

Table of Contents

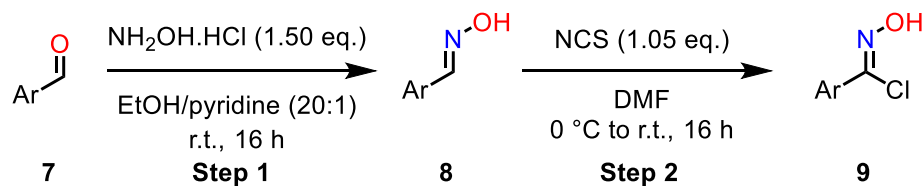
General Methods	2
General procedures for the synthesis of 9, 3b, 3c, 14, 18, 19, 22, 26, 3k, 3k and 5	3
Synthesis and experimental data for 2-Substituted-Alkynyl-1-Sulfonyl Fluorides (SASFs) 2a-2v	10
Synthesis and experimental data for compounds 4aa-4ax.....	20
Synthesis and experimental data for compounds 4ba-4aj.....	30
Synthesis and experimental data for compounds 4ca-4ar	35
Synthesis and experimental data for compounds 4da-4dk	44
Synthesis and experimental data for compounds 4ea-4ay	51
Synthesis and experimental data for compounds 4fa-4fl.....	64
Synthesis and experimental data for compounds 4ga-4an	71
Synthesis and experimental data for compounds 4ha-4hj.....	78
Synthesis and experimental data for compounds 4ia-4aj	84
Synthesis and experimental data for compounds 4ja-4jl	90
Synthesis and experimental data for compounds 4ka-4kj	97
Synthesis and experimental data for compounds 4la-4lq.....	104
Synthesis and experimental data for compounds 6a-6l	113
LLAMA and principle moments of inertia (PMI) analysis for DOC and SuFEx libraries	119
Antibacterial assays and method	121
References	123
X-ray crystallography data	124
Experimental procedure for SuFEx reactions performed in a 96 well plate format	138
Experimental procedure for SuFEx reactivity comparison	157
¹ H, ¹³ C and ¹⁹ F NMR spectra	158

General Methods

The petroleum ether used refers to the fraction with 40-60 °C boiling point. Commercial solvents and reagents were used as supplied. Unless otherwise stated, all reactions were monitored by TLC on Polygram® SIL/G₂₅ plates and visualized using UV light and stained using basic KMnO₄. ¹H, ¹³C and ¹⁹F NMR spectra were recorded on either a Bruker Ascend™ 400 (400 MHz), Bruker AV-400, Ultrashield™ 500 PLUS (500 MHz) or a Bruker AV-600 instrument as dilute solutions in the stipulated solvent. All chemical shifts (δ) are reported in parts per million (ppm) with ¹H and ¹³C NMR referenced to solvent signals [¹H NMR: CDCl₃ (7.27), DMSO-d₆ (2.50); ¹³C NMR: CDCl₃ (77.16), DMSO-d₆ (39.52)]. Coupling constants (*J*) are reported in Hertz (Hz) and recorded after averaging. The multiplicity of the ¹H NMR signals are designated by one of the following abbreviations: s=singlet, d=doublet, t=triplet, q=quartet, hept=heptet, m=multiplet, br=broad signal. Infra-red spectra were recorded using an Agilent Cary 660 FT-IR spectrophotometer in ATR mode, with the peaks recorded as ν_{\max} (cm⁻¹). HRMS were obtained using an Agilent 6530 accurate-mass Q-TOF LC/MS in electrospray ionization (ESI) mode, a Waters I-Class LC equipped with a Waters BEH C18 column using a water/acetonitrile/formic acid gradient and ionized in APCI negative mode on a Waters G2-XS TOF or on a Xevo G2-S in Atmospheric Solids Analysis Probe (ASAP) mode. LC/MS data was collected using at Agilent 1200 infinity series quaternary LC using a Waters XTerra MS C18 Column (3.5 μm 4.6 x 100 mm) together with a Thermo Scientific™ *MSQ Plus*™ Mass Detector in atmospheric pressure chemical ionization (APCI) mode. GC-MS data was recorded on an Agilent 7890A GC system with an Agilent 5975C Inert MSD system in electron ionization (EI) mode. Flash column chromatography was performed using a Biotage® Isolera™ on Biotage® KP-Sil SNAP cartridges. Melting points data were collected using a Gallenkamp melting point apparatus.

General procedures for the synthesis of 9, 3b, 3c, 14, 18, 19, 22, 26, 3k, 3k and 5

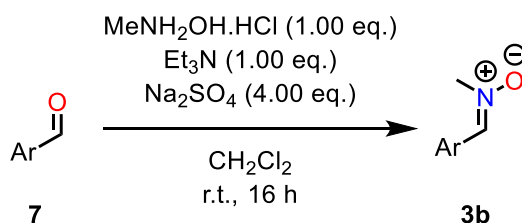
Synthesis of imidoyl chlorides 9



Step 1: Aldoximes 8 were synthesized by the dehydrative condensation of aldehydes 7 with NH_2OH .^[1] A typical procedure: To a solution of $\text{NH}_2\text{OH}\cdot\text{HCl}$ (1.50 eq.), in EtOH (1.00 mL/mmol), and pyridine (50.0 $\mu\text{L}/\text{mmol}$) was added the required aldehyde 7 (1.00 eq.) and stirred at room temperature for 16 h. The solvent was removed under reduced pressure and the residue extracted into ethyl acetate (5.00 mL/mmol), washed with H_2O (2 x 5.00 mL/mmol), dried over anhydrous MgSO_4 , filtered under vacuum and the solvent was removed under reduced pressure to obtain the analytically pure product.

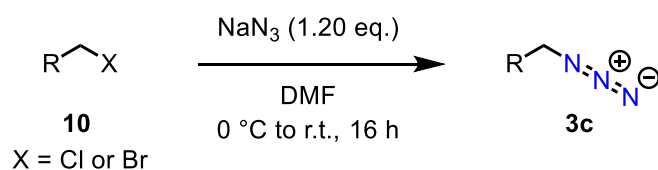
Step 2: Imidoyl chlorides 9 were synthesized through the chlorination of aldoximes 8 with *N*-chlorosuccinimide (NCS).^[2] A typical procedure: To a solution of the required aldoxime 8 (1.00 eq.) in DMF (3.00 mL/mmol) stirring at 0 °C was added NCS (1.05 eq.) portion wise over 10 min. The reaction was then removed from the cold bath and stirred at room temperature for 16 h. The reaction mixture was extracted into EtOAc (5.00 mL/mmol), washed with brine (2 x 5.00 mL/mmol) and H_2O (3 x 5.00 mL/mmol), dried over anhydrous MgSO_4 , filtered under vacuum and the solvent was removed under reduced pressure to obtain the analytically pure product.

Synthesis of nitrones 3b



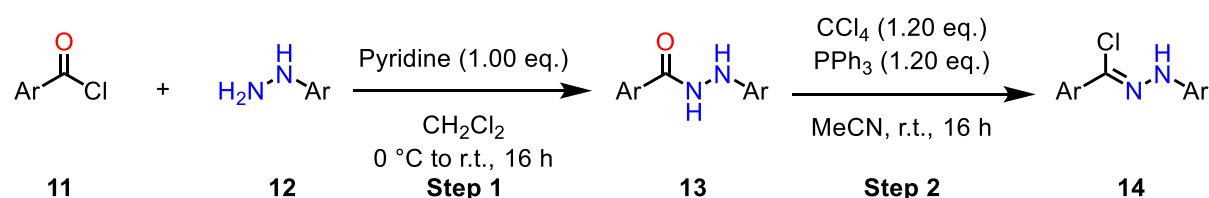
Nitrones 3b were synthesized by the dehydrative condensation of aldehydes 7 with MeNH_2OH .^[3] A typical procedure: To a mixture of the required aldehyde 7 (1.00 eq.), $\text{MeNH}_2\text{OH}\cdot\text{HCl}$ (1.00 eq.) and Na_2SO_4 (4.00 eq.) in CH_2Cl_2 (10.0 mL/mmol) was added Et_3N (1.00 eq.) and stirred at room temperature for 16 h. The solids were then removed by vacuum filtration and the solvent from the filtrate was removed under reduced pressure to obtain the analytically pure product.

Synthesis of alkyl azides **3c**



Azides **3c** were synthesized through the substitution reaction of alkyl halides **10** with NaN_3 .^[4] A typical procedure: To a solution of the required alkyl halide **10** (1.00 eq.) in DMF (3.00 mL/mmol) stirring at 0 °C was added NaN_3 (1.20 eq.) portion wise over 10 min. The reaction was then removed from the cold bath and stirred at room temperature for 16 h. The reaction mixture was extracted into EtOAc (5.00 mL/mmol), washed with brine (2 x 5.00 mL/mmol) and H_2O (3 x 5.00 mL/mmol), dried over anhydrous MgSO_4 , filtered under vacuum and the solvent was removed under reduced pressure to obtain the analytically pure product.

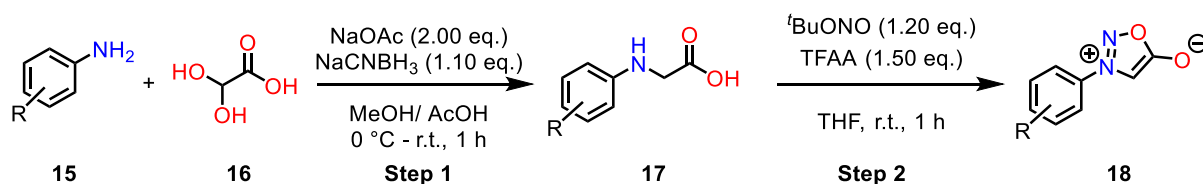
Synthesis of hydrazonoyl chlorides **14**



Step 1: Acylhydrazines **13** were synthesized through the coupling of acyl chlorides **11** and hydrazines **12**.^[5] A typical procedure: To a solution of the required hydrazine **12** (1.00 eq.) in CH_2Cl_2 (1.00 mL/mmol) at 0 °C was added pyridine (1.00 eq.) dropwise. To the reaction mixture was then added a solution of the required acyl chloride **11** (1.00 eq.) in CH_2Cl_2 (250 μL /mmol) dropwise and stirred at room temperature for 16 h. The reaction mixture was extracted into CH_2Cl_2 (5.00 mL/mmol), washed with brine (5.00 mL/mmol) and H_2O (5.00 mL/mmol), dried over anhydrous MgSO_4 , filtered under vacuum and the solvent was removed under reduced pressure. The crude mixture was then purified by flash column chromatography to obtain the analytically pure product.

Step 2: Hydrazonoyl chlorides **14** were synthesized through the chlorination of acylhydrazines **13** with carbon tetrachloride and triphenylphosphine.^[5] A typical procedure: To a solution of the required acylhydrazone **13** (1.00 eq.) in MeCN (2.00 mL/mmol) was added carbon tetrachloride (1.20 eq.) and triphenylphosphine (1.20 eq.) and stirred at room temperature for 16 h. The solvent was removed under reduced pressure and the crude mixture was then purified by flash column chromatography to obtain the analytically pure product.

Synthesis of Sydnones **18**

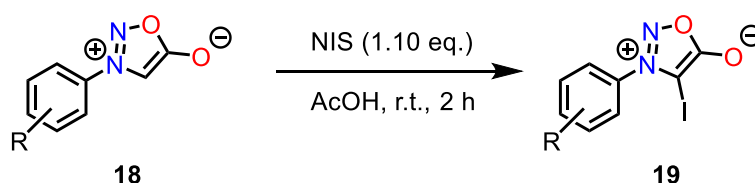


Sydnones **18** were synthesised from the corresponding aniline **15**, via a sequential reductive amination and cyclization reaction.^[6] A typical procedure:

Step 1: To a solution of the required aniline **15** (1.00 eq.) in MeOH (1.00 mL/mmol) and glacial acetic acid (4.00 eq.) at 0 °C was added sodium acetate (2.00 eq.), glyoxylic acid monohydrate (1.50 eq.) and NaCNBH₃ (1.10 eq.) sequentially. The solution was stirred for 1 h, slowly warming to room temperature. The reaction mixture was then filtered through a short pad of silica gel and washed with 1.00% glacial acetic acid in EtOAc (80.0 mL). The solution was then washed with brine (3 x 80.0 mL), dried over anhydrous MgSO₄, filtered under vacuum and the solvent was removed under reduced pressure to yield the crude carboxylic acid **17**, which was used without further purification.

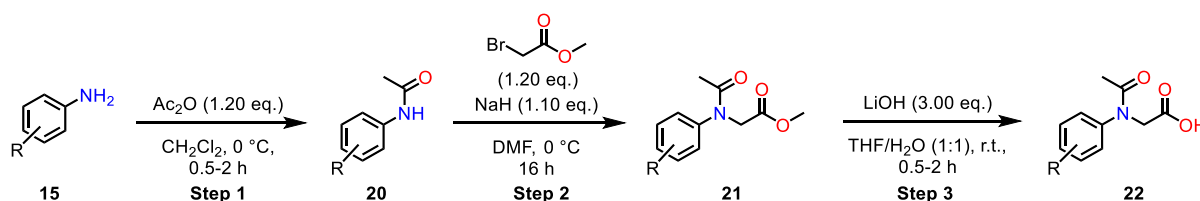
Step 2: The crude carboxylic **17** was dissolved in THF (2.00 mL/mmol) and ^tBuONO (1.20 eq.) was added. The mixture was stirred at room temperature for 30 min, then TFAA (1.50 eq.) was added, and the resultant mixture stirred at room temperature for 1 h. The reaction was quenched with NaHCO₃ (sat. aq.) (10.0 mL/mmol) and extracted with EtOAc (10.0 mL/mmol). The organic layer was dried over anhydrous MgSO₄, filtered under vacuum and the solvent was removed under reduced pressure. The crude product was purified by recrystallization (EtOH) to obtain the analytically pure product.

Synthesis of iodosydnones **19**



Iodosydnones **19** were synthesized by the iodination of Sydnones **18** with *N*-iodosuccinimide.^[7] A typical procedure: To a suspension of the required Sydnone **18** (1.00 eq.) in AcOH (2.00 mL/ mmol) at room temperature was added *N*-iodosuccinimide (NIS) (1.10 eq.), and stirred at room temperature for 2 h. The reaction mixture was diluted with water (40.0 mL/ mmol) and the precipitate formed filtered under vacuum and dried to obtain the analytically pure product.

Synthesis of substituted *N*-acetyl-*N*-arylglycines **22**



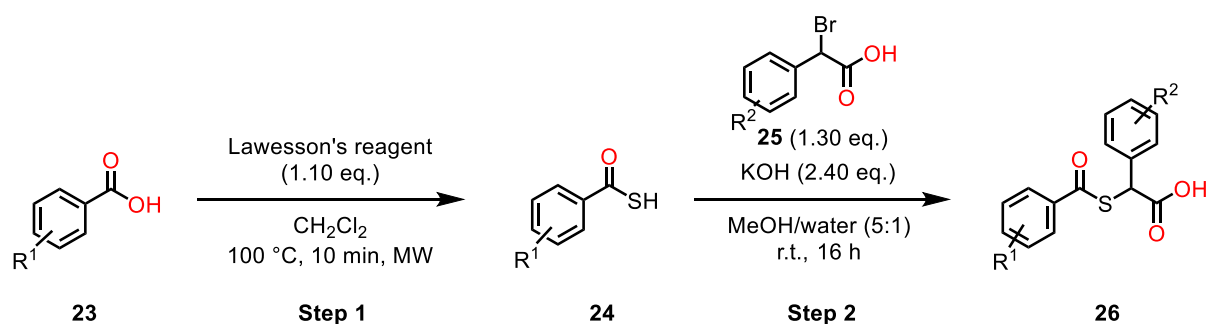
N-Acetyl-*N*-arylglycines **22** were synthesized from the corresponding aniline **15**, *via* sequential acetylation, alkylation and hydrolysis reactions.^[8-10] A typical procedure:

Step 1:^[8] To a solution of the required aniline **15** (1.00 eq.) in CH₂Cl₂ (1.00 mL/mmol) at 0 °C was added acetic anhydride (1.20 eq.). The reaction was stirred at room temperature for 0.5-2 h and the reaction mixture was then extracted with CH₂Cl₂ (5.00 mL/ mmol) and washed with NaHCO₃ (sat. aq.) (5.00 mL/mmol). The organic layer was dried over anhydrous MgSO₄, filtered under vacuum and the solvent was removed under reduced pressure to obtain the analytically pure product.

Step 2:^[9] To a solution of the required *N*-acetylaniline **20** (1.00 eq.) in anhydrous DMF (1.00 mL/mmol) at 0 °C was added sodium hydride (1.10 eq., 60% in mineral oil) in one portion. The reaction mixture was stirred at 0 °C for 0.5 h, then methyl bromoacetate (1.20 eq.) was added, and the reaction stirred at room temperature for a further 16 h. The reaction mixture was diluted with water (5.00 mL/mmol) and extracted into EtOAc (10.0 mL/mmol). The organic layer was washed with H₂O (5 x 5.00 mL/mmol) and brine (5.00 mL/mmol), dried over anhydrous MgSO₄, filtered under vacuum and the solvent was removed under reduced pressure. The crude product was purified by flash column chromatography to obtain the analytically pure product.

Step 3:^[10] To a solution of the required methyl *N*-acetyl-*N*-arylglycinate **21** (1.00 eq.) in THF (2.00 mL/mmol) and water (2.00 mL/mmol) at room temperature was added lithium hydroxide (3.00 eq.). The reaction was stirred at room temperature for 0.5-2 h, and then the solvent was removed under reduced pressure. The crude mixture was suspended in water (10.0 mL/mmol) and acidified with conc. HCl. The precipitate formed was collected by filtration under vacuum to obtain the analytically pure product.

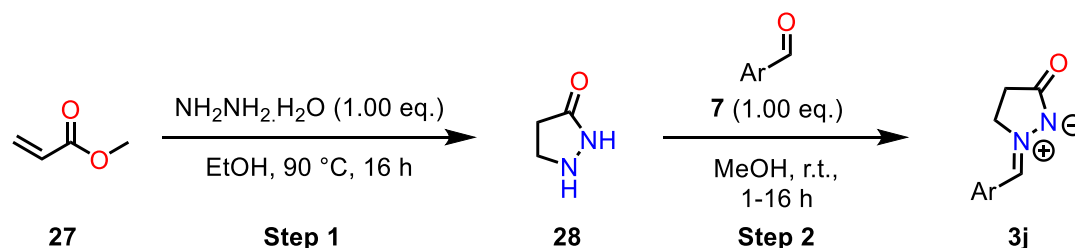
Synthesis of 2-(benzoylthio)-2-arylacetic acids **26**



Step 1: Thiobenzoic acids **24** were synthesized by reaction of benzoic acids **23** with Lawesson's reagent.^[11] A typical procedure: To a solution of the required benzoic acid **23** (1.00 eq.), in CH_2Cl_2 (3.50 mL/mmol) was added Lawesson's reagent (1.10 eq.), the reaction vial was sealed and irradiated with microwaves at 100°C for 10 min. The reaction mixture was extracted into CH_2Cl_2 (10.0 mL/mmol), washed with H_2O (2 x 10.0 mL/mmol), dried over anhydrous MgSO_4 , filtered under vacuum and the solvent was removed under reduced pressure. The crude mixture was purified by flash column chromatography (0-10% EtOAc in petroleum ether) to obtain analytically pure product.

Step 2: 2-(Benzoylthio)-2-arylacetic acids **26** were synthesized by reaction of thiobenzoic acids **24** with bromo-2-arylacetic acids **25** under basic conditions.^[12] A typical procedure: To a solution of KOH (3.50 eq.) in methanol (5.40 mL/mmol), and water (540 μL /mmol) was added the required thiobenzoic acid **24** (1.00 eq.) and the required bromo-2-arylacetic acid **25** (1.30 eq.), and stirred at room temperature for 16 h. The solvent was removed under reduced pressure and extracted into CH_2Cl_2 (15.0 mL/mmol), washed with 1M HCl (2 x 10.0 mL/mmol), dried over anhydrous MgSO_4 , filtered under vacuum and the solvent was removed under reduced pressure. The crude mixture was purified by flash column chromatography (40-80% EtOAc in petroleum ether) to obtain the analytically pure product.

Synthesis of azomethine imines **3j**



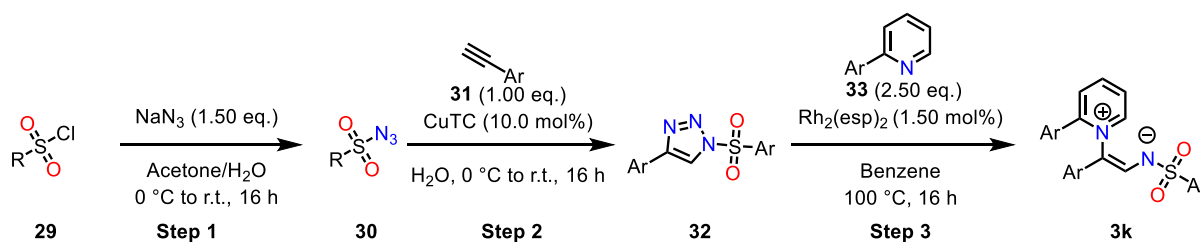
Azomethine imines **3j** were synthesized by the dehydrative condensation of the freshly prepared pyrazolidin-3-one **28** with benzaldehydes **7**.^[13] A typical procedure:

Step 1: To a solution of $\text{NH}_2\text{NH}_2\cdot\text{H}_2\text{O}$ (1.00 eq.) in EtOH (1.50 mL/mmol) at 0°C . was added methyl acrylate **27** (1.10 eq.) at 0°C . The reaction was stirred at room temperature for 10 min and then at 90°C for a further 16 h.

The solvent was removed under reduced pressure and the crude pyrazolidin-3-one **28** was used directly without further purification.

Step 2: The crude pyrazolidin-3-one **28** was dissolved in MeOH (1.00 mL/mmol) and the required aldehyde **7** (1.00 eq.) added at room temperature. The reaction mixture was stirred for 1-16 h, and the resultant precipitate collected by filtration under vacuum and washed with Et₂O (10.0 mL/ mmol) to obtain the analytically pure product.

Synthesis of azomethine ylides **3k**

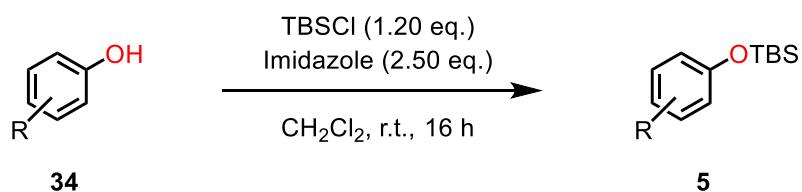


Step 1: Sulfonyl azides **30** were synthesized by the substitution reaction of sulfonyl chlorides **29** with sodium azide.^[14] A typical procedure: To a solution of the required sulfonyl chloride **29** (1.00 eq.) in acetone (1.50 mL/mmol) and H_2O (400 μL /mmol) at $0\text{ }^\circ\text{C}$ was added sodium azide portion wise over 10 min. The reaction was then stirred at room temperature for 16 h and the solvent was then removed under reduced pressure. The crude mixture was extracted into EtOAc (5.00 mL/mmol), washed with brine (5.00 mL/mmol) and H_2O (2 x 5.00 mL/mmol), dried over anhydrous MgSO_4 , filtered under vacuum and the solvent was removed under reduced pressure to obtain the analytically pure product.

Step 2: 1-Sulfonyltriazoles **32** were synthesized through a copper catalyzed azide-alkyne cycloaddition between sulfonyl azides **30** and alkynes **31**.^[15] A typical procedure: To a suspension of the required alkyne **31** (1.00 eq.) and copper(I) thiophene-2-carboxylate (CuTC , 10.0 mol%) in H_2O (5.00 mL/mmol) at $0\text{ }^\circ\text{C}$, was added the required sulfonyl azide **30** (1.00 eq.) portion wise over 10 min. The reaction mixture was then stirred at room temperature for 16 h and extracted into EtOAc (5.00 mL/mmol), washed with NH_4Cl (sat. aq.) (5.00 mL/mmol) and H_2O (5.00 mL/mmol), dried over anhydrous MgSO_4 , filtered under vacuum and the solvent was removed under reduced pressure. The crude mixture was purified by flash column chromatography to obtain the analytically pure product.

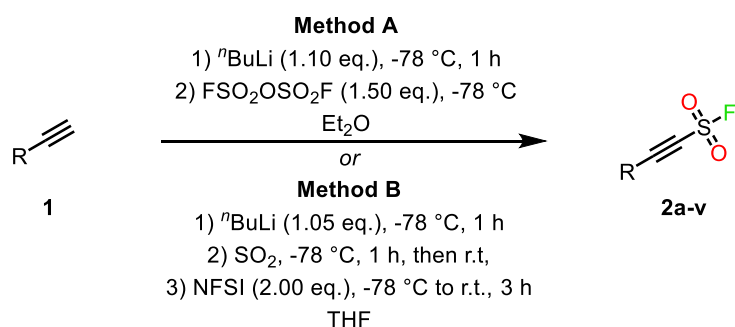
Step 3: Azomethine ylides **3k** were synthesized from the $\text{Rh}_2(\text{esp})_2$ catalyzed reaction of 1-sulfonyltriazoles **32** and the pyridines **33**.^[16] A typical procedure: To a solution of the required 1-sulfonyltriazole **32** (1.00 eq.) and $\text{Rh}_2(\text{esp})_2$ (1.50 mol%) in benzene (10.0 mL/mmol) at room temperature was added the required pyridine **33** (2.50 eq.). The reaction was stirred at $100\text{ }^\circ\text{C}$ for 16 h and the solvent was then removed under reduced pressure. The crude mixture was purified by flash column chromatography (0-10% MeOH in CH_2Cl_2) to obtain the analytically pure product.

Synthesis of aryl silyl ethers **5**

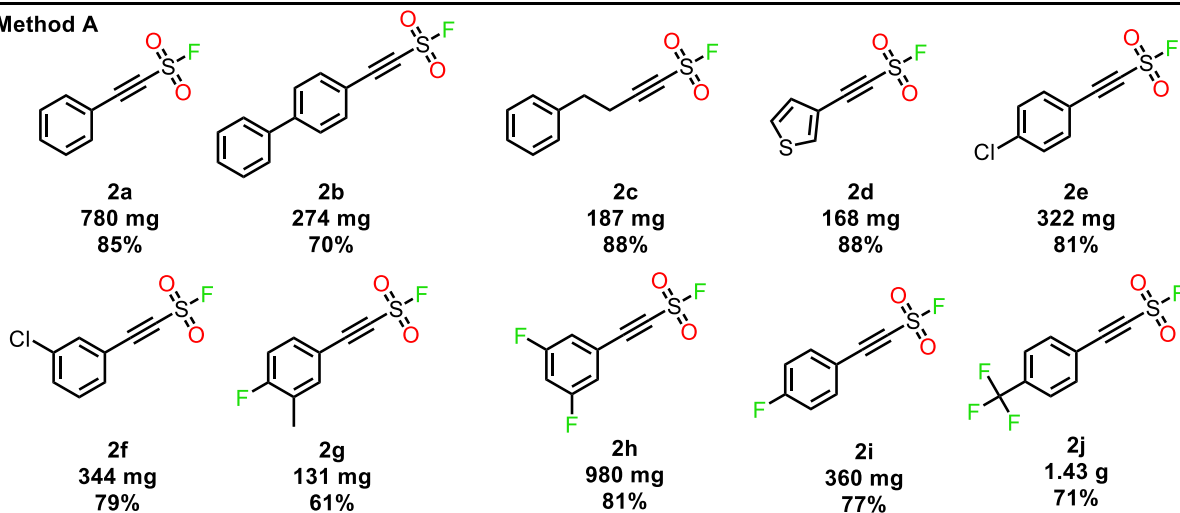


Aryl silyl ethers **5** were synthesised by the *tert*-butyldimethylsilyl (TBS) protection of phenols **34** with *tert*-butyldimethylsilyl chloride (TBSCl).^[17] A typical procedure: To a solution of phenol **34** (1.00 eq.) and TBSCl (1.20 eq.) in CH₂Cl₂ (4.00 mL/mmol) was added imidazole (2.50 eq.) and was stirred at room temperature for 16 h. The reaction mixture was extracted into EtOAc (5.00 mL/mmol), washed with 1N NaOH (5.00 mL/mmol) and H₂O (5.00 mL/mmol), dried over anhydrous MgSO₄, filtered under vacuum and the solvent was removed under reduced pressure. The crude mixture was purified by flash column chromatography to obtain the analytically pure product.

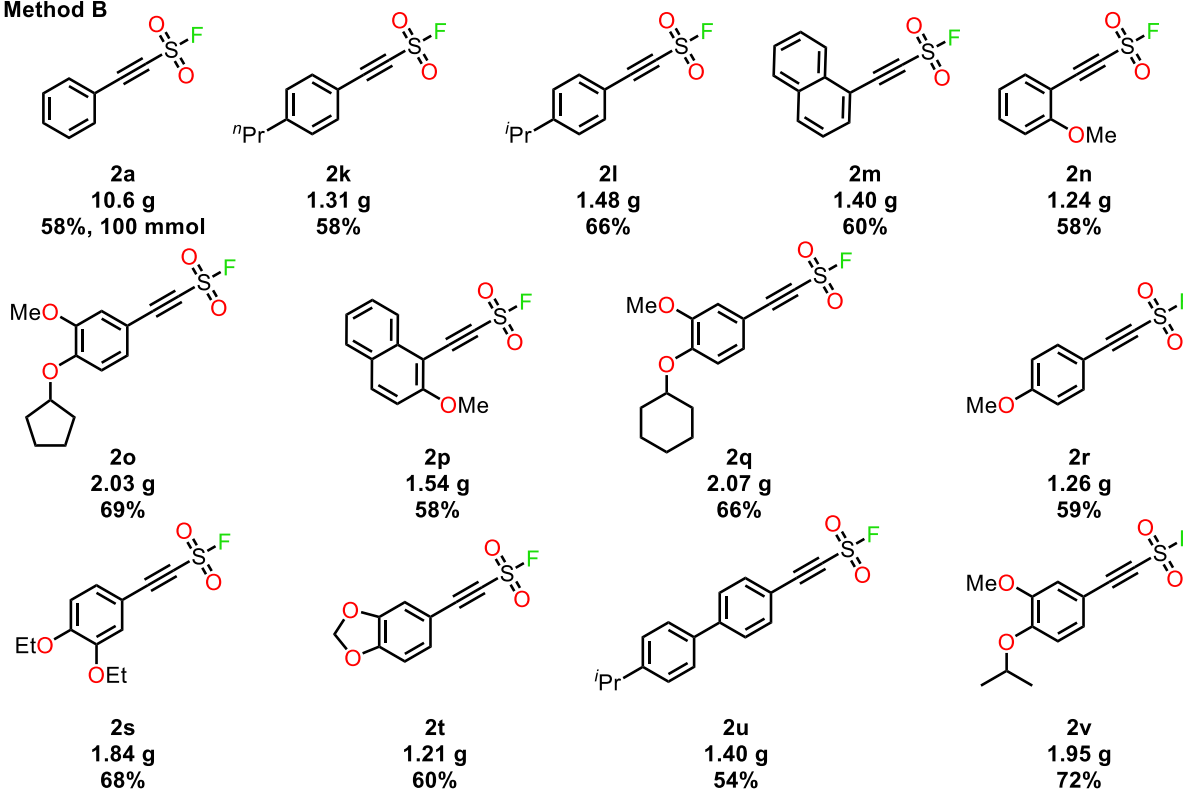
Synthesis and experimental data for 2-Substituted-Alkynyl-1-Sulfonyl Fluorides (SASFs) 2a-2v



Method A



Method B



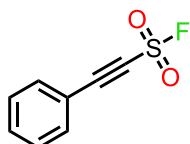
General Procedure A

To a solution of the required terminal alkyne **1a-1j** (1.00 mmol) in diethyl ether (5.00 mL) at -78 °C was added ⁿBuLi (1.05 eq.) dropwise. The resulting mixture was stirred at -78 °C for 0.5 h before the addition of fluorosulfonic anhydride (1.50 mmol, 1.50 eq.). After stirring at -78 °C for 0.5 h, the reaction was warmed to room temperature and the solvent was then removed under reduced pressure. The crude mixture was then purified by flash column chromatography (5% EtOAc in hexanes) to obtain the analytically pure product.

General Procedure B

To a solution of the required terminal alkyne **1a, 1k-1v** (10.0 mmol) in anhydrous THF (10.0 mL) at -78 °C was added ⁿBuLi (1.05 eq.) dropwise and stirred at -78 °C for 1 h. To the cooled reaction mixture was added a balloon of SO₂ by cannula, first bubbled through conc. H₂SO₄ (20.0 mL) and stirred at -78 °C for 1 h. The reaction mixture was warmed to room temperature and the remaining SO₂ removed by vacuum, purging with argon. The reaction mixture was then diluted with anhydrous THF (20.0 mL), cooled to -78 °C and *N*-fluorobenzenesulfonimide (6.30 g, 20.0 mmol) was added in one portion and removed from the cold bath, and stirred at room temperature for 3 h. The mixture was then filtered, the solvent removed under reduced pressure, redissolved in the minimum amount of diethyl ether, triturated with petroleum ether (50.0 mL), the solid removed by filtration and removal of the filtrate solvent under reduced pressure. The crude mixture was purified by flash column chromatography to obtain the analytically pure product.

2-Phenylethyne-1-sulfonyl fluoride (2a)



Method A

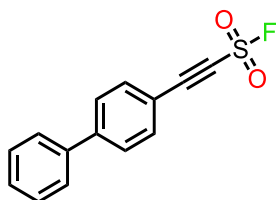
Following general procedure A (5.00 mmol of the required alkyne), the title compound was isolated as a yellow oil (780 mg, 85%). ¹H NMR (500 MHz, CDCl₃) δ = 7.70 – 7.67 (m, 2H), 7.64 – 7.60 (m, 1H), 7.51 – 7.47 (m, 2H); ¹³C NMR (126 MHz, CDCl₃) δ = 133.6, 133.2, 129.2, 95.4 (d, *J* = 8.1 Hz), 77.0 (d, *J* = 60.9 Hz); ¹⁹F NMR (471 MHz, CDCl₃) δ = 74.8; HRMS (ASAP): calculated for C₈H₆FO₂S [M+H]: *m/z* = 185.0072, *m/z* found 185.0069; IR ν_{max} (ATR)/cm⁻¹: 3069, 2192, 1422, 1249, 1203, 870.

Method B

Following general procedure B (100 mmol of the required alkyne), the title compound was isolated after purification by flash column chromatography (petroleum ether) as a yellow oil (10.6 g, 58%). ¹H NMR (500 MHz, CDCl₃) δ = 7.70 – 7.67 (m, 2H), 7.64 – 7.60 (m, 1H), 7.51 – 7.47 (m, 2H); ¹³C NMR (126 MHz, CDCl₃) δ = 133.6, 133.2, 129.2, 95.4 (d, *J* = 8.1 Hz), 77.0 (d, *J* = 60.9 Hz); ¹⁹F NMR (471 MHz, CDCl₃) δ = 74.8; HRMS (ASAP): calculated

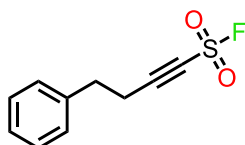
for $C_8H_6FO_2S$ $[M+H]^+$: $m/z = 185.0072$, m/z found 185.0069; **IR** v_{max} (ATR)/ cm^{-1} : 3069, 2192, 1422, 1249, 1203, 870.

1,1'-Biphenylethynyl sulfonyl fluoride (2b)



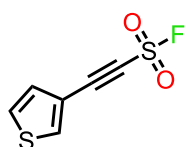
Following general procedure A (1.50 mmol of the required alkyne), the title compound was isolated as a pale yellow solid (274 mg, 70%). **m.p.** 92.4 °C; **1H NMR** (400 MHz, $CDCl_3$) δ 7.75 (appt. d, $J = 8.1$ Hz, 2H), 7.70 (appt. d, $J = 7.6$ Hz, 2H), 7.61 (appt. d, $J = 7.6$ Hz, 2H), 7.50 (appt. t, $J = 7.2$ Hz, 2H), 7.48 – 7.40 (m, 1H); **^{13}C NMR** (151 MHz, $CDCl_3$) δ 146.0, 139.3, 134.1, 129.3, 128.9, 127.8, 127.4, 114.5, 95.8 (d, $J = 8.1$ Hz), 77.5 (d, $J = 60.8$ Hz); **^{19}F NMR** (376 MHz, $CDCl_3$) δ 74.3; **GC-MS** (EI) calculated for $C_{14}H_9FO_2S$ $[M]^+$: calculated $m/z = 260.03$, m/z found 259.9.

4-Phenylbut-1-ynyl sulfonyl fluoride (2c)



Following general procedure A (1.00 mmol of the required alkyne), the title compound was isolated as a yellow oil (187 mg, 89%). **1H NMR** (400 MHz, $CDCl_3$) δ 7.40 – 7.32 (m, 2H), 7.32 – 7.26 (m, 1H), 7.24 – 7.18 (m, 2H), 2.97 (t, $J = 7.3$ Hz, 2H), 2.80 (td, $J = 7.3, 2.1$, 2H); **^{13}C NMR** (151 MHz, $CDCl_3$) δ 138.3, 129.0, 128.4, 127.4, 98.8 (d, $J = 7.8$ Hz), 70.6 (d, $J = 60.1$ Hz), 32.9, 21.3; **^{19}F NMR** (376 MHz, $CDCl_3$) δ 73.5; **HRMS** (APCI): calculated for $C_{10}H_8FO_2S$ $[M-H]^-$: $m/z = 211.0229$, m/z found 211.0231.

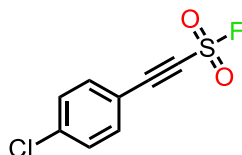
(Thiophen-3-yl)ethynyl sulfonyl fluoride (2d)



Following general procedure A (1.00 mmol of the required alkyne), the title compound was isolated as a yellow oil (168 mg, 88%). **1H NMR** (400 MHz, $CDCl_3$) δ 8.00 (dd, $J = 3.1, 1.6$ Hz, 1H), 7.51 – 7.36 (m, 1H), 7.32 (dd, $J = 5.2$,

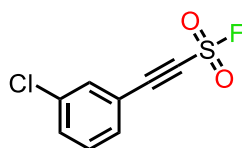
1.6 Hz, 1H); ^{13}C NMR (151 MHz, CDCl_3) δ 137.5, 130.0, 127.4, 115.3, 91.3 (d, $J = 8.2$ Hz), 77.1 (d, $J = 61.0$ Hz); ^{19}F NMR (376 MHz, CDCl_3) δ 74.3; HRMS (APCI): calculated for $\text{C}_7\text{H}_4\text{FO}_4\text{S}_2$ [$\text{M}+\text{CH}_2\text{O}_2$] $^-$: $m/z = 234.9535$, m/z found 234.9532.

4-Chlorophenylethynyl sulfonyl fluoride (2e)



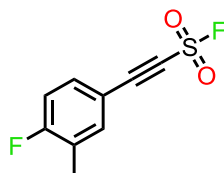
Following general procedure A (1.80 mmol of the required alkyne), the title compound was isolated as a yellow solid (322 mg, 81%). **m.p.** 78.6 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.62 (appt. d, $J = 8.2$ Hz, 2H), 7.47 (appt. d, $J = 8.2$ Hz, 2H); ^{13}C NMR (151 MHz, CDCl_3) δ 140.0, 134.7, 129.8, 114.4, 94.0 (d, $J = 8.2$ Hz), 77.8 (d, $J = 61.3$ Hz); ^{19}F NMR (376 MHz, CDCl_3) δ 74.4; HRMS (APCI): calculated for $\text{C}_9\text{H}_5\text{ClFO}_4\text{S}$ [$\text{M}+\text{CH}_2\text{O}_2$] $^-$: $m/z = 262.9581$, m/z found 262.9576.

3-Chlorophenylethynyl sulfonyl fluoride (2f)



Following general procedure A (2.00 mmol of the required alkyne), the title compound was isolated as a yellow oil (344 mg, 79%). ^1H NMR (400 MHz, CDCl_3) δ 7.67 (t, $J = 1.8$ Hz, 1H), 7.63 – 7.53 (m, 2H), 7.43 (t, $J = 7.9$ Hz, 1H); ^{13}C NMR (151 MHz, CDCl_3) δ 135.3, 133.5, 133.1, 131.6, 130.5, 117.7, 93.1 (d, $J = 8.1$ Hz), 77.7 (d, $J = 61.5$ Hz); ^{19}F NMR (376 MHz, CDCl_3) δ 74.4; HRMS (APCI): calculated for $\text{C}_9\text{H}_5\text{ClFO}_4\text{S}$ [$\text{M}+\text{CH}_2\text{O}_2$] $^-$: $m/z = 262.9581$, m/z found 262.9578.

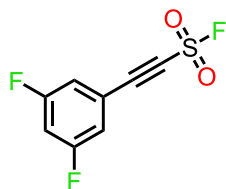
4-Fluoro-3-methylphenylethynyl sulfonyl fluoride (2g)



Following general procedure A (1.00 mmol of the required alkyne), the title compound was isolated as a yellow oil (131 mg, 61%). ^1H NMR (400 MHz, CDCl_3) δ 7.66 – 7.46 (m, 2H), 7.11 (t, $J = 8.8$ Hz, 1H), 2.32 (d, $J = 2.1$ Hz, 3H); ^{13}C NMR (151 MHz, CDCl_3) δ 164.2 (d, $J = 256.2$ Hz), 137.1 (d, $J = 6.7$ Hz), 133.5 (d, $J = 9.4$ Hz), 127.1 (d, $J = 18.6$

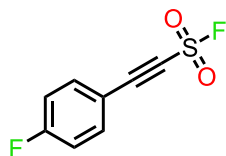
Hz), 116.5 (d, $J = 24.0$ Hz), 111.8 (d, $J = 4.1$ Hz), 95.0 (d, $J = 8.2$ Hz), 76.5 (d, $J = 1.7$ Hz), 14.5 (d, $J = 3.4$ Hz); ^{19}F NMR (376 MHz, CDCl_3) δ 74.3, -106.4; HRMS (APCI): calculated for $\text{C}_{10}\text{H}_7\text{F}_2\text{O}_4\text{S}$ [$\text{M}+\text{CH}_2\text{O}_2$]: $m/z = 261.0033$, m/z found 261.0034.

3,5-Difluoro-phenylethynyl sulfonyl fluoride (2h)



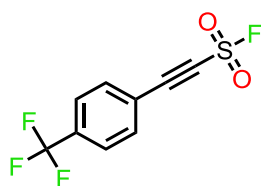
Following general procedure A (5.50 mmol of the required alkyne), the title compound was isolated as a pale yellow solid (980 mg, 81%). **m.p.** 48.8 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.24 – 7.20 (m, 2H), 7.09 (td, $J = 8.6, 2.1$ Hz, 1H); ^{13}C NMR (151 MHz, CDCl_3) δ 162.9 (dd, $J = 253.3, 12.7$ Hz), 118.6 (t, $J = 11.5$ Hz), 116.7 (dd, $J = 21.7, 6.6$ Hz), 109.7 (t, $J = 25.0$ Hz), 91.4 (dt, $J = 8.1, 4.0$ Hz), 78.0 (d, $J = 62.0$ Hz); ^{19}F NMR (376 MHz, CDCl_3) δ 74.5, -106.5; HRMS (APCI): calculated for $\text{C}_8\text{H}_3\text{F}_3\text{O}_2\text{S}$ [M]: $m/z = 219.9806$, m/z found 219.9813.

4-Fluorophenylethynyl sulfonyl fluoride (2i)



Following general procedure A (2.30 mmol of the required alkyne), the title compound was isolated as a yellow oil (360 mg, 77%). ^1H NMR (400 MHz, CDCl_3) δ 7.78 – 7.64 (m, 2H), 7.23 – 7.13 (m, 2H); ^{13}C NMR (151 MHz, CDCl_3) δ 165.4 (d, $J = 257.8$ Hz), 136.2 (d, $J = 9.5$ Hz), 117.0 (d, $J = 22.6$ Hz), 112.2 (d, $J = 3.5$ Hz), 94.4 (d, $J = 8.2$ Hz), 77.0 (dd, $J = 60.9, 1.6$ Hz); ^{19}F NMR (376 MHz, CDCl_3) δ 74.3, -101.9; GC-MS (EI) calculated for $\text{C}_8\text{H}_4\text{F}_4\text{O}_2\text{S}$ [M] $^+$: Calculated $m/z = 201.99$, m/z found: 202.0

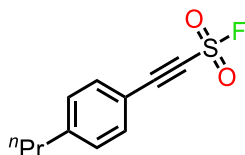
4-Trifluoromethylphenylethynyl sulfonyl fluoride (2j)



Following general procedure A (8.00 mmol of required alkyne), the title compound was isolated as a yellow solid (1.43 g, 71%). **m.p.** 74.6 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.82 (appt. d, $J = 8.1$ Hz, 2H), 7.75 (appt. d, $J = 8.1$ Hz,

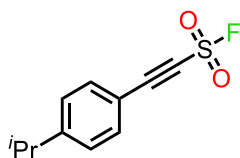
2H); $^{13}\text{C NMR}$ (151 MHz, CDCl_3) δ 134.6 (q, $J = 33.4$ Hz), 133.9, 126.2 (q, $J = 3.6$ Hz), 123.3 (q, $J = 273.0$ Hz), 119.8, 92.6 (d, $J = 8.2$ Hz), 78.4 (d, $J = 61.7$ Hz); $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ 74.5, -64.0; **HRMS** (APCI): calculated for $\text{C}_9\text{H}_4\text{F}_4\text{O}_2\text{S}$ $[\text{M}]^-$: $m/z = 251.9869$, HRMS found: 251.9868.

2-(4-Propylphenyl)ethyne-1-sulfonyl fluoride (2k)



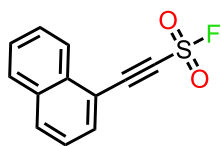
Following general procedure B, the title compound was isolated after purification by flash column chromatography (petroleum ether) as a yellow oil (1.31 g, 58%). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.60 (appt. d, $J = 8.4$ Hz, 2H), 7.29 (appt. d, $J = 8.5$ Hz, 2H), 2.70 – 2.61 (m, 2H), 1.74 – 1.60 (m, 2H), 0.96 (t, $J = 7.3$ Hz, 3H); $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 149.1, 133.6, 129.4, 113.1, 96.4 (d, $J = 8.1$ Hz), 76.7 (d, $J = 60.4$ Hz), 38.4, 24.2, 13.8. $^{19}\text{F NMR}$ (471 MHz, CDCl_3) δ 74.8; **HRMS** (ASAP): calculated for $\text{C}_{11}\text{H}_{12}\text{FO}_2\text{S}$ $[\text{M}+\text{H}]^+$: $m/z = 227.0542$, m/z found 227.0540; **IR** ν_{max} (ATR)/ cm^{-1} : 2964, 2874, 2187, 1605, 1422, 1206, 875, 785.

2-(4-isopropylphenyl)ethyne-1-sulfonyl fluoride (2l)



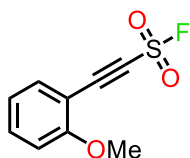
Following general procedure B, the title compound was isolated after purification by flash column chromatography (petroleum ether) as a yellow oil (1.48 g, 66%). $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.61 (appt. d, $J = 8.3$ Hz, 2H), 7.34 (appt. d, $J = 8.0$ Hz, 2H), 2.98 (hept, $J = 6.9$ Hz, 1H), 1.28 (d, $J = 6.9$ Hz, 7H); $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 155.1, 133.8, 127.5, 113.2, 96.4 (d, $J = 8.0$ Hz), 76.4, 34.7, 23.7; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ 74.8; **HRMS** (ASAP): calculated for $\text{C}_{11}\text{H}_{12}\text{FO}_2\text{S}$ $[\text{M}+\text{H}]^+$: $m/z = 227.0542$, m/z found 227.0544; **IR** ν_{max} (ATR)/ cm^{-1} : 2967, 2875, 2187, 1605, 1422, 1206, 874, 790.

2-(Naphthalen-1-yl)ethyne-1-sulfonyl fluoride (2m)



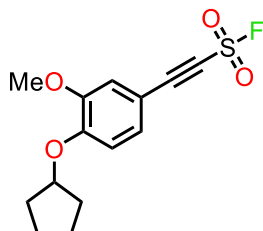
Following general procedure B, the title compound was isolated after purification by flash column chromatography (0-2% EtOAc in petroleum ether) as a brown solid (1.40 g, 60%). **m.p.** 39 - 40 °C; **¹H NMR** (500 MHz, CDCl₃) δ 8.21 – 8.18 (m, 1H), 8.11 (appt. d, *J* = 8.3 Hz, 1H), 7.98 (dd, *J* = 7.2, 1.2 Hz, 1H), 7.97 – 7.94 (m, 1H), 7.74 – 7.70 (m, 1H), 7.66 – 7.62 (m, 1H), 7.55 (dd, *J* = 8.3, 7.2 Hz, 1H); **¹³C NMR** (126 MHz, CDCl₃) δ 134.8, 134.1, 133.5, 133.0, 129.1, 128.9, 127.8, 125.2, 125.1, 113.4, 94.8 (d, *J* = 8.2 Hz), 81.3 (d, *J* = 60.9 Hz); **¹⁹F NMR** (376 MHz, CDCl₃) δ 75.2; **HRMS** (ASAP): calculated for C₁₂H₈FO₂S [M+H]: *m/z* = 234.0151, *m/z* found 234.0153; **IR** *v*_{max} (ATR)/cm⁻¹: 3060, 2182, 1416, 1202, 891, 797, 767.

2-(2-Methoxyphenyl)ethyne-1-sulfonyl fluoride (2n)



Following general procedure B, the title compound was isolated after purification by flash column chromatography (0-4% Et₂O in petroleum ether) as a low melting yellow solid (1.24 g, 58%). **¹H NMR** (500 MHz, CDCl₃) δ = 7.59 – 7.53 (m, 2H), 7.02 (td, *J* = 7.6, 0.8 Hz, 1H), 6.97 (d, *J* = 8.4 Hz, 1H), 3.94 (s, 3H); **¹³C NMR** (126 MHz, CDCl₃) δ = 162.9, 135.3, 135.0, 121.0, 111.3, 105.4, 93.8 (d, *J* = 8.1 Hz), 80.4 (d, *J* = 60.4 Hz), 56.1; **¹⁹F NMR** (376 MHz, CDCl₃) δ = 74.7; **HRMS** (ASAP): calculated for C₉H₈FO₃S [M+H]: *m/z* = 214.0100, *m/z* found 214.0098; **IR** *v*_{max} (ATR)/cm⁻¹: 2950, 2844, 2187, 1598, 1491, 1418, 1267, 1200, 1020, 880, 791, 752.

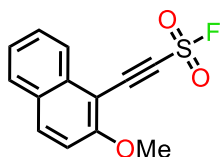
2-(4-(Cyclopentyloxy)-3-methoxyphenyl)ethyne-1-sulfonyl fluoride (2o)



Following general procedure B, the title compound was isolated after purification by flash column chromatography (0-5% EtOAc in petroleum ether) as a yellow solid (2.03 g, 69%). **m.p.** 59 - 61 °C; **¹H NMR** (500 MHz, CDCl₃) δ 7.30 (dd, *J* = 8.4, 1.8 Hz, 1H), 7.09 (d, *J* = 1.8 Hz, 1H), 6.89 (d, *J* = 8.4 Hz, 1H), 4.87 – 4.82 (m, 1H),

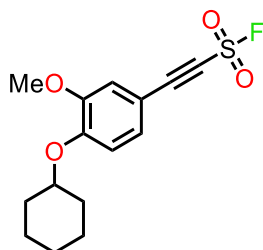
3.88 (s, 3H), 2.04 – 1.96 (m, 2H), 1.95 – 1.81 (m, 4H), 1.70 – 1.61 (m, 2H); ¹³C NMR (126 MHz, CDCl₃) δ 152.7, 149.9, 128.4, 115.7, 113.9, 106.7, 97.7 (d, *J* = 8.1 Hz), 81.0, 76.2 (d, *J* = 60.3 Hz), 56.3, 33.0, 24.3; ¹⁹F NMR (376 MHz, CDCl₃) δ 74.8; HRMS (ASAP): calculated for C₁₄H₁₆FO₄S [M+H]: *m/z* = 299.0753, *m/z* found 299.0754; IR ν_{max} (ATR)/cm⁻¹: 2966, 2952, 2170, 1592, 1507, 1415, 1212, 1054, 1025, 962, 799, 766.

2-(2-Methoxynaphthalen-1-yl)ethyne-1-sulfonyl fluoride (2p)



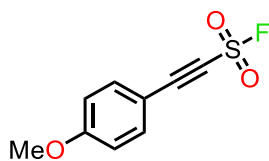
Following general procedure B, the title compound was isolated after purification by flash column chromatography (0-5% EtOAc in petroleum ether) as a yellow solid (1.54 g, 58%). **m.p.** 100 - 101 °C (decomposed); ¹H NMR (500 MHz, CDCl₃) δ 8.07 – 8.02 (m, 2H), 7.86 – 7.83 (m, 1H), 7.67 – 7.62 (m, 1H), 7.49 – 7.44 (m, 1H), 7.27 (d, *J* = 9.2 Hz, 1H), 4.08 (s, 3H); ¹³C NMR (126 MHz, CDCl₃) δ = 164.0, 136.1, 134.7, 129.6, 128.9, 128.1, 125.4, 124.0, 112.0, 98.6, 93.9 (d, *J* = 8.2 Hz), 85.6 (d, *J* = 60.1 Hz), 56.8; ¹⁹F NMR (471 MHz, CDCl₃) δ = 75.2; HRMS (ASAP): calculated for C₁₃H₁₀FO₃S [M+H]: *m/z* = 265.0335, *m/z* found 265.0335; IR ν_{max} (ATR)/cm⁻¹: 2928, 2854, 2167, 1589, 1508, 1408, 1263, 1214, 1187, 1098, 1056, 788, 753.

2-(4-(Cyclohexyloxy)-3-methoxyphenyl)ethyne-1-sulfonyl fluoride (2q)



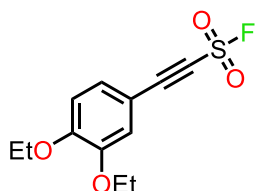
Following general procedure B, the title compound was isolated after purification by flash column chromatography (0-5% EtOAc in petroleum ether) as a yellow solid (2.07 g, 66%). **m.p.** 51 - 52 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.29 (dd, *J* = 8.4, 1.9 Hz, 1H), 7.10 (d, *J* = 1.9 Hz, 1H), 6.92 (d, *J* = 8.5 Hz, 1H), 4.39 – 4.30 (m, 1H), 3.89 (s, 3H), 2.09 – 1.99 (m, 2H), 1.90 – 1.81 (m, 2H), 1.67 – 1.58 (m, 3H), 1.45 – 1.29 (m, 3H); ¹³C NMR (126 MHz, CDCl₃) δ 152.3, 150.2, 128.4, 116.0, 114.2, 106.9, 97.6 (d, *J* = 8.1 Hz), 77.2, 76.2 (d, *J* = 60.3 Hz), 56.4, 31.7, 25.5, 24.0; ¹⁹F NMR (376 MHz, CDCl₃) δ 74.8; HRMS (ASAP): calculated for C₁₅H₁₈FO₄S [M+H]: *m/z* = 313.0910, *m/z* found 313.0910; IR ν_{max} (ATR)/cm⁻¹: 3091, 2939, 2857, 2162, 1590, 1507, 1414, 1275, 1245, 1212, 1141, 960, 815.

2-(4-Methoxyphenyl)ethyne-1-sulfonyl fluoride (2r)



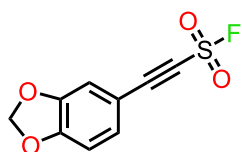
Following general procedure B, the title compound was isolated after purification by flash column chromatography (0-2% EtOAc in petroleum ether) as a yellow solid (1.26 g, 59%). **m.p.** 41 - 43 °C; **¹H NMR** (500 MHz, CDCl₃) δ = 7.63 (d, *J* = 9.0 Hz, 2H), 6.97 (d, *J* = 9.0 Hz, 2H), 3.89 (s, 3H); **¹³C NMR** (126 MHz, CDCl₃) δ = 163.5, 135.8, 115.0, 107.5, 97.1 (d, *J* = 8.1 Hz), 76.6 (d, *J* = 60.4 Hz), 55.8; **¹⁹F NMR** (471 MHz, CDCl₃) δ = 74.8; **HRMS** (ASAP): calculated for C₉H₈FO₃S [M+H]: *m/z* = 214.0100, *m/z* found 214.0096; **IR** *v*_{max} (ATR)/cm⁻¹: 2930, 2851, 2184, 1602, 1510, 1418, 1254, 1205, 1172, 877, 795.

2-(3,4-Diethoxyphenyl)ethyne-1-sulfonyl fluoride (2s)



Following general procedure B, the title compound was isolated after purification by flash column chromatography (0-4% EtOAc in petroleum ether) as a beige solid (1.84 g, 68%). **m.p.** 53 - 54 °C; **¹H NMR** (500 MHz, CDCl₃) δ = 7.30 (dd, *J* = 8.4, 1.9 Hz, 1H), 7.10 (d, *J* = 1.9 Hz, 1H), 6.89 (d, *J* = 8.4 Hz, 1H), 4.17 (q, *J* = 7.0 Hz, 2H), 4.11 (q, *J* = 7.0 Hz, 2H), 1.49 (appt. q, *J* = 7.0 Hz, 6H); **¹³C NMR** (126 MHz, CDCl₃) δ = 153.5, 148.8, 128.5, 116.9, 112.6, 107.1, 97.6 (d, *J* = 8.1 Hz), 76.2 (d, *J* = 60.4 Hz), 65.0, 64.9, 14.7, 14.7; **¹⁹F NMR** (376 MHz, CDCl₃) δ = 74.8; **HRMS** (ASAP): calculated for C₁₂H₁₄FO₄S [M+H]: *m/z* = 273.0597, *m/z* found 273.0597; **IR** *v*_{max} (ATR)/cm⁻¹: 2978, 2934, 2167, 1592, 1514, 1409, 1247, 1209, 1138, 1039, 806.

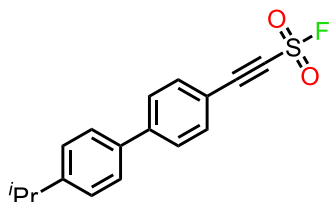
2-(Benzo[d][1,3]dioxol-5-yl)ethyne-1-sulfonyl fluoride (2t)



Following general procedure B, the title compound was isolated after purification by flash column chromatography (0-4% EtOAc in petroleum ether) as a brown solid (1.21 g, 60%). **m.p.** 82 - 84 °C; **¹H NMR** (500 MHz, CDCl₃) δ = 7.27 (dd, *J* = 8.1, 1.6 Hz, 1H), 7.05 (d, *J* = 1.6 Hz, 1H), 6.89 (d, *J* = 8.1 Hz, 1H), 6.10 (s, 2H); **¹³C NMR** (126 MHz, CDCl₃) δ = 152.2, 148.2, 130.4, 112.5, 109.4, 108.7, 102.5, 96.5 (d, *J* = 8.1 Hz), 76.0 (d, *J* = 60.7 Hz); **¹⁹F**

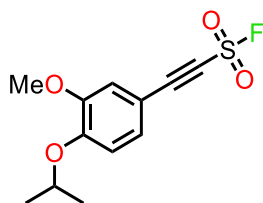
NMR (376 MHz, CDCl₃) δ = 74.8; **HRMS** (ASAP): calculated for C₉H₆FO₄S [M+H]: m/z = 228.9971, m/z found 228.9962; **IR** ν_{\max} (ATR)/cm⁻¹: 2928, 2181, 1614, 1477, 1417, 1256, 1201, 1035, 828, 775.

2-(4'-isopropyl-[1,1'-biphenyl]-4-yl)ethyne-1-sulfonyl fluoride (2u)



Following general procedure B, the title compound was isolated after purification by flash column chromatography (petroleum ether) as a yellow solid (1.40 g, 54%). **m.p.** 82 - 83 °C; **¹H NMR** (500 MHz, CDCl₃) δ = 7.74 (appt. d, *J* = 8.6 Hz, 2H), 7.70 (appt. d, *J* = 8.6 Hz, 2H), 7.55 (appt. d, *J* = 8.2 Hz, 2H), 7.31 (appt. d, *J* = 8.3 Hz, 2H), 2.69 – 2.64 (m, 2H), 1.75 – 1.66 (m, 2H), 0.99 (t, *J* = 7.3 Hz, 3H); **¹³C NMR** (126 MHz, CDCl₃) δ = 146.0, 143.9, 136.6, 134.1, 129.4, 127.5, 127.2, 114.1, 96.0 (d, *J* = 8.1 Hz), 77.7, 37.9, 24.6, 14.0; **¹⁹F NMR** (470 MHz, CDCl₃) δ = 74.9; **HRMS** (ASAP): calculated for C₁₇H₁₆FO₂S [M+H]: m/z = 303.0855, m/z found 303.0854; **IR** ν_{\max} (ATR)/cm⁻¹: 2954, 2870, 2187, 1599, 1418, 1204, 878, 781.

2-(4-isopropoxy-3-methoxyphenyl)ethyne-1-sulfonyl fluoride (2v)

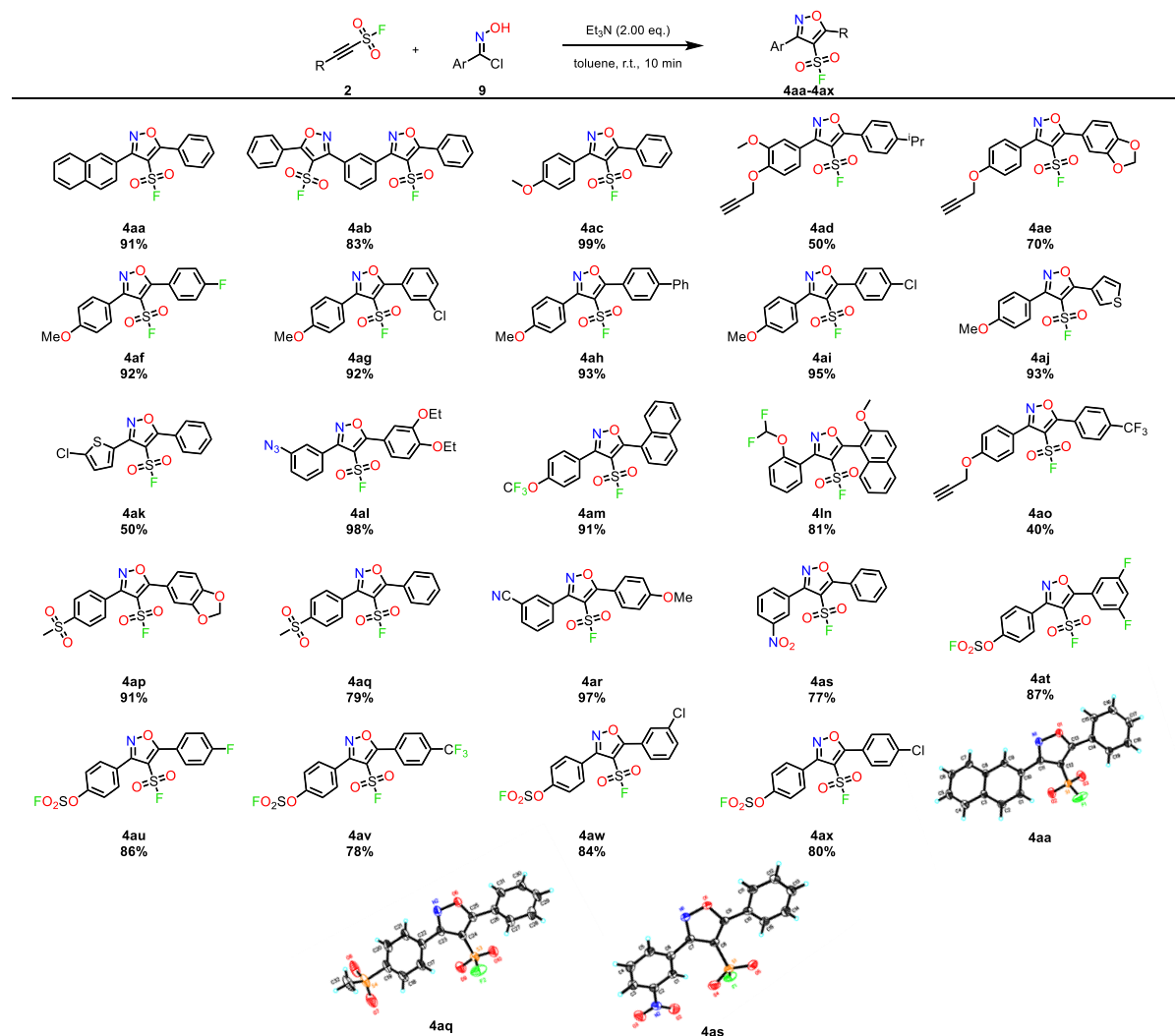


Following general procedure B, the title compound was isolated after purification by flash column chromatography (0-5% EtOAc in petroleum ether) as a yellow solid (1.95 g, 72%). **m.p.** 70 - 71 °C; **¹H NMR** (500 MHz, CDCl₃) δ 7.31 (dd, *J* = 8.4, 1.8 Hz, 1H), 7.10 (d, *J* = 1.8 Hz, 1H), 6.91 (d, *J* = 8.4 Hz, 1H), 4.67 (hept, *J* = 6.1 Hz, 1H), 3.89 (s, 3H), 1.43 (d, *J* = 6.1 Hz, 6H); **¹³C NMR** (126 MHz, CDCl₃) δ 152.3, 150.1, 128.4, 115.8, 113.9, 106.9, 97.5 (d, *J* = 8.1 Hz), 76.2 (d, *J* = 60.5 Hz), 71.7, 56.3, 22.0; **¹⁹F NMR** (376 MHz, CDCl₃) δ 74.8; **HRMS** (ASAP): calculated for C₁₂H₁₄FO₄S [M+H]: m/z = 273.0597, m/z found 273.0595; **IR** ν_{\max} (ATR)/cm⁻¹: 3022, 2906, 2169, 1592, 1506, 1417, 1272, 1247, 1211, 1141, 1107, 947, 850, 500, 770.

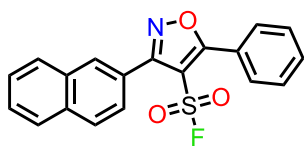
Synthesis and experimental data for compounds 4aa-4ax

General procedure C

To a solution of the required SASF **2** (500 μ mol) and the required imidoyl chloride **9** (500 μ mol) in toluene (1.50 mL) was added Et₃N (139 μ L, 1.00 mmol) and stirred at room temperature for 10 min. The reaction mixture was then extracted into EtOAc (10.0 mL) washed with brine (10.0 mL) and H₂O (2 x 10.0 mL). The aqueous layer was extracted with EtOAc (10.0 mL), the organic fractions combined, dried over anhydrous MgSO₄, filtered under vacuum and the solvent was removed under reduced pressure to obtain the analytically pure product.

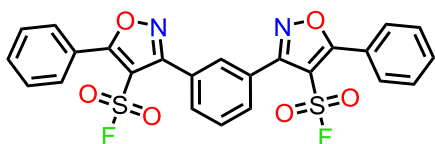


3-(Naphthalen-2-yl)-5-phenylisoxazole-4-sulfonyl fluoride (4aa)



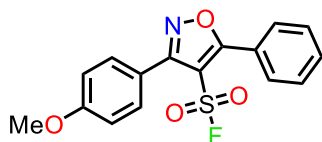
Following general procedure C, the title compound was isolated as a colourless solid (161 mg, 81%). **m.p.** 140 - 142 °C; **¹H NMR** (400 MHz, CDCl₃) δ 8.27 (s, 1H), 8.01 (d, *J* = 9.1 Hz, 1H), 8.00 – 7.90 (m, 4H), 7.79 (dd, *J* = 8.5, 1.8 Hz, 1H), 7.72 – 7.66 (m, 1H), 7.65 – 7.57 (m, 4H); **¹³C NMR** (101 MHz, CDCl₃) δ 175.8 (d, *J* = 2.9 Hz), 161.9, 134.4, 133.2, 132.9, 129.9, 129.8, 129.1, 128.9, 128.7, 128.0, 127.9, 127.1, 125.7, 124.6, 123.2, 110.1 (d, *J* = 34.8 Hz); **¹⁹F NMR** (376 MHz, CDCl₃) δ = 72.0; **HRMS** (ESI⁺): calculated for C₁₉H₁₂FNO₃SNa [M+Na⁺]: *m/z* = 376.0414, *m/z* found 376.0415; **IR** *v*_{max} (ATR)/cm⁻¹: 2923, 2863, 1498, 1551, 1417, 1215, 1056, 751.

3-(3-(4-(Fluorosulfonyl)-5-phenylisoxazol-3-yl)phenyl)-5-phenylisoxazole-4-sulfonyl fluoride (4ab)



Following general procedure C (1.50 mL of toluene used, 250 μmol of the required imidoyl chloride), the title compound was isolated as a colourless solid (110 mg, 83%). **m.p.** 185 - 186 °C; **¹H NMR** (500 MHz, CDCl₃) δ 8.12 (appt. t, *J* = 1.5 Hz, 1H), 7.96 (dd, *J* = 7.8, 1.8 Hz, 2H), 7.93 – 7.88 (m, 4H), 7.74 (t, *J* = 7.8 Hz, 1H), 7.71 – 7.67 (m, 2H), 7.64 – 7.58 (m, 4H); **¹³C NMR** (126 MHz, CDCl₃) δ = 176.0 (d, *J* = 2.9 Hz), 160.9, 133.3, 132.0, 130.2, 129.8, 129.4, 129.1, 126.8, 124.4, 110.0 (d, *J* = 35.2 Hz); **¹⁹F NMR** (376 MHz, CDCl₃) δ = 72.4; **HRMS** (ESI⁺): calculated for C₂₄H₁₄F₂N₂O₆S₂Na [M+Na⁺]: *m/z* = 551.0154, *m/z* found 551.0143; **IR** *v*_{max} (ATR)/cm⁻¹: 3067, 1551, 1420, 1217, 771.

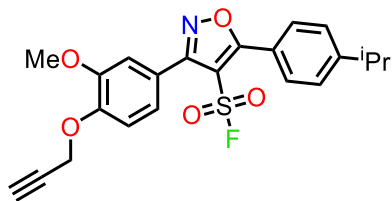
3-(4-Methoxyphenyl)-5-phenylisoxazole-4-sulfonyl fluoride (4ac)



Following general procedure C (250 μmol of the required SASF and the required imidoyl chloride used), the title compound was isolated as a colourless solid (82.0 mg, 99%). **m.p.** 104 - 106; **¹H NMR** (400 MHz, CDCl₃) δ 7.89 – 7.84 (m, 2H), 7.72 – 7.64 (m, 3H), 7.62 – 7.56 (m, 2H), 7.06 (appt. d, *J* = 8.9 Hz, 2H), 3.90 (s, 3H); **¹³C NMR** (101 MHz, CDCl₃) δ 175.8 (d, *J* = 3.1 Hz), 162.0, 161.4, 133.0, 130.9, 129.8, 129.0, 124.7, 118.0, 114.4, 109.8 (d, *J* = 34.5 Hz), 55.6; **¹⁹F NMR** (376 MHz, CDCl₃) δ 72.0; **HRMS** (ASAP): calculated for C₁₆H₁₃FNO₄S [M+H]: *m/z* =

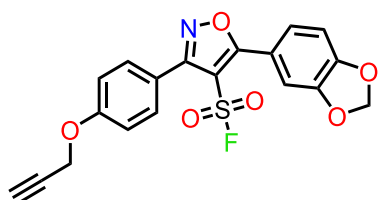
334.0549, m/z found 334.0547; IR ν_{\max} (ATR)/ cm^{-1} : 2922, 2863, 1603, 1550, 1523, 1414, 1381, 1258, 1216, 1017, 833, 764.

5-(4-Isopropylphenyl)-3-(3-methoxy-4-(prop-2-yn-1-yloxy)phenyl)isoxazole-4-sulfonyl fluoride (4ad)



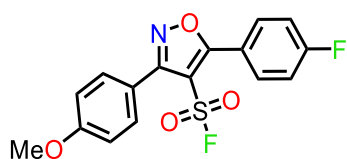
Following general procedure C, the title compound was isolated after purification by flash column chromatography (0-10% EtOAc in petroleum ether) as a colourless solid (108 mg, 50%). **m.p.** 103 - 104 °C; **^1H NMR** (500 MHz, CDCl_3) δ = 7.80 (d, J = 8.4 Hz, 2H), 7.44 (d, J = 8.4 Hz, 2H), 7.35 (dd, J = 8.3, 2.1 Hz, 1H), 7.30 (d, J = 2.1 Hz, 1H), 7.18 (d, J = 8.3 Hz, 1H), 4.86 (d, J = 2.4 Hz, 2H), 3.96 (s, 3H), 3.03 (hept, J = 6.9 Hz, 1H), 2.57 (t, J = 2.4 Hz, 1H), 1.32 (d, J = 6.9 Hz, 6H); **^{13}C NMR** (126 MHz, CDCl_3) δ = 176.0 (d, J = 3.0 Hz), 161.3, 154.7, 149.6, 149.3, 129.9, 127.2, 122.3, 122.0, 119.4, 113.7, 112.6, 109.2 (d, J = 34.5 Hz), 78.1, 76.5, 56.8, 56.2, 34.5, 23.8; **^{19}F NMR** (471 MHz, CDCl_3) δ = 71.3; **HRMS** (ESI⁺): calculated for $\text{C}_{22}\text{H}_{20}\text{FNO}_5\text{SNa}$ [$\text{M}+\text{Na}^+$]: m/z = 452.0938, m/z found 452.0936; IR ν_{\max} (ATR)/ cm^{-1} : 3264, 2963, 2927, 2855, 1606, 1498, 1430, 1258, 1218, 1139, 1007, 754.

5-(Benzo[d][1,3]dioxol-5-yl)-3-(4-(prop-2-yn-1-yloxy)phenyl)isoxazole-4-sulfonyl fluoride (4ae)



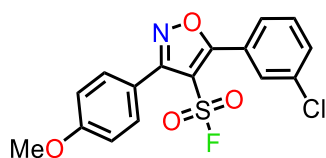
Following general procedure C, the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a colourless solid (140 mg, 70%). **m.p.** 154 - 156 °C; **^1H NMR** (500 MHz, CDCl_3) δ = 7.68 (appt. d, J = 8.9 Hz, 2H), 7.46 (dd, J = 8.2, 1.8 Hz, 1H), 7.32 (d, J = 1.8 Hz, 1H), 7.13 (d, J = 8.9 Hz, 2H), 7.00 (d, J = 8.2 Hz, 1H), 6.13 (s, 2H), 4.78 (d, J = 2.4 Hz, 2H), 2.58 (t, J = 2.4 Hz, 1H); **^{13}C NMR** (126 MHz, CDCl_3) δ = 175.2 (d, J = 2.7 Hz), 161.4, 159.9, 151.9, 148.4, 130.9, 125.7, 119.0, 118.0, 115.2, 109.5, 109.0, 108.7 (d, J = 34.5 Hz), 102.4, 78.1, 76.2, 56.0; **^{19}F NMR** (376 MHz, CDCl_3) δ = 71.8; **HRMS** (ESI⁺): calculated for $\text{C}_{19}\text{H}_{12}\text{FNO}_6\text{SNa}$ [$\text{M}+\text{Na}^+$]: m/z = 424.0262, m/z found 424.0264; IR ν_{\max} (ATR)/ cm^{-1} : 3283, 2923, 2854, 2119, 1607, 1555, 1490, 1460, 1427, 1251, 1226, 1030, 759.

5-(4-Fluorophenyl)-3-(4-methoxyphenyl)isoxazole-4-sulfonyl fluoride (4af)



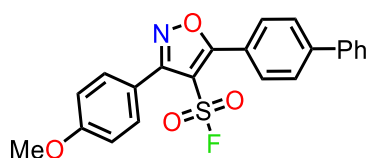
Following general procedure C (100 μ mol of the required SASF and the required imidoyl chloride used), the title compound was isolated as a white solid (32.0 mg, 92%). **m.p.** 121 $^{\circ}$ C; **1 H NMR** (400 MHz, CDCl_3) δ 7.97 – 7.83 (m, 2H), 7.67 (appt. d, J = 8.8 Hz, 2H), 7.36 – 7.23 (m, 2H), 7.04 (appt. d, J = 8.8 Hz, 2H), 3.89 (s, 3H); **13 C NMR** (151 MHz, CDCl_3) δ 174.7 (d, J = 3.0 Hz), 165.6 (d, J = 256.0 Hz), 162.0, 161.5, 132.3 (d, J = 9.3 Hz), 130.9, 120.9 (d, J = 3.4 Hz), 117.8, 116.5 (d, J = 22.3 Hz), 114.4, 109.7 (d, J = 34.6 Hz), 55.6; **19 F NMR** (376 MHz, CDCl_3) δ 71.3, -104.9; **HRMS** (APCI): calculated for $\text{C}_{16}\text{H}_{12}\text{F}_2\text{NO}_4\text{S}$ [$\text{M}+\text{H}^+$]: m/z = 352.0455, m/z found 352.0458

5-(3-Chlorophenyl)-3-(4-methoxyphenyl)isoxazole-4-sulfonyl fluoride (4ag)



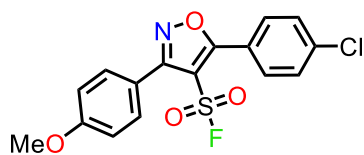
Following general procedure C (100 μ mol of the required SASF and the required imidoyl chloride used), the title compound was isolated as a pale yellow solid (34.0 mg, 92%). **m.p.** 127.8 $^{\circ}$ C; **1 H NMR** (400 MHz, CDCl_3) δ 7.83 (s, 1H), 7.74 (d, J = 7.8 Hz, 1H), 7.68 (appt. d, J = 8.2 Hz, 2H), 7.63 (d, J = 8.1 Hz, 1H), 7.53 (t, J = 7.8 Hz, 1H), 7.05 (appt. d, J = 8.2 Hz, 2H), 3.89 (s, 3H); **13 C NMR** (151 MHz, CDCl_3) δ 174.1 (d, J = 2.9 Hz), 162.1, 161.4, 135.2, 133.1, 130.9, 130.3, 129.6, 128.0, 126.2, 117.7, 114.5, 110.5 (d, J = 34.8 Hz), 55.6; **19 F NMR** (376 MHz, CDCl_3) δ 71.3; **HRMS** (APCI): calculated for $\text{C}_{16}\text{H}_{12}\text{ClFNO}_4\text{S}$ [$\text{M}+\text{H}^+$]: m/z = 368.0160, m/z found 368.0154

5-([1,1'-Biphenyl]-4-yl)-3-(4-methoxyphenyl)isoxazole-4-sulfonyl fluoride (4ah)



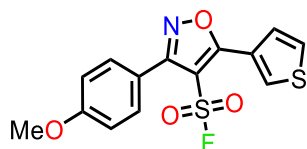
Following general procedure C (100 μ mol of the required SASF and the required imidoyl chloride used), the title compound was isolated as a white solid (38.0 mg, 93%). **m.p.** 173 $^{\circ}$ C; **1 H NMR** (400 MHz, CDCl_3) δ 7.95 (appt. d, J = 8.5 Hz, 2H), 7.80 (appt. d, J = 8.5 Hz, 2H), 7.73 – 7.63 (m, 4H), 7.55 – 7.46 (m, 2H), 7.48 – 7.39 (m, 1H), 7.05 (appt. d, J = 8.8 Hz, 2H), 3.89 (s, 3H); **13 C NMR** (151 MHz, CDCl_3) δ 175.5 (d, J = 2.8 Hz), 162.0, 161.5, 145.9, 139.6, 130.9, 130.3, 129.2, 128.7, 127.6, 127.4, 123.3, 118.0, 114.4, 109.5 (d, J = 34.5 Hz), 55.6; **19 F NMR** (376 MHz, CDCl_3) δ 71.2; **HRMS** (APCI): calculated for $\text{C}_{22}\text{H}_{17}\text{FNO}_4\text{S}$ [$\text{M}+\text{H}^+$]: m/z = 410.0862, m/z found 410.0862

5-(4-Chlorophenyl)-3-(4-methoxyphenyl)isoxazole-4-sulfonyl fluoride (4ai)



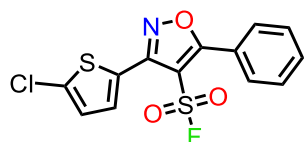
Following general procedure C (100 μ mol of the required SASF and the required imidoyl chloride used), the title compound was isolated as a pale yellow solid (35.0 mg, 95%). **m.p.** 105.5 $^{\circ}$ C; **1 H NMR** (400 MHz, CDCl_3) δ 7.80 (appt. d, J = 8.5 Hz, 2H), 7.67 (appt. d, J = 8.4 Hz, 2H), 7.57 (appt. d, J = 8.5 Hz, 2H), 7.04 (appt. d, J = 8.4 Hz, 2H), 3.89 (s, 3H); **13 C NMR** (151 MHz, CDCl_3) δ 174.6 (d, J = 3.0 Hz), 162.1, 161.5, 139.7, 131.1, 130.9, 129.5, 123.0, 117.8, 114.5, 110.0 (d, J = 34.8 Hz), 55.6; **19 F NMR** (376 MHz, CDCl_3) δ 71.3; **HRMS** (APCI): calculated for $\text{C}_{16}\text{H}_{12}\text{ClFNO}_4\text{S}$ [$\text{M}+\text{H}^+$]: m/z = 368.0160, m/z found 368.0166;

3-(4-Methoxyphenyl)-5-(thiophen-3-yl)isoxazole-4-sulfonyl fluoride (4aj)



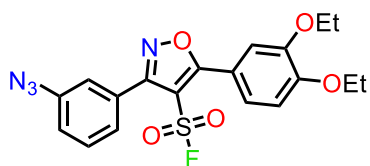
Following general procedure C (100 μ mol of the required SASF and the required imidoyl chloride used), the title compound was isolated as a yellow solid (31.5 mg, 93%). **m.p.** 93.7 $^{\circ}$ C; **1 H NMR** (400 MHz, CDCl_3) δ 8.34 (dd, J = 3.0, 1.3 Hz, 1H), 7.71 (dd, J = 5.2, 1.3 Hz, 1H), 7.63 (appt. d, J = 8.8 Hz, 2H), 7.51 (dd, J = 5.2, 3.0 Hz, 1H), 7.03 (appt. d, J = 8.9 Hz, 2H), 3.88 (s, 3H); **13 C NMR** (151 MHz, CDCl_3) δ 170.2 (d, J = 2.4 Hz), 161.9, 161.5, 132.5, 131.0, 127.6, 127.2, 125.0, 117.9, 114.3, 108.08 (d, J = 34.1 Hz), 55.5; **19 F NMR** (376 MHz, CDCl_3) δ 71.0; **HRMS** (APCI): calculated for $\text{C}_{14}\text{H}_{11}\text{FNO}_4\text{S}_2$ [$\text{M}+\text{H}^+$]: m/z = 340.0114, m/z found 340.0118;

3-(5-Chlorothiophen-2-yl)-5-phenylisoxazole-4-sulfonyl fluoride (4ak)



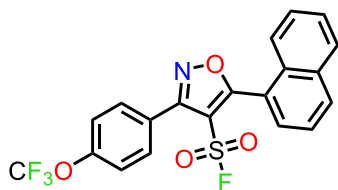
Following general procedure C, the title compound was isolated after purification by flash column chromatography (0-5% EtOAc in petroleum ether) as a yellow solid (134 mg, 77%). **m.p.** 94 - 96 $^{\circ}$ C; **1 H NMR** (400 MHz, CDCl_3) δ 7.83 - 7.78 (m, 2H), 7.70 - 7.65 (m, 1H), 7.63 (dd, J = 4.1, 0.6 Hz, 1H), 7.62 - 7.56 (m, 2H), 7.05 (d, J = 4.1 Hz, 1H); **13 C NMR** (101 MHz, CDCl_3) δ 176.6 (d, J = 3.6 Hz), 154.9, 135.3, 133.2, 131.4 (d, J = 1.7 Hz), 129.9, 129.0, 127.6, 124.2, 124.1, 108.9 (d, J = 35.7 Hz); **19 F NMR** (376 MHz, CDCl_3) δ 70.4; **HRMS** (ESI $^+$): calculated for $\text{C}_{13}\text{H}_8\text{ClFNO}_3\text{S}_2$ [$\text{M}+\text{H}^+$]: m/z = 343.9613, m/z found 343.9605; **IR** ν max (ATR)/ cm^{-1} : 3041, 2889, 2853, 1560, 1421, 1218, 1078, 1039, 770, 725.

3-(3-Azidophenyl)-5-(3,4-diethoxyphenyl)isoxazole-4-sulfonyl fluoride (4al)



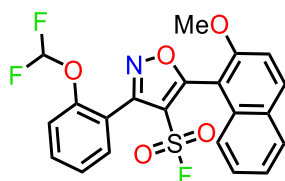
Following general procedure C, the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a beige solid (212 mg, 98%). **m.p.** 100 - 101 °C; **¹H NMR** (500 MHz, CDCl₃) δ = 7.57 – 7.49 (m, 2H), 7.47 – 7.44 (m, 1H), 7.43 (d, *J* = 2.2 Hz, 2H), 7.35 – 7.33 (m, 1H), 7.23 (ddd, *J* = 8.0, 2.3, 1.1 Hz, 1H), 7.03 (d, *J* = 8.5 Hz, 1H), 4.25 – 4.16 (m, 4H), 1.55 – 1.49 (m, 6H); **¹³C NMR** (126 MHz, CDCl₃) δ = 175.4 (d, *J* = 2.5 Hz), 161.2, 153.3, 148.7, 140.9, 130.2, 127.9, 125.9, 123.9, 121.6, 120.0, 116.1, 113.8, 112.4, 108.2 (d, *J* = 34.5 Hz), 65.0, 64.8, 14.8, 14.7; **¹⁹F NMR** (376 MHz, CDCl₃) δ = 72.1; **HRMS** (ESI⁺): calculated for C₁₉H₁₇FN₄O₅Na [M+Na⁺]: *m/z* = 455.0796, *m/z* found 455.0799; **IR** *v*_{max} (ATR)/cm⁻¹: 2987, 2933, 2892, 2099, 1599, 1517, 1420, 1272, 1209, 1136, 1039, 762.

5-(Naphthalen-1-yl)-3-(4-(trifluoromethoxy)phenyl)isoxazole-4-sulfonyl fluoride (4am)



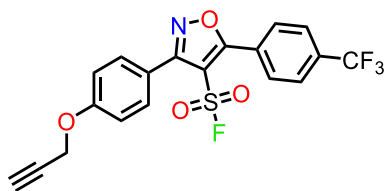
Following general procedure C (3.00 mL of toluene used), the title compound was isolated as a yellow solid (183 mg, 81%). **m.p.** 125 - 126 °C; **¹H NMR** (500 MHz, CDCl₃) δ = 8.17 – 8.14 (m, 1H), 8.02 – 7.98 (m, 1H), 7.94 – 7.90 (m, 2H), 7.81 – 7.76 (m, 2H), 7.68 – 7.62 (m, 3H), 7.46 – 7.42 (m, 2H); **¹³C NMR** (126 MHz, CDCl₃) δ = 176.8 (d, *J* = 3.2 Hz), 160.0, 151.6 – 151.5 (m), 133.6, 133.3, 131.2, 130.6 – 130.2 (m), 130.1, 129.0, 128.3, 127.2, 124.9, 124.4, 124.3, 121.67, 121.2, 120.5 (q, *J* = 258.8 Hz), 112.9 (d, *J* = 35.0 Hz); **¹⁹F NMR** (376 MHz, CDCl₃) δ = 71.8, -57.6; **HRMS** (ASAP): calculated for C₂₀H₁₂F₄NO₄S [M+H]: *m/z* = 438.0423, *m/z* found 438.0421; **IR** *v*_{max} (ATR)/cm⁻¹: 2967, 2937, 1601, 1559, 1431, 1254, 1217, 1159, 1055, 772.

3-(2-(Difluoromethoxy)phenyl)-5-(2-methoxynaphthalen-1-yl)isoxazole-4-sulfonyl fluoride (4an)



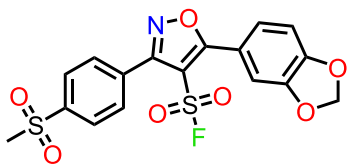
Following general procedure C (3.00 mL of toluene used), the title compound was isolated as a beige solid (200 mg, 91%). **m.p.** 90 - 91 °C; **¹H NMR** (500 MHz, CDCl₃) δ = 8.12 (d, *J* = 9.1 Hz, 1H), 7.89 (appt. d, *J* = 8.1 Hz, 1H), 7.70 (dd, *J* = 7.6, 1.7 Hz, 1H), 7.64 – 7.60 (m, 1H), 7.61 – 7.54 (m, 1H), 7.54 – 7.51 (m, 1H), 7.46 (ddd, *J* = 8.1, 6.6, 1.3 Hz, 1H), 7.42 (appt. td, *J* = 7.6, 1.1 Hz, 1H), 7.39 (d, *J* = 9.2 Hz, 1H), 7.38 – 7.33 (m, 1H), 6.59 (t, *J* = 73.3 Hz, 1H), 4.01 (s, 3H); **¹³C NMR** (126 MHz, CDCl₃) δ = 173.6 (d, *J* = 2.4 Hz), 157.8, 156.9, 149.8 (t, *J* = 2.7 Hz), 135.0, 132.7, 132.7, 131.7, 128.8, 128.7, 128.6, 125.6, 124.7, 123.3, 119.0, 118.7, 116.2 (t, *J* = 260.9 Hz), 114.9 (d, *J* = 33.8 Hz), 112.4, 106.6, 56.4; **¹⁹F NMR** (376 MHz, CDCl₃) δ = 67.2, -81.1 (t, *J* = 78.7 Hz); **HRMS** (ESI⁺): calculated for C₂₁H₁₄F₃NO₅Na [M+Na⁺]: *m/z* = 472.0437, *m/z* found 472.0437; **IR** *v*_{max} (ATR)/cm⁻¹: 2926, 2852, 15993, 1558, 1509, 1456, 1415, 1267, 1127, 1052, 779.

3-(4-(Prop-2-yn-1-yloxy)phenyl)-5-(4-(trifluoromethyl)phenyl)isoxazole-4-sulfonyl fluoride (4ao)



Following general procedure C (100 μmol of the required SASF and the required imidoyl chloride used), the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a colourless solid (17.0 mg, 40%). **m.p.** 124 – 125 °C; **¹H NMR** (500 MHz, CDCl₃) δ 7.99 (d, *J* = 8.2 Hz, 2H), 7.87 (d, *J* = 8.2 Hz, 2H), 7.72 (appt. d, *J* = 8.9 Hz, 2H), 7.15 (appt. d, *J* = 8.9 Hz, 2H), 4.79 (d, *J* = 2.4 Hz, 2H), 2.59 (t, *J* = 2.4 Hz, 1H); **¹³C NMR** (126 MHz, CDCl₃) δ 174.1 (d, *J* = 3.1 Hz), 161.4, 160.1, 134.6 (q, *J* = 33.2 Hz), 130.9, 130.3, 127.9, 126.1 (q, *J* = 3.7 Hz), 123.5 (q, *J* = 272.9 Hz), 118.5, 115.4, 111.0 (d, *J* = 35.1 Hz), 78.0, 76.3, 56.1; **¹⁹F NMR** (376 MHz, CDCl₃) δ 71.9, -63.3; **HRMS** (ESI⁺): calculated for C₁₉H₁₁F₄NO₄Na [M+Na⁺]: *m/z* = 448.0237, *m/z* found 448.0239; **IR** *v*_{max} (ATR)/cm⁻¹: 3280, 2898, 2133, 1608, 1564, 1432, 1320, 1244, 1223, 1181, 1140, 1069, 1021, 837, 770.

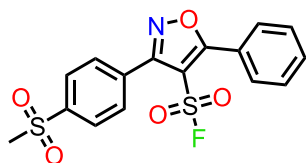
5-(Benzo[d][1,3]dioxol-5-yl)-3-(4-(methylsulfonyl)phenyl)isoxazole-4-sulfonyl fluoride (4p)



Following general procedure C, the title compound was isolated after washing with Et₂O as a yellow solid (193 mg, 91%). **m.p.** 180 °C (decomposition); **¹H NMR** (500 MHz, CDCl₃) δ = 8.12 (appt. d, *J* = 8.7 Hz, 2H), 7.91 (appt. d, *J* = 8.7 Hz, 2H), 7.50 (dd, *J* = 8.2, 1.8 Hz, 1H), 7.35 (d, *J* = 1.8 Hz, 1H), 7.02 (d, *J* = 8.2 Hz, 1H), 6.15 (s, 2H), 3.14 (s, 3H); **¹³C NMR** (126 MHz, CDCl₃) δ = 175.6 (d, *J* = 2.4 Hz), 160.6, 152.3, 148.5, 143.0, 131.5, 130.6, 127.9, 125.9,

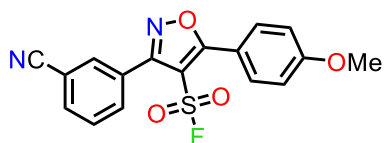
117.4, 109.4, 109.1, 108.7 (d, $J = 35.2$ Hz), 102.5, 44.6; ^{19}F NMR (376 MHz, CDCl_3) $\delta = 72.8$; HRMS (ESI $^+$): calculated for $\text{C}_{17}\text{H}_{12}\text{FNO}_7\text{S}_2\text{Na}$ [$\text{M}+\text{Na}^+$]: $m/z = 447.9931$, m/z found 447.9936; IR ν_{max} (ATR)/ cm^{-1} : 3015, 2359, 1556, 1486, 1420, 1311, 1264, 1244, 1214, 1149, 1034, 779.

3-(4-(Methylsulfonyl)phenyl)-5-phenylisoxazole-4-sulfonyl fluoride (4aq)



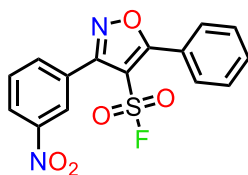
Following general procedure C, the title compound was isolated after washing with Et_2O as a yellow solid (151 mg, 79%). **m.p.** 139 - 141 $^\circ\text{C}$; ^1H NMR (500 MHz, CDCl_3) $\delta = 8.14$ (appt. d, $J = 8.7$ Hz, 2H), 7.95 (appt. d, $J = 8.7$ Hz, 2H), 7.92 - 7.89 (m, 2H), 7.73 - 7.68 (m, 1H), 7.65 - 7.60 (m, 2H), 3.14 (s, 4H); ^{13}C NMR (126 MHz, CDCl_3) $\delta = 176.2$ (d, $J = 2.8$ Hz), 160.4, 143.0, 133.5, 131.4, 130.6, 129.8, 129.2, 127.9, 124.0, 109.9 (d, $J = 35.4$ Hz), 44.6; ^{19}F NMR (376 MHz, CDCl_3) $\delta = 72.9$; HRMS (ESI $^+$): calculated for $\text{C}_{16}\text{H}_{12}\text{FNO}_5\text{S}_2\text{Na}$ [$\text{M}+\text{Na}^+$]: $m/z = 404.0033$, m/z found 404.0031; IR ν_{max} (ATR)/ cm^{-1} : 3074, 1606, 1557, 1424, 1306, 1219, 1146, 778.

3-(3-Cyanophenyl)-5-(4-methoxyphenyl)isoxazole-4-sulfonyl fluoride (4ar)



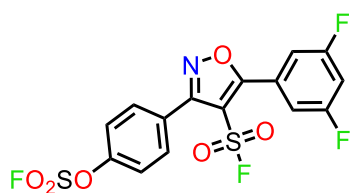
Following general procedure C, the title compound was isolated as a yellow solid (174 mg, 97%) ^1H NMR (400 MHz, CDCl_3) $\delta = 7.99$ (appt. td, $J = 1.7, 0.6$ Hz, 1H), 7.94 (ddd, $J = 7.9, 1.8, 1.2$ Hz, 1H), 7.90 (appt. d, $J = 9.1$ Hz, 2H), 7.87 (ddd, $J = 7.8, 1.6, 1.2$ Hz, 1H), 7.68 (appt. td, $J = 7.9, 0.6$ Hz, 1H), 7.10 (appt. d, $J = 9.0$ Hz, 2H), 3.93 (s, 3H); ^{13}C NMR (126 MHz, CDCl_3) $\delta = 175.8$ (d, $J = 2.5$ Hz), 163.8, 160.1, 134.4, 133.7, 132.8, 131.7, 129.7, 127.7, 117.9, 116.0, 114.7, 113.3, 108.0 (d, $J = 35.0$ Hz), 55.7; ^{19}F NMR (376 MHz, CDCl_3) $\delta = 72.9$; HRMS (ESI $^+$): calculated for $\text{C}_{17}\text{H}_{11}\text{FN}_2\text{O}_4\text{SNa}$ [$\text{M}+\text{Na}^+$]: $m/z = 381.0316$, m/z found 381.0322; IR ν_{max} (ATR)/ cm^{-1} : 2961, 2928, 2232, 1713, 1604, 1511, 1415, 1259, 1178, 1021, 803.

3-(3-Nitrophenyl)-5-phenylisoxazole-4-sulfonyl fluoride (4as)



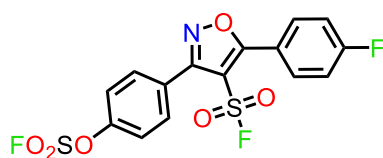
Following general procedure C, the title compound was isolated after purification by flash column chromatography (0-2% EtOAc in petroleum ether) as a beige solid (86.0 mg, 50%). **m.p.** 140 - 141 °C; **¹H NMR** (400 MHz, CDCl₃) δ 8.64 – 8.61 (m, 1H), 8.46 (ddd, *J* = 8.3, 2.3, 1.1 Hz, 1H), 8.06 (ddd, *J* = 7.7, 1.7, 1.1 Hz, 1H), 7.94 – 7.88 (m, 2H), 7.80 – 7.74 (m, 1H), 7.73 – 7.68 (m, 1H), 7.65 – 7.60 (m, 2H); **¹³C NMR** (101 MHz, CDCl₃) δ 176.3 (d, *J* = 2.7 Hz), 160.1, 148.4, 135.3, 133.6, 130.1, 129.8, 129.2, 127.7, 125.9, 124.7, 124.0, 109.8 (d, *J* = 35.5 Hz); **¹⁹F NMR** (376 MHz, CDCl₃) δ 73.2; **HRMS** (ESI⁺): calculated for C₁₅H₉FN₂O₅SNa [M+Na⁺]: *m/z* = 371.0108, *m/z* found 371.0100; **IR** ν_{\max} (ATR)/cm⁻¹: 2922, 2857, 1600, 1531, 1420, 1348, 1215, 1058, 767, 721.

4-(5-(3,5-Difluorophenyl)-4-(fluorosulfonyl)isoxazol-3-yl)phenyl fluorosulfate (4at)



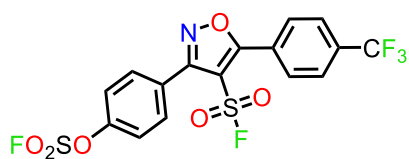
Following general procedure C (100 μmol of the required SASF and the required imidoyl chloride used), the title compound was isolated as a white solid (38.0 mg, 87%). **m.p.** 138.7 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.85 (appt. d, *J* = 8.5 Hz, 2H), 7.55 (appt. d, *J* = 8.5 Hz, 2H), 7.49 – 7.35 (m, 2H), 7.15 (td, *J* = 8.7, 2.4 Hz, 1H); **¹³C NMR** (151 MHz, CDCl₃) δ 173.2 (d, *J* = 3.1 Hz), 163.0 (dd, *J* = 252.3, 12.4 Hz), 160.4, 151.9, 131.9, 126.5 (t, *J* = 10.5 Hz), 126.1, 121.7, 113.3 (dd, *J* = 22.0, 6.7 Hz), 111.2 (d, *J* = 35.8 Hz), 109.0 (t, *J* = 24.9 Hz); **¹⁹F NMR** (376 MHz, CDCl₃) δ 72.3, 38.3, -106.6; **HRMS** (APCI): calculated for C₁₅H₈F₄NO₆S₂ [M+H⁺]: *m/z* = 437.9729, *m/z* found 437.9727;

4-(5-(4-Fluorophenyl)-4-(fluorosulfonyl)isoxazol-3-yl)phenyl fluorosulfate (4au)



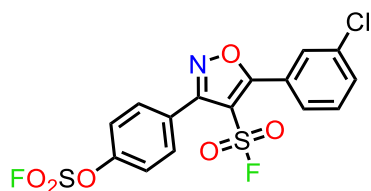
Following general procedure C (100 μmol of the required SASF and the required imidoyl chloride used), the title compound was isolated as a white solid (36.0 mg, 86%). **m.p.** 123.4 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.92 (dd, *J* = 8.5, 5.1 Hz, 2H), 7.85 (appt. d, *J* = 8.3 Hz, 2H), 7.54 (appt. d, *J* = 8.3 Hz, 2H), 7.30 (t, *J* = 8.3 Hz, 2H); **¹³C NMR** (151 MHz, CDCl₃) δ 175.1 (d, *J* = 2.7 Hz), 165.8 (d, *J* = 256.8 Hz), 160.3, 151.8, 132.4 (d, *J* = 9.3 Hz), 131.8, 126.6, 121.6, 120.3 (d, *J* = 3.4 Hz), 116.8 (d, *J* = 22.4 Hz), 109.8 (d, *J* = 35.6 Hz); **¹⁹F NMR** (376 MHz, CDCl₃) δ 72.2, 38.2, -104.0; **HRMS** (APCI): calculated for C₁₅H₉F₃NO₆S₂ [M+H⁺]: *m/z* = 419.9823, *m/z* found 419.9824;

4-(4-(Fluorosulfonyl)-5-(4-(trifluoromethyl)phenyl)isoxazol-3-yl)phenyl fluorosulfate (4av)



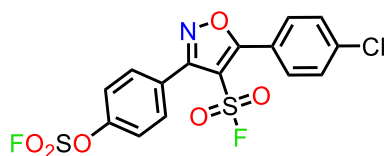
Following general procedure C (100 μ mol of the required SASF and the required imidoyl chloride used), the title compound was isolated as a white solid (36.5 mg, 78%). **m.p.** 134.4 $^{\circ}$ C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.01 (appt. d, $J = 8.1$ Hz, 2H), 7.93 – 7.75 (m, 4H), 7.55 (appt. d, $J = 8.3$ Hz, 2H); $^{13}\text{C NMR}$ (151 MHz, CDCl_3) δ 174.5 (d, $J = 2.8$ Hz), 160.3, 151.9, 135.0 (q, $J = 33.3$ Hz), 131.8, 130.4, 127.4, 126.2, 126.2 (q, $J = 3.8$ Hz), 123.4 (q, $J = 273.0$ Hz), 121.7, 111.1 (d, $J = 35.7$ Hz); $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ 72.3, 38.3, -63.8; **HRMS** (APCI): calculated for $\text{C}_{16}\text{H}_9\text{F}_5\text{NO}_6\text{S}_2$ [$\text{M}+\text{H}^+$]: $m/z = 469.9791$, m/z found 469.9780;

4-(5-(3-Chlorophenyl)-4-(fluorosulfonyl)isoxazol-3-yl)phenyl fluorosulfate (4aw)



Following general procedure C (100 μ mol of the required SASF and the required imidoyl chloride used), the title compound was isolated as a white solid (36.0 mg, 84%). **m.p.** 120.0 $^{\circ}$ C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.94 – 7.81 (m, 3H), 7.77 (d, $J = 7.9$ Hz, 1H), 7.66 (d, $J = 8.2$ Hz, 1H), 7.60 – 7.51 (m, 3H); $^{13}\text{C NMR}$ (151 MHz, CDCl_3) δ 174.6 (d, $J = 2.8$ Hz), 160.3, 151.9, 135.4, 133.5, 131.8, 130.5, 129.7, 128.0, 126.4, 125.7, 121.7, 110.6 (d, $J = 35.4$ Hz); $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ 72.3, 38.2; **HRMS** (APCI): calculated for $\text{C}_{15}\text{H}_9\text{ClF}_2\text{NO}_6\text{S}_2$ [$\text{M}+\text{H}^+$]: $m/z = 435.9528$, m/z found 435.9536;

4-(5-(4-Chlorophenyl)-4-(fluorosulfonyl)isoxazol-3-yl)phenyl fluorosulfate (4ax)

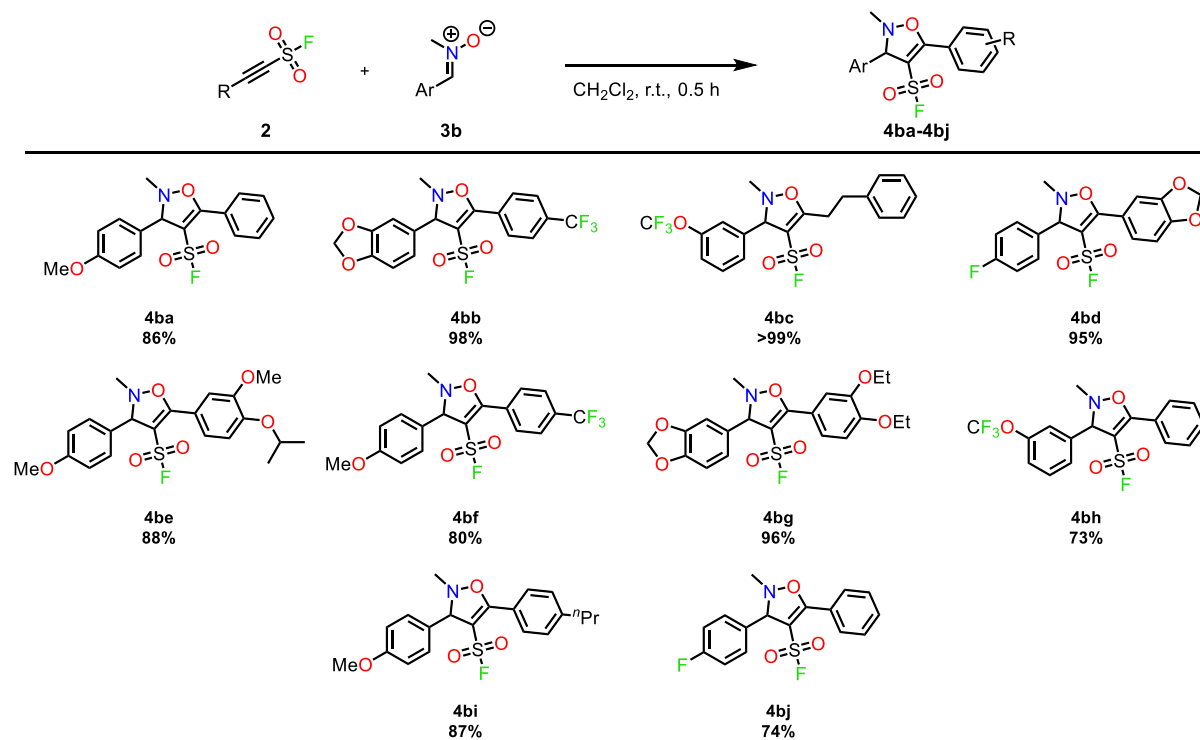


Following general procedure C (100 μ mol of the required SASF and the required imidoyl chloride used), the title compound was isolated as a white solid (35.0 mg, 80%). **m.p.** 96.3 $^{\circ}$ C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.92 – 7.74 (m, 4H), 7.59 (appt. d, $J = 8.0$ Hz, 2H), 7.54 (appt. d, $J = 8.4$ Hz, 2H); $^{13}\text{C NMR}$ (151 MHz, CDCl_3) δ 175.0 (d, $J = 2.6$ Hz), 160.4, 151.9, 140.3, 131.8, 131.1, 130.0, 129.7, 126.5, 122.5, 121.6, 110.2; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ 72.23, 38.20; **HRMS** (APCI): calculated for $\text{C}_{15}\text{H}_9\text{ClF}_2\text{NO}_6\text{S}_2$ [$\text{M}+\text{H}^+$]: $m/z = 435.9528$, m/z found 435.9524;

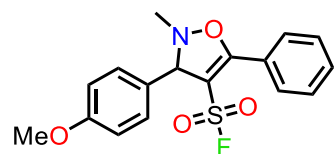
Synthesis and experimental data for compounds 4ba-4aj

General Procedure D

A solution of the required SASF **2** (100 μmol) and the required nitron **3b** (100 μmol) was stirred in CH_2Cl_2 (500 μL) at room temperature for 0.5 h and the solvent was then removed under reduced pressure to obtain the analytically pure product.

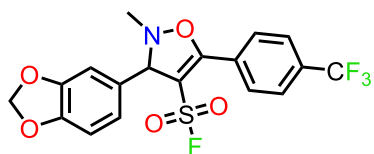


3-(4-Methoxyphenyl)-2-methyl-5-phenyl-2,3-dihydroisoxazole-4-sulfonyl fluoride (**4ba**)



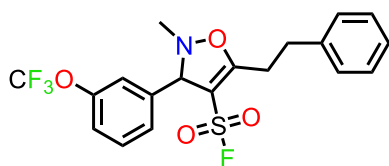
Following general procedure D, the title compound was isolated as a colourless oil (30.0 mg, 86%). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.76 (appt. d, $J = 8.5$ Hz, 1H), 7.60 (appt. t, $J = 7.5$ Hz, 1H), 7.50 (appt. t, $J = 7.6$ Hz, 1H), 7.37 (appt. d, $J = 8.7$ Hz, 1H), 6.94 (d, $J = 8.7$ Hz, 1H), 5.19 (s, 1H), 3.82 (s, 2H), 3.10 (s, 2H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 165.8 (d, $J = 2.3$ Hz), 160.1, 133.0, 130.6, 129.9, 128.6, 128.4, 124.5, 114.4, 102.9 (d, $J = 29.9$ Hz), 76.3, 55.3, 46.8. $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ 73.3; **HRMS** (ASAP): calculated for $\text{C}_{17}\text{H}_{17}\text{FNO}_4\text{S}$ [$\text{M}+\text{H}^+$]: $m/z = 350.0862$, m/z found 350.0861; **IR** ν_{max} (ATR)/ cm^{-1} : 3066, 2840, 1678, 1597, 761.

3-(Benzo[d][1,3]dioxol-5-yl)-2-methyl-5-(4-(trifluoromethyl)phenyl)-2,3-dihydroisoxazole-4-sulfonyl fluoride (4bb)



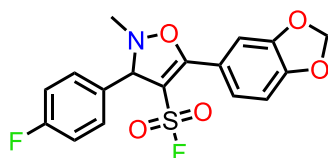
Following general procedure D, the title compound was isolated as a yellow solid (42.0 mg, 98%). **m.p.** 105 - 107 °C; **¹H NMR** (500 MHz, CDCl₃) δ 7.87 (appt. d, *J* = 8.2 Hz, 2H), 7.77 (appt. d, *J* = 8.2 Hz, 2H), 6.94 (d, *J* = 1.7 Hz, 1H), 6.91 (dd, *J* = 8.0, 1.7 Hz, 1H), 6.84 (d, *J* = 8.0 Hz, 1H), 6.00 (appt. q, *J* = 2.5 Hz, 2H), 5.19 (s, 1H), 3.12 (s, 3H); **¹³C NMR** (126 MHz, CDCl₃) δ 164.2, 148.5 (d, *J* = 3.0 Hz), 134.6 (q, *J* = 33.1 Hz), 131.9, 130.5, 128.1, 125.7 (q, *J* = 3.7 Hz), 123.5 (q, *J* = 272.8 Hz), 121.2, 108.7, 107.5, 104.8 (d, *J* = 30.5 Hz), 101.6, 76.8, 47.0; **¹⁹F NMR** (376 MHz, CDCl₃) δ 73.0, -63.3; **HRMS** (ESI⁺): calculated for C₁₈H₁₄F₄NO₅S [M+H⁺]: *m/z* = 432.0523, *m/z* found 435.0525; **IR** *v*_{max} (ATR)/cm⁻¹: 2966, 2892, 1687, 1632, 1607, 1490, 1449, 1406, 1320, 1247, 1211, 1172, 1125, 1063, 1038, 743.

2-Methyl-5-phenethyl-3-(3-(trifluoromethoxy)phenyl)-2,3-dihydroisoxazole-4-sulfonyl fluoride (4bc)



Following general procedure D, the title compound was isolated as a yellow oil (43.0 mg, >99%). **¹H NMR** (400 MHz, CDCl₃) δ 7.40 – 7.31 (m, 3H), 7.29 – 7.23 (m, 3H), 7.23 – 7.18 (m, 1H), 7.18 – 7.14 (m, 2H), 4.98 (s, 1H), 3.17 – 2.97 (m, 4H), 2.92 (s, 3H); **¹³C NMR** (101 MHz, CDCl₃) δ 170.7 (d, *J* = 2.8 Hz), 149.6 (q, *J* = 1.8 Hz), 140.7, 138.7, 130.3, 128.8, 128.7, 127.1, 125.8, 121.3, 120.6 (q, *J* = 257.5 Hz), 120.1, 103.5 (d, *J* = 29.6 Hz), 75.1, 47.4, 32.6, 27.7; **¹⁹F NMR** (376 MHz, CDCl₃) δ 71.3, -57.8; **HRMS** (ESI⁺): calculated for C₁₉H₁₇F₄NO₄SNa [M+Na⁺]: *m/z* = 454.0707, *m/z* found 454.0708; **IR** *v*_{max} (ATR)/cm⁻¹: 3031, 2963, 1736, 1621, 1404, 1256, 1207, 1161, 789, 757.

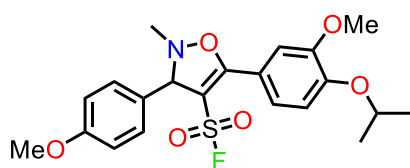
5-(Benzo[d][1,3]dioxol-5-yl)-3-(4-fluorophenyl)-2-methyl-2,3-dihydroisoxazole-4-sulfonyl fluoride (4bd)



Following general procedure D, the title compound was isolated as a yellow solid (36.0 mg, 95%). **m.p.** 92 - 93 °C; **¹H NMR** (500 MHz, CDCl₃) δ 7.44 – 7.40 (m, 2H), 7.37 (dd, *J* = 8.2, 1.8 Hz, 1H), 7.21 (d, *J* = 1.8 Hz, 1H), 7.12 – 7.07 (m, 2H), 6.92 (d, *J* = 8.2 Hz, 1H), 6.08 (s, 2H), 5.19 (s, 1H), 3.10 (s, 3H); **¹³C NMR** (126 MHz, CDCl₃) δ 165.6 (d, *J* =

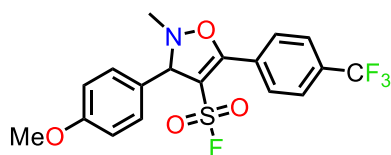
1.9 Hz), 163.1 (d, $J = 247.7$ Hz), 152.1, 148.1, 134.9, 128.9 (d, $J = 8.4$ Hz), 126.3, 117.7, 116.1 (d, $J = 21.7$ Hz), 110.0, 108.7, 102.3, 101.3 (d, $J = 30.4$ Hz), 76.0, 47.0; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ 73.9, -112.9; **HRMS** (ESI⁺): calculated for $\text{C}_{17}\text{H}_{14}\text{F}_2\text{NO}_5\text{S}$ [$\text{M}+\text{H}^+$]: $m/z = 382.0555$, m/z found 382.0557; **IR** ν_{max} (ATR)/ cm^{-1} : 2960, 2922, 2857, 1629, 1604, 1505, 1486, 1451, 1413, 1256, 1211, 1095, 1031, 791, 732.

5-(4-isopropoxy-3-methoxyphenyl)-3-(4-methoxyphenyl)-2-methyl-2,3-dihydroisoxazole-4-sulfonyl fluoride (4be)



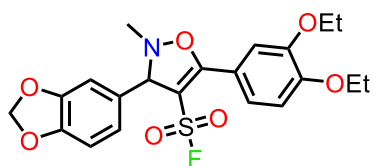
Following general procedure D, the title compound was isolated as a yellow oil (37.0 mg, 84%). $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.43 (appt. dd, $J = 8.5, 2.1$ Hz, 1H), 7.35 (appt. d, $J = 8.7$ Hz, 1H), 7.32 (appt. d, $J = 2.1$ Hz, 1H), 6.99 – 6.87 (m, 1H), 5.17 (s, 1H), 4.67 (hept, $J = 6.1$ Hz, 1H), 3.90 (s, 1H), 3.81 (s, 1H), 3.07 (s, 1H), 1.42 (d, $J = 6.1$ Hz, 1H); $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 165.5, 160.1, 152.0, 149.7, 128.4, 124.3, 116.2, 114.4, 113.4, 113.4, 100.7 (d, $J = 29.6$ Hz), 76.4, 71.5, 56.3, 55.4, 22.1, 22.0; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ 73.7; **HRMS** (ESI⁺): calculated for $\text{C}_{21}\text{H}_{25}\text{FNO}_6\text{S}$ [$\text{M}+\text{H}^+$]: $m/z = 438.1381$, m/z found 438.1391; **IR** ν_{max} (ATR)/ cm^{-1} : 2977, 2933, 2838, 1599, 1505, 740.

3-(4-Methoxyphenyl)-2-methyl-5-(4-(trifluoromethyl)phenyl)-2,3-dihydroisoxazole-4-sulfonyl fluoride (4bf)



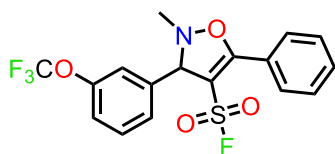
Following general procedure D, the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a yellow amorphous solid (33.0 mg, 80%). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.88 (appt. d, $J = 8.2$ Hz, 2H), 7.77 (appt. d, $J = 8.2$ Hz, 2H), 7.37 (appt. d, $J = 8.5$ Hz, 2H), 6.96 (appt. d, $J = 8.6$ Hz, 2H), 5.23 (s, 1H), 3.84 (s, 3H), 3.12 (s, 3H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 164.1, 160.4, 134.5 (q, $J = 33.0$ Hz), 130.5, 130.1, 128.7, 128.2, 125.7 (q, $J = 3.7$ Hz), 123.5 (q, $J = 272.9$ Hz), 114.6, 104.9 (d, $J = 30.3$ Hz), 76.6, 55.5, 47.0; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ 72.9, -63.3; **HRMS** (ASAP): calculated for $\text{C}_{18}\text{H}_{16}\text{F}_4\text{NO}_4\text{S}$ [$\text{M}+\text{H}^+$]: $m/z = 418.0736$, m/z found 418.0732; **IR** ν_{max} (ATR)/ cm^{-1} : 2924, 2841, 1606, 1513, 1408, 1322, 1246, 1212, 1176, 1124, 1066, 850, 731.

3-(Benzo[d][1,3]dioxol-5-yl)-5-(3,4-diethoxyphenyl)-2-methyl-2,3-dihydroisoxazole-4-sulfonyl fluoride (4bg)



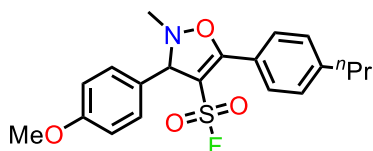
Following general procedure D, the title compound was isolated after purification by flash column chromatography (0-20% EtOAc in petroleum ether) as a yellow oil (43.0 mg, 96%). **¹H NMR** (500 MHz, CDCl₃) δ 7.42 (dd, *J* = 8.5, 2.1 Hz, 1H), 7.32 (d, *J* = 2.1 Hz, 1H), 6.94 (d, *J* = 8.5 Hz, 1H), 6.93 – 6.89 (m, 2H), 6.82 (d, *J* = 7.9 Hz, 1H), 5.98 (appt. q, *J* = 3.0 Hz, 2H), 5.13 (s, 1H), 4.21 – 4.12 (m, 4H), 3.08 (s, 3H), 1.50 (t, *J* = 7.0 Hz, 3H), 1.48 (t, *J* = 7.0 Hz, 3H); **¹³C NMR** (101 MHz, CDCl₃) δ 165.7 (d, *J* = 1.7 Hz), 153.3, 148.4, 148.2, 133.0, 124.4, 120.9, 116.2, 114.6, 112.1, 108.6, 107.4, 101.4, 100.5 (d, *J* = 29.5 Hz), 76.7, 65.0, 64.7, 46.9, 14.8, 14.7; **¹⁹F NMR** (376 MHz, CDCl₃) δ 73.8; **HRMS** (ESI⁺): calculated for C₂₁H₂₂FNO₇Na [M+Na⁺]: *m/z* = 474.0993, *m/z* found 474.0988; **IR** *v*_{max} (ATR)/cm⁻¹: 2985, 2931, 2884, 1735, 1599, 1505, 1398, 1241, 1201, 1036, 742.

2-Methyl-5-phenyl-3-(3-(trifluoromethoxy)phenyl)-2,3-dihydroisoxazole-4-sulfonyl fluoride (4bh)



Following general procedure D, the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a yellow oil (29.0 mg, 73%). **¹H NMR** (400 MHz, CDCl₃) δ 7.75 (appt. d, *J* = 7.2 Hz, 1H), 7.61 (appt. t, *J* = 6.9 Hz, 1H), 7.51 (appt. t, *J* = 7.6 Hz, 1H), 7.48 – 7.39 (m, 1H), 7.35 (br s, 1H), 7.24 (br d, *J* = 7.7 Hz, 1H), 5.25 (s, 1H), 3.14 (s, 3H); **¹³C NMR** (101 MHz, CDCl₃) δ 166.5 (d, *J* = 2.2 Hz), 149.8 (q, *J* = 1.7 Hz), 141.2, 133.4, 130.6, 130.2, 128.8, 125.4, 124.3, 121.4, 120.6 (q, *J* = 257.6 Hz), 119.8, 102.4 (d, *J* = 30.8 Hz), 76.0, 47.1; **¹⁹F NMR** (376 MHz, CDCl₃) δ 73.8, -57.8; **HRMS** (ESI⁺): calculated for C₁₇H₁₄F₄NO₄S [M+H⁺]: *m/z* = 404.0574, *m/z* found 404.0573; **IR** *v*_{max} (ATR)/cm⁻¹: 3068, 2857, 1704, 1408, 751.

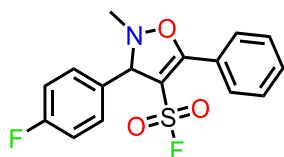
3-(4-Methoxyphenyl)-2-methyl-5-(4-propylphenyl)-2,3-dihydroisoxazole-4-sulfonyl fluoride (4bi)



Following general procedure D, the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a yellow oil (34.0 mg, 87%). **¹H NMR** (400 MHz, CDCl₃) δ 7.69 (appt. d, *J* = 8.3 Hz, 2H), 7.35 (appt. d, *J* = 8.7 Hz, 2H), 7.30 (appt. d, *J* = 8.2 Hz, 2H), 6.93 (appt. d, *J* = 8.7 Hz,

2H), 5.18 (s, 1H), 3.82 (s, 3H), 3.08 (s, 3H), 2.66 (t, $J = 7.5$ Hz, 2H), 1.74 – 1.62 (m, 2H), 0.96 (t, $J = 7.3$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 166.1, 160.2, 148.8, 130.1, 128.8, 128.5, 121.9, 114.5, 102.1 (d, $J = 29.9$ Hz), 76.4, 55.5, 46.9, 38.2, 24.3, 13.9; ^{19}F NMR (376 MHz, CDCl_3) δ 73.5; HRMS (ESI⁺): calculated for $\text{C}_{20}\text{H}_{23}\text{FNO}_4\text{S}$ [$\text{M}+\text{H}^+$]: $m/z = 392.1326$, m/z found 392.1332; IR ν_{max} (ATR)/ cm^{-1} : 2961, 2931, 2872, 1610, 1511, 748.

3-(4-Fluorophenyl)-2-methyl-5-phenyl-2,3-dihydroisoxazole-4-sulfonyl fluoride (4bj)

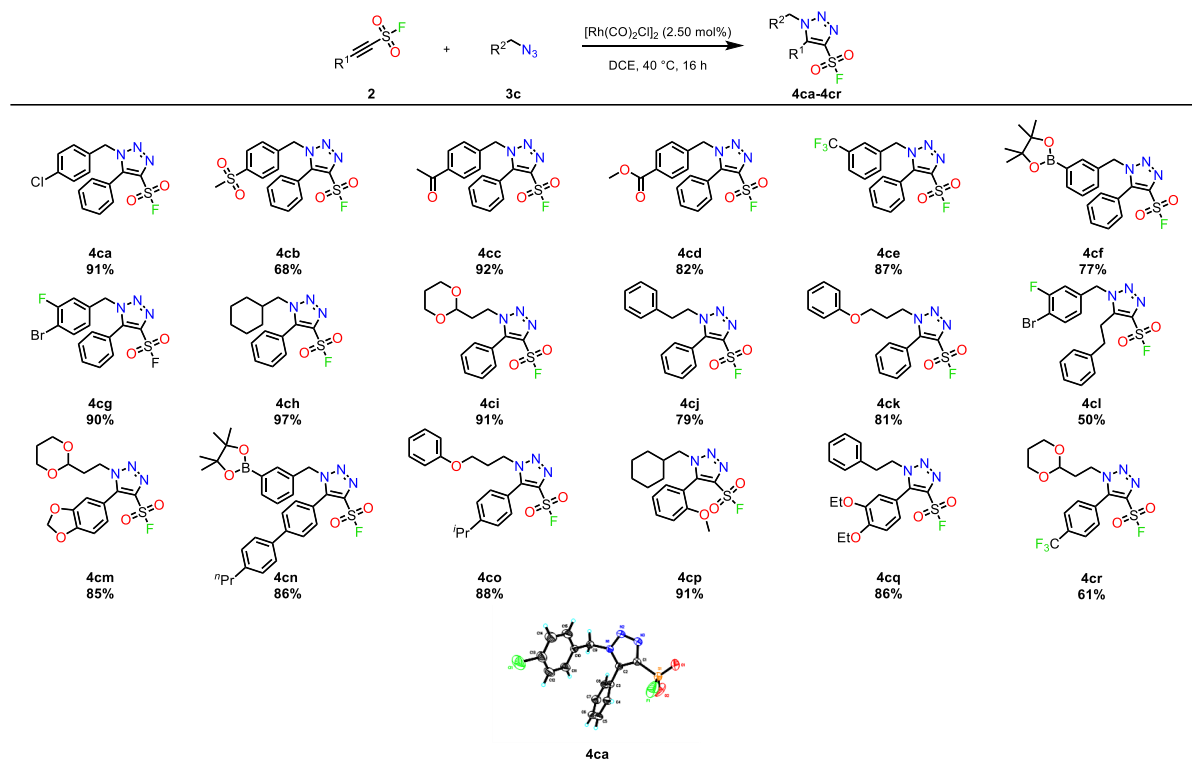


Following general procedure D, the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a yellow oil (28.0 mg, 74%). ^1H NMR (400 MHz, CDCl_3) δ 7.75 (appt. d, $J = 7.2$ Hz, 2H), 7.61 (appt. t, $J = 6.9$ Hz, 1H), 7.50 (appt. t, $J = 7.6$ Hz, 2H), 7.47 – 7.40 (m, 2H), 7.10 (appt. t, $J = 8.7$ Hz, 2H), 5.22 (s, 1H), 3.12 (s, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 166.1 (d, $J = 2.3$ Hz), 163.2 (d, $J = 247.9$ Hz), 134.7, 133.3, 130.1, 129.0 (d, $J = 8.4$ Hz), 128.8, 124.4, 116.1 (d, $J = 21.8$ Hz), 102.8 (d, $J = 30.4$ Hz), 76.1, 47.1; ^{19}F NMR (376 MHz, CDCl_3) δ 73.6, -112.8; HRMS (ESI⁺): calculated for $\text{C}_{16}\text{H}_{14}\text{F}_2\text{NO}_3\text{S}$ [$\text{M}+\text{H}^+$]: $m/z = 338.0657$, m/z found 338.0657; IR ν_{max} (ATR)/ cm^{-1} : 3069, 2967, 2923, 1509, 1403, 746.

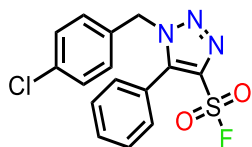
Synthesis and experimental data for compounds 4ca-4ar

General procedure E

To a solution of the required SASF **2** (100 μ mol) and the required azide **3c** (100 μ mol) in DCE (1.00 mL) was added $[\text{Rh}(\text{CO})_2\text{Cl}]_2$ (1.00 mg, 2.50 mol%) and stirred at 40 $^\circ\text{C}$ for 16 h.^[18] The solvent was removed under reduced pressure and the crude mixture was purified by flash column chromatography to obtain the analytically pure product.

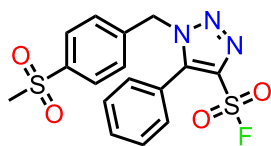


1-(4-Chlorobenzyl)-5-phenyl-1H-1,2,3-triazole-4-sulfonyl fluoride (4ca)



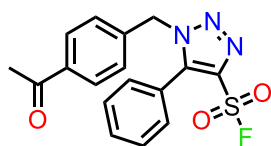
Following general procedure E, the title compound was isolated after purification by flash column chromatography (0-20% EtOAc in petroleum ether) as a colourless solid (32.0 mg, 91%). **m.p.** 129 - 130 $^\circ\text{C}$; **^1H NMR** (400 MHz, CDCl_3) δ 7.65 – 7.59 (m, 1H), 7.57 – 7.49 (m, 2H), 7.30 – 7.27 (m, 2H), 7.26 – 7.22 (m, 2H), 6.98 (appt. d, $J = 8.7$ Hz, 2H), 5.44 (s, 2H). **^{13}C NMR** (126 MHz, CDCl_3) δ 141.6 (d, $J = 2.7$ Hz), 138.3 (d, $J = 35.8$ Hz), 135.4, 131.9, 131.7, 129.7, 129.5, 129.4, 122.8, 52.5; **^{19}F NMR** (376 MHz, CDCl_3) δ 68.6; **HRMS** (ESI⁺): calculated for $\text{C}_{15}\text{H}_{12}\text{ClFN}_3\text{O}_2\text{S}$ $[\text{M}+\text{H}^+]$: $m/z = 352.0317$, m/z found 352.0314; **IR** ν_{max} (ATR)/ cm^{-1} : 3061, 2852, 1493, 1427, 1204, 1093, 783.

1-(4-(Methylsulfonyl)benzyl)-5-phenyl-1*H*-1,2,3-triazole-4-sulfonyl fluoride (4cb)



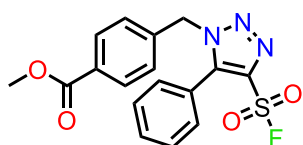
Following general procedure E, the title compound was isolated after purification by flash column chromatography (0-70% EtOAc in petroleum ether) as a colourless solid (27.0 mg, 68%). **m.p.** 98 - 99 °C; **¹H NMR** (500 MHz, CDCl₃) δ 7.89 (appt. d, *J* = 8.2 Hz, 2H), 7.62 (appt. t, *J* = 7.5 Hz, 1H), 7.54 (appt. t, *J* = 7.7 Hz, 2H), 7.26 (m, 4H), 5.55 (s, 2H), 3.04 (s, 3H). **¹³C NMR** (126 MHz, CDCl₃) δ 141.9 (d, *J* = 2.6 Hz), 141.5, 139.2, 138.4 (d, *J* = 36.2 Hz), 132.0, 129.6, 129.6, 128.9, 128.4, 122.5, 52.3, 44.5; **¹⁹F NMR** (376 MHz, CDCl₃) δ 68.6; **HRMS** (ESI⁺): calculated for C₁₆H₁₄F₂N₃O₄S₂Na [M+Na⁺]: *m/z* = 418.0302, *m/z* found 418.0305; **IR** *v*_{max} (ATR)/cm⁻¹: 2926, 2857, 1416, 1307, 1146, 722.

1-(4-Acetylbenzyl)-5-phenyl-1*H*-1,2,3-triazole-4-sulfonyl fluoride (4cc)



Following general procedure E, the title compound was isolated after purification by flash column chromatography (0-40% EtOAc in petroleum ether) as a colourless solid (33.0 mg, 92%). **m.p.** 111 - 112 °C; **¹H NMR** (500 MHz, CDCl₃) δ 7.89 (appt. d, *J* = 8.2 Hz, 2H), 7.60 (appt. t, *J* = 7.5 Hz, 1H), 7.51 (appt. t, *J* = 7.7 Hz, 2H), 7.23 (appt. d, *J* = 7.7 Hz, 2H), 7.13 (appt. d, *J* = 8.2 Hz, 2H), 5.52 (s, 2H), 2.58 (s, 3H); **¹³C NMR** (126 MHz, CDCl₃) δ 197.3, 141.8 (d, *J* = 2.6 Hz), 138.28 (d, *J* = 35.9 Hz), 138.3, 137.6, 131.8, 129.7, 129.4, 129.2, 128.1, 122.6, 52.7, 26.8; **¹⁹F NMR** (376 MHz, CDCl₃) δ 68.6; **HRMS** (ESI⁺): calculated for C₁₇H₁₄FN₃O₃SNa [M+Na⁺]: *m/z* = 382.0632, *m/z* found 382.0630; **IR** *v*_{max} (ATR)/cm⁻¹: 2925, 2855, 1681, 1610, 1428, 1201, 776.

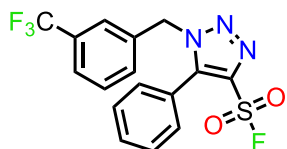
Methyl 4-((4-(fluorosulfonyl)-5-phenyl-1*H*-1,2,3-triazol-1-yl)methyl)benzoate (4cd)



Following general procedure E, the title compound was isolated after purification by flash column chromatography (0-30% EtOAc in petroleum ether) as a colourless solid (31.0 mg, 82%). **m.p.** 129 - 130 °C; **¹H**

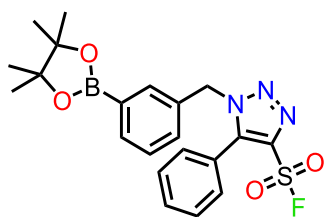
NMR (500 MHz, CDCl₃) δ 7.97 (appt. d, *J* = 8.5 Hz, 1H), 7.59 (appt. t, *J* = 6.9 Hz, 1H), 7.50 (appt. t, *J* = 8.0 Hz, 1H), 7.21 (appt. d, *J* = 7.1 Hz, 1H), 7.09 (appt. d, *J* = 8.6 Hz, 1H), 5.52 (s, 1H), 3.92 (s, 2H); **¹³C NMR** (126 MHz, CDCl₃) δ 166.4, 141.8 (d, *J* = 2.7 Hz), 138.3 (d, *J* = 36.0 Hz), 138.1, 131.8, 131.0, 130.5, 129.7, 129.4, 127.8, 122.7, 52.8, 52.5; **¹⁹F NMR** (376 MHz, CDCl₃) δ 68.6; **HRMS** (ESI⁺): calculated for C₁₇H₁₄FN₃O₄SNa [M+Na⁺]: *m/z* = 398.0581; *m/z* found 398.0579; **IR** *v*_{max} (ATR)/cm⁻¹: 2955, 2925, 2854, 1725, 1281, 768.

5-Phenyl-1-(3-(trifluoromethyl)benzyl)-1*H*-1,2,3-triazole-4-sulfonyl fluoride (4ce)



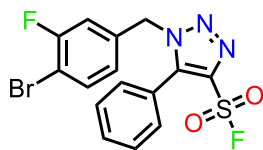
Following general procedure E, the title compound was isolated after purification by flash column chromatography (0-20% EtOAc in petroleum ether) as a yellow oil (34.0 mg, 87%) **¹H NMR** (500 MHz, CDCl₃) δ 7.65 – 7.58 (m, 2H), 7.56 – 7.51 (m, 2H), 7.47 (appt. t, *J* = 7.8 Hz, 1H), 7.30 (appt. d, *J* = 7.8 Hz, 1H), 7.24 – 7.19 (m, 3H), 5.54 (s, 2H); **¹³C NMR** (126 MHz, CDCl₃) δ 141.7 (d, *J* = 2.8 Hz), 138.4 (d, *J* = 36.1 Hz), 134.2, 131.9, 132.1 – 131.2 (m), 131.5, 130.0, 129.6, 129.5, 126.2 (q, *J* = 3.7 Hz), 125.2 (q, *J* = 3.8 Hz), 123.6 (q, *J* = 272.6 Hz), 122.7, 52.7; **¹⁹F NMR** (471 MHz, CDCl₃) δ 68.6, -62.9; **HRMS** (ESI⁺): calculated for C₁₆H₁₁F₄N₃O₂SNa [M+Na⁺]: *m/z* = 408.0400, *m/z* found 408.0400; **IR** *v*_{max} (ATR)/cm⁻¹: 3068, 2926, 1424, 1327, 1202, 1124, 1074, 779.

5-Phenyl-1-(3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1*H*-1,2,3-triazole-4-sulfonyl fluoride (4cf)



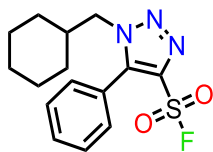
Following general procedure E, the title compound was isolated after purification by flash column chromatography (0-20% EtOAc in petroleum ether) as a colourless solid (34.0 mg, 77%). **m.p.** 108 - 109 °C; **¹H NMR** (500 MHz, CDCl₃) δ 7.75 (appt. dt, *J* = 7.4, 1.0 Hz, 1H), 7.61 – 7.56 (m, 1H), 7.53 – 7.46 (m, 3H), 7.30 (appt. t, *J* = 7.6 Hz, 1H), 7.25 – 7.21 (m, 2H), 7.09 (appt. ddd, *J* = 7.7, 1.9, 1.3 Hz, 1H), 5.48 (s, 2H), 1.34 (s, 12H); **¹³C NMR** (126 MHz, CDCl₃) δ 141.7 (d, *J* = 2.7 Hz), 138.1 (d, *J* = 35.7 Hz), 135.4, 134.4, 132.7, 131.5, 130.7, 129.8, 129.3, 128.6, 122.9, 84.2, 53.3, 25.0; **¹⁹F NMR** (471 MHz, CDCl₃) δ 68.6; **HRMS** (ESI⁺): calculated for C₂₁H₂₄BFN₃O₄S [M+H⁺]: *m/z* = 444.1559, *m/z* found 444.1564; **IR** *v*_{max} (ATR)/cm⁻¹: 2982, 2927, 2361, 1417, 1362, 1201, 1147, 780.

1-(4-Bromo-3-fluorobenzyl)-5-phenyl-1*H*-1,2,3-triazole-4-sulfonyl fluoride (4cg)



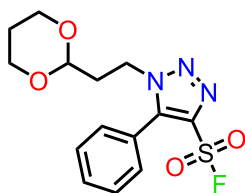
Following general procedure E, the title compound was isolated after purification by flash column chromatography (0-20% EtOAc in petroleum ether) as a colourless solid (37.0 mg, 90%). **m.p.** 103 - 104 °C; **¹H NMR** (500 MHz, CDCl₃) δ 7.64 (appt. t, *J* = 7.5 Hz, 1H), 7.56 (appt. t, *J* = 7.7 Hz, 2H), 7.24 (appt. d, *J* = 7.9 Hz, 2H), 7.20 (appt. dd, *J* = 6.2, 1.8 Hz, 1H), 7.05 (appt. t, *J* = 8.3 Hz, 1H), 7.00 – 6.94 (m, 1H), 5.42 (s, 2H); **¹³C NMR** (126 MHz, CDCl₃) δ 159.6 (d, *J* = 250.6 Hz), 141.6, 138.4 (d, *J* = 36.1 Hz), 133.6, 131.9, 130.6 (d, *J* = 4.0 Hz), 129.7, 129.5, 128.9 (d, *J* = 7.7 Hz), 122.7, 117.3 (d, *J* = 22.9 Hz), 110.0 (d, *J* = 21.6 Hz), 51.9; **¹⁹F NMR** (376 MHz, CDCl₃) δ 68.6, -105.6; **HRMS** (ESI⁺): calculated for C₁₅H₁₀BrF₂N₃O₂SNa [M+Na⁺]: *m/z* = 435.9537, *m/z* found 435.9542; **IR** *v*_{max} (ATR)/cm⁻¹: 3096, 2980, 2946, 1494, 1426, 771.

1-(Cyclohexylmethyl)-5-phenyl-1*H*-1,2,3-triazole-4-sulfonyl fluoride (4ch)



Following general procedure E, the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a colourless solid (31.0 mg, 97%). **m.p.** 71 - 72 °C; **¹H NMR** (500 MHz, CDCl₃) δ 7.65 – 7.56 (m, 3H), 7.40 – 7.35 (m, 2H), 4.11 (d, *J* = 7.4 Hz, 2H), 1.96 – 1.85 (m, 1H), 1.72 – 1.59 (m, 3H), 1.55 – 1.48 (m, 2H), 1.23 – 1.04 (m, 3H), 0.90 – 0.80 (m, 2H); **¹³C NMR** (126 MHz, CDCl₃) δ 141.8 (d, *J* = 2.7 Hz), 137.7 (d, *J* = 35.3 Hz), 131.5, 129.7, 129.4, 123.3, 55.2, 38.4, 30.4, 26.0, 25.4; **¹⁹F NMR** (471 MHz, CDCl₃) δ 68.6; **HRMS** (ESI⁺): calculated for C₁₅H₁₉FN₃O₂S [M+H⁺]: *m/z* = 324.1177, *m/z* found 324.1178; **IR** *v*_{max} (ATR)/cm⁻¹: 2922, 2851, 1422, 1201, 773.

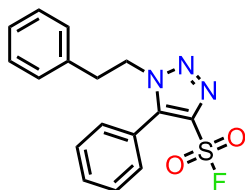
1-(2-(1,3-Dioxan-2-yl)ethyl)-5-phenyl-1*H*-1,2,3-triazole-4-sulfonyl fluoride (4ci)



Following general procedure E, the title compound was isolated after purification by flash column chromatography (0-35% EtOAc in petroleum ether) as a yellow oil (31.0 mg, 91%). **¹H NMR** (500 MHz, CDCl₃) δ

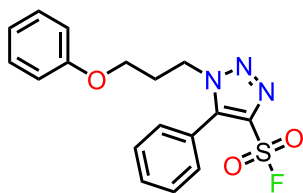
7.64 – 7.56 (m, 3H), 7.43 – 7.39 (m, 2H), 4.55 (t, $J = 4.6$ Hz, 1H), 4.43 (t, $J = 7.1$ Hz, 2H), 4.02 – 3.96 (m, 2H), 3.69 – 3.63 (m, 2H), 2.17 (td, $J = 7.1, 4.6$ Hz, 2H), 2.01 – 1.90 (m, 1H), 1.33 – 1.27 (m, 1H); $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 141.7 (d, $J = 2.7$ Hz), 137.7 (d, $J = 35.5$ Hz), 131.5, 129.7, 129.3, 123.1, 98.8, 66.9, 44.7, 34.7, 25.5; $^{19}\text{F NMR}$ (471 MHz, CDCl_3) δ 68.6; **HRMS** (ESI⁺): calculated for $\text{C}_{14}\text{H}_{16}\text{FN}_3\text{O}_4\text{SNa}$ [$\text{M}+\text{Na}^+$]: $m/z = 364.0738$, m/z found 364.0740; **IR** ν_{max} (ATR)/ cm^{-1} : 2962, 2926, 2855, 1421, 1201, 1142, 1005, 770.

1-Phenethyl-5-phenyl-1H-1,2,3-triazole-4-sulfonyl fluoride (4cj)



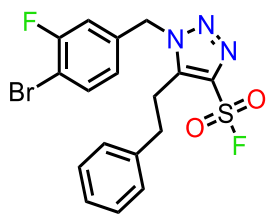
Following general procedure E, the title compound was isolated after purification by flash column chromatography (0-20% EtOAc in petroleum ether) as a yellow oil (26.0 mg, 79%). $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.57 – 7.52 (m, 1H), 7.46 – 7.41 (m, 2H), 7.27 – 7.21 (m, 3H), 6.93 – 6.89 (m, 2H), 6.89 – 6.84 (m, 2H), 4.48 (t, $J = 7.0$ Hz, 2H), 3.22 (t, $J = 7.0$ Hz, 2H); $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 142.1 (d, $J = 2.6$ Hz), 137.6 (d, $J = 35.6$ Hz), 136.1, 131.3, 129.5, 129.1, 129.1, 128.8, 127.6, 122.7, 50.6, 36.3; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ 68.7; **HRMS** (ESI⁺): calculated for $\text{C}_{16}\text{H}_{14}\text{FN}_3\text{O}_2\text{SNa}$ [$\text{M}+\text{Na}^+$]: $m/z = 332.0863$, m/z found 332.0864; **IR** ν_{max} (ATR)/ cm^{-1} : 3064, 3030, 2929, 1421, 1200, 773.

1-(3-Phenoxypropyl)-5-phenyl-1H-1,2,3-triazole-4-sulfonyl fluoride (4ck)



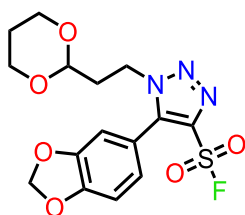
Following general procedure E, the title compound was isolated after purification by flash column chromatography (0-20% EtOAc in petroleum ether) as a yellow oil (29.0 mg, 81%) $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.61 – 7.55 (m, 1H), 7.53 – 7.47 (m, 2H), 7.36 – 7.31 (m, 2H), 7.30 – 7.24 (m, 3H), 7.00 – 6.94 (m, 1H), 6.74 – 6.69 (m, 2H), 4.52 (t, $J = 6.8$ Hz, 2H), 3.93 (appt. t, $J = 5.5$ Hz, 2H), 2.45 – 2.37 (m, 2H); $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 158.1 (s), 142.0 (d, $J = 2.8$ Hz), 137.8 (d, $J = 35.6$ Hz), 131.5, 129.7, 129.7, 129.4, 122.8, 121.4, 114.3, 63.6, 46.5, 29.5; $^{19}\text{F NMR}$ (471 MHz, CDCl_3) δ 68.6; **HRMS** (ESI⁺): calculated for $\text{C}_{17}\text{H}_{16}\text{FN}_3\text{O}_3\text{SNa}$ [$\text{M}+\text{Na}^+$]: $m/z = 362.0969$, m/z found 362.0966; **IR** ν_{max} (ATR)/ cm^{-1} : 2925, 1600, 1421, 1242, 1200, 754, 689.

1-(4-Bromo-3-fluorobenzyl)-5-phenethyl-1H-1,2,3-triazole-4-sulfonyl fluoride (4cl)



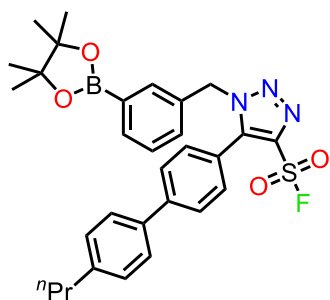
Following general procedure E, the title compound was isolated after purification by flash column chromatography (0-20% EtOAc in petroleum ether) as a yellow oil (22.0 mg, 50%). **¹H NMR** (500 MHz, CDCl₃) δ 7.36 – 7.27 (m, 4H), 7.12 (t, *J* = 8.3 Hz, 1H), 7.05 – 6.97 (m, 3H), 4.93 (s, 2H), 3.14 (t, *J* = 7.4 Hz, 2H), 2.84 (t, *J* = 7.4 Hz, 2H); **¹³C NMR** (126 MHz, CDCl₃) δ 159.6 (d, *J* = 250.8 Hz), 142.1 (d, *J* = 3.4 Hz), 138.6, 137.6 (d, *J* = 35.4 Hz), 133.0, 130.5 (d, *J* = 4.0 Hz), 129.4, 128.6, 128.4 (d, *J* = 7.6 Hz), 127.6, 117.6 (d, *J* = 22.9 Hz), 110.4 (d, *J* = 21.7 Hz), 51.2, 35.1, 25.7; **¹⁹F NMR** (376 MHz, CDCl₃) δ 68.7, -105.3 – -105.6 (m); **HRMS** (ESI⁺): calculated for C₁₇H₁₄BrF₂N₃O₂SNa [M+Na⁺]: *m/z* = 463.9850, *m/z* found 463.9853; **IR** *v*_{max} (ATR)/cm⁻¹: 3064, 3030, 2929, 2213, 1497, 1417, 1213, 779.

1-(2-(1,3-Dioxan-2-yl)ethyl)-5-(benzo[d][1,3]dioxol-5-yl)-1H-1,2,3-triazole-4-sulfonyl fluoride (4cm)



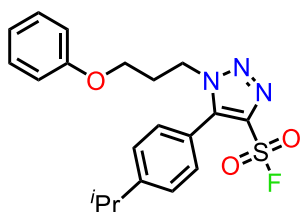
Following general procedure E, the title compound was isolated after purification by flash column chromatography (0-20% EtOAc in petroleum ether) as a yellow oil (33.0 mg, 85%). **¹H NMR** (400 MHz, CDCl₃) δ 6.98 (d, *J* = 8.0 Hz, 1H), 6.88 (dd, *J* = 8.0, 1.8 Hz, 1H), 6.85 (d, *J* = 1.8 Hz, 1H), 6.10 (s, 2H), 4.58 (t, *J* = 4.6 Hz, 1H), 4.43 (t, *J* = 7.1 Hz, 2H), 4.07 – 3.98 (m, 2H), 3.73 – 3.64 (m, 2H), 2.18 (td, *J* = 7.1, 4.6 Hz, 2H), 2.06 – 1.92 (m, 1H), 1.34 – 1.30 (m, 1H); **¹³C NMR** (101 MHz, CDCl₃) δ 150.4, 148.6, 141.4 (d, *J* = 2.8 Hz), 137.6 (d, *J* = 35.3 Hz), 124.4, 115.9, 109.7, 109.2, 102.2, 98.8, 66.9, 44.6, 34.7, 25.6; **¹⁹F NMR** (376 MHz, CDCl₃) δ 68.5; **HRMS** (ESI⁺): calculated for C₁₅H₁₆FN₃O₆Na [M+Na⁺]: *m/z* = 408.0636, *m/z* found 408.0636; **IR** *v*_{max} (ATR)/cm⁻¹: 2924, 2854, 1477, 1420, 1200, 1141, 1036, 772.

5-(4'-Propyl-[1,1'-biphenyl]-4-yl)-1-(3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)benzyl)-1H-1,2,3-triazole-4-sulfonyl fluoride (4cn)



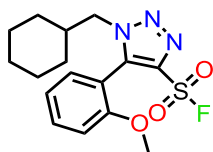
Following general procedure E, the title compound was isolated after purification by flash column chromatography (0-20% EtOAc in petroleum ether) as a yellow solid (48.0 mg, 86%). **m.p.** 123 - 124 °C **¹H NMR** (400 MHz, CDCl₃) δ 7.77 (appt. d, *J* = 7.4 Hz, 1H), 7.71 (appt. d, *J* = 8.5 Hz, 2H), 7.57 (appt. d, *J* = 8.2 Hz, 2H), 7.53 (s, 1H), 7.34 – 7.27 (m, 5H), 7.14 (appt. ddd, *J* = 7.7, 1.7, 1.3 Hz, 1H), 5.52 (s, 2H), 2.67 (appt. t, *J* = 7.4 Hz, 2H), 1.76 – 1.65 (m, 2H), 1.30 (s, 12H), 0.99 (t, *J* = 7.3 Hz, 3H); **¹³C NMR** (101 MHz, CDCl₃) δ 144.4, 143.3, 141.7 (d, *J* = 2.7 Hz), 138.1 (d, *J* = 35.5 Hz), 136.9, 135.5, 134.4, 132.8, 130.7, 130.2, 129.3, 128.6, 127.6, 127.2, 121.1, 84.2, 53.3, 37.8, 24.9, 24.6, 13.9; **¹⁹F NMR** (376 MHz, CDCl₃) δ 68.6; **HRMS** (ESI⁺): calculated for C₃₀H₃₃BFN₃O₄SNa [M+Na⁺]: *m/z* = 584.2161, *m/z* found 584.2163; **IR** *v*_{max} (ATR)/cm⁻¹: 2980, 2868, 2360, 1424, 1360, 1203, 776.

5-(4-Isopropylphenyl)-1-(3-phenoxypropyl)-1H-1,2,3-triazole-4-sulfonyl fluoride (4co)



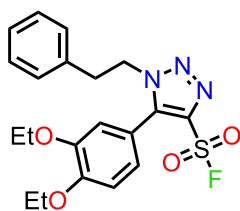
Following general procedure E, the title compound was isolated after purification by flash column chromatography (0-20% EtOAc in petroleum ether) as a yellow oil (35.0 mg, 88%). **¹H NMR** (500 MHz, CDCl₃) δ 7.37 (appt. d, *J* = 8.1 Hz, 1H), 7.31 – 7.25 (m, 4H), 7.01 – 6.96 (m, 1H), 6.74 (appt. d, *J* = 7.8 Hz, 1H), 4.54 (t, *J* = 6.8 Hz, 2H), 3.95 (appt. t, *J* = 5.6 Hz, 1H), 3.01 (hept, *J* = 6.9 Hz, 1H), 2.48 – 2.40 (m, 2H), 1.33 (d, *J* = 6.9 Hz, 6H); **¹³C NMR** (126 MHz, CDCl₃) δ 158.1, 152.6, 142.2 (d, *J* = 2.7 Hz), 137.6 (d, *J* = 35.4 Hz), 129.7, 129.6, 127.5, 121.4, 120.0, 114.4, 63.6, 46.3, 34.2, 29.5, 23.8; **¹⁹F NMR** (376 MHz, CDCl₃) δ 68.5; **HRMS** (ESI⁺): calculated for C₂₀H₂₃FN₃O₃SNa [M+H⁺]: *m/z* = 426.1258, *m/z* found 426.1254; **IR** *v*_{max} (ATR)/cm⁻¹: 2962, 2874, 1600, 1495, 1422, 1242, 1201, 777, 754.

1-(Cyclohexylmethyl)-5-(2-methoxyphenyl)-1*H*-1,2,3-triazole-4-sulfonyl fluoride (4cp)



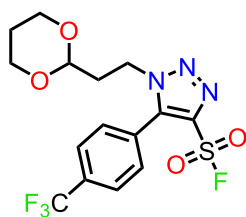
Following general procedure E, the title compound was isolated after purification by flash column chromatography (0-20% EtOAc in petroleum ether) as a yellow oil (32.0 mg, 91%). ¹H NMR (400 MHz, CDCl₃) δ 7.58 (ddd, *J* = 8.4, 7.5, 1.8 Hz, 1H), 7.22 (dd, *J* = 7.6, 1.7 Hz, 1H), 7.13 (appt. td, *J* = 7.5, 0.9 Hz, 1H), 7.07 (d, *J* = 8.4 Hz, 1H), 4.04 (ddd, *J* = 61.5, 13.5, 7.4 Hz, 2H), 3.81 (s, 3H), 1.95 – 1.82 (m, 1H), 1.70 – 1.58 (m, 3H), 1.59 – 1.44 (m, 2H), 1.22 – 1.06 (m, 3H), 0.89 – 0.77 (m, 2H); ¹³C NMR (101 MHz, CDCl₃) δ 157.3, 139.1 (d, *J* = 3.0 Hz), 138.0 (d, *J* = 35.1 Hz), 133.3, 131.2, 121.1, 112.2, 111.7, 55.8, 55.4, 38.1, 30.4 (d, *J* = 5.4 Hz), 26.0, 25.5; ¹⁹F NMR (376 MHz, CDCl₃) δ 66.9; HRMS (ESI⁺): calculated for C₁₆H₂₁FN₃O₃S [M+H⁺]: *m/z* = 354.1282, *m/z* found 354.1281; IR *v*_{max} (ATR)/cm⁻¹: 2927, 2852, 1609, 1486, 1419, 1200, 772.

5-(3,4-Diethoxyphenyl)-1-phenethyl-1*H*-1,2,3-triazole-4-sulfonyl fluoride (4cq)



Following general procedure E, the title compound was isolated after purification by flash column chromatography (0-20% EtOAc in petroleum ether) as a yellow oil (36.0 mg, 86%). ¹H NMR (500 MHz, CDCl₃) δ 7.26 – 7.21 (m, 3H), 6.91 – 6.86 (m, 3H), 6.52 – 6.48 (m, 2H), 4.51 (t, *J* = 7.1 Hz, 2H), 4.15 (q, *J* = 7.0 Hz, 2H), 4.00 (q, *J* = 7.0 Hz, 2H), 3.22 (t, *J* = 7.0 Hz, 2H), 1.50 (t, *J* = 7.0 Hz, 3H), 1.45 (t, *J* = 7.0 Hz, 3H); ¹³C NMR (126 MHz, CDCl₃) δ 151.3, 148.9, 142.1 (d, *J* = 2.5 Hz), 137.4 (d, *J* = 35.1 Hz), 136.2, 129.0, 128.8, 127.5, 122.5, 114.4, 114.2, 112.9, 65.0, 64.7, 50.5, 36.3, 14.8, 14.7; ¹⁹F NMR (376 MHz, CDCl₃) δ 68.4; HRMS (ESI⁺): calculated for C₂₀H₂₂FN₃O₄SNa [M+Na⁺]: *m/z* = 442.1207, *m/z* found 442.1209; IR *v*_{max} (ATR)/cm⁻¹: 2983, 2931, 1606, 1501, 1421, 1258, 1196, 1142, 1039, 770.

1-(2-(1,3-Dioxan-2-yl)ethyl)-5-(4-(trifluoromethyl)phenyl)-1*H*-1,2,3-triazole-4-sulfonyl fluoride (4cr)

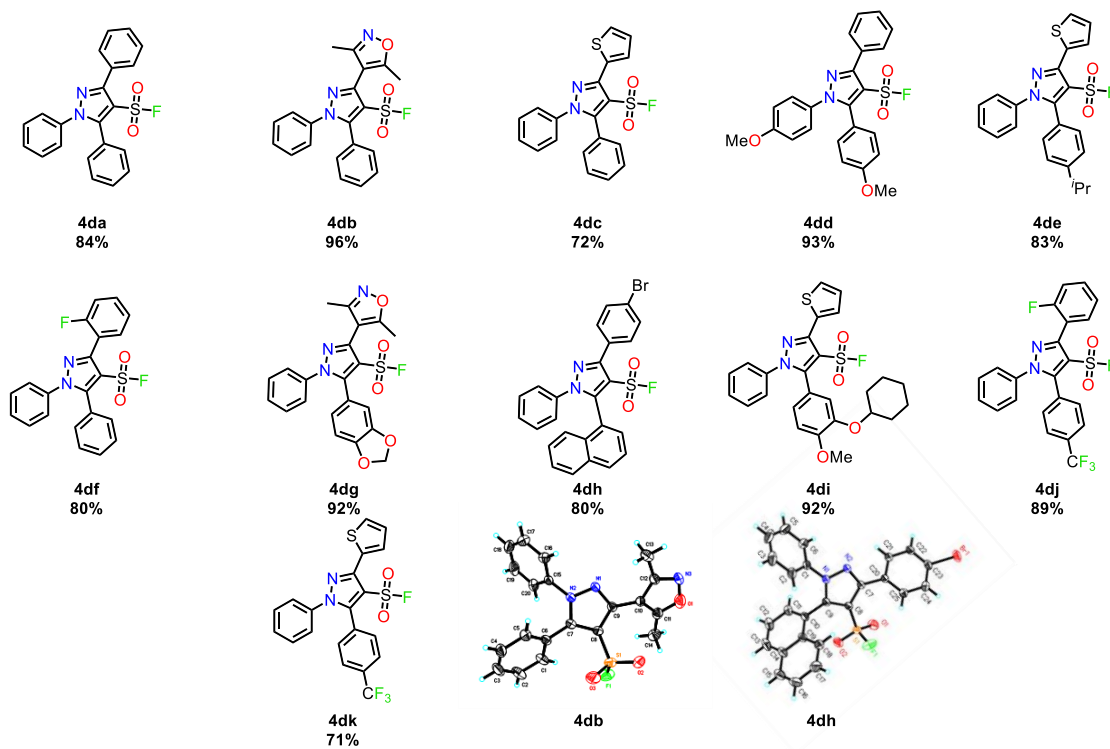
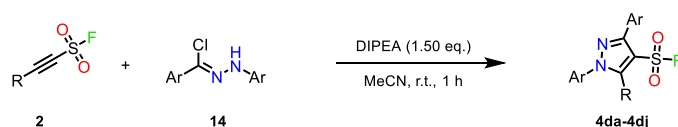


Following general procedure E, the title compound was isolated after purification by flash column chromatography (0-30% EtOAc in petroleum ether) as a yellow oil (25.0 mg, 61%). ¹H NMR (400 MHz, CDCl₃) δ 7.86 (d, *J* = 8.1 Hz, 2H), 7.58 (d, *J* = 8.0 Hz, 2H), 4.58 (t, *J* = 4.4 Hz, 1H), 4.44 (t, *J* = 7.0 Hz, 2H), 4.04 – 3.95 (m, 2H), 3.72 – 3.61 (m, 2H), 2.20 (td, *J* = 7.0, 4.5 Hz, 2H), 1.94 (qt, *J* = 12.5, 5.0 Hz, 1H), 1.35 – 1.28 (m, 1H); ¹³C NMR (101 MHz, CDCl₃) δ 140.2 (d, *J* = 2.9 Hz), 138.1 (d, *J* = 36.0 Hz), 133.6 (q, *J* = 33.3 Hz), 130.5, 127.0, 126.4 (q, *J* = 3.7 Hz), 123.5 (d, *J* = 272.8 Hz), 98.7, 66.9, 44.8, 34.6, 25.5; ¹⁹F NMR (376 MHz, CDCl₃) δ 68.7, -63.2; HRMS (ESI⁺): calculated for C₁₅H₁₅F₄N₃O₄Na [M+Na⁺]: *m/z* = 410.0792, *m/z* found 410.0793; IR *v*_{max} (ATR)/cm⁻¹: 2967, 2858, 1424, 1323, 1202, 1127, 1070, 1003, 849, 785

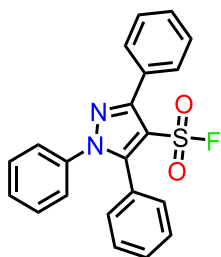
Synthesis and experimental data for compounds 4da-4dk

General procedure F

To a solution of the required SASF **2** (500 μmol) and the required hydrazonoyl chloride **14** (250 μmol) in MeCN (500 μL) was added DIPEA (65.2 μL , 375 μmol) and stirred at room temperature for 1 h. The reaction mixture was then extracted into EtOAc (10.0 mL) washed with brine (10.0 mL) and H_2O (2 x 10.0 mL). The aqueous layer was extracted with EtOAc (10.0 mL), the organic fractions combined, dried over anhydrous MgSO_4 , filtered under vacuum and the solvent was removed under reduced pressure. The crude mixture was purified by flash column chromatography to obtain the analytically pure product.

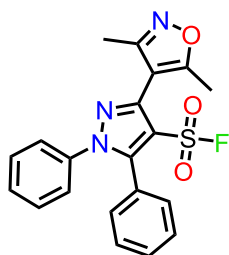


1,3,5-Triphenyl-1*H*-pyrazole-4-sulfonyl fluoride (4da)



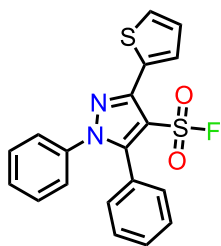
Following general procedure F, the title compound was isolated after purification by flash column chromatography (0-5% EtOAc in petroleum ether) as an off white solid (80.0 mg, 84%). **m.p.** 138 - 139 °C; **¹H NMR** (500 MHz, CDCl₃) δ = 7.85 – 7.81 (m, 2H), 7.53 – 7.46 (m, 4H), 7.44 – 7.40 (m, 2H), 7.39 – 7.36 (m, 2H), 7.36 – 7.32 (m, 3H), 7.30 – 7.27 (m, 2H); **¹³C NMR** (126 MHz, CDCl₃) δ = 152.8, 147.8 (d, *J* = 2.0 Hz), 138.4, 130.6, 130.5, 130.3, 129.8, 129.5, 129.2, 129.0, 128.7, 128.5, 126.8, 125.6, 112.3 (d, *J* = 31.2 Hz); **¹⁹F NMR** (376 MHz, CDCl₃) δ = 73.7; **HRMS** (ESI⁺): calculated for C₂₁H₁₆FN₂O₂S [M+H⁺]: *m/z* = 379.0911, *m/z* found 379.0912; **IR** *v*_{max} (ATR)/cm⁻¹: 3069, 2925, 2853, 1961, 1594, 1494, 1449, 1408, 1221, 1184, 774, 690.

3-(3,5-Dimethylisoxazol-4-yl)-1,5-diphenyl-1*H*-pyrazole-4-sulfonyl fluoride (4db)



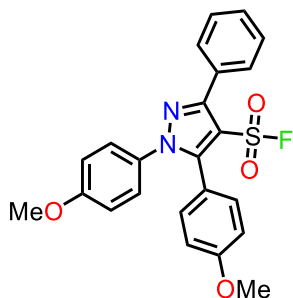
Following general procedure F, the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as an off white solid (95.0 mg, 96%). **m.p.** 158 - 161 °C; **¹H NMR** (500 MHz, CDCl₃) δ = 7.52 – 7.48 (m, 1H), 7.46 – 7.41 (m, 2H), 7.40 – 7.33 (m, 5H), 7.26 – 7.23 (m, 2H), 2.51 (s, 3H), 2.37 (s, 3H); **¹³C NMR** (126 MHz, CDCl₃) δ = 169.2, 159.5, 147.5, 143.2, 138.1, 130.9, 130.3, 129.4, 129.3, 128.9, 126.2, 125.3, 114.0 (d, *J* = 30.4 Hz), 106.9, 12.2, 10.8; **¹⁹F NMR** (376 MHz, CDCl₃) δ = 73.5; **HRMS** (ESI⁺): calculated for C₂₀H₁₆FN₃O₃SNa [M+Na⁺]: *m/z* = 420.0789, *m/z* found 420.0787; **IR** *v*_{max} (ATR)/cm⁻¹: 3067, 2926, 1642, 1496, 1409, 1221, 1188, 769.

1,5-Diphenyl-3-(thiophen-2-yl)-1H-pyrazole-4-sulfonyl fluoride (4dc)



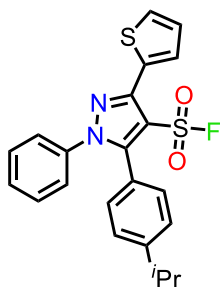
Following general procedure F, the title compound was isolated after purification by flash column chromatography (0-5% EtOAc in petroleum ether) as a beige solid (69.0 mg, 72%). **m.p.** 142 - 143 °C; **¹H NMR** (400 MHz, CDCl₃) δ = 7.85 – 7.79 (m, 1H), 7.51 – 7.42 (m, 2H), 7.45 – 7.36 (m, 2H), 7.37 – 7.29 (m, 5H), 7.28 – 7.21 (m, 2H), 7.18 (dd, *J* = 5.1, 3.7 Hz, 1H); **¹³C NMR** (126 MHz, CDCl₃) δ = 148.3 (d, *J* = 2.6 Hz), 146.3, 138.1, 131.1, 130.6, 130.5, 129.7 (d, *J* = 1.4 Hz), 129.2, 129.1, 128.6, 128.0, 128.0, 126.6, 125.7, 111.2 (d, *J* = 31.9 Hz); **¹⁹F NMR** (471 MHz, CDCl₃) δ = 71.5; **HRMS** (ESI⁺): calculated for C₁₉H₁₄FN₂O₂S₂ [M+H⁺]: *m/z* = 385.0475, *m/z* found 385.0481; **IR** ν_{max} (ATR)/cm⁻¹: 2967, 2923, 1477, 1403, 1214, 1056, 765.

1,5-Bis(4-methoxyphenyl)-3-phenyl-1H-pyrazole-4-sulfonyl fluoride (4dd)



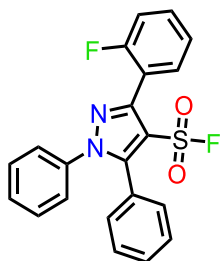
Following general procedure F, the title compound was isolated after purification by flash column chromatography (0-10% EtOAc in petroleum ether) as a colourless solid (102 mg, 93%). **m.p.** 183 - 184 °C; **¹H NMR** (500 MHz, CDCl₃) δ = 7.82 – 7.77 (m, 2H), 7.52 – 7.47 (m, 3H), 7.28 (appt. d, *J* = 8.8 Hz, 2H), 7.20 (appt. d, *J* = 9.0 Hz, 2H), 6.92 (appt. d, *J* = 8.8 Hz, 2H), 6.84 (appt. d, *J* = 9.0 Hz, 2H), 3.84 (s, 3H), 3.81 (s, 3H); **¹³C NMR** (126 MHz, CDCl₃) δ = 161.1, 159.8, 152.5, 147.7, 131.9, 131.5, 130.5, 129.7, 129.5, 128.4, 127.0, 118.8, 114.4, 114.2, 111.6 (d, *J* = 31.0 Hz), 55.7, 55.4; **¹⁹F NMR** (471 MHz, CDCl₃) δ = 73.7; **HRMS** (ESI⁺): calculated for C₂₃H₂₀FN₂O₄S [M+H⁺]: *m/z* = 439.1122, *m/z* found 439.1142; **IR** ν_{max} (ATR)/cm⁻¹: 3010, 2971, 2923, 1610, 1519, 1478, 1405, 1250, 1226, 1169, 1027, 757.

5-(4-isopropylphenyl)-1-phenyl-3-(thiophen-2-yl)-1H-pyrazole-4-sulfonyl fluoride (4de)



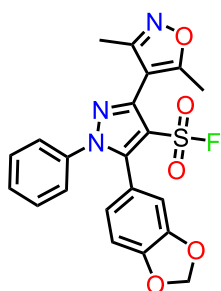
Following general procedure F, the title compound was isolated after purification by flash column chromatography (0-10% EtOAc in petroleum ether) as a Yellow solid (89.0 mg, 83%) **m.p.** 134 - 136 °C; **¹H NMR** (400 MHz, CDCl₃) δ = 7.81 (ddd, *J* = 3.7, 1.2, 0.5 Hz, 1H), 7.46 (dd, *J* = 5.1, 1.1 Hz, 1H), 7.36 – 7.30 (m, 3H), 7.26 – 7.22 (m, 6H), 7.17 (dd, *J* = 5.1, 3.7 Hz, 1H), 2.93 (hept, *J* = 6.9 Hz, 1H), 1.26 (d, *J* = 6.9 Hz, 6H); **¹³C NMR** (126 MHz, CDCl₃) δ = 151.5, 148.5 (d, *J* = 2.5 Hz), 146.3, 138.3, 131.2, 130.4, 129.7 (d, *J* = 1.6 Hz), 129.1, 129.0, 128.0, 127.9, 126.7, 125.7, 123.8, 111.0 (d, *J* = 31.7 Hz), 34.1, 23.8; **¹⁹F NMR** (376 MHz, CDCl₃) δ = 71.4; **HRMS** (ESI⁺): calculated for C₂₂H₂₀FN₂O₂S₂ [M+H⁺]: *m/z* = 427.0945, *m/z* found 427.0951; **IR** *v*_{max} (ATR)/cm⁻¹: 2962, 2927, 1410, 1217, 1191, 839, 757, 708.

3-(2-Fluorophenyl)-1,5-diphenyl-1H-pyrazole-4-sulfonyl fluoride (4df)



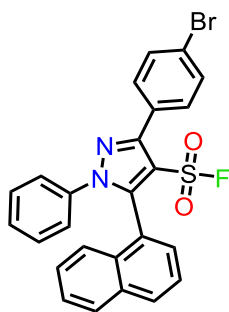
Following general procedure F, the title compound was isolated after purification by flash column chromatography (0-5% EtOAc in petroleum ether) as an off white solid (79.0 mg, 80%). **m.p.** 168 - 169 °C; **¹H NMR** (500 MHz, CDCl₃) δ = 7.65 (appt. td, *J* = 7.4, 1.8 Hz, 1H), 7.53 – 7.46 (m, 2H), 7.45 – 7.39 (m, 4H), 7.37 – 7.33 (m, 3H), 7.32 – 7.27 (m, 3H), 7.23 (ddd, *J* = 9.6, 8.3, 1.1 Hz, 1H); **¹³C NMR** (126 MHz, CDCl₃) δ = 160.8 (d, *J* = 249.8 Hz), 147.8, 147.3, 138.3, 131.9 (d, *J* = 8.3 Hz), 131.6 (d, *J* = 2.5 Hz), 130.7, 130.4, 129.3, 129.1, 128.8, 126.5, 125.6, 124.2 (d, *J* = 3.6 Hz), 118.8 (d, *J* = 15.0 Hz), 115.9 (d, *J* = 21.4 Hz), 113.8 (d, *J* = 31.0 Hz); **¹⁹F NMR** (376 MHz, CDCl₃) δ = 72.0 (d, *J* = 11.5 Hz), -112.8 – -113.2 (m); **HRMS** (ESI⁺): calculated for C₂₁H₁₅F₂N₂O₅S [M+H⁺]: *m/z* = 397.0817, *m/z* found 397.0835; **IR** *v*_{max} (ATR)/cm⁻¹: 3064, 2923, 2853, 1497, 1458, 1405, 1216, 1182, 764.

5-(Benzo[d][1,3]dioxol-5-yl)-3-(3,5-dimethylisoxazol-4-yl)-1-phenyl-1H-pyrazole-4-sulfonyl fluoride (4dg)



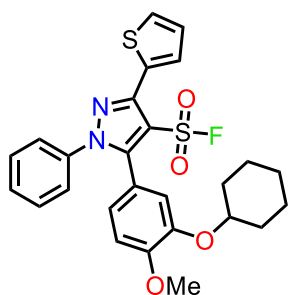
Following general procedure F, the title compound was isolated after purification by flash column chromatography (0-20% EtOAc in petroleum ether) as a colourless solid (101 mg, 92%). **m.p.** 178 - 180 °C; **¹H NMR** (500 MHz, CDCl₃) δ = 7.42 – 7.36 (m, 3H), 7.31 – 7.27 (m, 2H), 6.88 – 6.83 (m, 2H), 6.82 – 6.79 (m, 1H), 6.05 (s, 2H), 2.49 (s, 3H), 2.35 (s, 3H); **¹³C NMR** (126 MHz, CDCl₃) δ = 169.2, 159.5, 149.9, 148.2, 147.1, 143.2, 138.2, 129.5, 129.3, 125.2, 125.1, 119.2, 113.9 (d, *J* = 30.1 Hz), 110.3, 108.9, 107.0, 102.0, 12.2, 10.9; **¹⁹F NMR** (471 MHz, CDCl₃) δ = 73.4; **HRMS** (ESI⁺): calculated for C₂₁H₁₇FN₃O₅S [M+H⁺]: *m/z* = 442.0867, *m/z* found 442.0874; **IR** *v*_{max} (ATR)/cm⁻¹: 3070, 1646, 1484, 1454, 1417, 1236, 1217, 1178, 1039, 756.

3-(4-Bromophenyl)-5-(naphthalen-1-yl)-1-phenyl-1H-pyrazole-4-sulfonyl fluoride (4dh)



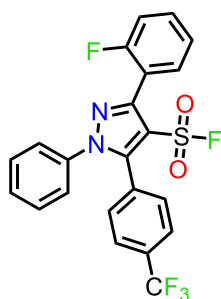
Following general procedure F (0.1 mmol of the required hydrazonoyl chloride), the title compound was isolated after purification by flash column chromatography (0-10% EtOAc in petroleum ether) as a colourless solid (41.0 mg, 80%). **m.p.** 161 - 164 °C; **¹H NMR** (500 MHz, CDCl₃) δ = 8.01 – 7.95 (m, 1H), 7.93 – 7.88 (m, 1H), 7.83 – 7.79 (m, 2H), 7.71 – 7.66 (m, 2H), 7.60 – 7.57 (m, 1H), 7.55 – 7.48 (m, 4H), 7.25 – 7.15 (m, 5H); **¹³C NMR** (126 MHz, CDCl₃) δ = 151.5, 146.9 (d, *J* = 1.9 Hz), 138.4, 133.3, 132.1, 131.8, 131.4, 131.1, 129.6, 129.1, 129.1, 128.9, 127.8, 126.8, 125.1, 124.7, 124.5, 124.4, 124.3, 113.8 (d, *J* = 31.4 Hz). **¹⁹F NMR** (376 MHz, CDCl₃) δ = 72.73; **HRMS** (ESI⁺): calculated for C₂₅H₁₇BrFN₂O₂S [M+H⁺]: *m/z* = 507.0173, *m/z* found 507.0156; **IR** *v*_{max} (ATR)/cm⁻¹: 3056, 2924, 2854, 1595, 1492, 1414, 1226, 767, 745.

5-(3-(Cyclohexyloxy)-4-methoxyphenyl)-1-phenyl-3-(thiophen-2-yl)-1H-pyrazole-4-sulfonyl fluoride (4di)



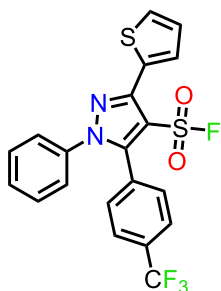
Following general procedure F (100 μ mol of the required hydrazoneyl chloride), the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a colourless solid (47.0 mg, 92%). **m.p.** 69 - 71 $^{\circ}$ C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.79 (d, J = 3.7 Hz, 1H), 7.46 (dd, J = 5.1, 0.9 Hz, 1H), 7.36 - 7.31 (m, 3H), 7.29 - 7.24 (m, 2H), 7.17 (dd, J = 5.1, 3.8 Hz, 1H), 6.92 - 6.84 (m, 2H), 6.76 (d, J = 1.4 Hz, 1H), 4.30 - 4.20 (m, 1H), 3.70 (s, 3H), 2.08 - 2.00 (m, 2H), 1.88 - 1.79 (m, 2H), 1.64 - 1.52 (m, 3H), 1.42 - 1.28 (m, 3H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 150.0, 149.2, 148.2 (d, J = 2.5 Hz), 146.4, 138.4, 131.2, 129.7, 129.7, 129.2, 128.9, 128.0, 127.9, 125.6, 123.8, 118.3, 114.6, 114.3, 110.8 (d, J = 31.6 Hz), 56.2, 32.0, 25.6, 24.2; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ 71.4; **HRMS** (ESI $^+$): calculated for $\text{C}_{26}\text{H}_{26}\text{FN}_2\text{O}_4\text{S}_2$ [$\text{M}+\text{H}^+$]: m/z = 513.1313, m/z found 513.1309; **IR** ν_{max} (ATR)/ cm^{-1} : 3104, 2934, 2854, 1604, 1488, 1410, 1254, 1215, 1139, 763.

3-(2-Fluorophenyl)-1-phenyl-5-(4-(trifluoromethyl)phenyl)-1H-pyrazole-4-sulfonyl fluoride (4dj)



Following general procedure F (100 μ mol of the required hydrazoneyl chloride), the title compound was isolated after purification by flash column chromatography (0-10% EtOAc in petroleum ether) as a yellow solid (41.0 mg, 89%). **m.p.** 164 - 165 $^{\circ}$ C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.83 - 7.78 (m, 2H), 7.68 (d, J = 8.0 Hz, 2H), 7.51 (d, J = 8.0 Hz, 2H), 7.41 - 7.34 (m, 3H), 7.27 - 7.24 (m, 2H), 7.21 (appt. t, J = 8.7 Hz, 2H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 164.0 (d, J = 250.0 Hz), 152.0, 146.3, 137.9, 132.6 (q, J = 33.0 Hz), 131.5 (d, J = 8.6 Hz), 131.1, 130.4 (appt. d, J = 1.1 Hz), 129.6, 129.6, 126.0 (d, J = 3.4 Hz), 125.8 (q, J = 3.7 Hz), 125.7, 123.7 (q, J = 272.7 Hz), 115.8 (d, J = 21.9 Hz), 112.8 (d, J = 31.8 Hz); $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ 74.0, -63.0, -110.8 - -110.9 (m); **HRMS** (ESI $^+$): calculated for $\text{C}_{22}\text{H}_{14}\text{F}_5\text{N}_2\text{O}_2\text{S}$ [$\text{M}+\text{H}^+$]: m/z = 465.0691, m/z found 465.0690; **IR** ν_{max} (ATR)/ cm^{-1} : 3024, 2885, 2785, 1608, 1493, 1410, 1334, 1219, 1159, 1122, 1070, 838, 787, 761, 689.

1-Phenyl-3-(thiophen-2-yl)-5-(4-(trifluoromethyl)phenyl)-1H-pyrazole-4-sulfonyl fluoride (4dk)

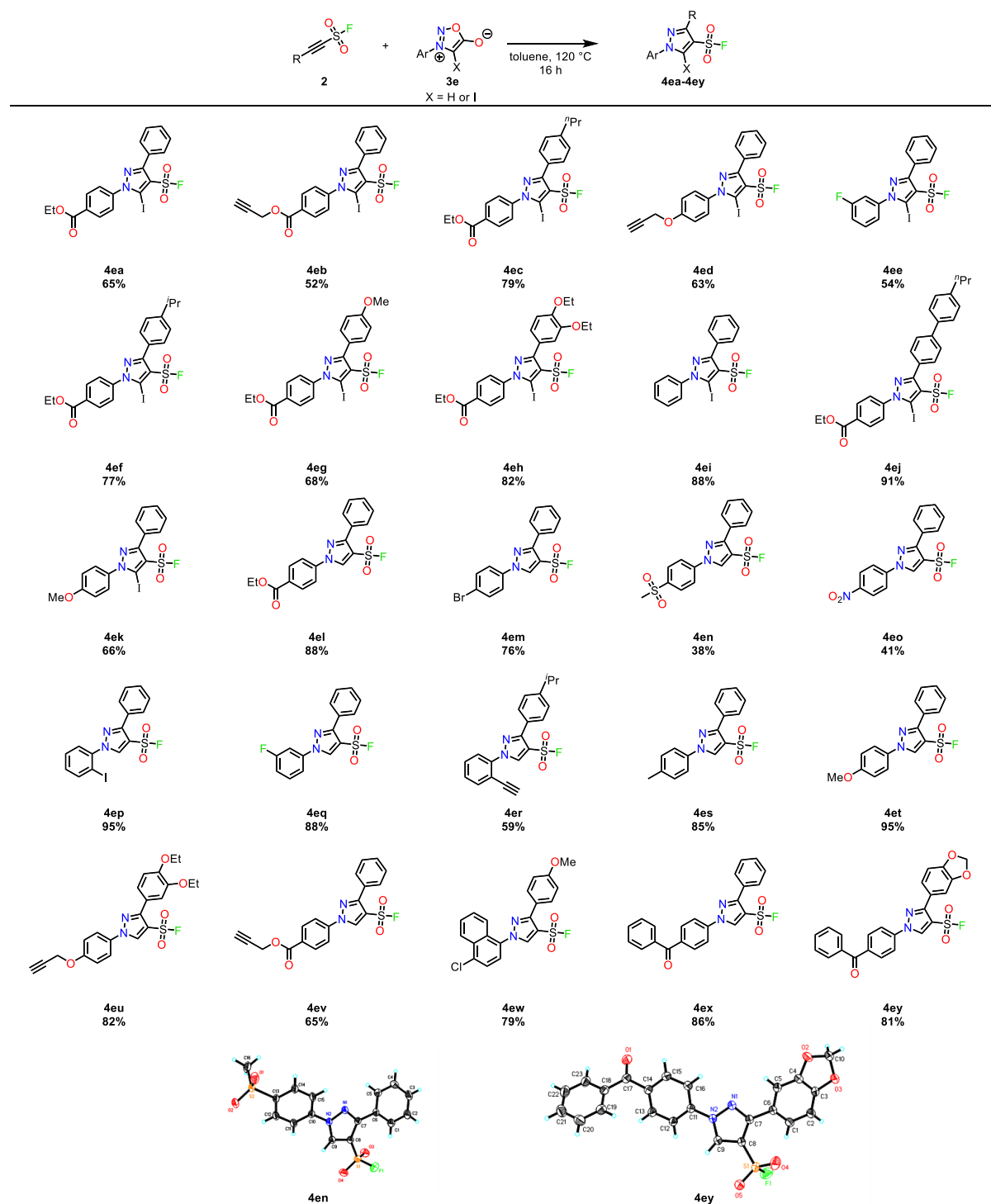


Following general procedure F (100 μ mol of the required hydrazoneyl chloride), the title compound was isolated after purification by flash column chromatography (0-10% EtOAc in petroleum ether) as a yellow solid (32.0 mg, 71%). **m.p.** 175 - 177 $^{\circ}$ C; **1 H NMR** (400 MHz, CDCl_3) δ 7.83 (d, J = 3.6 Hz, 1H), 7.67 (d, J = 8.2 Hz, 2H), 7.50 – 7.47 (m, 2H), 7.47 (s, 1H), 7.39 – 7.33 (m, 3H), 7.26 – 7.21 (m, 2H), 7.19 (dd, J = 5.1, 3.8 Hz, 1H); **13 C NMR** (101 MHz, CDCl_3) δ 146.5, 137.6, 132.4 (q, J = 33.0 Hz), 131.0, 130.6, 130.2 (q, J = 1.0 Hz), 129.8 (d, J = 1.4 Hz), 129.4, 129.4, 128.1, 128.0, 125.6, 125.5 (q, J = 3.7 Hz), 123.5 (q, J = 272.6 Hz), 111.6 (d, J = 32.5 Hz); **19 F NMR** (376 MHz, CDCl_3) δ 71.5, -63.0. **HRMS** (ESI $^+$): calculated for $\text{C}_{20}\text{H}_{13}\text{F}_4\text{N}_2\text{O}_2\text{S}_2$ [$\text{M}+\text{H}^+$]: m/z = 453.0349, m/z found 453.0350; **IR** ν_{max} (ATR)/ cm^{-1} : 3112, 3084, 2923, 1410, 1322, 1218, 1170, 1141, 1122, 1066, 850, 767, 709.

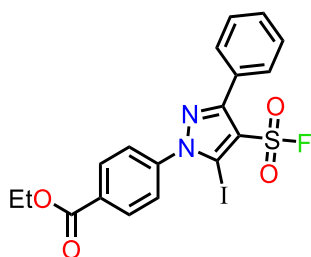
Synthesis and experimental data for compounds 4ea-4ay

General Procedure G

A vial containing a solution of the required SASF **2** (250 μmol) and the required Sydnone **3e** (250 μmol) in toluene (2.00 mL) was sealed and stirred at 120 $^{\circ}\text{C}$ for 16 h. The reaction mixture was cooled, unsealed and the solvent was removed under reduced pressure. The crude product was purified by flash column chromatography to obtain the analytically pure product.

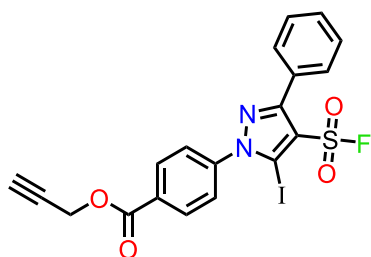


Ethyl 4-(4-(fluorosulfonyl)-5-iodo-3-phenyl-1H-pyrazol-1-yl)benzoate (4ea)



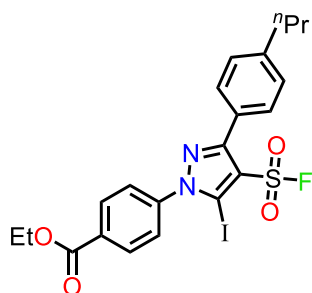
Following general procedure G, the title compound was isolated by flash column chromatography (0-30% EtOAc in petroleum ether) as a pale yellow solid (81.0 mg, 65%). **m.p.**; 137 - 138 °C; **¹H NMR** (400 MHz, CDCl₃) δ 8.26 (appt. d, *J* = 8.6 Hz, 2H), 7.74 – 7.63 (m, 4H), 7.52 – 7.44 (m, 3H), 4.45 (q, *J* = 7.1 Hz, 2H), 1.45 (t, *J* = 7.1 Hz, 3H); **¹³C NMR** (101 MHz, CDCl₃) δ 165.3, 155.3, 142.7, 132.3, 130.8, 130.2, 129.6, 129.4, 128.5, 127.1, 119.6 (d, *J* = 32.5 Hz), 91.8, 61.8, 14.4; **¹⁹F NMR** (376 MHz, CDCl₃) δ 72.2; **HRMS** (ESI⁺): calculated for C₁₈H₁₅FIN₂O₄S [M+H⁺]: *m/z* = 500.9776, *m/z* found 500.9775; **IR** *v*_{max} (ATR)/cm⁻¹: 3073, 3003, 1702, 1606, 1411, 1281.

