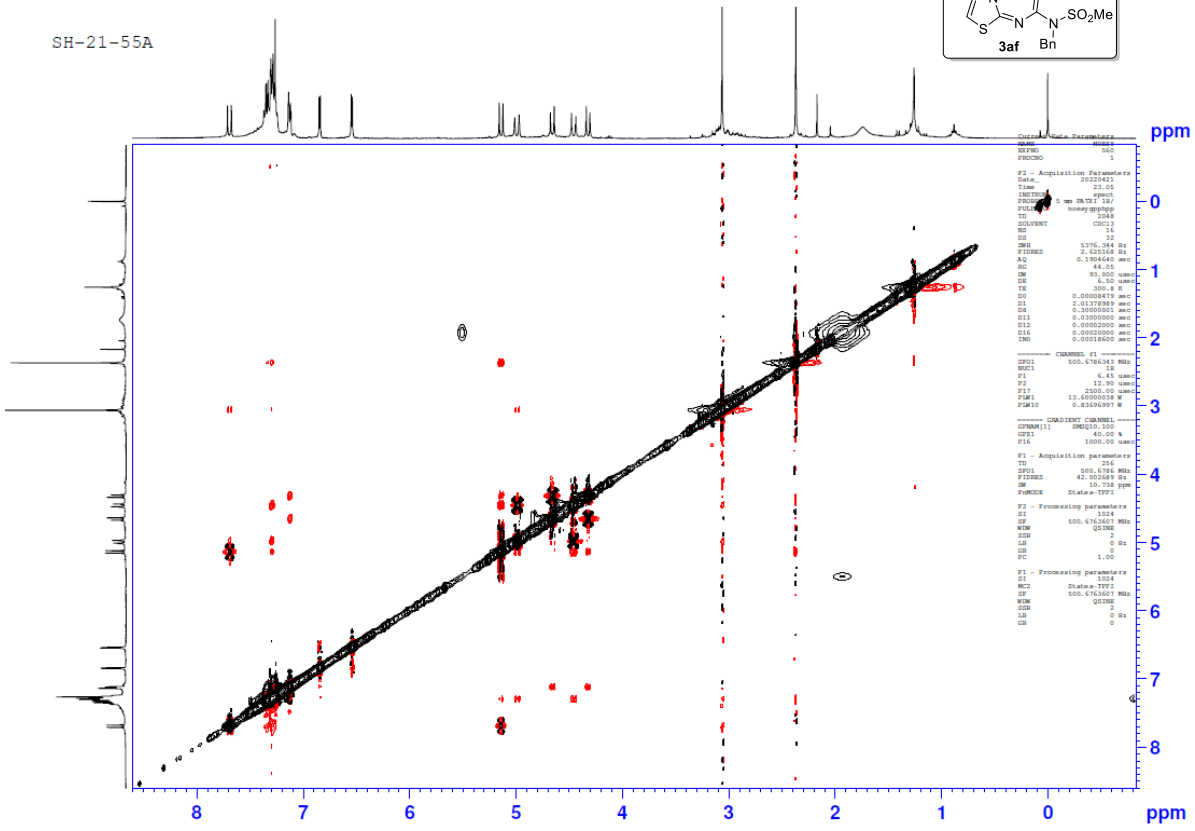




NOESY NMR, $CDCl_3$, 500 MHz

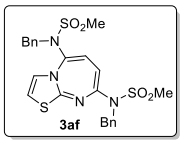
SH-21-55A

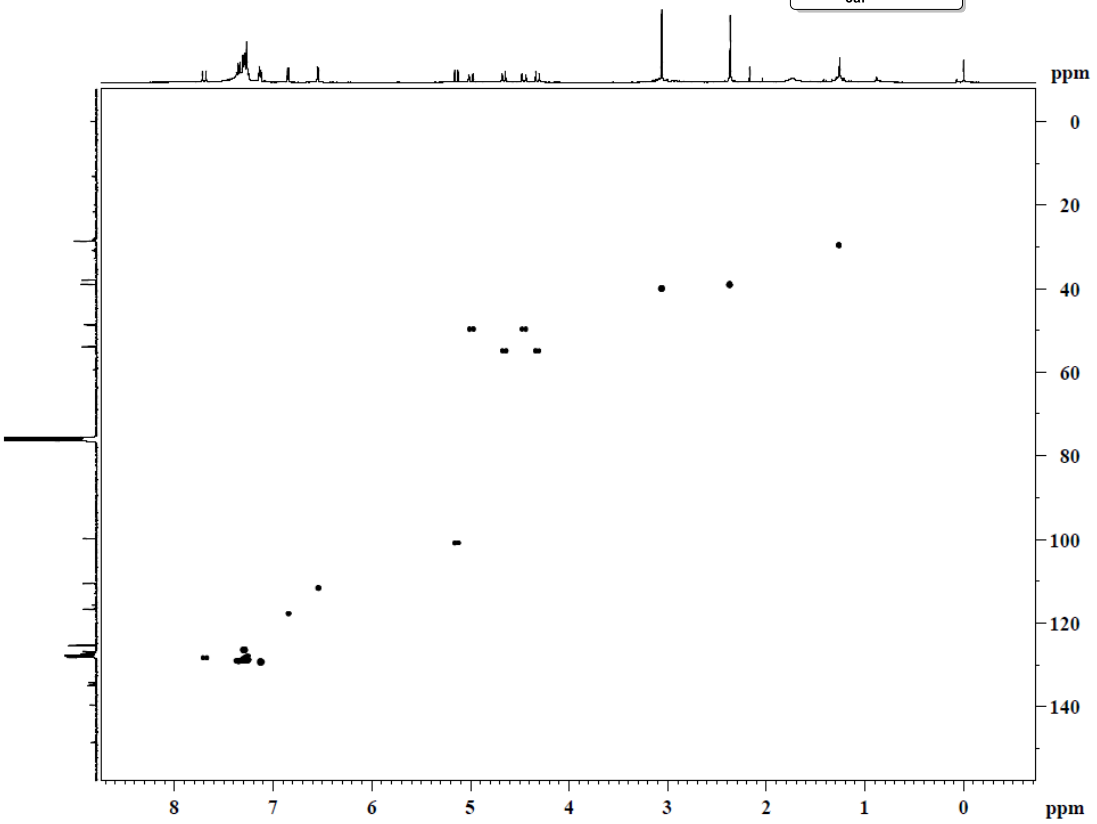




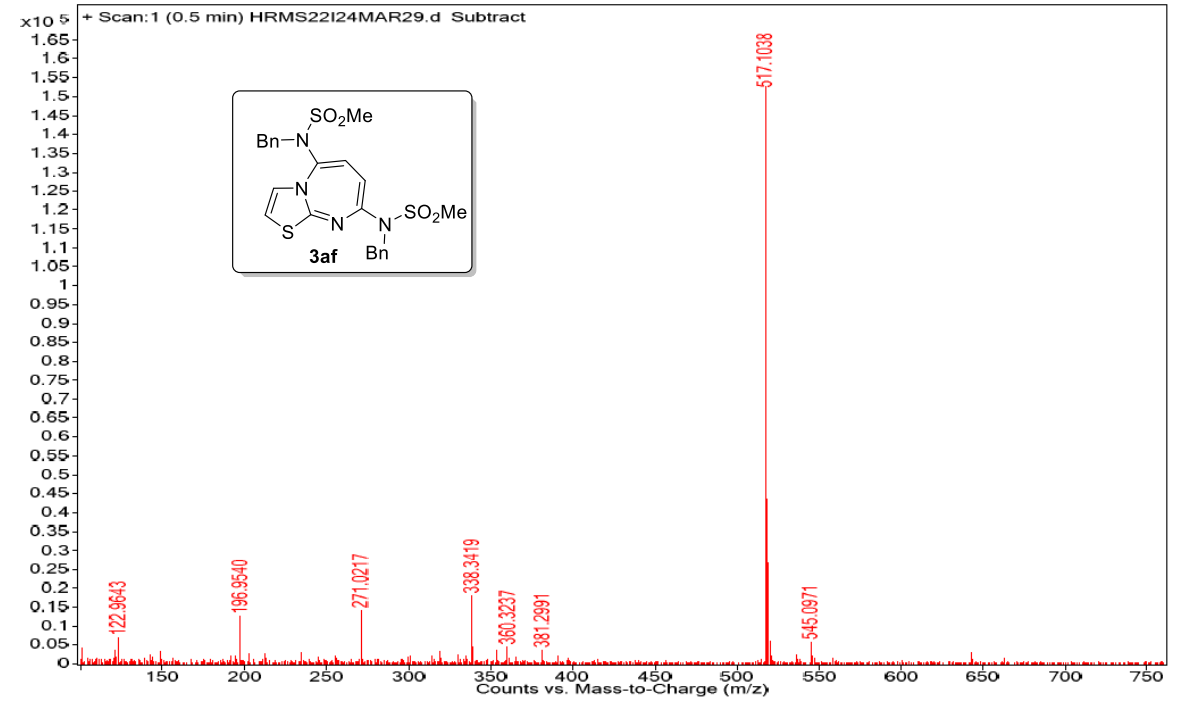
SH-21-55A

HSQC NMR, $CDCl_3$, 101 MHz

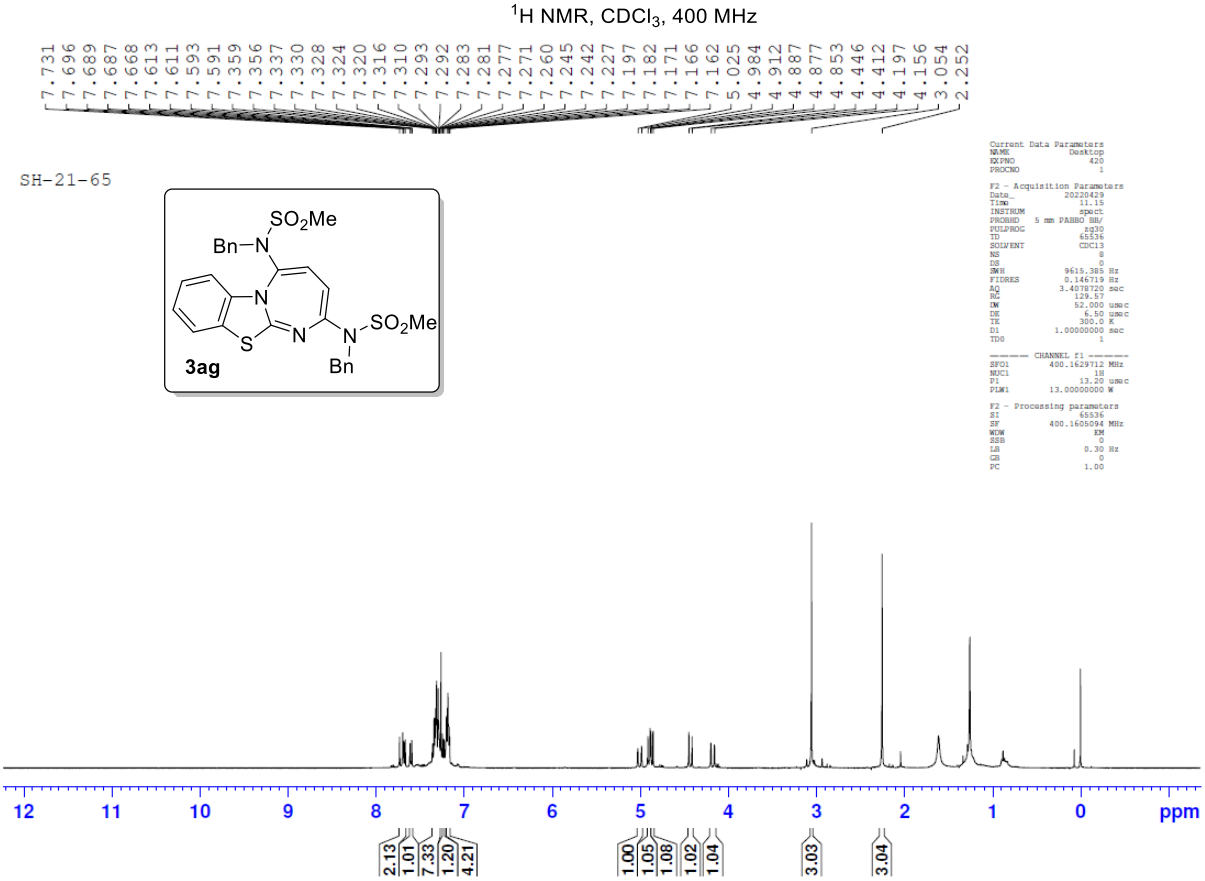




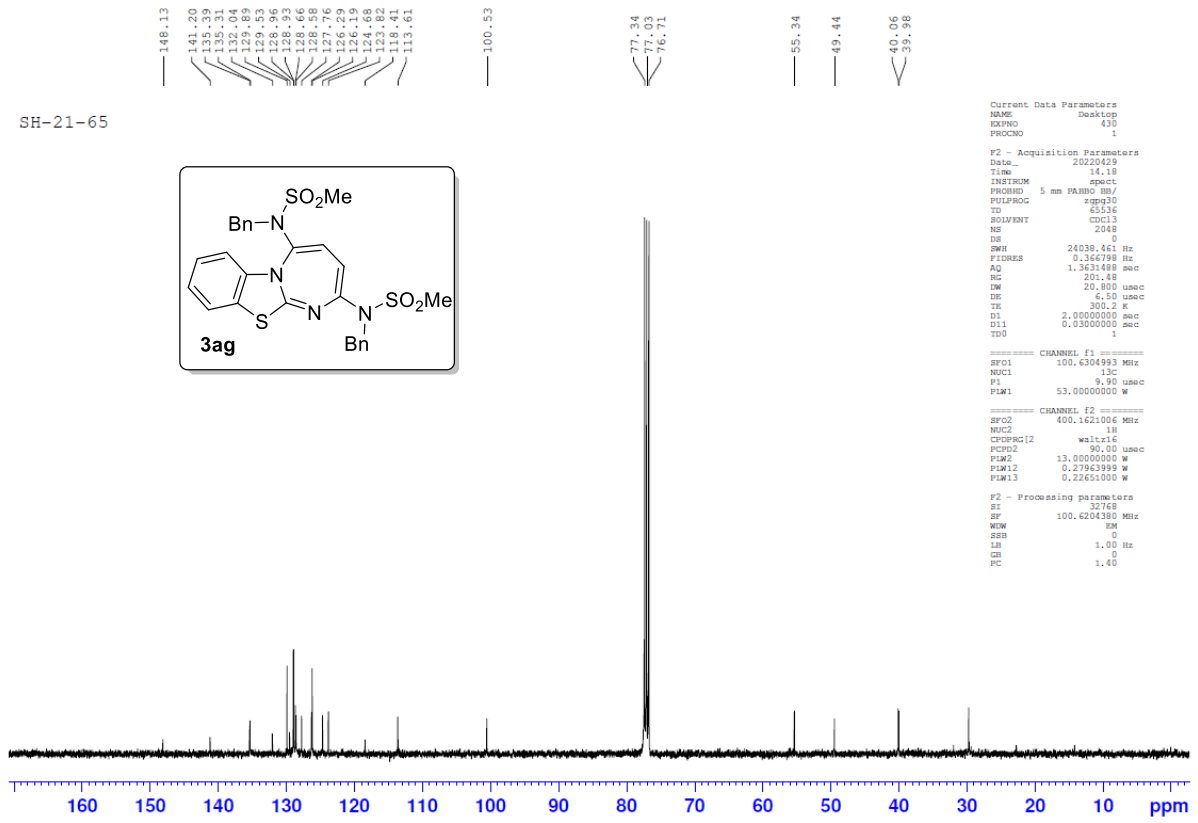
Sample Name	GR-22-13	**Position**	Vial 29	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	HRMS22I24MAR29.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	3/24/2022 12:51:13 PM



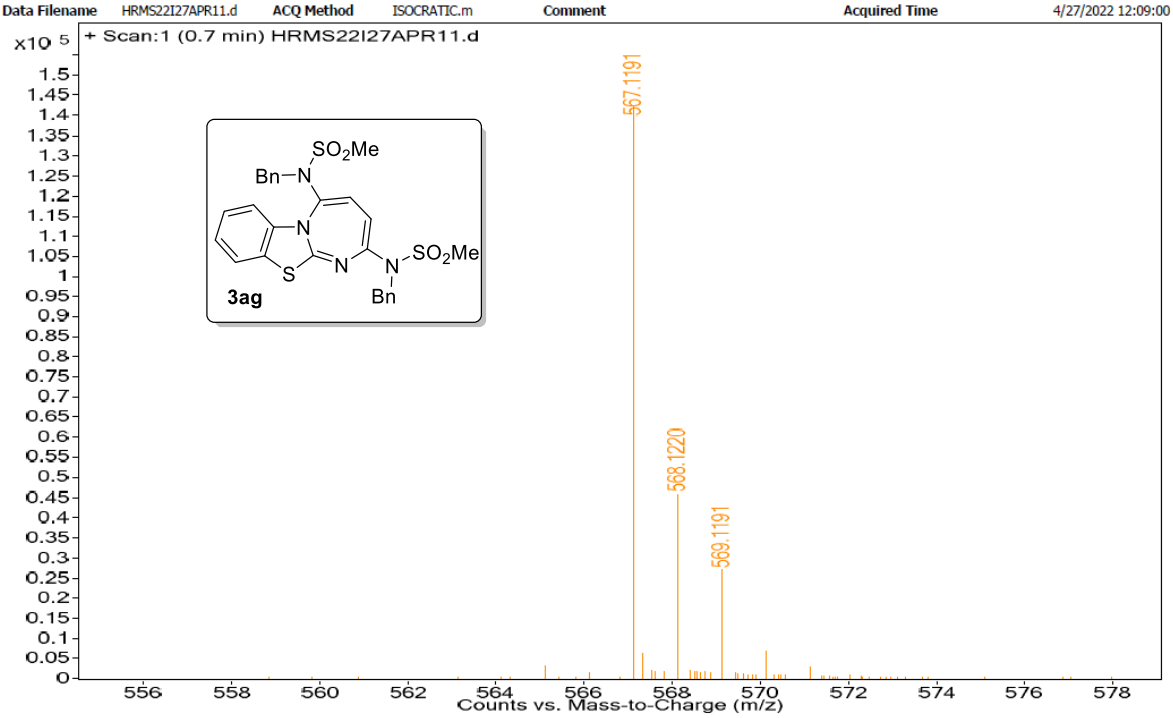
^{1}H NMR, $CDCl_3$, 400 MHz



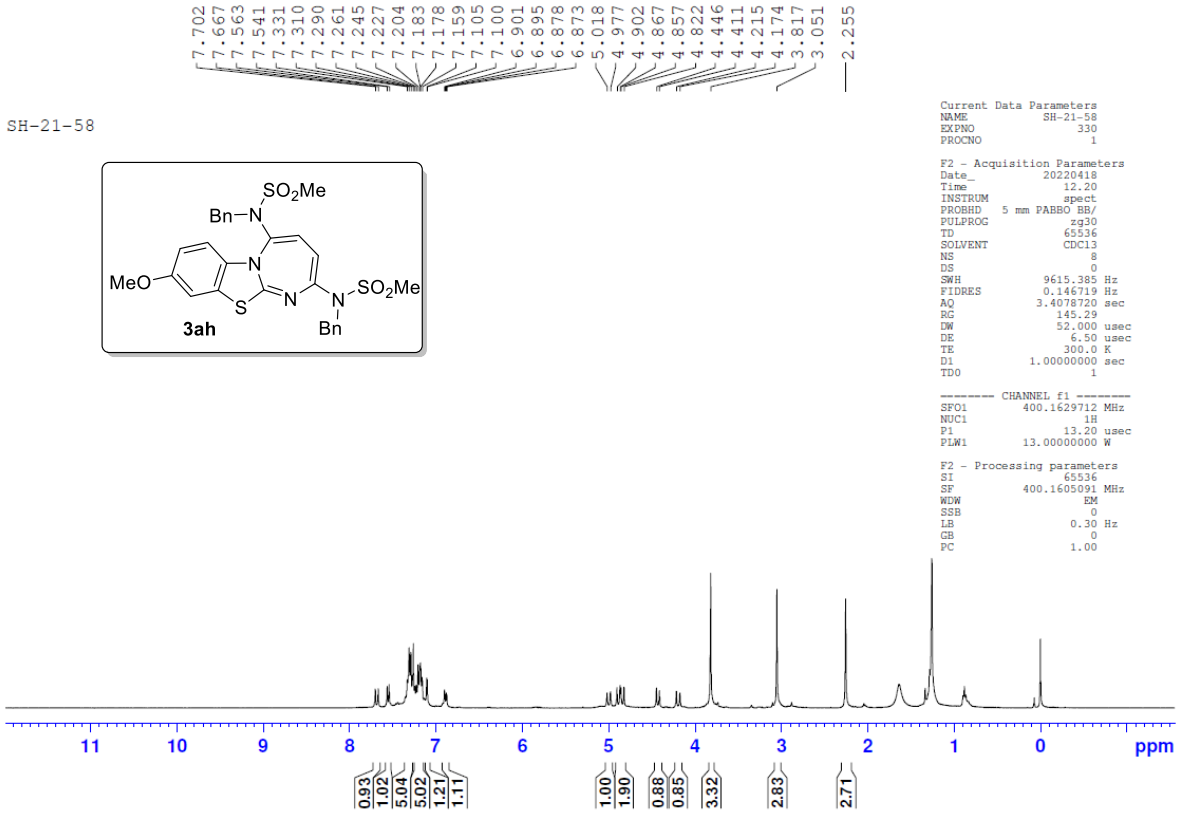
^{13}C NMR, $CDCl_3$, 101 MHz



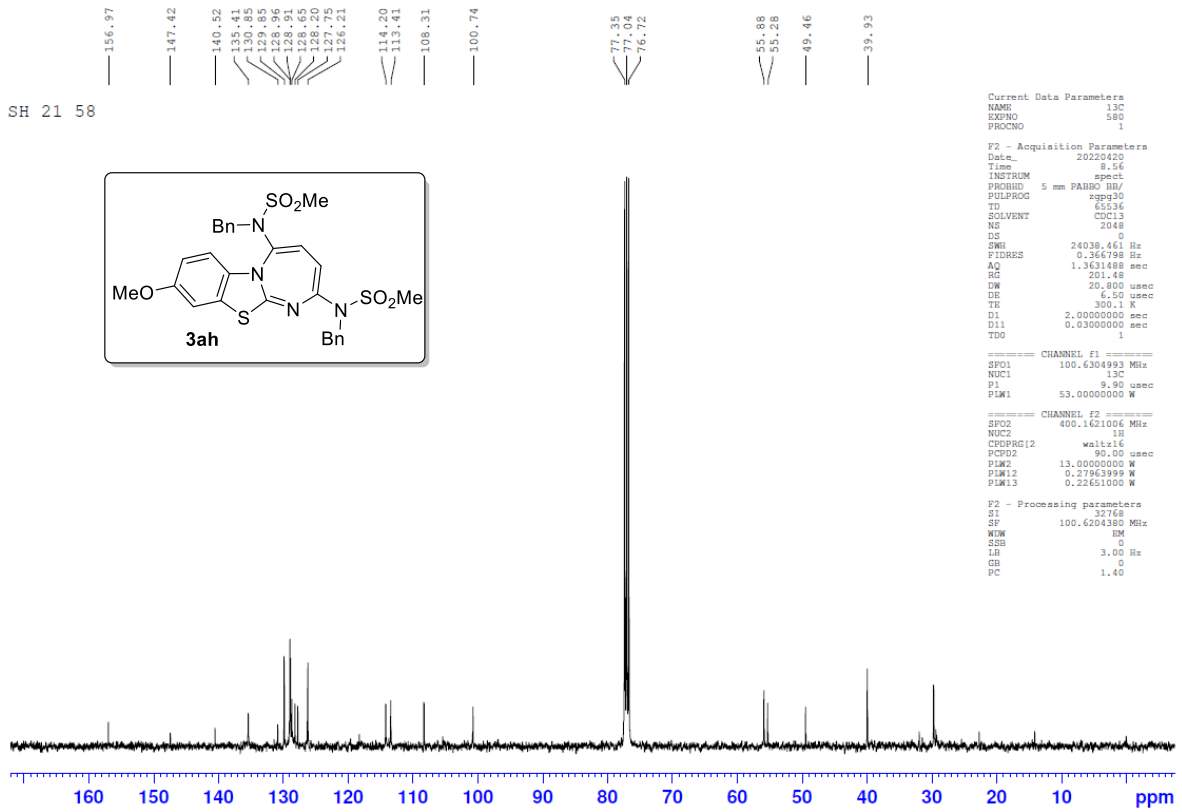
Sample Name	SH-21-09	**Position**	Vial 11	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	HRMS22I27APR11.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	4/27/2022 12:09:00 PM

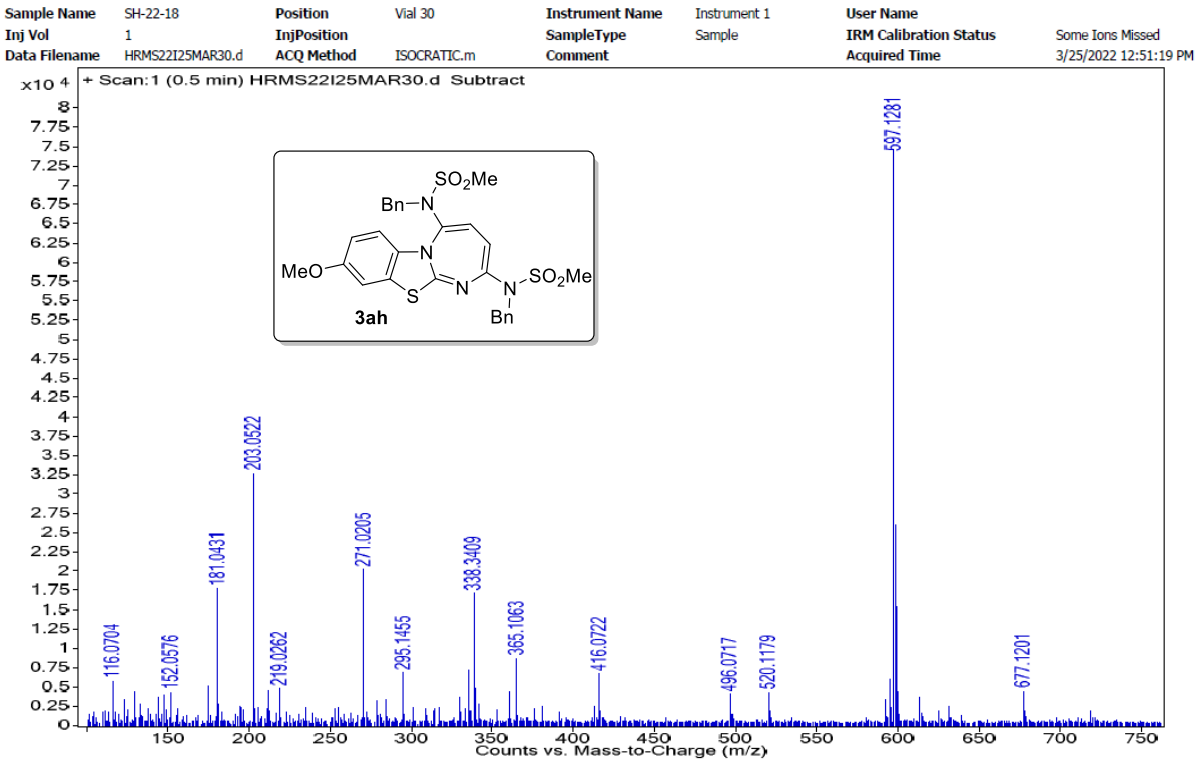


^{1}H NMR, $CDCl_3$, 400 MHz

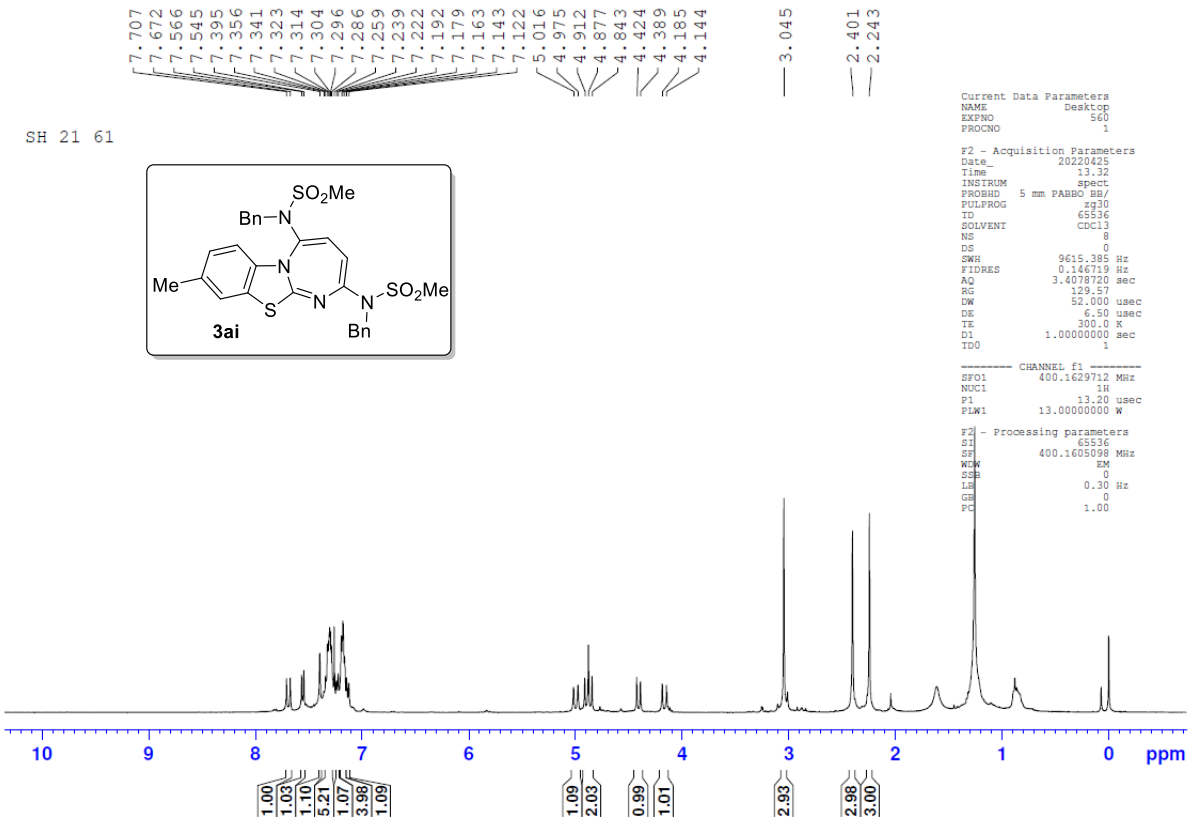


^{13}C NMR, $CDCl_3$, 101 MHz

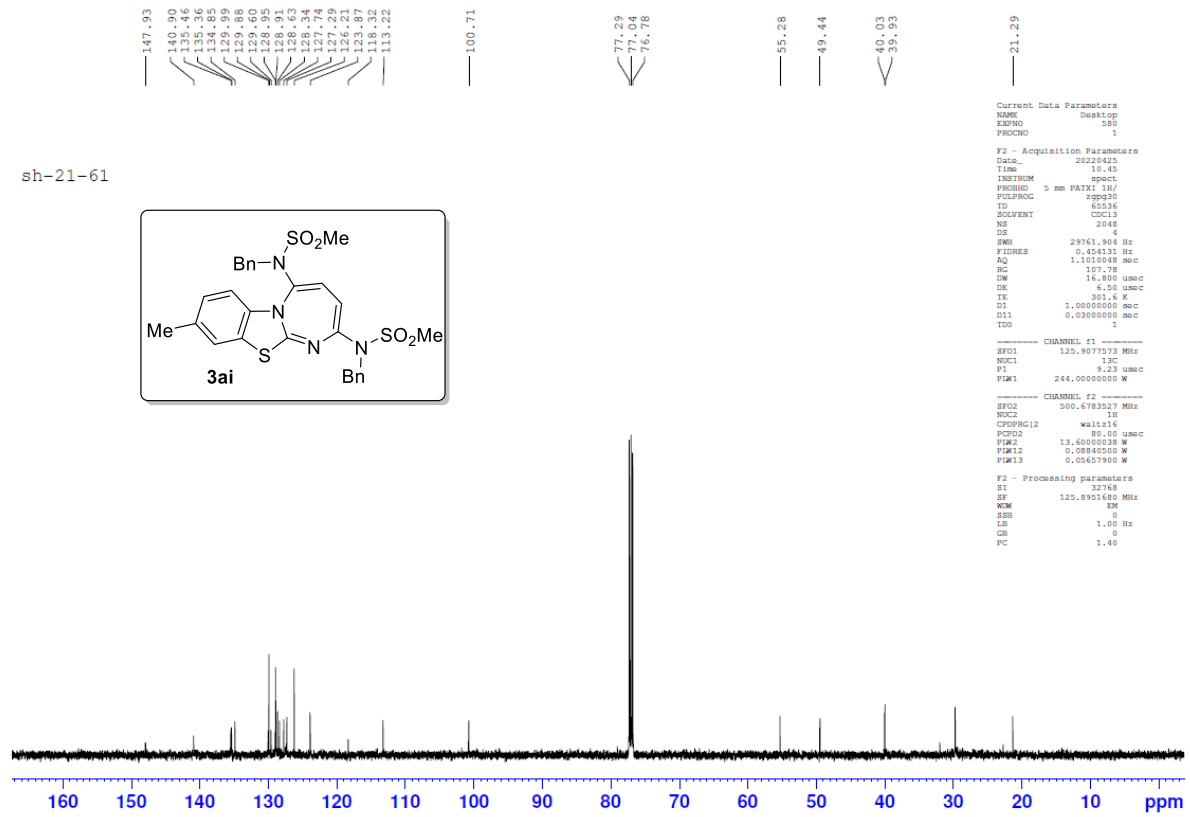




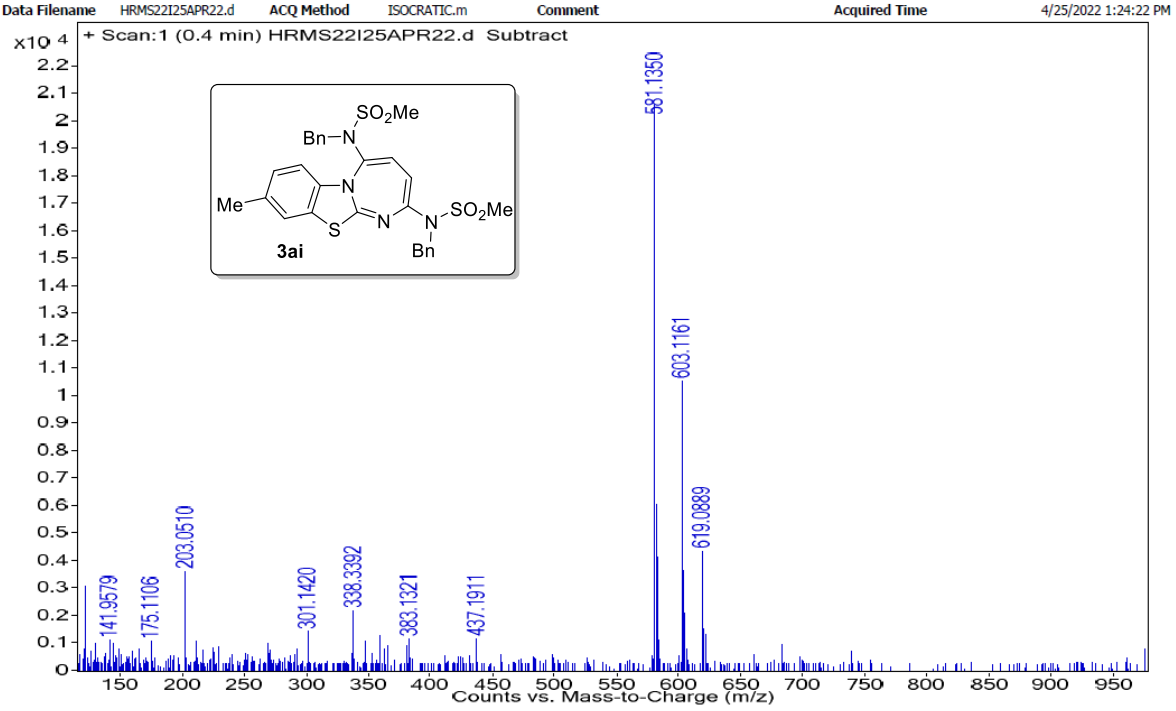
^{1}H NMR, $CDCl_3$, 400 MHz



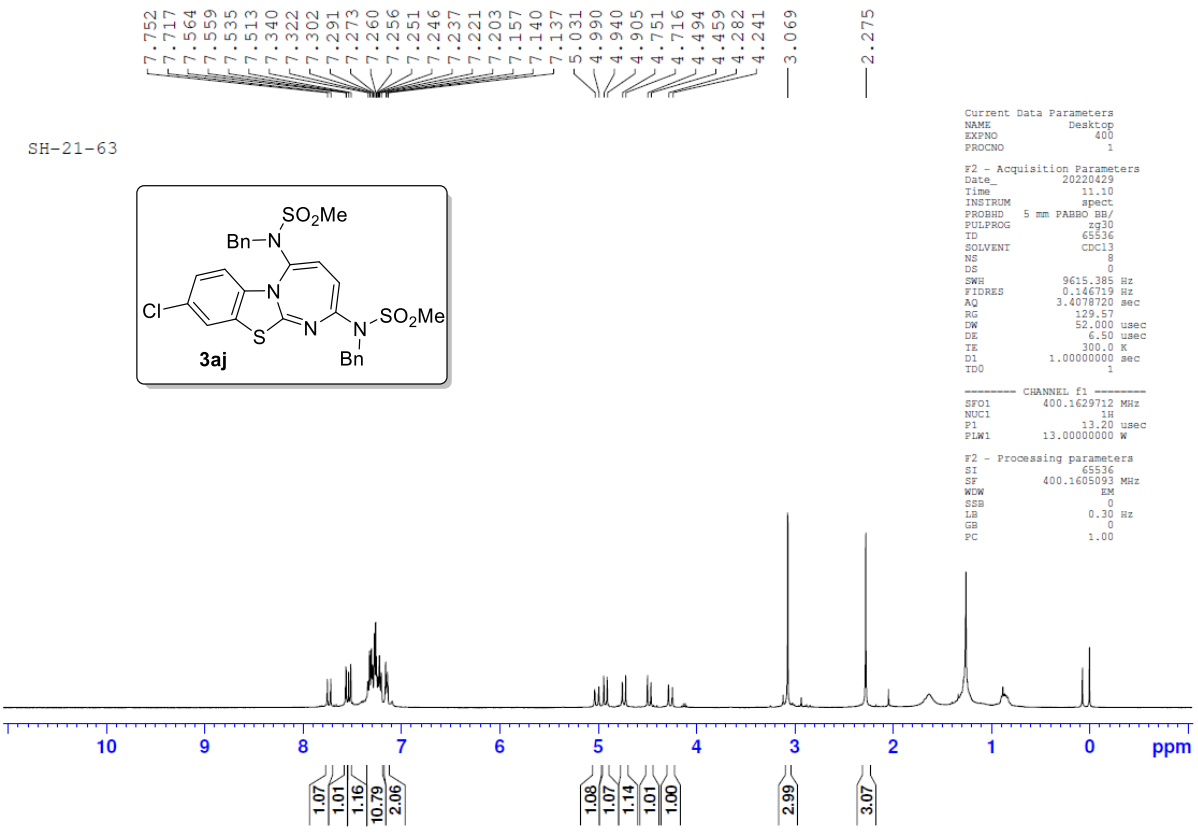
^{13}C NMR, $CDCl_3$, 125 MHz



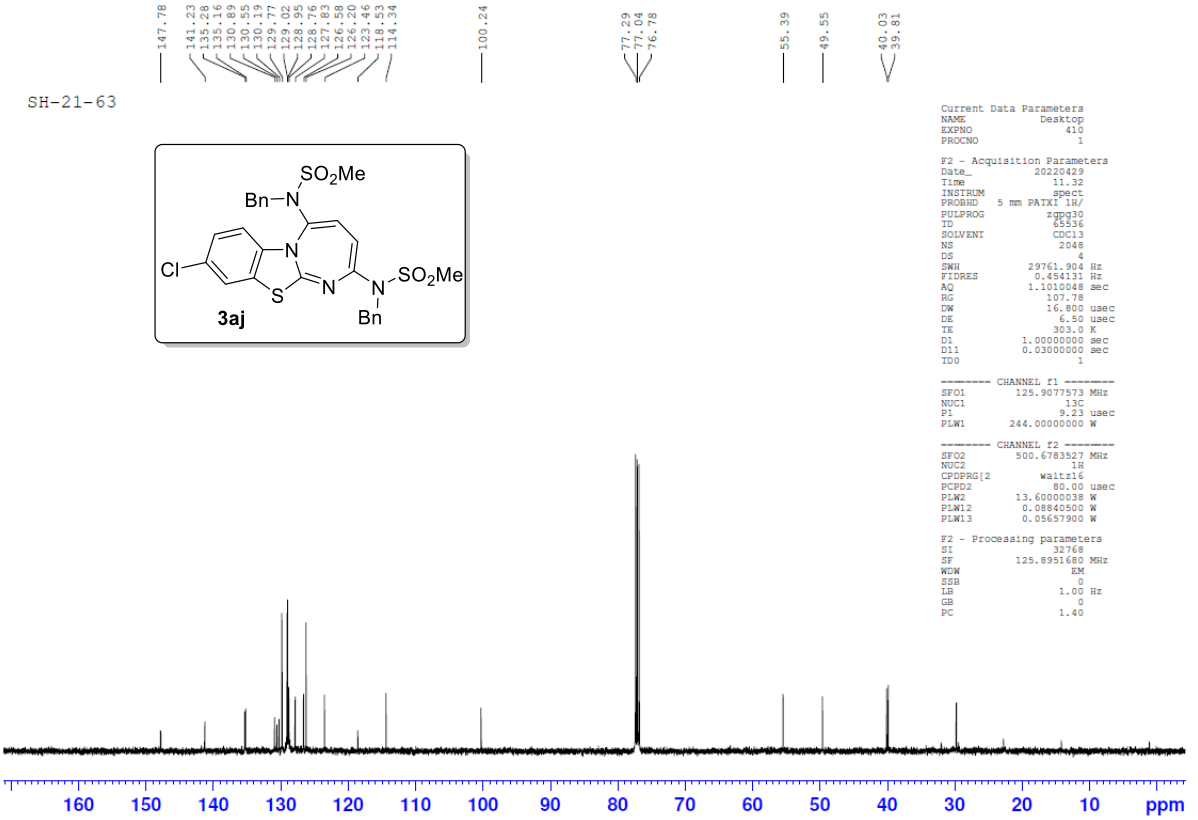
Sample Name	GR-22-22	**Position**	Vial 22	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	HRMS22I25APR22.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	4/25/2022 1:24:22 PM