Prop-2-yn-1-yl 4-(4-(fluorosulfonyl)-5-iodo-3-phenyl-1H-pyrazol-1-yl)benzoate (4eb)



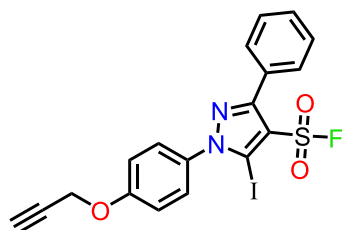
Following general procedure G (110 μmol of the required SASF), the title compound was isolated by flash column chromatography (0-10% EtOAc in petroleum ether) as a colourless oil (29.0 mg, 52%). **¹H NMR** (400 MHz, CDCl₃) δ 8.29 (appt. d, *J* = 8.0 Hz, 2H), 7.71–7.67 (m, 4H), 7.53 – 7.45 (m, 3H), 5.00 (d, *J* = 2.5 Hz, 2H), 2.57 (t, *J* = 2.5 Hz, 1H); **¹³C NMR** (101 MHz, CDCl₃) δ 164.6, 155.4, 143.1, 131.2, 131.1, 130.3, 129.6, 129.5, 128.5, 127.2, 119.8 (d, *J* = 32.5 Hz), 91.7, 75.7, 53.2; **¹⁹F NMR** (376 MHz, CDCl₃) δ 72.2; **HRMS** (ESI⁺): calculated for C₁₉H₁₃FIN₂O₄S [M+H⁺]: *m/z* = 510.9619, *m/z* found 510.9638. **IR** *v*_{max} (ATR)/cm⁻¹: 3304, 2928, 1728, 1607, 1413, 1267.

Ethyl 4-(4-(fluorosulfonyl)-5-iodo-3-(4-propylphenyl)-1H-pyrazol-1-yl)benzoate (4ec)



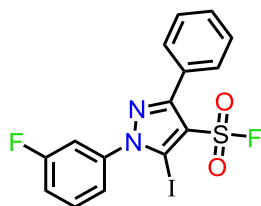
Following general procedure G, the title compound was isolated by flash column chromatography (0-8% EtOAc in petroleum ether) as white solid (43.0 mg, 79%). **m.p.** 139 - 140 °C; **¹H NMR** (400 MHz, CDCl₃) δ 8.25 (appt. d, *J* = 8.7 Hz, 2H), 7.67 (appt. d, *J* = 8.7 Hz, 2H), 7.60 (appt. d, *J* = 8.2 Hz, 2H), 7.29 (appt. d, *J* = 8.3 Hz, 2H), 4.45 (q, *J* = 7.1 Hz, 2H), 2.68 – 2.63 (m, 2H), 1.74 – 1.63 (m, 2H), 1.45 (t, *J* = 7.1 Hz, 3H), 0.97 (t, *J* = 7.3 Hz, 3H); **¹³C NMR** (126 MHz, CDCl₃) δ 165.4, 155.4, 145.1, 142.8, 132.3, 130.8, 129.3, 128.7, 127.1, 126.9, 119.4 (d, *J* = 32.4 Hz), 91.6, 61.8, 38.0, 24.4, 14.4, 14.0; **¹⁹F NMR** (471 MHz, CDCl₃) δ 72.1; **HRMS** (ESI⁺): calculated for C₂₁H₂₁FIN₂O₄S [M+H⁺]: *m/z* = 543.0245, *m/z* found 543.0248; **IR** *v*_{max} (ATR)/cm⁻¹: 2989, 2875, 1714, 1606, 1456, 1410, 1280, 1220.

5-Iodo-3-phenyl-1-(4-(prop-2-yn-1-yloxy)phenyl)-1H-pyrazole-4-sulfonyl fluoride (4ed)



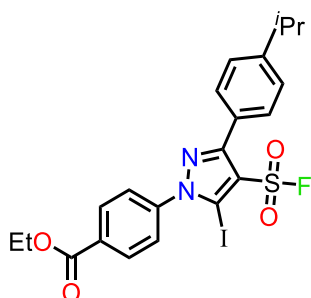
Following general procedure G, the title compound was isolated by flash column chromatography (0-15% EtOAc in petroleum ether) as an off-white solid (88.0 mg, 63%). **¹H NMR** (400 MHz, CDCl₃) δ 7.71 – 7.66 (m, 2H), 7.52 – 7.43 (m, 5H), 7.14 (appt. d, *J* = 9.0 Hz, 2H), 4.79 (d, *J* = 2.4 Hz, 2H), 2.59 (t, *J* = 2.4 Hz, 1H); **¹³C NMR** (101 MHz, CDCl₃) δ 158.9, 154.9, 133.2, 130.1, 129.9, 129.5, 128.5, 118.6 (d, *J* = 32.4 Hz), 115.6, 92.8, 77.8, 76.5, 56.3; **¹⁹F NMR** (376 MHz, CDCl₃) δ 72.2; **HRMS** (ESI⁺): calculated for C₁₈H₁₃FIN₂O₃S [M+H⁺]: *m/z* = 482.9670, *m/z* found 482.9670; **IR** *v*_{max} (ATR)/cm⁻¹: 3287, 3073, 2926, 1601, 1513, 1409, 1218.

1-(3-Fluorophenyl)-5-iodo-3-phenyl-1H-pyrazole-4-sulfonyl fluoride (4ee)



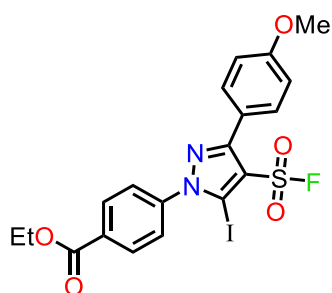
Following general procedure G, the title compound was isolated by flash column chromatography (0-8% EtOAc in petroleum ether) as a pale yellow solid (24.0 mg, 54%). **m.p.** 119 - 120 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.70 – 7.66 (m, 2H), 7.56 (tdd, *J* = 8.1, 5.9, 0.6 Hz, 1H), 7.53 – 7.44 (m, 3H), 7.41 – 7.37 (m, 1H), 7.35 – 7.27 (m, 2H); **¹³C NMR** (126 MHz, CDCl₃) δ 162.6 (d, *J* = 250.3 Hz), 155.2, 140.5 (d, *J* = 9.9 Hz), 130.8 (d, *J* = 8.9 Hz), 130.2, 129.6, 129.4, 128.5, 123.1 (d, *J* = 3.5 Hz), 119.4 (d, *J* = 32.4 Hz), 117.8 (d, *J* = 20.9 Hz), 115.1 (d, *J* = 24.7 Hz), 92.1; **¹⁹F NMR** (471 MHz, CDCl₃) δ 72.2, -109.5 – -109.6 (m); **HRMS** (ESI⁺): calculated for C₁₅H₁₀F₂IN₂O₂S [M+H⁺]: *m/z* = 446.9470, *m/z* found 446.9470; **IR** *v*_{max} (ATR)/cm⁻¹: 2981, 2865, 1600, 1416, 1222, 1055, 766.

Ethyl 4-(4-(fluorosulfonyl)-5-iodo-3-(4-isopropylphenyl)-1H-pyrazol-1-yl)benzoate (4ef)



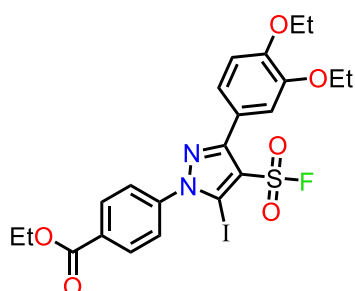
Following general procedure G, the title compound was isolated by flash column chromatography (0-10% EtOAc in petroleum ether) as a pale yellow solid (42.0 mg, 77%). **m.p.** 121 - 122 °C; **¹H NMR** (400 MHz, CDCl₃) δ 8.25 (appt. d, *J* = 8.8 Hz, 2H), 7.67 (appt. d, *J* = 8.8 Hz, 2H), 7.62 (appt. d, *J* = 8.2 Hz, 2H), 7.34 (appt. d, *J* = 8.5 Hz, 2H), 4.45 (q, *J* = 7.1 Hz, 2H), 2.98 (hept, *J* = 6.8 Hz, 1H), 1.45 (t, *J* = 7.1 Hz, 3H), 1.29 (d, *J* = 6.9 Hz, 6H); **¹³C NMR** (126 MHz, CDCl₃) δ 165.4, 155.4, 151.2, 142.8, 132.3, 130.8, 129.4, 127.1, 127.0, 126.7, 119.4 (d, *J* = 32.3 Hz), 91.6, 61.8, 34.2, 24.0, 14.4; **¹⁹F NMR** (376 MHz, CDCl₃) δ 72.1; **HRMS** (ESI⁺): calculated for C₂₁H₂₁FIN₂O₄S [M+H⁺]: *m/z* = 543.0245, *m/z* found 543.0246; **IR** *v*_{max} (ATR)/cm⁻¹: 2960, 2927, 1723, 1607, 1462, 1413, 1274.

Ethyl 4-(4-(fluorosulfonyl)-5-iodo-3-(4-methoxyphenyl)-1H-pyrazol-1-yl)benzoate (4eg)



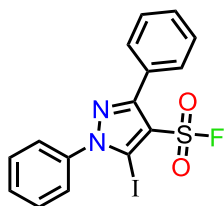
Following general procedure G, the title compound was isolated by flash column chromatography (0-12% EtOAc in petroleum ether) as a pale yellow solid (36.0 mg, 68%). **m.p.** 154 - 156 °C; **¹H NMR** (400 MHz, CDCl₃) δ 8.25 (appt. d, *J* = 8.8 Hz, 2H), 7.69 – 7.61 (m, 4H), 7.00 (appt. d, *J* = 8.9 Hz, 2H), 4.45 (q, *J* = 7.1 Hz, 2H), 3.87 (s, 3H), 1.45 (t, *J* = 7.1 Hz, 3H); **¹³C NMR** (126 MHz, CDCl₃) δ 165.4, 161.2, 155.1, 142.8, 132.3, 130.8, 130.8, 127.1, 121.9, 119.2 (d, *J* = 32.3 Hz), 114.0, 91.7, 61.8, 55.5, 14.4; **¹⁹F NMR** (471 MHz, CDCl₃) δ 72.1; **HRMS** (ESI⁺): calculated for C₁₉H₁₇FIN₂O₅S [M+H⁺]: *m/z* = 530.9881, *m/z* found 500.9884; **IR** *v*_{max} (ATR)/cm⁻¹: 2982, 2832, 1714, 1612, 1462, 1412, 1280.

Ethyl 4-(3-(3,4-diethoxyphenyl)-4-(fluorosulfonyl)-5-iodo-1H-pyrazol-1-yl)benzoate (4eh)



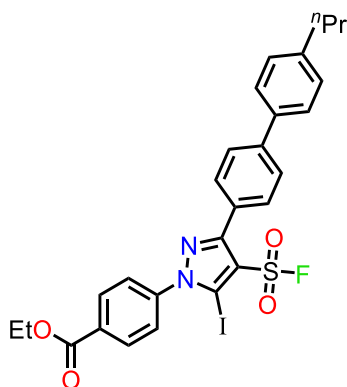
Following general procedure G, the title compound was isolated by flash column chromatography (0-12% EtOAc in petroleum ether) as an off-white solid (48.0 mg, 82%). **m.p.** 127 - 128°C; **¹H NMR** (400 MHz, CDCl₃) δ 8.25 (appt. d, *J* = 8.3 Hz, 2H), 7.66 (appt. d, *J* = 8.3 Hz, 2H), 7.26 (appt. d, *J* = 8.4 Hz, 2H), 6.95 (d, *J* = 8.7 Hz, 1H), 4.45 (q, *J* = 7.1 Hz, 2H), 4.20 – 4.11 (m, *J* = 6.9, 4.5 Hz, 4H), 1.52 – 1.41 (m, 9H); **¹³C NMR** (126 MHz, CDCl₃) δ 165.4, 155.1, 150.5, 148.4, 142.8, 132.3, 130.8, 127.2, 122.4, 121.9, 119.2 (d, *J* = 32.4 Hz), 114.4, 112.7, 91.7, 64.8, 64.6, 61.8, 14.9, 14.9, 14.4; **¹⁹F NMR** (376 MHz, CDCl₃) δ 71.8; **HRMS** (ESI⁺): calculated for C₂₂H₂₃FIN₂O₆S [M+H⁺]: *m/z* = 589.0300, *m/z* found 589.0301; **IR** *v*_{max} (ATR)/cm⁻¹: 2981, 2935, 1716, 1607, 1472, 1420, 1294.

5-Iodo-1,3-diphenyl-1H-pyrazole-4-sulfonyl fluoride (4ei)



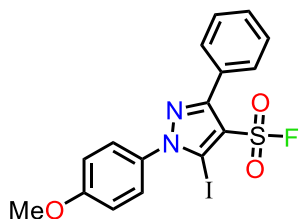
Following general procedure G (250 μmol of SASF), the title compound was isolated by flash column chromatography (0-10% EtOAc in petroleum ether) as a white solid (94.0 mg, 88%). **m.p.** 159 - 160 $^{\circ}\text{C}$; **$^1\text{H NMR}$** (400 MHz, CDCl_3) δ 7.72 – 7.67 (m, 2H), 7.61 – 7.53 (m, 5H), 7.51 – 7.44 (m, 3H); **$^{13}\text{C NMR}$** (126 MHz, CDCl_3) δ 155.0, 139.5, 130.5, 130.1, 129.9, 129.5, 129.5, 128.7, 128.5, 127.1, 126.8, 118.9 (d, $J = 32.4$ Hz), 92.2; **$^{19}\text{F NMR}$** (471 MHz, CDCl_3) δ 72.2; **HRMS** (ESI $^+$): calculated for $\text{C}_{15}\text{H}_{11}\text{FIN}_2\text{O}_2\text{S}$ [$\text{M}+\text{H}^+$]: $m/z = 428.9564$, m/z found 428.9565; **IR** ν_{max} (ATR)/ cm^{-1} : 3075, 2925, 1494, 1468, 1417, 1228.

Ethyl 4-(4-(fluorosulfonyl)-5-iodo-3-(4'-propyl-[1,1'-biphenyl]-4-yl)-1H-pyrazol-1-yl)benzoate (4ej)



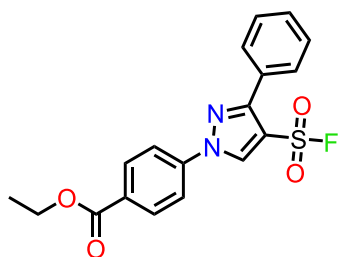
Following general procedure G, the title compound was isolated by flash column chromatography (0-8% EtOAc in petroleum ether) as a pale yellow solid (56.0 mg, 91%). **m.p.** 128 - 129 $^{\circ}\text{C}$; **$^1\text{H NMR}$** (400 MHz, CDCl_3) δ 8.27 (appt. d, $J = 8.8$ Hz, 2H), 7.77 (appt. d, $J = 8.6$ Hz, 2H), 7.72 – 7.67 (m, 4H), 7.57 (appt. d, $J = 8.3$ Hz, 2H), 7.28 (appt. d, $J = 8.4$ Hz, 2H), 4.46 (q, $J = 7.1$ Hz, 2H), 2.69 – 2.62 (m, 2H), 1.75 – 1.65 (m, 2H), 1.45 (t, $J = 7.1$ Hz, 3H), 0.99 (t, $J = 7.3$ Hz, 3H); **$^{13}\text{C NMR}$** (126 MHz, CDCl_3) δ 165.4, 155.1, 143.0, 142.7, 142.6, 137.7, 132.3, 130.8, 129.8, 129.1, 128.1, 127.2, 127.1, 127.0, 119.5 (d, $J = 32.5$ Hz), 91.8, 61.8, 37.9, 24.7, 14.4, 14.0; **$^{19}\text{F NMR}$** (471 MHz, CDCl_3) δ 72.2; **HRMS** (ESI $^+$): calculated for $\text{C}_{27}\text{H}_{25}\text{FIN}_2\text{O}_4\text{S}$ [$\text{M}+\text{H}^+$]: $m/z = 619.0558$, m/z found 619.0556; **IR** ν_{max} (ATR)/ cm^{-1} : 2960, 2926, 1705, 1607, 1464, 1409, 1279, 1218, 772.

5-Iodo-1-(4-methoxyphenyl)-3-phenyl-1H-pyrazole-4-sulfonyl fluoride (4ek)



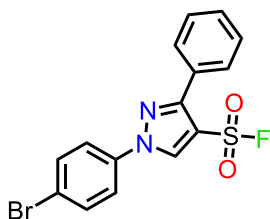
Following general procedure G (250 μ mol of the required SASF), the title compound was isolated by flash column chromatography (0-10% EtOAc in petroleum ether) as a white solid (75.0 mg, 66%). **m.p.** 146 - 148 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.71 – 7.66 (m, 2H), 7.52 – 7.43 (m, 5H), 7.05 (appt. d, J = 9.0 Hz, 2H), 3.90 (s, 3H); **¹³C NMR** (126 MHz, CDCl₃) δ 160.9, 154.8, 132.4, 130.0, 129.9, 129.4, 128.4, 118.4 (d, J = 32.2 Hz), 114.5 (d, J = 9.9 Hz), 93.0, 55.8; **¹⁹F NMR** (471 MHz, CDCl₃) δ 72.2; **HRMS** (ESI⁺): calculated for C₁₆H₁₃FIN₂O₃S [M+H⁺]: m/z = 458.9670, m/z found 458.9670; **IR** ν_{\max} (ATR)/cm⁻¹: 2925, 2851, 1510, 1417, 1254.

Ethyl 4-(4-(fluorosulfonyl)-3-phenyl-1H-pyrazol-1-yl)benzoate (4el)



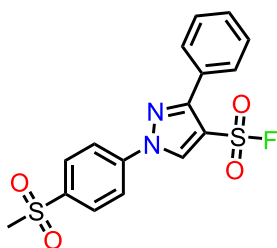
Following general procedure G, the title compound was isolated by flash column chromatography (0-30% EtOAc in petroleum ether) as a white solid (82.0 mg, 88%). **m.p.** 106 - 108 °C; **¹H NMR** (400 MHz, CDCl₃) δ 8.71 (d, J = 1.1 Hz, 1H), 8.24 (appt. d, J = 9.0 Hz, 2H), 7.93 – 7.86 (m, 4H), 7.55 – 7.49 (m, 3H), 4.44 (q, J = 7.1 Hz, 2H), 1.44 (t, J = 7.1 Hz, 3H); **¹³C NMR** (101 MHz, CDCl₃) δ 165.3, 152.2, 141.3, 133.9, 131.4, 130.6, 130.2, 129.1, 128.7, 119.2, 115.1 (d, J = 33.3 Hz), 61.5, 14.3; **¹⁹F NMR** (376 MHz, CDCl₃) δ 69.8; **HRMS** (ESI⁺): calculated for C₁₈H₁₆FN₂O₄S [M+H⁺]: m/z = 375.0809, m/z found 375.0806; **IR** ν_{\max} (ATR)/cm⁻¹: 3107, 2977, 2927, 1699, 1608, 1521, 1410, 1280, 1211, 764.

1-(4-Bromophenyl)-3-phenyl-1H-pyrazole-4-sulfonyl fluoride (4em)



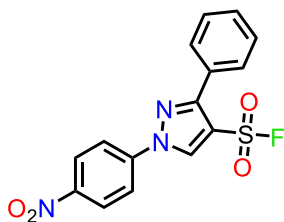
Following general procedure G, the title compound was isolated by flash column chromatography (0-10% EtOAc in petroleum ether) as an off white solid (72.0 mg, 76%). **m.p.** 133 - 135 °C; **¹H NMR** (400 MHz, CDCl₃) δ 8.61 (d, *J* = 1.1 Hz, 1H), 7.90 – 7.86 (m, 2H), 7.68 (s, 4H), 7.54 – 7.49 (m, 3H); **¹³C NMR** (101 MHz, CDCl₃) δ 152.1, 137.5, 133.8, 133.2, 130.3, 129.3, 128.9, 122.6, 121.4, 114.8 (d, *J* = 33.3 Hz); **¹⁹F NMR** (376 MHz, CDCl₃) δ 69.9 (d, *J* = 16.0 Hz); **HRMS** (ESI⁺): calculated for C₁₅H₁₁BrFN₂O₂S [M+H⁺]: *m/z* = 380.9703, *m/z* found 380.9695; **IR** ν_{max} (ATR)/cm⁻¹: 3137, 2965, 2865, 1505, 1402, 1207, 1060, 764.

1-(4-(Methylsulfonyl)phenyl)-3-phenyl-1H-pyrazole-4-sulfonyl fluoride (4en)



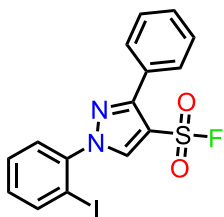
Following general procedure G, the title compound was isolated by flash column chromatography (0-10% EtOAc in petroleum ether) as a white solid (36.0 mg, 38%). **m.p.** 202 - 204 °C; **¹H NMR** (500 MHz, CDCl₃) δ 8.76 (d, *J* = 0.9 Hz, 1H), 8.15 (appt. d, *J* = 8.9 Hz, 2H), 8.04 (appt. d, *J* = 8.9 Hz, 2H), 7.92 – 7.87 (m, 2H), 7.55 – 7.51 (m, 3H), 3.13 (s, 3H); **¹³C NMR** (126 MHz, CDCl₃) δ 152.7, 142.1, 140.6, 134.2 (d, *J* = 2.4 Hz), 130.6, 129.7, 129.0, 128.9, 128.8, 120.3, 115.9 (d, *J* = 33.7 Hz), 44.7; **¹⁹F NMR** (376 MHz, CDCl₃) δ 69.8. **HRMS** (ESI⁺): calculated for C₁₆H₁₄FN₂O₄S₂ [M+H⁺]: *m/z* = 381.0374, *m/z* found 381.0371; **IR** ν_{max} (ATR)/cm⁻¹: 3140, 2926, 1598, 1521, 1393, 1298, 1213, 1147.

1-(4-Nitrophenyl)-3-phenyl-1H-pyrazole-4-sulfonyl fluoride (4eo)



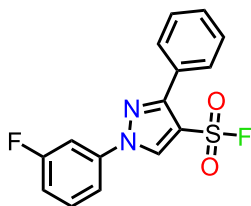
Following general procedure G, the title compound was isolated by flash column chromatography (0-10% EtOAc in petroleum ether) as a pale yellow solid (36.0 mg, 41%). **m.p.** 200 - 202 °C; **¹H NMR** (400 MHz, CDCl₃) δ 8.76 (d, *J* = 1.2 Hz, 1H), 8.45 (appt. d, *J* = 9.4 Hz, 2H), 8.02 (appt. d, *J* = 9.4 Hz, 2H), 7.93 – 7.87 (m, 2H), 7.56 – 7.51 (m, 3H); **¹³C NMR** (101 MHz, CDCl₃) δ 152.9, 147.3, 142.6, 134.3, 130.6, 129.0, 128.9, 128.8, 125.8, 120.0, 116.2 (d, *J* = 33.8 Hz); **¹⁹F NMR** (376 MHz, CDCl₃) δ 69.7; **HRMS** (ESI⁺): calculated for C₁₅H₁₁FN₃O₄S [M+H⁺]: *m/z* = 348.0449, *m/z* found 348.0439; **IR** *v*_{max} (ATR)/cm⁻¹: 3142, 2926, 2853, 1595, 1514, 1398, 1338, 1203;

1-(2-Iodophenyl)-3-phenyl-1H-pyrazole-4-sulfonyl fluoride (4ep)



Following general procedure G, the title compound was isolated by flash column chromatography (0-10% EtOAc in petroleum ether) as a white solid (102 mg, 95%). **m.p.** 85 - 87 °C; **¹H NMR** (400 MHz, CDCl₃) δ 8.44 (s, 1H), 8.03 (appt. d, *J* = 7.9 Hz, 1H), 7.94 – 7.88 (m, 2H), 7.56 – 7.47 (m, 5H), 7.30 – 7.24 (m, 1H) **¹³C NMR** (101 MHz, CDCl₃) δ 151.7, 141.6, 140.6, 138.3 (d, *J* = 2.4 Hz), 131.8, 130.1, 129.6, 129.4, 128.9, 128.8, 128.0, 113.7 (d, *J* = 33.1 Hz), 93.9; **¹⁹F NMR** (376 MHz, CDCl₃) δ 70.1; **HRMS** (ESI⁺): calculated for C₁₅H₁₁FIN₂O₂S [M+H⁺]: *m/z* = 428.9564, *m/z* found 428.9564; **IR** *v*_{max} (ATR)/cm⁻¹: 3126, 1504, 1448, 1402, 1203, 761.

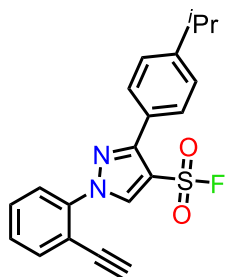
1-(3-Fluorophenyl)-3-phenyl-1H-pyrazole-4-sulfonyl fluoride (4eq)



Following general procedure G, the title compound was isolated by flash column chromatography (0-10% EtOAc in petroleum ether) as a white solid (70.0 mg, 88%). **m.p.** 105 - 107 °C; **¹H NMR** (400 MHz, CDCl₃) δ 8.63 (d, *J* =

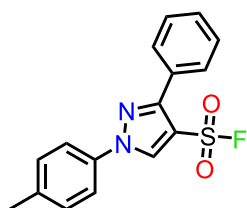
1.1 Hz, 1H), 7.92 – 7.86 (m, 2H), 7.59 (ddt, $J = 8.5, 3.5, 1.6$ Hz, 1H), 7.56 – 7.49 (m, 5H), 7.20 – 7.14 (m, 1H); ^{13}C NMR (101 MHz, CDCl_3) δ 163.4 (d, $J = 249.1$ Hz), 152.1, 139.7 (d, $J = 10.2$ Hz), 134.0 (d, $J = 2.5$ Hz), 131.5 (d, $J = 9.0$ Hz), 130.3, 129.3, 128.9, 115.8 (d, $J = 21.2$ Hz), 115.1 (d, $J = 3.3$ Hz), 114.9 (d, $J = 33.4$ Hz), 108.1 (d, $J = 26.6$ Hz); ^{19}F NMR (376 MHz, CDCl_3) δ 69.9, -109.2 (m); HRMS (ESI⁺): calculated for $\text{C}_{15}\text{H}_{11}\text{F}_2\text{N}_2\text{O}_2\text{S}$ [$\text{M}+\text{H}^+$]: $m/z = 321.0504$, m/z found 321.0507; IR ν_{max} (ATR)/ cm^{-1} : 3142, 2964, 2923, 1606, 1504, 1407, 1209, 1056, 865, 766.

1-(2-Ethynylphenyl)-3-(4-isopropylphenyl)-1H-pyrazole-4-sulfonyl fluoride (4er)



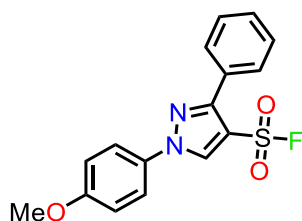
Following general procedure G, the title compound was isolated by flash column chromatography (0-10% EtOAc in petroleum ether) as a pale yellow (54.0 mg, 59%). **m.p.** 89 - 90 °C; ^1H NMR (400 MHz, CDCl_3) δ 8.97 (d, $J = 1.1$ Hz, 1H), 7.85 – 7.79 (m, 3H), 7.70 (dd, $J = 7.8, 1.5$ Hz, 1H), 7.58 – 7.52 (appt. td, $J = 7.6, 1.3$ Hz, 1H), 7.45 (appt. td, $J = 7.6, 1.3$ Hz, 1H), 7.36 (appt. d, $J = 8.1$ Hz, 2H), 3.42 (s, 1H), 2.99 (hept, $J = 6.9$ Hz, 1H), 1.30 (d, $J = 6.9$ Hz, 6H); ^{13}C NMR (101 MHz, CDCl_3) δ 151.7, 151.1, 139.9, 137.6, 134.8, 130.4, 129.0, 128.8, 126.9, 126.9, 125.1, 115.7, 113.3 (d, $J = 32.9$ Hz), 84.6, 79.2, 34.2, 24.0; ^{19}F NMR (376 MHz, CDCl_3) δ 69.8; HRMS (ESI⁺): calculated for $\text{C}_{20}\text{H}_{18}\text{FN}_2\text{O}_2\text{S}$ [$\text{M}+\text{H}^+$]: $m/z = 369.1068$, m/z found 369.1072; IR ν_{max} (ATR)/ cm^{-1} : 3280, 3136, 2970, 1506, 1406, 1206, 771.

3-Phenyl-1-(*p*-tolyl)-1H-pyrazole-4-sulfonyl fluoride (4es)



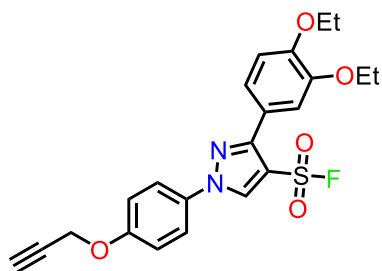
Following general procedure G, the title compound was isolated by flash column chromatography (0-10% EtOAc in petroleum ether) as a pale yellow solid (67.0 mg, 85%). **m.p.** 126 °C; ^1H NMR (400 MHz, CDCl_3) δ 8.58 (s, 1H), 7.89 (dd, $J = 6.6, 2.9$ Hz, 2H), 7.65 (d, $J = 8.4$ Hz, 2H), 7.53 – 7.47 (m, 3H), 7.34 (d, $J = 8.5$ Hz, 2H), 2.44 (s, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 151.8, 139.1, 136.3, 128.9, 128.8, 120.0, 114.0 (d, $J = 32.9$ Hz), 21.2; ^{19}F NMR (376 MHz, CDCl_3) δ 70.1; HRMS (ESI⁺): calculated for $\text{C}_{16}\text{H}_{14}\text{FN}_2\text{O}_2\text{S}$ [$\text{M}+\text{H}^+$]: $m/z = 317.0755$, m/z found 317.0754; IR ν_{max} (ATR)/ cm^{-1} : 3134, 2927, 2853, 1527, 1446, 1407, 1204, 752.

1-(4-Methoxyphenyl)-3-phenyl-1H-pyrazole-4-sulfonyl fluoride (4et)



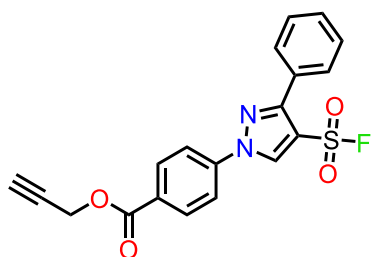
Following general procedure G, the title compound was isolated by flash column chromatography (0-20% EtOAc in petroleum ether) as a white solid (79.0 mg, 95%). **m.p.** 125 - 126 °C; **¹H NMR** (400 MHz, CDCl₃) δ 8.51 (d, *J* = 1.1 Hz, 1H), 7.91 – 7.86 (m, 2H), 7.67 (appt. d, *J* = 9.2 Hz, 2H), 7.53 – 7.47 (m, 3H), 7.04 (appt. d, *J* = 9.2 Hz, 2H); **¹³C NMR** (101 MHz, CDCl₃) δ 159.9, 151.5, 133.6 (d, *J* = 2.2 Hz), 131.9, 129.9, 129.5, 128.7, 128.6, 121.6, 114.9, 113.6 (d, *J* = 32.6 Hz), 55.7; **¹⁹F NMR** (376 MHz, CDCl₃) δ 70.2; **HRMS** (ESI⁺): calculated for C₁₆H₁₄FN₂O₃S [M+H⁺]: *m/z* = 333.0704, *m/z* found 333.0708; **IR** *v*_{max} (ATR)/cm⁻¹: 3114, 2971, 2939, 1522, 1397, 1204, 1018, 752.

3-(3,4-Diethoxyphenyl)-1-(4-(prop-2-yn-1-yloxy)phenyl)-1H-pyrazole-4-sulfonyl fluoride (4eu)



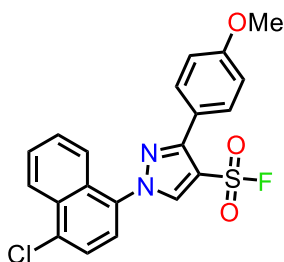
Following general procedure G, the title compound was isolated by flash column chromatography (0-10% EtOAc in petroleum ether) as a white solid (91.0 mg, 82%). **m.p.** 109 - 110 °C; **¹H NMR** (400 MHz, CDCl₃) δ 8.50 (d, *J* = 0.9 Hz, 1H), 7.69 (appt. d, *J* = 9.1 Hz, 2H), 7.49 – 7.44 (m, 3H), 7.12 (appt. d, *J* = 9.1 Hz, 2H), 6.98 (appt. d, *J* = 9.0 Hz, 1H), 4.77 (d, *J* = 2.4 Hz, 2H), 4.18 (qd, *J* = 7.0, 5.6 Hz, 5H), 2.59 – 2.56 (m, 1H), 1.49 (td, *J* = 7.0, 1.1 Hz, 6H); **¹³C NMR** (101 MHz, CDCl₃) δ 157.8, 151.5, 150.4, 148.7, 133.9, 132.8, 122.1, 121.7, 116.1, 113.7, 113.4 (d, *J* = 32.6 Hz), 113.0, 78.0, 76.3, 64.8, 64.6, 56.3, 14.9, 14.9; **¹⁹F NMR** (376 MHz, CDCl₃) δ 69.2; **HRMS** (ESI⁺): calculated for C₂₂H₂₂FN₂O₅S [M+H⁺]: *m/z* = 445.1228, *m/z* found 445.1228; **IR** *v*_{max} (ATR)/cm⁻¹: 3252, 3136, 1518, 1456, 1407, 1209, 761.

Prop-2-yn-1-yl 4-(4-(fluorosulfonyl)-3-phenyl-1H-pyrazol-1-yl)benzoate (4ev)



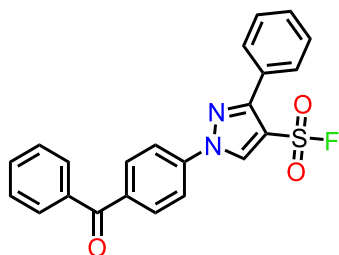
Following general procedure G, the title compound was isolated by flash column chromatography (0-10% EtOAc in petroleum ether) as a white solid (62.0 mg, 65%). **m.p.** 155 - 156 °C **¹H NMR** (400 MHz, CDCl₃) δ 8.72 (d, *J* = 1.0 Hz, 1H), 8.27 (d, *J* = 8.9 Hz, 2H), 7.95 – 7.85 (m, 4H), 7.56 – 7.49 (m, 3H), 4.98 (d, *J* = 2.5 Hz, 2H), 2.57 (t, *J* = 2.5 Hz, 1H); **¹³C NMR** (101 MHz, CDCl₃) δ 164.7, 152.4, 141.8, 134.1 (d, *J* = 2.5 Hz), 131.9, 130.4, 129.6, 129.2, 128.9, 128.9, 119.5, 115.4 (d, *J* = 33.4 Hz), 75.6, 53.1; **¹⁹F NMR** (376 MHz, CDCl₃) δ 69.9; **HRMS** (ESI⁺): calculated for C₁₉H₁₄FN₂O₄S [M+H⁺]: *m/z* = 385.0653, *m/z* found 385.0670. **IR** *v*_{max} (ATR)/cm⁻¹: 3262, 3142, 2133, 1719, 1608, 1529, 1404, 1273, 1204.

1-(4-Chloronaphthalen-1-yl)-3-(4-methoxyphenyl)-1H-pyrazole-4-sulfonyl fluoride (4ew)



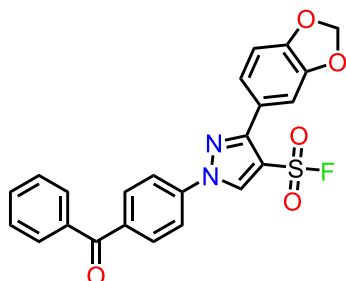
Following general procedure G, the title compound was isolated by flash column chromatography (0-10% EtOAc in petroleum ether) as a white solid (82.0 mg, 79%). **m.p.** 109 - 110 °C; **¹H NMR** (400 MHz, CDCl₃) δ 8.46 (d, *J* = 0.9 Hz, 1H), 8.44 – 8.39 (m, 1H), 7.89 (appt. d, *J* = 8.9 Hz, 2H), 7.85 – 7.81 (m, 1H), 7.77 – 7.65 (m, 3H), 7.55 (d, *J* = 7.9 Hz, 1H), 7.02 (appt. d, *J* = 9.0 Hz, 2H), 3.87 (s, 3H); **¹³C NMR** (101 MHz, CDCl₃) δ 161.2, 151.8, 138.8 (d, *J* = 2.2 Hz), 134.7, 134.5, 131.6, 130.3, 129.5, 129.0, 128.5, 125.4, 125.3, 123.7, 123.0, 121.7, 114.3, 113.3 (d, *J* = 32.9 Hz), 55.5; **¹⁹F NMR** (376 MHz, CDCl₃) δ 69.7; **HRMS** (ESI⁺): calculated for C₂₀H₁₅ClFN₂O₃S [M+H⁺]: *m/z* = 417.0470, *m/z* found 417.0468; **IR** *v*_{max} (ATR)/cm⁻¹: 3142, 3005, 2841, 1612, 1504, 1429, 1401, 1255, 1210, 959, 762.

1-(4-Benzoylphenyl)-3-phenyl-1H-pyrazole-4-sulfonyl fluoride (4ex)



Following general procedure G, the title compound was isolated by flash column chromatography (0-10% EtOAc in petroleum ether) as a white solid (87.0 mg, 86%). **m.p.** 126 - 127 °C; **¹H NMR** (400 MHz, CDCl₃) δ 8.74 (d, *J* = 1.1 Hz, 1H), 8.01 (appt. d, *J* = 8.9 Hz, 2H), 7.95 – 7.88 (m, 4H), 7.85 – 7.81 (m, 2H), 7.68 – 7.62 (m, 1H), 7.56 – 7.50 (m, 5H); **¹³C NMR** (101 MHz, CDCl₃) δ 195.2, 152.4, 141.0, 137.6, 137.2, 134.1 (d, *J* = 2.4 Hz), 133.1, 132.0, 130.4, 130.1, 129.2, 128.9, 128.9, 128.7, 119.4, 115.2 (d, *J* = 33.3 Hz); **¹⁹F NMR** (376 MHz, CDCl₃) δ 69.9; **HRMS** (ESI⁺): calculated for C₂₂H₁₆FN₂O₃S [M+H⁺]: *m/z* = 407.0860, *m/z* found 407.0856; **IR** *v*_{max} (ATR)/cm⁻¹: 3139, 1662, 1602, 1524, 1395, 1215, 771.

3-(Benzo[d][1,3]dioxol-5-yl)-1-(4-benzoylphenyl)-1H-pyrazole-4-sulfonyl fluoride (4ey)

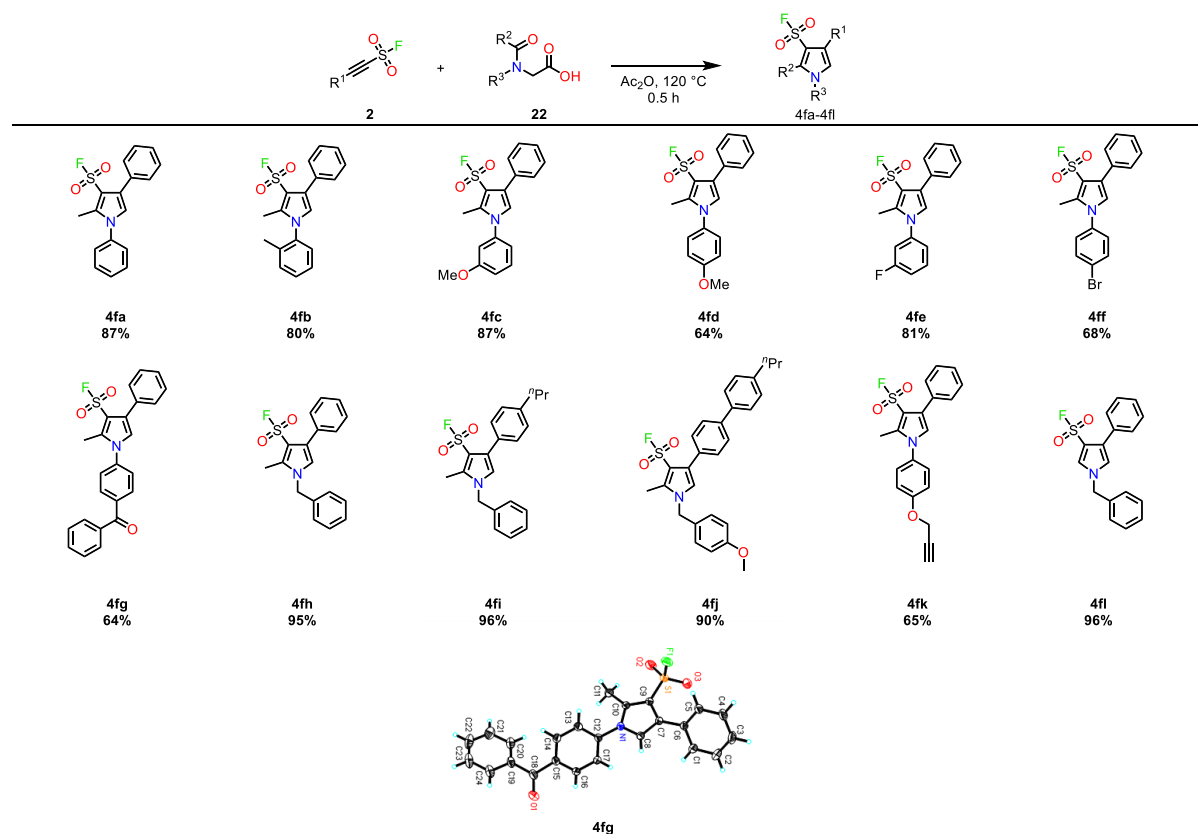


Following general procedure G, the title compound was isolated by flash column chromatography (0-10% EtOAc in petroleum ether) as a white solid (91.0 mg, 81%). **m.p.** 138 - 140 °C; **¹H NMR** (400 MHz, CDCl₃) δ 8.70 (d, *J* = 0.8 Hz, 1H), 8.00 (appt. d, *J* = 8.7 Hz, 2H), 7.91 (appt. d, *J* = 8.7 Hz, 2H), 7.85 – 7.80 (m, 2H), 7.68 – 7.62 (m, 1H), 7.58 – 7.51 (m, 2H), 7.44 (dd, *J* = 8.1, 1.8 Hz, 1H), 7.40 – 7.38 (m, 1H), 6.95 (d, *J* = 8.1 Hz, 1H), 6.06 (s, 2H); **¹³C NMR** (101 MHz, CDCl₃) δ 195.2, 152.0, 149.5, 148.2, 141.0, 137.6, 137.2, 134.1 (d, *J* = 2.4 Hz), 133.1, 132.0, 130.1, 128.7, 123.4, 123.0, 119.3, 114.9 (d, *J* = 33.1 Hz), 109.1, 108.7, 101.7; **¹⁹F NMR** (376 MHz, CDCl₃) δ 69.5; **HRMS** (ESI⁺): calculated for C₂₃H₁₆FN₂O₅S [M+H⁺]: *m/z* = 451.0758, *m/z* found 451.0759; **IR** *v*_{max} (ATR)/cm⁻¹: 3129, 1651, 1605, 1516, 1461, 1408, 1207, 763.

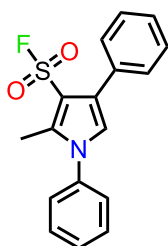
Synthesis and experimental data for compounds 4fa-4fl

General Procedure H

To a suspension of required substituted glycine **22** (100 μ mol) in acetic anhydride (1.00 mL) was added the required SASF **2** (120 μ mol) at room temperature. The reaction mixture was stirred at 120 $^{\circ}$ C for 0.5 h. The resultant solution was cooled to room temperature, the Ac₂O was removed under a stream of nitrogen, and the crude product was purified by flash column chromatography to obtain the analytically pure product.



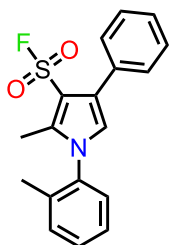
2-Methyl-1,4-diphenyl-1H-pyrrole-3-sulfonyl fluoride (4fa)



Following general procedure H, the title compound was isolated by flash column chromatography (0-6% EtOAc in petroleum ether) as a white solid (67.0 mg, 87%). **m.p.** 91 - 92 $^{\circ}$ C; **¹H NMR** (400 MHz, CDCl₃) δ 7.61 – 7.48 (m, 5H), 7.46 – 7.33 (m, 5H), 6.82 (s, 1H), 2.49 (d, J = 1.1 Hz, 3H); **¹³C NMR** (126 MHz, CDCl₃) δ 137.9, 137.8 (d, J = 2.8 Hz), 132.3, 129.9, 129.5, 129.4, 128.3, 127.9, 126.6, 126.1, 122.3, 111.1 (d, J = 28.6 Hz), 12.4; **¹⁹F NMR** (471 MHz,

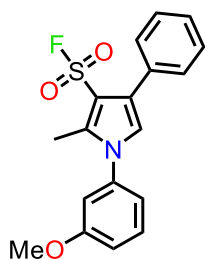
CDCl₃) δ 74.0; **HRMS** (ESI⁺): calculated for C₁₇H₁₄FNO₂SNa [M+Na⁺]: m/z = 338.0621, m/z found 338.0621; **IR** ν_{max} (ATR)/cm⁻¹: 3137, 2926, 1597, 1500, 1399, 1208, 758, 728.

2-Methyl-4-phenyl-1-(*o*-tolyl)-1*H*-pyrrole-3-sulfonyl fluoride (4fb)



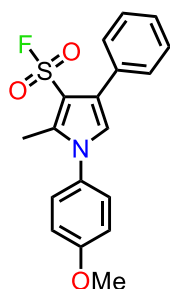
Following general procedure H, the title compound was isolated by flash column chromatography (0-6% EtOAc in petroleum ether) as a white solid (66.0 mg, 80%). **m.p.** 95 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.55 – 7.34 (m, 8H), 7.25 (s, 1H), 6.71 (s, 1H), 2.34 (s, 3H), 2.12 (s, 3H); **¹³C NMR** (126 MHz, CDCl₃) δ 138.3 (d, *J* = 2.8 Hz), 136.8, 135.7, 132.4, 131.5, 130.0, 129.5, 128.3, 127.8, 127.8, 127.3, 126.1, 121.8, 110.3 (d, *J* = 28.6 Hz), 17.4, 11.9; **¹⁹F NMR** (471 MHz, CDCl₃) δ 74.0; **HRMS** (ESI⁺): calculated for C₁₈H₁₆FNO₂SNa [M+Na⁺]: m/z = 352.0778, m/z found 352.0775; **IR** ν_{max} (ATR)/cm⁻¹: 3127, 2926, 2853, 1498, 1397, 1212, 752.

1-(3-Methoxyphenyl)-2-methyl-4-phenyl-1*H*-pyrrole-3-sulfonyl fluoride (4fc)



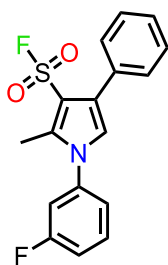
Following general procedure H, the title compound was isolated by flash column chromatography (0-10% EtOAc in petroleum ether) as a colourless oil (75.0 mg, 87%). **¹H NMR** (400 MHz, CDCl₃) δ 7.52 – 7.34 (m, 6H), 7.05 (ddd, *J* = 8.4, 2.5, 0.9 Hz, 1H), 6.93 (ddd, *J* = 7.8, 2.0, 0.9 Hz, 1H), 6.87 (t, *J* = 2.2 Hz, 1H), 6.82 (s, 1H), 3.88 (s, 3H), 2.50 (d, *J* = 1.0 Hz, 3H); **¹³C NMR** (126 MHz, CDCl₃) δ 160.6, 138.9, 137.8 (d, *J* = 2.7 Hz), 132.3, 130.6, 129.5, 128.3, 127.9, 126.1, 122.2, 118.8, 114.9, 112.6, 111.1 (d, *J* = 28.5 Hz), 55.8, 12.5; **¹⁹F NMR** (471 MHz, CDCl₃) δ 74.0; **HRMS** (ESI⁺): calculated for C₁₈H₁₆FNO₃SNa [M+Na⁺]: m/z = 368.0727, m/z found 368.0715; **IR** ν_{max} (ATR)/cm⁻¹: 3135, 3064, 2932, 1605, 1496, 1399, 1200, 733.

1-(4-Methoxyphenyl)-2-methyl-4-phenyl-1H-pyrrole-3-sulfonyl fluoride (4fd)



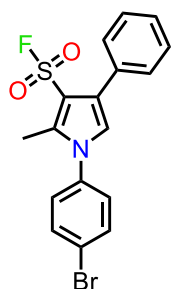
Following General general procedure H, the title compound was isolated by flash column chromatography (0-10% EtOAc in petroleum ether) as a white solid (67.0 mg, 64%). **m.p.** 106 - 108 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.53 – 7.47 (m, 2H), 7.46 – 7.33 (m, 3H), 7.26 (appt. d, *J* = 9.0 Hz, 2H), 7.04 (appt. d, *J* = 9.0 Hz, 2H), 6.78 (s, 1H), 3.89 (s, 3H), 2.46 (d, *J* = 1.0 Hz, 3H); **¹³C NMR** (126 MHz, CDCl₃) δ 160.2, 138.1 (d, *J* = 2.6 Hz), 132.4, 130.6, 129.5, 128.3, 127.8, 125.8, 122.5, 114.9, 110.6 (d, *J* = 28.5 Hz), 55.8, 12.4; **¹⁹F NMR** (471 MHz, CDCl₃) δ 74.0; **HRMS** (ESI⁺): calculated for C₁₈H₁₆FNO₃SNa [M+Na⁺]: *m/z* = 368.0727, *m/z* found 368.0729; **IR** *v*_{max} (ATR)/cm⁻¹: 3149, 2926, 2853, 1515, 1398, 1250, 1205, 1027, 752.

1-(3-Fluorophenyl)-2-methyl-4-phenyl-1H-pyrrole-3-sulfonyl fluoride (4fe)



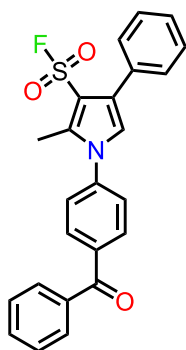
Following general procedure H, the title compound was isolated by flash column chromatography (0-6% EtOAc in petroleum ether) as a white solid (91.0 mg, 81%). **m.p.** 84 - 86 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.60 – 7.45 (m, 3H), 7.45 – 7.35 (m, 3H), 7.28 – 7.22 (m, 1H), 7.19 – 7.15 (m, 1H), 7.11 (appt. dt, *J* = 8.8, 2.2 Hz, 1H), 6.81 (s, 1H), 2.51 (d, *J* = 1.1 Hz, 3H); **¹³C NMR** (126 MHz, CDCl₃) δ 163.0 (d, *J* = 250.4 Hz), 139.2 (d, *J* = 9.7 Hz), 137.6 (d, *J* = 3.0 Hz), 132.1, 131.2 (d, *J* = 9.0 Hz), 129.5, 128.3, 128.0, 126.5, 122.5 (d, *J* = 3.4 Hz), 122.0 (d, *J* = 1.4 Hz), 116.6 (d, *J* = 21.9 Hz), 114.4 (d, *J* = 23.7 Hz), 111.8 (d, *J* = 28.8 Hz), 12.4; **¹⁹F NMR** (471 MHz, CDCl₃) δ 73.9, -109.5 – -109.6 (m); **HRMS** (ESI⁺): calculated for C₁₇H₁₃F₂NO₂SNa [M+Na⁺]: *m/z* = 356.0527, *m/z* found 356.0531; **IR** *v*_{max} (ATR)/cm⁻¹: 3135, 2926, 1599, 1495, 1399, 1216, 876, 759, 733.

1-(4-Bromophenyl)-2-methyl-4-phenyl-1H-pyrrole-3-sulfonyl fluoride (4ff)



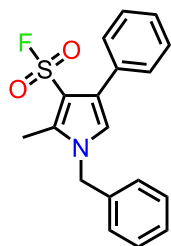
Following general procedure H, the title compound was isolated by flash column chromatography (0-10% EtOAc in petroleum ether) as a white solid (67.0 mg, 68%). **m.p.** 116 - 118 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.69 (appt. d, *J* = 8.8 Hz, 2H), 7.51 – 7.46 (m, 2H), 7.44 – 7.35 (m, 3H), 7.24 (appt. d, *J* = 8.8 Hz, 2H), 6.79 (s, 1H), 2.48 (d, *J* = 1.0 Hz, 3H); **¹³C NMR** (101 MHz, CDCl₃) δ 137.6 (d, *J* = 2.9 Hz), 136.8, 133.1, 132.1, 129.5, 128.3, 128.2, 128.0, 126.4, 123.5, 122.0, 111.6 (d, *J* = 28.8 Hz), 12.4; **¹⁹F NMR** (471 MHz, CDCl₃) δ 74.0; **HRMS** (ESI⁺): calculated for C₁₇H₁₃BrFKNO₂S [M+K⁺]: *m/z* = 433.9446, *m/z* found 433.9440; **IR** *v*_{max} (ATR)/cm⁻¹: 3142, 2927, 2853, 1494, 1398, 1204.

1-(4-Benzoylphenyl)-2-methyl-4-phenyl-1H-pyrrole-3-sulfonyl fluoride (4fg)



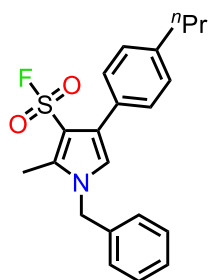
Following general procedure H, the title compound was isolated by flash column chromatography (0-10% EtOAc in petroleum ether) as an off-white solid (67.0 mg, 64%). **m.p.** 160 - 162 °C; **¹H NMR** (400 MHz, CDCl₃) δ 8.00 (appt. d, *J* = 8.7 Hz, 2H), 7.88 – 7.84 (m, 2H), 7.69 – 7.63 (m, 1H), 7.58 – 7.47 (m, 6H), 7.46 – 7.35 (m, 3H), 6.87 (s, 1H), 2.56 (d, *J* = 0.8 Hz, 3H); **¹³C NMR** (101 MHz, CDCl₃) δ 195.3, 141.0, 138.4, 137.5 (d, *J* = 3.0 Hz), 137.0, 133.2, 132.0, 131.6, 130.2, 129.5, 128.7, 128.3, 128.1, 126.7, 126.5, 121.9, 112.1 (d, *J* = 28.8 Hz), 12.6; **¹⁹F NMR** (376 MHz, CDCl₃) δ 73.9; **HRMS** (ESI⁺): calculated for C₂₄H₁₈FNO₃SNa [M+Na⁺]: *m/z* = 442.0884, *m/z* found 442.0890; **IR** *v*_{max} (ATR)/cm⁻¹: 3064, 2925, 1661, 1601, 1511, 1400, 1207, 741.

1-Benzyl-2-methyl-4-phenyl-1H-pyrrole-3-sulfonyl fluoride (4fh)



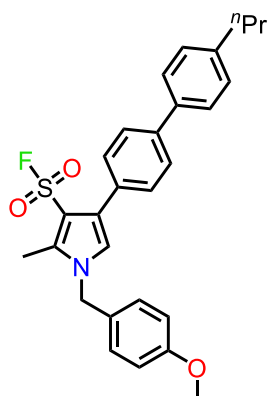
Following general procedure H, the title compound was isolated by flash column chromatography (0-10% EtOAc in petroleum ether) as a colourless oil (78.0 mg, 95%). **¹H NMR** (400 MHz, CDCl₃) δ 7.49 – 7.31 (m, 8H), 7.15 – 7.10 (m, 2H), 6.69 (s, 1H), 5.12 (s, 2H), 2.54 (d, *J* = 1.0 Hz, 3H); **¹³C NMR** (101 MHz, CDCl₃) δ 137.5 (d, *J* = 2.7 Hz), 135.3, 132.6, 129.5, 129.4, 128.5, 128.2, 127.7, 126.9, 126.0, 121.7, 110.4 (d, *J* = 28.3 Hz), 51.4, 11.4; **¹⁹F NMR** (471 MHz, CDCl₃) δ 74.1; **HRMS** (ESI⁺): calculated for C₁₈H₁₆FNO₂SNa [M+Na⁺]: *m/z* = 352.0778, *m/z* found 352.0779; **IR** *v*_{max} (ATR)/cm⁻¹: 3064, 3033, 1605, 1506, 1397, 1229, 1183, 739.

1-Benzyl-2-methyl-4-(4-propylphenyl)-1H-pyrrole-3-sulfonyl fluoride (4fi)



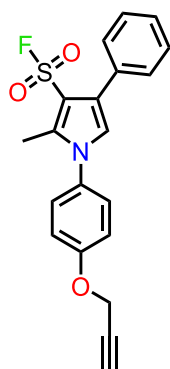
Following general procedure H, the title compound was isolated by flash column chromatography (0-10% EtOAc in petroleum ether) as a colourless oil (89.0 mg, 96%). **¹H NMR** (400 MHz, CDCl₃) δ 7.43 – 7.33 (m, 5H), 7.19 (appt. d, *J* = 8.4 Hz, 2H), 7.14 – 7.09 (m, 2H), 6.67 (s, 1H), 5.11 (s, 2H), 2.64 – 2.59 (m, 2H), 2.53 (d, *J* = 0.9 Hz, 3H), 1.73 – 1.62 (m, 2H), 1.00 – 0.95 (m, 3H); **¹³C NMR** (126 MHz, CDCl₃) δ 142.3, 137.3 (d, *J* = 2.4 Hz), 135.4, 129.8, 129.4, 129.3, 128.5, 128.3, 126.9, 126.0, 121.6, 110.3 (d, *J* = 28.0 Hz), 51.3, 37.9, 24.6, 14.1, 11.5; **¹⁹F NMR** (471 MHz, CDCl₃) δ 74.0; **HRMS** (ESI⁺): calculated for C₂₁H₂₂FNO₂SNa [M+Na⁺]: *m/z* = 394.1247, *m/z* found 394.1249; **IR** *v*_{max} (ATR)/cm⁻¹: 3031, 2959, 2927, 2870, 1508, 1456, 1397, 1229, 1183, 733.

1-(4-Methoxybenzyl)-4-(4'-propyl-[1,1'-biphenyl]-4-yl)-1H-pyrrole-3-sulfonyl fluoride (4fj)



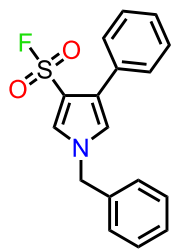
Following general procedure H, the title compound was isolated by flash column chromatography (0-10% EtOAc in petroleum ether) as a yellow solid (107 mg, 90%). **m.p.** 130 - 132 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.60 (appt. d, *J* = 8.6 Hz, 2H), 7.54 (appt. d, *J* = 8.3 Hz, 2H), 7.50 (appt. d, *J* = 8.6 Hz, 2H), 7.28 – 7.24 (m, 2H), 7.08 (appt. d, *J* = 8.9 Hz, 2H), 6.93 (appt. d, *J* = 8.8 Hz, 2H), 6.69 (s, 1H), 5.05 (s, 2H), 3.83 (s, 3H), 2.68 – 2.61 (m, 2H), 2.56 (d, *J* = 0.8 Hz, 3H), 1.70 (dp, *J* = 8.9, 7.3 Hz, 2H), 0.99 (t, *J* = 7.3 Hz, 3H); **¹³C NMR** (101 MHz, CDCl₃) δ 159.8, 142.0, 140.5, 138.3, 137.5 (d, *J* = 2.7 Hz), 131.3, 129.8, 129.0, 128.5, 127.1, 127.0, 126.8, 125.6, 121.6, 114.8, 110.2 (d, *J* = 28.1 Hz), 55.5, 51.0, 37.9, 24.6, 14.0, 11.5; **¹⁹F NMR** (471 MHz, CDCl₃) δ 74.1; **HRMS** (ESI⁺): calculated for C₂₈H₂₈FNO₃Na [M+Na⁺]: *m/z* = 500.1666, *m/z* found 500.1666; **IR** *v*_{max} (ATR)/cm⁻¹: 3127, 2927, 1609, 1509, 1388, 1245, 1182, 744.

2-Methyl-4-phenyl-1-(4-(prop-2-yn-1-yloxy)phenyl)-1H-pyrrole-3-sulfonyl fluoride (4fk)



Following general procedure H, the title compound was isolated by flash column chromatography (0-10% EtOAc in petroleum ether) as a yellow oil (60.0 mg, 65%). **¹H NMR** (500 MHz, CDCl₃) δ 7.51 – 7.47 (m, 2H), 7.43 – 7.34 (m, 3H), 7.28 (appt. d, *J* = 9.4 Hz, 2H), 7.13 (appt. d, *J* = 8.9 Hz, 2H), 6.78 (s, 1H), 4.78 (d, *J* = 2.4 Hz, 2H), 2.59 (t, *J* = 2.4 Hz, 1H), 2.46 (d, *J* = 0.6 Hz, 3H); **¹³C NMR** (126 MHz, CDCl₃) δ 158.0, 138.1 (d, *J* = 2.7 Hz), 132.4, 131.4, 129.5, 128.3, 127.9, 126.0, 122.5, 116.0, 110.8 (d, *J* = 28.4 Hz), 78.0, 76.4, 56.3, 12.4; **¹⁹F NMR** (376 MHz, CDCl₃) δ 73.9; **HRMS** (ESI⁺): calculated for C₂₀H₁₆FNO₃Na [M+Na⁺]: *m/z* = 392.0727, *m/z* found 392.0727; **IR** *v*_{max} (ATR)/cm⁻¹: 3290, 3057, 1512, 1398, 1204.

1-Benzyl-4-phenyl-1H-pyrrole-3-sulfonyl fluoride (4fl)

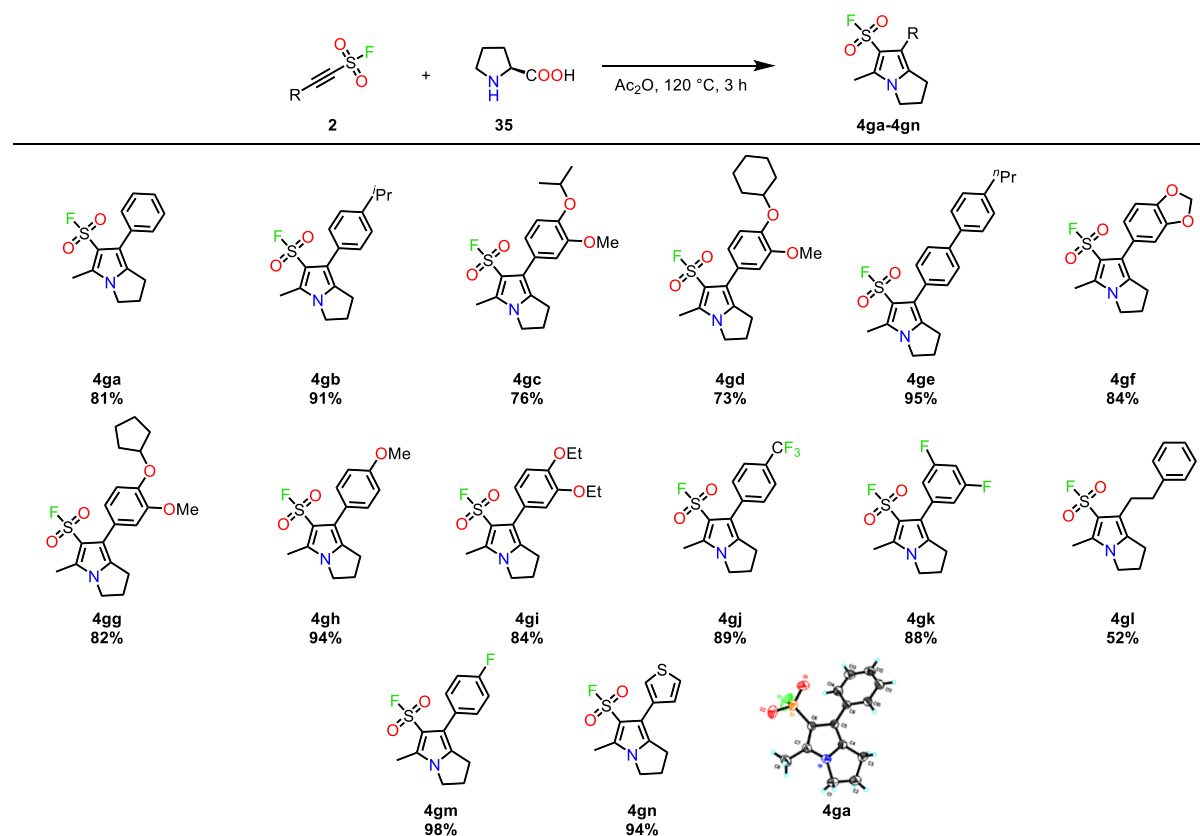


Following general procedure H, the title compound was isolated by flash column chromatography (0-10% EtOAc in petroleum ether) as a colourless oil (75.0 mg, 96%). **¹H NMR** (400 MHz, CDCl₃) δ 7.54 – 7.49 (m, 3H), 7.46 – 7.32 (m, 6H), 7.28 – 7.24 (m, 2H), 6.83 (d, *J* = 2.6 Hz, 1H), 5.11 (s, 2H); **¹³C NMR** (126 MHz, CDCl₃) δ 134.9, 131.7, 129.4, 129.4, 129.0, 128.8, 128.6, 128.0, 127.9, 126.2, 122.7, 113.1 (d, *J* = 30.1 Hz), 54.6; **¹⁹F NMR** (471 MHz, CDCl₃) δ 70.0; **HRMS** (ESI⁺): calculated for C₁₇H₁₄FNO₂SNa [M+Na⁺]: *m/z* = 338.0621, *m/z* found 338.0613; **IR** *v*_{max} (ATR)/cm⁻¹: 3132, 2926, 2854, 1604, 1507, 1394, 1176, 749.

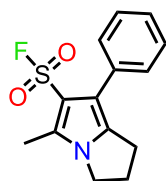
Synthesis and experimental data for compounds 4ga-4an

General Procedure I

To a solution of *L*-proline **35** (100 μ mol) in Ac₂O (400 μ L) was added the required SASF **2** (120 μ mol) and heated to 120 °C for 3 h. The Ac₂O was removed under a stream of nitrogen and the crude product purified by flash column chromatography to obtain the analytically pure product.

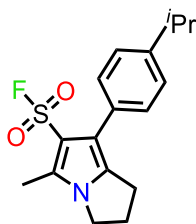


5-Methyl-7-phenyl-2,3-dihydro-1*H*-pyrrolizine-6-sulfonyl fluoride (4ga)



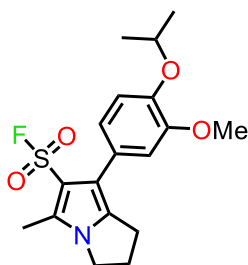
Following general procedure I (250 μ mol of *L*-proline used), the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a colourless solid (57.0 mg, 81%). **m.p.** 138 - 141 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.43 – 7.28 (m, 5H), 3.99 (appt. t, *J* = 7.3 Hz, 2H), 2.89 (appt. t, *J* = 7.4 Hz, 2H), 2.63 – 2.49 (m, 5H); **¹³C NMR** (101 MHz, CDCl₃) δ 135.0 (d, *J* = 1.4 Hz), 133.1, 132.4 (d, *J* = 2.8 Hz), 129.5, 128.2, 127.1, 116.6, 110.9 (d, *J* = 27.3 Hz), 45.6, 26.9, 24.1, 12.1; **¹⁹F NMR** (376 MHz, CDCl₃) δ 74.1; **HRMS** (ESI⁺): calculated for C₁₄H₁₄FNO₂SNa [M+Na⁺]: *m/z* = 302.0621, *m/z* found 302.0623; **IR** ν_{max} : 3070, 2926, 2853, 1501, 1372, 1240, 1171, 764, 739, 703.

7-(4-Isopropylphenyl)-5-methyl-2,3-dihydro-1H-pyrrolizine-6-sulfonyl fluoride (4gb)



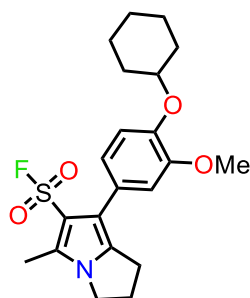
Following general procedure I, the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a beige solid (29.0 mg, 91%). **m.p.** 105 - 106 °C; **¹H NMR** (500 MHz, CDCl₃) δ 7.33 (appt. d, *J* = 8.3 Hz, 2H), 7.25 – 7.22 (m, 2H), 3.97 (appt. t, *J* = 7.1 Hz, 2H), 2.97 – 2.88 (m, 3H), 2.57 – 2.49 (m, 5H), 1.28 (d, *J* = 6.9 Hz, 6H); **¹³C NMR** (126 MHz, CDCl₃) δ 147.5, 134.8, 132.3 (d, *J* = 2.6 Hz), 130.3, 130.1, 129.3, 126.3, 116.7, 110.8 (d, *J* = 27.1 Hz), 45.5, 33.9, 26.9, 24.2, 24.0, 12.1; **¹⁹F NMR** (376 MHz, CDCl₃) δ 74.0; **HRMS** (ESI⁺): calculated for C₁₇H₂₀FNO₂SNa [M+Na⁺]: *m/z* = 344.1091, *m/z* found 344.1091; **IR** ν_{max} (ATR)/cm⁻¹: 2959, 2927, 2854, 1515, 1394, 1238, 1172, 724.

7-(4-Isopropoxy-3-methoxyphenyl)-5-methyl-2,3-dihydro-1H-pyrrolizine-6-sulfonyl fluoride (4gc)



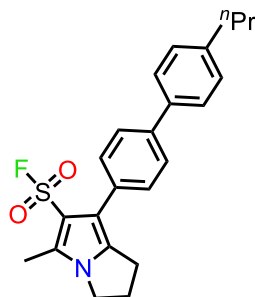
Following general procedure I, the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a colourless solid (29.0 mg, 76%). **m.p.** 93 - 94 °C; **¹H NMR** (500 MHz, CDCl₃) δ 7.01 – 6.98 (m, 1H), 6.90 – 6.88 (m, 2H), 4.55 (hept, *J* = 6.1 Hz, 1H), 3.97 (appt. t, *J* = 7.1 Hz, 2H), 3.86 (s, *J* = 3.6 Hz, 3H), 2.91 (appt. t, *J* = 7.4 Hz, 2H), 2.57 – 2.50 (m, 5H), 1.39 (d, *J* = 6.1 Hz, 6H); **¹³C NMR** (126 MHz, CDCl₃) δ 149.8, 146.6, 134.7, 132.2, 125.8, 121.6, 116.5, 115.1, 113.8, 110.9 (d, *J* = 27.0 Hz), 71.4, 56.1, 45.5, 26.9, 24.3, 22.3, 12.2; **¹⁹F NMR** (376 MHz, CDCl₃) δ 73.6; **HRMS** (ESI⁺): calculated for C₁₈H₂₂FNO₄SNa [M+Na⁺]: *m/z* = 390.1146, *m/z* found 390.1146; **IR** ν_{max} (ATR)/cm⁻¹: 2978, 2928, 1510, 1464, 1240, 1211, 1165, 1139, 1109, 719.

7-(4-(Cyclohexyloxy)-3-methoxyphenyl)-5-methyl-2,3-dihydro-1H-pyrrolizine-6-sulfonyl fluoride (4gd)



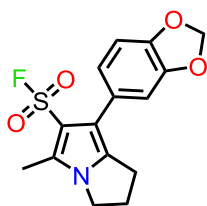
Following general procedure I, the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a beige solid (30.0 mg, 73%). **m.p.** 138 °C (decomposed); **¹H NMR** (400 MHz, CDCl₃) δ 6.99 (d, *J* = 1.2 Hz, 1H), 6.92 – 6.86 (m, 2H), 4.24 – 4.16 (m, 1H), 3.96 (appt. t, *J* = 7.1 Hz, 2H), 3.85 (s, 3H), 2.90 (appt. t, *J* = 7.4 Hz, 2H), 2.59 – 2.48 (m, 5H), 2.12 – 2.02 (m, 2H), 1.88 – 1.79 (m, 2H), 1.64 – 1.52 (m, 3H), 1.41 – 1.28 (m, 3H); **¹³C NMR** (101 MHz, CDCl₃) δ 150.0, 146.6, 134.7, 132.2 (d, *J* = 2.8 Hz), 125.9, 121.6, 116.5, 115.6, 113.9, 110.9 (d, *J* = 27.3 Hz), 77.4, 56.1, 45.5, 32.3, 26.9, 25.8, 24.4, 24.3, 12.1; **¹⁹F NMR** (376 MHz, CDCl₃) δ 73.7; **HRMS** (ESI⁺): calculated for C₂₁H₂₆FNO₄SNa [M+Na⁺]: *m/z* = 430.1459, *m/z* found 430.1457; **IR** *v*_{max} (ATR)/cm⁻¹: 2925, 2853, 1512, 1392, 1237, 1162, 1137, 732.

5-Methyl-7-(4'-propyl-[1,1'-biphenyl]-4-yl)-2,3-dihydro-1H-pyrrolizine-6-sulfonyl fluoride (4ge)



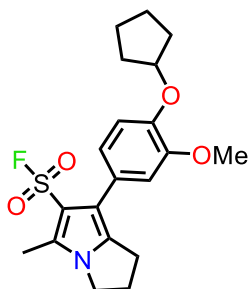
Following general procedure I, the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a beige solid (38.0 mg, 95%). **m.p.** 116 - 117 °C; **¹H NMR** (500 MHz, CDCl₃) δ 7.61 (appt. d, *J* = 8.5 Hz, 2H), 7.56 (appt. d, *J* = 8.2 Hz, 2H), 7.47 (appt. d, *J* = 8.5 Hz, 2H), 7.27 (appt. d, *J* = 8.3 Hz, 2H), 3.97 (appt. t, *J* = 7.2 Hz, 2H), 2.93 (appt. t, *J* = 7.4 Hz, 2H), 2.68 – 2.63 (m, 2H), 2.58 – 2.50 (m, 5H), 1.75 – 1.66 (m, 2H), 1.00 (t, *J* = 7.3 Hz, 3H); **¹³C NMR** (126 MHz, CDCl₃) δ 141.9, 139.8, 138.3, 135.1, 132.5 (d, *J* = 2.5 Hz), 131.7, 129.7, 129.0, 127.0 (s, *J* = 4.2 Hz), 126.8, 116.3, 110.9 (d, *J* = 27.3 Hz), 45.6, 37.8, 26.9, 24.7, 24.3, 14.0, 12.1; **¹⁹F NMR** (376 MHz, CDCl₃) δ 74.1; **HRMS** (ESI⁺): calculated for C₂₃H₂₄FNO₂SNa [M+Na⁺]: *m/z* = 420.1404, *m/z* found 420.1400; **IR** *v*_{max} (ATR)/cm⁻¹: 2926, 2869, 1499, 1394, 1241, 1173, 738.

7-(Benzo[d][1,3]dioxol-5-yl)-5-methyl-2,3-dihydro-1H-pyrrolizine-6-sulfonyl fluoride (4gf)



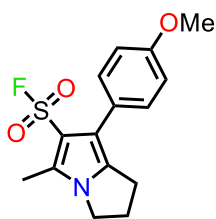
Following general procedure I, the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a beige solid (27.0 mg, 84%). **m.p.** 145 - 146 °C; **¹H NMR** (500 MHz, CDCl₃) δ 6.89 – 6.80 (m, 3H), 5.98 (s, 2H), 3.97 (appt. t, *J* = 7.1 Hz, 2H), 2.87 (appt. t, *J* = 7.4 Hz, 2H), 2.59 – 2.43 (m, 5H); **¹³C NMR** (126 MHz, CDCl₃) δ 147.5, 146.9, 134.8, 132.1, 126.7, 123.0, 116.3, 111.0 (d, *J* = 27.1 Hz), 110.1, 108.2, 101.2, 45.6, 26.9, 24.0, 12.1; **¹⁹F NMR** (376 MHz, CDCl₃) δ 74.0; **HRMS** (ESI⁺): calculated for C₁₅H₁₄FNO₄SNa [M+Na⁺]: *m/z* = 346.0520, *m/z* found 346.0521; **IR** *v*_{max} (ATR)/cm⁻¹: 3071, 2905, 2856, 1489, 1449, 1375, 1225, 1175, 730.

7-(4-(Cyclopentyloxy)-3-methoxyphenyl)-5-methyl-2,3-dihydro-1H-pyrrolizine-6-sulfonyl fluoride (4gg)



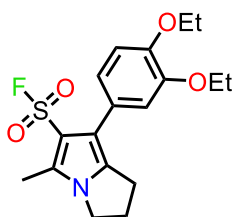
Following general procedure I, the title compound was isolated after purification by flash column chromatography (0-20% EtOAc in petroleum ether) as a brown solid (32.0 mg, 82%). **m.p.** 105 - 107 °C; **¹H NMR** (500 MHz, CDCl₃) δ 6.98 (d, *J* = 1.9 Hz, 1H), 6.90 (dd, *J* = 8.3, 1.9 Hz, 1H), 6.86 (d, *J* = 8.3 Hz, 1H), 4.81 – 4.76 (m, 1H), 3.96 (appt. t, *J* = 7.1 Hz, 2H), 3.85 (s, 3H), 2.90 (appt. t, *J* = 7.4 Hz, 2H), 2.57 – 2.50 (m, 5H), 1.97 – 1.91 (m, 4H), 1.88 – 1.81 (m, 2H), 1.65 – 1.58 (m, 2H); **¹³C NMR** (126 MHz, CDCl₃) δ 149.4, 147.1, 134.7, 132.2, 125.4, 121.6, 116.6, 114.3, 113.8, 110.9 (d, *J* = 27.1 Hz), 80.4, 56.2, 45.5, 33.1, 26.9, 24.3, 24.2, 12.1; **¹⁹F NMR** (376 MHz, CDCl₃) δ 73.6; **HRMS** (ESI⁺): calculated for C₂₀H₂₄FNO₄SNa [M+Na⁺]: *m/z* = 416.1302, *m/z* found 416.1304; **IR** *v*_{max} (ATR)/cm⁻¹: 2952, 2888, 1513, 1392, 1238, 1163, 1140, 1027, 733.

7-(4-Methoxyphenyl)-5-methyl-2,3-dihydro-1H-pyrrolizine-6-sulfonyl fluoride (4gh)



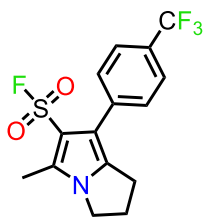
Following general procedure I, the title compound was isolated after purification by flash column chromatography (0-20% EtOAc in petroleum ether) as a colourless solid (29.0 mg, 94%). **m.p.** 78 - 81 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.32 (appt. d, *J* = 8.8 Hz, 2H), 6.92 (appt. d, *J* = 8.8 Hz, 2H), 3.96 (appt. t, *J* = 7.2 Hz, 2H), 3.83 (s, 3H), 2.87 (appt. t, *J* = 7.4 Hz, 2H), 2.58 – 2.49 (m, 5H); **¹³C NMR** (101 MHz, CDCl₃) δ 158.8, 134.7, 132.1 (d, *J* = 2.7 Hz), 130.6, 125.3, 116.3, 113.7, 110.9 (d, *J* = 26.7 Hz), 55.3, 45.5, 26.9, 24.0, 12.1; **¹⁹F NMR** (376 MHz, CDCl₃) δ 73.9; **HRMS** (ESI⁺): calculated for C₁₅H₁₆FNO₃SNa [M+Na⁺]: *m/z* = 332.0727, *m/z* found 332.0727; **IR** ν_{max} (ATR)/cm⁻¹: 2926, 2854, 1513, 1391, 1237, 1169, 1028, 836, 735.

7-(3,4-Diethoxyphenyl)-5-methyl-2,3-dihydro-1H-pyrrolizine-6-sulfonyl fluoride (4gi)



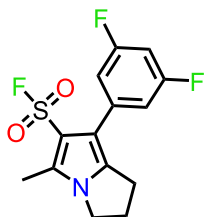
Following general procedure I, the title compound was isolated after purification by flash column chromatography (0-20% EtOAc in petroleum ether) as a beige solid (31.0 mg, 84%); **m.p.** 136 - 137 °C; **¹H NMR** (400 MHz, CDCl₃) δ 6.99 (d, *J* = 1.5 Hz, 1H), 6.91 – 6.86 (m, 2H), 4.12 (appt. q, *J* = 7.0 Hz, 4H), 3.96 (appt. t, *J* = 7.1 Hz, 2H), 2.89 (appt. t, *J* = 7.4 Hz, 2H), 2.57 – 2.49 (m, 5H), 1.48 – 1.42 (m, 6H); **¹³C NMR** (101 MHz, CDCl₃) δ 148.2, 148.0, 134.7, 132.2 (d, *J* = 2.8 Hz), 125.6, 121.7, 116.5, 115.3, 113.1, 110.8 (d, *J* = 27.0 Hz), 64.6, 64.6, 45.5, 26.9, 24.2, 15.0, 14.9; **¹⁹F NMR** (376 MHz, CDCl₃) δ 73.7; **HRMS** (ESI⁺): calculated for C₁₈H₂₂FNO₄SNa [M+Na⁺]: *m/z* = 390.1146, *m/z* found 390.1148; **IR** ν_{max} (ATR)/cm⁻¹: 2981, 2926, 1515, 1393, 1239, 1140, 1040, 723.

5-Methyl-7-(4-(trifluoromethyl)phenyl)-2,3-dihydro-1H-pyrrolizine-6-sulfonyl fluoride (4gj)



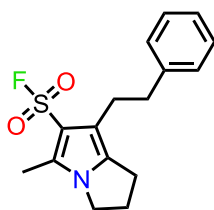
Following general procedure I, the title compound was isolated after purification by flash column chromatography (0-20% EtOAc in petroleum ether) as a colourless solid (31.0 mg, 89%). **m.p.** 106 - 108 °C; **¹H NMR** (500 MHz, CDCl₃) δ 7.63 (appt. d, *J* = 8.1 Hz, 2H), 7.51 (appt. d, *J* = 8.0 Hz, 2H), 4.00 (appt. t, *J* = 7.2 Hz, 2H), 2.90 (appt. t, *J* = 7.4 Hz, 2H), 2.61 – 2.53 (m, 5H); **¹³C NMR** (126 MHz, CDCl₃) δ 136.9, 135.7, 133.0 (d, *J* = 2.7 Hz), 129.7, 129.1 (q, *J* = 32.4 Hz), 125.2 (q, *J* = 3.8 Hz), 124.4 (q, *J* = 272.1 Hz), 115.4, 111.1 (d, *J* = 27.9 Hz), 45.7 (s), 26.9, 24.2, 12.1; **¹⁹F NMR** (376 MHz, CDCl₃) δ 74.1, -62.5; **HRMS** (ESI⁺): calculated for C₁₅H₁₃F₄NO₂SNa [M+Na⁺]: *m/z* = 370.0495, *m/z* found 370.0490; **IR** *v*_{max} (ATR)/cm⁻¹: 3036, 2960, 2851, 1618, 1397, 1323, 1244, 1161, 1109, 1067, 1039, 726.

7-(3,5-Difluorophenyl)-5-methyl-2,3-dihydro-1H-pyrrolizine-6-sulfonyl fluoride (4gk)



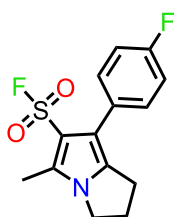
Following general procedure I, the title compound was isolated after purification by flash column chromatography (0-25% EtOAc in petroleum ether) as a colourless solid (28.0 mg, 88%). **m.p.** 158 - 159 °C; **¹H NMR** (500 MHz, CDCl₃) δ 6.91 (dt, *J* = 6.4, 3.2 Hz, 2H), 6.75 (tt, *J* = 9.0, 2.3 Hz, 1H), 3.99 (appt. t, *J* = 7.2 Hz, 2H), 2.90 (appt. t, *J* = 7.4 Hz, 2H), 2.62 – 2.50 (m, 5H); **¹³C NMR** (126 MHz, CDCl₃) δ 162.8 (dd, *J* = 247.6, 13.4 Hz), 136.3 (t, *J* = 10.5 Hz), 135.7, 133.0 (d, *J* = 2.7 Hz), 114.6, 112.4 (dd, *J* = 20.0, 6.2 Hz), 111.0 (d, *J* = 28.1 Hz), 102.6 (t, *J* = 25.3 Hz), 45.7, 26.8, 24.2, 12.1. **¹⁹F NMR** (376 MHz, CDCl₃) δ 74.1, -110.5 (t, *J* = 8.1 Hz); **HRMS** (ESI⁺): calculated for C₁₄H₁₂F₃NO₂SNa [M+Na⁺]: *m/z* = 338.0433, *m/z* found 338.0439; **IR** *v*_{max} (ATR)/cm⁻¹: 3088, 2927, 2852, 1625, 1594, 1434, 1396, 1280, 1213, 1160, 1118, 984, 863, 733.

5-Methyl-7-phenethyl-2,3-dihydro-1H-pyrrolizine-6-sulfonyl fluoride (4gl)



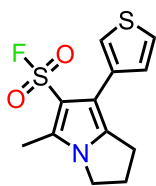
Following general procedure I, the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a colourless solid (16.0 mg, 52%). **m.p.** 59 - 60 °C; **¹H NMR** (500 MHz, CDCl₃) δ 7.27 – 7.23 (m, 2H), 7.20 – 7.16 (m, 1H), 7.15 – 7.11 (m, 2H), 3.84 – 3.79 (m, 2H), 2.90 – 2.83 (m, 4H), 2.46 (d, *J* = 0.9 Hz, 3H), 2.37 – 2.33 (m, 4H); **¹³C NMR** (126 MHz, CDCl₃) δ 142.1, 134.6, 131.5, 128.9 (*s*, *J* = 9.1 Hz), 128.3, 125.9, 114.3, 110.6 (*d*, *J* = 25.2 Hz), 45.1, 36.8, 27.6, 27.1, 23.1, 11.8; **¹⁹F NMR** (376 MHz, CDCl₃) δ 72.7; **HRMS** (ESI⁺): calculated for C₁₆H₁₈FNO₂SNa [M+Na⁺]: *m/z* = 330.0934, *m/z* found 330.0933; **IR** ν_{max} (ATR)/cm⁻¹: 3023, 2865, 1453, 1375, 1212, 1150, 753, 721.

7-(4-Fluorophenyl)-5-methyl-2,3-dihydro-1H-pyrrolizine-6-sulfonyl fluoride (4gm)



Following general procedure I, the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a yellow solid (42.0 mg, 94%). **m.p.** 143.5 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.40 – 7.29 (m, 2H), 7.15 – 6.98 (m, 2H), 3.97 (t, *J* = 7.2 Hz, 2H), 2.86 (t, *J* = 7.4 Hz, 2H), 2.61 – 2.49 (m, 2H), 2.51 (*s*, 3H); **¹³C NMR** (151 MHz, CDCl₃) δ 162.2 (*d*, *J* = 246.0 Hz), 135.0, 132.4 (*d*, *J* = 2.7 Hz), 131.2 (*d*, *J* = 8.1 Hz), 129.0 (*d*, *J* = 3.3 Hz), 115.6, 115.2 (*d*, *J* = 21.5 Hz), 111.1 (*d*, *J* = 27.1 Hz), 45.6, 26.9, 24.0, 12.1; **¹⁹F NMR** (376 MHz, CDCl₃) δ 73.45, -115.89; **HRMS** (APCI): calculated for C₁₄H₁₄F₂NO₂S [M+H⁺]: *m/z* = 298.0713, *m/z* found 298.0711.

5-Methyl-7-(thiophen-3-yl)-2,3-dihydro-1H-pyrrolizine-6-sulfonyl fluoride (4gn)

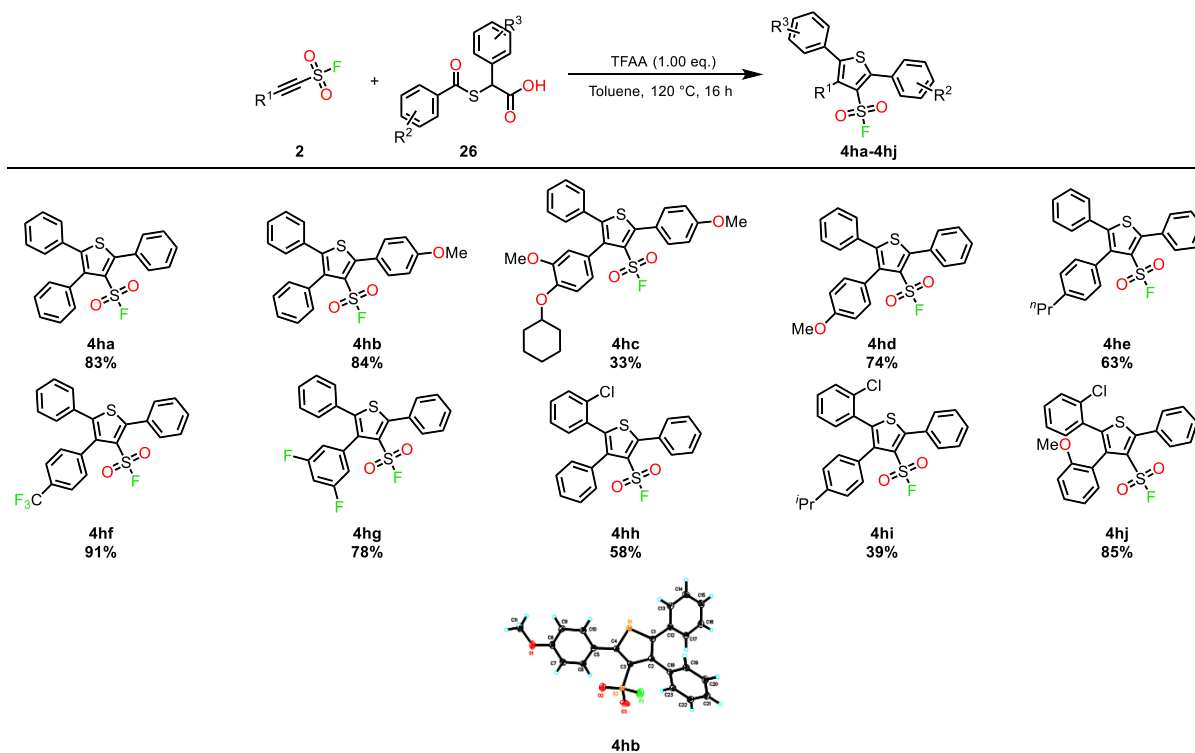


Following general procedure I, the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a yellow solid (40.0 mg, 93%). **m.p.** 125.1 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.37 – 7.29 (m, 2H), 7.21 (d, *J* = 4.9 Hz, 1H), 3.96 (t, *J* = 7.3 Hz, 2H), 2.93 (t, *J* = 7.4 Hz, 2H), 2.60 – 2.52 (m, 2H), 2.51 (s, 3H). **¹³C NMR** (151 MHz, CDCl₃) δ 135.1, 132.7, 132.4 (d, *J* = 3.2 Hz), 128.6, 125.0, 122.7, 111.4, 110.7 (d, *J* = 27.6 Hz), 45.6, 26.9, 24.6, 12.2; **¹⁹F NMR** (376 MHz, CDCl₃) δ 71.7; **HRMS** (APCI): calculated for C₁₂H₁₃FNO₂S₂ [M+H⁺]: *m/z* = 286.0372, *m/z* found 286.0374.

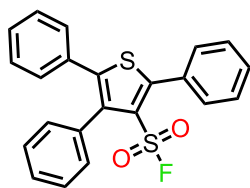
Synthesis and experimental data for compounds 4ha-4hj

General Procedure J

To a solution of the required 2-(benzoylthio)-2-arylacetic acid **26** (100 μmol) in toluene (500 μL) was added TFAA (120 μmol), the required SASF **2** (120 μmol) and stirred at 120 °C for 16 h. The solvent was removed under reduced pressure and the crude product was purified by flash column chromatography to obtain the analytically pure product.

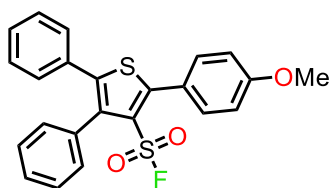


2,4,5-Triphenylthiophene-3-sulfonyl fluoride (4ha)



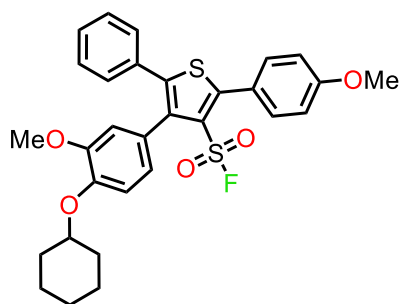
Following general procedure J (300 μ mol of the required 2-(benzoylthio)-2-arylacetic acid used), the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a colourless solid (33.0 mg, 83%). **m.p.** 135 - 136 $^{\circ}$ C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.64 – 7.58 (m, 2H), 7.54 – 7.47 (m, 3H), 7.40 – 7.31 (m, 5H), 7.26 – 7.21 (m, 3H), 7.19 – 7.14 (m, 2H); $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 151.7, 141.7, 137.5, 133.5, 132.0, 131.1, 130.6, 130.0, 129.3, 128.7, 128.6 (d, J = 2.6 Hz), 128.54, 128.4, 127.9 (d, J = 23.7 Hz), 116.0; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ 69.8; **HRMS** (ESI $^+$): calculated for $\text{C}_{22}\text{H}_{15}\text{FO}_2\text{S}_2\text{Na}$ [$\text{M}+\text{Na}^+$]: m/z = 417.0389, m/z found 417.0393; **IR** ν_{max} (ATR)/ cm^{-1} : 3054, 2926, 2852, 2359, 1410, 1201, 746.

5-(4-Methoxyphenyl)-2,4-diphenylthiophene-3-sulfonyl fluoride (4hb)



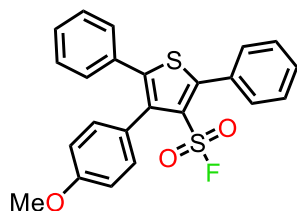
Following general procedure J, the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a colourless solid (36.0 mg, 84%). **m.p.** 110 – 112 $^{\circ}$ C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.58 – 7.51 (m, 2H), 7.40 – 7.30 (m, 5H), 7.24 (m, 3H), 7.19 – 7.12 (m, 2H), 7.07 – 6.98 (m, 2H), 3.89 (s, 3H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 161.1, 152.0, 141.1, 137.5, 133.7, 132.2, 131.4, 130.6, 129.3, 128.7, 128.5, 128.5, 128.4, 123.3, 114.1, 55.5; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ 69.7; **HRMS** (ESI $^+$): calculated for $\text{C}_{23}\text{H}_{17}\text{FO}_3\text{S}_2\text{Na}$ [$\text{M}+\text{Na}^+$]: m/z = 447.0495, m/z found 447.0498; **IR** ν_{max} (ATR)/ cm^{-1} : 3060, 3001, 2927, 2851, 1604, 1407, 1200, 755.

4-(4-(Cyclohexyloxy)-3-methoxyphenyl)-5-(4-methoxyphenyl)-2-phenylthiophene-3-sulfonyl fluoride (4hc)



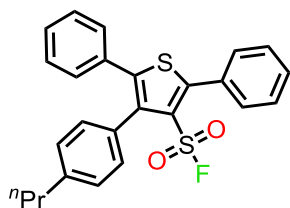
Following general procedure J, the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a colourless solid (18.0 mg, 33%). **m.p.** 72 - 74 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.54 (appt. d, *J* = 8.8 Hz, 2H), 7.27 – 7.20 (m, 3H), 7.21 – 7.13 (m, 2H), 7.01 (appt. d, *J* = 8.8 Hz, 2H), 6.91 – 6.78 (m, 3H), 4.23 (appt. ddd, *J* = 13.8, 9.6, 3.8 Hz, 1H), 3.88 (s, 3H), 3.74 (s, 3H), 2.17 – 1.94 (m, 2H), 1.94 – 1.71 (m, 2H), 1.71 – 1.48 (m, 3H), 1.48 – 1.15 (m, 3H); **¹³C NMR** (101 MHz, CDCl₃) δ 161.1, 151.6, 150.1, 147.4, 140.8, 137.4, 132.4, 131.3, 129.2, 128.7, 128.4, 127.5 (d, *J* = 22.8 Hz), 126.1, 123.4, 123.2, 115.5, 114.7, 114.0, 77.3, 56.1, 55.53, 32.1, 25.7, 24.3; **¹⁹F NMR** (376 MHz, CDCl₃) δ 68.8; **HRMS** (ESI⁺): calculated for C₃₀H₂₉FO₅S₂Na [M+Na⁺]: *m/z* = 575.1332, *m/z* found 575.1338; **IR** *v*_{max} (ATR)/cm⁻¹: 3069, 2931, 2854, 2360, 1605, 1605, 1249, 753.

4-(4-Methoxyphenyl)-2,5-diphenylthiophene-3-sulfonyl fluoride (4hd)



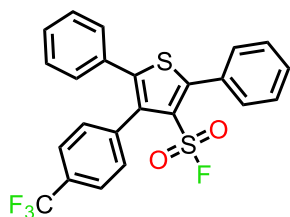
Following general procedure J, the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a colourless solid (31.0 mg, 74%). **m.p.** 149 - 150 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.65 – 7.57 (m, 2H), 7.50 (m, 3H), 7.31 – 7.22 (m, 5H), 7.23 – 7.15 (m, 2H), 6.94 – 6.83 (m, 2H), 3.84 (s, 3H); **¹³C NMR** (101 MHz, CDCl₃) δ 159.7, 151.5 (d, *J* = 2.1 Hz), 141.6 (d, *J* = 2.0 Hz), 137.2, 132.2, 131.8, 131.2, 130.0, 129.9, 129.2, 128.7, 128.5, 128.0 (d, *J* = 23.3 Hz), 125.6, 113.9, 55.3; **¹⁹F NMR** (376 MHz, CDCl₃) δ 69.3; **HRMS** (ESI⁺): calculated for C₂₃H₁₇FO₃S₂Na [M+Na⁺]: *m/z* = 447.0495, *m/z* found 447.0499; **IR** *v*_{max} (ATR)/cm⁻¹: 2934, 2859, 2359, 1605, 1404, 1031, 752.

2,5-Diphenyl-4-(4-propylphenyl)thiophene-3-sulfonyl fluoride (4he)



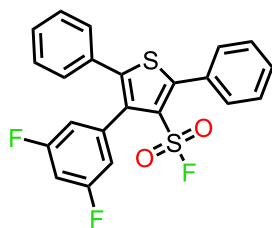
Following general procedure J, the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a colourless solid (28.0 mg, 63%). **m.p.** 157 - 159 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.66 – 7.57 (m, 2H), 7.55 – 7.46 (m, 3H), 7.26 – 7.20 (m, 5H), 7.20 – 7.13 (m, 4H), 2.68 – 2.58 (m, 2H), 1.74 – 1.62 (m, 2H), 0.94 (t, *J* = 7.3 Hz, 3H); **¹³C NMR** (101 MHz, CDCl₃) δ 151.5 (d, *J* = 2.2 Hz), 143.0, 141.5 (d, *J* = 1.8 Hz), 137.7, 132.2, 131.2, 130.7, 130.4, 130.0, 129.9, 129.2, 128.6, 128.5, 128.5, 128.0 (d, *J* = 23.4 Hz), 37.9, 24.3, 13.8; **¹⁹F NMR** (376 MHz, CDCl₃) δ 69.5; **HRMS** (ESI⁺): calculated for C₂₅H₂₁FO₂S₂Na [M+Na⁺]: *m/z* = 459.0859, *m/z* found 459.0854; **IR** *v*_{max} (ATR)/cm⁻¹: 3040, 2958, 2924, 2860, 1408, 1202 750.

2,5-Diphenyl-4-(4-(trifluoromethyl)phenyl)thiophene-3-sulfonyl fluoride (4hf)



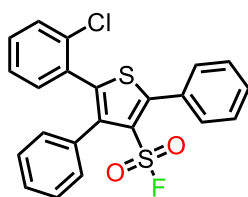
Following general procedure J, the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a colourless solid (42.0 mg, 91%). **m.p.** 199 - 200 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.66 – 7.59 (m, 4H), 7.52 (m, 3H), 7.47 (d, *J* = 8.1 Hz, 2H), 7.34 – 7.24 (m, 3H), 7.14 (appt. dd, *J* = 8.0, 1.6 Hz, 2H); **¹³C NMR** (101 MHz, CDCl₃) δ 152.4 (d, *J* = 2.1 Hz), 142.5, 137.4, 135.8, 131.5, 131.1, 130.8, 130.2, 130.6 (d, *J* = 32.5 Hz), 130.0, 129.3, 129.0, 128.9, 128.6, 127.6 (d, *J* = 24.5 Hz), 125.3 (q, *J* = 3.7 Hz), 124.1 (q, *J* = 272.3 Hz); **¹⁹F NMR** (376 MHz, CDCl₃) δ 70.4, -62.6; **HRMS** (ESI⁺): calculated for C₂₃H₁₄F₄O₂S₂Na [M+Na⁺]: *m/z* = 485.0263, *m/z* found 485.0264; **IR** *v*_{max} (ATR)/cm⁻¹: 3066, 2960, 2924, 2854, 1326, 1124, 746.

4-(3,5-Difluorophenyl)-2,5-diphenylthiophene-3-sulfonyl fluoride (4hg)



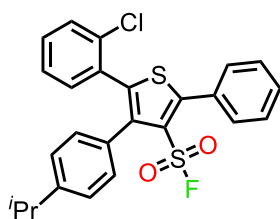
Following general procedure J, the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a colourless solid (34.0 mg, 78%). **m.p.** 165 - 168 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.65 – 7.56 (m, 2H), 7.56 – 7.45 (m, 3H), 7.38 – 7.25 (m, 3H), 7.22 – 7.14 (m, 2H), 6.96 – 6.77 (m, 3H); **¹³C NMR** (101 MHz, CDCl₃) δ 164.0 (d, *J* = 13.0 Hz), 161.5 (d, *J* = 13.0 Hz), 152.4 (d, *J* = 2.1 Hz), 142.6 (d, *J* = 1.9 Hz), 136.6 (t, *J* = 10.3 Hz), 134.7, 131.3, 130.7, 130.2, 130.0, 129.2, 129.1, 129.0, 128.6, 127.4 (d, *J* = 24.8 Hz), 114.0 (d, *J* = 26.0 Hz), 114.0 (d, *J* = 11.5 Hz), 104.3 (t, *J* = 25.1 Hz); **¹⁹F NMR** (376 MHz, CDCl₃) δ 70.6, -106.0 – -115.6 (m); **HRMS** (ESI⁺): calculated for C₂₂H₁₃F₃O₂S₂Na [M+Na⁺]: *m/z* = 453.0201, *m/z* found 453.0200; **IR** *v*_{max} (ATR)/cm⁻¹: 3087, 2927, 2852, 1620, 1202, 1118, 752.

2-(2-Chlorophenyl)-4,5-diphenylthiophene-3-sulfonyl fluoride (4hh)



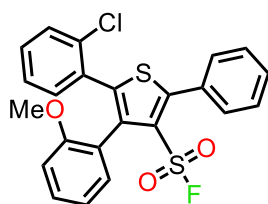
Following general procedure J, the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a colourless solid (25.0 mg, 58%). **m.p.** 95 - 96 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.70 – 7.60 (m, 2H), 7.58 – 7.45 (m, 3H), 7.42 – 7.36 (m, 1H), 7.34 – 7.21 (m, 6H), 7.21 – 7.11 (m, 2H); **¹³C NMR** (101 MHz, CDCl₃) δ 153.1, 140.4, 138.1, 134.7, 133.2, 133.1, 131.0, 130.7, 130.5, 130.1, 130.0, 129.9, 128.5, 128.4, 128.0, 126.9 (d, *J* = 24.4 Hz), 126.7; **¹⁹F NMR** (376 MHz, CDCl₃) δ 70.0; **HRMS** (ESI⁺): calculated for C₂₂H₁₄ClFO₂S₂Na [M+Na⁺]: *m/z* = 451.0000, *m/z* found 451.0005; **IR** *v*_{max} (ATR)/cm⁻¹: 3065, 2925, 2853, 1409, 1201, 744.

2-(2-Chlorophenyl)-4-(4-isopropylphenyl)-5-phenylthiophene-3-sulfonyl fluoride (4hi)



Following general procedure J, the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a colourless solid (18.0 mg, 39%). **m.p.** 112 - 114 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.68 – 7.60 (m, 2H), 7.55 – 7.47 (m, 3H), 7.39 (appt. dd, *J* = 8.0, 0.7 Hz, 1H), 7.27 – 7.17 (m, 3H), 7.14 (m, 4H), 2.93 – 2.77 (m, 1H), 1.21 (d, *J* = 6.9 Hz, 6H); **¹³C NMR** (101 MHz, CDCl₃) δ 152.9, 148.9, 140.6, 137.9, 134.7, 133.2, 131.1, 130.9, 130.3, 130.0, 130.0, 129.8, 128.5, 127.0 (d, *J* = 23.9 Hz), 126.6, 126.0, 33.8, 23.9; **¹⁹F NMR** (376 MHz, CDCl₃) δ 69.6; ; **HRMS** (ESI⁺): calculated for C₂₅H₂₀ClFO₂S₂Na [M+Na⁺]: *m/z* =493.0469, *m/z* found 493.0470; **IR** *v*_{max} (ATR)/cm⁻¹:3071, 2960, 2926, 2889, 2360, 1202, 749.

2-(2-Chlorophenyl)-4-(2-methoxyphenyl)-5-phenylthiophene-3-sulfonyl fluoride (4hj)

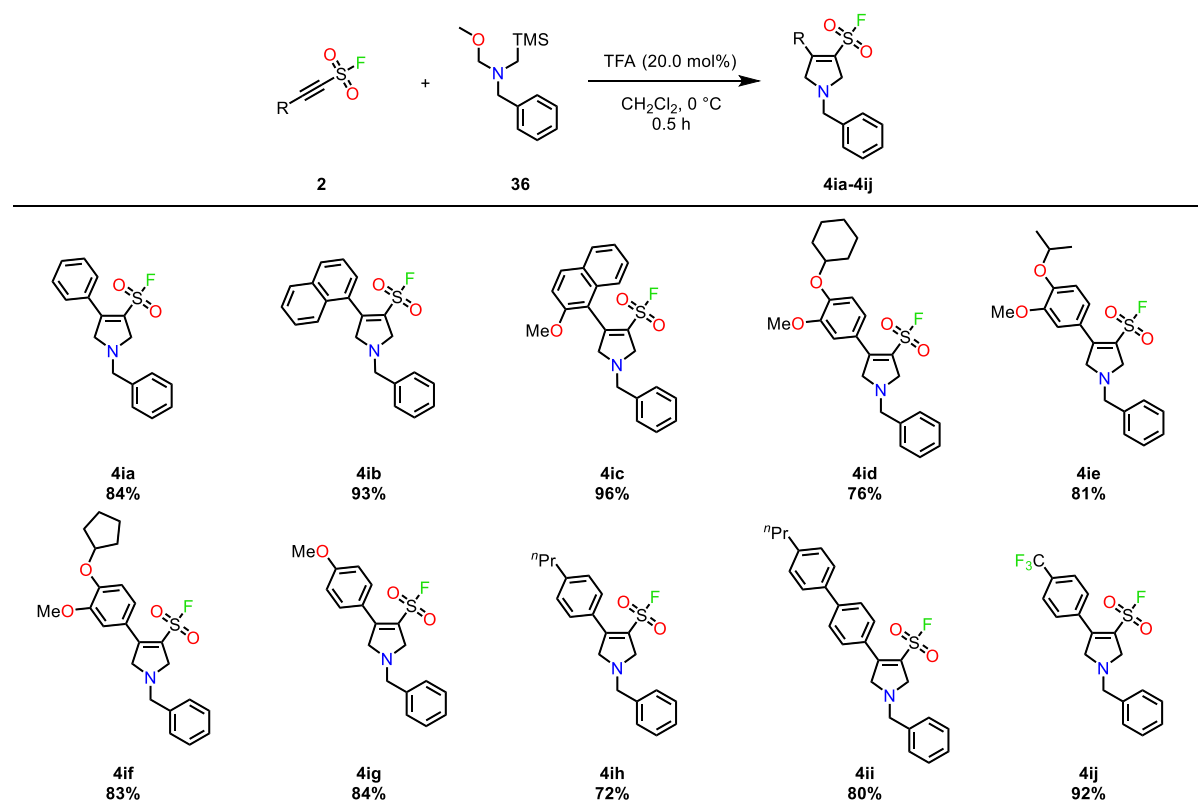


Following general procedure J, the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a colourless solid (39.0 mg, 85%). **m.p.** 119 - 120 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.69 (appt. dd, *J* = 6.5, 2.8 Hz, 2H), 7.54 – 7.47 (m, 3H), 7.39 (d, *J* = 7.9 Hz, 1H), 7.30 – 7.22 (m, 2H), 7.22 – 7.16 (m, 1H), 7.16 – 7.09 (m, 1H), 7.07 (appt. d, *J* = 7.4 Hz, 1H), 6.88 – 6.78 (m, 2H), 3.80 (s, 3H); **¹³C NMR** (101 MHz, CDCl₃) δ 157.6, 152.7, 137.9, 137.1, 134.5, 132.9, 131.4, 131.3, 131.2, 130.2, 130.2, 130.1, 130.0, 129.8, 128.5, 127.5 (d, *J* = 24.0 Hz), 126.6, 122.4, 120.2, 110.4, 55.3; **¹⁹F NMR** (376 MHz, CDCl₃) δ 69.0; **HRMS** (ESI⁺): calculated for C₂₃H₁₆ClFO₃S₂Na [M+Na⁺]: *m/z* =481.0105, *m/z* found 481.0108; **IR** *v*_{max} (ATR)/cm⁻¹:2979, 2934, 1674, 1590, 1412, 1265, 787.

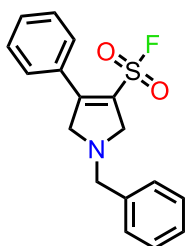
Synthesis and experimental data for compounds 4ia-4aj

General Procedure K

To a solution of the required SASF **2** (100 μmol) in CH_2Cl_2 (500 μL) at 0 $^\circ\text{C}$ was added *N*-(methoxymethyl)-*N*-(trimethylsilylmethyl)benzylamine **36** (92%, 110 μmol , 30.0 μL) and TFA (1.50 μL , 20.0 mol%).^[19] The solution was stirred at 0 $^\circ\text{C}$ for 0.5 h and the solvent was then removed under reduced pressure. The crude product was purified by flash column chromatography to obtain the analytically pure product.

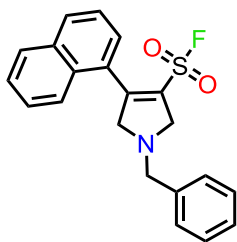


1-Benzyl-4-phenyl-2,5-dihydro-1H-pyrrole-3-sulfonyl fluoride (4ia)



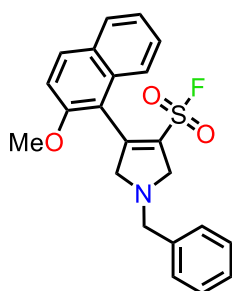
Following general procedure K (250 μmol of SASF), the title compound was isolated by flash column chromatography (0-15% EtOAc in petroleum ether) as a pale yellow oil (66.0 mg, 84%). **¹H NMR** (400 MHz, CDCl_3) δ 7.46 – 7.29 (m, 10H), 4.13 – 4.05 (m, 4H), 3.90 (s, 2H); **¹³C NMR** (101 MHz, CDCl_3) δ 156.2 (d, $J = 3.1$ Hz), 137.7, 130.6, 130.6, 128.8, 128.8, 128.56, 128.1 (d, $J = 0.9$ Hz), 127.8, 125.5 (d, $J = 26.9$ Hz), 65.4 (d, $J = 1.6$ Hz), 60.8, 59.7; **¹⁹F NMR** (376 MHz, CDCl_3) δ 63.8; **HRMS** (ESI⁺): calculated for $\text{C}_{17}\text{H}_{17}\text{FNO}_2\text{S}$ [$\text{M}+\text{H}^+$]: $m/z = 318.0959$, m/z found 318.0966; **IR** ν_{max} (ATR)/ cm^{-1} : 3029, 1603, 1552, 1396, 1207, 1176.

1-Benzyl-4-(naphthalen-1-yl)-2,5-dihydro-1H-pyrrole-3-sulfonyl fluoride (4ib)



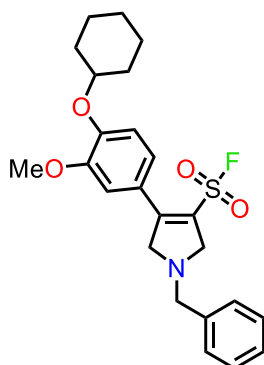
Following general procedure K, the title compound was isolated by flash column chromatography (0-12% EtOAc in petroleum ether) as a pale yellow oil (34.0 mg, 93%). **¹H NMR** (400 MHz, CDCl₃) δ 7.90 (d, *J* = 7.6 Hz, 2H), 7.79 (d, *J* = 7.7 Hz, 1H), 7.60 – 7.47 (m, 3H), 7.44 – 7.28 (m, 6H), 4.27 – 4.04 (m, 4H), 3.95 (s, 2H); **¹³C NMR** (101 MHz, CDCl₃) δ 157.2, 137.7, 133.5, 130.2, 129.9, 129.4 (d, *J* = 27.2 Hz), 128.8, 127.8, 127.2, 126.6, 125.3, 125.1, 124.5, 66.7, 59.9, 59.9; **¹⁹F NMR** (376 MHz, CDCl₃) δ 62.3; **HRMS** (ESI⁺): calculated for C₂₁H₁₉FNO₂S [M+H⁺]: *m/z* = 368.1115, *m/z* found 368.1124; **IR** ν_{max} (ATR)/cm⁻¹: 2925, 1503, 1396, 1194, 1121.

1-Benzyl-4-(2-methoxynaphthalen-1-yl)-2,5-dihydro-1H-pyrrole-3-sulfonyl fluoride (4ic)



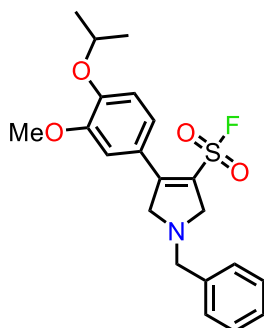
Following general procedure K, the title compound was isolated by flash column chromatography (0-12% EtOAc in petroleum ether) as a pale yellow oil (38.0 mg, 96%). **¹H NMR** (500 MHz, CDCl₃) δ 7.89 (d, *J* = 9.0 Hz, 1H), 7.81 (d, *J* = 8.2 Hz, 1H), 7.65 – 7.62 (m, 1H), 7.52 (ddd, *J* = 8.4, 6.8, 1.3 Hz, 1H), 7.43 (t, *J* = 6.9 Hz, 2H), 7.41 – 7.35 (m, 3H), 7.33 – 7.28 (m, 2H), 4.30 – 4.22 (m, 1H), 4.18 – 4.03 (m, 2H), 4.03 – 3.92 (m, 6H); **¹³C NMR** (126 MHz, CDCl₃) δ 154.3, 153.4, 138.0, 131.5, 131.3, 129.5 (d, *J* = 27.6 Hz), 128.9, 128.8, 128.7, 128.5, 127.7, 127.6, 124.2, 123.4, 113.5, 112.8, 65.0, 60.1, 59.7, 56.5; **¹⁹F NMR** (376 MHz, CDCl₃) δ 59.8; **HRMS** (ESI⁺): calculated for C₂₂H₂₁FNO₃S [M+H⁺]: *m/z* = 398.1221, *m/z* found 398.1230; **IR** ν_{max} (ATR)/cm⁻¹: 2936, 2838, 1620, 1593, 1506, 1396, 1251, 1202, 1065.

1-Benzyl-4-(4-(cyclohexyloxy)-3-methoxyphenyl)-2,5-dihydro-1H-pyrrole-3-sulfonyl fluoride (4id)



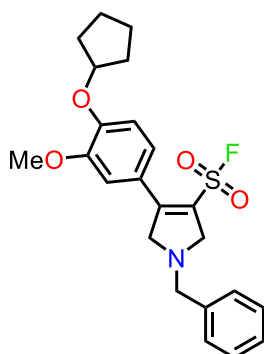
Following general procedure K, the title compound was isolated by flash column chromatography (0-15% EtOAc in petroleum ether) as a pale yellow oil (34.0 mg, 76%). **¹H NMR** (400 MHz, CDCl₃) δ 7.41 – 7.28 (m, 5H), 7.07 (d, *J* = 2.2 Hz, 1H), 6.99 (dd, *J* = 8.5, 2.2 Hz, 1H), 6.88 (d, *J* = 8.5 Hz, 1H), 4.32 – 4.23 (m, 1H), 4.07 (s, 4H), 3.86 (s, 5H), 2.10 – 1.99 (m, 2H), 1.89 – 1.79 (m, 2H), 1.65 – 1.53 (m, 3H), 1.43 – 1.23 (m, 3H); **¹³C NMR** (126 MHz, CDCl₃) δ 155.5, 149.8, 149.8, 137.9, 128.8, 128.7, 127.7, 123.2 (d, *J* = 25.9 Hz), 122.7, 121.8, 114.3, 112.8, 77.1, 65.1, 61.2, 59.8, 56.3, 32.0, 25.6, 24.2; **¹⁹F NMR** (376 MHz, CDCl₃) δ 64.0; **HRMS** (ESI⁺): calculated for C₂₄H₂₉FNO₄S [M+H⁺]: *m/z* = 446.1796, *m/z* found 446.1785; **IR** *v*_{max} (ATR)/cm⁻¹: 2933, 2856, 1501, 1453, 1395, 1203.

1-Benzyl-4-(4-isopropoxy-3-methoxyphenyl)-2,5-dihydro-1H-pyrrole-3-sulfonyl fluoride (4ie)



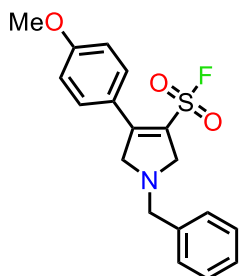
Following general procedure K, the title compound was isolated by flash column chromatography (0-15% EtOAc in petroleum ether) as a pale yellow oil (33.0 mg, 81%). **¹H NMR** (400 MHz, CDCl₃) δ 7.40 – 7.28 (m, 5H), 7.08 (d, *J* = 2.1 Hz, 1H), 6.99 (dd, *J* = 8.5, 2.1 Hz, 1H), 6.87 (d, *J* = 8.5 Hz, 1H), 4.60 (hept, *J* = 6.0 Hz, 1H), 4.07 (s, 4H), 3.87 (s, 5H), 1.40 (d, *J* = 6.1 Hz, 6H); **¹³C NMR** (101 MHz, CDCl₃) δ 155.5 (d, *J* = 2.8 Hz), 149.9, 149.7, 137.9, 128.8, 128.7, 127.7, 123.2 (d, *J* = 26.0 Hz), 122.7, 121.8, 113.9, 112.6, 71.4, 65.1, 61.2, 59.8, 56.2, 22.1; **¹⁹F NMR** (376 MHz, CDCl₃) δ 64.0; **HRMS** (ESI⁺): calculated for C₂₁H₂₅FNO₄S [M+H⁺]: *m/z* = 406.1483, *m/z* found 406.1498; **IR** *v*_{max} (ATR)/cm⁻¹: 2976, 2931, 1599, 1510, 1402, 1257, 1200, 1140.

1-Benzyl-4-(4-(cyclopentyloxy)-3-methoxyphenyl)-2,5-dihydro-1H-pyrrole-3-sulfonyl fluoride (4if)



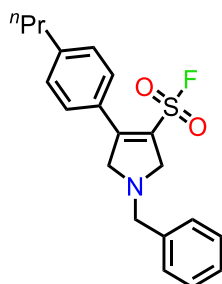
Following general procedure K, the title compound was isolated by flash column chromatography (0-15% EtOAc in petroleum ether) as a colourless oil (36.0 mg, 83%). **¹H NMR** (400 MHz, CDCl₃) δ 7.41 – 7.28 (m, 5H), 7.07 (d, *J* = 2.1 Hz, 1H), 6.99 (dd, *J* = 8.5, 2.1 Hz, 1H), 6.85 (d, *J* = 8.5 Hz, 1H), 4.80 (appt. dt, *J* = 9.4, 3.1 Hz, 1H), 4.07 (s, 4H), 3.86 (d, *J* = 7.2 Hz, 5H), 2.02 – 1.78 (m, 6H), 1.69 – 1.58 (m, 2H); **¹³C NMR** (101 MHz, CDCl₃) δ 155.5, 150.3, 149.5, 137.7, 128.8, 128.8, 127.8, 123.0 (d, *J* = 26.0 Hz), 122.3, 121.9, 113.6, 112.6, 80.6, 65.0, 61.1, 59.8, 56.3, 33.0, 24.3; **¹⁹F NMR** (376 MHz, CDCl₃) δ 64.1; **HRMS** (ESI⁺): calculated for C₂₃H₂₇FNO₄S [M+H⁺]: *m/z* = 432.1639, *m/z* found 432.1645; **IR** *v*_{max} (ATR)/cm⁻¹: 2956, 2870, 1501, 1395, 1236, 1203, 1165, 1031.

1-Benzyl-4-(4-methoxyphenyl)-2,5-dihydro-1H-pyrrole-3-sulfonyl fluoride (4ig)



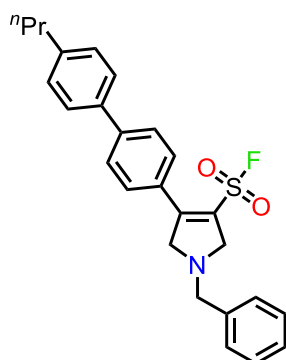
Following general procedure K, the title compound was isolated by flash column chromatography (0-15% EtOAc in petroleum ether) as a white solid (29.0 mg, 84%). **m.p.** 73 - 75 °C **¹H NMR** (400 MHz, CDCl₃) δ 7.42 (appt. d, *J* = 9.0 Hz, 2H), 7.39 – 7.29 (m, 5H), 6.92 (appt. d, *J* = 9.0 Hz, 2H), 4.07 (s, 4H), 3.87 (s, 2H), 3.84 (s, 3H); **¹³C NMR** (101 MHz, CDCl₃) δ 161.7, 155.6, 137.7, 130.3, 128.8, 127.8, 123.2 (d, *J* = 26.3 Hz), 122.6, 114.0, 65.1, 61.0, 59.8, 55.5; **¹⁹F NMR** (376 MHz, CDCl₃) δ 64.0; **HRMS** (ESI⁺): calculated for C₁₈H₁₉FNO₃S [M+H⁺]: *m/z* = 348.1064, *m/z* found 348.1077; **IR** *v*_{max} (ATR)/cm⁻¹: 2926, 2816, 1599, 1514, 1400, 1259, 1186, 1023.

1-Benzyl-4-(4-propylphenyl)-2,5-dihydro-1H-pyrrole-3-sulfonyl fluoride (4ih)



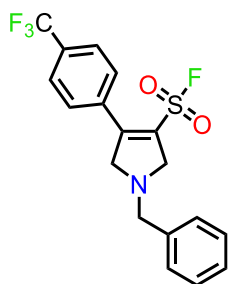
Following general procedure K, the title compound was isolated by flash column chromatography (0-10% EtOAc in petroleum ether) as a colourless oil (26.0 mg, 72%). **¹H NMR** (400 MHz, CDCl₃) δ 7.40 – 7.28 (m, 7H), 7.22 (appt. d, *J* = 8.2 Hz, 2H), 4.12 – 4.03 (m, 4H), 3.88 (s, 2H), 2.64 – 2.59 (m, 2H), 1.73 – 1.58 (m, 2H), 0.95 (t, *J* = 7.3 Hz, 3H); **¹³C NMR** (101 MHz, CDCl₃) δ 156.2 (d, *J* = 2.9 Hz), 145.9, 137.7, 128.8, 128.8, 128.7, 128.3, 128.3, 127.8, 124.4 (d, *J* = 26.6 Hz), 65.3, 60.9, 59.8, 38.0, 24.3, 13.9; **¹⁹F NMR** (376 MHz, CDCl₃) δ 63.8; **HRMS** (ESI⁺): calculated for C₂₀H₂₃FNO₂S [M+H⁺]: *m/z* = 360.1428, *m/z* found 360.1435; **IR** *v*_{max} (ATR)/cm⁻¹: 2958, 2928, 1556, 1497, 1397, 1206, 1176, 1112.

1-Benzyl-4-(4'-propyl-[1,1'-biphenyl]-4-yl)-2,5-dihydro-1H-pyrrole-3-sulfonyl fluoride (4ii)



Following general procedure K, the title compound was isolated by flash column chromatography (0-10% EtOAc in petroleum ether) as a white solid (35.0 mg, 80%). **m.p.** 123 - 125 °C **¹H NMR** (400 MHz, CDCl₃) δ 7.63 (appt. d, *J* = 8.3 Hz, 2H), 7.54 – 7.46 (m, 4H), 7.40 – 7.25 (m, 7H), 4.10 (s, 4H), 3.89 (s, 2H), 2.67 – 2.61 (m, 2H), 1.75 – 1.64 (m, 2H), 0.98 (t, *J* = 7.3 Hz, 3H); **¹³C NMR** (101 MHz, CDCl₃) δ 155.8, 143.5, 142.9, 137.8, 137.3, 129.2, 129.0, 128.8, 128.8, 127.8, 127.1, 127.0, 125.0 (d, *J* = 26.7 Hz), 65.3, 61.0, 59.8, 37.8, 24.6, 14.0; **¹⁹F NMR** (376 MHz, CDCl₃) δ 63.9; **HRMS** (ESI⁺): calculated for C₂₆H₂₇FNO₂S [M+H⁺]: *m/z* = 436.1741, *m/z* found 436.1727; **IR** *v*_{max} (ATR)/cm⁻¹: 2870, 2800, 1603, 1402, 1201, 1155.

1-Benzyl-4-(4-(trifluoromethyl)phenyl)-2,5-dihydro-1H-pyrrole-3-sulfonyl fluoride (4ij)

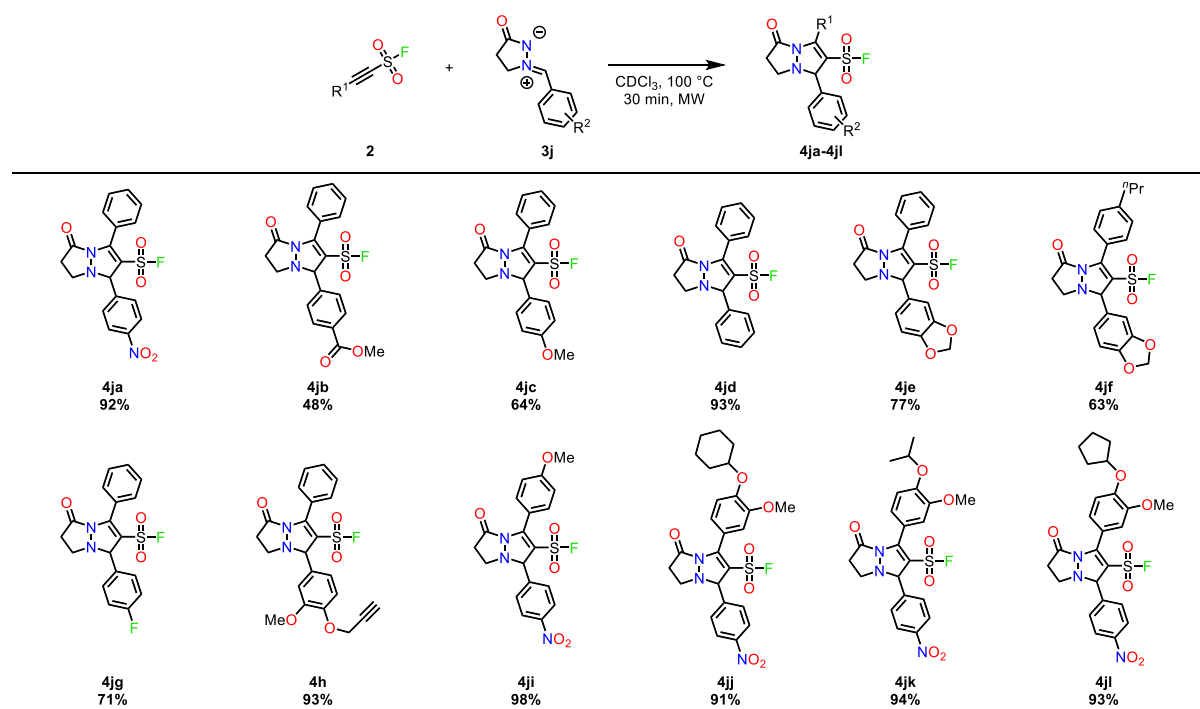


Following general procedure K, the title compound was isolated by flash column chromatography (0-10% EtOAc in petroleum ether) as a white solid (36.0 mg, 94%). **m.p.** 72 - 73 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.68 (d, *J* = 8.2 Hz, 2H), 7.51 (d, *J* = 8.2 Hz, 2H), 7.40 – 7.29 (m, 5H), 4.12 (t, *J* = 4.6 Hz, 2H), 4.07 (dd, *J* = 3.9, 2.4 Hz, 2H), 3.89 (s, 2H); **¹³C NMR** (101 MHz, CDCl₃) δ 154.6 (d, *J* = 3.2 Hz), 137.6, 134.2, 132.3 (q, *J* = 32.9 Hz), 128.9, 128.7, 128.5 (d, *J* = 0.8 Hz), 127.9, 127.7 (d, *J* = 27.5 Hz), 125.6 (q, *J* = 3.7 Hz), 123.8 (q, *J* = 272.5 Hz), 65.4 (d, *J* = 1.4 Hz), 60.7, 59.7; **¹⁹F NMR** (376 MHz, CDCl₃) δ 63.6, -63.0; **HRMS** (ESI⁺): calculated for C₁₈H₁₆F₄NO₂S [M+H⁺]: *m/z* = 386.0832, *m/z* found 386.0839; **IR** *v*_{max} (ATR)/cm⁻¹: 2928, 2802, 1616, 1409, 1320, 1206, 1172, 1124, 1067.

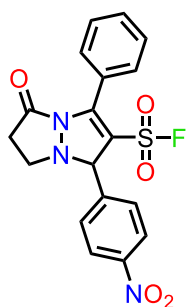
Synthesis and experimental data for compounds 4ja-4jl

General Procedure L

To a solution of the required azomethine imine **3j** (100 μ mol) in CDCl_3 (1.00 mL) was added the required SASF **2** (100 μ mol). The reaction vial was sealed and irradiated with microwaves at 100 $^\circ\text{C}$ for 0.5 h. The solvent was then removed under reduced pressure and the crude product was purified by flash column chromatography to obtain the analytically pure product.



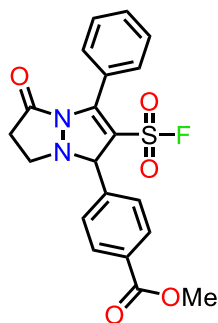
1-(4-Nitrophenyl)-5-oxo-3-phenyl-6,7-dihydro-1H,5H-pyrazolo[1,2-a]pyrazole-2-sulfonyl fluoride (**4ja**)



Following general procedure L (250 μ mol of SASF), the title compound was isolated by flash column chromatography (0-50% EtOAc in petroleum ether) as a yellow foam (93.0 mg, 92%). **m.p.** 72 - 74 $^\circ\text{C}$; **$^1\text{H NMR}$** (400 MHz, CDCl_3) δ 8.32 (appt. d, $J = 8.8$ Hz, 2H), 7.77 (appt. d, $J = 8.8$ Hz, 2H), 7.62 - 7.56 (m, 3H), 7.54 - 7.48 (m, 2H), 5.52 (s, 1H), 3.47 (appt. td, $J = 8.4, 2.8$ Hz, 1H), 3.21 (ddd, $J = 11.2, 8.8, 7.5$ Hz, 1H), 3.00 (ddd, $J = 17.0, 11.2, 8.2$ Hz, 1H), 2.86 (ddd, $J = 17.0, 7.5, 2.8$ Hz, 1H); **$^{13}\text{C NMR}$** (101 MHz, CDCl_3) δ 164.8, 148.8, 148.6 (d, $J = 2.7$ Hz), 143.6, 132.2, 129.9, 129.8, 128.3, 124.1, 123.2, 110.3 (d, $J = 29.1$ Hz), 73.4, 51.0, 36.0; **$^{19}\text{F NMR}$** (376 MHz,

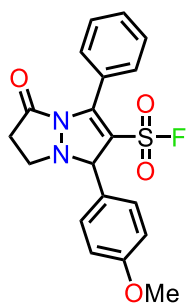
CDCl_3) δ 73.0; **HRMS** (ESI⁺): calculated for $\text{C}_{18}\text{H}_{14}\text{FN}_3\text{O}_5\text{SNa}$ [$\text{M}+\text{Na}^+$]: $m/z = 426.0530$, m/z found 426.0531; **IR** ν_{max} (ATR)/ cm^{-1} : 2927, 2855, 1739, 1520, 1404, 1345, 1190.

Methyl 4-(2-(fluorosulfonyl)-5-oxo-3-phenyl-6,7-dihydro-1H,5H-pyrazolo[1,2- α]pyrazol-1-yl)benzoate (4jb)



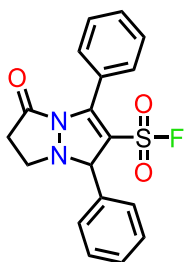
Following general procedure L, the title compound was isolated by flash column chromatography (0-40% EtOAc in petroleum ether) as a yellow foam (24.0 mg, 58%). **m.p.** 70 - 72 °C; **¹H NMR** (400 MHz, CDCl_3) δ 8.14 (d, $J = 8.4$ Hz, 2H), 7.64 – 7.55 (m, 5H), 7.50 (appt. t, $J = 7.4$ Hz, 2H), 5.48 (s, 1H), 3.95 (s, 3H), 3.34 (appt. td, $J = 7.9, 4.7$ Hz, 1H), 3.17 (dd, $J = 17.4, 9.1$ Hz, 1H), 2.98 – 2.82 (m, 2H); **¹³C NMR** (101 MHz, CDCl_3) δ 166.6, 164.9, 148.2 (d, $J = 2.8$ Hz), 140.7, 132.1, 131.5, 130.3, 129.9, 129.0, 128.3, 123.5, 111.2 (d, $J = 28.8$ Hz), 73.2, 52.4, 50.1, 36.2; **¹⁹F NMR** (376 MHz, CDCl_3) δ 72.7; **HRMS** (ESI⁺): calculated for $\text{C}_{20}\text{H}_{18}\text{FN}_2\text{O}_5\text{S}$ [$\text{M}+\text{H}^+$]: $m/z = 417.0915$, m/z found 417.0908; **IR** ν_{max} (ATR)/ cm^{-1} : 2927, 2856, 1719, 1612, 1404, 1365, 1278, 1190.

1-(4-Methoxyphenyl)-5-oxo-3-phenyl-6,7-dihydro-1H,5H-pyrazolo[1,2- α]pyrazole-2-sulfonyl fluoride (4jc)



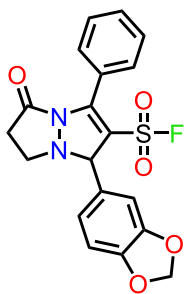
Following general procedure L, the title compound was isolated by flash column chromatography (0-40% EtOAc in petroleum ether) as a yellow foam (25.0 mg, 64%). **m.p.** 108 - 110 °C; **¹H NMR** (400 MHz, CDCl_3) δ 7.64 – 7.55 (m, 3H), 7.50 (appt. t, $J = 7.4$ Hz, 2H), 7.41 (d, $J = 8.7$ Hz, 2H), 6.99 (d, $J = 8.7$ Hz, 2H), 5.43 (s, 1H), 3.85 (s, 3H), 3.21 – 3.06 (m, 2H), 2.95 – 2.77 (m, 2H); **¹³C NMR** (101 MHz, CDCl_3) δ 165.1, 160.8, 147.6, 132.1, 130.2, 130.0, 128.3, 126.7, 123.8, 114.6, 112.1 (d, $J = 28.1$ Hz), 72.0, 55.5, 48.4, 36.4; **¹⁹F NMR** (376 MHz, CDCl_3) δ 72.8; **HRMS** (ESI⁺): calculated for $\text{C}_{19}\text{H}_{18}\text{FN}_2\text{O}_4\text{S}$ [$\text{M}+\text{H}^+$]: $m/z = 389.0966$, m/z found 389.0960; **IR** ν_{max} (ATR)/ cm^{-1} : 2934, 1727, 1610, 1404, 1369, 1191.

5-Oxo-1,3-diphenyl-6,7-dihydro-1*H*,5*H*-pyrazolo[1,2-*a*]pyrazole-2-sulfonyl fluoride (4jd)



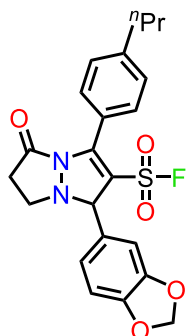
Following general procedure L (250 μ mol of the required SASF), the title compound was isolated by flash column chromatography (0-40% EtOAc in petroleum ether) as a yellow foam (84.0 mg, 93%). **m.p.** 58 - 60 $^{\circ}$ C; **1 H NMR** (400 MHz, CDCl_3) δ 7.64 – 7.55 (m, 3H), 7.53 – 7.43 (m, 7H), 5.46 (s, 1H), 3.26 – 3.18 (m, 1H), 3.12 (appt. td, J = 8.8, 4.4 Hz, 1H), 2.93 – 2.79 (m, 2H); **13 C NMR** (101 MHz, CDCl_3) δ 165.0, 147.8 (d, J = 2.7 Hz), 135.1, 132.0, 129.9, 129.8, 129.2, 128.9, 128.3, 123.7, 111.9 (d, J = 28.5 Hz), 72.9, 49.0, 36.4; **19 F NMR** (376 MHz, CDCl_3) δ 72.7; **HRMS** (ESI $^+$): calculated for $\text{C}_{18}\text{H}_{16}\text{FN}_2\text{O}_3\text{S}$ [$\text{M}+\text{H}^+$]: m/z = 359.0855, m/z found 359.0861; **IR** ν_{max} (ATR)/ cm^{-1} : 2926, 1736, 1571, 1402, 1364, 1191.

1-(Benzo[*d*][1,3]dioxol-5-yl)-5-oxo-3-phenyl-6,7-dihydro-1*H*,5*H*-pyrazolo[1,2-*a*]pyrazole-2-sulfonyl fluoride (4je)



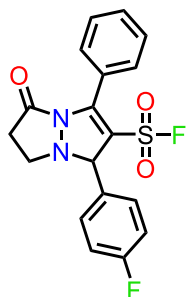
Following general procedure L, the title compound was isolated by flash column chromatography (0-60% EtOAc in petroleum ether) as a yellow foam (37.0 mg, 92%). **m.p.** 172 - 173 $^{\circ}$ C; **1 H NMR** (400 MHz, CDCl_3) δ 7.63 – 7.55 (m, J = 7.5, 6.8, 4.1 Hz, 3H), 7.53 – 7.47 (appt. dd, J = 11.4, 4.4 Hz, 2H), 7.00 (d, J = 1.6 Hz, 1H), 6.94 (dd, J = 8.0, 1.7 Hz, 1H), 6.87 (d, J = 7.9 Hz, 1H), 6.03 (s, 2H), 5.39 (s, 1H), 3.23 (appt. td, J = 8.3, 6.4 Hz, 1H), 3.11 (dd, J = 16.5, 8.4 Hz, 1H), 2.95 – 2.79 (m, 2H); **13 C NMR** (101 MHz, CDCl_3) δ 164.9, 148.9, 148.6, 147.7, 132.1, 129.9, 128.7, 128.3, 123.7, 122.9, 111.8 (d, J = 28.3 Hz), 108.8, 108.7, 101.7, 72.4, 48.6, 36.4; **19 F NMR** (376 MHz, CDCl_3) δ 72.8; **HRMS** (ESI $^+$): calculated for $\text{C}_{19}\text{H}_{16}\text{FN}_2\text{O}_5\text{S}$ [$\text{M}+\text{H}^+$]: m/z = 403.0753, m/z found 403.0748; **IR** ν_{max} (ATR)/ cm^{-1} : 2926, 1722, 1411, 1371, 1252, 1193.

1-(Benzo[d][1,3]dioxol-5-yl)-5-oxo-3-(4-propylphenyl)-6,7-dihydro-1*H*,5*H*-pyrazolo[1,2-*a*]pyrazole-2-sulfonyl fluoride (4jf)



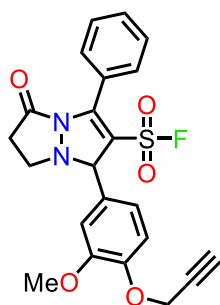
Following general procedure L, the title compound was isolated by flash column chromatography (0-40% EtOAc in petroleum ether) as a yellow foam (29.0 mg, 65%). **m.p.** 144 - 145 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.53 (appt. d, *J* = 8.2 Hz, 2H), 7.29 (appt. d, *J* = 8.3 Hz, 2H), 6.97 (d, *J* = 1.6 Hz, 1H), 6.92 (dd, *J* = 7.9, 1.7 Hz, 1H), 6.87 (d, *J* = 7.9 Hz, 1H), 6.03 (s, 1H), 5.37 (s, 1H), 3.19 (dd, *J* = 15.5, 8.5 Hz, 1H), 3.10 (dd, *J* = 16.8, 7.8 Hz, 1H), 2.95 – 2.77 (m, 1H), 2.71 – 2.63 (m, 1H), 1.75 – 1.65 (m, 1H), 0.98 (t, *J* = 7.3 Hz, 2H); **¹³C NMR** (101 MHz, CDCl₃) δ 165.0, 148.9, 148.6, 148.1 (d, *J* = 2.6 Hz), 147.4, 130.1, 128.7, 128.3, 122.9, 120.7, 111.3 (d, *J* = 28.2 Hz), 108.8, 108.7, 101.7, 72.2, 48.3, 38.3, 36.4, 24.1, 14.0; **¹⁹F NMR** (376 MHz, CDCl₃) δ 73.0; **HRMS** (ESI⁺): calculated for C₂₂H₂₂FN₂O₅S [M+H⁺]: *m/z* = 445.1222, *m/z* found 445.1221; **IR** *v*_{max} (ATR)/cm⁻¹: 2926, 1731, 1504, 1407, 1358, 1178.

1-(4-Fluorophenyl)-5-oxo-3-phenyl-6,7-dihydro-1*H*,5*H*-pyrazolo[1,2-*a*]pyrazole-2-sulfonyl fluoride (4jg)



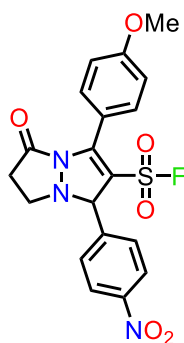
Following general procedure L, the title compound was isolated by flash column chromatography (0-40% EtOAc in petroleum ether) as a yellow foam (29.0 mg, 77%). **m.p.** 121 - 123 °C; **¹H NMR** (500 MHz, CDCl₃) δ 7.62 – 7.55 (m, 3H), 7.54 – 7.47 (m, 4H), 7.19 – 7.13 (m, 2H), 5.44 (s, 1H), 3.29 (ddd, *J* = 9.0, 7.3, 5.7 Hz, 1H), 3.16 – 3.09 (m, 1H), 2.91 – 2.86 (m, 2H); **¹³C NMR** (126 MHz, CDCl₃) δ 164.9, 163.5 (d, *J* = 249.0 Hz), 147.9, 132.1, 131.4 (d, *J* = 3.1 Hz), 130.7 (d, *J* = 8.5 Hz), 129.9, 128.8 (d, *J* = 7.7 Hz), 128.3, 123.6, 116.1 (d, *J* = 21.8 Hz), 111.6 (d, *J* = 28.6 Hz), 72.6, 49.5, 36.3; **¹⁹F NMR** (376 MHz, CDCl₃) δ 72.62, -111.6; **HRMS** (ESI⁺): calculated for C₁₈H₁₅F₂N₂O₃S [M+H⁺]: *m/z* = 377.0760, *m/z* found 377.0755; **IR** *v*_{max} (ATR)/cm⁻¹: 2924, 1735, 1605, 1508, 1404, 1369, 1189.

1-(3-Methoxy-4-(prop-2-yn-1-yloxy)phenyl)-5-oxo-3-phenyl-6,7-dihydro-1H,5H-pyrazolo[1,2-*a*]pyrazole-2-sulfonyl fluoride (4jh)



Following general procedure L (250 μ mol of the required SASF), the title compound was isolated by flash column chromatography (0-50% EtOAc in petroleum ether) as a yellow foam (103 mg, 93%). **m.p.** 62 - 63 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.64 – 7.55 (m, 3H), 7.52 – 7.47 (m, 2H), 7.11 (d, *J* = 8.2 Hz, 1H), 7.06 – 7.01 (m, 2H), 5.41 (s, 1H), 4.81 (d, *J* = 2.4 Hz, 2H), 3.93 (s, 3H), 3.23 (appt. td, *J* = 8.1, 6.2 Hz, 1H), 3.11 (dd, *J* = 16.6, 8.6 Hz, 1H), 2.94 – 2.80 (m, 2H), 2.55 (t, *J* = 2.4 Hz, 1H); **¹³C NMR** (126 MHz, CDCl₃) δ 165.0, 150.1, 148.1, 147.7, 132.1, 129.9, 128.5, 128.3, 123.7, 121.4, 114.0, 112.0, 111.8 (d, *J* = 28.4 Hz), 78.4, 76.3, 72.6, 56.8, 56.2, 48.8, 36.4; **¹⁹F NMR** (376 MHz, CDCl₃) δ 72.6; **HRMS** (ESI⁺): calculated for C₂₂H₂₀FN₂O₅S [M+H⁺]: *m/z* = 443.1071, *m/z* found 443.1060; **IR** ν_{max} (ATR)/cm⁻¹: 3284, 2936, 1733, 1511, 1402, 1365, 1191.

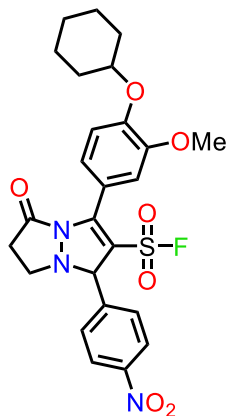
3-(4-Methoxyphenyl)-1-(4-nitrophenyl)-5-oxo-6,7-dihydro-1H,5H-pyrazolo[1,2-*a*]pyrazole-2-sulfonyl fluoride (4ji)



Following general procedure L, the title compound was isolated by flash column chromatography (0-40% EtOAc in petroleum ether) as a yellow foam (42.0 mg, 98%). **m.p.** 65 - 66 °C; **¹H NMR** (400 MHz, CDCl₃) δ 8.31 (appt. d, *J* = 8.6 Hz, 2H), 7.74 (appt. d, *J* = 8.7 Hz, 2H), 7.58 (appt. d, *J* = 8.7 Hz, 2H), 7.00 (appt. d, *J* = 8.8 Hz, 2H), 5.48 (s, 1H), 3.88 (s, 3H), 3.41 (appt. td, *J* = 8.5, 3.4 Hz, 1H), 3.22 – 3.13 (m, 1H), 2.98 (ddd, *J* = 18.9, 10.7, 8.3 Hz, 1H), 2.86 (ddd, *J* = 16.8, 7.6, 3.2 Hz, 1H); **¹³C NMR** (101 MHz, CDCl₃) δ 165.0, 162.9, 148.8, 148.7, 143.6, 132.2, 130.0, 124.1, 114.7, 113.8, 108.9 (d, *J* = 28.5 Hz), 73.3, 55.5, 50.7, 36.1; **¹⁹F NMR** (376 MHz, CDCl₃) δ 73.4; **HRMS** (ESI⁺):

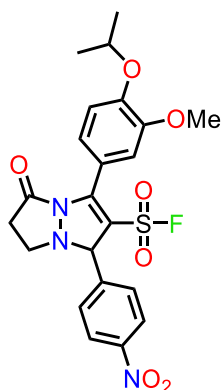
calculated for $C_{19}H_{17}FN_3O_6S$ [$M+H^+$]: $m/z = 434.0817$, m/z found 434.0832; IR ν_{max} (ATR)/ cm^{-1} : 2925, 2853, 1738, 1607, 1504, 1345, 1174.

3-(4-(Cyclohexyloxy)-3-methoxyphenyl)-1-(4-nitrophenyl)-5-oxo-6,7-dihydro-1H,5H-pyrazolo[1,2-*a*]pyrazole-2-sulfonyl fluoride (4jj)



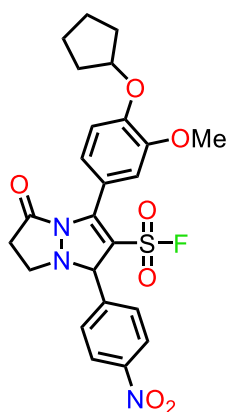
Following general procedure L, the title compound was isolated by flash column chromatography (0-60% EtOAc in petroleum ether) as a yellow foam (48.0 mg, 91%). **m.p.** 76 - 78 °C; **1H NMR** (400 MHz, $CDCl_3$) δ 8.30 (appt. d, $J = 8.7$ Hz, 2H), 7.74 (appt. d, $J = 8.7$ Hz, 2H), 7.22 (dd, $J = 8.4, 2.1$ Hz, 1H), 7.13 (d, $J = 2.1$ Hz, 1H), 6.95 (d, $J = 8.6$ Hz, 1H), 5.48 (s, 1H), 4.37 - 4.28 (m, 1H), 3.88 (s, 3H), 3.40 (appt. td, $J = 8.4, 3.5$ Hz, 1H), 3.23 - 3.13 (m, 1H), 2.98 (ddd, $J = 18.6, 10.4, 8.2$ Hz, 1H), 2.87 (ddd, $J = 16.8, 7.7, 3.5$ Hz, 1H), 2.07 (dd, $J = 9.6, 6.5$ Hz, 2H), 1.90 - 1.79 (m, 2H), 1.67 - 1.55 (m, 2H), 1.45 - 1.22 (m, 4H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 164.9, 151.3, 149.3, 148.8, 148.7, 143.7, 130.0, 124.4, 124.1, 114.2, 114.2, 113.2, 108.8 (d, $J = 28.3$ Hz), 77.1, 73.3, 56.3, 50.5, 36.2, 32.0, 25.6, 24.2; **^{19}F NMR** (376 MHz, $CDCl_3$) δ 73.8; **HRMS** (ESI^+): calculated for $C_{25}H_{27}FN_3O_7S$ [$M+H^+$]: $m/z = 532.1548$, m/z found 532.1538; IR ν_{max} (ATR)/ cm^{-1} : 2933, 2857, 1735, 1503, 1344, 1189.

3-(4-Isopropoxy-3-methoxyphenyl)-1-(4-nitrophenyl)-5-oxo-6,7-dihydro-1H,5H-pyrazolo[1,2-*a*]pyrazole-2-sulfonyl fluoride (4jk)



Following general procedure L, the title compound was isolated by flash column chromatography (0-60% EtOAc in petroleum ether) as a yellow foam (46.0 mg, 94%). **m.p.** 58 - 60 °C; **¹H NMR** (400 MHz, CDCl₃) δ 8.31 (appt. d, *J* = 8.7 Hz, 2H), 7.74 (appt. d, *J* = 8.7 Hz, 2H), 7.23 (dd, *J* = 8.4, 2.1 Hz, 1H), 7.13 (d, *J* = 2.1 Hz, 1H), 6.94 (d, *J* = 8.6 Hz, 1H), 5.48 (s, 1H), 4.66 (hept, *J* = 6.2 Hz, 1H), 3.89 (s, 3H), 3.40 (appt. td, *J* = 8.4, 3.5 Hz, 1H), 3.19 (appt. dt, *J* = 10.4, 8.3 Hz, 1H), 2.98 (ddd, *J* = 18.8, 10.6, 8.2 Hz, 1H), 2.87 (ddd, *J* = 16.9, 7.7, 3.5 Hz, 1H), 1.43 (d, *J* = 6.1 Hz, 6H); **¹³C NMR** (101 MHz, CDCl₃) δ 164.9, 151.4, 149.2, 148.8, 148.7, 143.7, 130.0, 124.4, 124.1, 114.3, 114.0, 112.8, 108.9 (d, *J* = 28.4 Hz), 73.3, 71.4, 56.3, 50.6, 36.2, 22.1, 22.1; **¹⁹F NMR** (376 MHz, CDCl₃) δ 73.7; **HRMS** (ESI⁺): calculated for C₂₂H₂₃FN₃O₇S [M+H⁺]: *m/z* = 492.1235, *m/z* found 492.1223; **IR** *v*_{max} (ATR)/cm⁻¹: 2925, 2854, 1742, 1504, 1346, 1190.

3-(4-(Cyclopentyloxy)-3-methoxyphenyl)-1-(4-nitrophenyl)-5-oxo-6,7-dihydro-1H,5H-pyrazolo[1,2-*a*]pyrazole-2-sulfonyl fluoride (4jl)



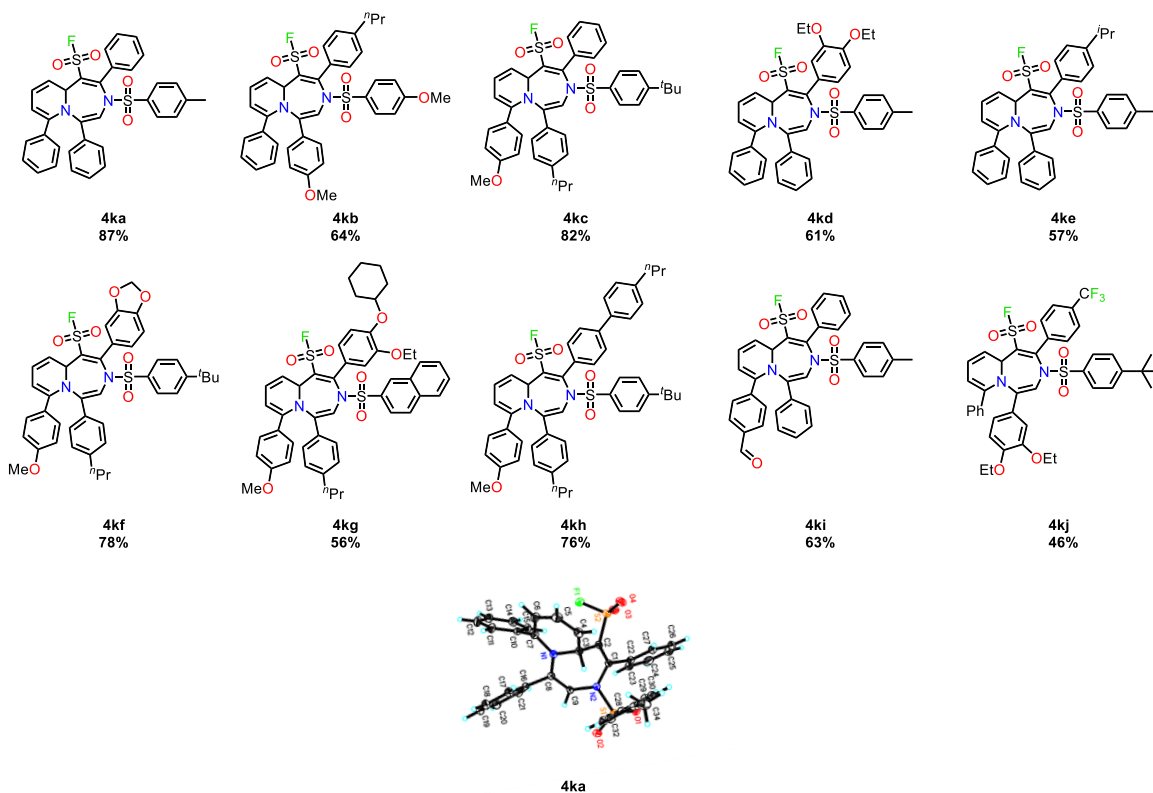
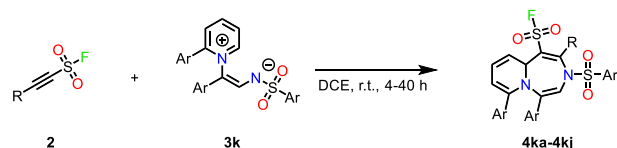
Following general procedure L, the title compound was isolated by flash column chromatography (0-60% EtOAc in petroleum ether) as a yellow foam (48.0 mg, 93%). **m.p.** 64 - 66 °C; **¹H NMR** (400 MHz, CDCl₃) δ 8.31 (appt. d, *J* = 8.7 Hz, 2H), 7.74 (appt. d, *J* = 8.7 Hz, 2H), 7.23 (dd, *J* = 8.4, 2.1 Hz, 1H), 7.12 (d, *J* = 2.1 Hz, 1H), 6.93 (d, *J* = 8.5 Hz, 1H), 5.48 (s, 1H), 4.88 – 4.82 (m, 1H), 3.88 (s, 3H), 3.40 (appt. td, *J* = 8.5, 3.7 Hz, 1H), 3.23 – 3.14 (m, 1H), 2.98

(ddd, $J = 18.6, 10.6, 8.2$ Hz, 1H), 2.88 (ddd, $J = 16.8, 7.7, 3.6$ Hz, 1H), 2.03 – 1.91 (m, 4H), 1.91 – 1.80 (m, 2H), 1.70 – 1.58 (m, 2H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 164.9, 151.8, 149.1, 148.9 (d, $J = 2.2$ Hz), 148.7, 143.7, 130.0, 124.4, 124.1, 114.1, 114.0, 112.8, 108.8 (d, $J = 28.4$ Hz), 80.7, 73.3, 56.3, 50.5, 36.1, 33.0 (d, $J = 2.8$ Hz), 24.3; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ 73.8; **HRMS** (ESI^+): calculated for $\text{C}_{24}\text{H}_{25}\text{FN}_3\text{O}_7\text{S}$ [$\text{M}+\text{H}^+$]: $m/z = 518.1392$, m/z found 518.1383; **IR** ν_{max} (ATR)/ cm^{-1} : 2963, 1742, 1503, 1345, 1189.

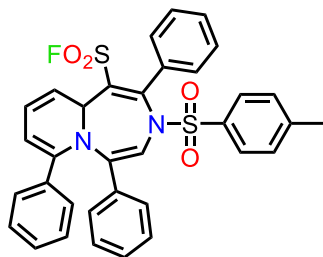
Synthesis and experimental data for compounds 4ka-4kj

General Procedure M

A solution of the required SASF **2** (100 μmol) and the required azomethine imine **3k** (100 μmol) in DCE (1.00 mL) was stirred at room temperature for 4 h. The solvent was then removed under reduced pressure and the crude product was purified by flash column chromatography to obtain the analytically pure product.

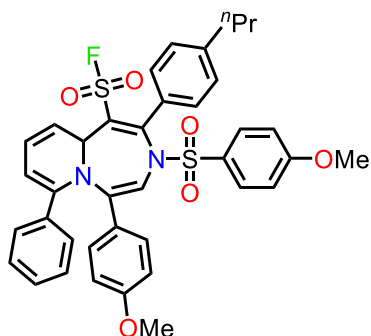


2,5,7-Triphenyl-3-tosyl-3,10a-dihydropyrido[1,2-d][1,4]diazepine-1-sulfonyl fluoride (4ka)



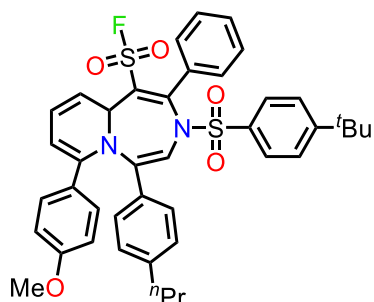
Following general procedure M, the title compound was isolated after purification by flash column chromatography (0-10% EtOAc in petroleum ether) as a purple solid (53.0 mg, 87%). **m.p.** 148 – 149 °C; **¹H NMR** (500 MHz, CDCl₃) δ = 7.58 – 7.53 (m, 3H), 7.47 – 7.41 (m, 2H), 7.41 – 7.36 (m, 2H), 7.33 – 7.27 (m, 2H), 7.07 – 6.98 (m, 3H), 6.98 – 6.87 (m, 5H), 6.82 (dt, *J* = 15.6, 7.0 Hz, 2H), 5.99 (s, 1H), 5.46 – 5.39 (m, 1H), 5.33 (dd, *J* = 5.9, 1.4 Hz, 1H), 5.23 (d, *J* = 5.6 Hz, 1H), 2.49 (s, 3H); **¹³C NMR** (126 MHz, CDCl₃) δ = 157.3 (d, *J* = 5.7 Hz), 145.4, 140.8, 138.8, 138.1, 137.6 (d, *J* = 25.6 Hz), 137.1, 134.8, 134.3, 131.7, 130.8, 130.1, 128.2, 128.0, 127.9, 127.8, 127.5, 127.4, 127.2, 126.5, 126.2, 114.4, 109.6, 106.2, 56.8, 21.9; **¹⁹F NMR** (376 MHz, CDCl₃) δ 63.5; **HRMS** (ESI⁺): calculated for C₃₄H₂₈FN₂O₄S₂ [M+H⁺]: *m/z* = 611.1469, *m/z* found 611.1462; **IR** *v*_{max} (ATR)/cm⁻¹: 2988, 2803, 1565, 1394, 1370, 1175, 1148, 750.

5-(4-Methoxyphenyl)-3-((4-methoxyphenyl)sulfonyl)-7-phenyl-2-(4-propylphenyl)-3,10a-dihydropyrido[1,2-d][1,4]diazepine-1-sulfonyl fluoride (4kb)



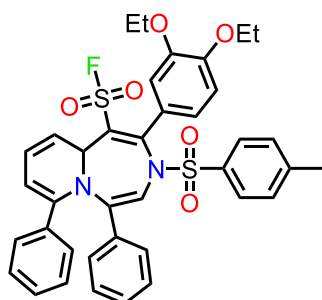
Following general procedure M, the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a purple solid (45.0 mg, 64%). **m.p.** 148 - 150 °C; **¹H NMR** (500 MHz, CDCl₃) δ = 7.46 – 7.40 (m, 4H), 7.24 (appt. d, *J* = 8.3 Hz, 2H), 6.98 – 6.90 (m, 5H), 6.85 – 6.78 (m, 4H), 6.54 (appt. d, *J* = 8.9 Hz, 2H), 6.25 (ddd, *J* = 9.4, 5.7, 1.5 Hz, 1H), 5.90 (s, 1H), 5.47 (ddd, *J* = 9.3, 6.0, 0.4 Hz, 1H), 5.39 (dd, *J* = 5.9, 1.3 Hz, 1H), 5.21 (d, *J* = 5.6 Hz, 1H), 3.91 (s, 3H), 3.71 (s, 3H), 2.71 – 2.65 (m, 2H), 1.76 – 1.68 (m, 2H), 1.01 (t, *J* = 7.3 Hz, 3H); **¹³C NMR** (126 MHz, CDCl₃) δ = 164.1, 159.2, 157.8 (d, *J* = 5.6 Hz), 147.0, 140.9, 139.1, 136.7, 136.8 – 136.5 (m), 132.3, 130.8, 130.7, 129.8, 129.1, 128.7, 128.3, 127.9, 127.2, 126.4, 126.0, 114.6, 114.6, 113.5, 108.8, 106.2, 56.8, 56.0, 55.4, 38.2, 24.4, 14.0; **¹⁹F NMR** (376 MHz, CDCl₃) δ = 63.4 **HRMS** (ESI⁺): calculated for C₃₈H₃₆FN₂O₆S₂ [M+H⁺]: *m/z* = 699.1993, *m/z* found 699.1995; **IR** *v*_{max} (ATR)/cm⁻¹: 3027, 2888, 1593, 1575, 1393, 1373, 1248, 1168.

3-((4-(*tert*-Butyl)phenyl)sulfonyl)-7-(4-methoxyphenyl)-2-phenyl-5-(4-propylphenyl)-3,10a-dihydropyrido[1,2-*d*][1,4]diazepine-1-sulfonyl fluoride (4kc)



Following general procedure M, the title compound was isolated after purification by flash column chromatography (0-10% EtOAc in petroleum ether) as a purple solid (59.0 mg, 82%). **m.p.** 116 - 117 °C; **¹H NMR** (500 MHz, CDCl₃) δ = 7.55 – 7.49 (m, 5H), 7.46 – 7.38 (m, 4H), 6.86 – 6.79 (m, 4H), 6.73 (appt. d, *J* = 8.5 Hz, 1H), 6.48 (appt. d, *J* = 8.8 Hz, 1H), 6.21 (ddd, *J* = 9.4, 5.7, 1.5 Hz, 1H), 5.31 (dd, *J* = 9.4, 5.9 Hz, 1H), 5.18 (dd, *J* = 5.9, 1.2 Hz, 1H), 5.14 (d, *J* = 5.6 Hz, 1H), 3.66 (s, 3H), 2.46 (t, *J* = 7.4 Hz, 2H), 1.59 – 1.51 (m, 2H), 1.39 (s, 9H), 0.87 (t, *J* = 7.4 Hz, 3H); **¹³C NMR** (126 MHz, CDCl₃) δ = 158.8, 158.4, 157.3 (d, *J* = 5.5 Hz), 142.2, 140.7, 137.6 (d, *J* = 26.0 Hz), 137.3, 135.6, 134.9, 134.1, 131.7, 131.6, 130.8, 128.1, 127.5, 127.3, 127.2, 126.5, 113.8, 113.4, 109.1, 105.2, 56.8, 55.3, 37.6, 35.5, 31.2, 24.6, 13.7; **¹⁹F NMR** (376 MHz, CDCl₃) δ 63.22; **HRMS** (ESI⁺): calculated for C₄₁H₄₂FN₂O₅S₂ [M+H⁺]: *m/z* = 725.2514, *m/z* found 725.2513; **IR** ν_{max} (ATR)/cm⁻¹: 2963, 2871, 1603, 1579, 1508, 1400, 1372, 1247, 1172.

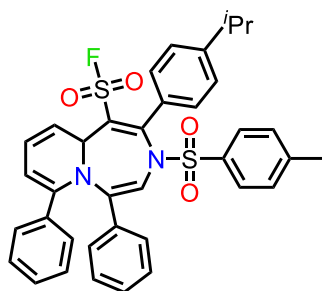
2-(3,4-Diethoxyphenyl)-5,7-diphenyl-3-tosyl-3,10a-dihydropyrido[1,2-*d*][1,4]diazepine-1-sulfonyl fluoride (4kd)



Following general procedure M (stirring for 20 h at room temperature), the title compound was isolated after purification by flash column chromatography (0-10% EtOAc in petroleum ether) as an orange solid (43.0 mg, 61%). **m.p.** 56 - 57 °C; **¹H NMR** (500 MHz, CDCl₃) δ = 7.41 (appt. d, *J* = 8.3 Hz, 2H), 7.31 (appt. d, *J* = 8.0 Hz, 2H), 7.09 (dd, *J* = 8.3, 2.1 Hz, 1H), 7.04 – 6.98 (m, 4H), 6.96 – 6.88 (m, 5H), 6.85 (d, *J* = 8.4 Hz, 1H), 6.83 – 6.78 (m, 2H), 6.24 (ddd, *J* = 9.5, 5.7, 1.6 Hz, 1H), 5.96 (s, 1H), 5.42 (dd, *J* = 9.2, 5.9 Hz, 1H), 5.24 (dd, *J* = 5.9, 1.5 Hz, 1H), 5.20 (d, *J* = 5.6 Hz, 1H), 4.21 – 4.06 (m, 4H), 2.49 (s, 3H), 1.52 (t, *J* = 7.0 Hz, 3H), 1.50 (t, *J* = 7.0 Hz, 3H); **¹³C NMR** (126 MHz, CDCl₃) δ = 157.6, 152.5, 148.2, 145.3, 140.8, 139.0, 138.3, 137.3, 135.2 (d, *J* = 25.8 Hz), 134.6, 130.0, 127.9,

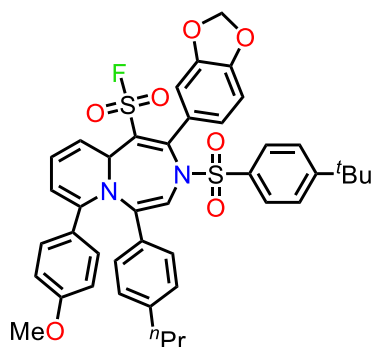
127.8, 127.7, 127.6, 127.5, 127.1, 126.8, 126.3, 126.3, 124.8, 116.1, 114.7, 111.8, 109.5, 106.0, 64.9, 64.6, 56.8, 21.9, 14.9, 14.9; ¹⁹F NMR (376 MHz, CDCl₃) δ = 63.1; HRMS (ESI⁺): calculated for C₃₈H₃₅FN₂O₆S₂ [M+H⁺]: m/z = 699.1993, m/z found 699.1991; IR ν_{max} (ATR)/cm⁻¹: 2924, 2857, 1595, 1565, 1509, 1403, 1374, 1273, 1171, 1052.

2-(4-Isopropylphenyl)-5,7-diphenyl-3-tosyl-3,10a-dihydropyrido[1,2-d][1,4]diazepine-1-sulfonyl fluoride (4ke)



Following general procedure M (stirring for 7 h at room temperature), the title compound was isolated after purification by flash column chromatography (0-6% EtOAc in petroleum ether) as an orange solid (37.0 mg, 57%). **m.p.** 153 - 155 °C; ¹H NMR (500 MHz, CDCl₃) δ = 7.43 (appt. d, J = 8.2 Hz, 2H), 7.35 (appt. d, J = 8.3 Hz, 2H), 7.28 - 7.24 (m, 4H), 7.04 - 6.99 (m, 3H), 6.97 - 6.87 (m, 5H), 6.84 - 6.79 (m, 2H), 6.26 (ddd, J = 9.3, 5.7, 1.4 Hz, 1H), 5.97 (s, 1H), 5.48 - 5.46 (ddd, J = 9.2, 6.0, 0.4 Hz, 1H), 5.41 (dd, J = 6.0, 1.2 Hz, 1H), 5.22 (d, J = 5.6 Hz, 1H), 3.05 - 2.95 (hept, J = 6.9 Hz, 1H), 2.48 (s, 3H), 1.32 (d, J = 6.9 Hz, 6H); ¹³C NMR (126 MHz, CDCl₃) δ = 157.5, 153.1, 145.2, 140.8, 138.9, 138.2, 137.0, 136.8 (d, J = 25.6 Hz), 134.7, 132.0, 130.9, 130.0, 128.0, 127.9, 127.7, 127.5, 127.4, 127.2, 126.4, 126.3, 126.3, 114.6, 109.7, 106.2, 56.9, 34.3, 23.9, 21.9; ¹⁹F NMR (376 MHz, CDCl₃) δ = 63.4; HRMS (ESI⁺): calculated for C₃₇H₃₄FN₂O₄S₂ [M+H⁺]: m/z = 653.1939, m/z found 653.1935; IR ν_{max} (ATR)/cm⁻¹: 2967, 1937, 1400, 1373, 1176, 1055, 1014.

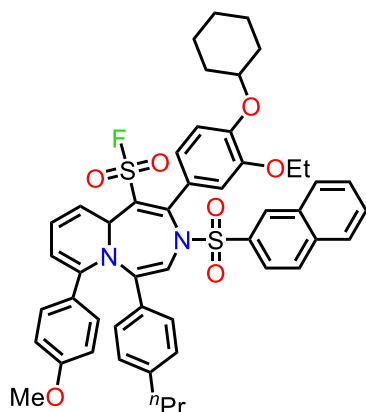
2-(Benzo[d][1,3]dioxol-5-yl)-3-((4-(tert-butyl)phenyl)sulfonyl)-7-(4-methoxyphenyl)-5-(4-propylphenyl)-3,10a-dihydropyrido[1,2-d][1,4]diazepine-1-sulfonyl fluoride (4kf)



Following general procedure M (stirring for 20 h at room temperature), the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a purple solid (60.0 mg, 78%).

m.p. 128 - 130 °C; $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.57 (appt. d, $J = 8.7$ Hz, 2H), 7.52 (appt. d, $J = 8.8$ Hz, 2H), 7.11 (dd, $J = 8.0, 1.8$ Hz, 1H), 6.97 (d, $J = 1.7$ Hz, 1H), 6.85 (d, $J = 8.0$ Hz, 1H), 6.84 – 6.77 (m, 4H), 6.71 (appt. d, $J = 8.5$ Hz, 2H), 6.47 (appt. d, $J = 8.8$ Hz, 2H), 6.17 (ddd, $J = 9.4, 5.7, 1.6$ Hz, 1H), 6.07 (dd, $J = 4.4, 1.5$ Hz, 2H), 5.98 (s, 1H), 5.24 (dd, $J = 9.4, 5.9$ Hz, 1H), 5.10 (d, $J = 5.6$ Hz, 1H), 4.93 (dd, $J = 5.9, 1.5$ Hz, 1H), 3.66 (s, 3H), 2.45 (t, $J = 7.4$ Hz, 2H), 1.59 – 1.49 (m, 2H), 1.40 (s, 9H), 0.87 (t, $J = 7.3$ Hz, 3H); $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 158.8, 158.6, 156.9 (d, $J = 5.3$ Hz), 151.1, 147.7, 142.2, 140.6, 137.7, 136.1 (d, $J = 25.9$ Hz), 135.7, 133.8, 131.8, 128.8, 128.1, 127.9, 127.4, 127.2, 126.5, 126.3, 113.9, 113.3, 110.8, 108.8, 108.2, 105.0, 102.0, 56.7, 55.3, 37.6, 35.6, 31.3, 24.6, 13.7; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ 62.8; **HRMS** (ESI $^+$): calculated for $\text{C}_{42}\text{H}_{42}\text{FN}_2\text{O}_7\text{S}_2$ [$\text{M}+\text{H}^+$]: $m/z = 769.2412$, m/z found 769.2404; **IR** ν_{max} (ATR)/ cm^{-1} : 2963, 2925, 2869, 1607, 1578, 1507, 1485, 1399, 1374, 1248, 1173, 1034, 781, 730.

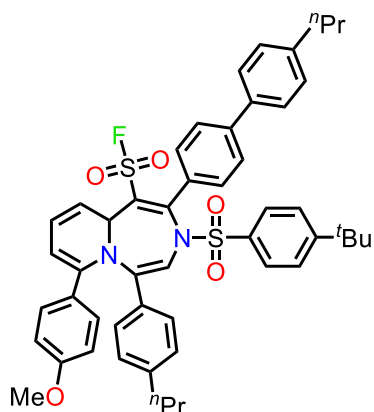
2-(4-(Cyclohexyloxy)-3-methoxyphenyl)-7-(4-methoxyphenyl)-3-(naphthalen-2-ylsulfonyl)-5-(4-propylphenyl)-3,10a-dihydropyrido[1,2-d][1,4]diazepine-1-sulfonyl fluoride (4kg)



Following general procedure M (stirring for 40 h at room temperature), the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a purple solid (49.0 mg, 58%).

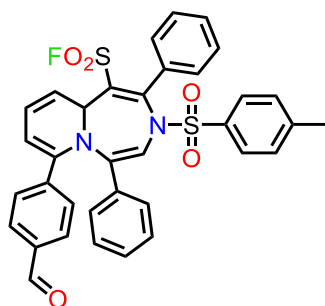
m.p. 112 - 113 °C; $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.96 – 7.90 (m, 3H), 7.85 – 7.82 (m, 1H), 7.73 – 7.69 (m, 1H), 7.66 – 7.62 (m, 1H), 7.54 (dd, $J = 8.7, 1.9$ Hz, 1H), 7.01 – 6.97 (m, 2H), 6.84 – 6.78 (m, 4H), 6.74 – 6.69 (m, 2H), 6.66 (d, $J = 8.4$ Hz, 1H), 6.48 – 6.42 (m, 2H), 6.16 (ddd, $J = 9.4, 5.7, 1.6$ Hz, 1H), 6.01 (s, 1H), 5.53 (dd, $J = 5.8, 1.3$ Hz, 1H), 5.35 (dd, $J = 9.4, 5.9$ Hz, 1H), 5.12 (d, $J = 5.6$ Hz, 1H), 4.18 – 4.07 (m, 1H), 3.80 (s, 3H), 3.65 (s, 3H), 2.46 (t, $J = 7.4$ Hz, 2H), 2.08 – 2.03 (m, 1H), 1.99 – 1.94 (m, 1H), 1.88 – 1.82 (m, 2H), 1.65 – 1.52 (m, 6H), 1.40 – 1.29 (m, 4H), 0.87 (t, $J = 7.3$ Hz, 3H); $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 158.7, 157.4 (d, $J = 4.5$ Hz), 151.1, 149.5, 142.2, 140.7, 137.3, 135.7, 135.4 – 135.2 (m), 135.1, 134.9, 132.0, 131.9, 129.7, 129.6, 129.5, 129.5, 128.1, 128.1, 127.5, 127.3, 126.5, 125.9, 124.9, 122.0, 115.0, 114.3, 113.3, 113.0, 109.1, 105.4, 57.2, 56.3, 55.3, 37.6, 32.1, 32.0, 25.6, 24.6, 24.3, 24.3, 13.7; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ 63.4; **HRMS** (ESI $^+$): calculated for $\text{C}_{48}\text{H}_{48}\text{FN}_2\text{O}_7\text{S}_2$ [$\text{M}+\text{H}^+$]: $m/z = 847.2881$, m/z found 847.2890; **IR** ν_{max} (ATR)/ cm^{-1} : 3056, 2933, 2854, 1602, 1505, 1400, 1270, 1247, 1169, 1143, 809, 748.

3-((4-(*tert*-Butyl)phenyl)sulfonyl)-7-(4-methoxyphenyl)-2-(4'-propyl-[1,1'-biphenyl]-4-yl)-5-(4-propylphenyl)-3,10a-dihydropyrido[1,2-*d*][1,4]diazepine-1-sulfonyl fluoride (4kh)



Following general procedure M (stirring for 16 h at room temperature), the title compound was isolated after purification by flash column chromatography (0-10% EtOAc in petroleum ether) as a purple solid (64.0 mg, 76%). **m.p.** 112 - 114 °C; **¹H NMR** (500 MHz, CDCl₃) δ 7.61 – 7.54 (m, 6H), 7.49 – 7.42 (m, 4H), 7.30 (appt. d, *J* = 8.1 Hz, 2H), 6.86 – 6.82 (m, 4H), 6.75 (appt. d, *J* = 8.4 Hz, 2H), 6.49 (appt. d, *J* = 8.7 Hz, 2H), 6.24 (ddd, *J* = 9.3, 5.7, 1.3 Hz, 1H), 6.03 (s, 1H), 5.38 (dd, *J* = 9.3, 6.0 Hz, 1H), 5.31 (dd, *J* = 5.9, 1.0 Hz, 1H), 5.16 (d, *J* = 5.6 Hz, 1H), 3.67 (s, 3H), 2.67 (t, *J* = 7.6 Hz, 2H), 2.47 (t, *J* = 7.4 Hz, 2H), 1.76 – 1.68 (m, 2H), 1.60 – 1.52 (m, 2H), 1.32 (s, 9H), 1.00 (t, *J* = 7.3 Hz, 3H), 0.88 (t, *J* = 7.3 Hz, 3H); **¹³C NMR** (126 MHz, CDCl₃) δ 158.8, 158.3, 157.1 (d, *J* = 5.4 Hz), 144.4, 142.9, 142.2, 140.7, 137.4, 137.2, 137.0 (d, *J* = 25.6 Hz), 135.7, 134.5, 133.1, 131.8, 131.4, 129.2, 128.1, 127.5, 127.2, 126.5, 126.4, 126.4, 113.9, 113.4, 109.3, 105.2, 56.9, 55.3, 37.8, 37.6, 35.4, 31.2, 24.6, 24.6, 13.9, 13.7; **¹⁹F NMR** (376 MHz, CDCl₃) δ 63.4; **HRMS** (ESI⁺): calculated for C₅₀H₅₂FN₂O₅S₂ [M+H⁺]: *m/z* = 843.3296, *m/z* found 843.3299; **IR** *v*_{max} (ATR)/cm⁻¹: 3063, 2888, 2815, 1637m 1580m 1508, 1401, 1247, 1198, 1084, 1039, 983.

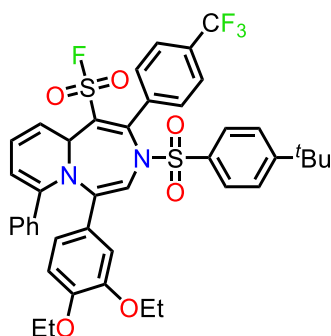
7-(4-Formylphenyl)-2,5-diphenyl-3-tosyl-3,10a-dihydropyrido[1,2-*d*][1,4]diazepine-1-sulfonyl fluoride (4ki)



Following general procedure M (50.0 μmol of the required SASF used, stirring for 20 h at room temperature), the title compound was isolated after purification by flash column chromatography (0-20% EtOAc in petroleum ether) as an orange solid (20.0 mg, 63%). **m.p.** 153 - 154 °C; **¹H NMR** (500 MHz, CDCl₃) δ 9.81 (s, 1H), 7.58 – 7.51 (m, 3H), 7.49 – 7.41 (m, 4H), 7.36 (appt. d, *J* = 8.4 Hz, 1H), 7.30 (appt. d, *J* = 8.0 Hz, 1H), 7.07 – 6.96 (m, 5H), 6.93 – 6.88 (m, 2H), 6.29 (ddd, *J* = 9.5, 5.7, 1.7 Hz, 1H), 6.01 (s, 1H), 5.52 (dd, *J* = 9.1, 5.9 Hz, 1H), 5.40 (dd, *J* = 5.9, 1.5

Hz, 1H), 5.31 (d, $J = 5.7$ Hz, 1H), 2.49 (s, 3H); $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 191.6, 157.7, 145.5, 144.8, 139.5, 137.6, 137.2 (d, $J = 25.2$ Hz), 136.6, 134.9, 134.4, 134.3, 131.9, 130.9, 130.2, 129.5, 128.3, 128.3, 128.2, 127.6, 127.6, 126.6, 126.2, 115.9, 110.0, 107.8, 56.7, 21.9; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ 63.9; **HRMS** (ESI⁺): calculated for $\text{C}_{35}\text{H}_{28}\text{FN}_2\text{O}_5\text{S}_2$ [$\text{M}+\text{H}^+$]: $m/z = 639.1418$, m/z found 639.1412; **IR** ν_{max} (ATR)/ cm^{-1} : 3052, 2926, 2865, 1696, 1602, 1404, 1356, 1198, 1160, 794, 747.

3-((4-(*tert*-butyl)Phenyl)sulfonyl)-5-(3,4-diethoxyphenyl)-7-phenyl-2-(4-(trifluoromethyl)phenyl)-3,10a-dihydropyrido[1,2-*d*][1,4]diazepine-1-sulfonyl fluoride (4kj)



Following general procedure M, the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a purple solid (37.0 mg, 46%). **m.p.** 146 - 148 °C; $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.61 (s, 4H), 7.49 (appt. d, $J = 8.7$ Hz, 2H), 7.38 (appt. d, $J = 8.7$ Hz, 2H), 7.01 – 6.96 (m, 3H), 6.85 – 6.80 (m, 2H), 6.53 (d, $J = 8.3$ Hz, 1H), 6.46 – 6.37 (m, 2H), 6.27 (ddd, $J = 9.0, 5.7, 1.0$ Hz, 1H), 5.99 (s, 1H), 5.45 – 5.36 (m, 2H), 5.24 (d, $J = 5.6$ Hz, 1H), 4.03 – 3.88 (m, 4H), 1.43 – 1.38 (m, 6H), 1.38 (s, 9H); $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 158.7, 155.4 (d, $J = 5.3$ Hz), 148.7, 148.4, 141.0, 139.1 (d, $J = 26.0$ Hz), 138.8, 137.9, 136.9, 134.4, 133.0 (q, $J = 32.7$ Hz), 131.2, 130.8, 127.9, 127.3, 127.0, 126.7, 126.6, 126.1, 125.0 (q, $J = 3.5$ Hz), 124.9 – 122.6 (m), 120.5, 114.1, 113.4, 113.3, 109.0, 106.3, 64.9, 64.8, 56.8, 35.5, 31.2, 15.0, 14.8; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ 64.0, -62.8; **HRMS** (ESI⁺): calculated for $\text{C}_{42}\text{H}_{41}\text{F}_4\text{N}_2\text{O}_6\text{S}_2$ [$\text{M}+\text{H}^+$]: $m/z = 809.2337$, m/z found 809.2338; **IR** ν_{max} (ATR)/ cm^{-1} : 3098, 2975, 2870, 1567, 1512, 1396, 1321, 1172, 1129, 1066, 844.

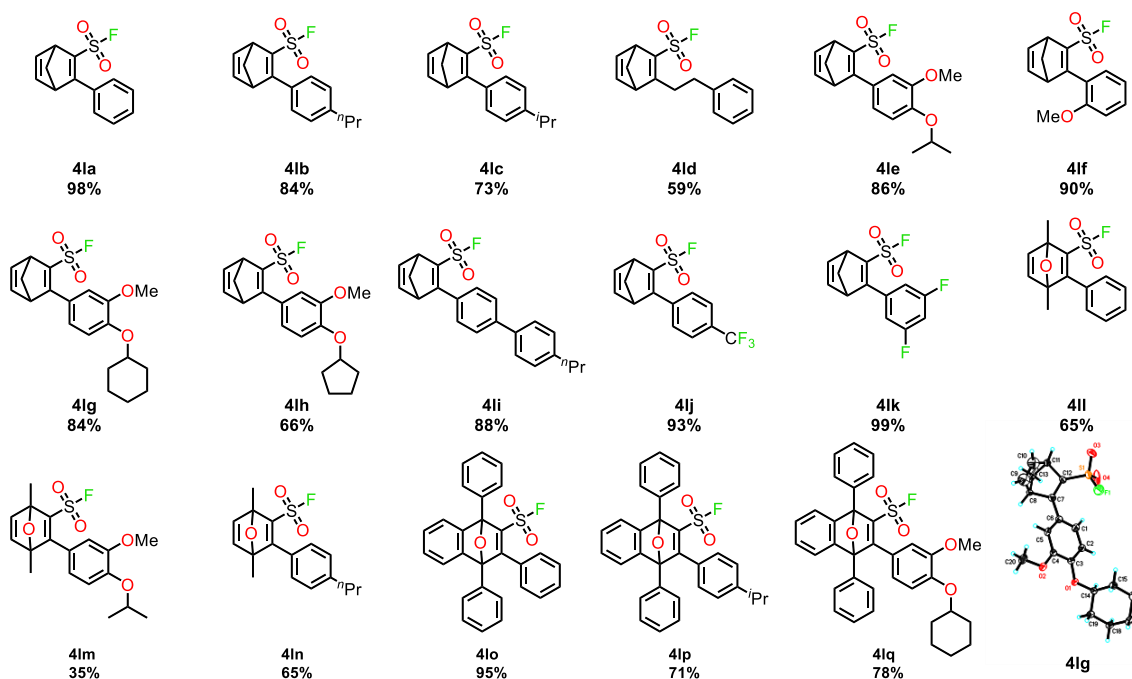
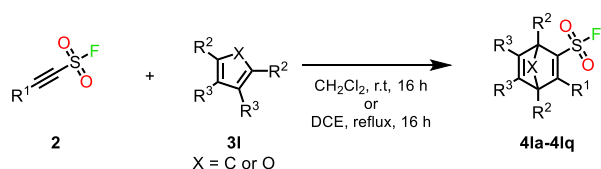
Synthesis and experimental data for compounds 41a-41q

General Procedure N

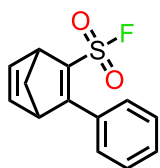
To a solution of the required diene **3I** (100 μ mol) in CH_2Cl_2 (500 μL) was added the required SASF **2** (100 μ mol) and stirred at room temperature for 2-16 h. The solvent was then removed under reduced pressure and the crude product was then purified by flash column chromatography to obtain the analytically pure product.

General Procedure O

To a solution of required diene **3I** (100 μ mol) in DCE (500 μL) was added the required SASF **2** (100 μ mol) and refluxed for 16 h. The solvent was then removed under reduced pressure and the crude product was then purified by flash column chromatography to obtain the analytically pure product.

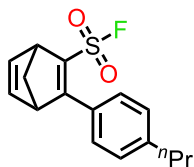


3-Phenylbicyclo[2.2.1]hepta-2,5-diene-2-sulfonyl fluoride (4la)



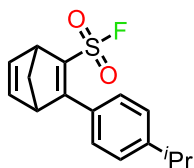
Following general procedure N (500 μmol of the required diene used), the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a colourless solid (24.0 mg, 98%). **m.p.** 48 - 49 $^{\circ}\text{C}$; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.51 – 7.37 (m, 5H), 7.13 (dd, $J = 4.9, 3.1$ Hz, 1H), 7.00 (dd, $J = 4.7, 3.5$ Hz, 1H), 4.28 – 4.13 (m, 1H), 4.11 – 3.99 (m, 1H), 2.49 (appt. dt, $J = 7.1, 1.5$ Hz, 1H), 2.30 – 2.15 (m, 1H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 172.1 (d, $J = 5.1$ Hz), 143.4, 140.6, 138.2 (d, $J = 27.9$ Hz), 132.3, 130.6, 128.4, 128.0 (d, $J = 1.1$ Hz), 71.2, 59.7 (d, $J = 1.4$ Hz), 54.4; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ 61.4; **HRMS** (ESI $^+$): calculated for $\text{C}_{13}\text{H}_{11}\text{FO}_2\text{SNa}$ [$\text{M}+\text{Na}^+$]: $m/z = 273.0356$, m/z found 273.0356; **IR** ν_{max} (ATR)/ cm^{-1} : 3069, 2959, 2881, 1578, 1395, 739.

3-(4-Propylphenyl)bicyclo[2.2.1]hepta-2,5-diene-2-sulfonyl fluoride (4lb)



Following general procedure N, the title compound was isolated after purification by flash column chromatography (0-20% EtOAc in petroleum ether) as a colourless solid (29.0 mg, 86%). **m.p.** 48 - 50 $^{\circ}\text{C}$; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.42 (appt. d, $J = 8.3$ Hz, 2H), 7.25 (appt. d, $J = 8.4$ Hz, 2H), 7.11 (dd, $J = 4.9, 3.0$ Hz, 1H), 6.98 (dd, $J = 4.7, 3.4$ Hz, 1H), 4.21 – 4.16 (m, 1H), 4.08 – 4.02 (m, 1H), 2.69 – 2.59 (m, 2H), 2.50 – 2.42 (m, 1H), 2.26 – 2.15 (m, 1H), 1.75 – 1.60 (m, 2H), 0.97 (t, $J = 7.3$ Hz, 3H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 171.0 (d, $J = 5.1$ Hz), 144.7, 142.1, 139.3 (d, $J = 1.0$ Hz), 135.7 (d, $J = 27.5$ Hz), 127.3, 127.1 (d, $J = 1.2$ Hz), 69.7 (d, $J = 0.9$ Hz), 58.5 (d, $J = 1.4$ Hz), 53.3, 36.9, 23.2, 12.8; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ 61.7; **HRMS** (ESI $^+$): calculated for $\text{C}_{16}\text{H}_{17}\text{FO}_2\text{SNa}$ [$\text{M}+\text{Na}^+$]: $m/z = 315.0826$, m/z found 315.0826; **IR** ν_{max} (ATR)/ cm^{-1} : 3077, 2960, 2873, 2188, 1398, 1209, 1183, 745.

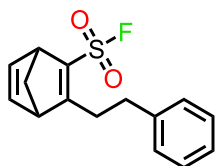
3-(4-Isopropylphenyl)bicyclo[2.2.1]hepta-2,5-diene-2-sulfonyl fluoride (4lc)



Following general procedure N, the title compound was isolated after purification by flash column chromatography (0-10% EtOAc in petroleum ether) as a yellow oil (21.0 mg, 73%); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.44 (appt. d, $J = 8.3$ Hz, 2H), 7.30 (appt. d, $J = 8.4$ Hz, 2H), 7.11 (dd, $J = 4.9, 3.0$ Hz, 1H), 6.98 (dd, $J = 4.7, 3.3$ Hz,

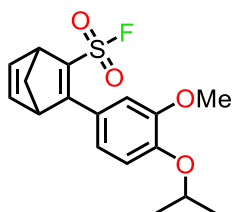
1H), 4.21 – 4.14 (m, 1H), 4.09 – 4.02 (m, 1H), 2.95 (hept, $J = 7.0$ Hz, 1H), 2.53 – 2.38 (m, 1H), 2.24 – 2.14 (m, 1H), 1.28 (d, $J = 6.9$ Hz, 6H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 172.1 (d, $J = 5.0$ Hz), 152.0, 143.3, 140.5, 136.9 (d, $J = 27.5$ Hz), 129.7, 128.4 (d, $J = 0.9$ Hz), 126.5, 70.9, 59.6 (d, $J = 1.2$ Hz), 54.4, 34.2, 23.8 (d, $J = 2.6$ Hz); $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ 61.6; **HRMS** (ESI⁺): calculated for $\text{C}_{16}\text{H}_{17}\text{FO}_2\text{SNa}$ [$\text{M}+\text{Na}^+$]: $m/z = 315.0826$, m/z found 315.0829; **IR** ν_{max} (ATR)/ cm^{-1} : 3075, 2962, 2873, 1398, 1208, 757.

3-Phenethylbicyclo[2.2.1]hepta-2,5-diene-2-sulfonyl fluoride (4ld)



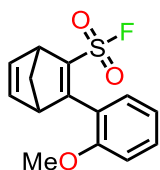
Following general procedure N, the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a yellow oil (16.0 mg, 59%); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.39 – 7.17 (m, 5H), 6.97 (dd, $J = 4.9, 3.0$ Hz, 1H), 6.70 – 6.62 (m, 1H), 4.00 – 3.90 (m, 1H), 3.72 – 3.62 (m, 1H), 3.14 – 2.93 (m, 2H), 2.91 – 2.71 (m, 2H), 2.20 – 2.12 (m, 1H), 2.11 – 1.99 (m, 1H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 177.5 (d, $J = 5.7$ Hz), 143.2, 140.5 (d, $J = 1.4$ Hz), 139.9, 139.1 (d, $J = 27.6$ Hz), 128.6, 128.5, 126.6, 71.7 (d, $J = 1.0$ Hz), 57.2 (d, $J = 1.4$ Hz), 52.6, 33.1 (d, $J = 1.3$ Hz), 31.3; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ 60.7; **HRMS** (ESI⁺): calculated for $\text{C}_{15}\text{H}_{15}\text{FO}_2\text{SNa}$ [$\text{M}+\text{Na}^+$]: $m/z = 301.0669$, m/z found 301.0674; **IR** ν_{max} (ATR)/ cm^{-1} : 3065, 3028, 2968, 2930, 2873, 2213, 1612, 1395, 1208, 748.

3-(4-Isopropoxy-3-methoxyphenyl)bicyclo[2.2.1]hepta-2,5-diene-2-sulfonyl fluoride (4le)



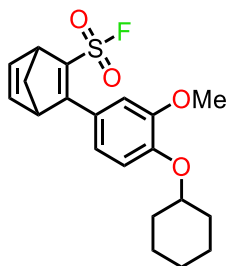
Following general procedure N, the title compound was isolated after purification by flash column chromatography (0-20% EtOAc in petroleum ether) as a colourless solid (29.0 mg, 86%). **m.p.** 48 - 50 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.18 (d, $J = 2.1$ Hz, 1H), 7.12 – 7.06 (m, 2H), 6.95 (dd, $J = 4.5, 3.5$ Hz, 1H), 6.91 (d, $J = 8.5$ Hz, 1H), 4.63 (hept, $J = 6.1$ Hz, 1H), 4.20 – 4.13 (m, 1H), 4.12 – 4.04 (m, 1H), 3.89 (s, 3H), 2.44 – 2.38 (m, 1H), 2.21 – 2.13 (m, 1H), 1.41 (d, $J = 6.0$ Hz, 3H), 1.14 (d, $J = 6.1$ Hz, 6H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 171.6 (d, $J = 4.8$ Hz), 149.8 (d, $J = 34.0$ Hz), 143.3, 140.1, 135.0 (d, $J = 26.6$ Hz), 128.4, 124.6, 121.8, 115.7, 113.8, 112.9 (d, $J = 1.3$ Hz), 71.4, 70.3 (d, $J = 1.1$ Hz), 59.34 (d, $J = 1.3$ Hz), 56.2, 54.6, 22.1 (d, $J = 1.7$ Hz); $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ 62.1; **HRMS** (ESI⁺): calculated for $\text{C}_{17}\text{H}_{19}\text{FO}_4\text{SNa}$ [$\text{M}+\text{Na}^+$]: $m/z = 361.0880$, m/z found 361.0882; **IR** ν_{max} (ATR)/ cm^{-1} : 3074, 2978, 2939, 2172, 1597, 1504, 744.

3-(2-Methoxyphenyl)bicyclo[2.2.1]hepta-2,5-diene-2-sulfonyl fluoride (4f)



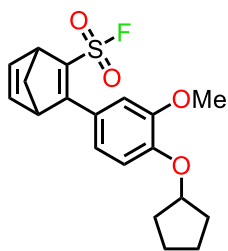
Following general procedure N, the title compound was isolated after purification by flash column chromatography (0-10% EtOAc in petroleum ether) as a yellow oil (25.0 mg, 90%); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.42 – 7.35 (m, 1H), 7.20 (dd, J = 7.6, 1.7 Hz, 1H), 7.08 (dd, J = 4.9, 3.0 Hz, 1H), 6.99 (td, J = 7.5, 0.9 Hz, 1H), 6.96 – 6.90 (m, 2H), 4.13 (d, J = 2.1 Hz, 1H), 4.01 (s, 1H), 3.85 (s, 3H), 2.52 – 2.46 (m, 1H), 2.20 (d, J = 6.9 Hz, 1H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 170.7 (d, J = 5.4 Hz), 156.9, 142.5, 141.7, 139.7 (d, J = 28.6 Hz), 131.6, 129.5, 122.1, 120.3, 111.0, 71.2, 59.5 (d, J = 1.2 Hz), 55.5, 53.3; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ 59.5; **HRMS** (ESI^+): calculated for $\text{C}_{14}\text{H}_{13}\text{FO}_3\text{SNa}$ [$\text{M}+\text{Na}^+$]: m/z = 303.0462, m/z found 303.0460; **IR** ν_{max} (ATR)/ cm^{-1} : 3074, 3002, 2943, 2842, 1592, 1395, 1207, 1188, 743.

3-(4-(Cyclohexyloxy)-3-methoxyphenyl)bicyclo[2.2.1]hepta-2,5-diene-2-sulfonyl fluoride (4g)



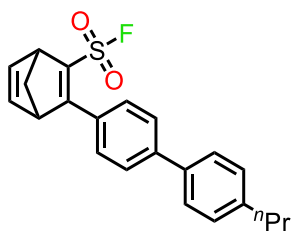
Following general procedure N, the title compound was isolated after purification by flash column chromatography (0-20% EtOAc in petroleum ether) as a colourless solid (32.0 mg, 84%). **m.p.** 99 - 100 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.18 (d, J = 2.1 Hz, 1H), 7.11 – 7.05 (m, 2H), 6.97 – 6.89 (m, 2H), 4.35 – 4.25 (m, 1H), 4.20 – 4.15 (m, 1H), 4.11 – 4.05 (m, 1H), 3.89 (s, 3H), 2.46 – 2.38 (m, 1H), 2.21 – 2.15 (m, 1H), 2.10 – 1.99 (m, 2H), 1.90 – 1.78 (m, 2H), 1.67 – 1.53 (m, 3H), 1.44 – 1.21 (m, 3H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 171.6 (d, J = 4.8 Hz), 149.9, 149.8, 143.3, 140.1, 134.9 (d, J = 26.6 Hz), 124.6, 121.8, 114.2, 113.0, 77.1, 70.3, 59.3, 56.3, 54.6, 31.9 (d, J = 1.5 Hz), 25.6, 24.1; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ 62.2; **HRMS** (ESI^+): calculated for $\text{C}_{20}\text{H}_{23}\text{FO}_4\text{SNa}$ [$\text{M}+\text{Na}^+$]: m/z = 401.1193, m/z found 401.1188; **IR** ν_{max} (ATR)/ cm^{-1} : 3003, 2933, 2859, 1595, 1508, 1396, 723.

3-(4-(Cyclopentyloxy)-3-methoxyphenyl)bicyclo[2.2.1]hepta-2,5-diene-2-sulfonyl fluoride (4lh)



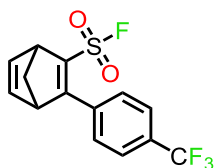
Following general procedure N, the title compound was isolated after purification by flash column chromatography (0-20% EtOAc in petroleum ether) as a colourless solid (24.0 mg, 66%). **m.p.** 62 - 63 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.17 (d, *J* = 2.1 Hz, 1H), 7.12 – 7.06 (m, 2H), 6.94 (dd, *J* = 4.6, 3.5 Hz, 1H), 6.90 (d, *J* = 8.5 Hz, 1H), 4.83 (tt, *J* = 6.3, 3.2 Hz, 1H), 4.21 – 4.14 (m, 1H), 4.12 – 4.04 (m, 1H), 3.88 (s, 3H), 2.46 – 2.38 (m, 1H), 2.21 – 2.15 (m, 1H), 2.05 – 1.78 (m, 6H), 1.71 – 1.58 (m, 2H); **¹³C NMR** (101 MHz, CDCl₃) δ 171.6 (d, *J* = 4.8 Hz), 150.4, 149.4, 143.3, 140.1, 134.8 (d, *J* = 26.5 Hz), 124.3, 121.8, 113.4, 112.9, 80.6, 70.3, 59.3 (d, *J* = 1.3 Hz), 56.3, 54.6, 33.0 (d, *J* = 3.0 Hz), 24.2; **¹⁹F NMR** (376 MHz, CDCl₃) δ 62.2; **HRMS** (ESI⁺): calculated for C₁₉H₂₁FO₄SNa [M+Na⁺]: *m/z* = 387.1037, *m/z* found 387.1030; **IR** *v*_{max} (ATR)/cm⁻¹: 2958, 2875, 2361, 1598, 1394, 728.

3-(4'-Propyl-[1,1'-biphenyl]-4-yl)bicyclo[2.2.1]hepta-2,5-diene-2-sulfonyl fluoride (4li)



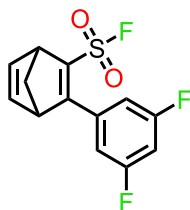
Following general procedure N, the title compound was isolated after purification by flash column chromatography (0-10% EtOAc in petroleum ether) as a colourless solid (32.0 mg, 88%). **m.p.** 80 - 82 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.69 – 7.63 (m, 2H), 7.60 – 7.51 (m, 4H), 7.31 – 7.25 (m, 2H), 7.14 (dd, *J* = 4.9, 3.0 Hz, 1H), 7.01 (dd, *J* = 4.8, 3.3 Hz, 1H), 4.25 – 4.17 (m, 1H), 4.15 – 4.04 (m, 1H), 2.71 – 2.59 (m, 2H), 2.54 – 2.43 (m, 1H), 2.29 – 2.18 (m, 1H), 1.78 – 1.62 (m, 2H), 1.00 (t, *J* = 7.3 Hz, 3H); **¹³C NMR** (101 MHz, CDCl₃) δ 171.7 (d, *J* = 5.0 Hz), 143.4, 143.3, 142.8, 140.5, 137.6 (d, *J* = 27.7 Hz), 137.4, 130.7, 129.2, 128.8 (d, *J* = 1.0 Hz), 127.0, 126.8, 71.0, 59.6 (d, *J* = 1.2 Hz), 54.5, 37.8, 24.6, 14.0; **¹⁹F NMR** (376 MHz, CDCl₃) δ 61.6; **HRMS** (ESI⁺): calculated for C₂₂H₂₁FO₂SNa [M+Na⁺]: *m/z* = 391.1138, *m/z* found 391.1139; **IR** *v*_{max} (ATR)/cm⁻¹: 2955, 2867, 1581, 1390, 1180, 1055, 746.

3-(4-(Trifluoromethyl)phenyl)bicyclo[2.2.1]hepta-2,5-diene-2-sulfonyl fluoride (4lj)



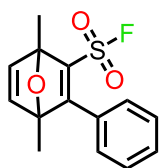
Following general procedure N, the title compound was isolated after purification by flash column chromatography (0-10% EtOAc in petroleum ether) as a colourless solid (30.0 mg, 93%). **m.p.** 55 - 56 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.69 (appt. d, *J* = 8.3 Hz, 2H), 7.54 (appt. d, *J* = 8.3 Hz, 2H), 7.16 (dd, *J* = 4.8, 3.2 Hz, 1H), 7.03 (dd, *J* = 4.6, 3.4 Hz, 1H), 4.29 – 4.08 (m, 1H), 4.08 – 3.94 (m, 1H), 2.59 – 2.43 (m, 1H), 2.30 – 2.10 (m, 1H); **¹³C NMR** (101 MHz, CDCl₃) δ 170.2 (d, *J* = 5.3 Hz), 143.5, 140.9 (d, *J* = 28.7 Hz), 140.6, 135.8, 132.1 (q, *J* = 32.9 Hz), 128.2 (d, *J* = 1.0 Hz), 123.8 (q, *J* = 272.4 Hz), 125.4 (q, *J* = 3.8 Hz), 71.8, 59.8, 54.5; **¹⁹F NMR** (376 MHz, CDCl₃) δ 61.1, -63.0; **HRMS** (ASAP): calculated for C₁₄H₁₁F₄O₂S [M+H]: *m/z* = 319.0416, *m/z* found 319.0412; **IR** *v*_{max} (ATR)/cm⁻¹: 2967, 2642, 2579, 1603, 1393, 1322, 1130, 764.

3-(3,5-Difluorophenyl)bicyclo[2.2.1]hepta-2,5-diene-2-sulfonyl fluoride (4lk)



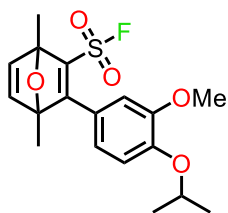
Following general procedure N, the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a colourless solid (28.0 mg, 99%). **m.p.** 56 - 58 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.15 (dd, *J* = 4.7, 3.3 Hz, 1H), 7.05 – 7.00 (m, 1H), 7.00 – 6.92 (m, 2H), 6.92 – 6.82 (m, 1H), 4.27 – 4.15 (m, 1H), 4.04 – 3.90 (m, 1H), 2.56 – 2.42 (m, 1H), 2.33 – 2.20 (m, 1H); **¹³C NMR** (101 MHz, CDCl₃) δ 169.1 (d, *J* = 5.1 Hz), 162.7 (dd, *J* = 250.1, 12.8 Hz), 143.5, 141.2 (d, *J* = 28.6 Hz), 140.6 (d, *J* = 1.1 Hz), 135.2 (t, *J* = 10.0 Hz), 111.1 (dd, *J* = 26.8, 1.2 Hz), 111.1 (dd, *J* = 11.5, 1.1 Hz), 105.7 (t, *J* = 25.2 Hz), 71.7, 59.7 (d, *J* = 1.4 Hz), 54.6; **¹⁹F NMR** (376 MHz, CDCl₃) δ 61.4, -108.6 (t, *J* = 7.6 Hz); **HRMS** (ASAP): calculated for C₁₃H₁₀F₃O₂S [M+H]: *m/z* = 287.0354, *m/z* found 287.0350; **IR** *v*_{max} (ATR)/cm⁻¹: 3094, 2987, 2947, 2878, 1593, 1397, 1204, 737.

1,4-Dimethyl-3-phenyl-7-oxabicyclo[2.2.1]hepta-2,5-diene-2-sulfonyl fluoride (4II)



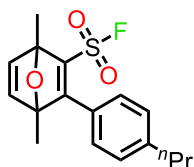
Following general procedure O, the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a yellow oil (26.0 mg, 95%); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.50 – 7.42 (m, 3H), 7.21 – 7.13 (m, 3H), 7.03 (d, J = 5.1 Hz, 1H), 1.98 (s, 3H), 1.64 (s, 3H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 175.7 (d, J = 4.8 Hz), 148.9, 145.3, 143.8 (d, J = 31.3 Hz), 130.3, 130.0, 128.5, 126.6, 95.1, 92.5, 15.9, 15.7; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ 65.8; **HRMS** (ESI⁺): calculated for $\text{C}_{14}\text{H}_{13}\text{FO}_3\text{SNa}$ [$\text{M}+\text{Na}^+$]: m/z = 303.0461, m/z found 303.0463; **IR** ν_{max} (ATR)/ cm^{-1} : 2987, 2939, 1619, 1403, 1196, 747.

3-(4-Isopropoxy-3-methoxyphenyl)-1,4-dimethyl-7-oxabicyclo[2.2.1]hepta-2,5-diene-2-sulfonyl fluoride (4Im)



Following general procedure O, the title compound was isolated after purification by flash column chromatography (0-20% EtOAc in petroleum ether) as a yellow oil (13.0 mg, 35%); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.15 (d, J = 5.1 Hz, 1H), 7.00 (d, J = 5.1 Hz, 1H), 6.92 (d, J = 8.3 Hz, 1H), 6.78 – 6.69 (m, 2H), 4.68 – 4.54 (m, 1H), 3.87 (s, 3H), 1.97 (s, 3H), 1.69 (s, 3H), 1.41 (dd, J = 6.0, 2.0 Hz, 6H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 175.7 (d, J = 4.7 Hz), 149.7 (d, J = 3.6 Hz), 148.6, 144.9, 141.3 (d, J = 30.5 Hz), 121.9, 120.0, 113.8, 111.9, 95.0, 92.3, 71.4, 56.3, 22.1 (d, J = 2.5 Hz), 16.3, 15.9; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ 66.6; **HRMS** (ASAP): calculated for $\text{C}_{18}\text{H}_{22}\text{FO}_5\text{S}$ [$\text{M}+\text{H}$]: m/z = 369.1172, m/z found 369.1169; **IR** ν_{max} (ATR)/ cm^{-1} : 2979, 2934, 1674, 1412, 1265, 787.

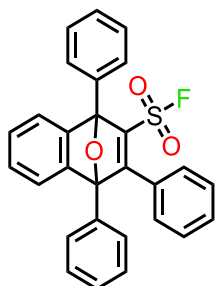
1,4-Dimethyl-3-(4-propylphenyl)-7-oxabicyclo[2.2.1]hepta-2,5-diene-2-sulfonyl fluoride (4In)



Following general procedure O, the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a yellow oil (25.0 mg, 78%); $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.28 – 7.23 (m, 2H), 7.17 (appt. d, J = 5.1 Hz, 1H), 7.12 – 7.07 (m, 2H), 7.02 (appt. d, J = 5.1 Hz, 1H), 2.68 – 2.58

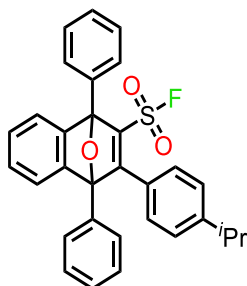
(m, 2H), 2.01 – 1.93 (m, 3H), 1.74 – 1.66 (m, 2H), 1.65 (s, 3H), 0.97 (t, $J = 7.3$ Hz, 3H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 175.8 (d, $J = 4.9$ Hz), 148.6, 145.4, 145.0, 142.5 (d, $J = 30.9$ Hz), 128.4, 127.0, 126.6, 94.9, 92.2, 37.9, 24.1, 15.9, 15.6, 13.8; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ 66.1; **HRMS** (ESI⁺): calculated for $\text{C}_{17}\text{H}_{19}\text{FO}_3\text{SNa}$ [$\text{M}+\text{Na}^+$]: $m/z = 345.0931$, m/z found 345.0932; **IR** ν_{max} (ATR)/ cm^{-1} : 3028, 2961, 2935, 2872, 1613, 1404, 1197, 759.

1,3,4-Triphenyl-1,4-dihydro-1,4-epoxynaphthalene-2-sulfonyl fluoride (4lo)



Following general procedure N (0.30 mmol of the required diene used), the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a colourless solid (43.0 mg, 95%). **m.p.** 69 - 70 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.97 (d, $J = 7.6$ Hz, 2H), 7.76 – 7.68 (m, 1H), 7.59 – 7.53 (m, 1H), 7.53 – 7.40 (m, 3H), 7.38 (dd, $J = 5.8, 2.2$ Hz, 2H), 7.34 – 7.14 (m, 8H), 7.01 (d, $J = 7.1$ Hz, 2H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 173.3 (d, $J = 2.9$ Hz), 149.3, 147.7, 145.1 (d, $J = 26.5$ Hz), 132.6, 132.0, 130.5, 130.4, 129.6, 129.2, 128.9, 128.8, 128.5, 128.3, 128.2, 127.6, 126.9, 126.4, 122.7 (d, $J = 8.2$ Hz), 96.3, 94.1; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ 67.4; **HRMS** (ESI⁺): calculated for $\text{C}_{28}\text{H}_{19}\text{FO}_3\text{SK}$ [$\text{M}+\text{K}^+$]: $m/z = 493.0670$, m/z found 493.0676; **IR** ν_{max} (ATR)/ cm^{-1} : 3062, 2932, 2601, 2359, 1590, 1406, 1199, 748.

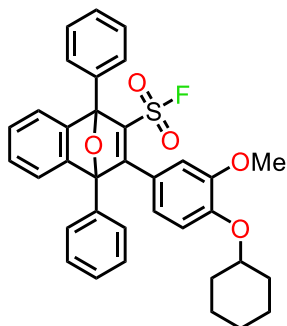
3-(4-Isopropylphenyl)-1,4-diphenyl-1,4-dihydro-1,4-epoxynaphthalene-2-sulfonyl fluoride (4lp)



Following general procedure N, the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a colourless solid (35.0 mg, 71%). **m.p.** 116 - 118 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.07 – 8.00 (m, 2H), 7.81 – 7.75 (m, 1H), 7.69 – 7.63 (m, 1H), 7.59 – 7.44 (m, 5H), 7.41 – 7.31 (m, 3H), 7.30 – 7.24 (m, 2H), 7.18 (appt. d, $J = 8.2$ Hz, 2H), 7.08 (appt. d, $J = 8.3$ Hz, 2H), 2.96 – 2.83 (m, 1H), 1.23 (dd, $J = 6.9, 1.6$ Hz, 6H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 173.3 (d, $J = 2.7$ Hz), 151.8, 149.4, 147.7, 143.9 (d, $J = 25.7$ Hz), 132.8, 132.2, 129.6, 129.4, 129.3, 128.8, 128.5, 128.4, 127.9, 127.7, 126.9, 126.4, 126.3, 122.9, 96.5,

94.4, 34.1, 23.7 (d, $J = 7.9$ Hz); ^{19}F NMR (376 MHz, CDCl_3) δ 67.5; HRMS (ESI $^+$): calculated for $\text{C}_{31}\text{H}_{25}\text{FO}_3\text{SNa}$ [$\text{M}+\text{Na}^+$]: $m/z = 519.1400$, m/z found 519.1402; IR ν_{max} (ATR)/ cm^{-1} : 3063, 2963, 2869, 2361, 1400, 1198, 996, 748.

3-(4-(Cyclohexyloxy)-3-methoxyphenyl)-1,4-diphenyl-1,4-dihydro-1,4-epoxynaphthalene-2-sulfonyl fluoride (4lq)

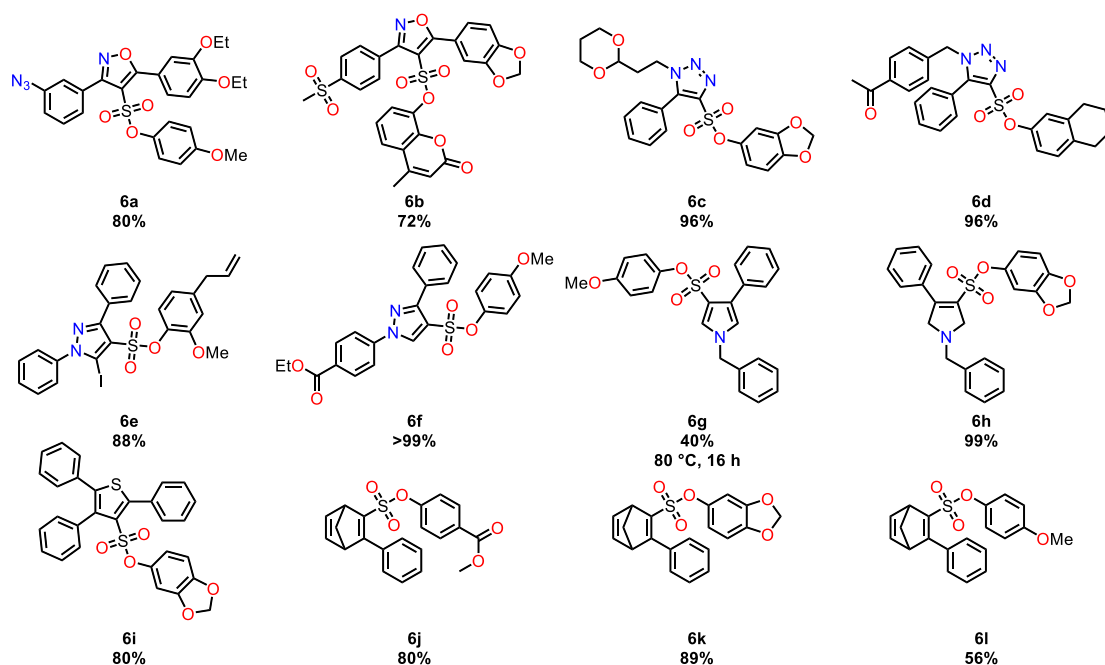
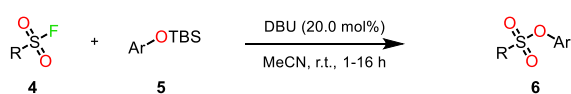


Following general procedure N, the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a colourless solid (45.0 mg, 78%). **m.p.** 87 - 88 °C; ^1H NMR (400 MHz, CDCl_3) δ 8.05 – 7.98 (m, 2H), 7.80 – 7.74 (m, 1H), 7.69 – 7.65 (m, 1H), 7.59 – 7.47 (m, 5H), 7.42 – 7.33 (m, 3H), 7.31 – 7.23 (m, 2H), 6.84 – 6.70 (m, 3H), 4.31 – 4.11 (m, 1H), 3.68 (s, 3H), 2.08 – 1.96 (m, 2H), 1.89 – 1.77 (m, 2H), 1.66 – 1.48 (m, 3H), 1.41 – 1.20 (m, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 172.8, 149.7, 149.5, 149.4, 147.6, 141.9 (d, $J = 24.4$ Hz), 133.0, 132.4, 129.7, 129.6, 129.3, 128.8, 128.6, 128.6, 126.9, 126.2, 113.9, 112.4, 96.5, 94.6, 77.1, 56.0, 31.9 (d, $J = 2.2$ Hz), 25.6, 24.1; ^{19}F NMR (376 MHz, CDCl_3) δ 67.2; HRMS (ESI $^+$): calculated for $\text{C}_{35}\text{H}_{31}\text{FO}_5\text{SNa}$ [$\text{M}+\text{Na}^+$]: $m/z = 605.1768$, m/z found 605.1768; IR ν_{max} (ATR)/ cm^{-1} : 3157, 2936, 2861, 1503, 1402, 1004, 751.

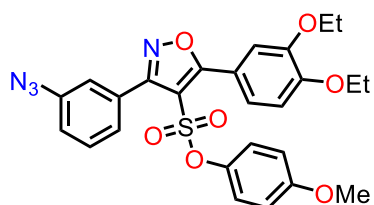
Synthesis and experimental data for compounds 6a-6l

General Procedure P

To a solution of the required sulfonyl fluoride **4** (100 μ mol) and the required aryl silyl-ether **5** (100 μ mol) in a solution of MeCN (400 μ L) was added DBU (3.00 μ L, 20.0 μ mol). The reaction mixture was stirred at room temperature for 1 h. The reaction mixture was extracted into EtOAc (5.00 mL), then washed with brine (10.0 mL) and H₂O (10.0 mL). The aqueous layer was then extracted with EtOAc (5.00 mL), the organic fractions combined, dried over anhydrous MgSO₄, filtered under vacuum and the solvent was removed under reduced pressure to obtain the analytically pure product.



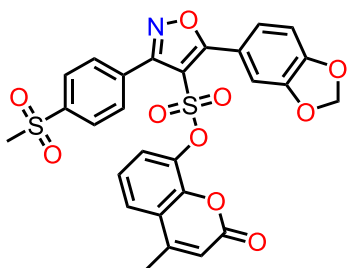
4-Methoxyphenyl 3-(3-azidophenyl)-5-(3,4-diethoxyphenyl)isoxazole-4-sulfonate (**6a**)



Following general procedure P, the title compound was isolated after purification by flash column chromatography (0-15% EtOAc in petroleum ether) as a brown solid (43.0 mg, 80%). **m.p.** 87 - 88 °C; **¹H NMR** (500 MHz, CDCl₃) δ 7.53 (dd, J = 8.5, 2.2 Hz, 1H), 7.49 (d, J = 2.1 Hz, 1H), 7.41 (appt. t, J = 7.9 Hz, 1H), 7.25 (ddd,

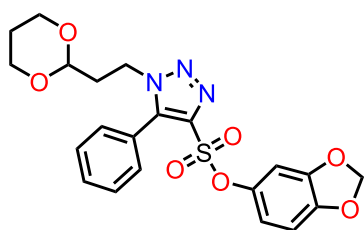
$J = 7.7, 1.5, 1.0$ Hz, 1H), 7.15 (ddd, $J = 8.1, 2.3, 1.0$ Hz, 1H), 7.08 – 7.06 (m, 1H), 6.98 (d, $J = 8.5$ Hz, 1H), 6.90 (appt. d, $J = 9.2$ Hz, 2H), 6.80 (appt. d, $J = 9.2$ Hz, 2H), 4.20 (q, $J = 7.0$ Hz, 2H), 4.13 (q, $J = 7.0$ Hz, 2H), 3.79 (s, 3H), 1.52 (t, $J = 6.4$ Hz, 3H), 1.49 (t, $J = 6.4$ Hz, 3H); ^{13}C NMR (126 MHz, CDCl_3) δ 173.8, 161.8, 158.8, 152.6, 148.5, 142.5, 140.3, 129.7, 128.7, 126.1, 123.5, 123.3, 121.0, 120.2, 117.1, 114.9, 113.9, 112.4, 110.1, 64.9, 64.7, 55.8, 14.8, 14.8; HRMS (ESI⁺): calculated for $\text{C}_{26}\text{H}_{25}\text{N}_4\text{O}_7\text{S}$ [M+H⁺]: $m/z = 537.1438$, m/z found 537.1440; IR ν_{max} (ATR)/ cm^{-1} : 3079, 2976, 2100, 1603, 1562, 1500, 1450, 1388, 1290, 1252, 1149, 1032, 845, 794.

4-Methyl-2-oxo-2H-chromen-8-yl 5-(benzo[d][1,3]dioxol-5-yl)-3-(4-(methylsulfonyl)phenyl)isoxazole-4-sulfonate (6b)



Following general procedure P, the title compound was isolated after filtration and washing with Et_2O as a colourless solid (42.0 mg, 72%). **m.p.** 230 - 231 °C; ^1H NMR (500 MHz, DMSO) δ 8.03 (appt. d, $J = 8.6$ Hz, 2H), 7.79 – 7.76 (m, 3H), 7.32 (dd, $J = 8.2, 1.8$ Hz, 1H), 7.25 (d, $J = 2.4$ Hz, 1H), 7.18 (d, $J = 1.7$ Hz, 1H), 7.13 (dd, $J = 8.7, 2.4$ Hz, 1H), 7.10 (d, $J = 8.2$ Hz, 1H), 6.48 (q, $J = 1.3$ Hz, 1H), 6.16 (s, 2H), 3.31 (s, 3H), 2.44 (d, $J = 1.3$ Hz, 3H); ^{13}C NMR (126 MHz, DMSO) δ 174.1, 160.5, 159.2, 153.4, 152.5, 151.1, 149.7, 147.5, 142.6, 131.1, 130.5, 127.2, 126.8, 125.3, 119.4, 118.4, 117.3, 114.8, 110.8, 110.4, 108.9, 108.6, 102.4, 43.2, 18.1; HRMS (ESI⁺): calculated for $\text{C}_{27}\text{H}_{19}\text{NO}_{10}\text{S}_2\text{Na}$ [M+Na⁺]: $m/z = 604.0343$, m/z found 604.0345; IR ν_{max} (ATR)/ cm^{-1} : 3094, 3073, 3001, 2884, 2795, 1721, 1607, 1579, 1485, 1370, 1257, 1145, 1109, 813, 774.

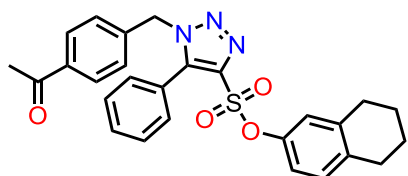
Benzo[d][1,3]dioxol-5-yl 1-(2-(1,3-dioxan-2-yl)ethyl)-5-phenyl-1H-1,2,3-triazole-4-sulfonate (6c)



Following general procedure P, the title compound was isolated after purification by flash column chromatography (0-20% EtOAc in petroleum ether) as an orange gum (44.0 mg, 96%). ^1H NMR (500 MHz, CDCl_3) δ 7.56 – 7.52 (m, 1H), 7.50 – 7.45 (m, 2H), 7.20 – 7.17 (m, 2H), 6.66 (d, $J = 8.5$ Hz, 1H), 6.59 (d, $J = 2.4$ Hz, 1H), 6.51 (dd, $J = 8.5, 2.4$ Hz, 1H), 5.97 (s, 2H), 4.50 (t, $J = 4.7$ Hz, 1H), 4.35 (t, $J = 7.0$ Hz, 2H), 4.01 – 3.96 (m, 2H), 3.68 – 3.61 (m, 2H), 2.12 (td, $J = 7.0, 4.7$ Hz, 2H), 1.96 (dddt, $J = 22.5, 17.5, 12.5, 5.0$ Hz, 1H), 1.29 (dtt, $J = 13.5, 2.6,$

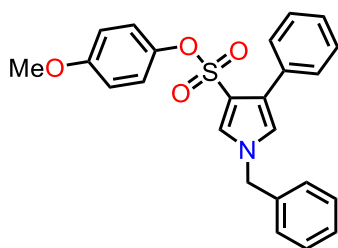
1.3 Hz, 1H); $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 148.2, 146.7, 143.6, 141.0, 139.6, 130.9, 129.7, 128.9, 123.8, 115.4, 108.0, 104.6, 102.1, 98.8, 66.9, 44.5, 34.8, 25.6; **HRMS** (ESI^+): calculated for $\text{C}_{21}\text{H}_{21}\text{N}_3\text{O}_7\text{SNa}$ [$\text{M}+\text{Na}^+$]: $m/z = 482.0992$, m/z found 482.0990; **IR** ν_{max} (ATR)/ cm^{-1} : 3063, 2960, 2928, 2851, 1483, 1381, 1240, 1161, 1138, 1115, 1093, 1038, 945, 860, 834, 769, 738.

5,6,7,8-Tetrahydronaphthalen-2-yl 1-(4-acetylbenzyl)-5-phenyl-1H-1,2,3-triazole-4-sulfonate (6d)



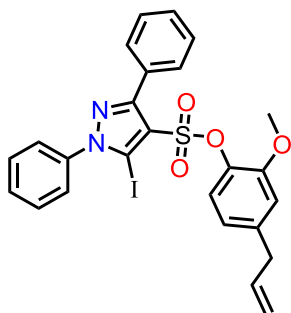
Following general procedure P, the title compound was isolated as an orange gum (47.0 mg, 96%). $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.86 (appt. d, $J = 8.5$ Hz, 2H), 7.51 – 7.46 (m, 1H), 7.34 (appt. t, $J = 7.8$ Hz, 2H), 7.04 (appt. d, $J = 8.5$ Hz, 2H), 6.90 (d, $J = 8.3$ Hz, 1H), 6.83 – 6.79 (m, 2H), 6.72 – 6.66 (m, 2H), 5.44 (s, 2H), 2.69 (t, $J = 6.0$ Hz, 2H), 2.60 – 2.56 (m, 5H), 1.78 – 1.69 (m, 4H); $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 197.3, 147.00, 141.3, 140.2, 139.0, 138.9, 137.4, 136.6, 131.0, 130.2, 129.7, 129.0, 128.8, 127.8, 123.5, 122.6, 119.4, 52.3, 29.4, 29.0 (s), 26.8, 23.0, 22.8; **HRMS** (ESI^+): calculated for $\text{C}_{27}\text{H}_{26}\text{N}_3\text{O}_4\text{S}$ [$\text{M}+\text{H}^+$]: $m/z = 488.1639$, m/z found 488.1640; **IR** ν_{max} (ATR)/ cm^{-1} : 2927, 2857, 1681, 1609, 1492, 1386, 1264, 1174, 923, 788, 695.

4-Methoxyphenyl 1-benzyl-4-phenyl-1H-pyrrole-3-sulfonate (6e)



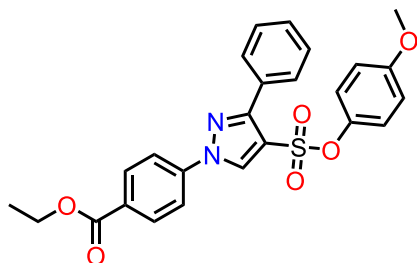
Following general procedure P (heating to 80 °C for 1.5 h), the title compound was isolated after purification by flash column chromatography (0-20% EtOAc in petroleum ether) as a colourless gum (17.0 mg, 40%). $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.70 – 7.66 (m, 2H), 7.42 – 7.30 (m, 6H), 7.18 (d, $J = 2.6$ Hz, 1H), 7.09 – 7.05 (m, 2H), 6.90 (appt. d, $J = 9.2$ Hz, 2H), 6.82 (d, $J = 2.6$ Hz, 1H), 6.73 (appt. d, $J = 9.2$ Hz, 2H), 5.02 (s, 2H), 3.77 (s, 3H); $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 158.1, 143.5, 135.6, 132.5, 129.8, 129.2, 128.9, 128.7, 128.5, 127.6, 127.4, 125.6, 123.5, 122.5, 115.1, 114.4, 55.7, 54.3; **HRMS** (ESI^+): calculated for $\text{C}_{24}\text{H}_{21}\text{NO}_4\text{SNa}$ [$\text{M}+\text{Na}^+$]: $m/z = 442.1083$, m/z found 442.1084; **IR** ν_{max} (ATR)/ cm^{-1} : 3128, 2925, 2852, 1500, 1361, 1249, 1149, 1106, 836, 788, 730, 694.

4-Allyl-2-methoxyphenyl 5-iodo-1,3-diphenyl-1H-pyrazole-4-sulfonate (6f)



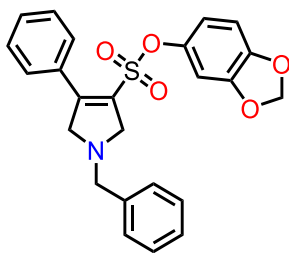
Following general procedure P (3.00 mL MeCN used, stirring for 18 h at room temperature), the title compound was isolated after purification by flash column chromatography (0-20% EtOAc in petroleum ether) as a white solid (50.0 mg, 88%). **m.p.** 140 - 142 °C **¹H NMR** (400 MHz, CDCl₃) δ 7.70 – 7.63 (m, 2H), 7.50 (ddd, *J* = 16.6, 7.5, 5.4 Hz, 5H), 7.44 – 7.37 (m, 3H), 7.23 (d, *J* = 8.2 Hz, 1H), 6.77 (d, *J* = 8.3 Hz, 1H), 6.73 (s, 1H), 5.95 (ddt, *J* = 16.3, 9.6, 6.6 Hz, 1H), 5.15 – 5.11 (m, 1H), 5.09 (s, 1H), 3.62 (s, 3H), 3.38 (d, *J* = 6.4 Hz, 2H); **¹³C NMR** (101 MHz, CDCl₃) δ 155.1, 151.8, 140.9, 139.9, 136.9, 136.4, 130.8, 130.1, 129.6, 129.5, 129.4, 128.0, 127.1, 125.2, 121.5, 121.2, 116.5, 113.0, 92.1, 55.7, 40.2; **HRMS** (ESI⁺): calculated for C₂₅H₂₂I₂N₂O₄S [M+H⁺]: *m/z* = 573.0339, *m/z* found 573.0355; **IR** *v*_{max} (ATR)/cm⁻¹: 3072, 2929, 1596, 1497, 1466, 1381.

Ethyl 4-((4-methoxyphenoxy)sulfonyl)-3-phenyl-1H-pyrazol-1-yl)benzoate (6g)



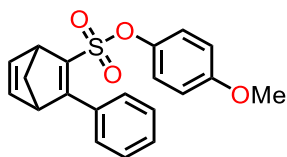
Following general procedure P, the title compound was isolated after purification by flash column chromatography (0-20% EtOAc in petroleum ether) as a colourless oil (48.0 mg, quantitative). **¹H NMR** (400 MHz, CDCl₃) δ 8.42 (s, 1H), 8.18 (d, *J* = 9.0 Hz, 2H), 8.12 – 8.07 (m, 2H), 7.81 (d, *J* = 9.0 Hz, 2H), 7.55 – 7.48 (m, 3H), 6.94 (d, *J* = 9.3 Hz, 2H), 6.75 (d, *J* = 9.3 Hz, 2H), 4.42 (q, *J* = 7.1 Hz, 2H), 3.74 (s, 3H), 1.43 (t, *J* = 7.1 Hz, 3H); **¹³C NMR** (101 MHz, CDCl₃) δ 165.6, 158.5, 151.5, 143.0, 141.6, 134.0, 131.4, 130.2, 130.1, 130.0, 128.9, 128.8, 123.3, 119.0, 117.6, 114.8, 61.5, 55.7, 14.5; **HRMS** (ESI⁺): calculated for C₂₅H₂₃N₂O₆S [M+H⁺]: *m/z* = 479.1271, *m/z* found 479.1275; **IR** *v*_{max} (ATR)/cm⁻¹: 3124, 1705, 1607, 1504, 1375, 1279, 1194.

Benzo[d][1,3]dioxol-5-yl 1-benzyl-4-phenyl-2,5-dihydro-1H-pyrrole-3-sulfonate (6h)



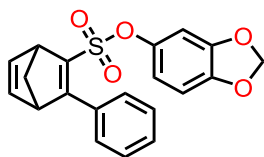
Following general procedure P (10.0 μmol DBU used instead), the title compound was isolated after purification by flash column chromatography (0-20% EtOAc in petroleum ether) as a colourless oil (43.0 mg, 99%). $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.40 – 7.27 (m, 10H), 6.73 (d, J = 8.4 Hz, 1H), 6.66 (d, J = 2.4 Hz, 1H), 6.62 (dd, J = 8.4, 2.4 Hz, 1H), 5.99 (s, 2H), 3.99 (dq, J = 8.0, 4.1 Hz, 4H), 3.83 (s, 2H); $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 153.4, 148.2, 146.7, 143.5, 138.0, 131.3, 130.0, 128.7, 128.7, 128.2, 128.2, 127.9, 127.6, 115.2, 108.1, 104.5, 102.1, 65.5, 61.7, 59.8; **HRMS** (ESI $^+$): calculated for $\text{C}_{24}\text{H}_{22}\text{NO}_5\text{S}$ [$\text{M}+\text{H}^+$]: m/z = 436.1213, m/z found 436.1231; **IR** ν_{max} (ATR)/ cm^{-1} : 2901, 1606, 1478, 1363, 1246, 1158, 1089, 1034.

4-Methoxyphenyl 3-phenylbicyclo[2.2.1]hepta-2,5-diene-2-sulfonate (6i)



Following general procedure P, the title compound was isolated after purification by flash column chromatography (0-25% Et_2O in petroleum ether) as a colourless solid (20.0 mg, 56%). **m.p.** 79 - 80 $^\circ\text{C}$; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.35 (s, 5H), 7.05 – 6.99 (m, 2H), 6.99 – 6.95 (m, 1H), 6.95 – 6.89 (m, 1H), 6.83 – 6.76 (m, 2H), 4.12 – 4.03 (m, 1H), 4.01 – 3.90 (m, 1H), 3.79 (s, 3H), 2.42 – 2.29 (m, 1H), 2.18 – 2.03 (m, 1H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 168.6, 158.3, 143.3, 142.9, 140.9, 140.5, 133.1, 129.8, 128.1, 127.9, 123.5, 114.5, 70.8, 59.6, 55.7, 55.0; **HRMS** (ESI $^+$): calculated for $\text{C}_{20}\text{H}_{18}\text{O}_4\text{SNa}$ [$\text{M}+\text{Na}^+$]: m/z = 377.0818, m/z found 377.0818; **IR** ν_{max} (ATR)/ cm^{-1} : 3013, 2937, 2873, 2844, 1449, 1362, 769.

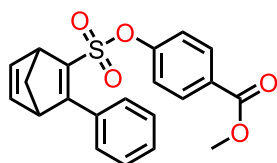
Benzo[d][1,3]dioxol-5-yl 3-phenylbicyclo[2.2.1]hepta-2,5-diene-2-sulfonate (6j)



Following general procedure P, the title compound was isolated after purification by flash column chromatography (0-25% Et_2O in petroleum ether) as a colourless solid (33.0 mg, 89%). **m.p.** 71 - 72 $^\circ\text{C}$; $^1\text{H NMR}$

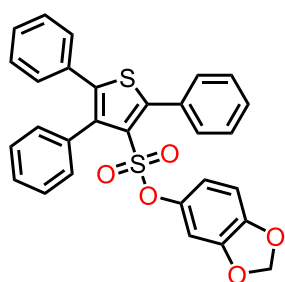
(400 MHz, CDCl₃) δ 7.44 – 7.29 (m, 5H), 7.00 (dd, J = 4.8, 2.9 Hz, 1H), 6.97 – 6.90 (m, 1H), 6.67 (d, J = 8.4 Hz, 1H), 6.60 (d, J = 2.4 Hz, 1H), 6.54 (dd, J = 8.4, 2.4 Hz, 1H), 5.97 (s, 2H), 4.13 – 4.06 (m, 1H), 3.97 (s, 1H), 2.42 – 2.31 (m, 1H), 2.18 – 2.09 (m, 1H); ¹³C NMR (101 MHz, CDCl₃) δ 168.8, 148.0, 146.4, 143.6, 143.3, 140.8, 140.5, 133.1, 129.9, 128.2, 127.9, 115.4, 107.8, 104.6, 102.0, 70.9, 59.7, 55.0; HRMS (ESI⁺): calculated for C₂₀H₁₆O₅SNa [M+Na⁺]: m/z = 391.0610, m/z found 391.0614; IR ν_{\max} (ATR)/cm⁻¹: 3065, 2983, 2939, 1478, 1365, 1158, 824.

Methyl 4-(((3-phenylbicyclo[2.2.1]hepta-2,5-dien-2-yl)sulfonyl)oxy)benzoate (6k)



Following general procedure P, the title compound was isolated after purification by flash column chromatography (0-25% Et₂O in petroleum ether) as a colourless solid (31.0 mg, 80%). **m.p.** 63 - 64 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.96 (appt. d, J = 8.7 Hz, 2H), 7.36 (s, 5H), 7.08 (appt. d, J = 8.8 Hz, 2H), 6.91 (dt, J = 8.3, 5.2 Hz, 2H), 4.14 – 4.07 (m, 1H), 3.99 – 3.92 (m, 1H), 3.91 (s, 3H), 2.37 – 2.31 (m, 1H), 2.15 – 2.10 (m, 1H); ¹³C NMR (101 MHz, CDCl₃) δ 169.3, 166.1, 153.0, 143.2, 140.8, 140.5, 133.0, 131.3, 130.1, 128.7, 128.2, 127.9, 122.1, 71.0, 59.7, 54.9, 52.4; HRMS (ESI⁺): calculated for C₂₁H₁₈O₅SNa [M+Na⁺]: m/z = 405.0767, m/z found 405.0764; IR ν_{\max} (ATR)/cm⁻¹: 3069, 2998, 2949, 2873, 1721, 1277, 1148, 865.

Benzo[d][1,3]dioxol-5-yl 2,4,5-triphenylthiophene-3-sulfonate (6l)



Following general procedure P, the title compound was isolated after purification by flash column chromatography (0-25% Et₂O in petroleum ether) as a colourless solid (41.0 mg, 80%). **m.p.** 152 - 154 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.51 – 7.39 (m, 5H), 7.39 – 7.33 (m, 5H), 7.26 – 7.20 (m, 3H), 7.20 – 7.14 (m, 2H), 6.64 (d, J = 8.4 Hz, 1H), 6.34 – 6.27 (m, 1H), 6.28 – 6.20 (m, 1H), 5.96 (s, 2H); ¹³C NMR (101 MHz, CDCl₃) δ 150.7, 148.0, 146.4, 142.9, 141.3, 137.8, 134.4, 132.5, 131.8, 131.2, 130.23, 130.1, 129.5, 129.4, 128.6, 128.3, 128.2, 128.1, 128.1, 115.3, 107.9, 104.7, 101.9; HRMS (ESI⁺): calculated for C₂₉H₂₀O₅S₂Na [M+Na⁺]: m/z = 535.0644, m/z found 534.0647; IR ν_{\max} (ATR)/cm⁻¹: 2929, 2865, 1481, 1373, 1054, 835.

LLAMA and principle moments of inertia (PMI) analysis for DOC and SuFEx libraries

LLAMA (Lead-Likeness and Molecular Analysis) is an open access tool for assessing the lead-likeness and novelty of molecular scaffolds where the plot possesses a lead-like chemical space (Alog P of -1 to 3 and RMM of 200 to 350) and a 'Lipinski' space (Alog P <5 and RMM <500). Using a set of criteria each molecule or scaffold is also given a lead-likeness penalty. The lead-likeness penalty is a measure of how far outside lead-like space a compound lies. For each of the key properties (heavy atom count, AlogP, number of aromatic rings and an undesirable functional group filter) the compound gains penalty points dependent on how far outside the ideal space it lies. The overall score is the sum of these individual penalties (for more information on the scoring criteria see Colomer *et al.*, 2016).^[20]

Using the LLAMA package the principal moments of inertia (PMI) of each molecule can also be determined to assess the shape distribution of a library. The three vertices of the triangular plot represent the extremes of molecular geometry. The top left-hand corner represents a linear molecule (diacetylene), the top right-hand corner represents a spherical molecule (adamantane) and the bottom corner represents a disc-like molecule (benzene). To generate the PMI coordinates for each molecule, the system randomly generates a number of 3D conformers, minimises their energy and selects the lowest-energy conformer. The system then calculates the moments of inertia in the x, y and z axes. The PMI plot I1 coordinates are calculated by dividing inertia(x) by inertia(z). The I2 coordinates are calculated by dividing inertia(y) by inertia(z).^[21]

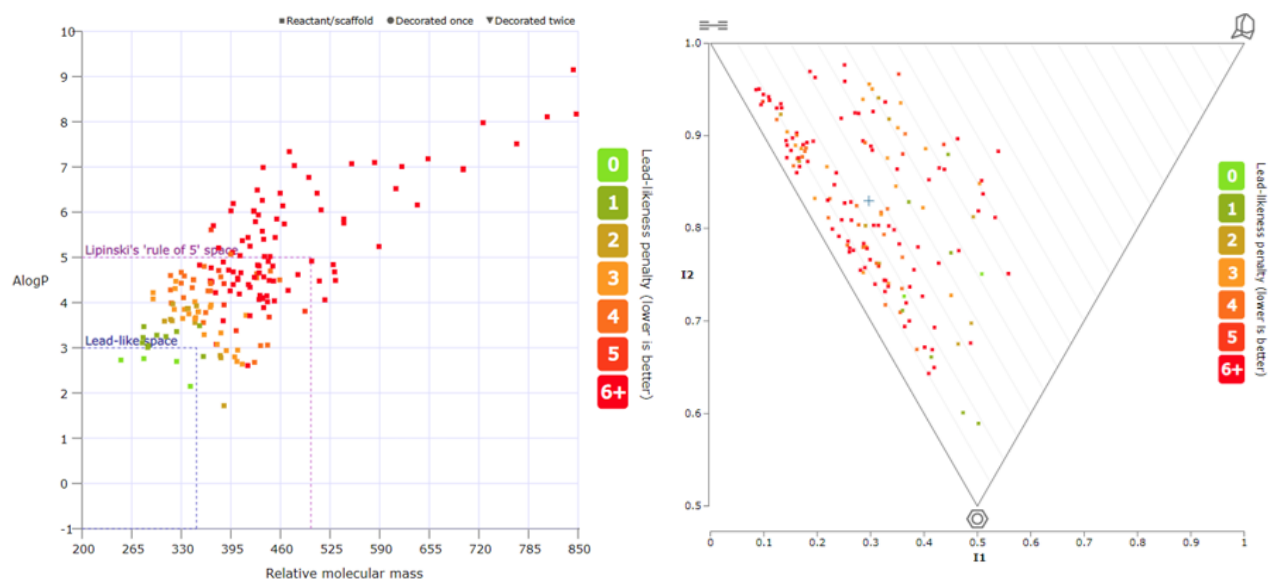


Figure S1. Lead-likeness analysis and PMI analysis of the DOC library using the LLAMA software package.

Of the 173 DOC compounds (Figure S-1) 2.30% were deemed to be in lead-like chemical space with 68.0% in 'Lipinski' space; indicating that the majority of the library could be considered 'drug-like'. A principle moment of inertia (PMI) plot for the library suggests that the majority of the library lies between the linear (diacetylene) and the flat disc-like (benzene) sections— this is inherently related to the nature of the cycloaddition reactions and the sp^2 rich products. In comparison, the sulfonate modification compound library 0% were deemed to be

in lead-like chemical space and 28.3% in 'Lipinski' space, most likely due to the increase in molecular weight and the addition of an extra ring through SuFEx modification. The sulfonate products however, had a more diverse PMI compared to the DOC library and can be used to increase the 3D nature of the library.

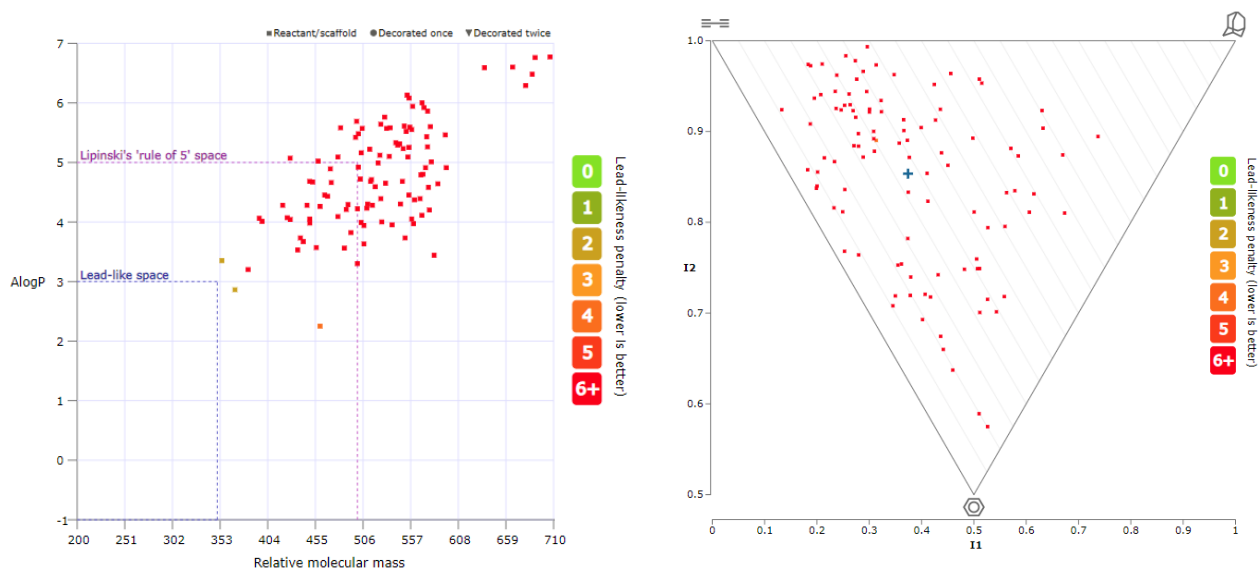
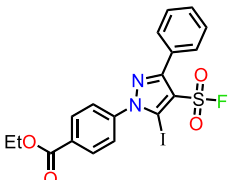
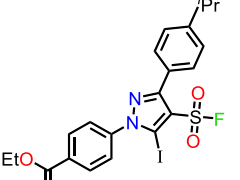
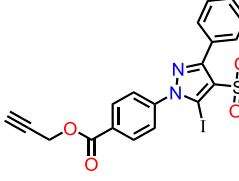
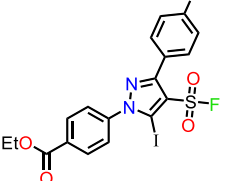
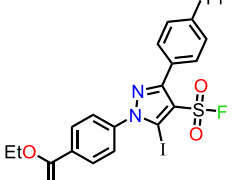
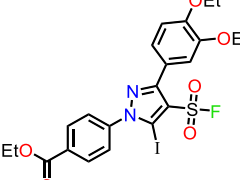
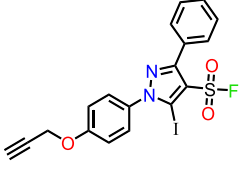
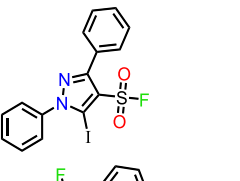
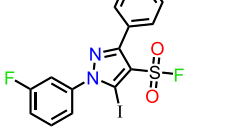
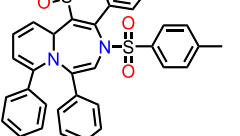


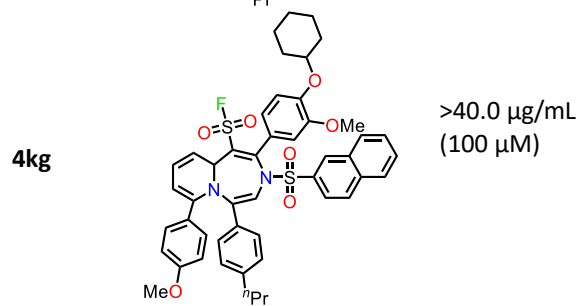
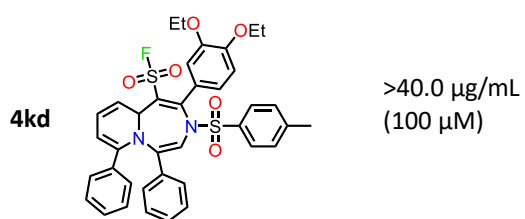
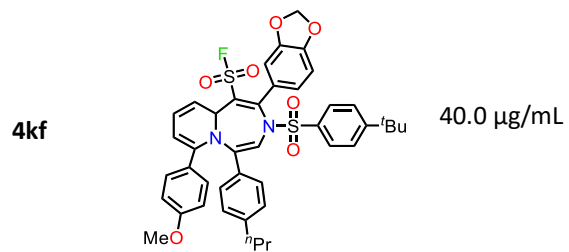
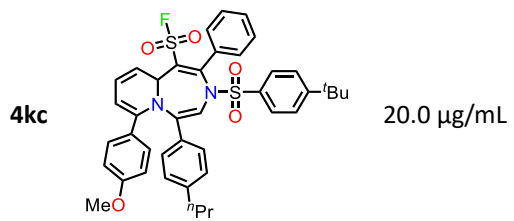
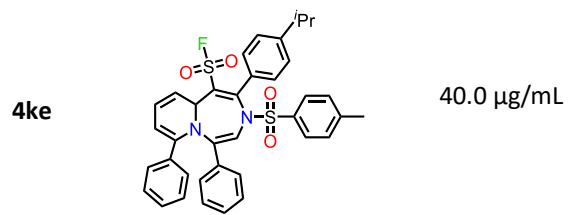
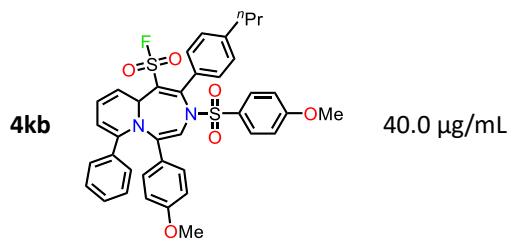
Figure S2. Lead-likeness analysis and PMI analysis of the sulfonate library using the LLAMA software package.

Antibacterial assays and method

Effects of molecules on bacterial growth and minimum inhibitory concentrations (MIC) were determined as described previously,^[22,23] using a modified CLSI microbroth dilution method in 100 μ L of Mueller Hinton broth containing 2-fold dilutions of molecules with 2% DMSO at the highest concentration. DMSO vehicle or methicillin were used as controls. The MIC was defined as the lowest concentration of molecules that resulted in no increase in growth relative to background at 24 hours.

Table 1. Hit compounds after screening **4a-h** and **4k-l** library originally at 200 μ M, followed by the determination of MICs

#	Structure	MIC (<i>Staphylococcus aureus</i> USA300)	#	Structure	MIC (<i>Staphylococcus aureus</i> USA300)
	Methicillin	5.00 μ g/mL			
4ea		0.625 μ g/mL	4ef		5.00 μ g/mL
4eb		0.625 μ g/mL	4eg		5.00 μ g/mL
4ec		2.50 μ g/mL	4eh		5.00-10.0 μ g/mL
4ed		1.25 μ g/mL	4ei		~20.0 μ g/mL
4ee		0.625 μ g/mL	4ka		>40.0 μ g/mL (100 μ M)

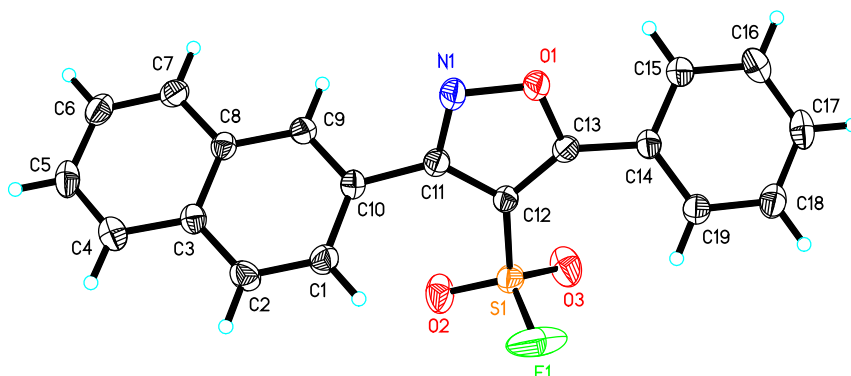


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X-ray crystallography data

X-ray data of structure **4aa** (CCDC1983970)



Datablock: d8v19481

Bond precision: C-C = 0.0041 Å Wavelength=0.71073
Cell: a=6.0654(9) b=14.541(3) c=18.061(3)
alpha=90 beta=90.981(4) gamma=90
Temperature: 293 K

	Calculated	Reported
Volume	1592.7(5)	1592.7(4)
Space group	P 21/n	P 21/n
Hall group	-P 2yn	-P 2yn
Moiety formula	C19 H12 F N O3 S	?
Sum formula	C19 H12 F N O3 S	C19 H12 F N O3 S
Mr	353.36	353.36
Dx, g cm ⁻³	1.474	1.474
Z	4	4
Mu (mm ⁻¹)	0.233	0.233
F000	728.0	728.0
F000'	728.87	
h,k,lmax	7,17,21	7,17,21
Nref	2961	2954
Tmin,Tmax	0.959,0.975	0.658,0.746
Tmin'	0.959	

Correction method= # Reported T Limits: Tmin=0.658 Tmax=0.746 AbsCorr = MULTI-SCAN

Data completeness= 0.998

Theta(max)= 25.496

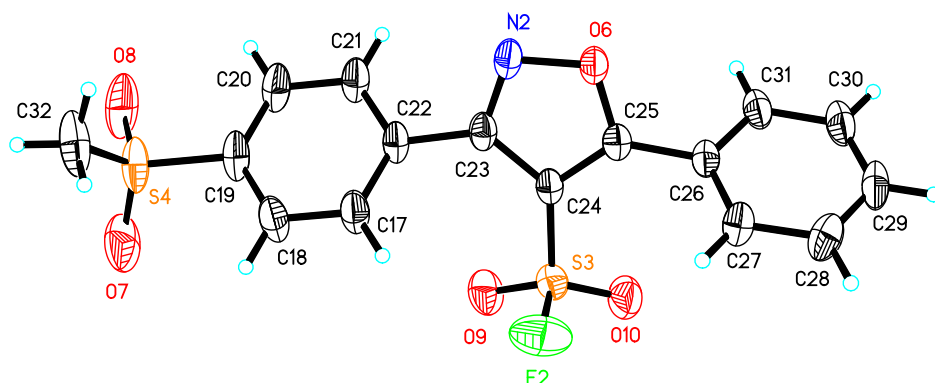
R(reflections)= 0.0573(2245)

wR2(reflections)= 0.1469(2954)

S = 1.081

Npar= 226

X-ray data of structure 4aq (CCDC1983972)



Datablock: mo_d8v19492_0m

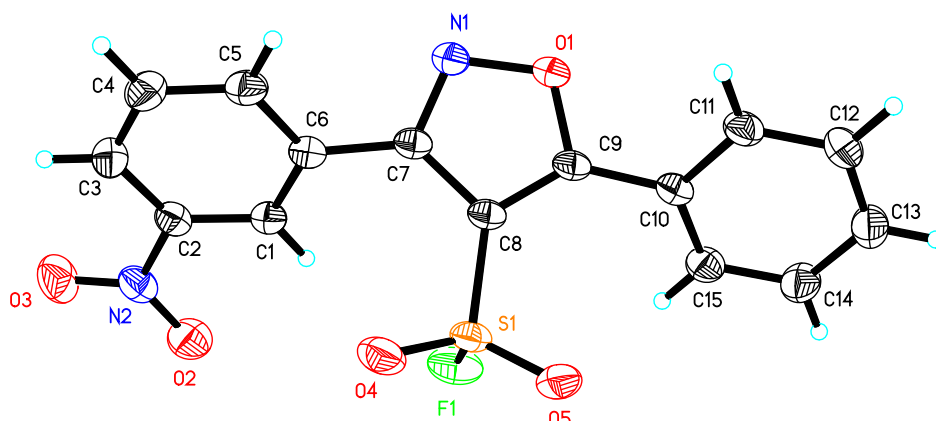
Bond precision: C-C = 0.0036 Å Wavelength=0.71073
 Cell: a=7.8645(3) b=15.0207(6) c=15.6640(6)
 alpha=68.380(1) beta=75.602(1) gamma=86.551(1)
 Temperature: 293 K

	Calculated	Reported
Volume	1665.17(11)	1665.17(11)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C16 H12 F N O5 S2	?
Sum formula	C16 H12 F N O5 S2	C16 H12 F N O5 S2
Mr	381.39	381.39
Dx, g cm-3	1.521	1.521
Z	4	4
Mu (mm-1)	0.358	0.358
F000	784.0	784.0
F000'	785.42	
h,k,lmax	9,18,19	9,18,19
Nref	6552	6509
Tmin,Tmax	0.942,0.965	0.665,0.746
Tmin'	0.934	

Correction method= # Reported T Limits: Tmin=0.665 Tmax=0.746 AbsCorr = MULTI-SCAN

Data completeness= 0.993 Theta(max)= 25.997
 R(reflections)= 0.0444(5152) wR2(reflections)= 0.1227(6509)
 S = 1.045 Npar= 453

X-ray data of structure 4as (CCDC1983971)



Datablock: mo_d8v19491_0m

Bond precision: C-C = 0.0028 Å Wavelength=0.71073
 Cell: a=8.5194(2) b=11.1364(3) c=15.8544(3)
 alpha=90 beta=101.678(1) gamma=90
 Temperature: 293 K

	Calculated	Reported
Volume	1473.06(6)	1473.06(6)
Space group	P 21/c	P 21/c
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C15 H9 F N2 O5 S	?
Sum formula	C15 H9 F N2 O5 S	C15 H9 F N2 O5 S
Mr	348.30	348.30
Dx, g cm ⁻³	1.571	1.571
Z	4	4
Mu (mm ⁻¹)	0.262	0.262
F000	712.0	712.0
F000'	712.92	
h,k,lmax	10,13,19	10,13,19
Nref	2902	2887
Tmin,Tmax	0.957,0.974	0.653,0.746
Tmin'	0.954	

Correction method= # Reported T Limits: Tmin=0.653 Tmax=0.746 AbsCorr = MULTI-SCAN

Data completeness= 0.995

Theta(max)= 25.997

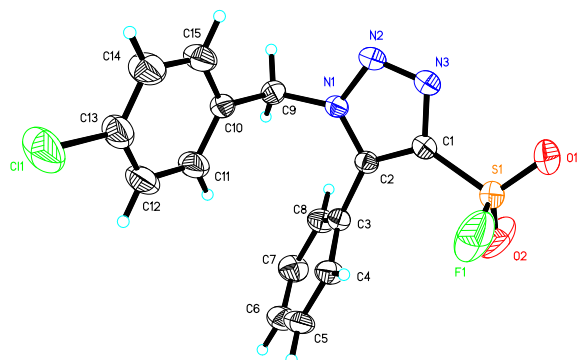
R(reflections)= 0.0369(2366)

wR2(reflections)= 0.1017(2887)

S = 1.066

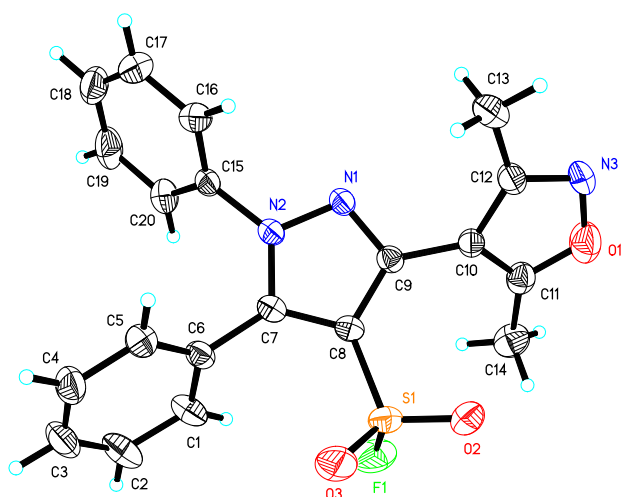
Npar= 218

X-ray data of structure 4ca (CCDC1983969)



Datablock: mo_d8v191015_0m

Bond precision:	C-C = 0.0051 Å	Wavelength=0.71073
Cell:	a=25.8046(10) b=14.9084(7) c=8.5482(4)	
	alpha=90 beta=105.211(1) gamma=90	
Temperature: 293 K		
	Calculated	Reported
Volume	3173.3(2)	3173.3(2)
Space group	C 2/c	C 2/c
Hall group	-C 2yc	-C 2yc
Moiety formula	C15 H11 Cl F N3 O2 S	?
Sum formula	C15 H11 Cl F N3 O2 S	C15 H11 Cl F N3 O2 S
Mr	351.78	351.78
Dx, g cm ⁻³	1.473	1.473
Z	8	8
Mu (mm ⁻¹)	0.394	0.394
F000	1440.0	1440.0
F000'	1442.79	
h,k,lmax	31,18,10	31,18,10
Nref	3125	3105
Tmin,Tmax	0.936,0.958	0.654,0.746
Tmin'	0.935	
Correction method= # Reported T Limits: Tmin=0.654 Tmax=0.746 AbsCorr = MULTI-SCAN		
Data completeness= 0.994		Theta(max)= 25.990
R(reflections)= 0.0610(2364)		wR2(reflections)= 0.1806(3105)
S = 1.037	Npar= 208	
X-ray data of structure 4db (CCDC1983973)		



Datablock: mo_d8v19493_0m

Bond precision: C-C = 0.0042 Å Wavelength=0.71073
 Cell: a=39.5801(10) b=11.7992(3) c=17.4500(4)
 alpha=90 beta=109.001(1) gamma=90

Temperature: 293 K

	Calculated	Reported
Volume	7705.4(3)	7705.3(3)
Space group	C 2/c	C 2/c
Hall group	-C 2yc	-C 2yc
Moiety formula	C20 H16 F N3 O3 S	?
Sum formula	C20 H16 F N3 O3 S	C20 H16 F N3 O3 S
Mr	397.42	397.42
Dx, g cm ⁻³	1.370	1.370
Z	16	16
Mu (mm ⁻¹)	0.203	0.203
F000	3296.0	3296.0
F000'	3299.56	
h,k,lmax	48,14,21	48,14,21
Nref	7589	7568
Tmin,Tmax	0.964,0.978	0.605,0.746
Tmin'	0.960	

Correction method= # Reported T Limits: Tmin=0.605 Tmax=0.746 AbsCorr = MULTI-SCAN

Data completeness= 0.997

Theta(max)= 26.000

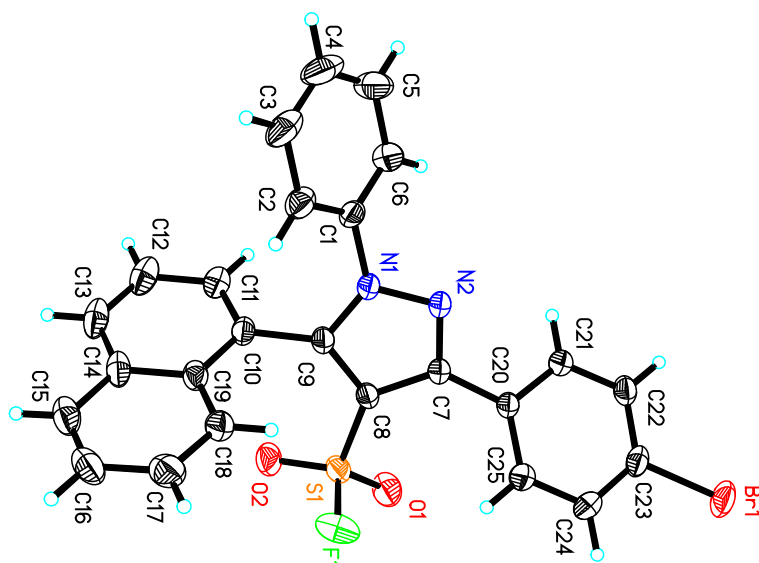
R(reflections)= 0.0538(5736)

wR2(reflections)= 0.1542(7568)

S = 1.026

Npar= 518

X-ray data of structure 4dh (CCDC1983977)



Datablock: mo_d8v19487_0m

Bond precision: C-C = 0.0043 Å Wavelength=0.71073

Cell: a=10.0429(4) b=10.1977(5) c=11.6072(5)
 alpha=96.715(2) beta=111.783(1) gamma=91.036(2)

Temperature: 293 K

	Calculated	Reported
Volume	1093.85(8)	1093.85(8)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C ₂₅ H ₁₆ Br F N ₂ O ₂ S	?
Sum formula	C ₂₅ H ₁₆ Br F N ₂ O ₂ S	C ₂₅ H ₁₆ Br F N ₂ O ₂ S
Mr	507.36	507.37
Dx, g cm ⁻³	1.540	1.540
Z	2	2
Mu (mm ⁻¹)	2.009	2.009
F ₀₀₀	512.0	512.0
F ₀₀₀ '	511.88	
h,k,lmax	12,12,14	12,12,14
Nref	4297	4254
Tmin,Tmax	0.732,0.818	0.587,0.746
Tmin'	0.718	

Correction method= # Reported T Limits: Tmin=0.587 Tmax=0.746 AbsCorr = MULTI-SCAN

Data completeness= 0.990

Theta(max)= 25.999

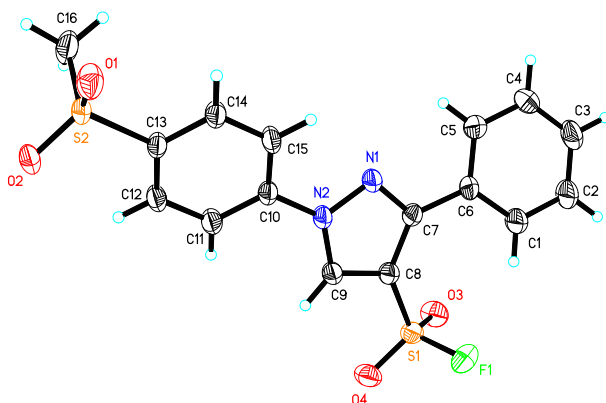
R(reflections)= 0.0363(3279)

wR2(reflections)= 0.0951(4254)

S = 1.026

Npar= 290

X-ray data of structure 4en (CCDC1983974)



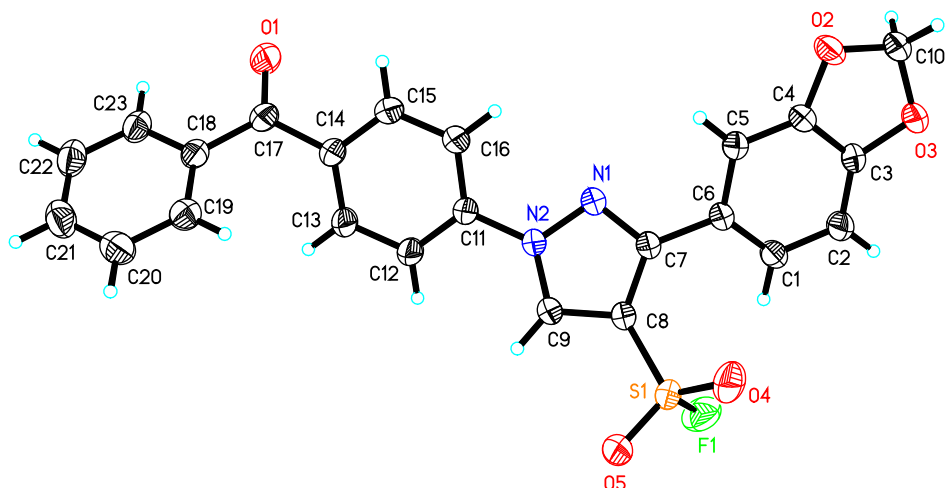
Datablock: mo_d8v19490_0m

Bond precision: C-C = 0.0052 Å Wavelength=0.71073
 Cell: a=4.9232(4) b=10.7857(9) c=15.7702(14)
 alpha=91.102(3) beta=95.522(3) gamma=93.169(3)
 Temperature: 293 K

	Calculated	Reported
Volume	831.99(12)	831.99(12)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C16 H13 F N2 O4 S2	?
Sum formula	C16 H13 F N2 O4 S2	C16 H13 F N2 O4 S2
Mr	380.40	380.40
Dx, g cm ⁻³	1.518	1.518
Z	2	2
Mu (mm ⁻¹)	0.355	0.355
F000	392.0	392.0
F000'	392.69	
h,k,lmax	5,13,19	5,13,19
Nref	3079	3057
Tmin,Tmax	0.950,0.975	0.617,0.746
Tmin'	0.931	

Correction method= # Reported T Limits: Tmin=0.617 Tmax=0.746 AbsCorr = MULTI-SCAN

Data completeness= 0.993 Theta(max)= 25.499
 R(reflections)= 0.0573(2480) wR2(reflections)= 0.1739(3057)
 S = 1.091 Npar= 227



Datablock: mo_d8v19489_0m

Bond precision: C-C = 0.0033 Å Wavelength=0.71073
 Cell: a=12.2503(8) b=12.4284(10) c=14.2741(11)
 alpha=90 beta=110.834(2) gamma=90

Temperature: 293 K

	Calculated	Reported
Volume	2031.2(3)	2031.2(3)
Space group	P 21/n	P 21/n
Hall group	-P 2yn	-P 2yn
Moiety formula	C23 H15 F N2 O5 S	?
Sum formula	C23 H15 F N2 O5 S	C23 H15 F N2 O5 S
Mr	450.43	450.43
Dx, g cm ⁻³	1.473	1.473
Z	4	4
Mu (mm ⁻¹)	0.209	0.209
F000	928.0	928.0
F000'	929.00	
h,k,lmax	15,15,17	15,15,17
Nref	3991	3984
Tmin,Tmax	0.961,0.973	0.598,0.746
Tmin'	0.959	

Correction method= # Reported T Limits: Tmin=0.598 Tmax=0.746 AbsCorr = MULTI-SCAN

Data completeness= 0.998

Theta(max)= 25.999

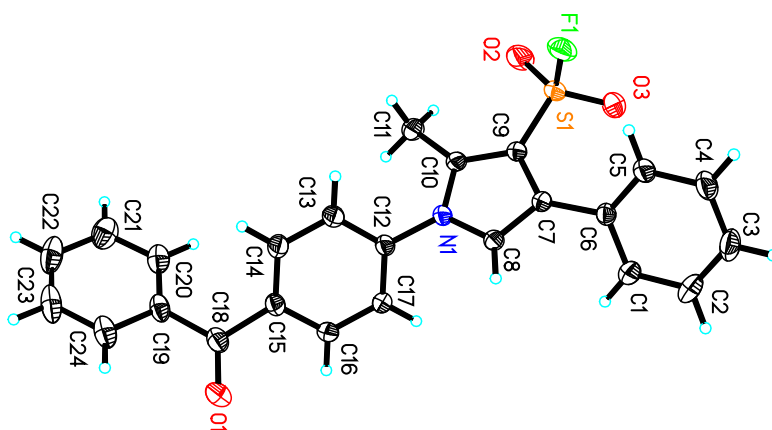
R(reflections)= 0.0429(2951)

wR2(reflections)= 0.1190(3984)

S = 1.017

Npar= 290

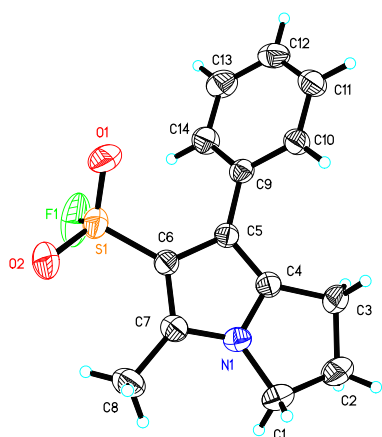
X-ray data of structure 4fg (CCDC1983975)



Datablock: mo_d8v19488_0m

Bond precision:	C-C = 0.0028 Å	Wavelength=0.71073
Cell:	a=7.8823(2) b=10.9580(3) c=12.2338(3)	
	alpha=78.789(1) beta=76.764(1) gamma=83.985(1)	
Temperature:	293 K	
	Calculated	Reported
Volume	1006.97(5)	1006.97(5)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C ₂₄ H ₁₈ F N O ₃ S	?
Sum formula	C ₂₄ H ₁₈ F N O ₃ S	C ₂₄ H ₁₈ F N O ₃ S
Mr	419.45	419.45
Dx, g cm ⁻³	1.383	1.383
Z	2	2
Mu (mm ⁻¹)	0.196	0.196
F ₀₀₀	436.0	436.0
F ₀₀₀ '	436.46	
h,k,lmax	9,13,15	9,13,15
Nref	3958	3916
Tmin,Tmax	0.970,0.981	0.680,0.746
Tmin'	0.969	
Correction method= # Reported T Limits: Tmin=0.680 Tmax=0.746 AbsCorr = MULTI-SCAN		
Data completeness=	0.989	Theta(max)= 25.996
R(reflections)=	0.0409(3227)	wR2(reflections)= 0.1121(3916)
S =	1.033	Npar= 273

X-ray data of structure 4ga (CCDC1983978)



Datablock: mo_d8v191016_0m

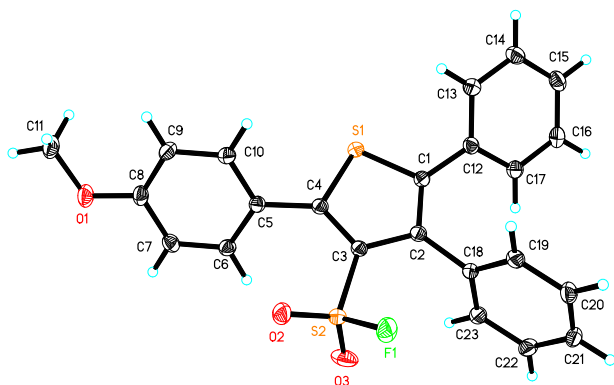
Bond precision: C-C = 0.0038 Å Wavelength=0.71073
 Cell: a=8.2800(5) b=16.9766(11) c=9.6583(6)
 alpha=90 beta=101.364(2) gamma=90
 Temperature: 293 K

	Calculated	Reported
Volume	1331.02(14)	1331.01(14)
Space group	P 21/c	P 21/c
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C14 H14 F N O2 S	?
Sum formula	C14 H14 F N O2 S	C14 H14 F N O2 S
Mr	279.32	279.32
Dx, g cm ⁻³	1.394	1.394
Z	4	4
Mu (mm ⁻¹)	0.252	0.252
F000	584.0	584.0
F000'	584.78	
h,k,lmax	10,20,11	10,20,11
Nref	2603	2591
Tmin,Tmax	0.956,0.973	0.674,0.746
Tmin'	0.956	

Correction method= # Reported T Limits: Tmin=0.674 Tmax=0.746 AbsCorr = MULTI-SCAN

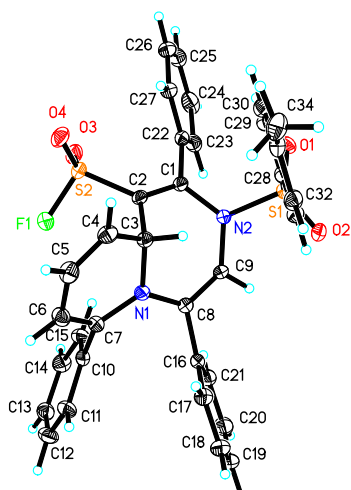
Data completeness= 0.995 Theta(max)= 25.994
 R(reflections)= 0.0500(2013) wR2(reflections)= 0.1484(2591)
 S = 1.064 Npar= 173

X-ray data of structure 4hb (CCDC1983963)



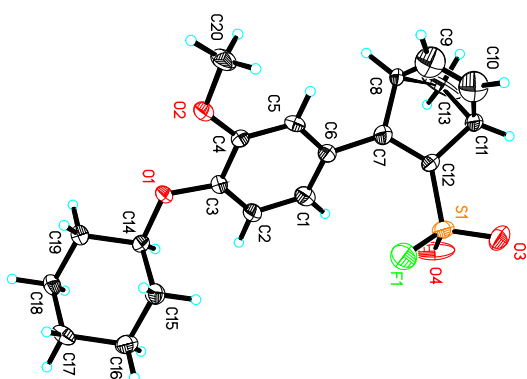
Datablock: d8v191004

Bond precision:	C-C = 0.0025 Å	Wavelength=0.71073	
Cell:	a=14.8154(7)	b=12.1306(6)	c=12.0053(6)
	alpha=90	beta=113.789(1)	gamma=90
Temperature: 193 K			
	Calculated	Reported	
Volume	1974.27(17)	1974.27(17)	
Space group	P 21/c	P 21/c	
Hall group	-P 2ybc	-P 2ybc	
Moiety formula	C23 H17 F O3 S2	?	
Sum formula	C23 H17 F O3 S2	C23 H17 F O3 S2	
Mr	424.49	424.48	
Dx,g cm ⁻³	1.428	1.428	
Z	4	4	
Mu (mm ⁻¹)	0.302	0.301	
F000	880.0	880.0	
F000'	881.41		
h,k,lmax	18,14,14	18,14,14	
Nref	3883	3877	
Tmin,Tmax	0.947,0.965	0.646,0.746	
Tmin'	0.947		
Correction method= # Reported T Limits: Tmin=0.646 Tmax=0.746 AbsCorr = MULTI-SCAN			
Data completeness= 0.998	Theta(max)= 25.997		
R(reflections)= 0.0332(3449)	wR2(reflections)= 0.0835(3877)		
S = 1.060	Npar= 263		
X-ray data of structure 4ka (CCDC1983965)			



Datablock: mo_d8v191009_0m

Bond precision:	C-C = 0.0031 Å	Wavelength=0.71073
Cell:	a=9.3192(2)	b=29.3986(8)
	alpha=90	beta=102.820(1)
		c=10.7679(3)
		gamma=90
Temperature: 193 K		
	Calculated	Reported
Volume	2876.56(13)	2876.56(13)
Space group	P 21/n	P 21/n
Hall group	-P 2yn	-P 2yn
Moiety formula	C34 H27 F N2 O4 S2	?
Sum formula	C34 H27 F N2 O4 S2	C34 H27 F N2 O4 S2
Mr	610.70	610.69
Dx, g cm ⁻³	1.410	1.410
Z	4	4
Mu (mm ⁻¹)	0.235	0.235
F000	1272.0	1272.0
F000'	1273.56	
h,k,lmax	11,36,13	11,36,13
Nref	5654	5621
Tmin,Tmax	0.959,0.970	0.673,0.746
Tmin'	0.959	
Correction method= # Reported T Limits: Tmin=0.673 Tmax=0.746 AbsCorr = MULTI-SCAN		
Data completeness= 0.994	Theta(max)= 26.000	
R(reflections)= 0.0405(4618)	wR2(reflections)= 0.1024(5621)	
S = 1.027	Npar= 390	
X-ray data of structure 4lg (CCDC1983966)		



Datablock: d8v191012

Bond precision: C-C = 0.0109 Å Wavelength=0.71073
 Cell: a=13.6756(6) b=14.5111(6) c=9.2765(4)
 alpha=90 beta=98.364(1) gamma=90

Temperature: 193 K

	Calculated	Reported
Volume	1821.32(14)	1821.32(14)
Space group	P c	P c
Hall group	P -2yc	P -2yc
Moiety formula	C ₂₀ H ₂₃ F O ₄ S	?
Sum formula	C ₂₀ H ₂₃ F O ₄ S	C ₂₀ H ₂₃ F O ₄ S
Mr	378.44	378.44
D _x , g cm ⁻³	1.380	1.380
Z	4	4
Mu (mm ⁻¹)	0.210	0.210
F ₀₀₀	800.0	800.0
F ₀₀₀ ^l	800.92	
h,k,l _{max}	16,17,11	16,17,11
N _{ref}	7163[3590]	7107
T _{min} ,T _{max}	0.963,0.975	0.653,0.746
T _{min} ^l	0.963	

Correction method= # Reported T Limits: T_{min}=0.653 T_{max}=0.746 AbsCorr = MULTI-SCAN

Data completeness= 1.98/0.99

Theta(max)= 25.999

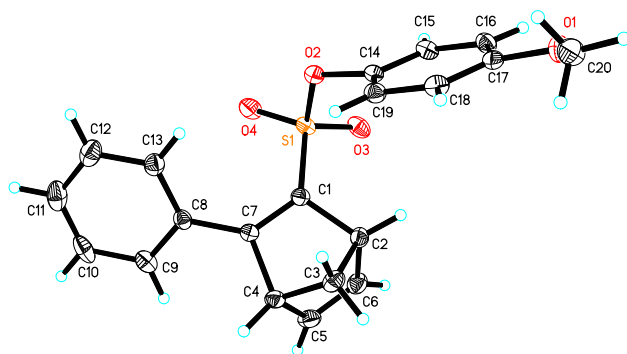
R(reflections)= 0.0532(5726)

wR2(reflections)= 0.1322(7107)

S = 1.028

Npar= 472

X-ray data of structure 6l (CCDC1983962)



Datablock: mo_d8v191003_0m

Bond precision: C-C = 0.0048 Å Wavelength=0.71073
 Cell: a=9.9964(4) b=7.9770(3) c=11.5581(5)
 alpha=90 beta=113.947(1) gamma=90

Temperature: 193 K

	Calculated	Reported
Volume	842.32(6)	842.32(6)
Space group	P 21	P 21
Hall group	P 2yb	P 2yb
Moiety formula	C20 H18 O4 S	?
Sum formula	C20 H18 O4 S	C20 H18 O4 S
Mr	354.40	354.40
Dx, g cm ⁻³	1.397	1.397
Z	2	2
Mu (mm ⁻¹)	0.214	0.214
F000	372.0	372.0
F000'	372.43	
h,k,lmax	12,9,14	12,9,14
Nref	3131[1687]	2942
Tmin,Tmax	0.967,0.983	0.672,0.746
Tmin'	0.962	

Correction method= # Reported T Limits: Tmin=0.672 Tmax=0.746 AbsCorr = MULTI-SCAN

Data completeness= 1.74/0.94

Theta(max)= 25.497

R(reflections)= 0.0317(2758)

wR2(reflections)= 0.0718(2942)

S = 1.070

Npar= 228

Experimental procedure for SuFEx reactions performed in a 96 well plate format

Method

Stock solutions of heterocyclic sulfonyl fluorides (**4eq**, **4ey**, **4ei**, **4hg**, **4hb**, **4al**, **4aq**, **4an**, **4aa**, **4ad**, **4dg**, **4df**, **4ce**, **4cn**, **4ch** and **4de**) (3.00 μmol), TBS ethers **5a-5f** (3.00 μmol) and DBU (600 nmol) in MeCN were added to a 96 well plate totalling a volume of 500 μL in each well. The plate was agitated at 310 rpm at room temperature for 16 h and 150 μL aliquots were taken from the reaction mixture and analyzed by LCMS (70-90 % $\text{CH}_3\text{CN}/\text{water}$ 15.9 min) or TLC.

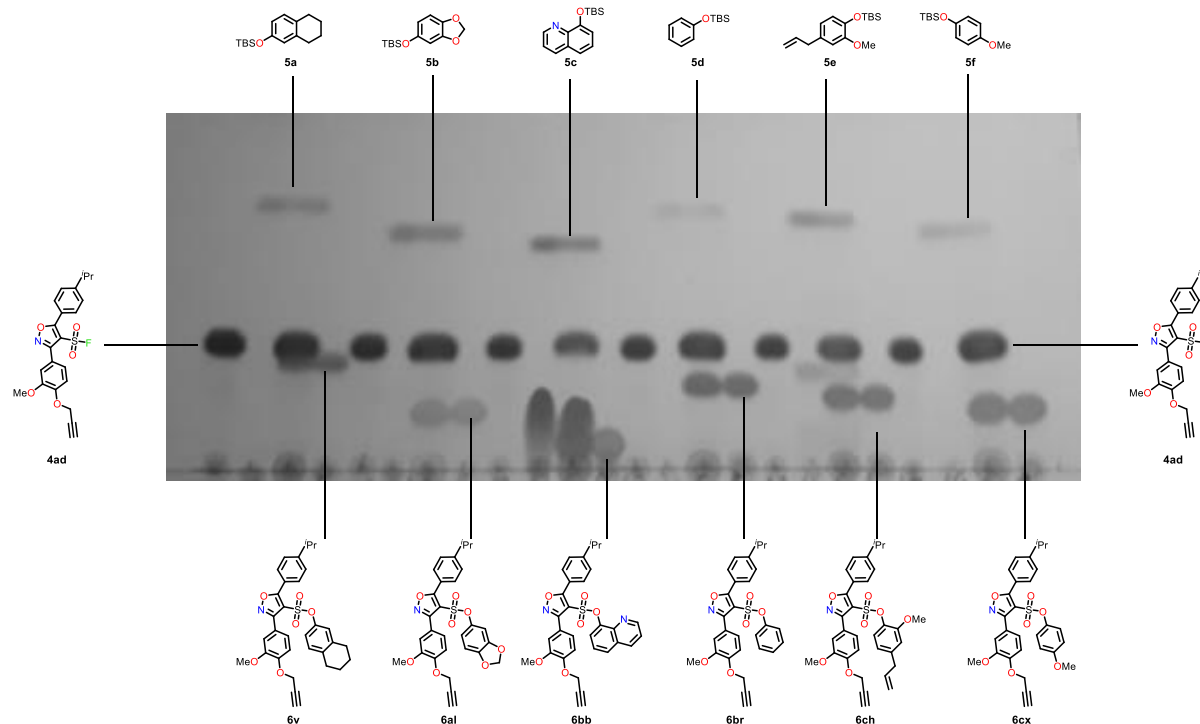
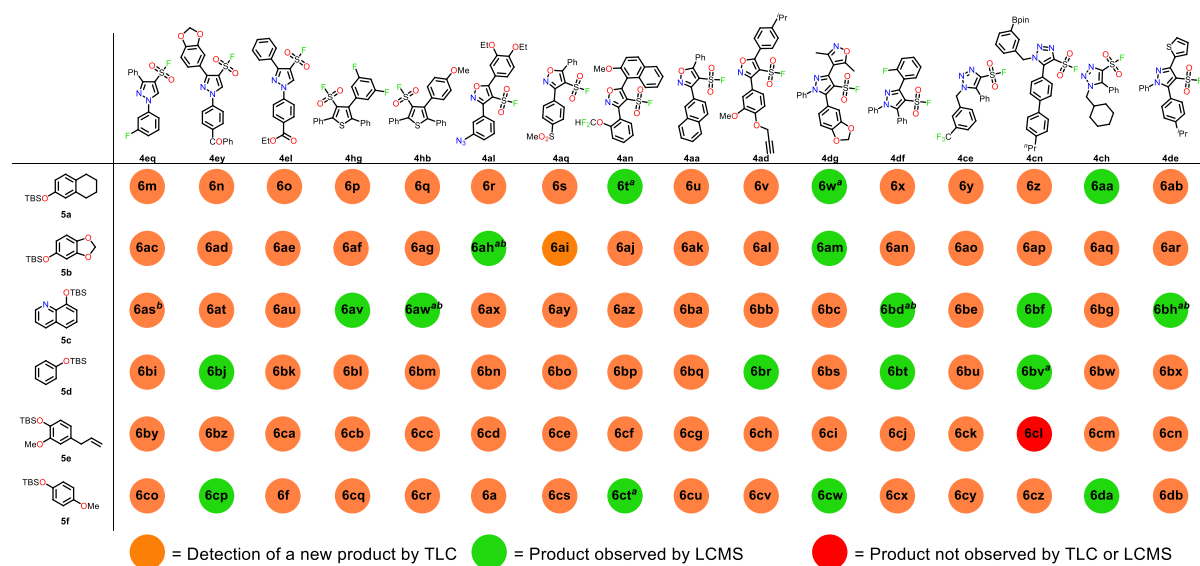
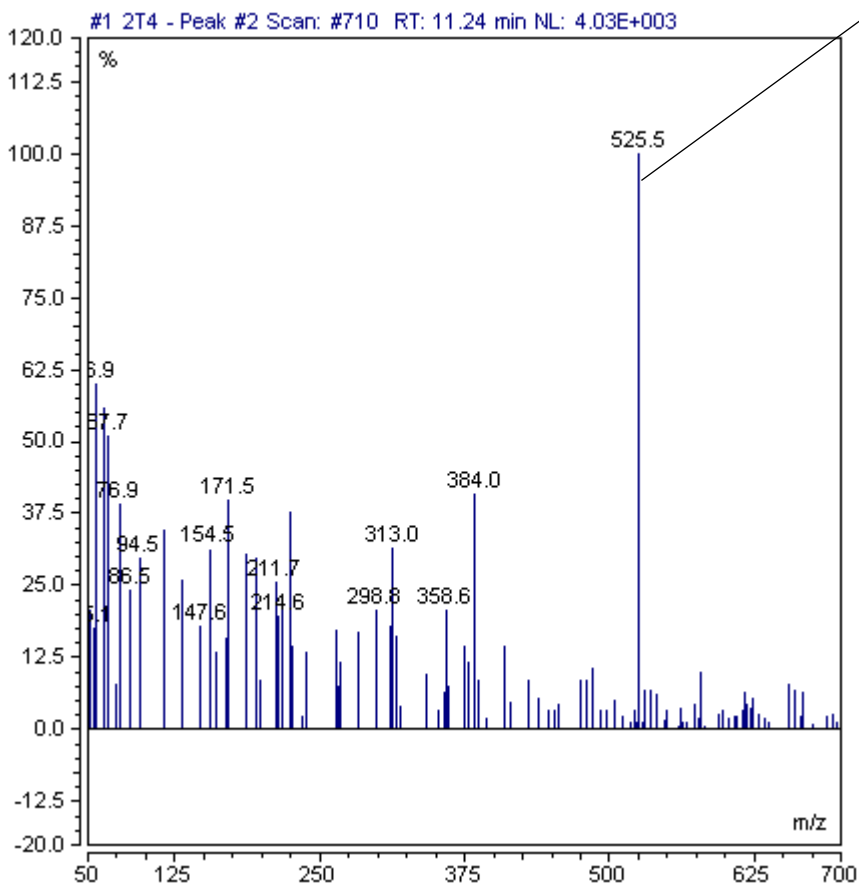
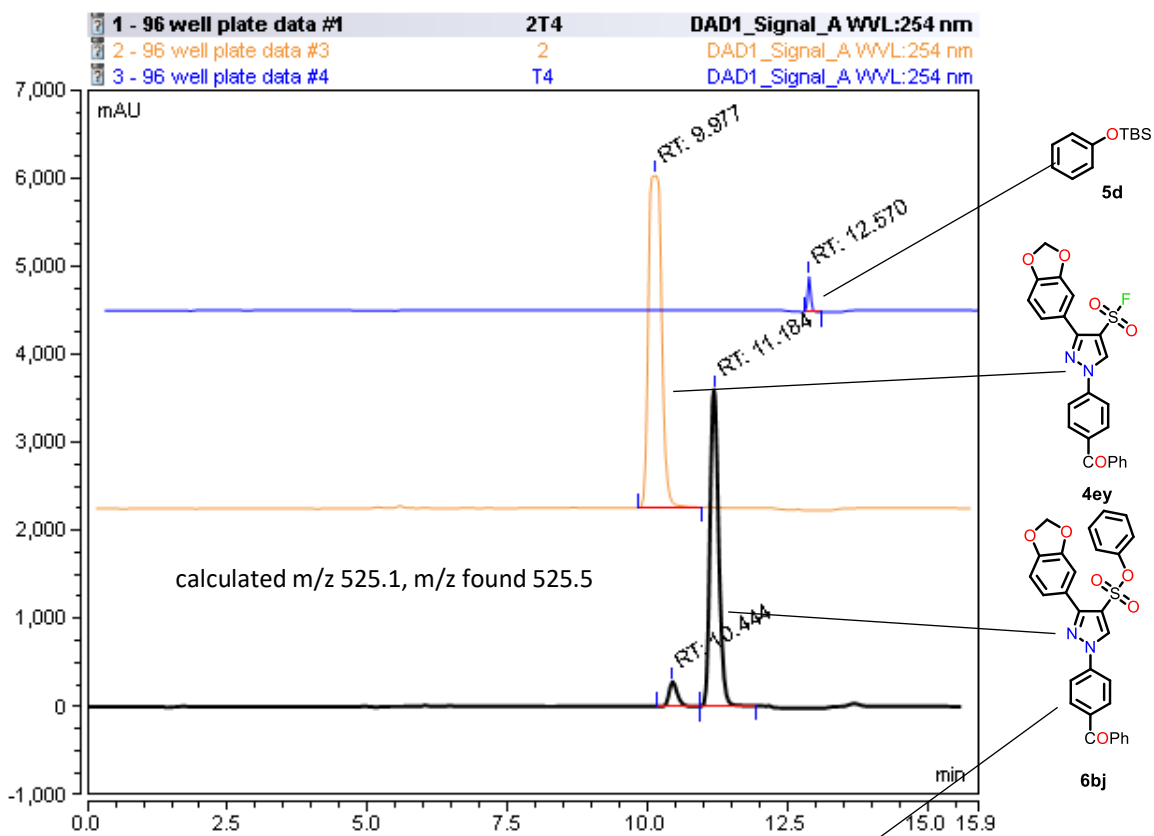
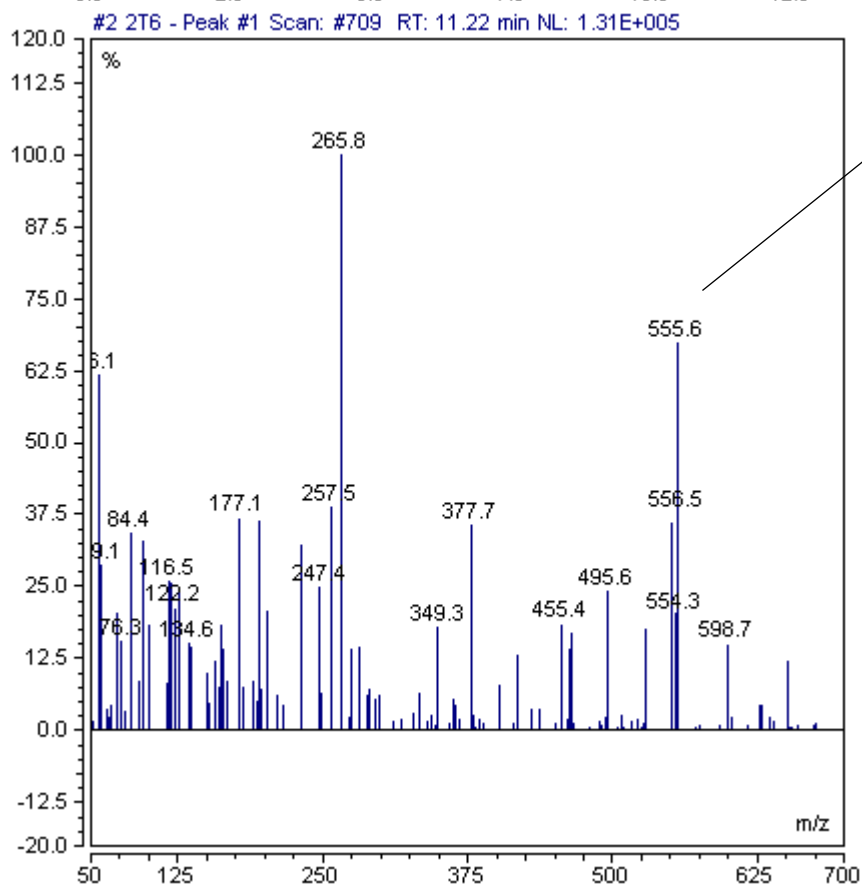
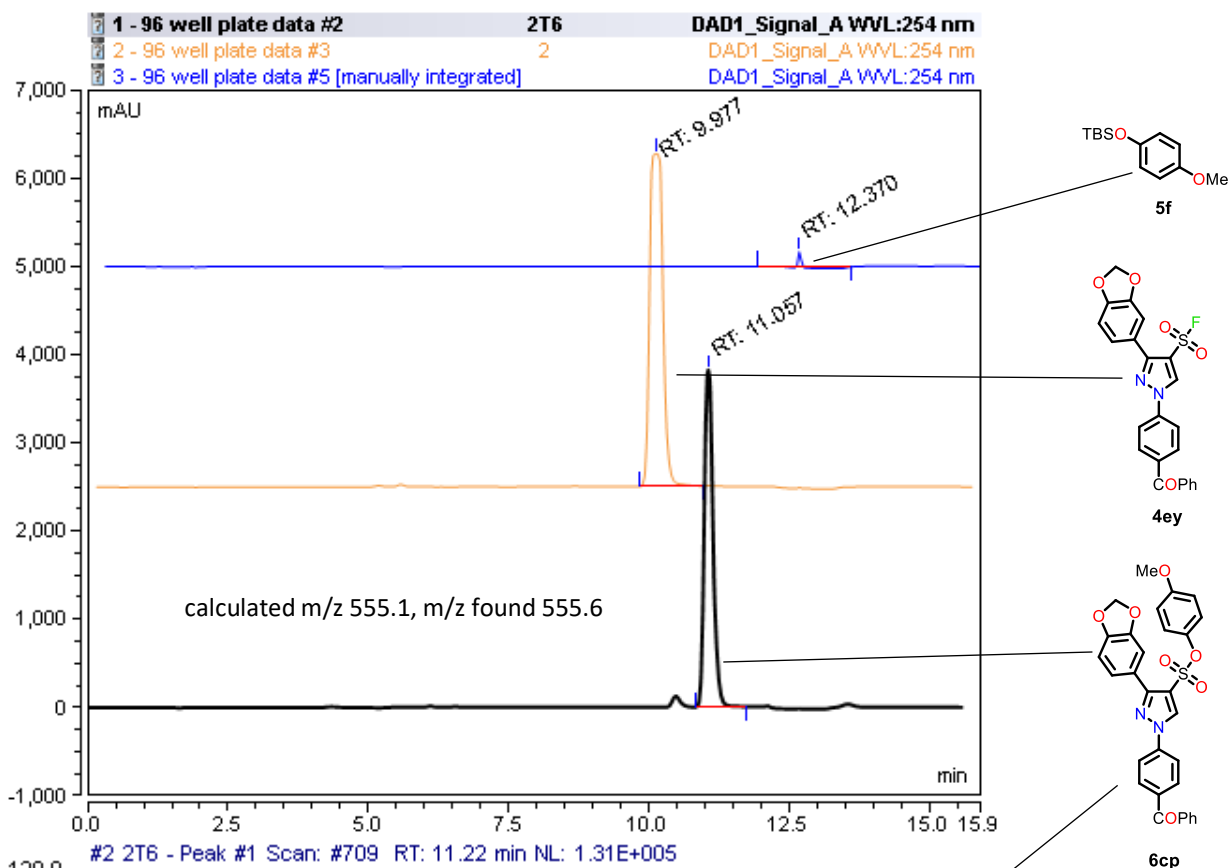
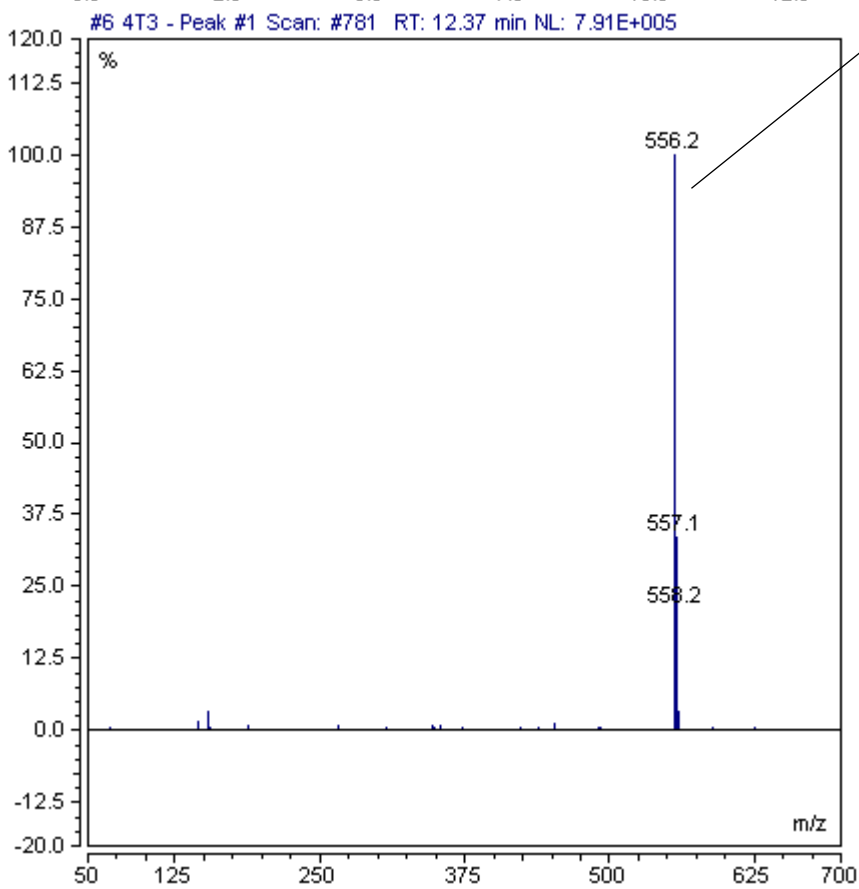
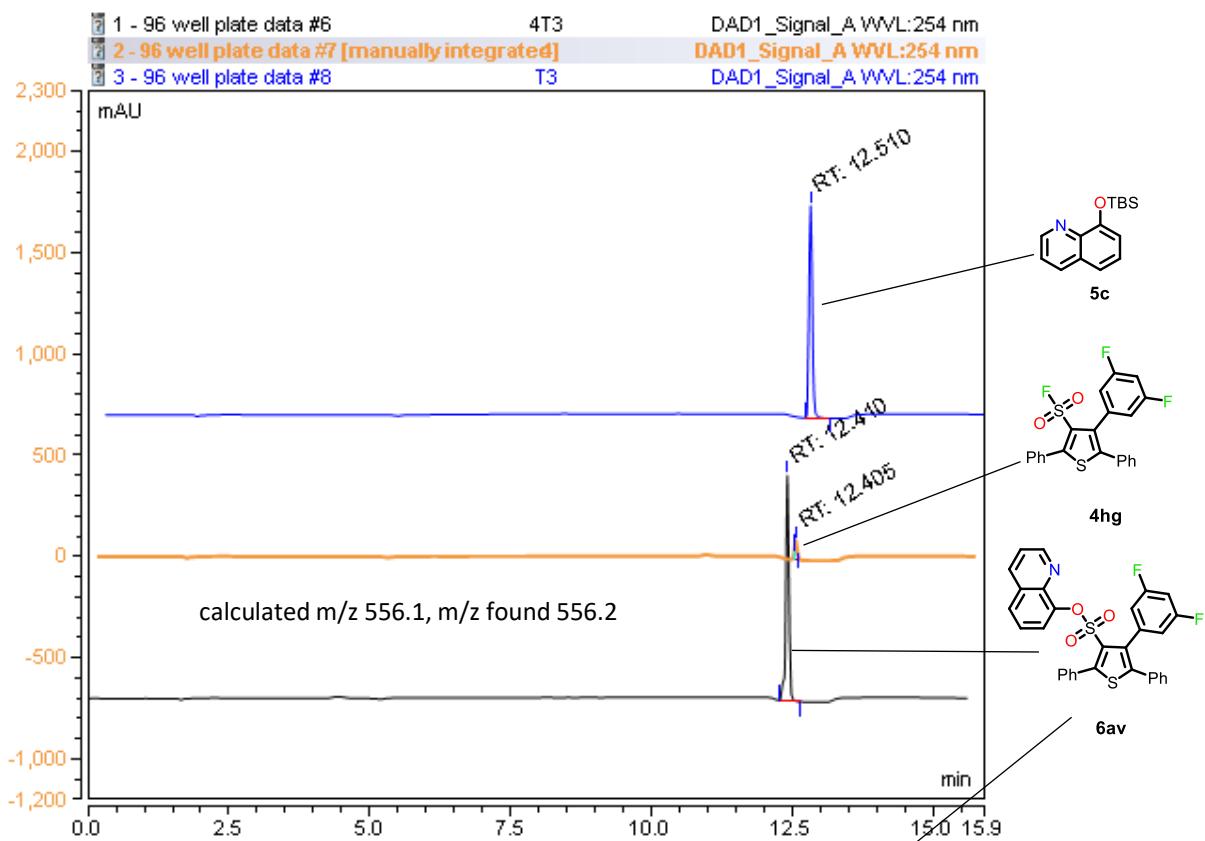
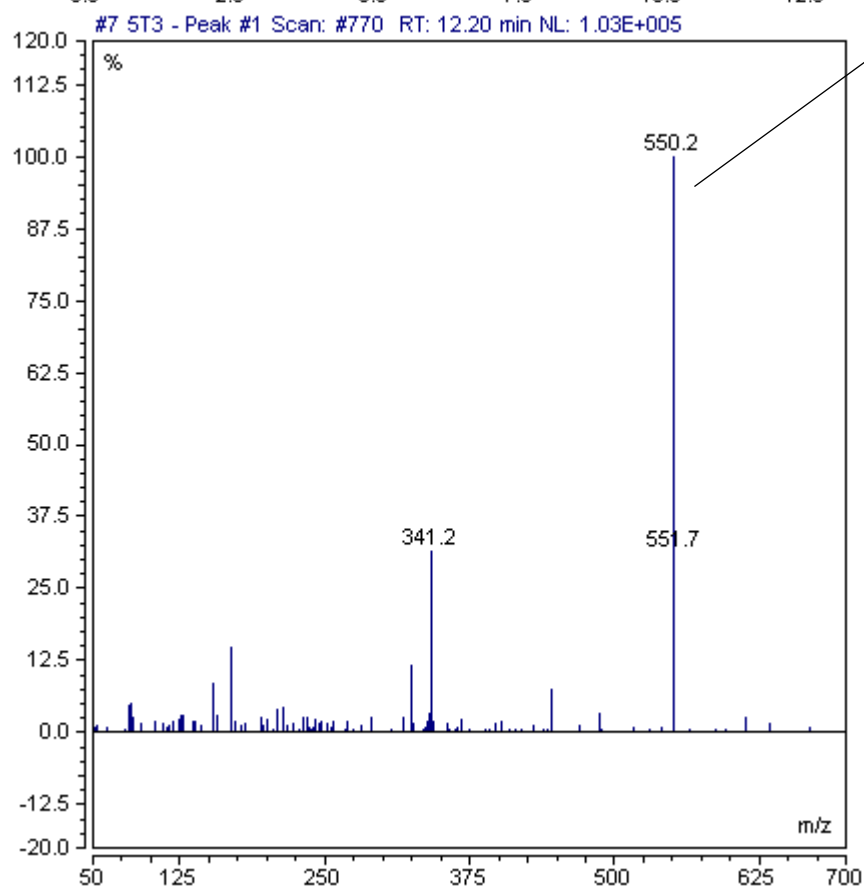
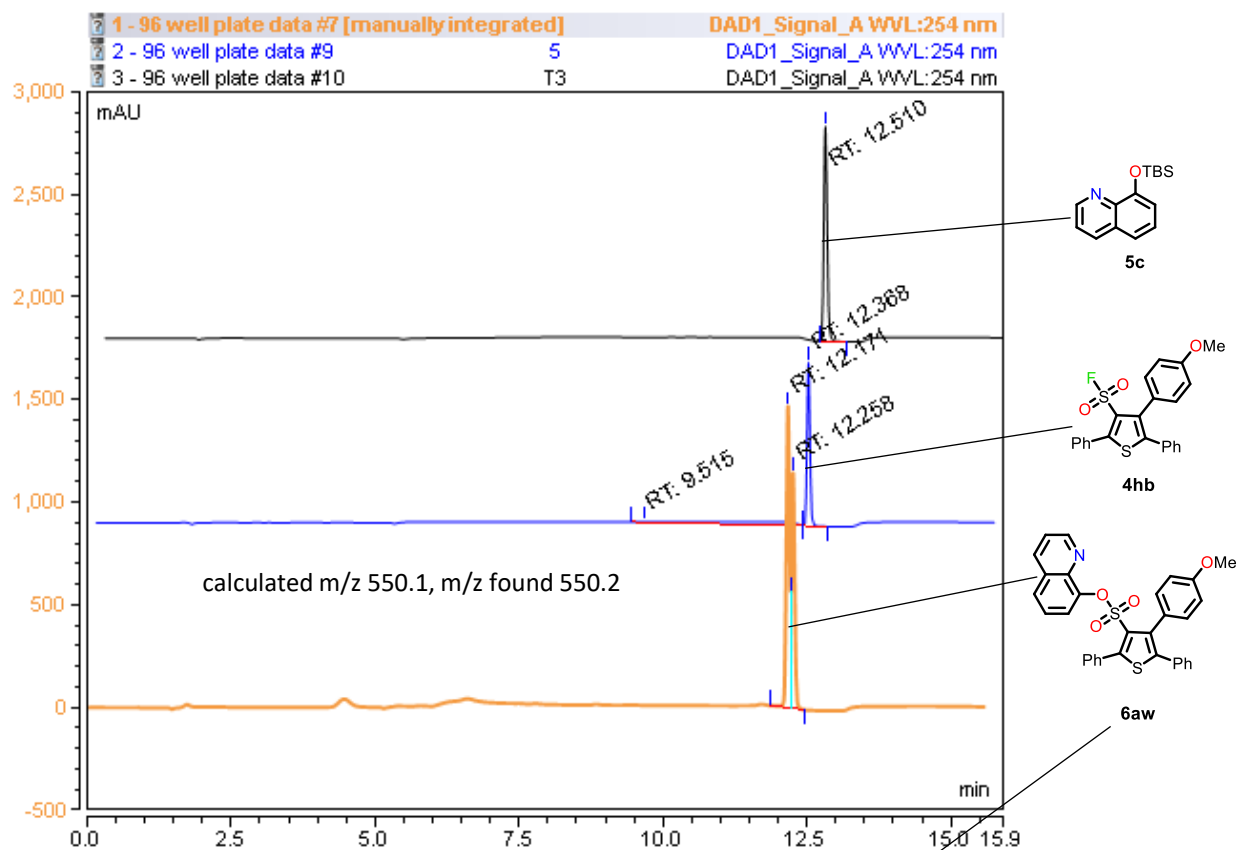


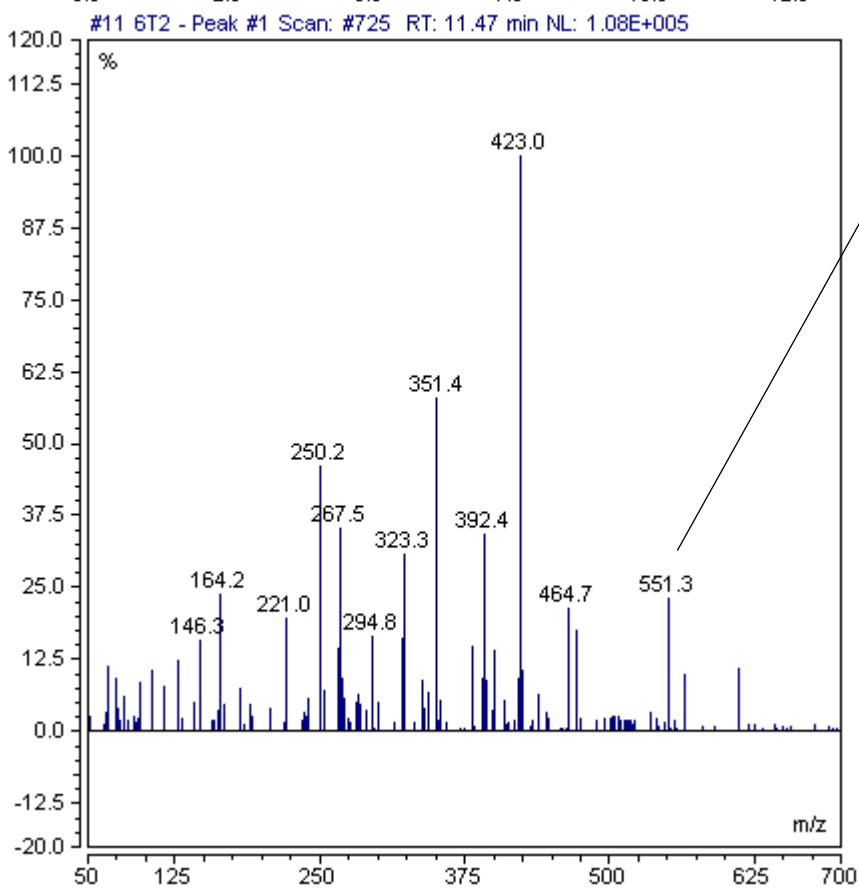
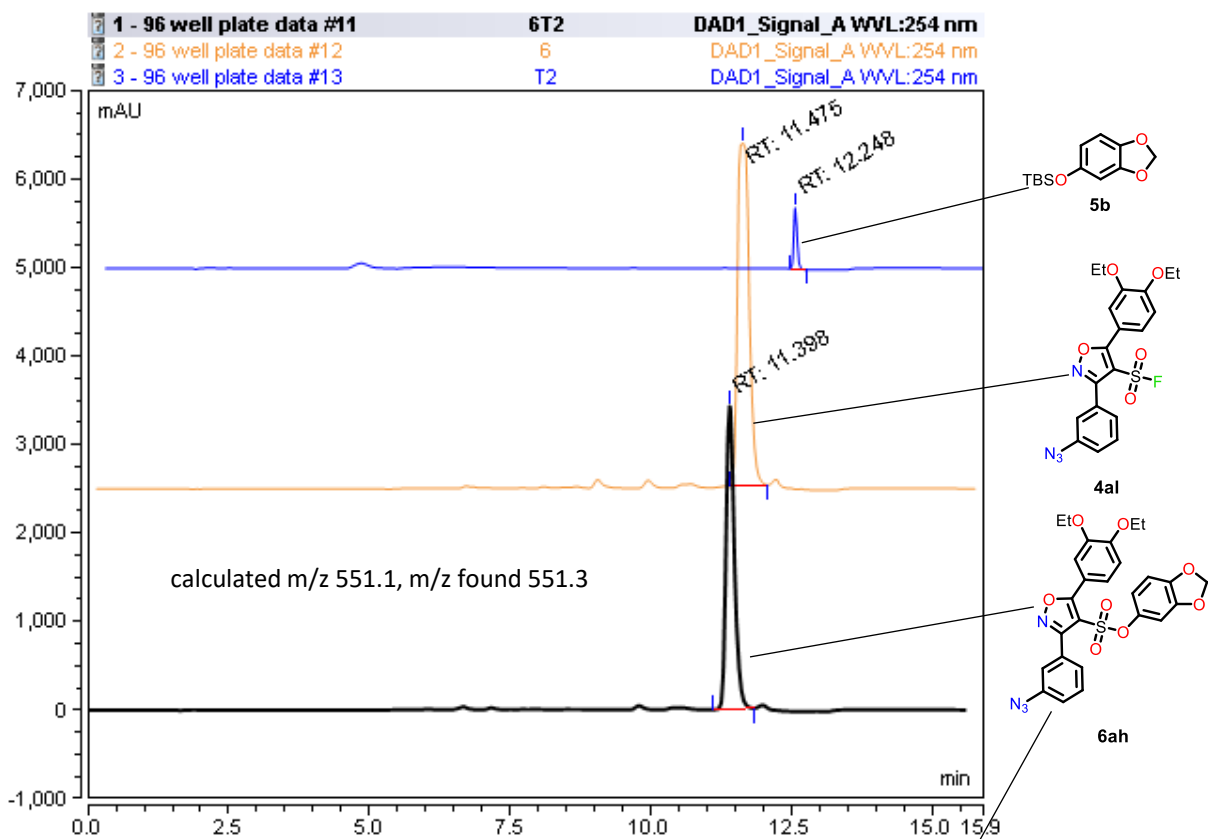
Figure S3. Representative TLC of SuFEx reactions performed in a 96 well plate format, Eluent: 15% EtOAc in petroleum ether

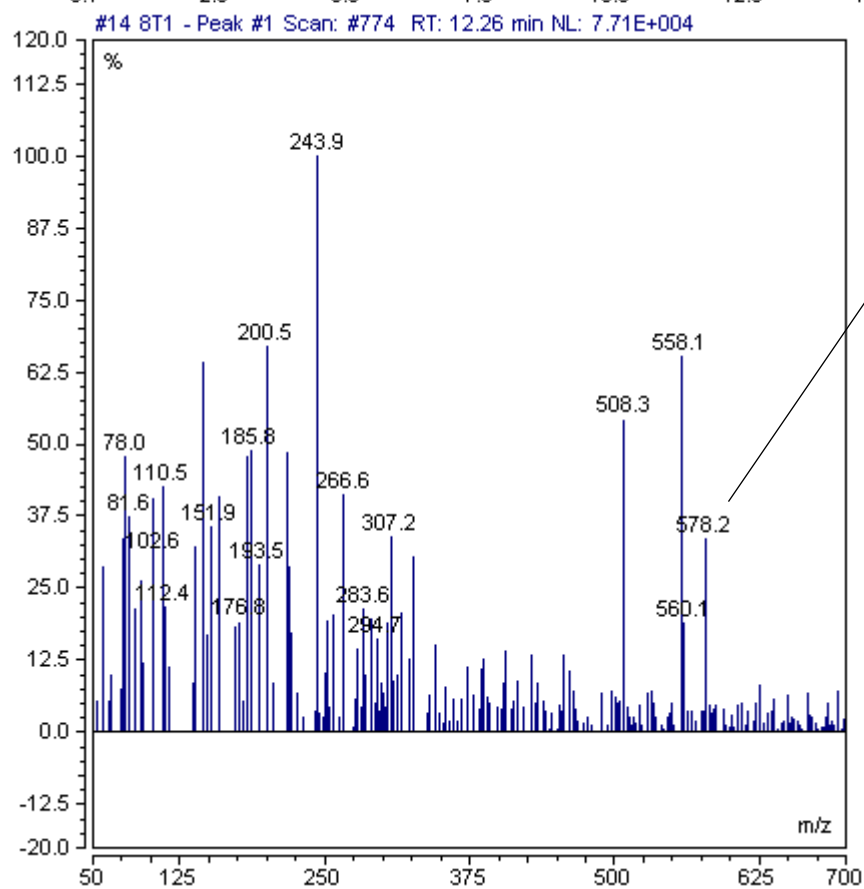
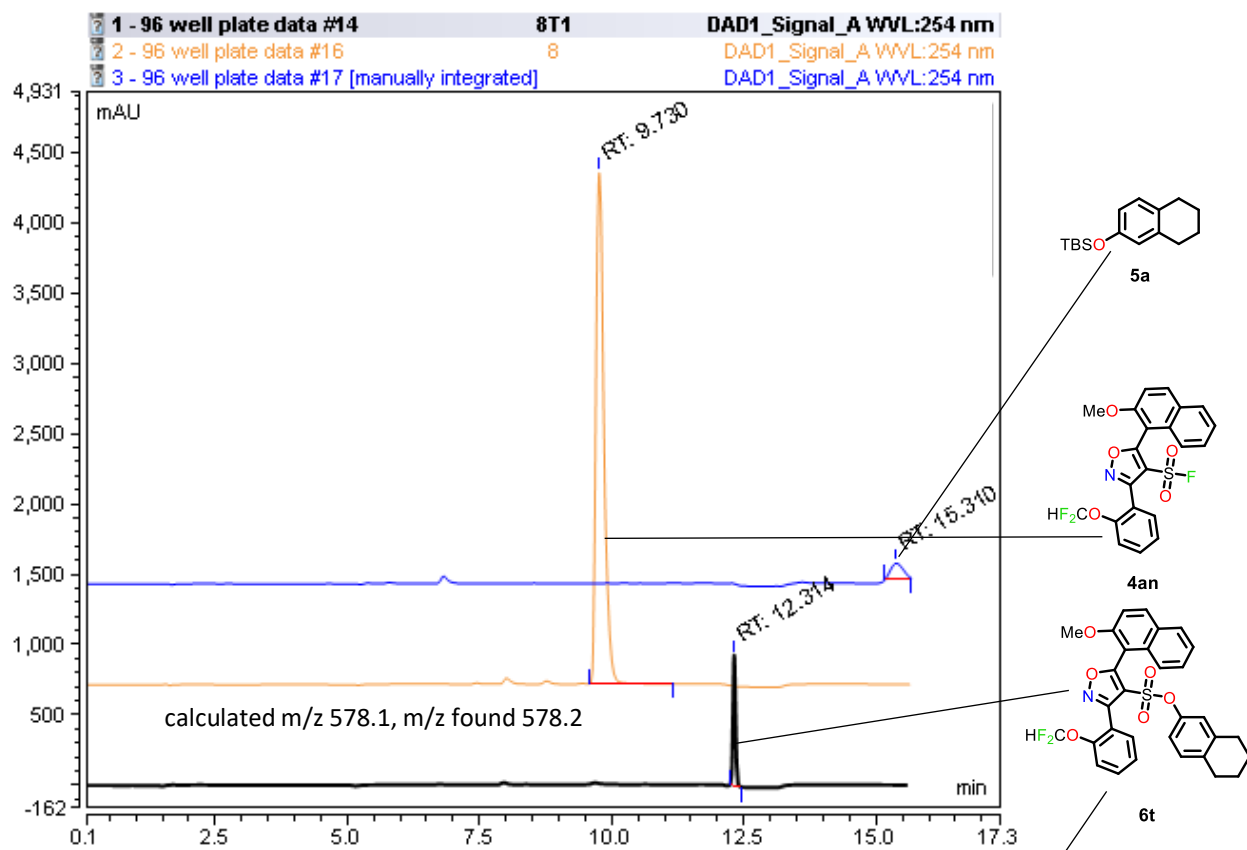


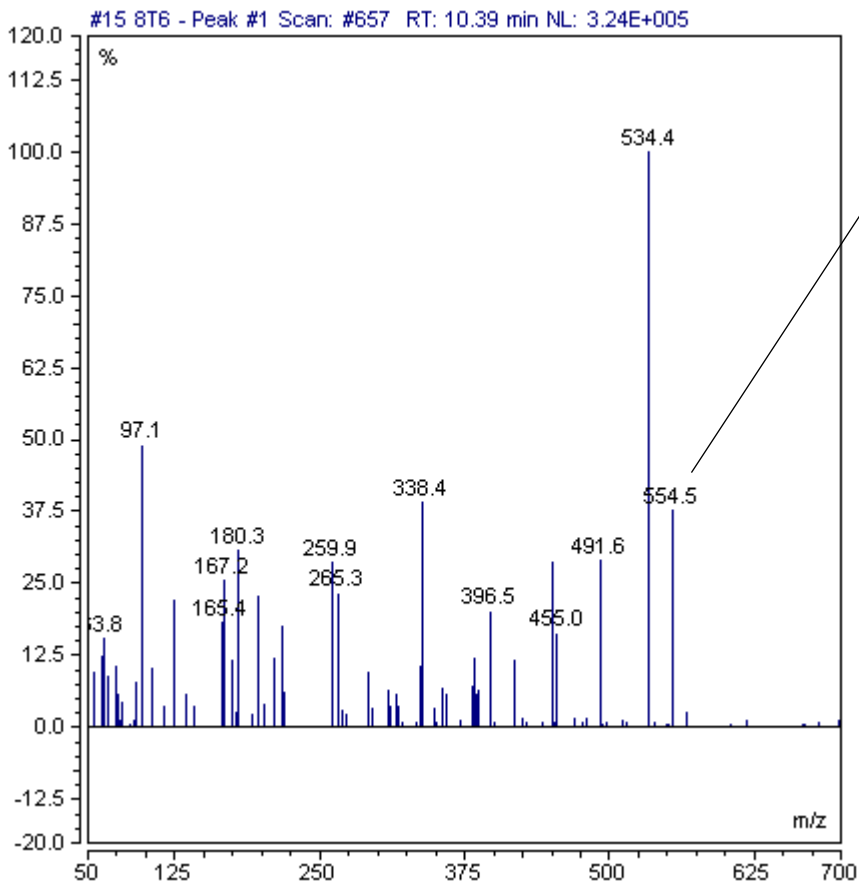
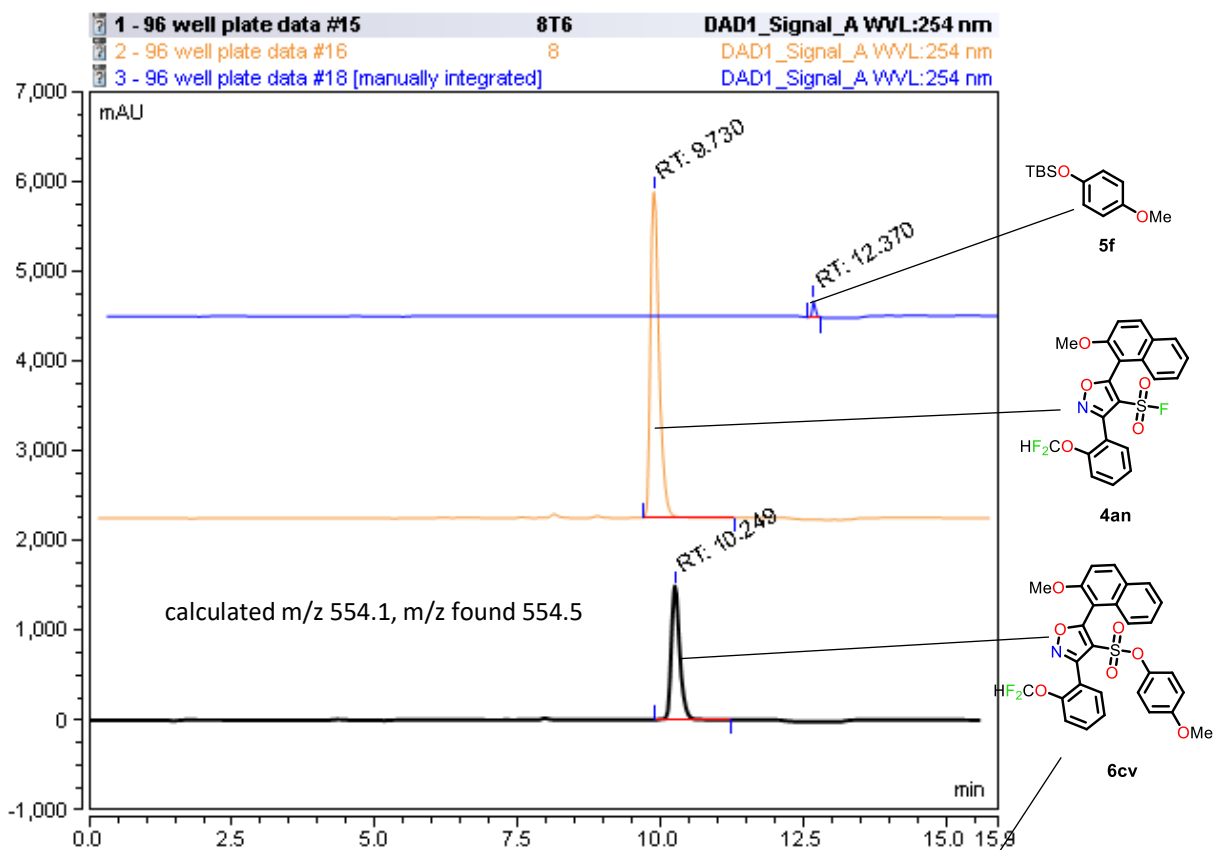


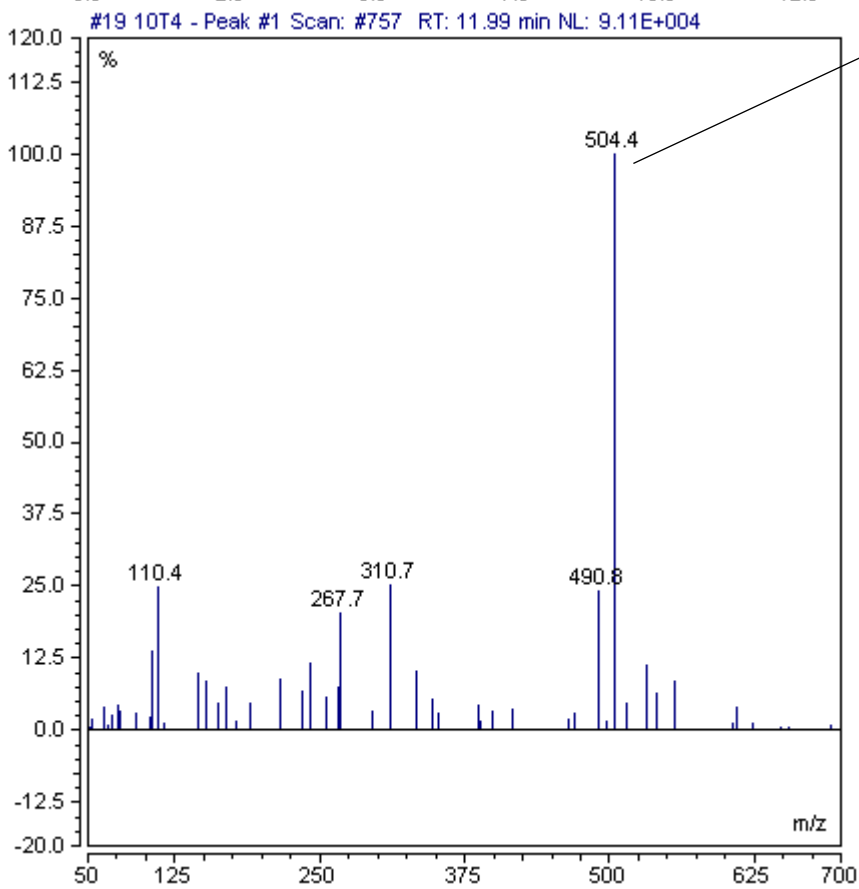
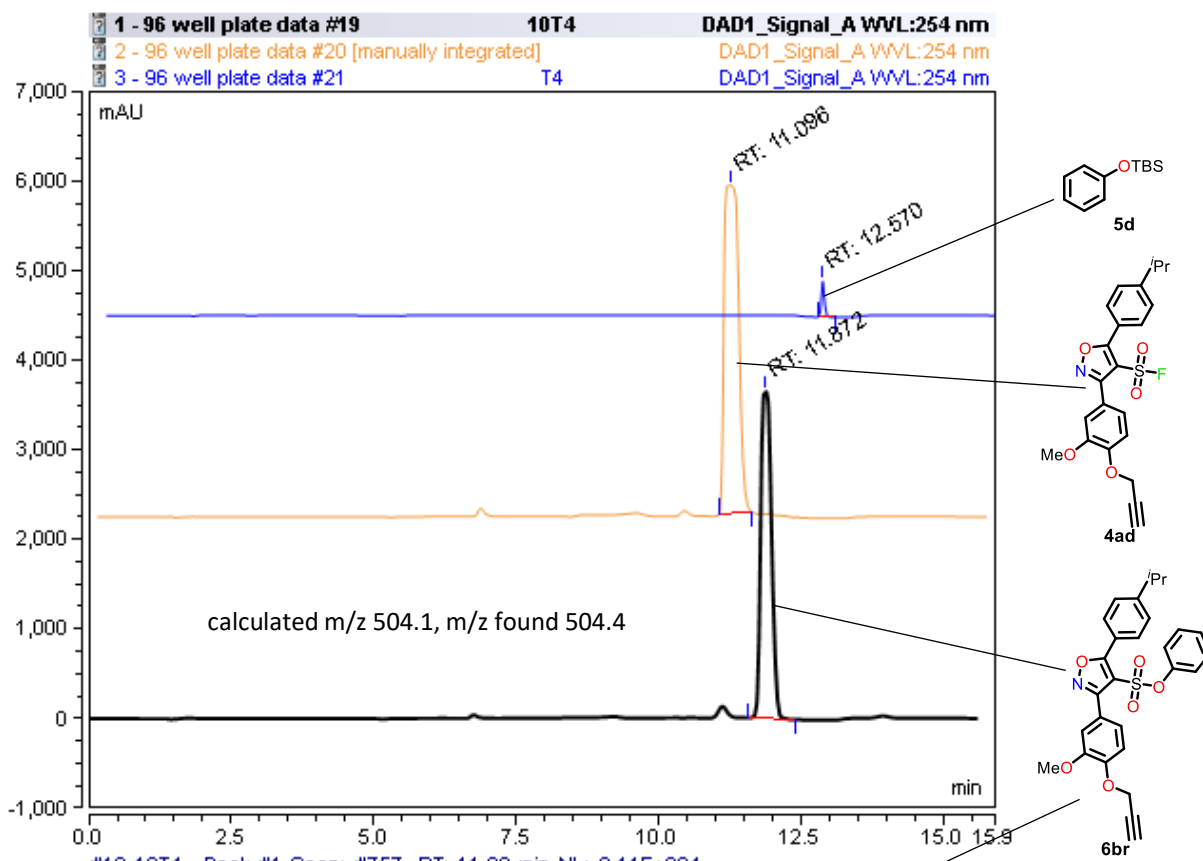


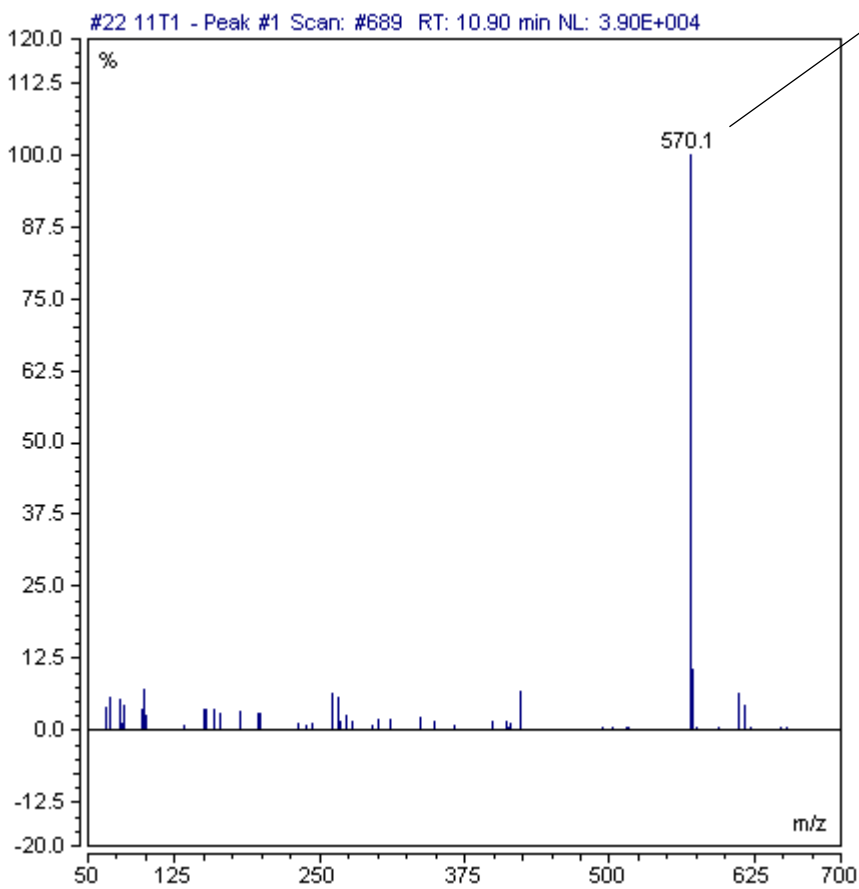
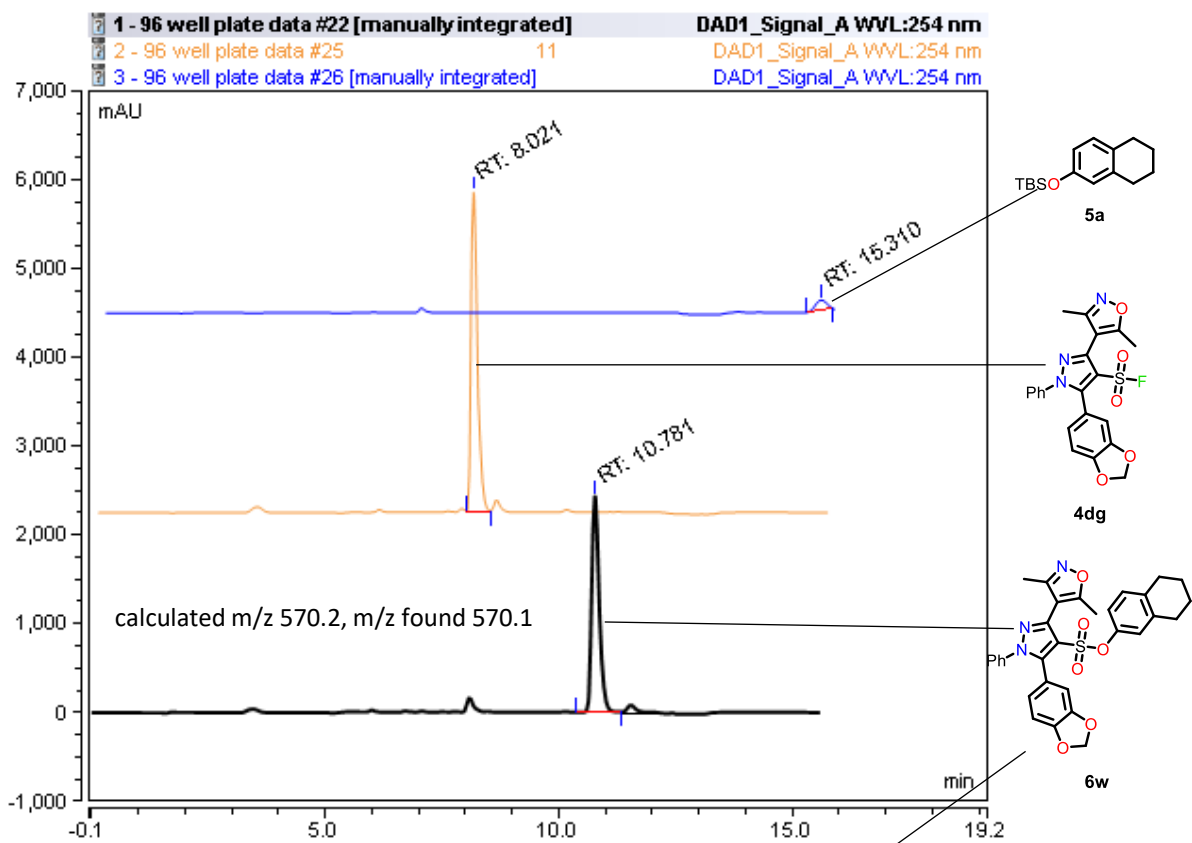


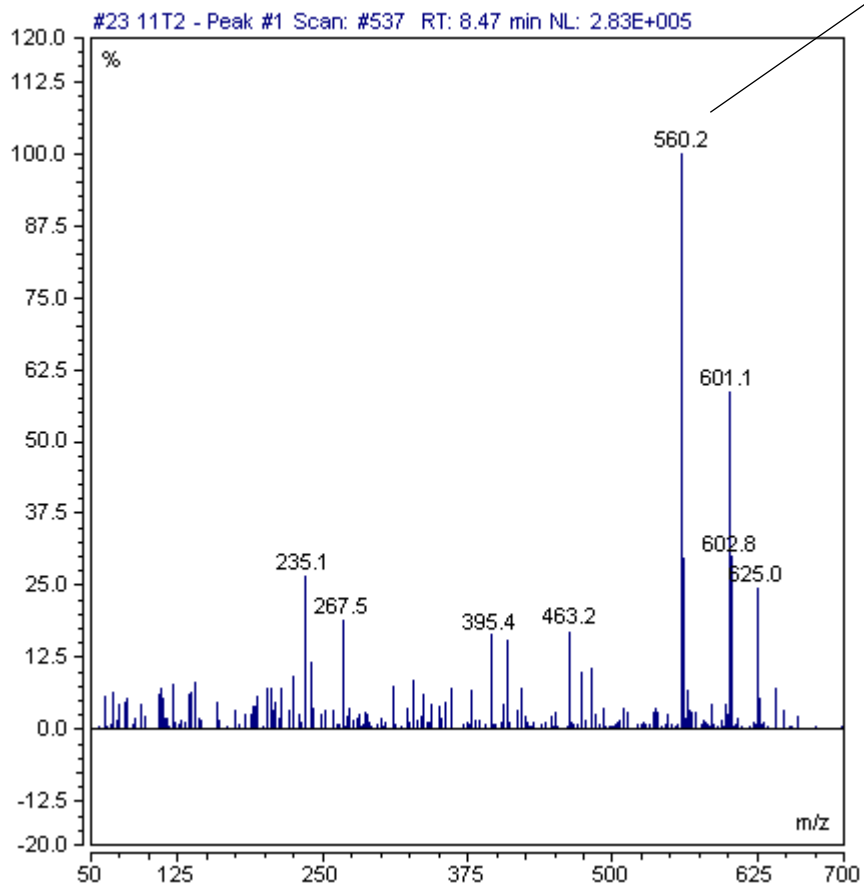
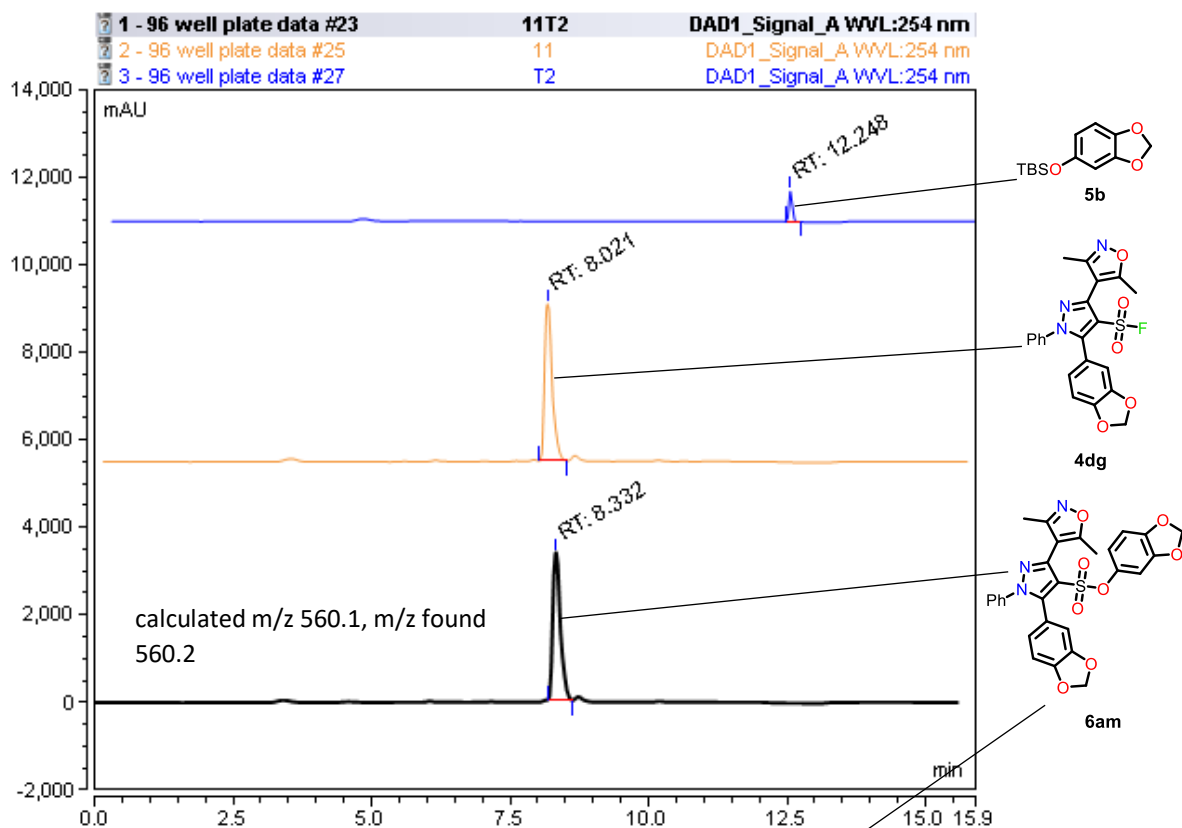


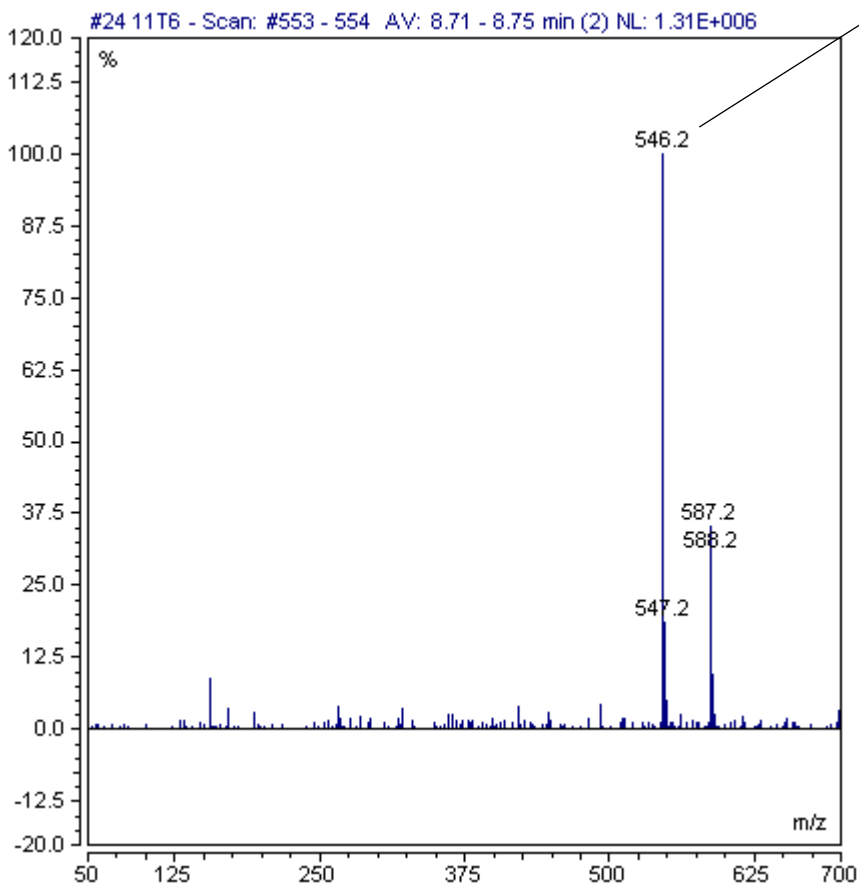
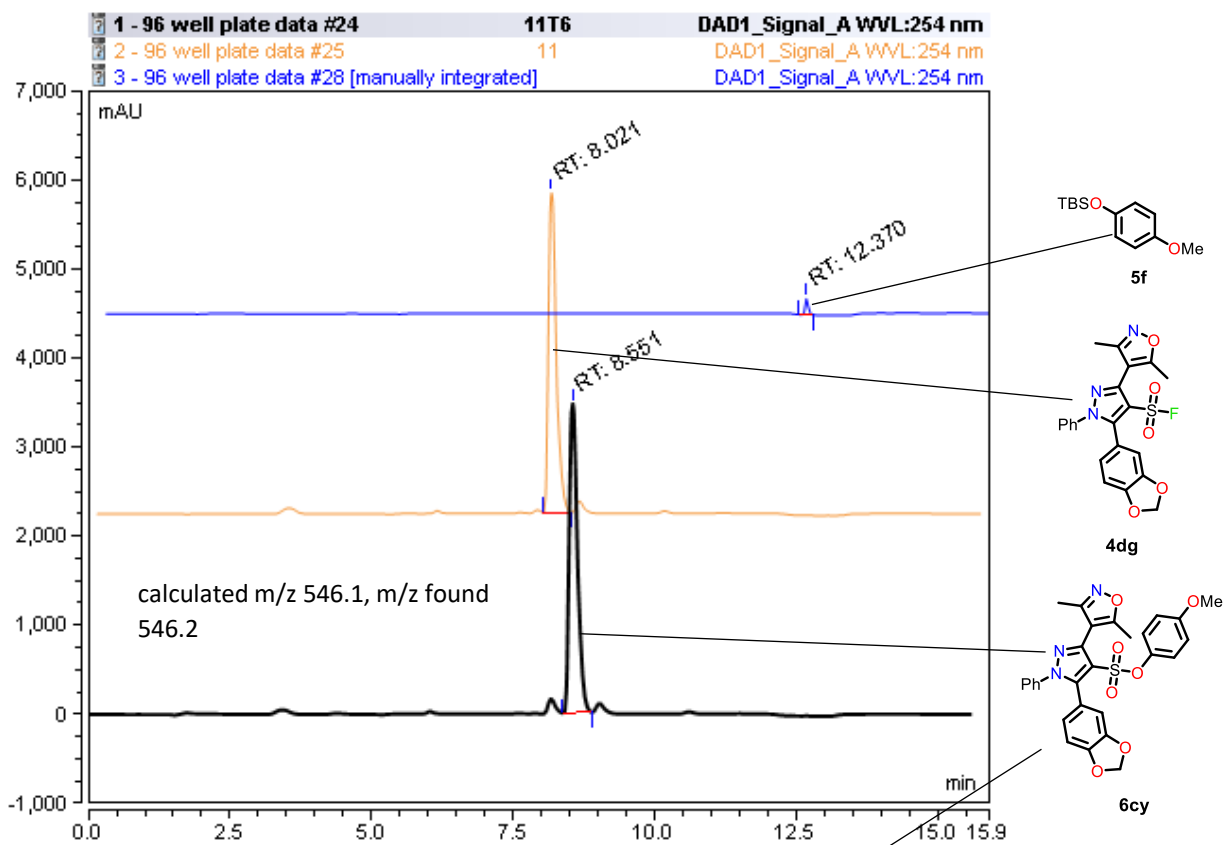


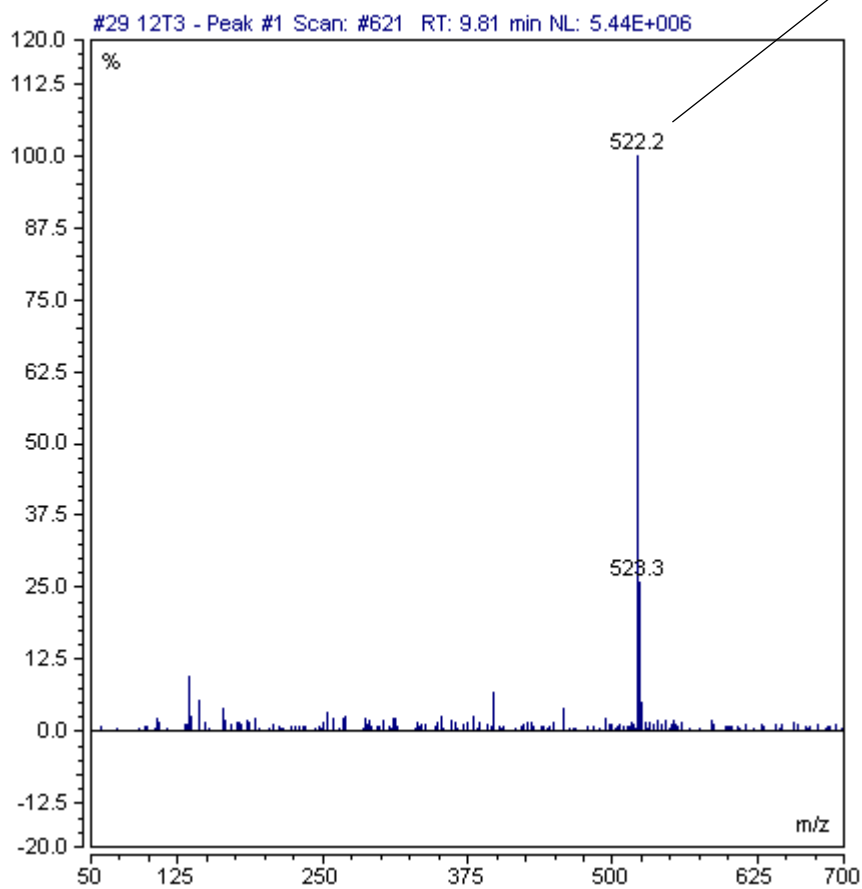
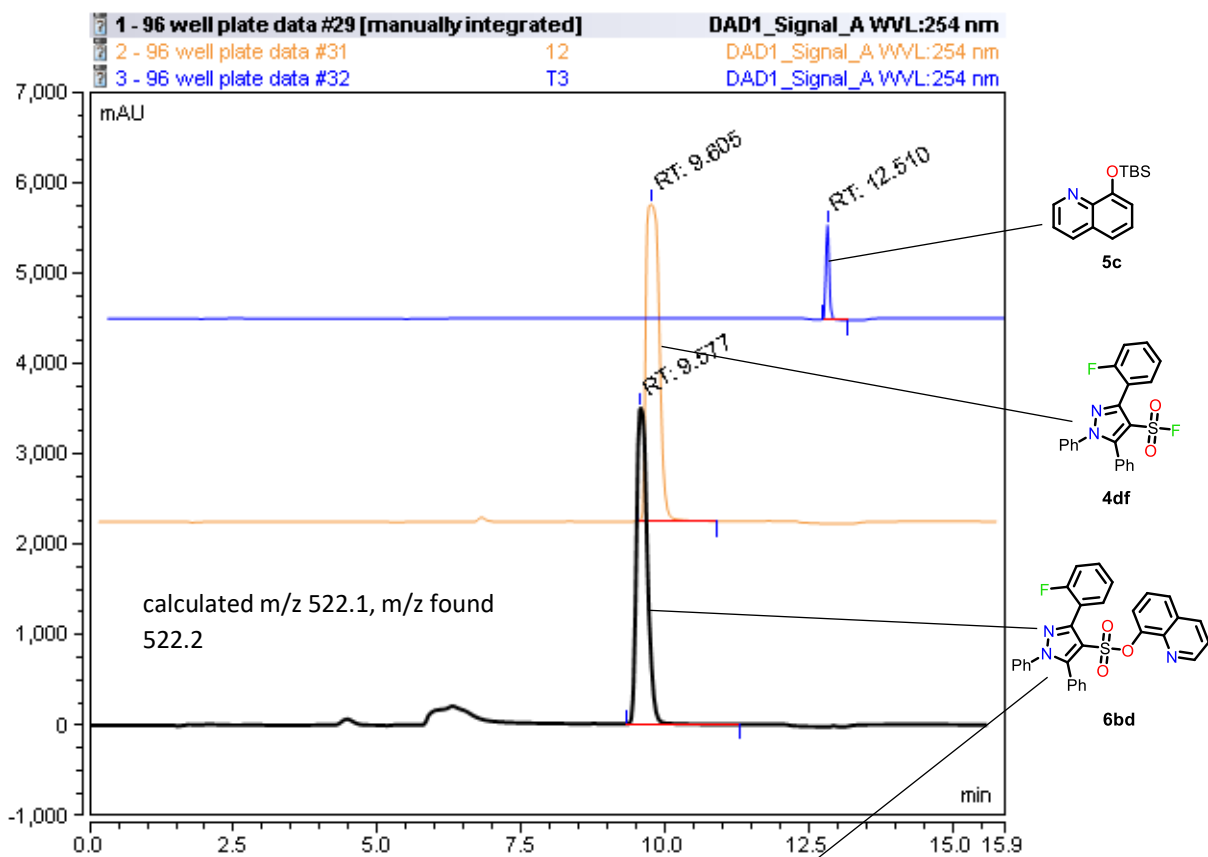


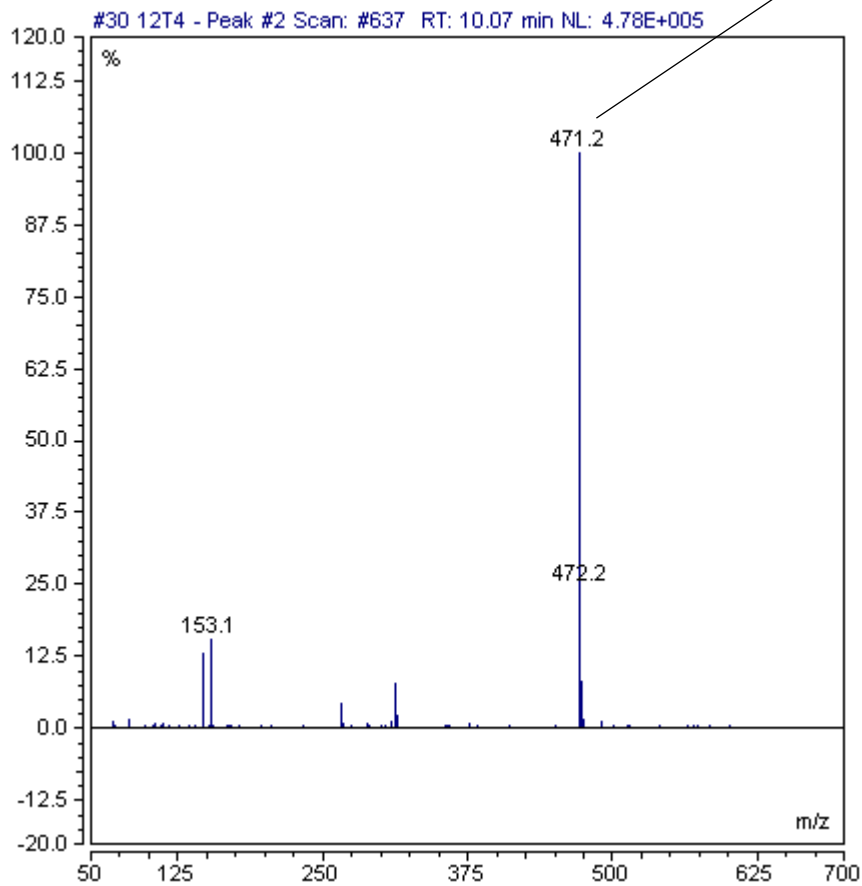
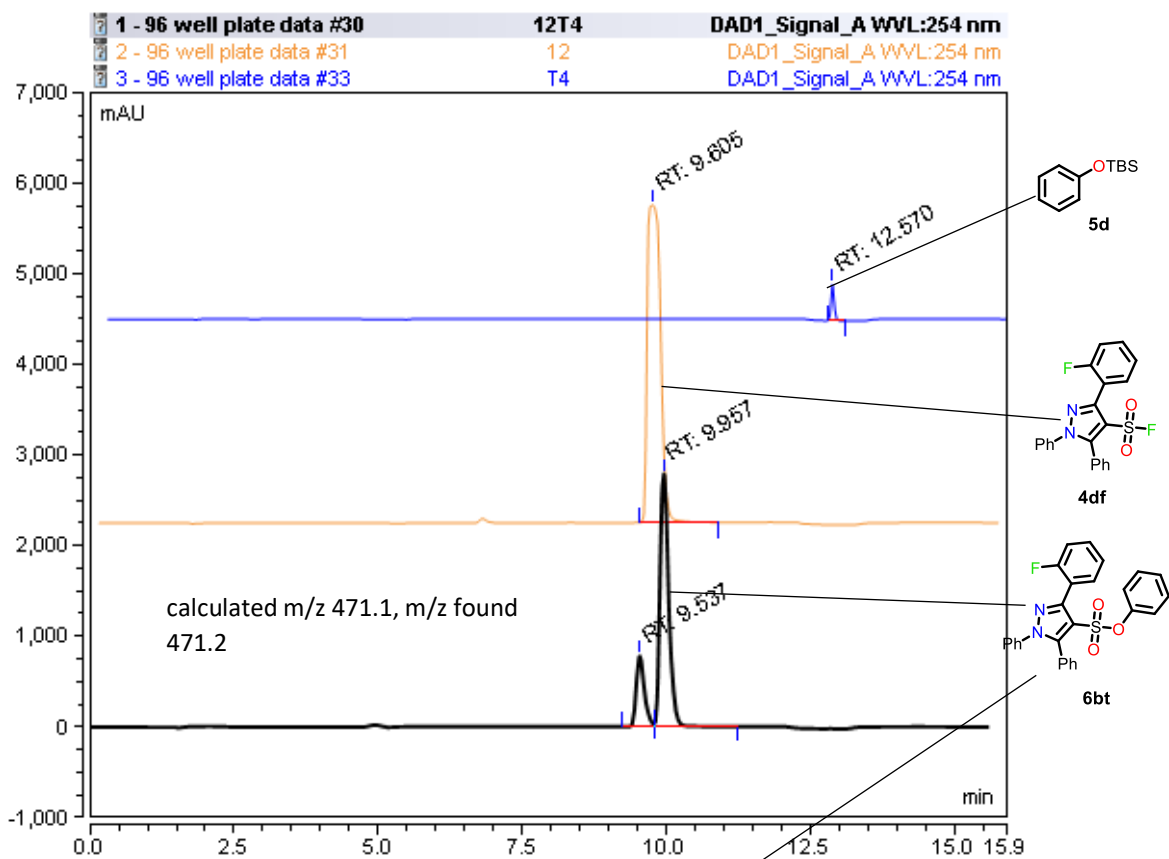


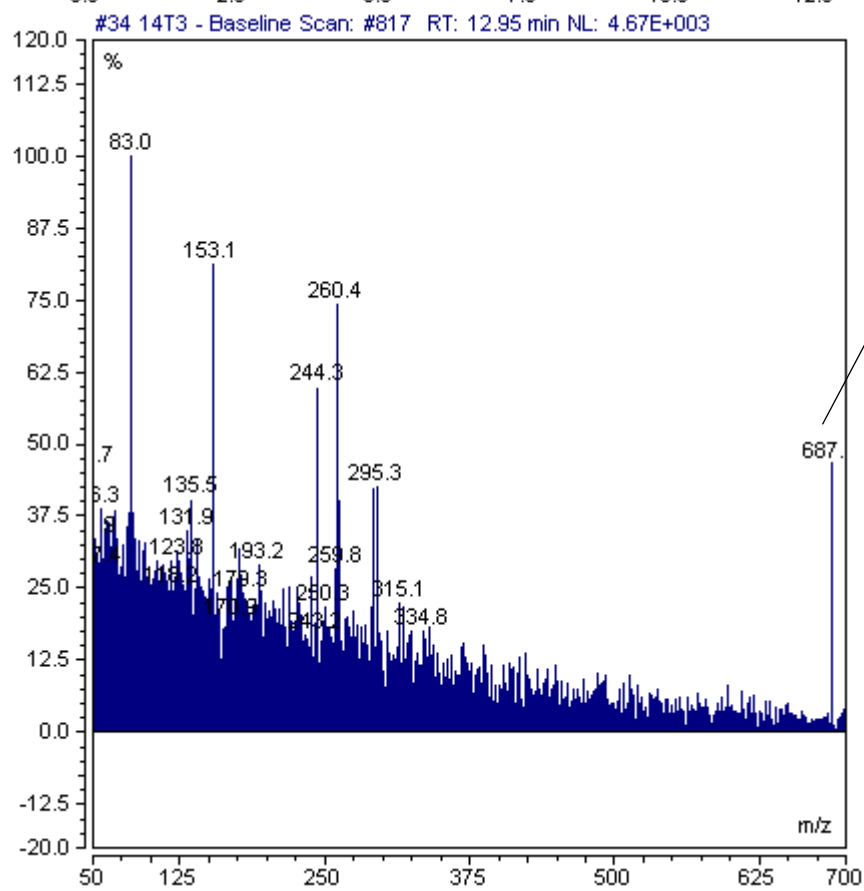
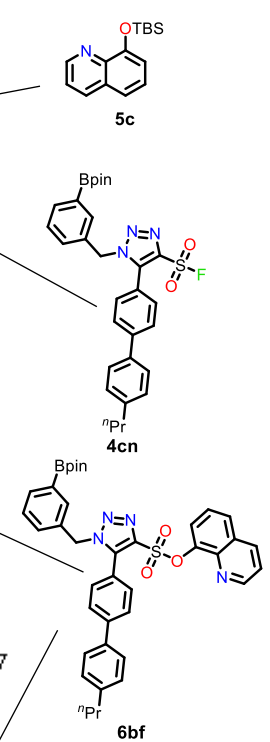
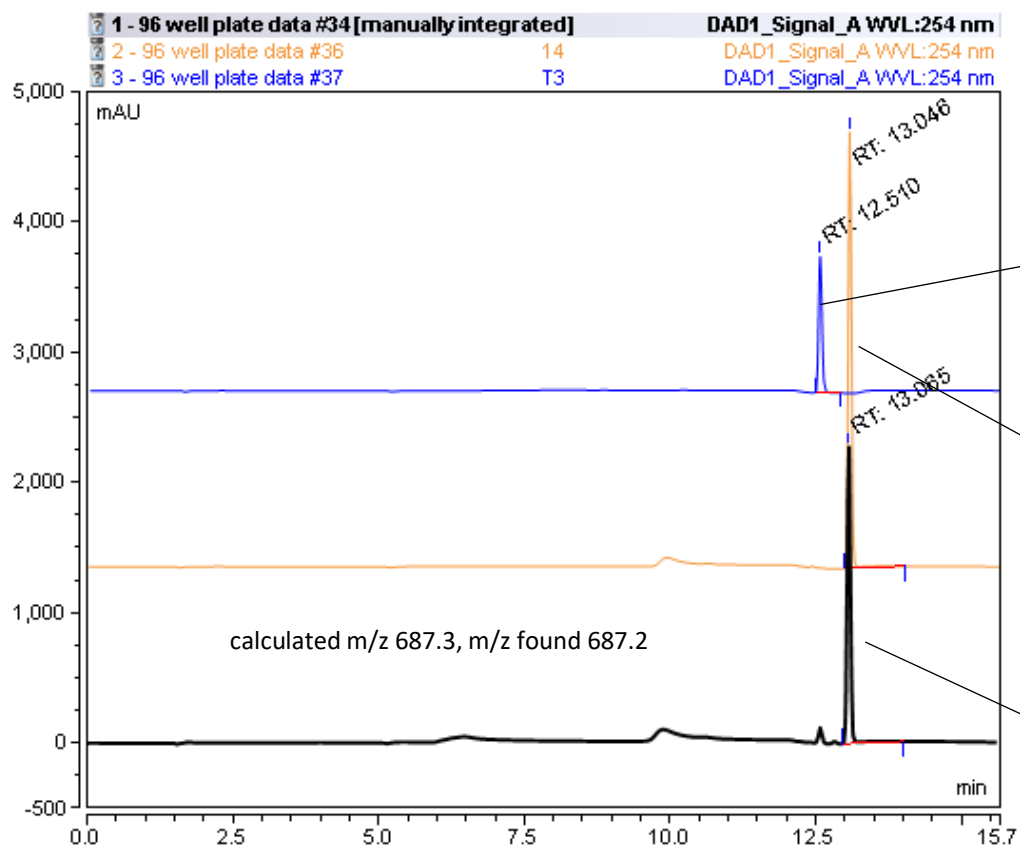


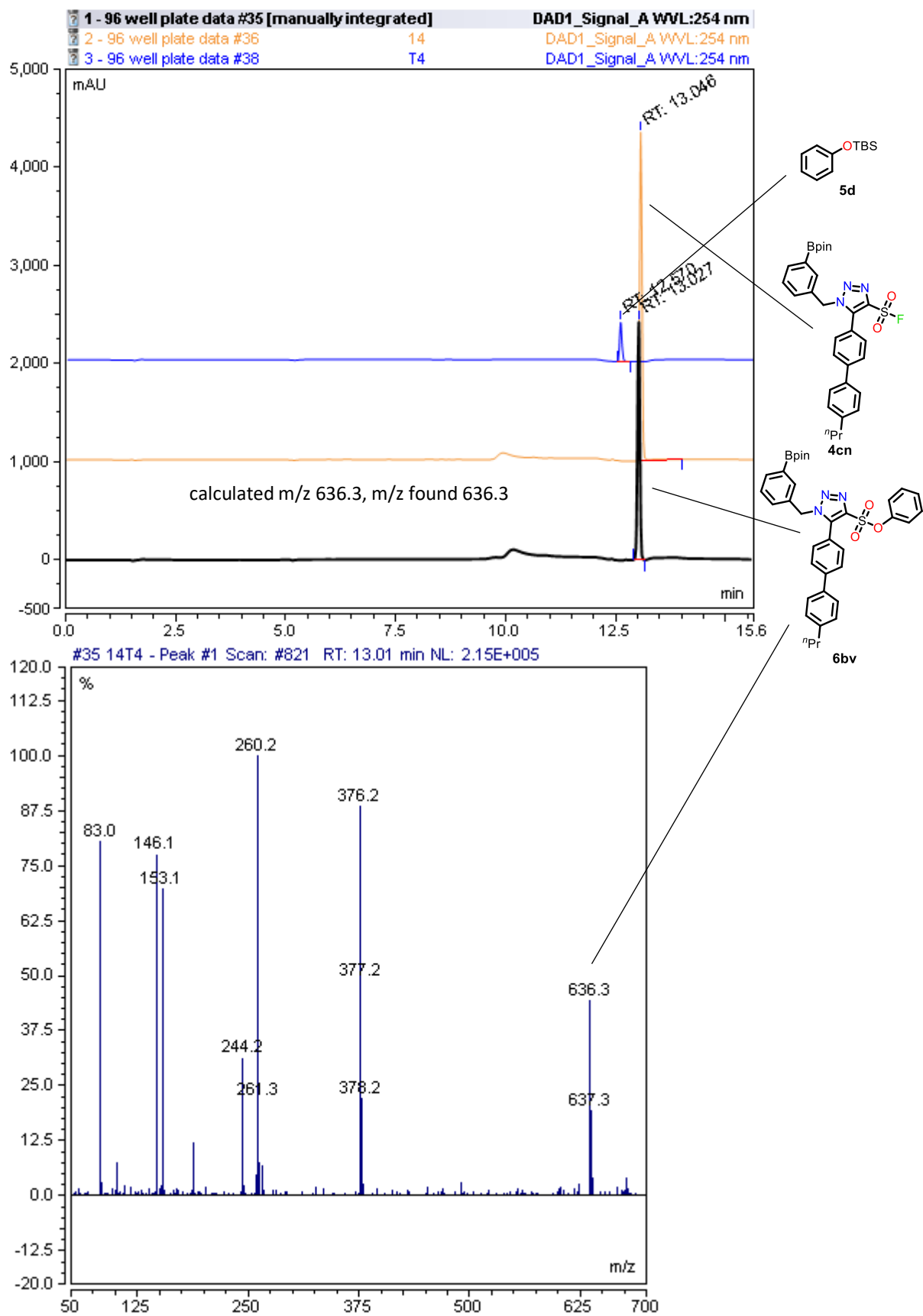


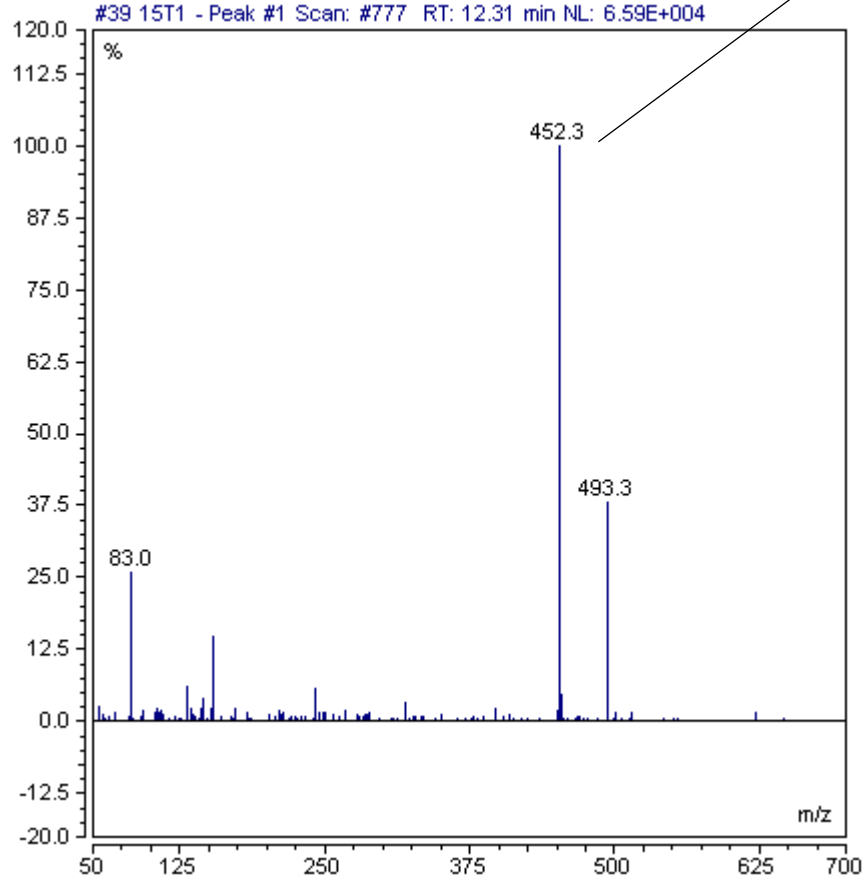
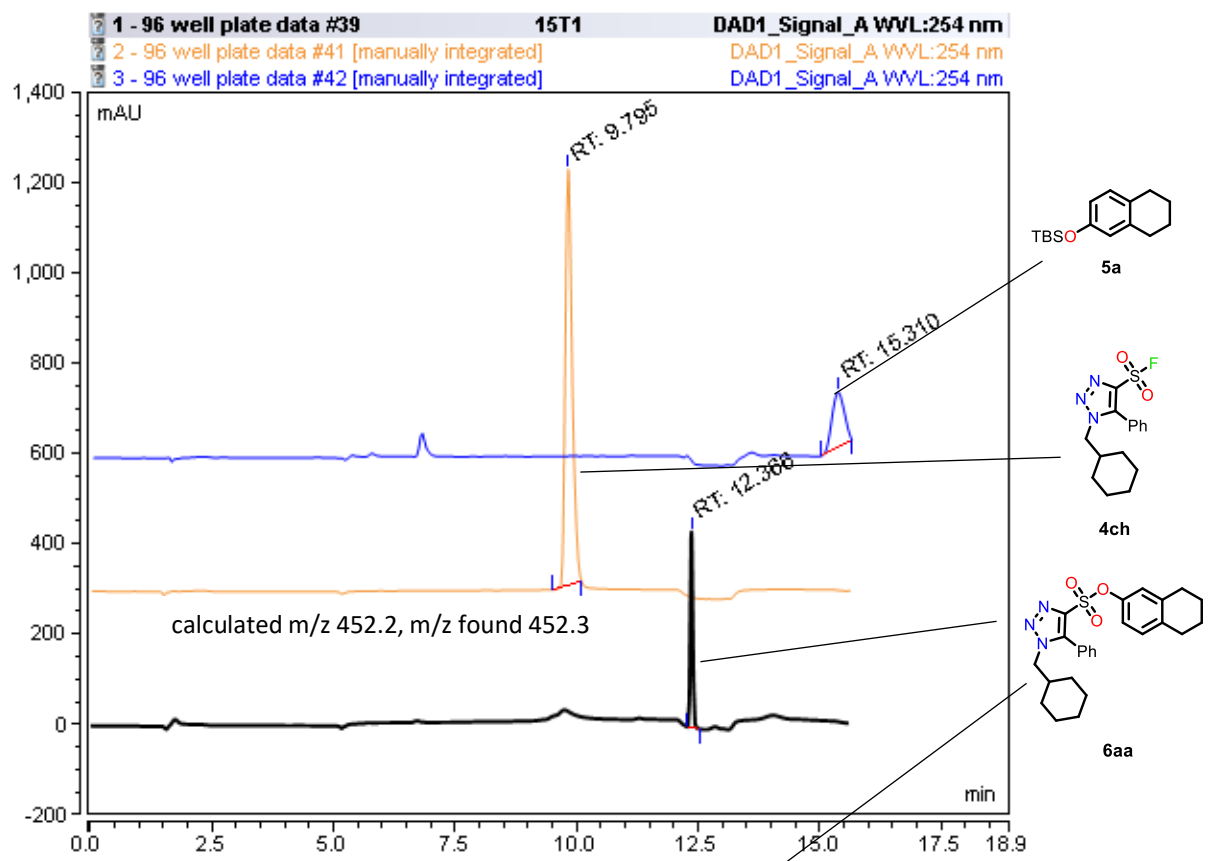


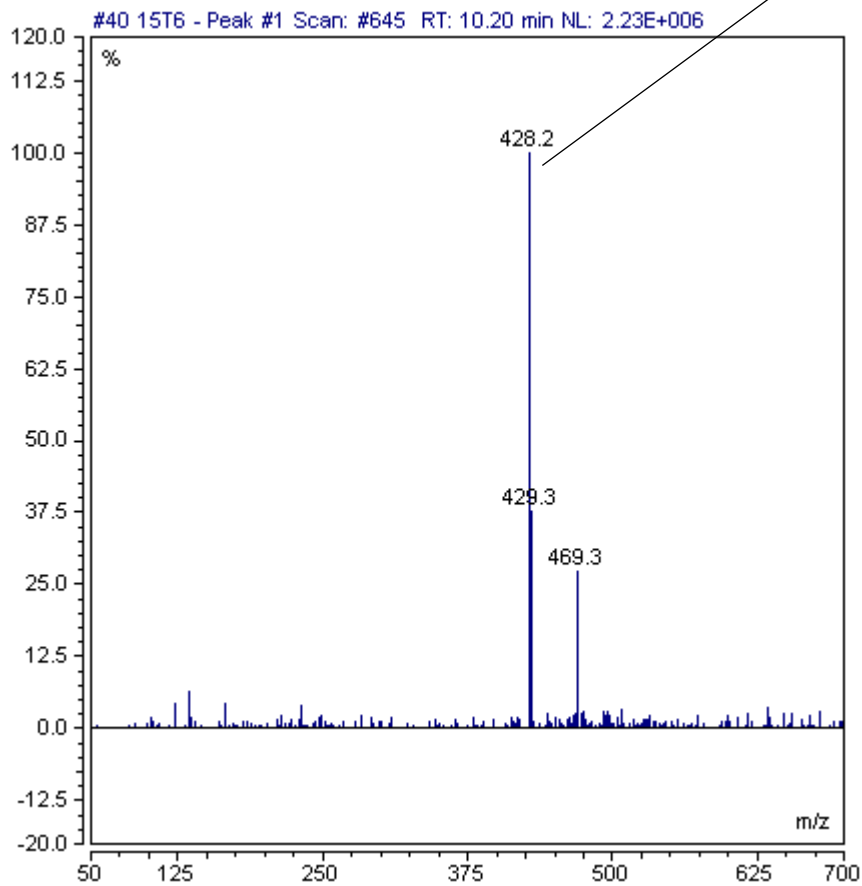
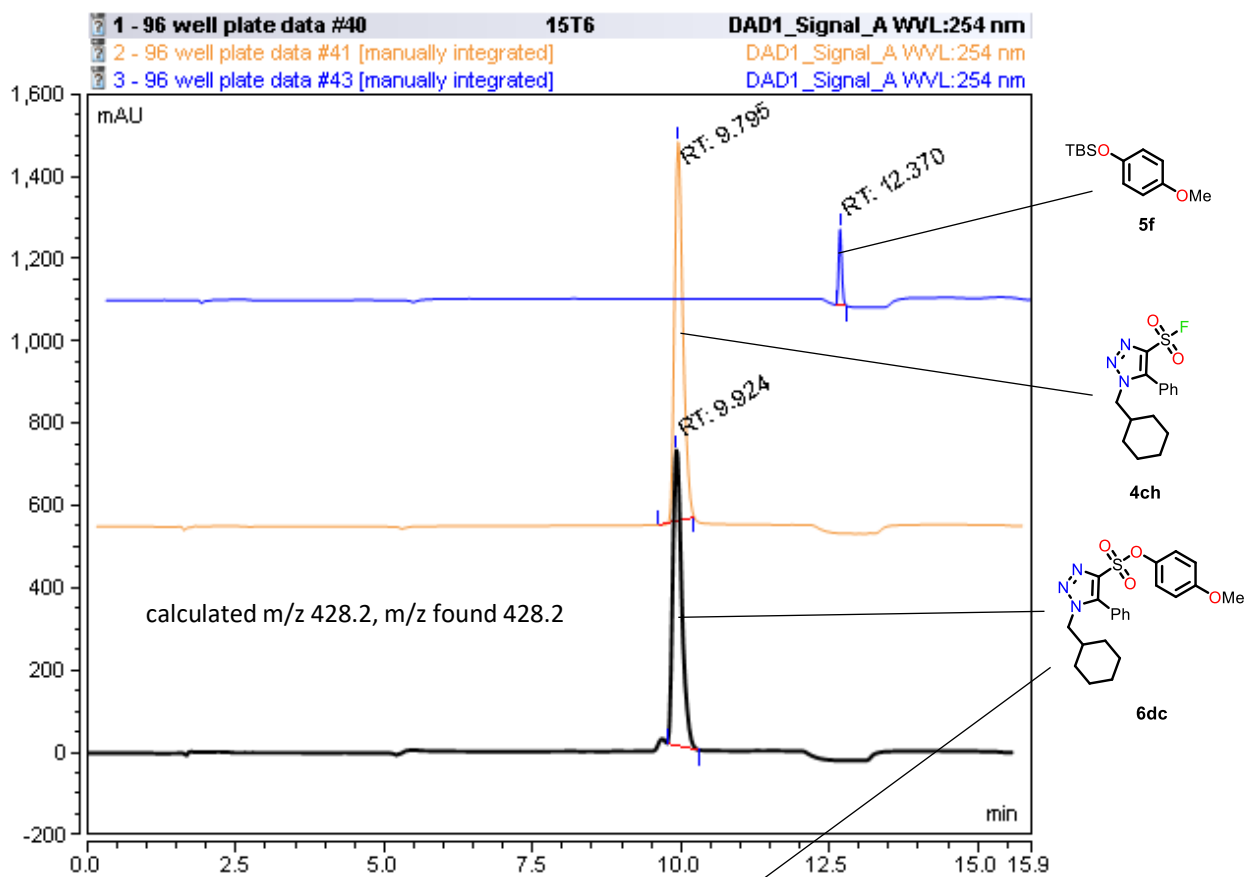


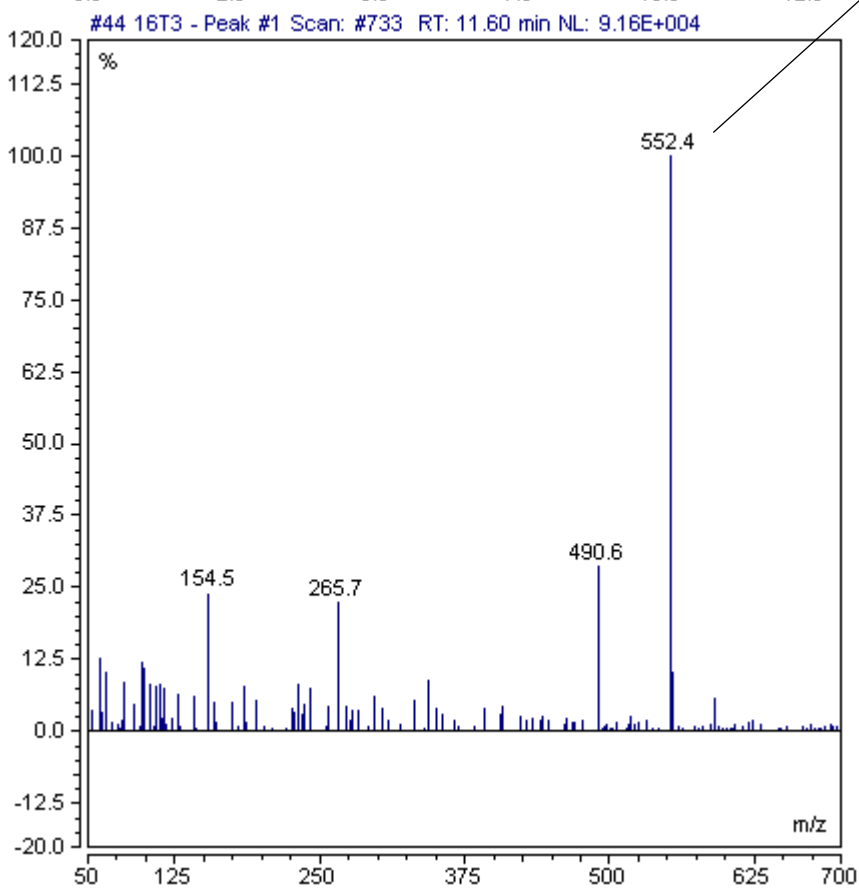
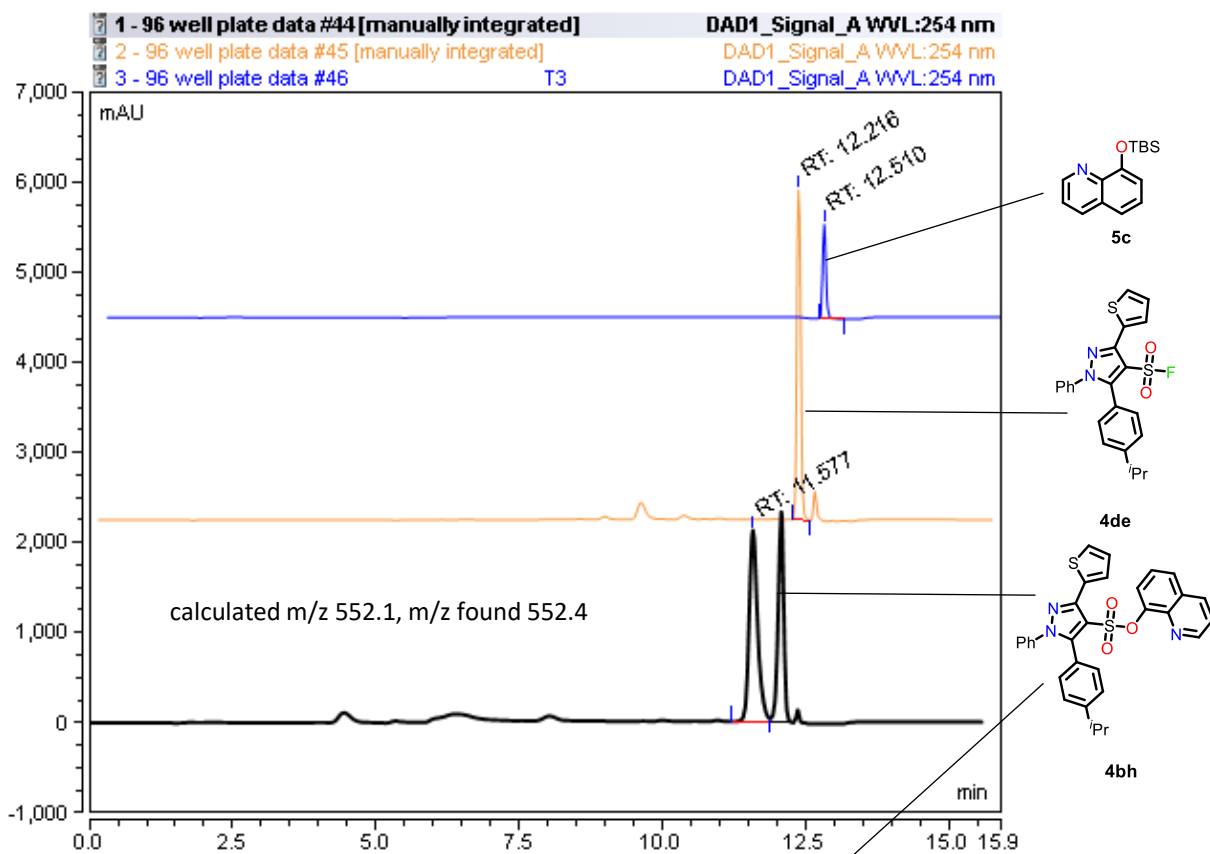












Experimental procedure for SuFEx reactivity comparison

Method

Stock solutions of heterocyclic sulfonyl fluorides (5.00 μmol) or Diels-Alder adducts (5.00 μmol), TBS ether (5.00 μmol) and DBU (100 nmol) in CD_3CN were added to a 96 well plate totalling a volume of 500 μL in each well. The plate was agitated at 310 rpm at r.t. for 16 h. The reaction mixture was analysed directly by ^{19}F qNMR. Integrals extend $\pm 32\times$ the half height width of the peak except for the competition between compounds 1 and 8 which extends $\pm 15\times$ the half height width of the peak to avoid peak overlap. The ratio of starting materials remaining was used to determine their relative reactivity.