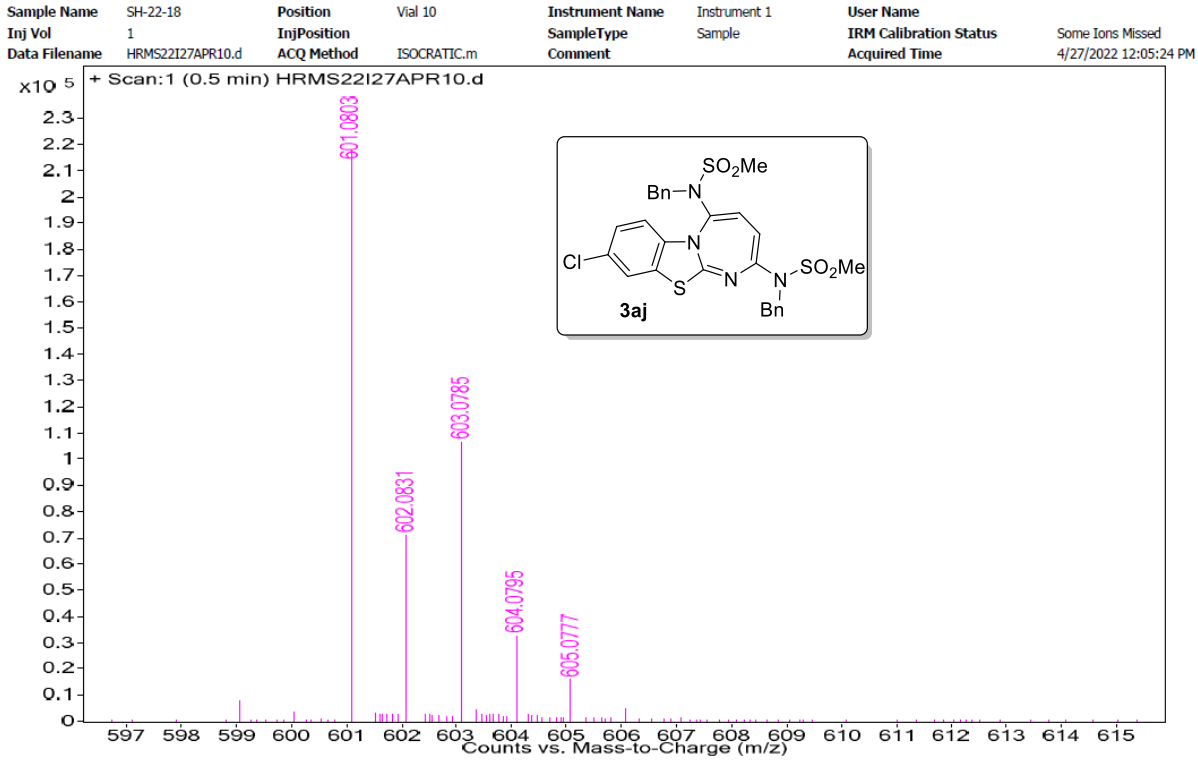


^{1}H NMR, $CDCl_3$, 400 MHz

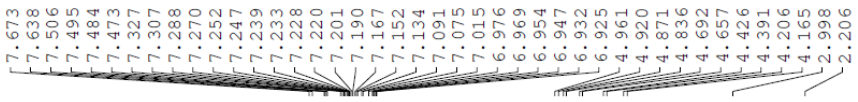


^{13}C NMR, $CDCl_3$, 125 MHz

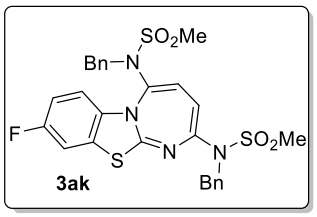


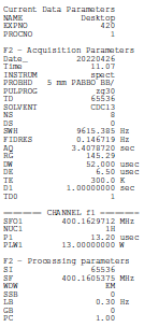


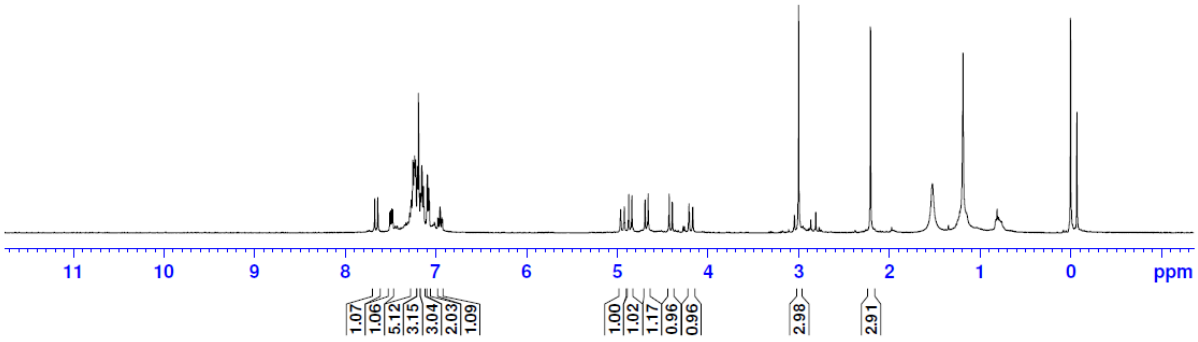
^{1}H NMR, $CDCl_3$, 400 MHz



SH-21-64

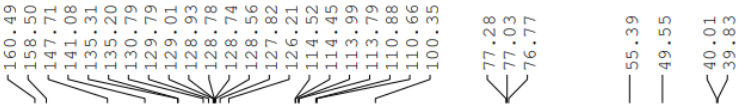


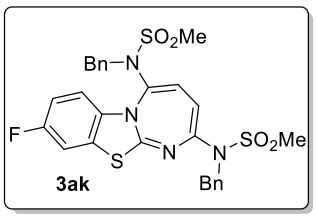


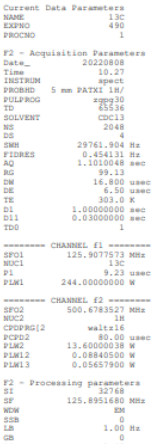


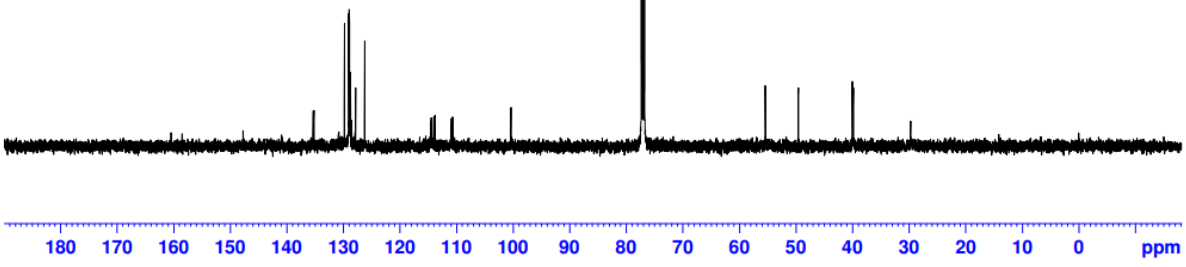
^{13}C NMR, $CDCl_3$, 125 MHz

SH-21-64

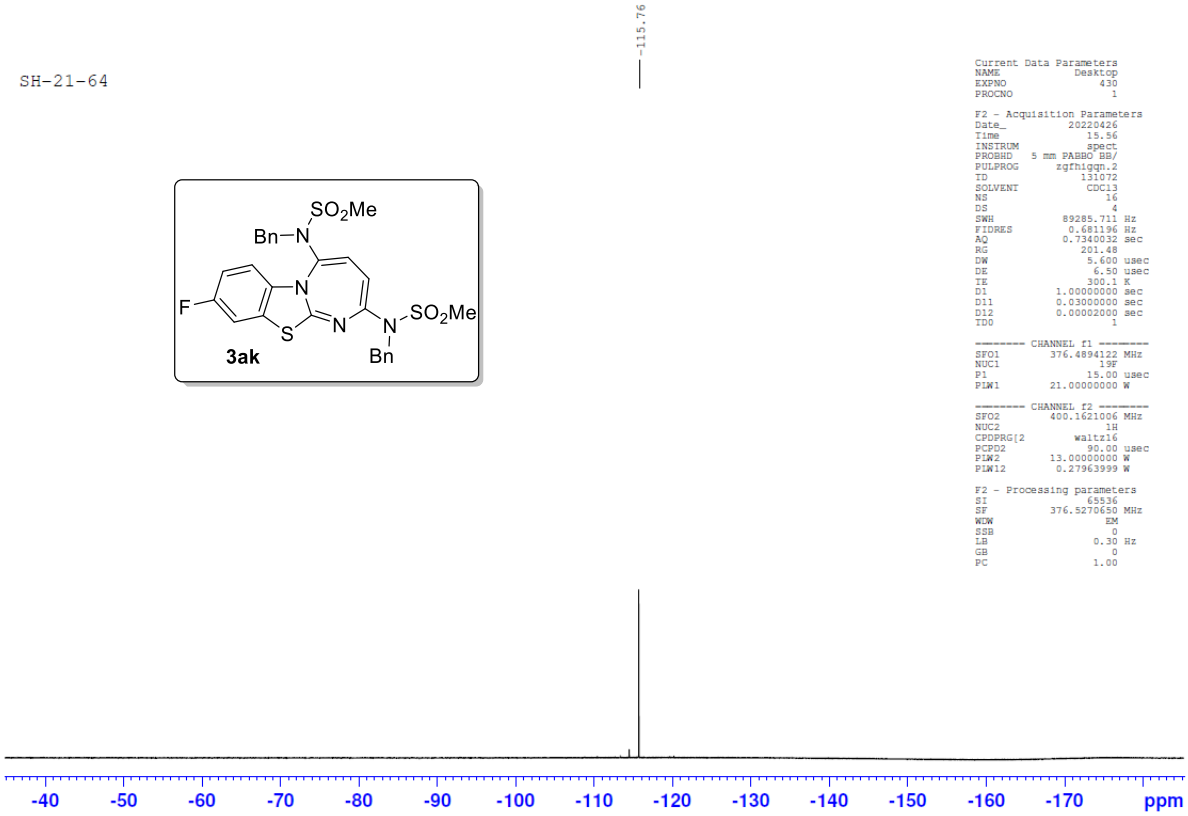


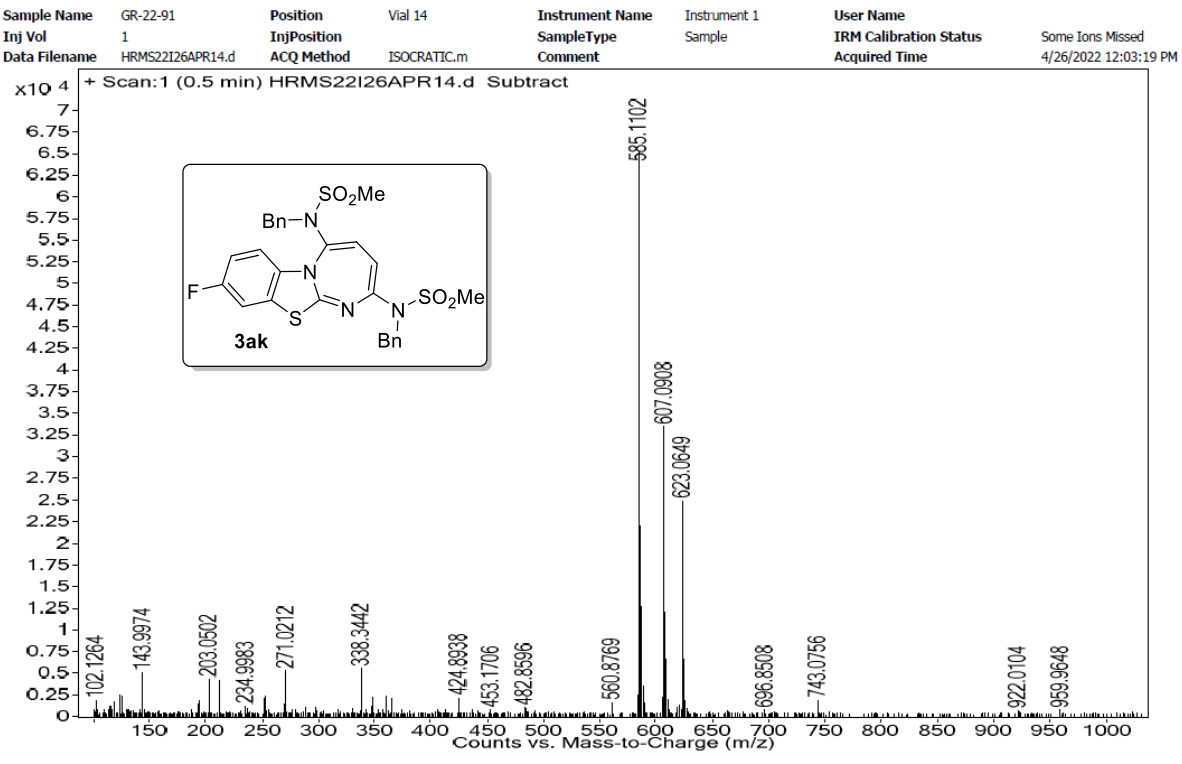






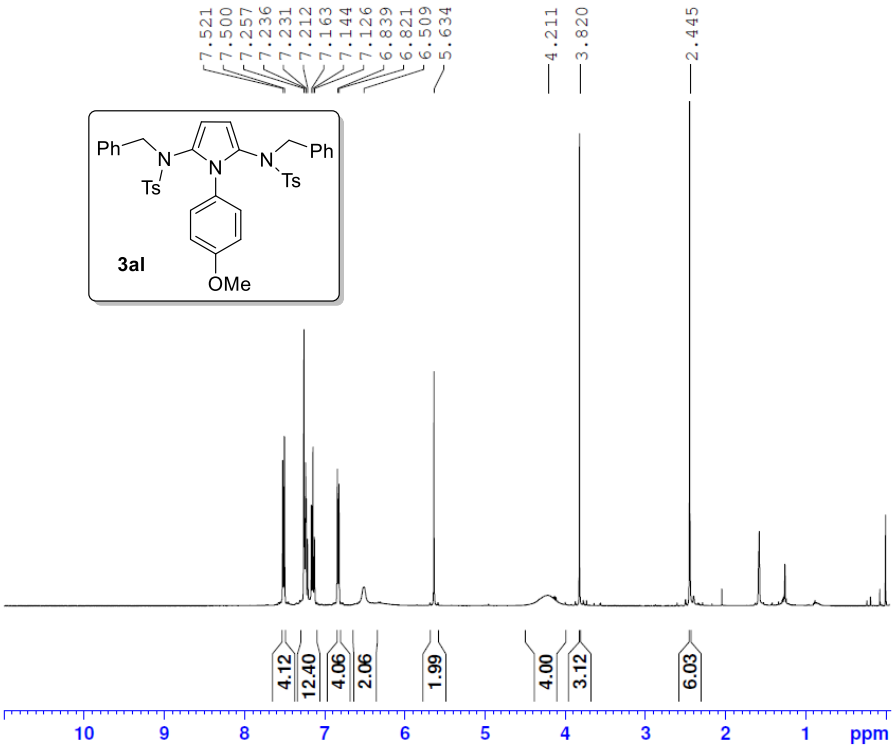
^{19}F NMR, $CDCl_3$, 376 MHz





gr-21-61

^{1}H NMR, $CDCl_3$, 400 MHz




```
Current Data Parameters
NAME            GR-21-61
EXPNO                560
PROCNO                 1

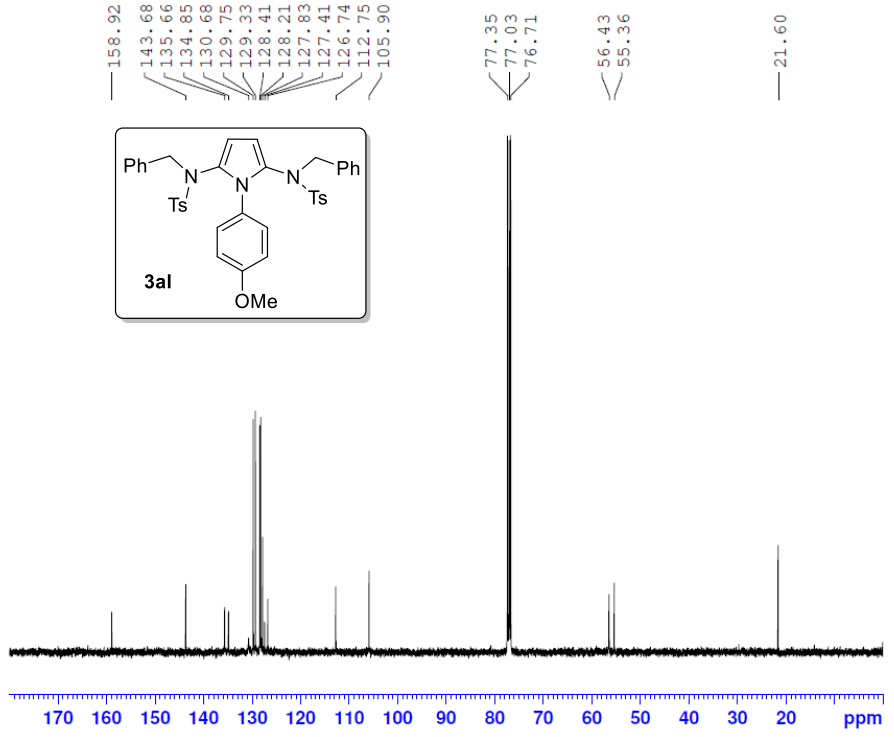
F2 - Acquisition Parameters
Date_          20211123
Time             21.11
INSTRUM          spect
PROBHD   5 mm PABBO BB/
PULPROG          zg30
TD              65536
SOLVENT          CDC13
NS                   8
DS                   0
SWH           9615.385 Hz
FIDRES        0.146719 Hz
AQ            3.4078720 sec
RG               114.26
DW               52.000 usec
DE                6.50 usec
TE               300.0 K
D1            1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1         400.1629712 MHz
NUC1                1H
P1                13.20 usec
PLW1         13.00000000 W

F2 - Processing parameters
SI                65536
SF           400.1605106 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

Q-IV-01-2

^{13}C NMR, $CDCl_3$, 101 MHz




```
Current Data Parameters
NAME            GR-21-61
EXPNO                570
PROCNO                 1

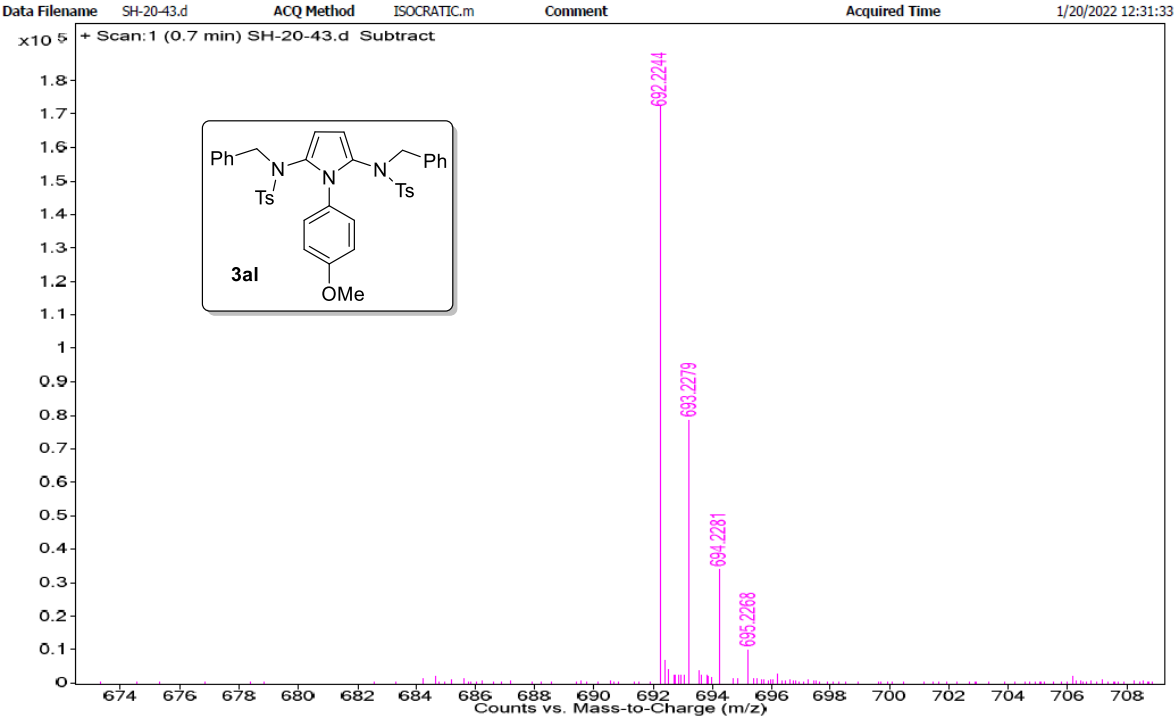
F2 - Acquisition Parameters
Date_          20211123
Time             21.46
INSTRUM          spect
PROBHD   5 mm PABBO BB/
PULPROG        zgpg30
TD              65536
SOLVENT          CDC13
NS                1024
DS                   0
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631488 sec
RG               201.48
DW               20.800 usec
DE                6.50 usec
TE               300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1         100.6304993 MHz
NUC1                13C
P1                 9.90 usec
PLW1         53.00000000 W