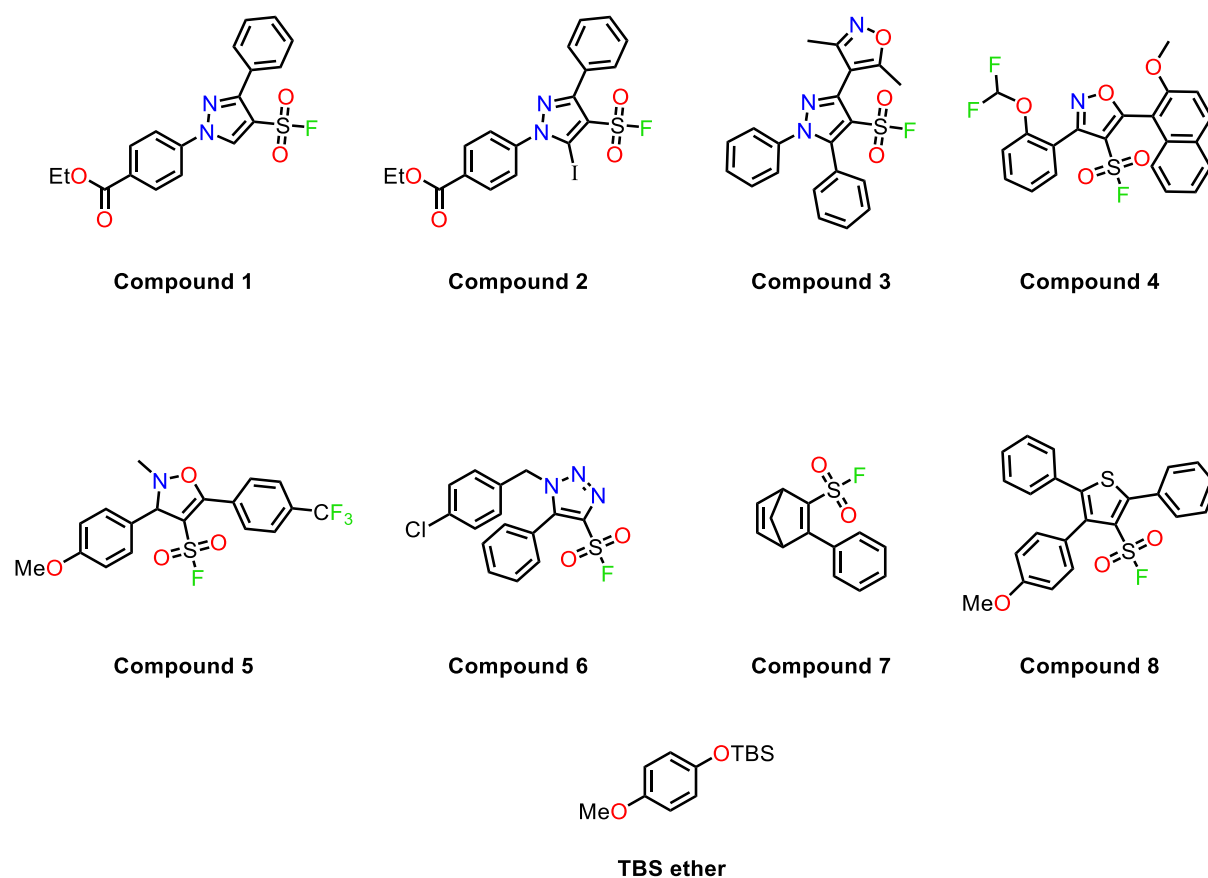
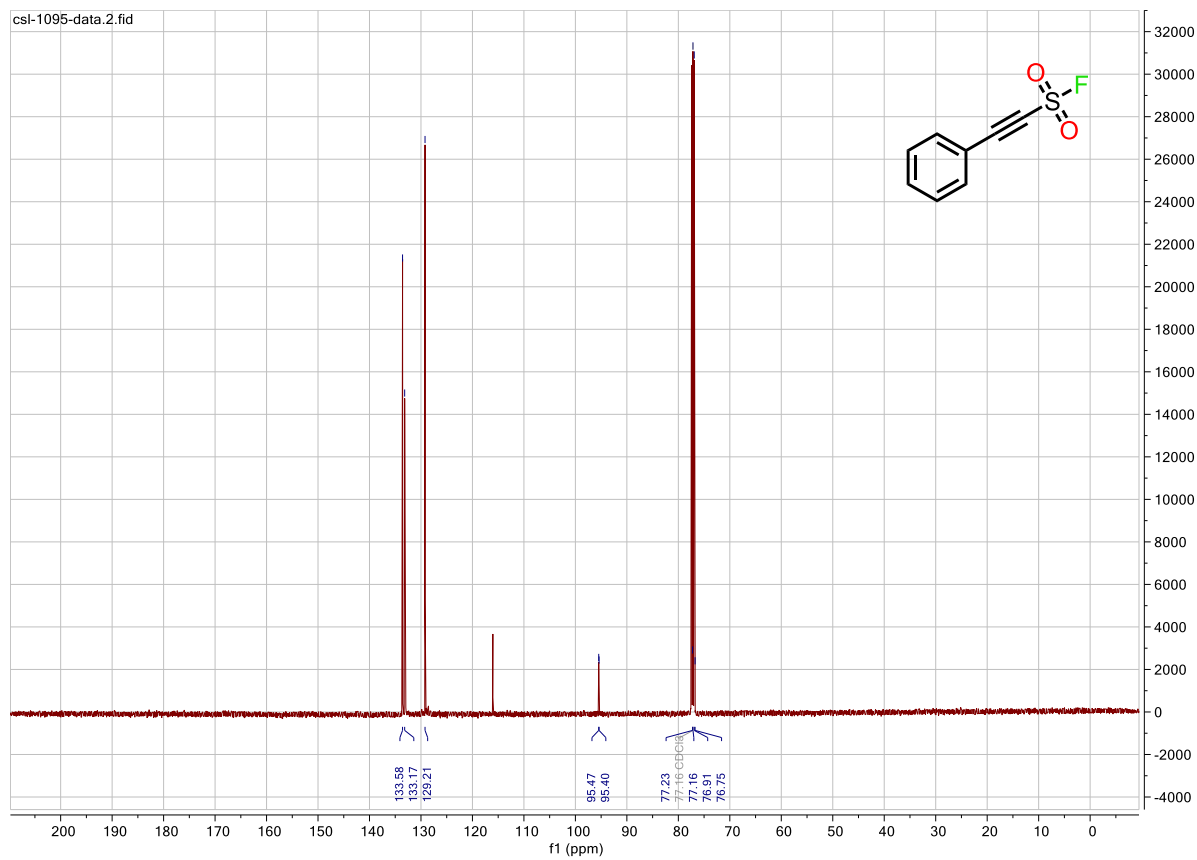
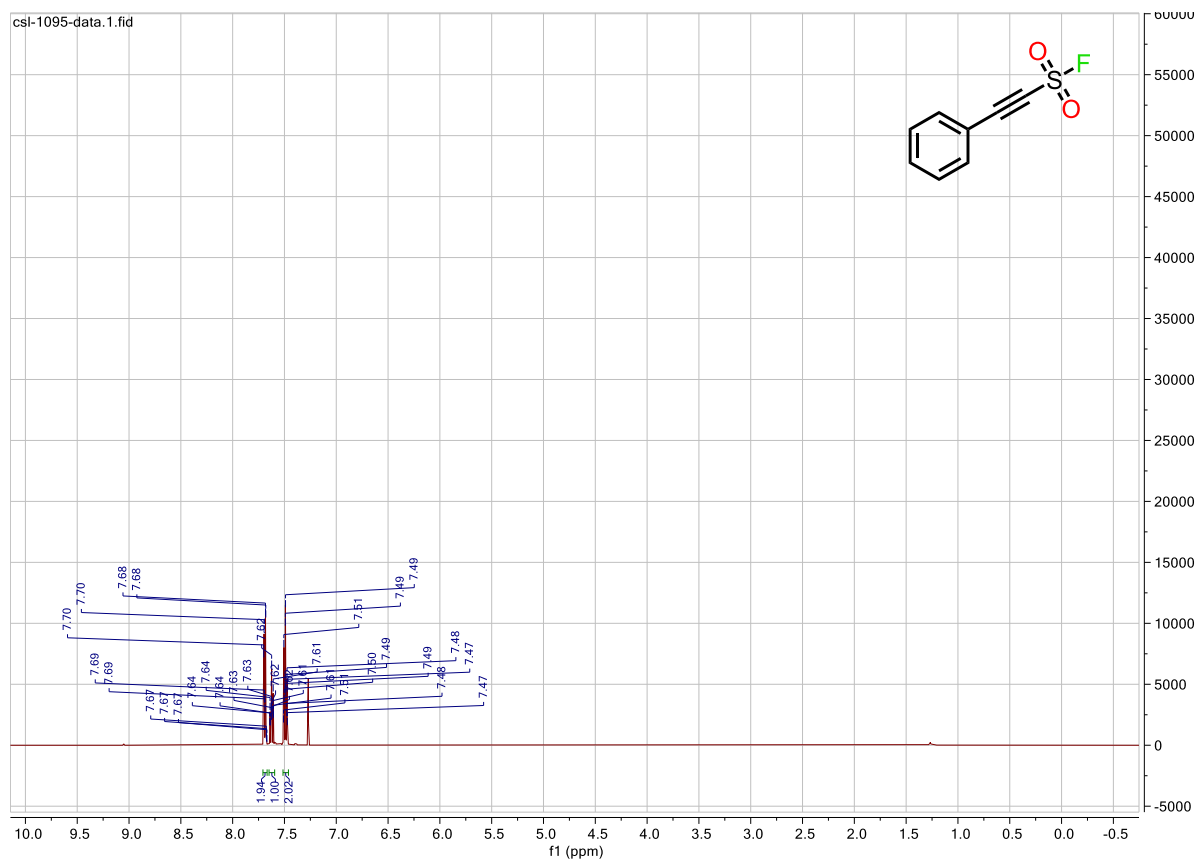
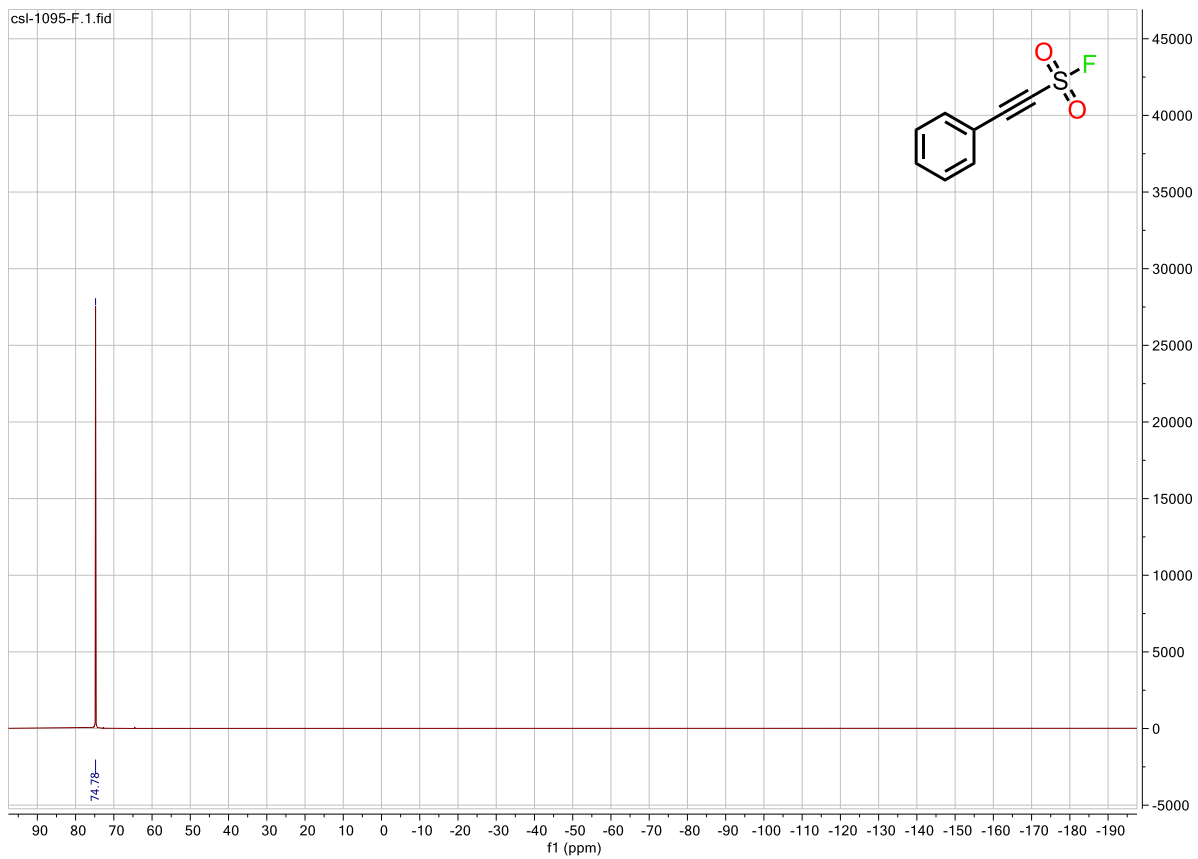


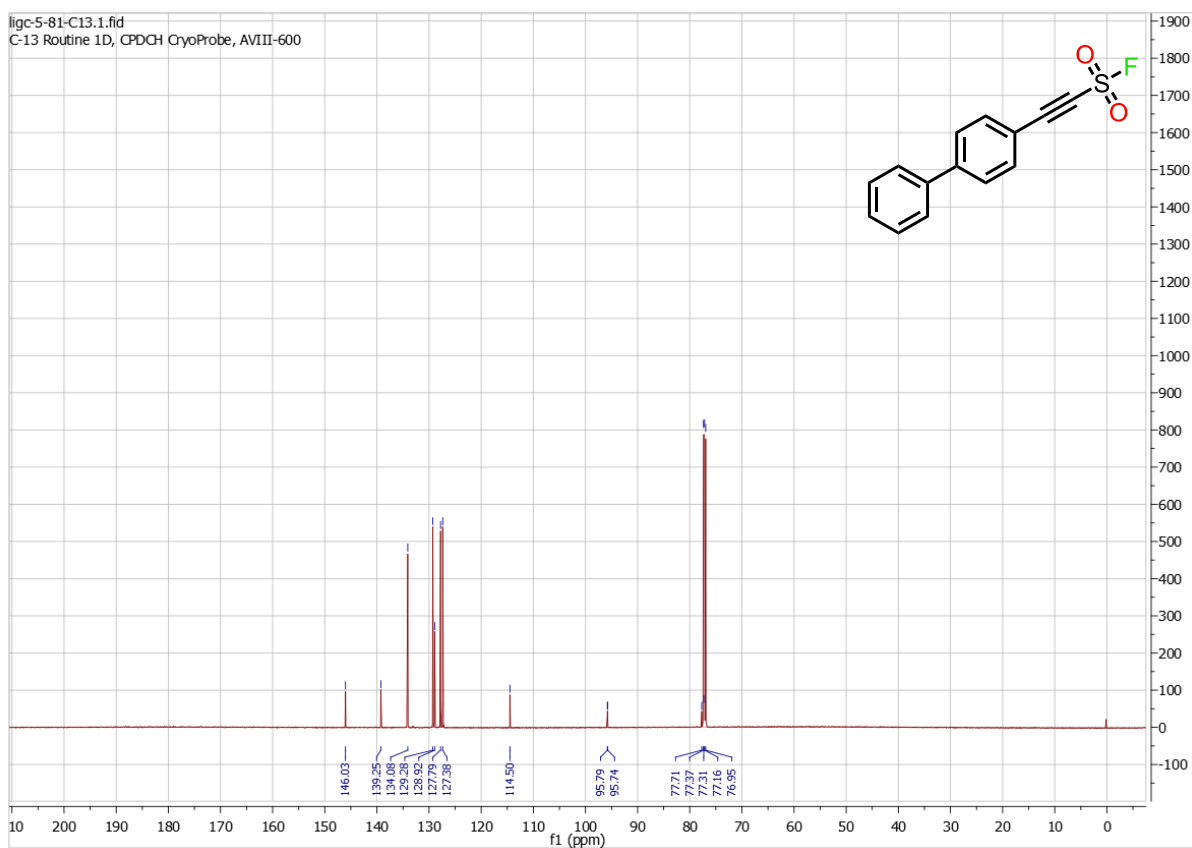
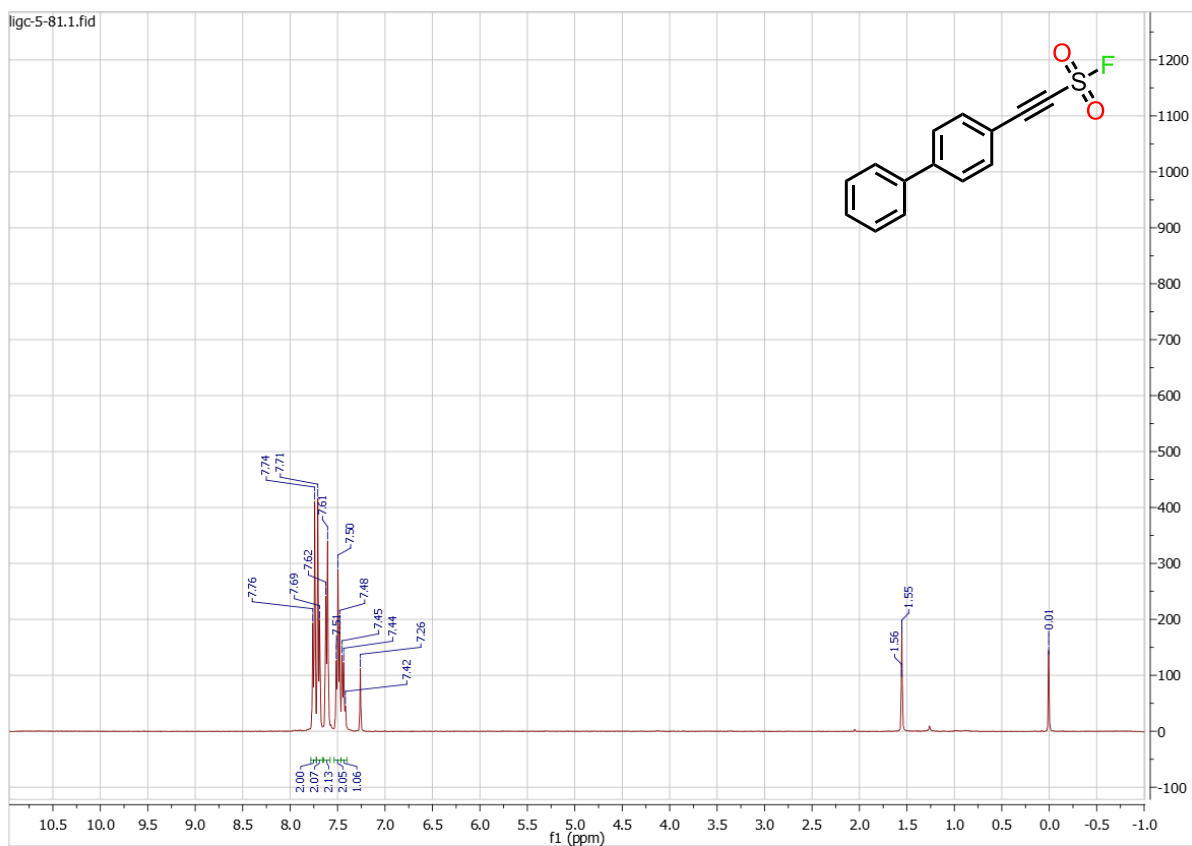
Table 2. SuFEx Competition studies.

Compound	1	2	3	4	5	6	7	8
1		2>1	1>3	4>1	5>1	6>1	1>7	8>1
2	2>1		2>3	2>4	2 \approx 5	6>2	2>7	2>8
3	1>3	2>3		4>3	5>3	6>3	3>7	8>3
4	4>1	2>4	4>3		5>4	6>4	4>7	4>8
5	5>1	2 \approx 5	5>3	5>4		6>5	5>7	8>5
6	6>1	6>2	6>3	6>4	6>5		6>7	6>8
7	1>7	2>7	3>7	4>7	5>7	6>7		8>7
8	8>1	2>8	8>3	4>8	8>5	6>8	8>7	

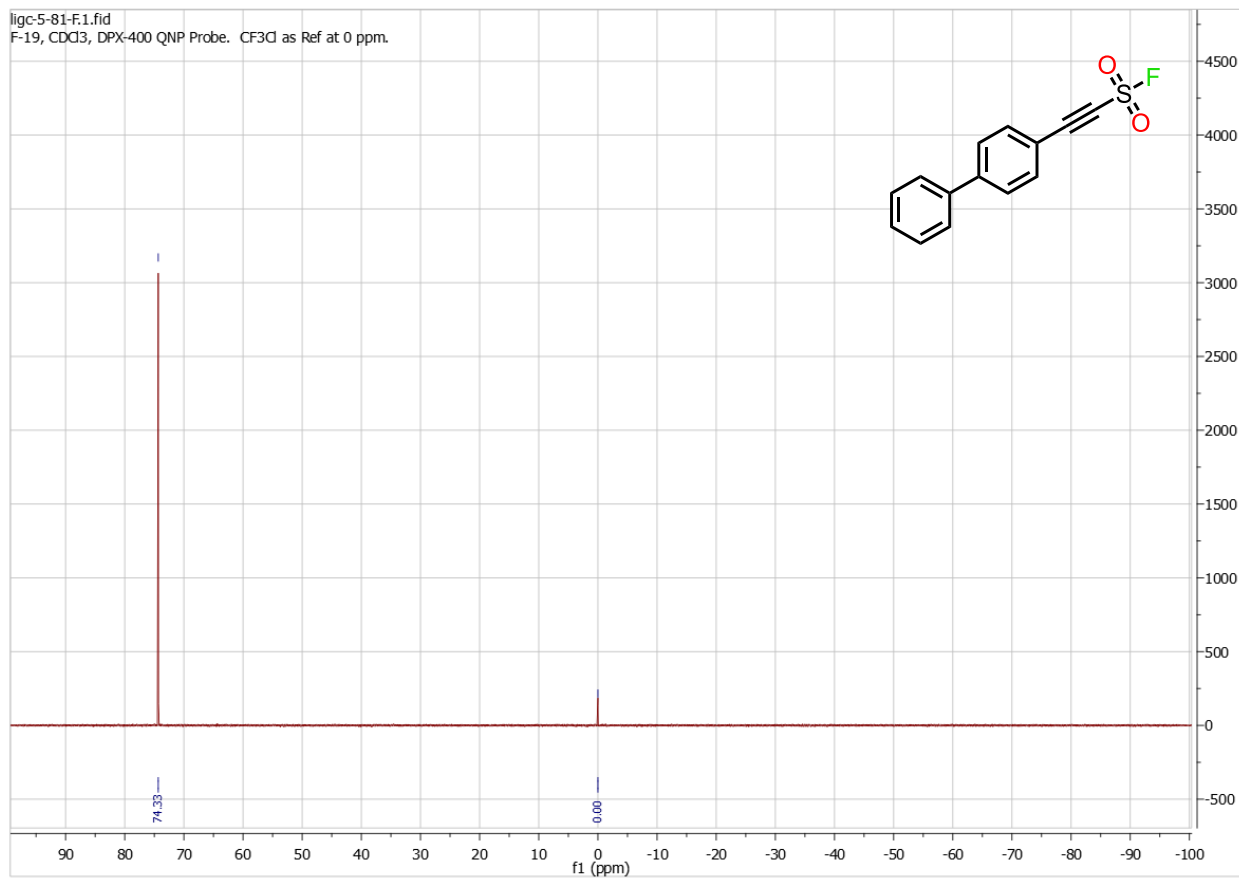
¹H, ¹³C and ¹⁹F NMR spectra

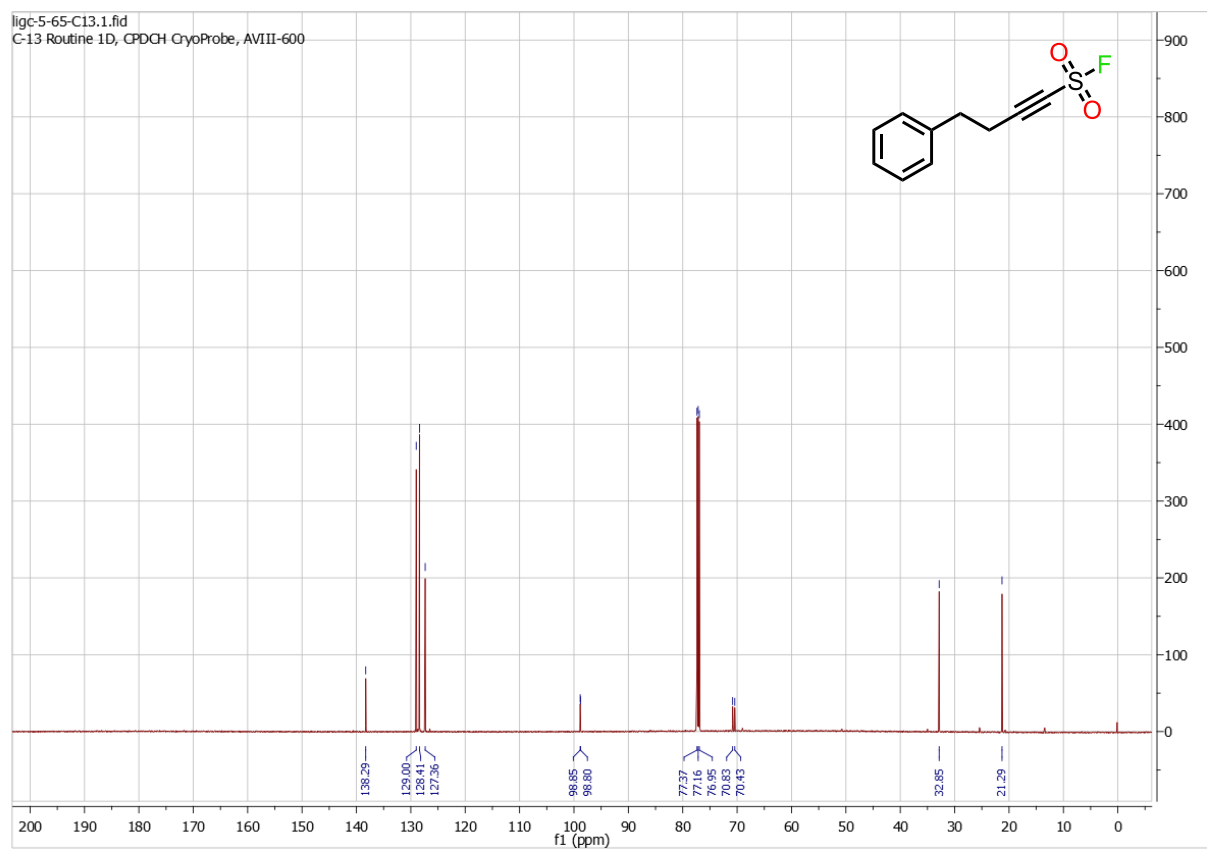
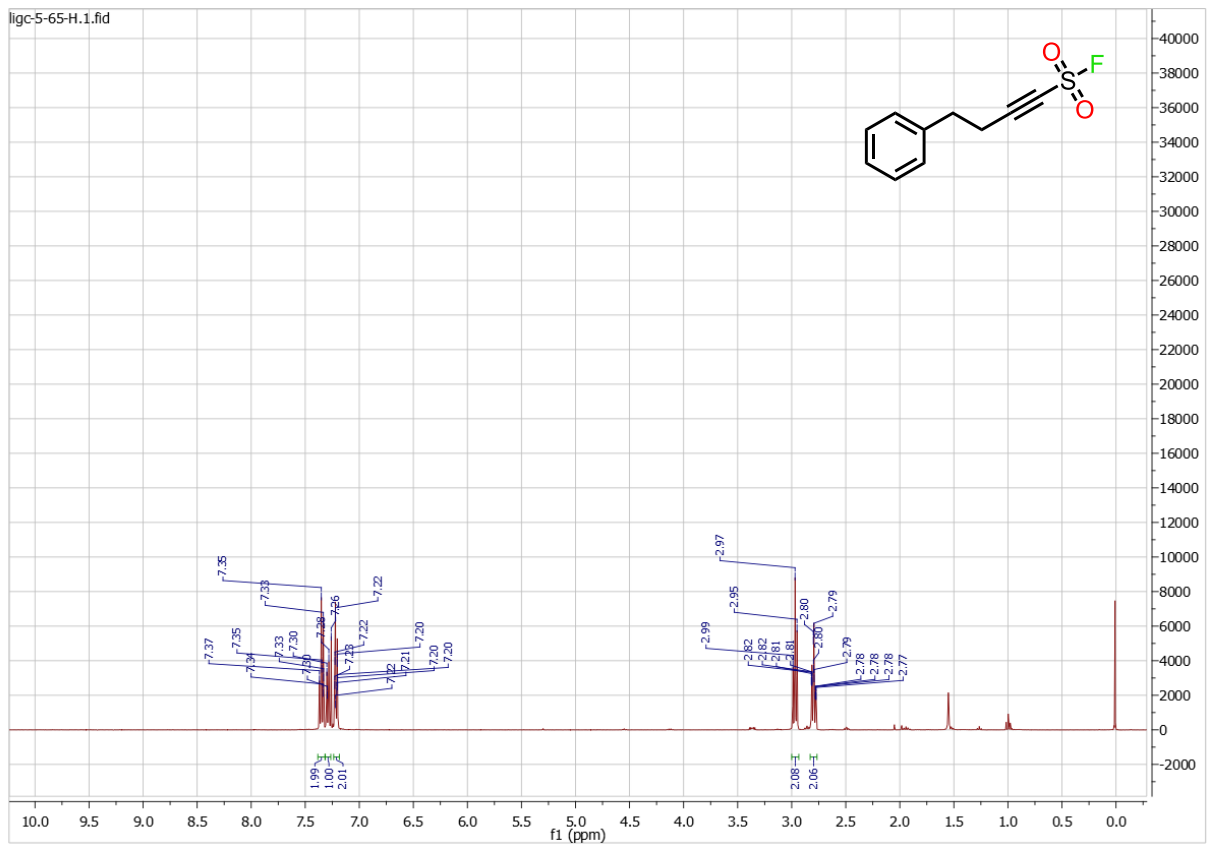




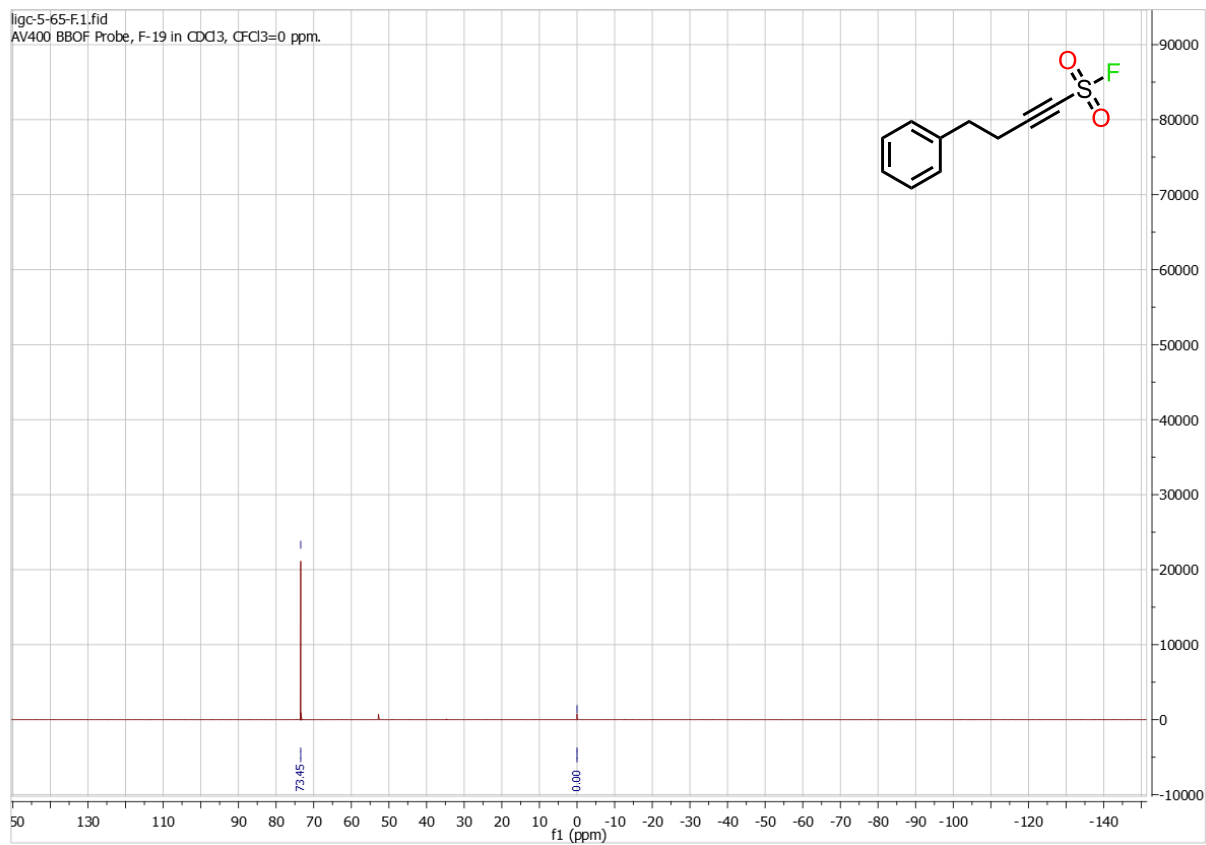


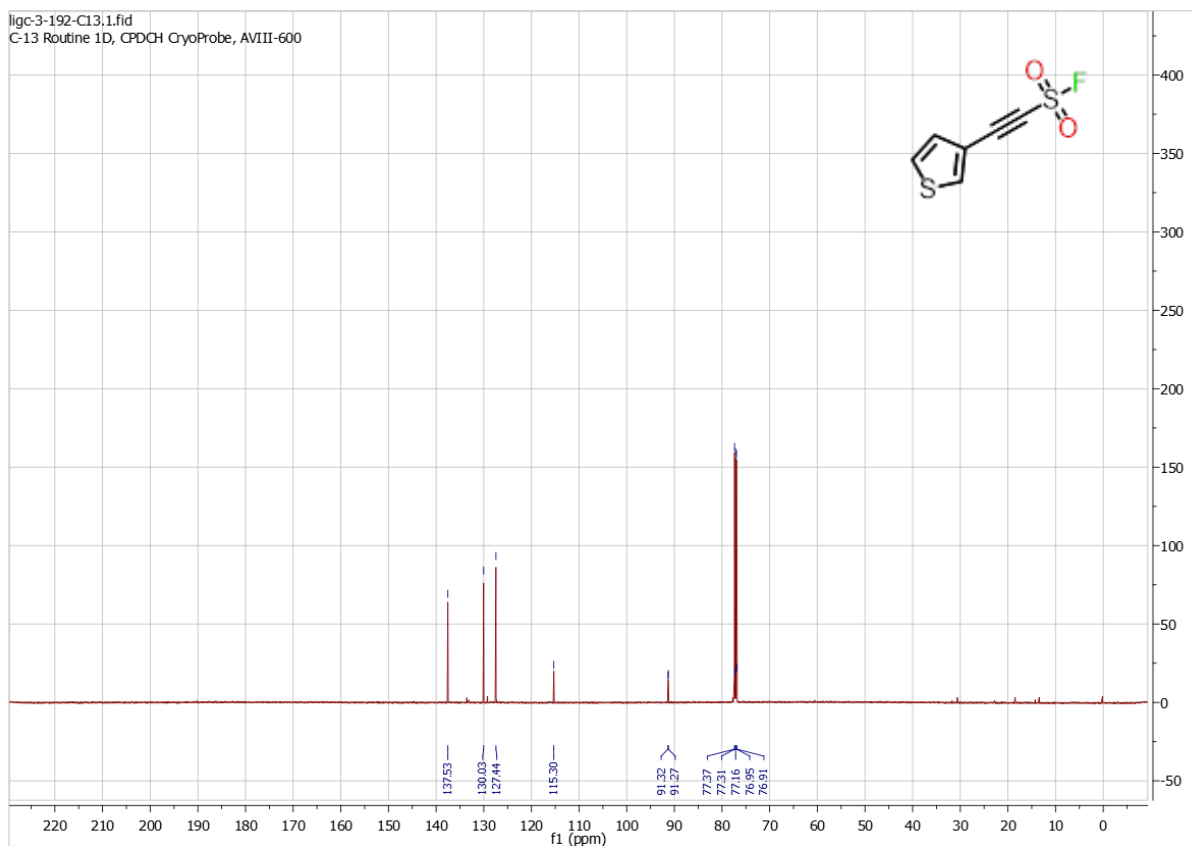
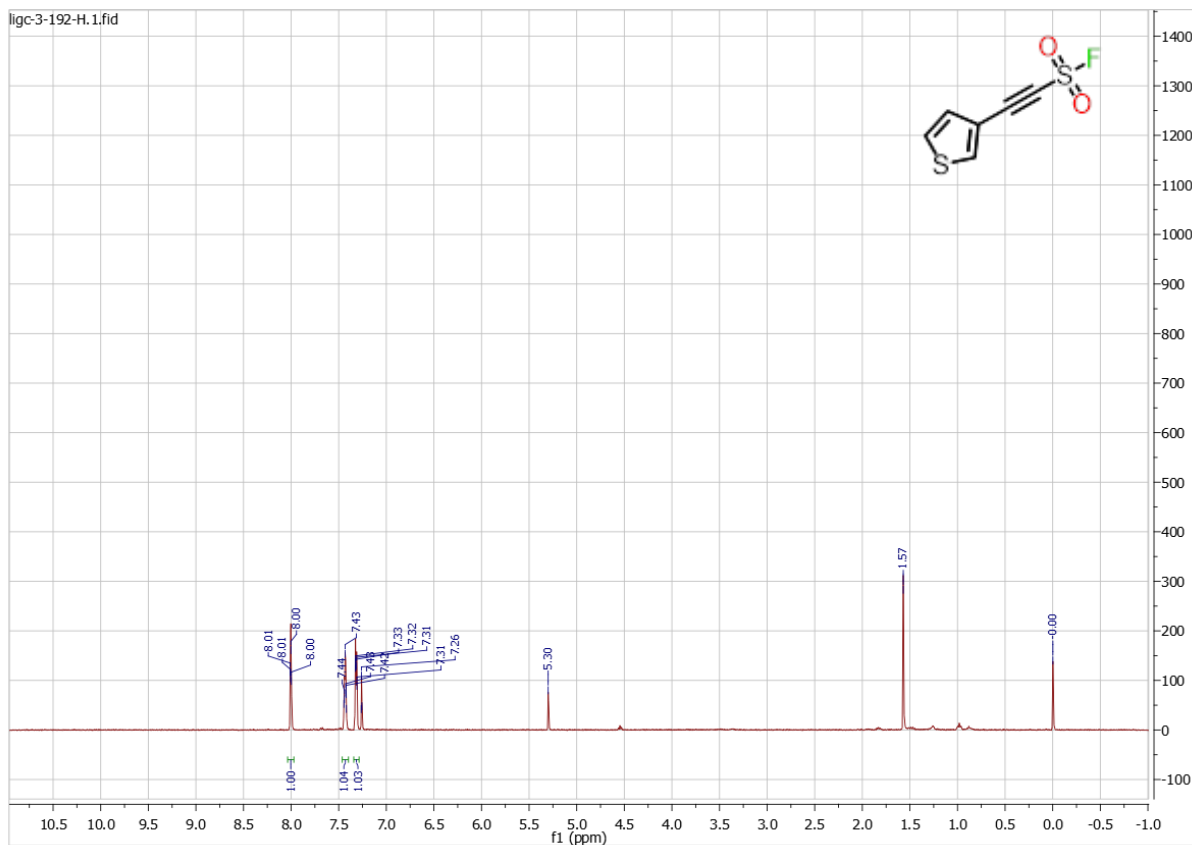
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F-19, CDCl3, DPX-400 QNP Probe. CF3Cl as Ref at 0 ppm.



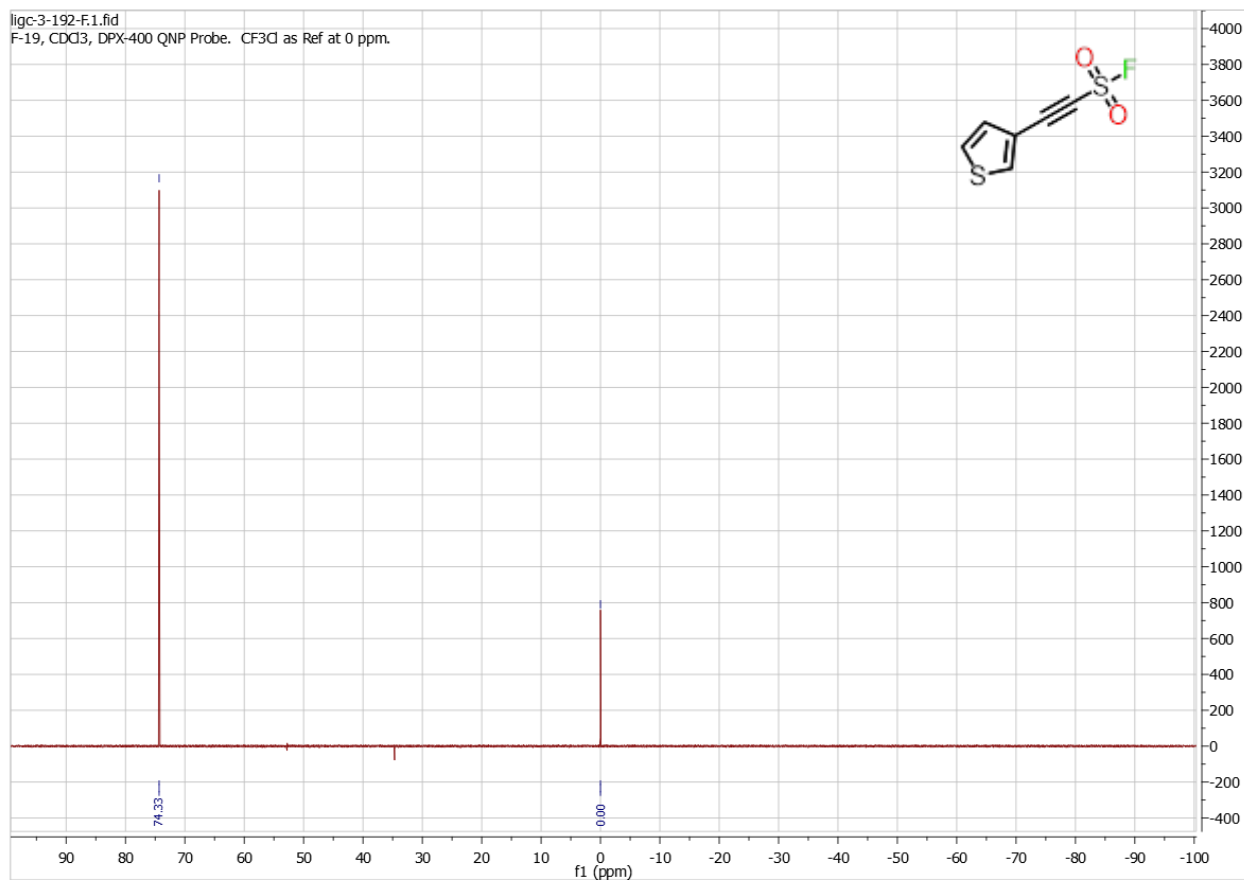


lgc-5-65-F.1.fid
AV400 BBOF Probe, F-19 in CDCl3, CFC13=0 ppm.





ligc-3-192-F.1.fid
F-19, CDCl₃, DPX-400 QNP Probe. CF₃Cl as Ref at 0 ppm.



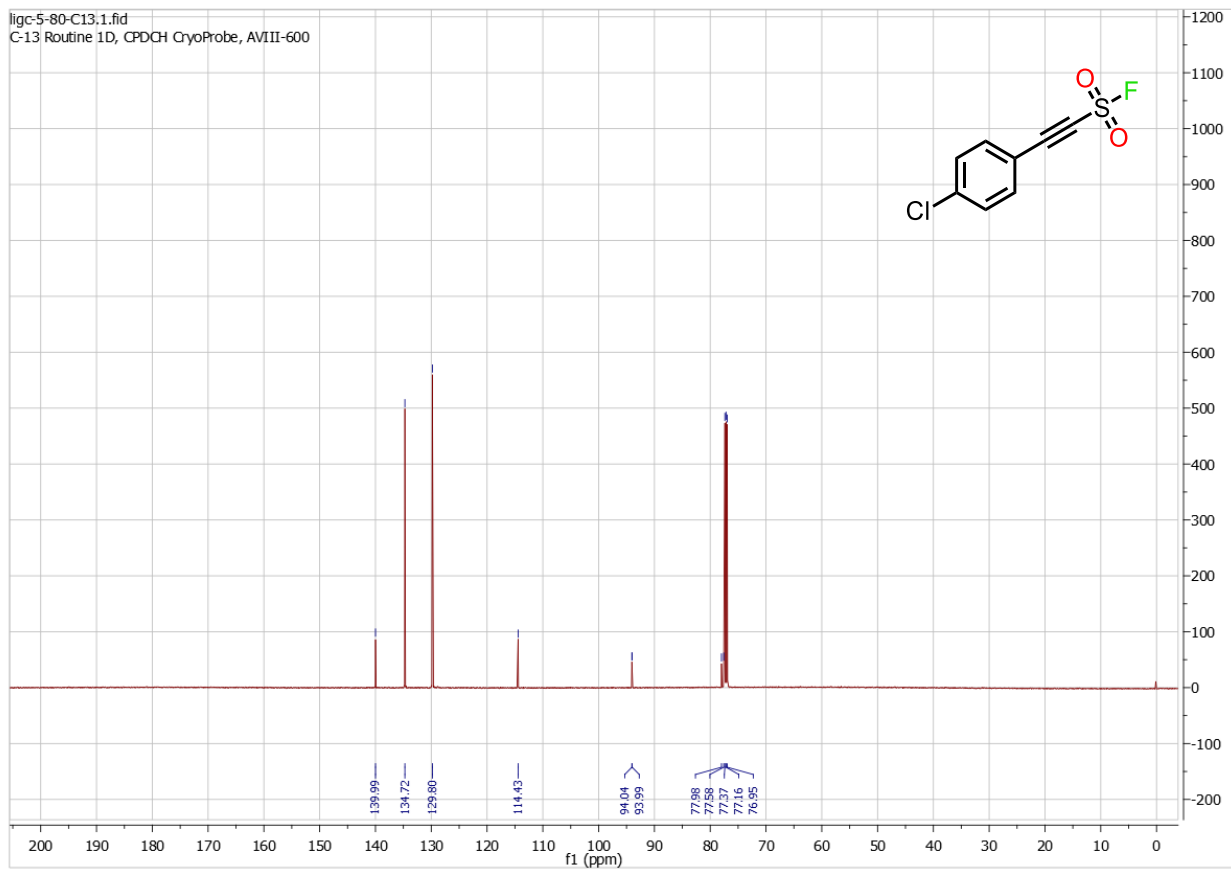
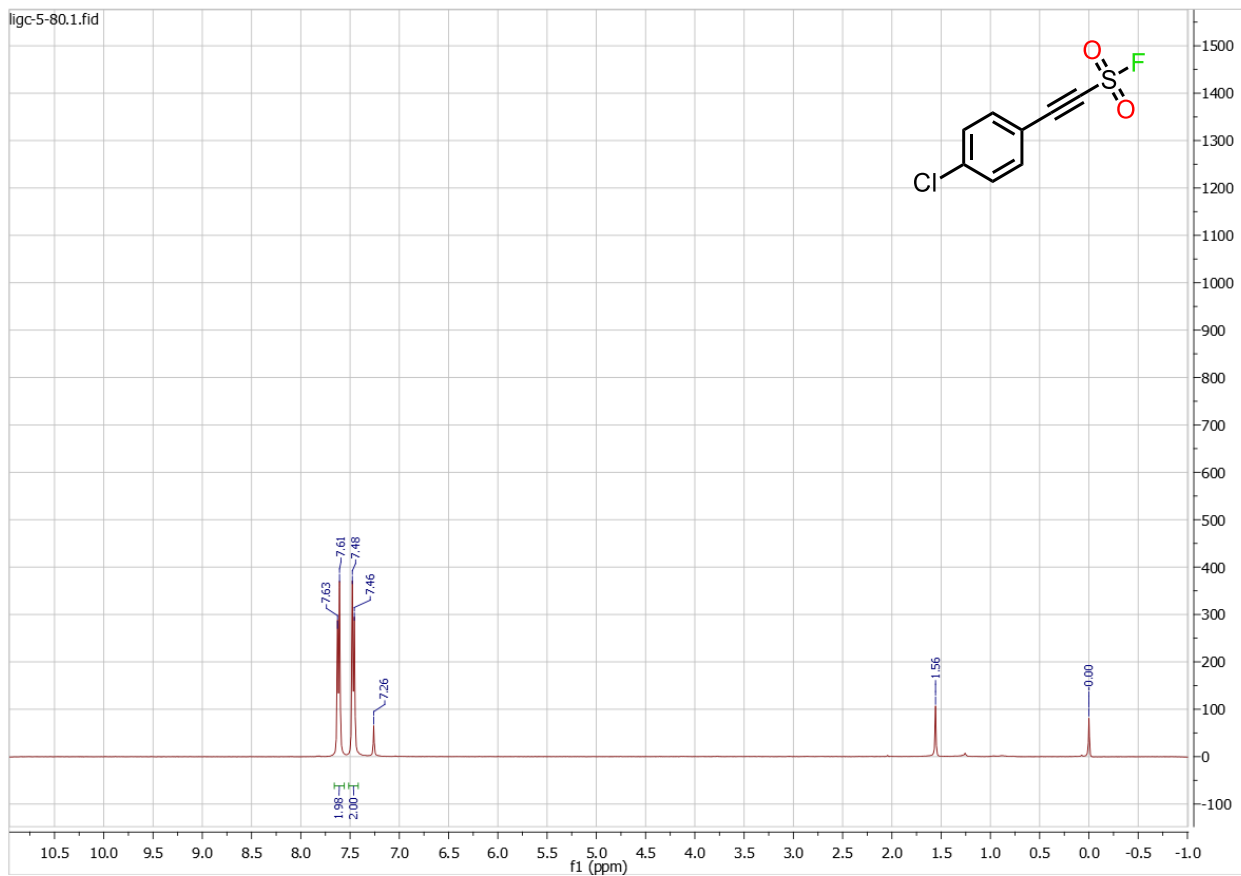
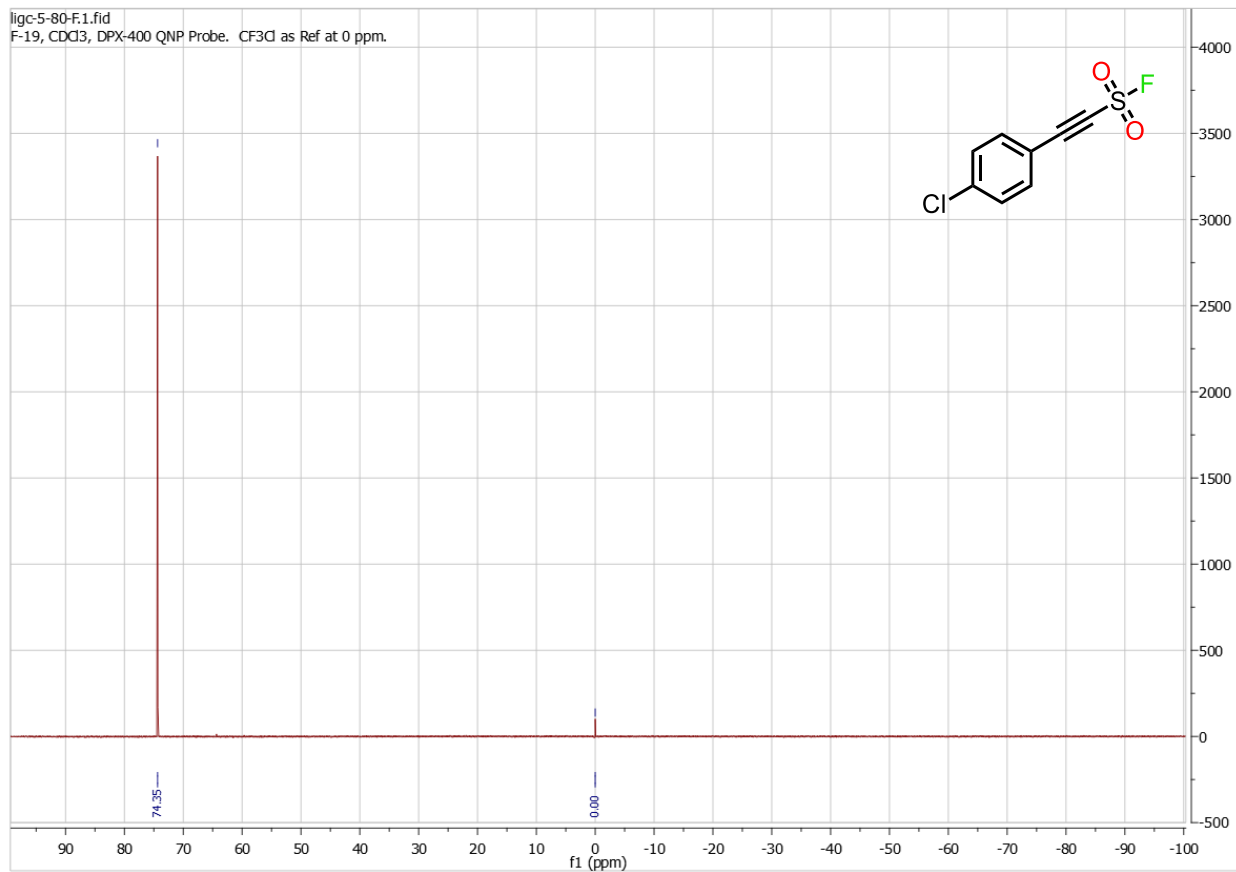
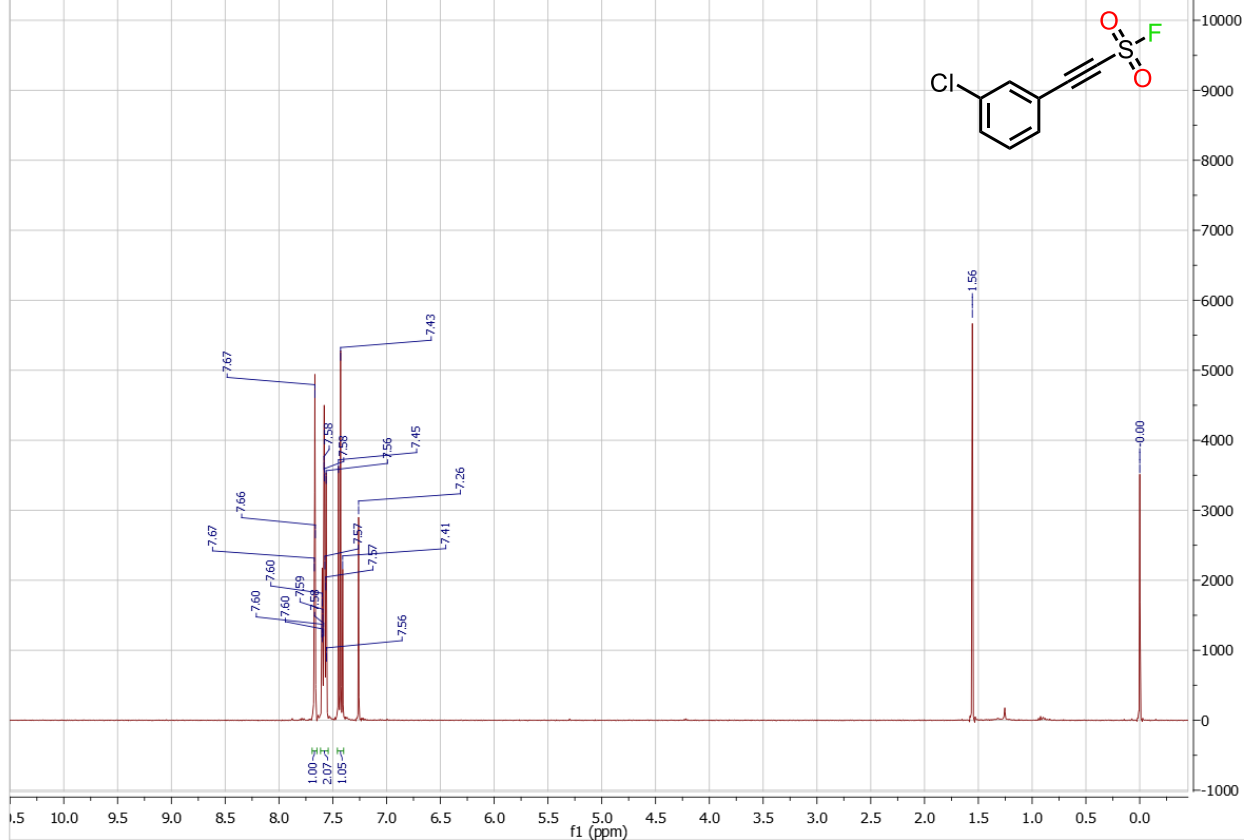


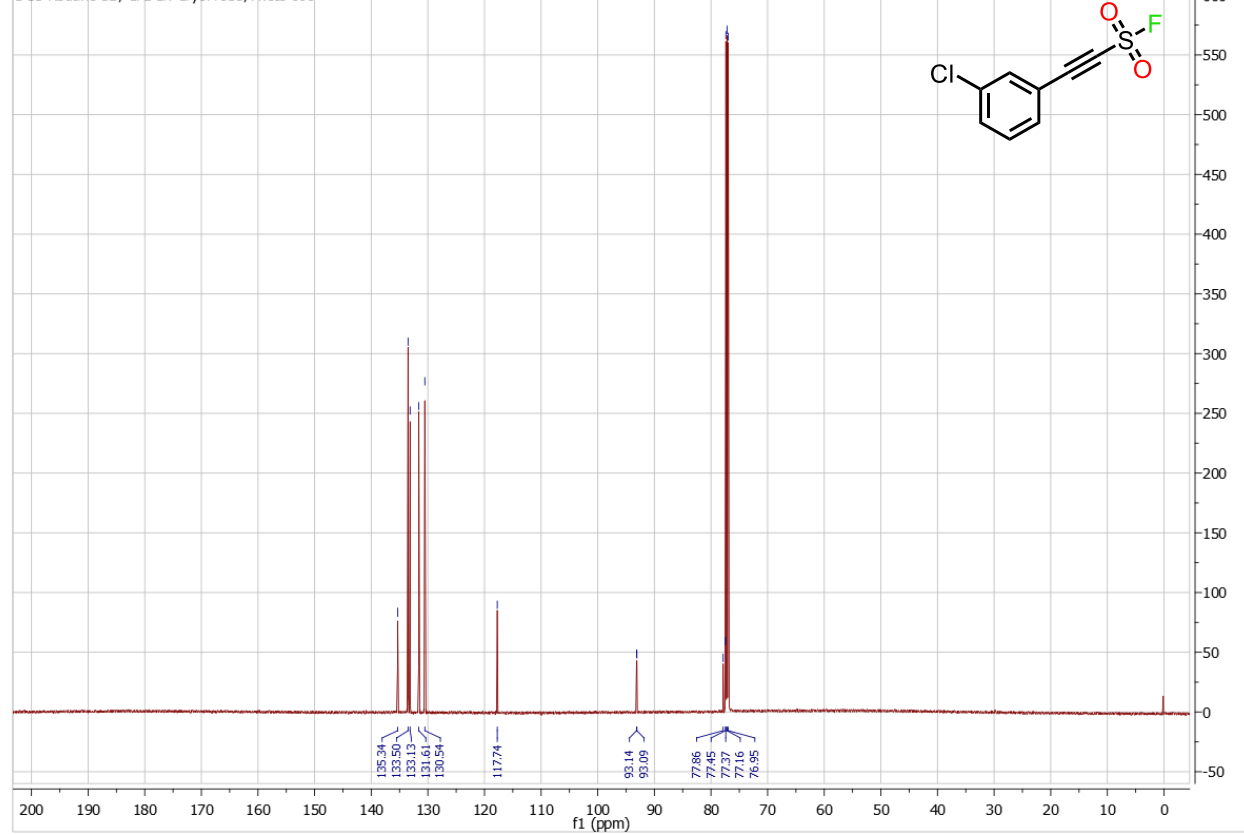
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F-19, CDCl3, DPX-400 QNP Probe. CF3Cl as Ref at 0 ppm.



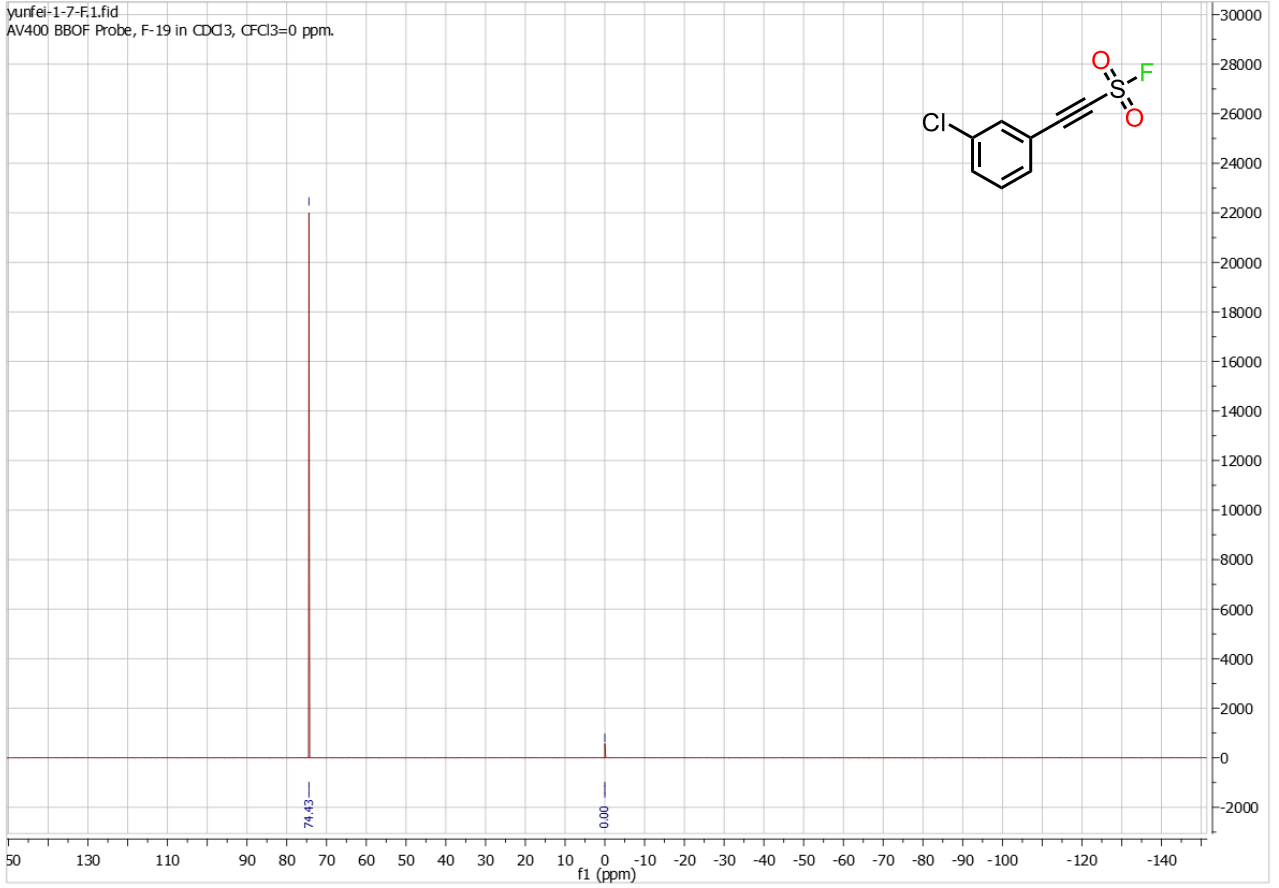
yunfei-1-7-H.1.fid

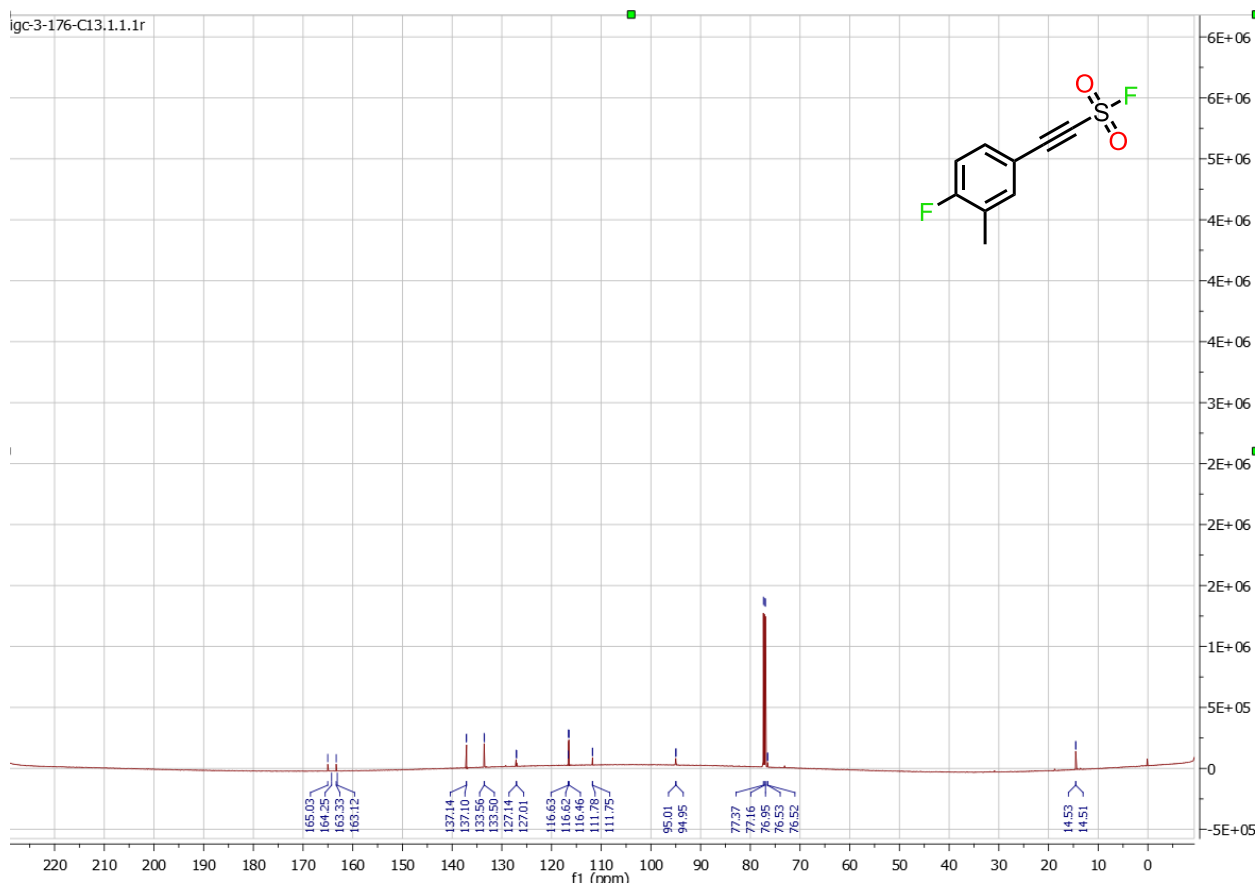
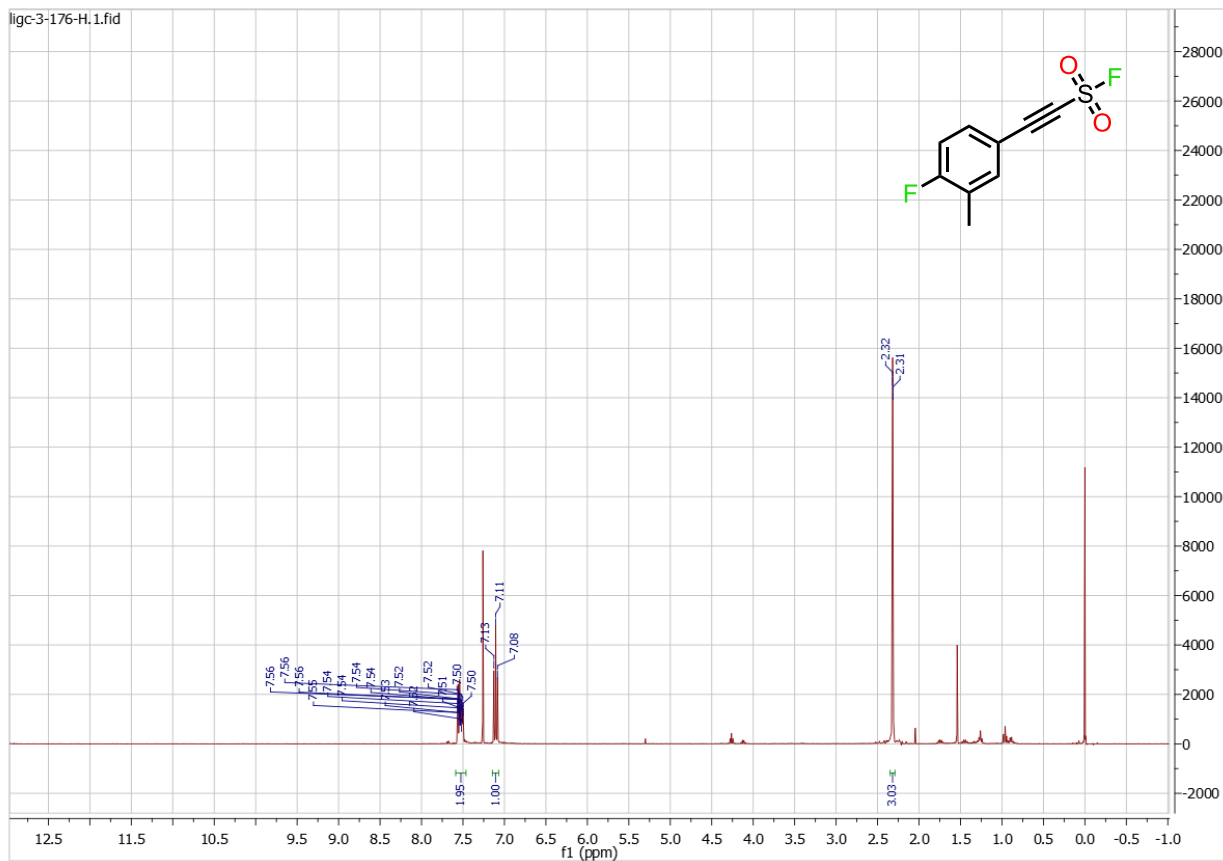


yunfei-1-7-C13.1.fid
C-13 Routine 1D, CPDCH CryoProbe, AVIII-600

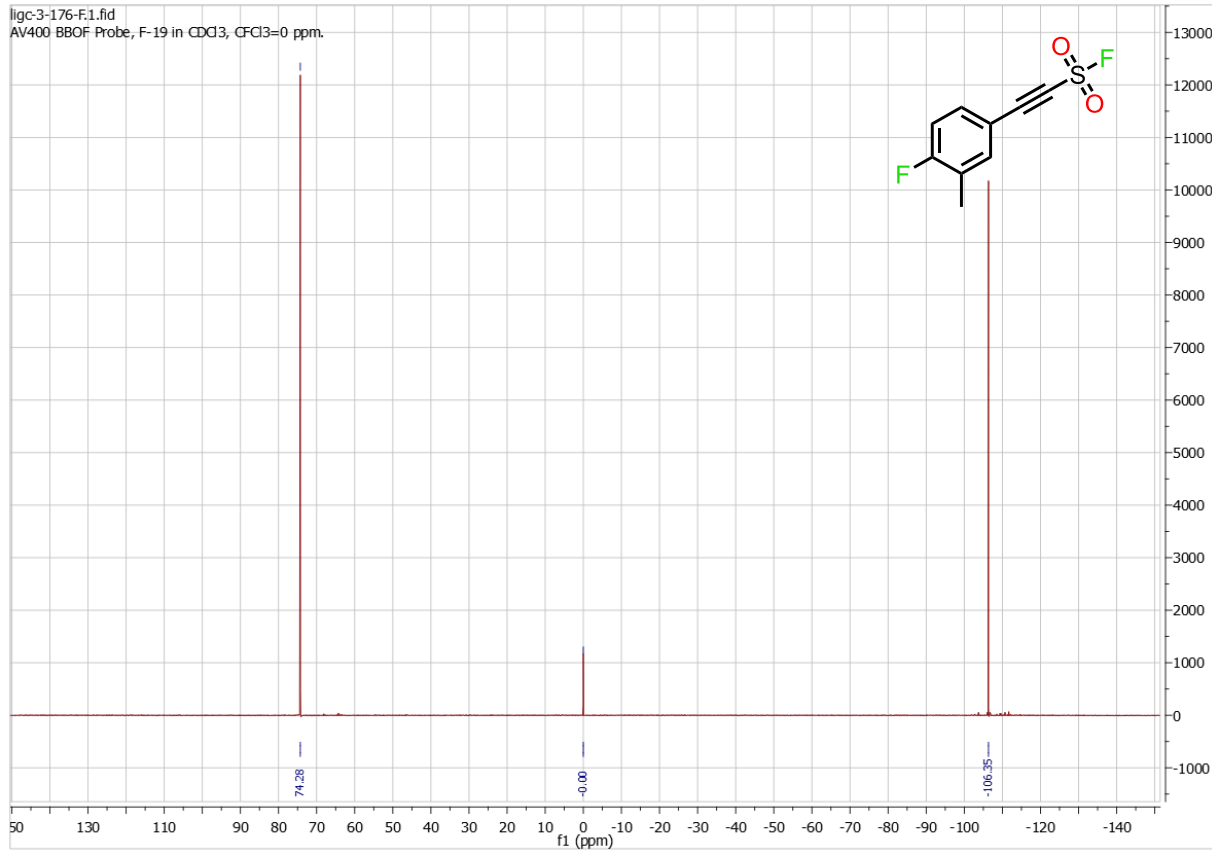


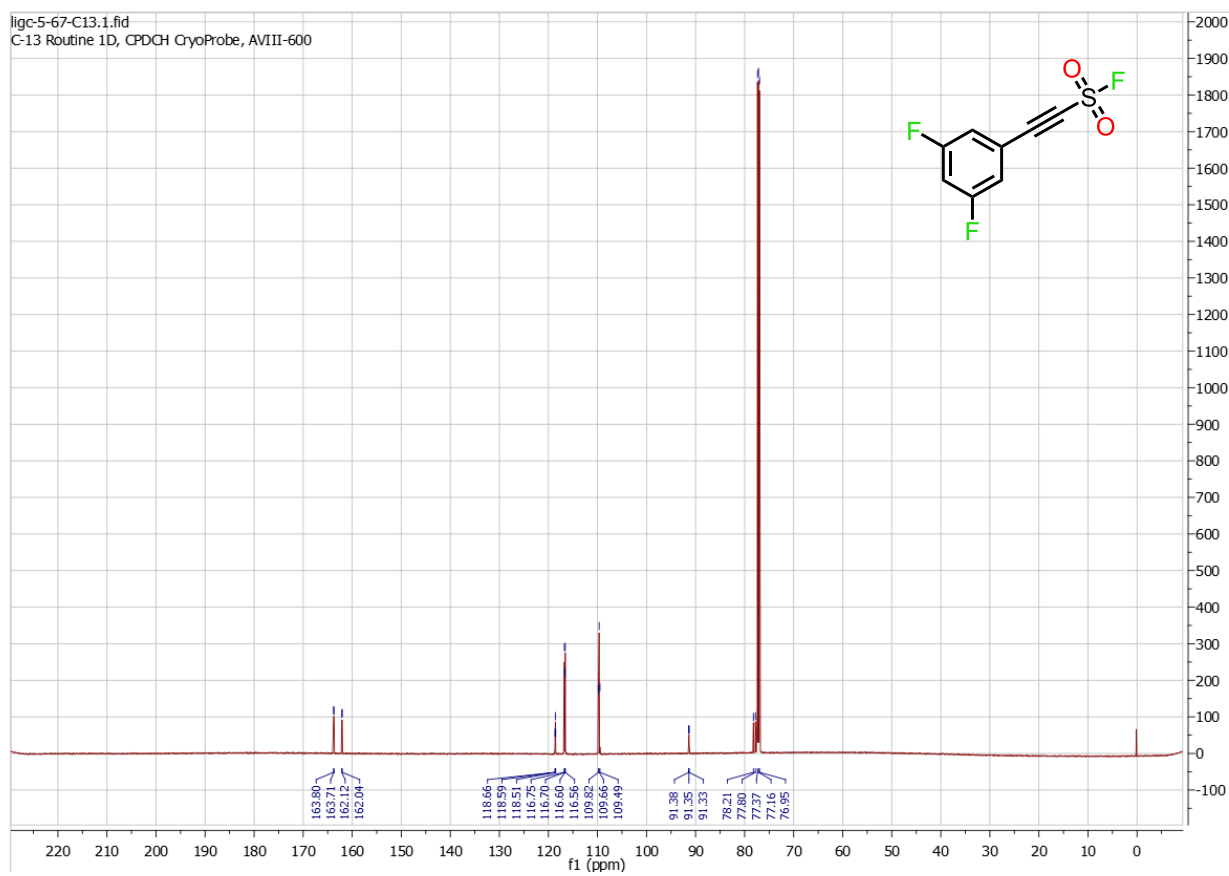
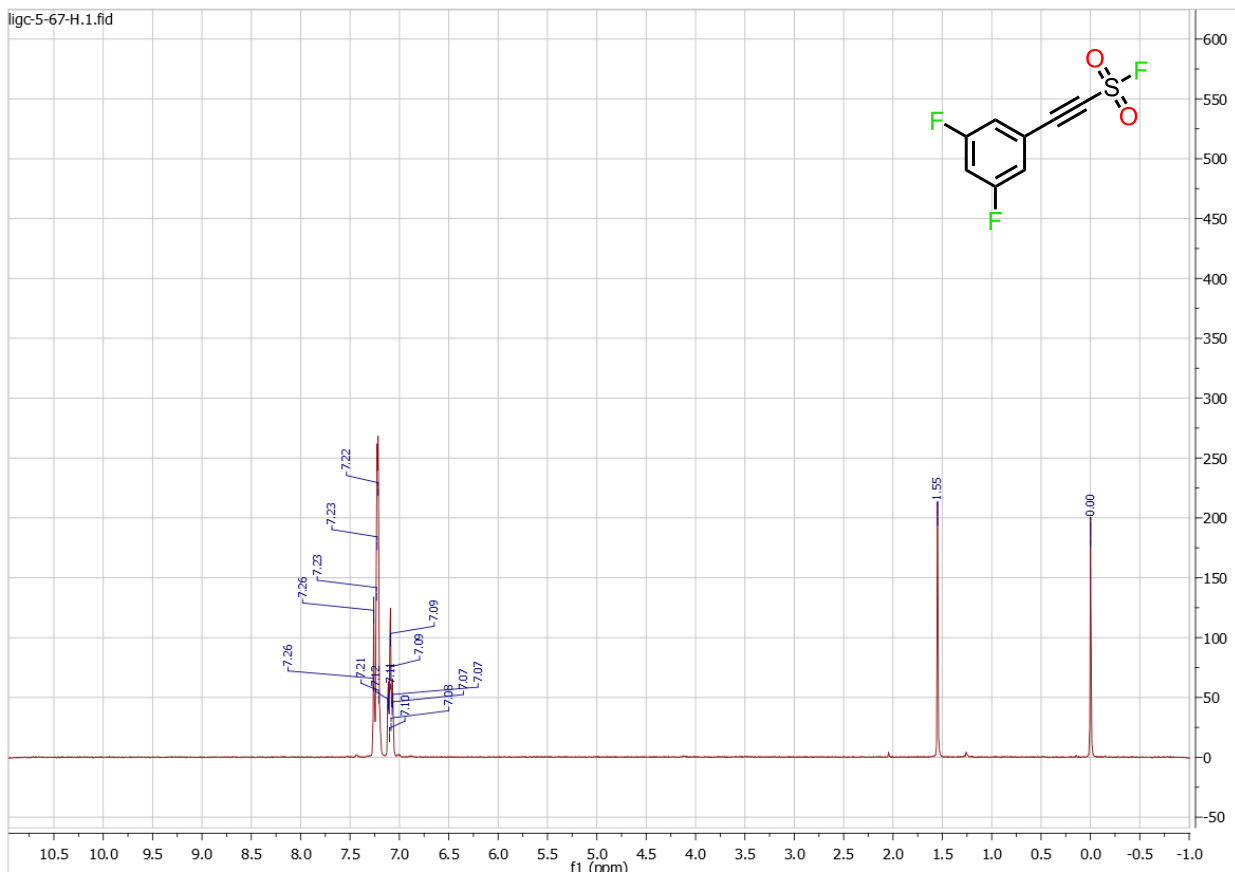
yunfei-1-7-F.1.fid
AV400 BBOF Probe, F-19 in CDCl3, CFCl3=0 ppm.



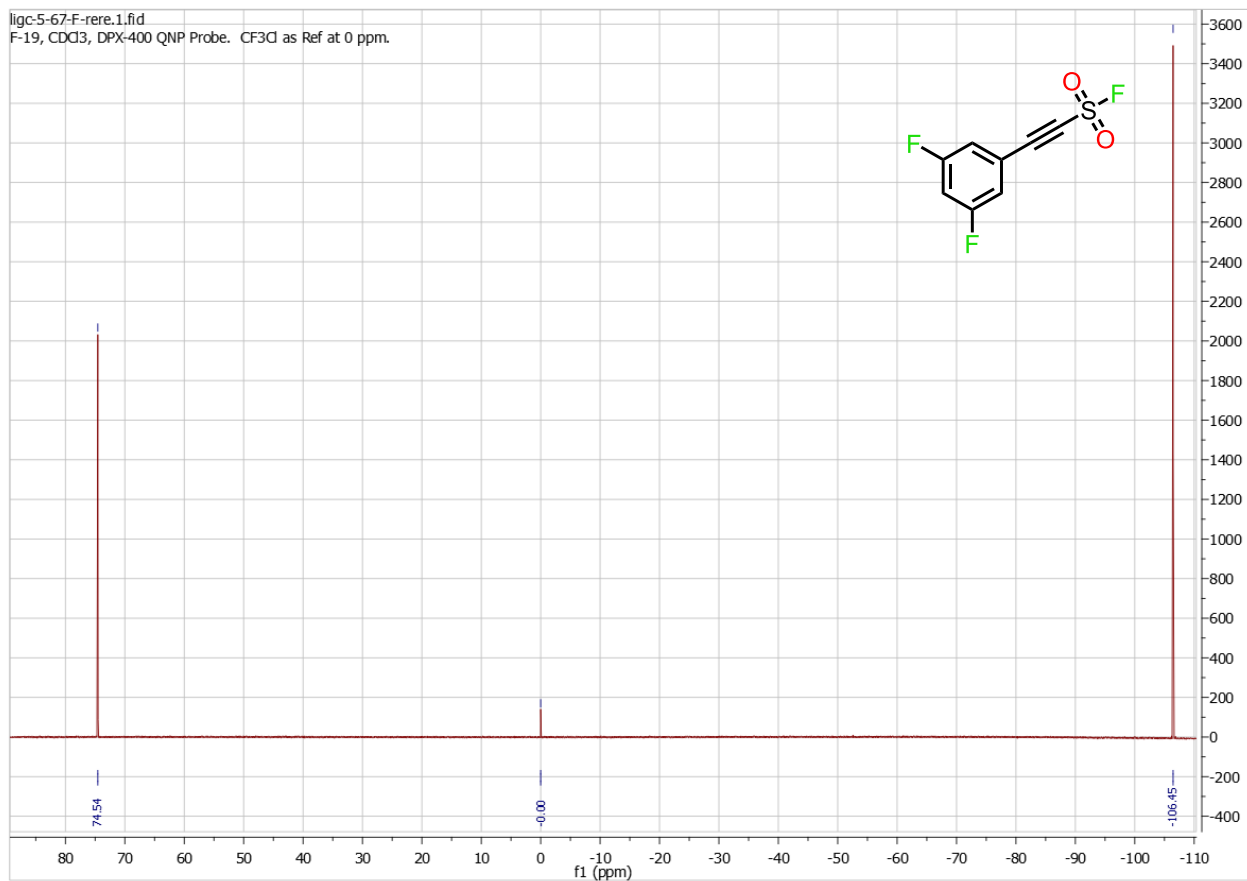


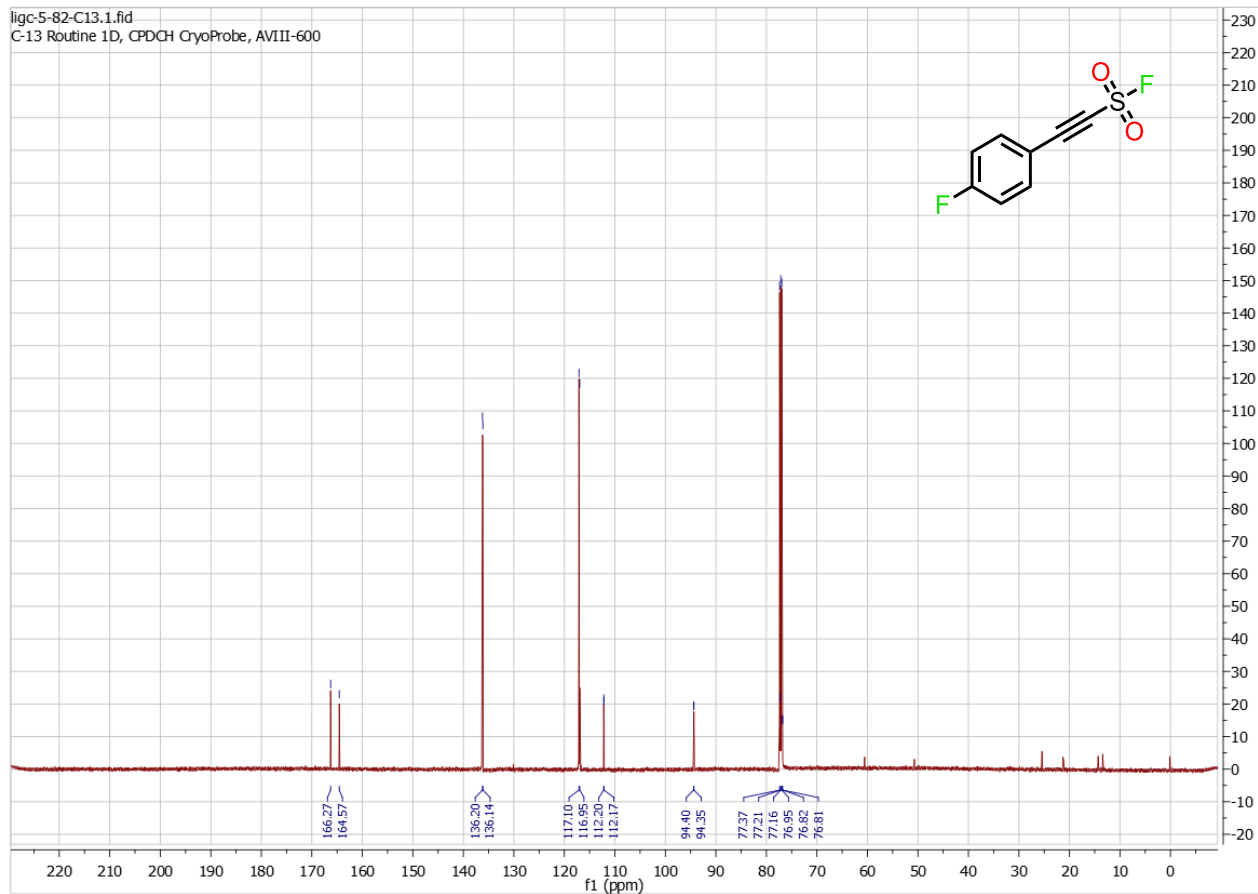
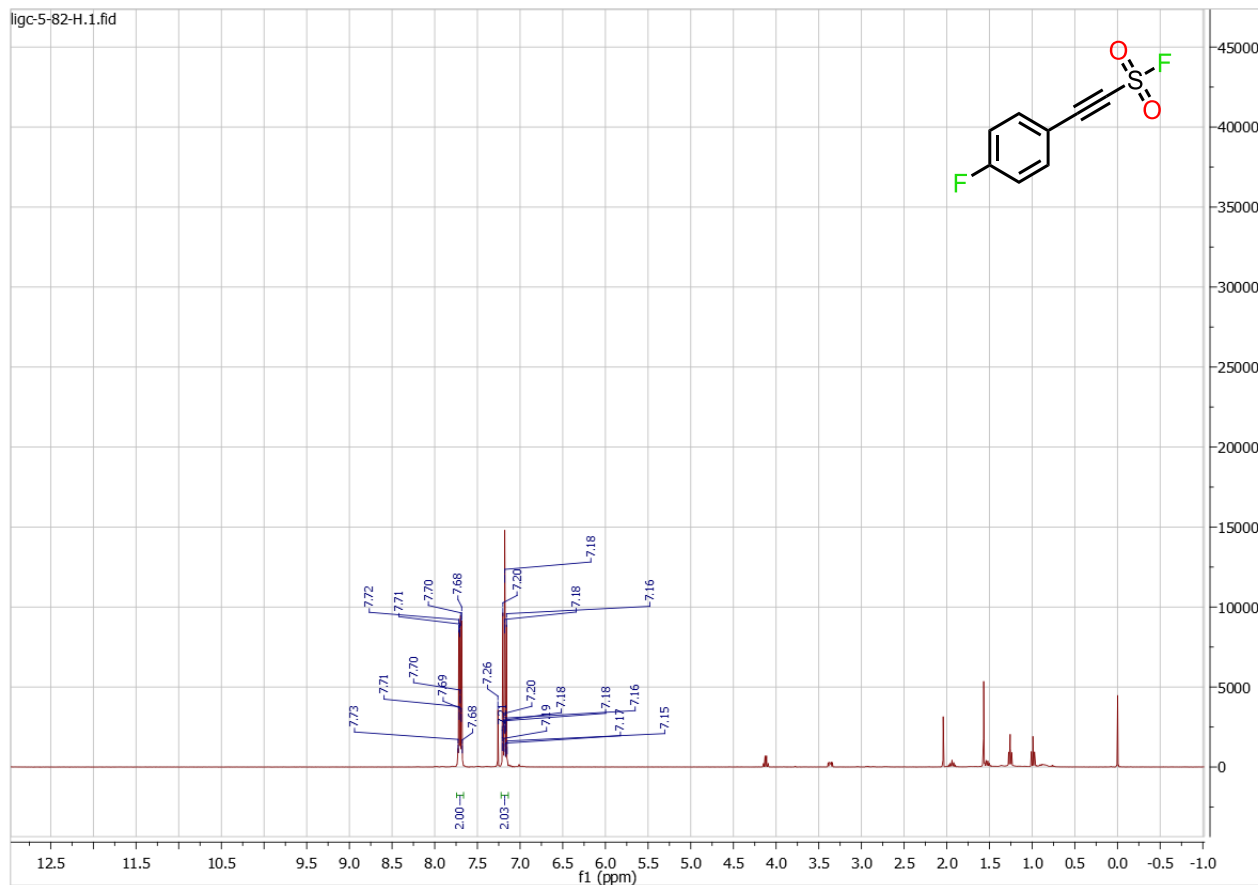
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AV400 BBOF Probe, F-19 in CDCl3, CFCl3=0 ppm.



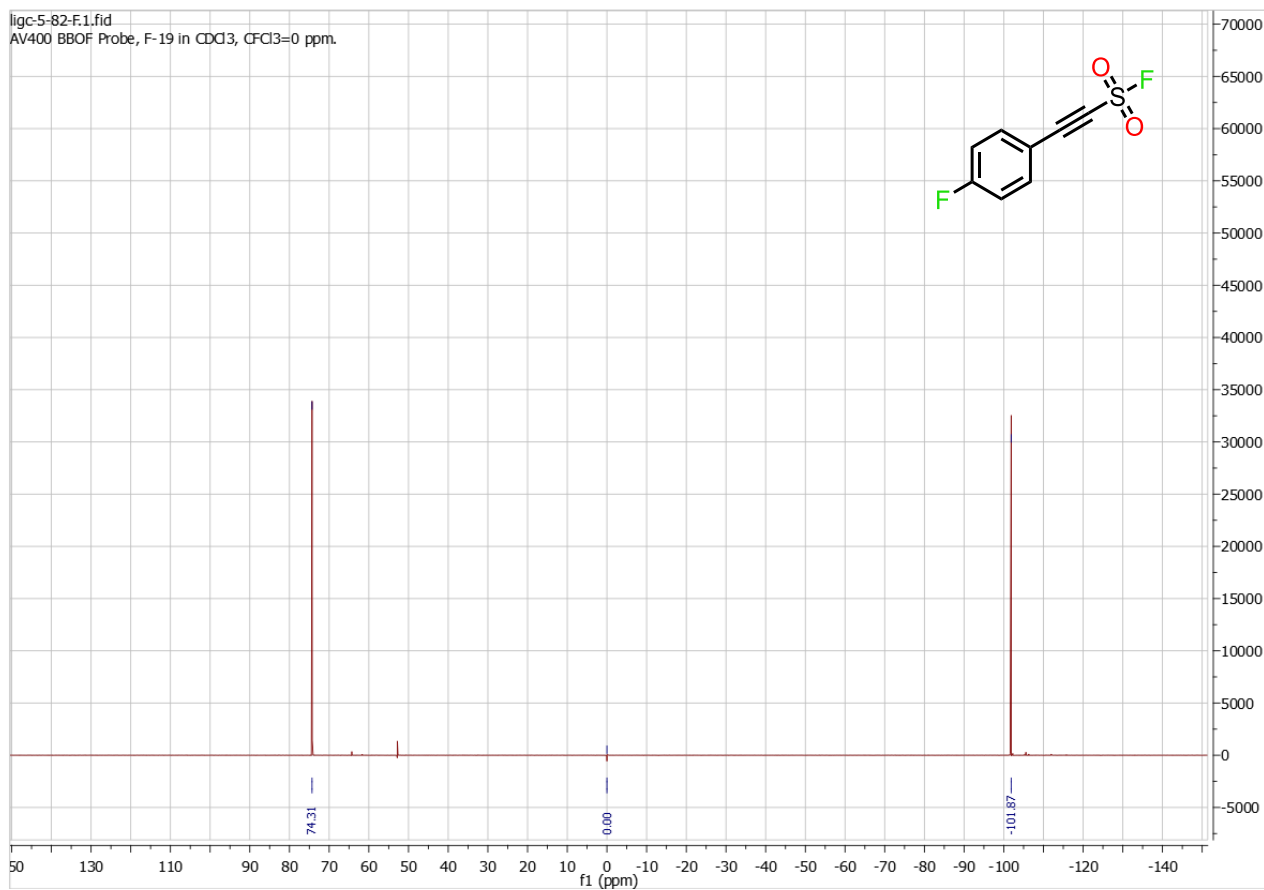


ligc-5-67-F-rere.1.fid
F-19, CDCl₃, DPX-400 QNP Probe. CF₃Cl as Ref at 0 ppm.





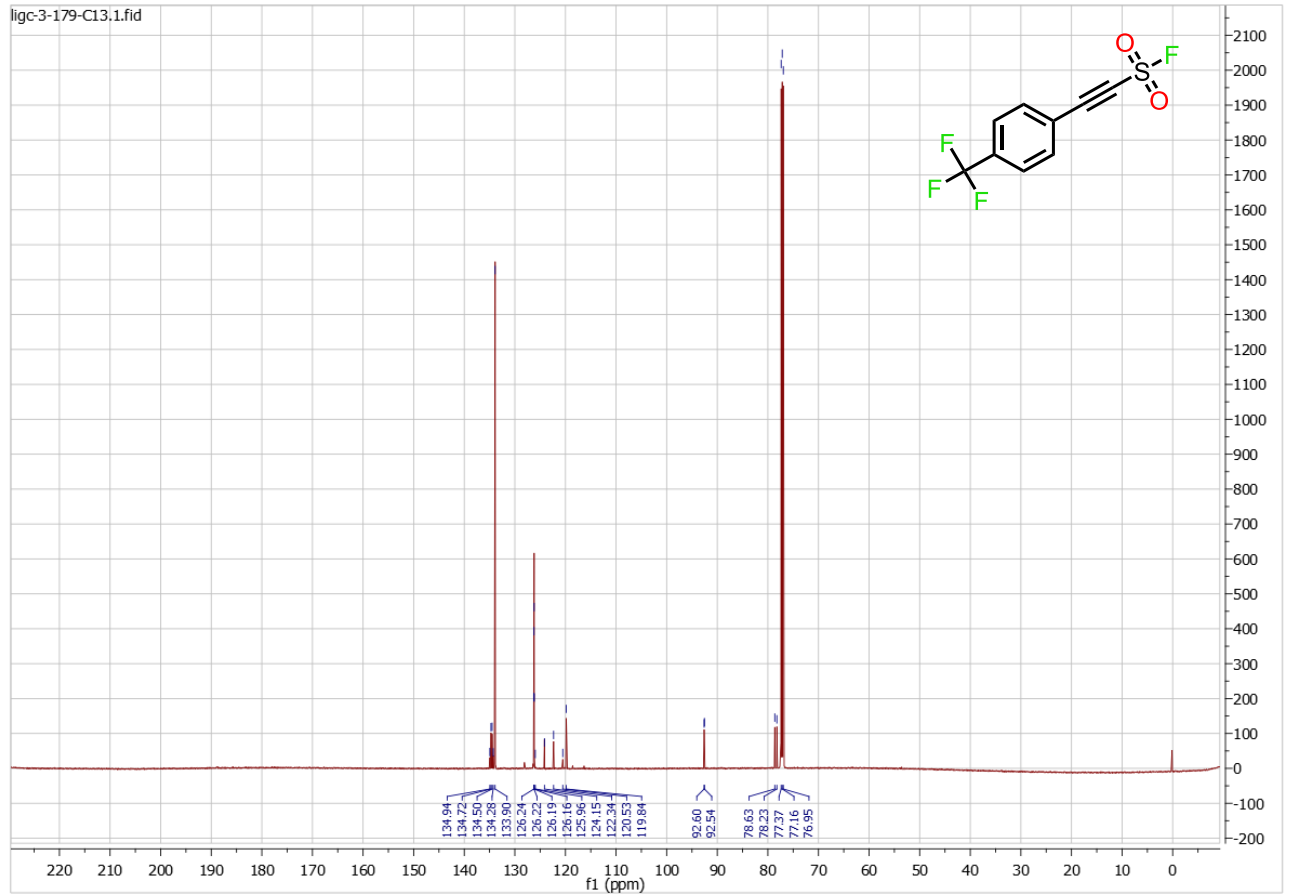
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AV400 BBOF Probe, F-19 in CDCl3, CFCl3=0 ppm.



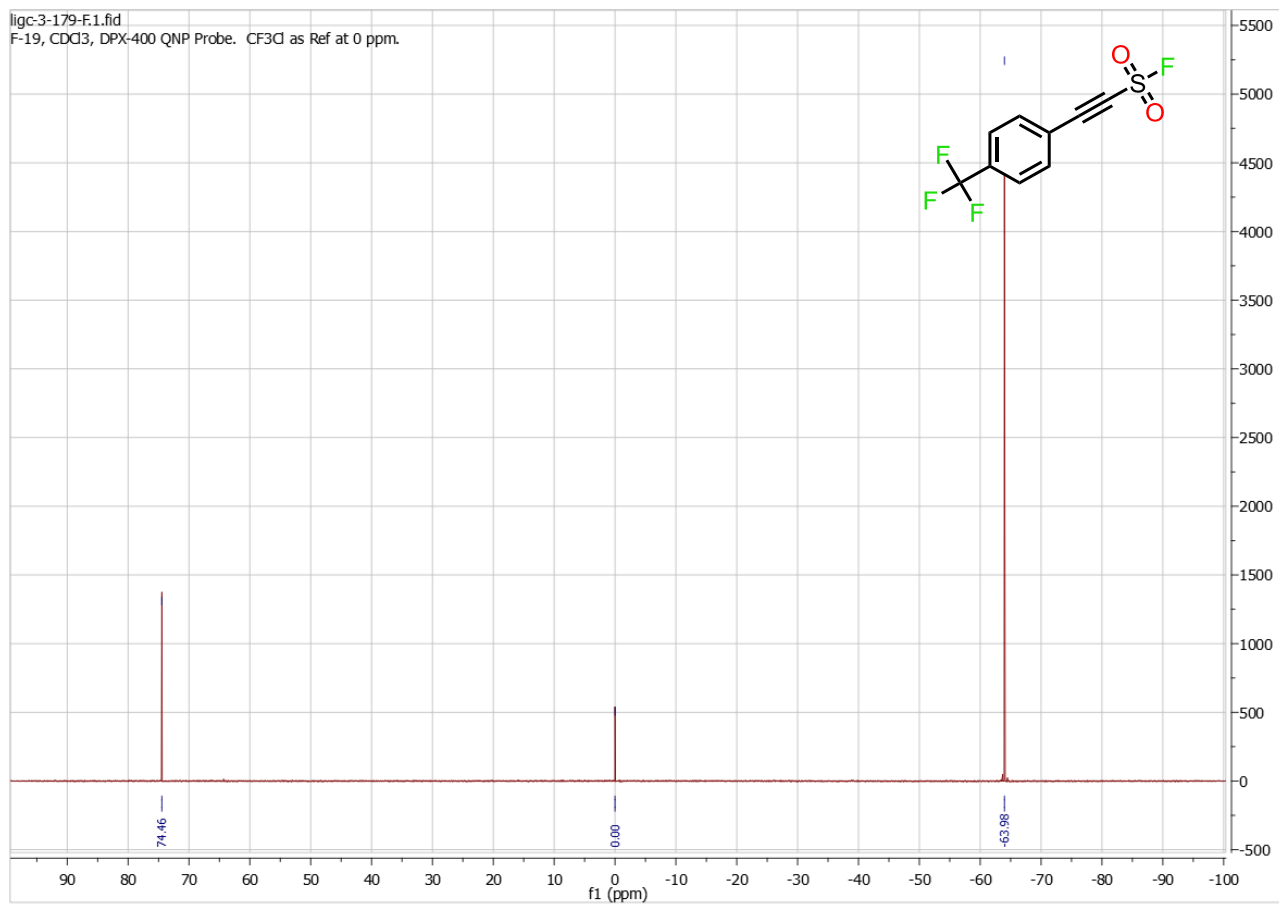
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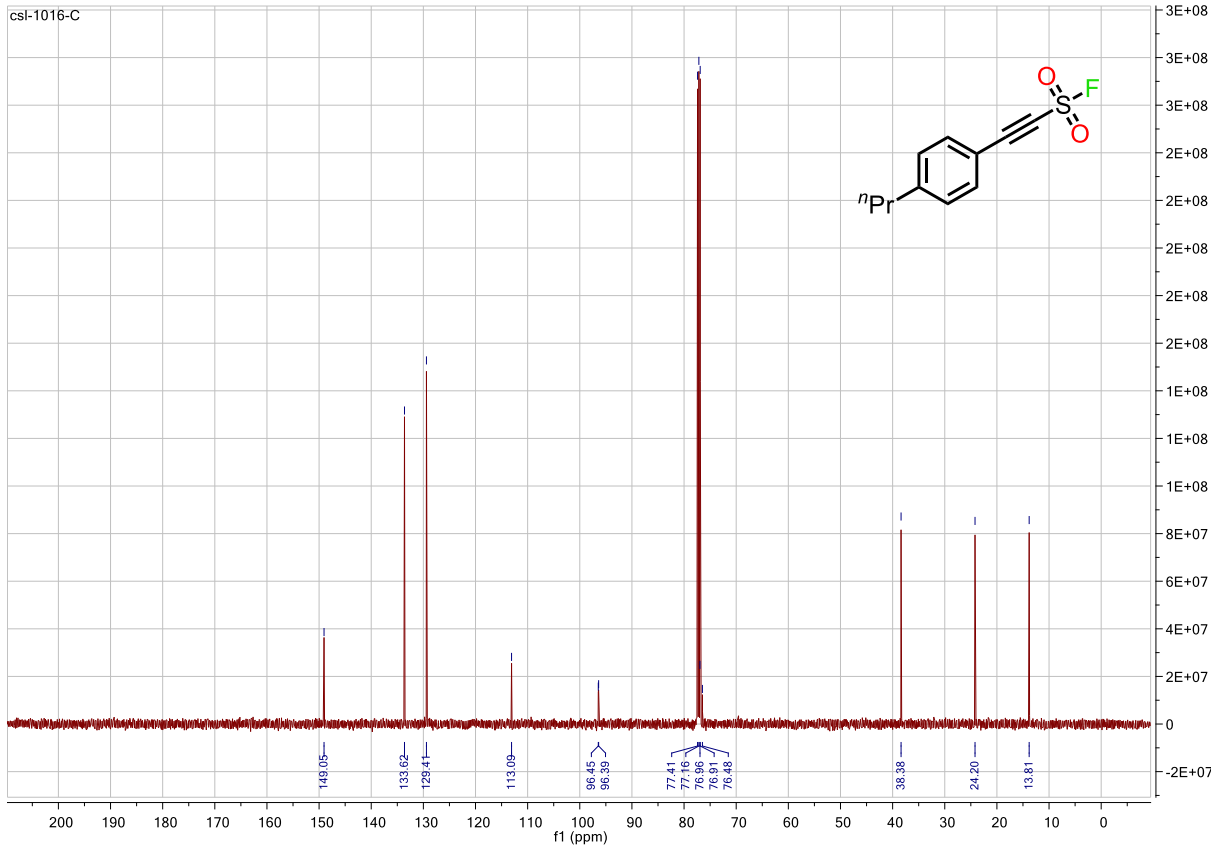
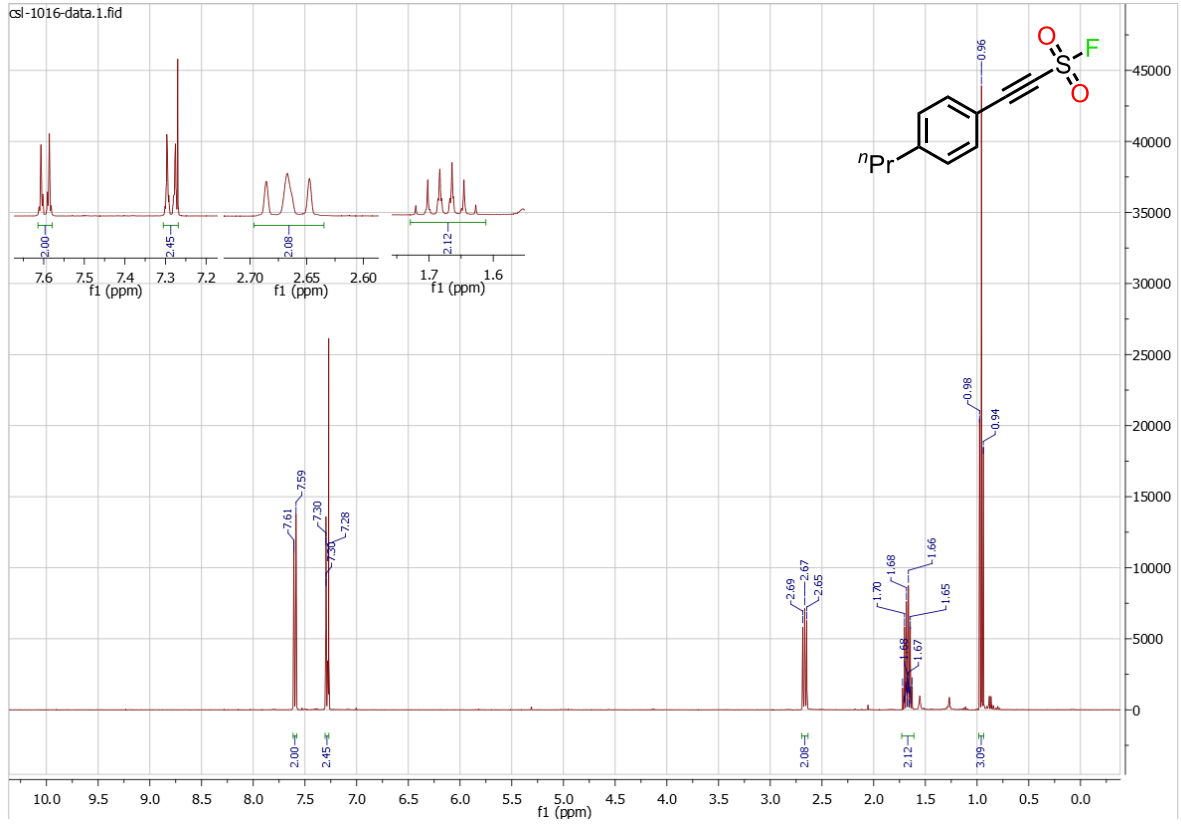


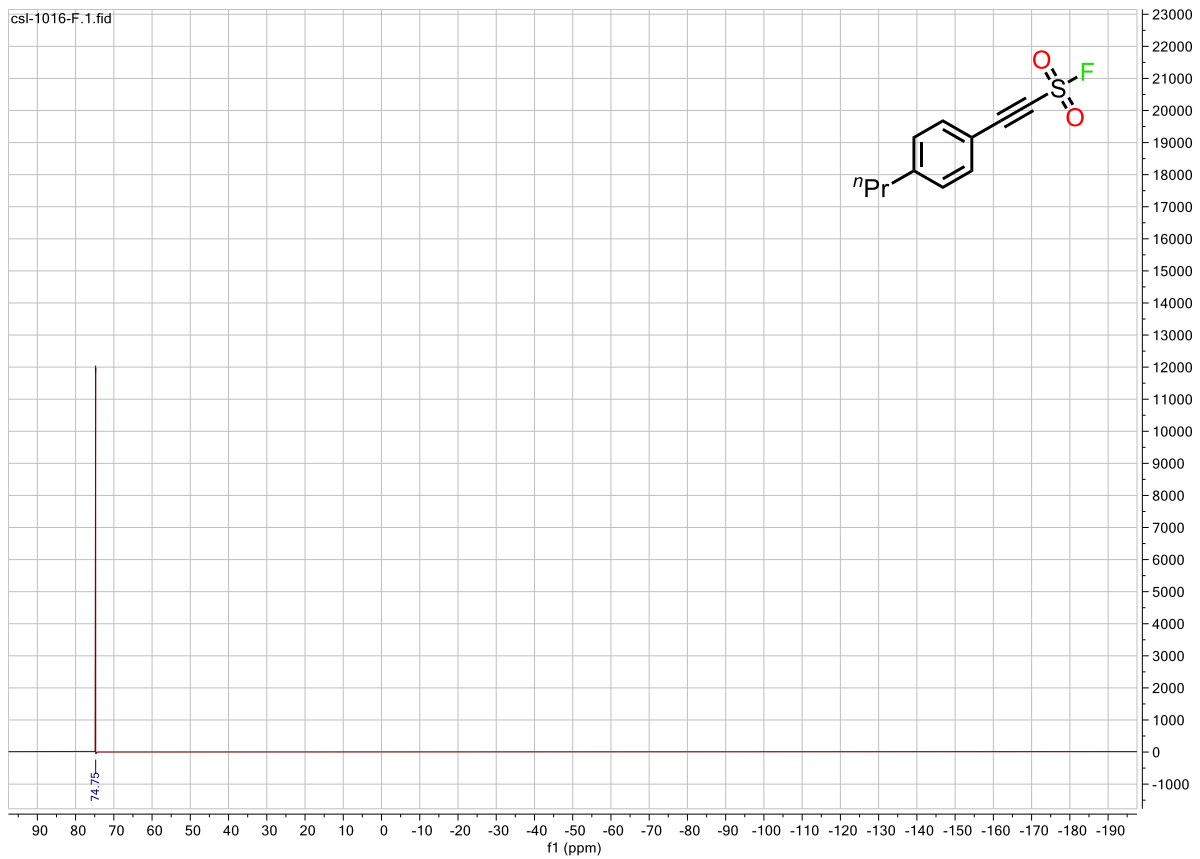
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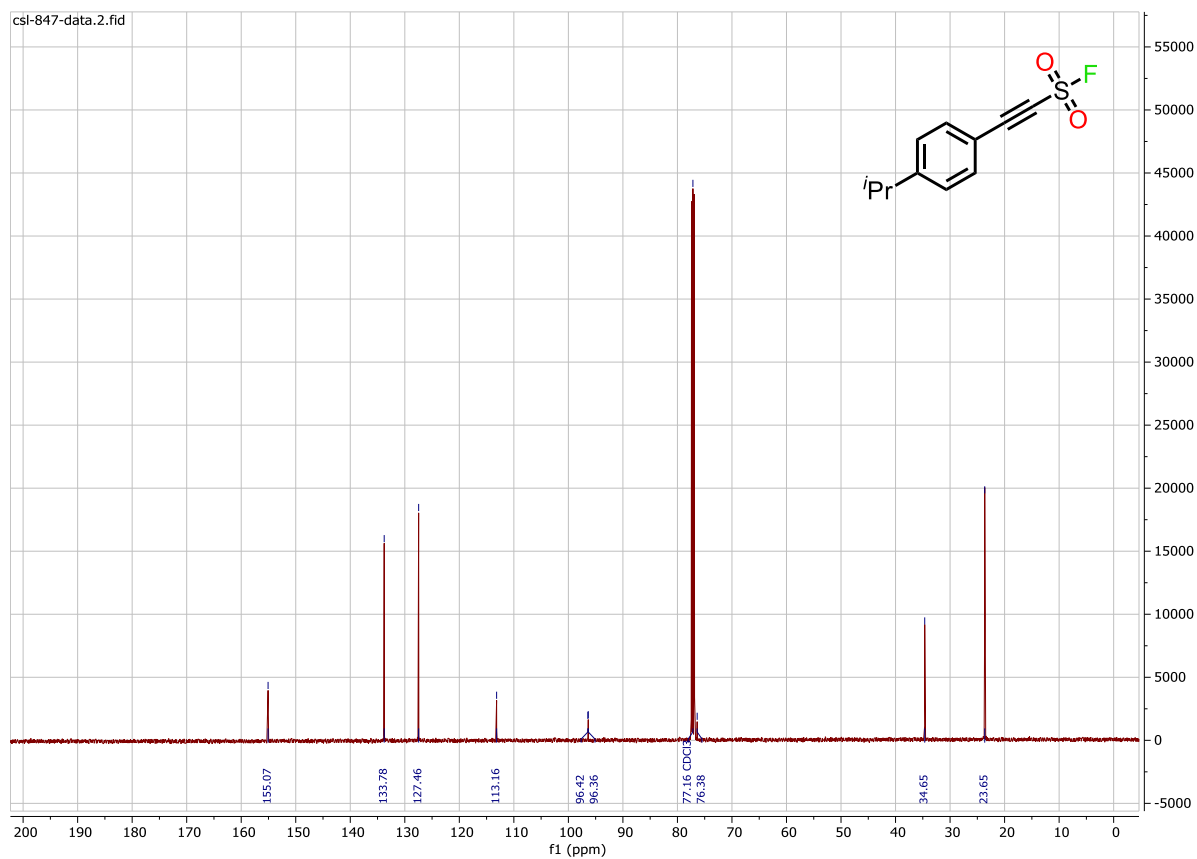
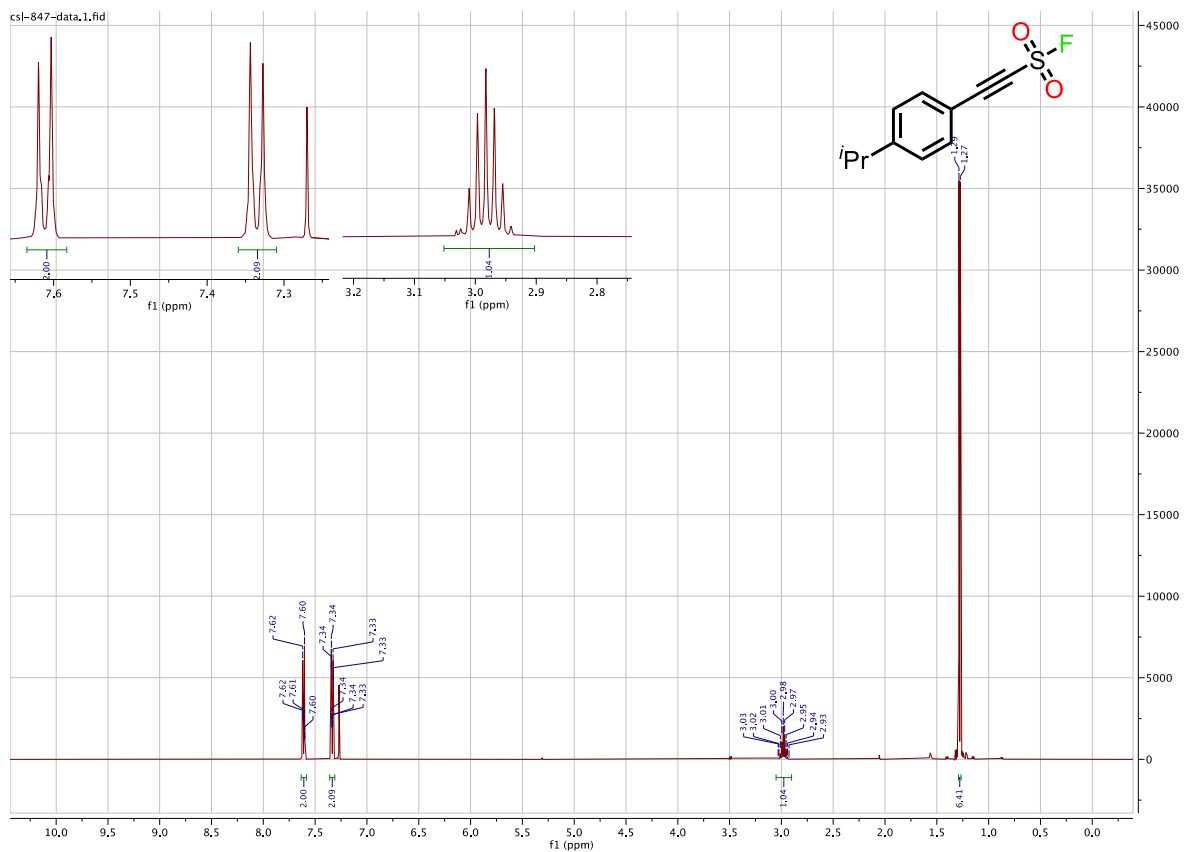


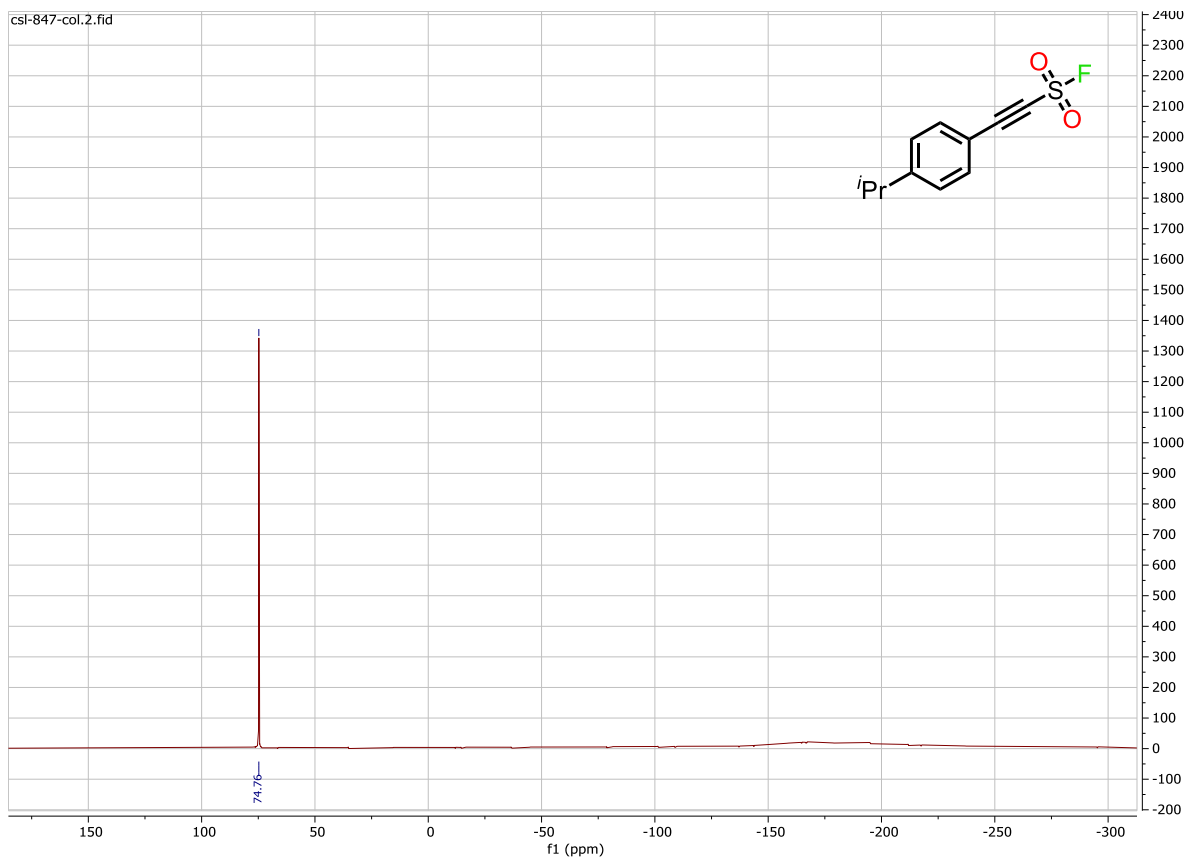
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F-19, CDCl3, DPX-400 QNP Probe. CF3Cl as Ref at 0 ppm.

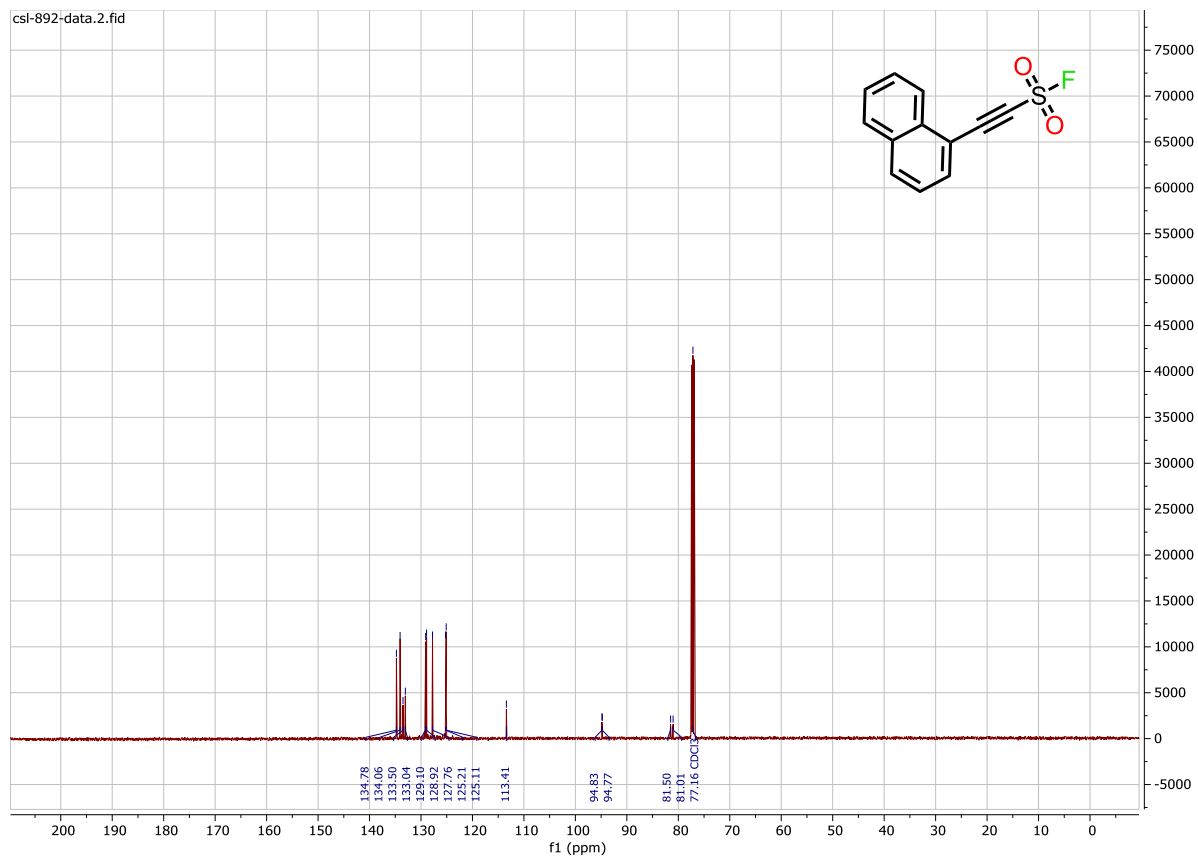
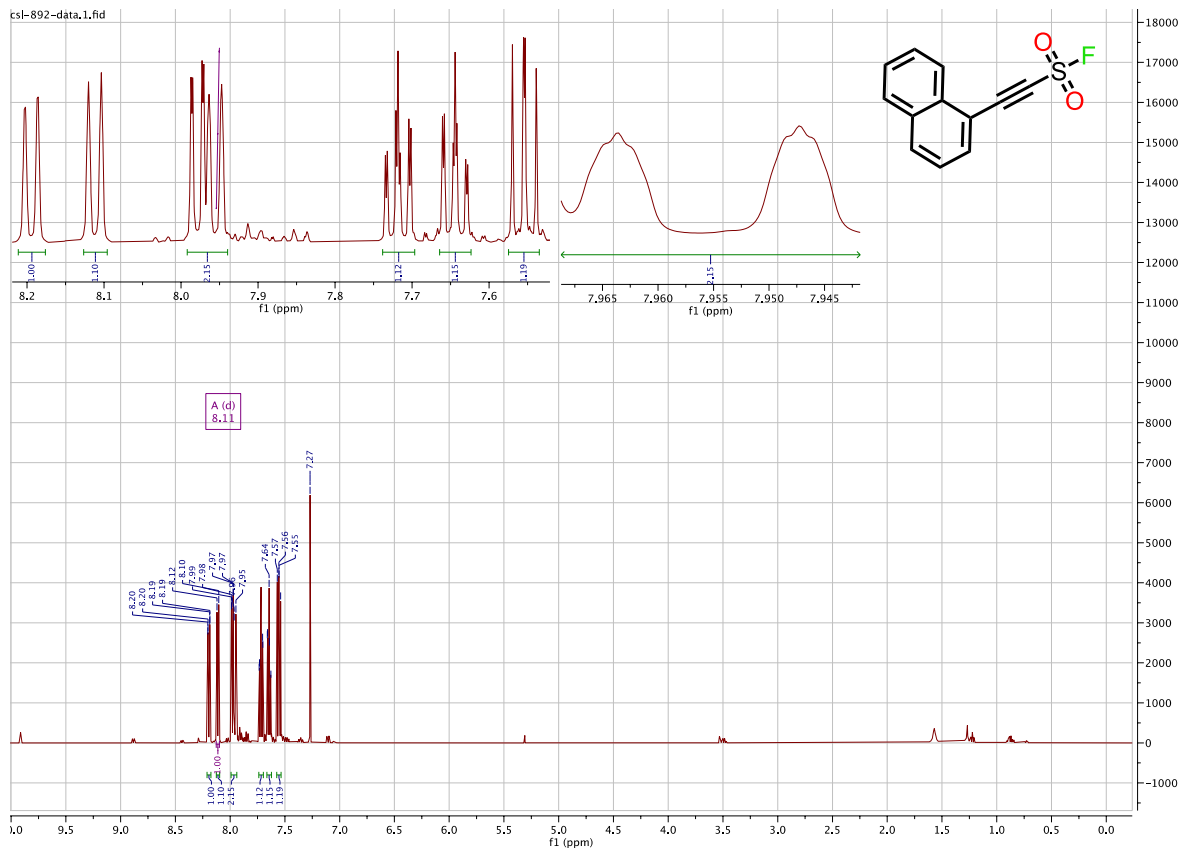


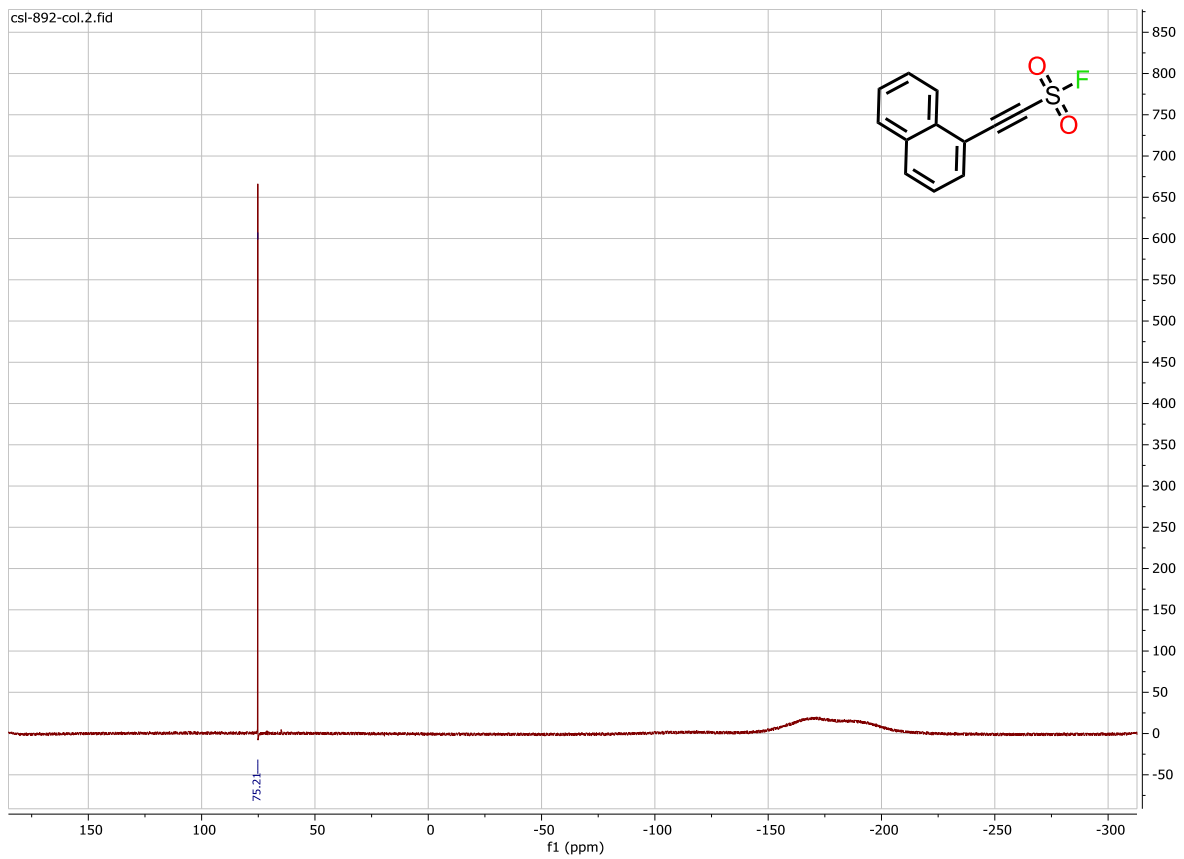


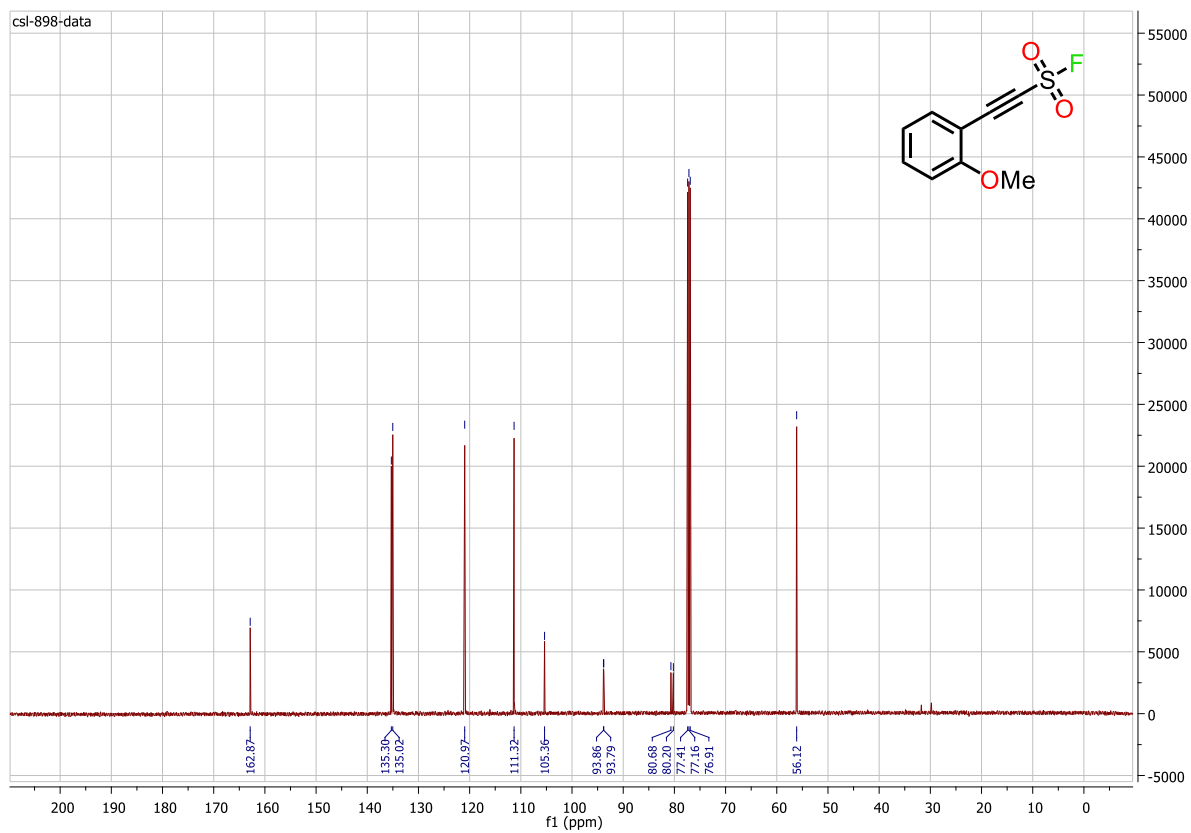
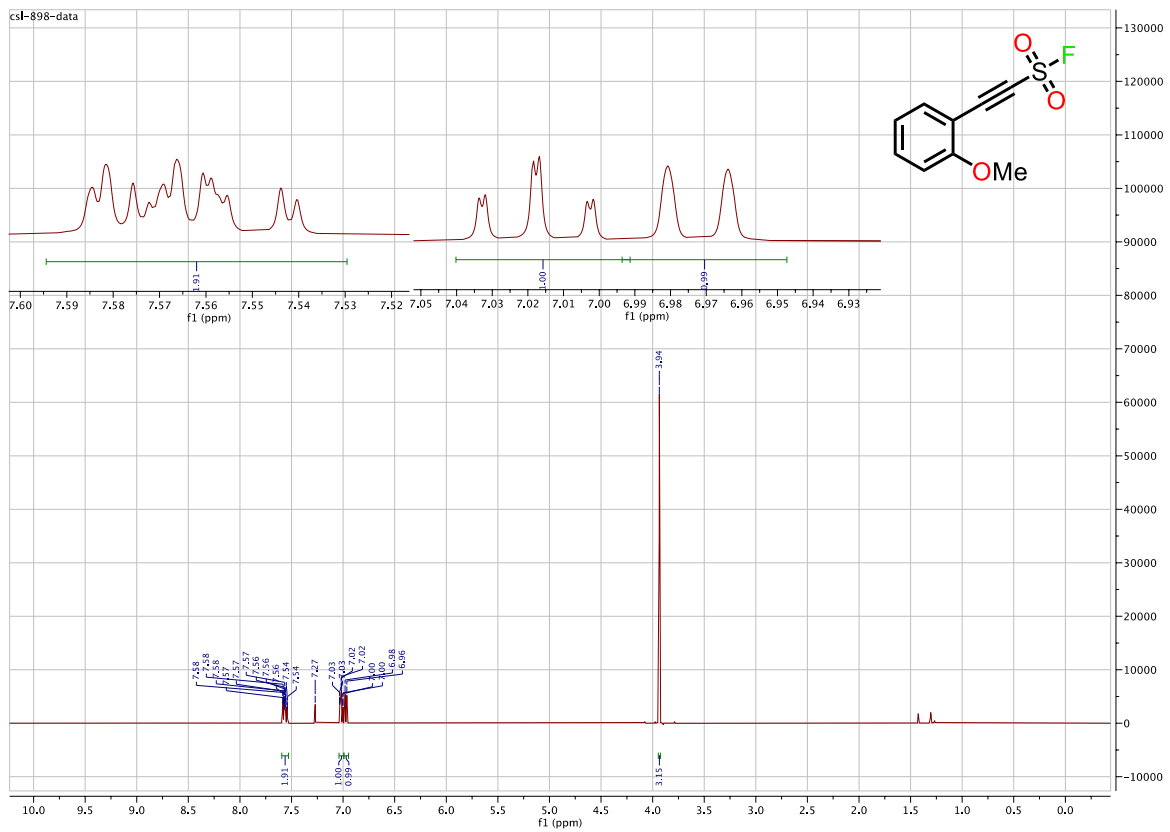


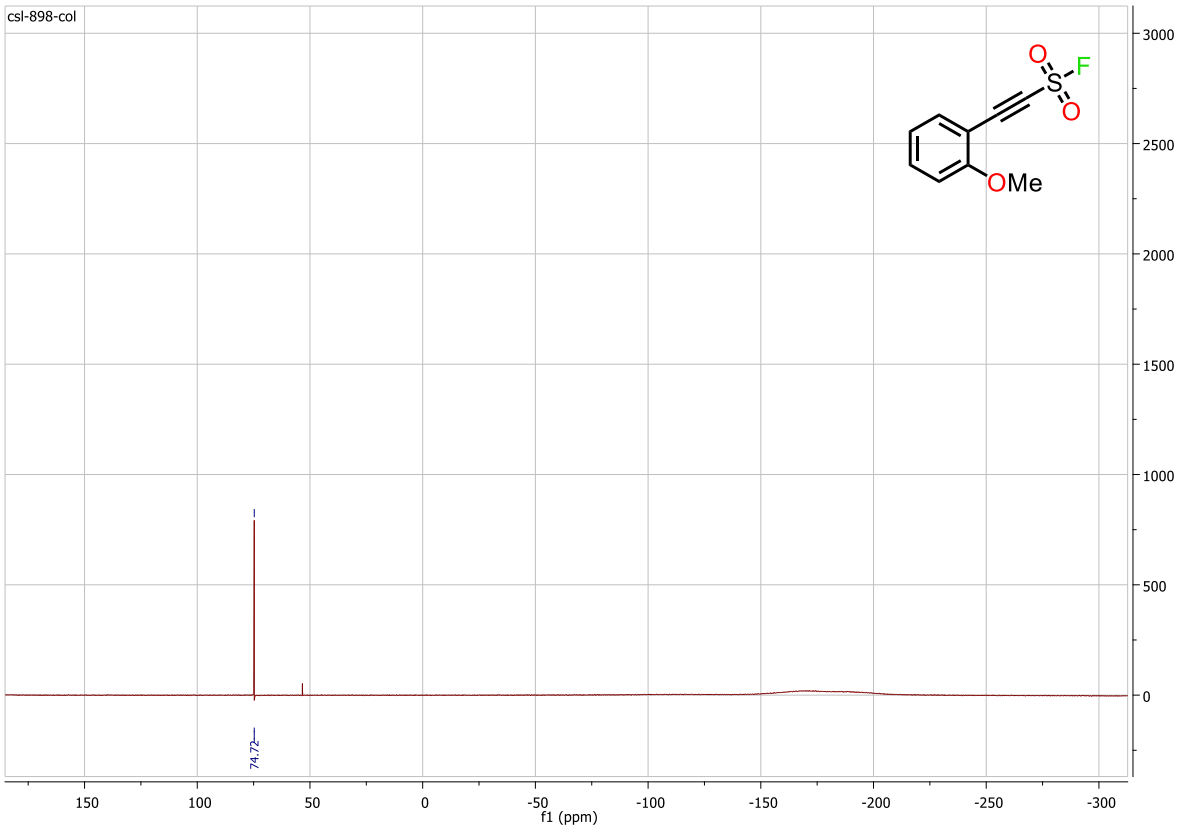


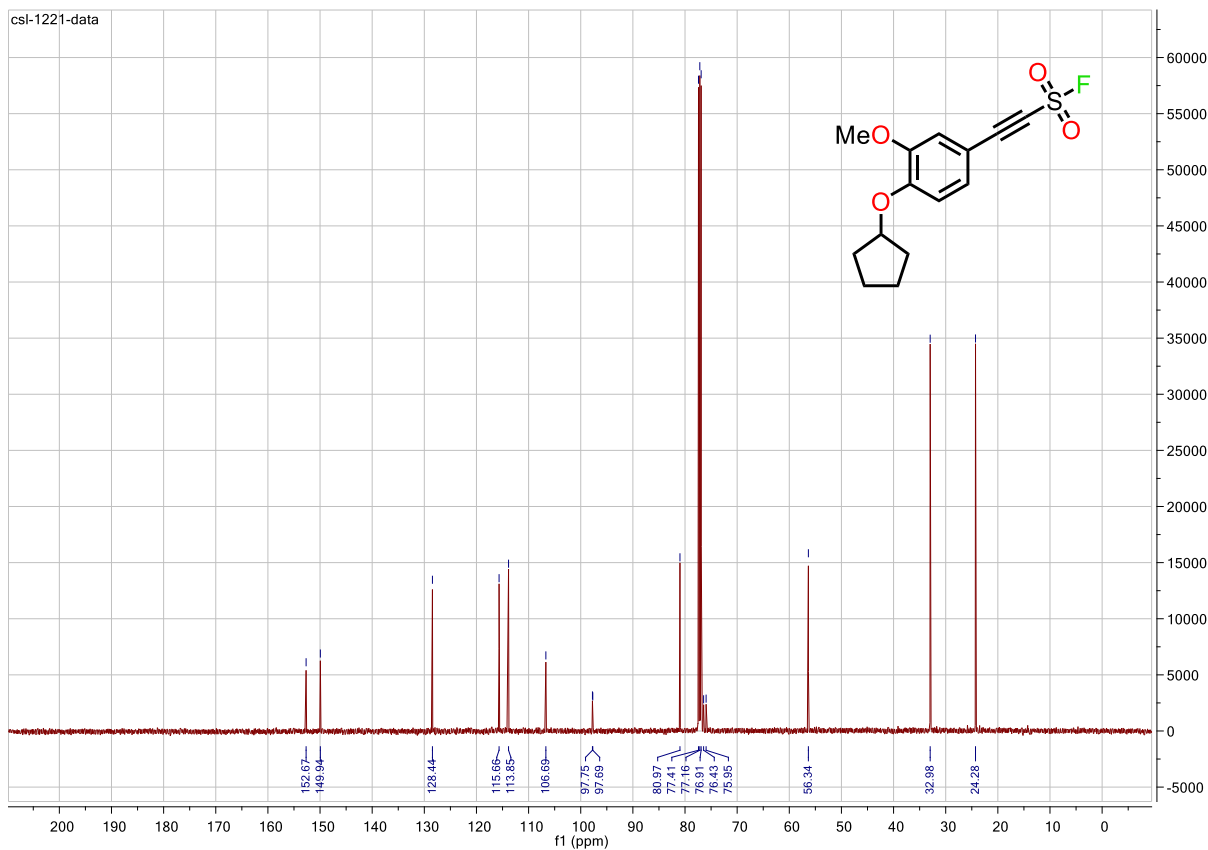
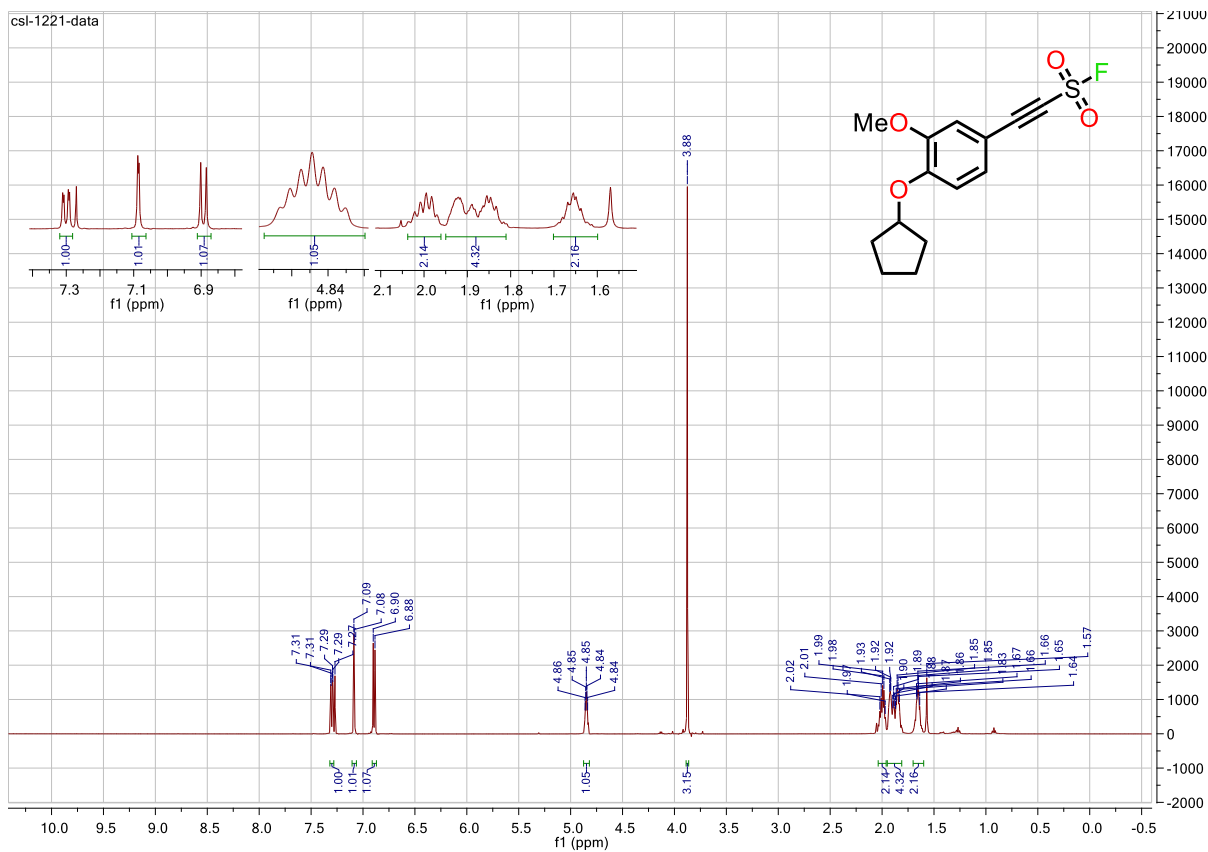


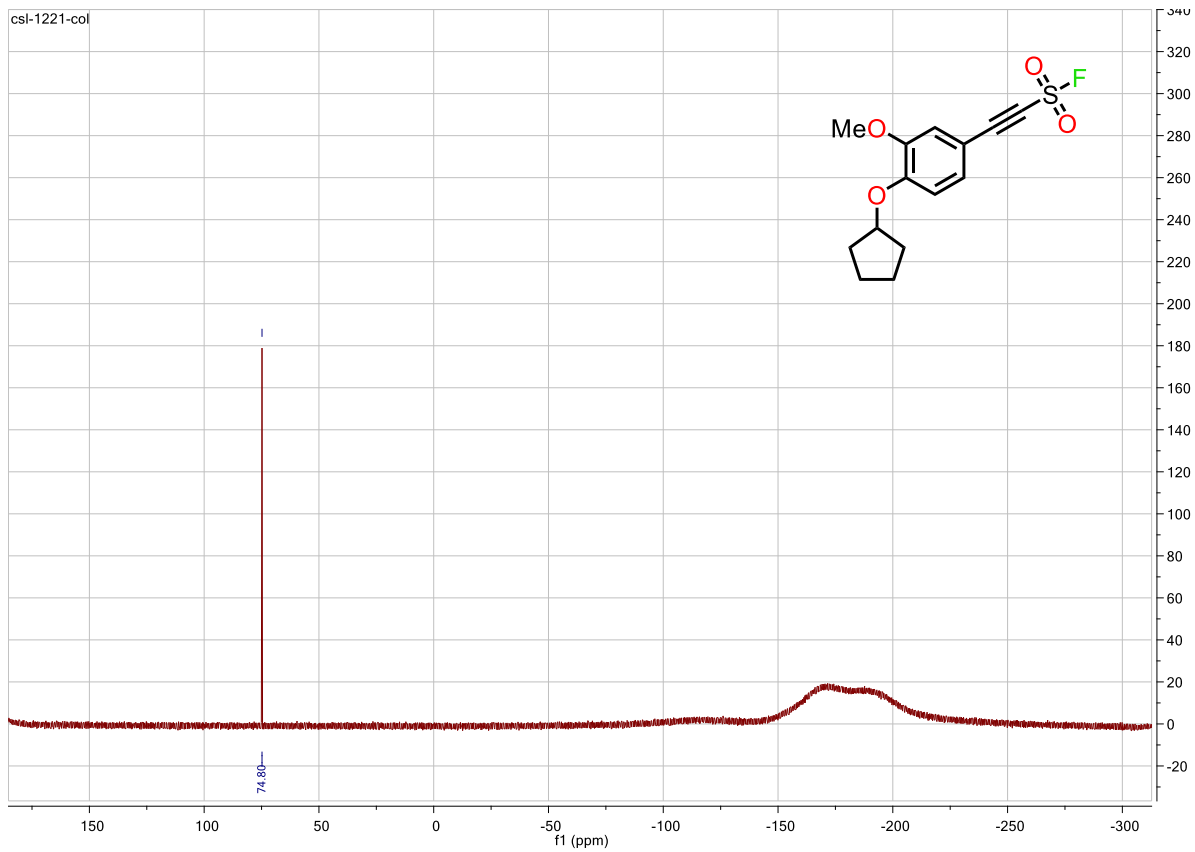


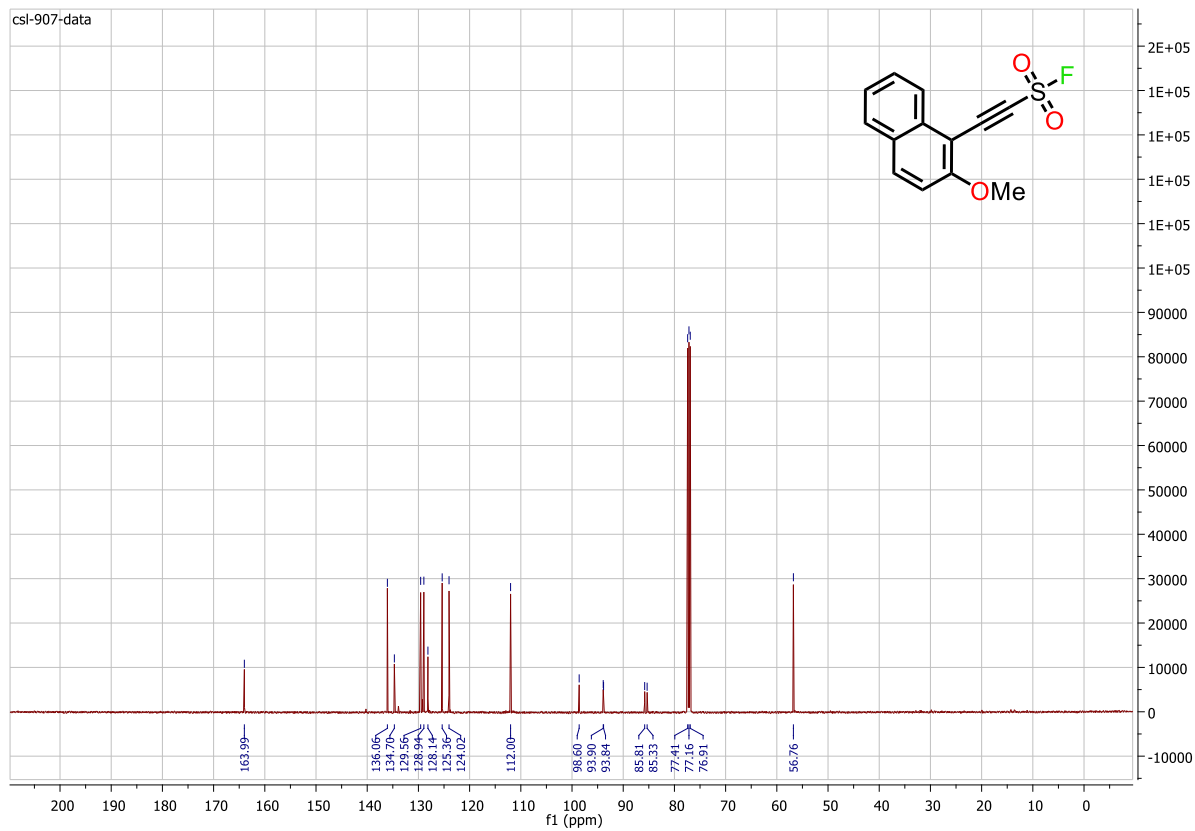
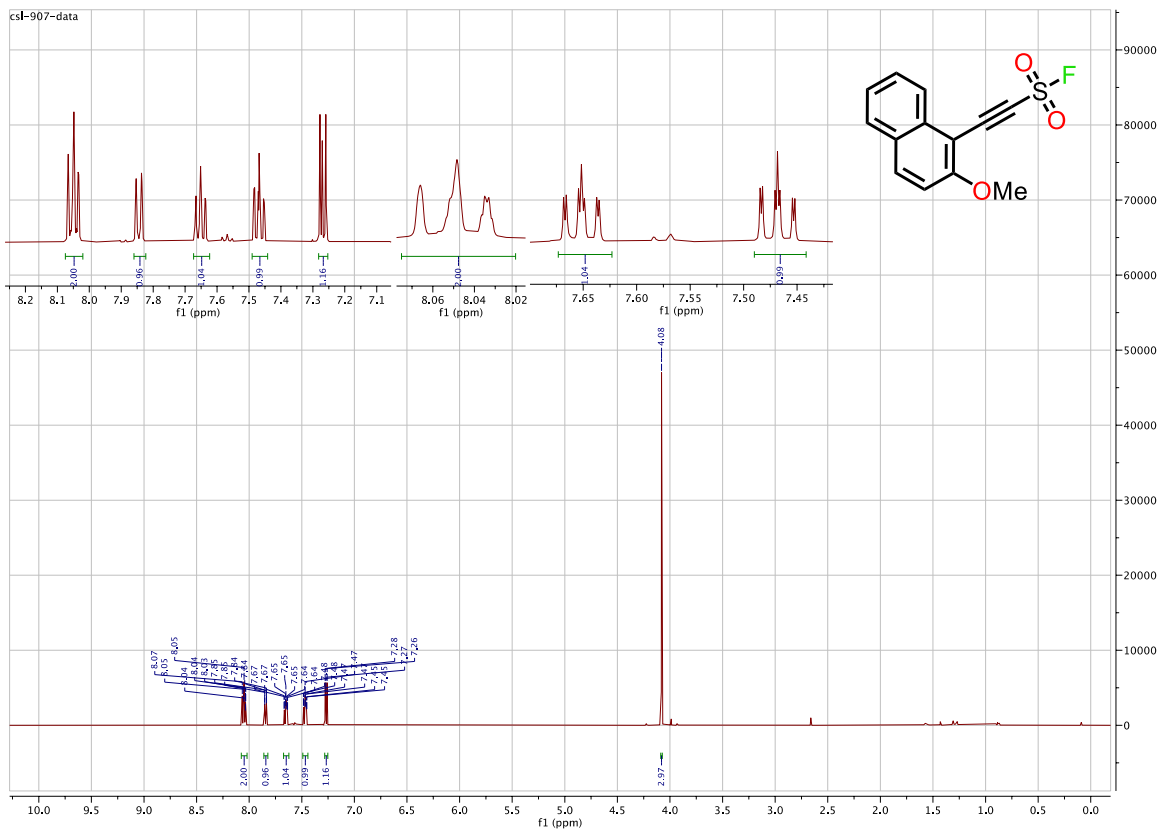


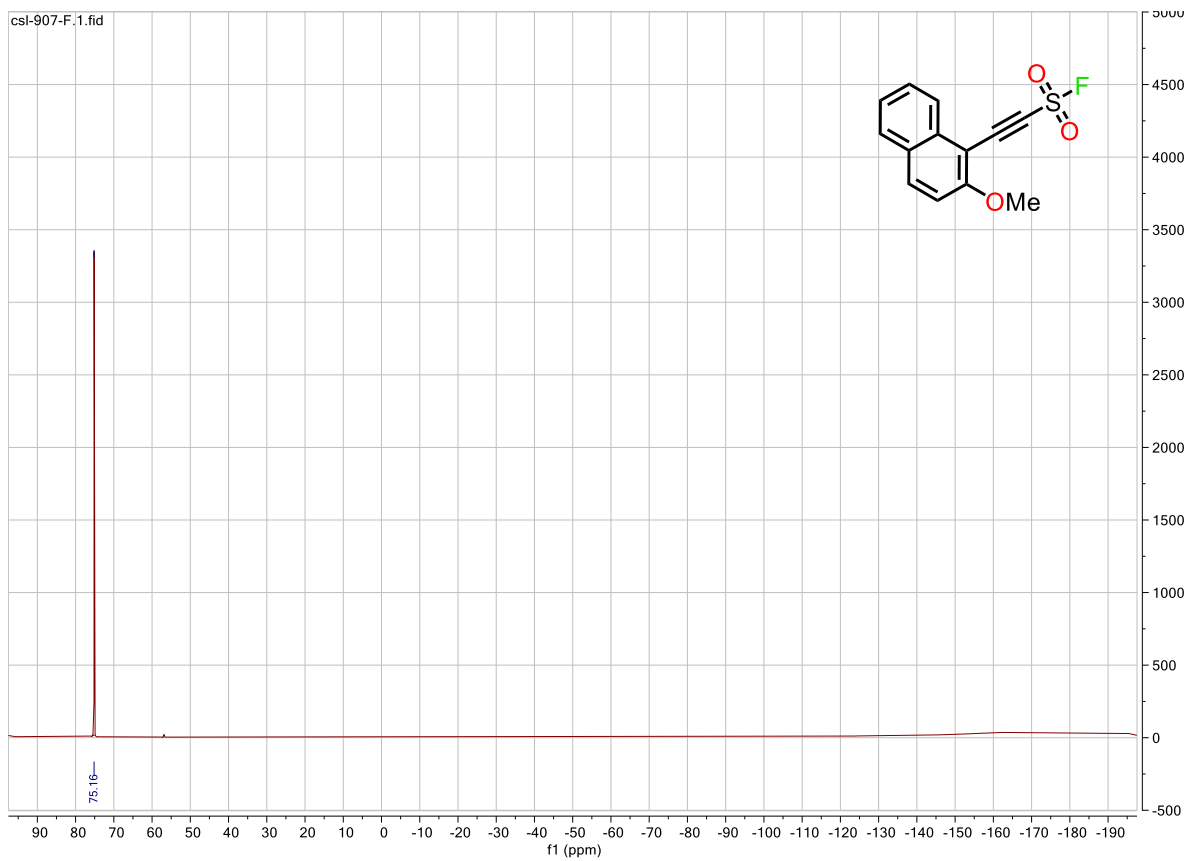


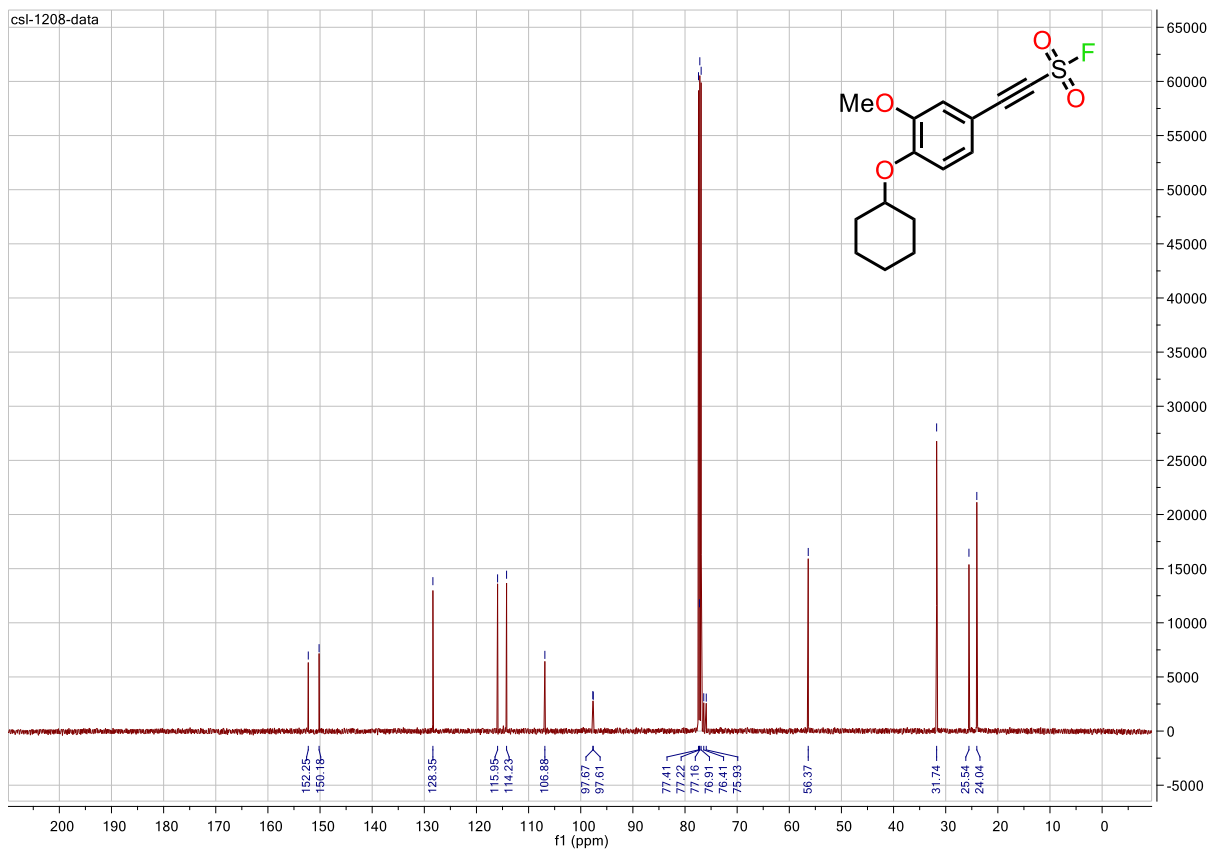
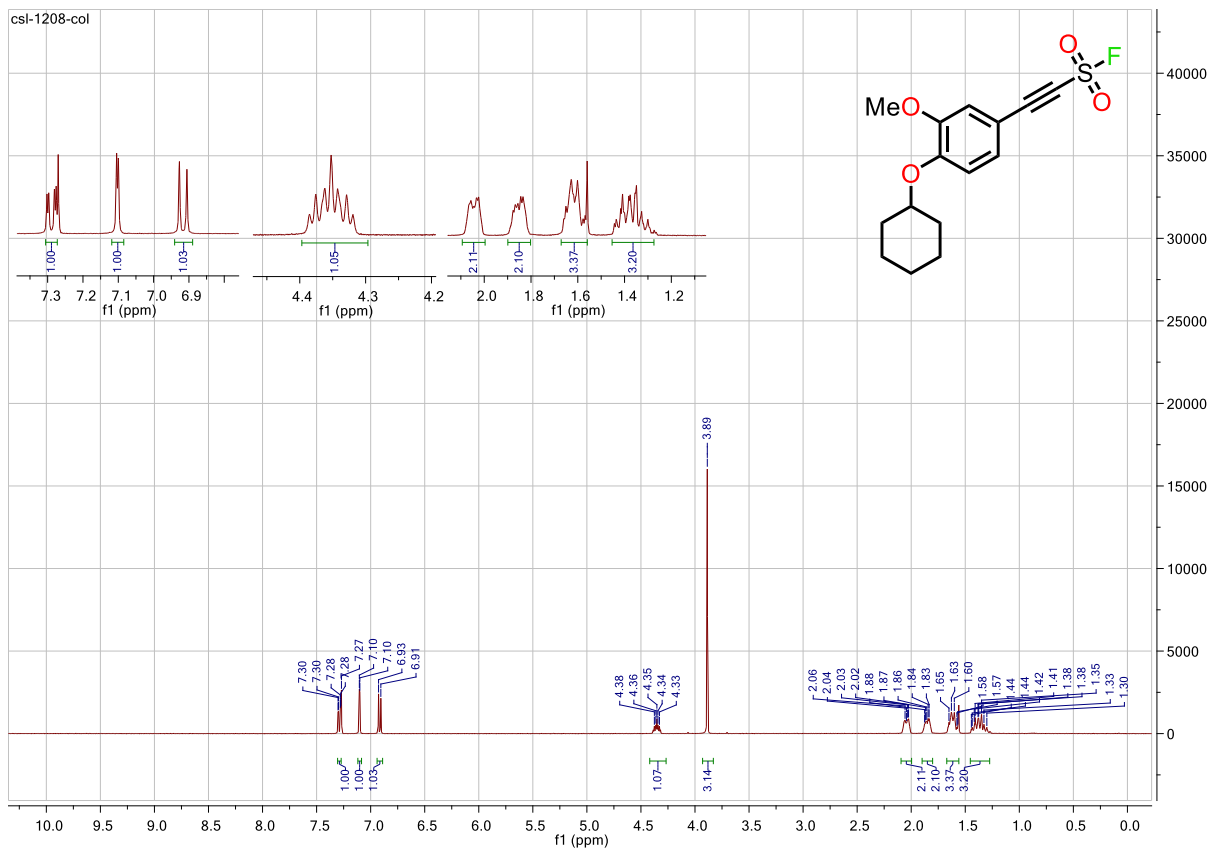


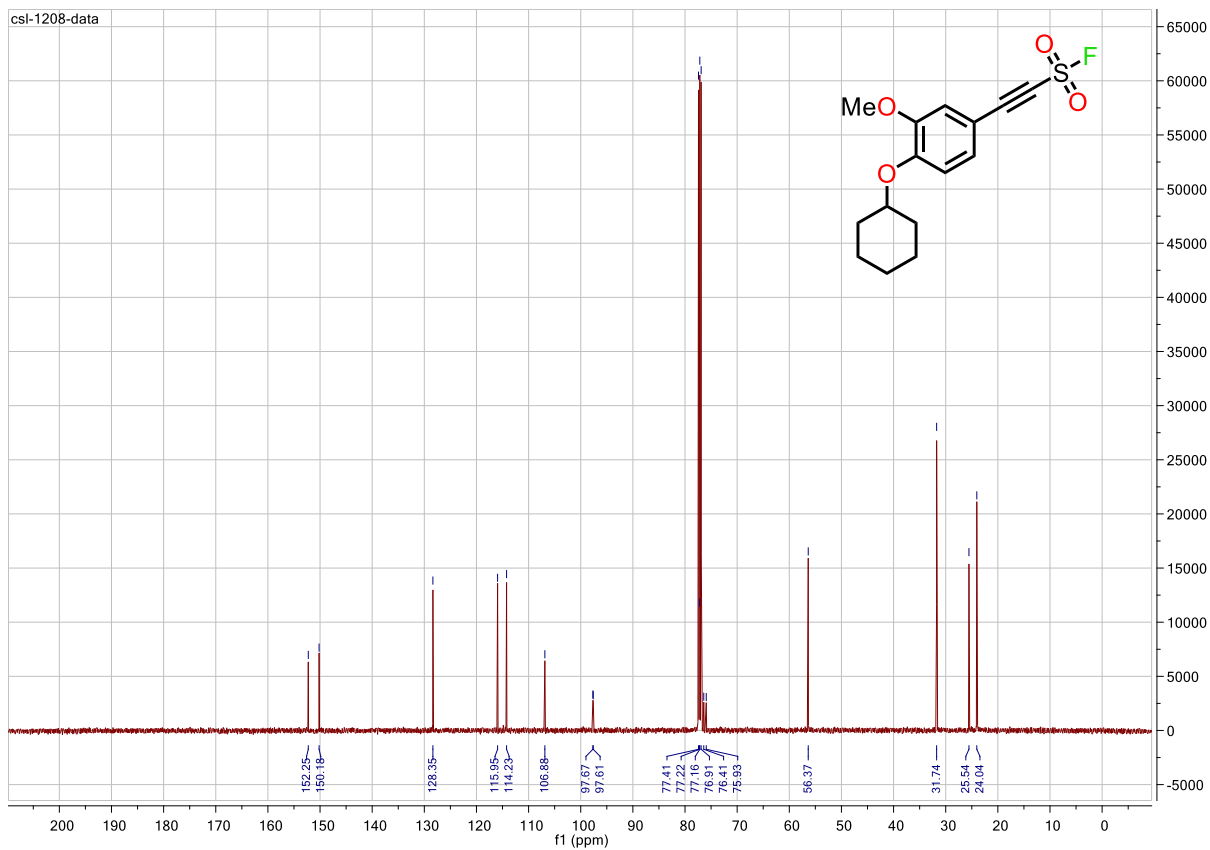


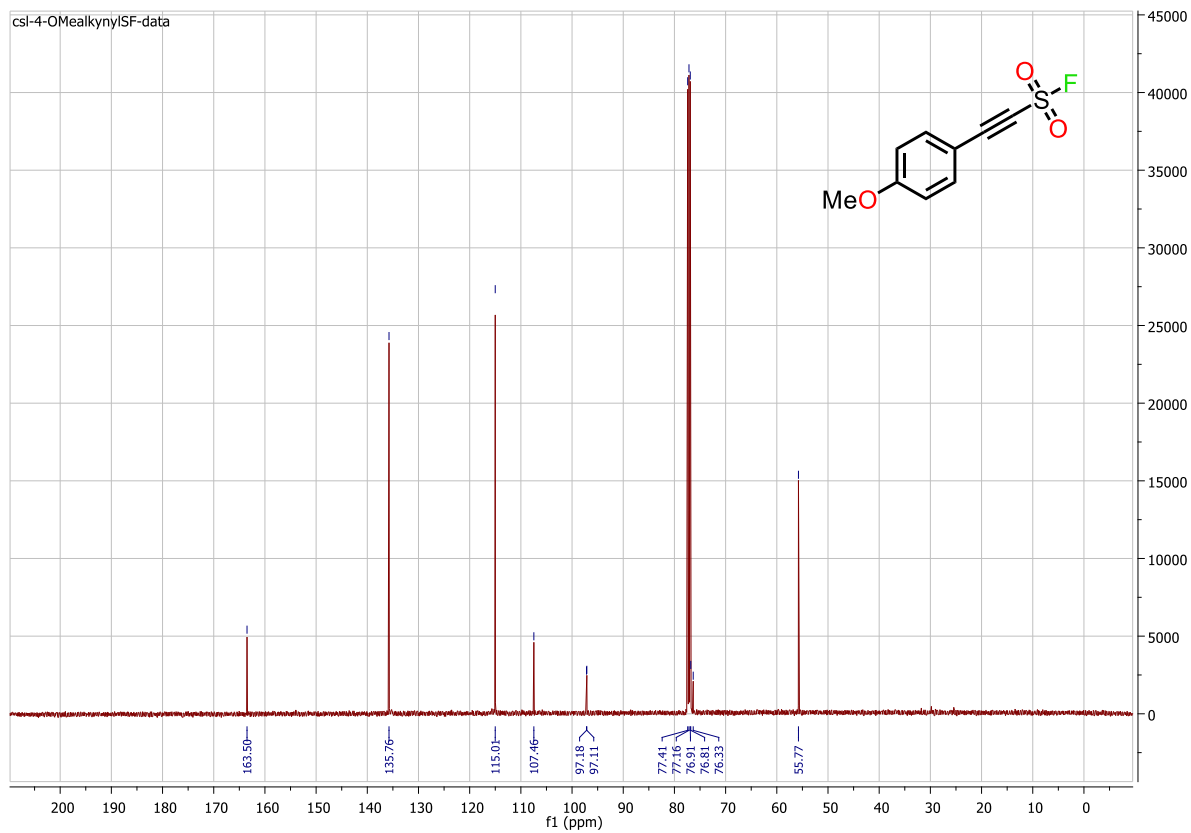
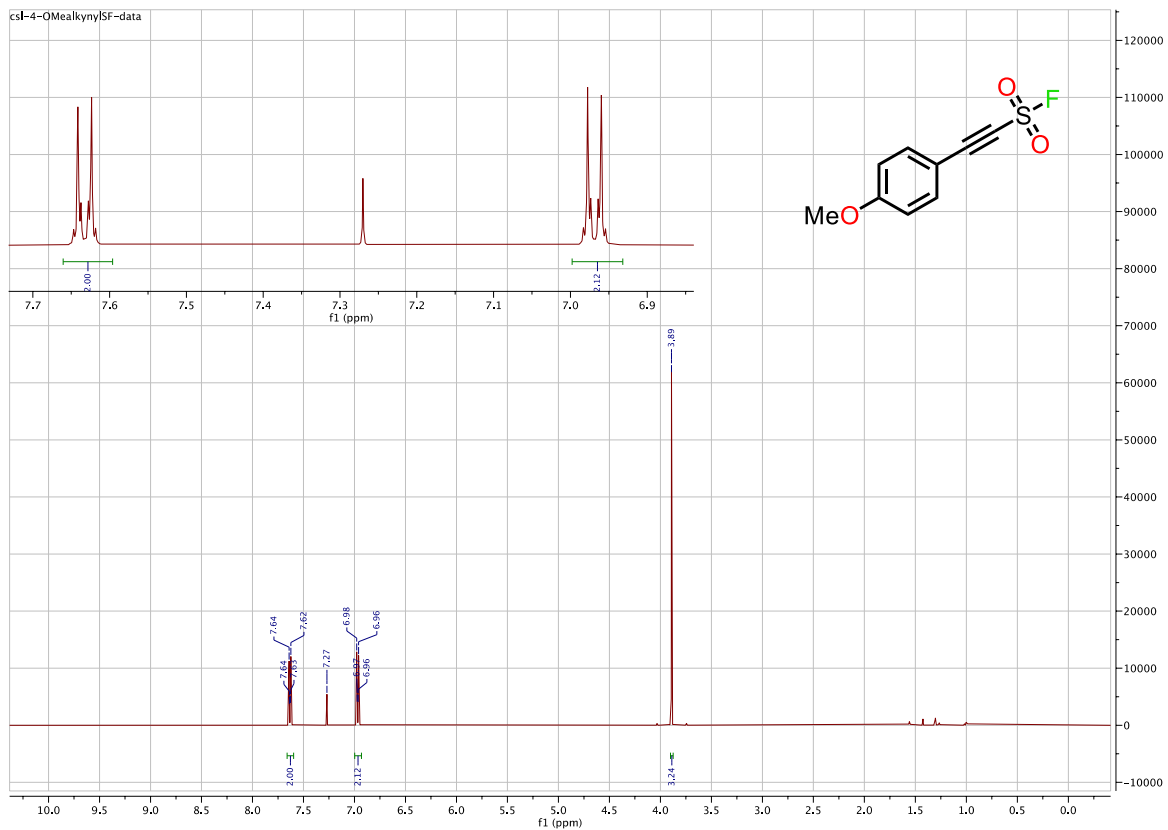