======== CHANNEL f2 ========
SFO2         400.1621006 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         13.00000000 W
PLW12         0.27963999 W
PLW13         0.22651000 W

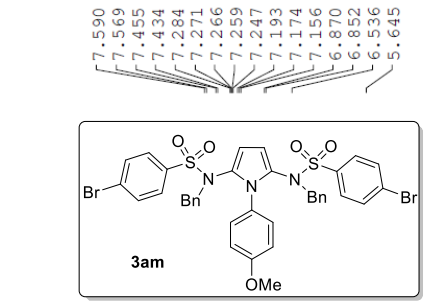
F2 - Processing parameters
SI                32768
SF           100.6204380 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

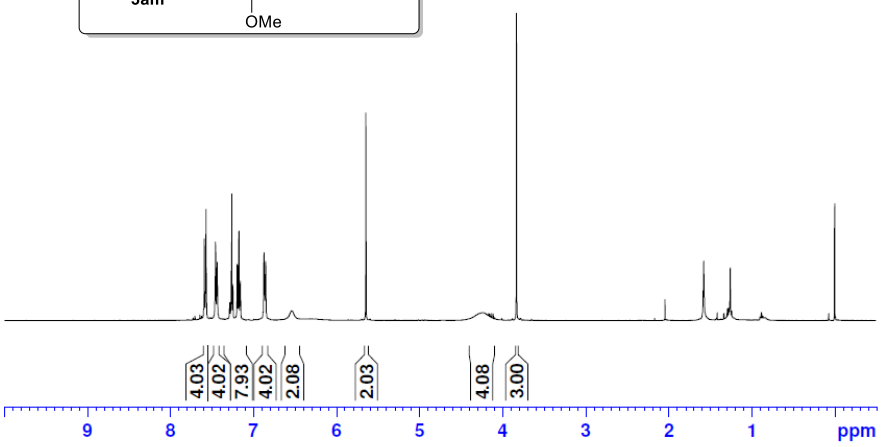
Sample Name	HRMS22I20JAN23	**Position**	Vial 23	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	SH-20-43.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	1/20/2022 12:31:33 PM



GR-21-58

^{1}H NMR, $CDCl_3$, 400 MHz






```
Current Data Parameters
NAME                  GR-21-58
EXPNO                      320
PROCNO                       1

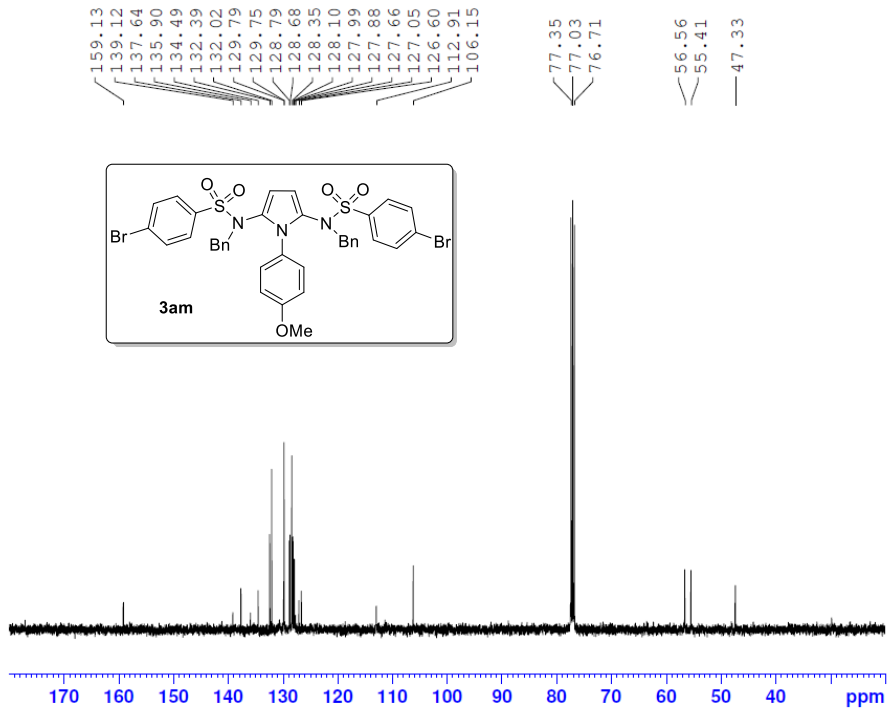
F2 - Acquisition Parameters
Date_                 20211125
Time                     12.26
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                      65536
SOLVENT                  CDC13
NS                           8
DS                           0
SWH                   9615.385 Hz
FIDRES                0.146719 Hz
AQ                    3.4078720 sec
RG                       129.57
DW                       52.000 usec
DE                        6.50 usec
TE                       300.0 K
D1                    1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                400.1629712 MHz
NUC1                        1H
P1                       13.20 usec
PLW1                13.00000000 W

F2 - Processing parameters
SI                      65536
SF                  400.1605101 MHz
WDW                        EM
SSB                  0
LB                       0.30 Hz
GB                   0
PC                       1.00
```

^{13}C NMR, $CDCl_3$, 101 MHz

GR-21-58




```
Current Data Parameters
NAME                  GR-21-58
EXPNO                      330
PROCNO                       1

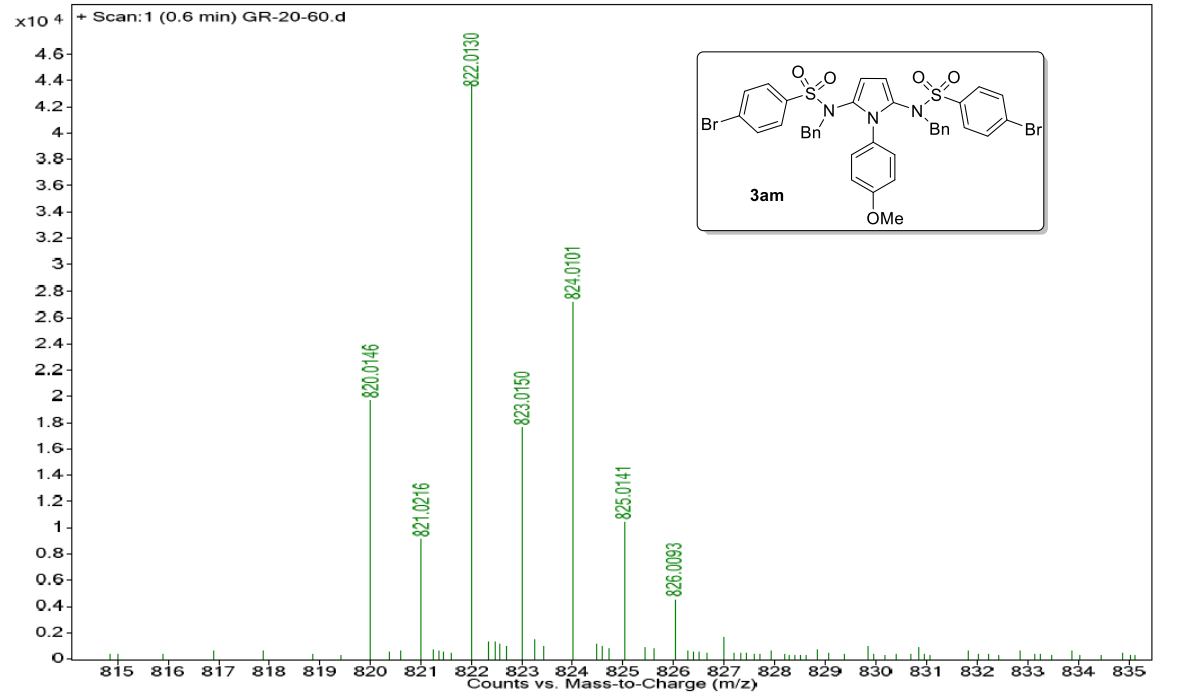
F2 - Acquisition Parameters
Date_                 20211125
Time                     15.18
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                         2048
DS                            4
SWH                  24038.461 Hz
FIDRES                0.366798 Hz
AQ                    1.3631488 sec
RG                       201.48
DW                       20.800 usec
DE                        6.50 usec
TE                       300.0 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                100.6304993 MHz
NUC1                        13C
P1                         9.90 usec
PLW1                53.00000000 W

======== CHANNEL f2 ========
SFO2                400.1621006 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                     90.00 usec
PLW2                13.00000000 W
PLW12                0.27963999 W
PLW13                0.22651000 W

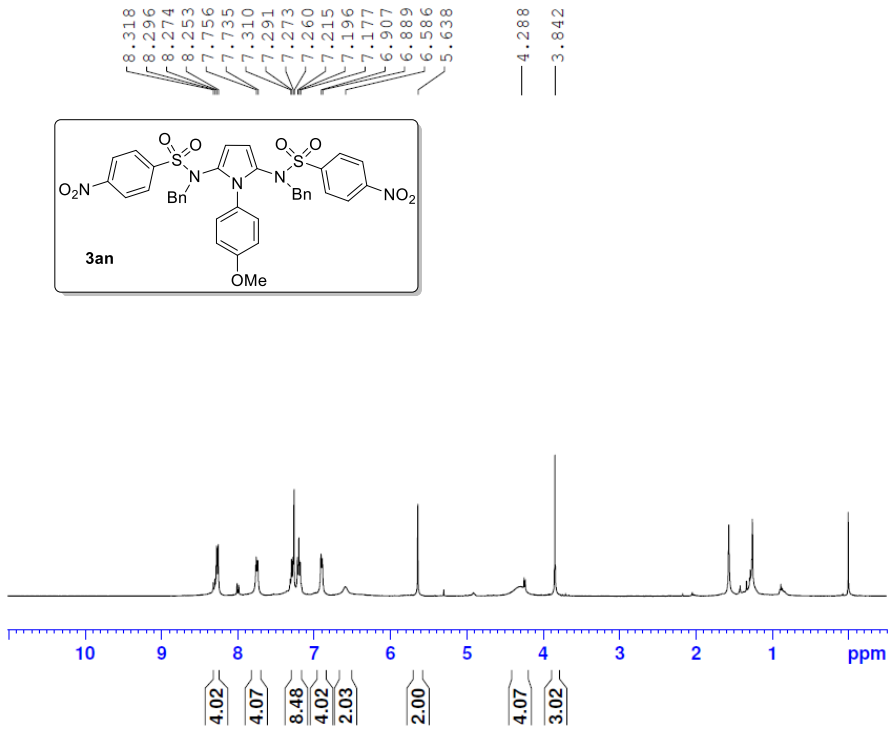
F2 - Processing parameters
SI                        32768
SF                  100.6204380 MHz
WDW                          EM
SSB                 0
LB                        1.00 Hz
GB                  0
PC                        1.40
```

Sample Name	HRMS22I21JAN12	**Position**	Vial 12	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	GR-20-60.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	1/21/2022 11:58:10 AM



sh 20 59

^{1}H NMR, $CDCl_3$, 400 MHz




```
Current Data Parameters
NAME                 GR-21-59
EXPNO                     540
PROCNO                      1

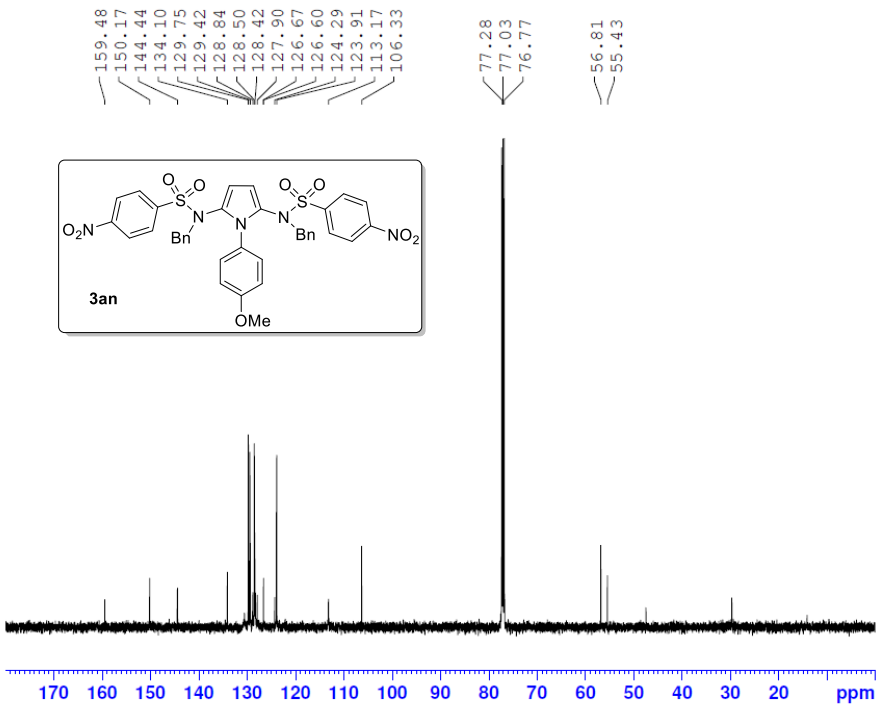
F2 - Acquisition Parameters
Date_               20211208
Time                    7.39
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                         8
DS                         0
SWH                 9615.385 Hz
FIDRES              0.146719 Hz
AQ                  3.4078720 sec
RG                     145.29
DW                     52.000 usec
DE                      6.50 usec
TE                     300.0 K
D1                  1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1               400.1629712 MHz
NUC1                       1H
P1                      13.20 usec
PLW1               13.00000000 W

F2 - Processing parameters
SI                     65536
SF                 400.1605095 MHz
WDW                       EM
SSB          0
LB                      0.30 Hz
GB           0
PC                      1.00
```

GR-21-59

^{13}C NMR, $CDCl_3$, 125 MHz




```
Current Data Parameters
NAME                 GR-21-59
EXPNO                     550
PROCNO                      1

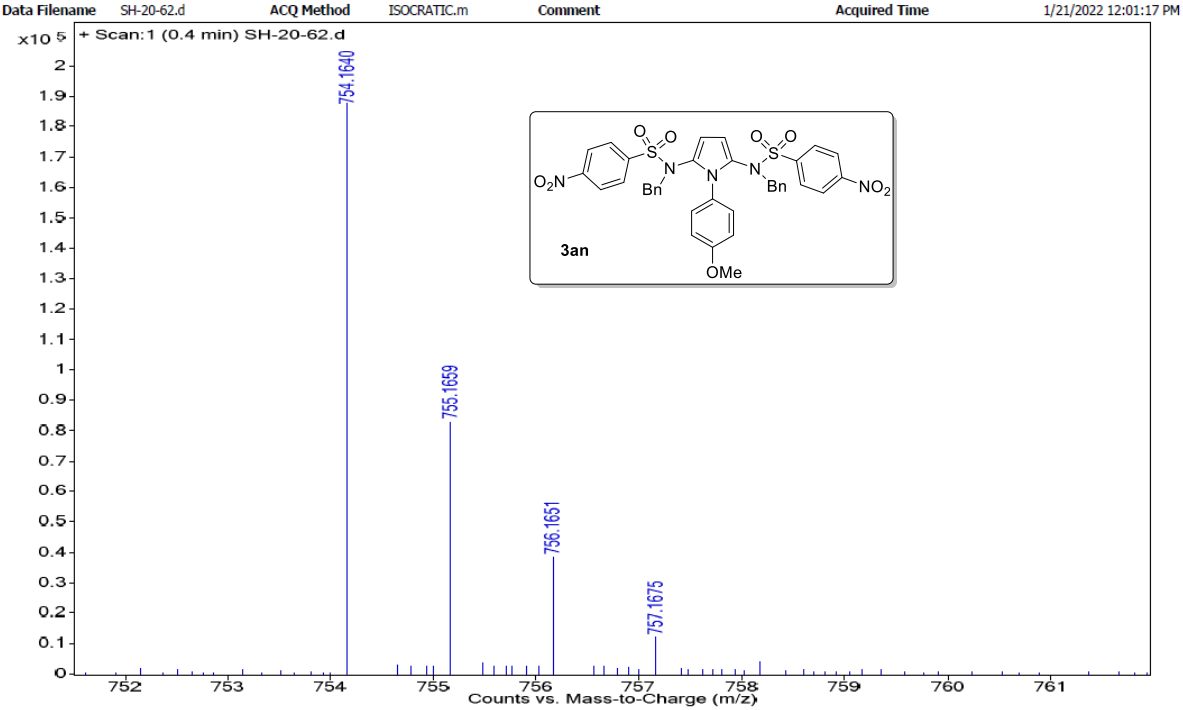
F2 - Acquisition Parameters
Date_               20211207
Time                   16.34
INSTRUM                spect
PROBHD    5 mm PATXI 1H/
PULPROG             zgpg30
TD                     65536
SOLVENT                CDCl3
NS                      2048
DS                         4
SWH                29761.904 Hz
FIDRES              0.454131 Hz
AQ                  1.1010048 sec
RG                     56.22
DW                     16.800 usec
DE                      6.50 usec
TE                     303.0 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1               125.9077573 MHz
NUC1                       13C
P1                       9.23 usec
PLW1               244.00000000 W

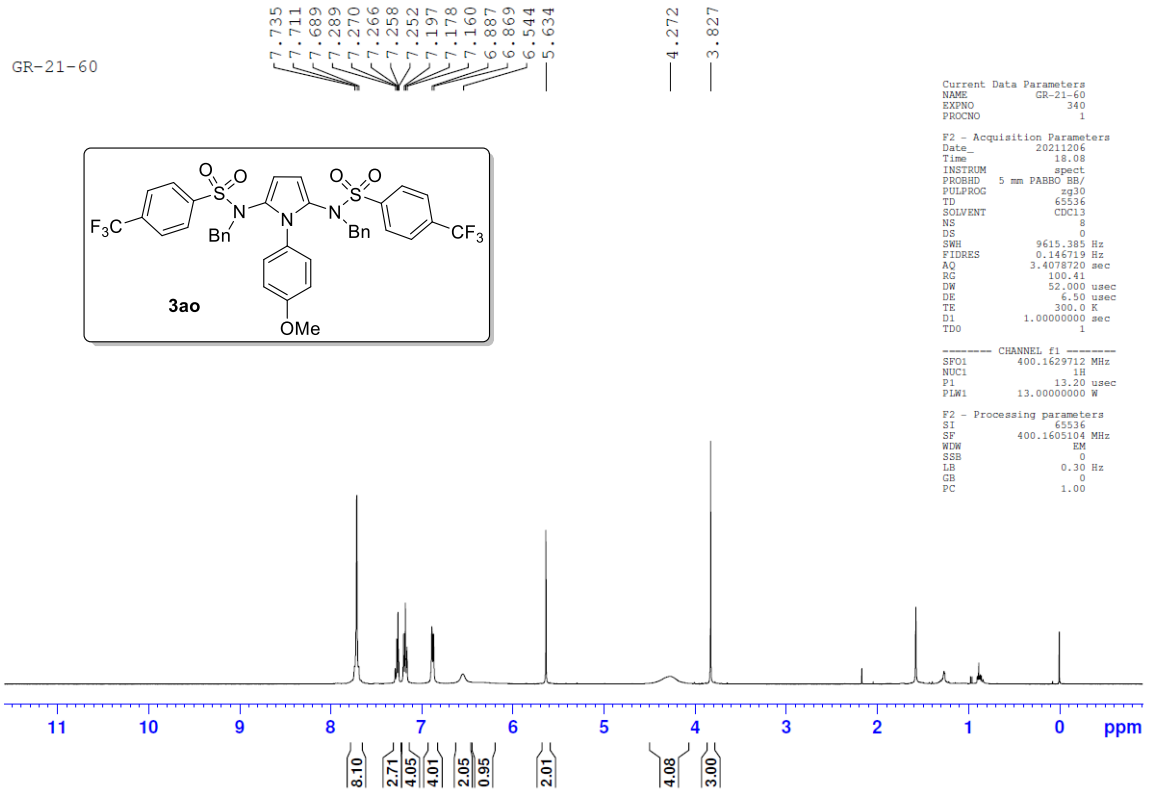
======== CHANNEL f2 ========
SFO2               500.6783527 MHz
NUC2                       1H
CPDPRG[2             waltz16
PCPD2                   80.00 usec
PLW2               13.60000038 W
PLW12               0.08840500 W
PLW13               0.05657900 W

F2 - Processing parameters
SI                     32768
SF                 125.8951680 MHz
WDW                       EM
SSB          0
LB                      1.00 Hz
GB           0
PC                      1.40
```

Sample Name	HRMS22I21JAN13	**Position**	Vial 13	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	SH-20-62.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	1/21/2022 12:01:17 PM



^{1}H NMR, $CDCl_3$, 400 MHz



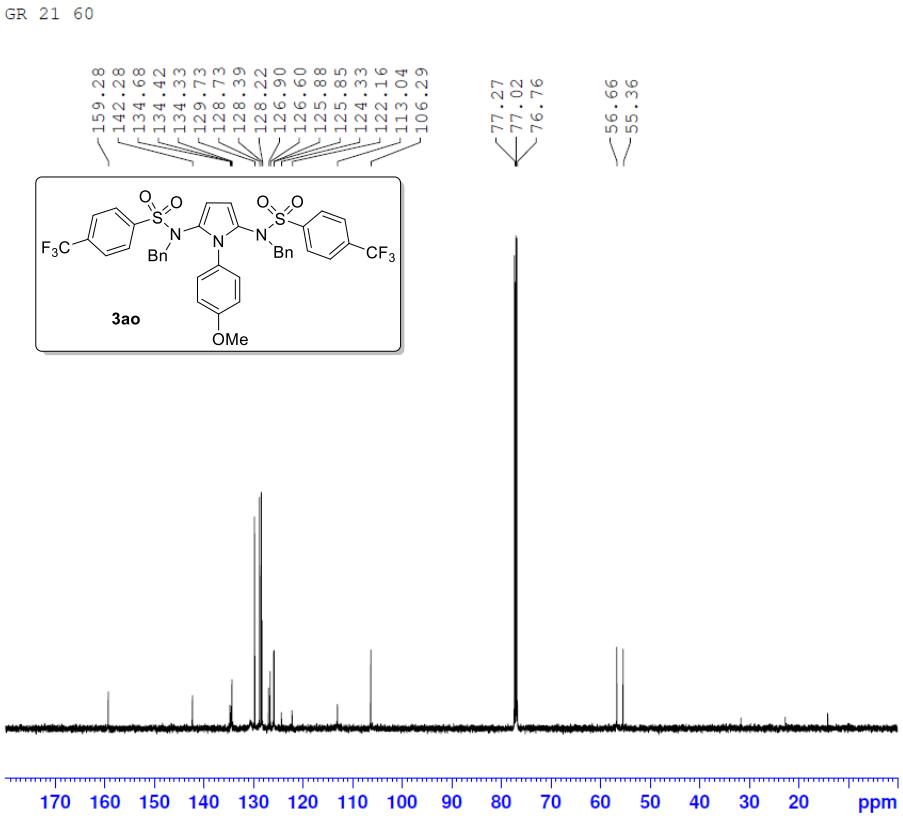
Current Data Parameters
NAME GR-21-60
EXPNO 340
PROCNO 1

F2 - Acquisition Parameters
Date_ 20211206
Time 18.08
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 9615.385 Hz
FIDRES 0.146719 Hz
AQ 3.4078720 sec
RG 100.41
DW 52.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1629712 MHz
NUC1 1H
P1 13.20 usec
PLW1 13.00000000 W

F2 - Processing parameters
SI 65536
SF 400.1605104 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

^{13}C NMR, $CDCl_3$, 125 MHz




```
Current Data Parameters
NAME                  GR-21-60
EXPNO                      370
PROCNO                       1

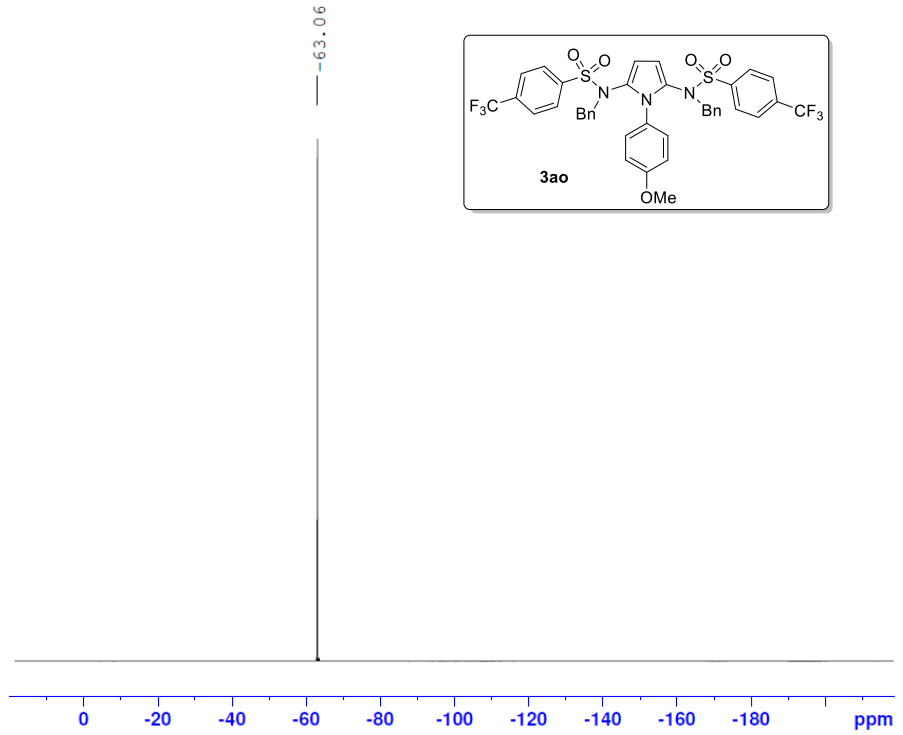
F2 - Acquisition Parameters
Date_                 20211206
Time                     14.58
INSTRUM                  spect
PROBHD    5 mm PATXI 1H/
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                       2048
DS                          4
SWH                 29761.904 Hz
FIDRES                0.454131 Hz
AQ                    1.1010048 sec
RG                        56.22
DW                       16.800 usec
DE                        6.50 usec
TE                       303.0 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1               125.9077573 MHz
NUC1                        13C
P1                         9.23 usec
PLW1              244.00000000 W

======== CHANNEL f2 ========
SFO2               500.6783527 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                     80.00 usec
PLW2               13.60000038 W
PLW12               0.08840500 W
PLW13               0.05657900 W

F2 - Processing parameters
SI                          32768
SF                 125.8951680 MHz
WDW                            EM
SSB                             0
LB                           1.00 Hz
GB                              0
PC                           1.40
```

^{19}F NMR, $CDCl_3$, 376 MHz




```
Current Data Parameters
NAME                  GR-21-60
EXPNO                      350
PROCNO                       1

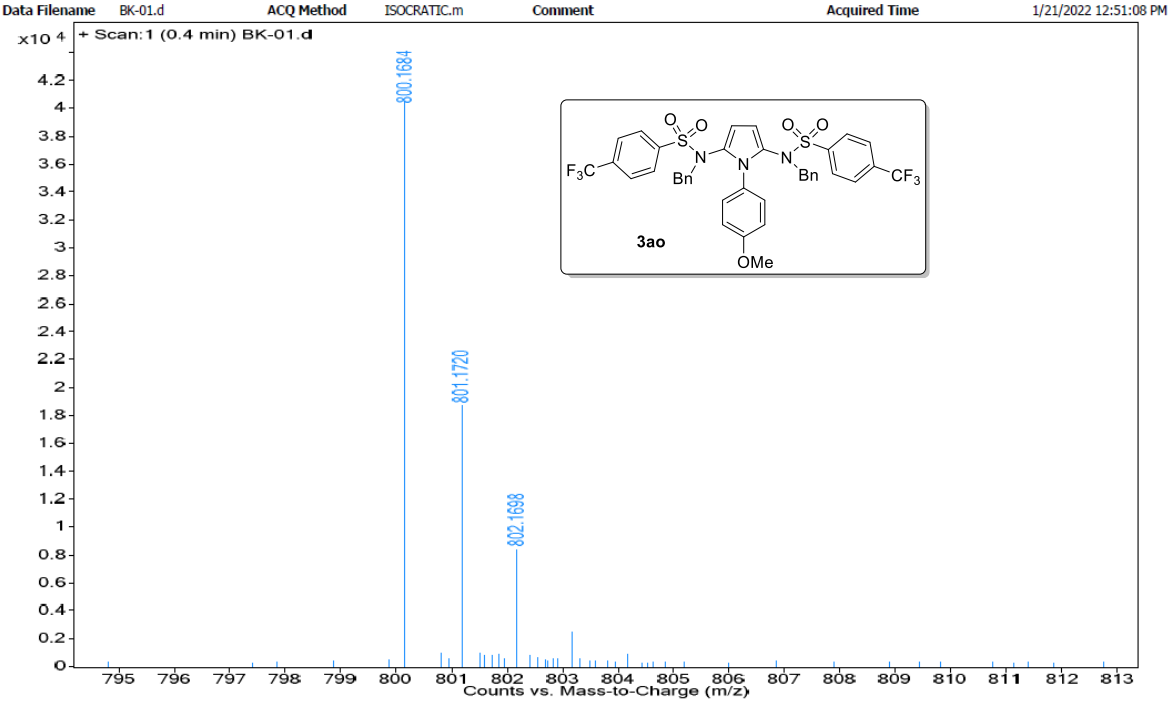
F2 - Acquisition Parameters
Date_                 20211206
Time                     18.10
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG             zgfhigqn.2
TD                      131072
SOLVENT                 CDCl3
NS                          16
DS                           4
SWH                 89285.711 Hz
FIDRES                0.681196 Hz
AQ                    0.7340032 sec
RG                       201.48
DW                        5.600 usec
DE                        6.50 usec
TE                       300.0 K
D1                  1.00000000 sec
D11                 0.03000000 sec
D12                 0.00002000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1               376.4894122 MHz
NUC1                        19F
P1                        15.00 usec
PLW1              21.00000000 W

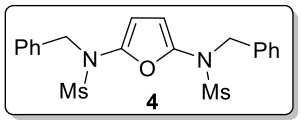
======== CHANNEL f2 ========
SFO2               400.1621006 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                     90.00 usec
PLW2               13.00000000 W
PLW12               0.27963999 W

F2 - Processing parameters
SI                          65536
SF                 376.5270650 MHz
WDW                            EM
SSB                 0
LB                           0.30 Hz
GB                  0
PC                           1.00
```

Sample Name	HRMS22I21JAN29	**Position**	Vial 29	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	BK-01.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	1/21/2022 12:51:08 PM



^{1}H NMR, $CDCl_3$, 400 MHz

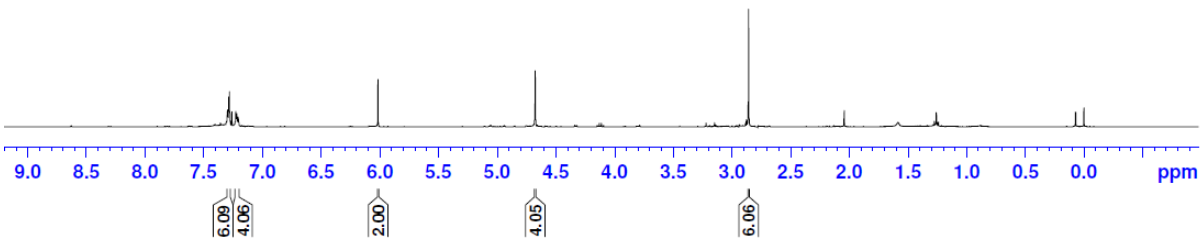



```
Current Data Parameters
NAME                  Desktop
EXPNO                     440
PROCNO                      1

F2 - Acquisition Parameters
Date_                20220104
Time                    12.07
INSTRUM                 spect
PROBHD    5 mm PABBO BB/
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                          8
DS                          0
SWH                 9615.385 Hz
FIDRES              0.146719 Hz
AQ                  3.4078720 sec
RG                     129.57
DW                     52.000 usec
DE                      6.50 usec
TE                     300.0 K
D1                  1.00000000 sec
TD0                         1

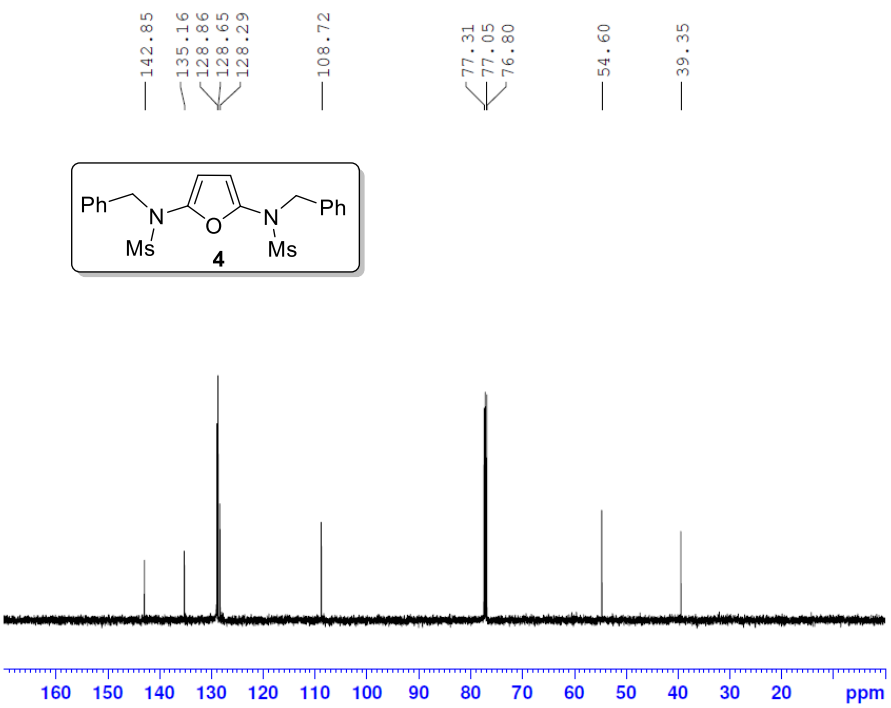
======== CHANNEL f1 ========
SFO1               400.1629712 MHz
NUC1                       1H
P1                      13.20 usec
PLW1               13.00000000 W

F2 - Processing parameters
SI                         65536
SF                 400.1605097 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```



SH-21-09

^{13}C NMR, $CDCl_3$, 125 MHz




```
Current Data Parameters
NAME                  SH-21-09
EXPNO                      520
PROCNO                       1

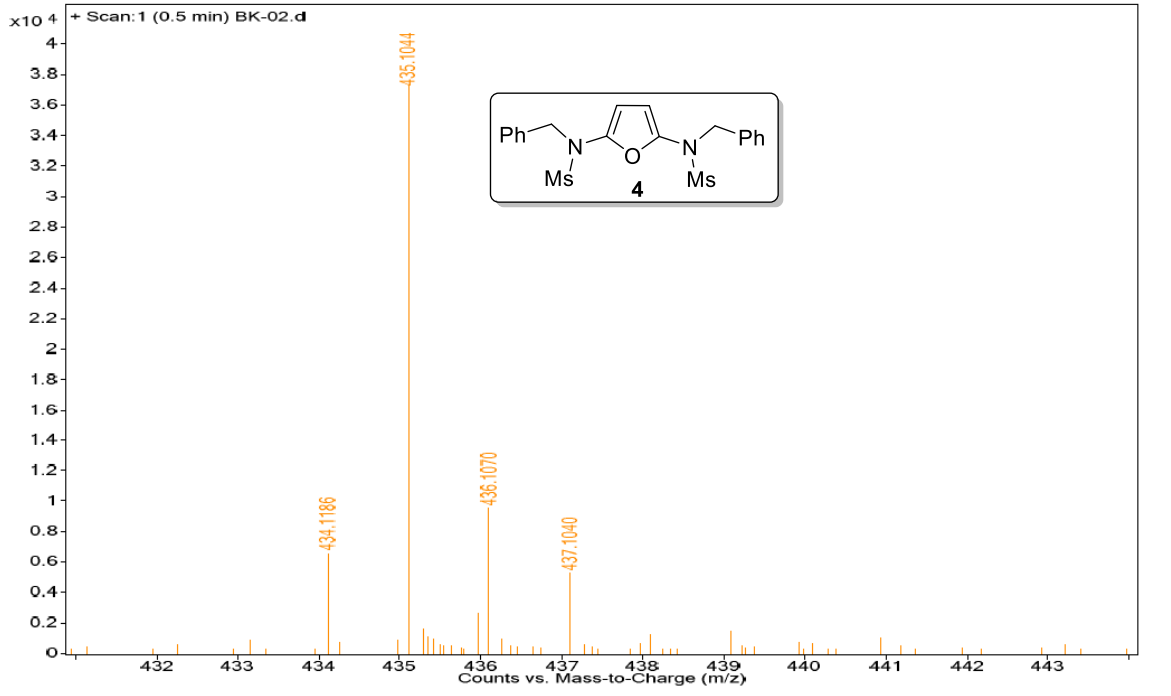
F2 - Acquisition Parameters
Date_                 20220105
Time                     13.26
INSTRUM                  spect
PROBHD    5 mm PATXI 1H/
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        2048
DS                           0
SWH                  29761.904 Hz
FIDRES                0.454131 Hz
AQ                    1.1010048 sec
RG                       107.78
DW                       16.800 usec
DE                        6.50 usec
TE                       300.0 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                125.9077573 MHz
NUC1                        13C
P1                         9.23 usec
PLW1               244.00000000 W

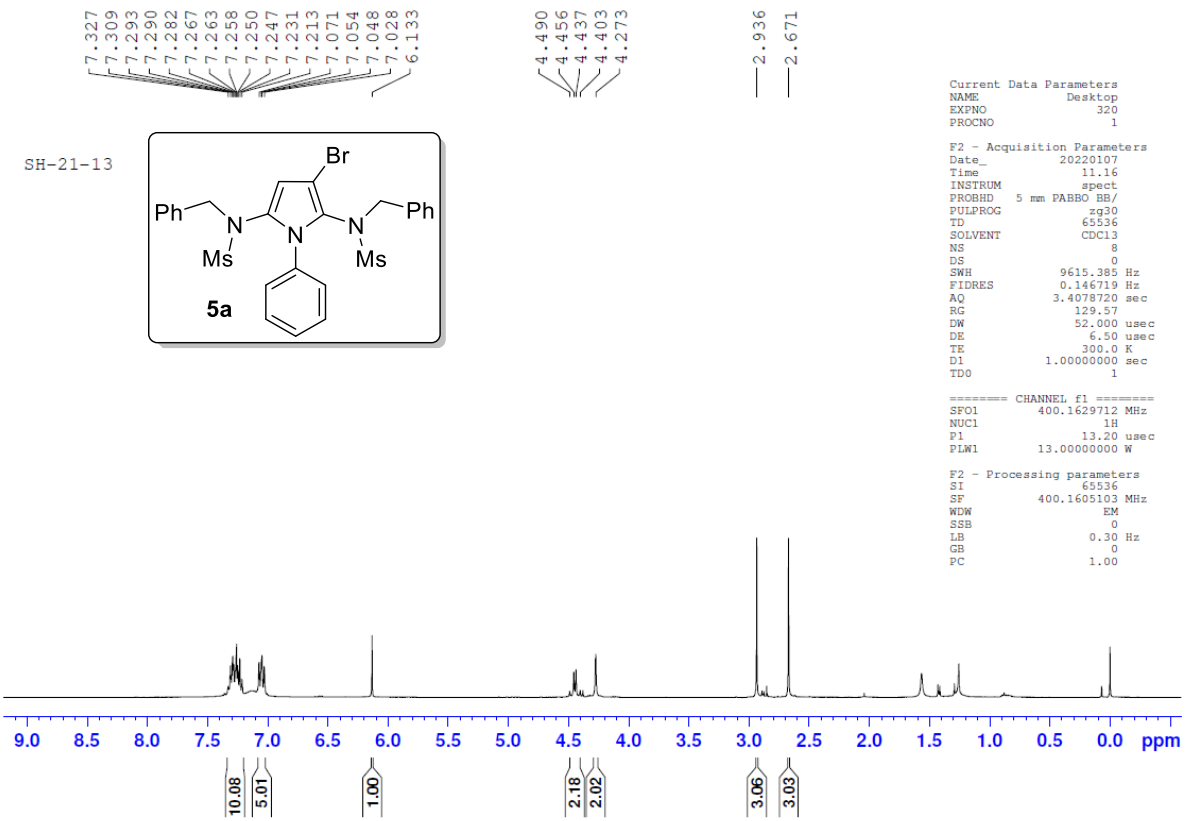
======== CHANNEL f2 ========
SFO2                500.6783527 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                     80.00 usec
PLW2               13.60000038 W
PLW12               0.08840500 W
PLW13               0.05657900 W