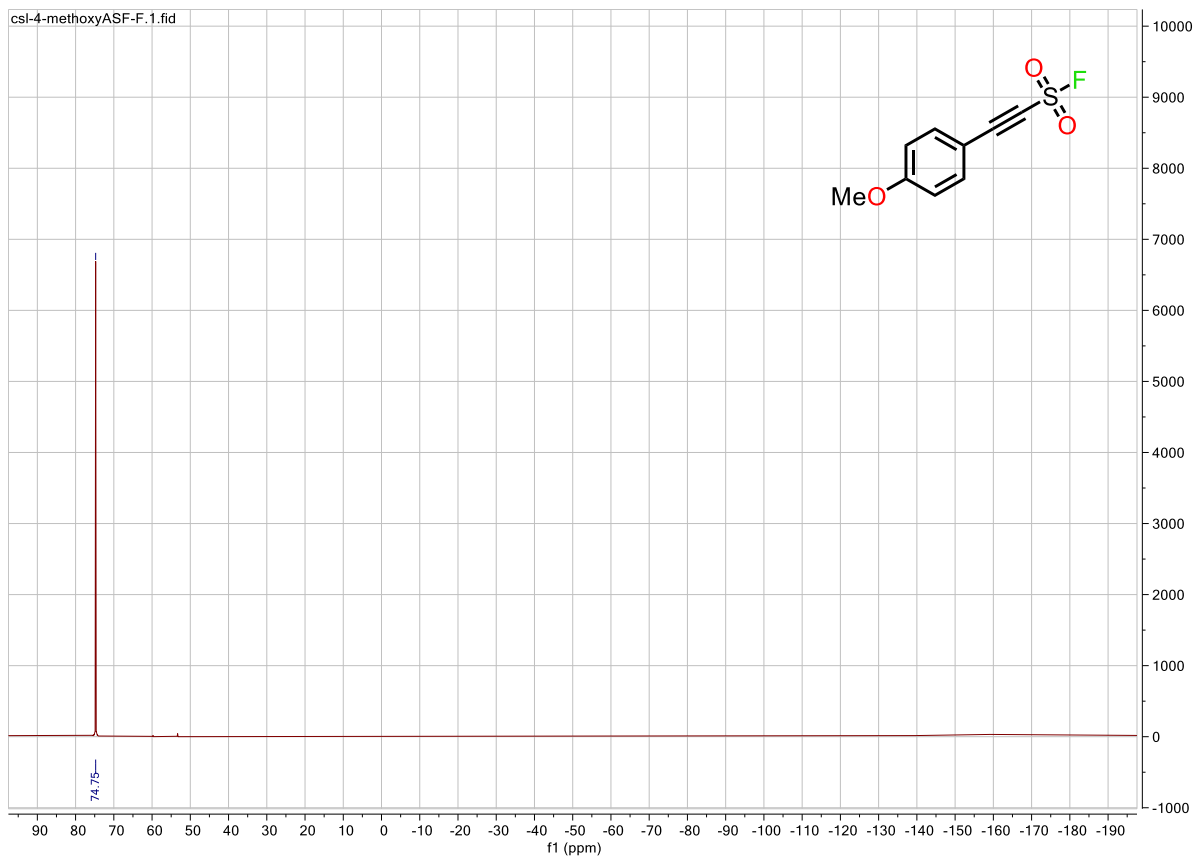


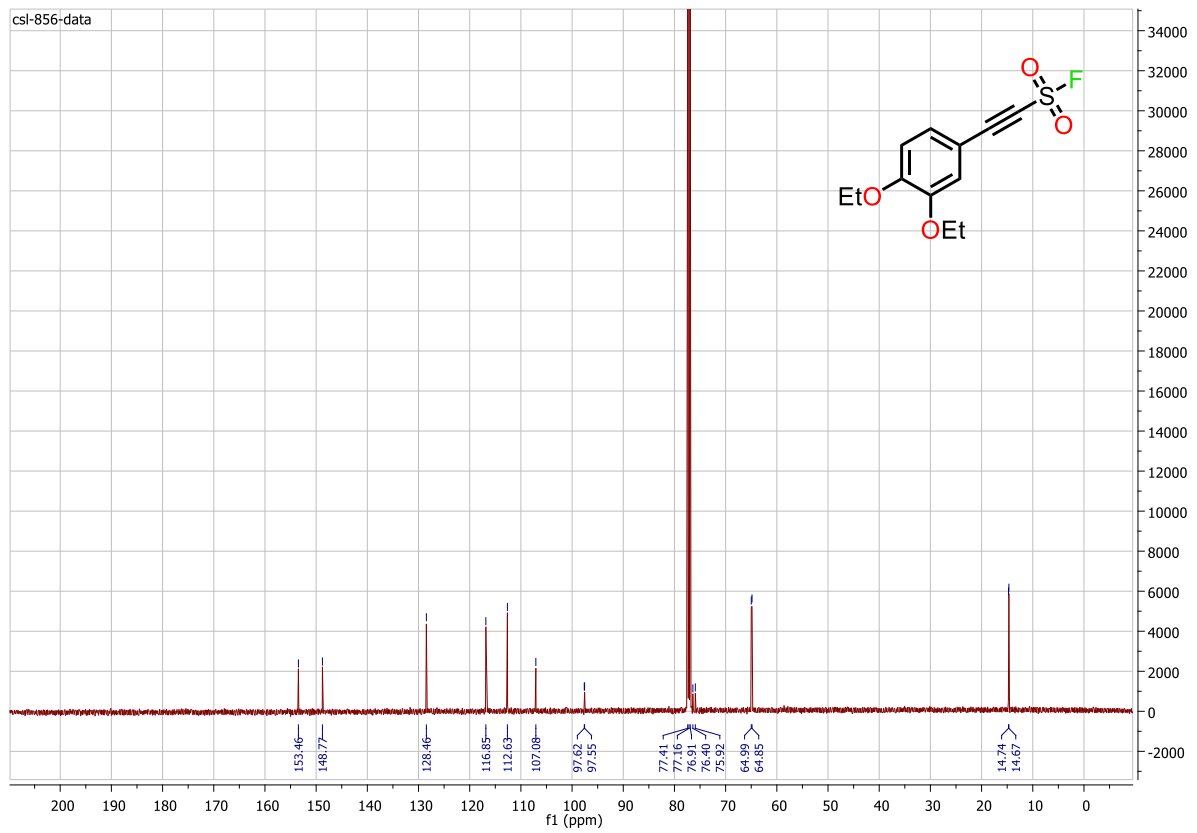
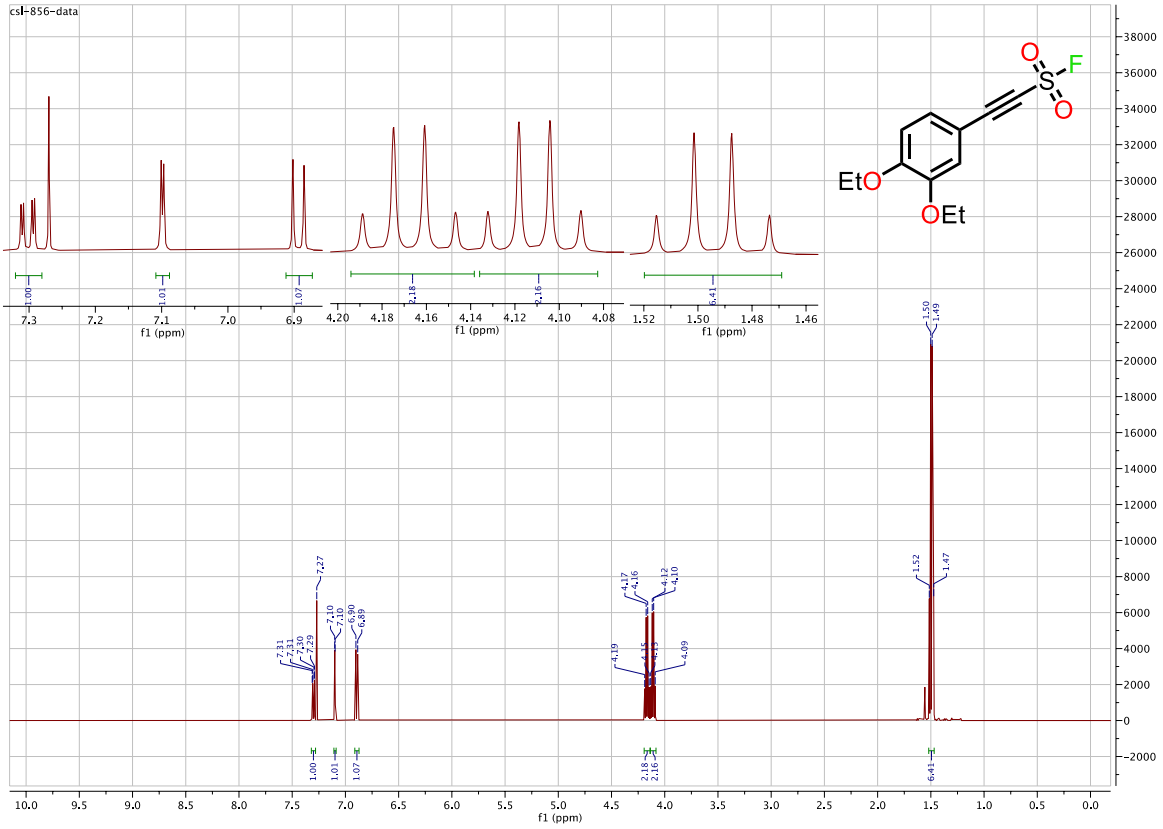


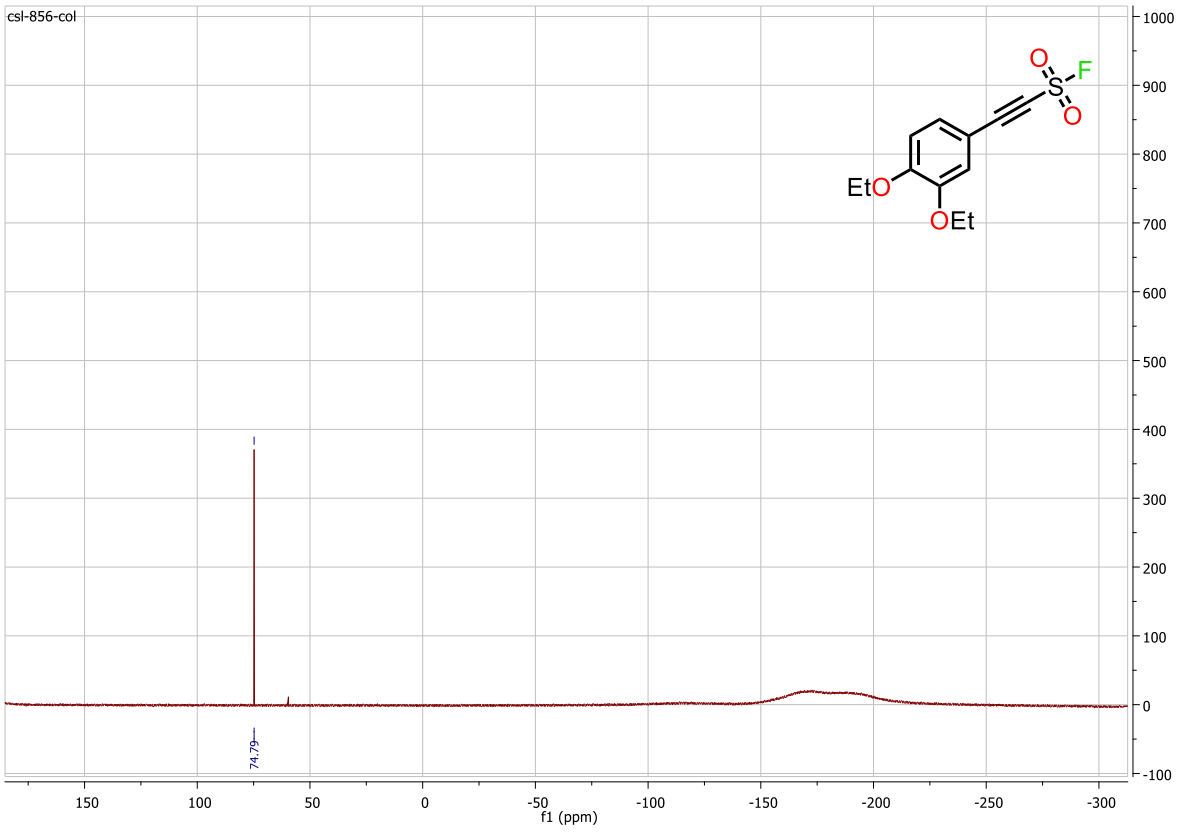


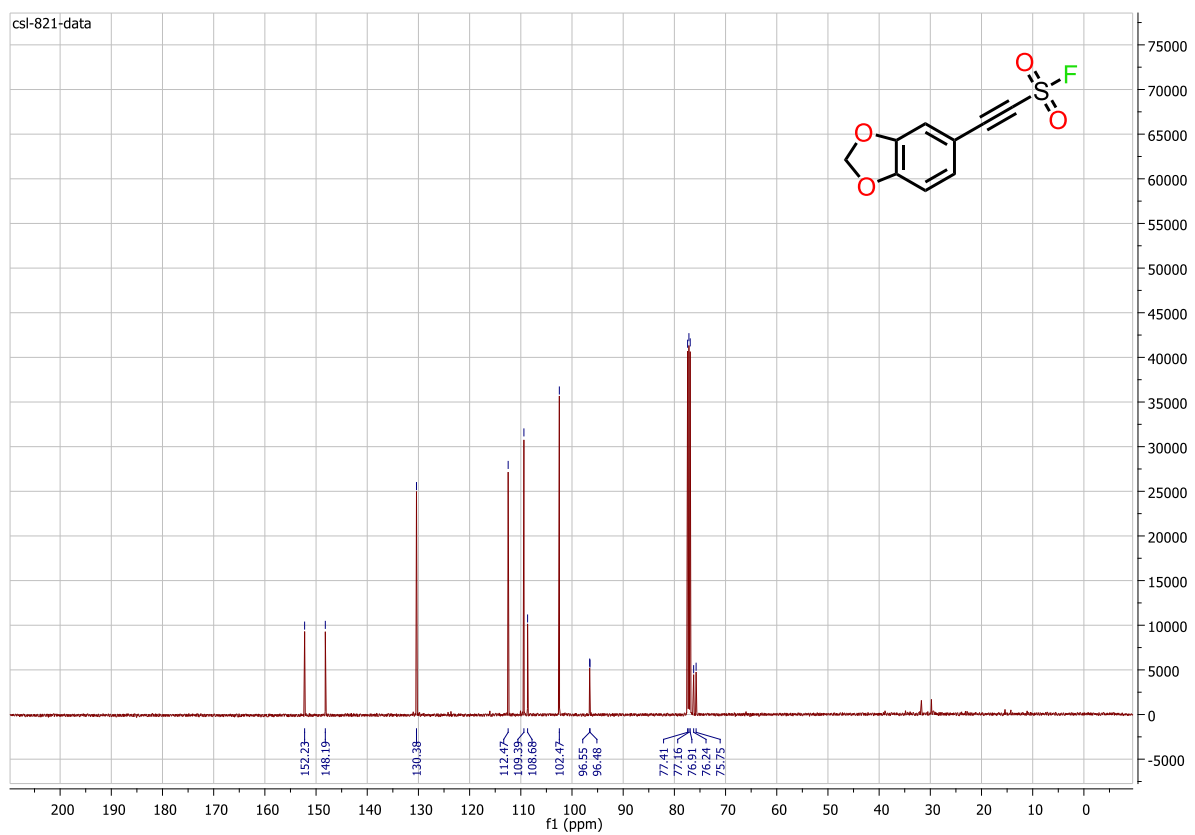
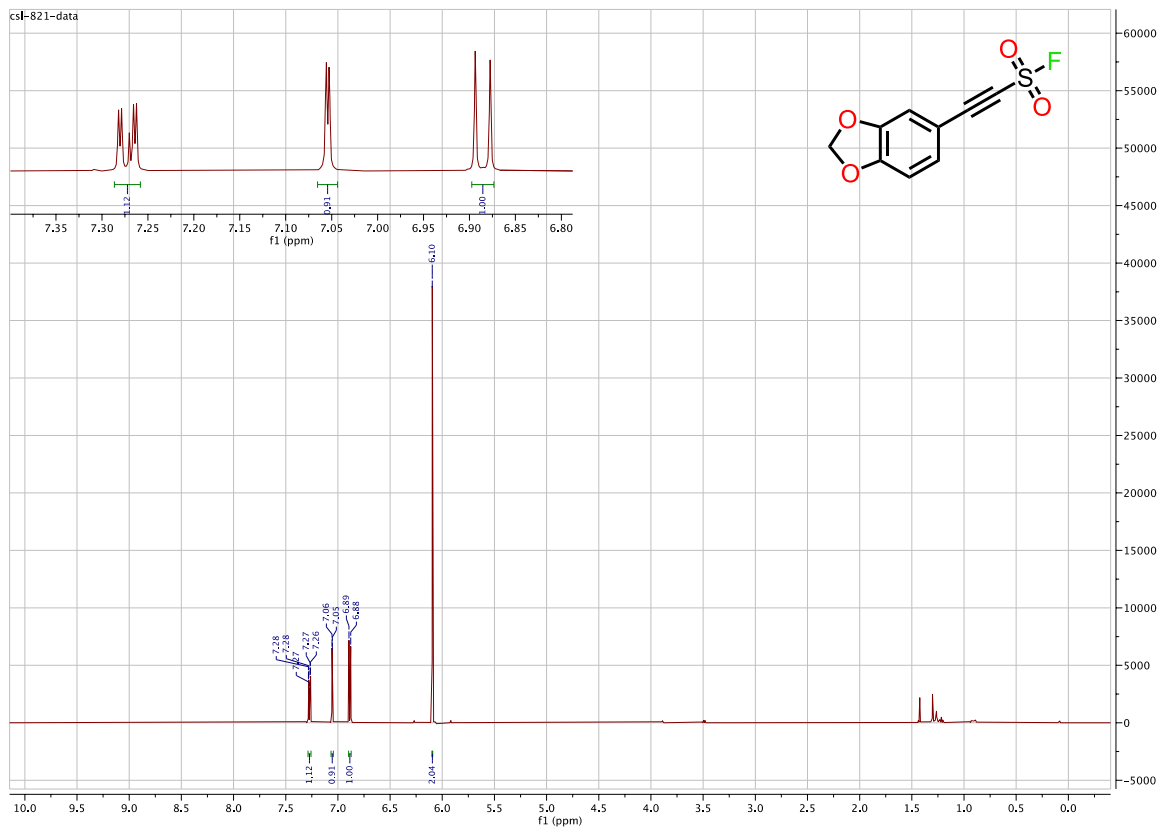




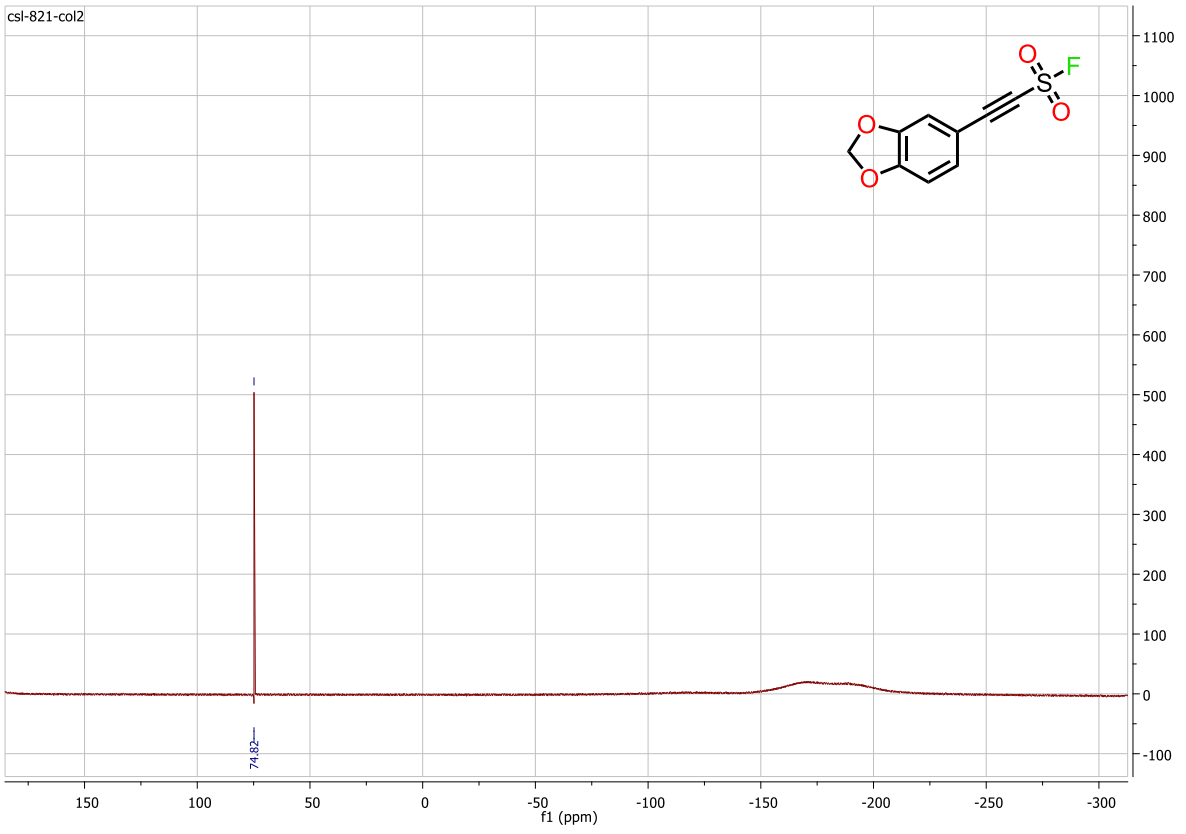


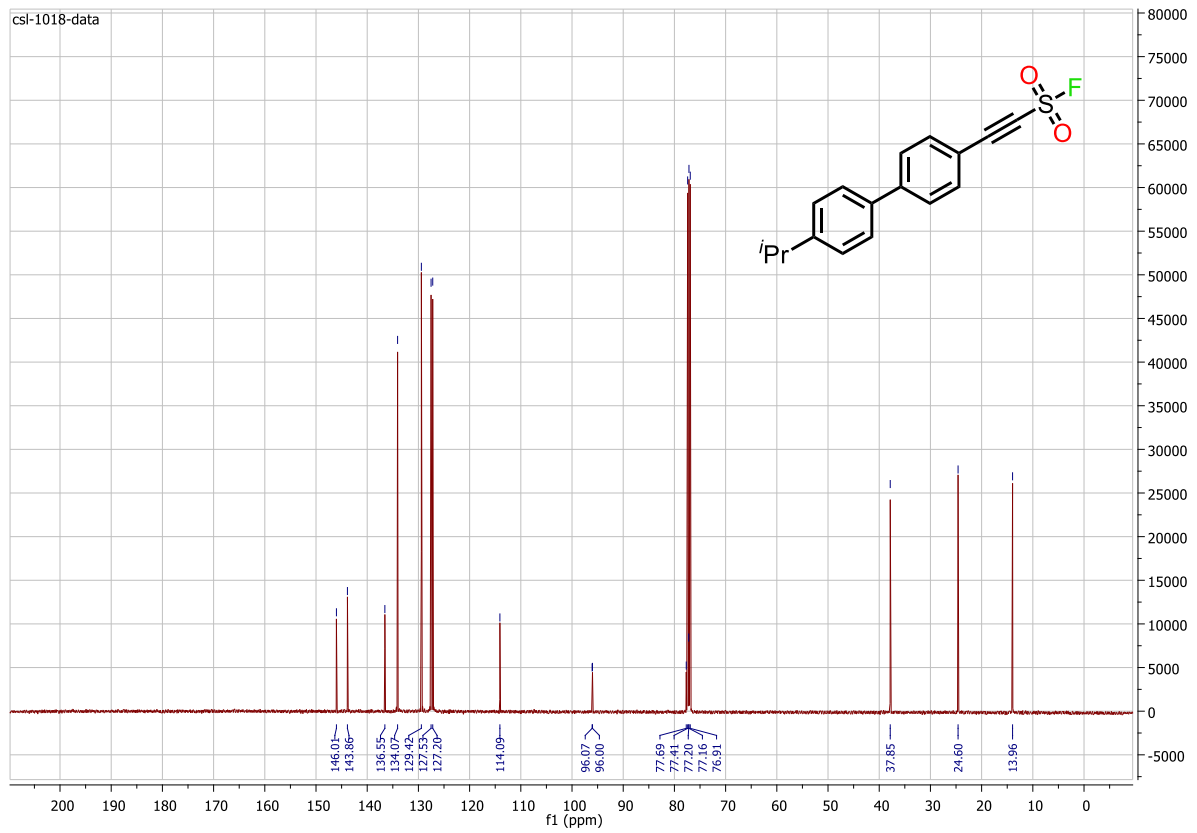
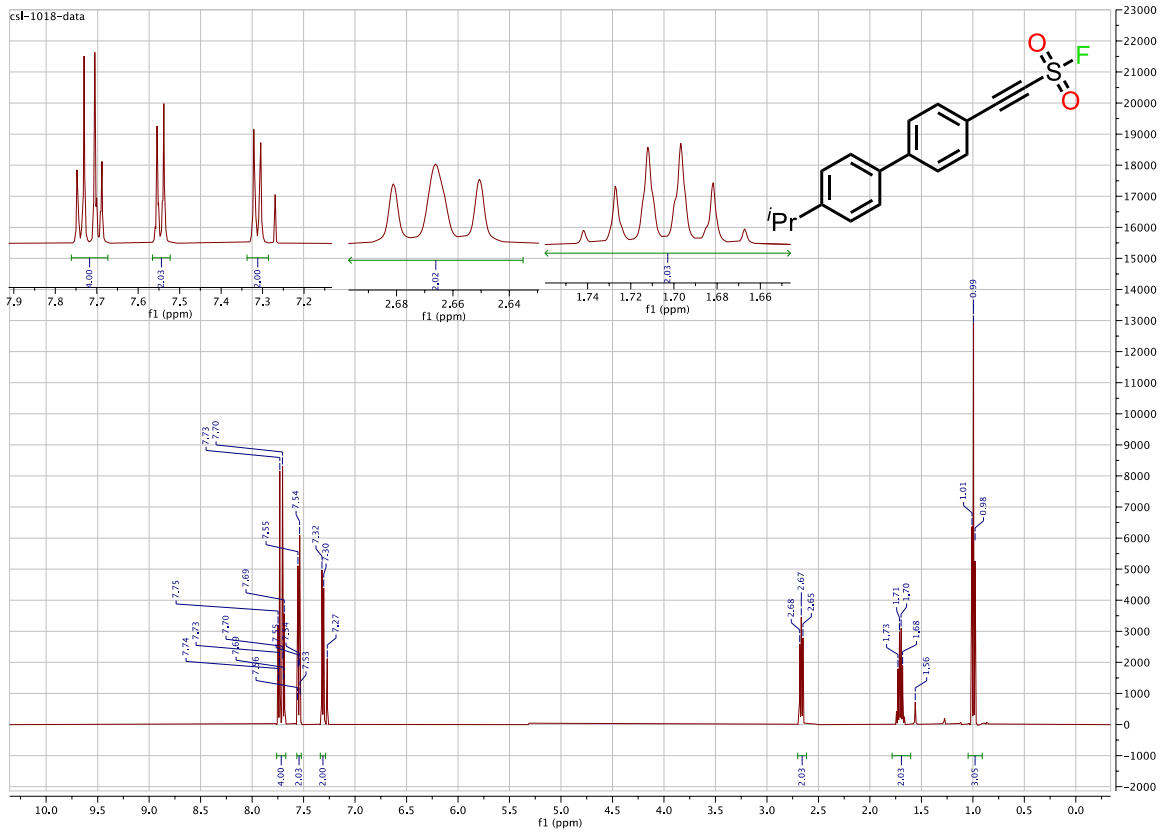




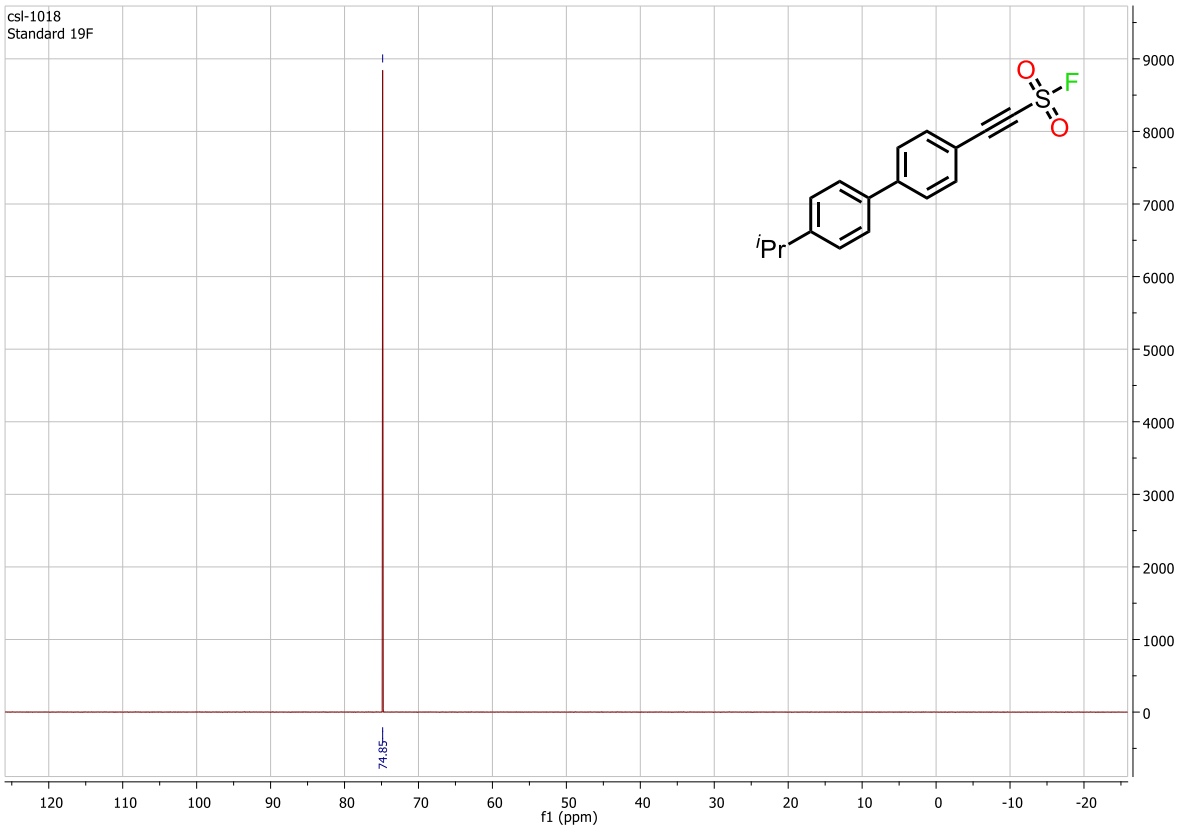


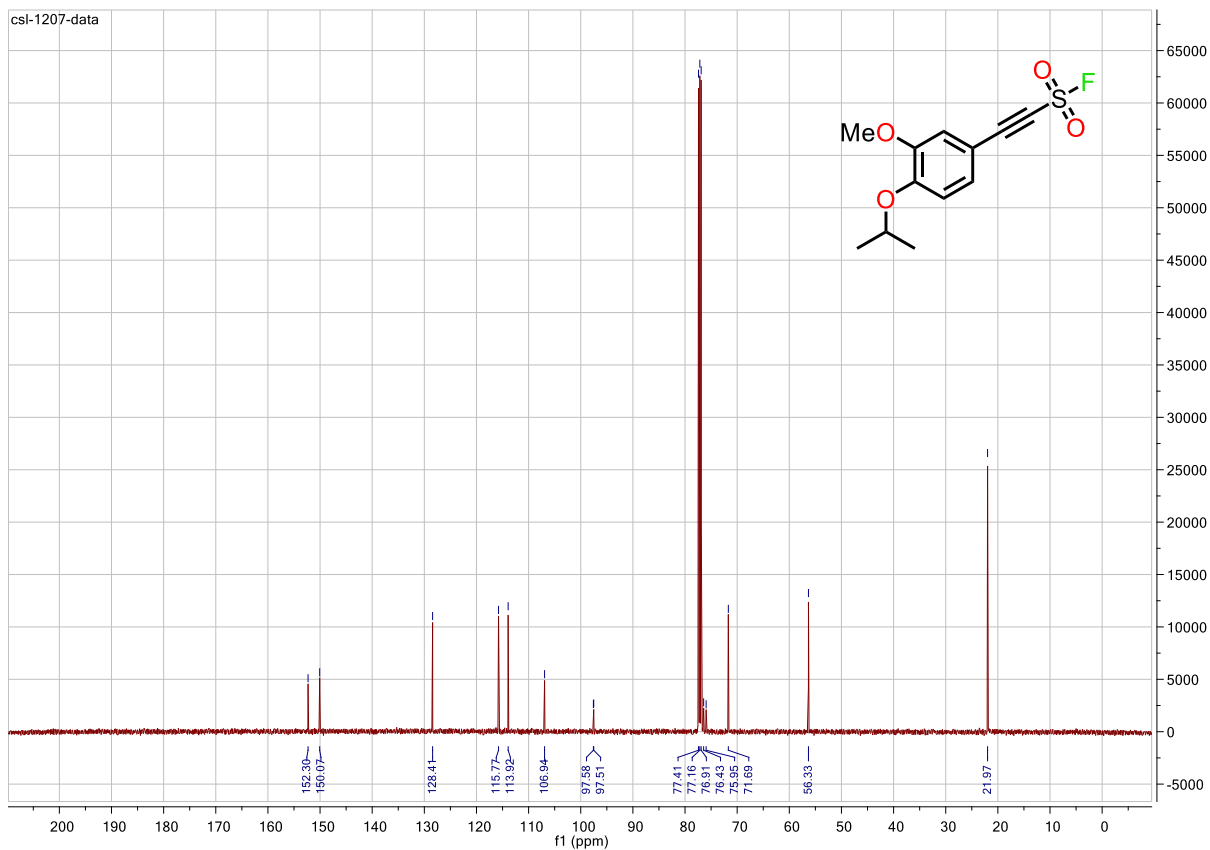
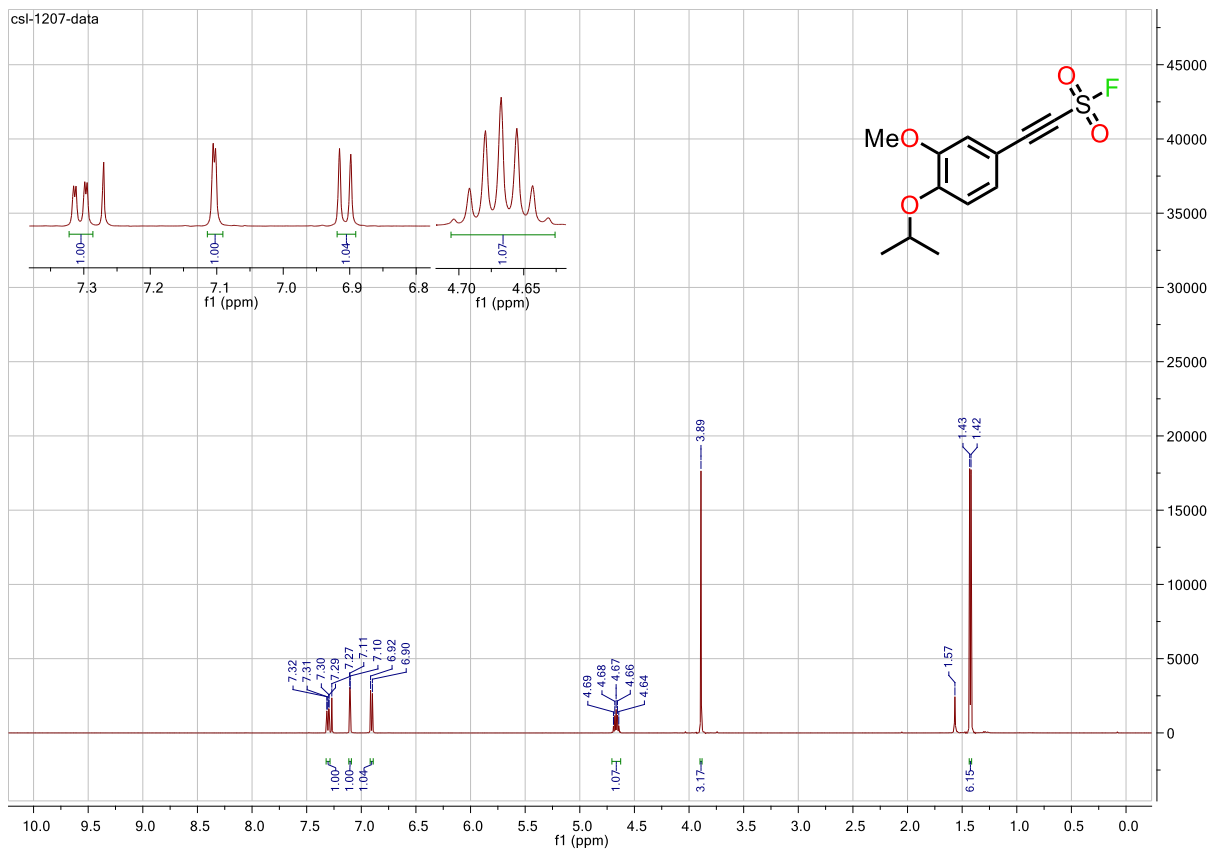
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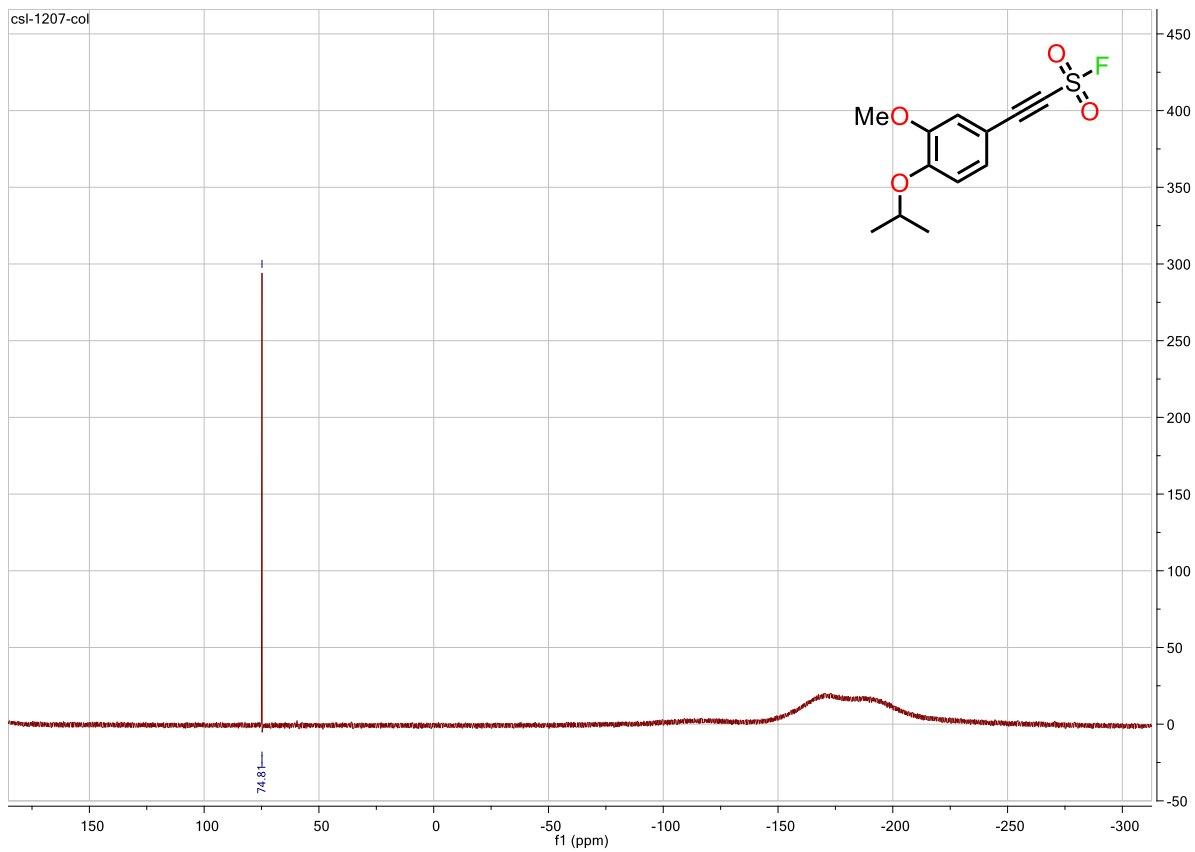




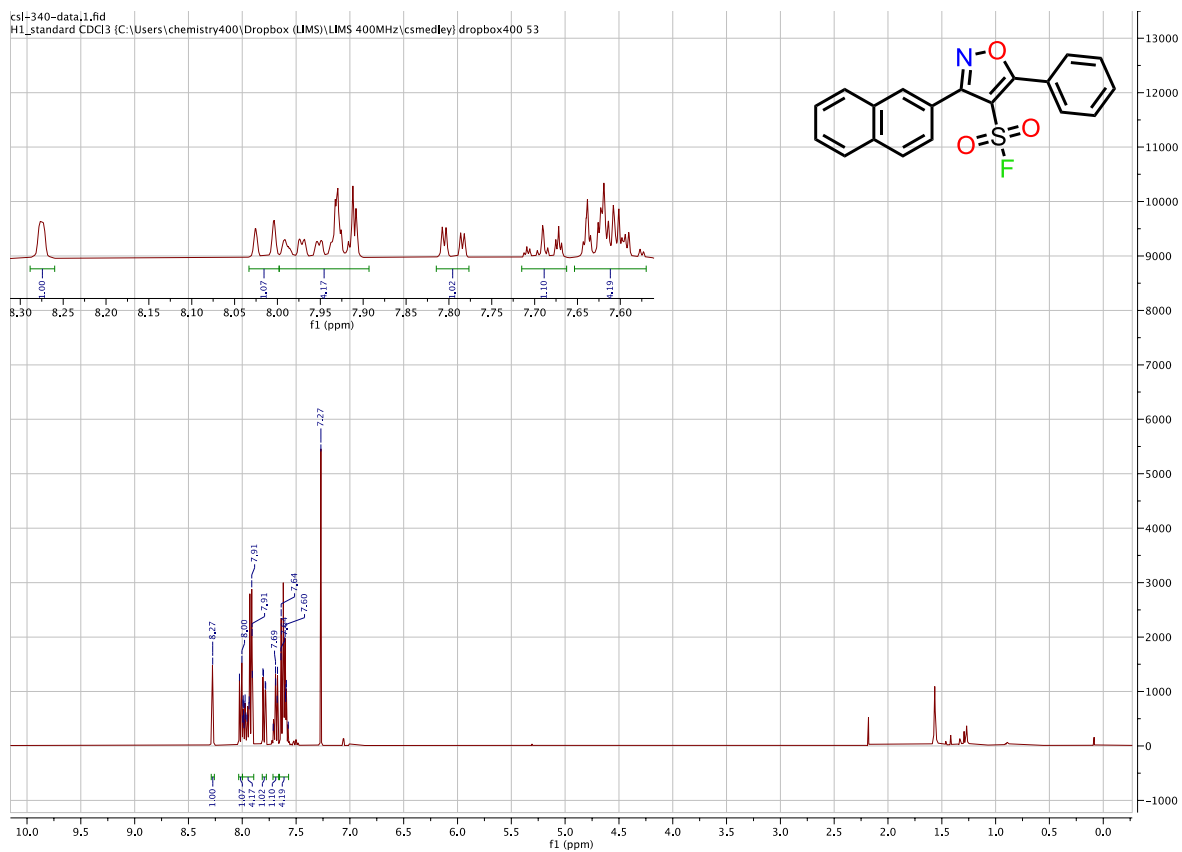
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Standard 19F



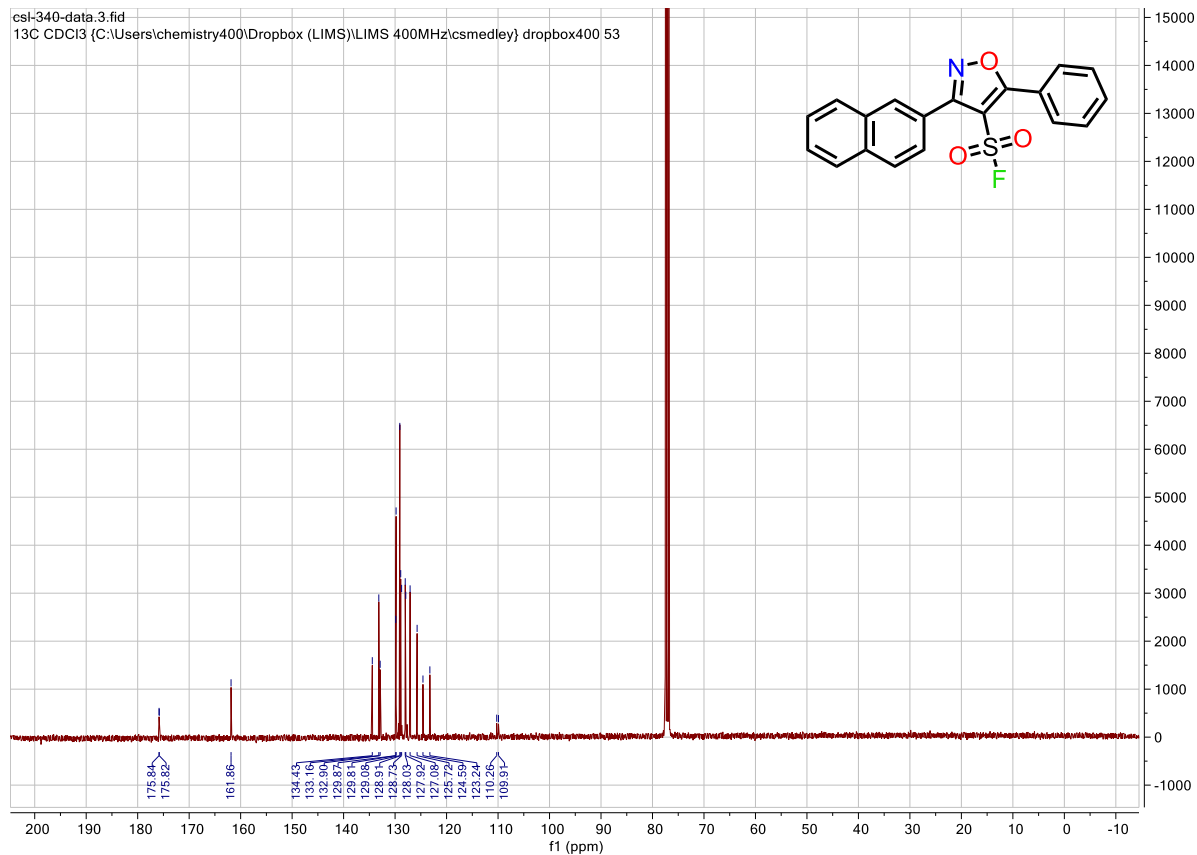




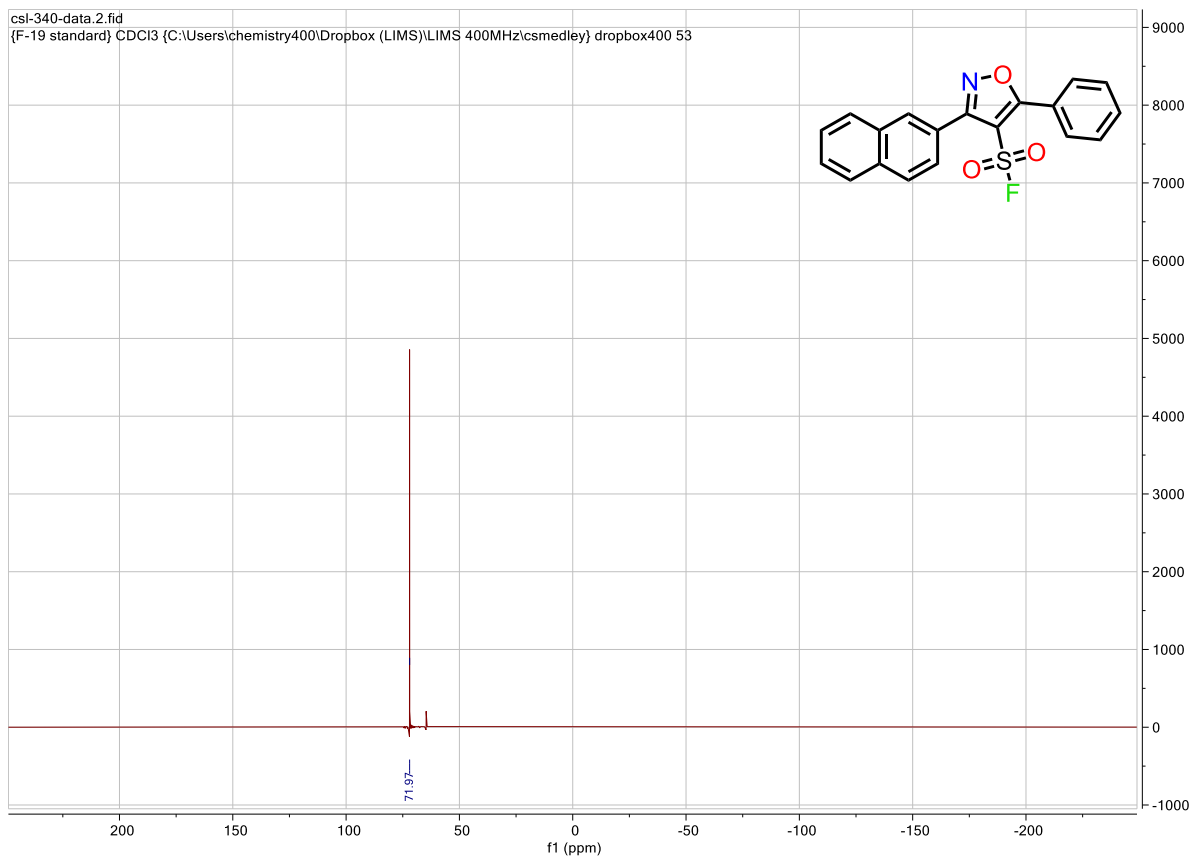
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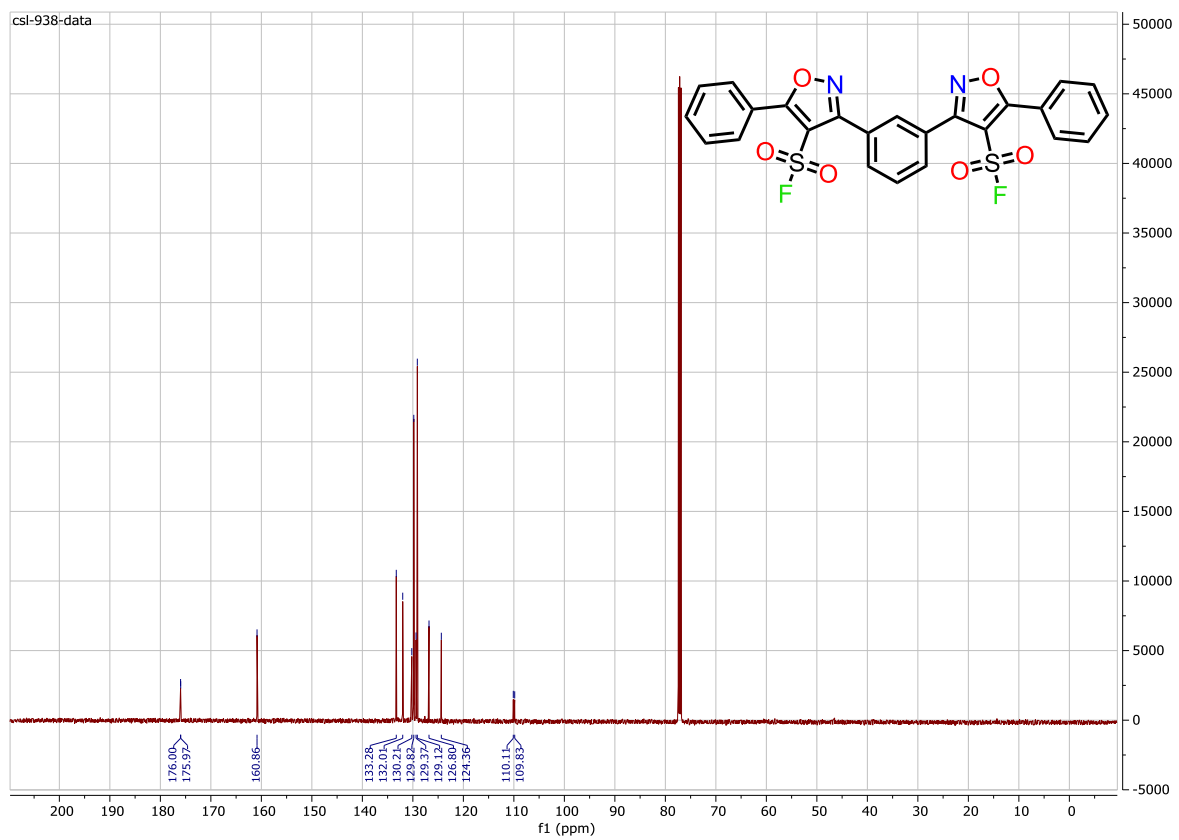
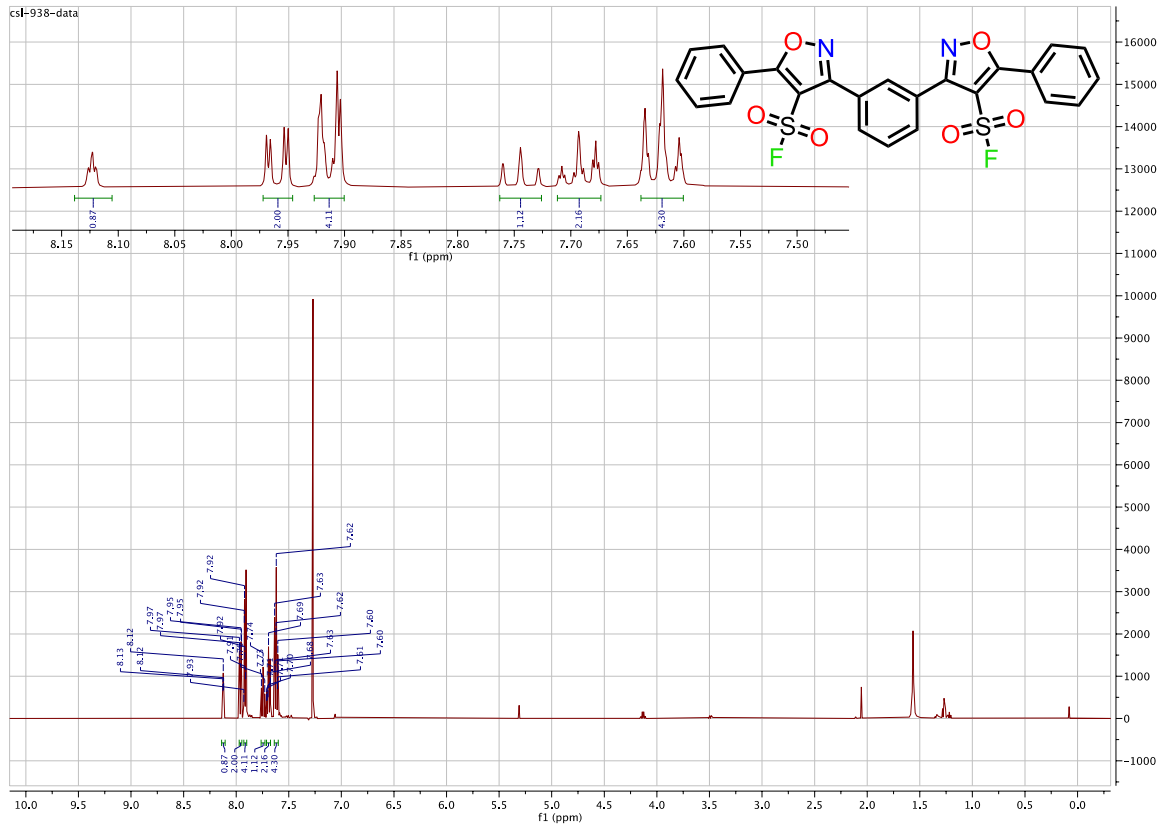


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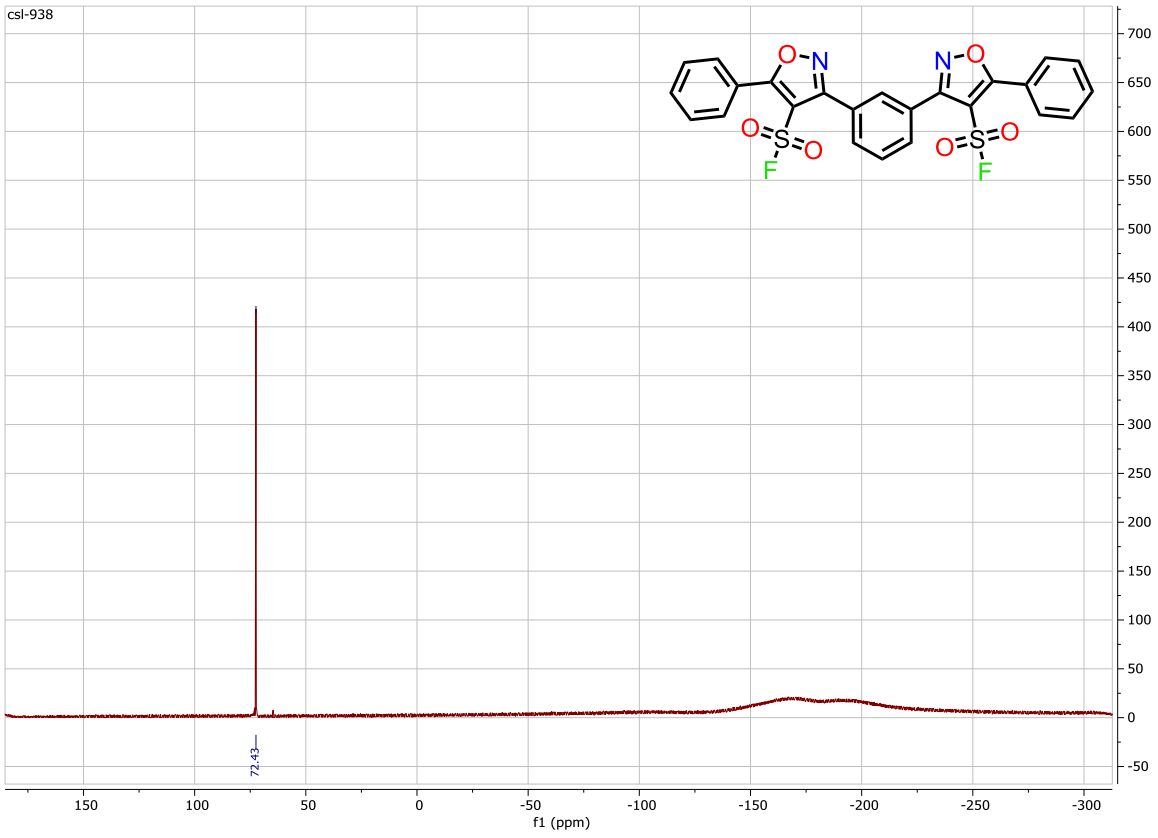


csi-340-data.2.fid
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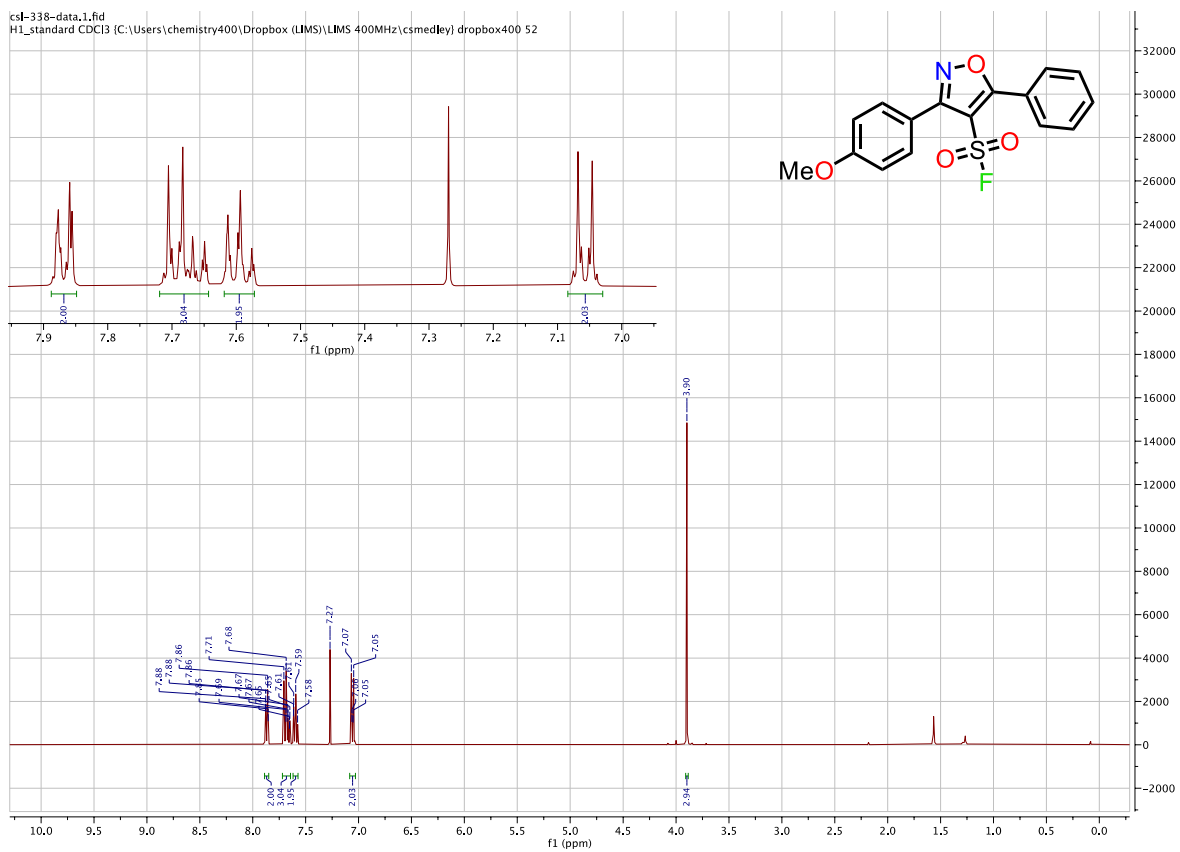




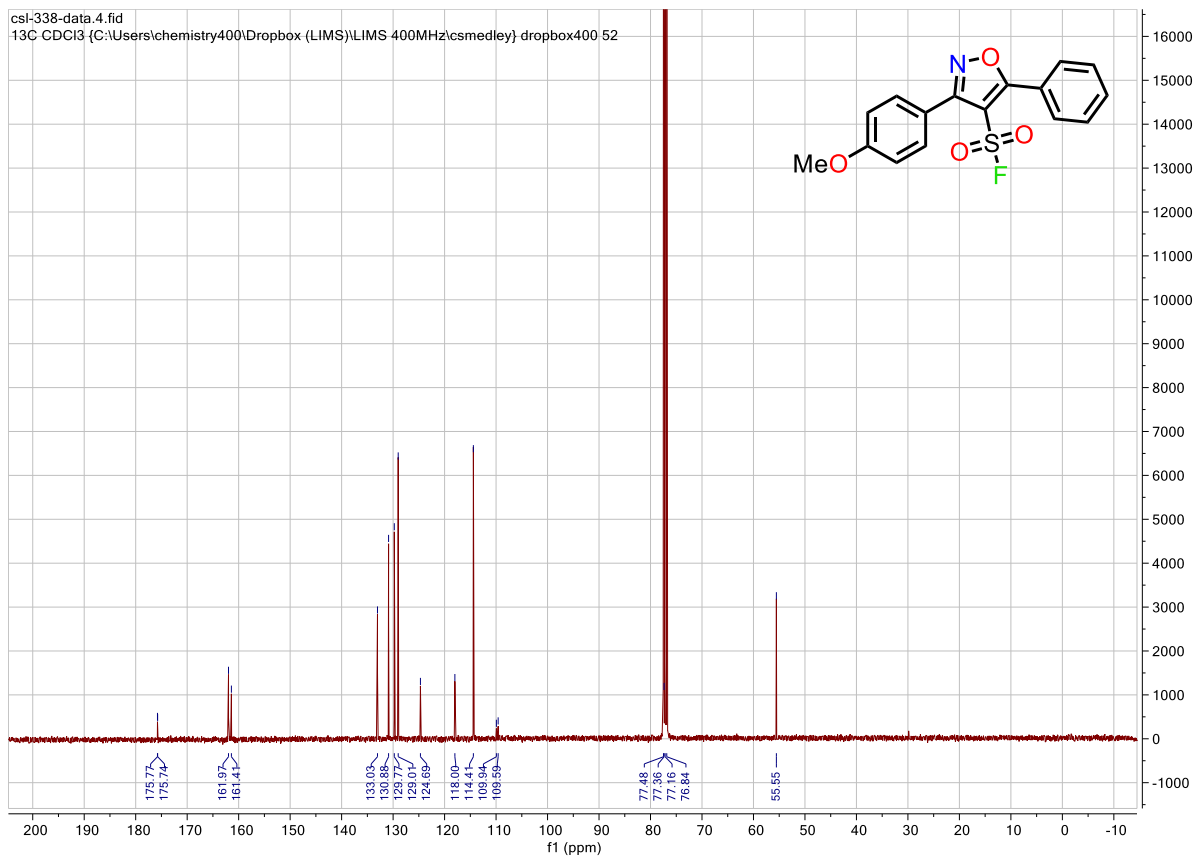
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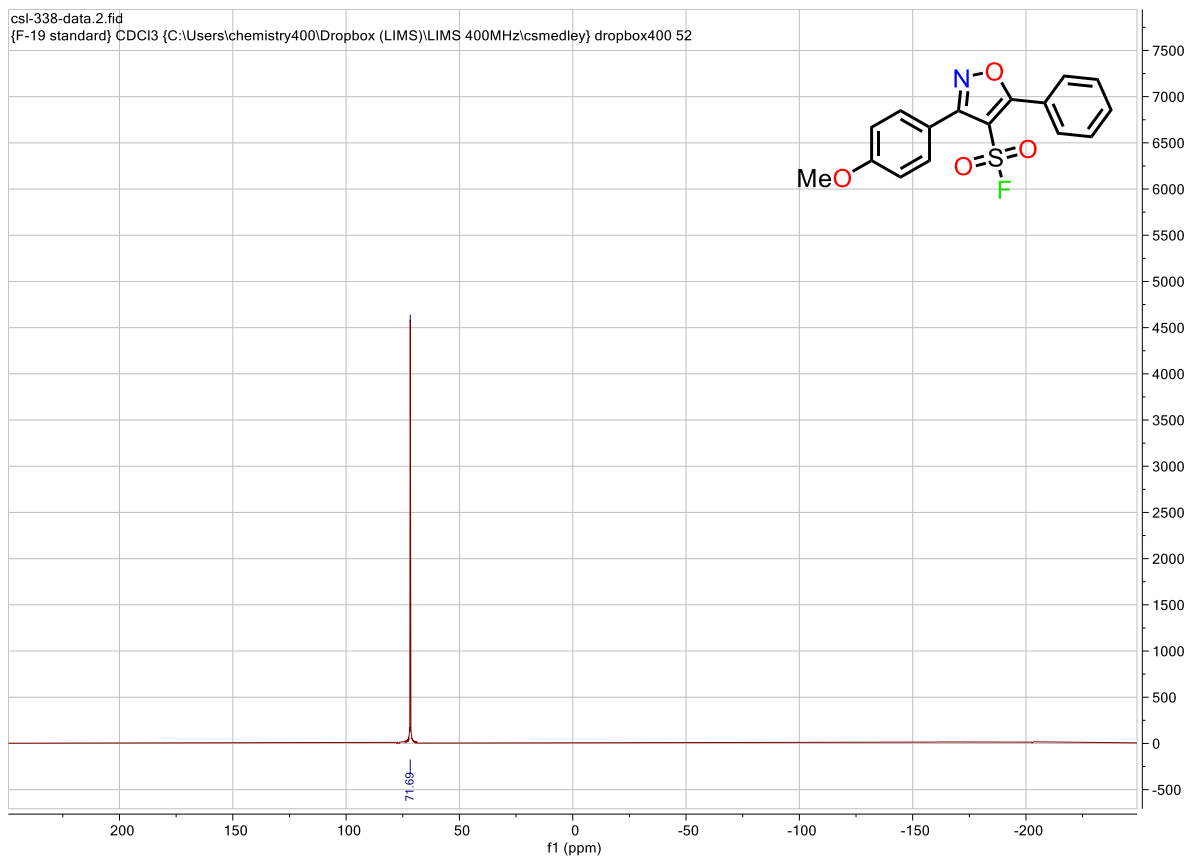
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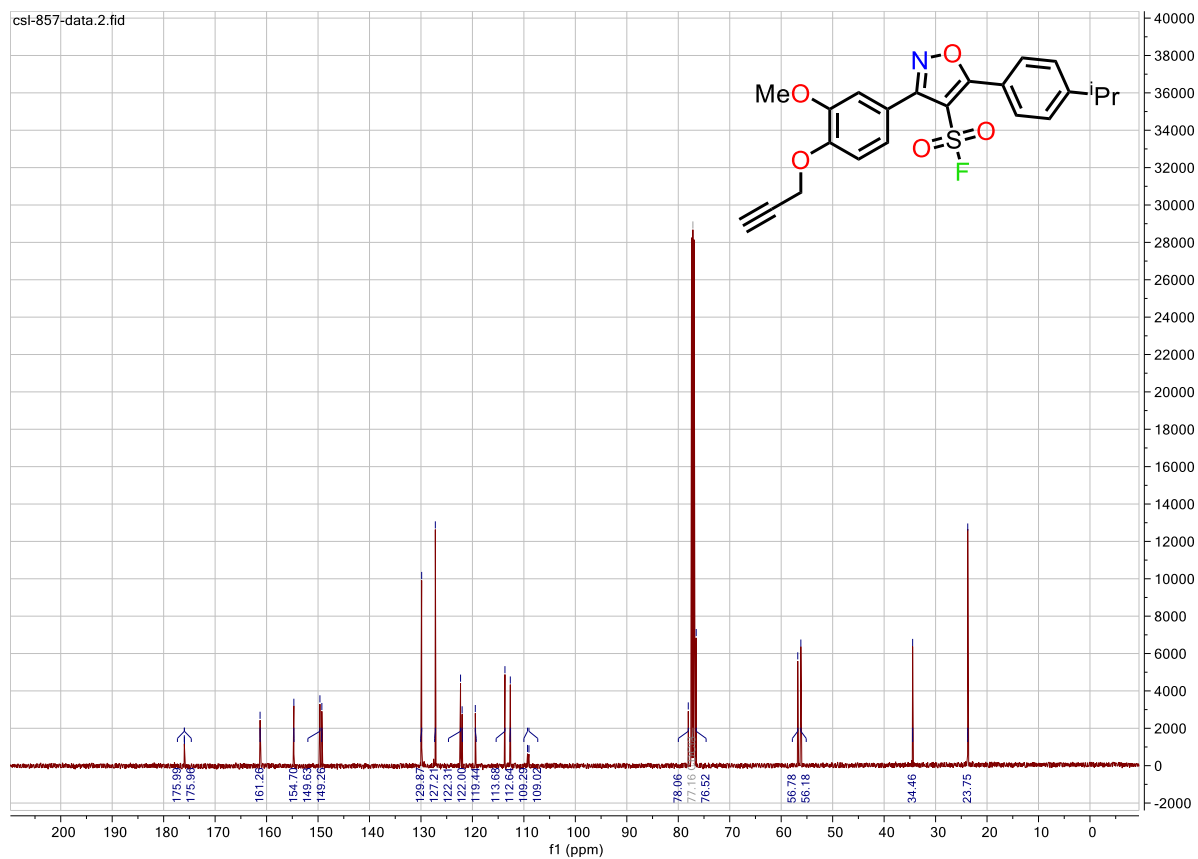
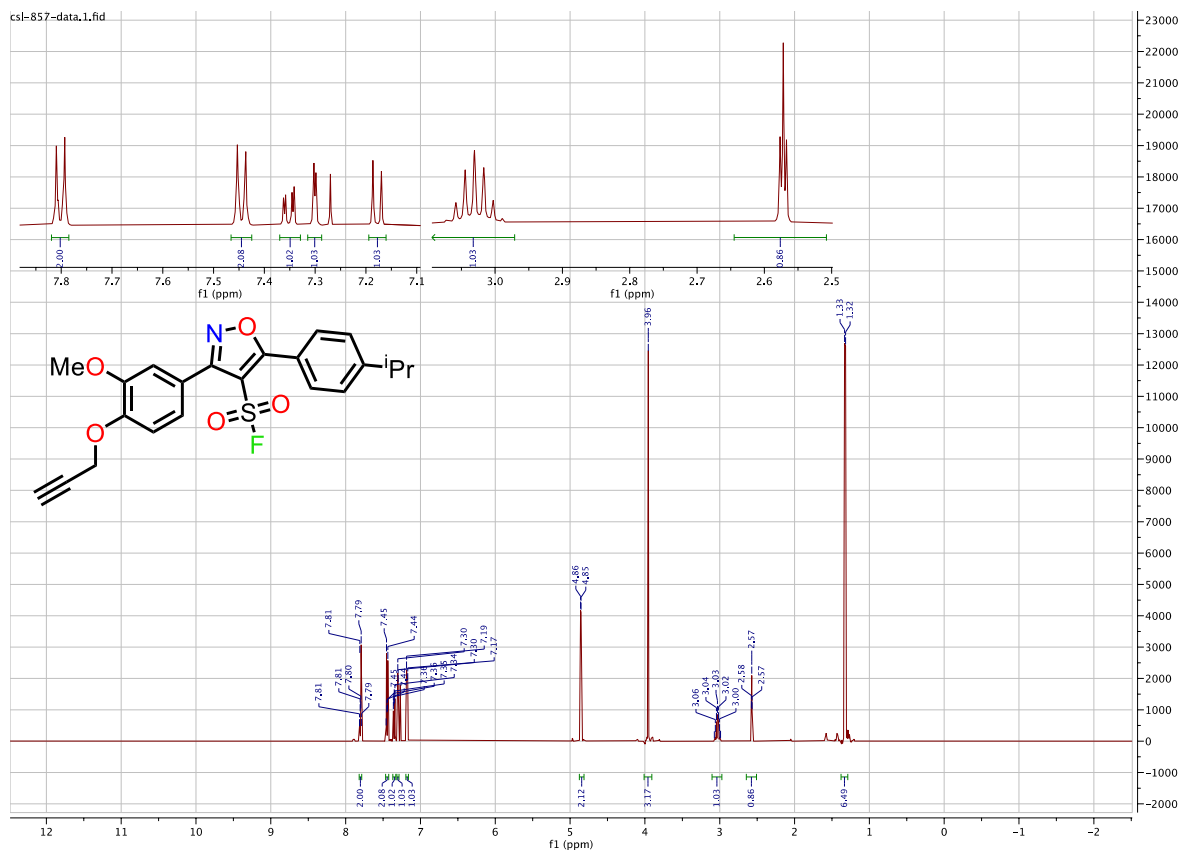


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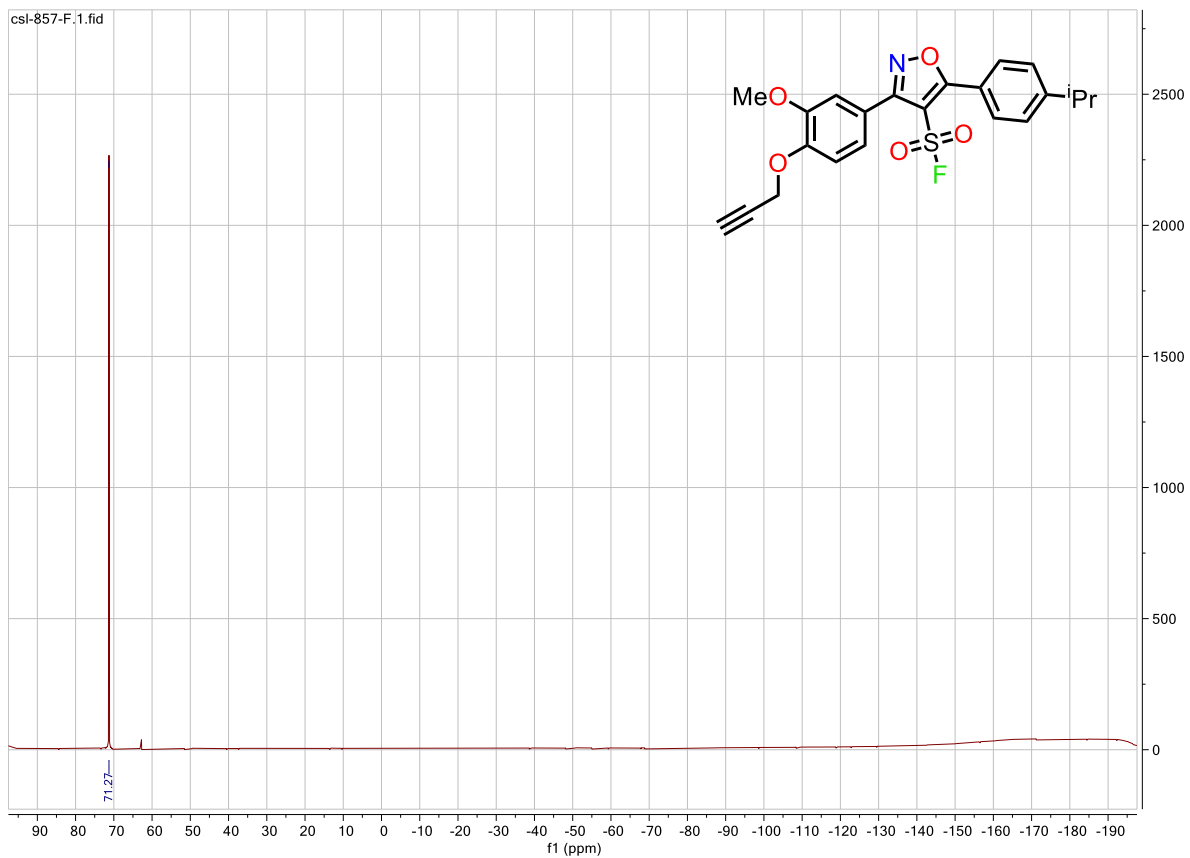


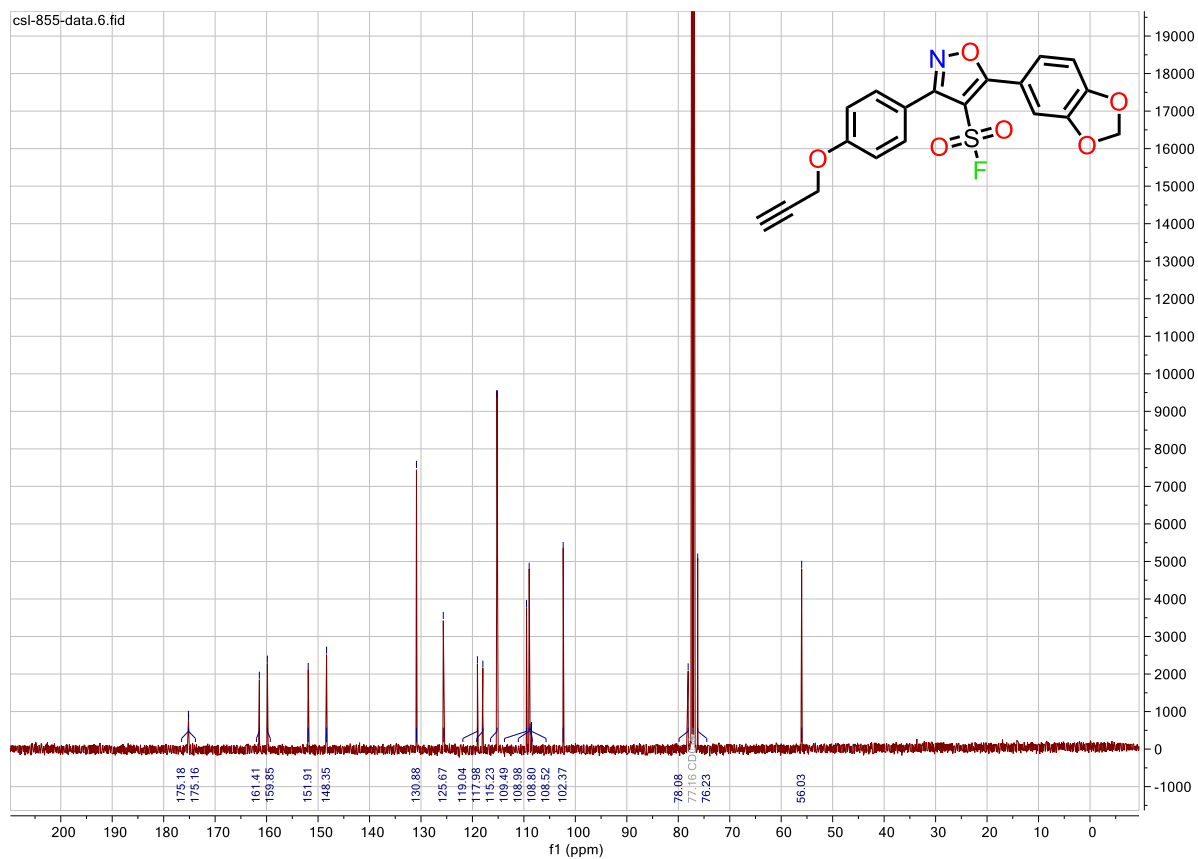
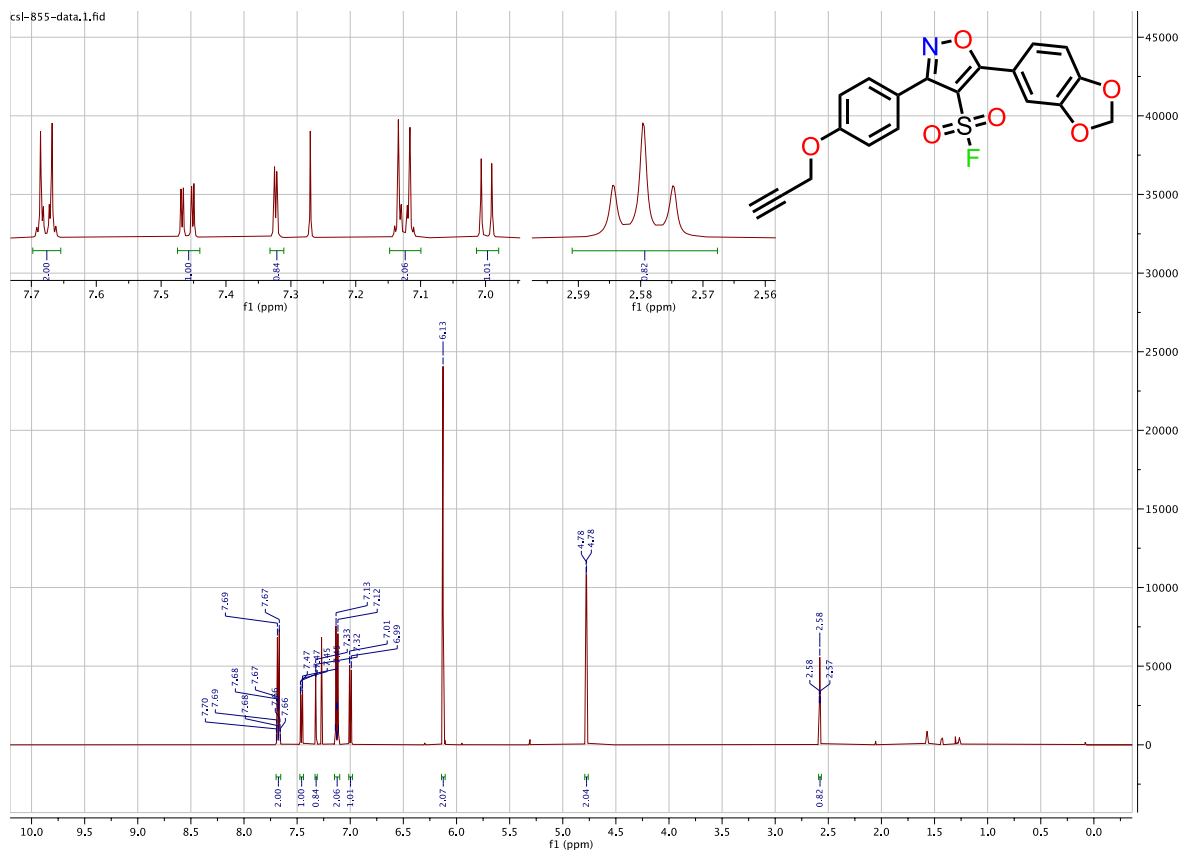
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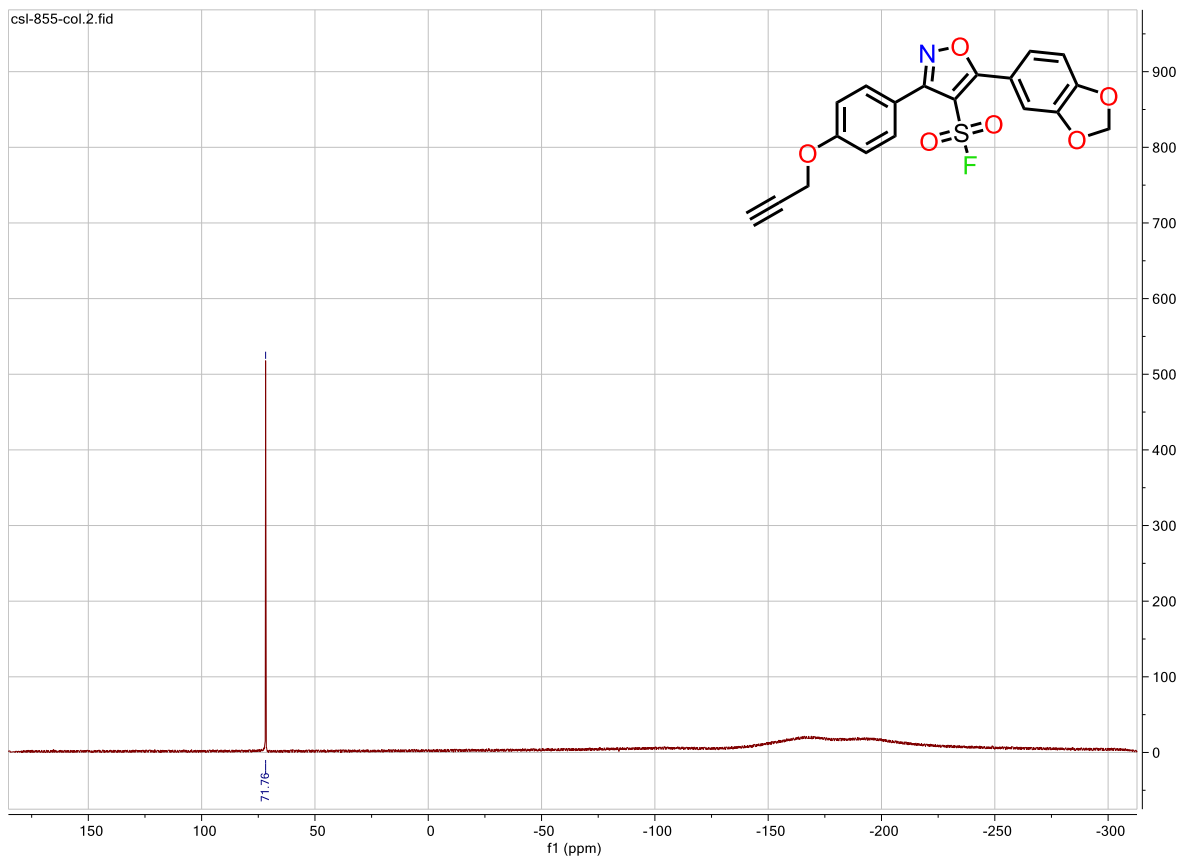


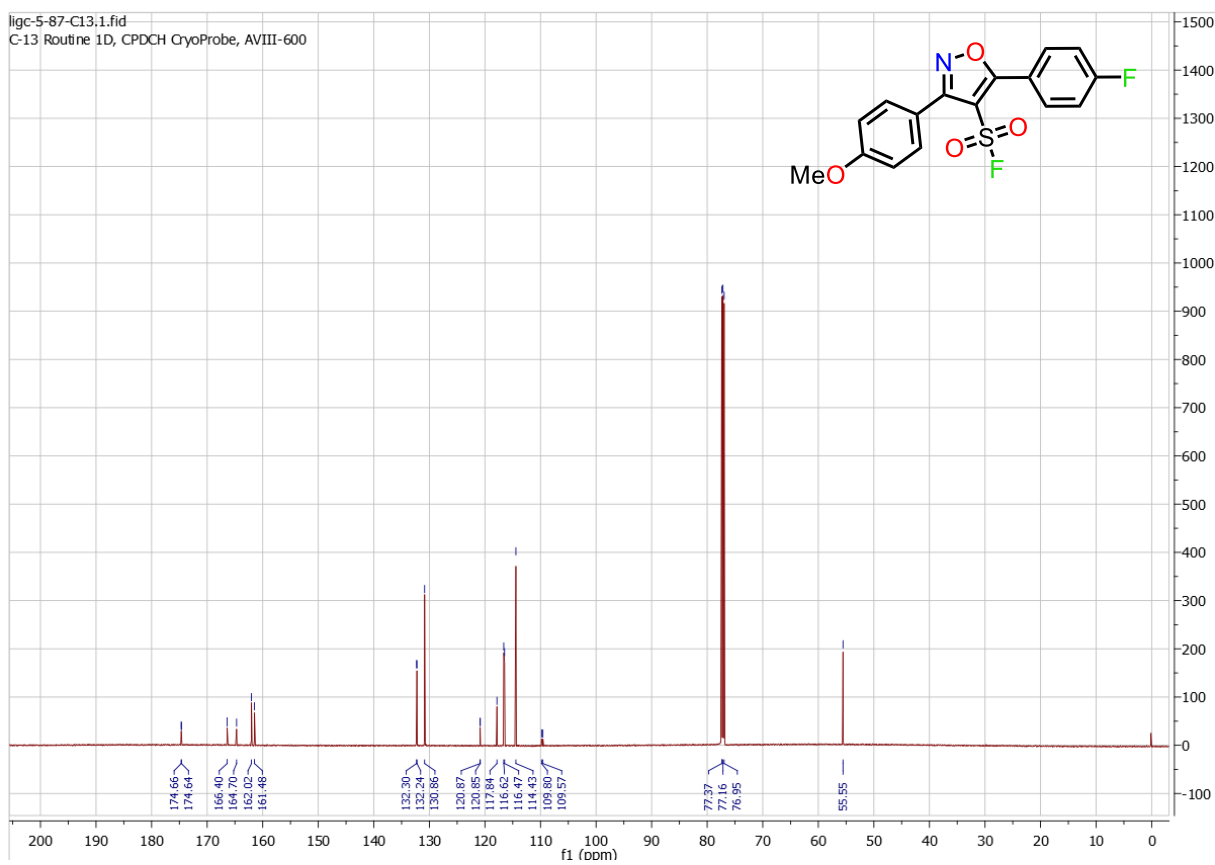
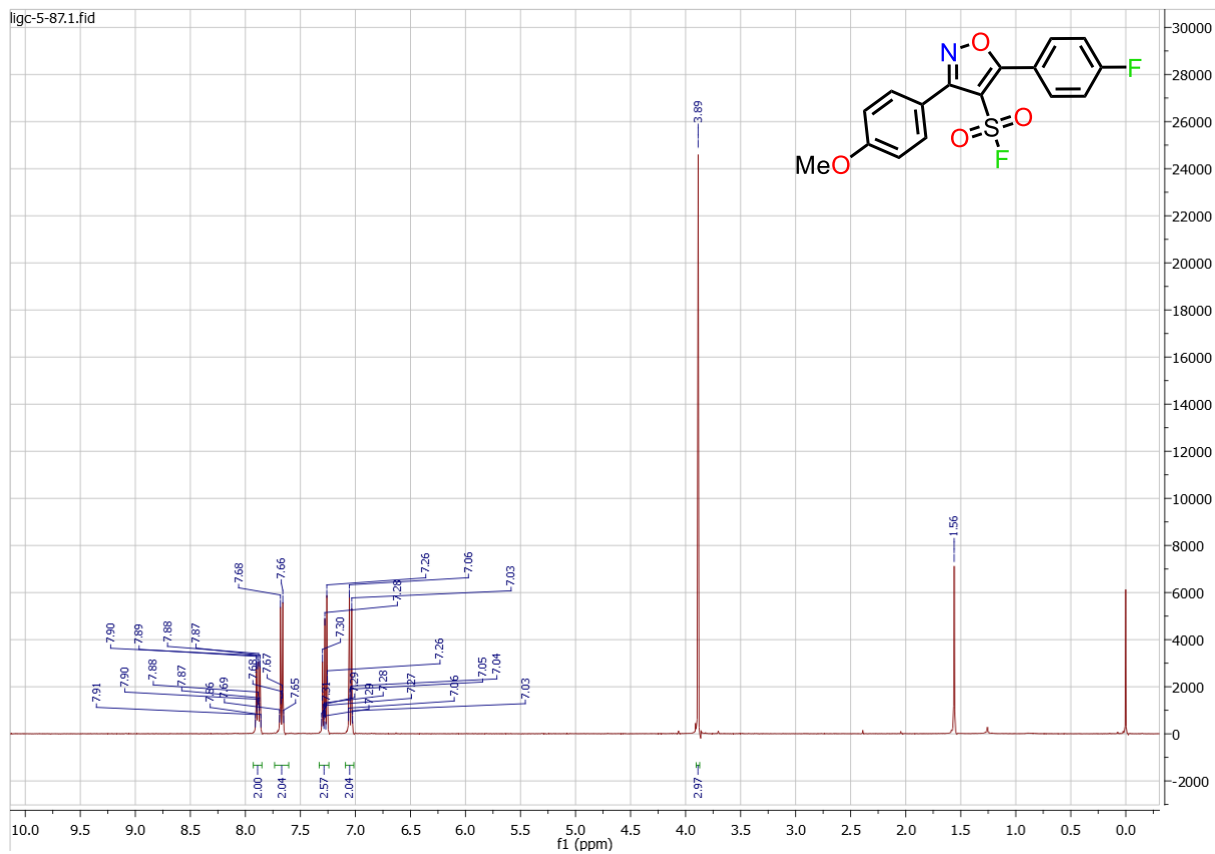


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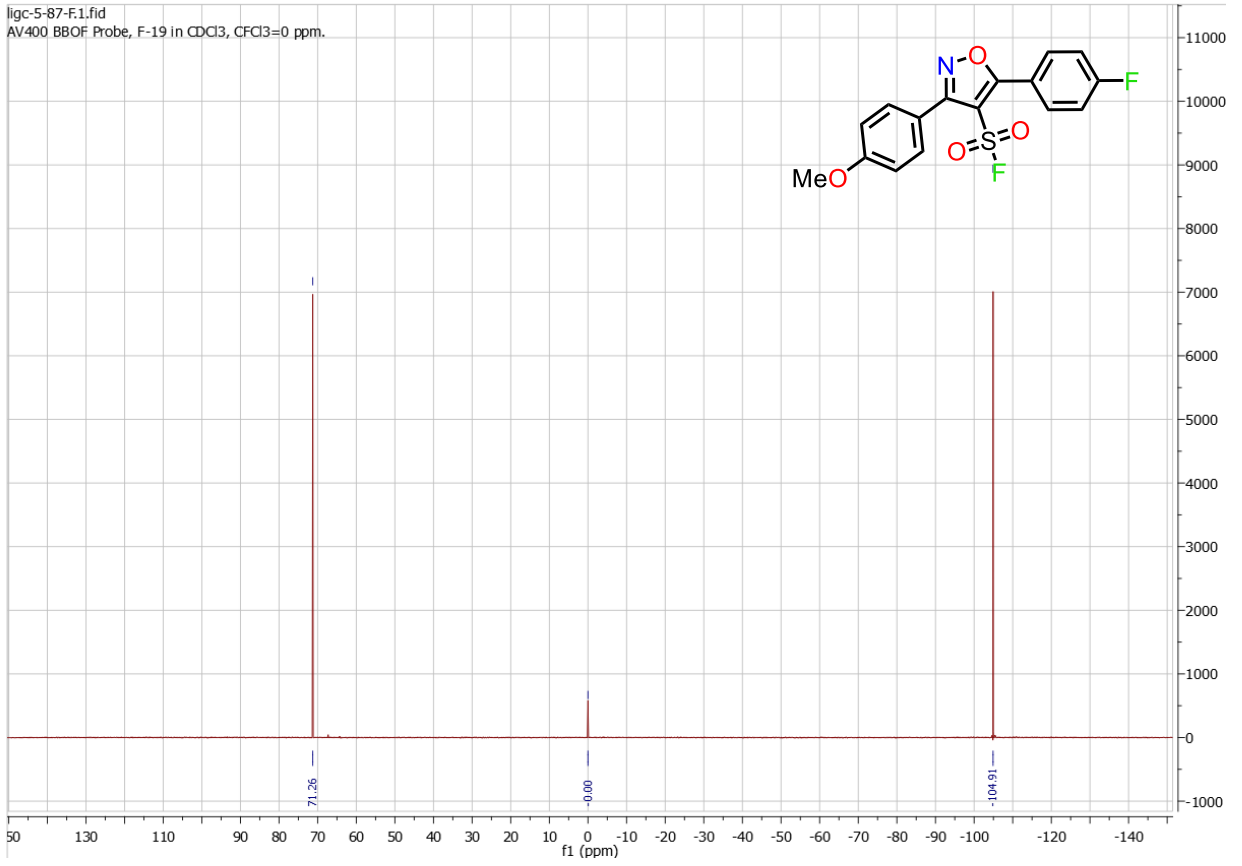


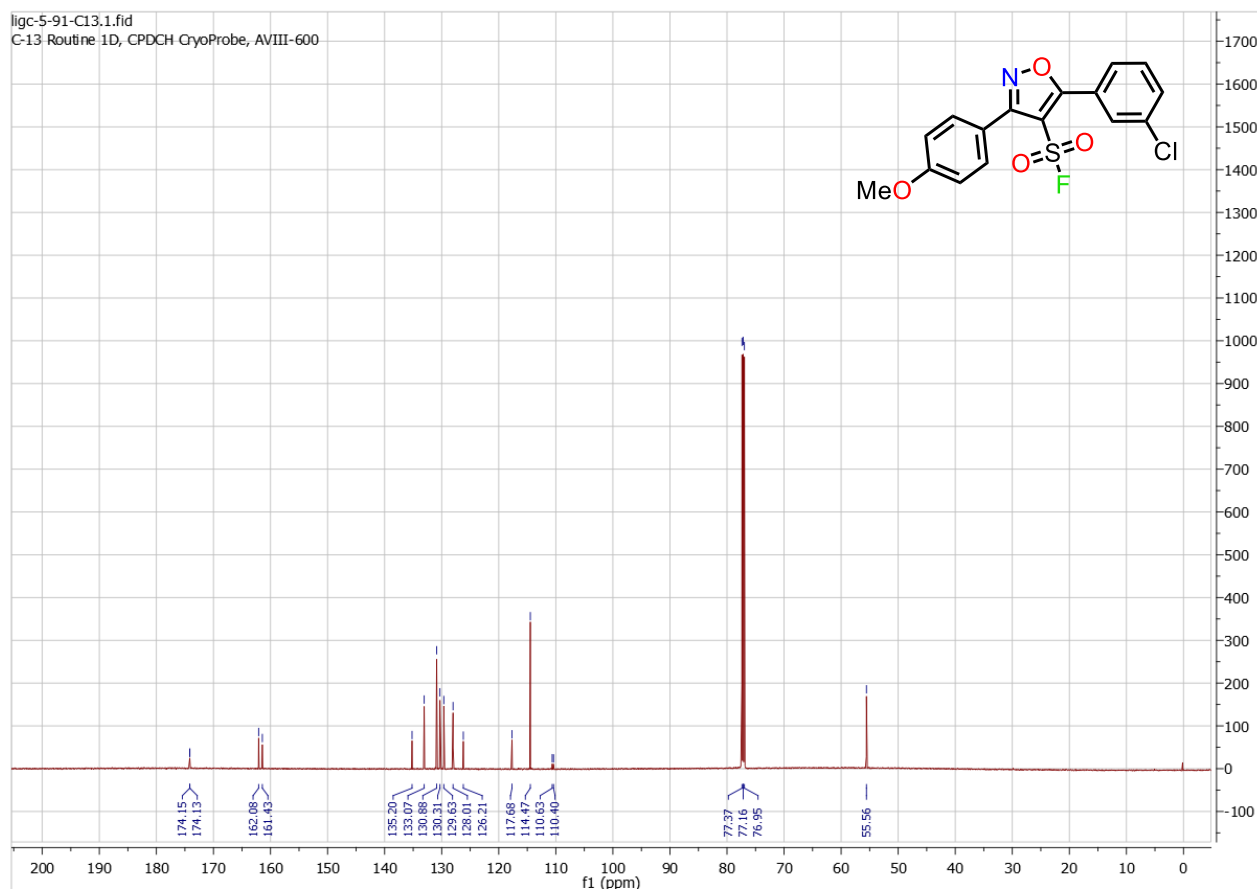
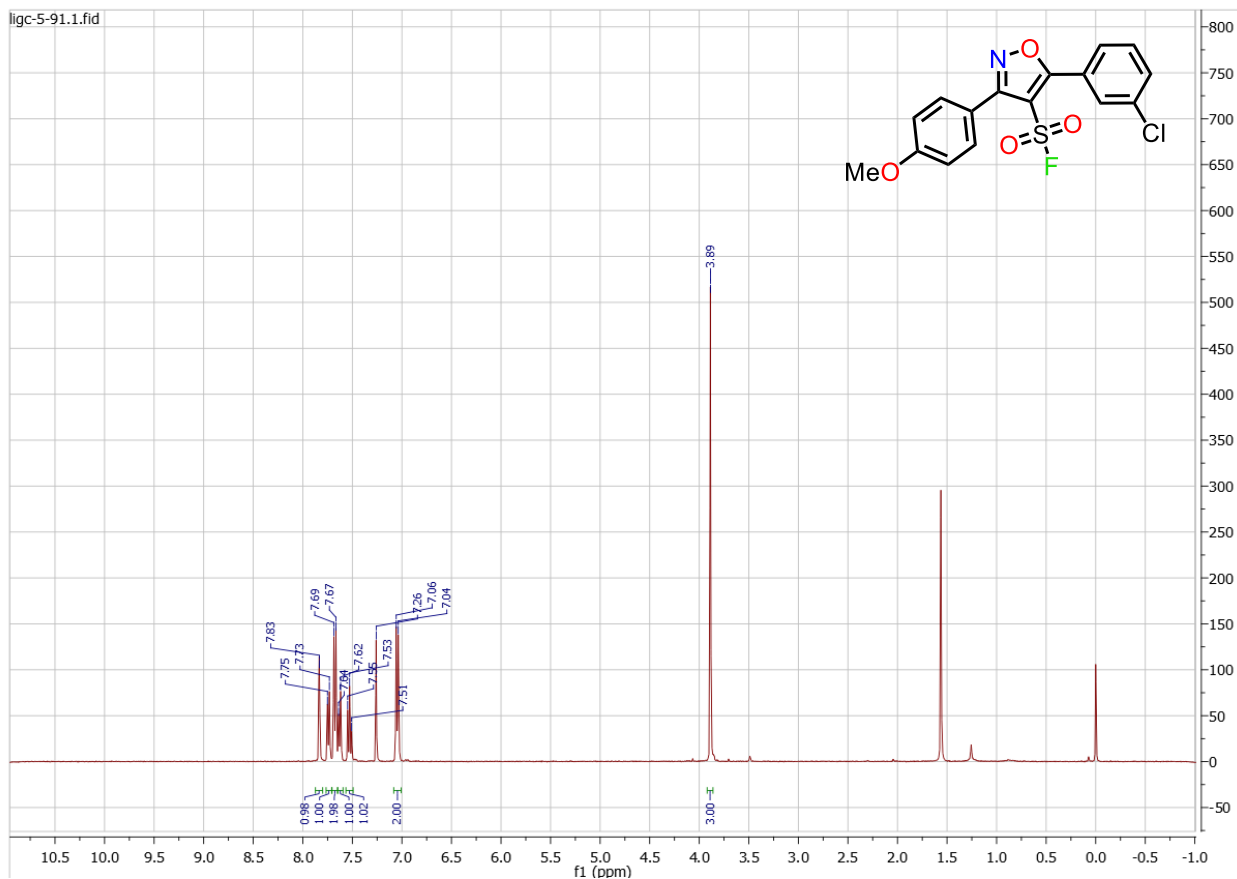




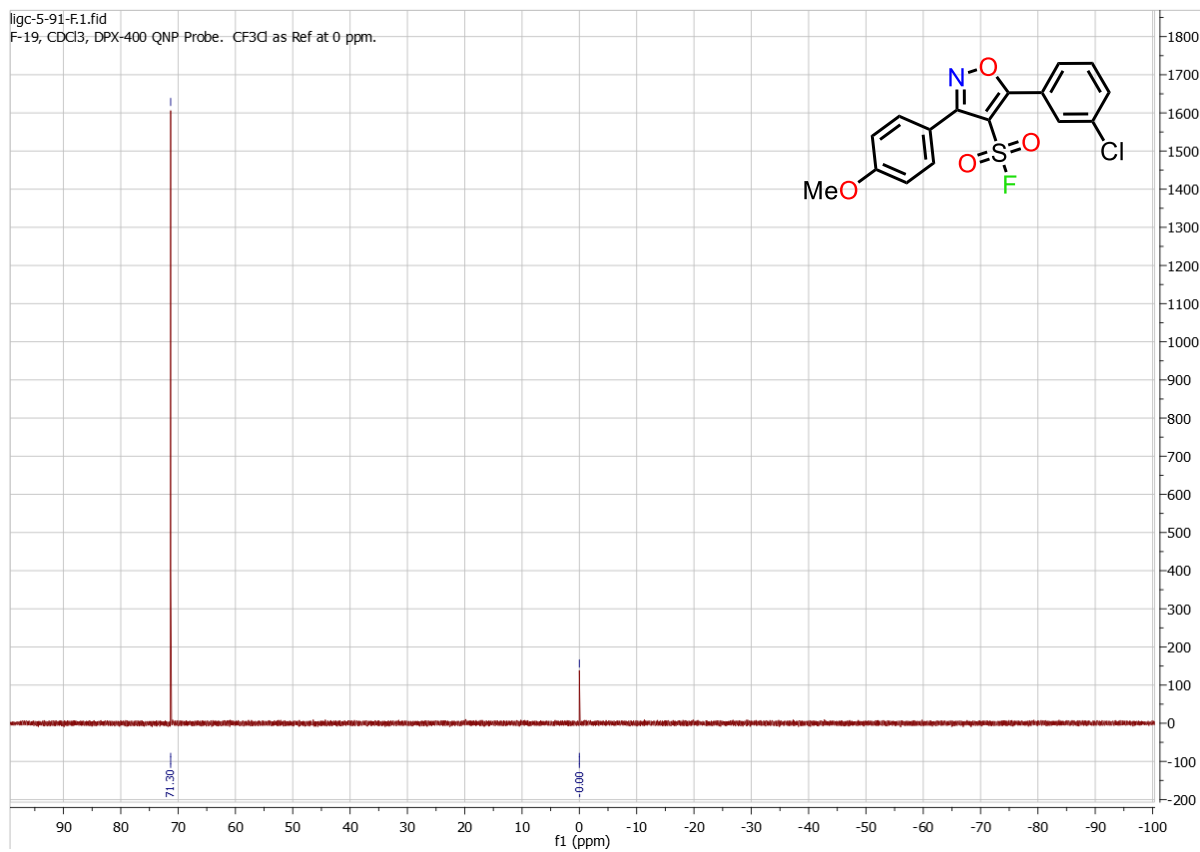


ligc-5-87-F.1.fid
AV400 BBOF Probe, F-19 in CDCl3, CFCl3=0 ppm.

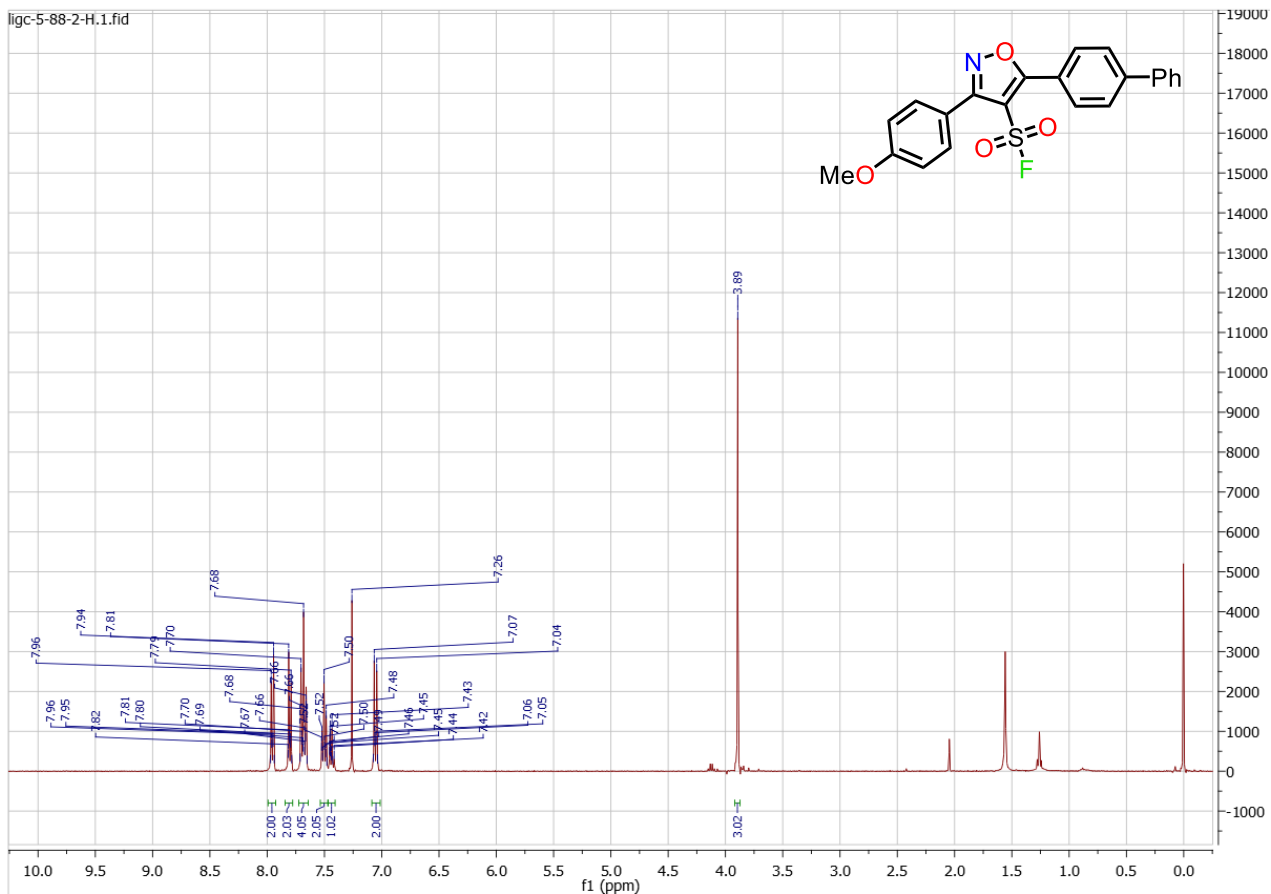




ligc-5-91-F1.fid
F-19, CDCl3, DPX-400 QNP Probe. CF3Cl as Ref at 0 ppm.

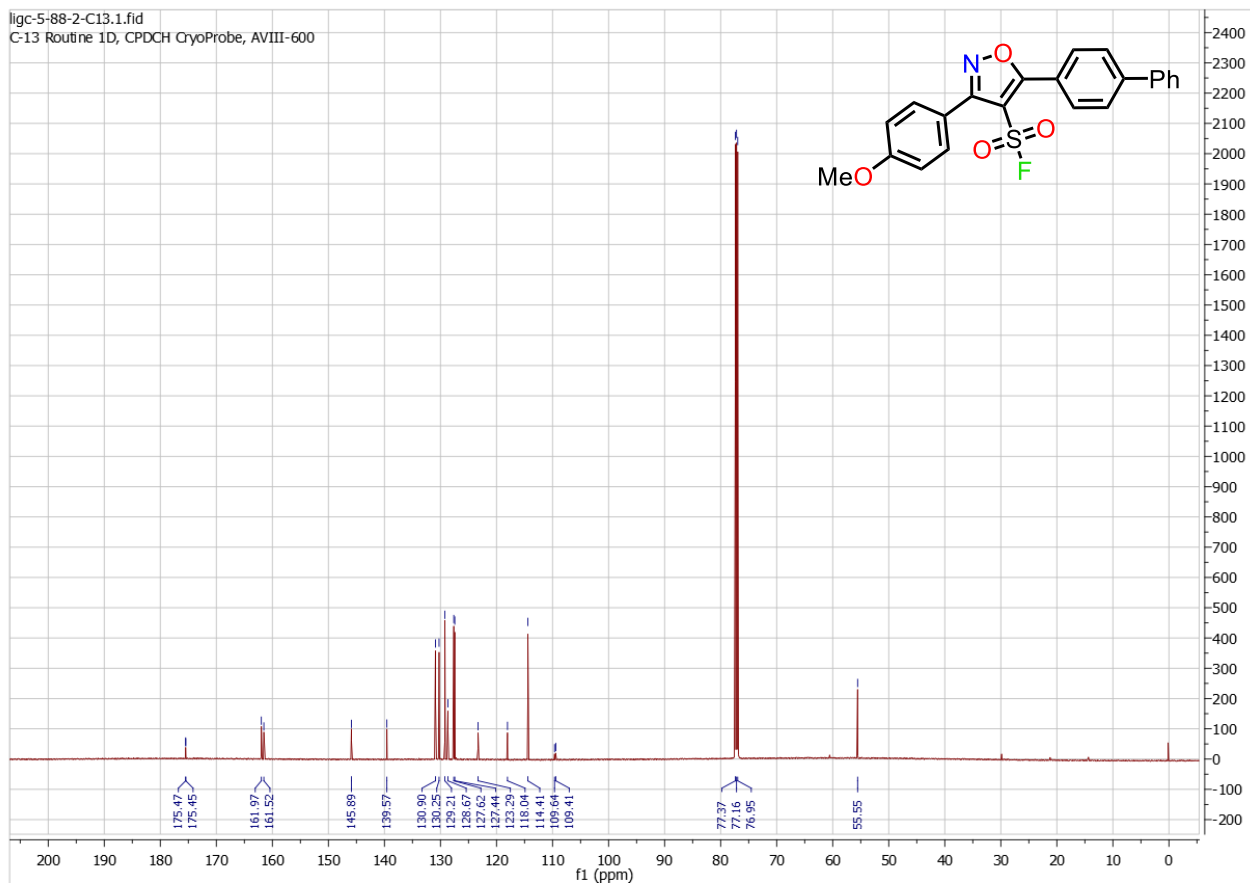


ligc-5-88-2-H.1.fid

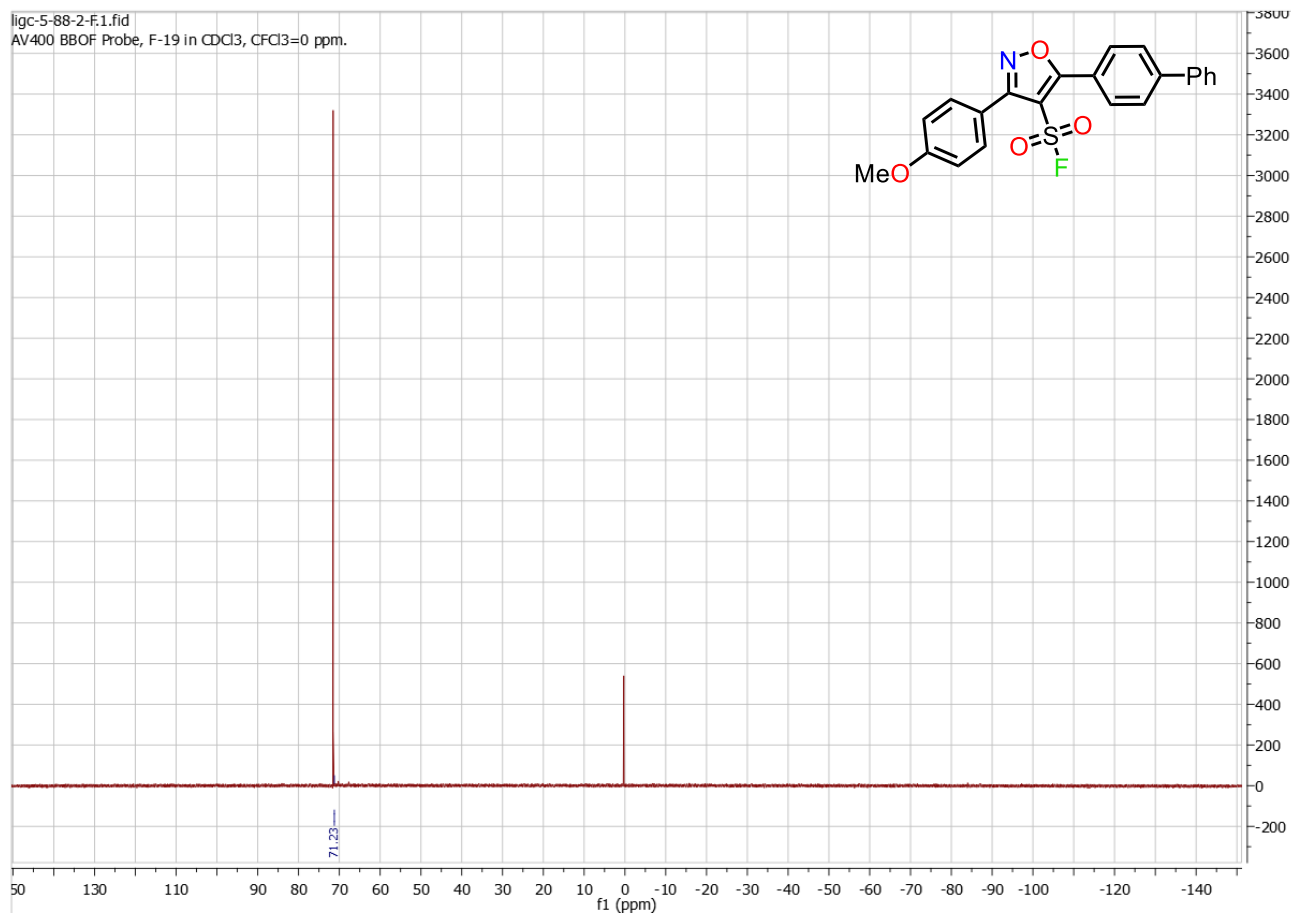


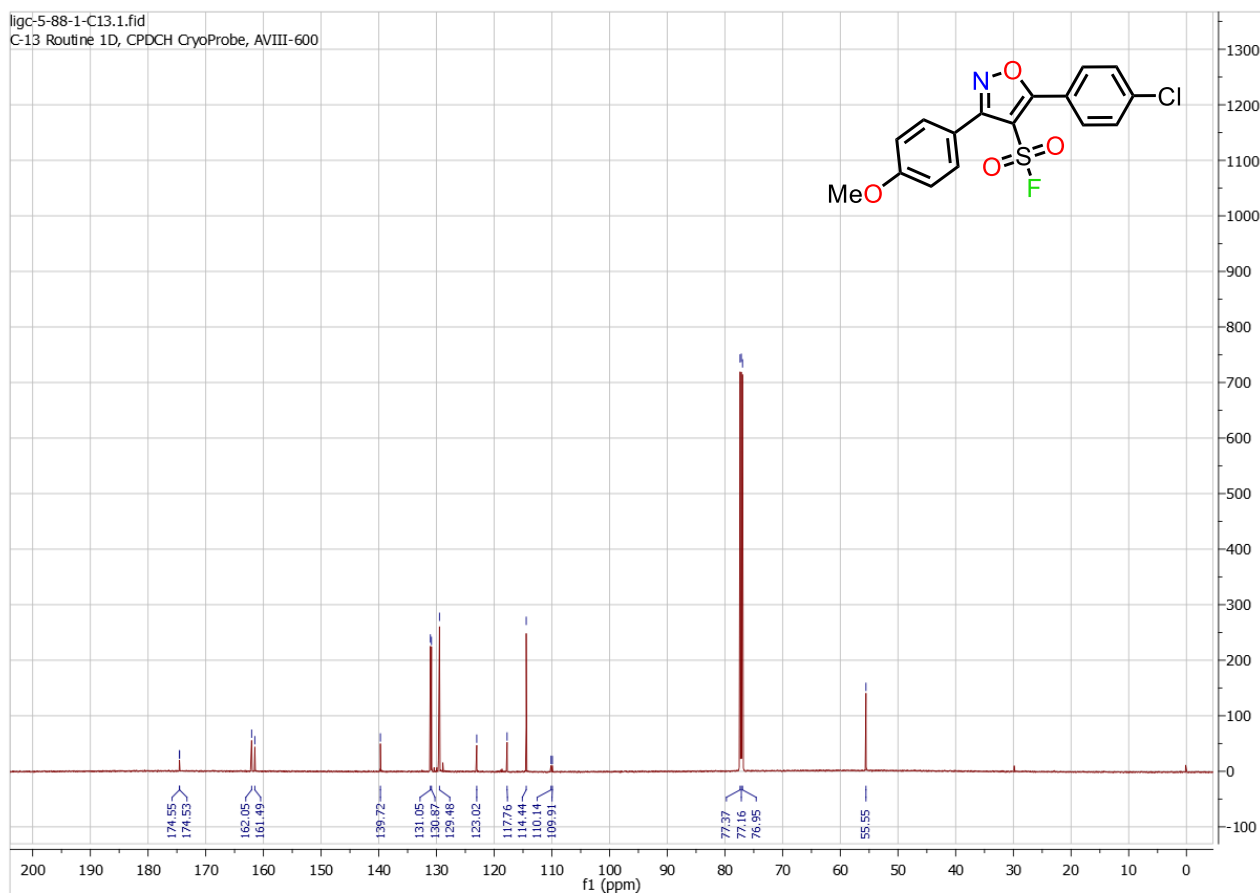
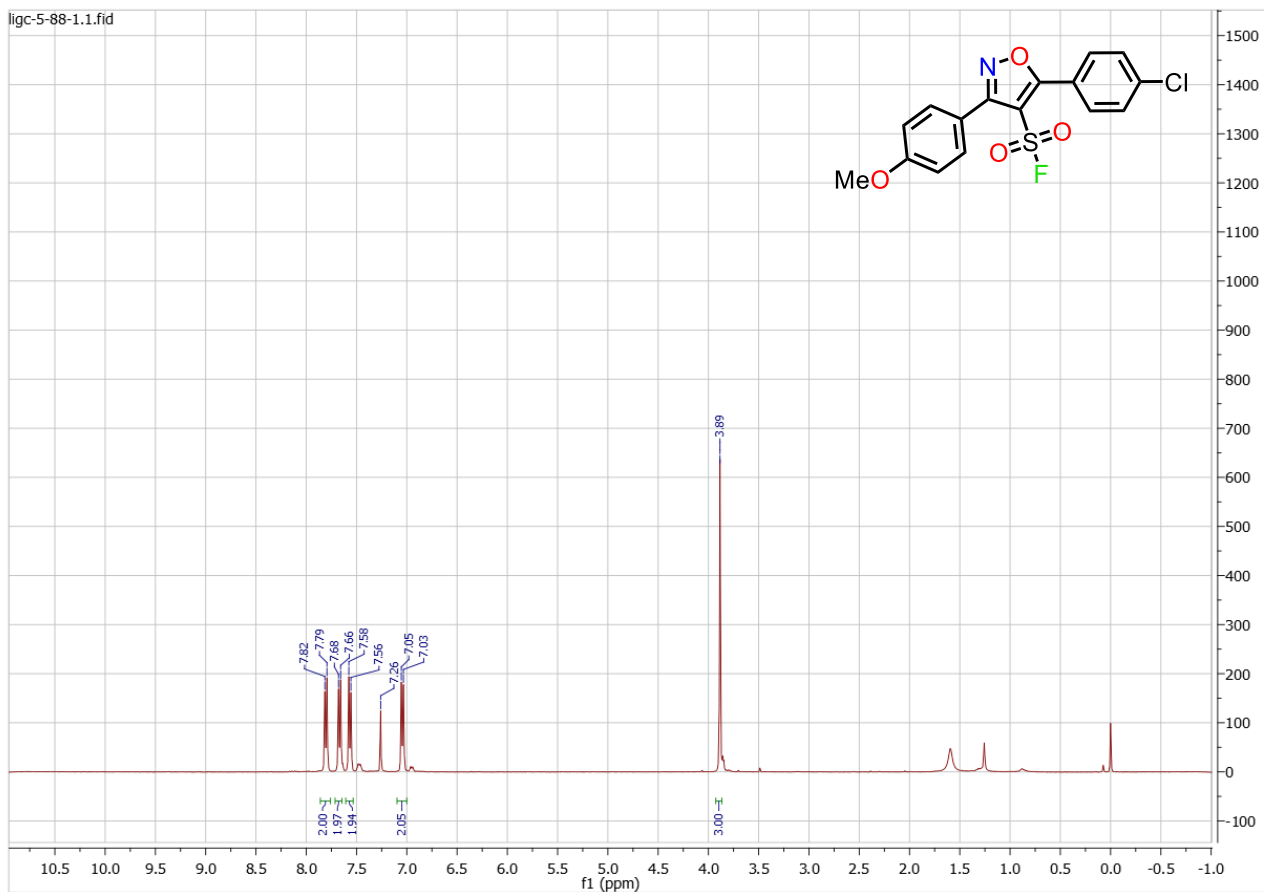
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C-13 Routine 1D, CPDCH CryoProbe, AVIII-600

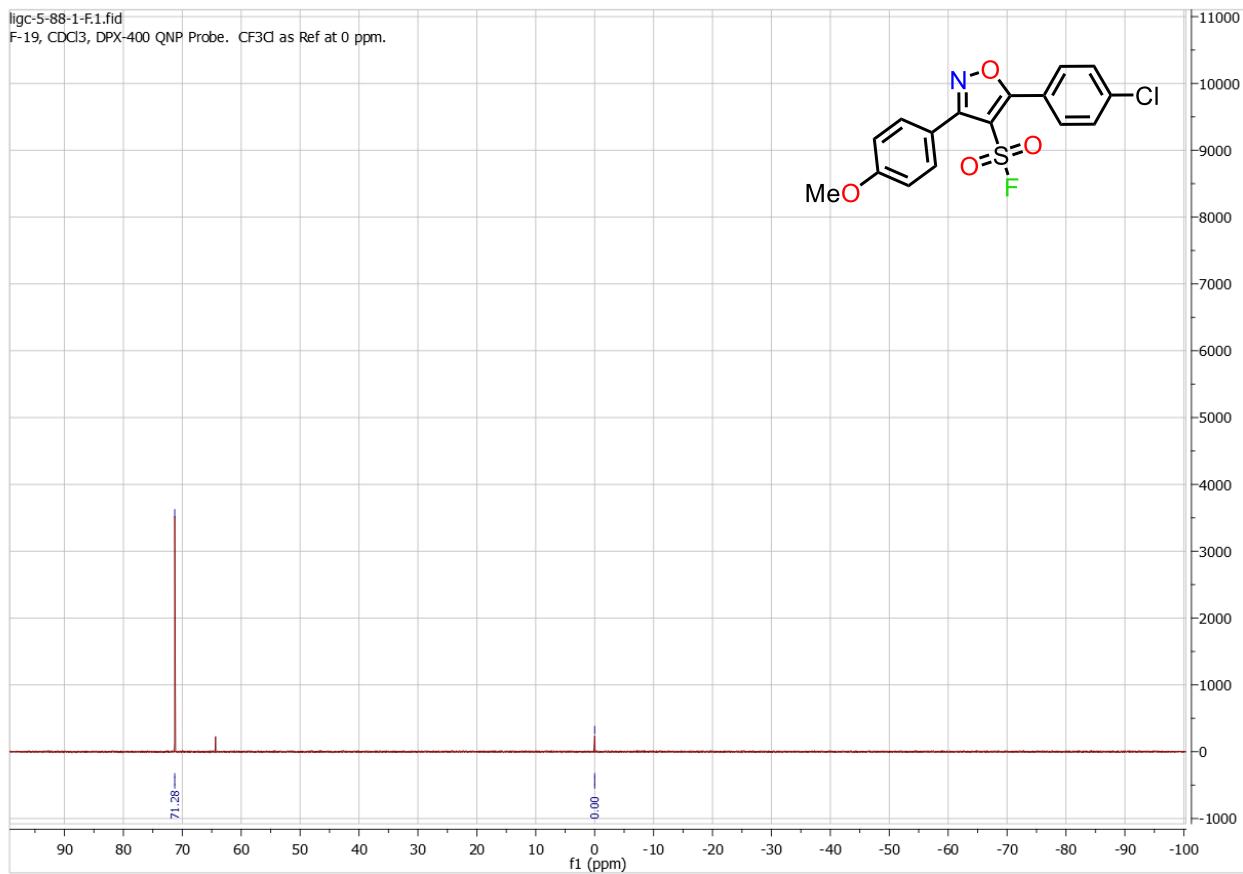


ligc-5-88-2-F.1.fid
AV400 BBOF Probe, F-19 in CDCl₃, CFCl₃=0 ppm.

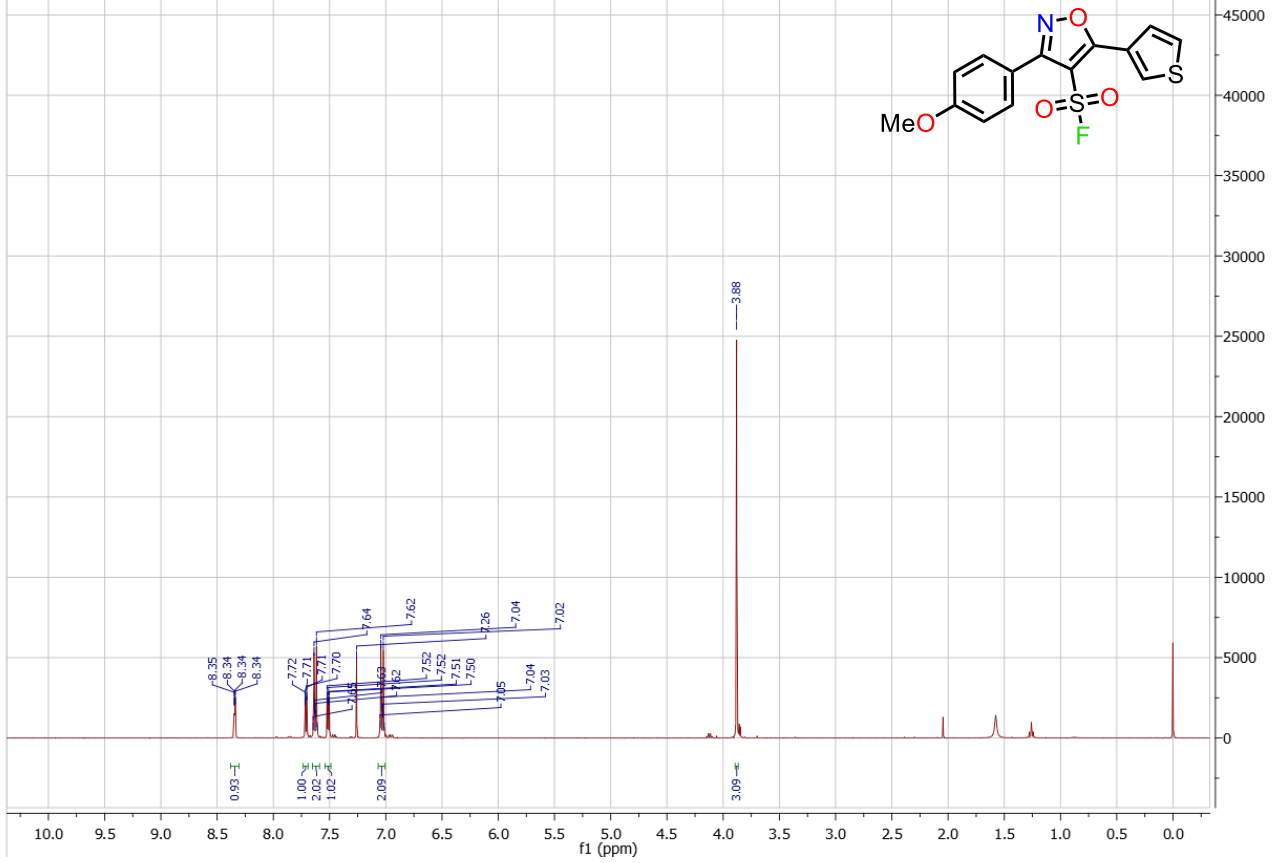




ligc-5-88-1-F1.fid
F-19, CDCl3, DPX-400 QNP Probe. CF3Cl as Ref at 0 ppm.

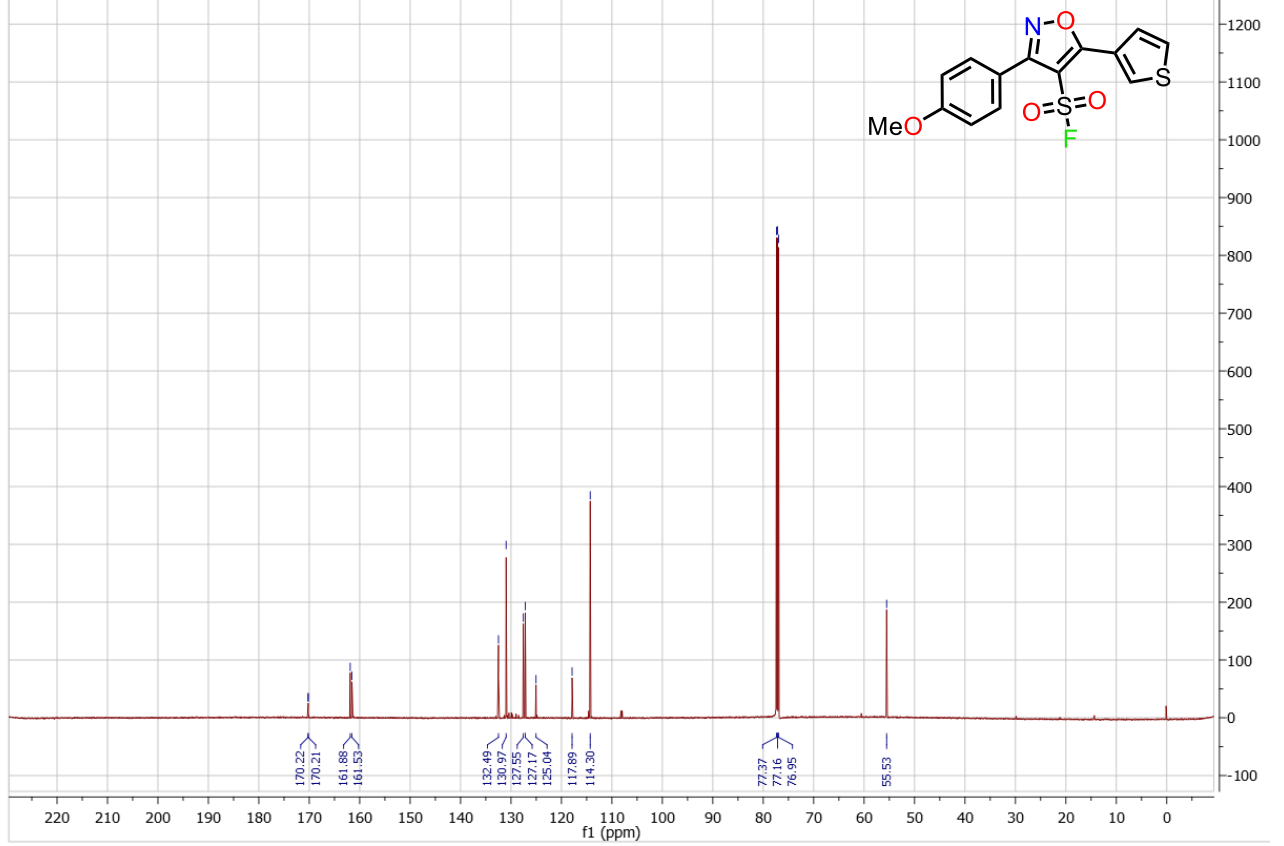


ligc-5-86.1.fid

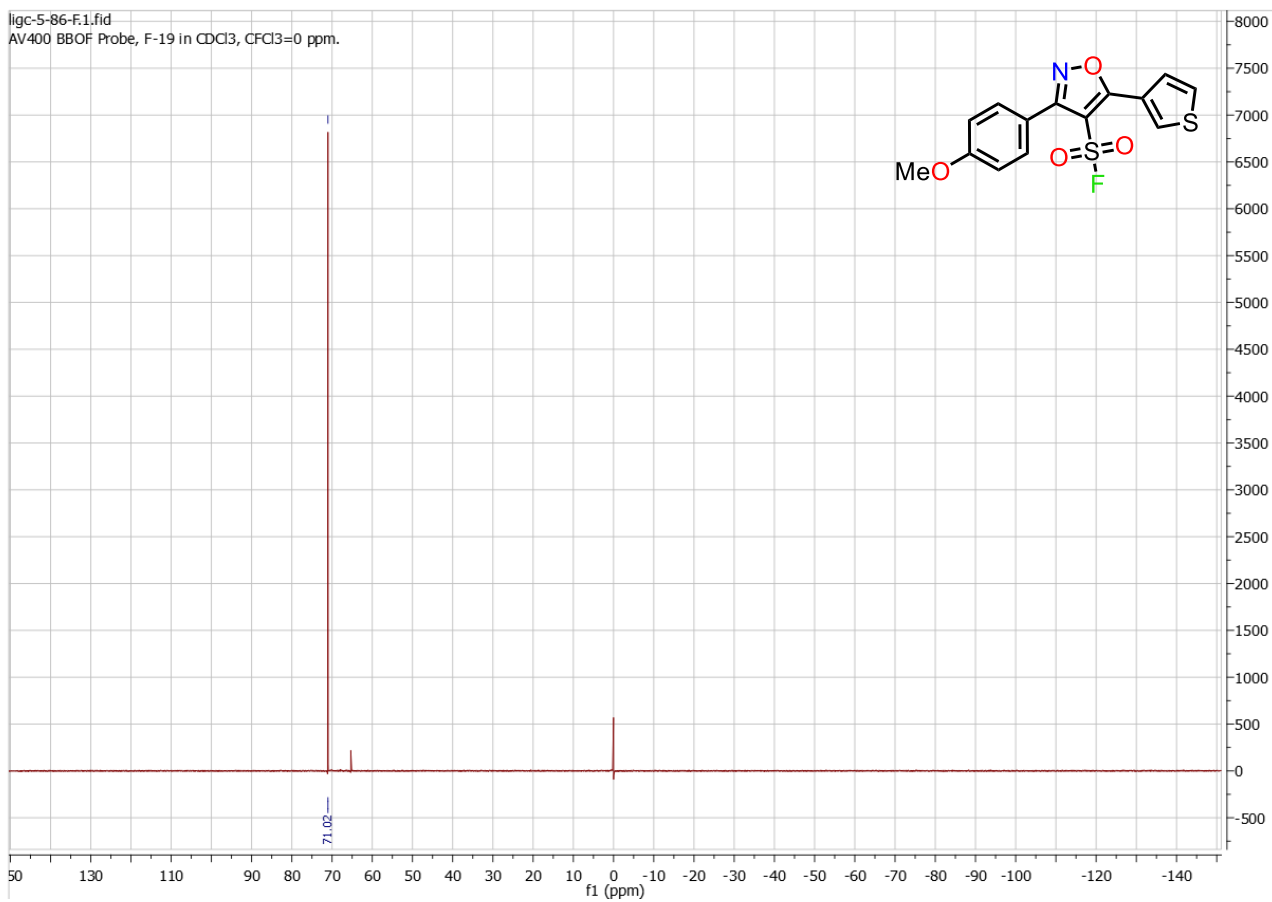


ligc-5-86-C13.1.fid

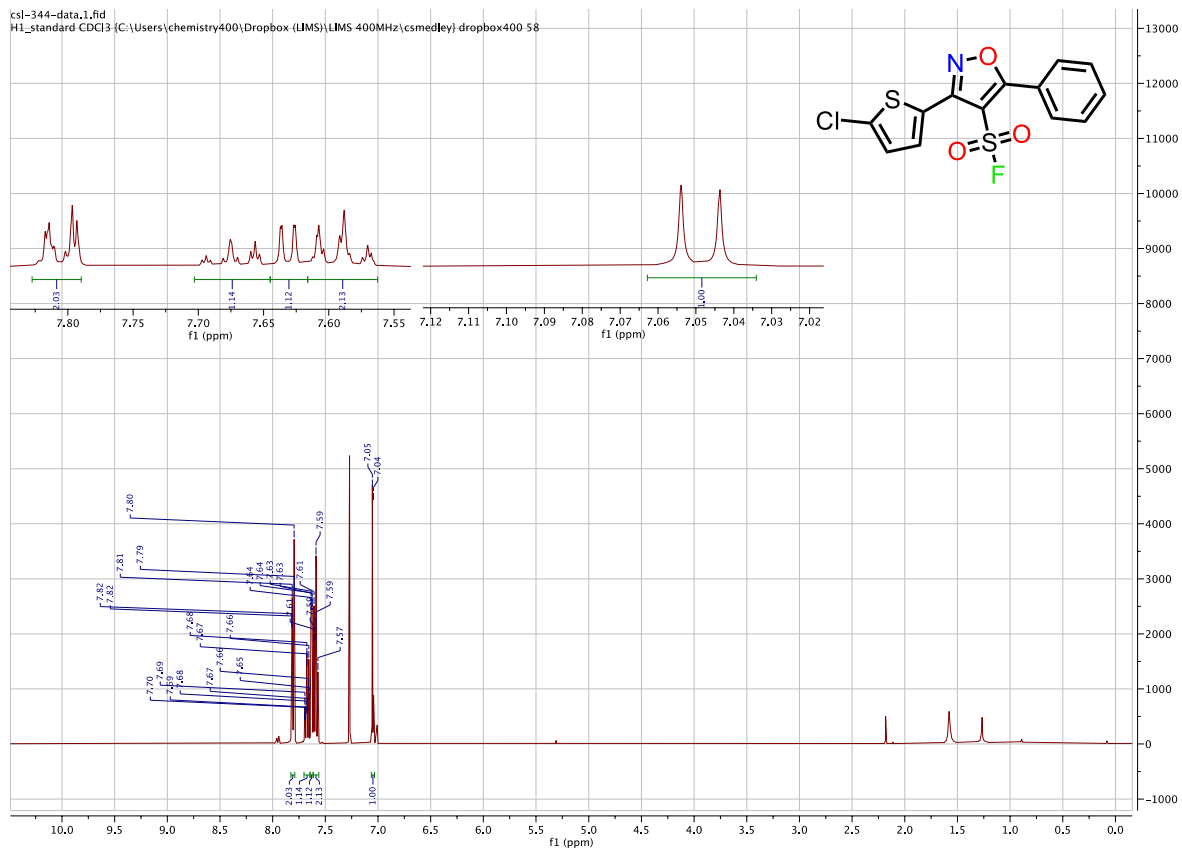
C-13 Routine 1D, CPDCH CryoProbe, AVIII-600



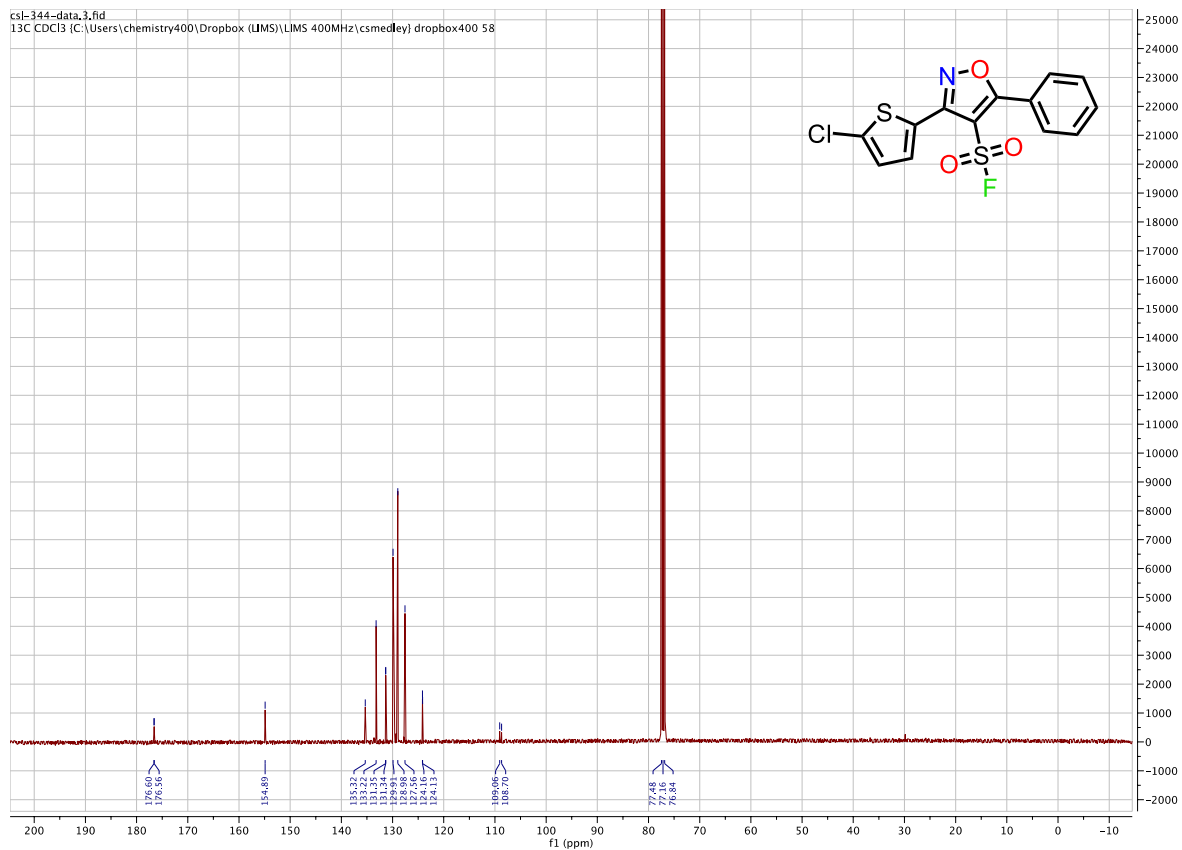
ligc-5-86-F1.fid
AV400 BBOF Probe, F-19 in CDCl3, CFCl3=0 ppm.



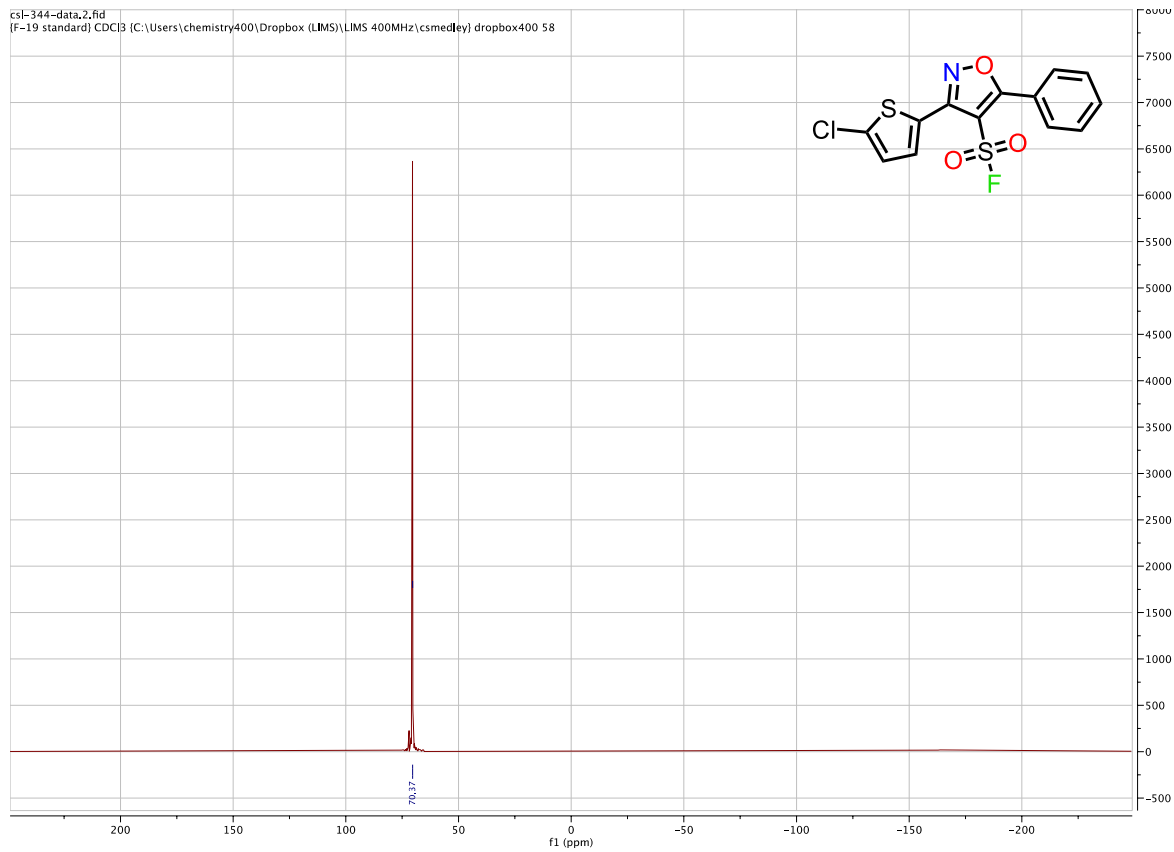
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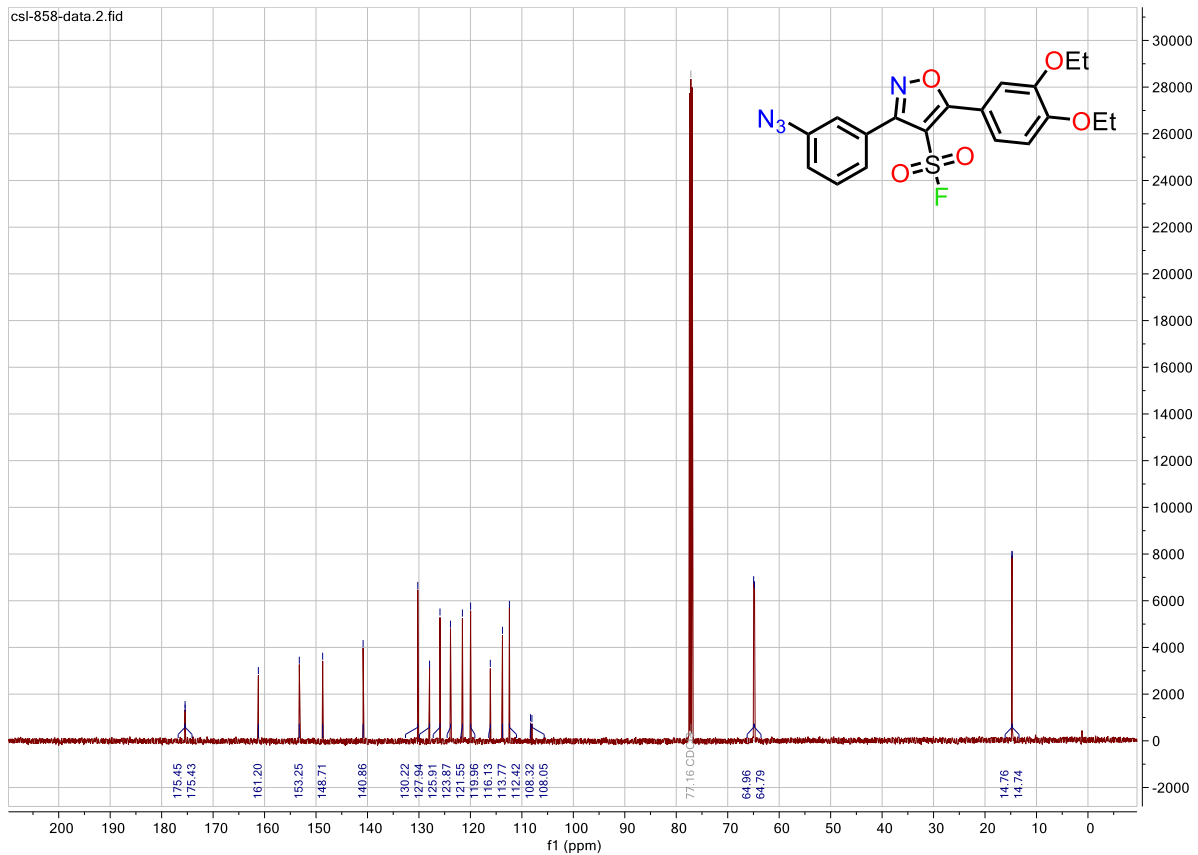
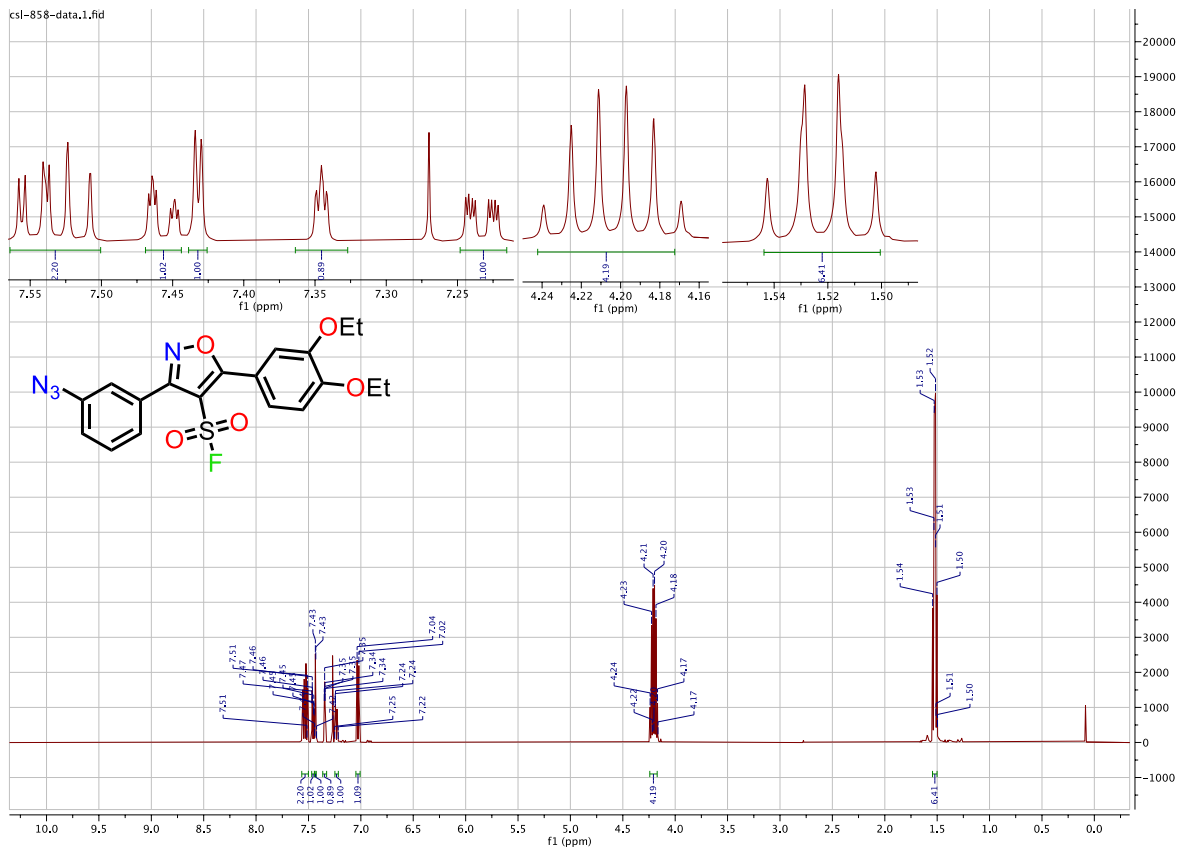


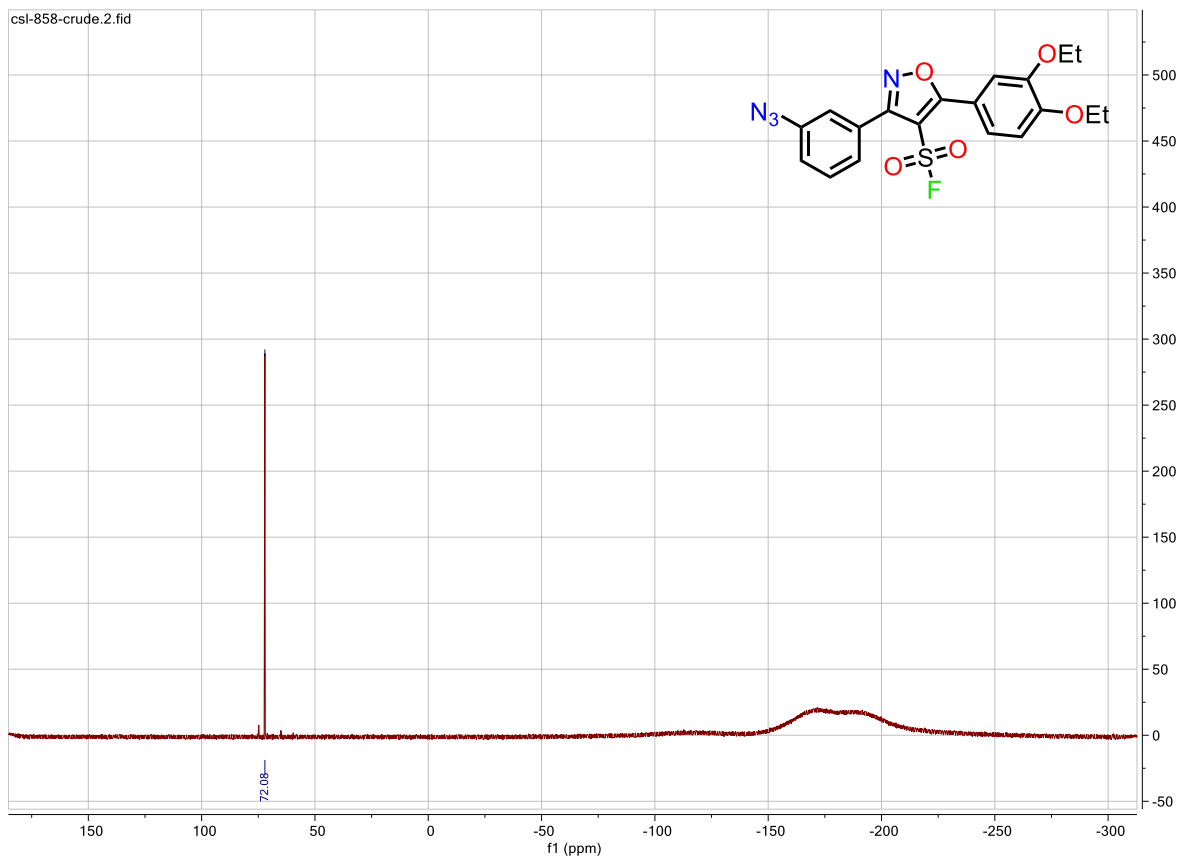
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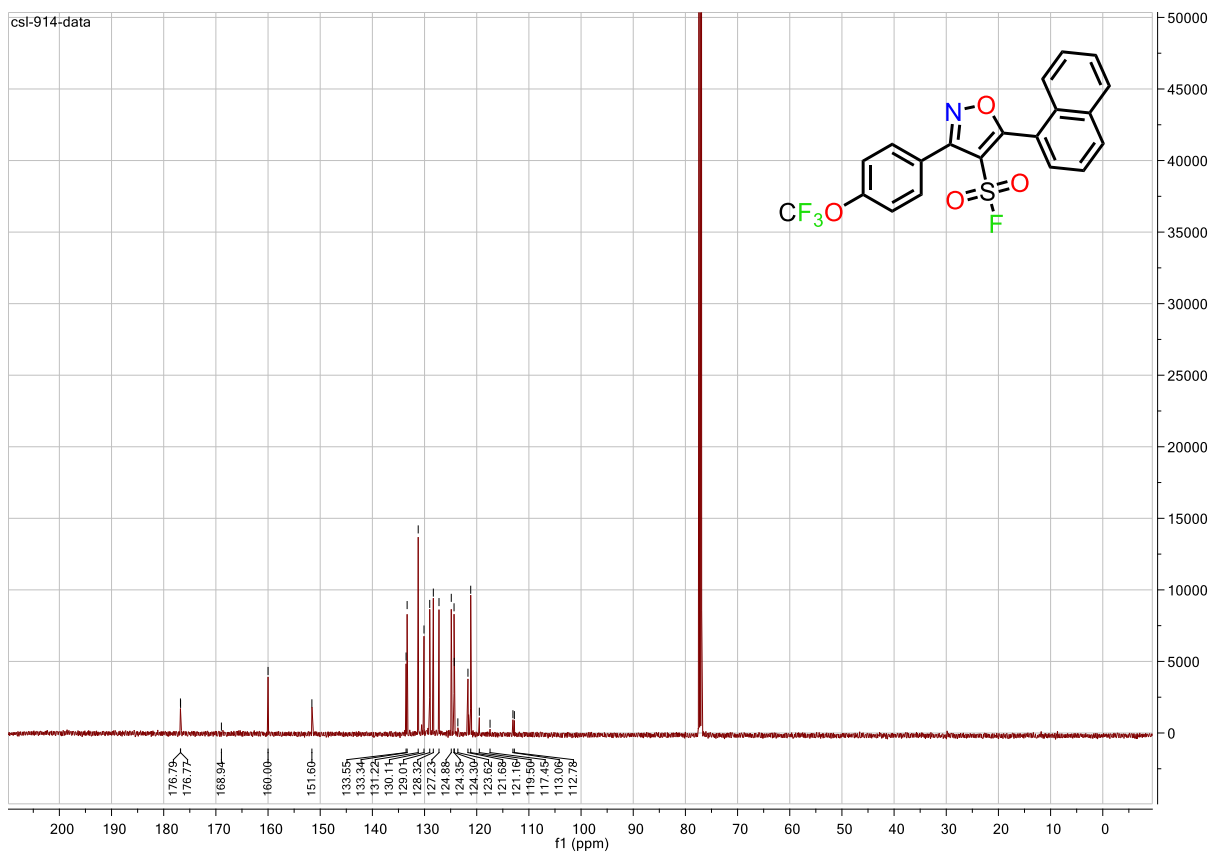
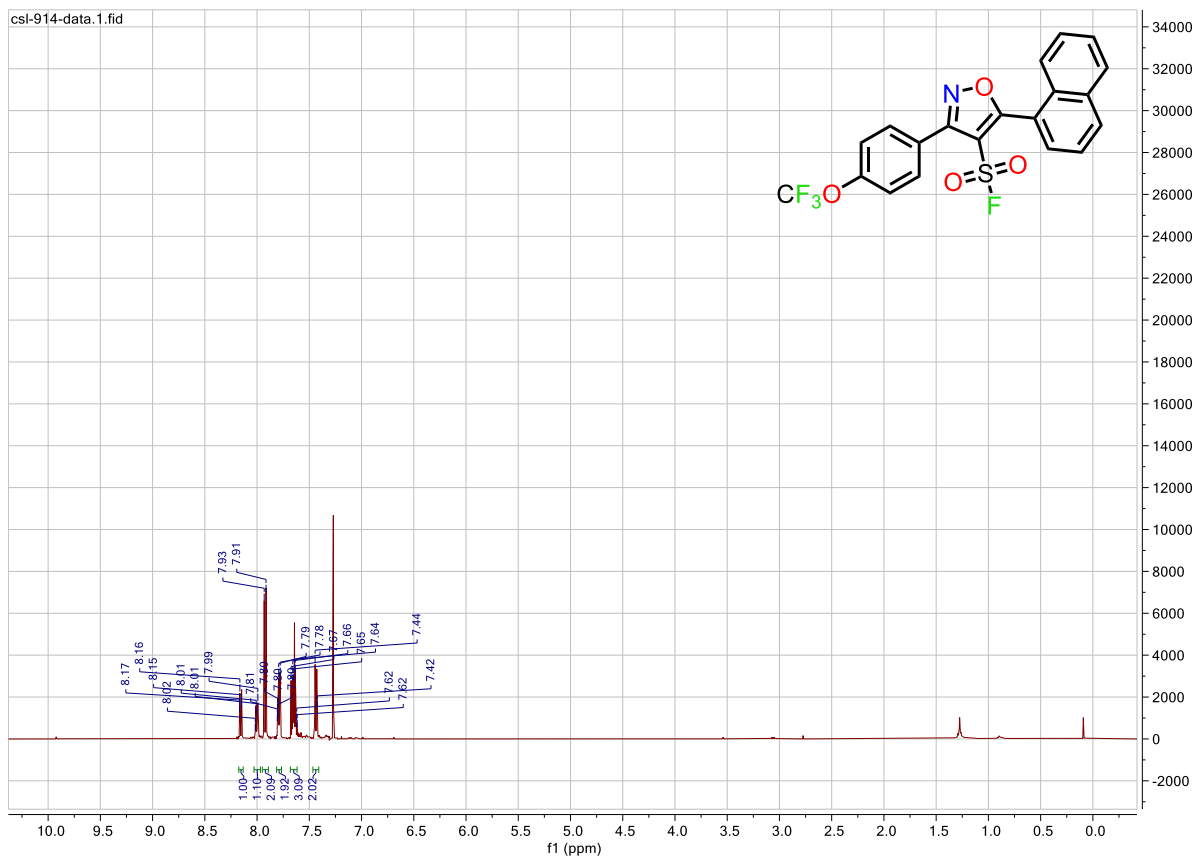


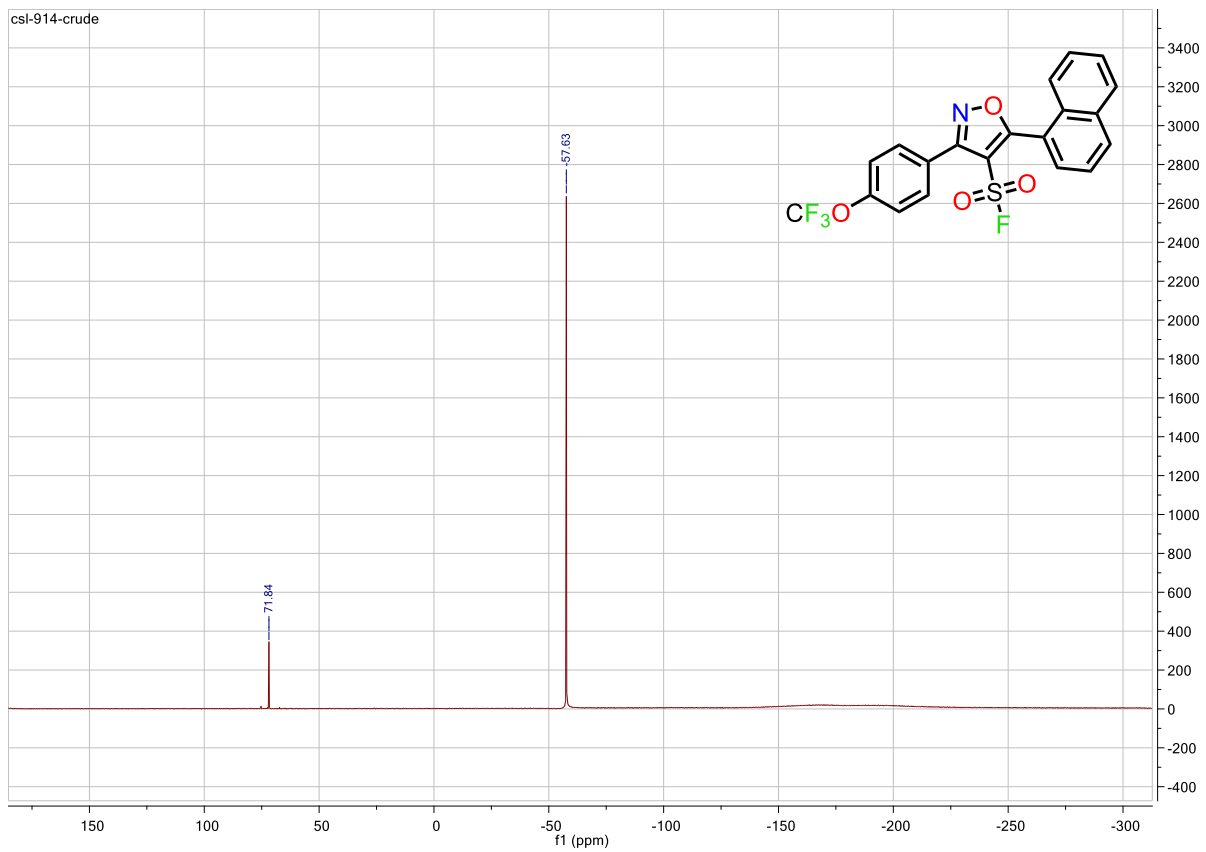
csi-344--data,2.fid
(F-19 standard) CDCl₃ (C:\Users\chemistry400\Dropbox (LIMS)\LIMS 400MHz\csmedley\ dropbox400 58



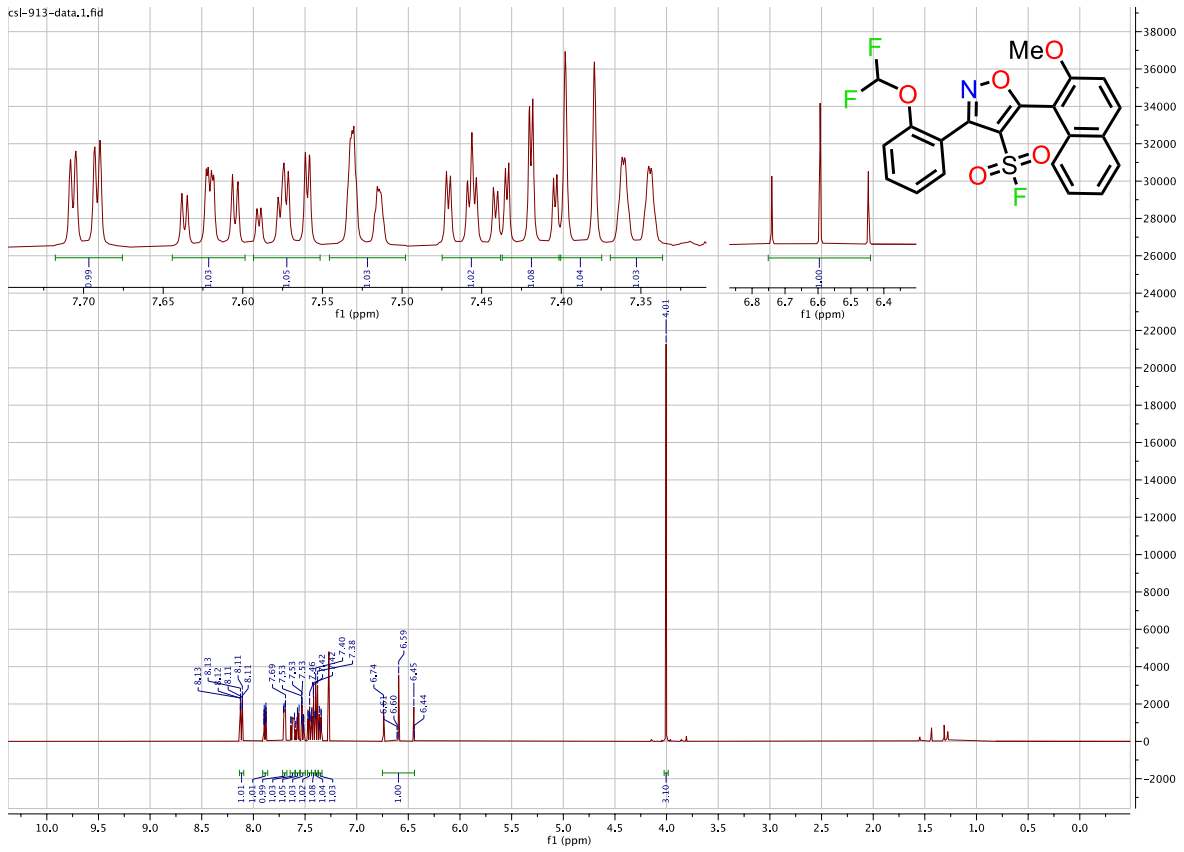




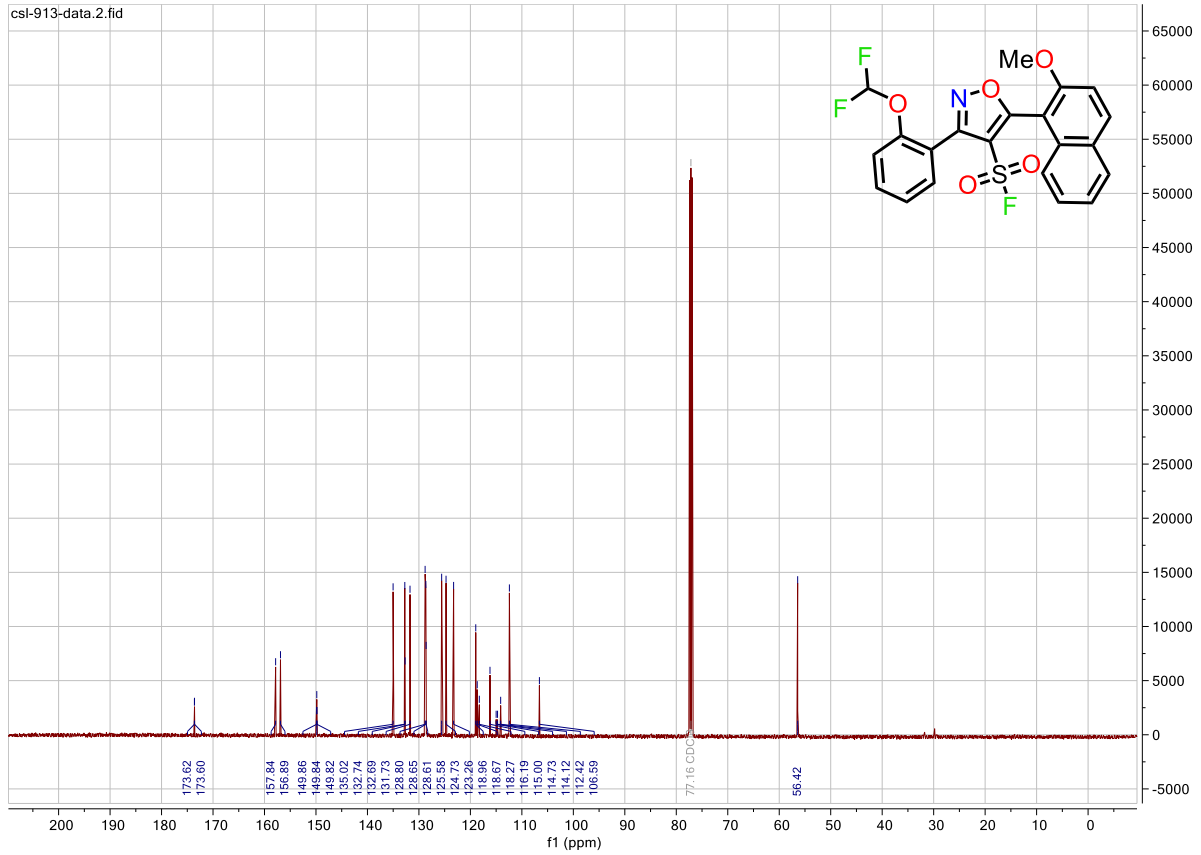


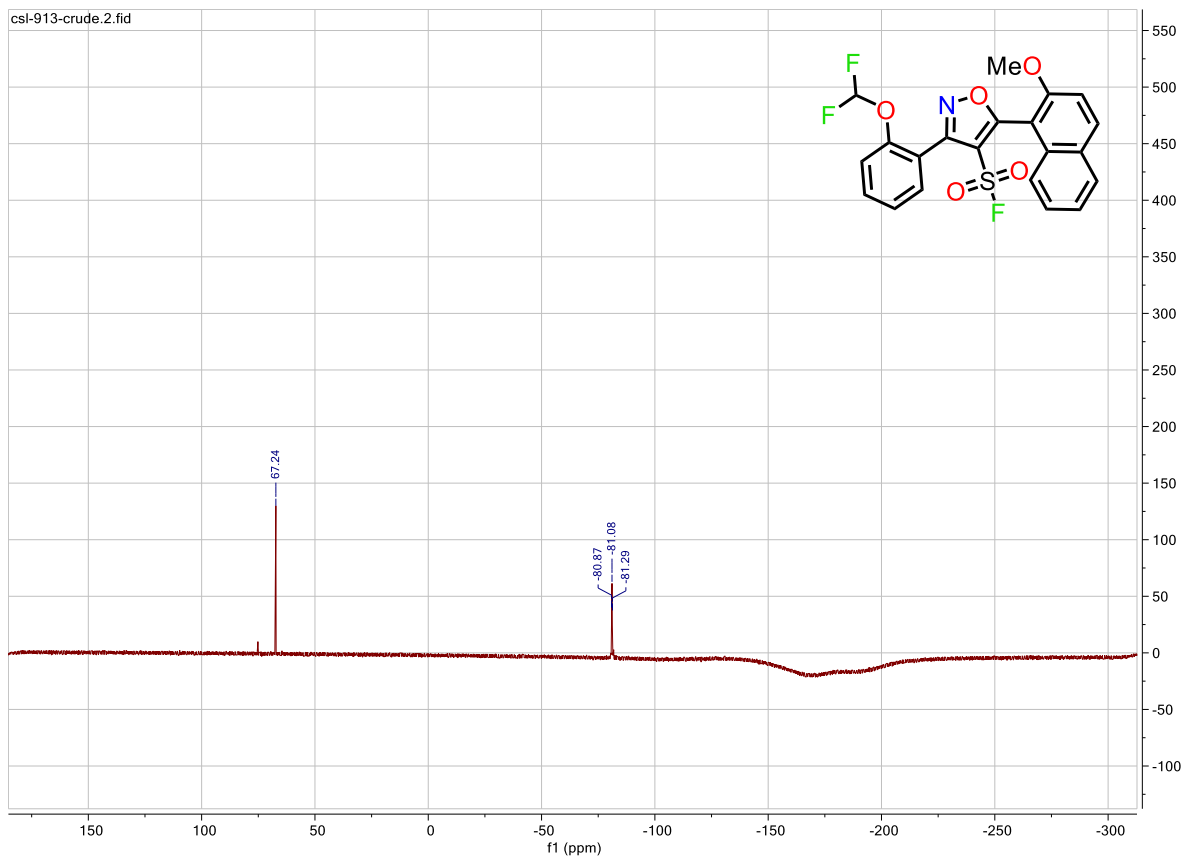


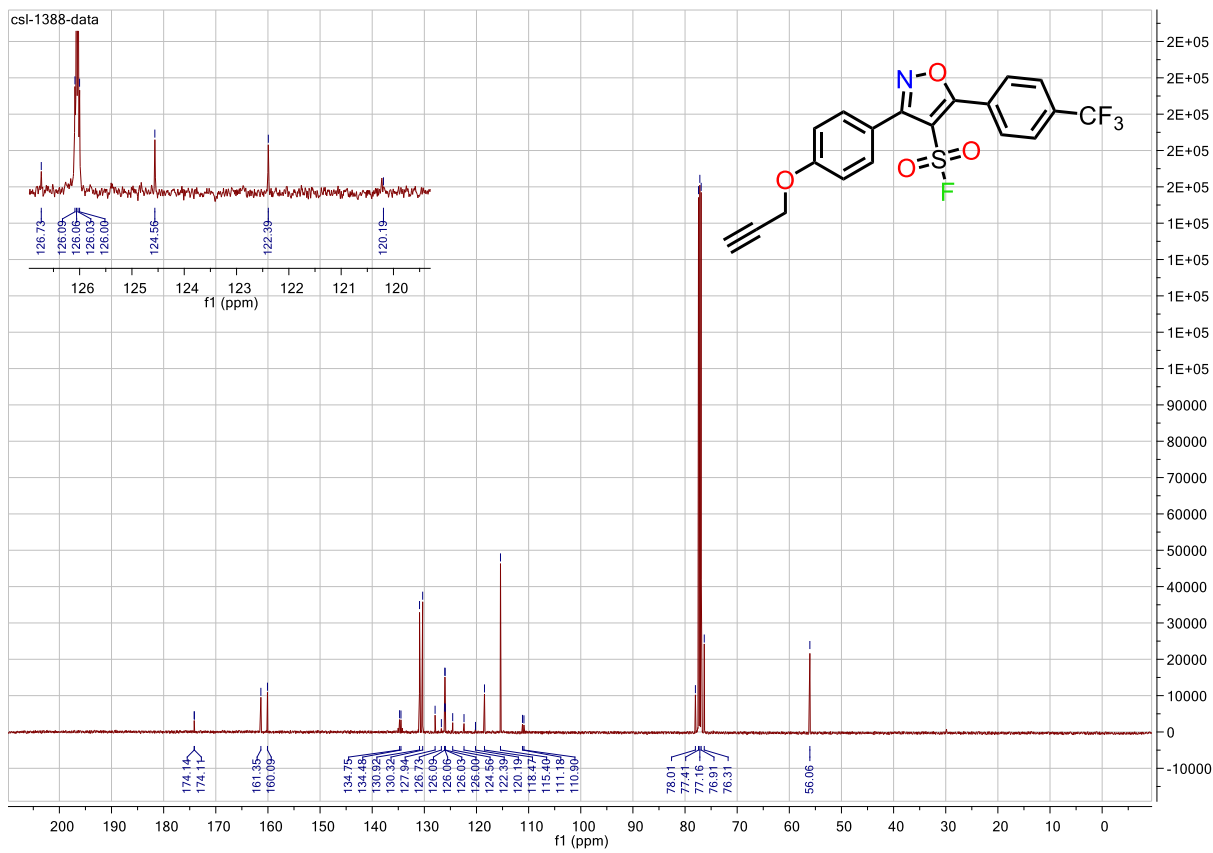
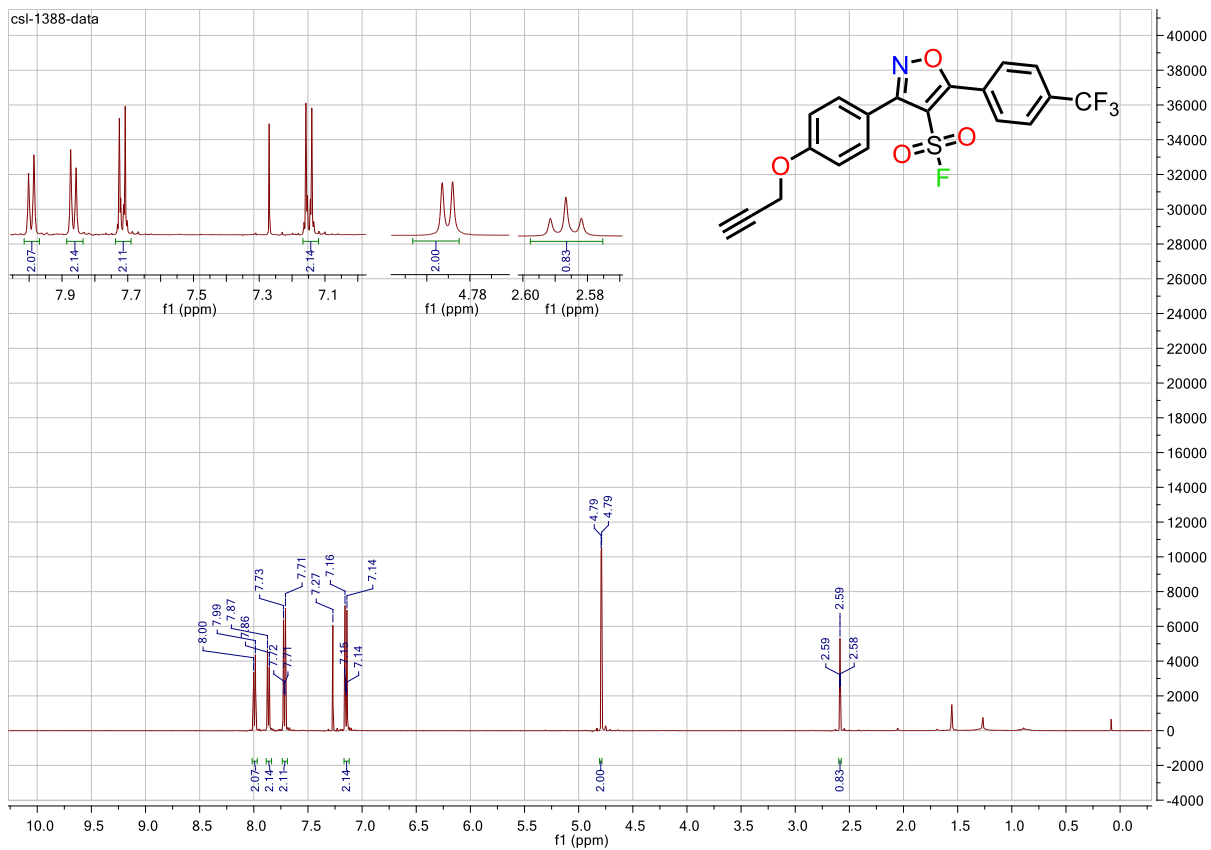
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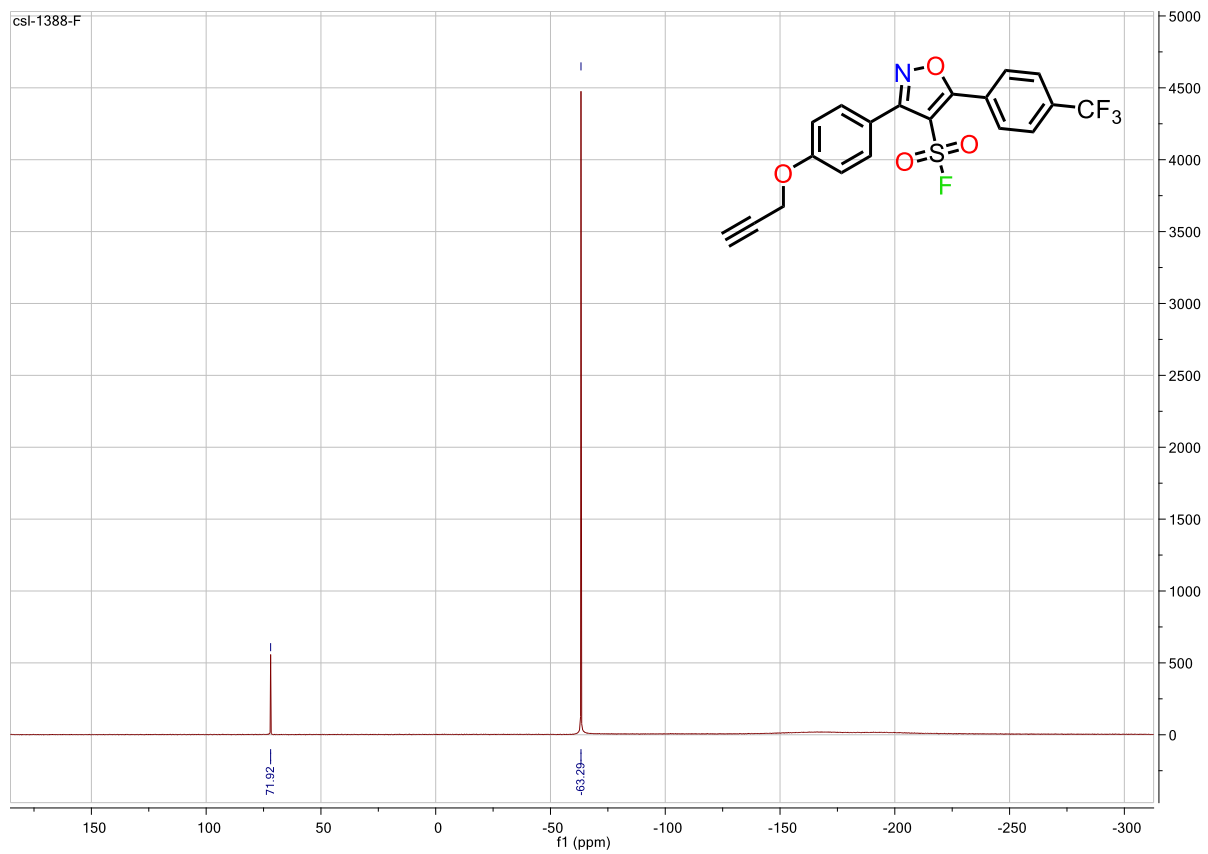