F2 - Processing parameters
SI                        32768
SF                  125.8951680 MHz
WDW                          EM
SSB                  0
LB                         1.00 Hz
GB                   0
PC                         1.40
```

Sample Name	HRMS22I21JAN30	**Position**	Vial 30	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	BK-02.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	1/21/2022 12:54:19 PM

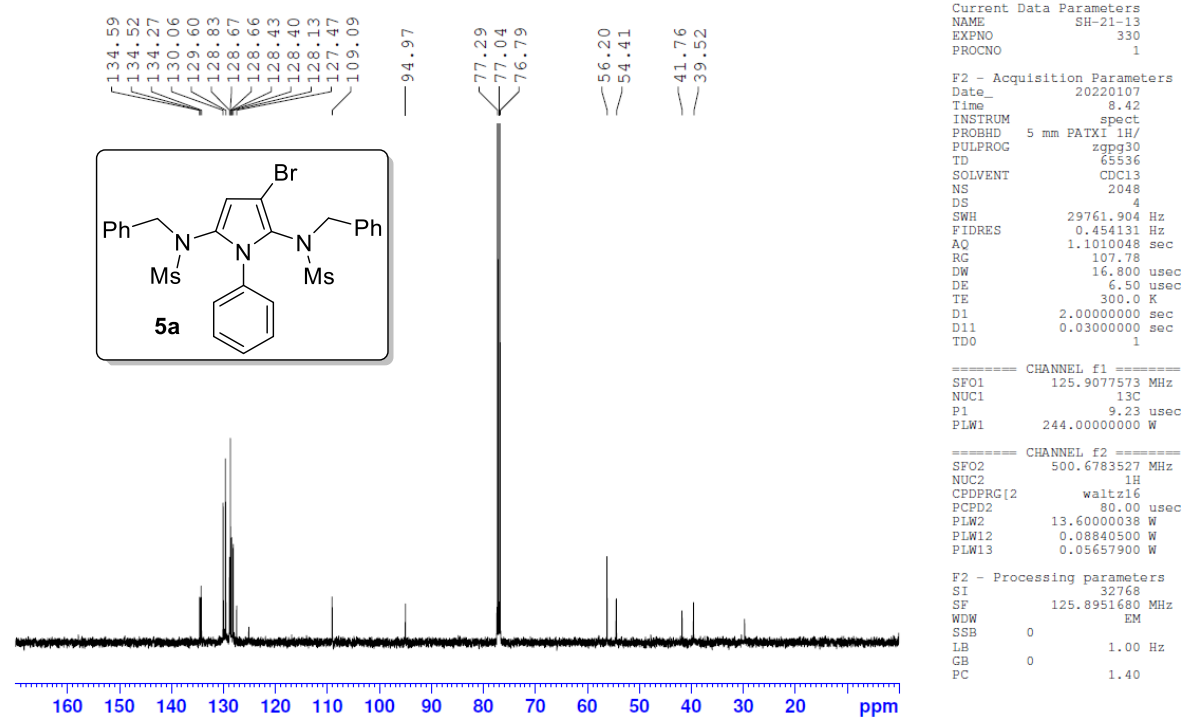


^{1}H NMR, $CDCl_3$, 400 MHz

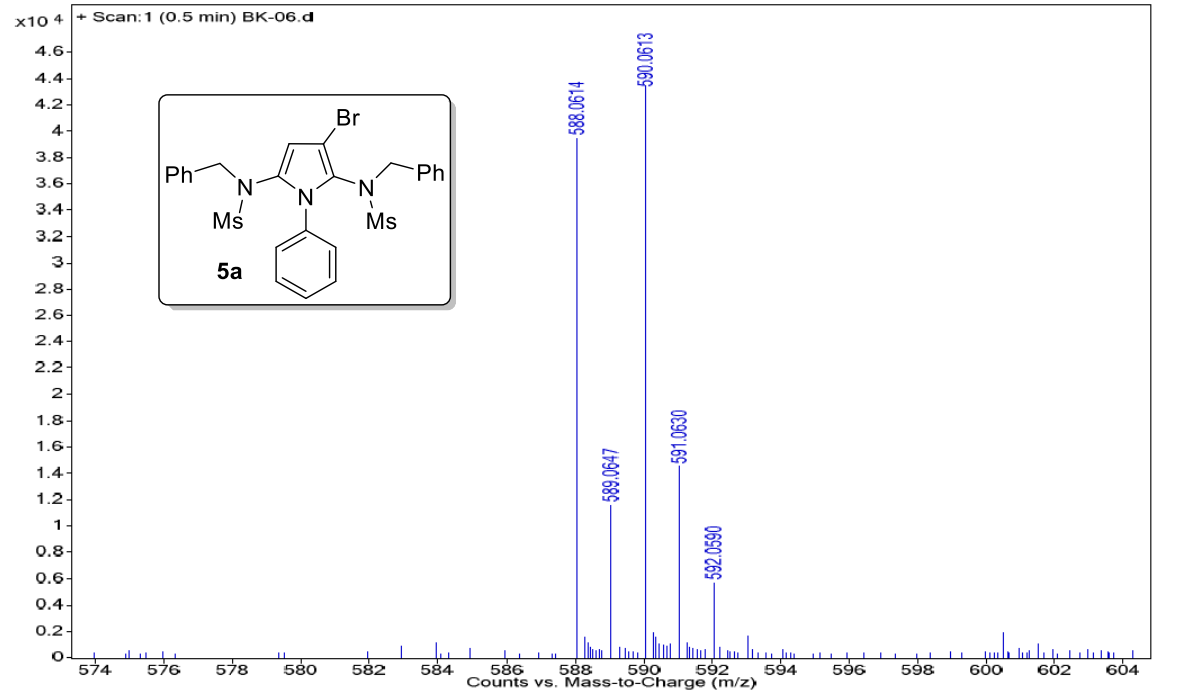


SH 21 13

^{13}C NMR, $CDCl_3$, 125 MHz

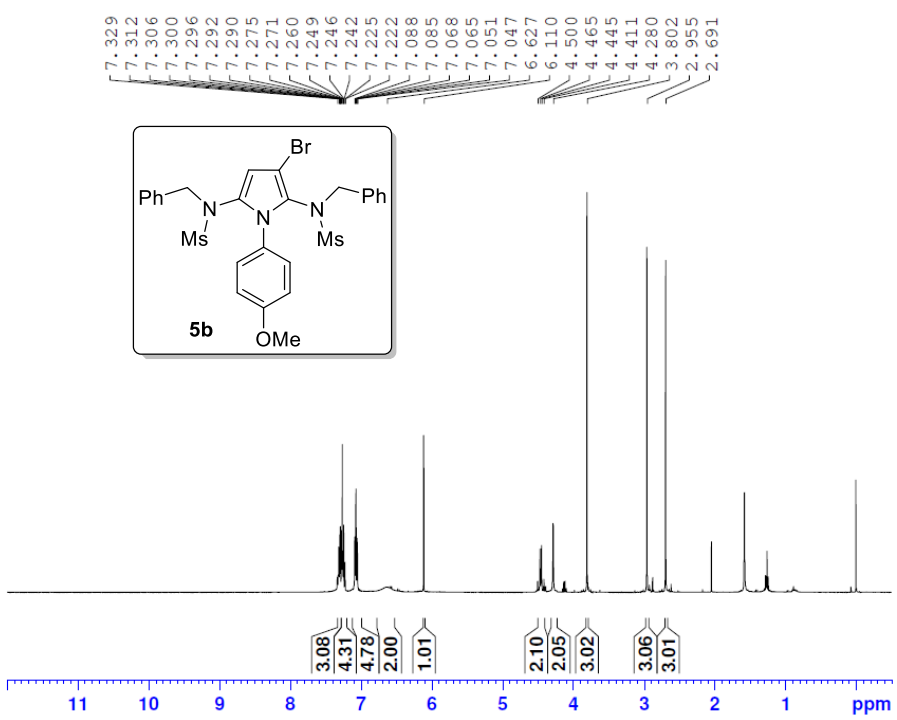


Sample Name	HRMS22I28JAN23	**Position**	Vial 23	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	BK-06.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	1/28/2022 12:45:37 PM



SH 21 18B

^{1}H NMR, $CDCl_3$, 400 MHz




```
Current Data Parameters
NAME                  SH-21-18
EXPNO                      330
PROCNO                       1

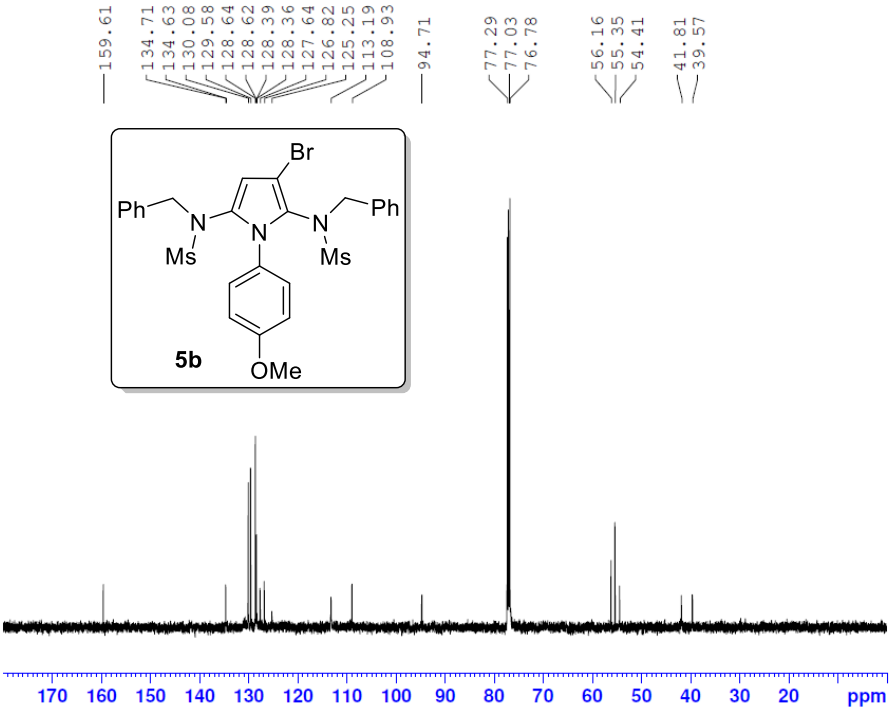
F2 - Acquisition Parameters
Date_                 20220124
Time                     12.45
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                          8
DS                          0
SWH                  9615.385 Hz
FIDRES               0.146719 Hz
AQ                   3.4078720 sec
RG                      129.57
DW                      52.000 usec
DE                       6.50 usec
TE                      300.0 K
D1                   1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                400.1629712 MHz
NUC1                        1H
P1                      13.20 usec
PLW1                13.00000000 W

F2 - Processing parameters
SI                          65536
SF                  400.1605095 MHz
WDW                            EM
SSB                  0
LB                       0.30 Hz
GB                   0
PC                       1.00
```

^{13}C NMR, $CDCl_3$, 125 MHz

SH 21 18B




```
Current Data Parameters
NAME                  SH-21-18
EXPNO                      410
PROCNO                       1

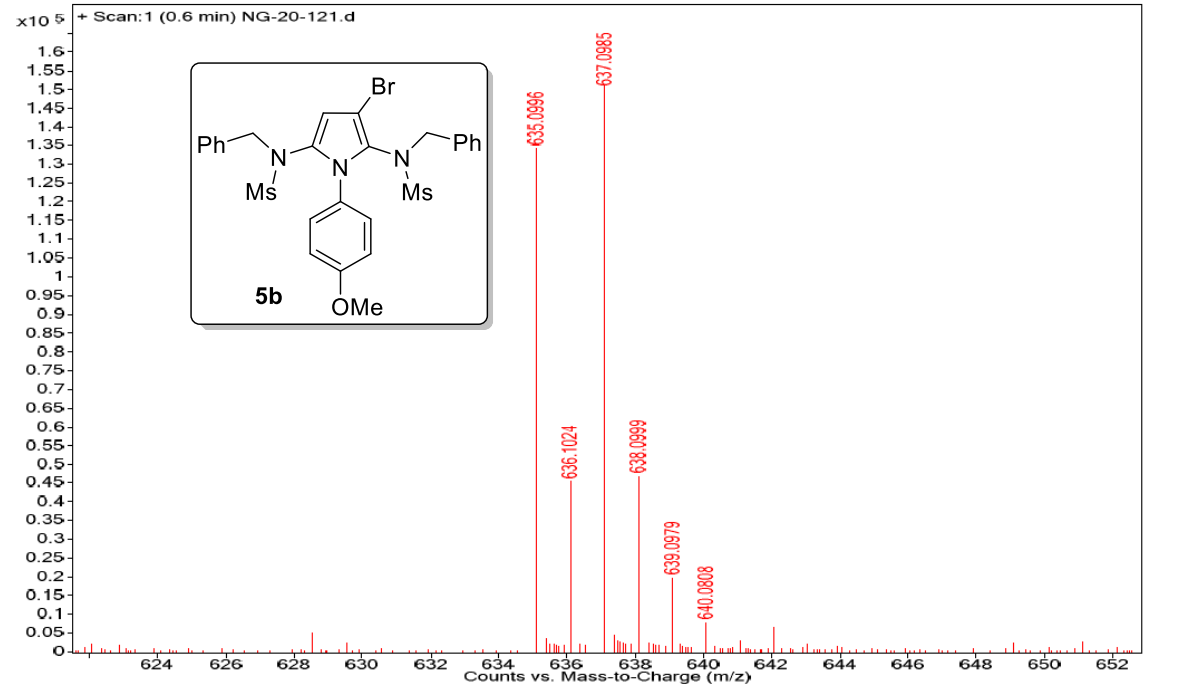
F2 - Acquisition Parameters
Date_                 20220127
Time                     13.23
INSTRUM                  spect
PROBHD    5 mm PATXI 1H/
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        2048
DS                           4
SWH                  29761.904 Hz
FIDRES               0.454131 Hz
AQ                   1.1010048 sec
RG                      107.78
DW                      16.800 usec
DE                       6.50 usec
TE                      303.0 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                125.9077573 MHz
NUC1                        13C
P1                         9.23 usec
PLW1               244.00000000 W

======== CHANNEL f2 ========
SFO2                500.6783527 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                    80.00 usec
PLW2                13.60000038 W
PLW12                0.08840500 W
PLW13                0.05657900 W

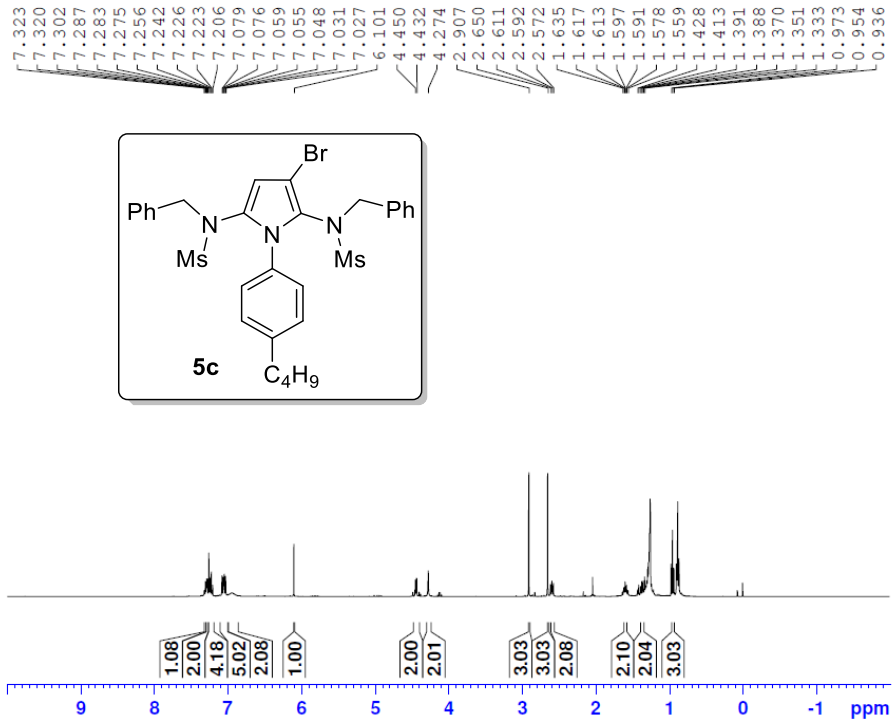
F2 - Processing parameters
SI                         32768
SF                  125.8951680 MHz
WDW                           EM
SSB                 0
LB                        1.00 Hz
GB                  0
PC                        1.40
```

Sample Name	HRMS22I25JAN24	**Position**	Vial 24	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	NG-20-121.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	1/25/2022 12:47:09 PM



SH-21-06

^{1}H NMR, $CDCl_3$, 400 MHz

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Current Data Parameters
NAME                 SH-21-06
EXPNO                     550
PROCNO                      1

F2 - Acquisition Parameters
Date_               20220104
Time                   18.31
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                         8
DS                         0
SWH                 9615.385 Hz
FIDRES              0.146719 Hz
AQ                  3.4078720 sec
RG                     58.68
DW                    52.000 usec
DE                     6.50 usec
TE                     300.0 K
D1                  1.00000000 sec
TD0                         1

======== CHANNEL f1 =======
SFO1              400.1629712 MHz
NUC1                       1H
P1                      13.20 usec
PLW1              13.00000000 W

F2 - Processing parameters
SI                         65536
SF                400.1605108 MHz
WDW                           EM
SSB          0
LB                      0.30 Hz
GB           0
PC                      1.00
```
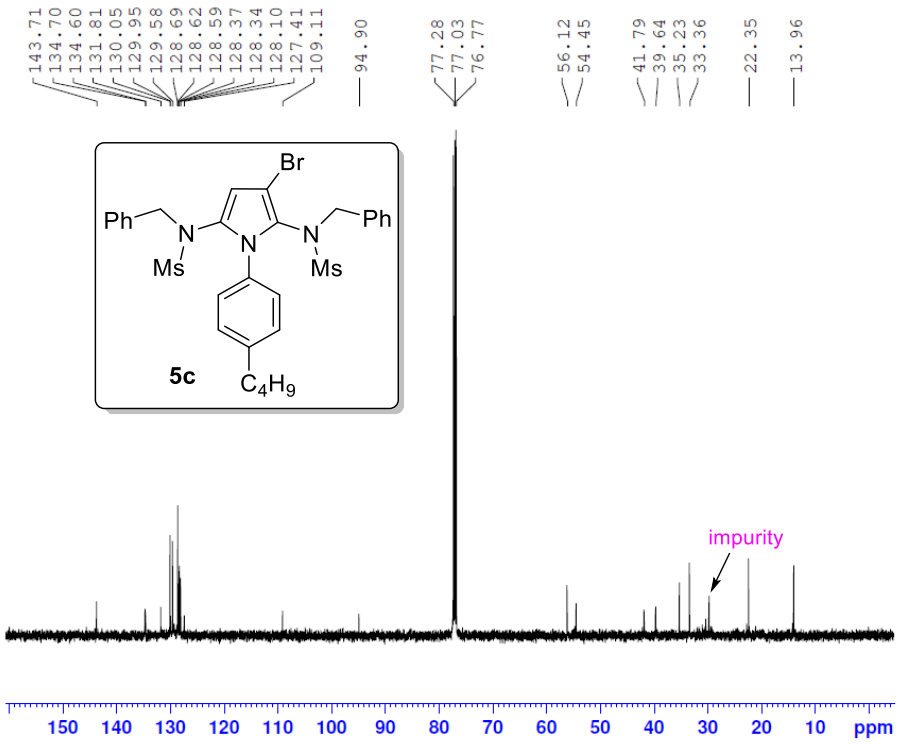
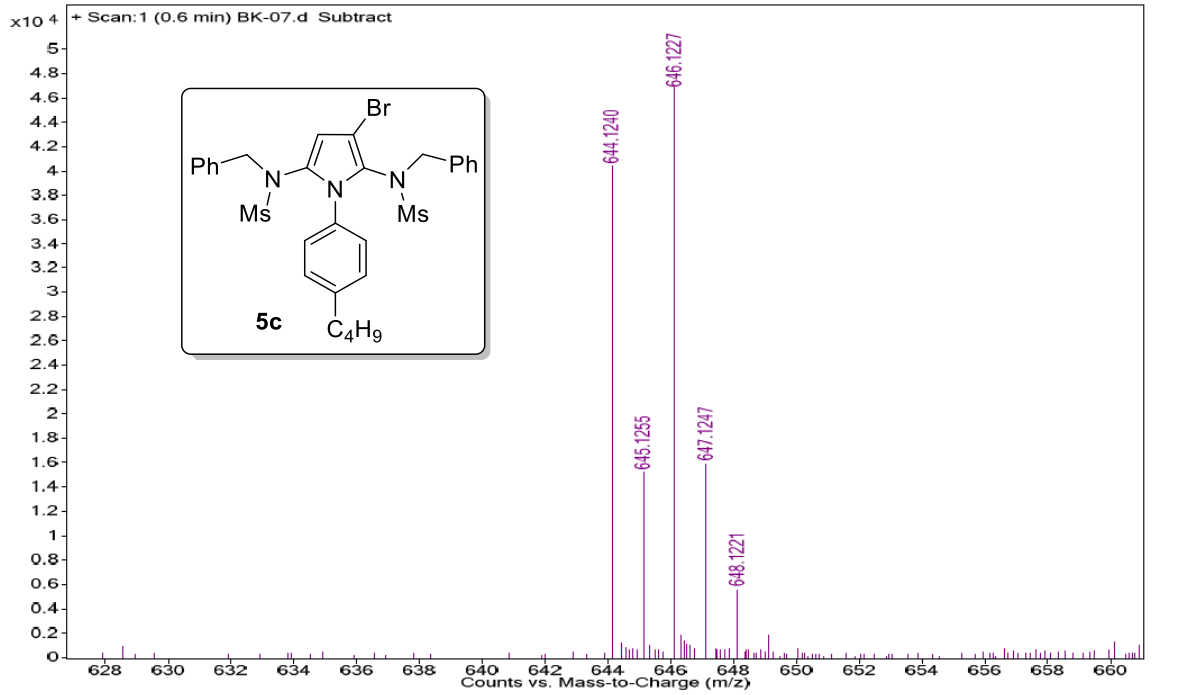