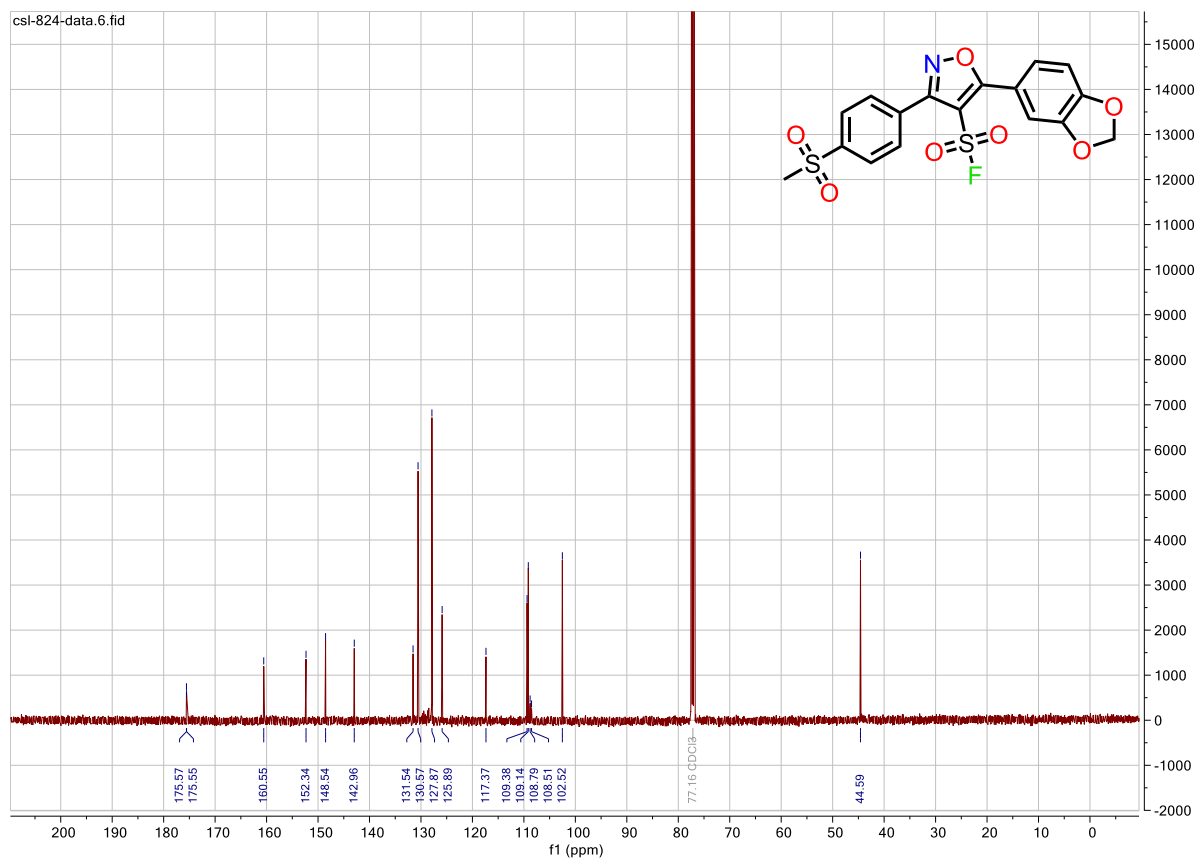
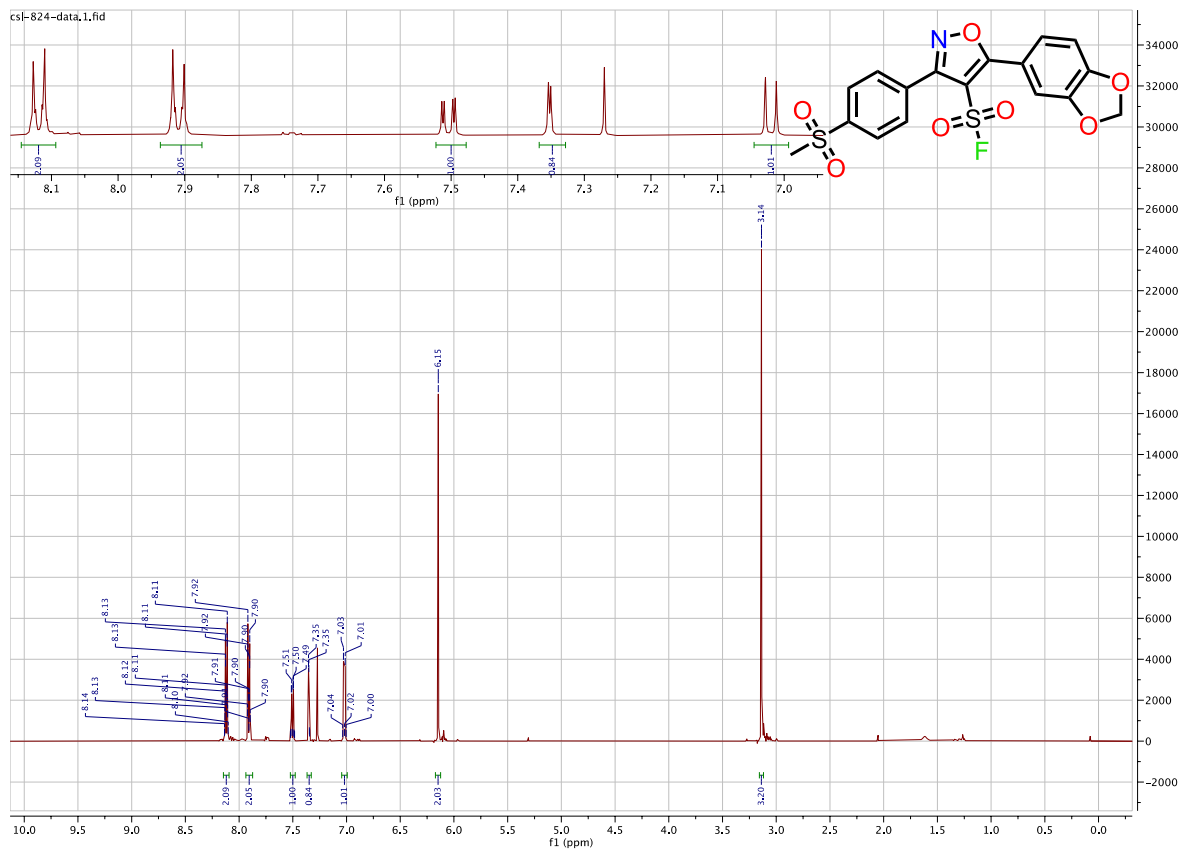
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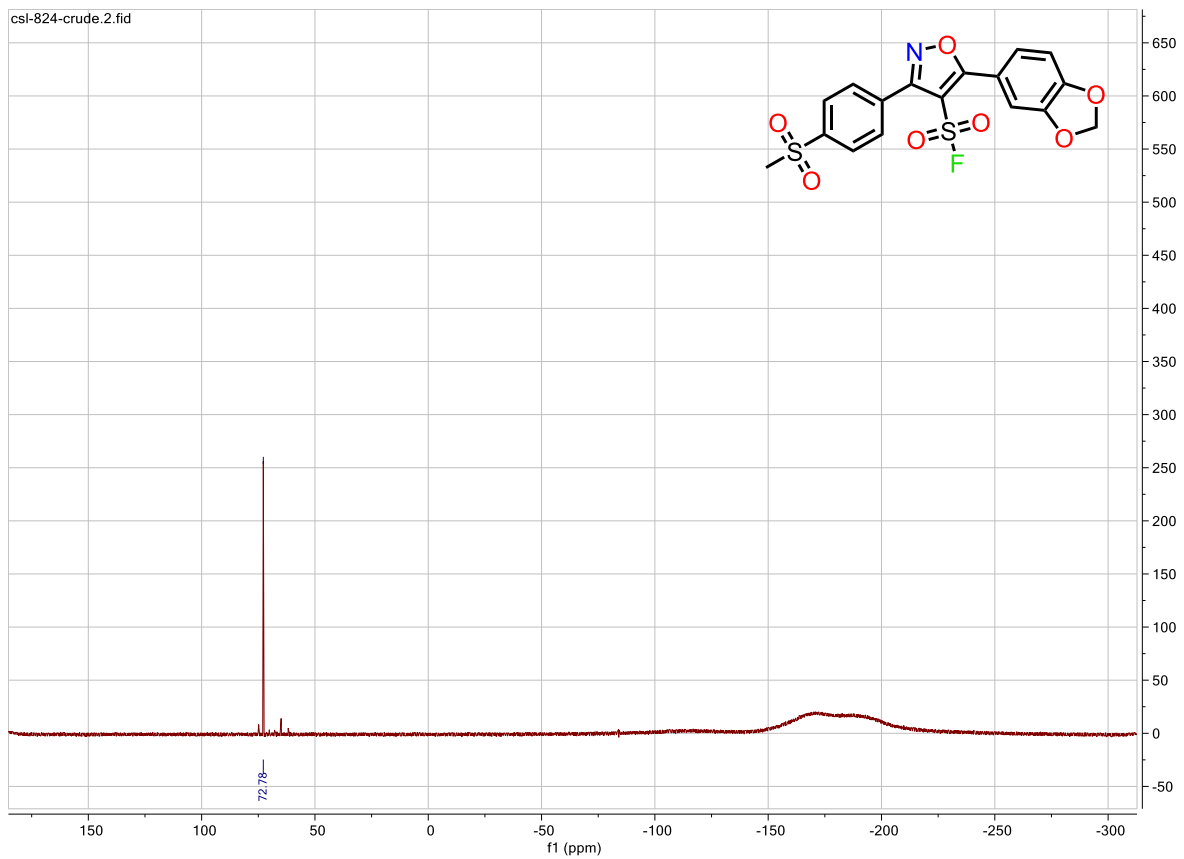


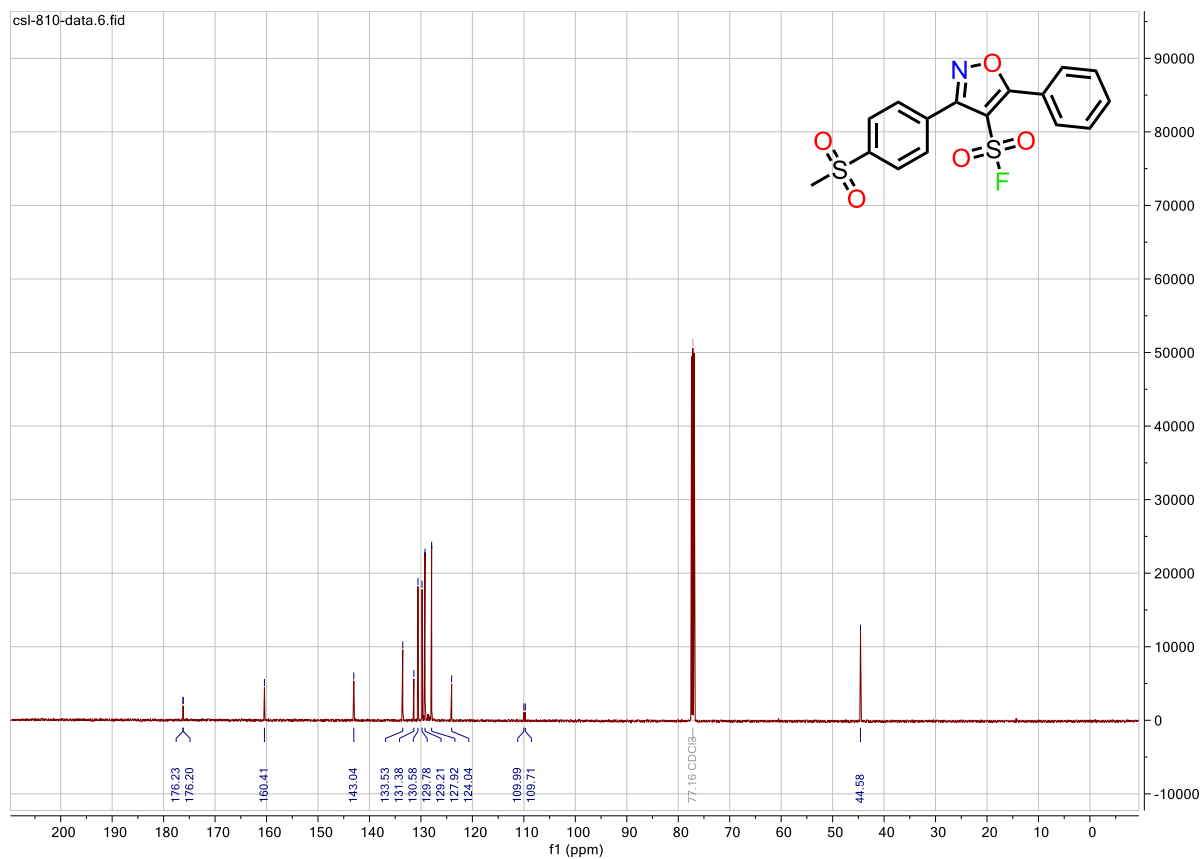
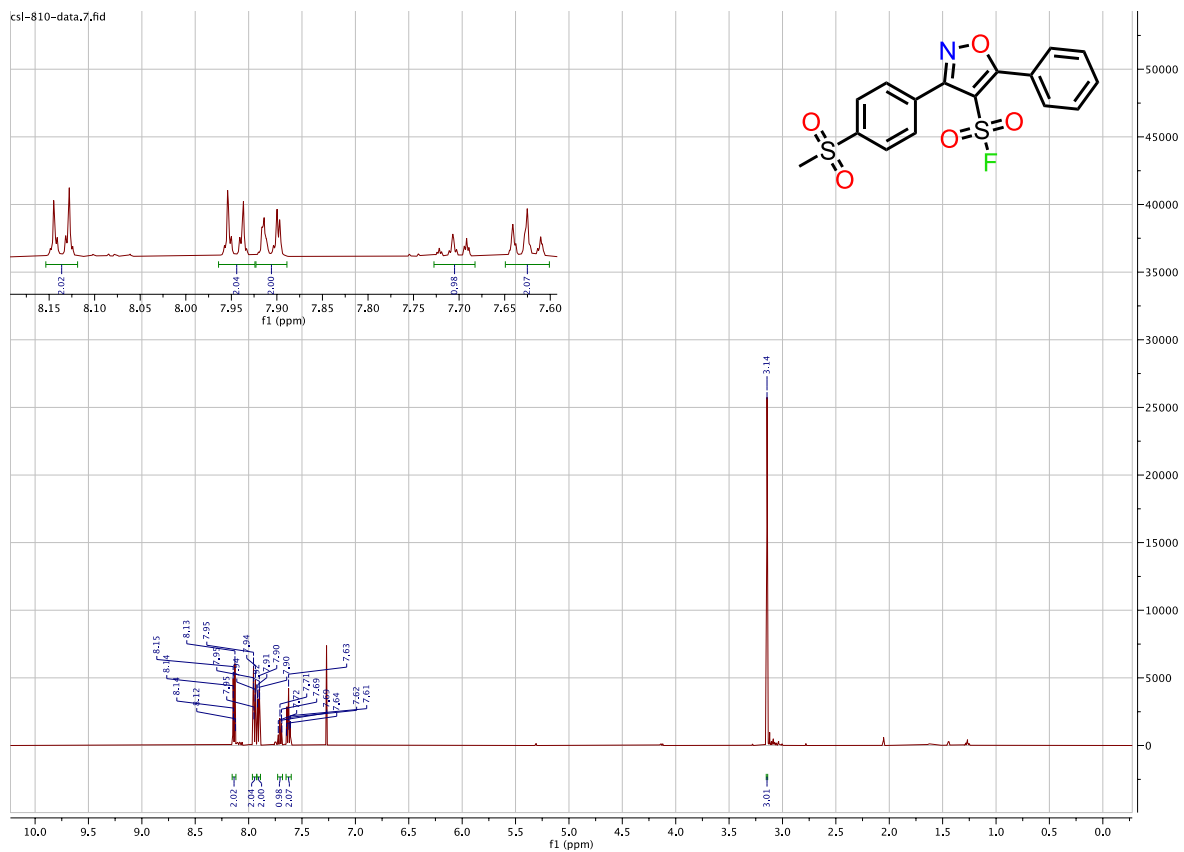


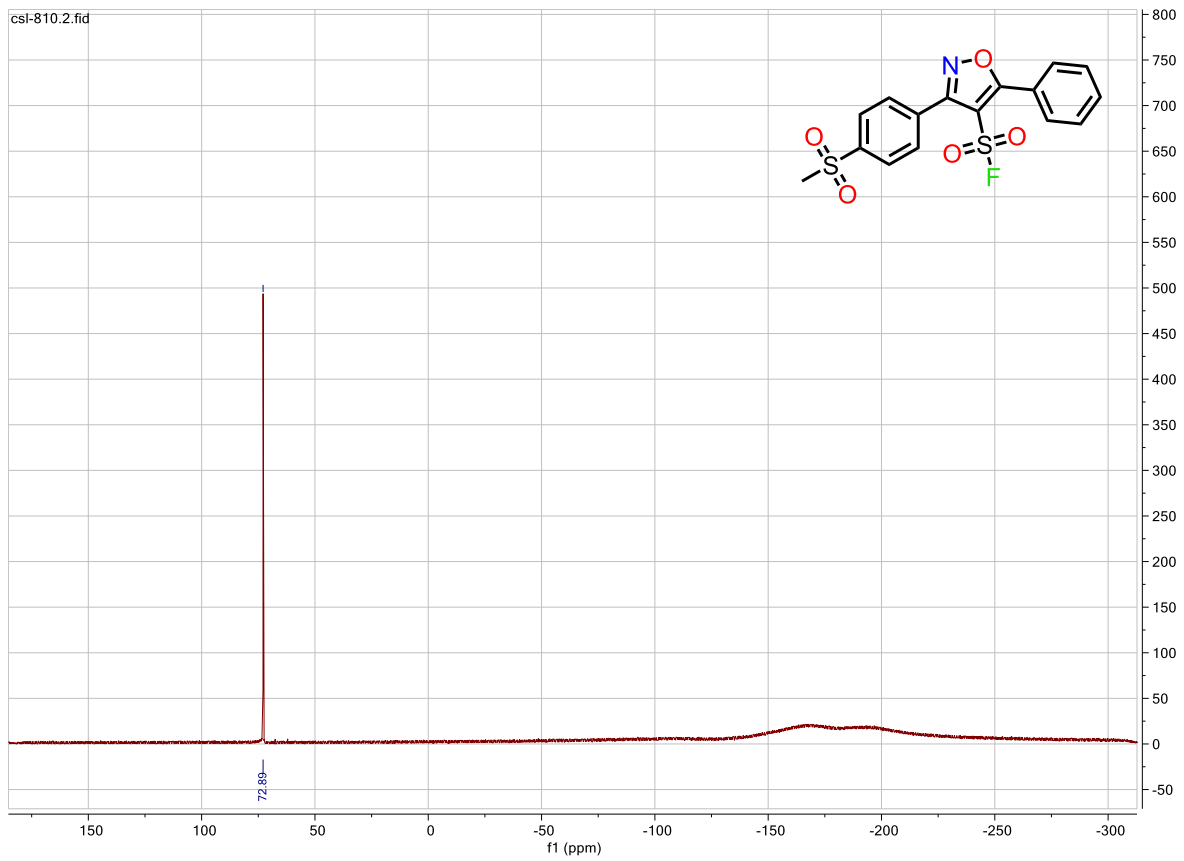


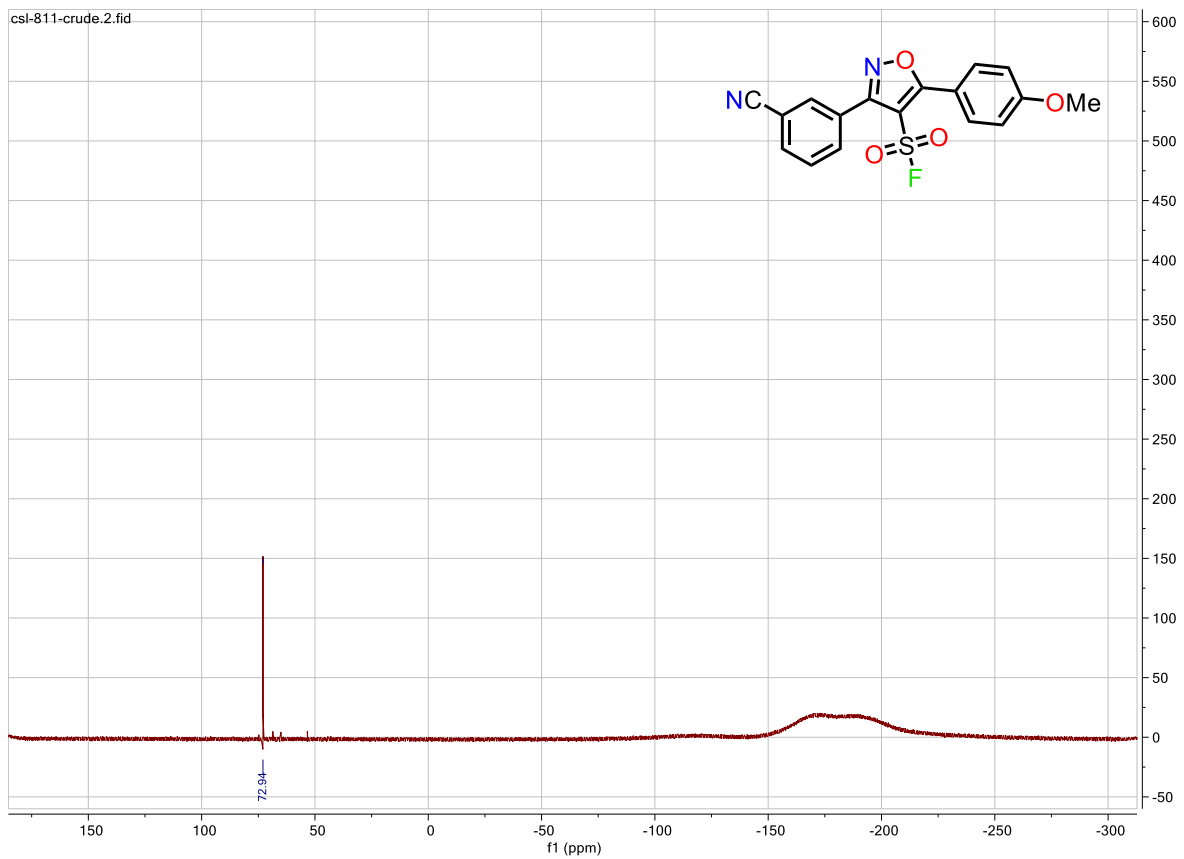


csi-824-crude.2.fid

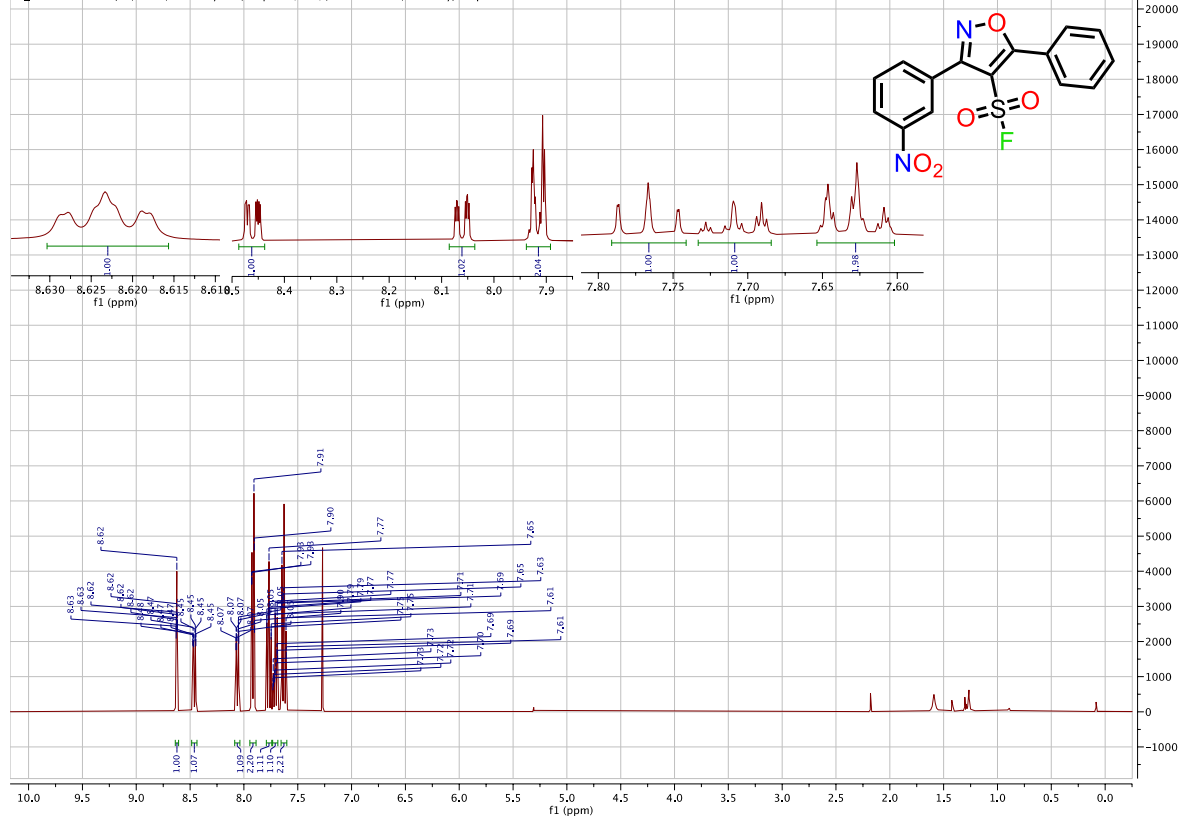




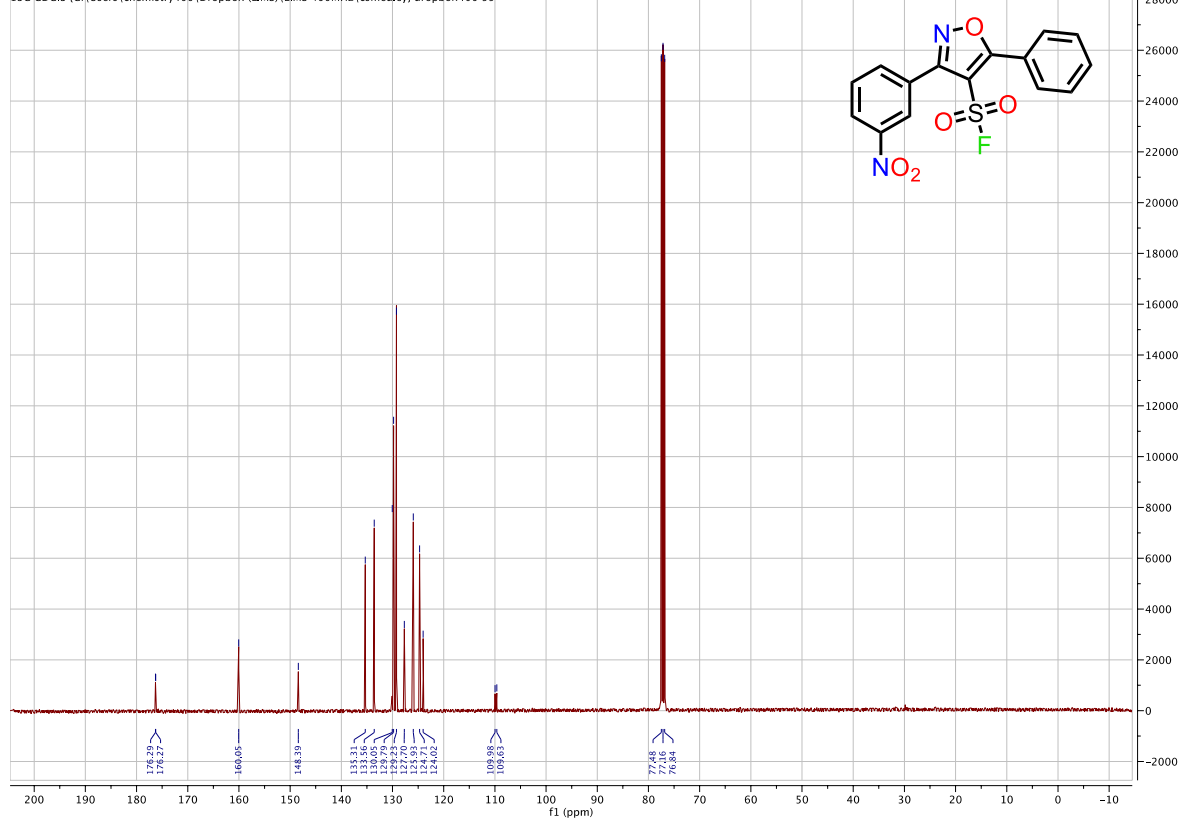




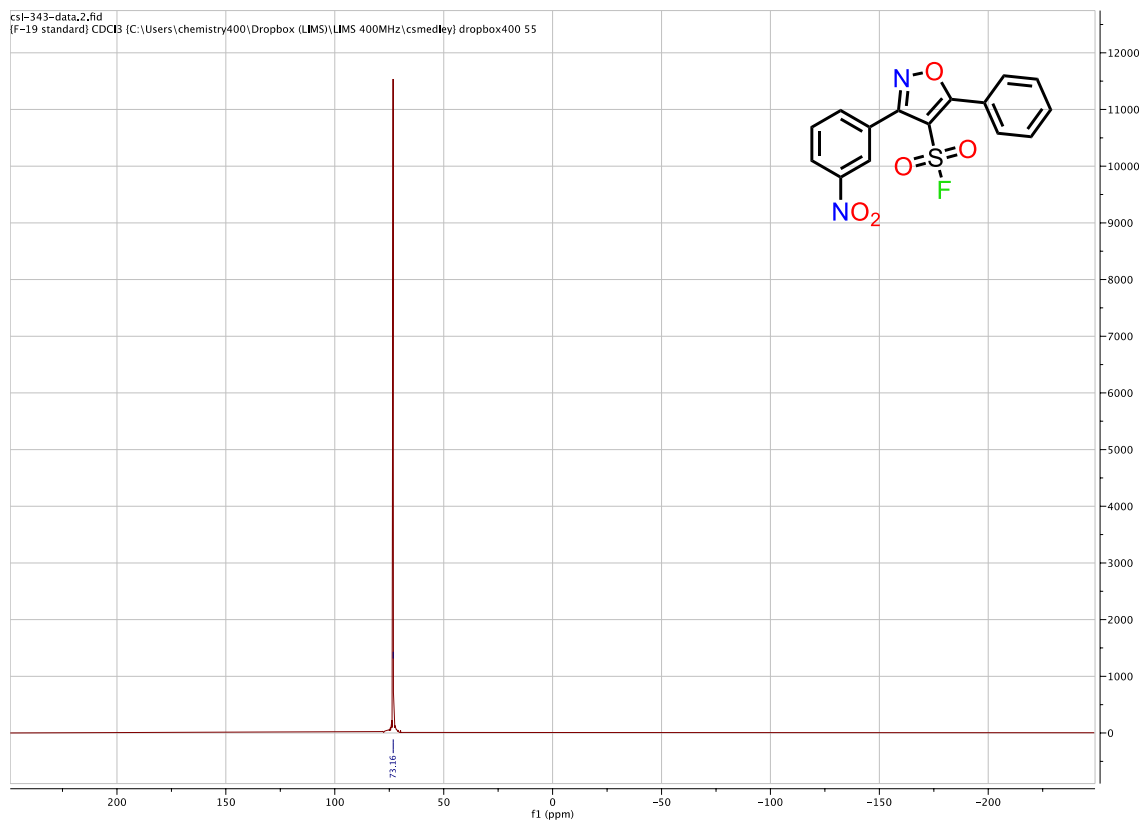
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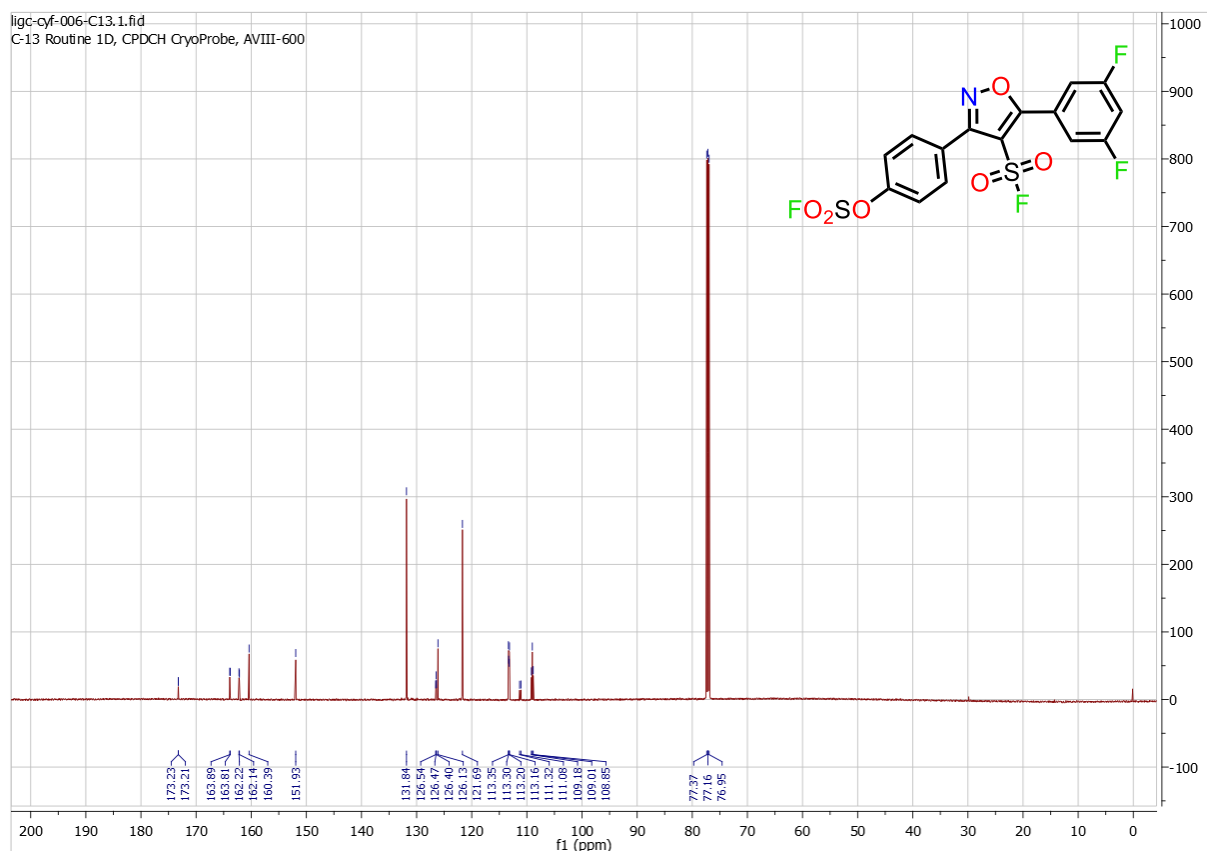
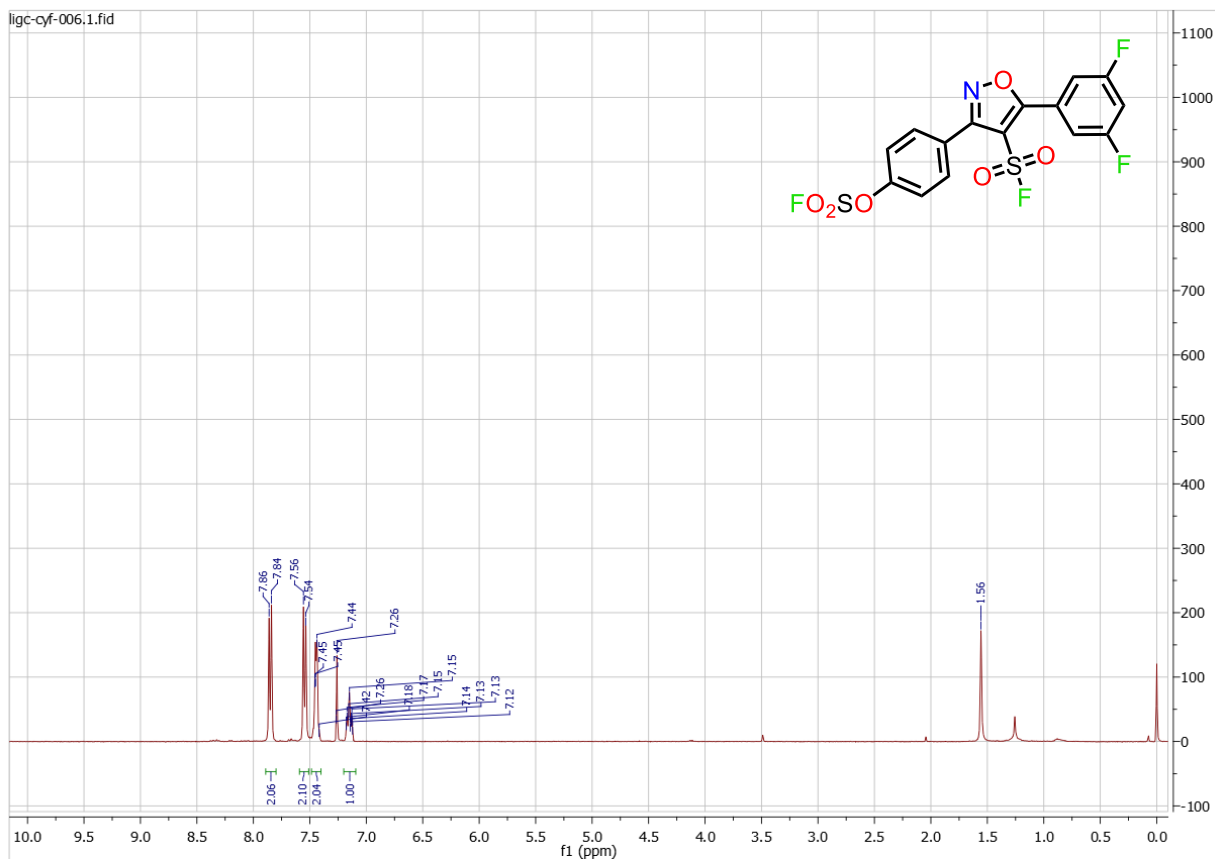


csi-343-data_3.fid
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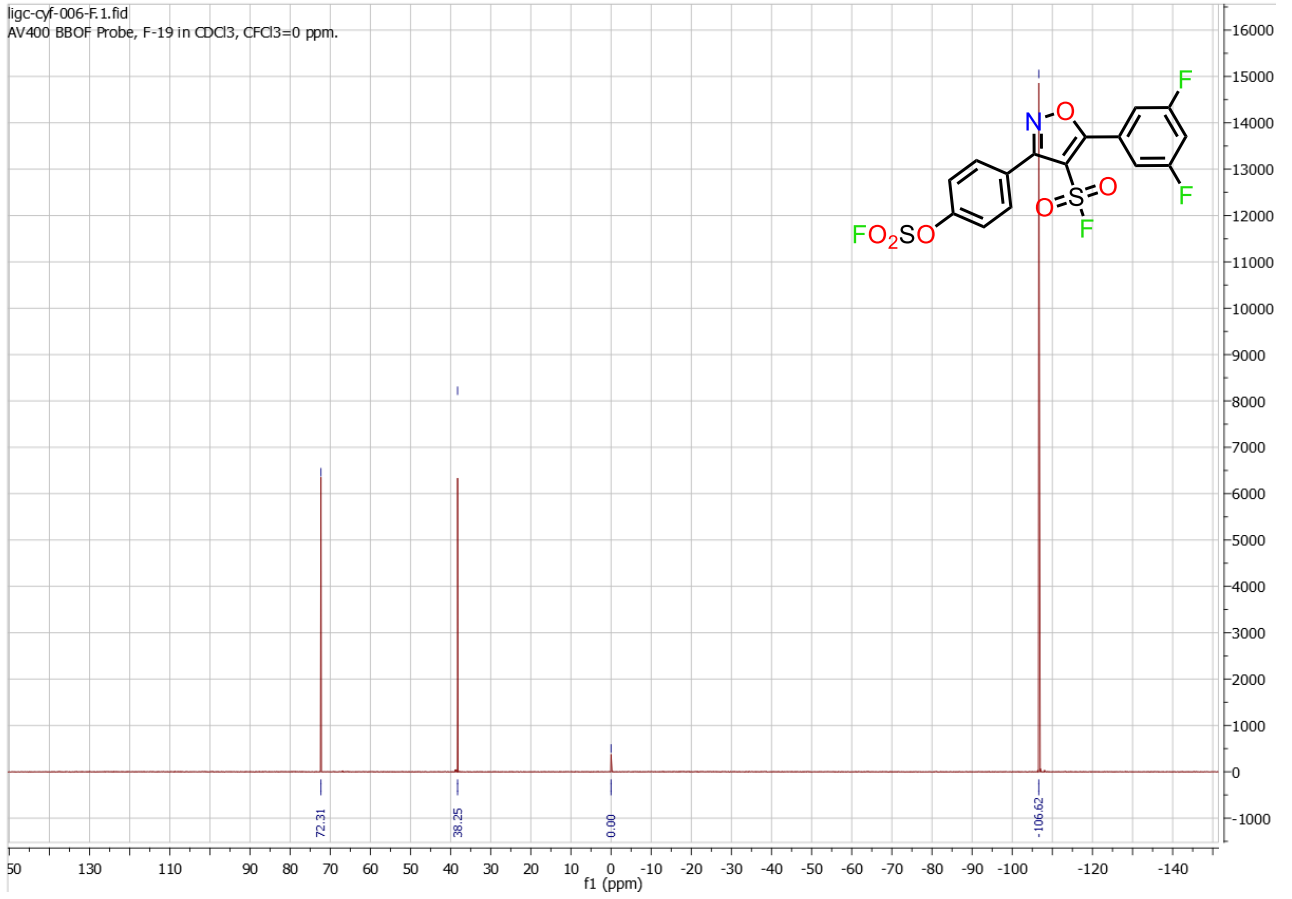


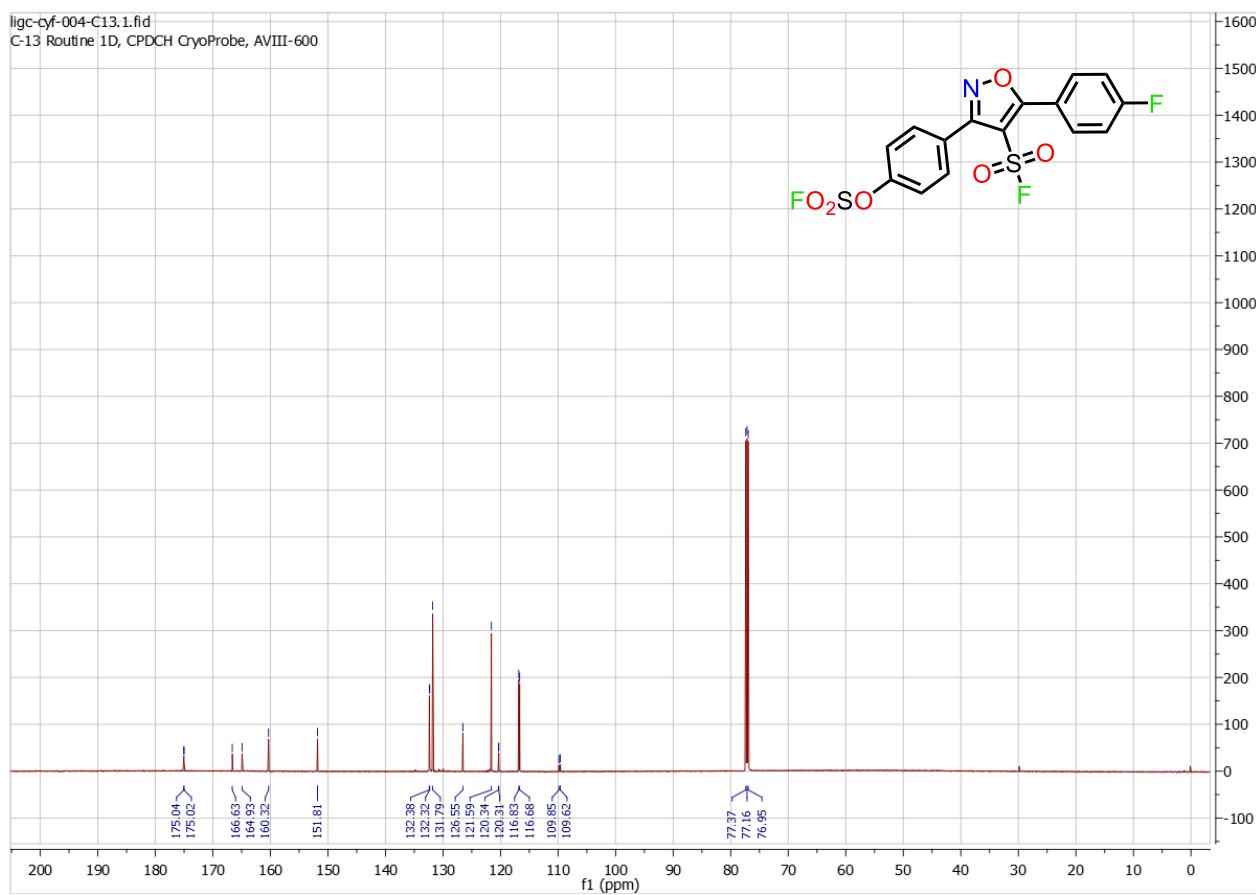
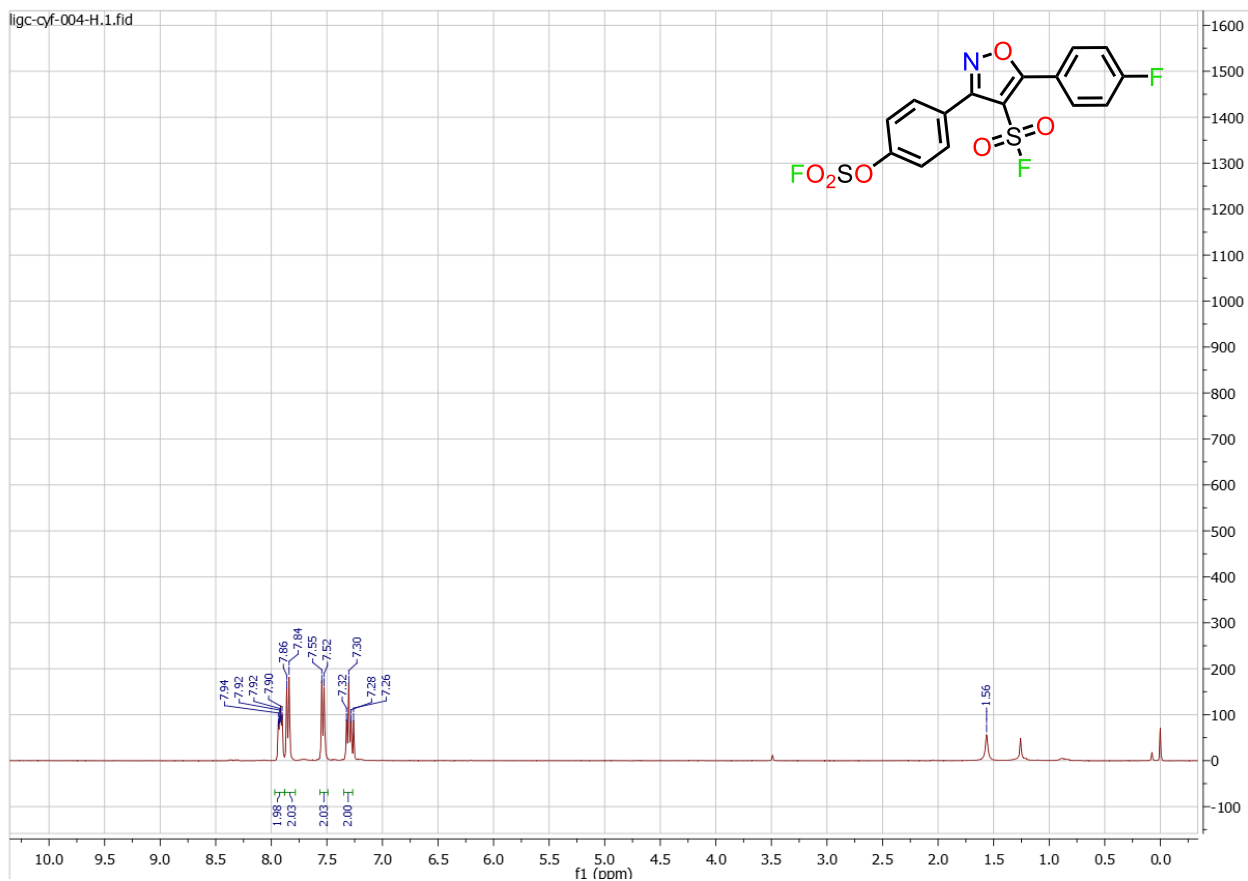
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F-19 standard) CDCl3 (C:\Users\chemistry400\Dropbox (LIMS)\LIMS 400MHz\csmedley) dropbox400 55



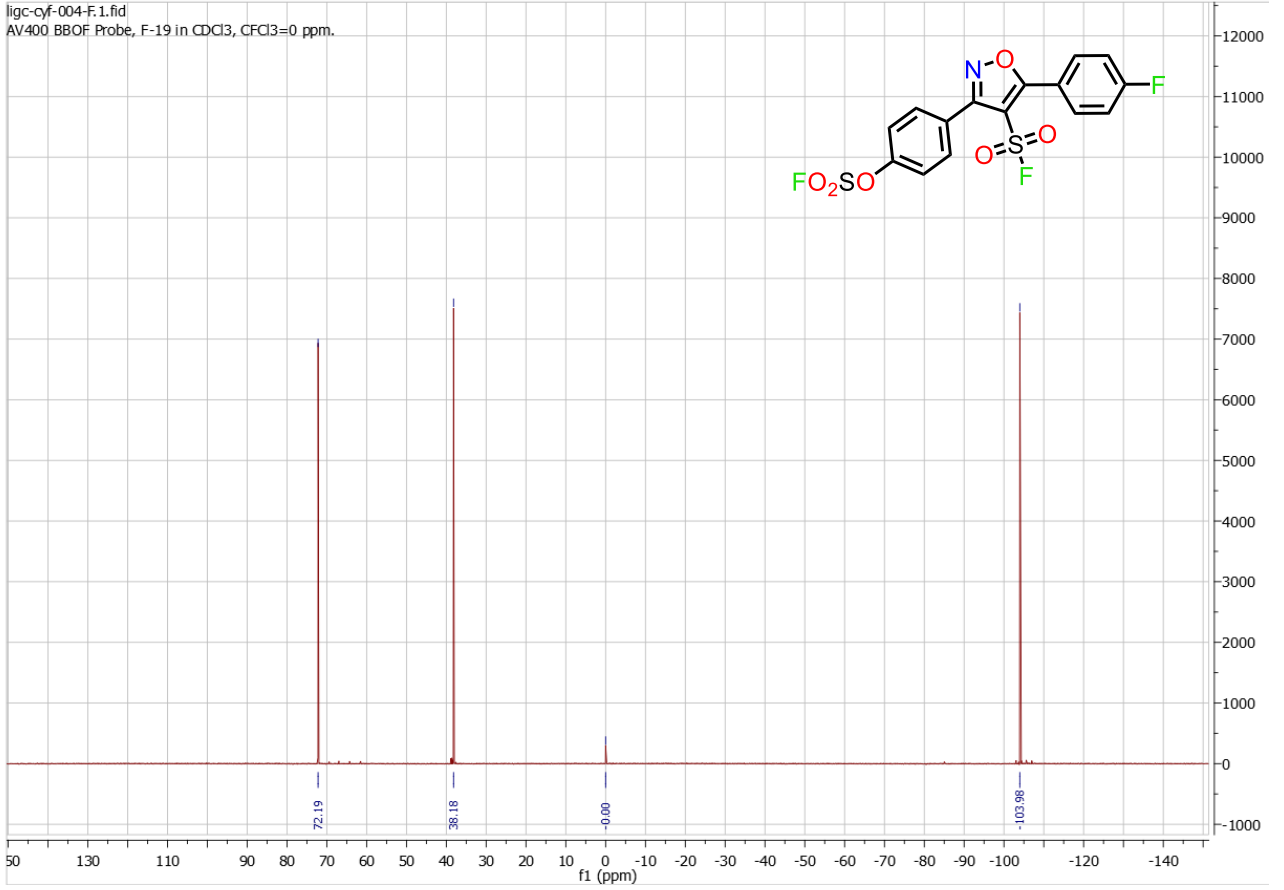


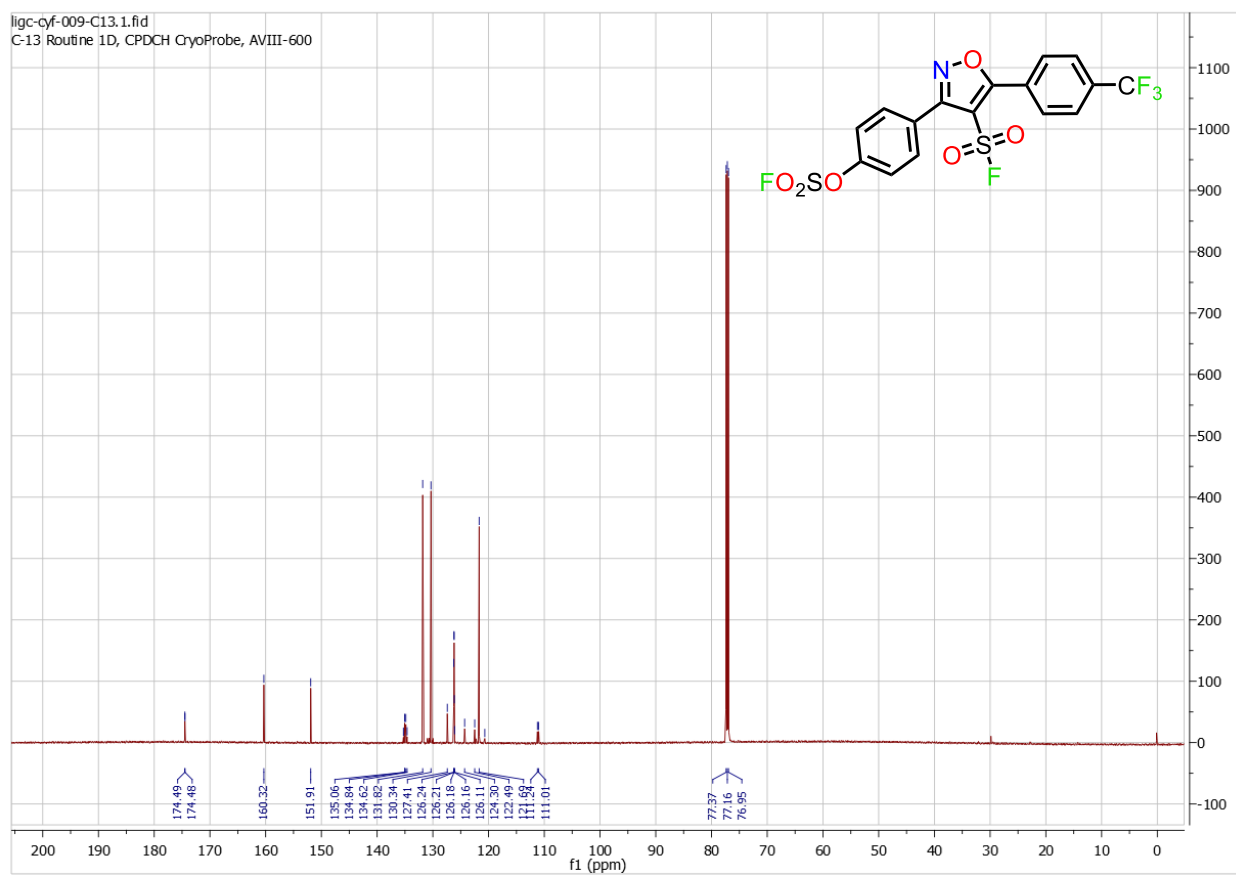
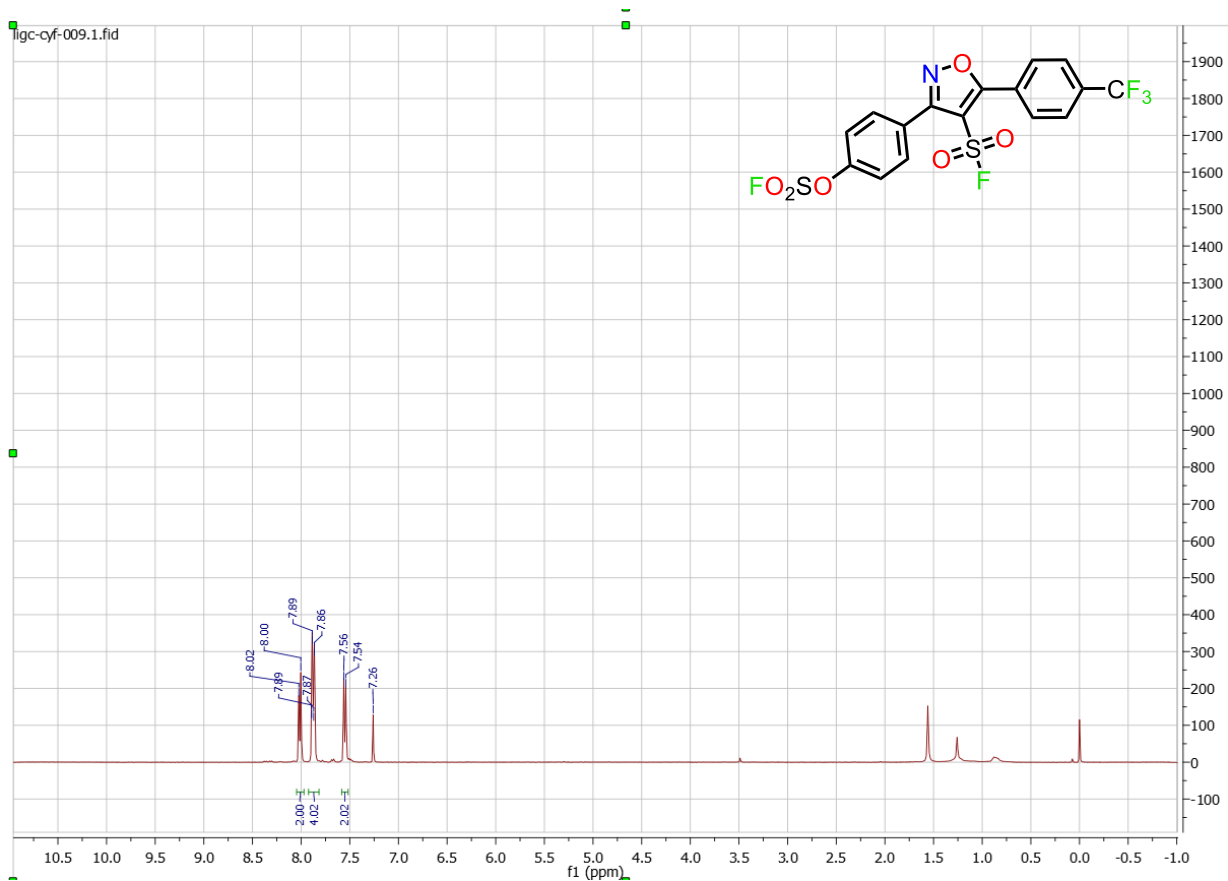
ligc-cyf-006-F.1.fid
AV400 BBOF Probe, F-19 in CDCl3, CFCl3=0 ppm.



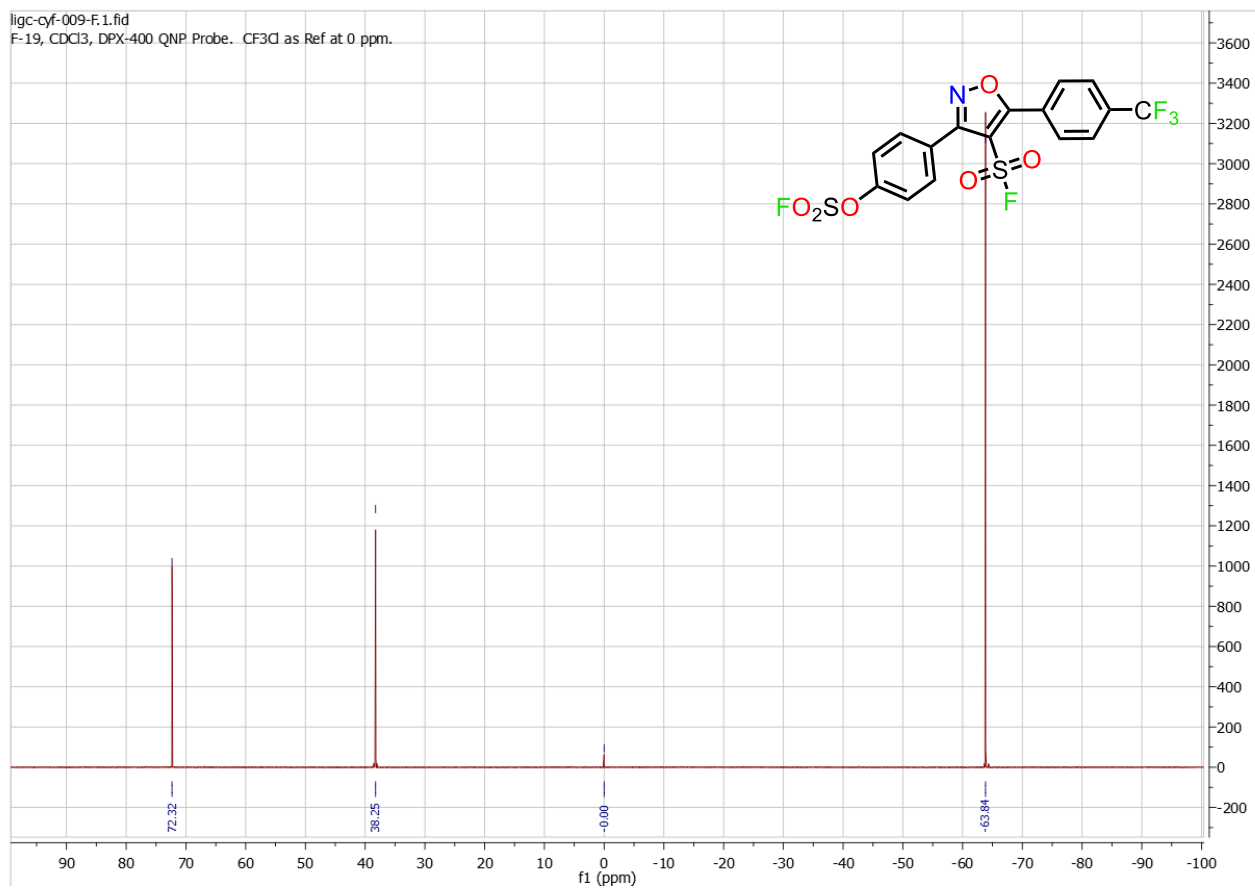


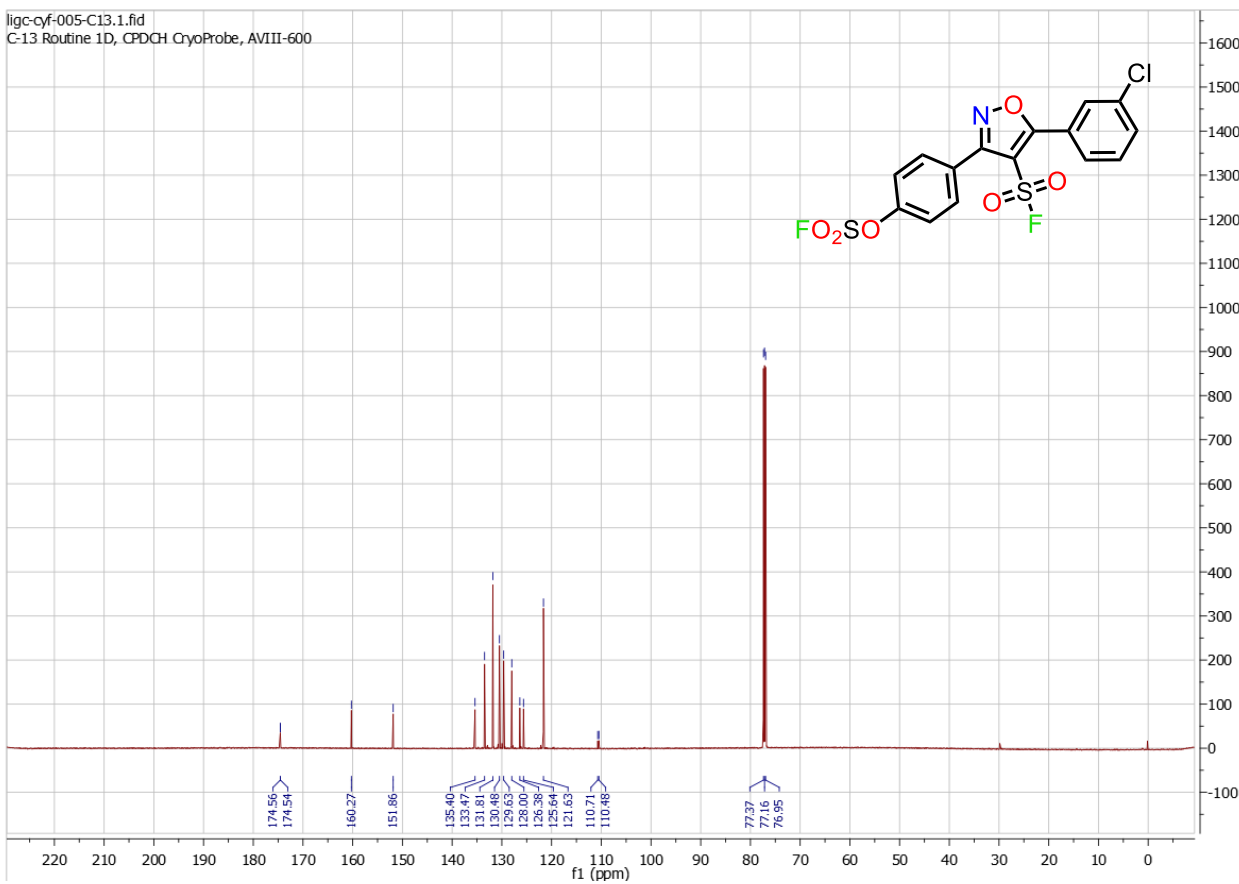
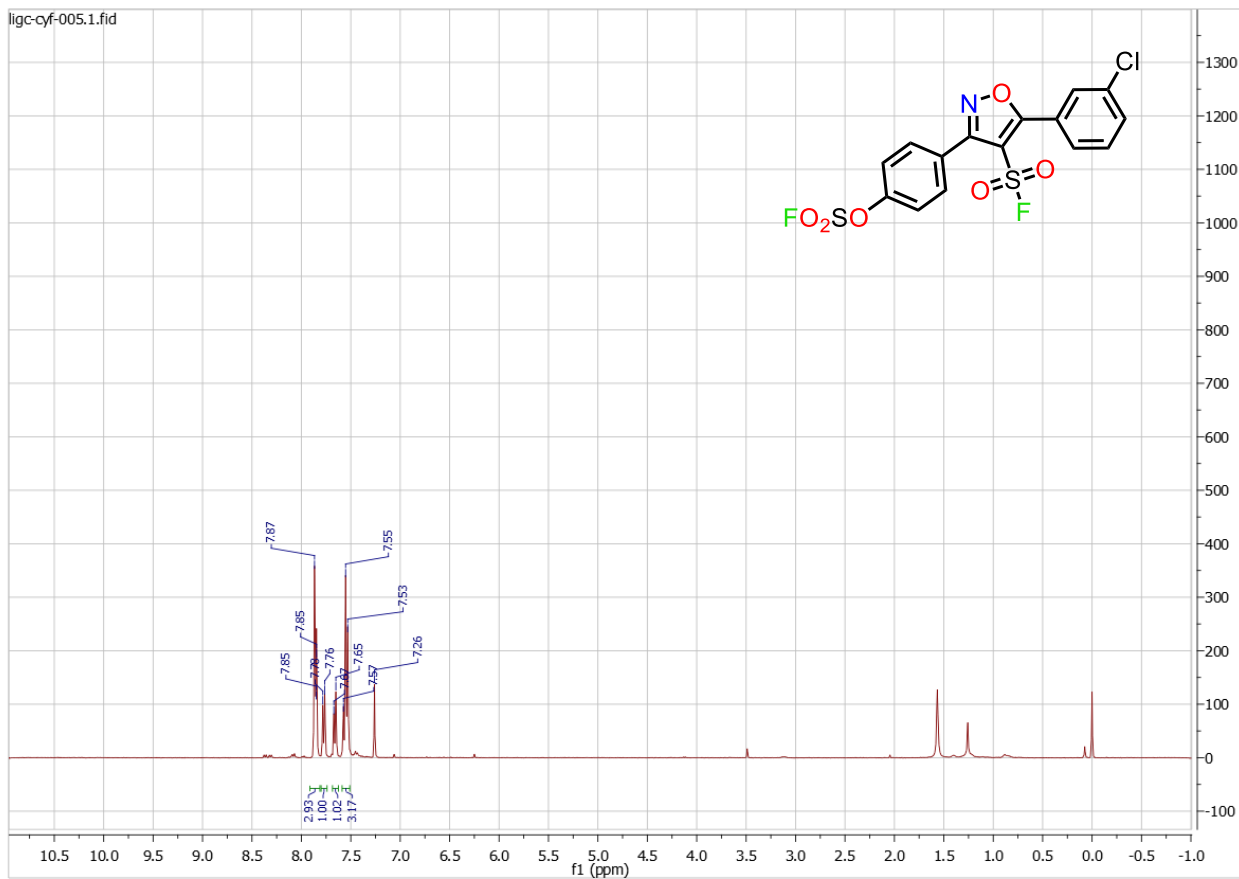
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AV400 BBOF Probe, F-19 in CDCl3, CFCl3=0 ppm.



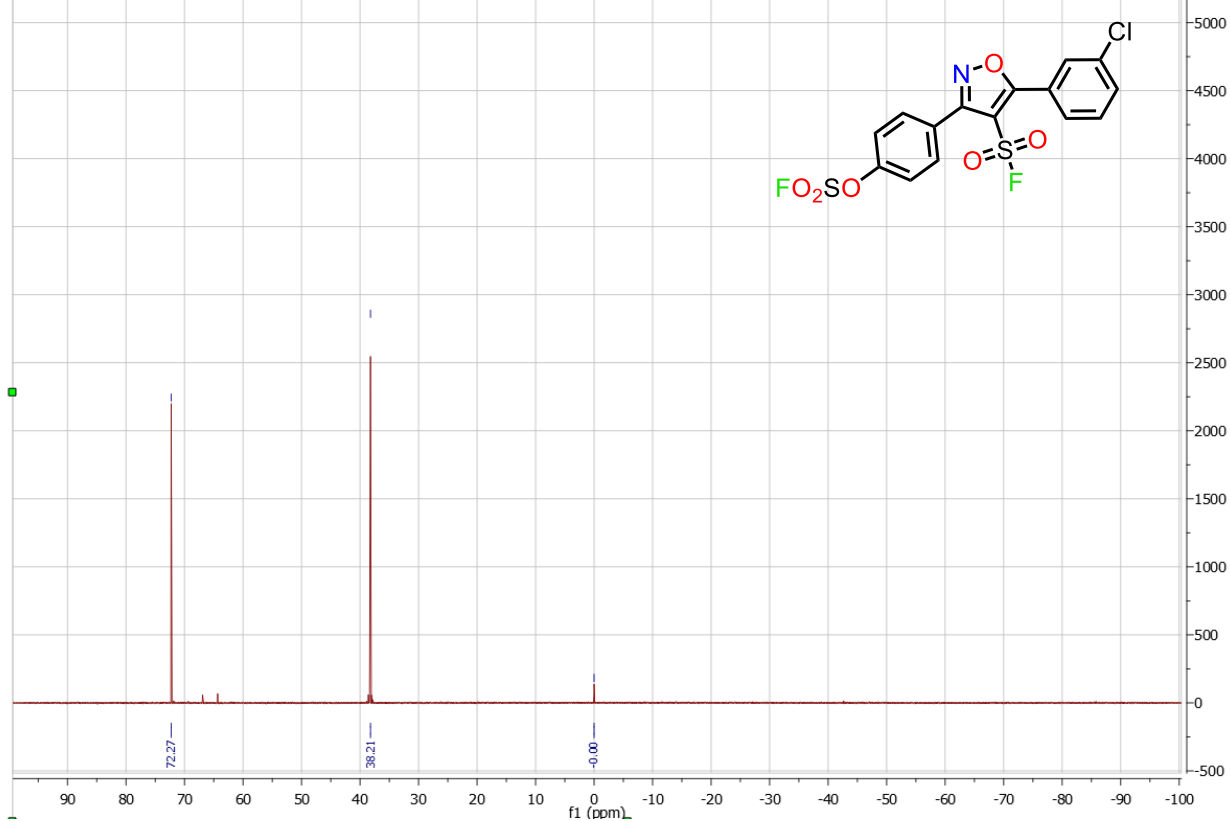


ligc-cyf-009-F.1.fid
F-19, CDCl₃, DPX-400 QNP Probe. CF₃Cl as Ref at 0 ppm.

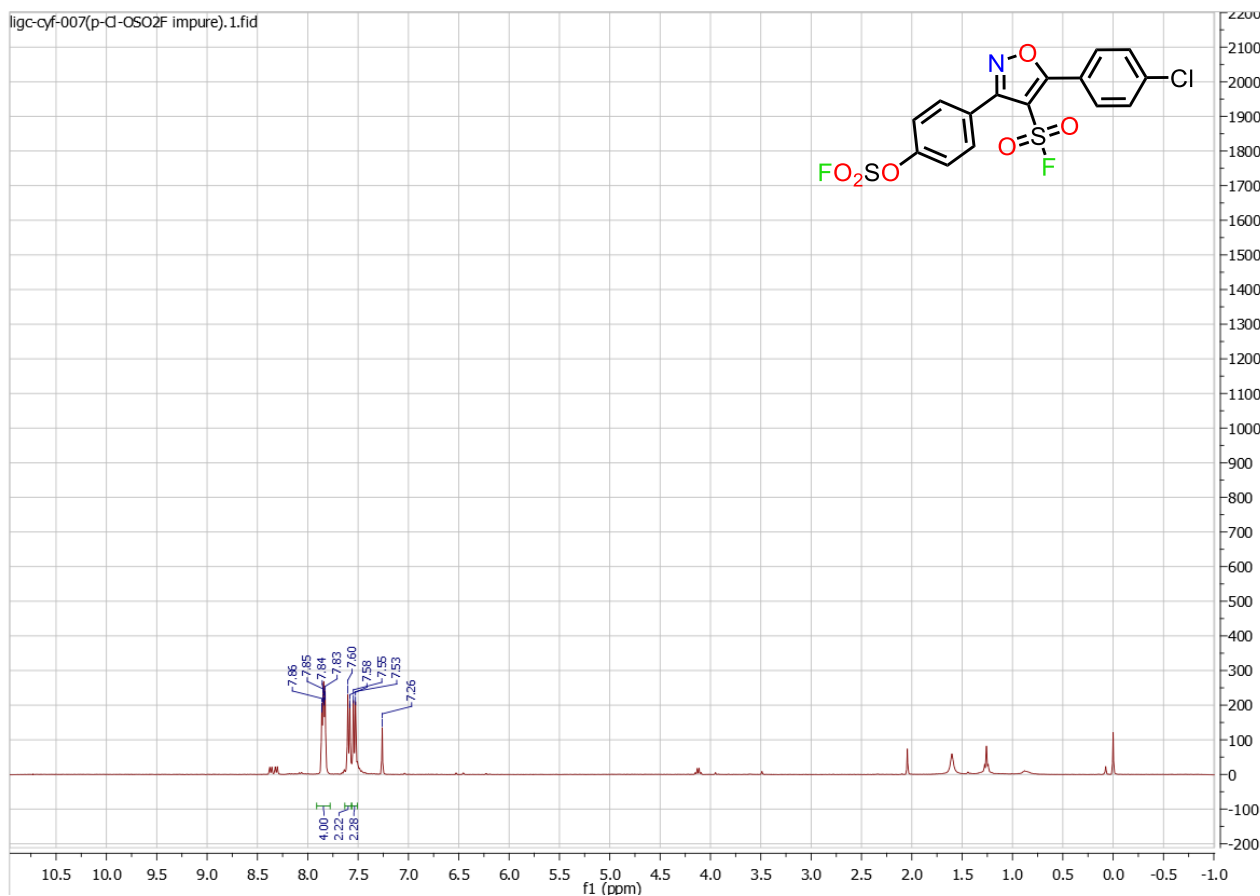




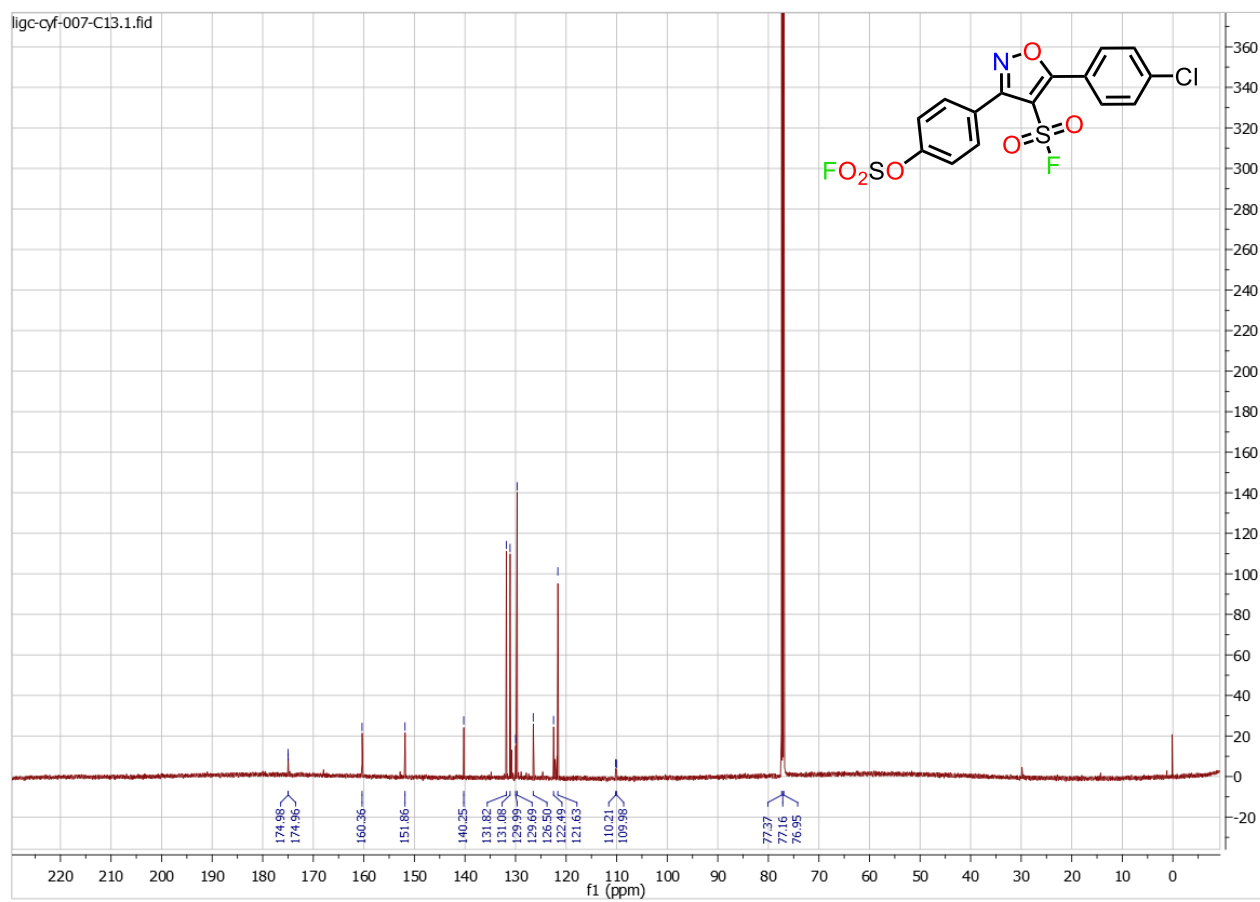
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F-19, CDCl3, DPX-400 QNP Probe. CF3Cl as Ref at 0 ppm.



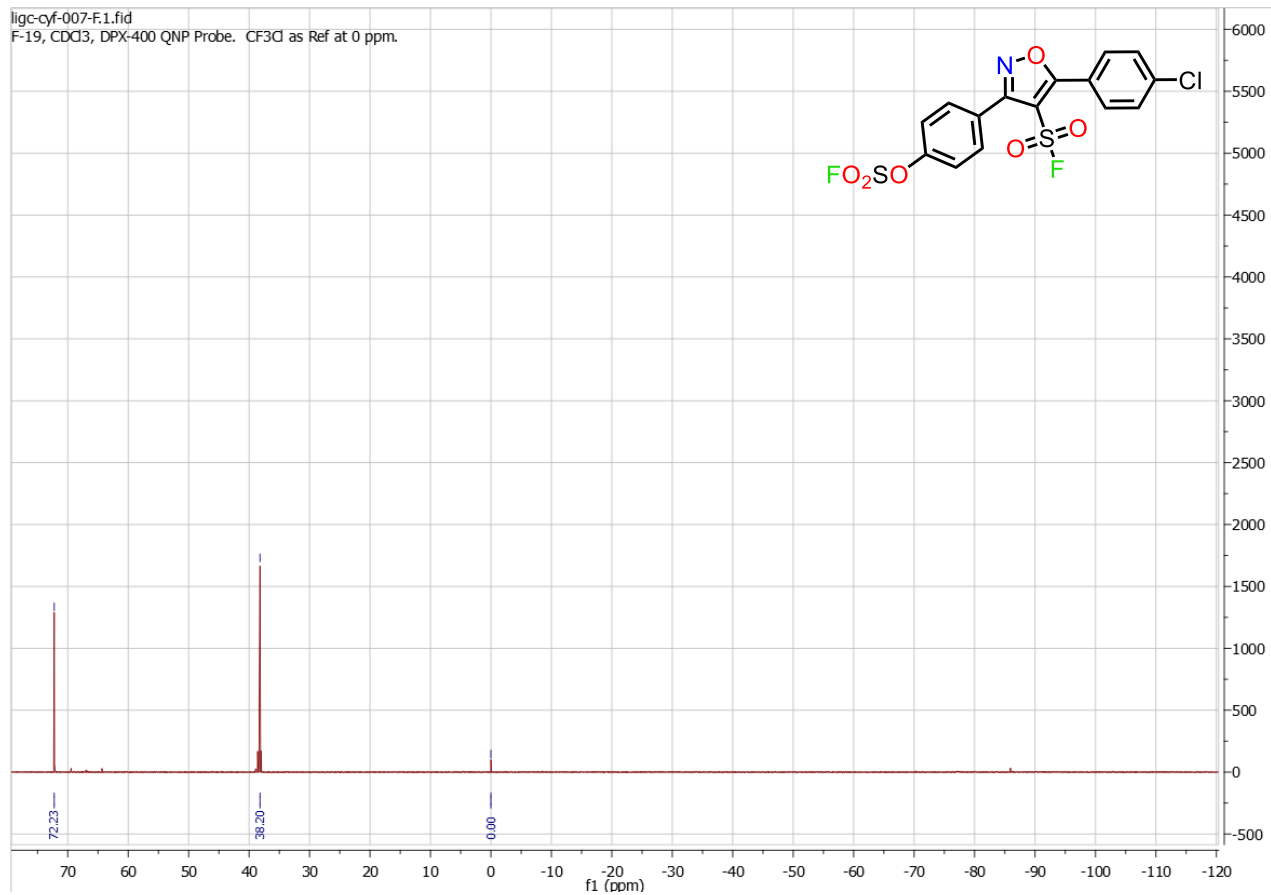
ligc-cyf-007(p-Cl-OSO2F impure), 1.fid



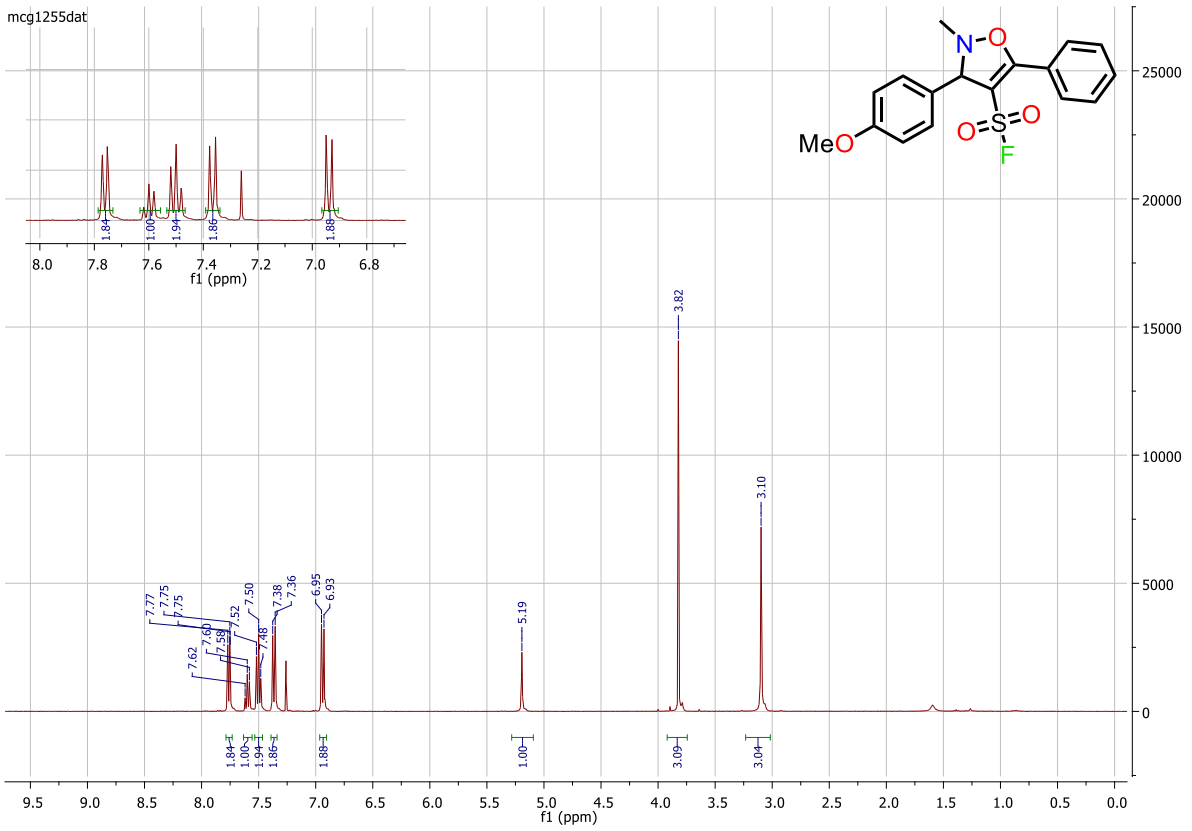
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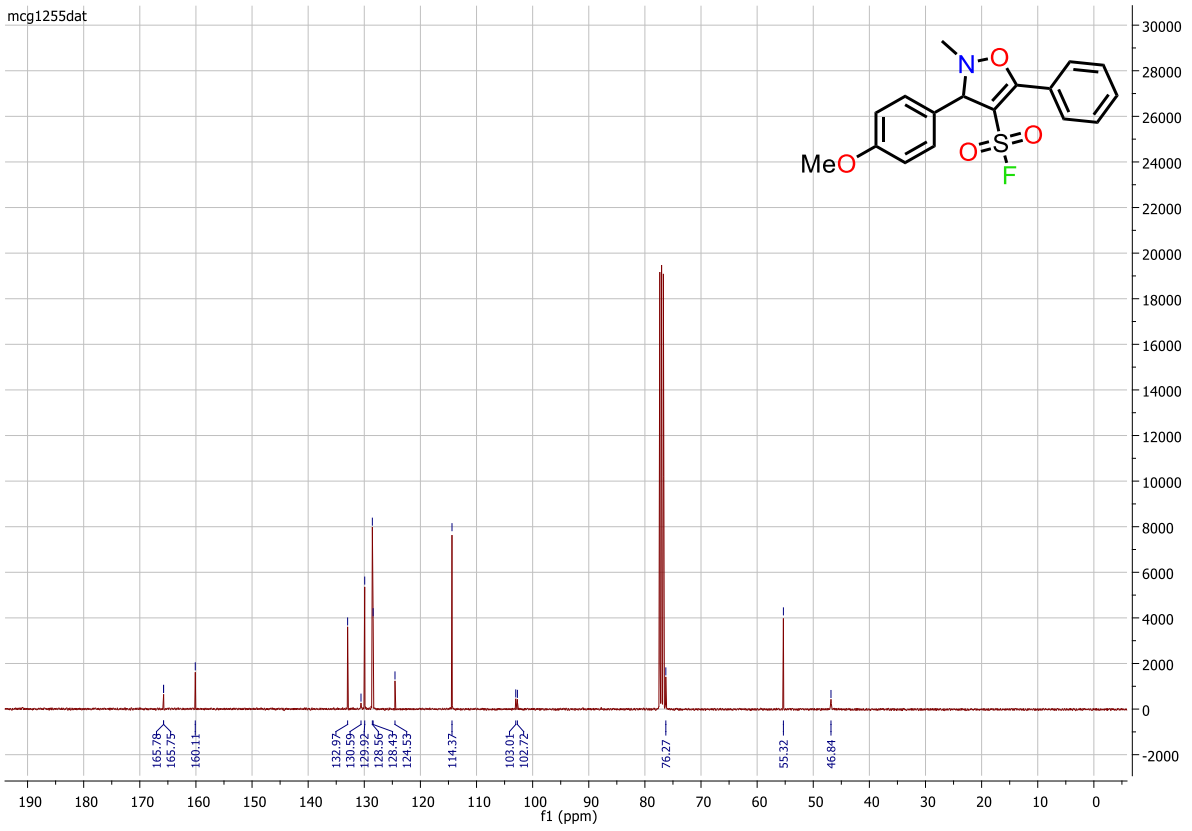
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F-19, CDCl3, DPX-400 QNP Probe. CF3Cl as Ref at 0 ppm.



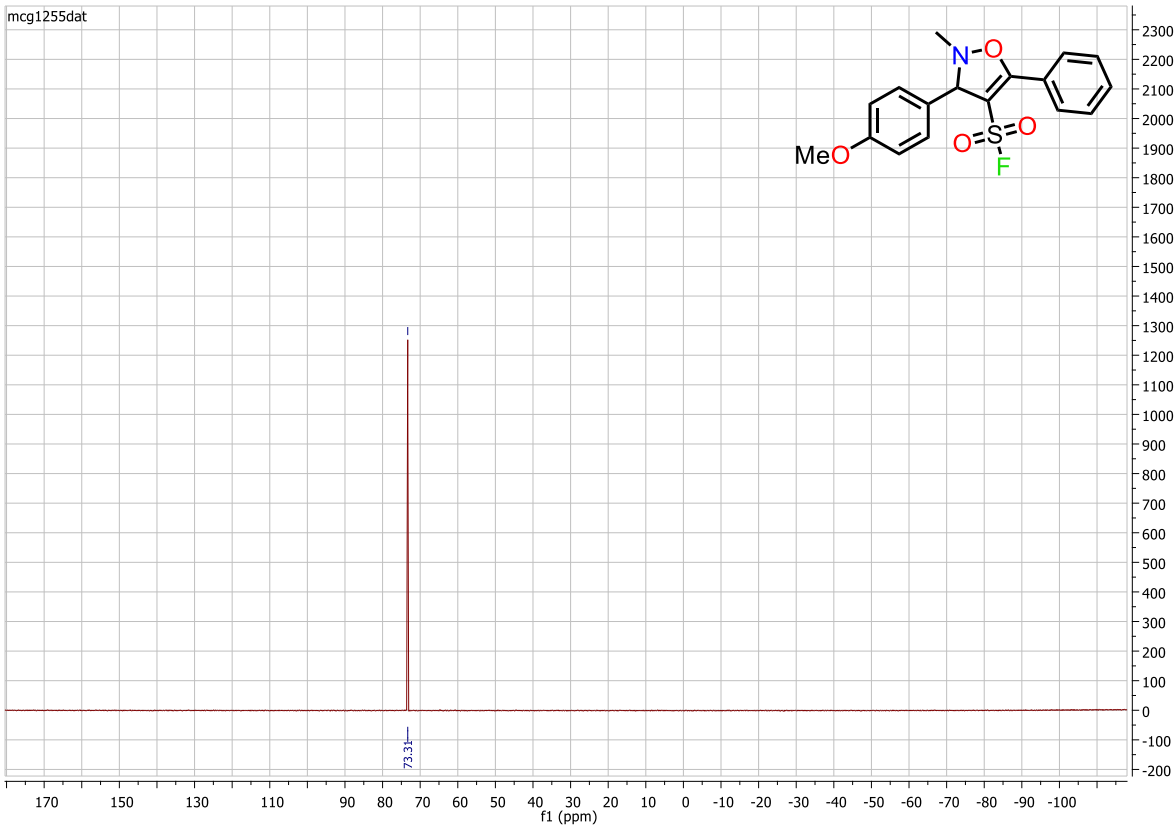
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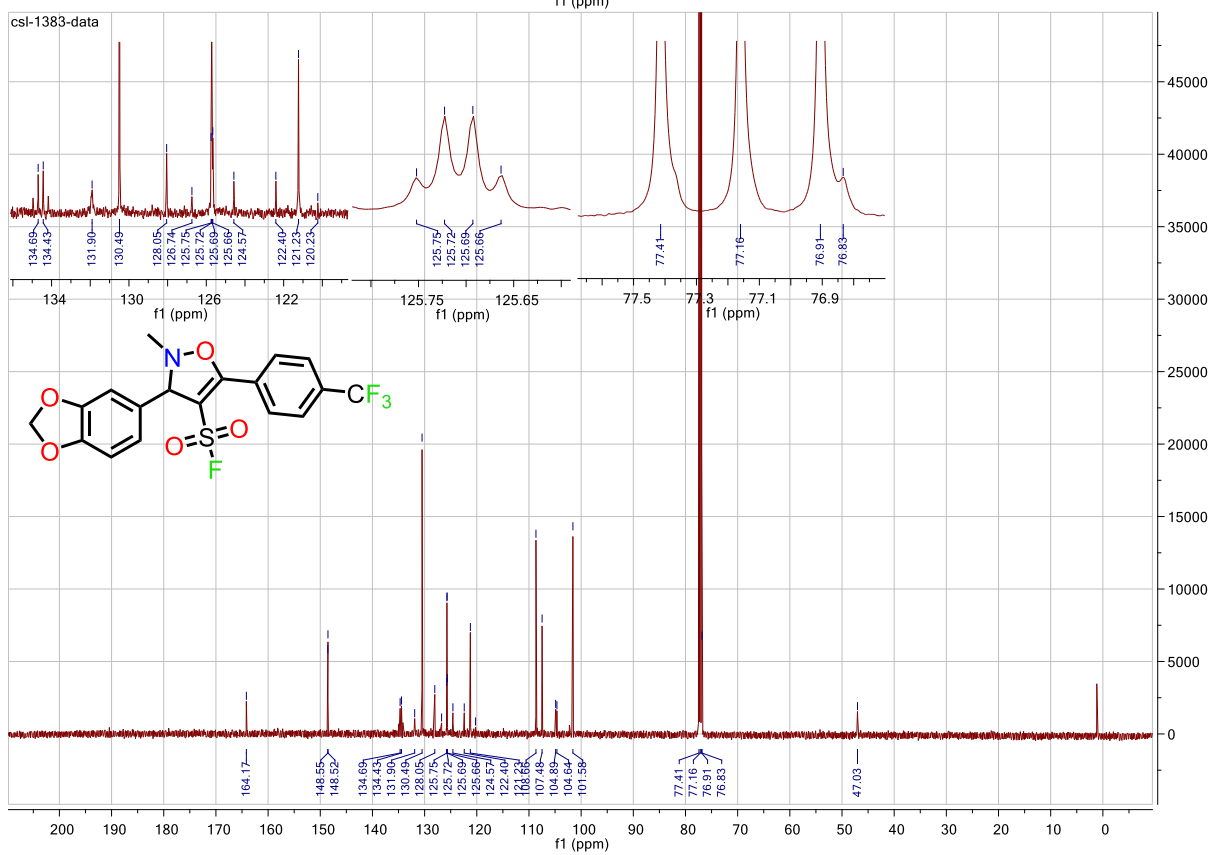
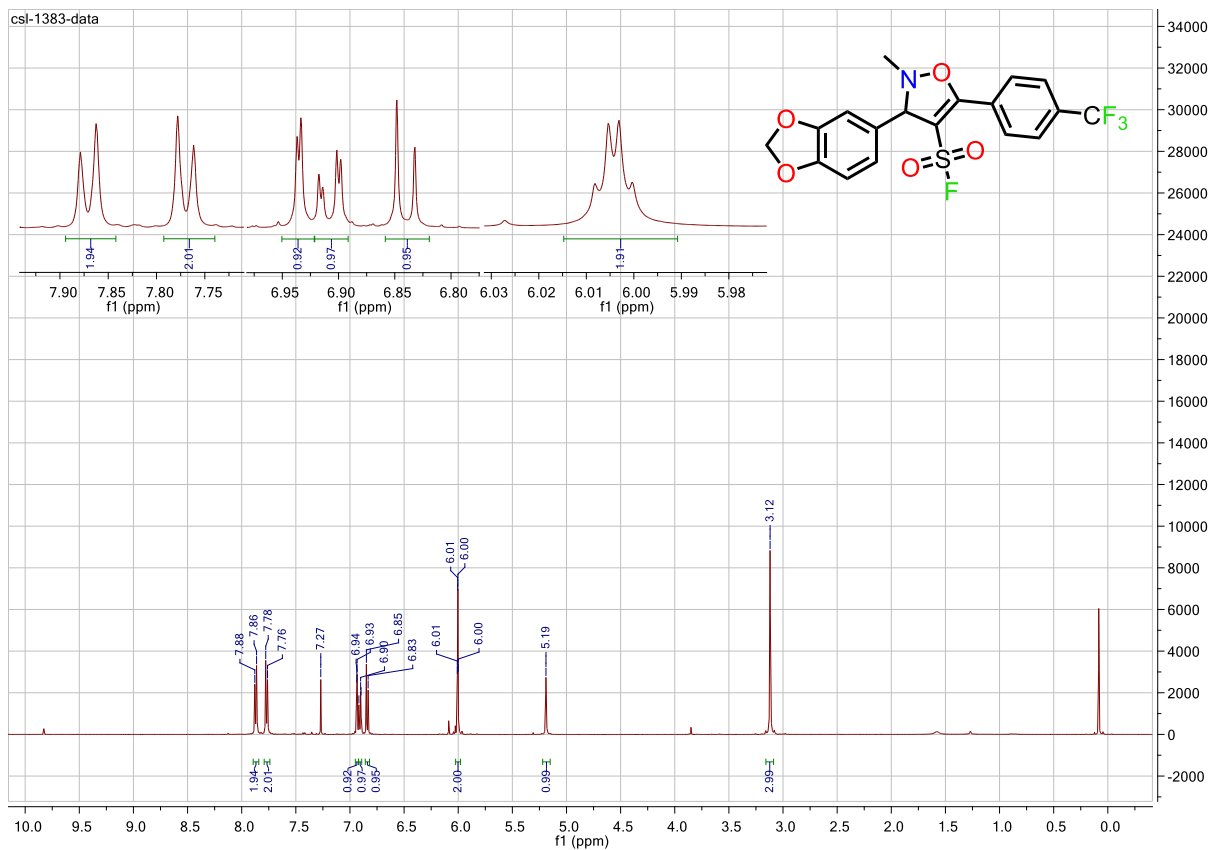


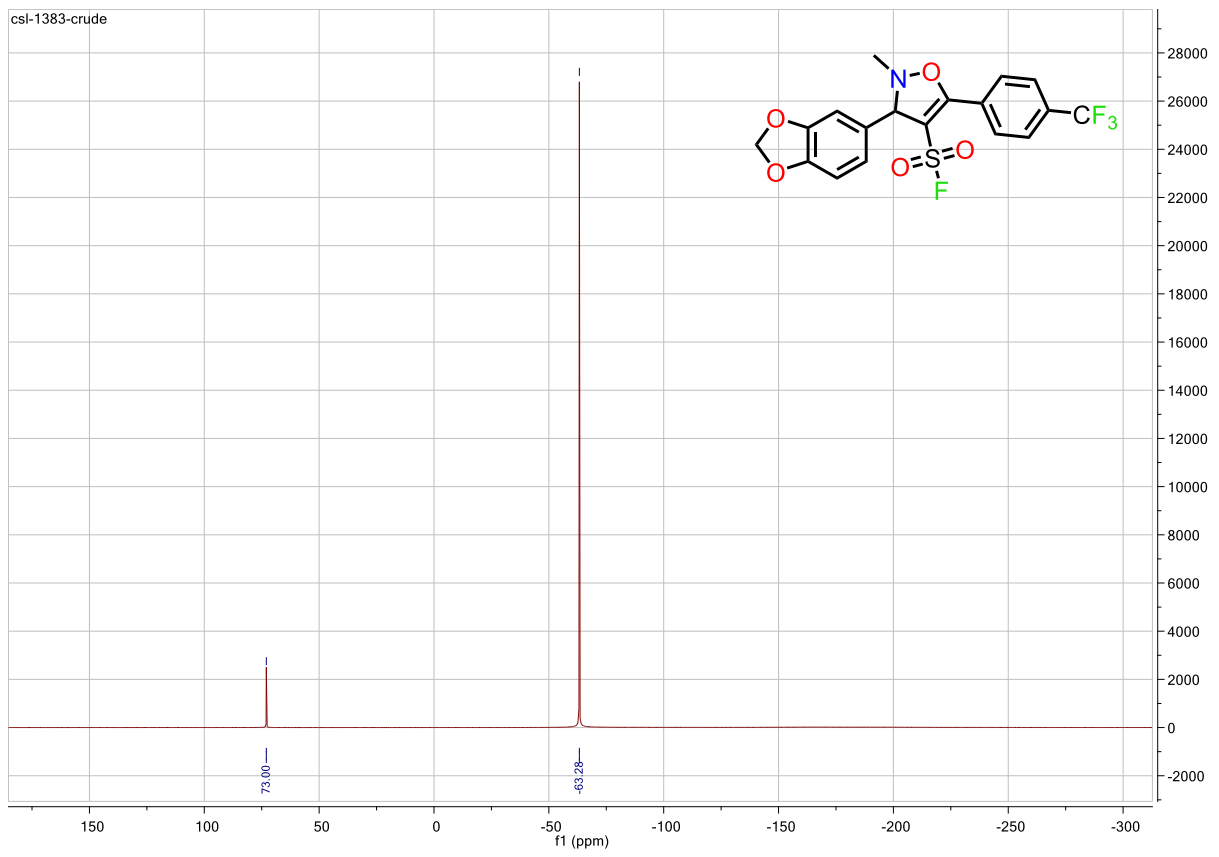
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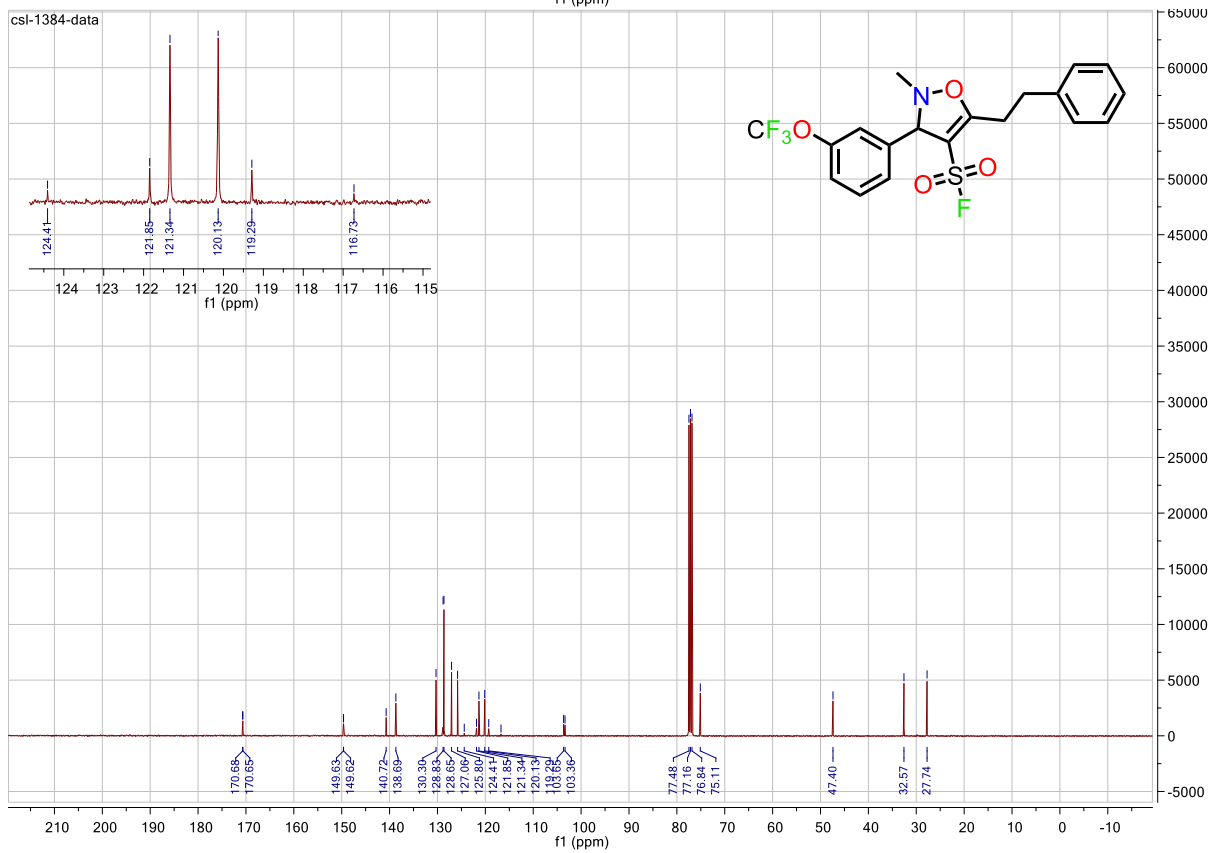
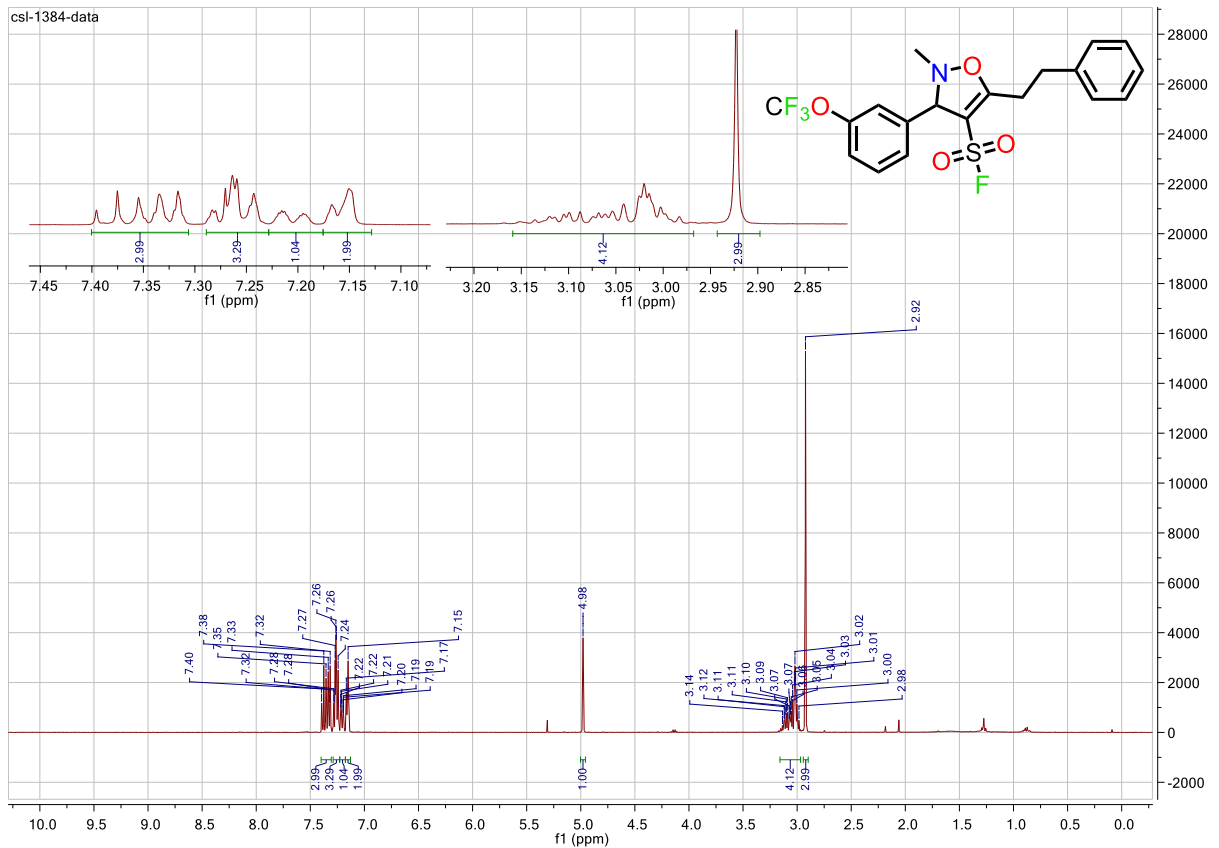


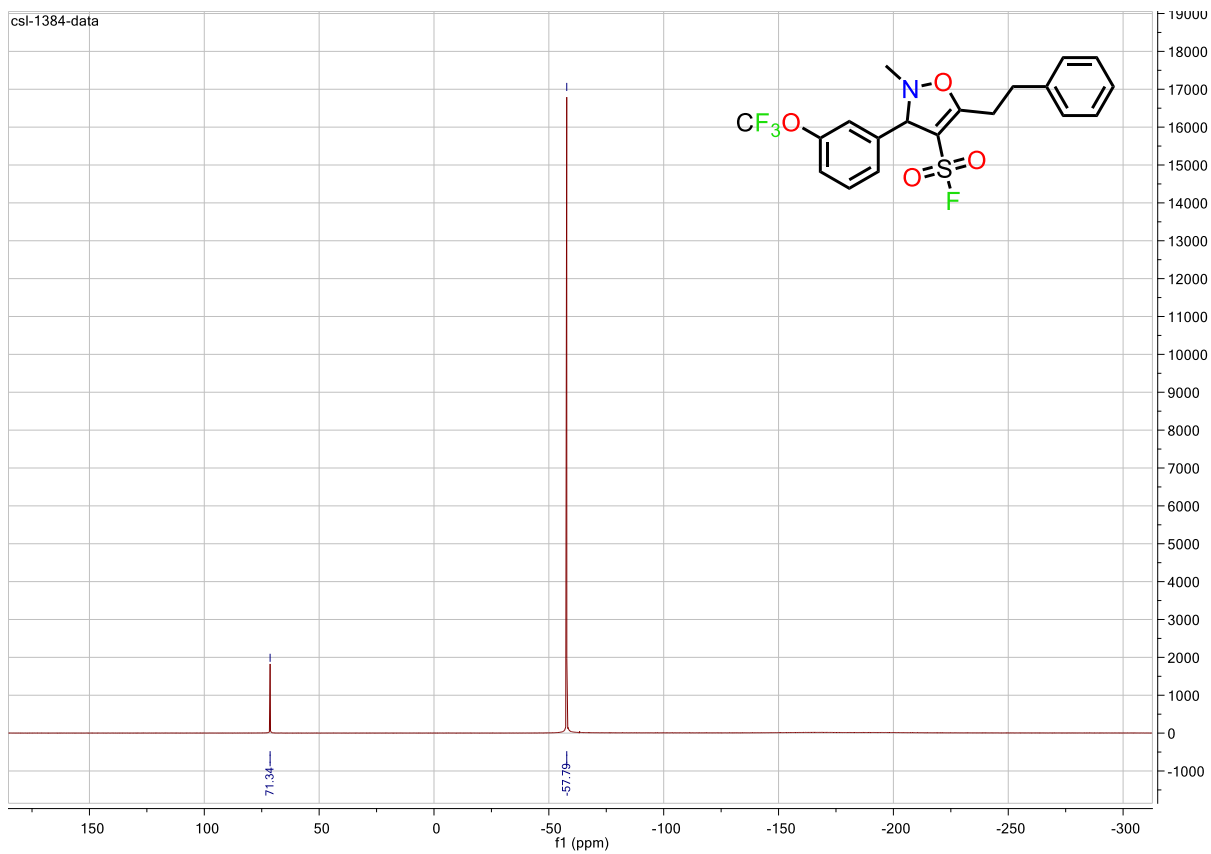
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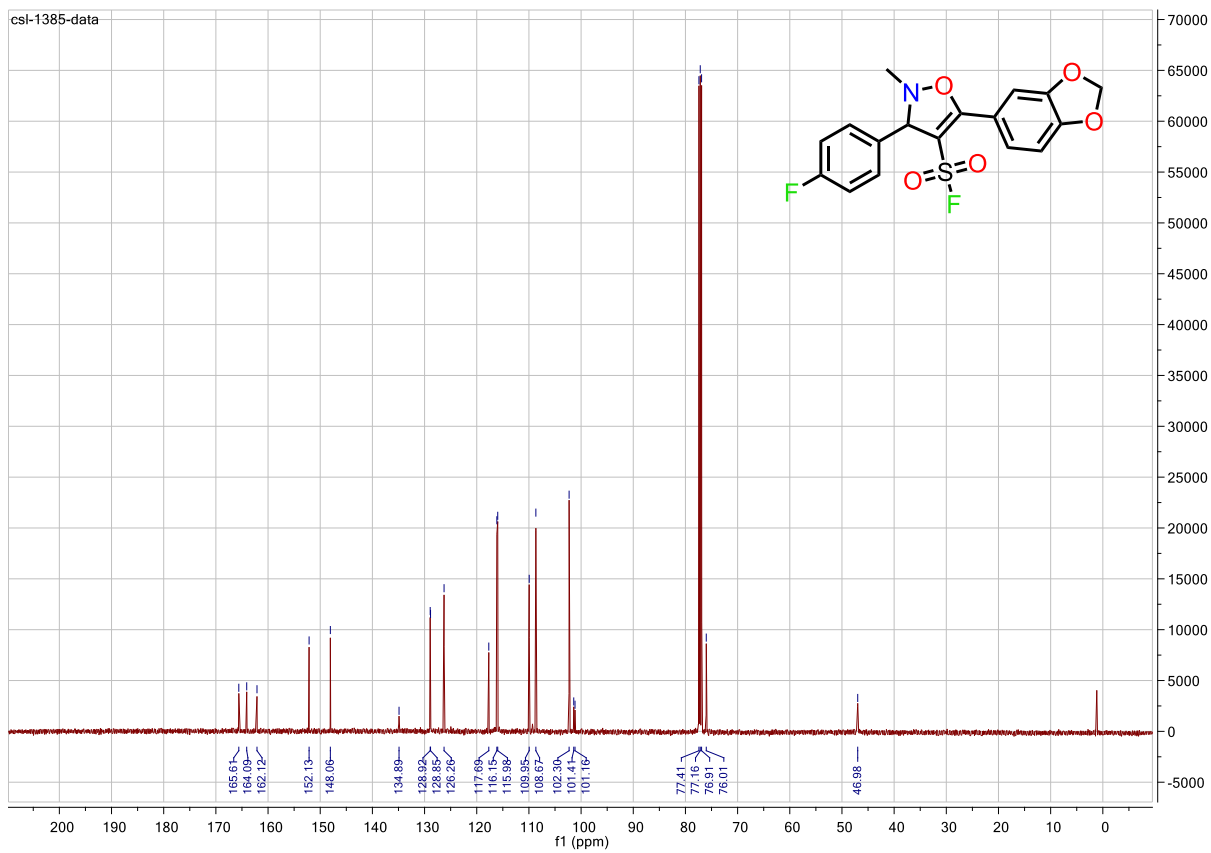
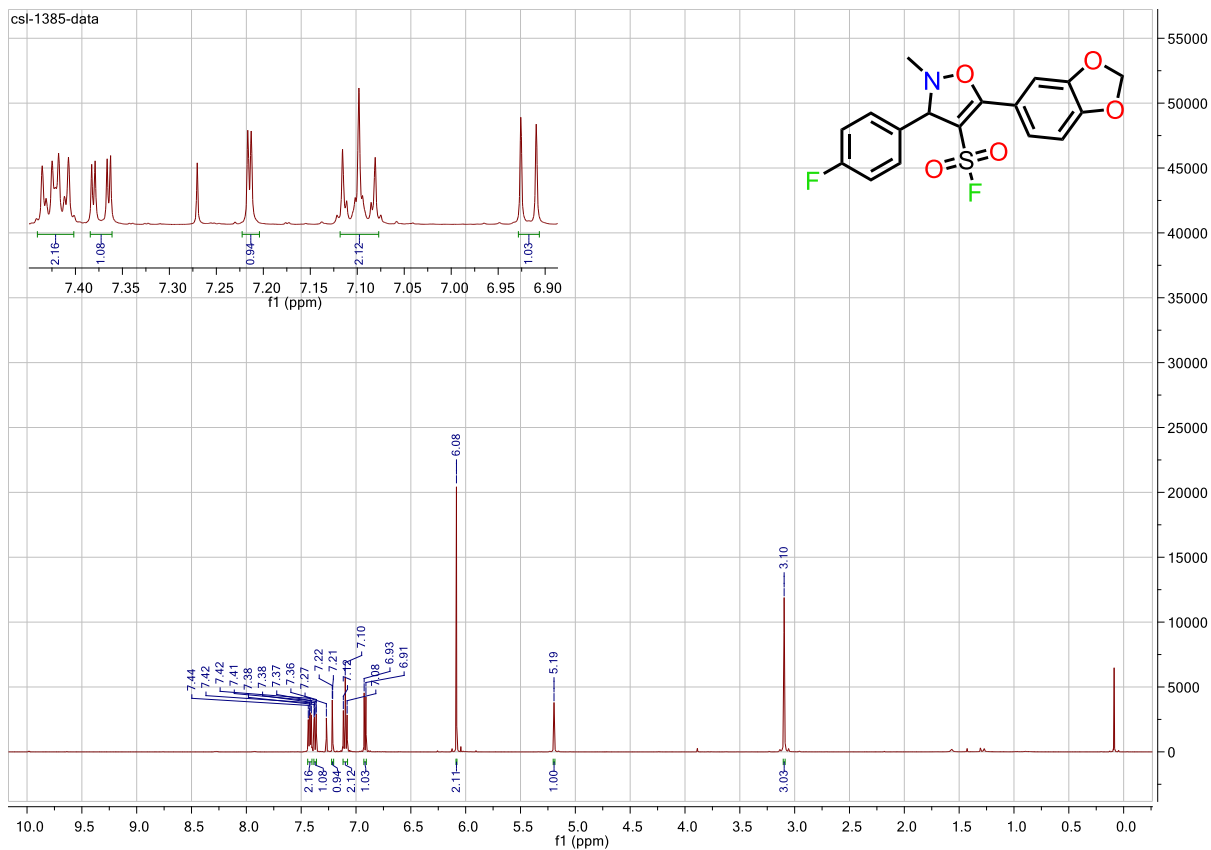


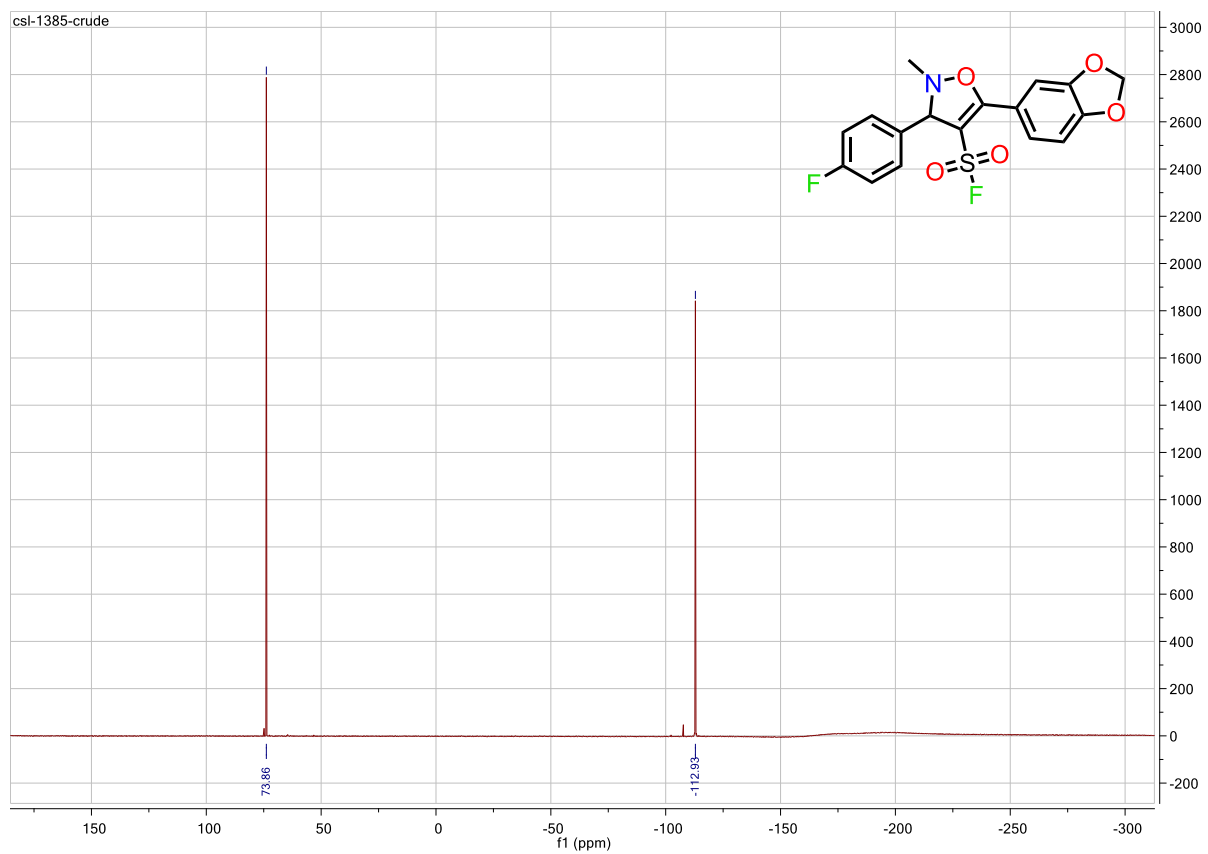




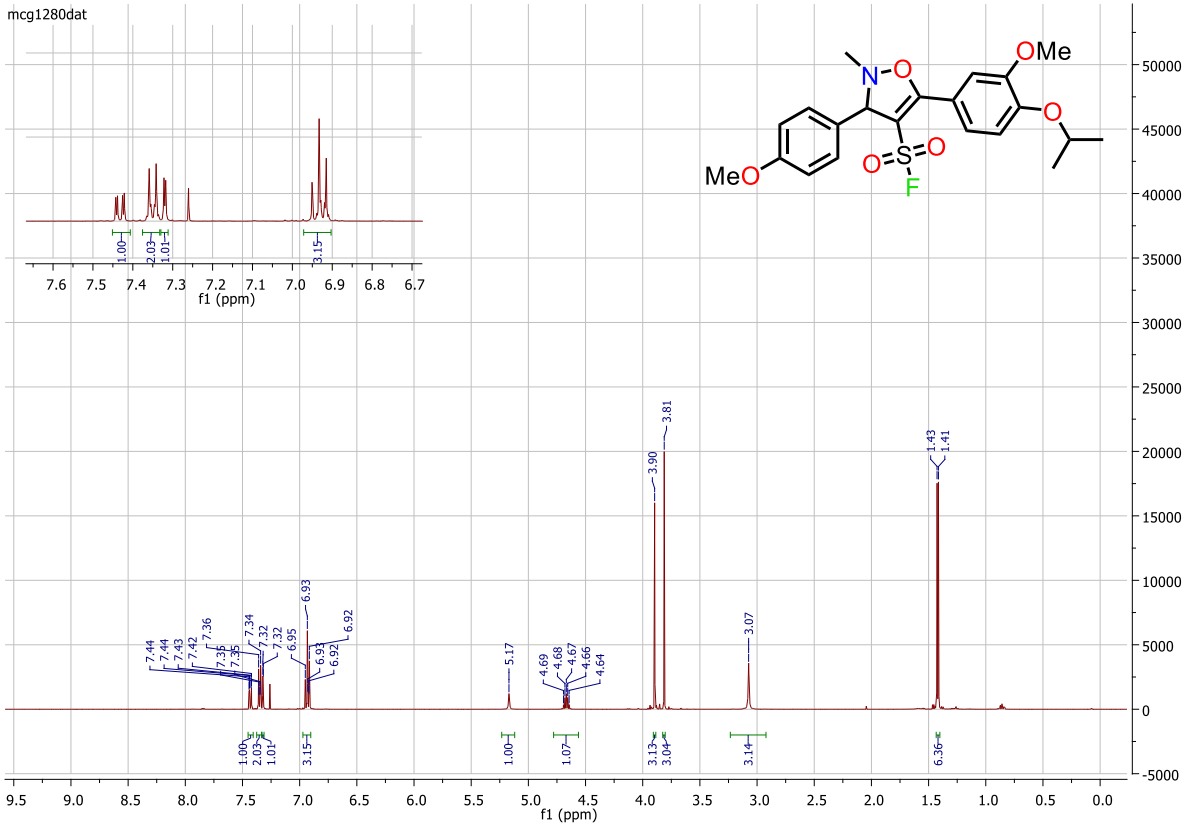




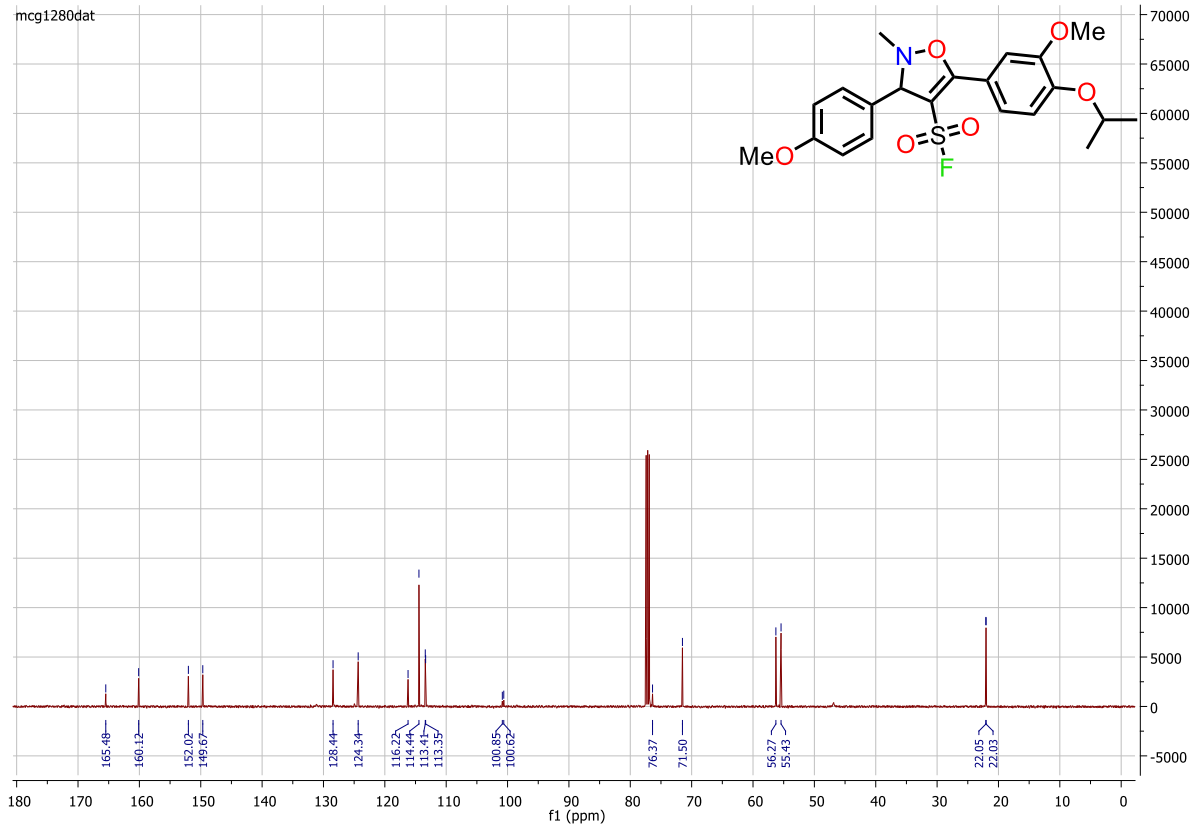




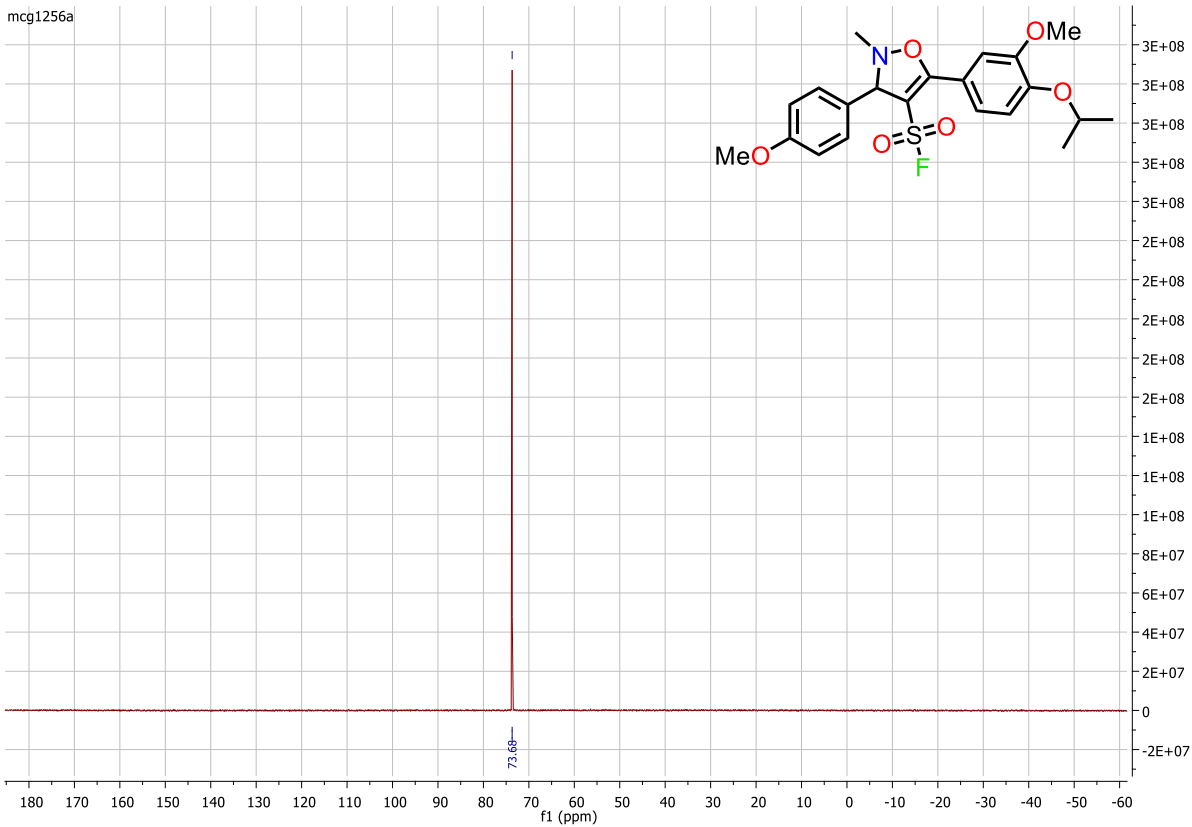
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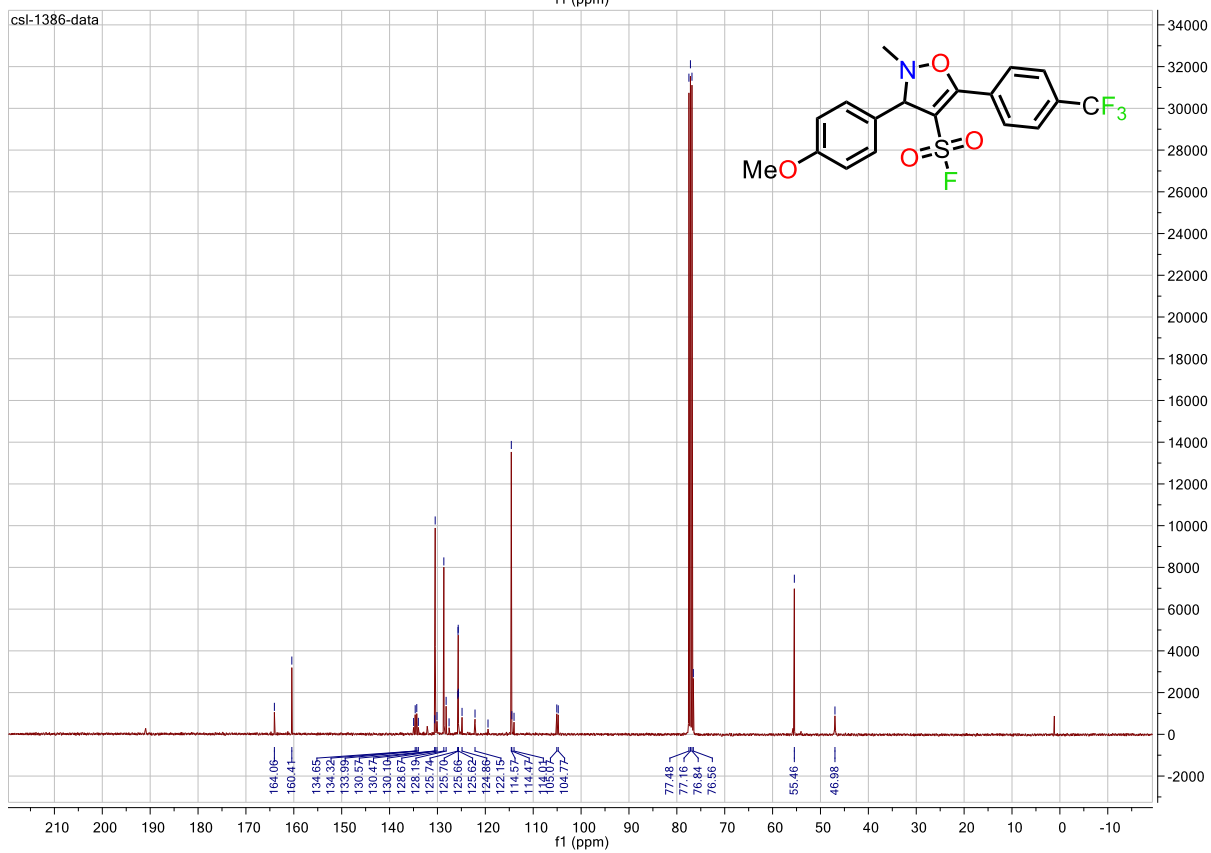
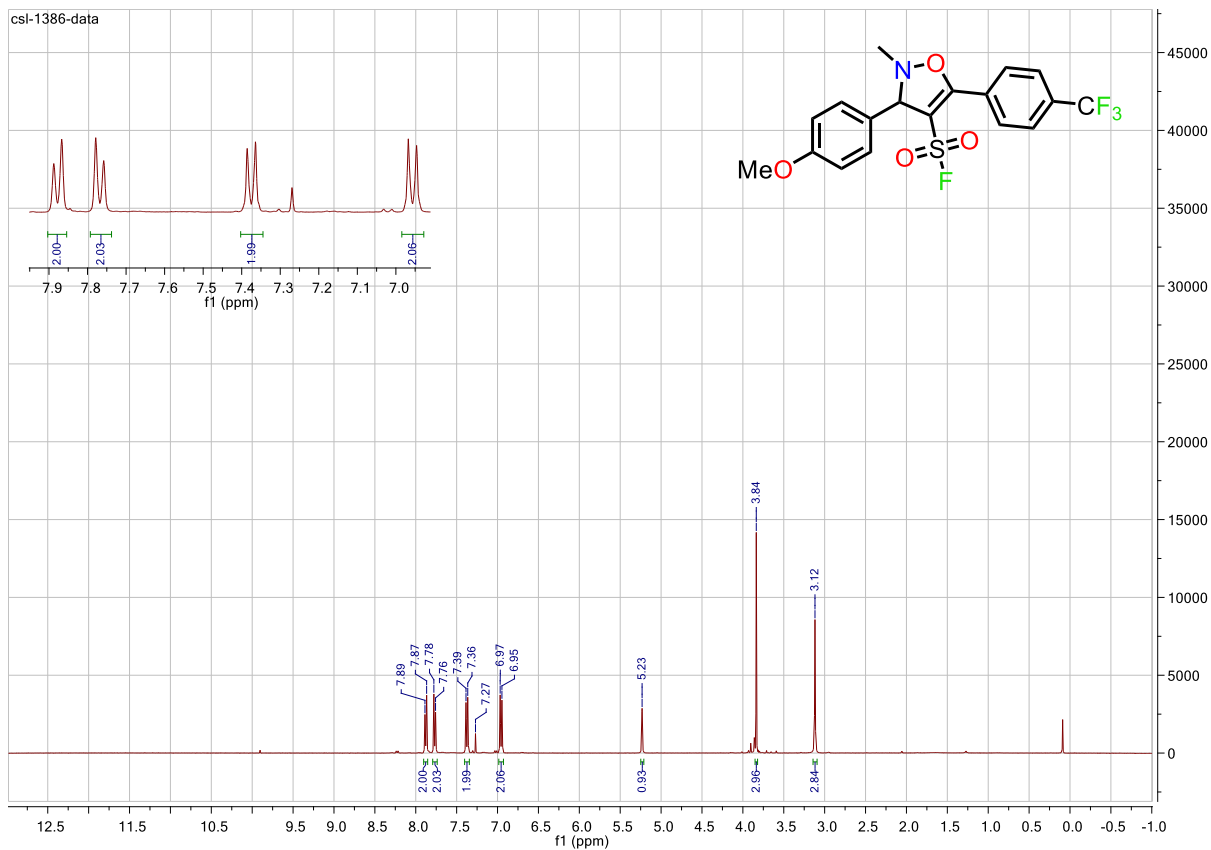


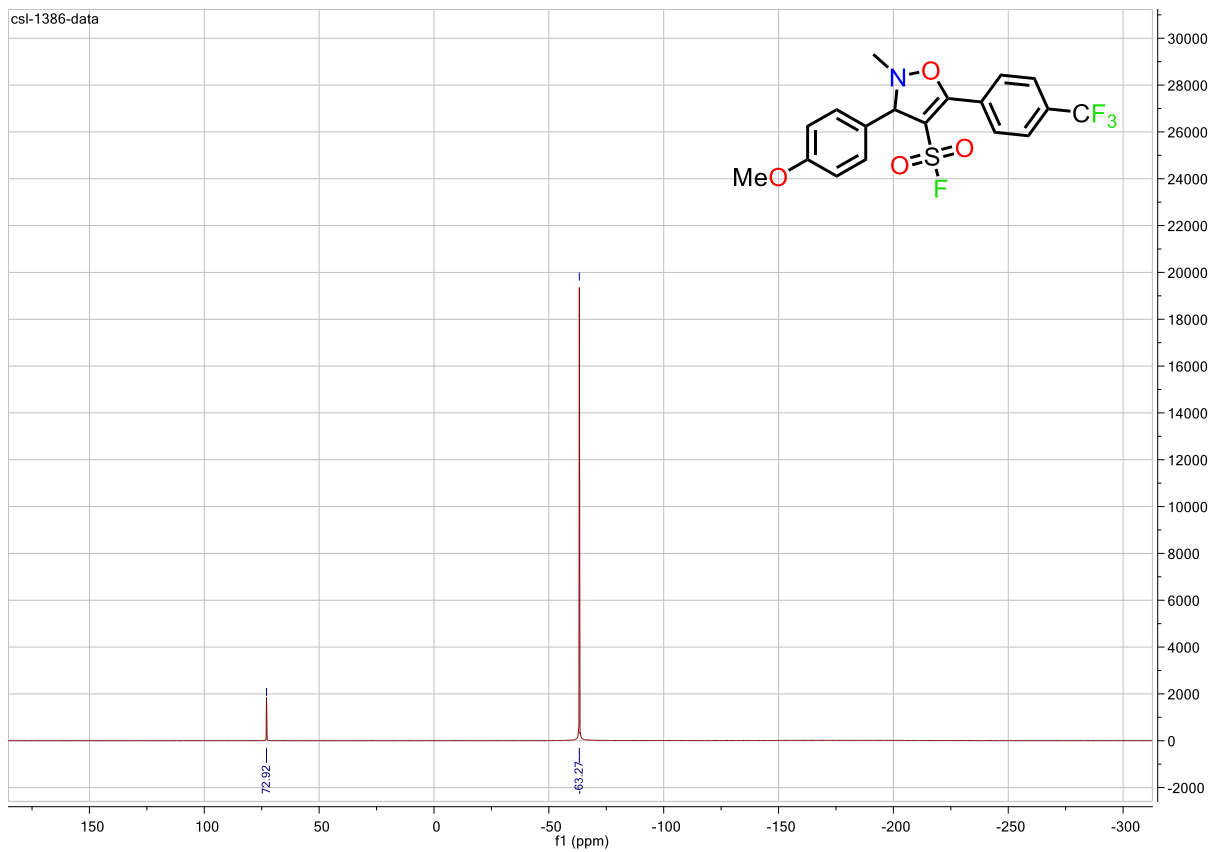
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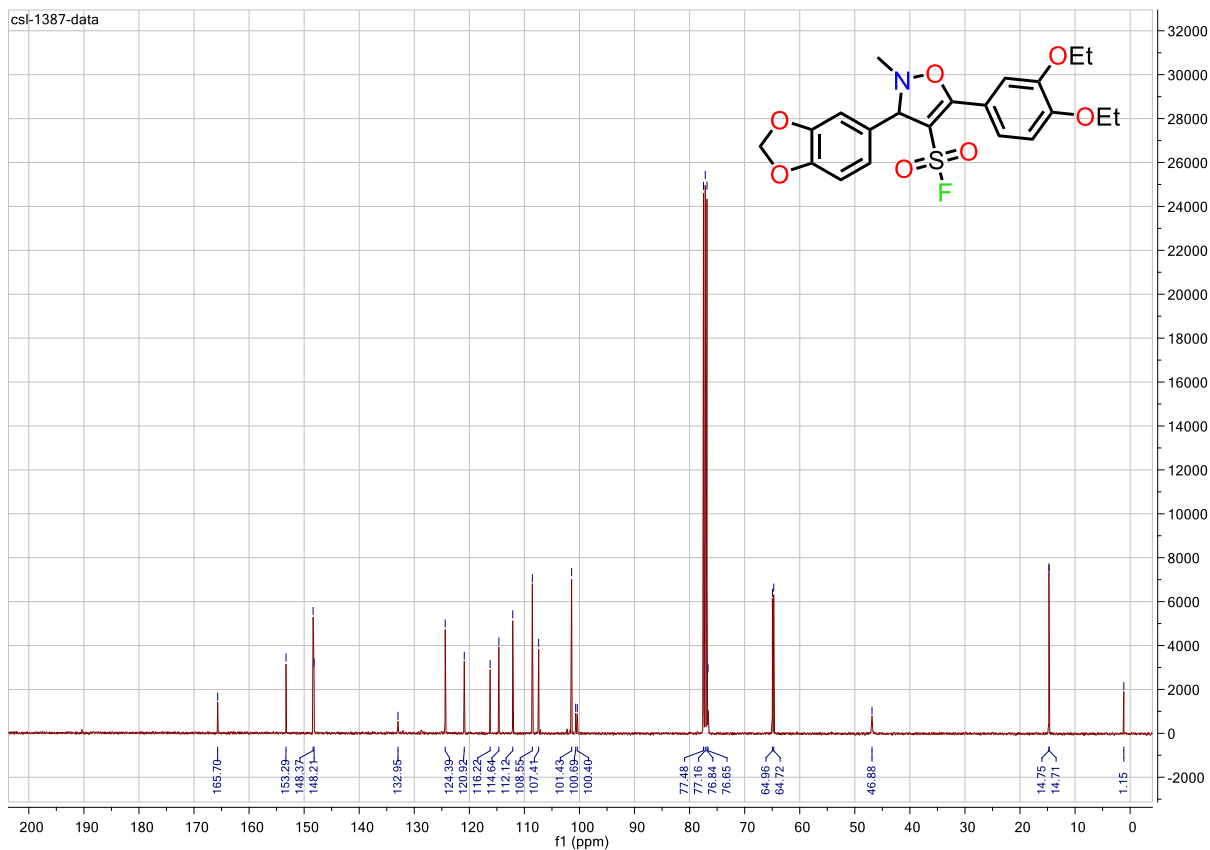
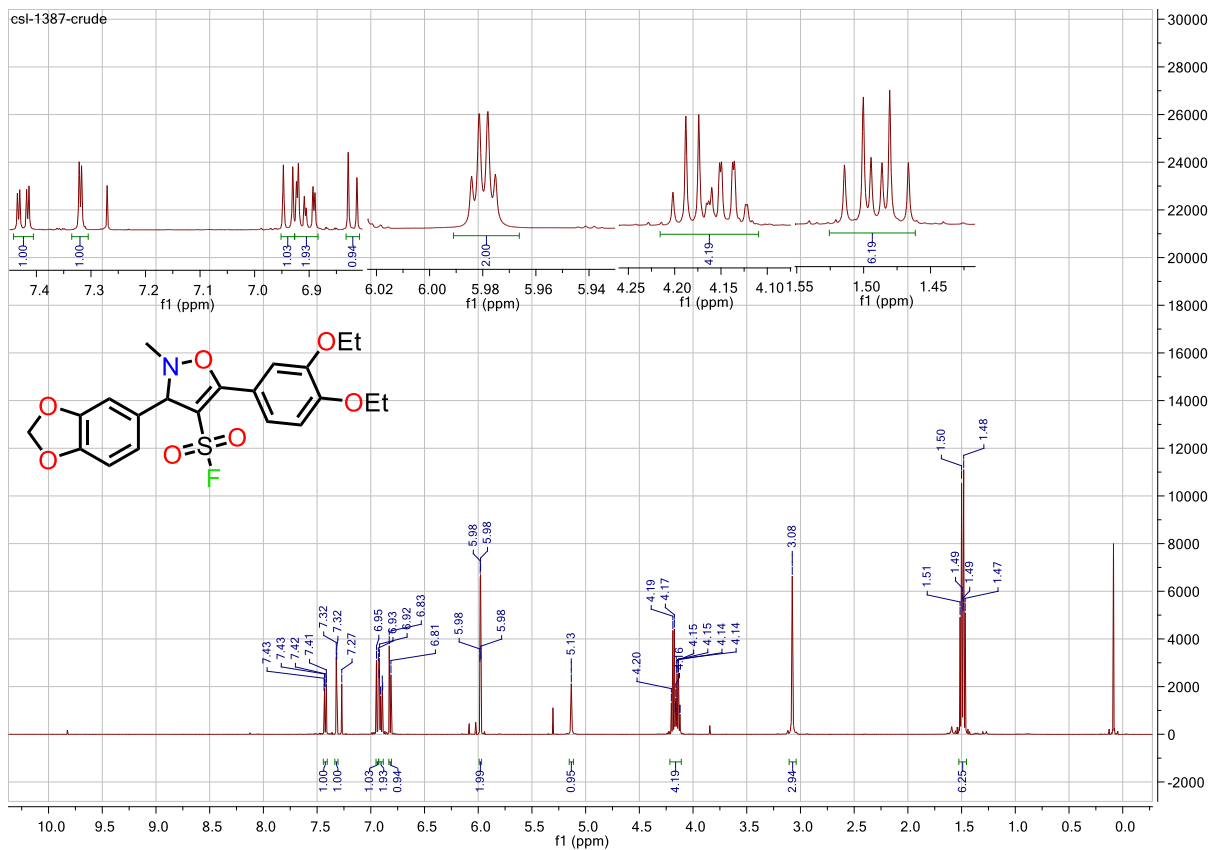


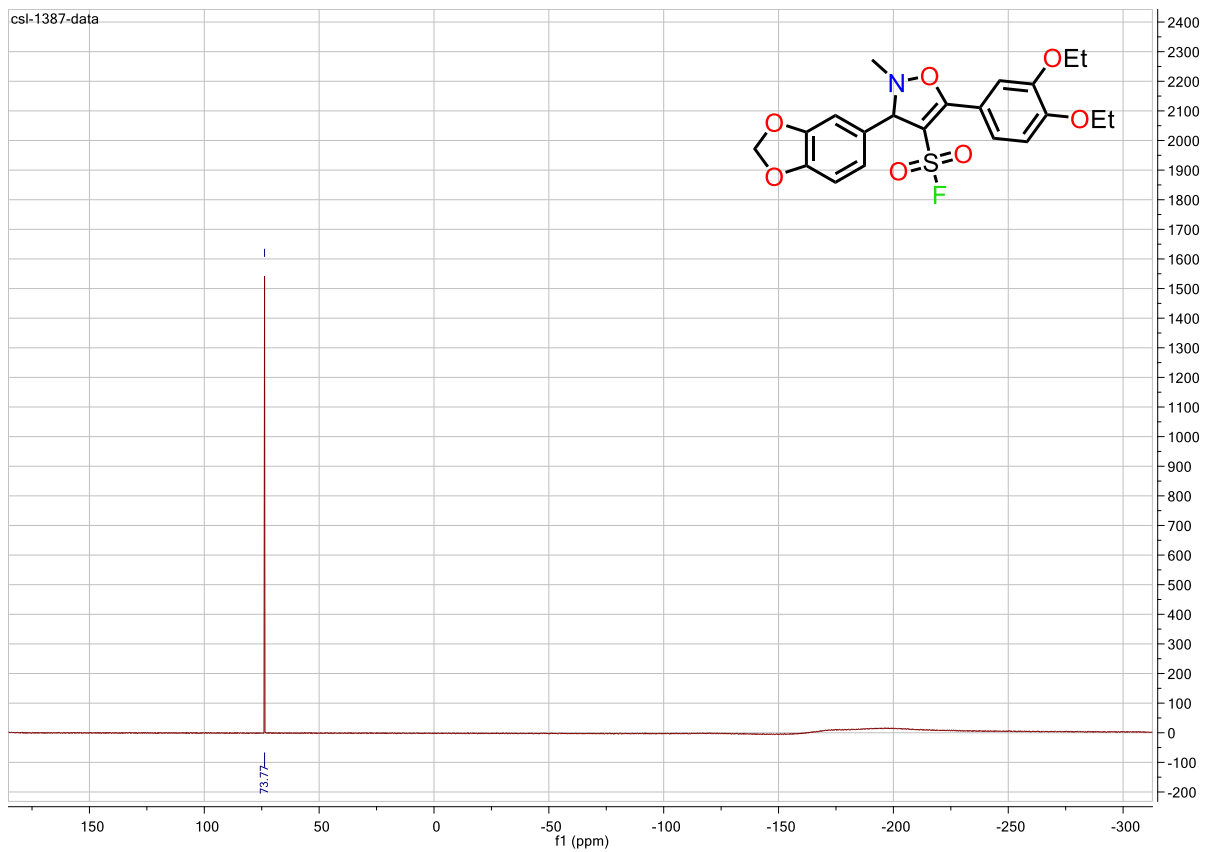
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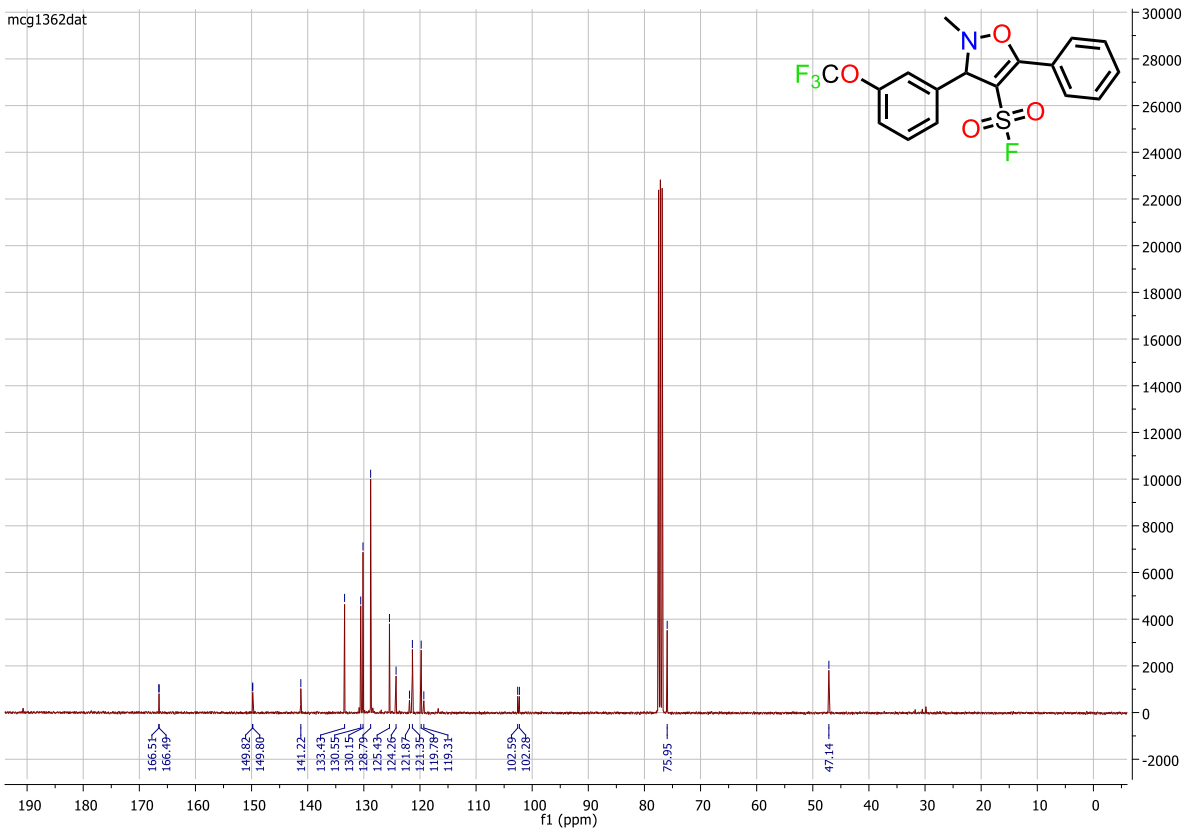
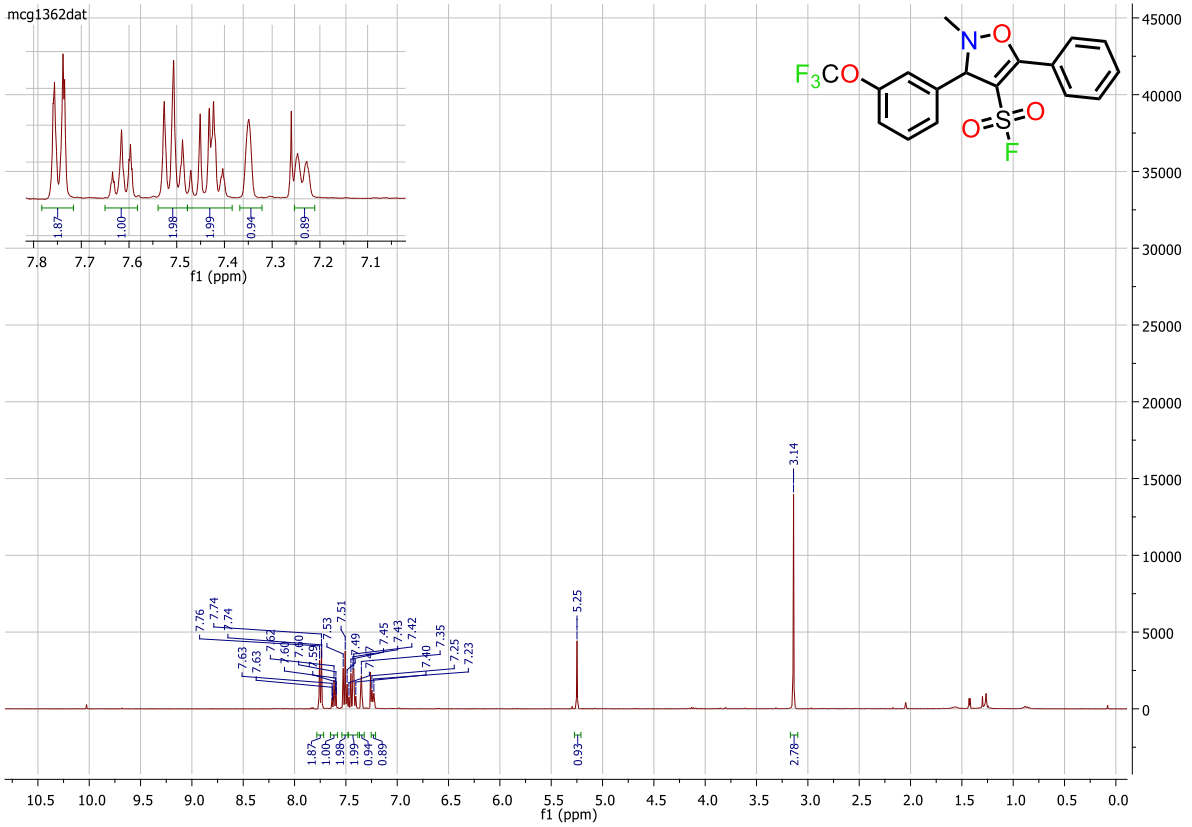




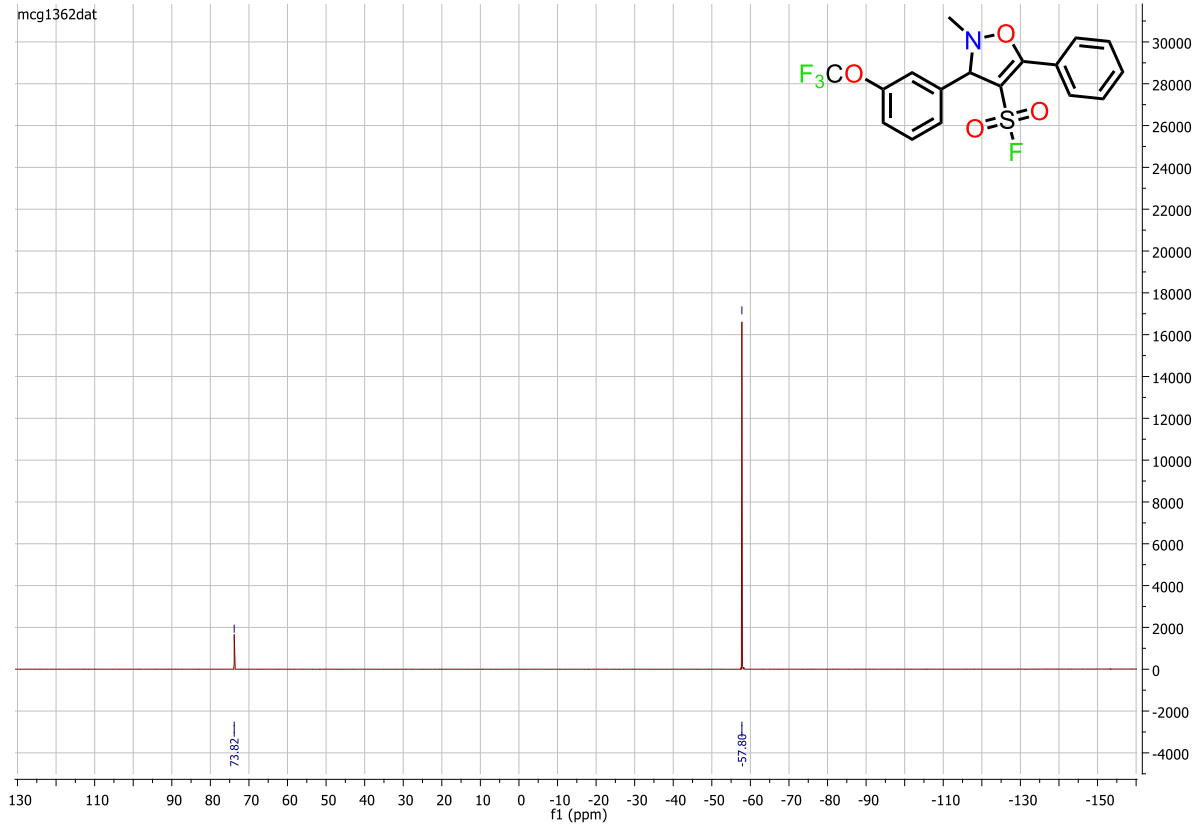




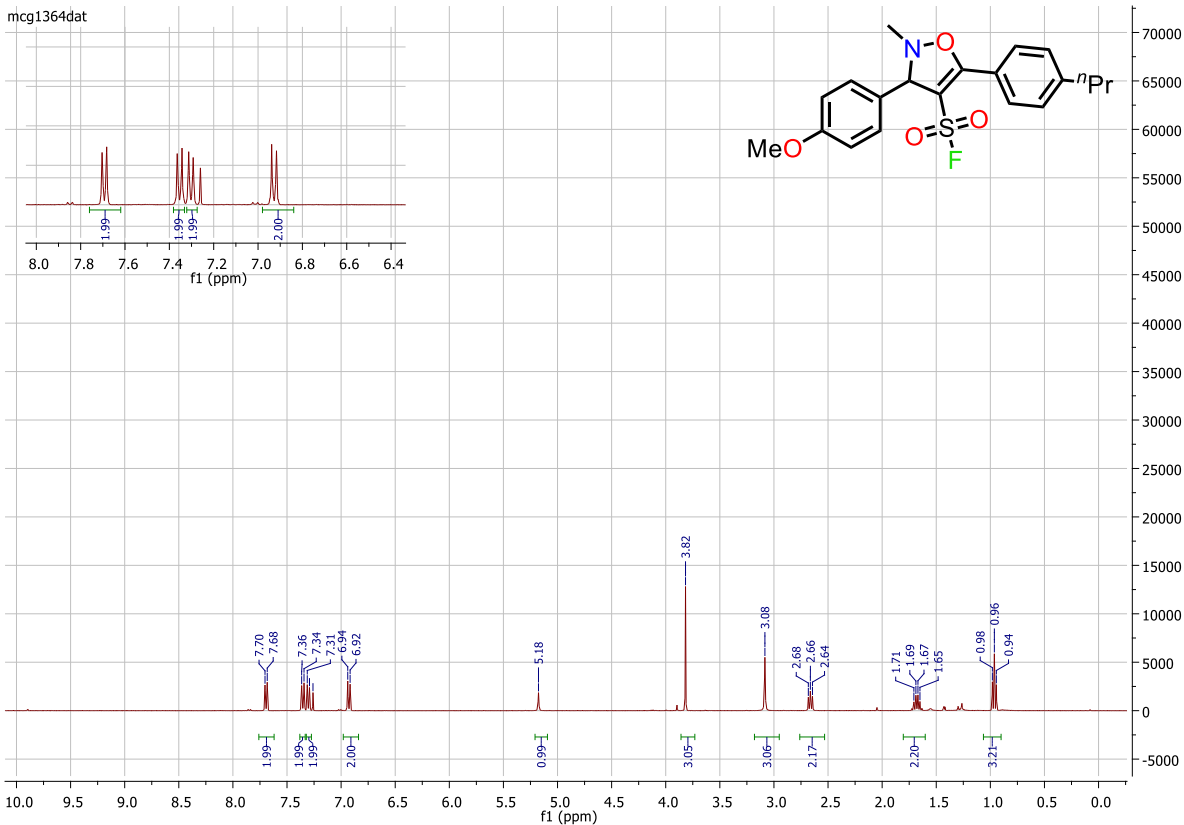




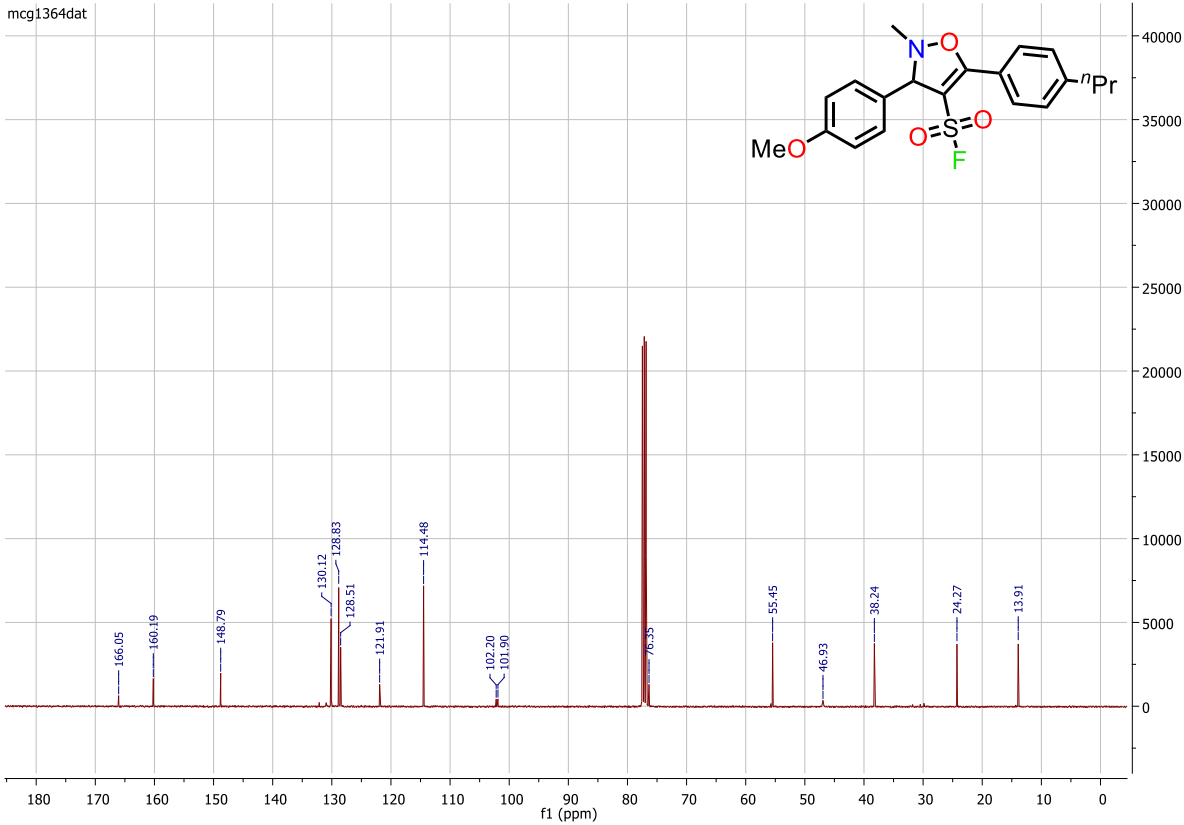
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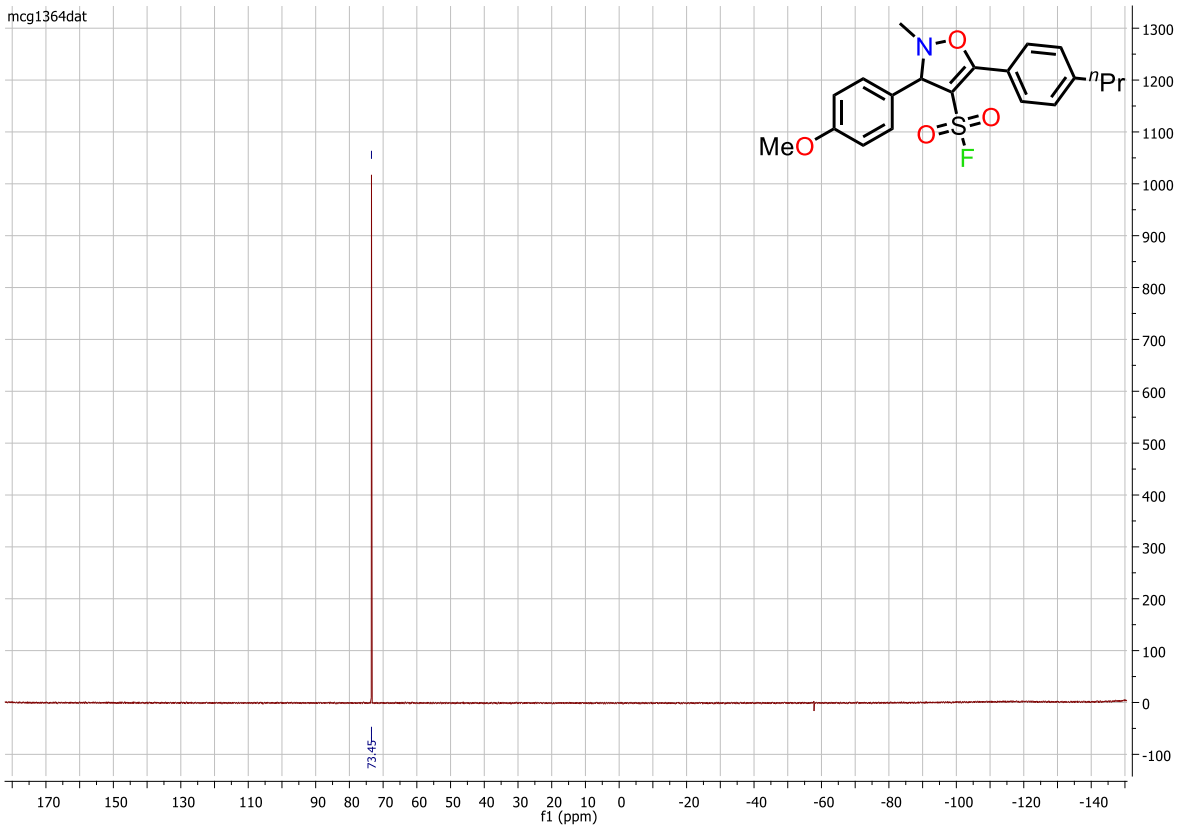
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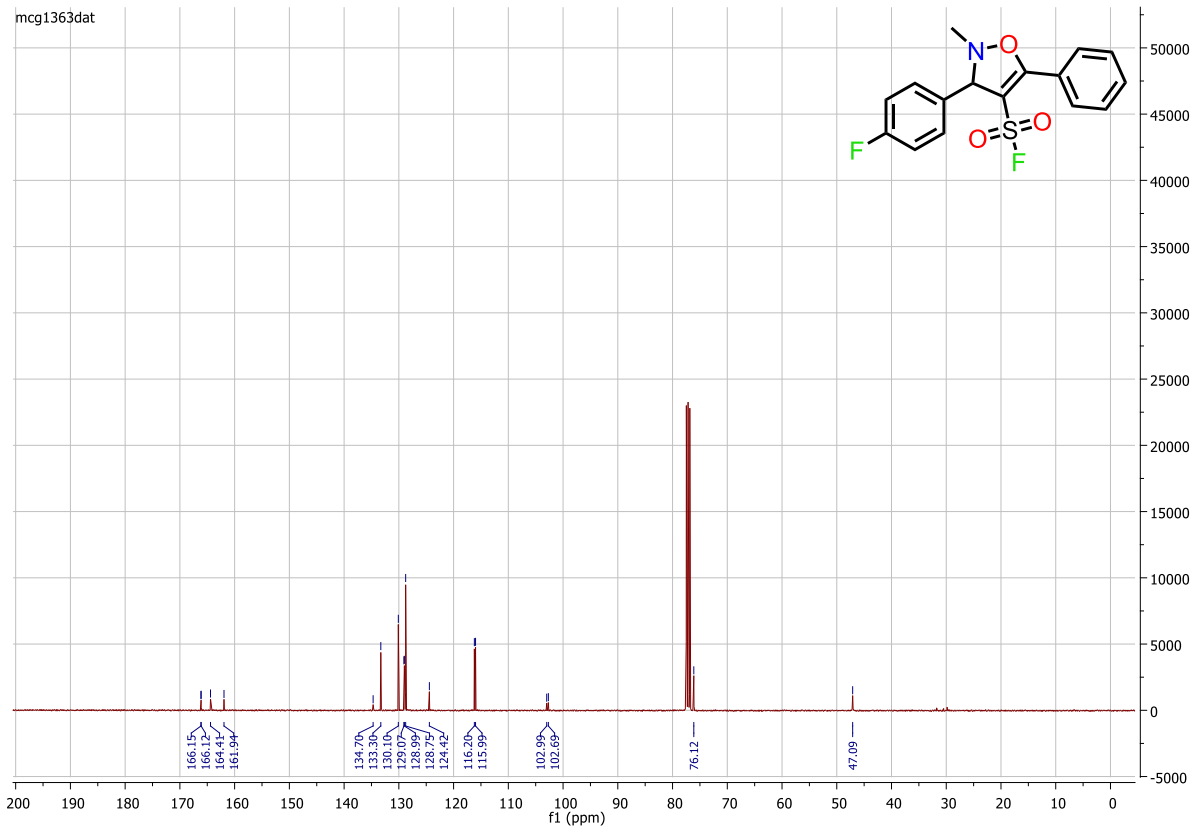
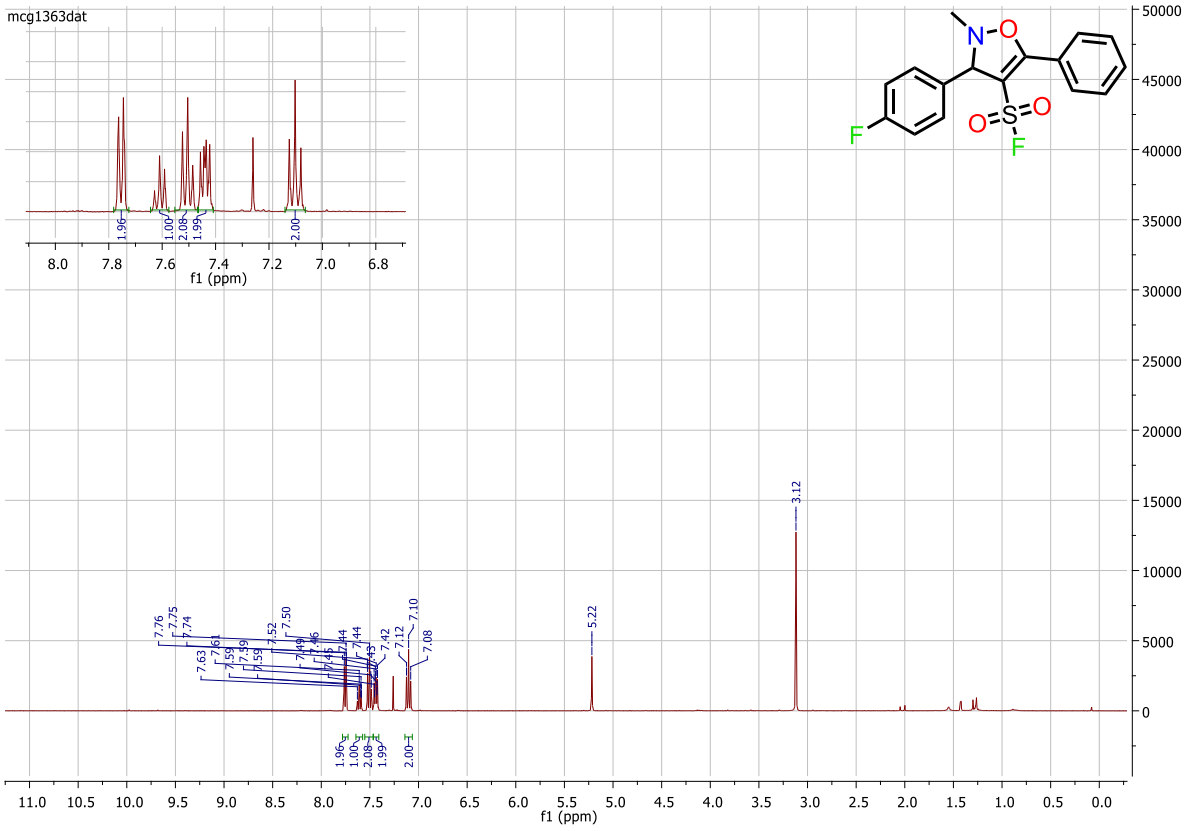


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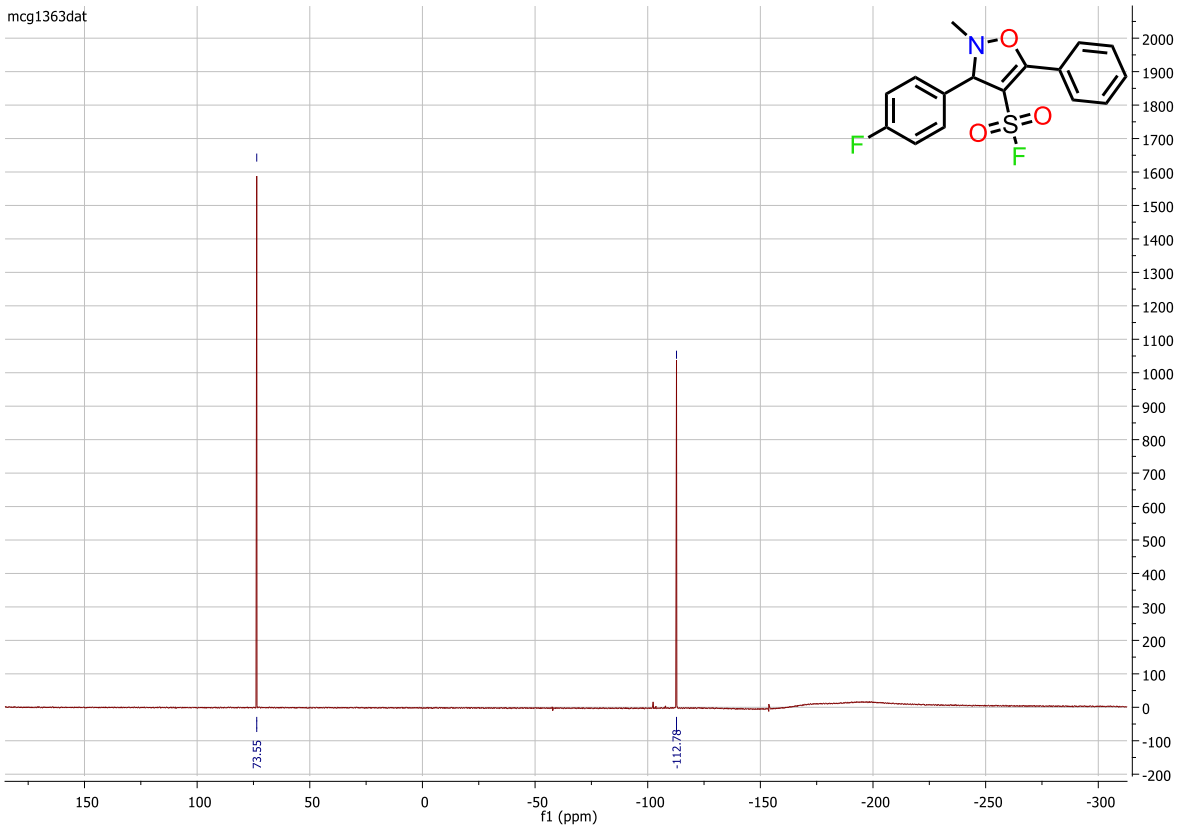