SH 21 06

^{13}C NMR, $CDCl_3$, 125 MHz



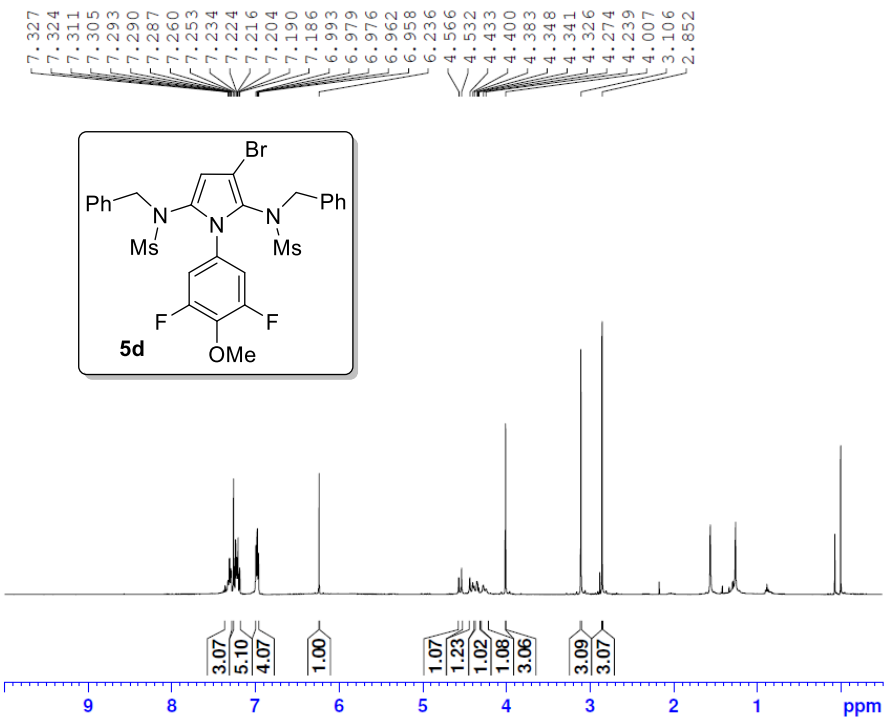

```
Current Data Parameters
NAME                 SH-21-06
EXPNO                     370
PROCNO                      1

F2 - Acquisition Parameters
Date_               20220322
Time                   21.49
INSTRUM                spect
PROBHD    5 mm PATXI 1H/
PULPROG             zgpg30
TD                     65536
SOLVENT                CDCl3
NS                      2048
DS                         4
SWH                29761.904 Hz
FIDRES              0.454131 Hz
AQ                  1.1010048 sec
RG                     107.78
DW                    16.800 usec
DE                     6.50 usec
TE                     300.2 K
D1                  1.00000000 sec
D11                 0.03000000 sec
TD0                         1

======== CHANNEL f1 =======
SFO1              125.9077573 MHz
NUC1                       13C
P1                      9.23 usec
PLW1             244.00000000 W

======== CHANNEL f2 =======
SFO2              500.6783527 MHz
NUC2                       1H
CPDPRG[2              waltz16
PCPD2                  80.00 usec
PLW2             13.60000038 W
PLW12             0.08840500 W
PLW13             0.05657900 W

F2 - Processing parameters
SI                         32768
SF                125.8951680 MHz
WDW                           EM
SSB          0
LB                      1.00 Hz
GB           0
PC                      1.40
```

Sample Name	HRMS22I28JAN24	**Position**	Vial 24	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	BK-07.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	1/28/2022 12:48:44 PM



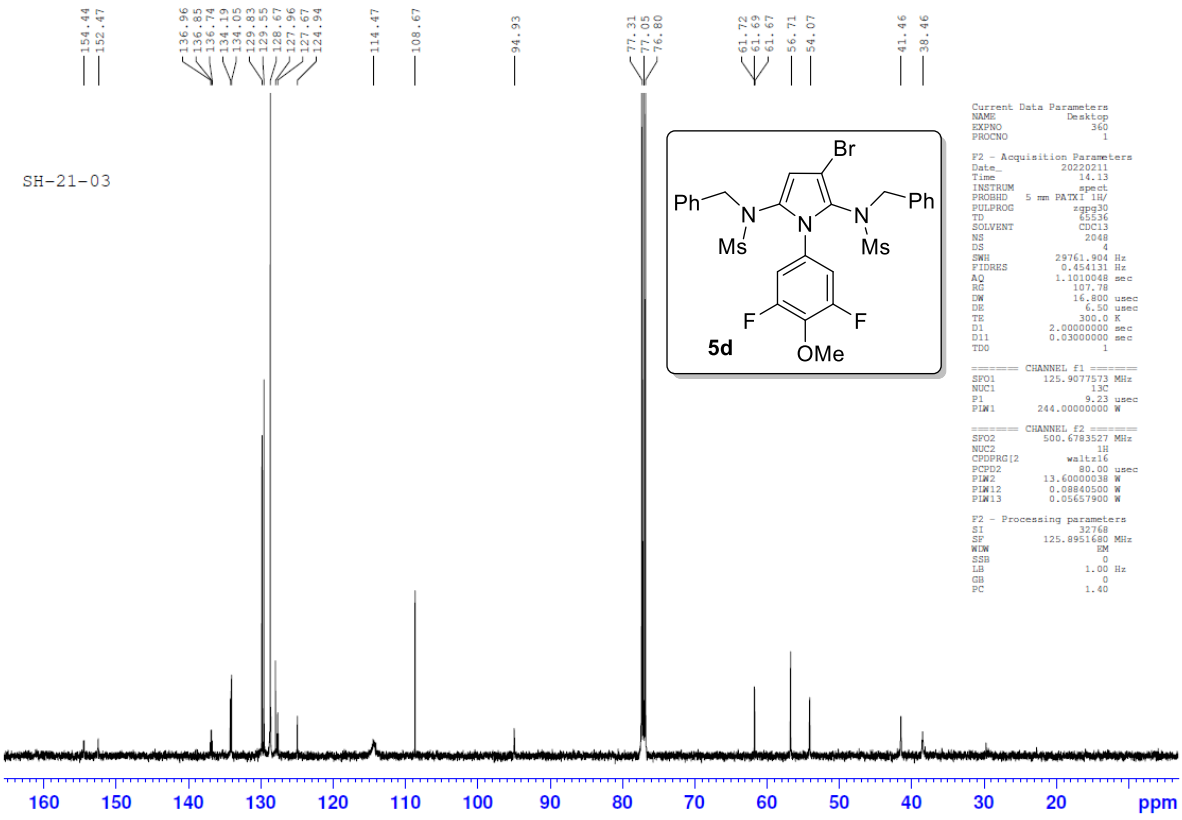
SH-21-03

^{1}H NMR, $CDCl_3$, 400 MHz

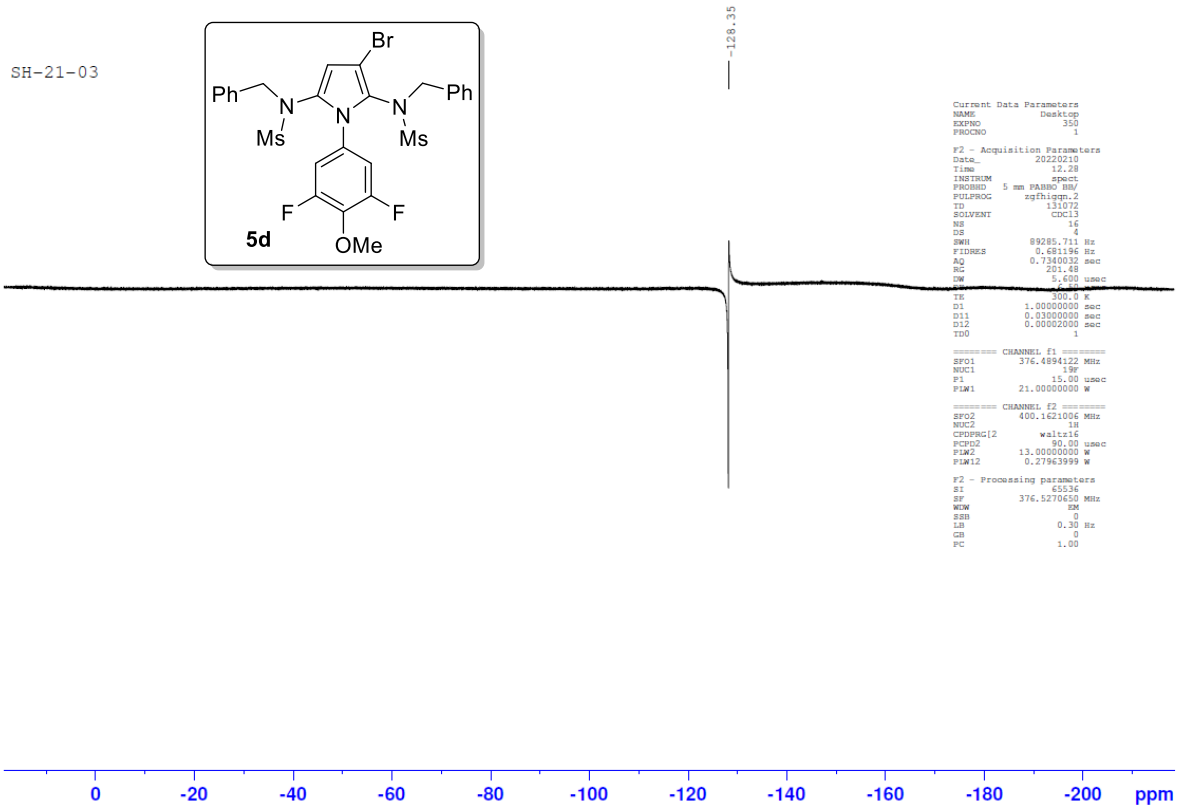


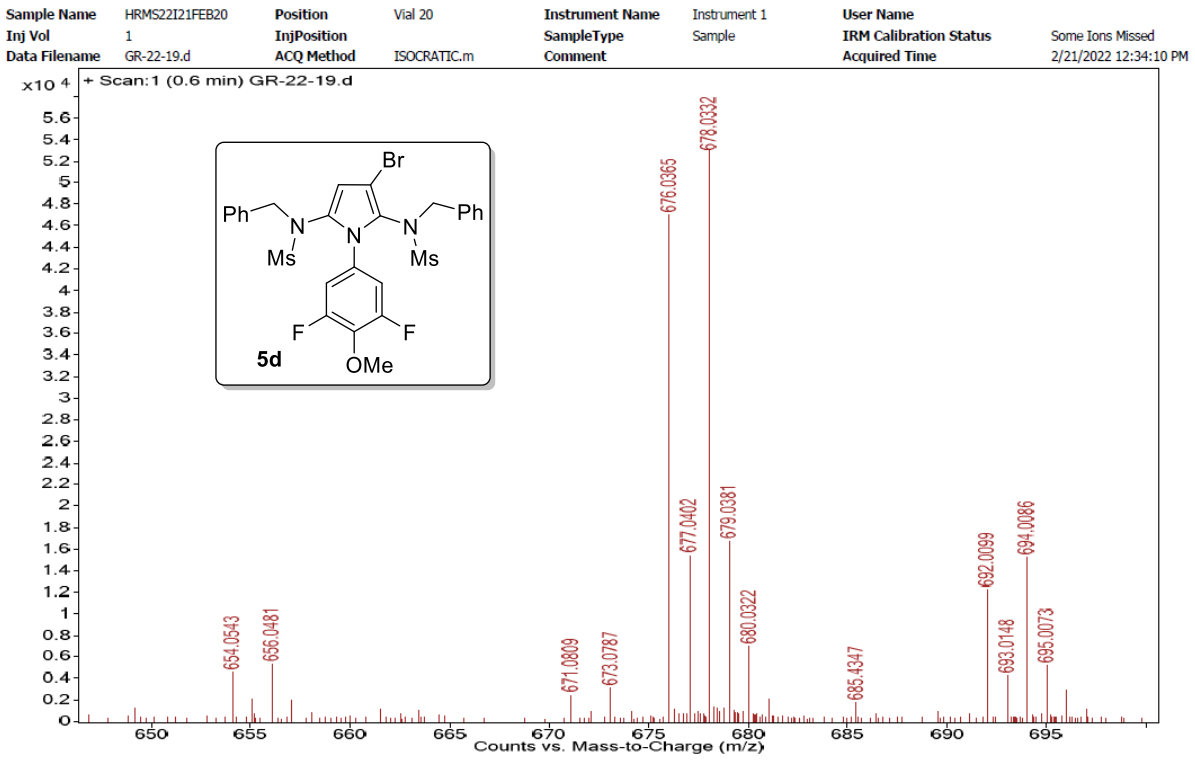

```
Current Data Parameters
NAME                  SH-21-03
EXPNO                      490
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20211222
Time                      13.23
INSTRUM                   spect
PROBHD     5 mm PABBO BB/
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                           8
DS                           0
SWH                   9615.385 Hz
FIDRES                0.146719 Hz
AQ                    3.4078720 sec
RG                       145.29
DW                       52.000 usec
DE                        6.50 usec
TE                       300.0 K
D1                    1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                400.1629712 MHz
NUC1                        1H
P1                       13.20 usec
PLW1               13.00000000 W

F2 - Processing parameters
SI                       65536
SF                  400.1605098 MHz
WDW                         EM
SSB                  0
LB                        0.30 Hz
GB                   0
PC                        1.00
```