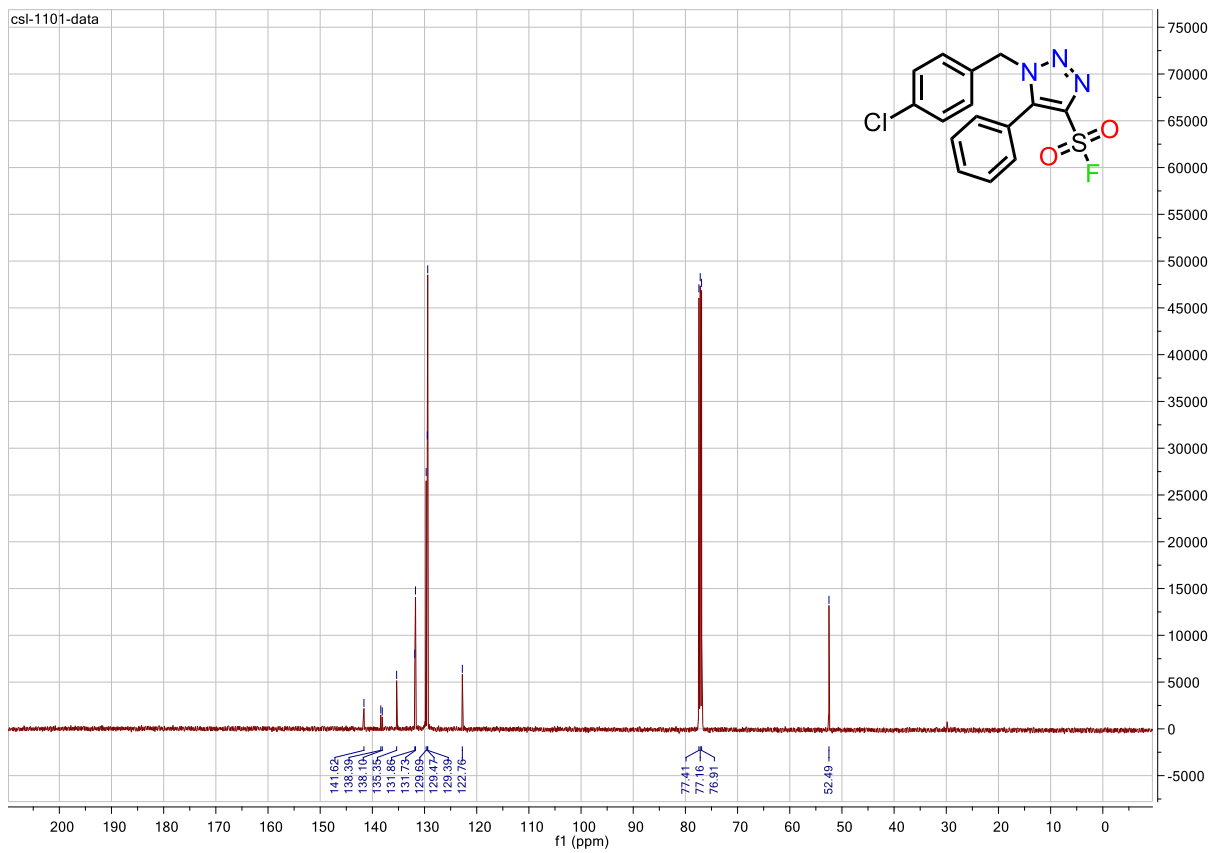
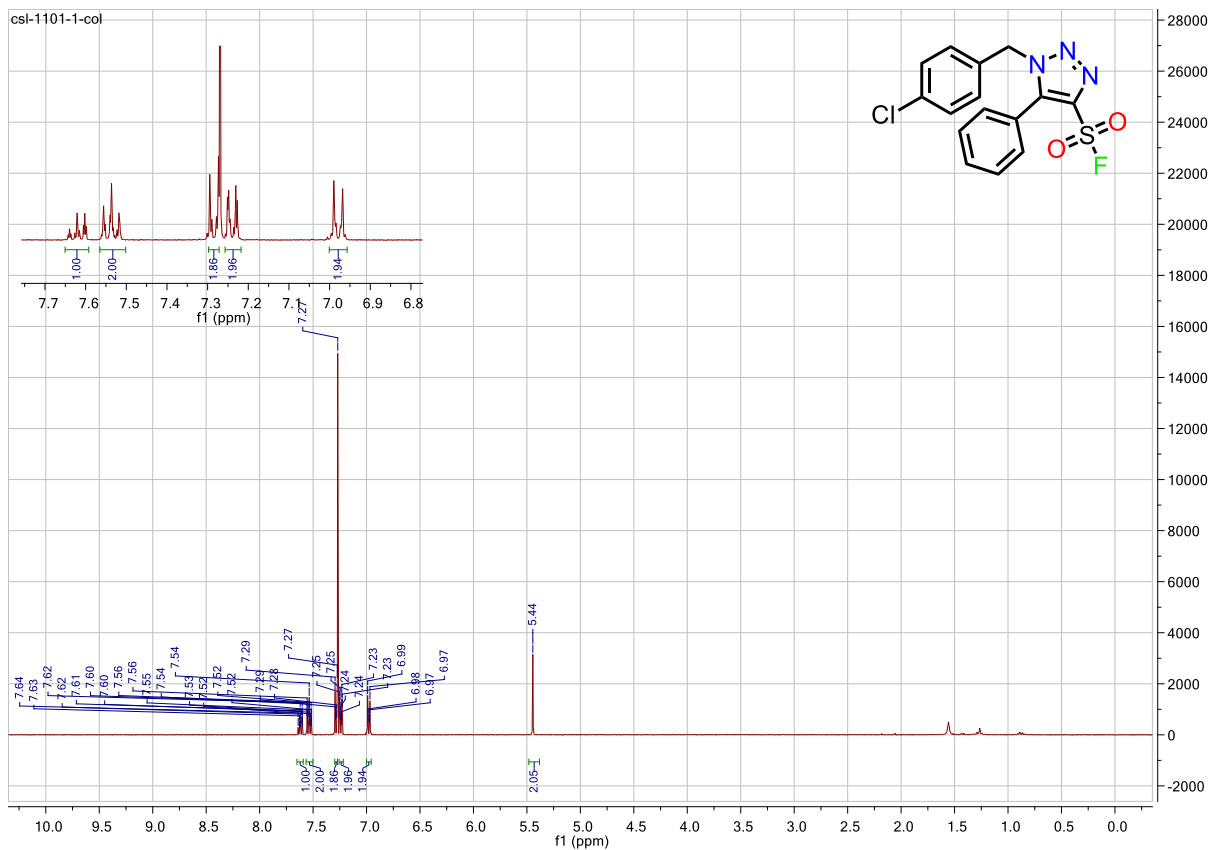
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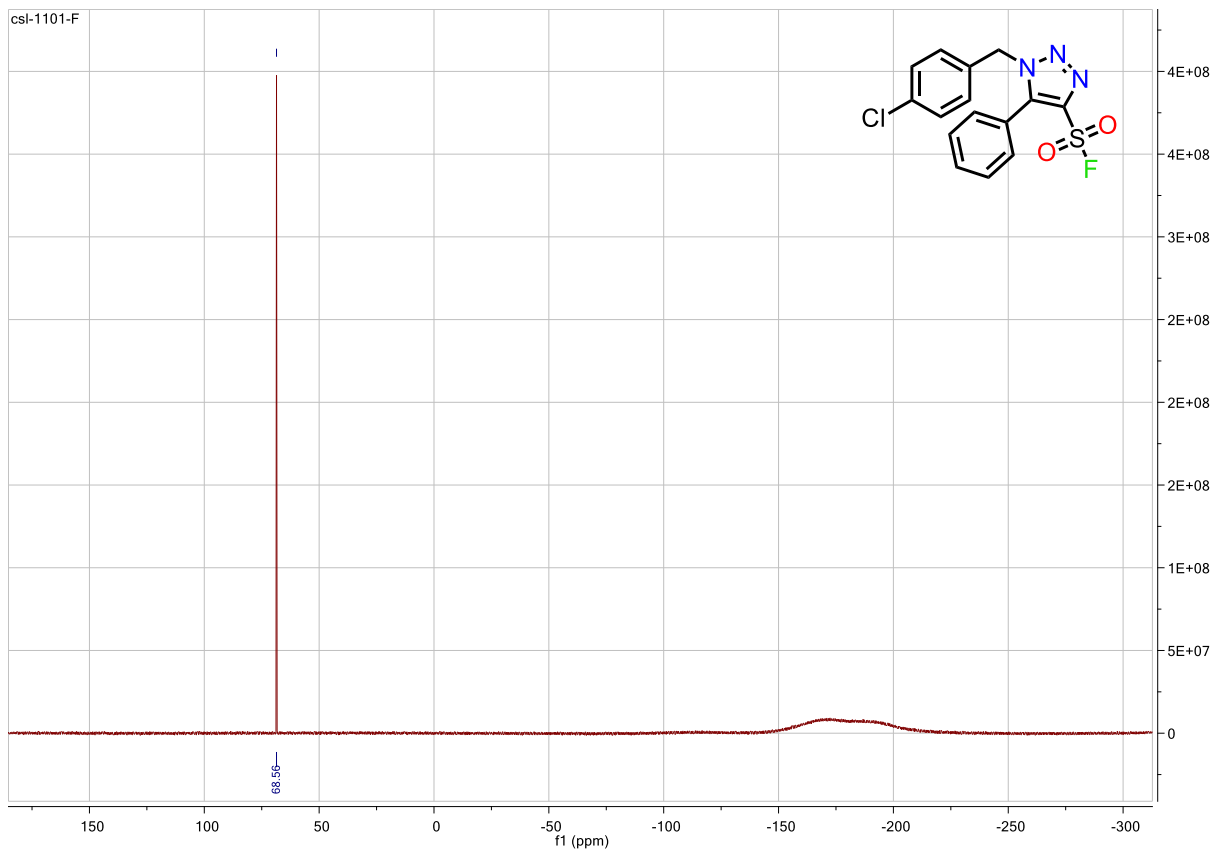




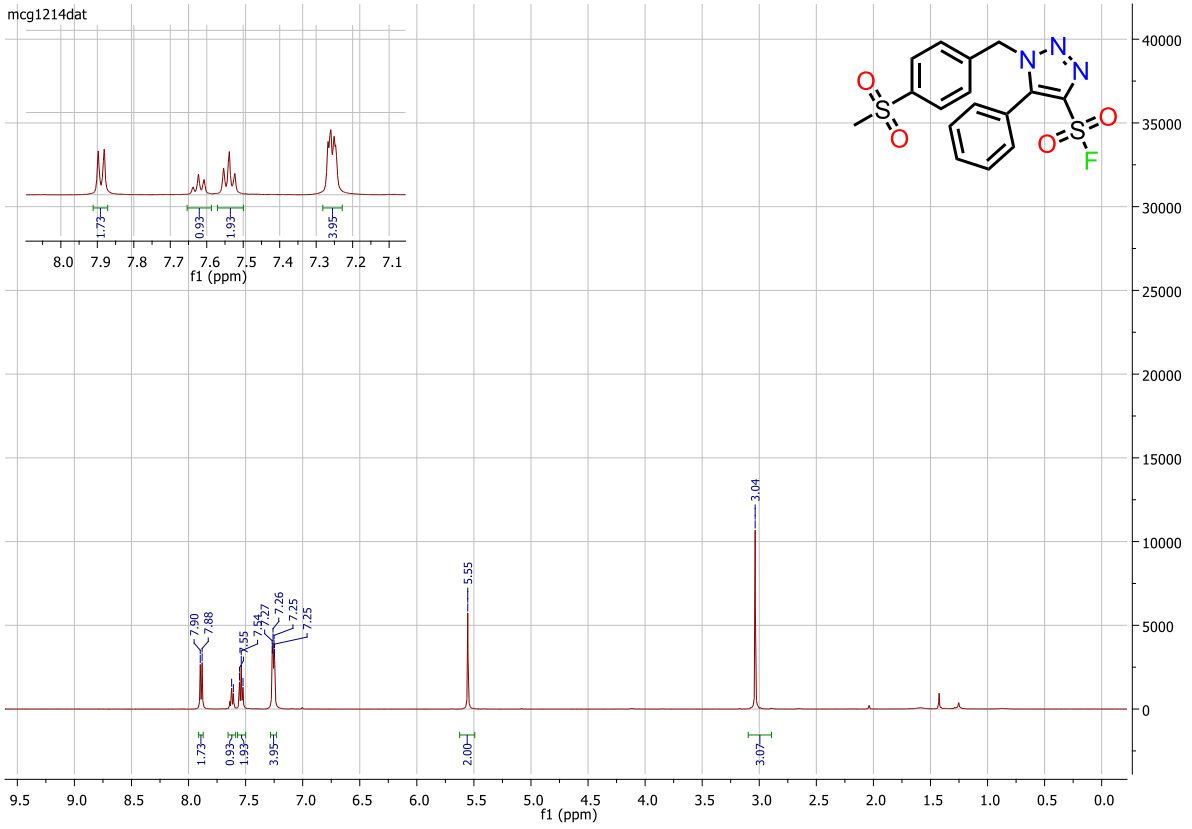
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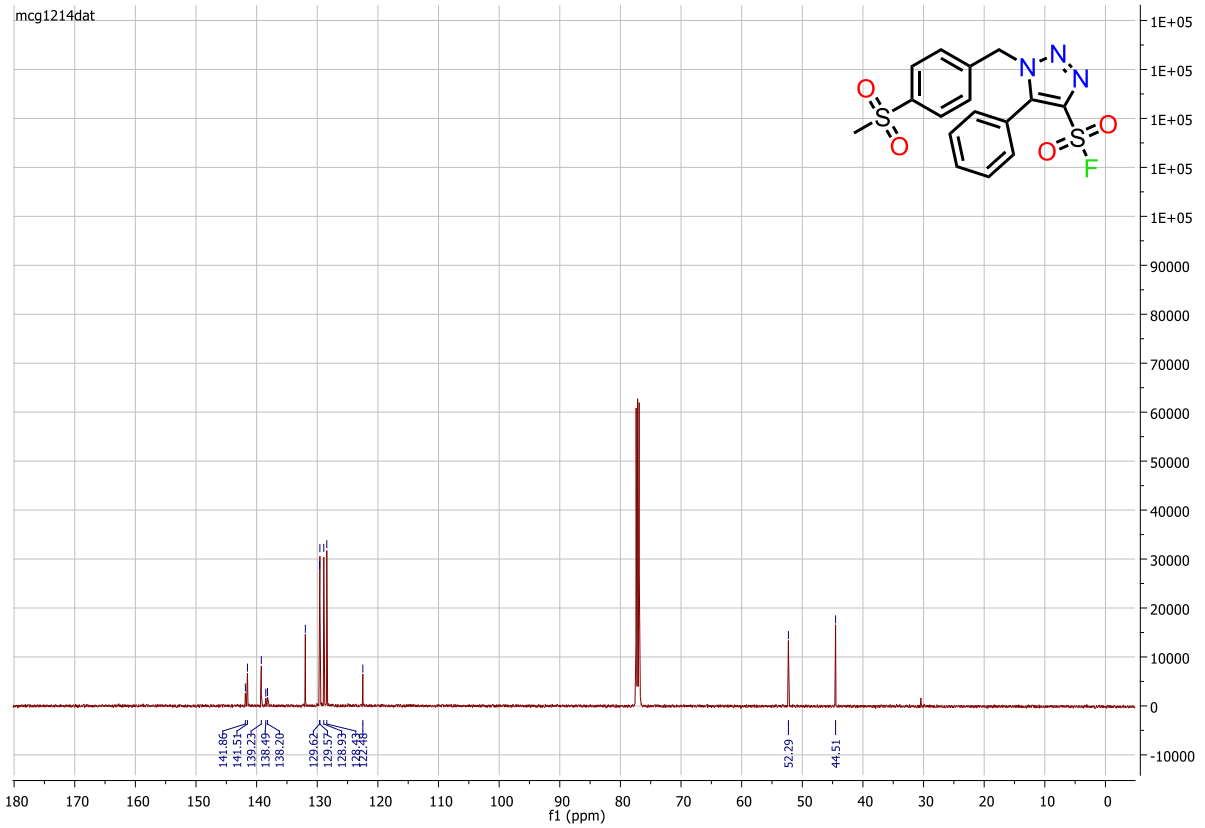




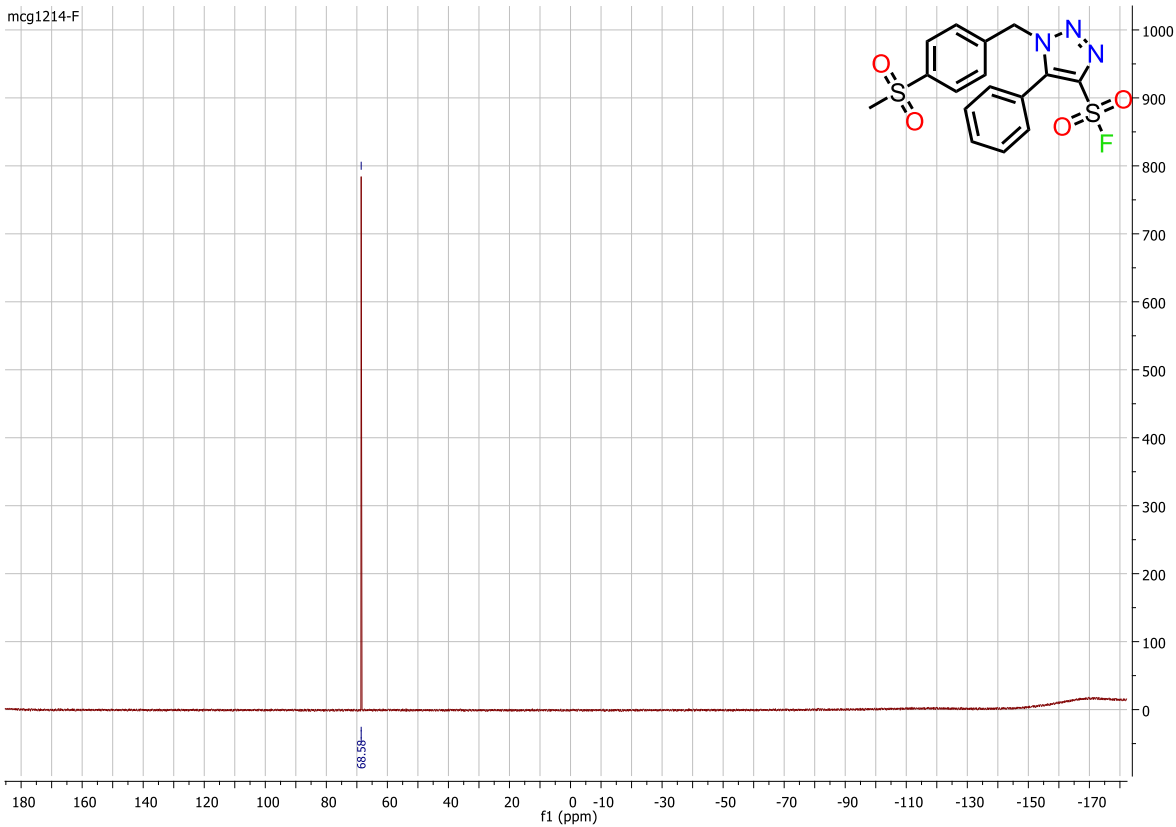
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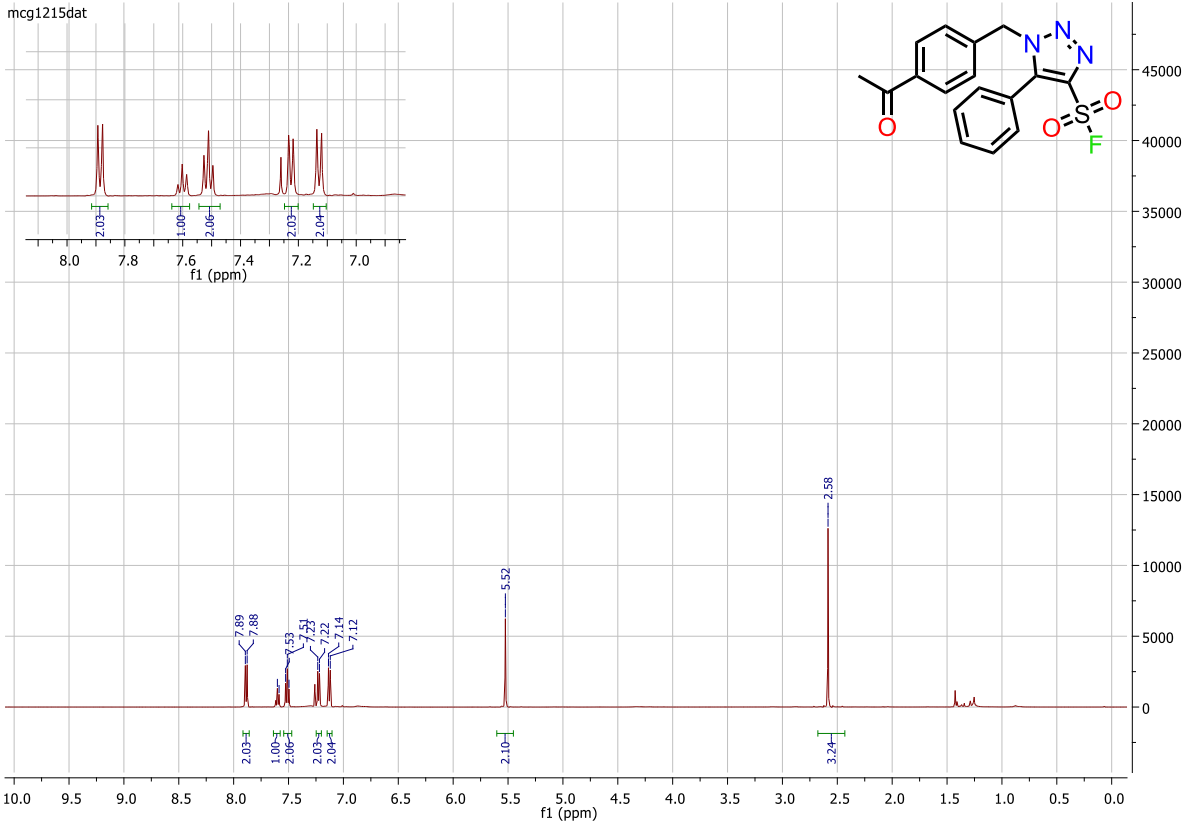
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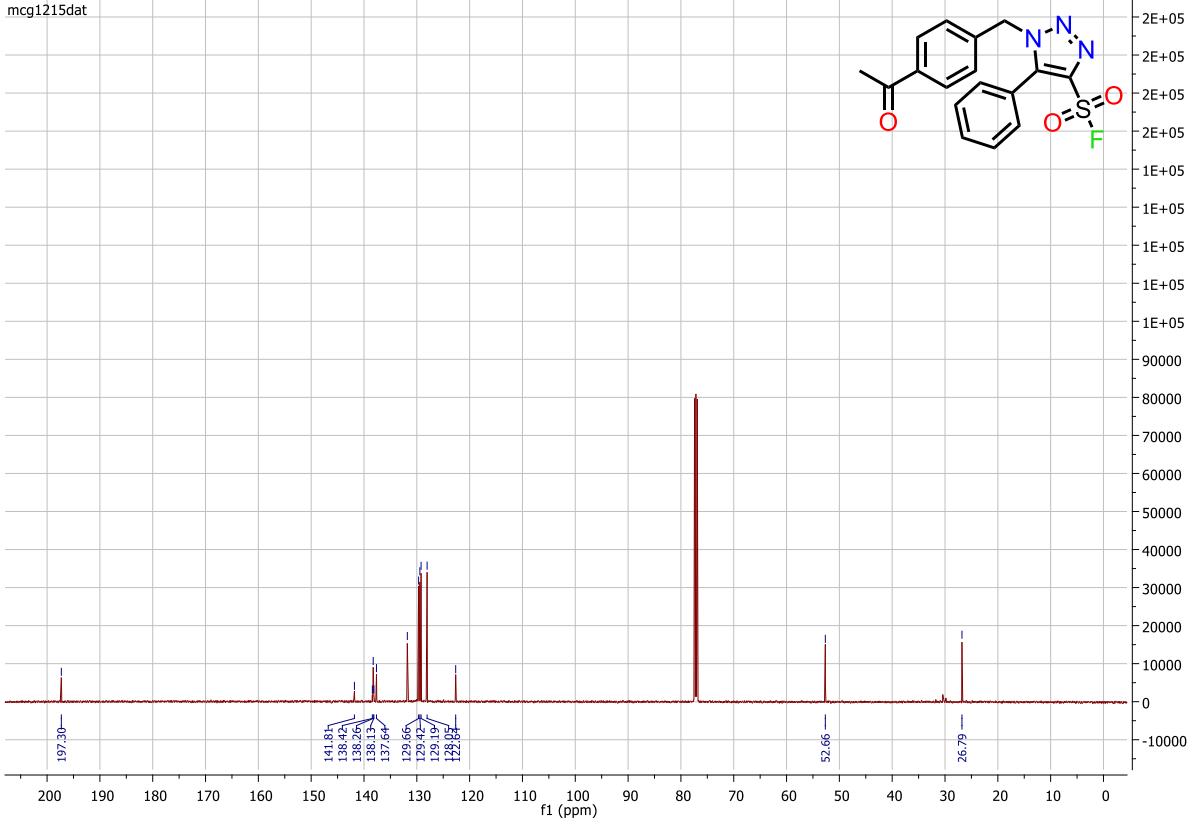
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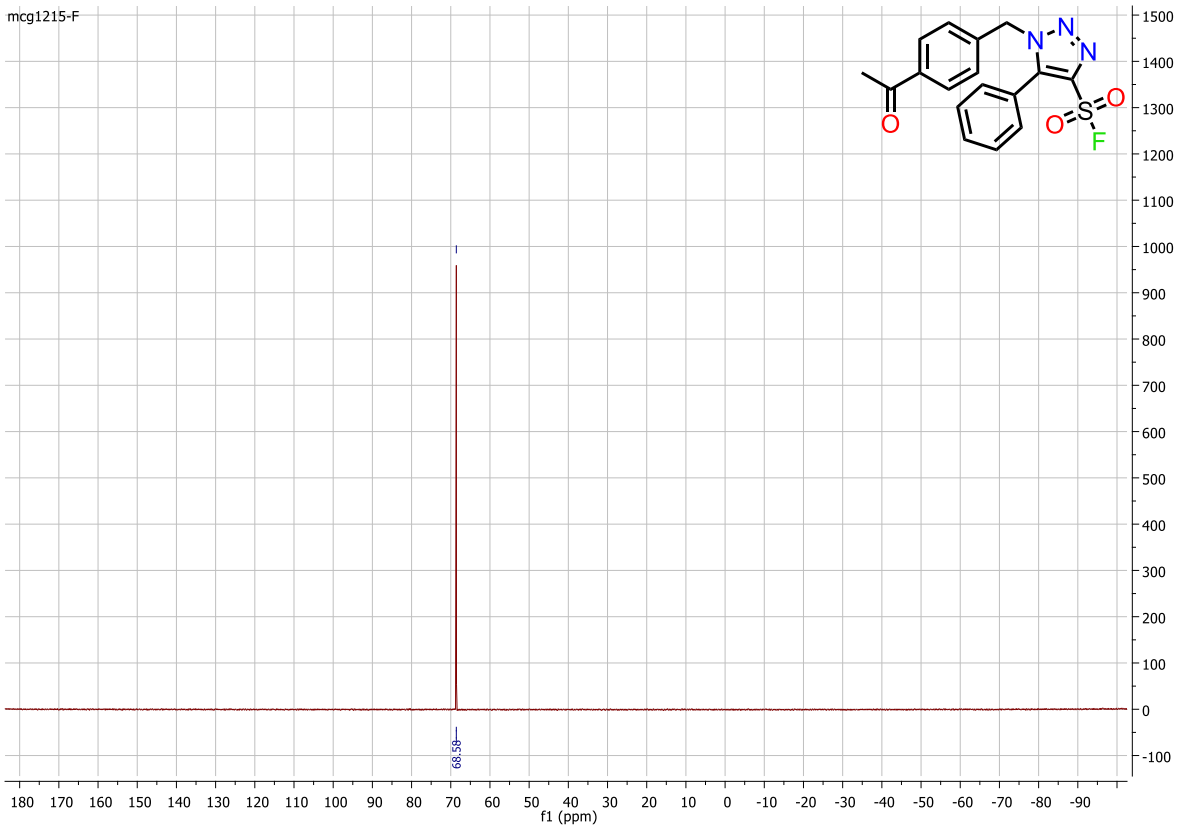
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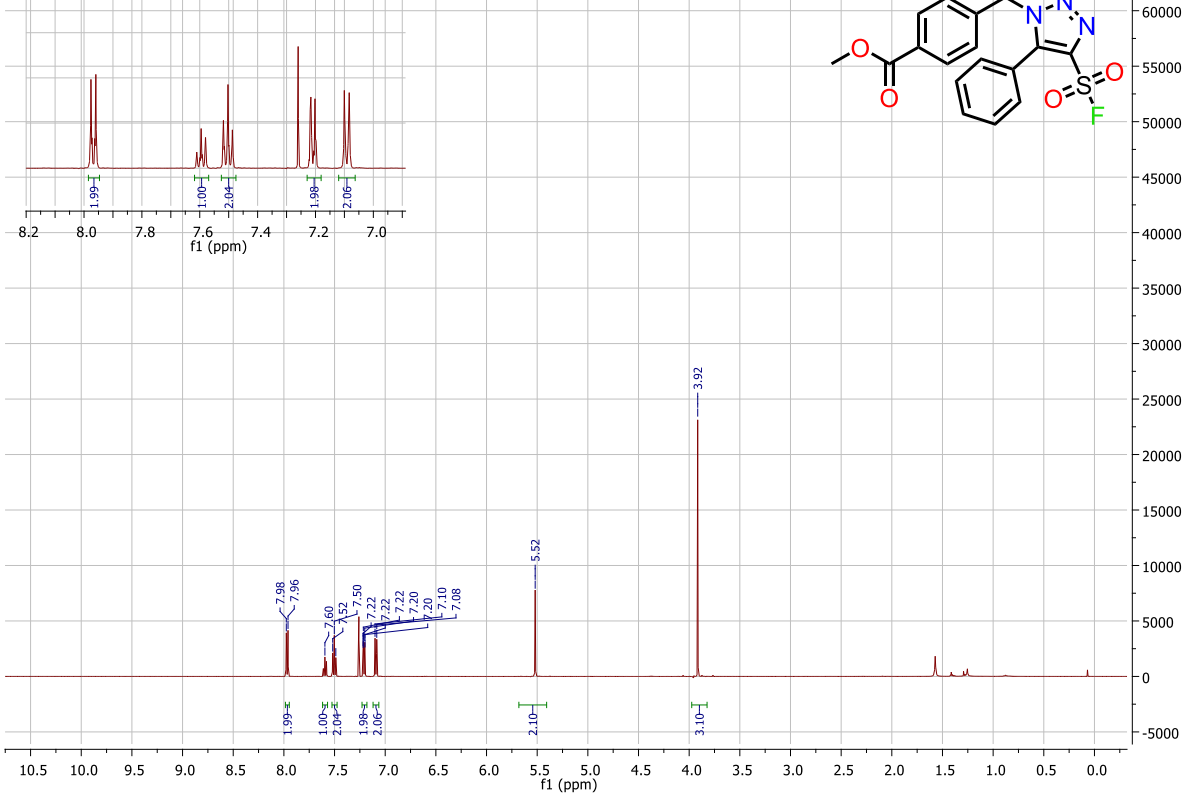
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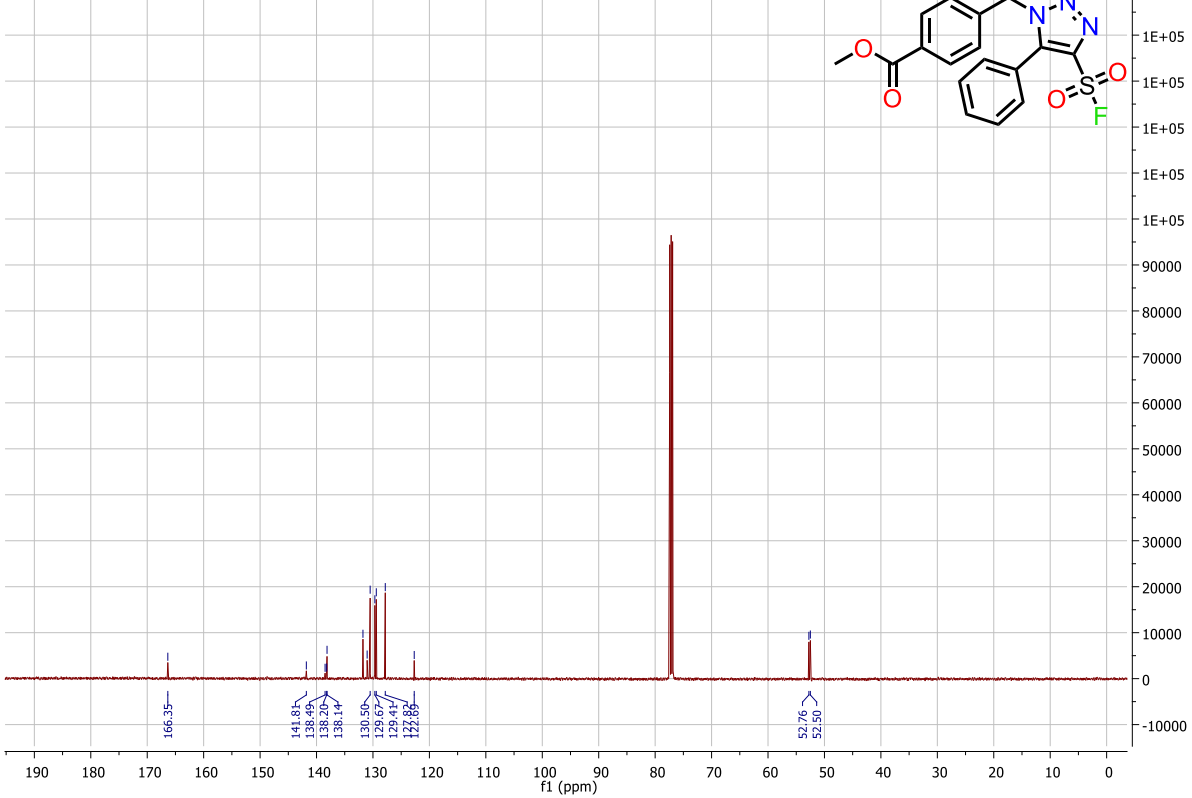
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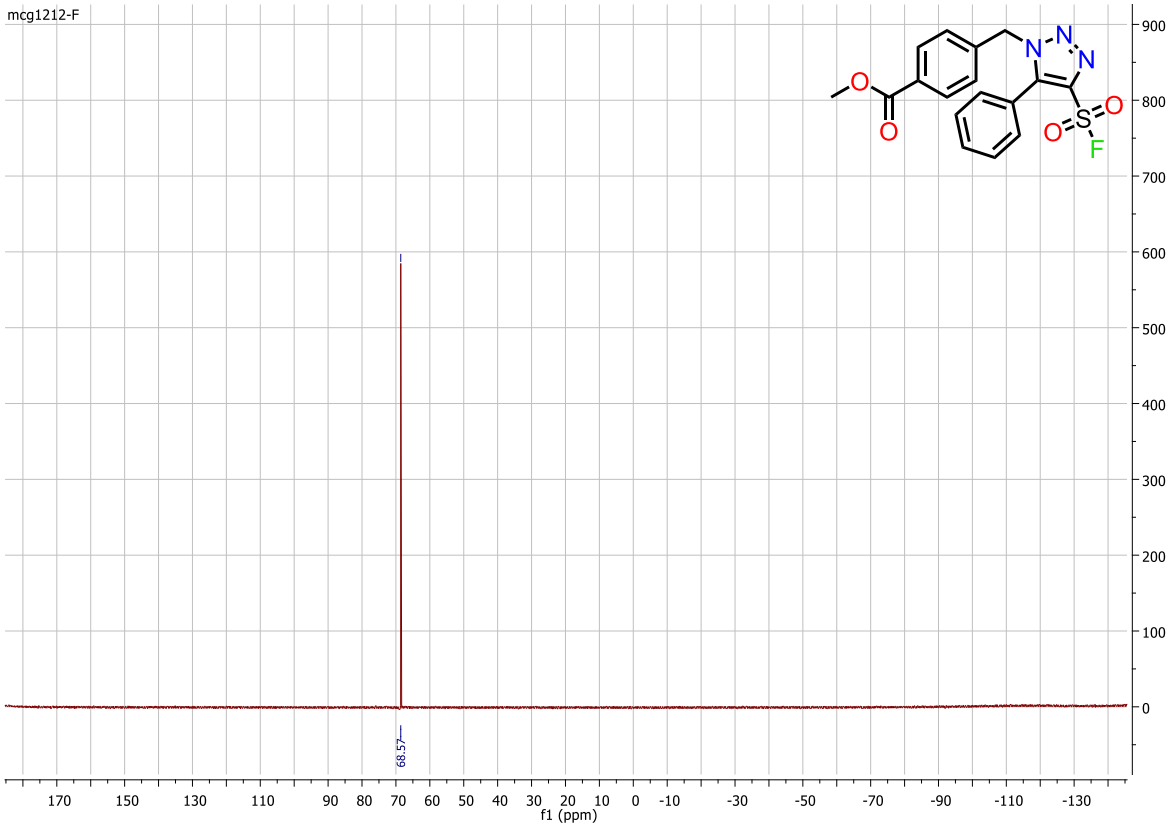
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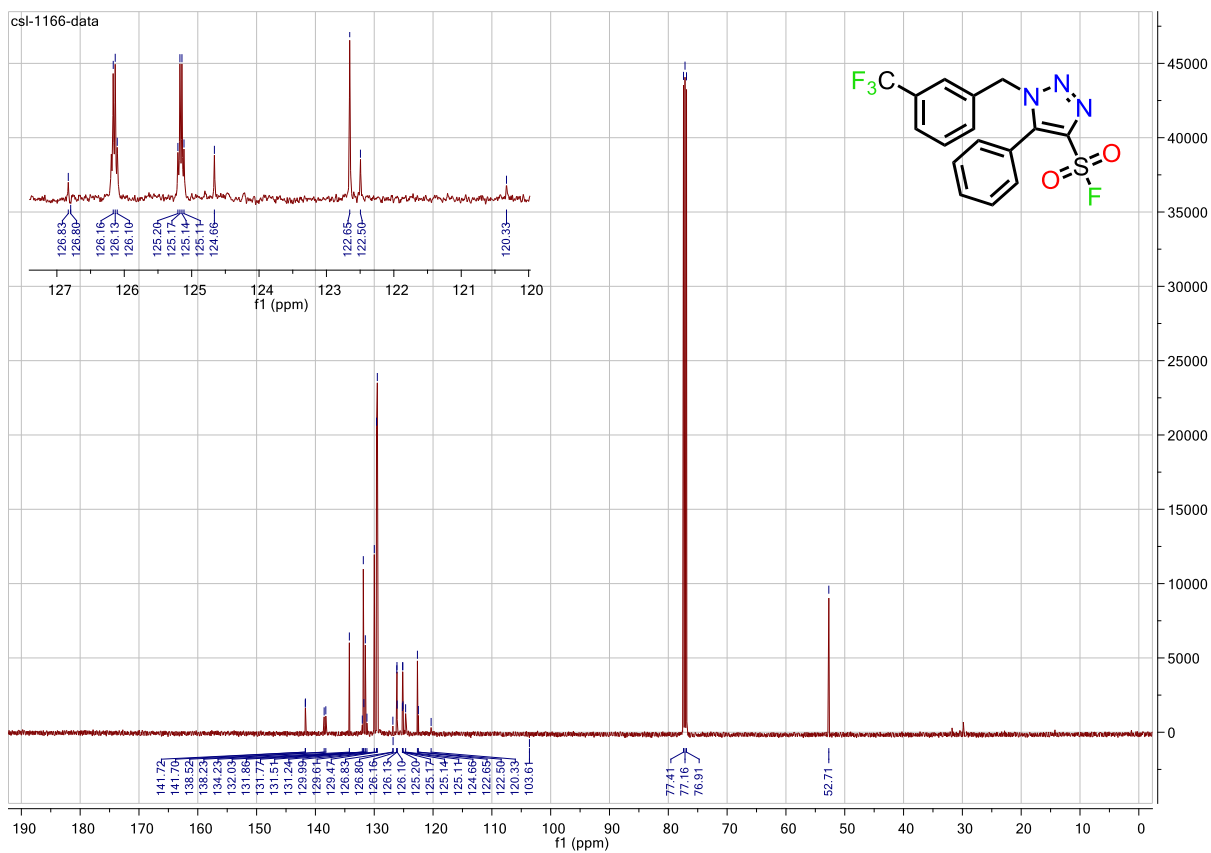
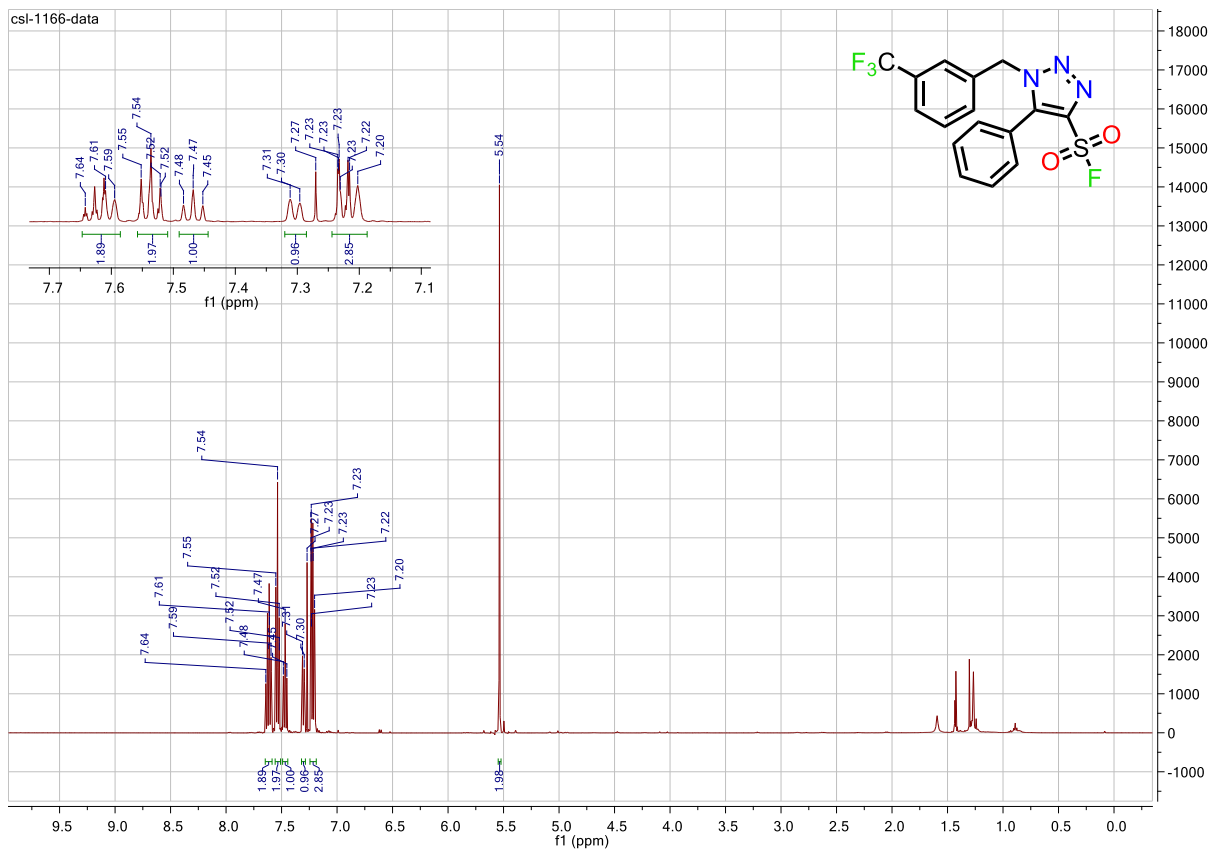


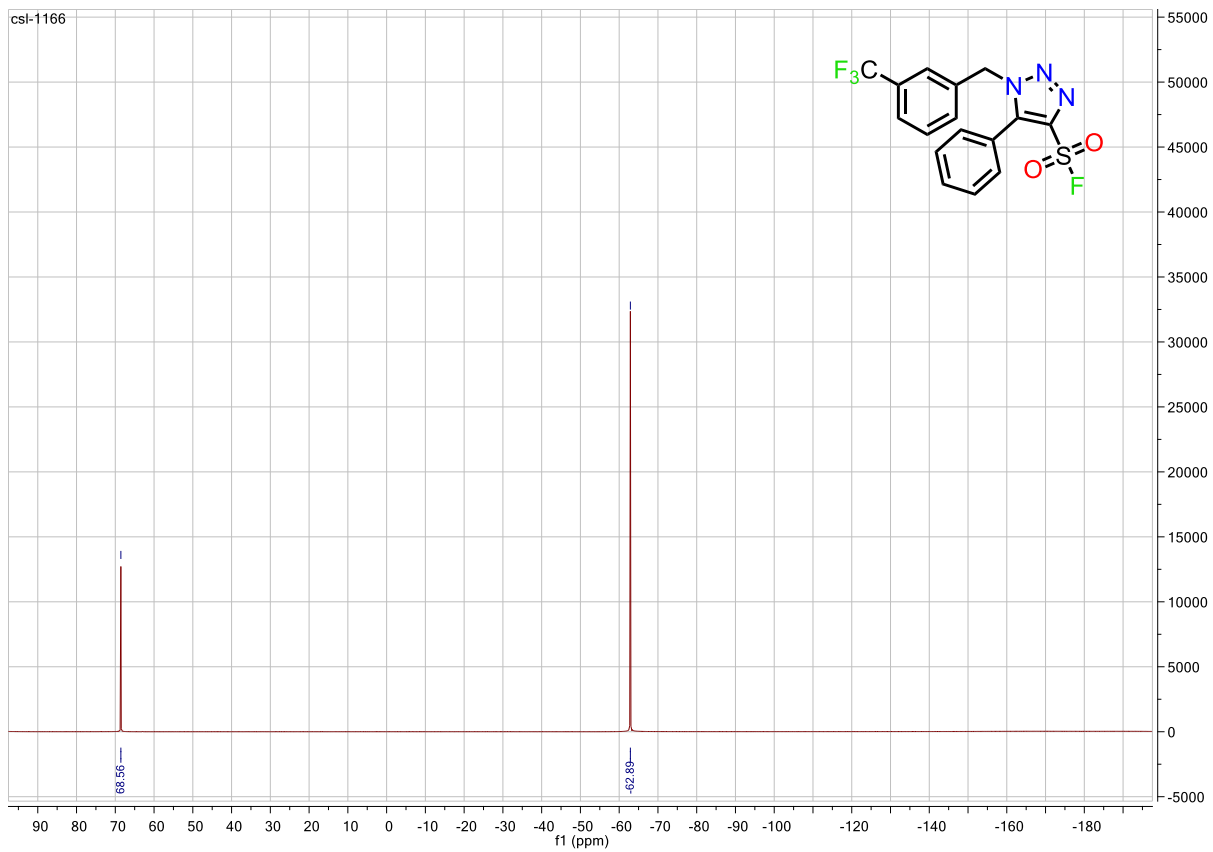
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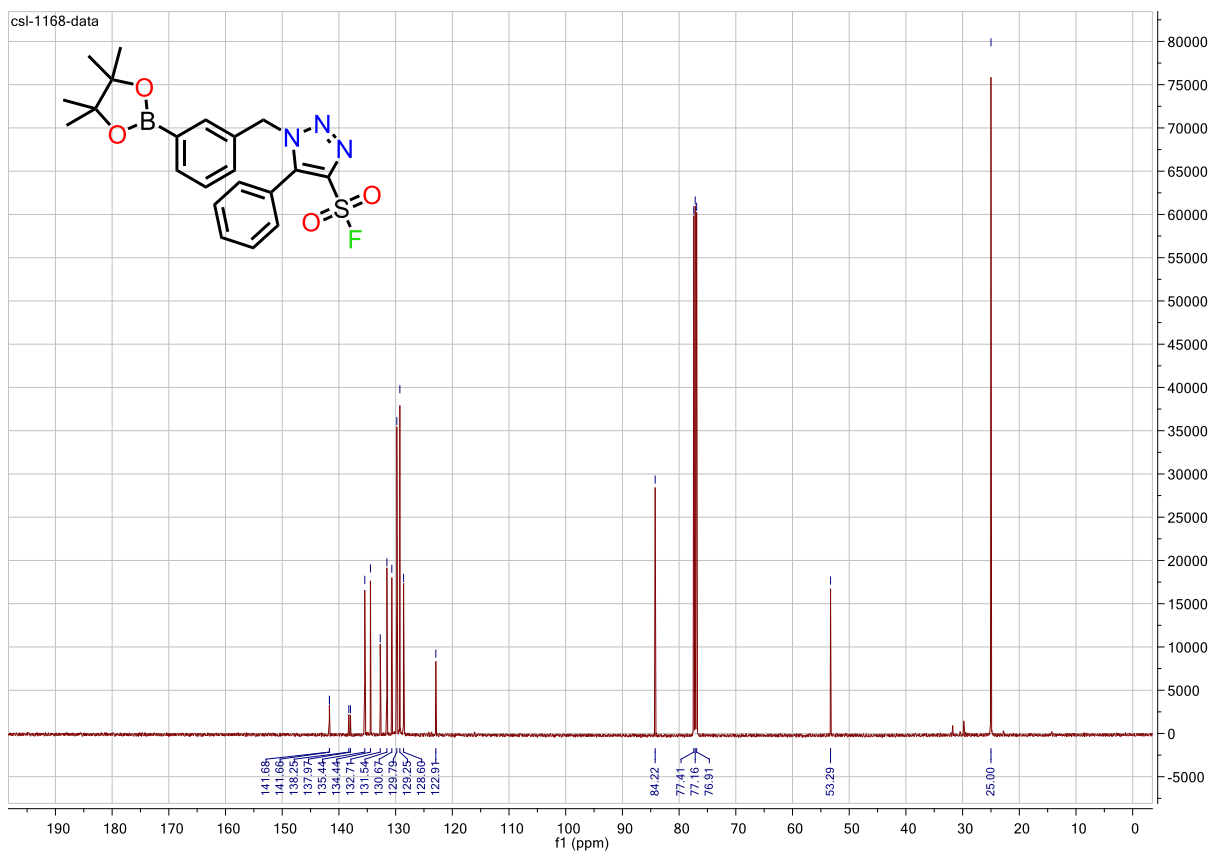
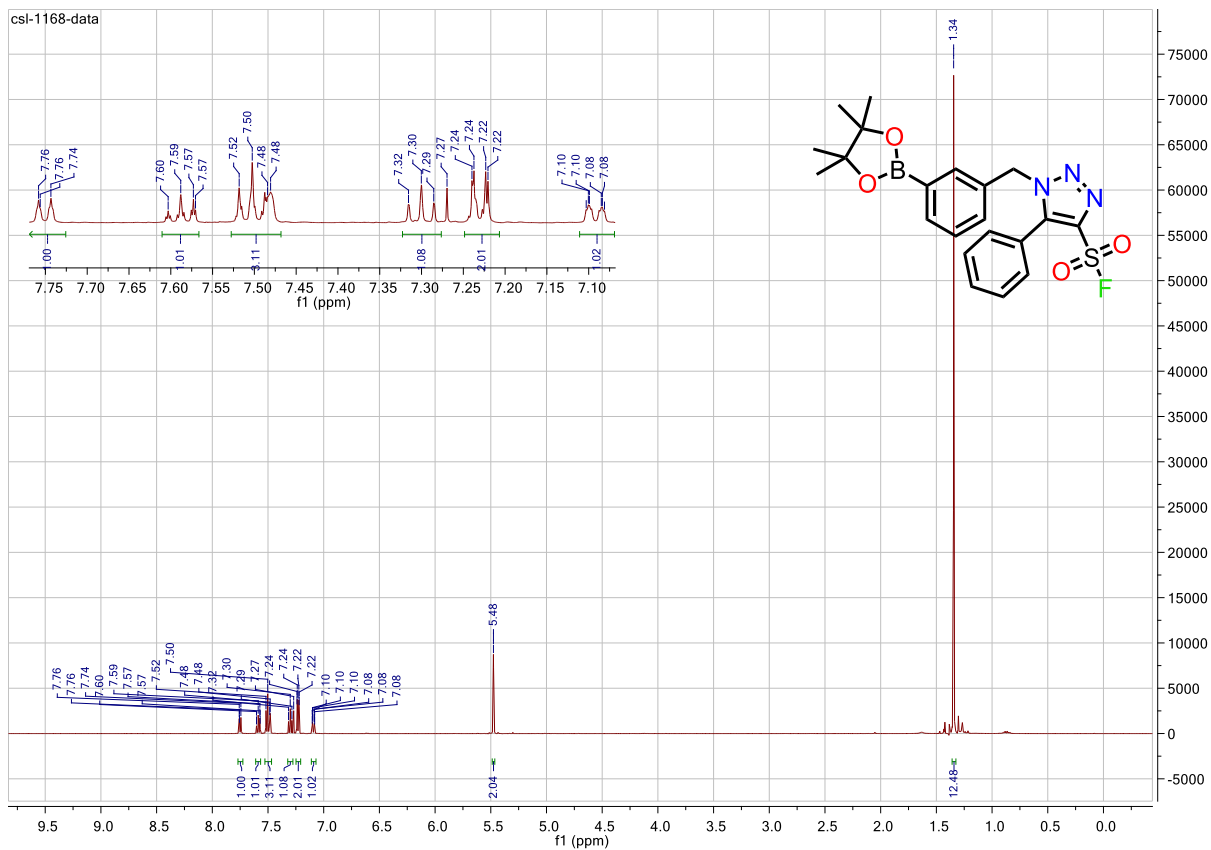


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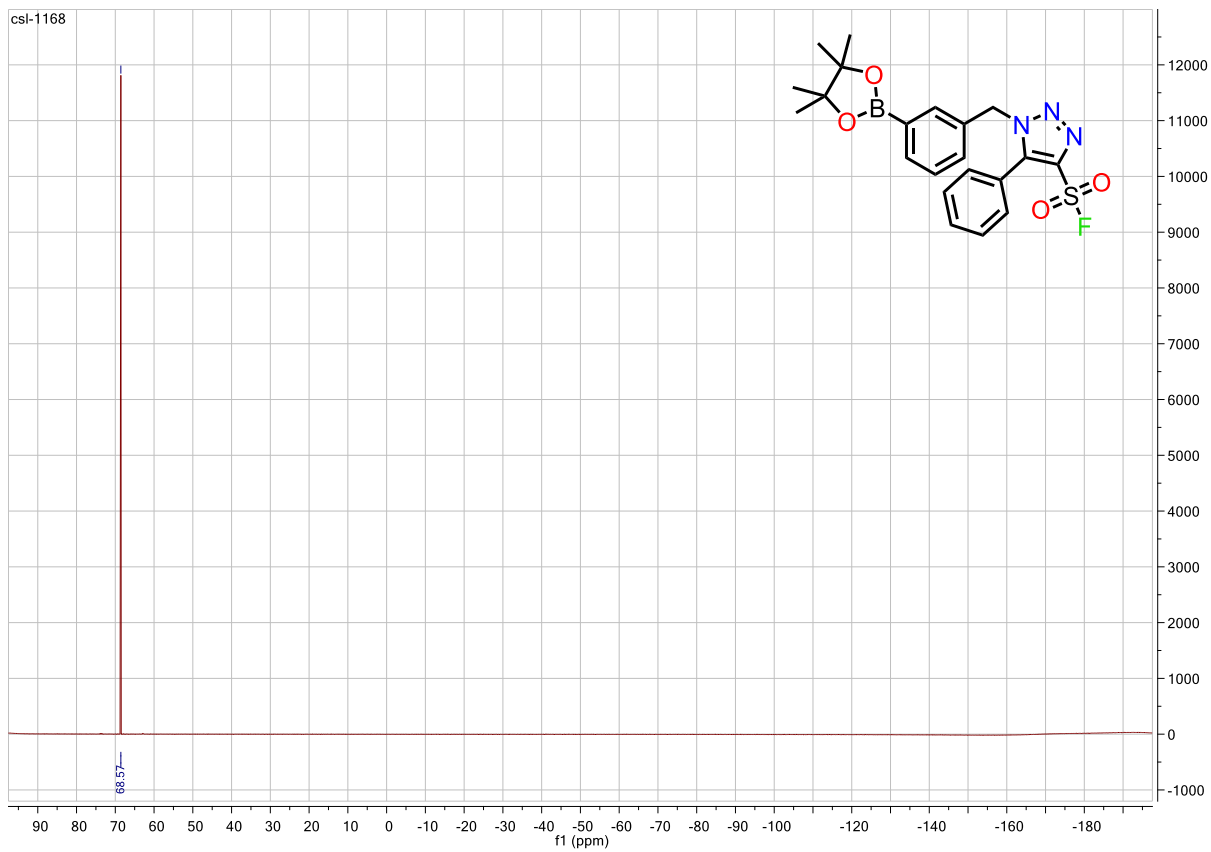




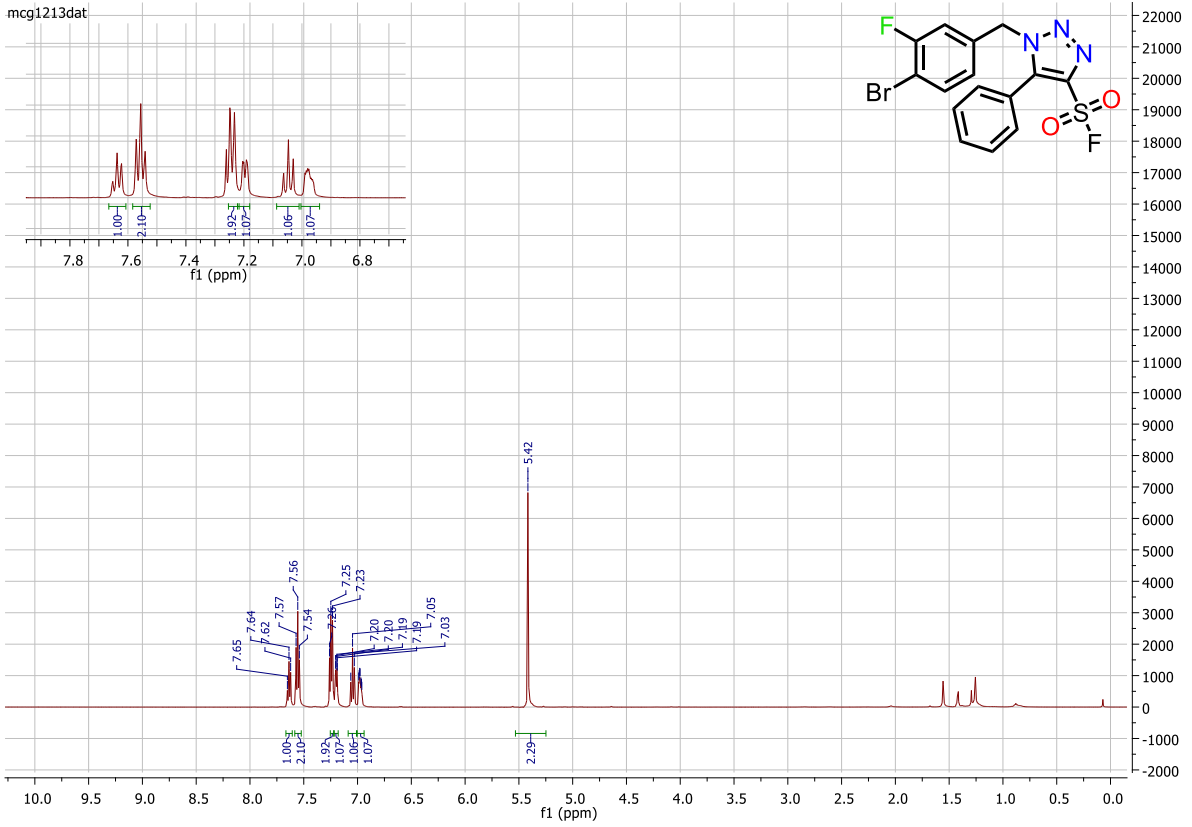




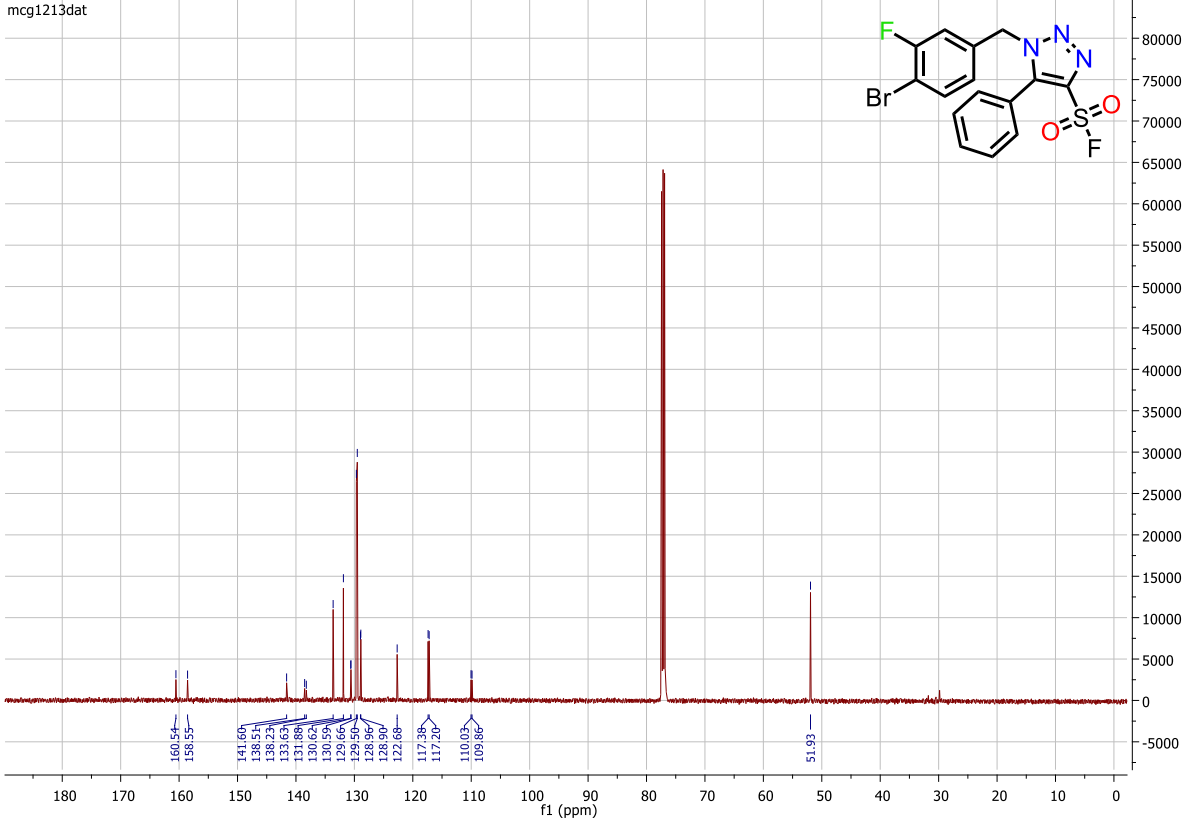
csi-1168



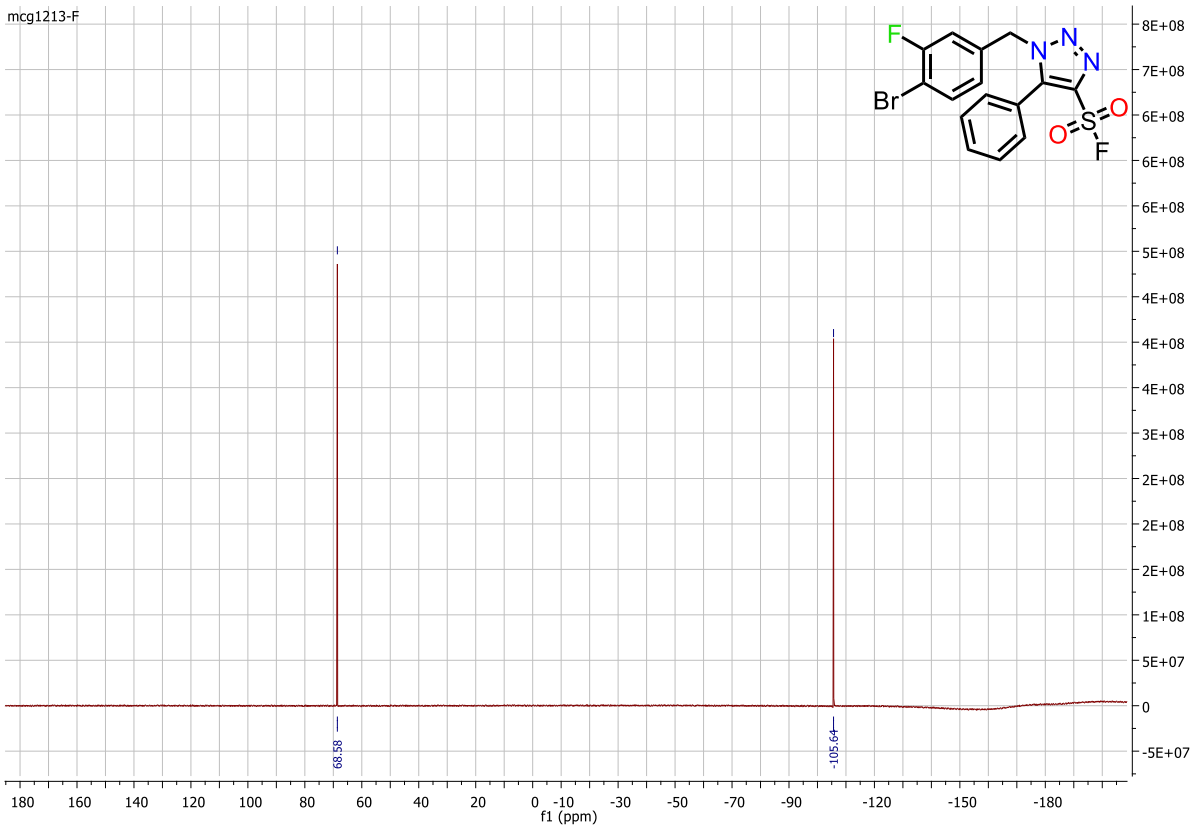
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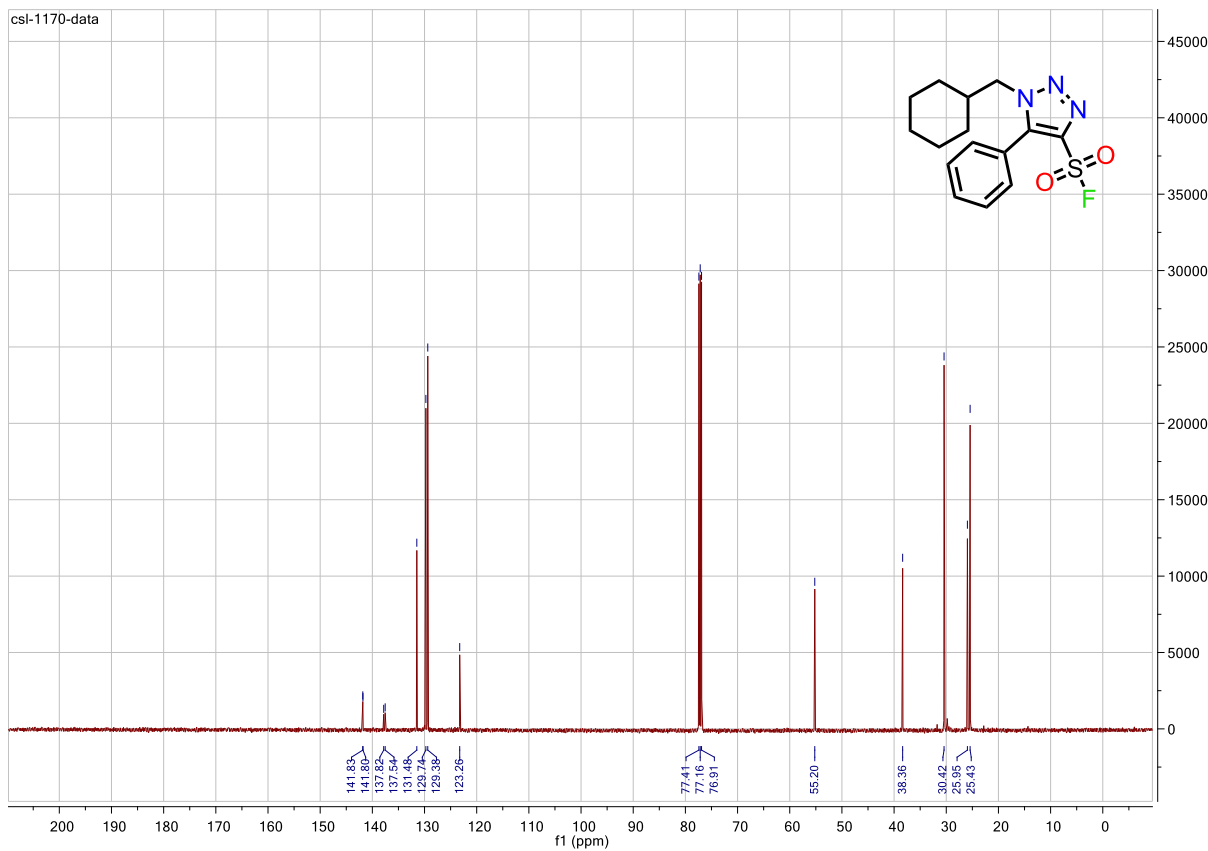
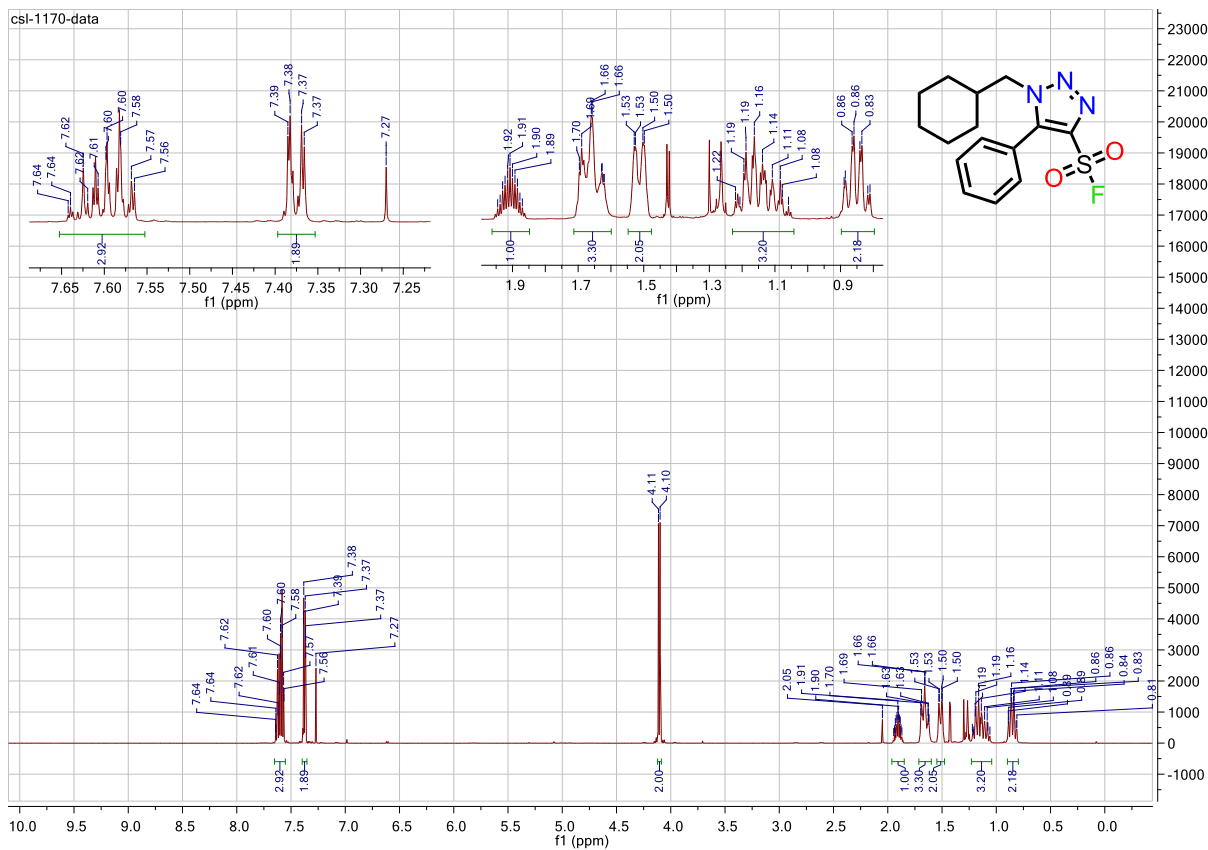


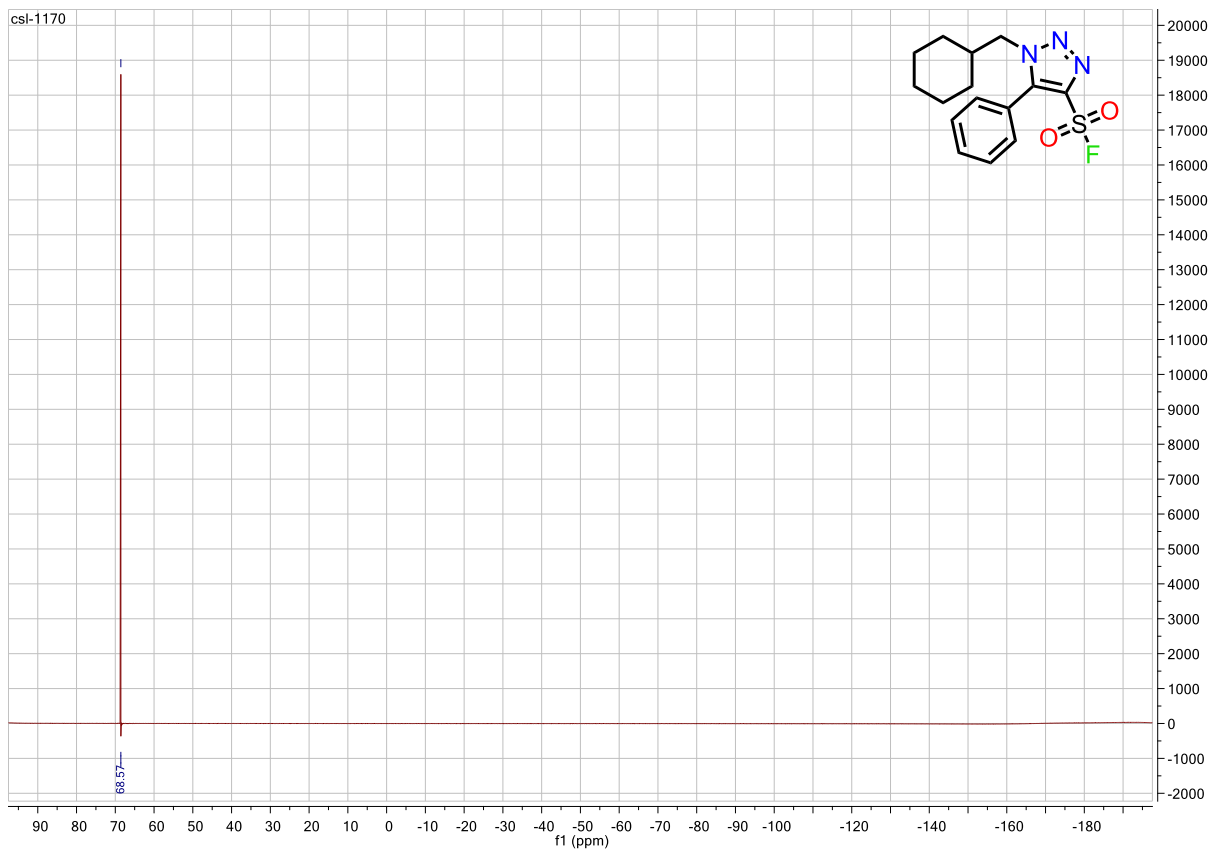
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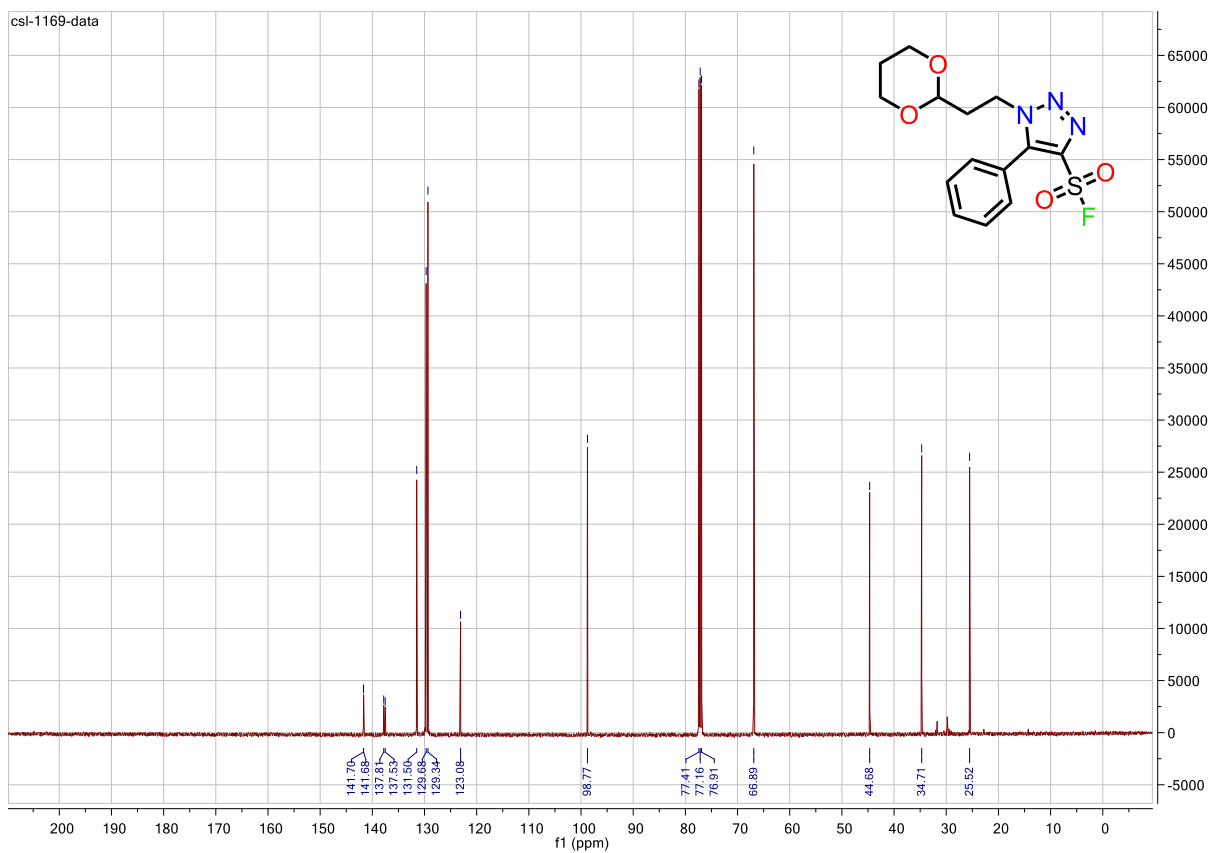
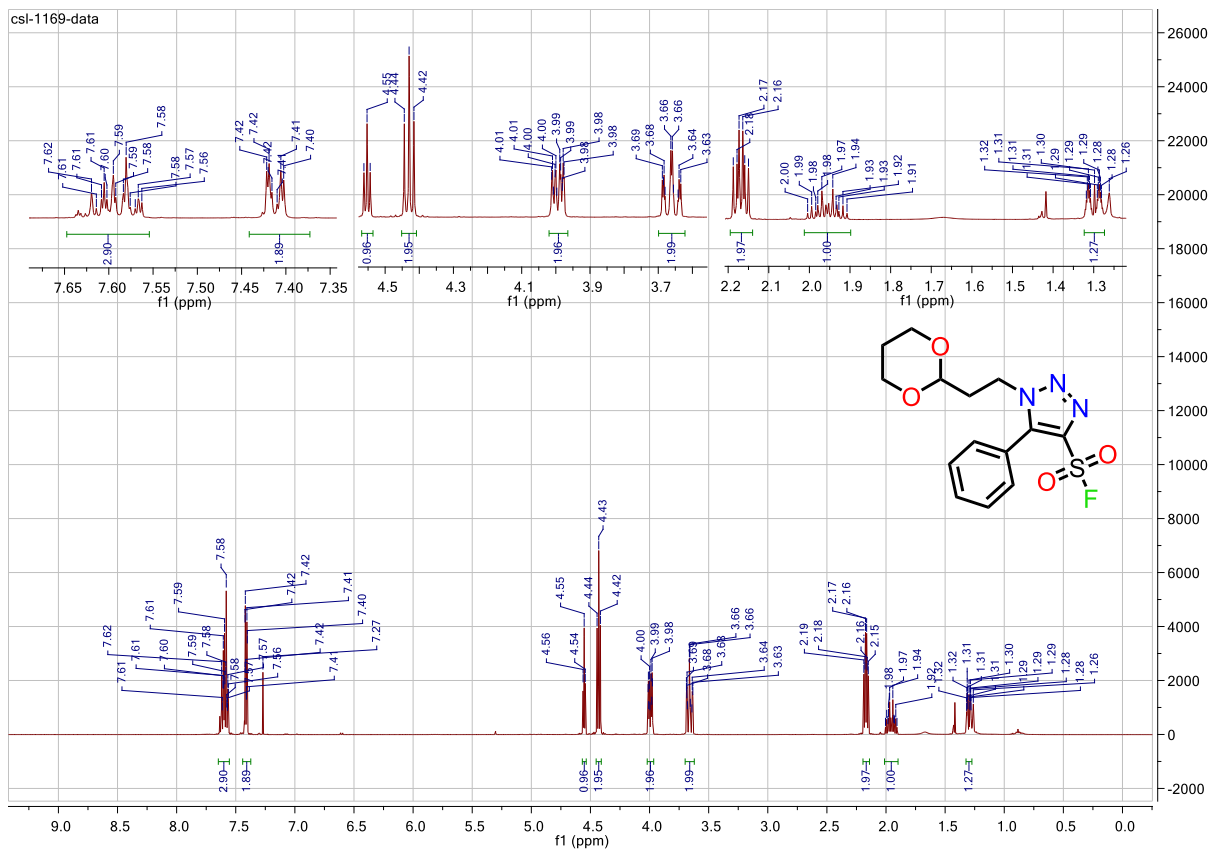


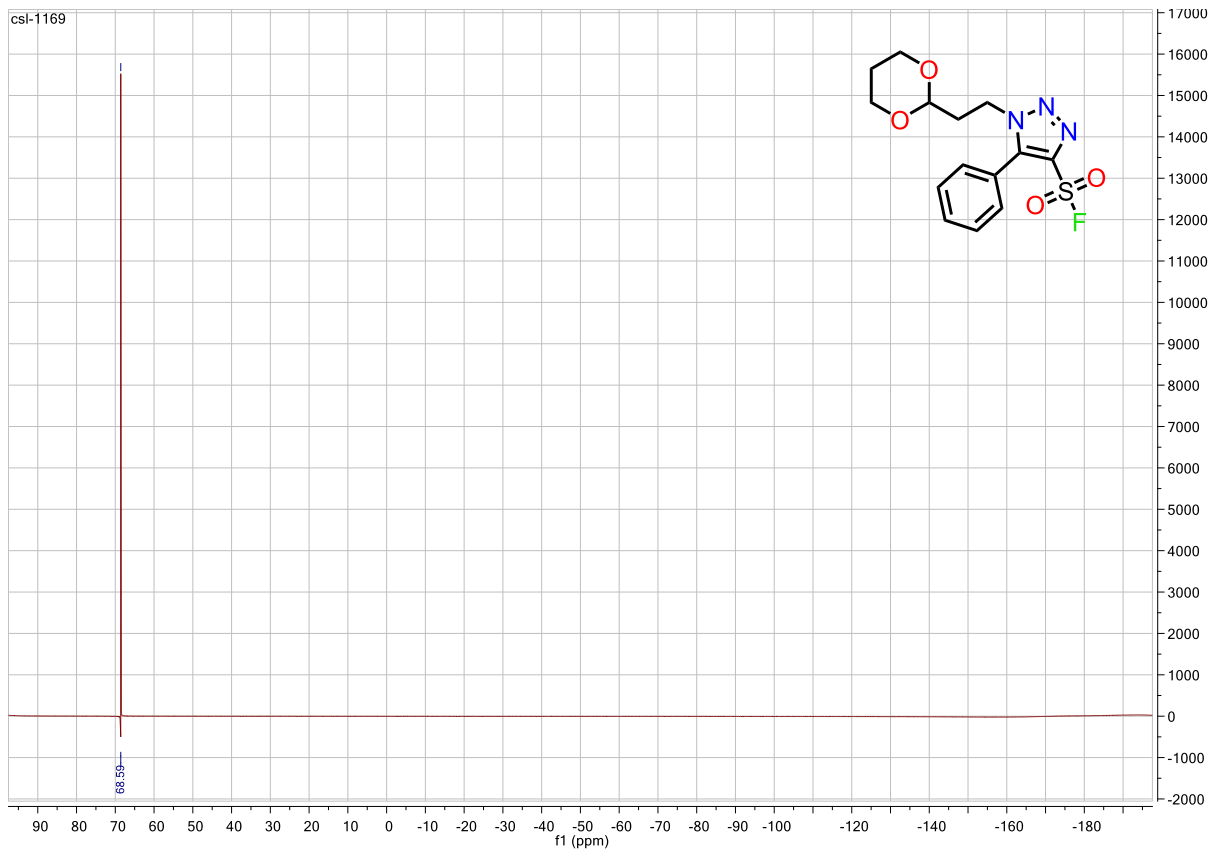
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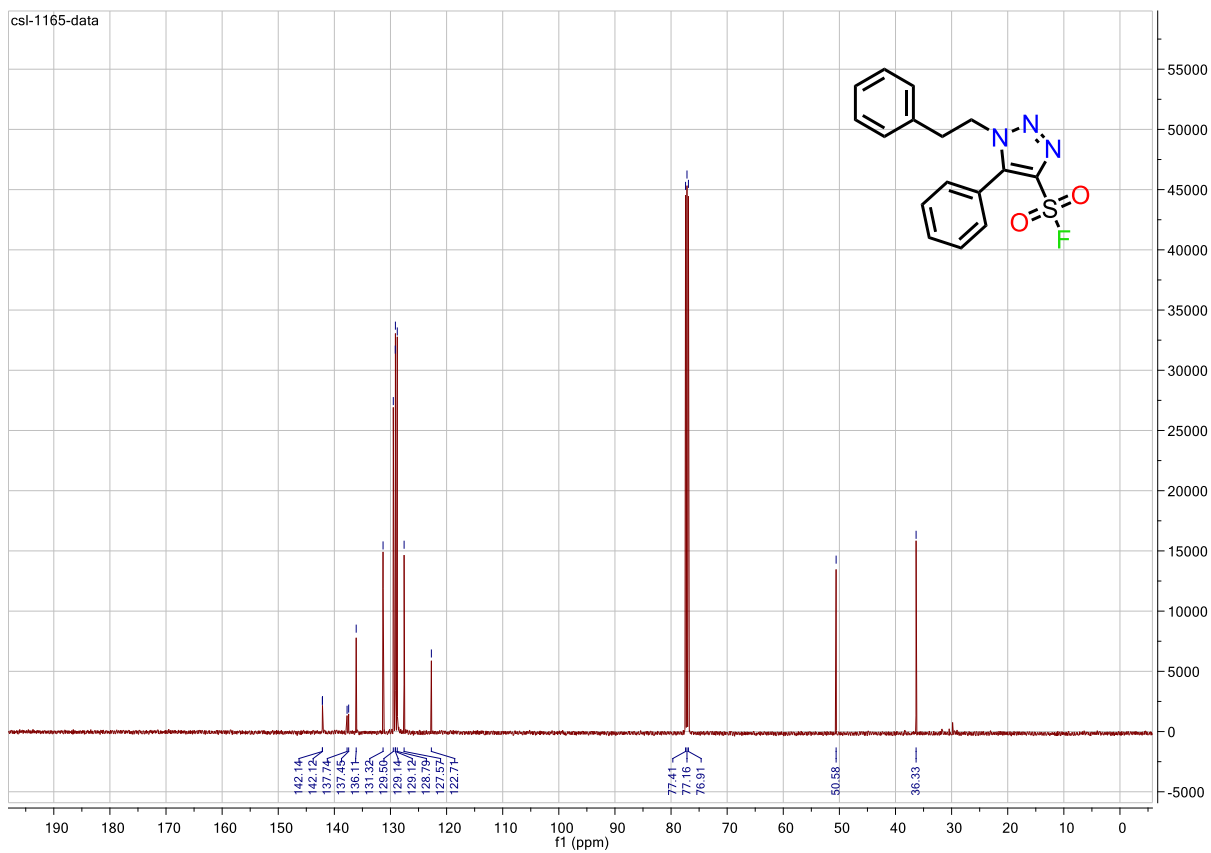
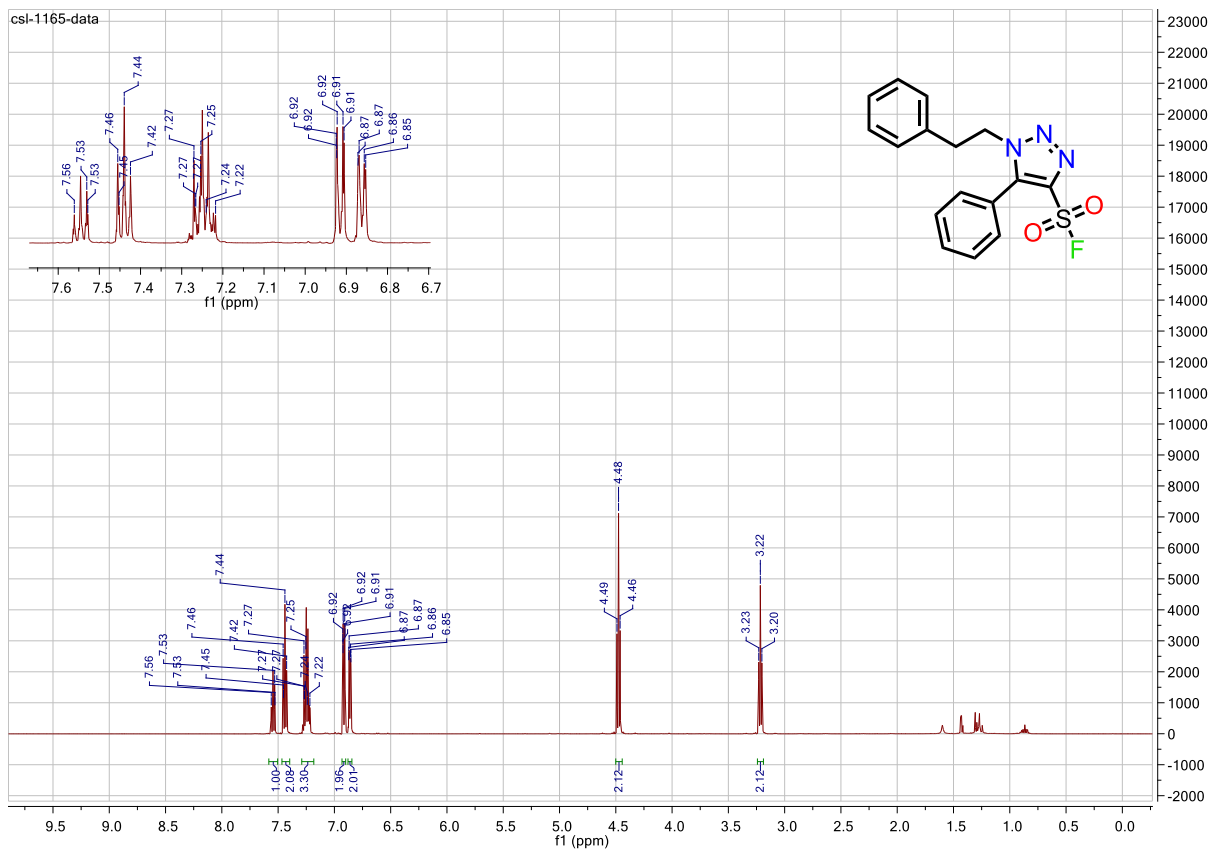


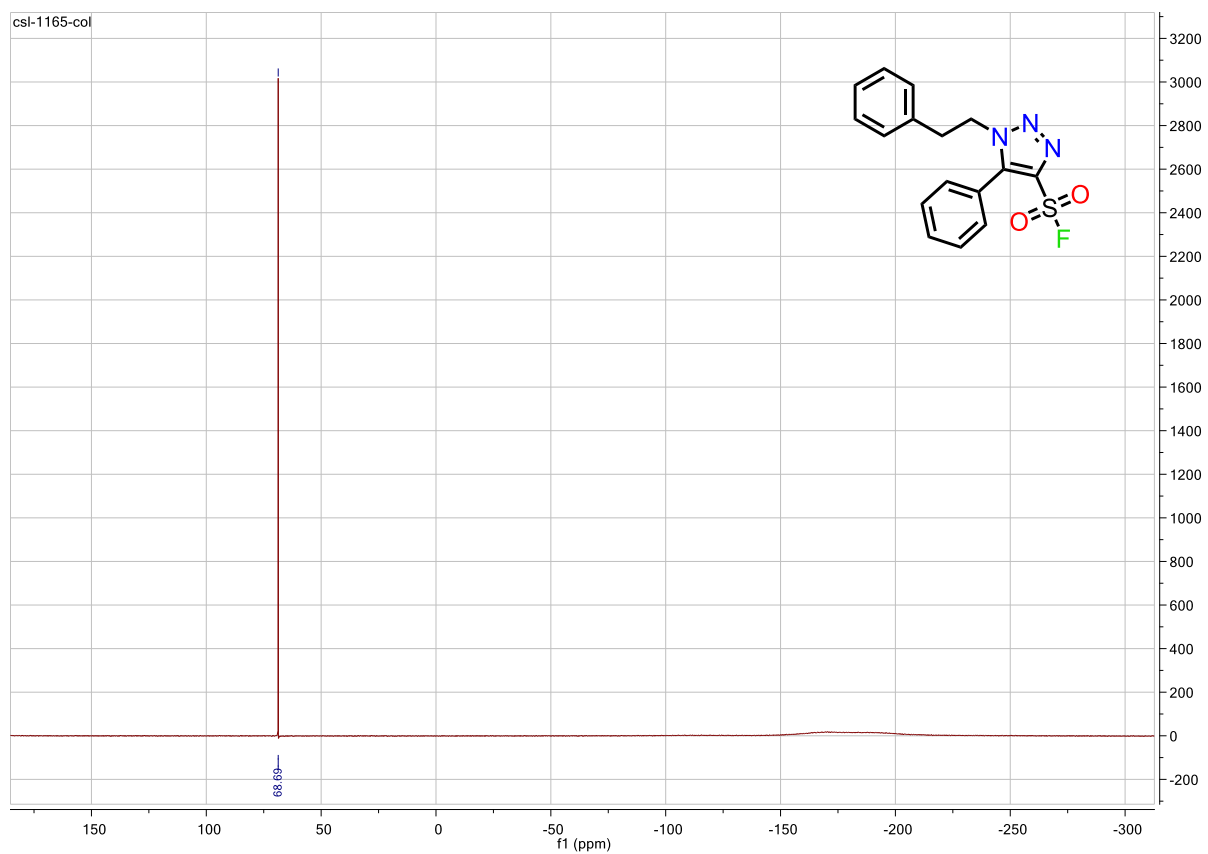


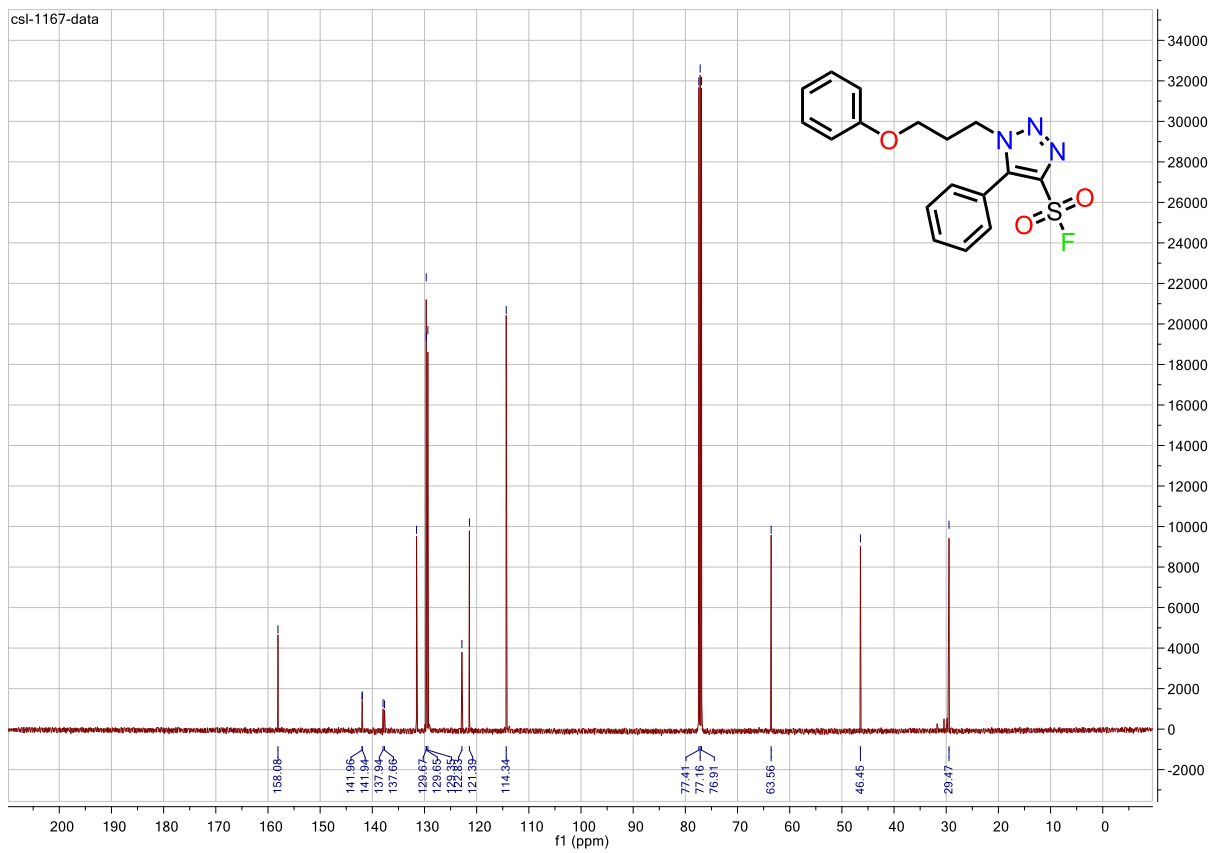
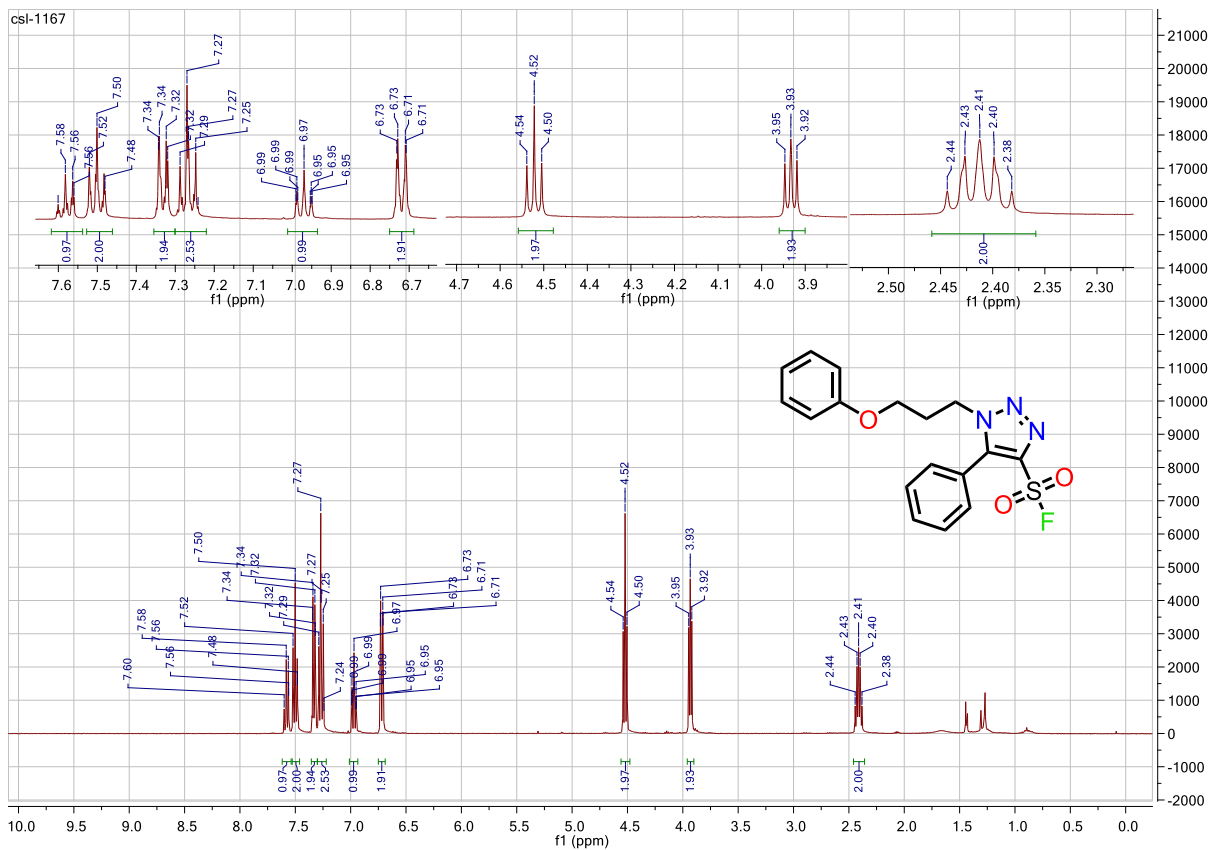


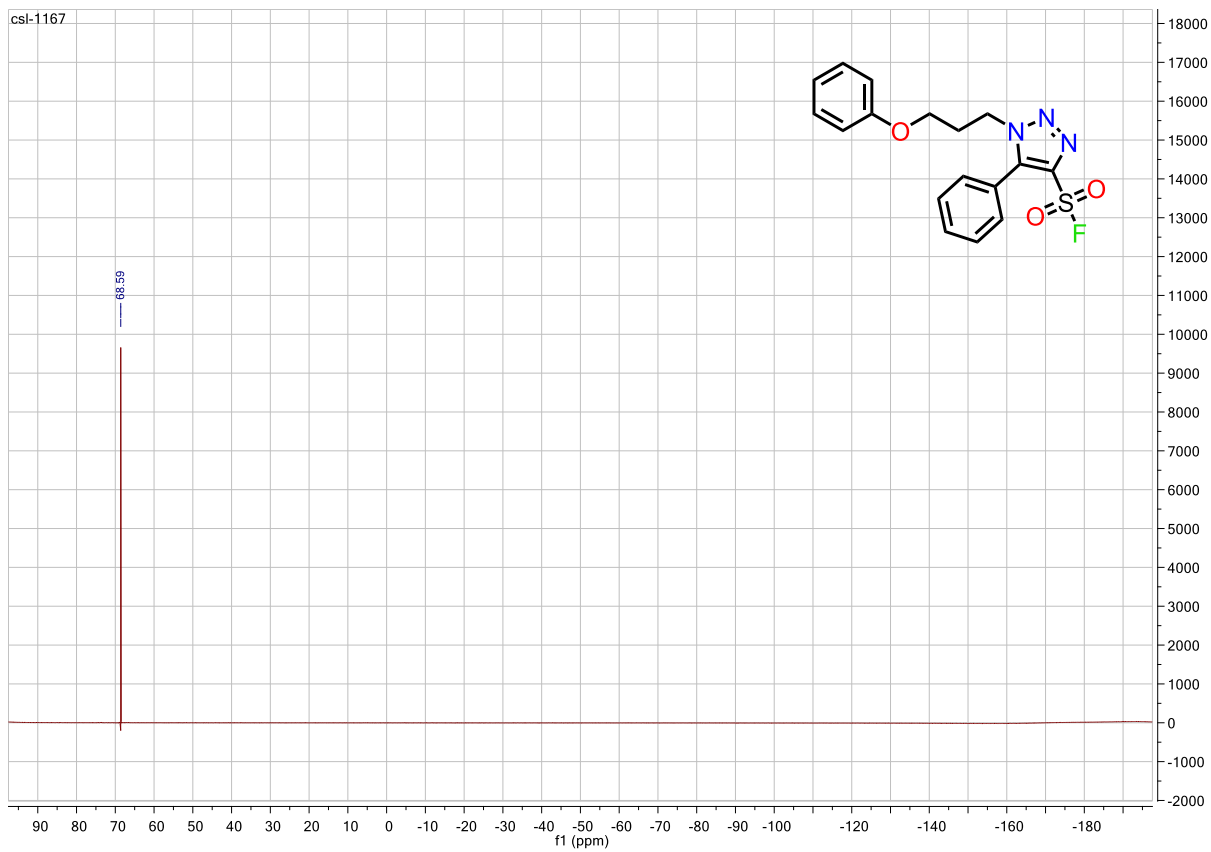


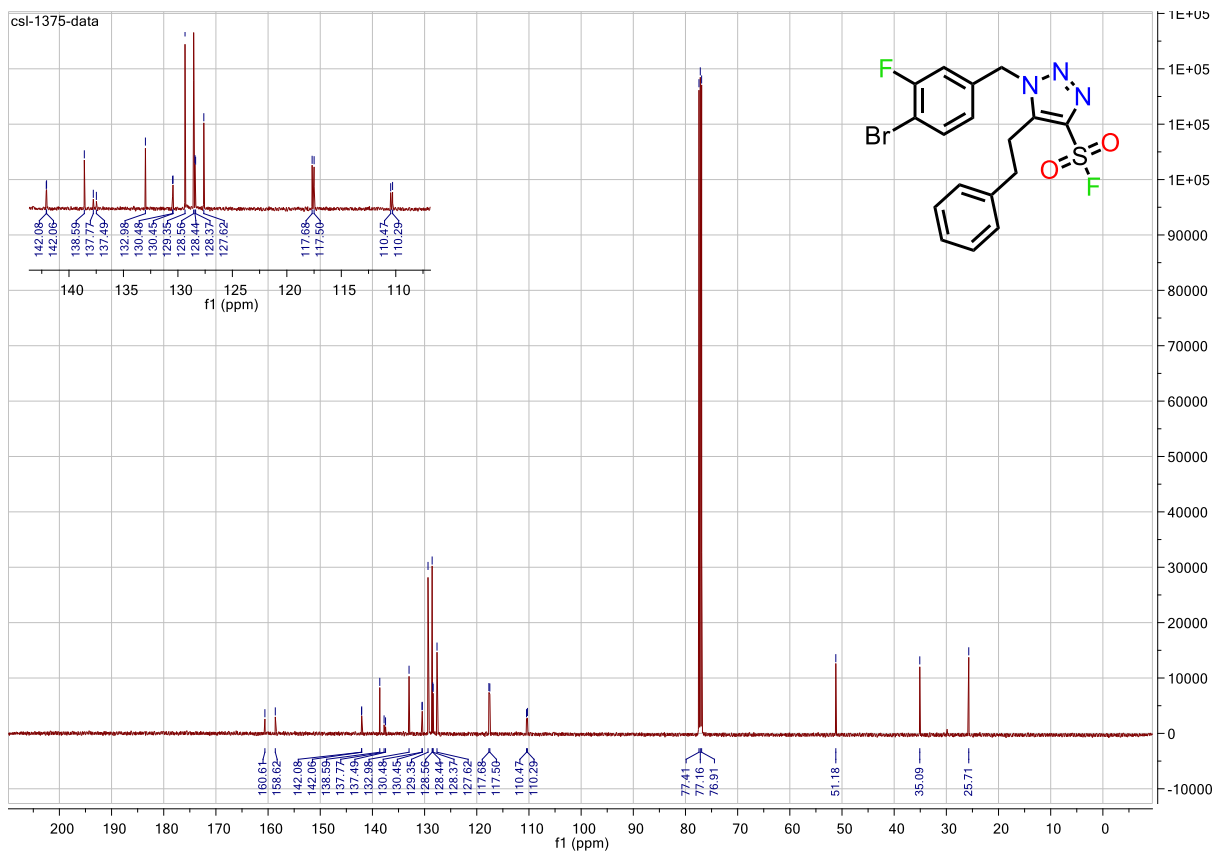
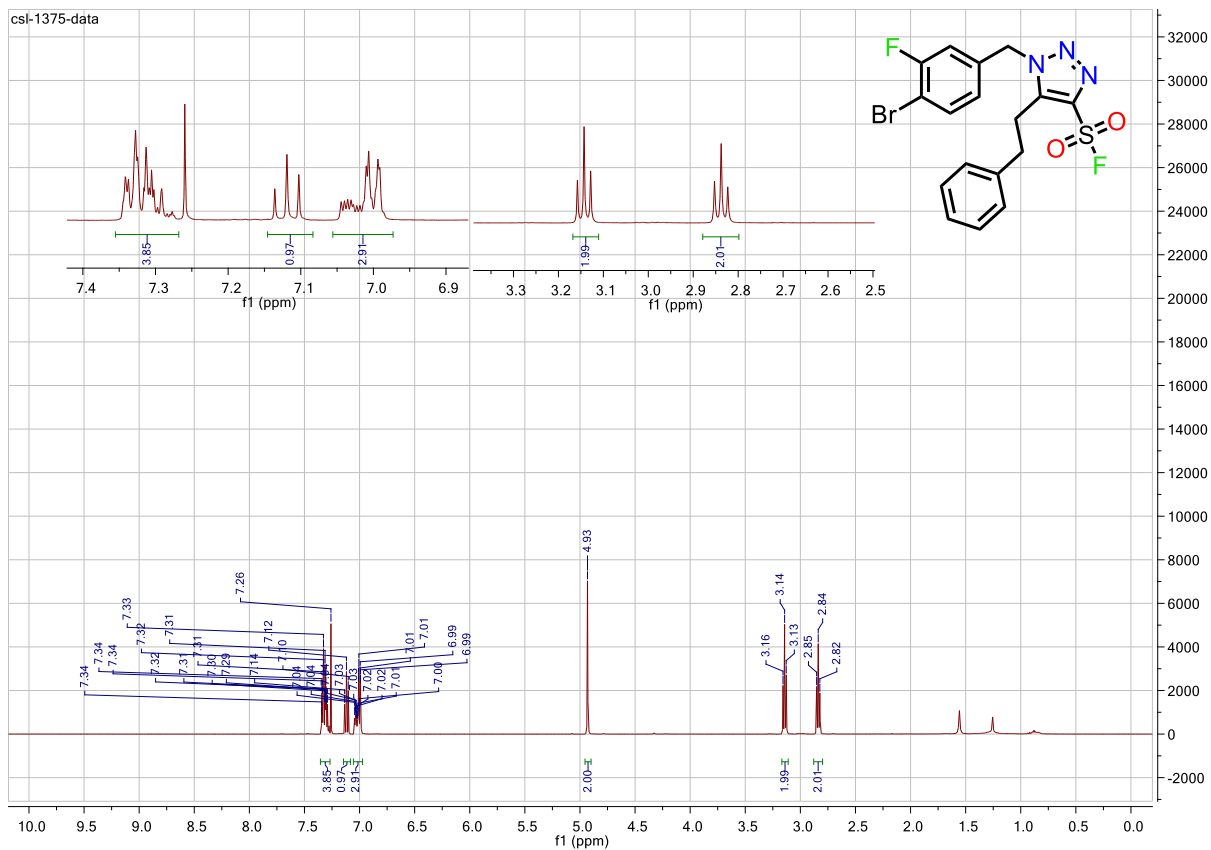


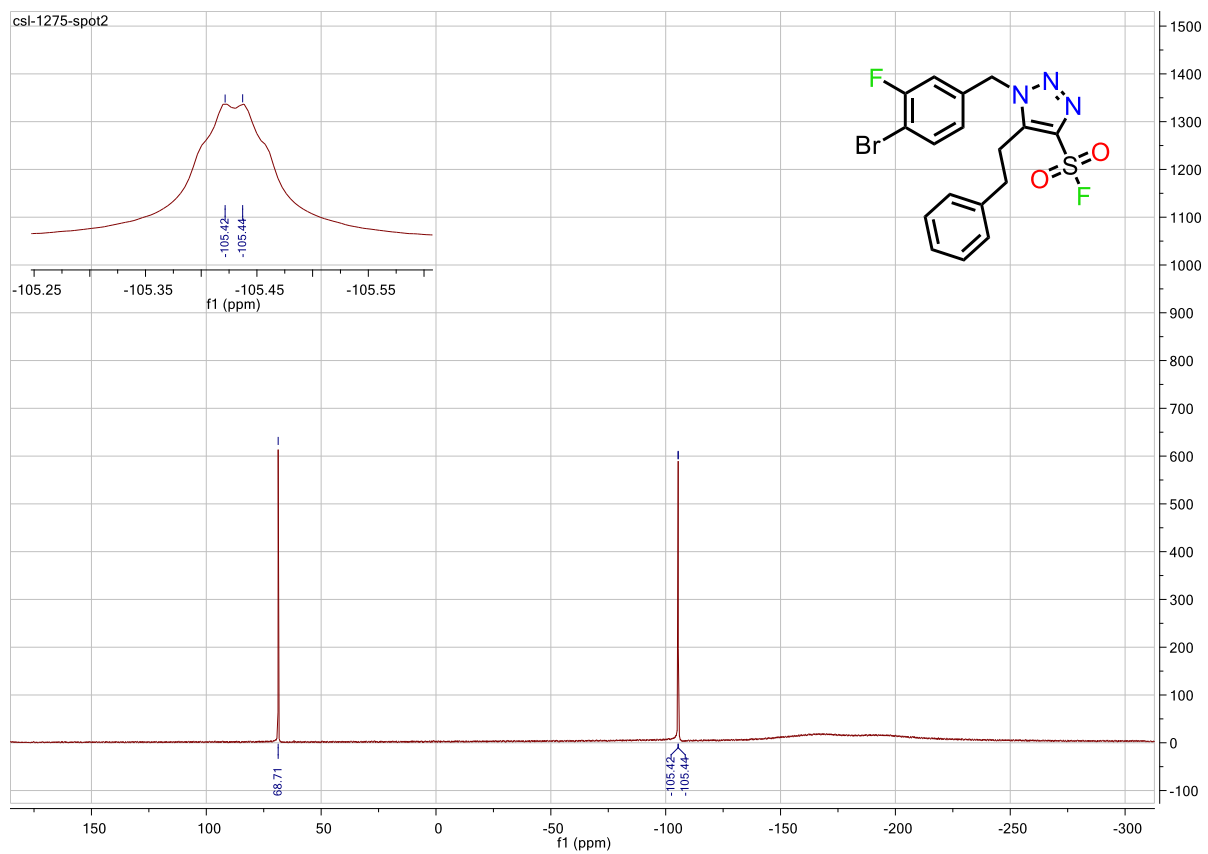


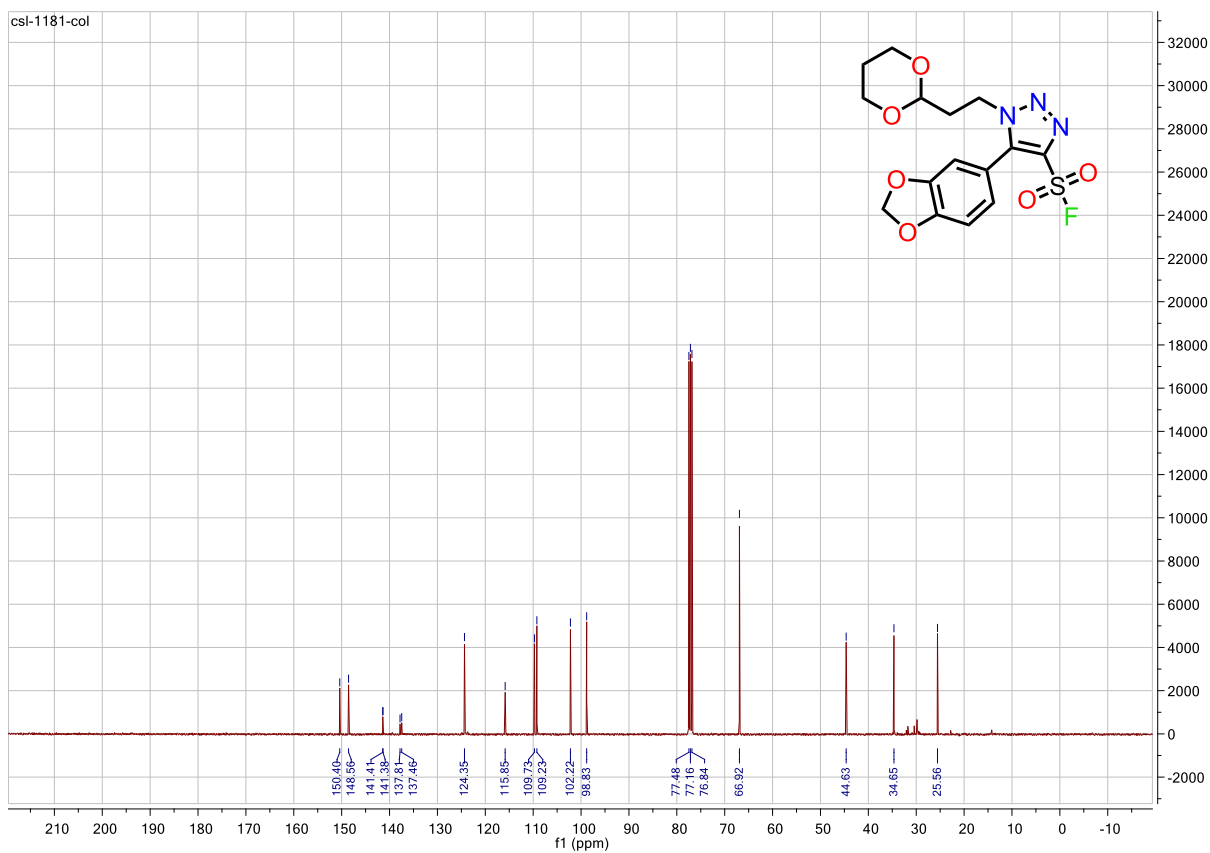
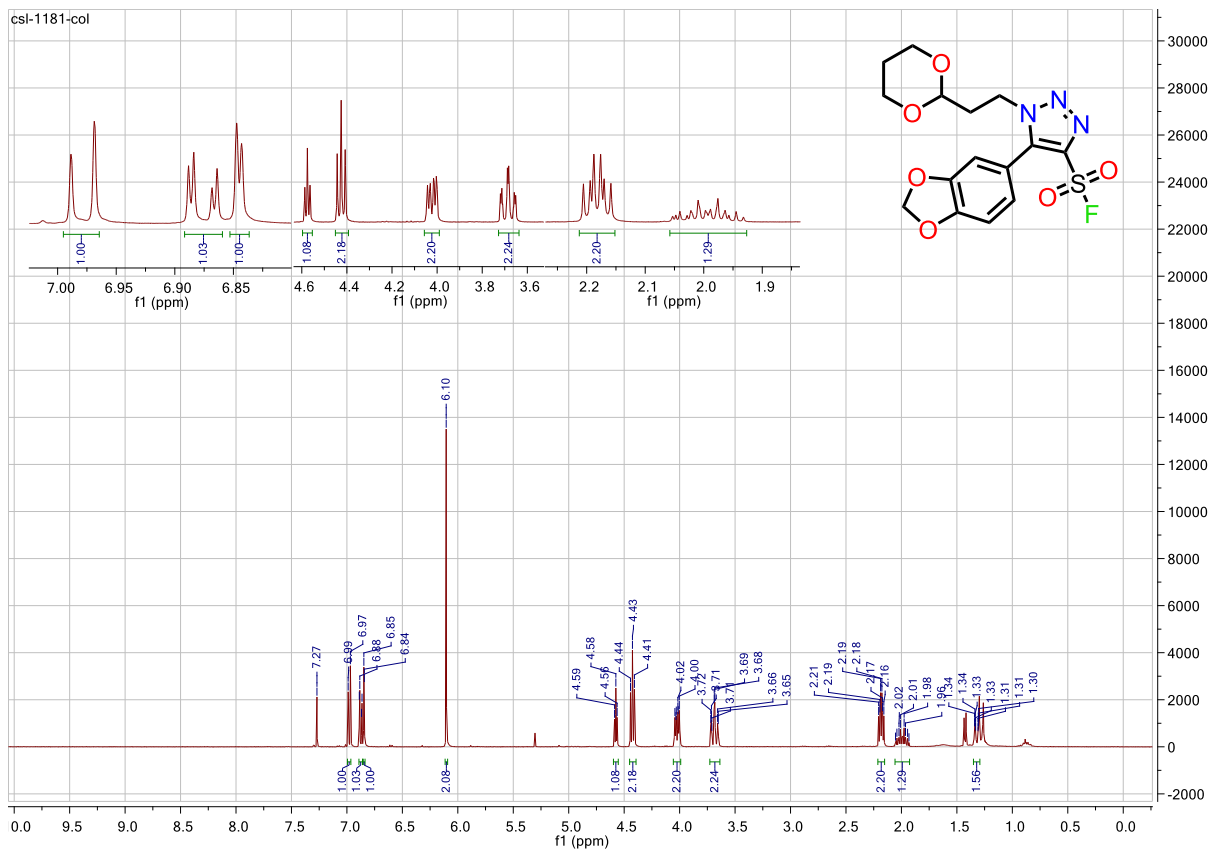


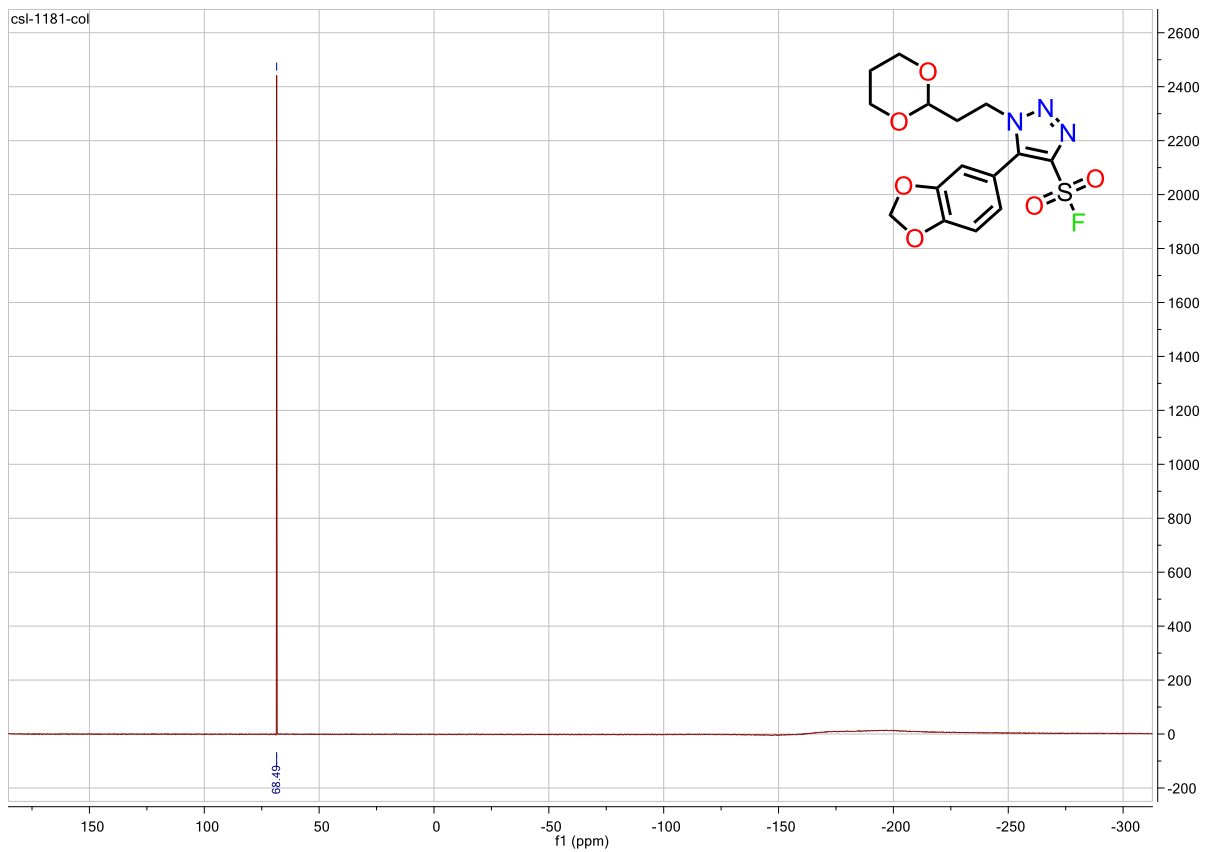


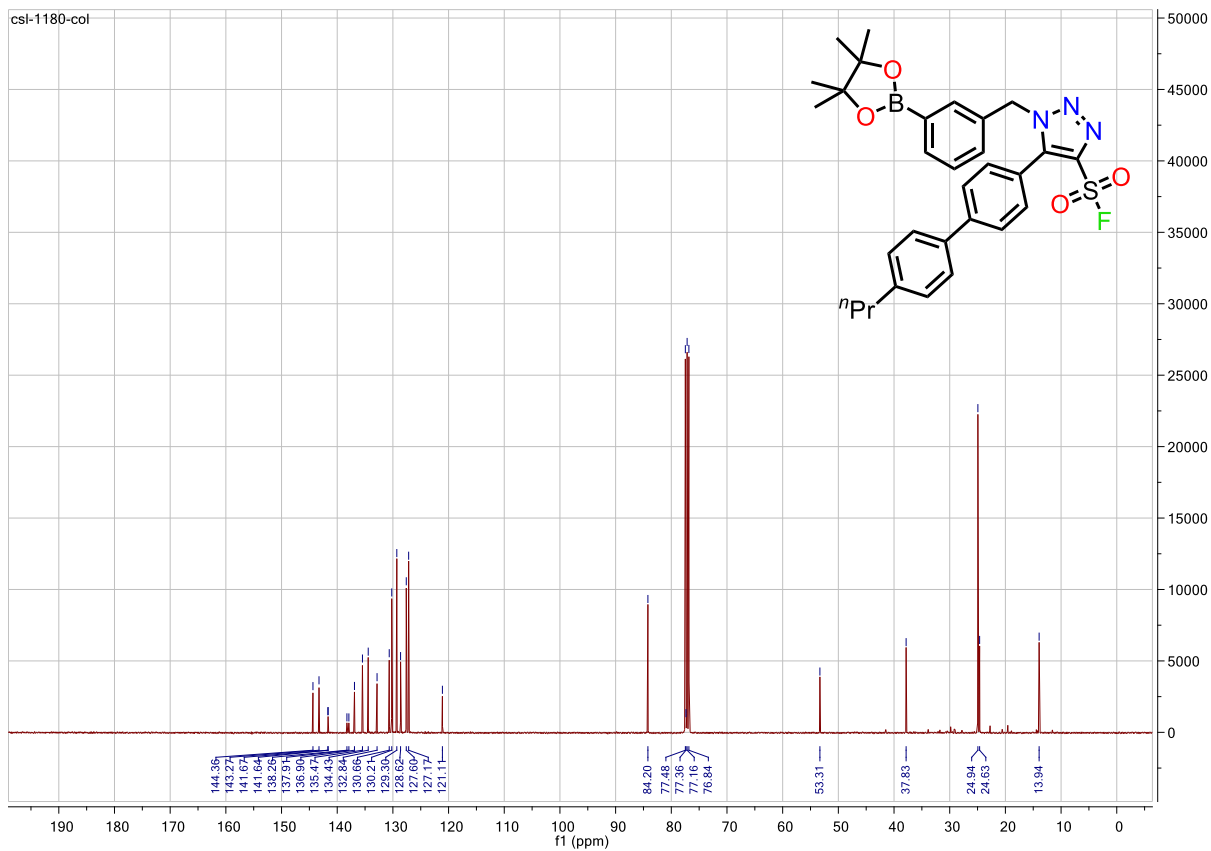
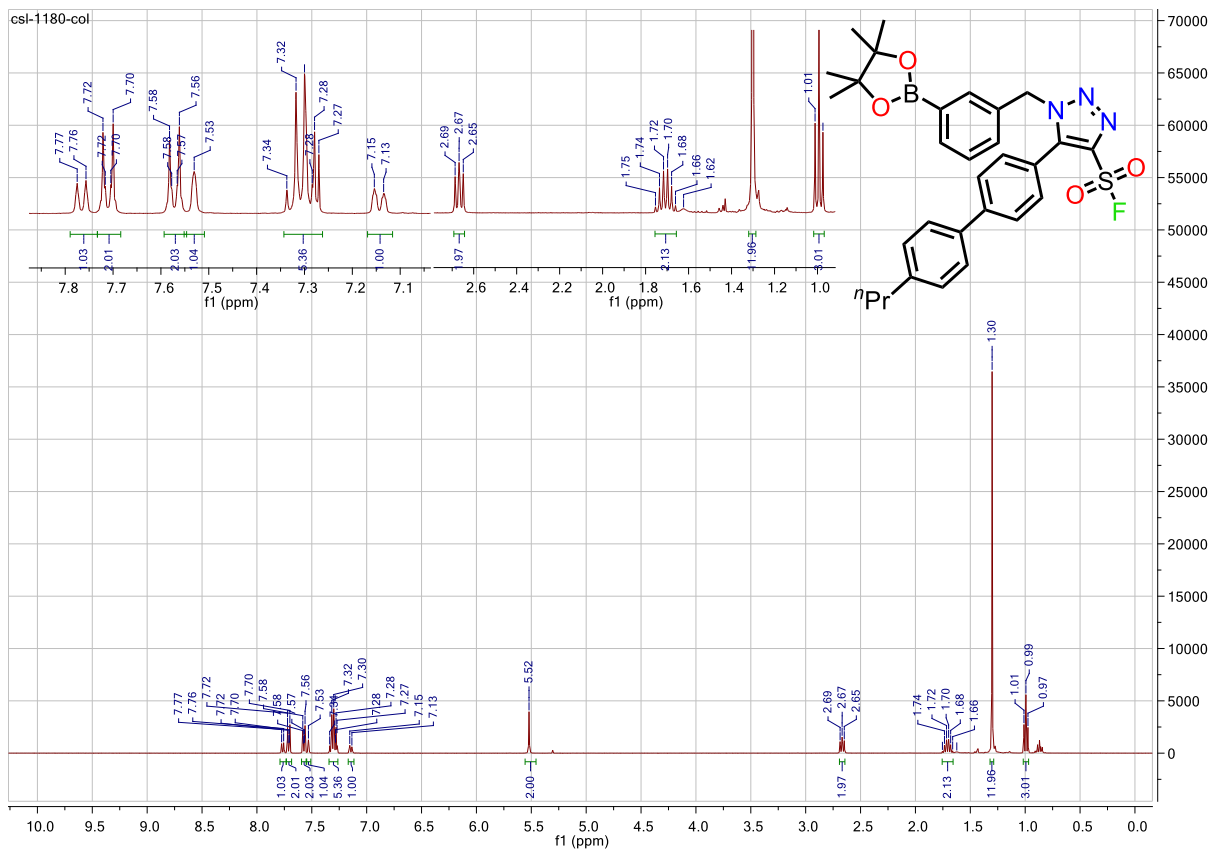


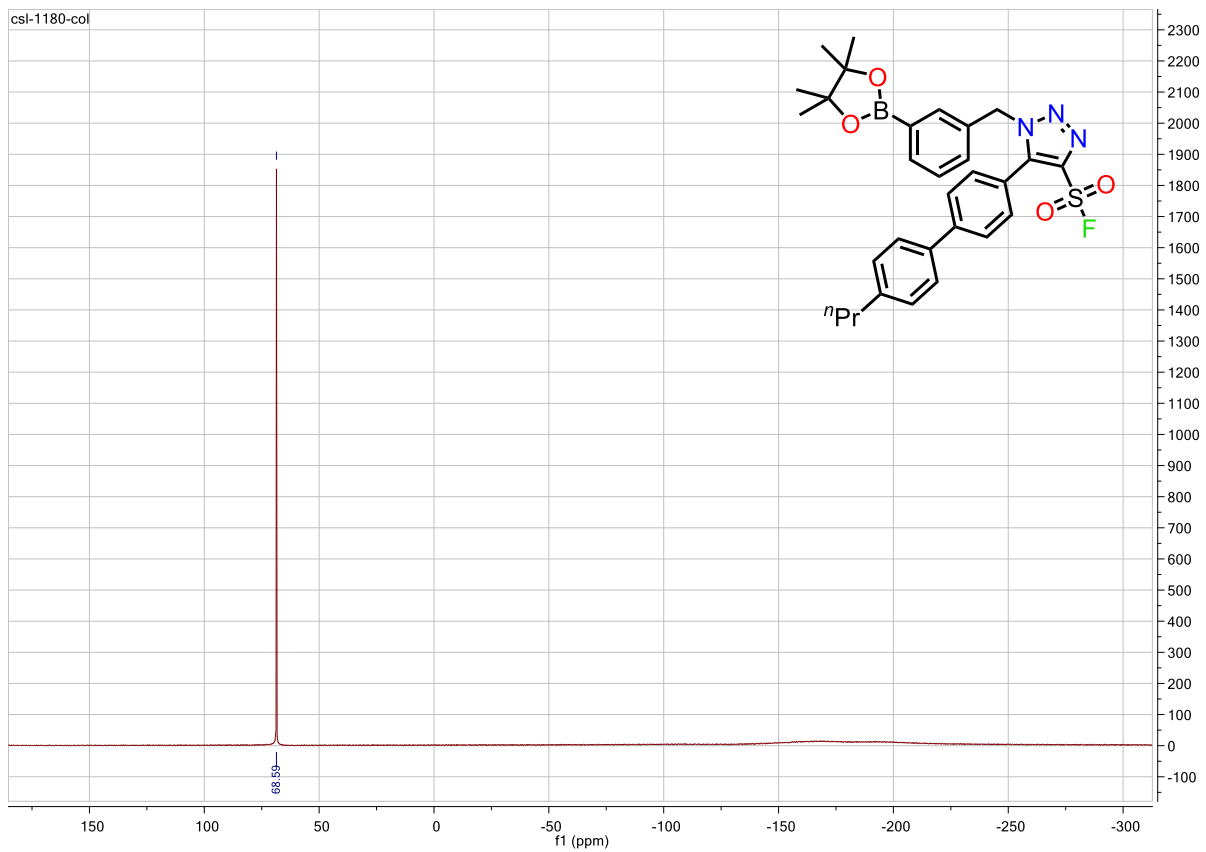


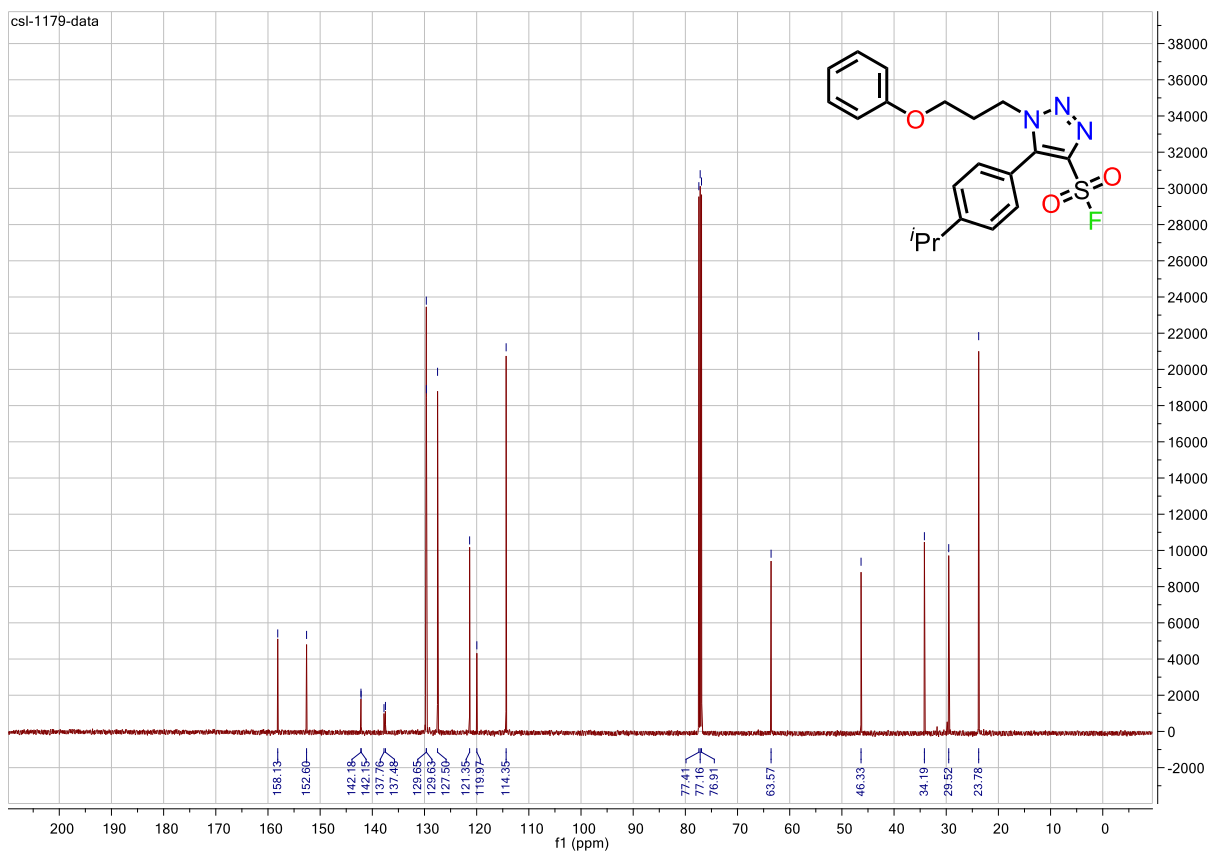
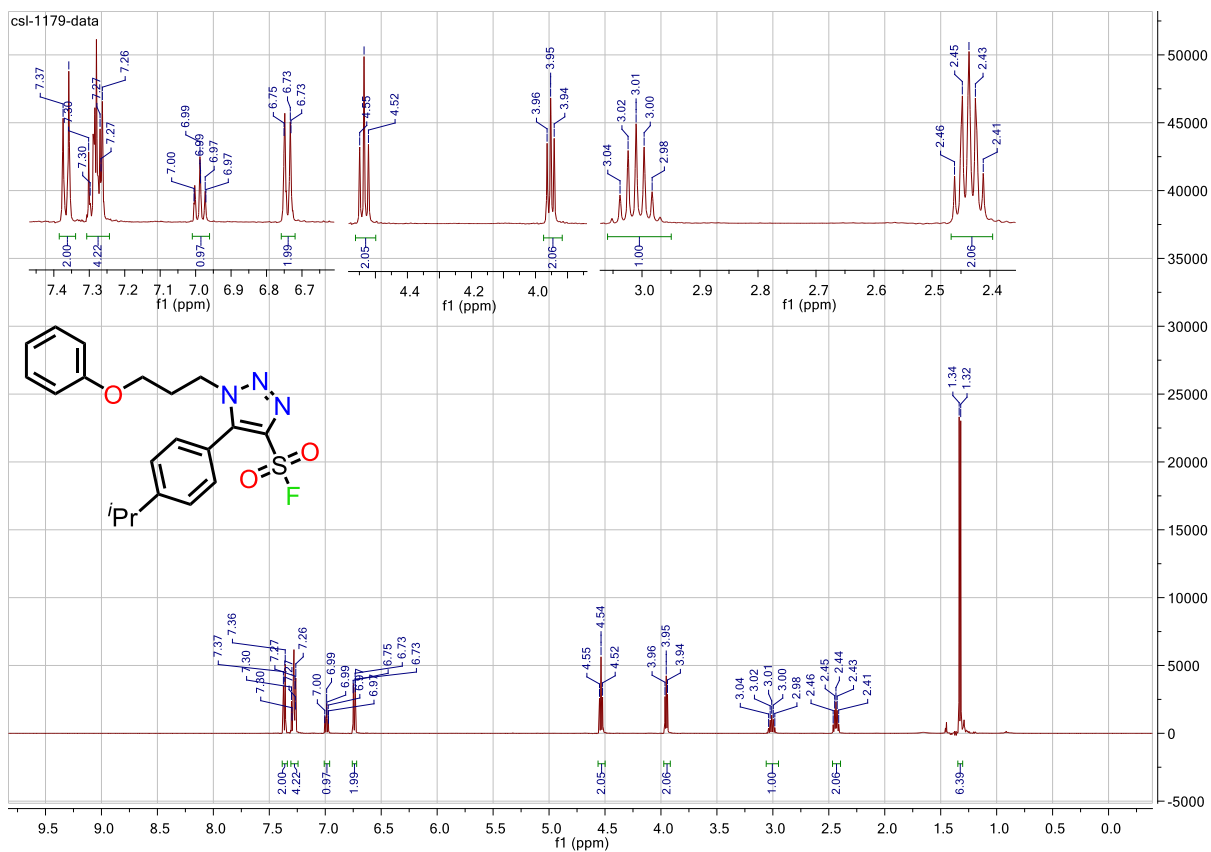


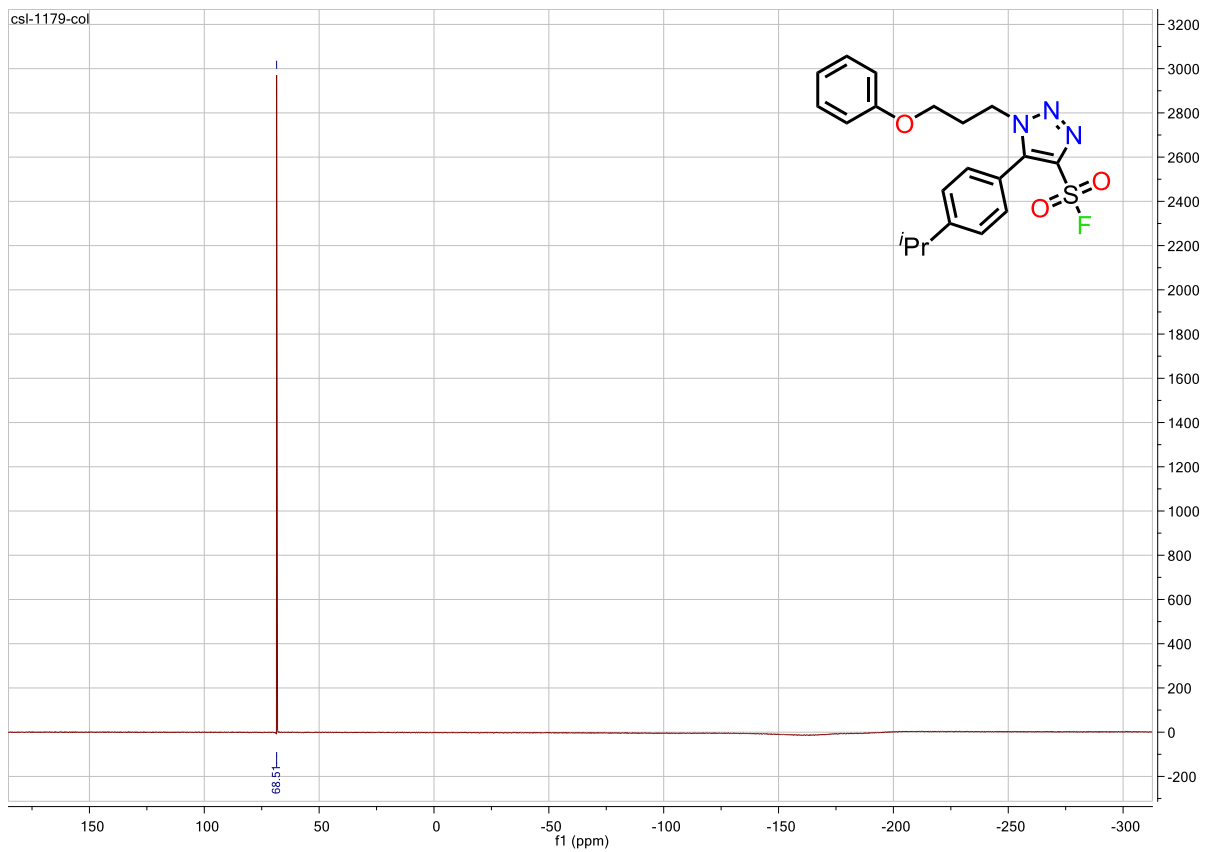


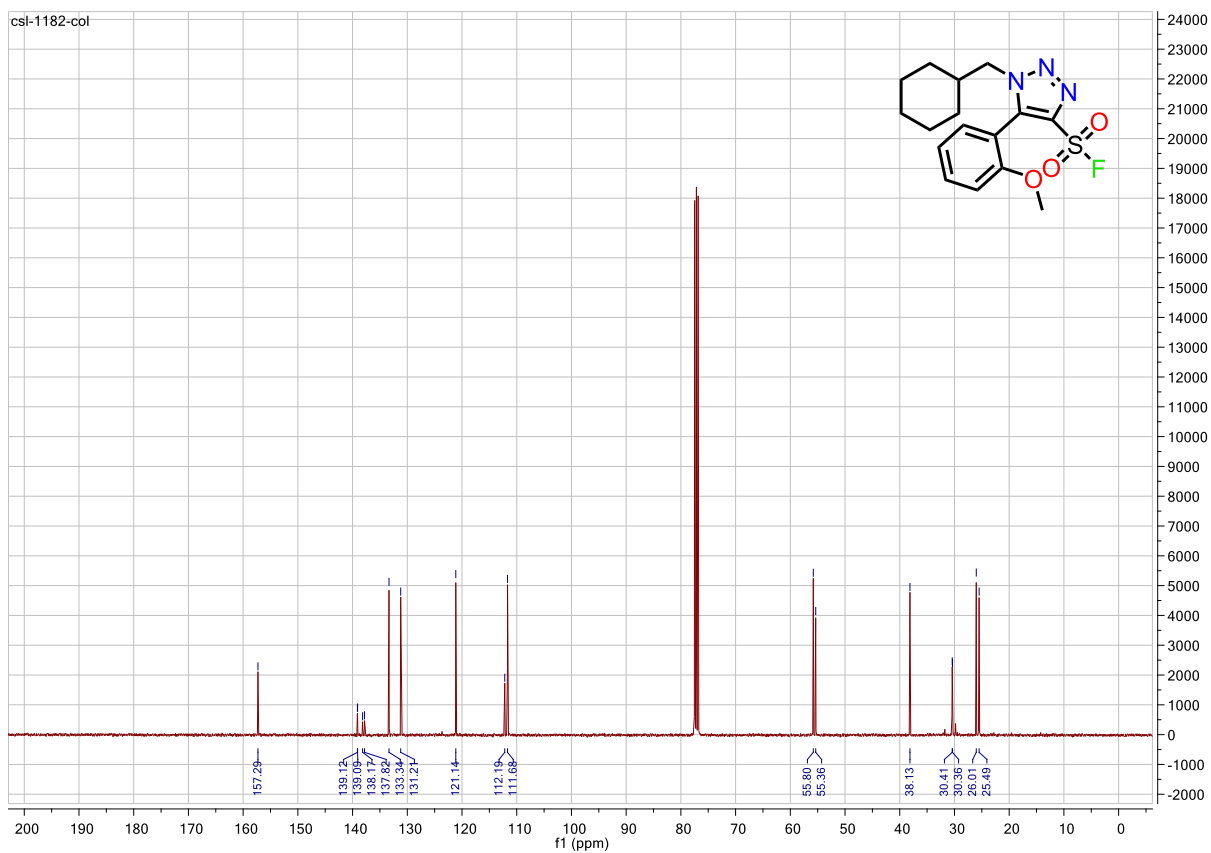
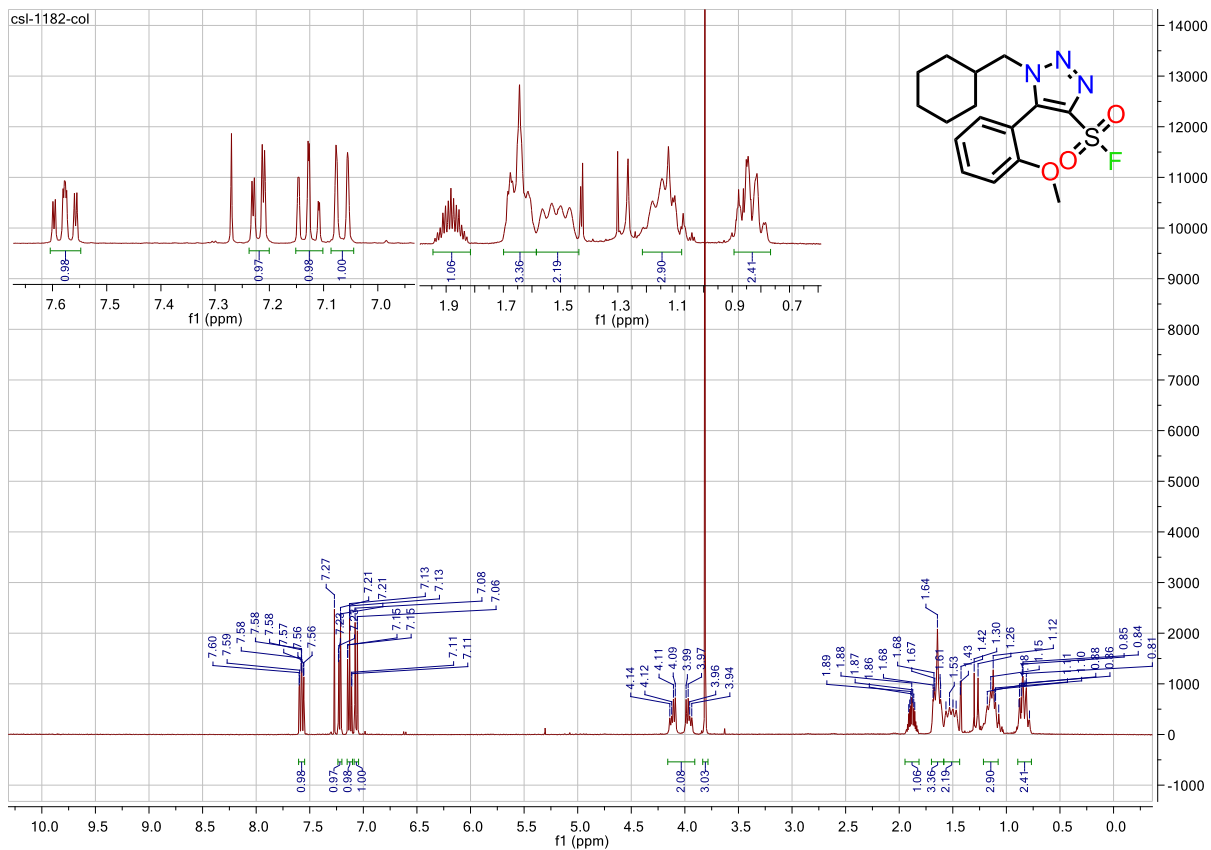


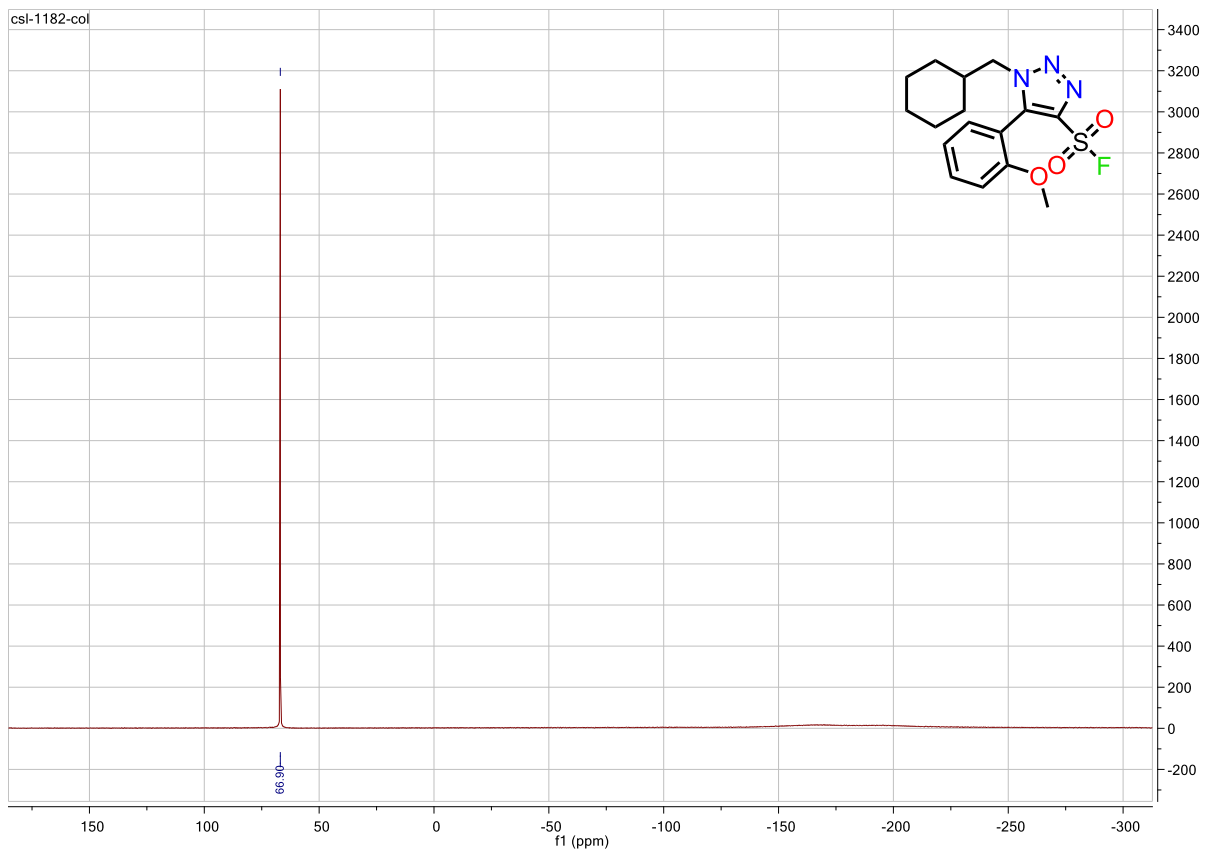


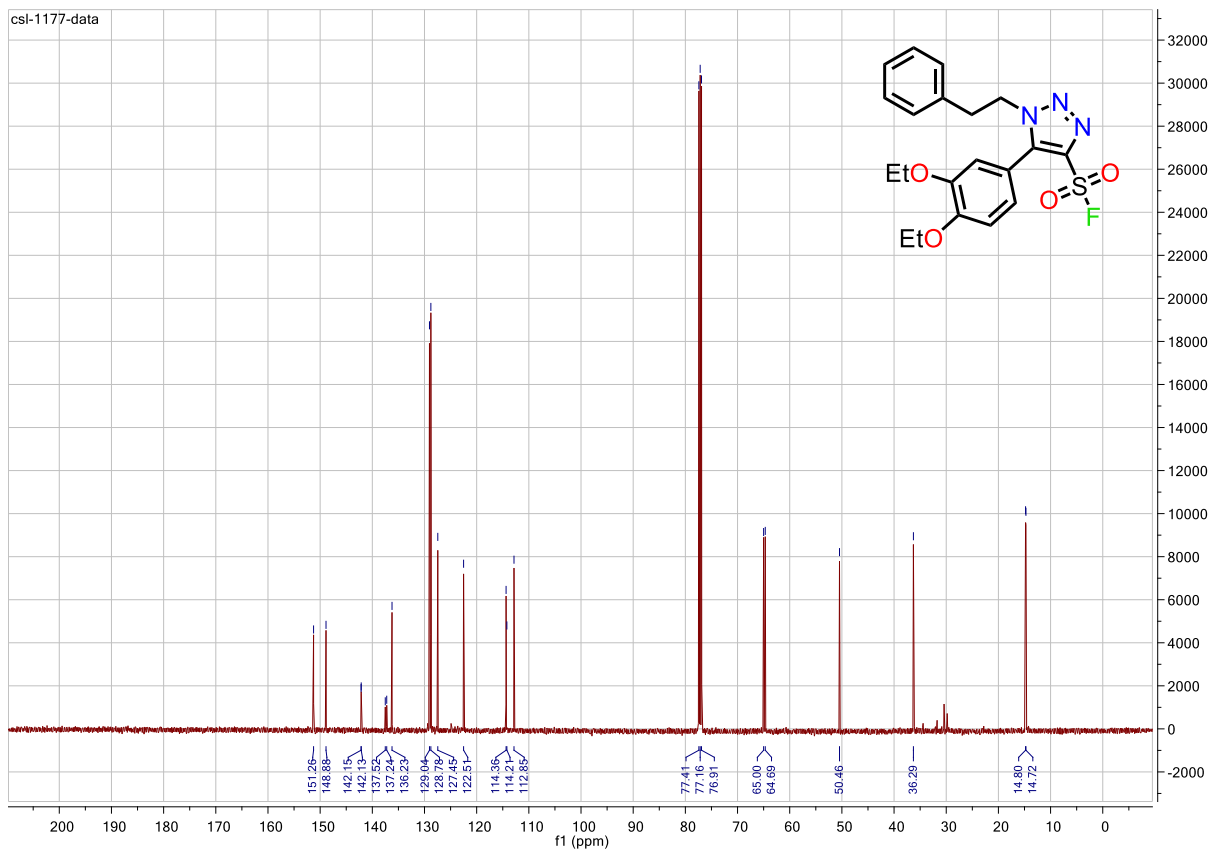
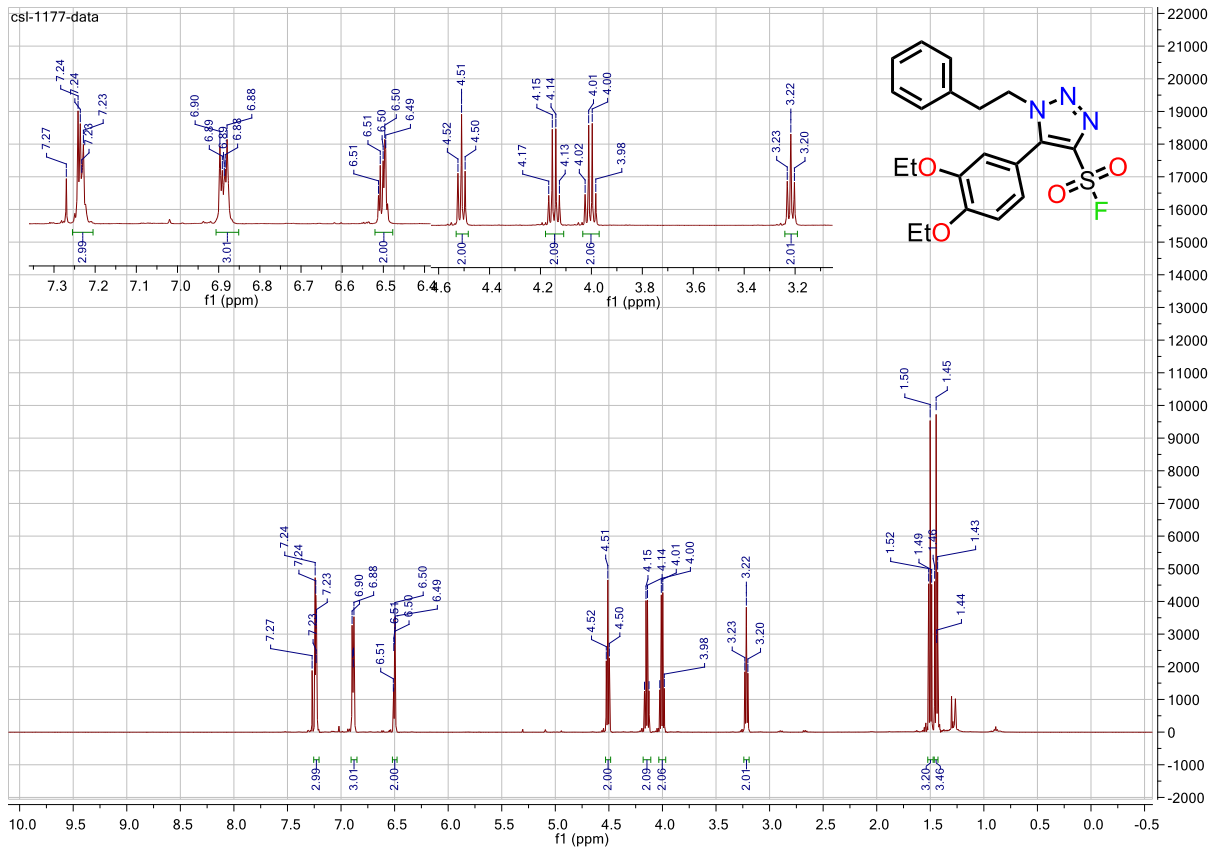


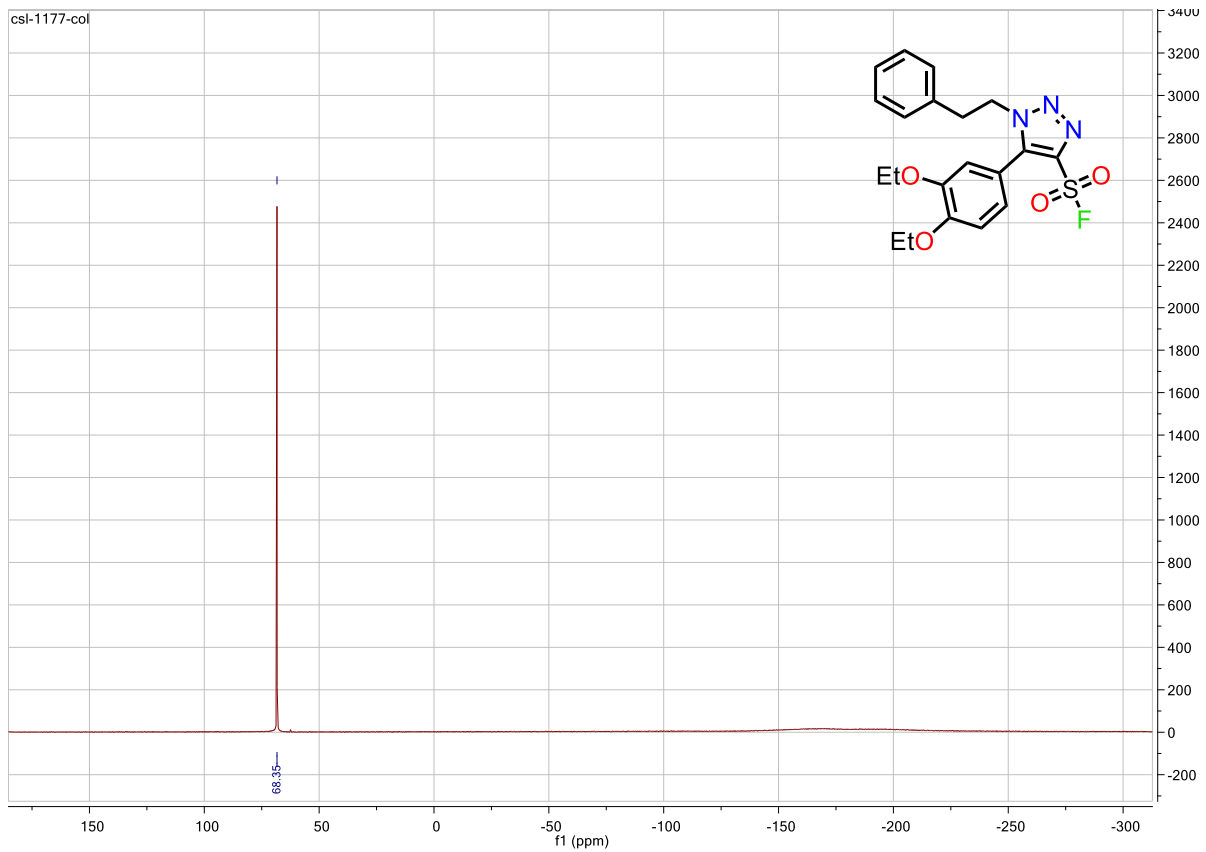


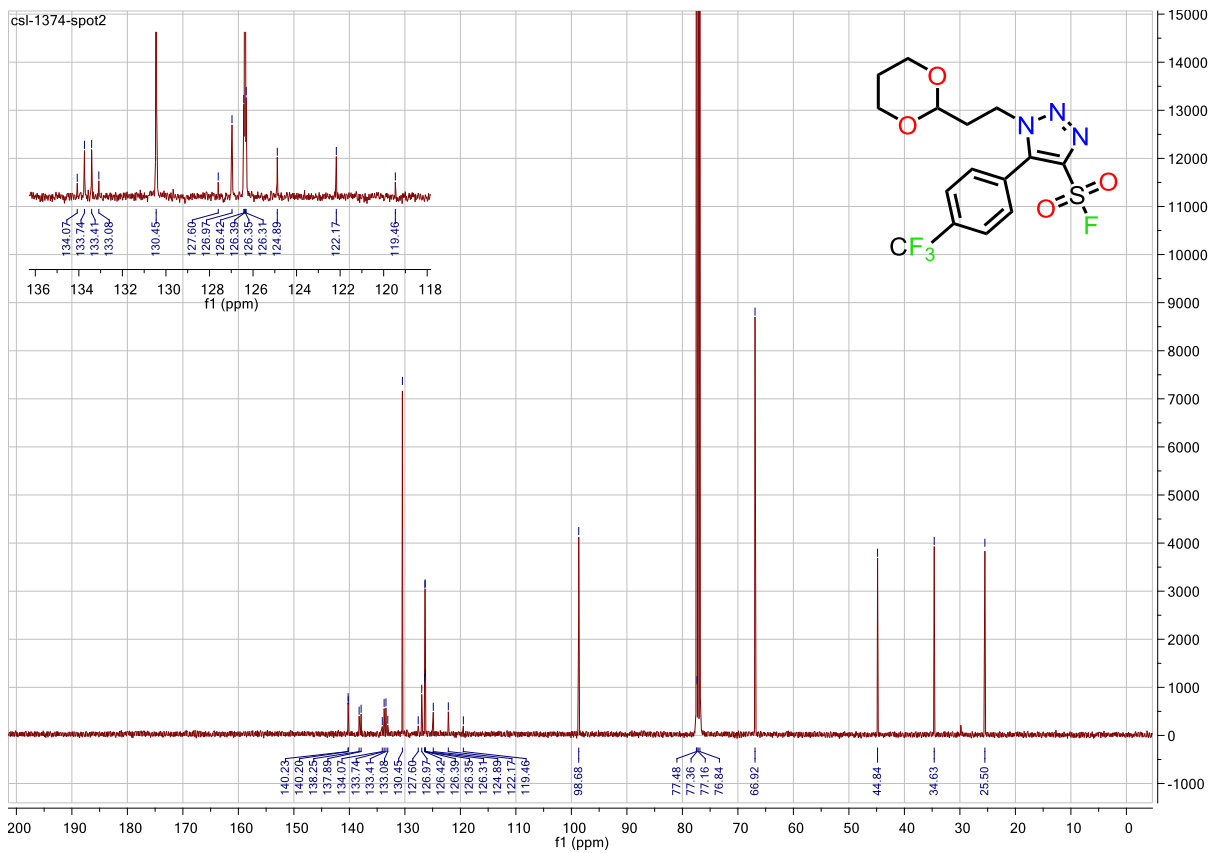
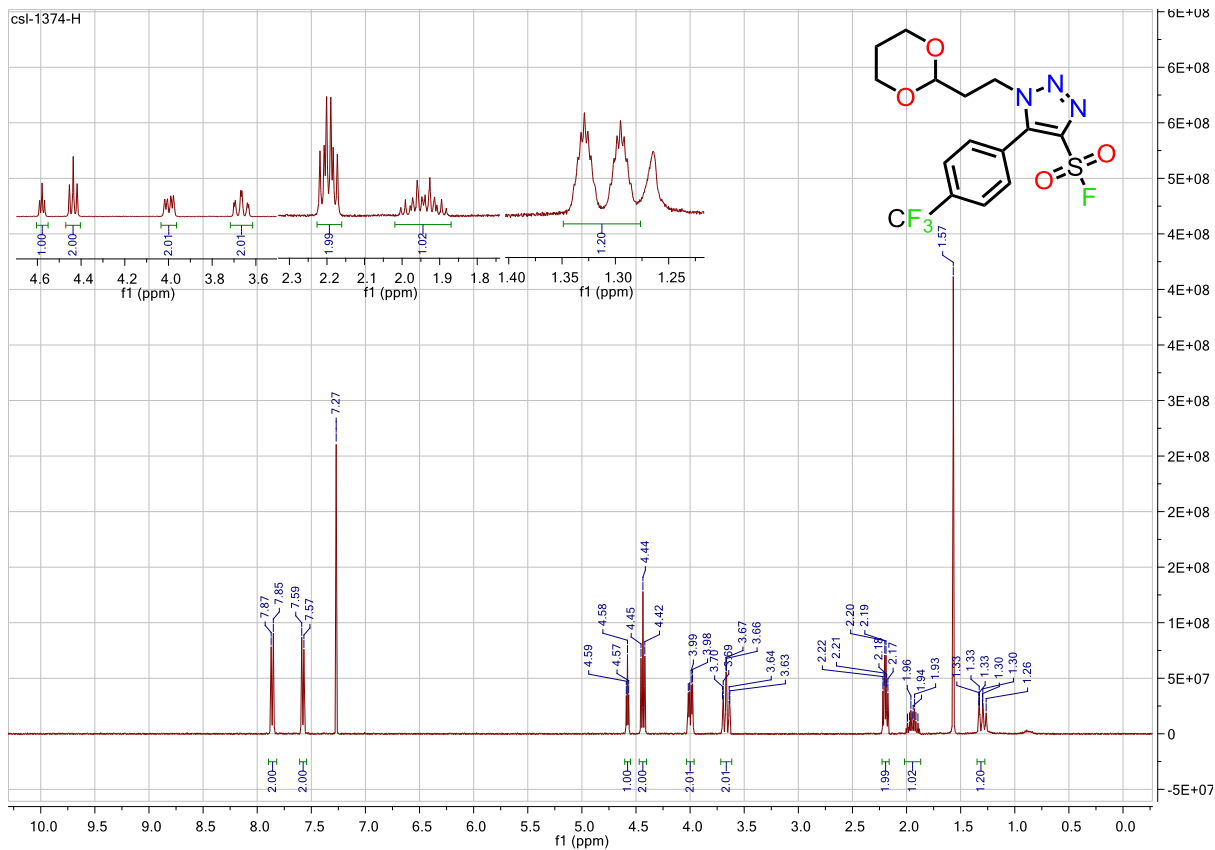


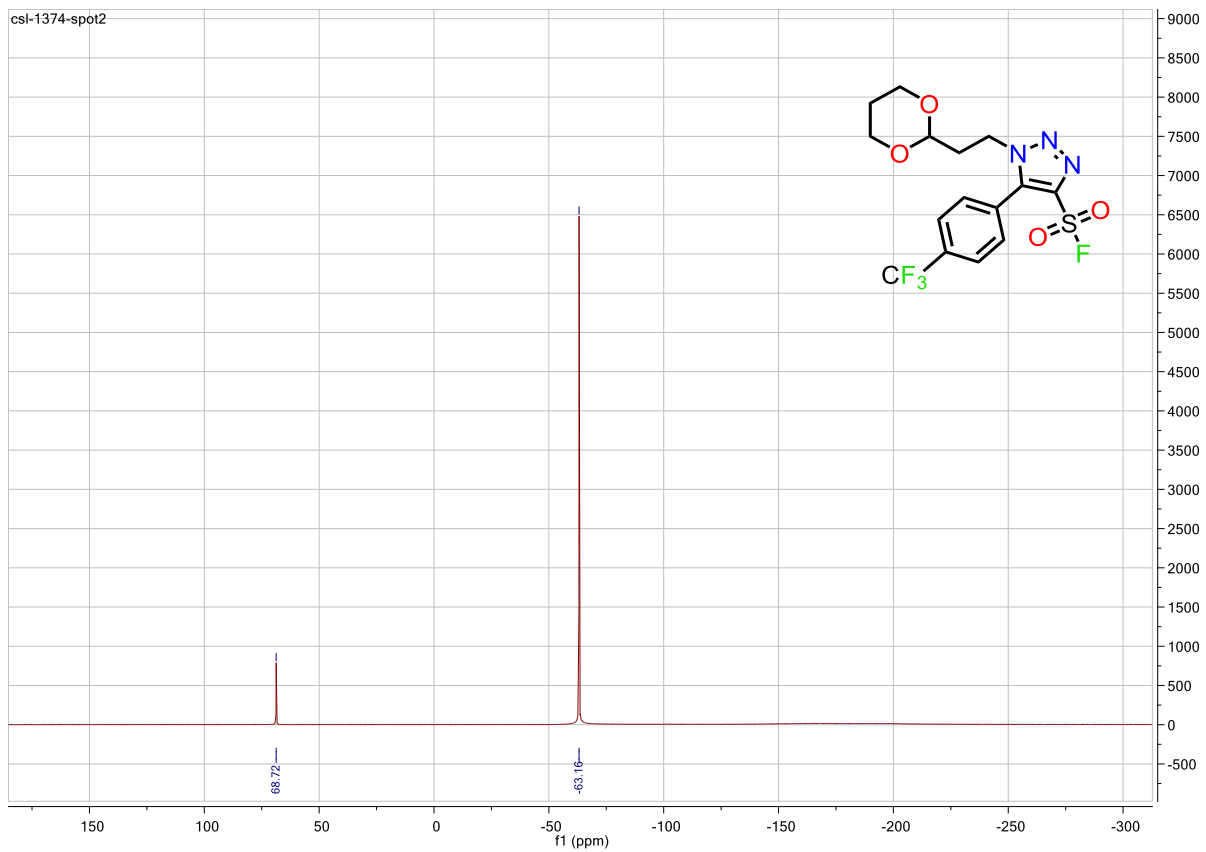


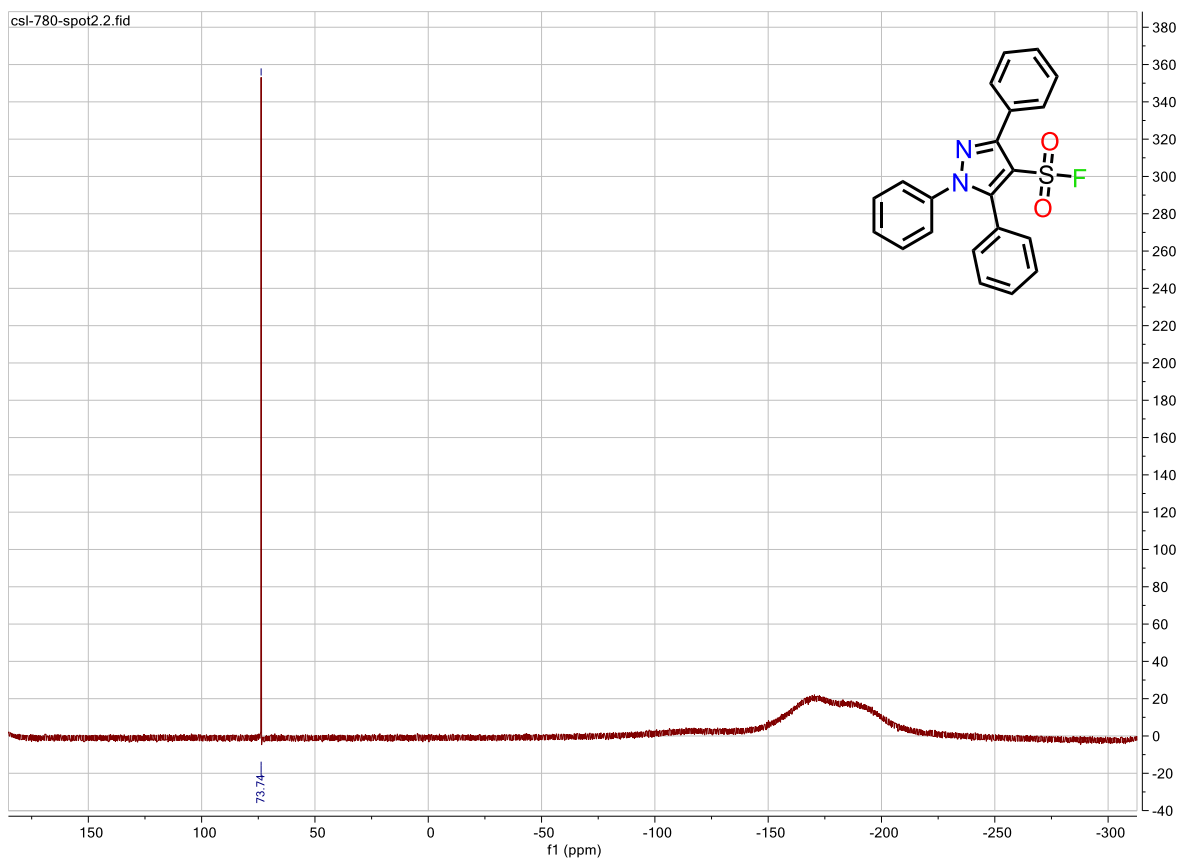
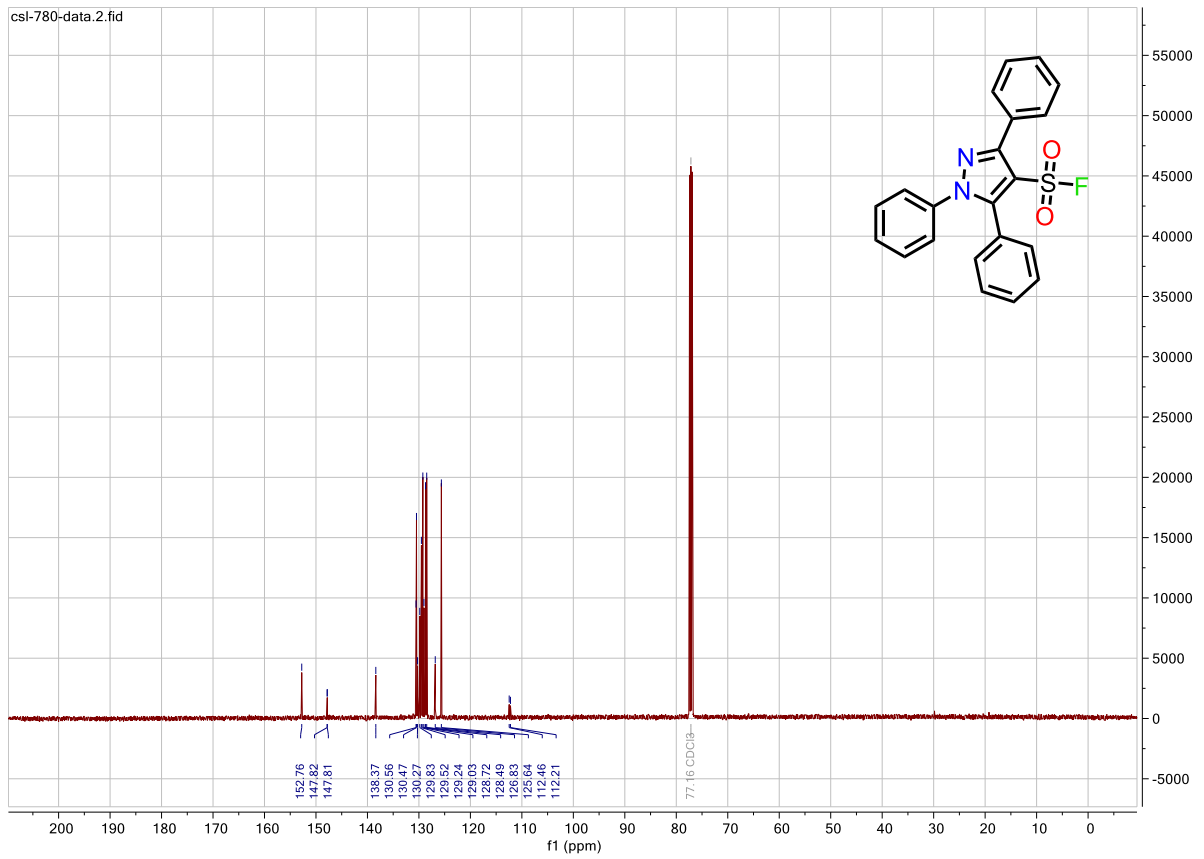


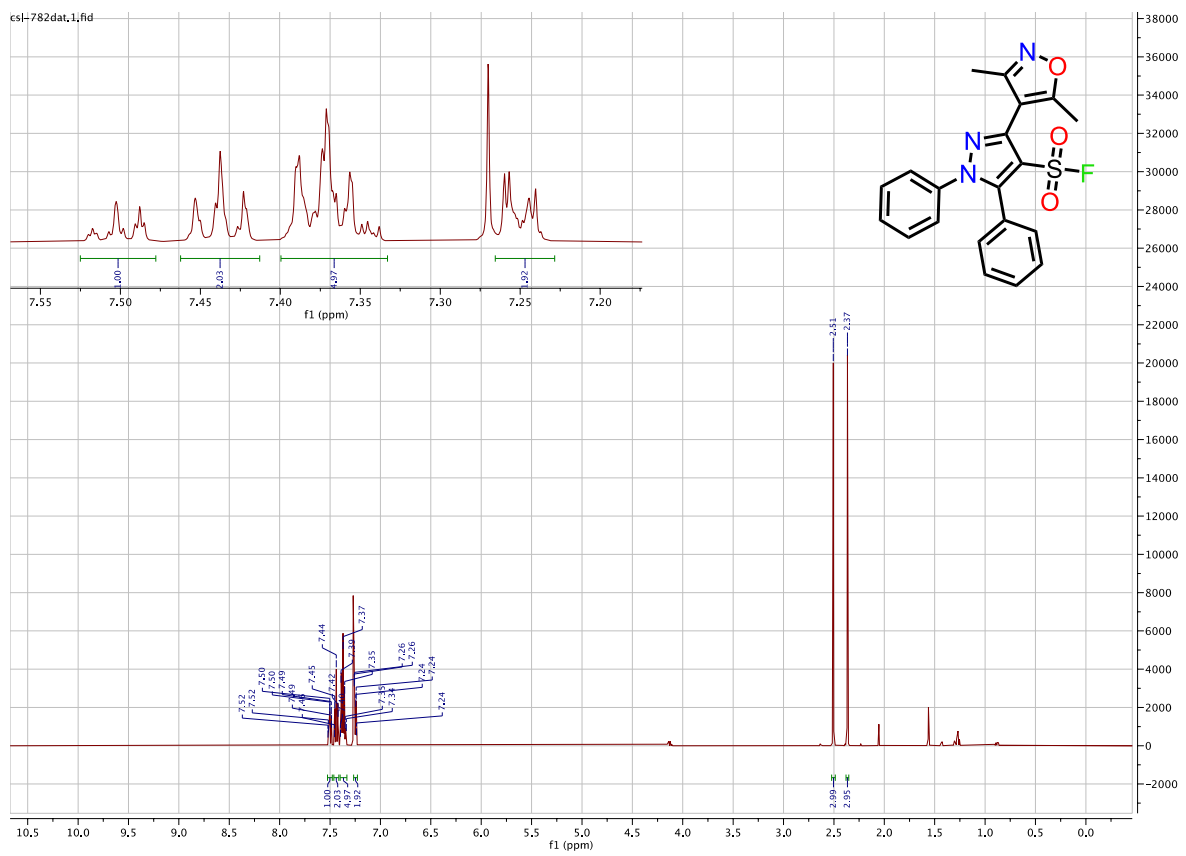


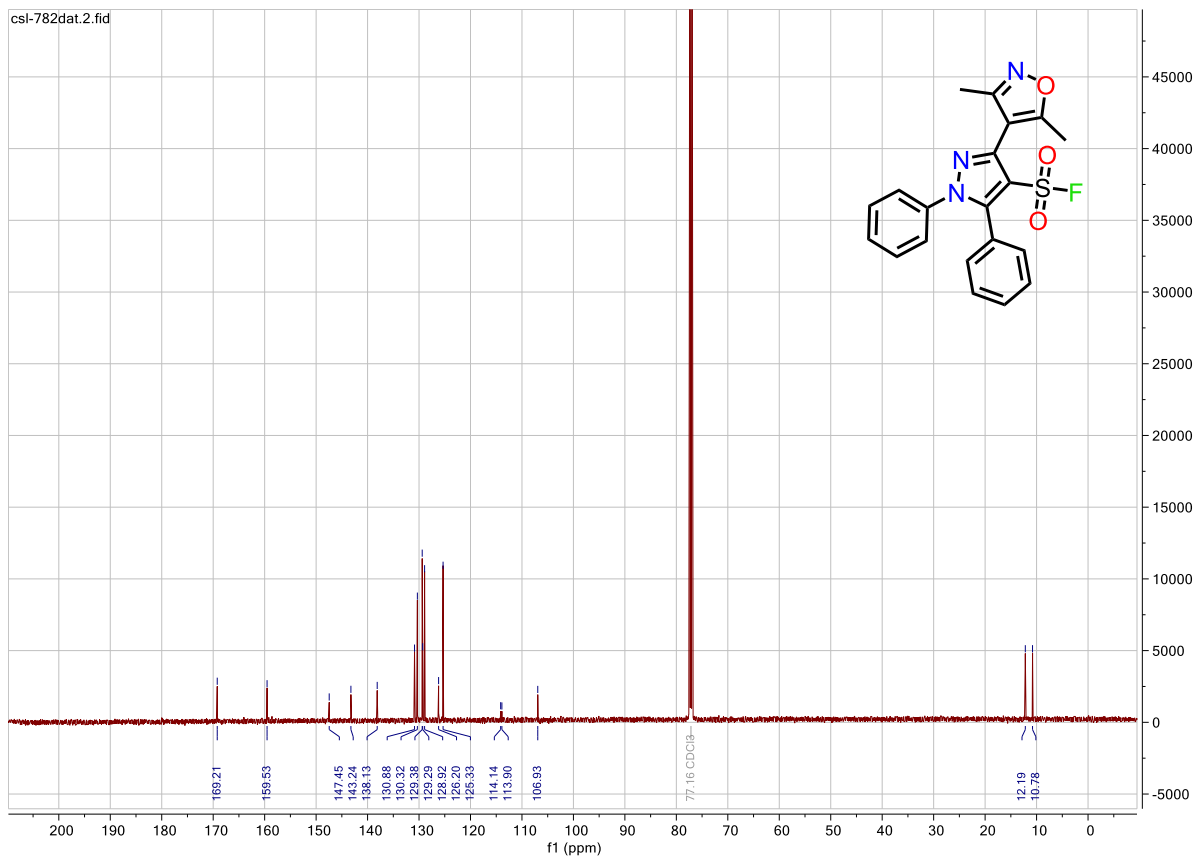


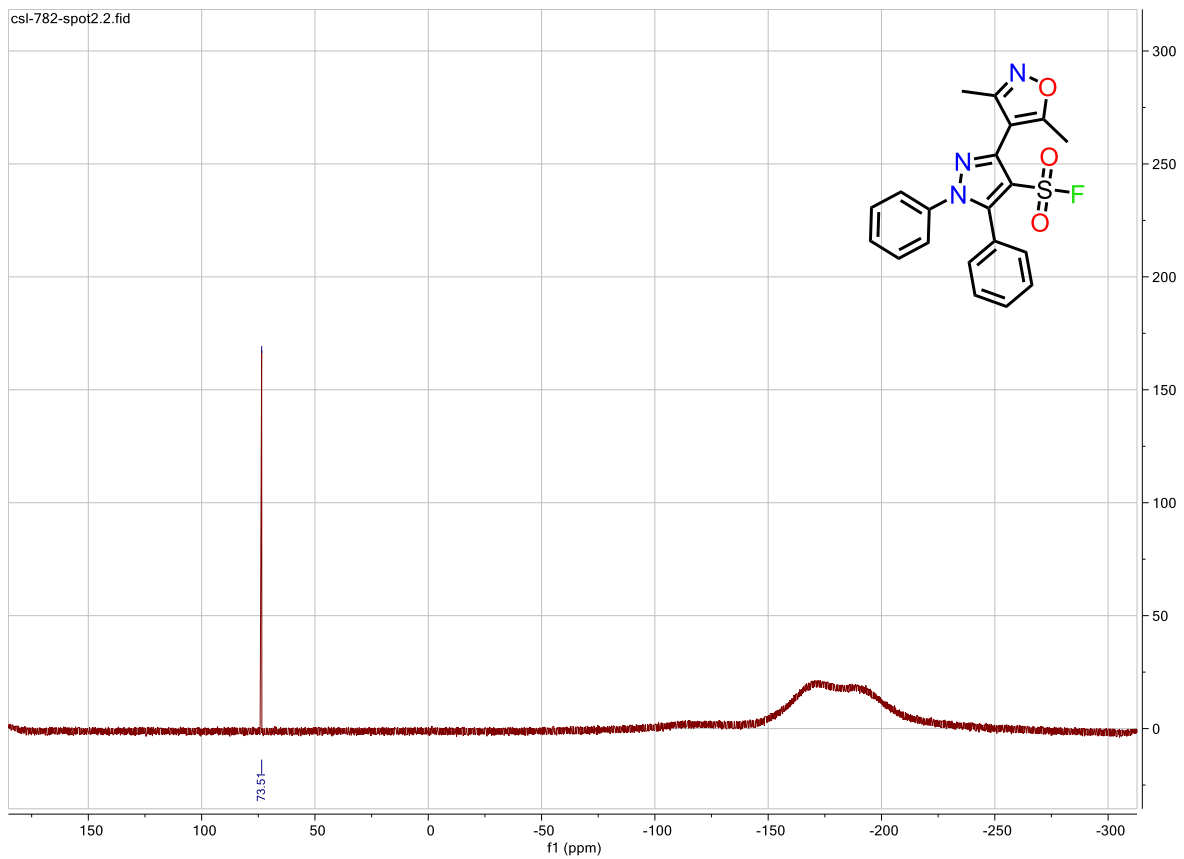


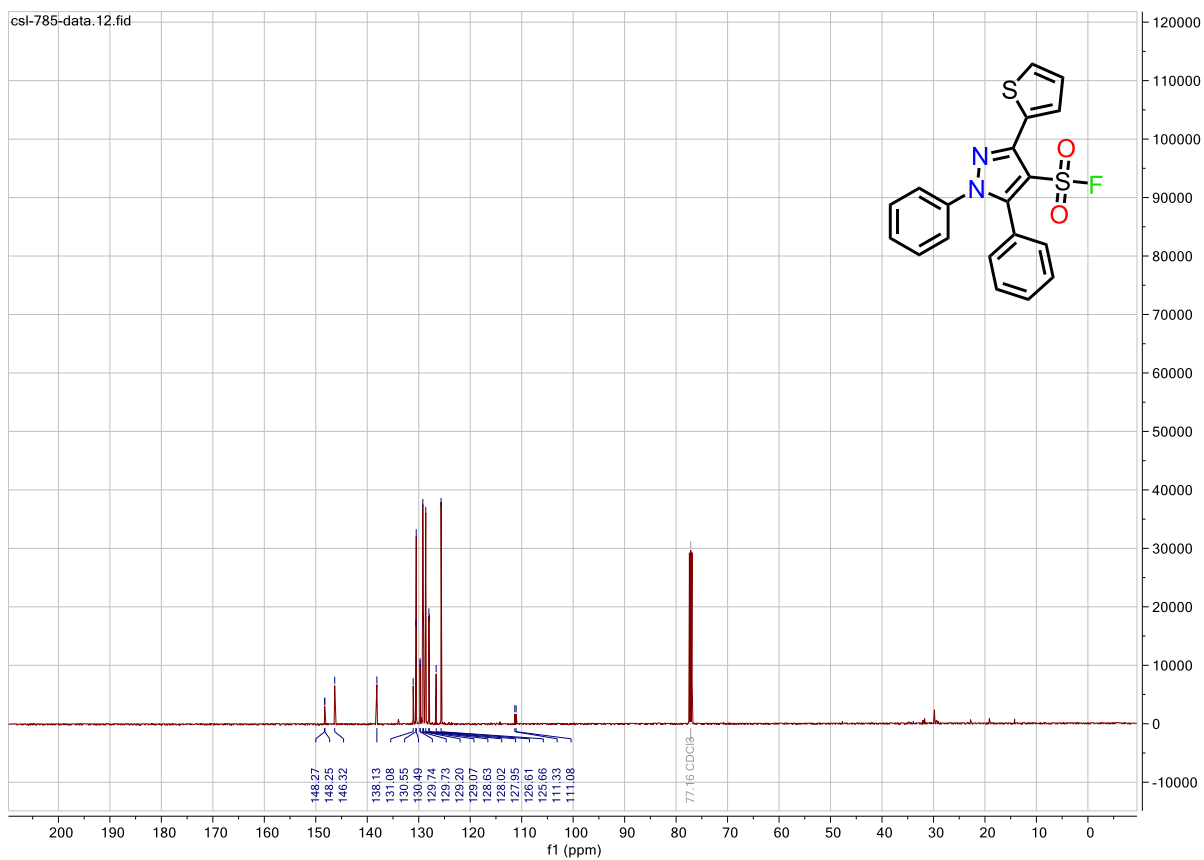
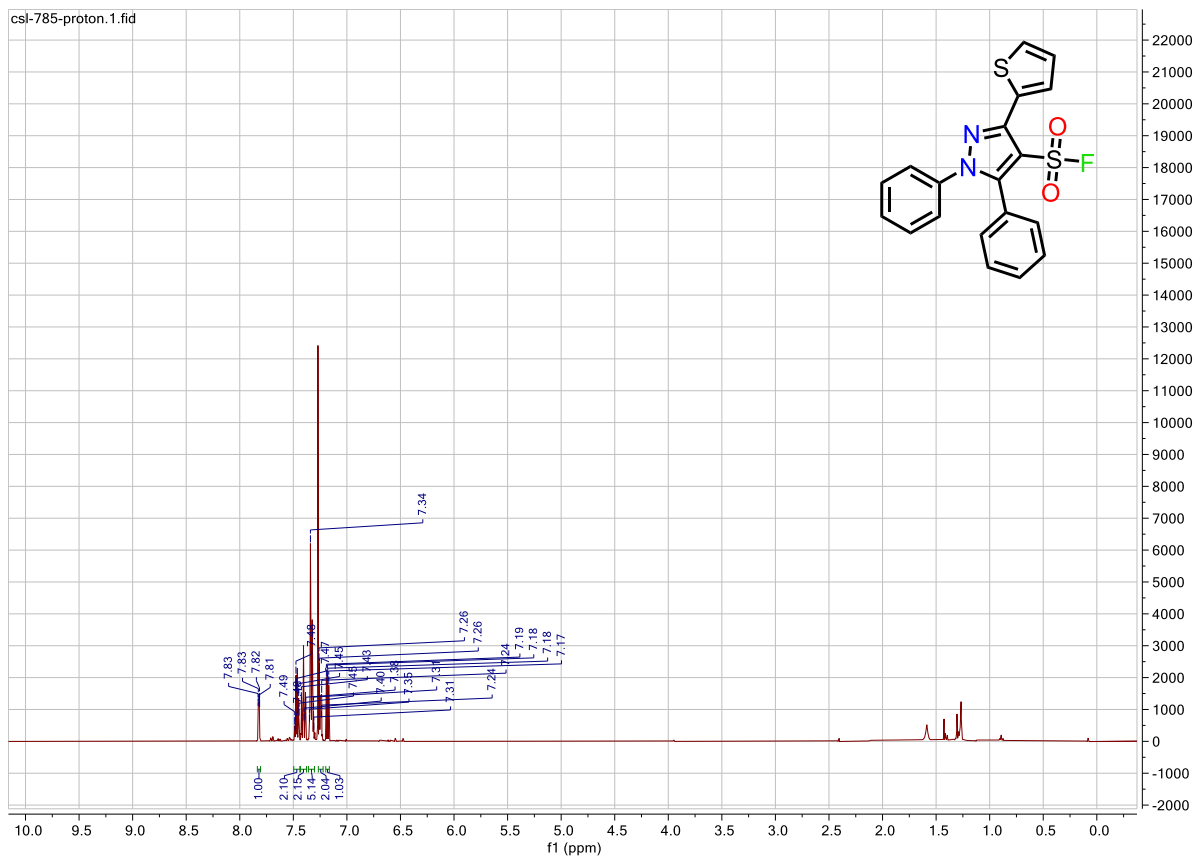