^{13}C NMR, $CDCl_3$, 125 MHz



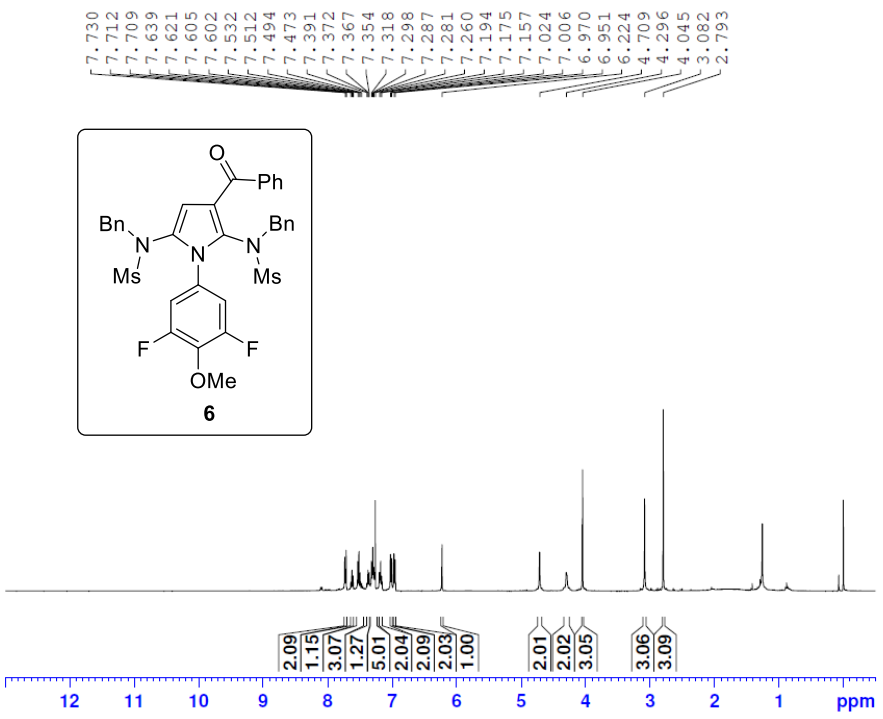
^{19}F NMR, $CDCl_3$, 376 MHz





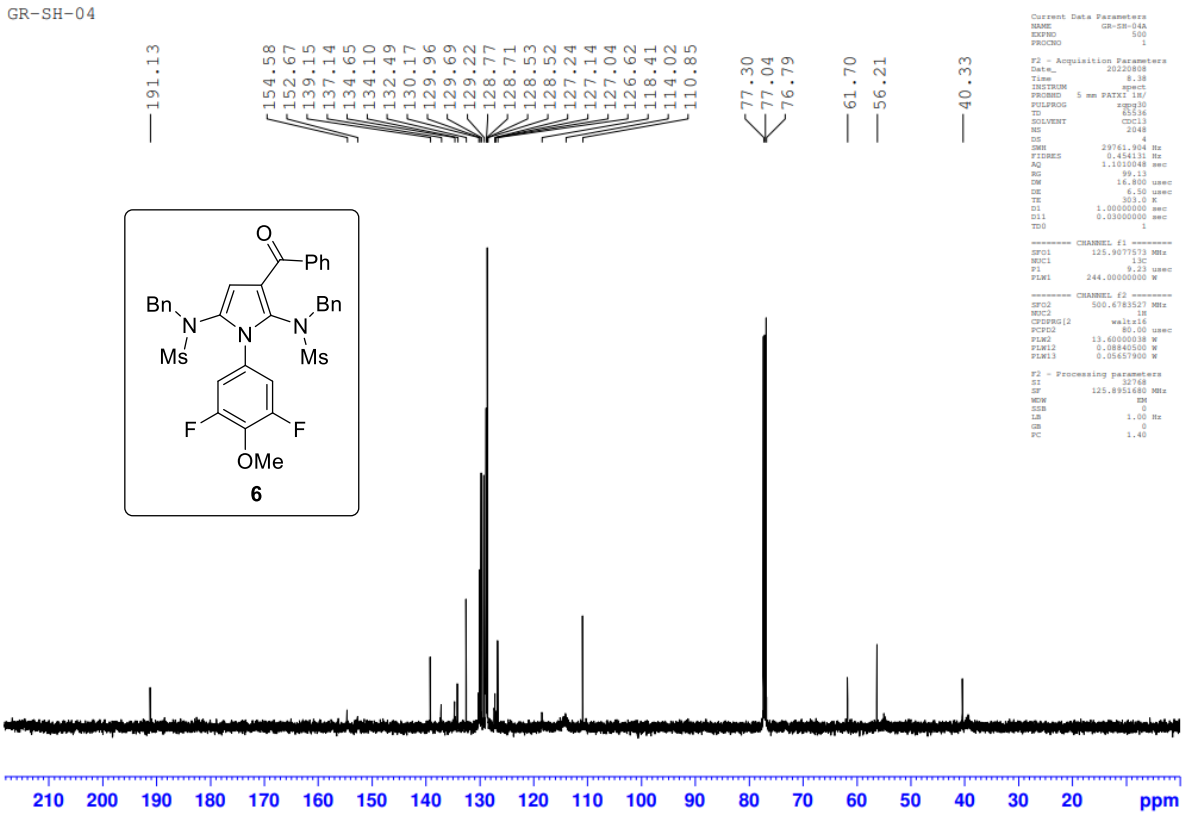
GR SH 04

^{1}H NMR, $CDCl_3$, 400 MHz



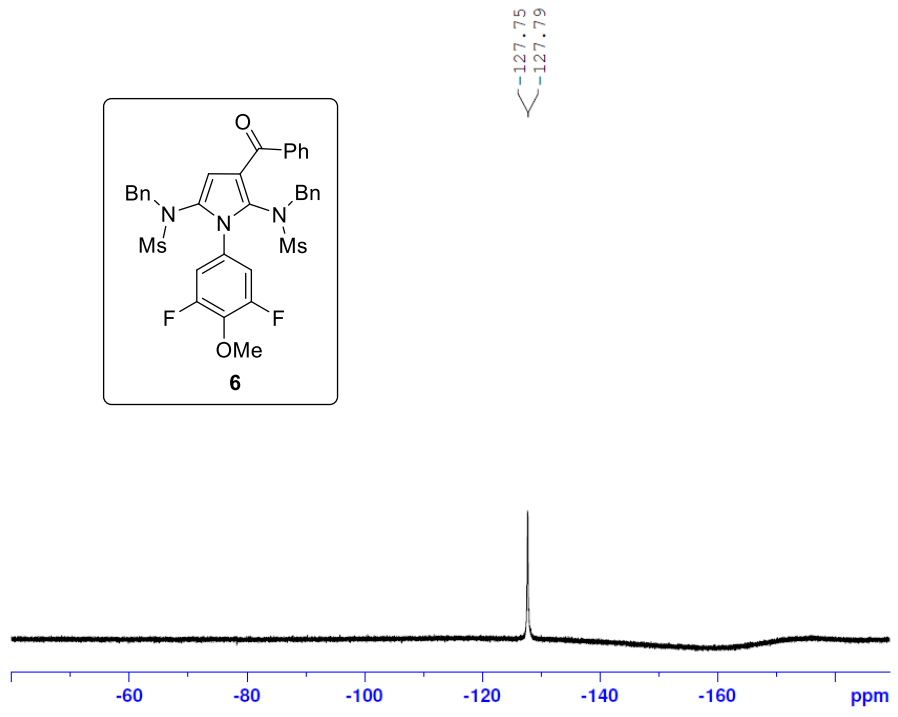
^{13}C NMR, $CDCl_3$, 101 MHz

GR-SH-04



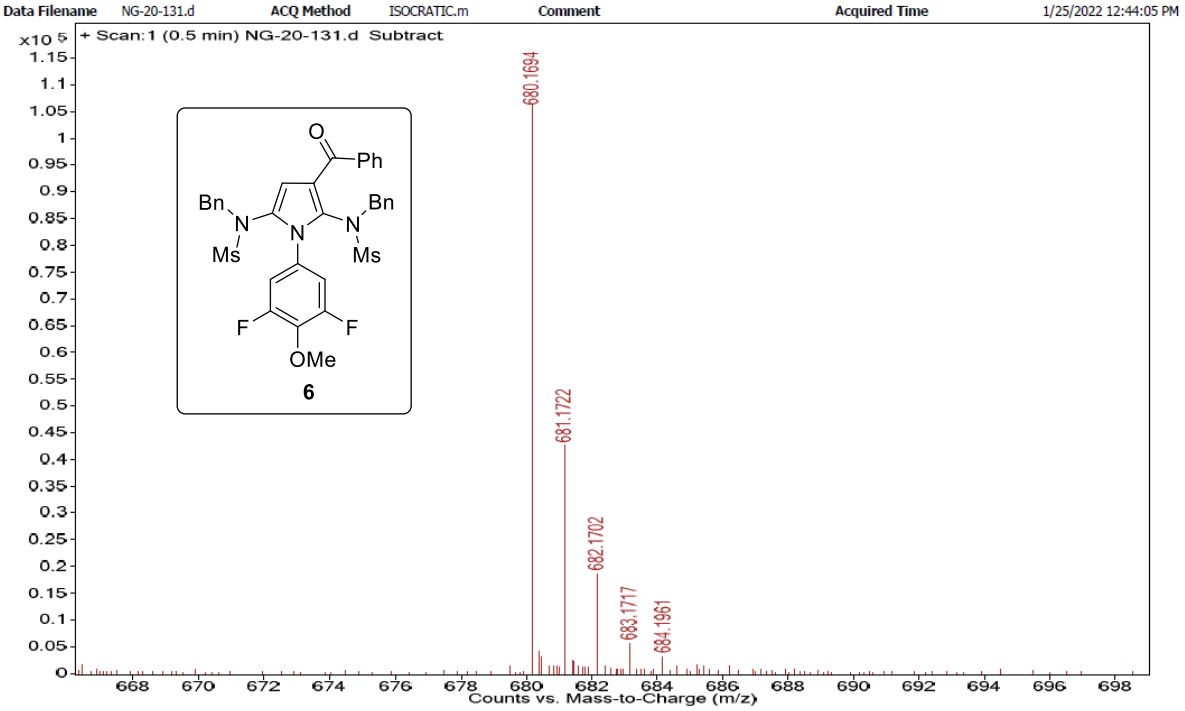
GR SH 04

^{19}F NMR, $CDCl_3$, 376 MHz

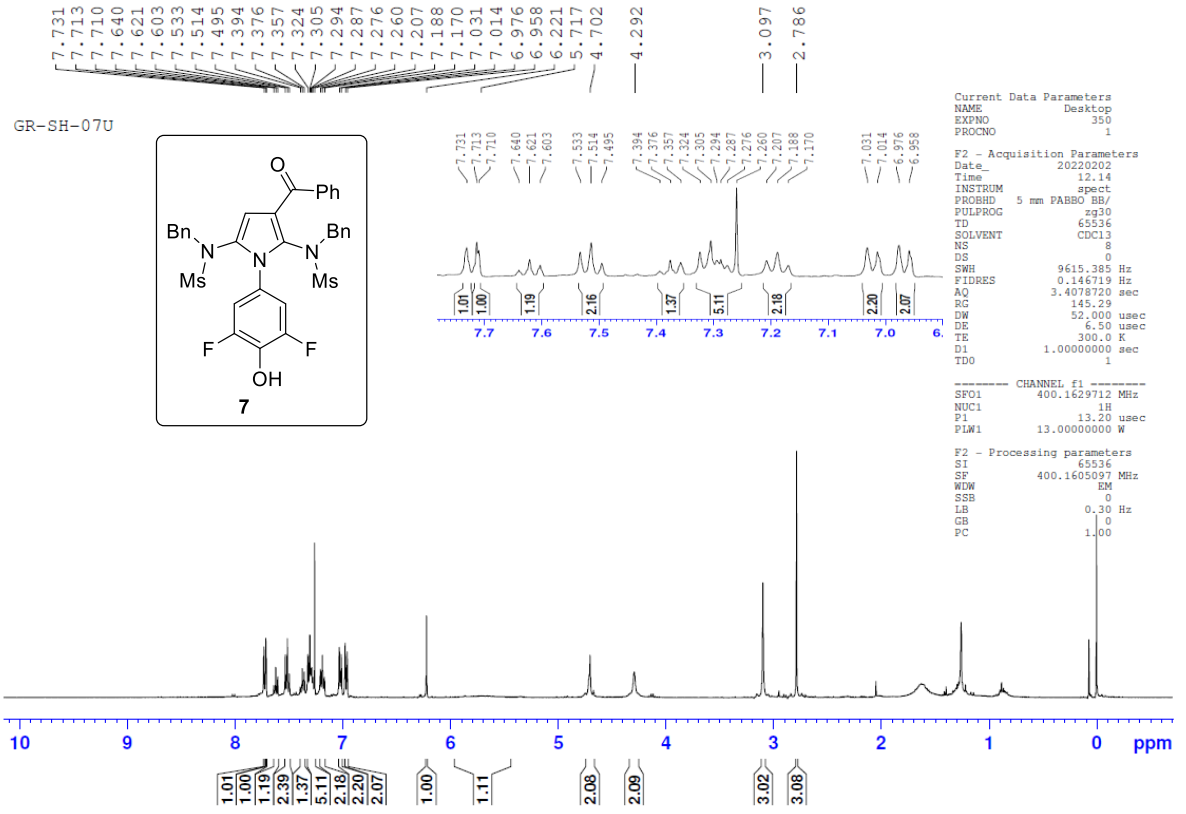



```
Current Data Parameters
NAME                  GR-SH-04
EXPNO                      370
PROCNO                       1

F2 - Acquisition Parameters
Date_               20220124
Time                     13.11
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG          zgfhigqn.2
TD                     131072
SOLVENT                 CDCl3
NS                         16
DS                          4
SWH                89285.711 Hz
FIDRES                 0.681196 Hz
AQ                     0.7340032 sec
RG                       201.48
DW                       5.600 usec
DE                       6.50 usec
TE                       300.1 K
D1                  1.00000000 sec
D11                 0.03000000 sec
D12                 0.00002000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1               376.4894122 MHz
NUC1                       19F
P1                       15.00 usec
PLW1               21.00000000 W

======== CHANNEL f2 ========
SFO2               400.1621006 MHz
NUC2                       1H
CPDPRG[2                waltz16
PCPD2                    90.00 usec
PLW2               13.00000000 W
PLW12                0.27963999 W

F2 - Processing parameters
SI                         65536
SF                 376.5270650 MHz
WDW                          EM
SSB          0
LB                       0.30 Hz
GB           0
PC                       1.00
```

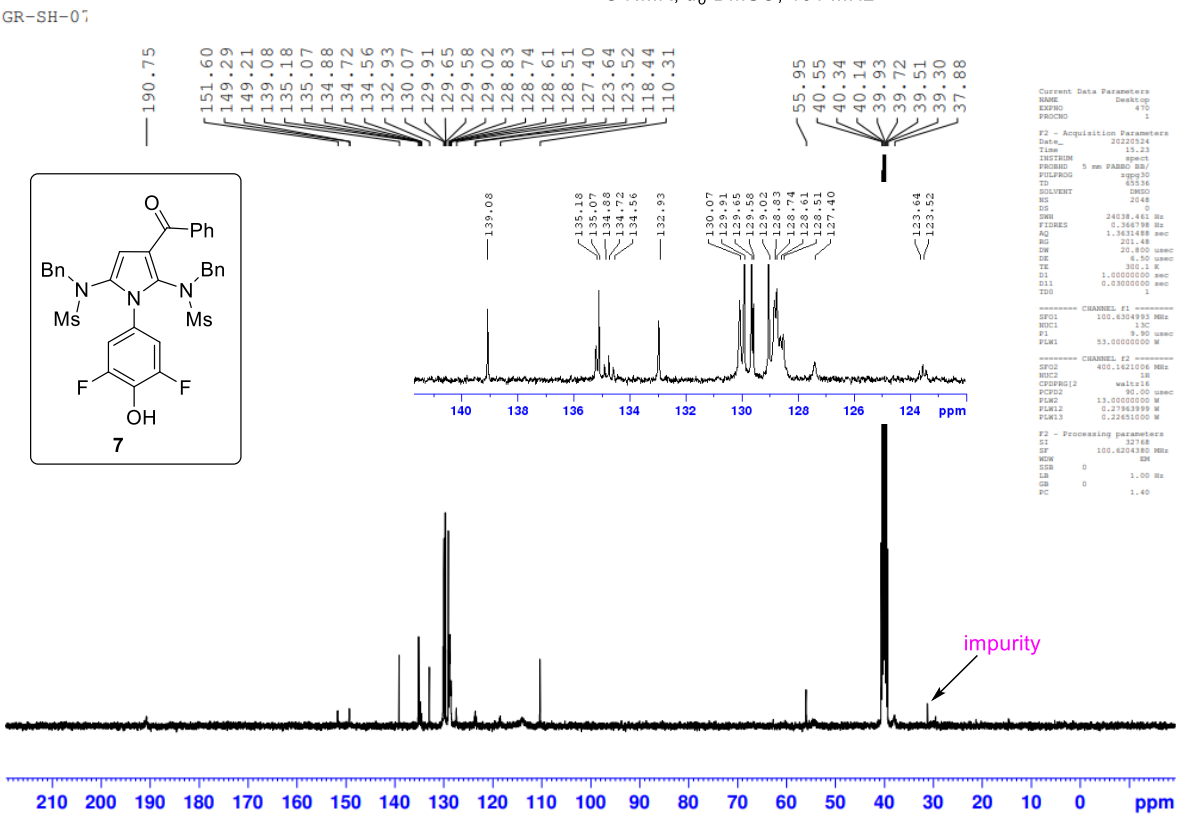
Sample Name	HRMS22I25JAN23	**Position**	Vial 23	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	NG-20-131.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	1/25/2022 12:44:05 PM



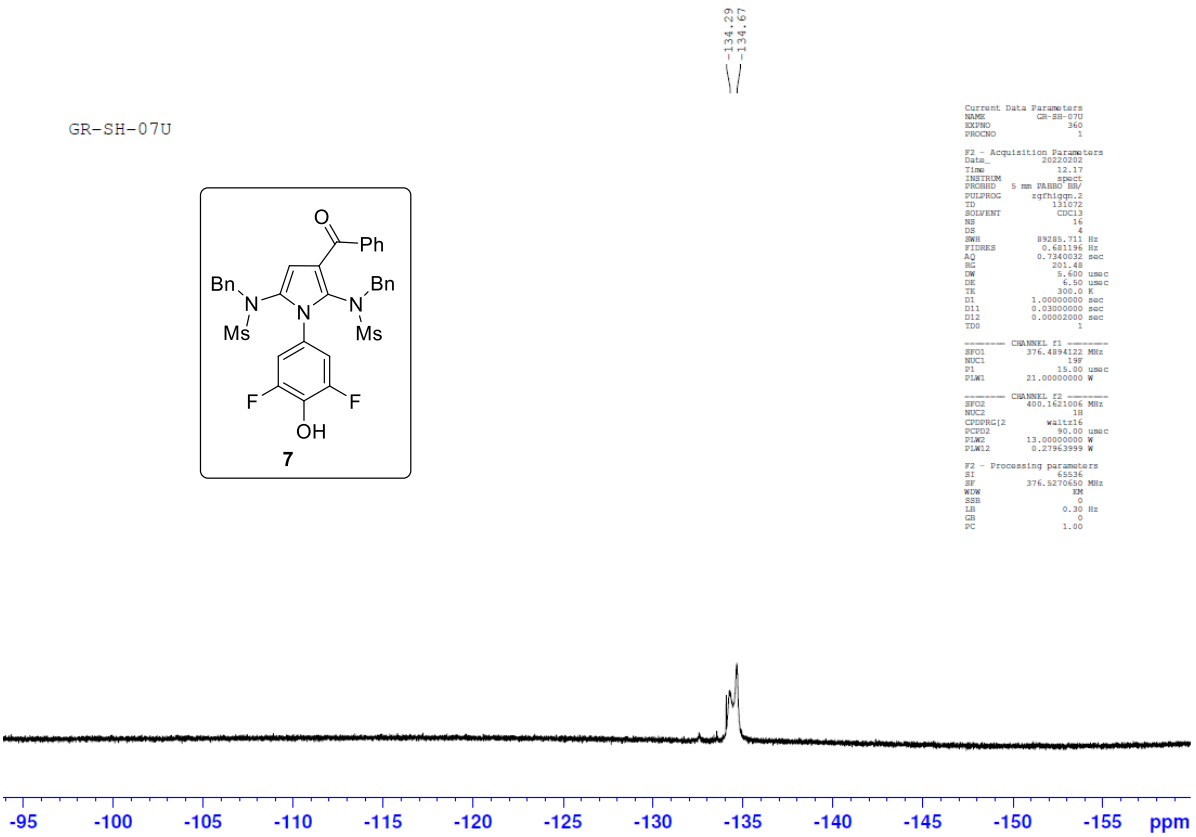
^{1}H NMR, $CDCl_3$, 400 MHz



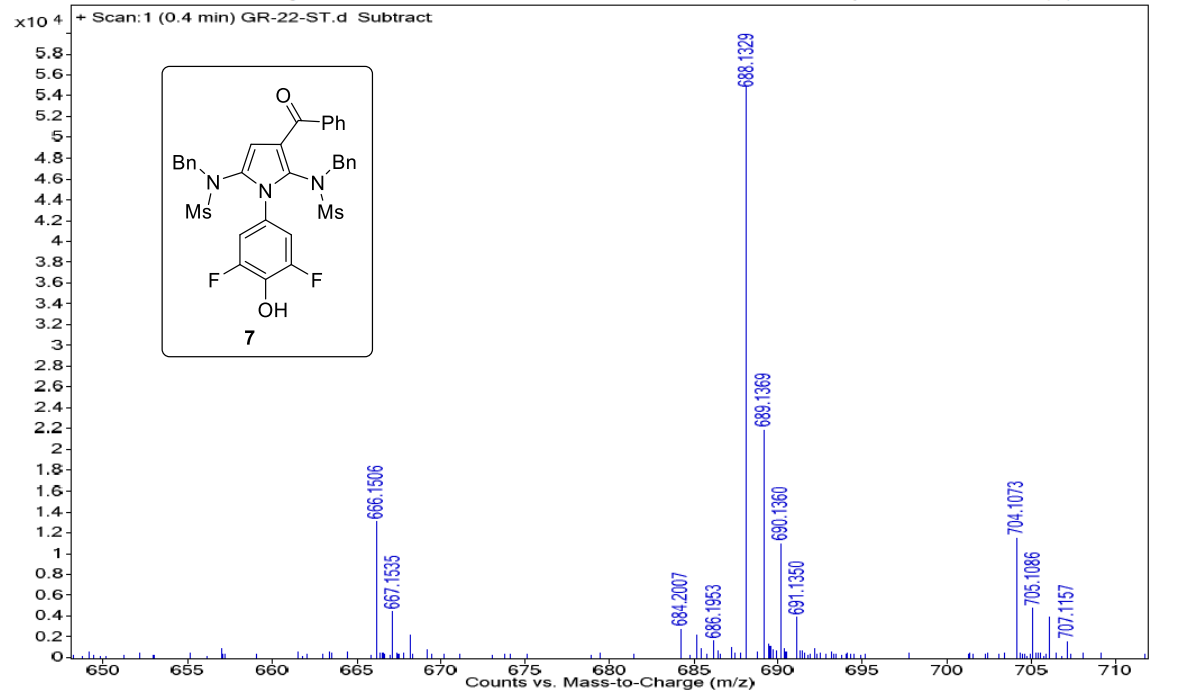
^{13}C NMR, d_6-DMSO, 101 MHz



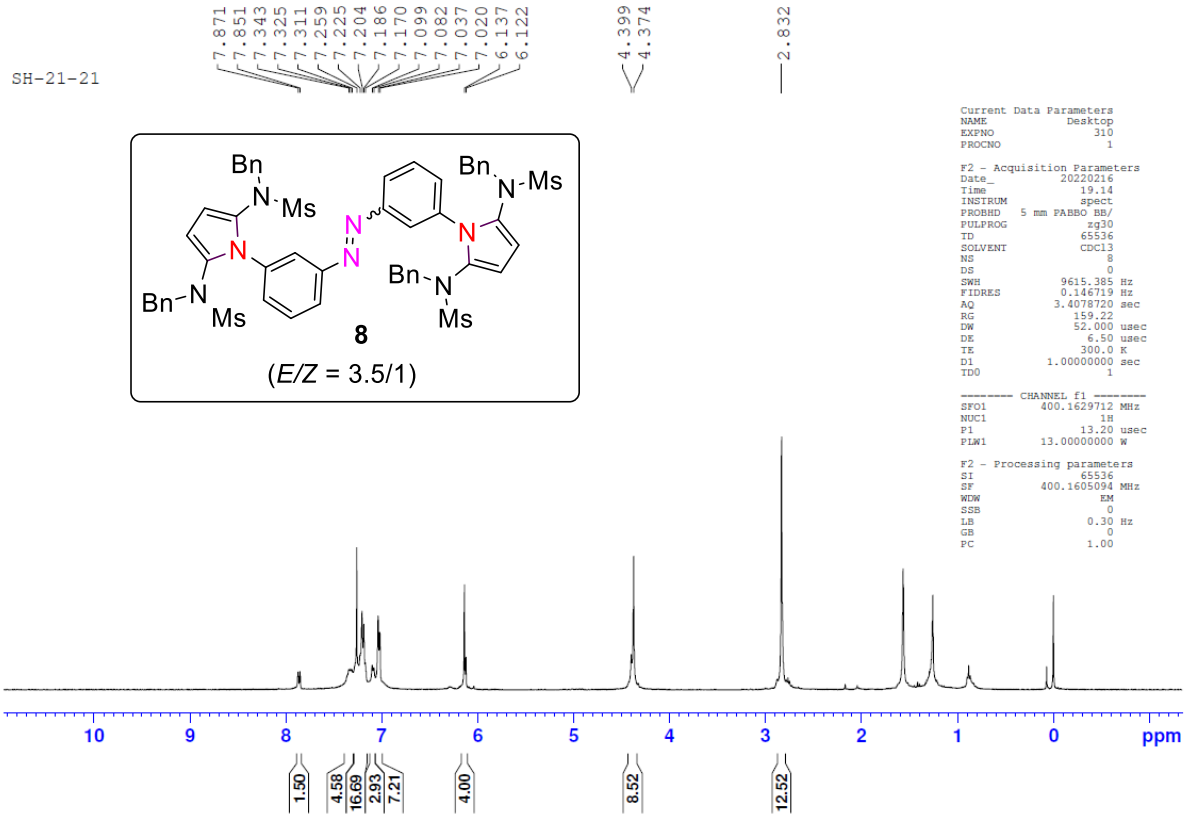
^{19}F NMR, $CDCl_3$, 376 MHz



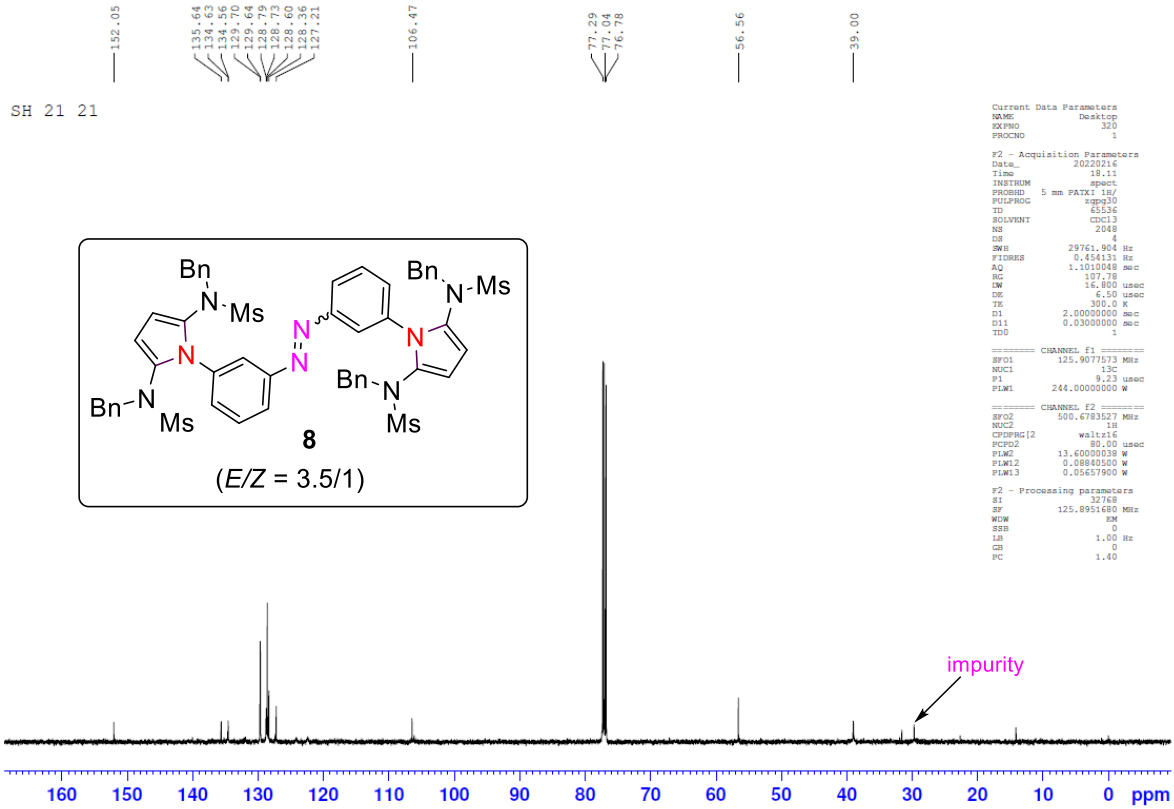
Sample Name	HRMS22I21FEB19	**Position**	Vial 19	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	GR-22-ST.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	2/21/2022 12:31:31 PM



^{1}H NMR, $CDCl_3$, 400 MHz



^{13}C NMR, $CDCl_3$, 125 MHz



Sample Name	HRMS22I11FEB26	**Position**	Vial 26	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	SH-21-10.d	**ACQ Method**	ISOCRATIC.m	**Comment**		**Acquired Time**	2/11/2022 12:56:40 PM