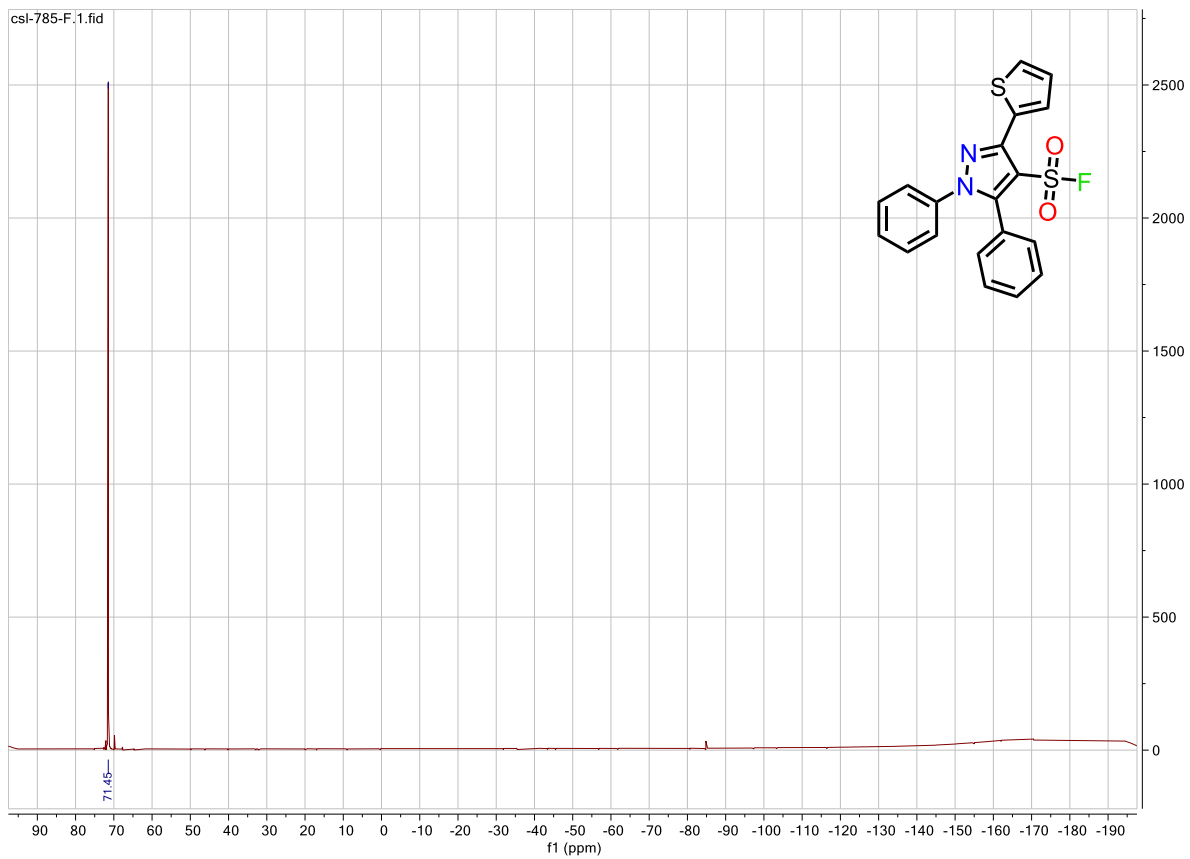


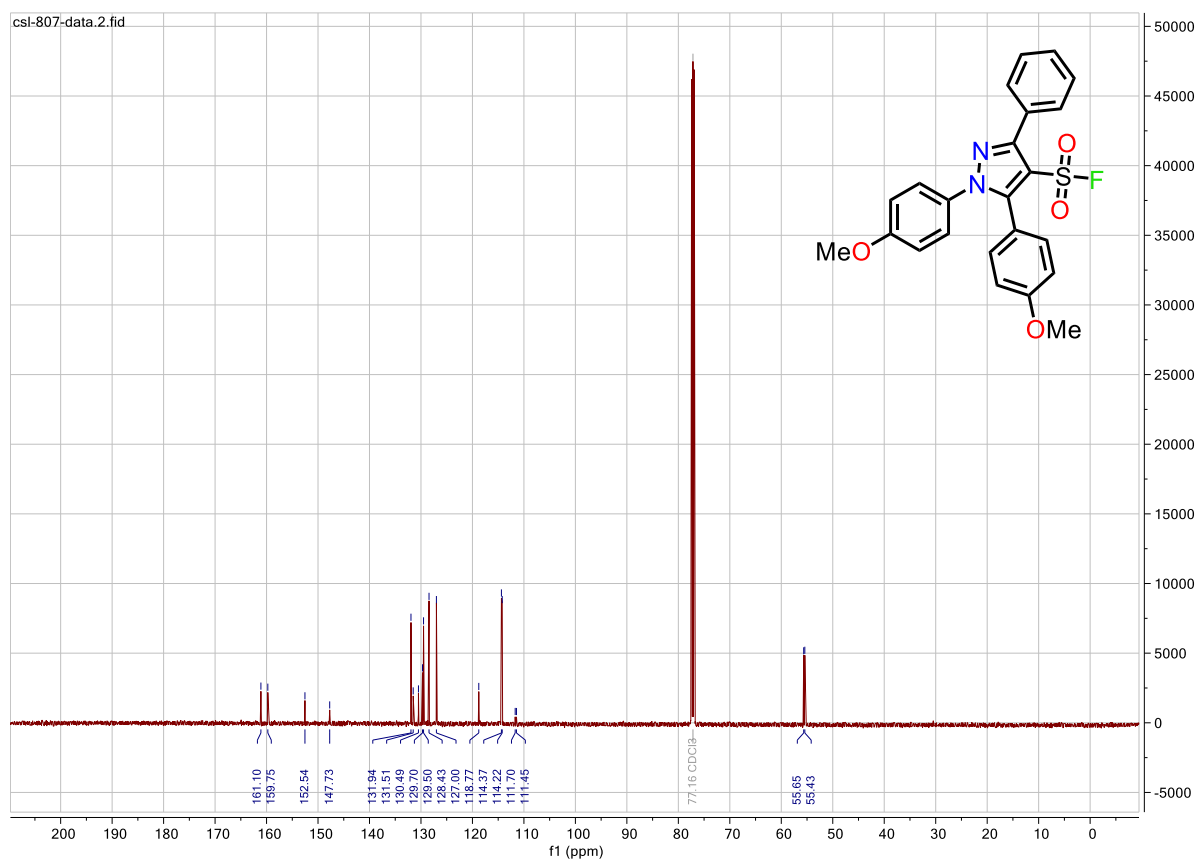
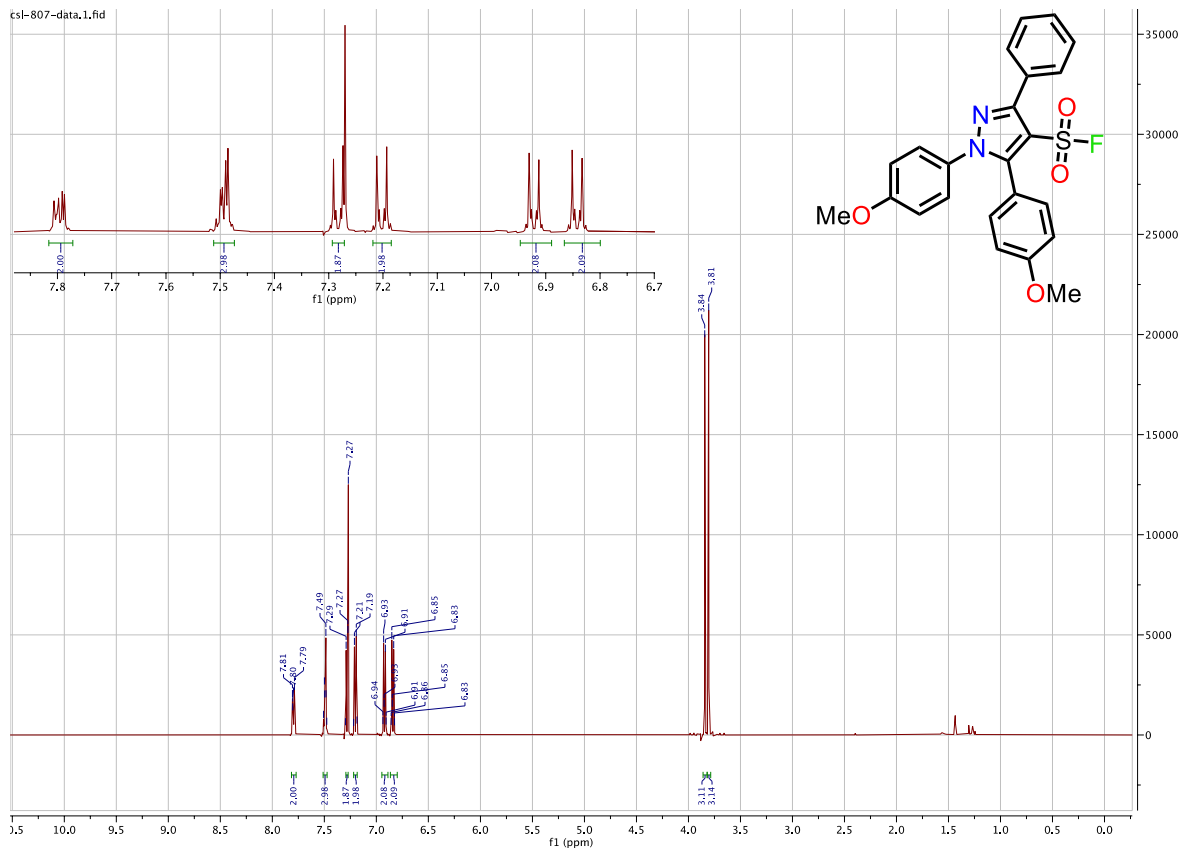




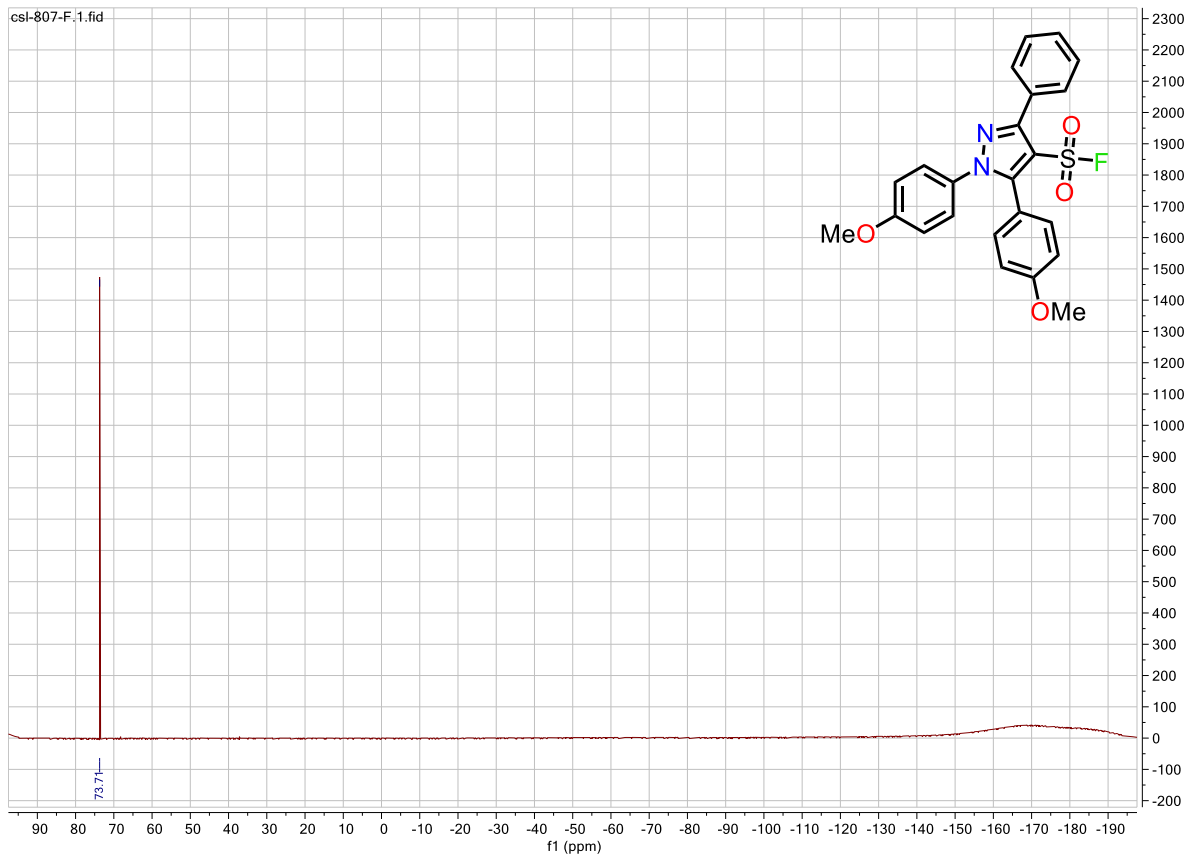


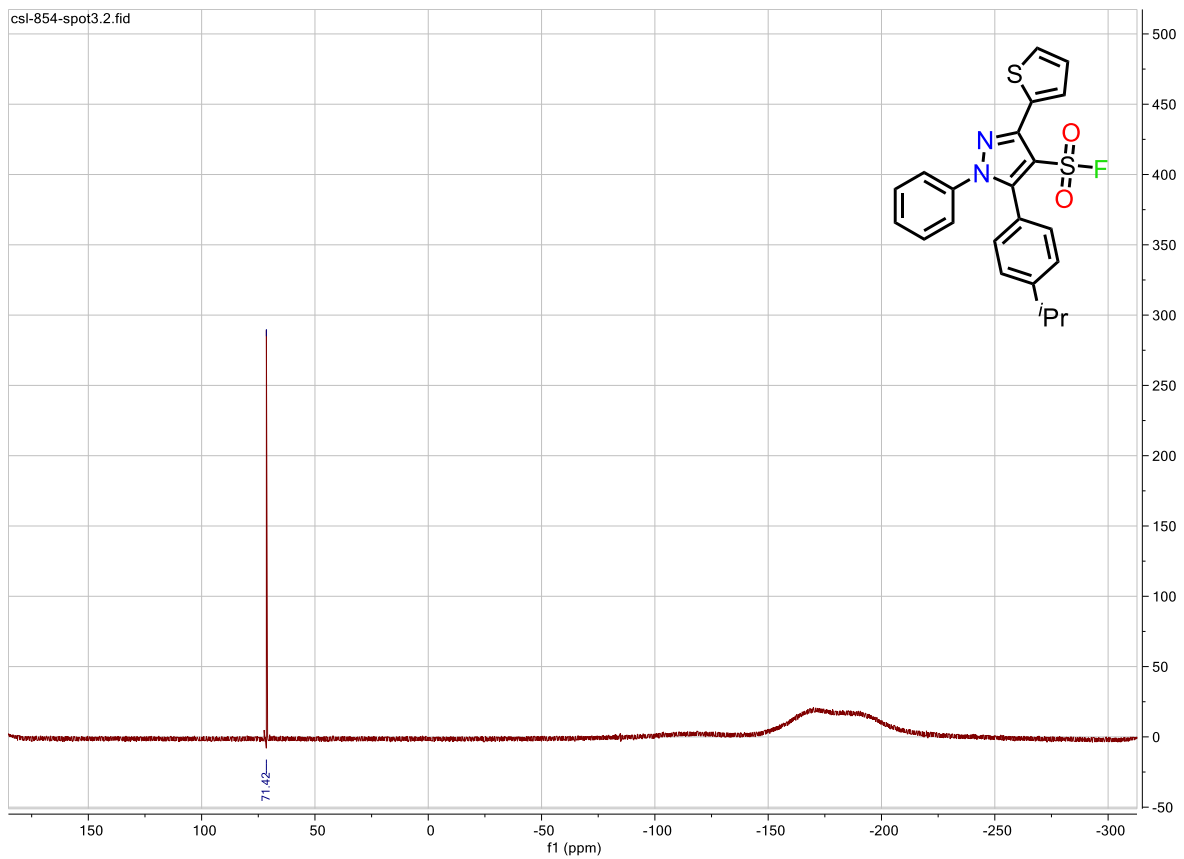


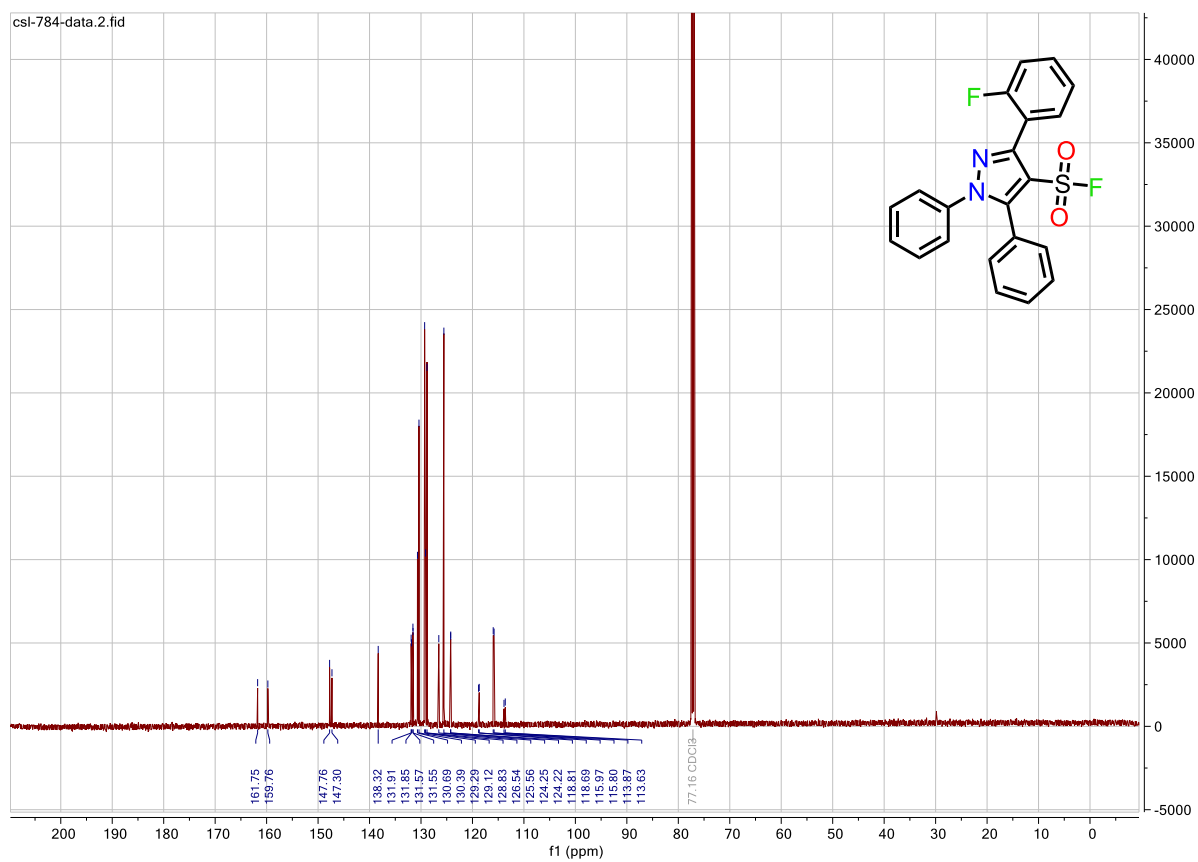
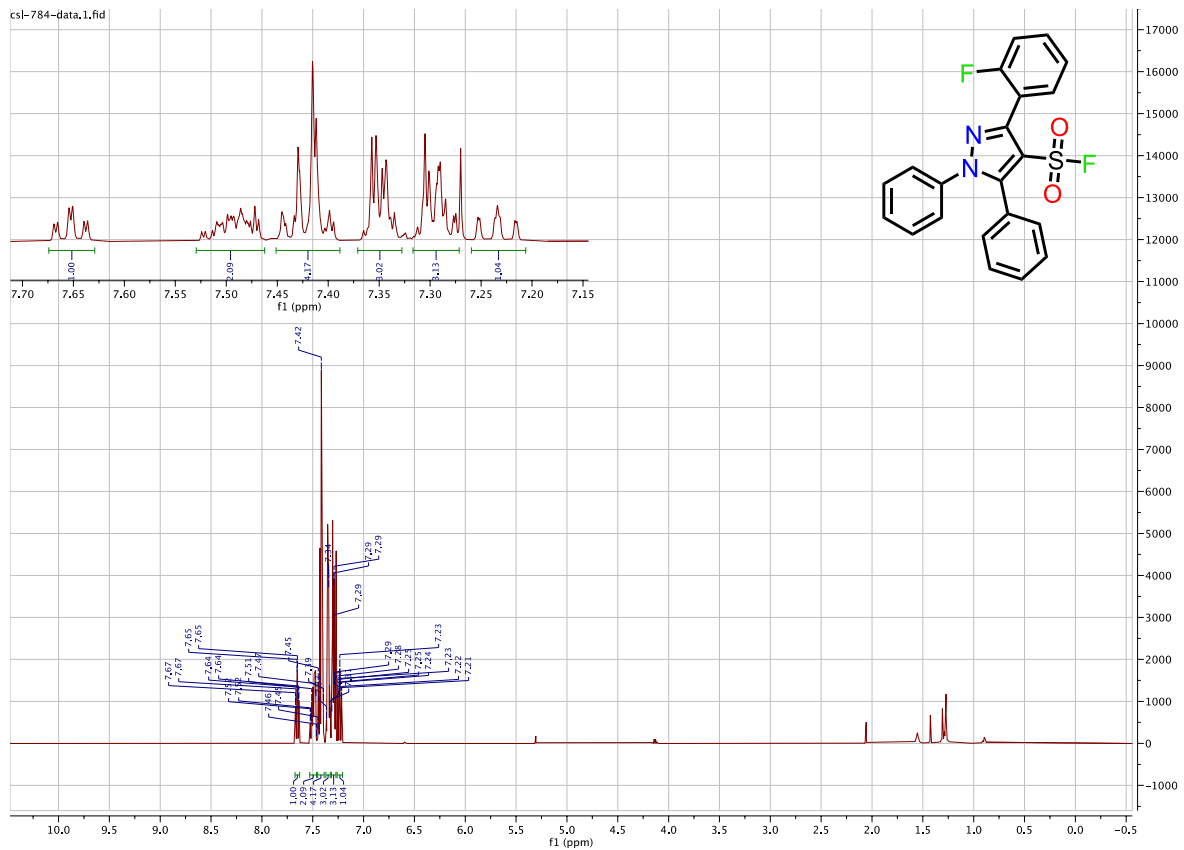


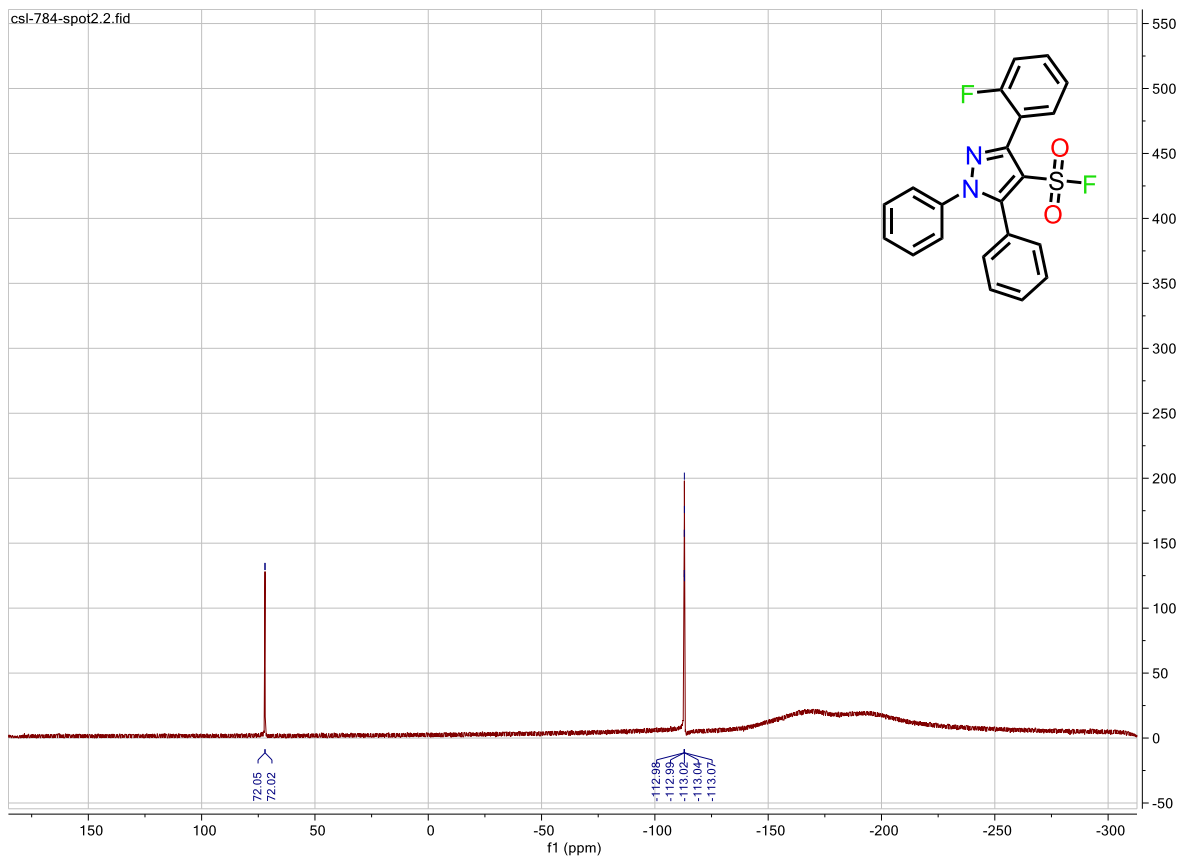


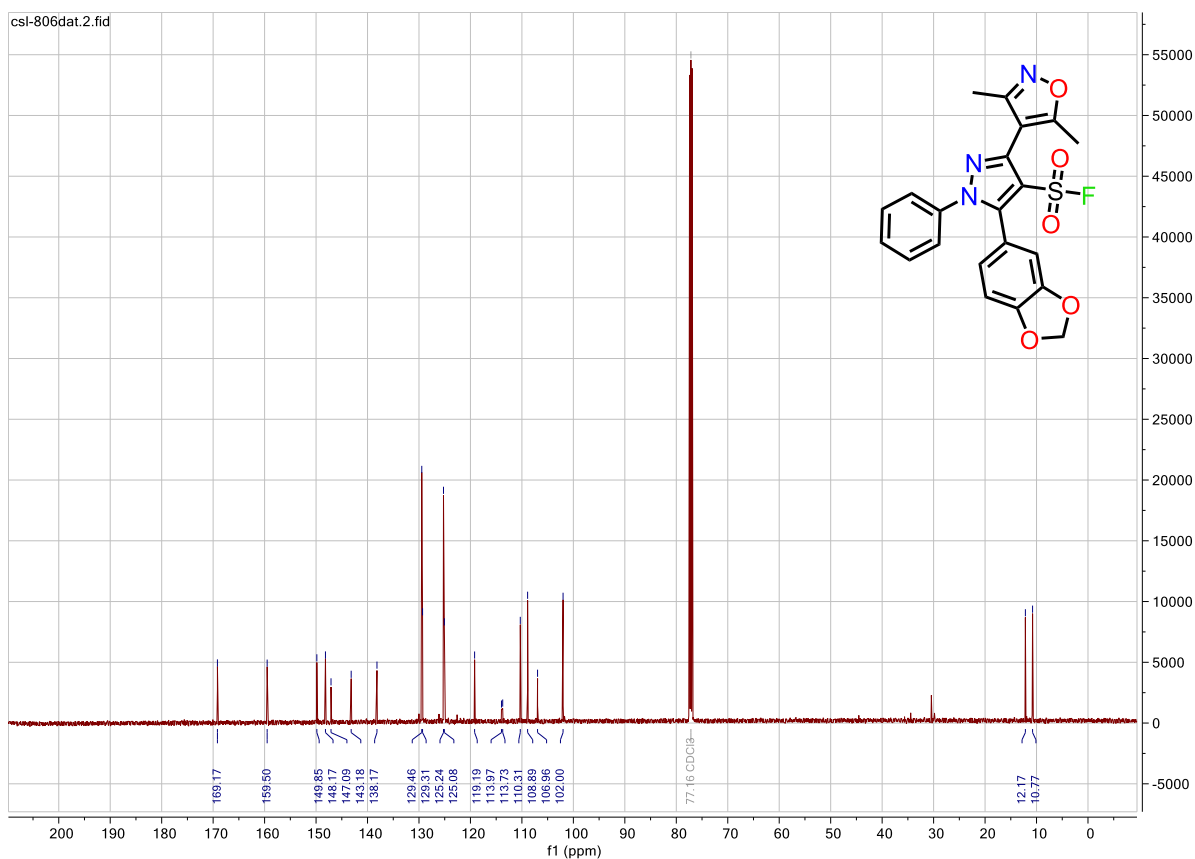
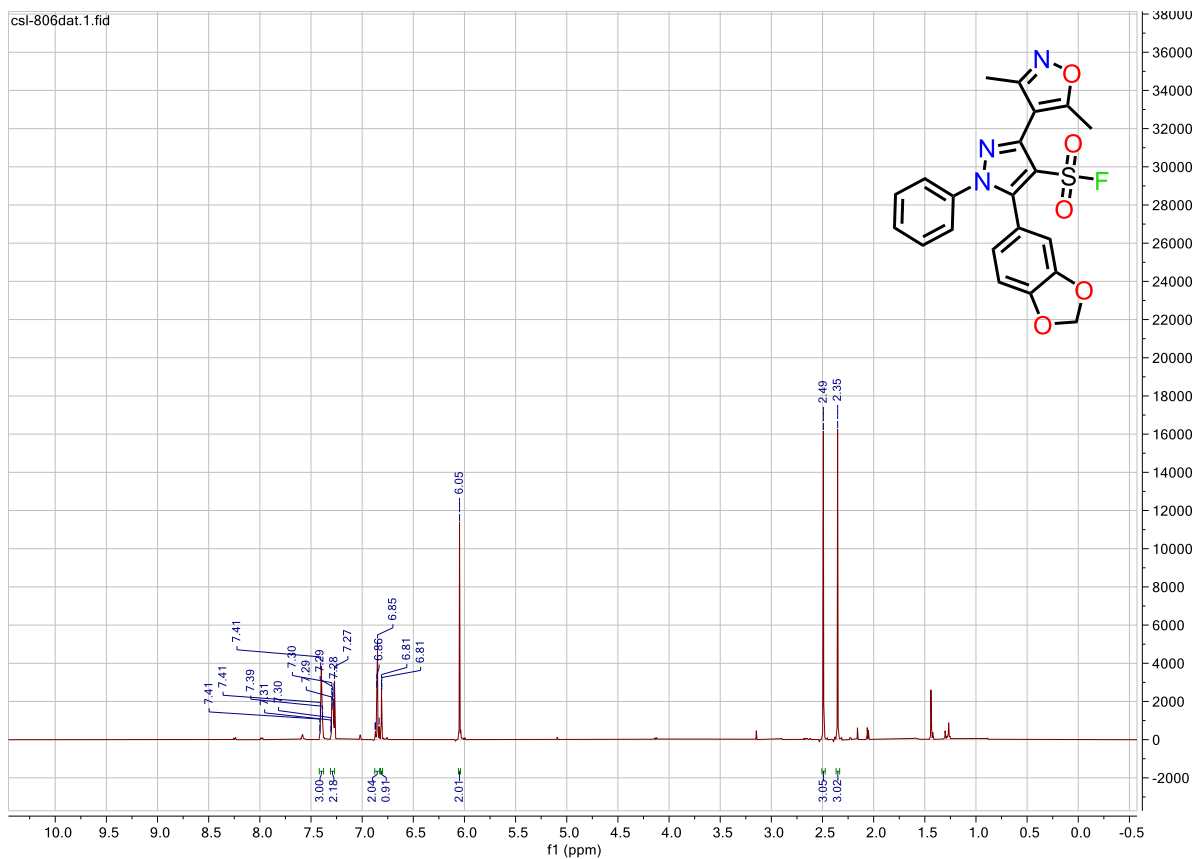
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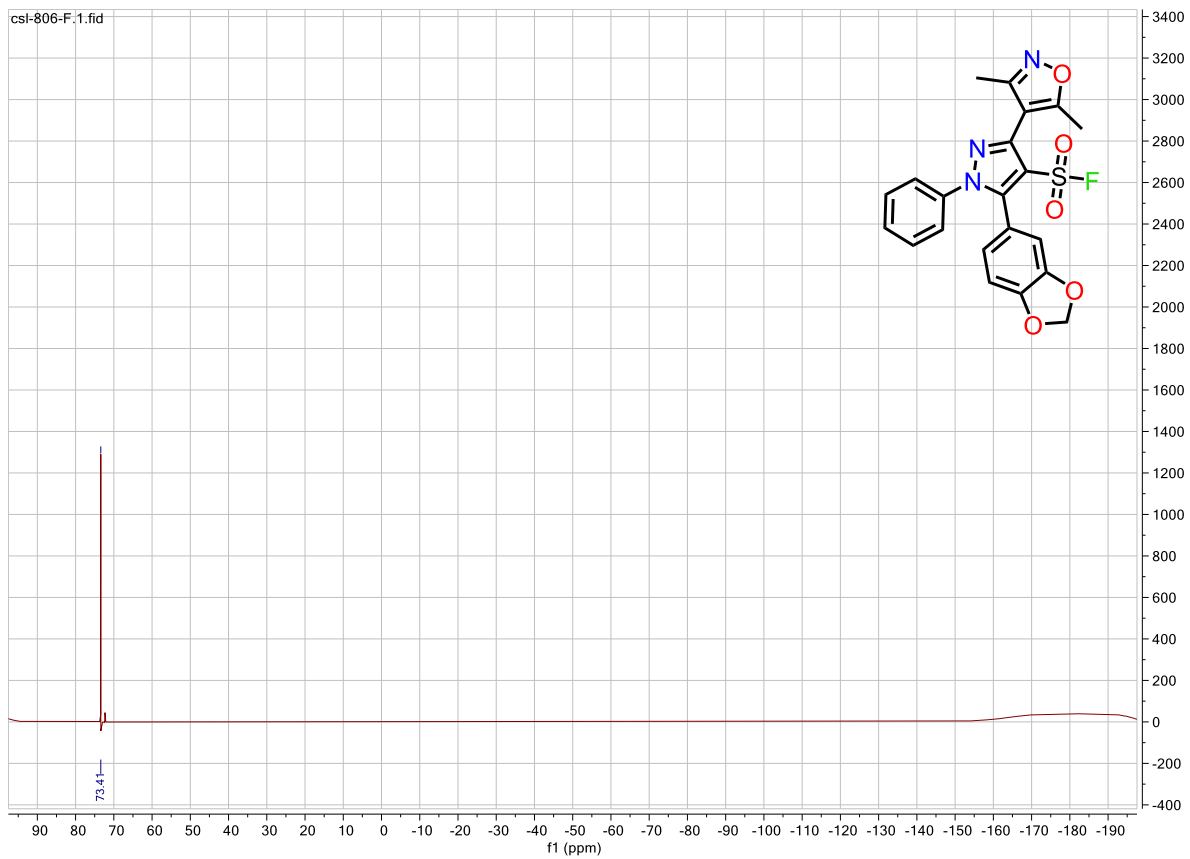


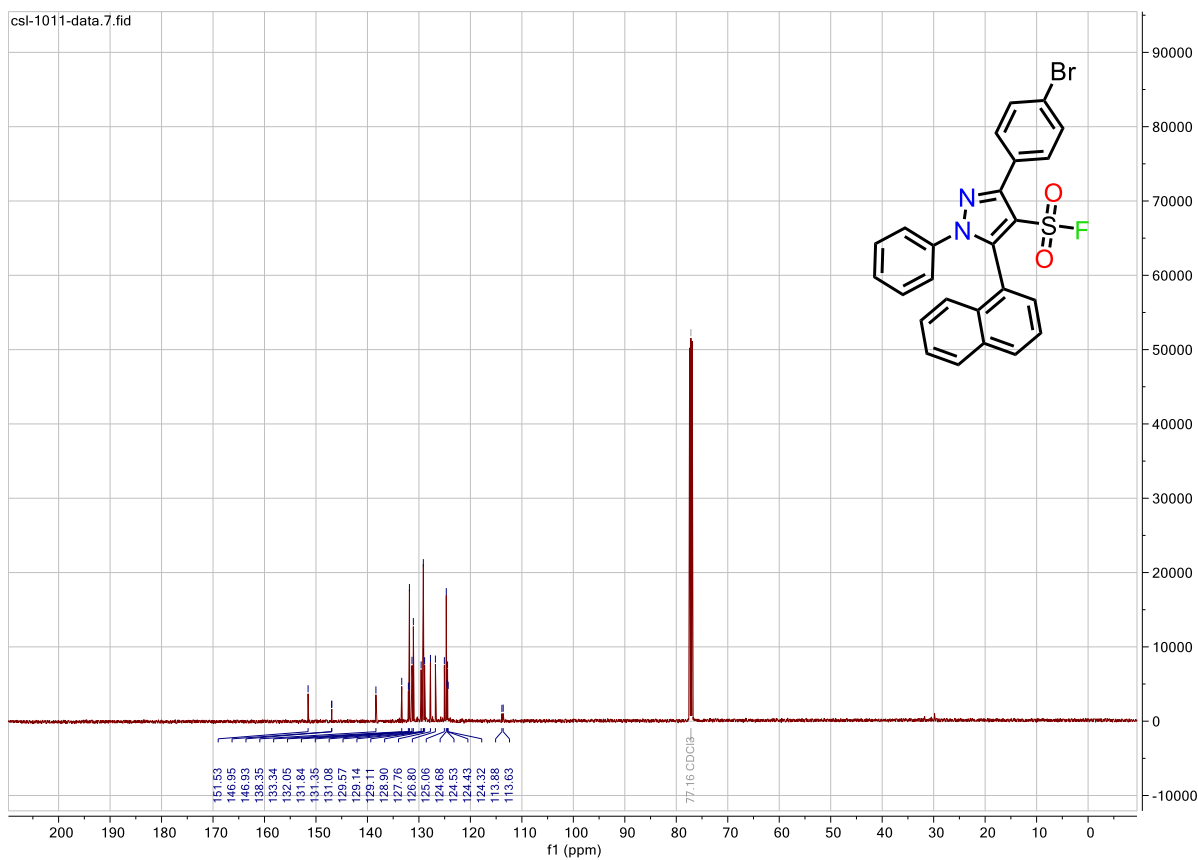
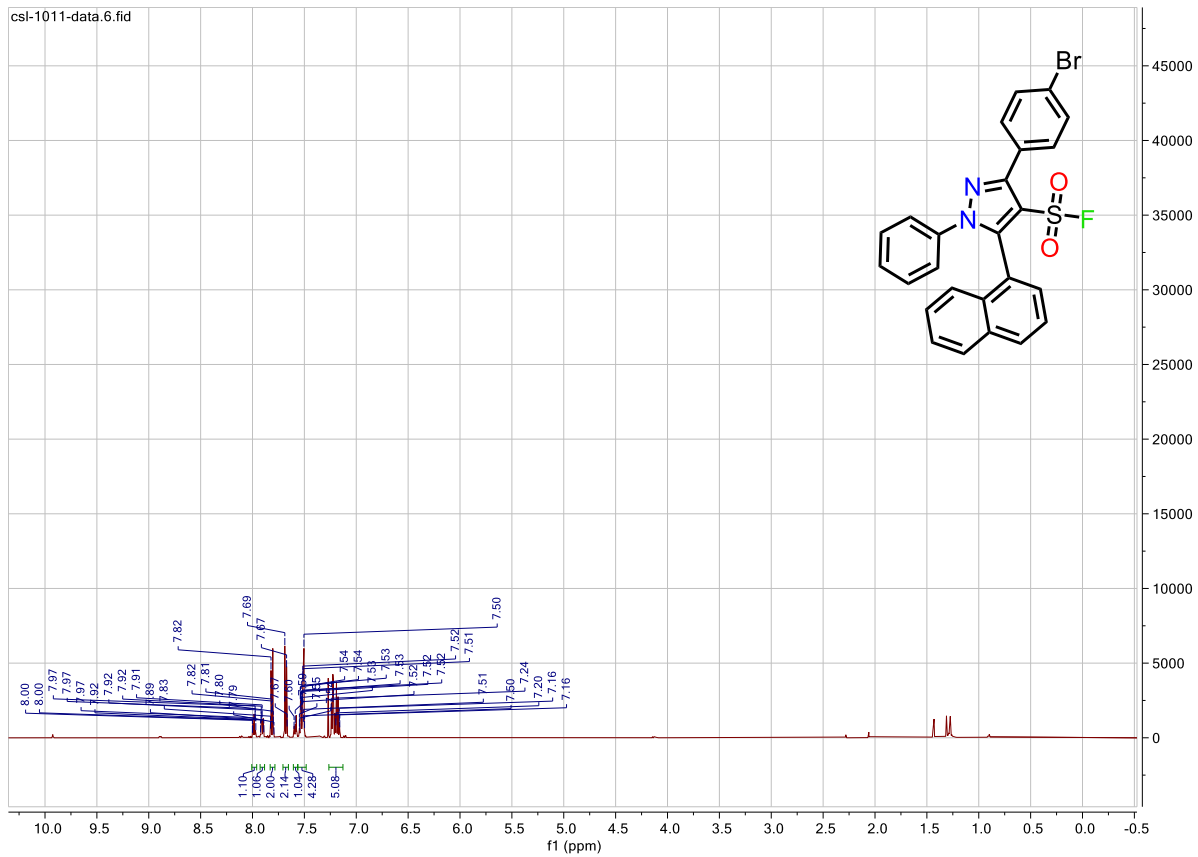


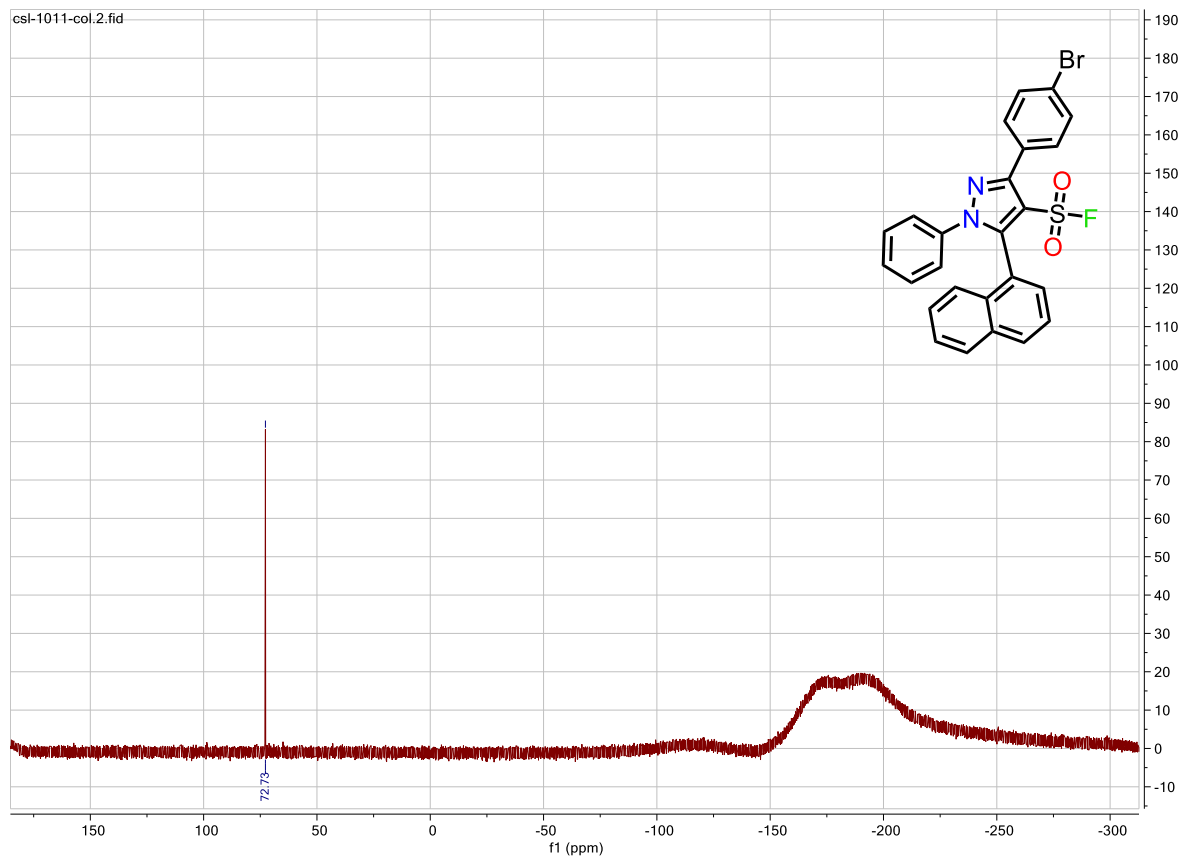


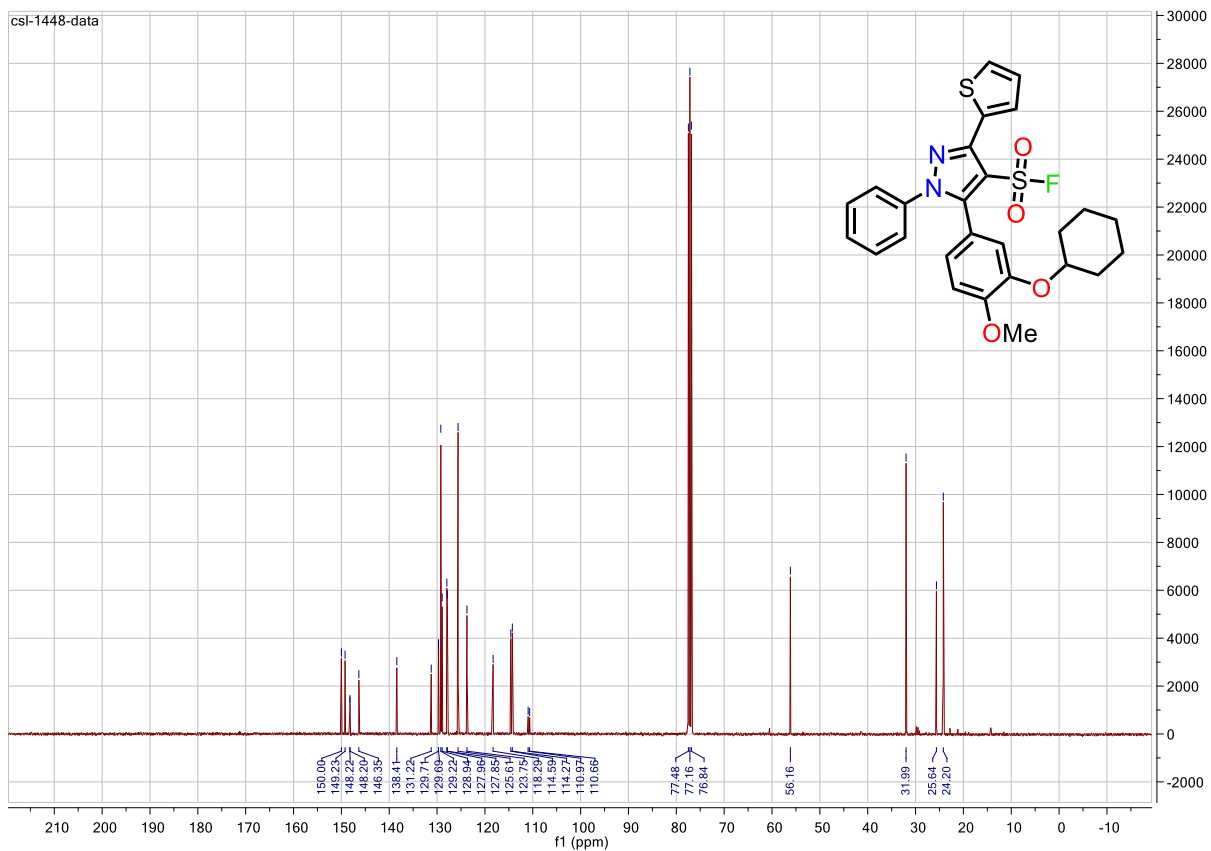
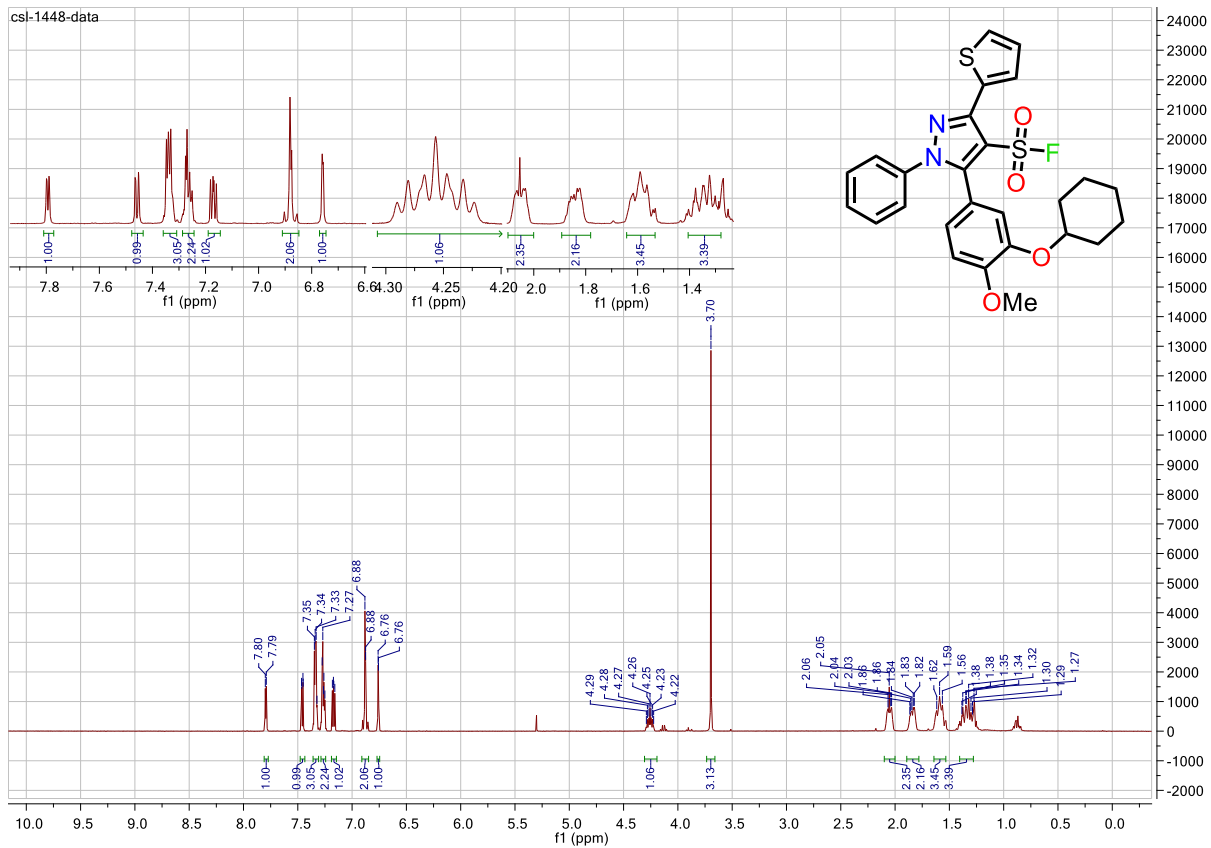


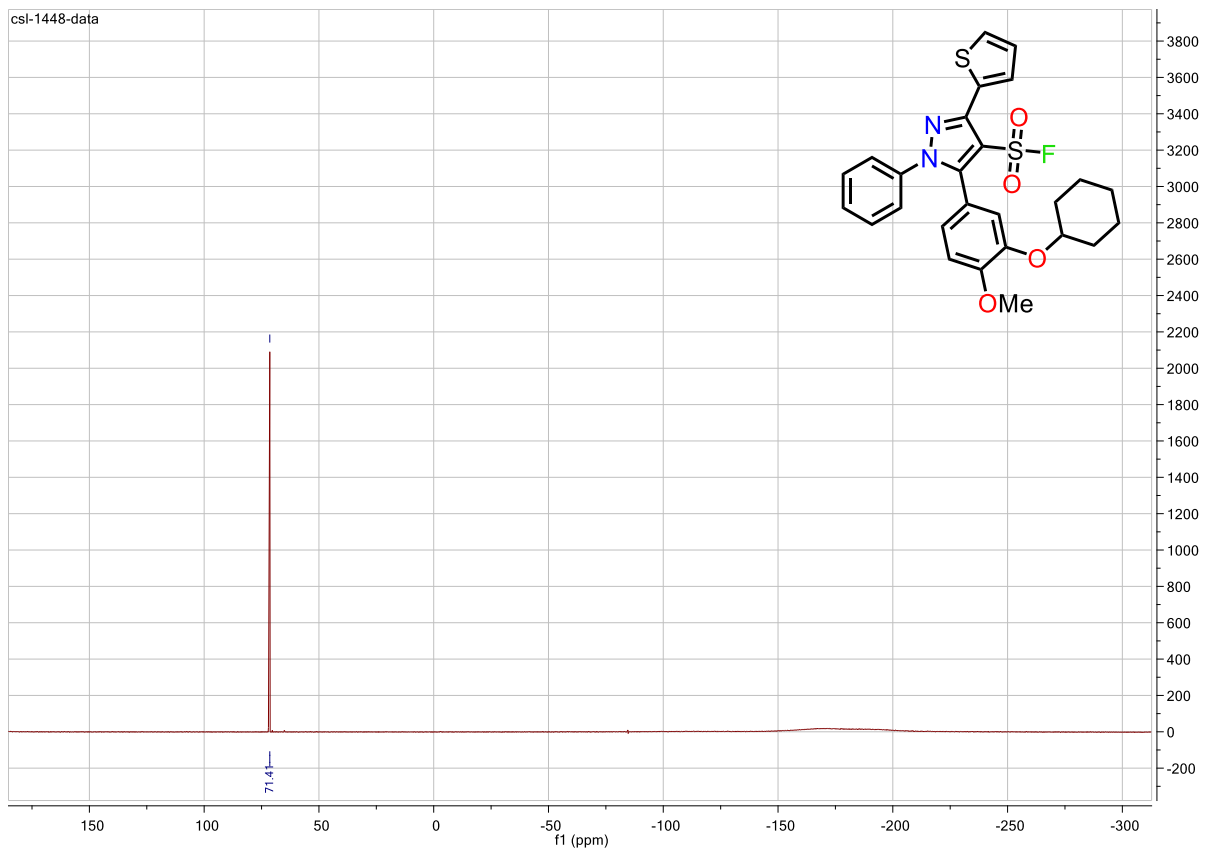


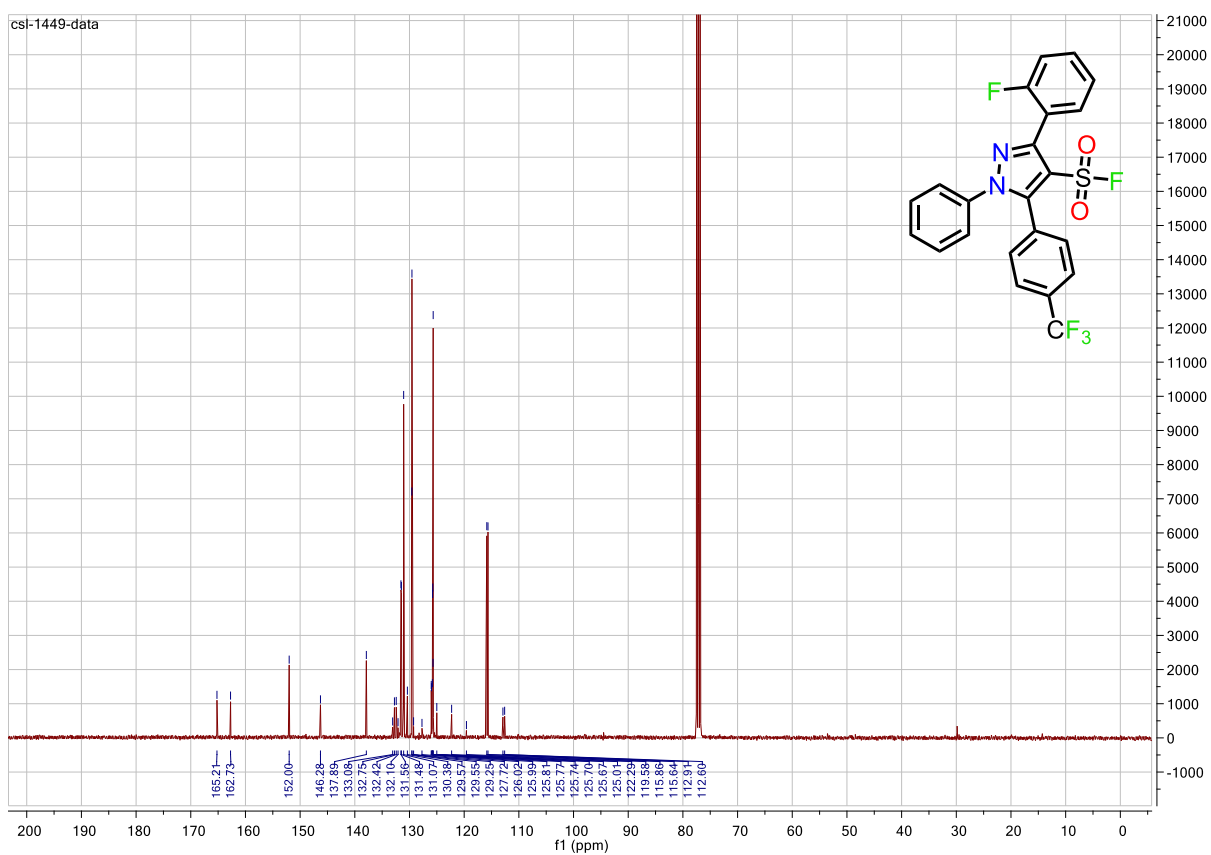
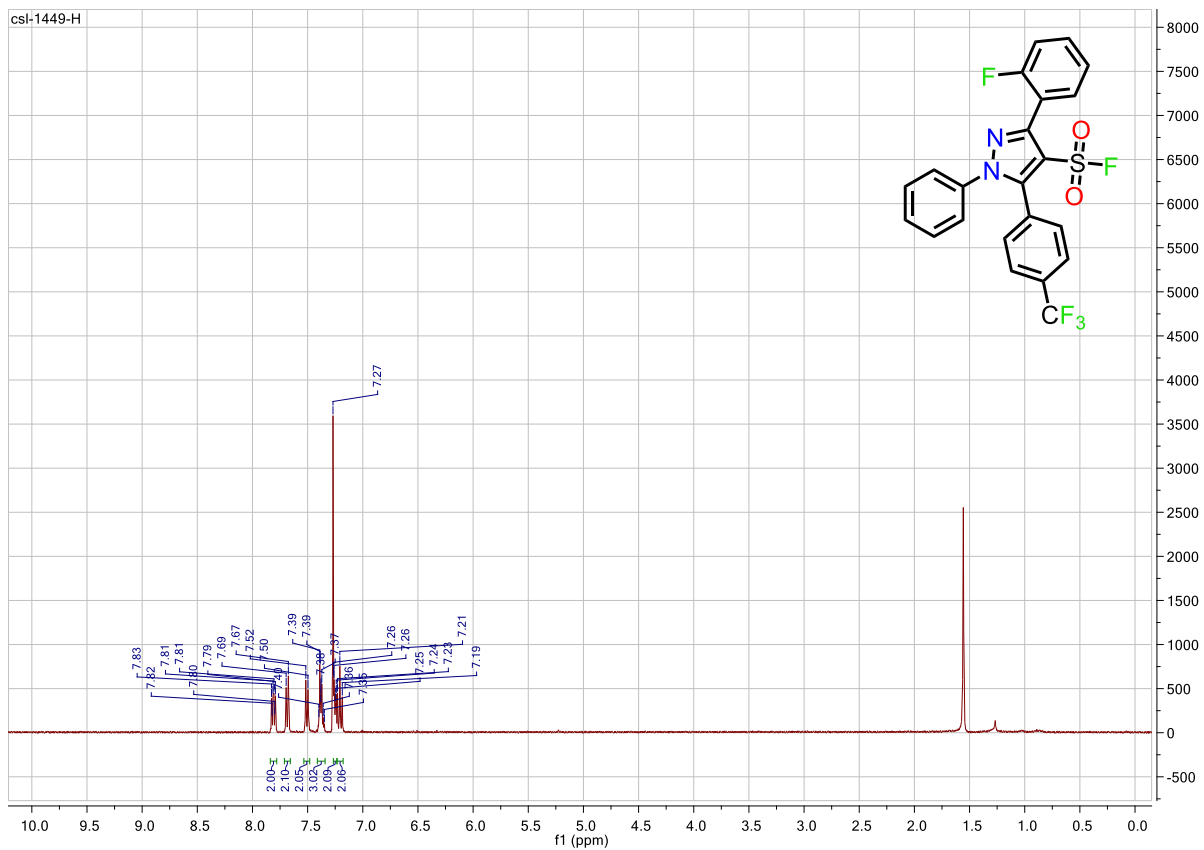


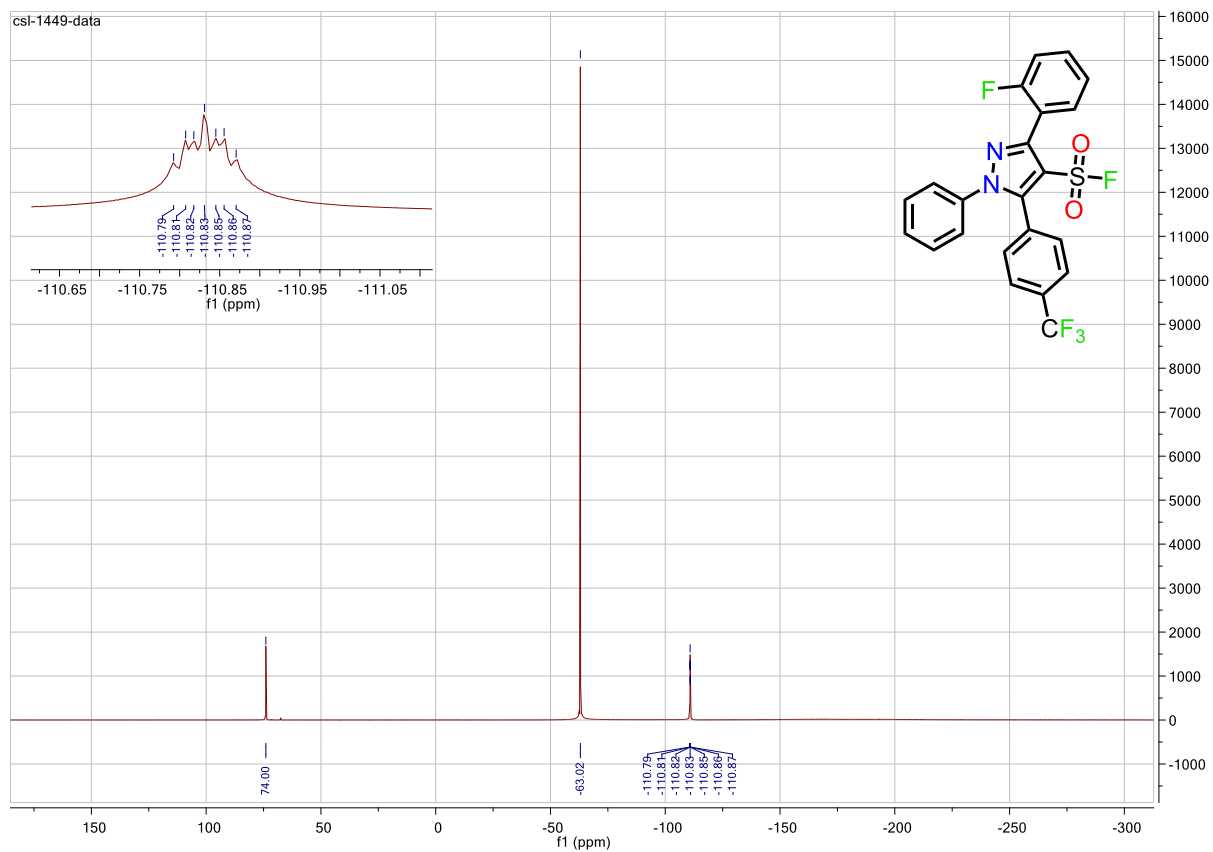


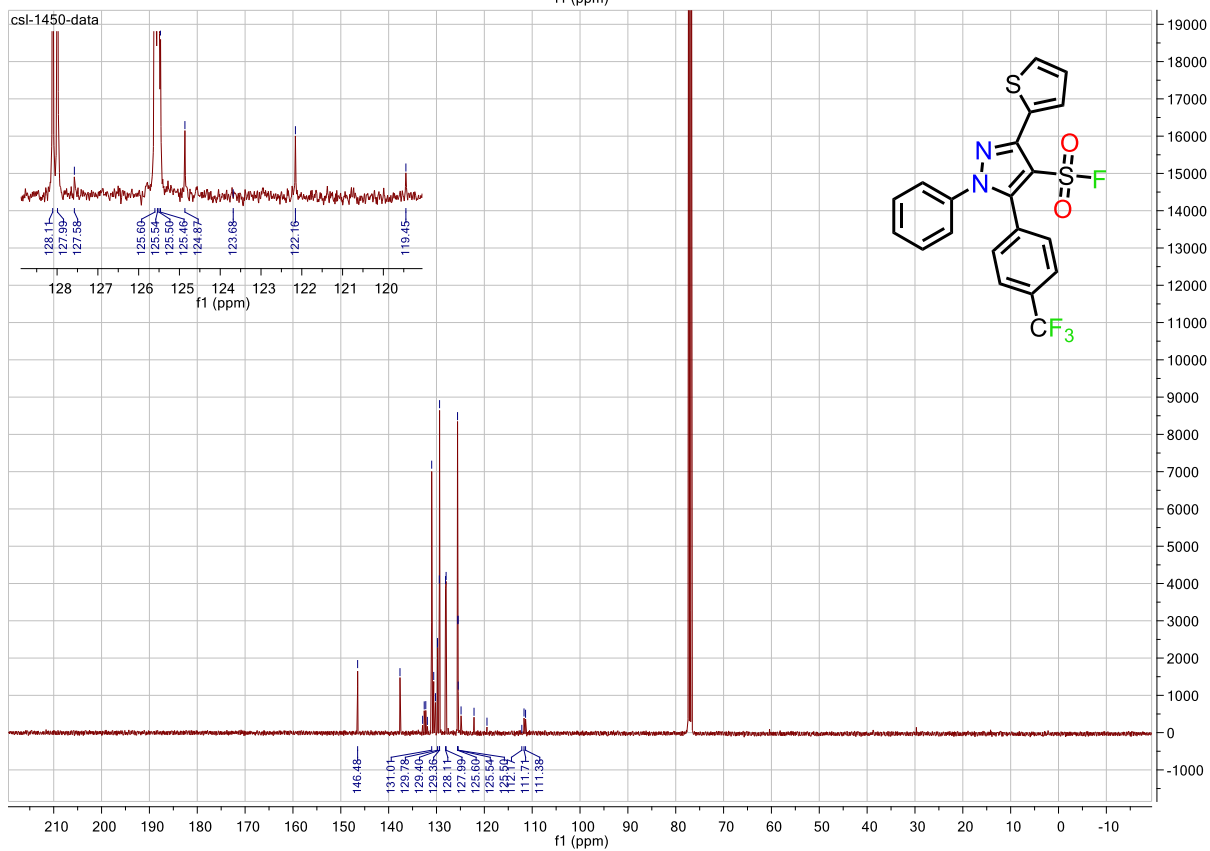
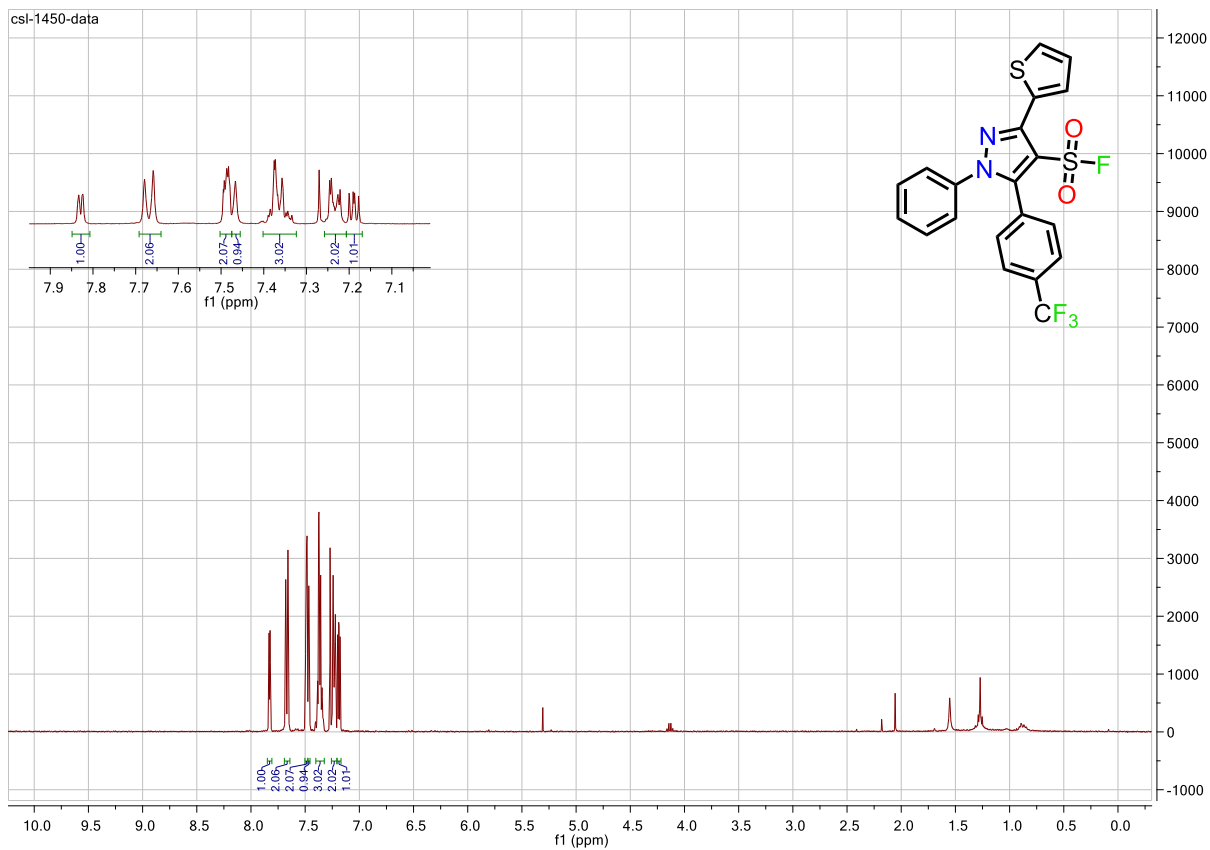




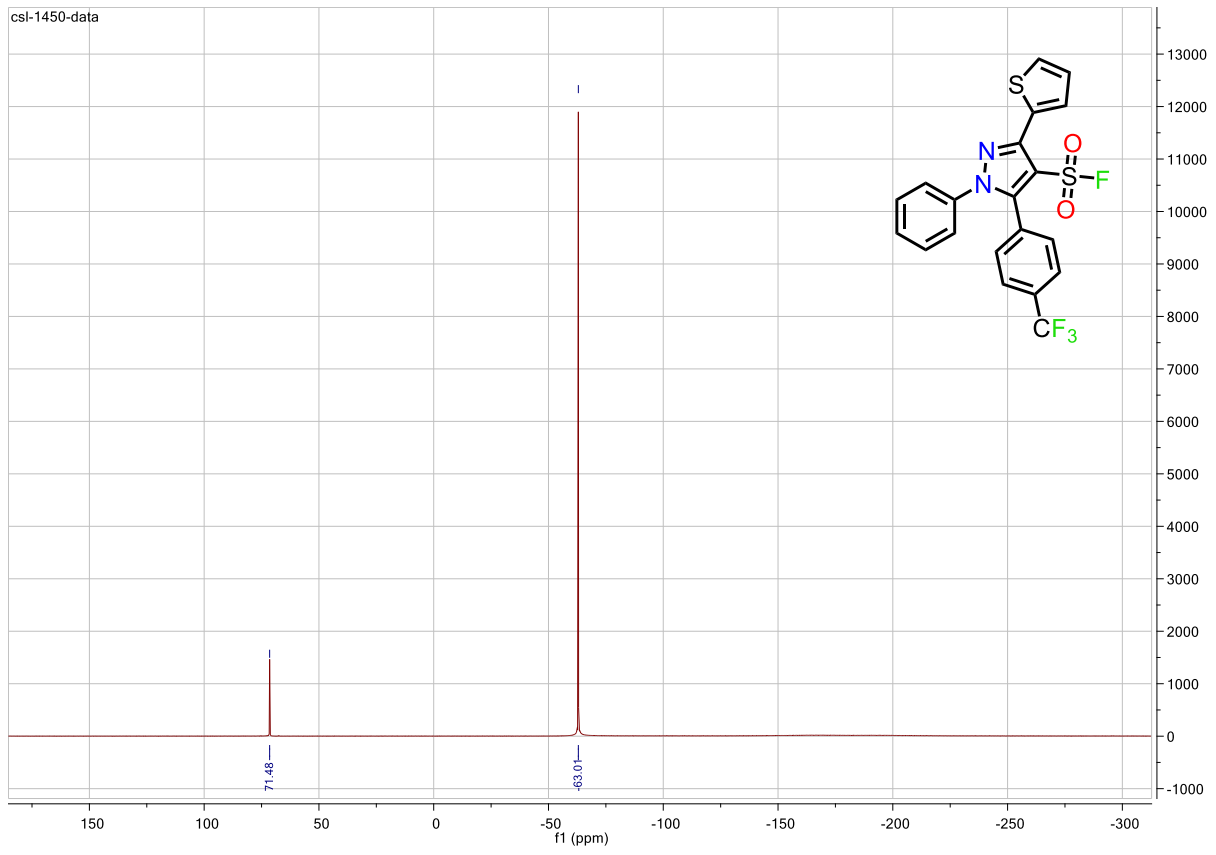


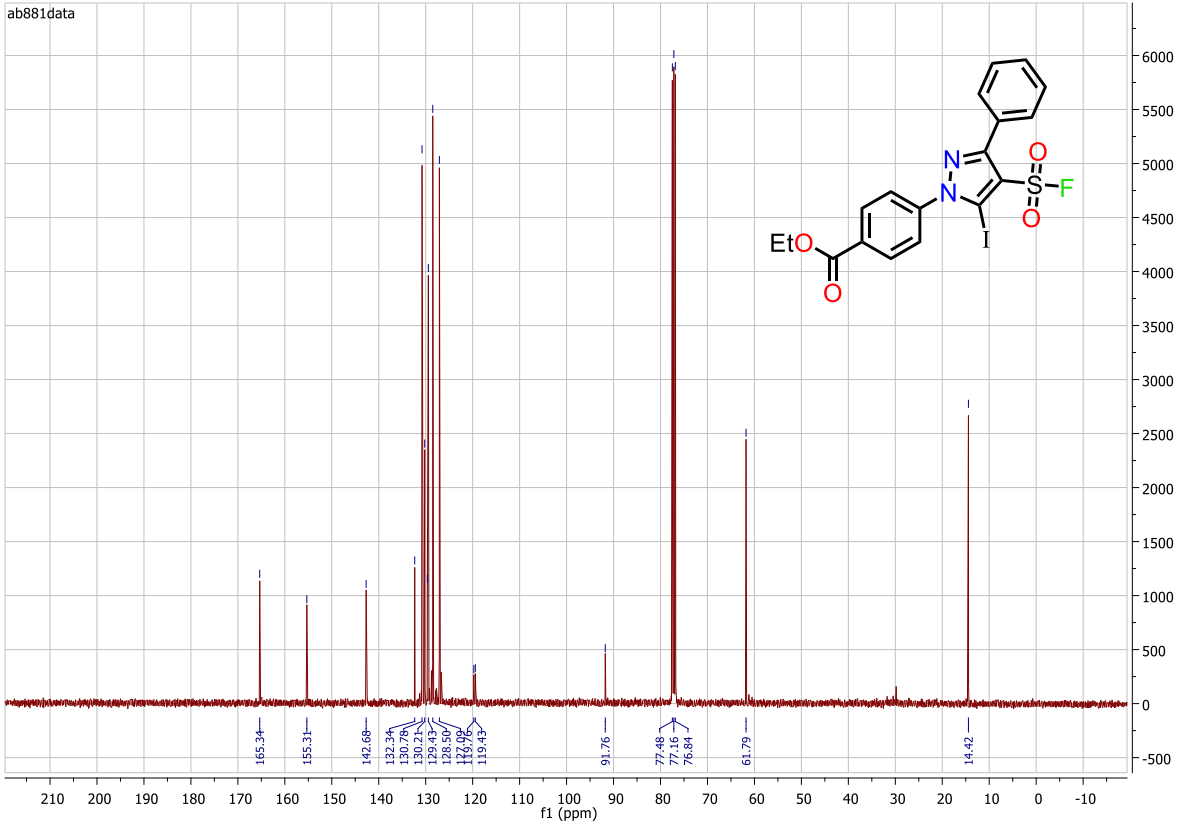
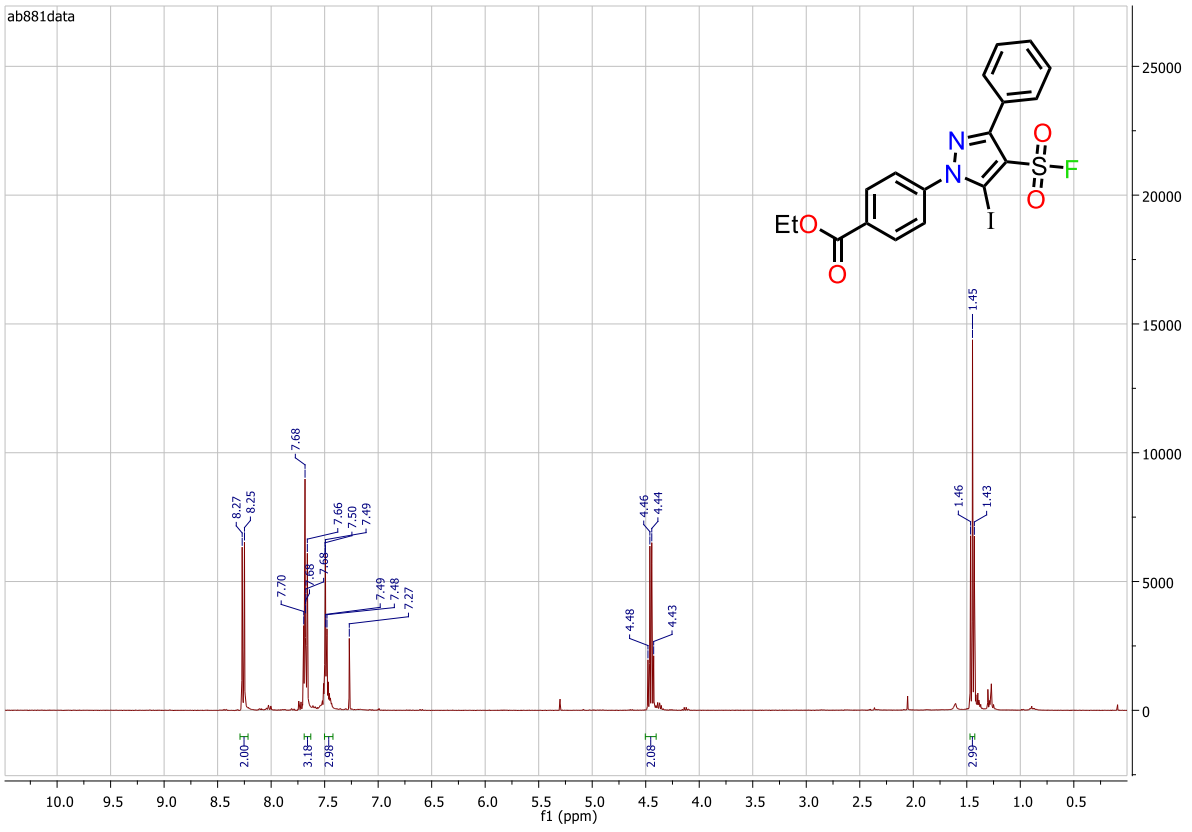


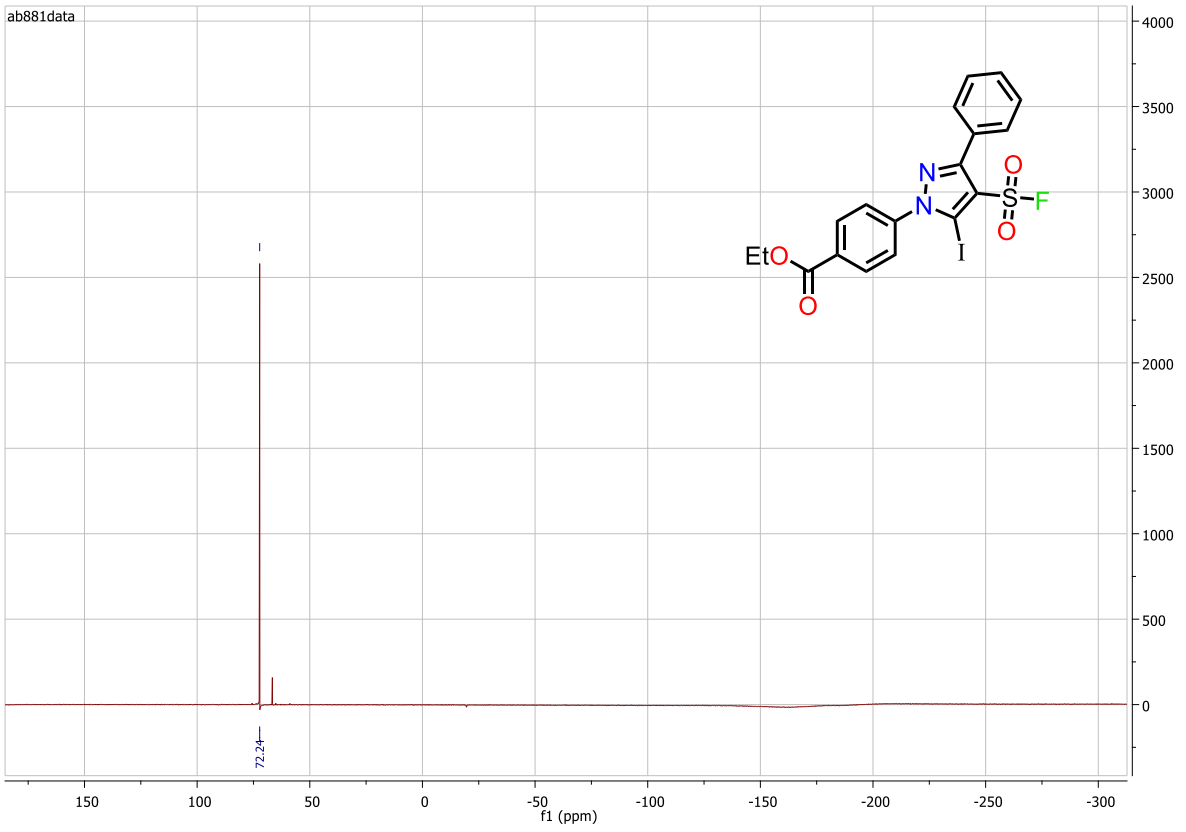


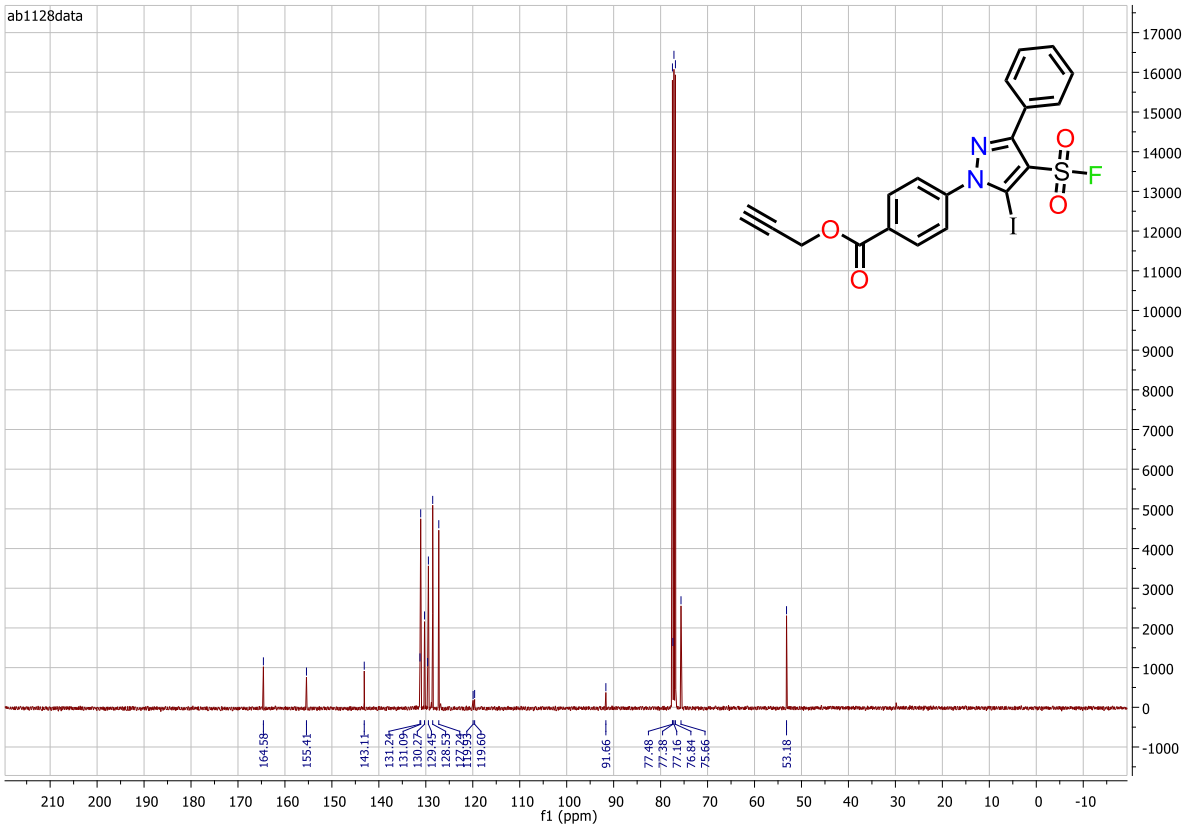
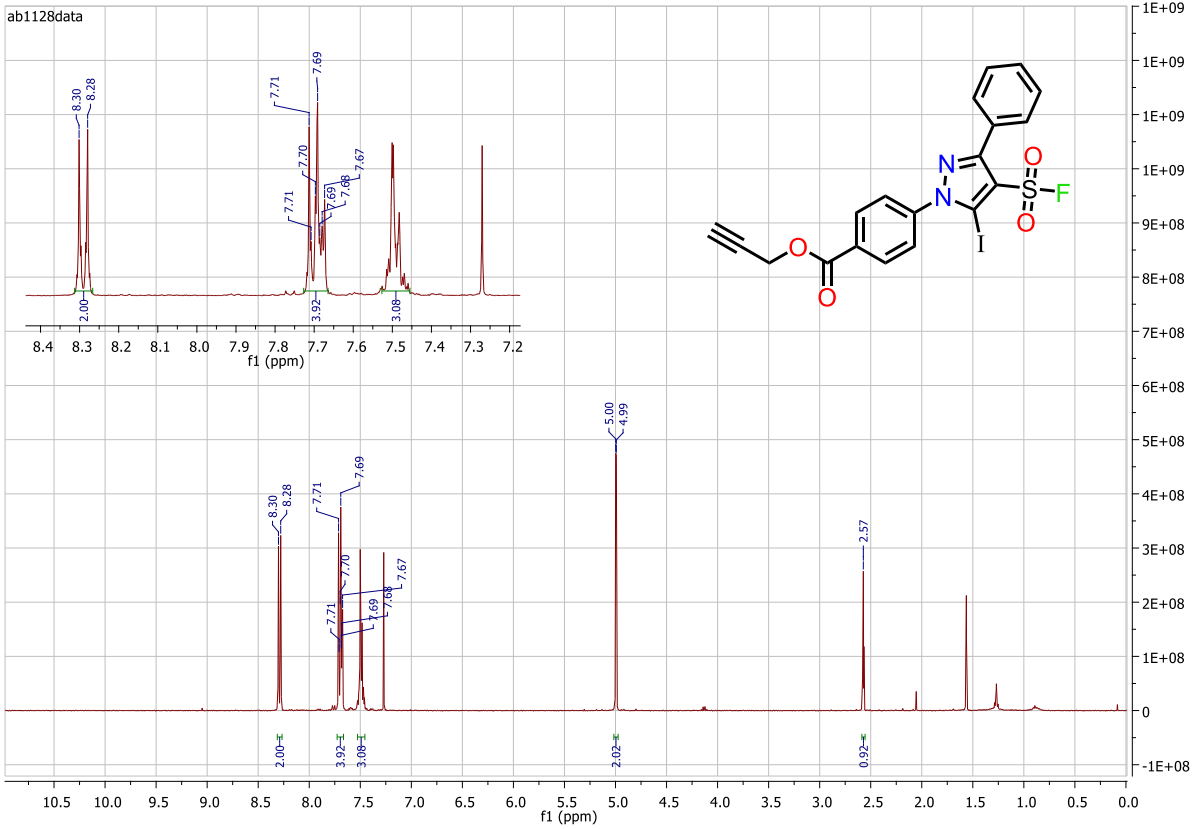


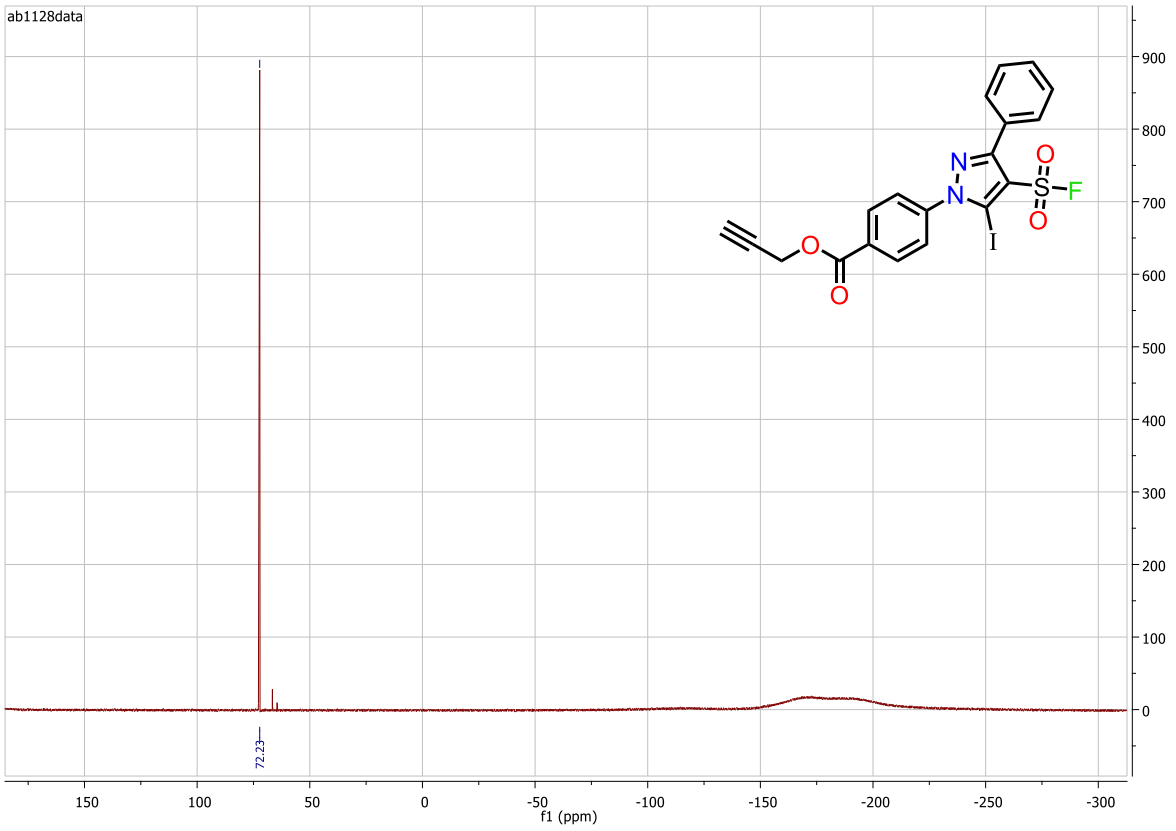
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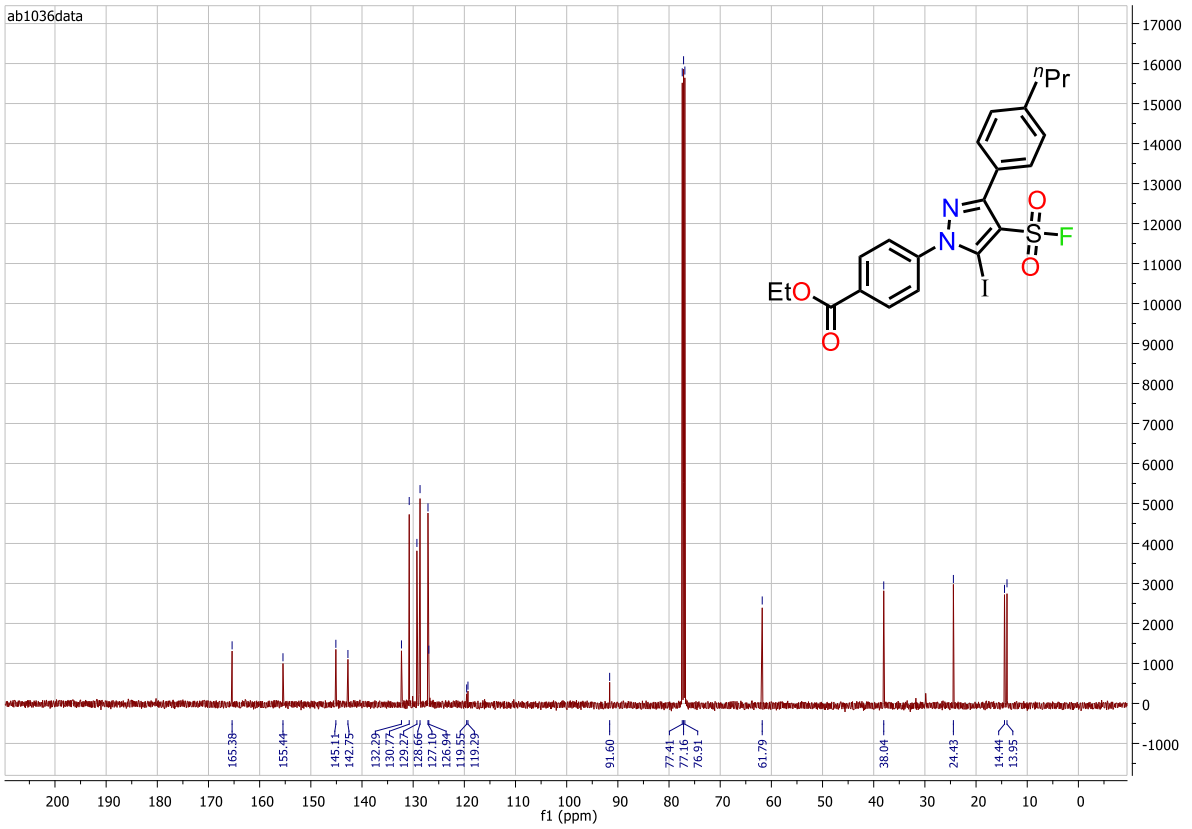
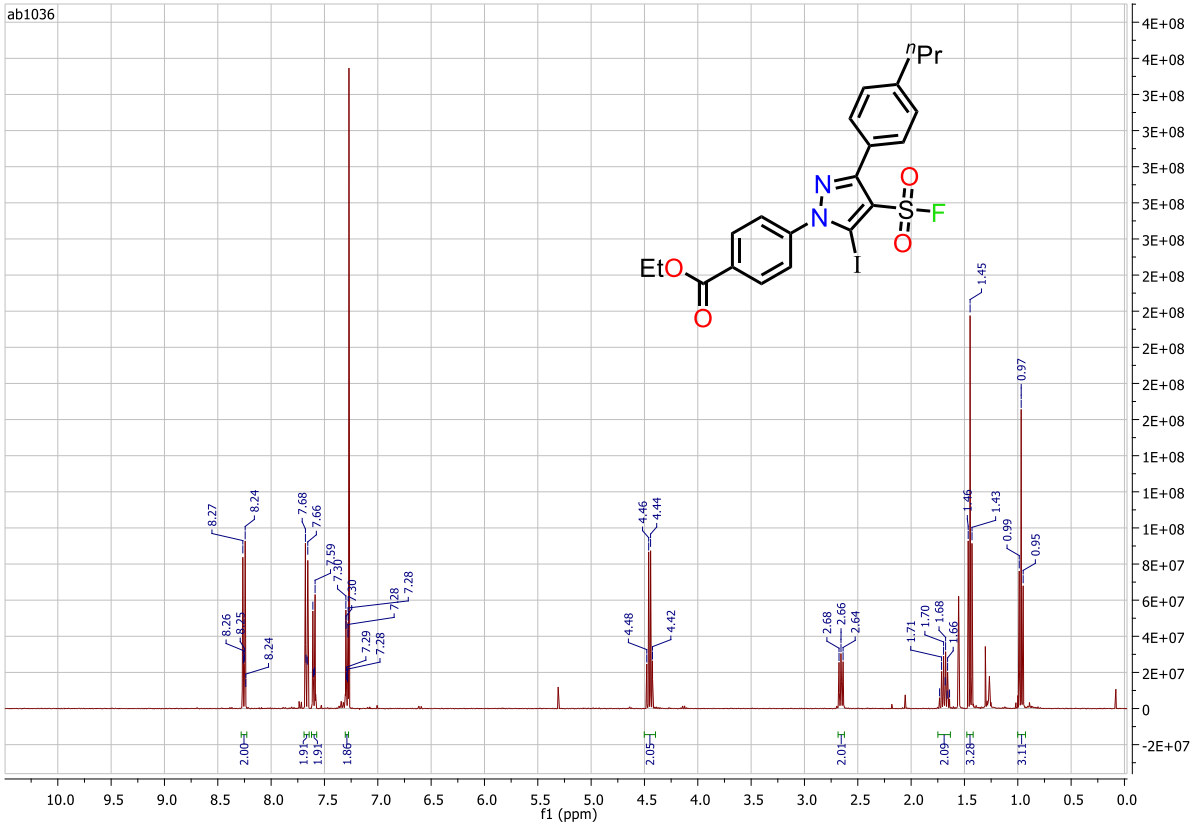




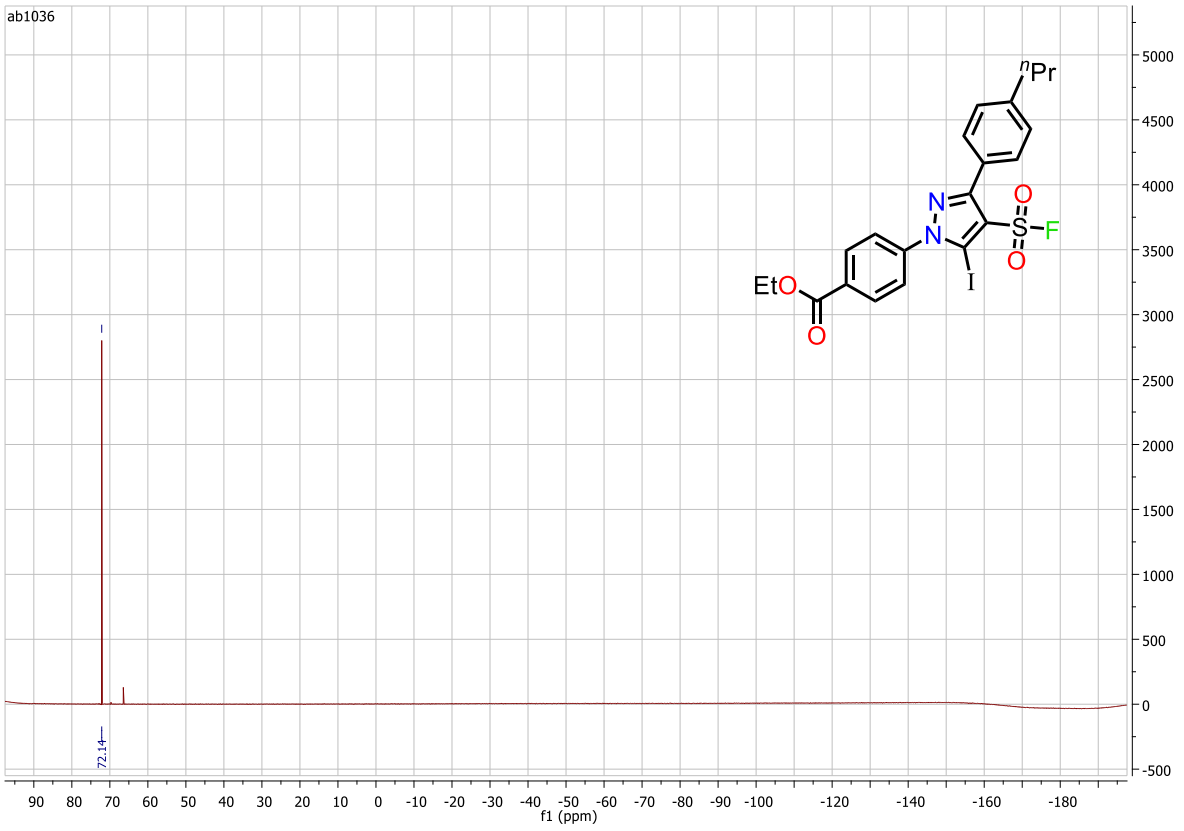


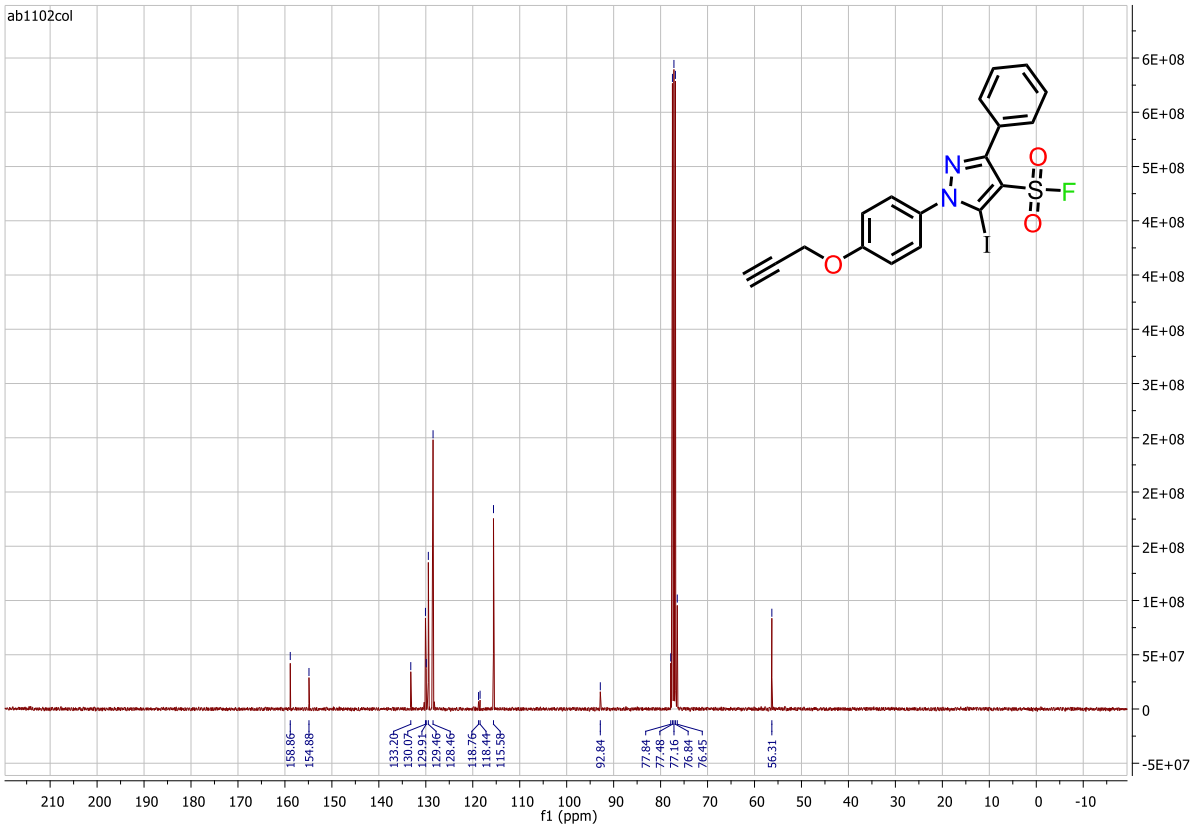
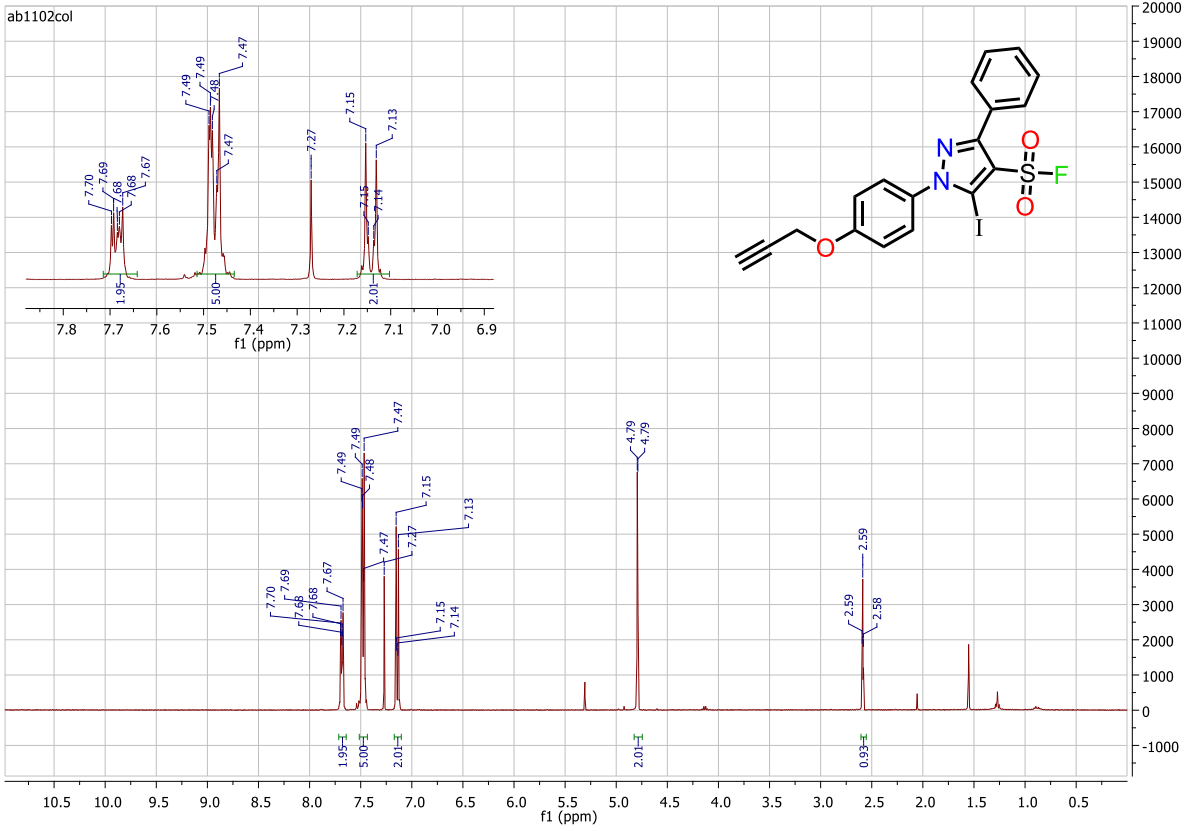




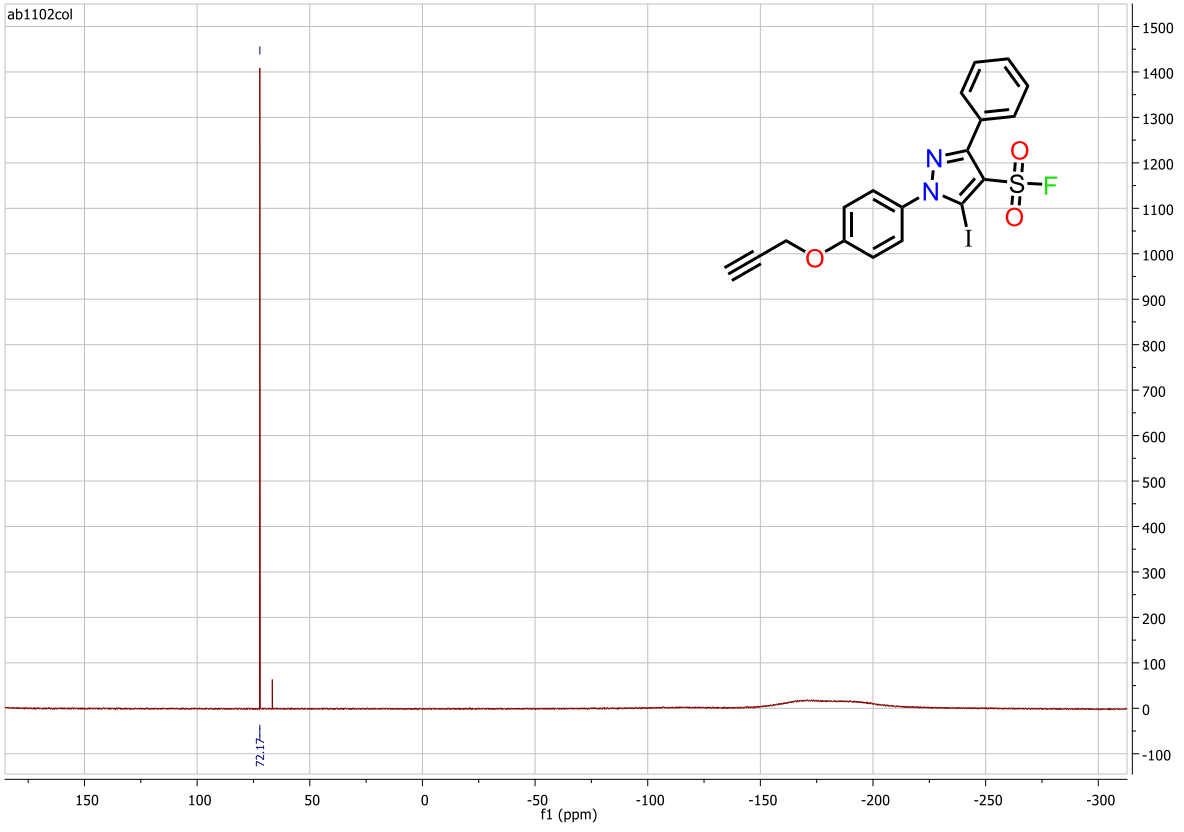


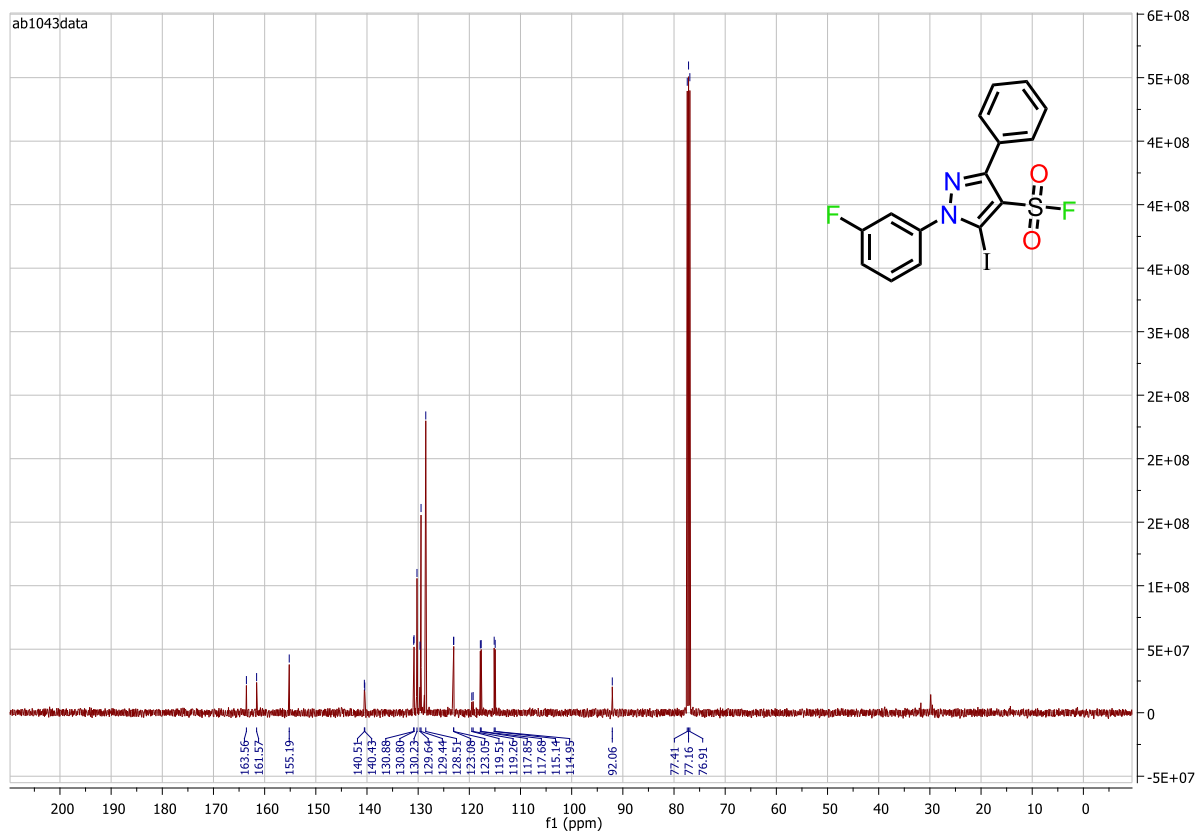
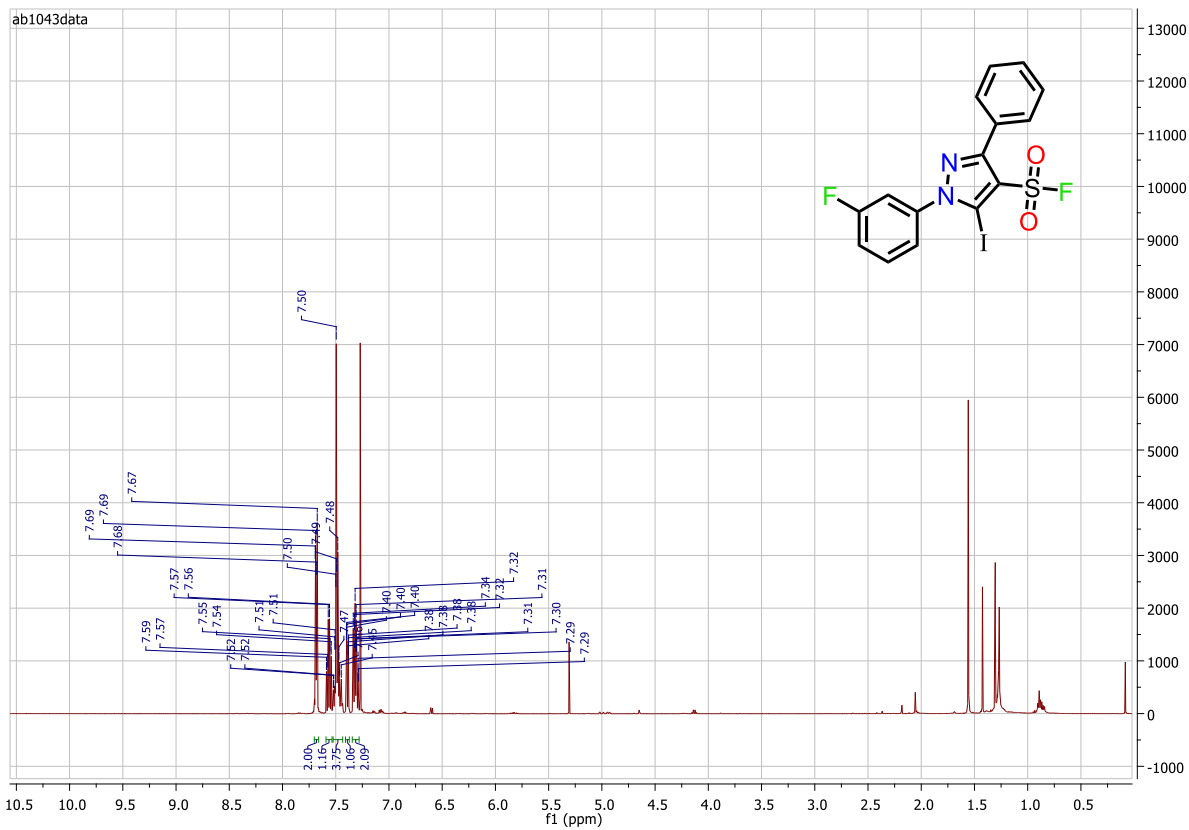
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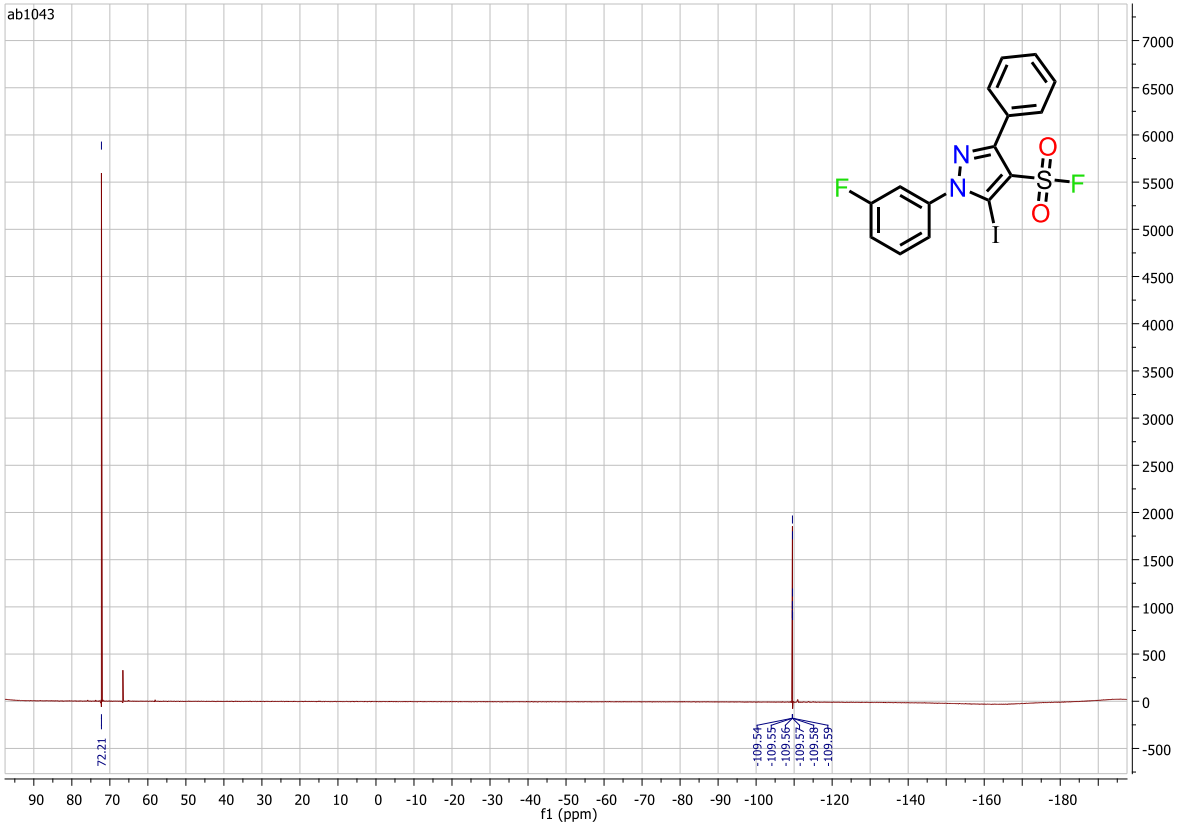


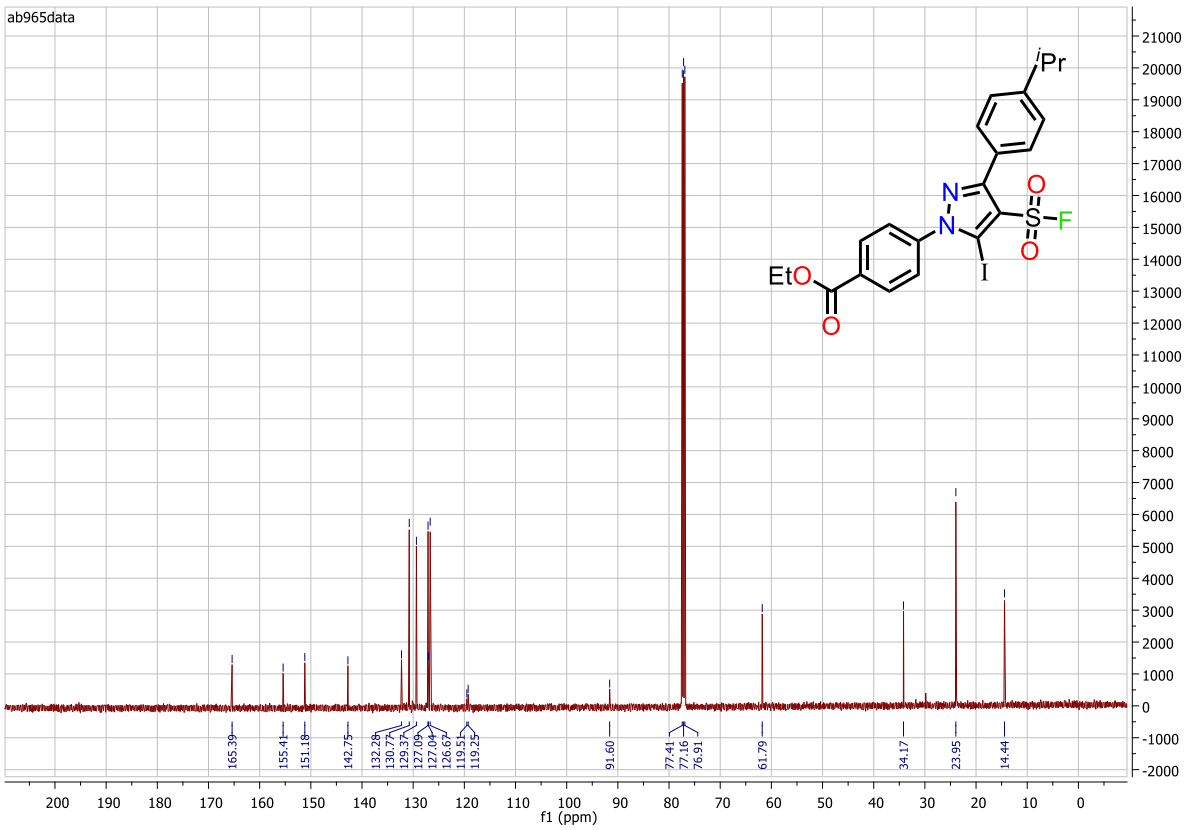
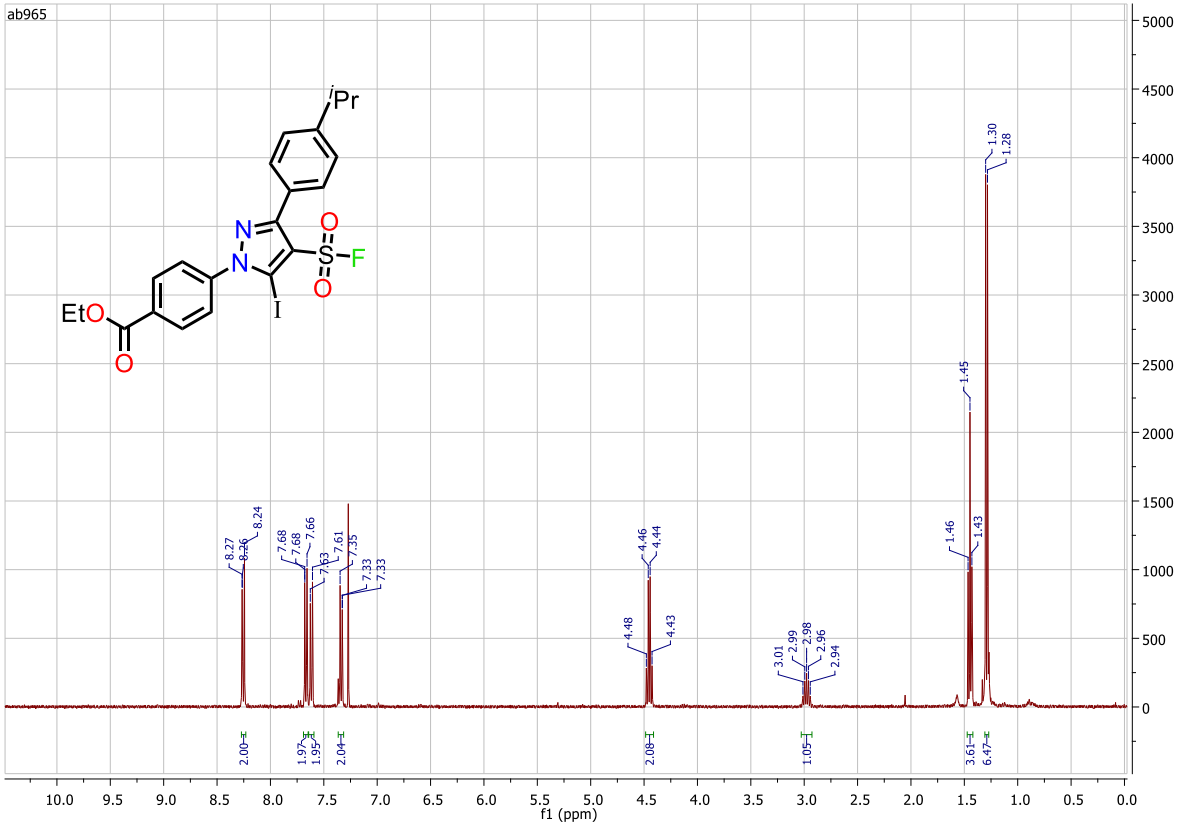
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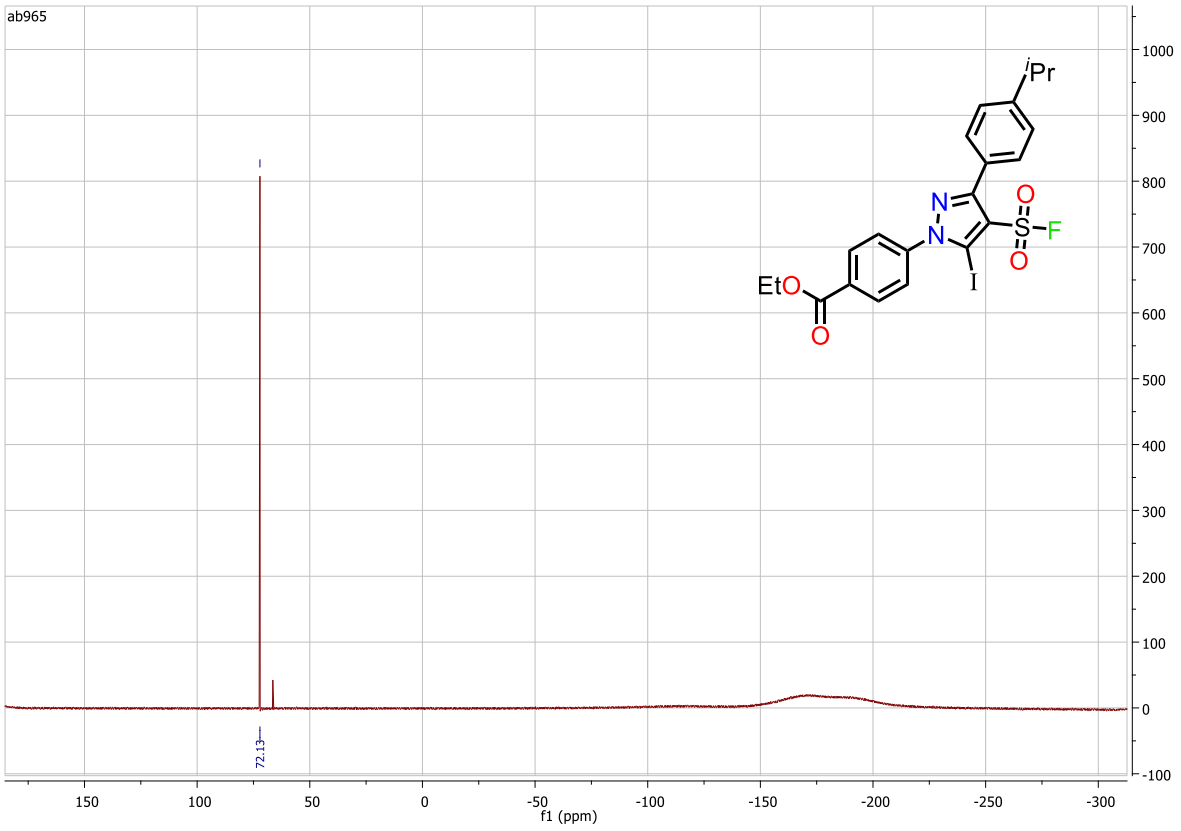


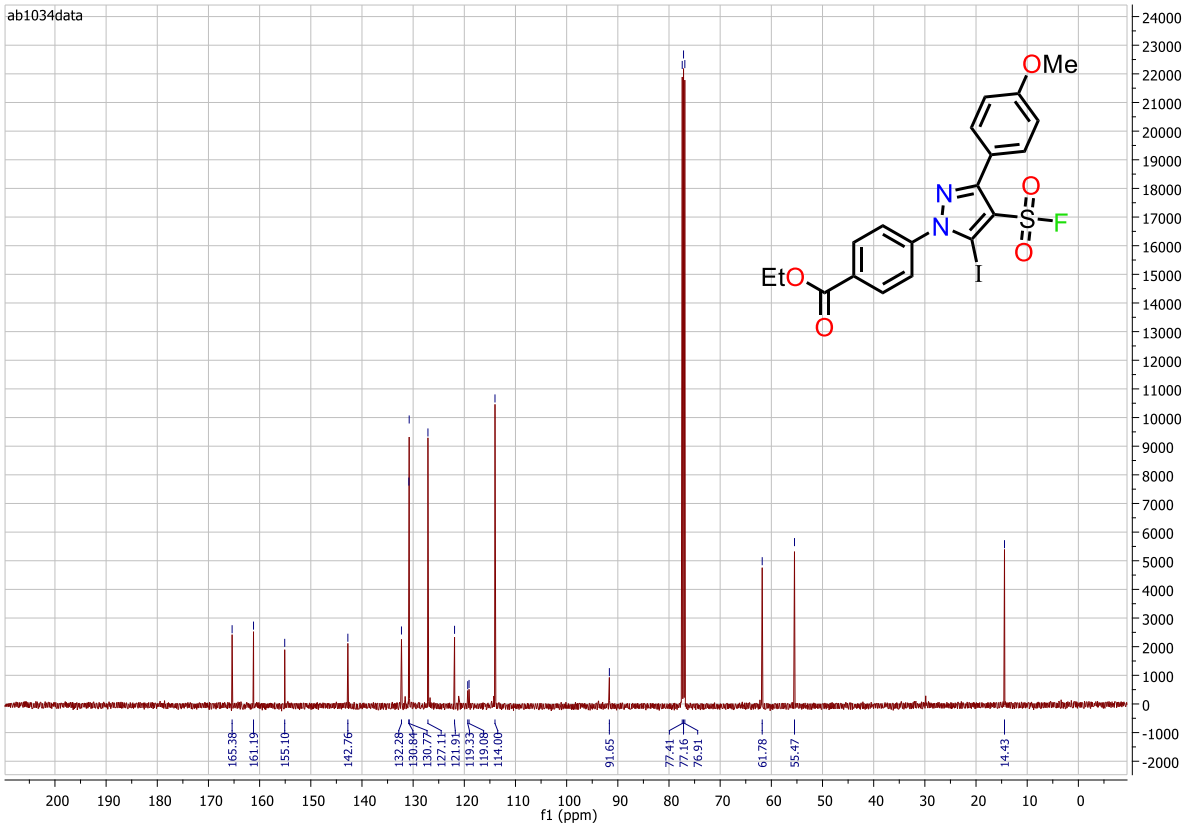
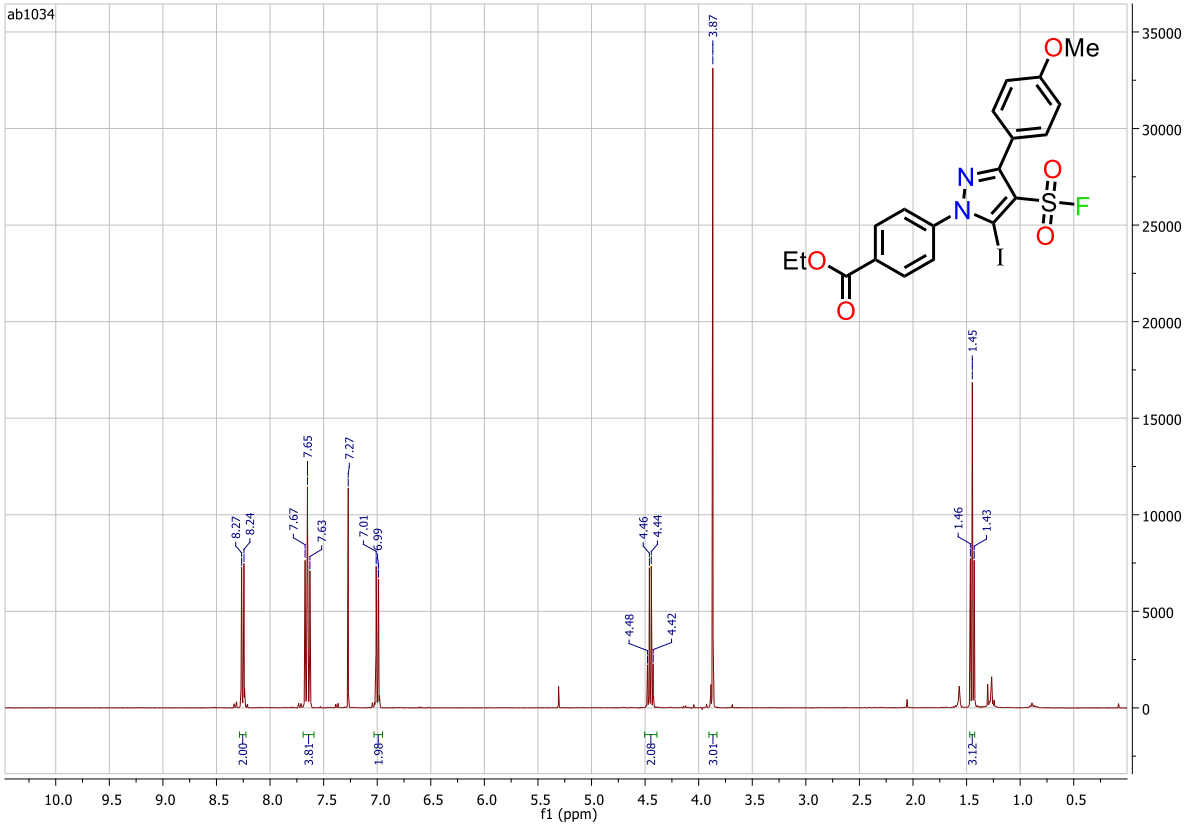
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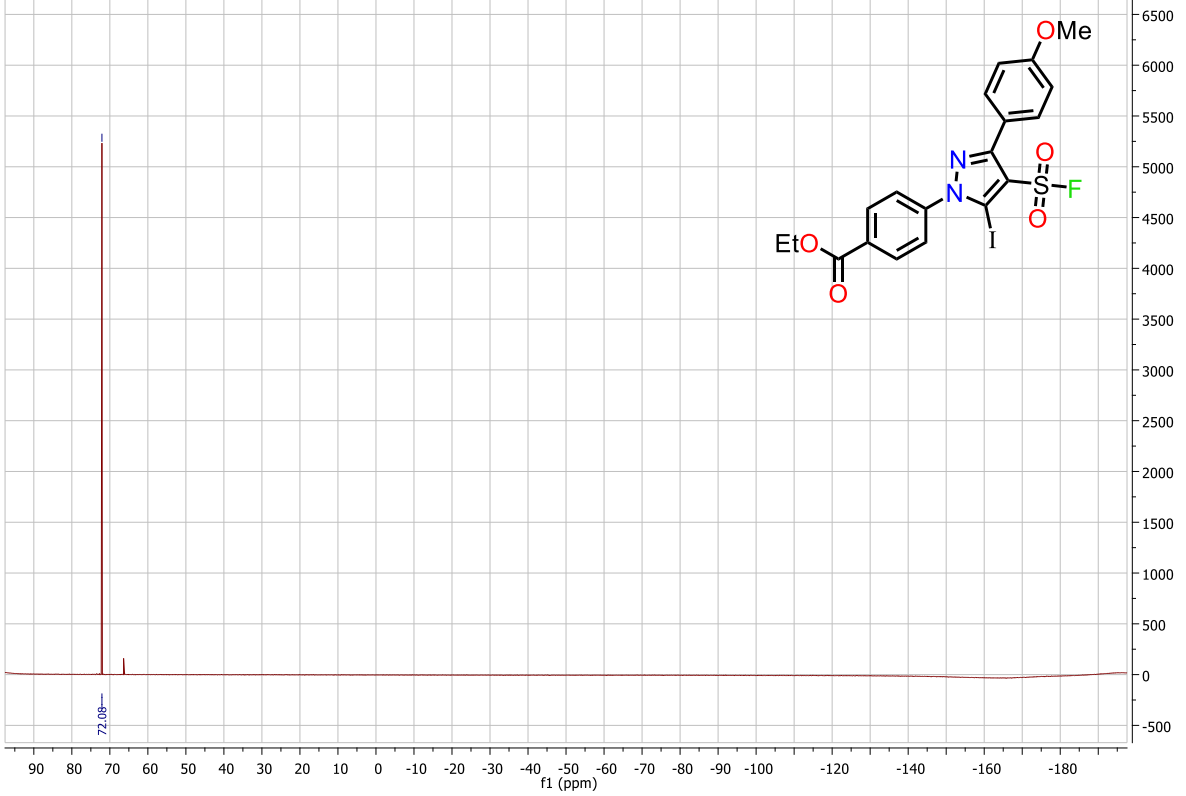


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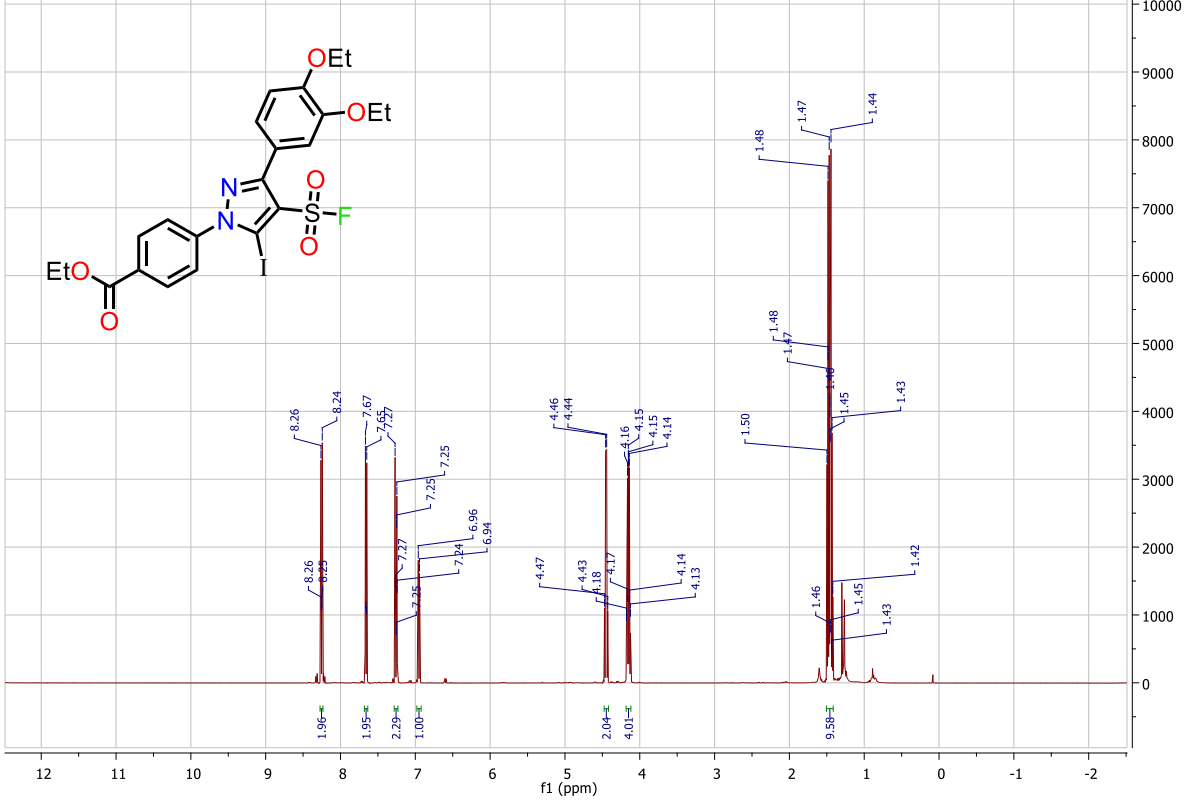


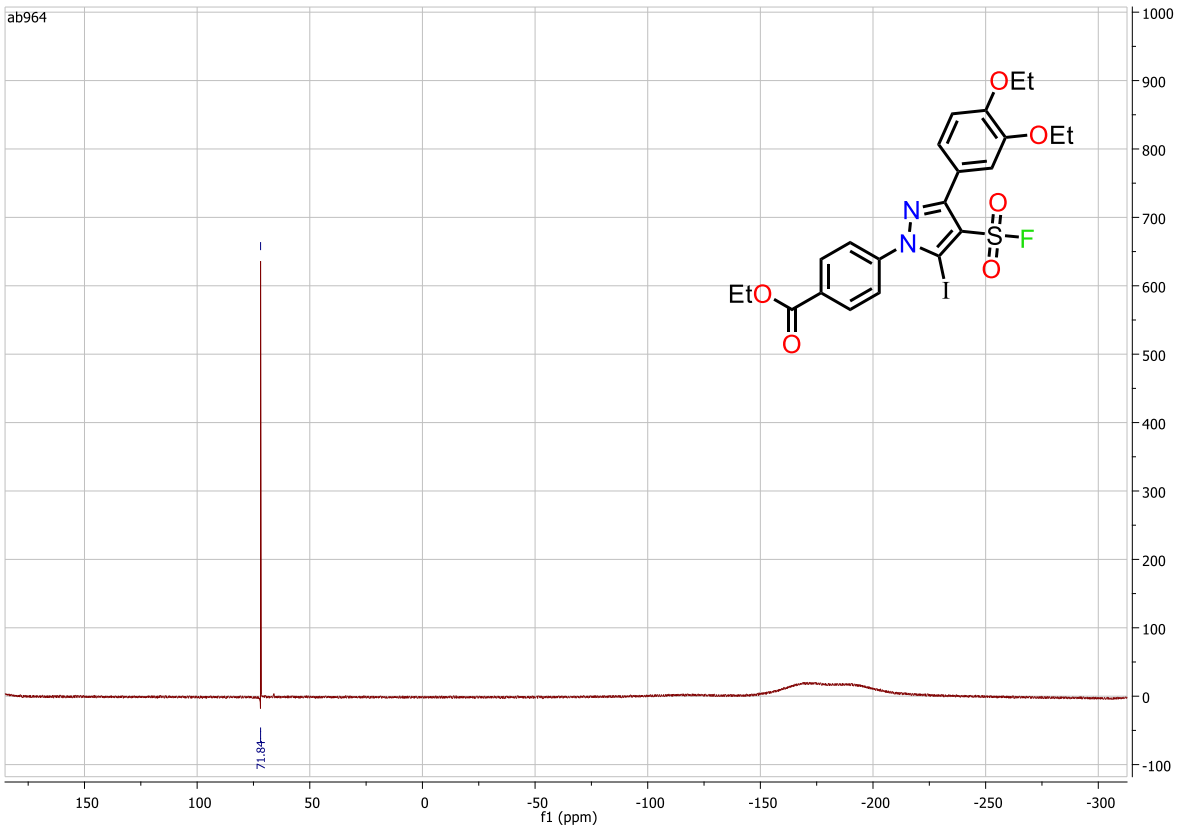
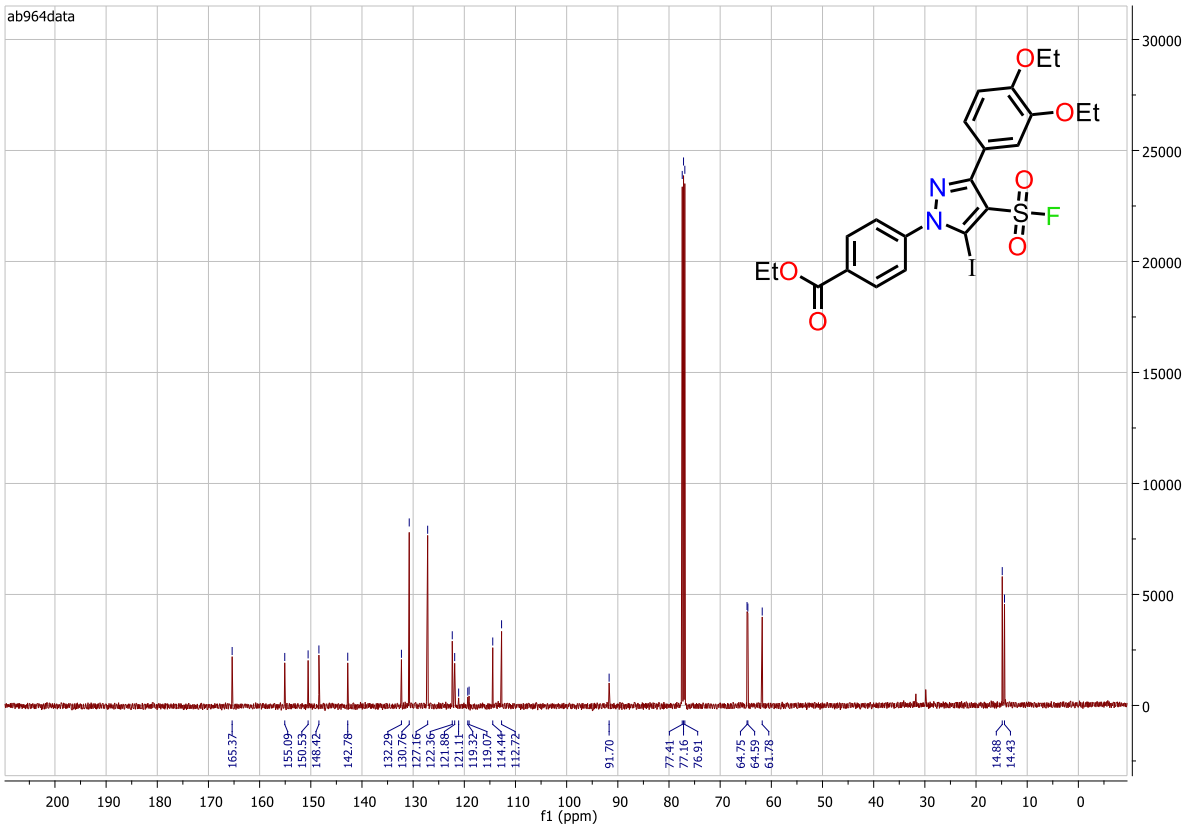


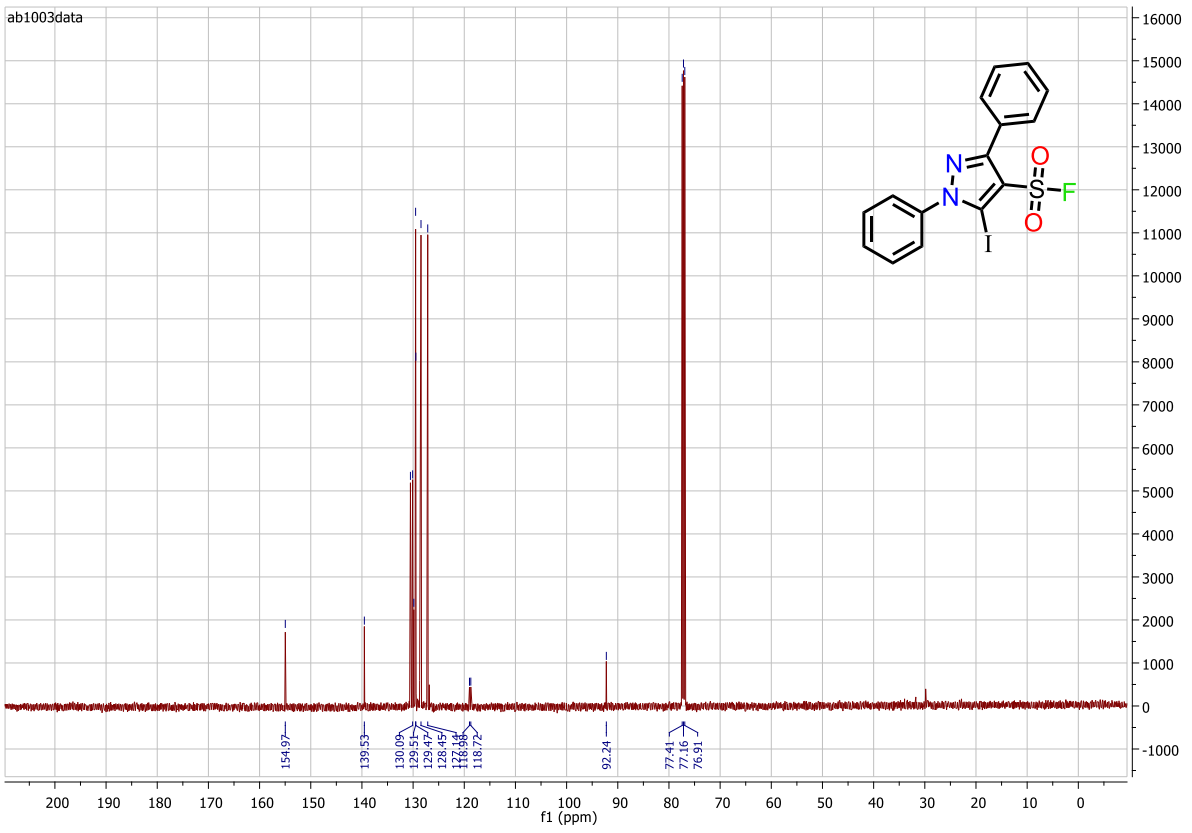
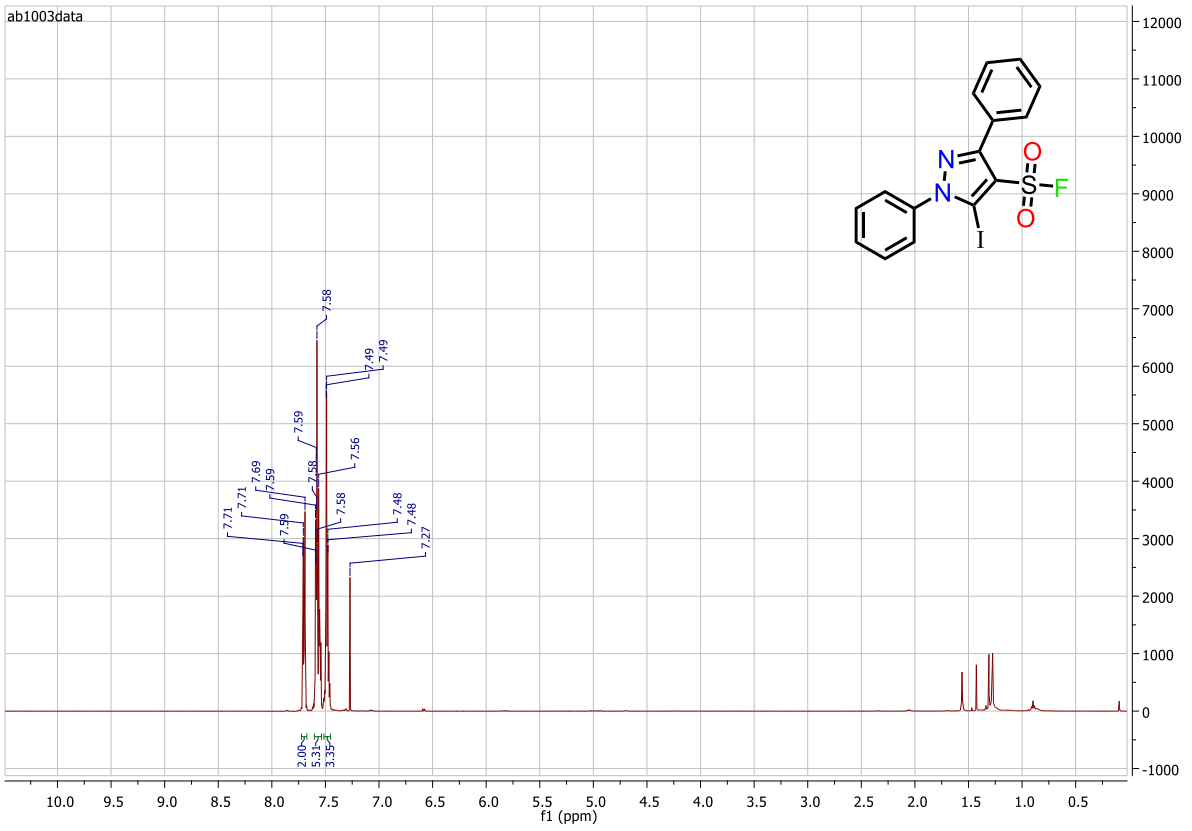
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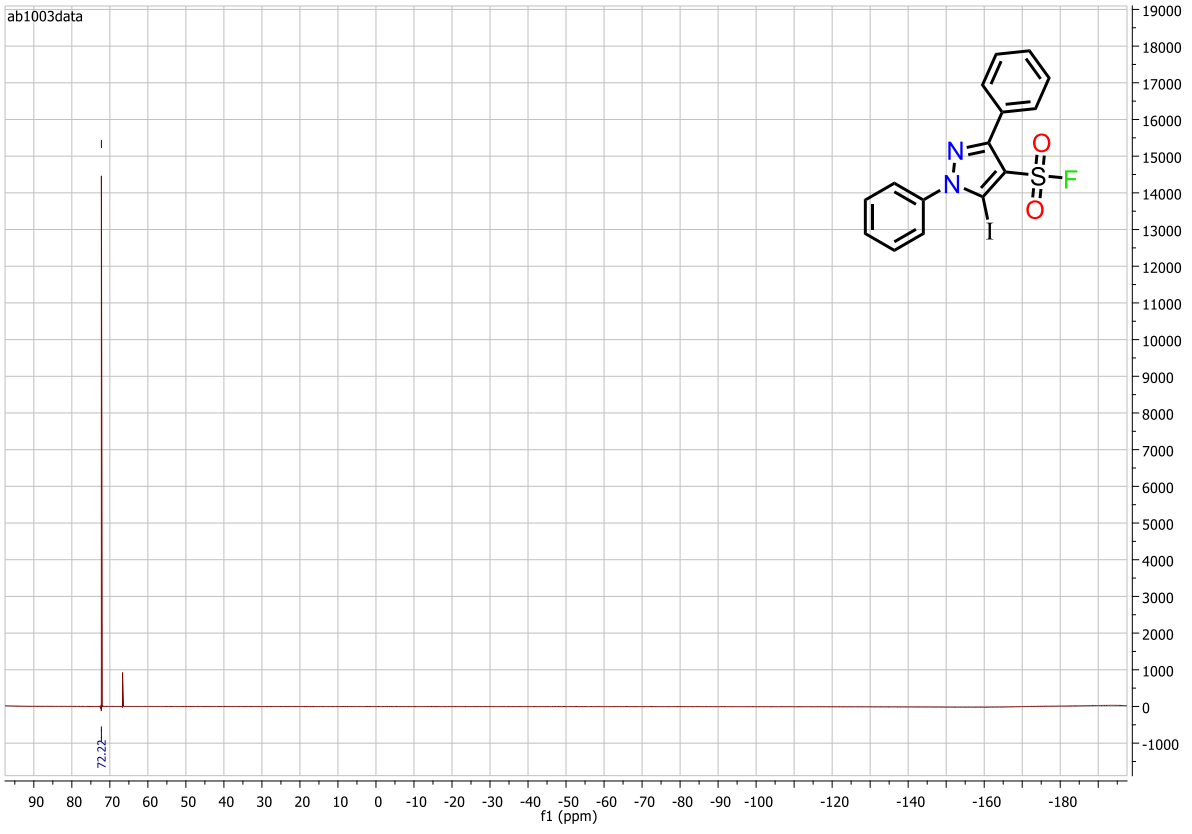
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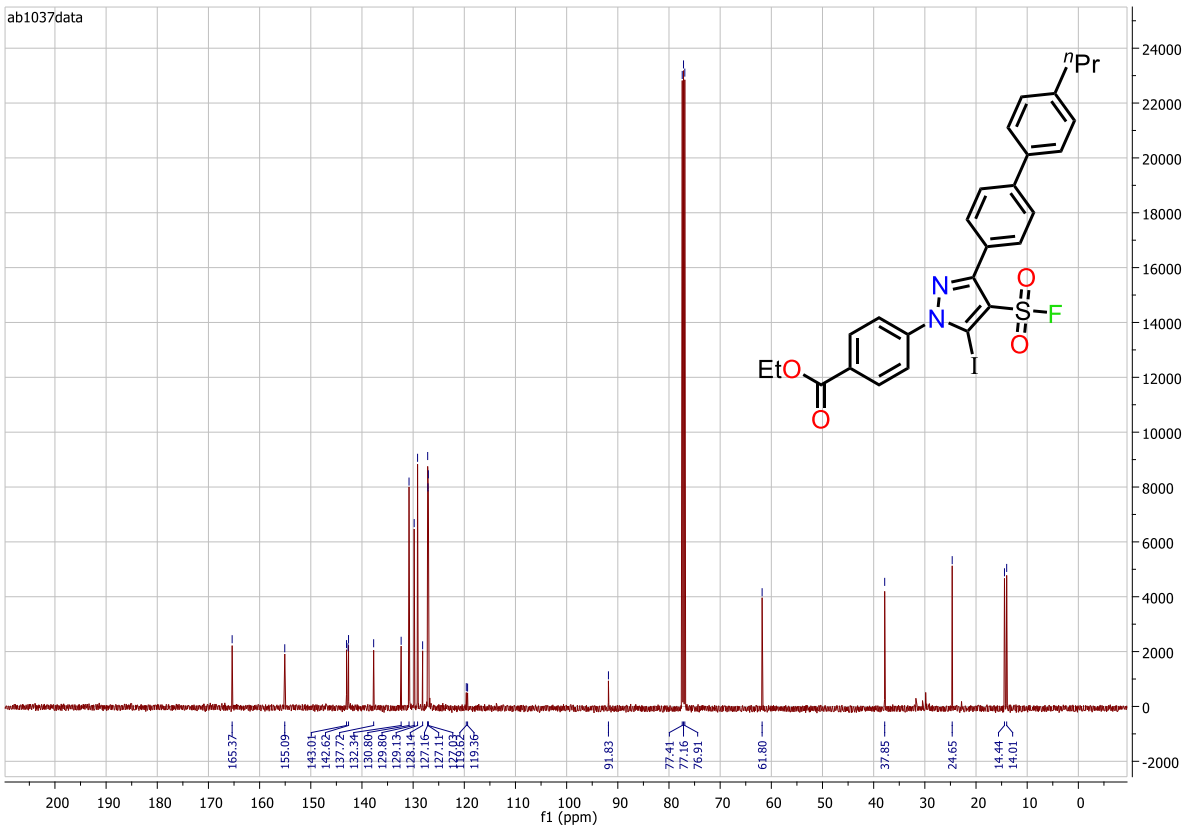
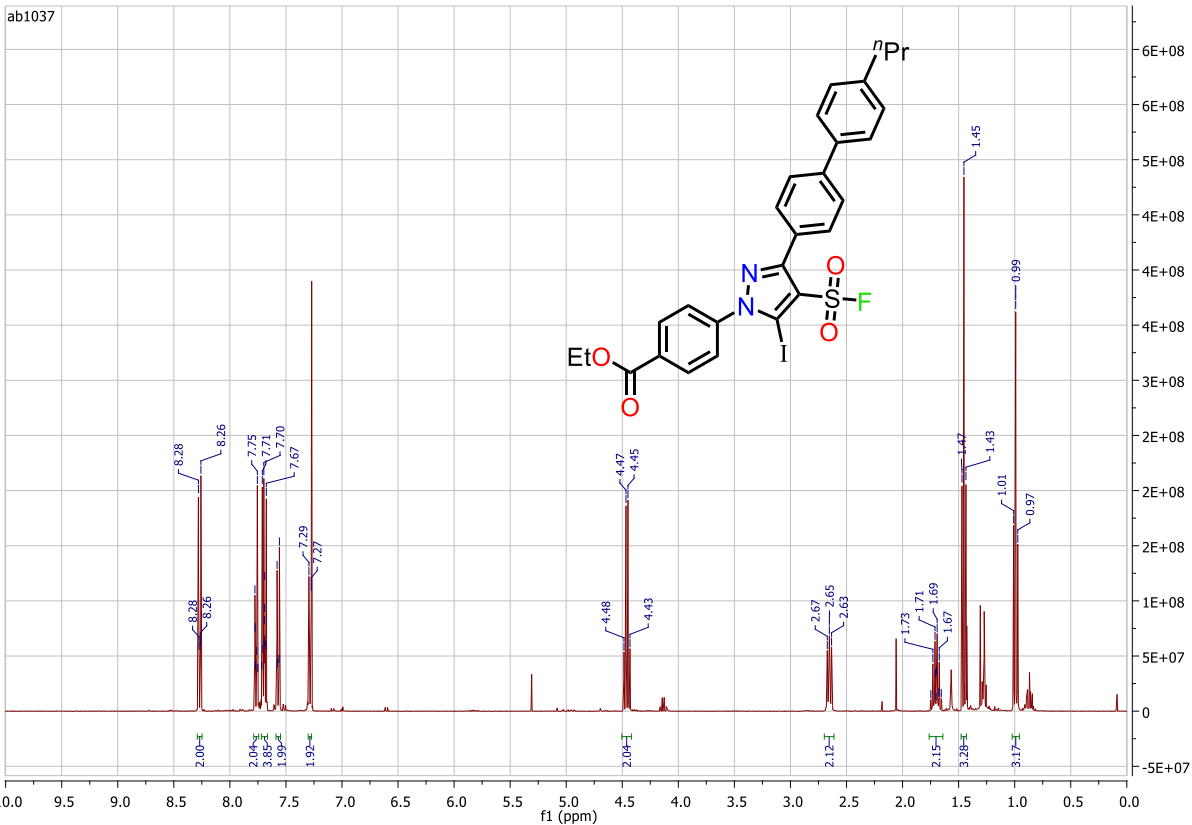




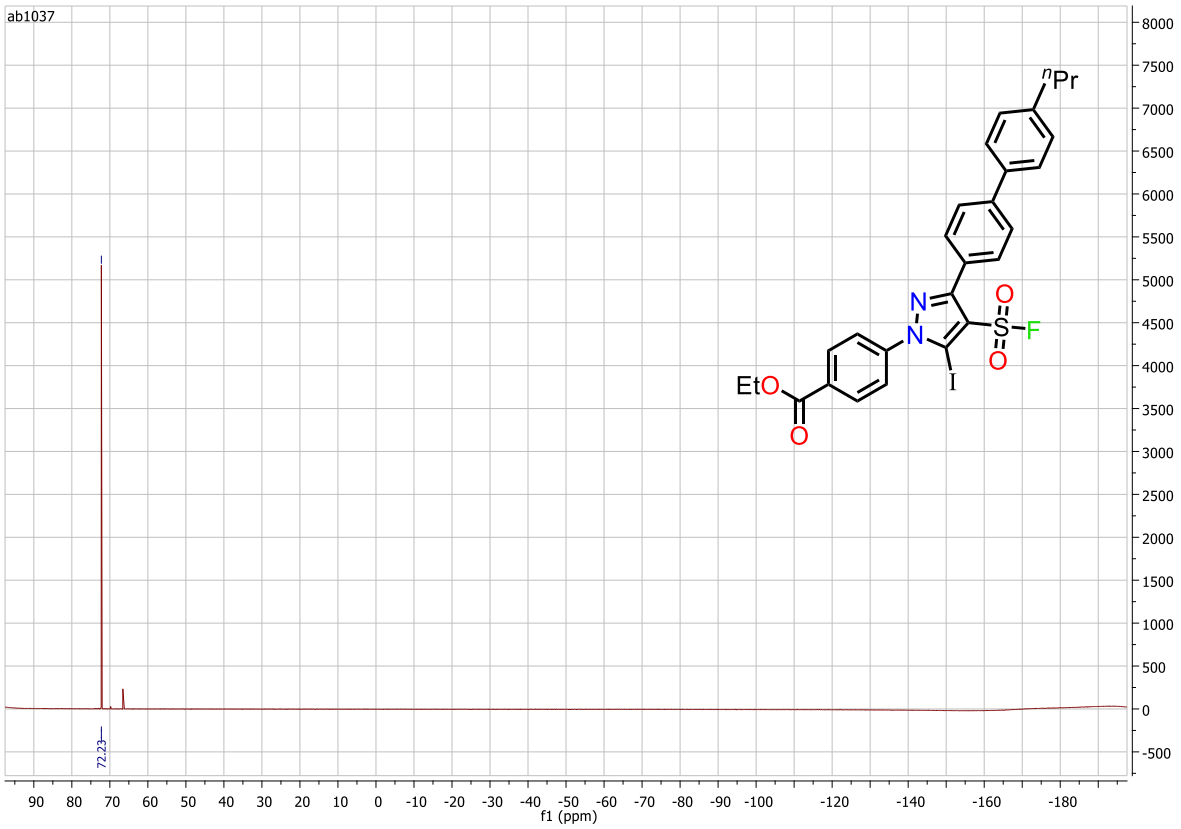


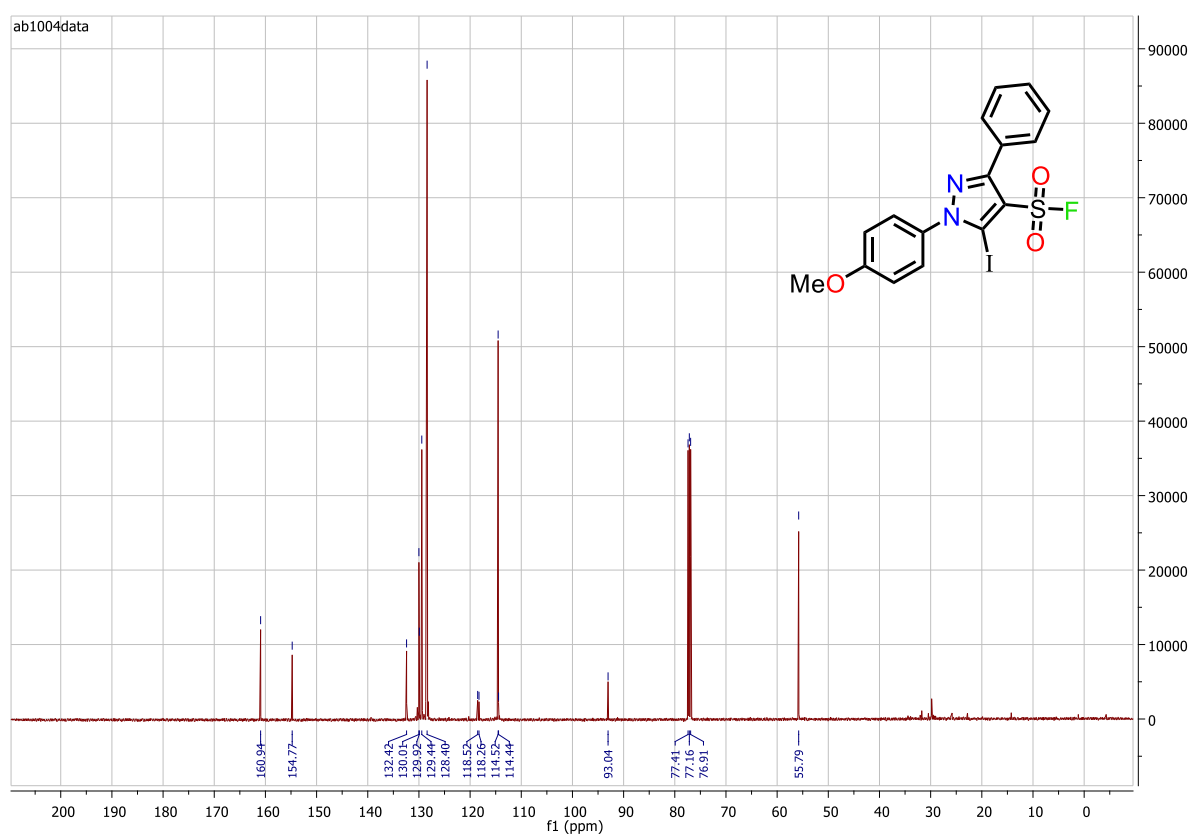
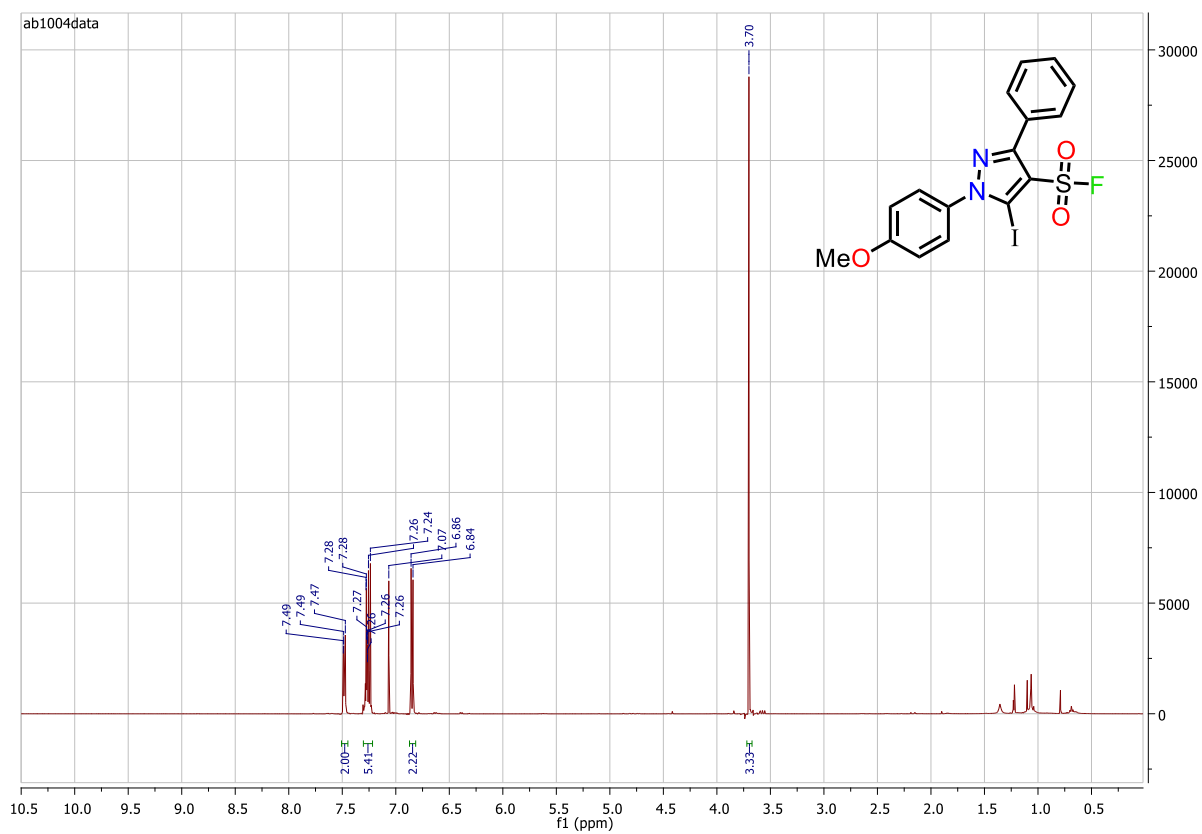
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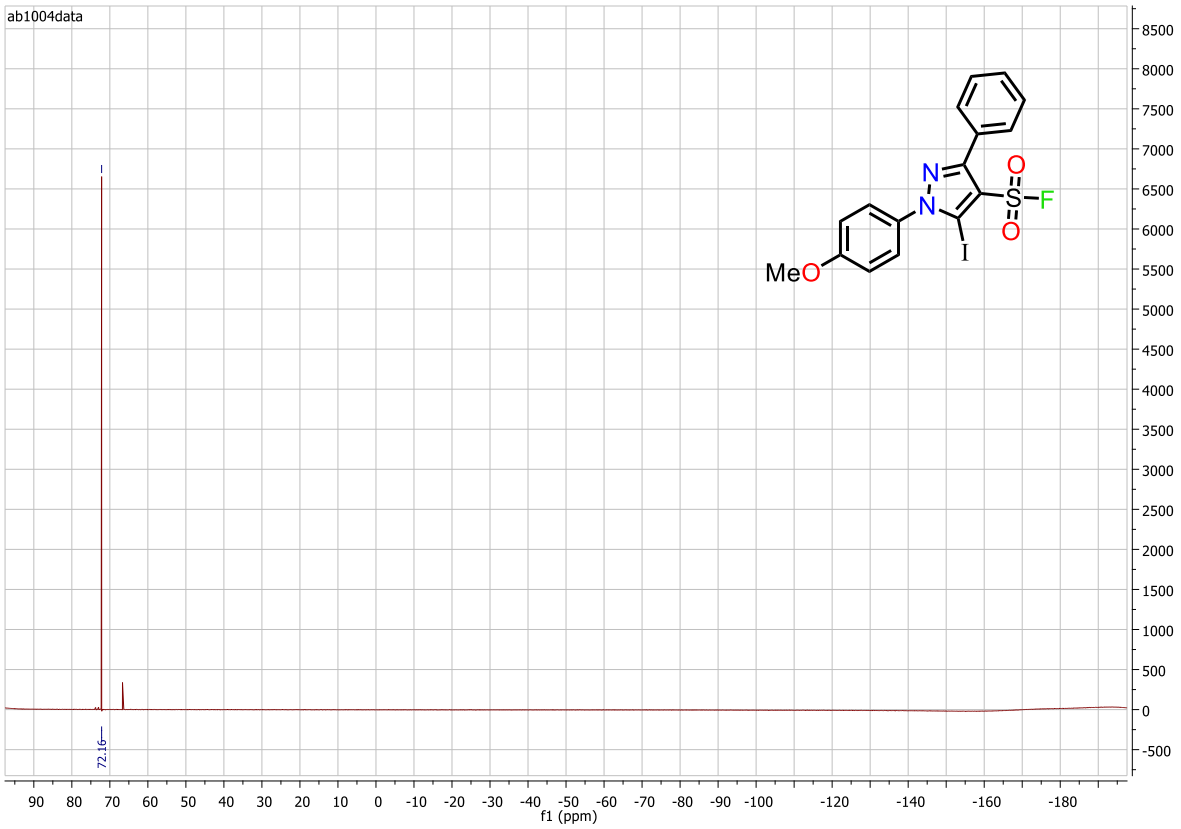


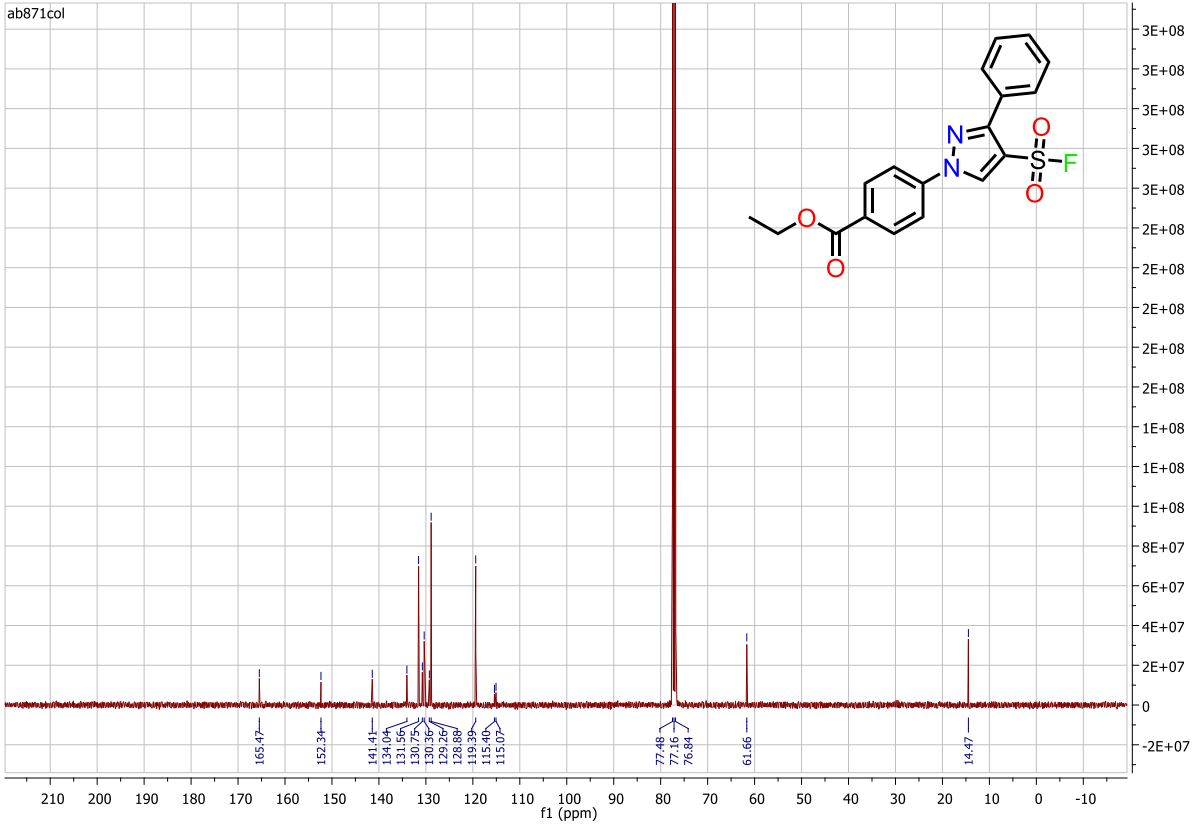
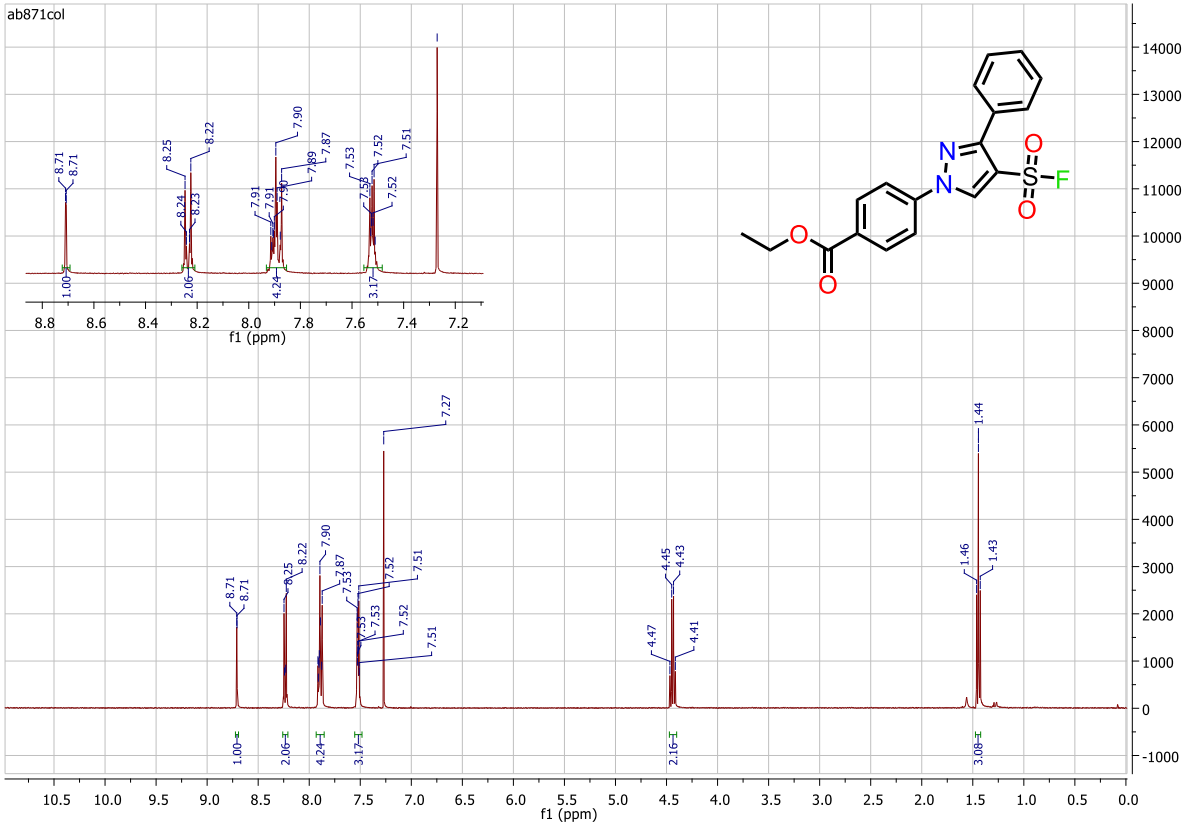
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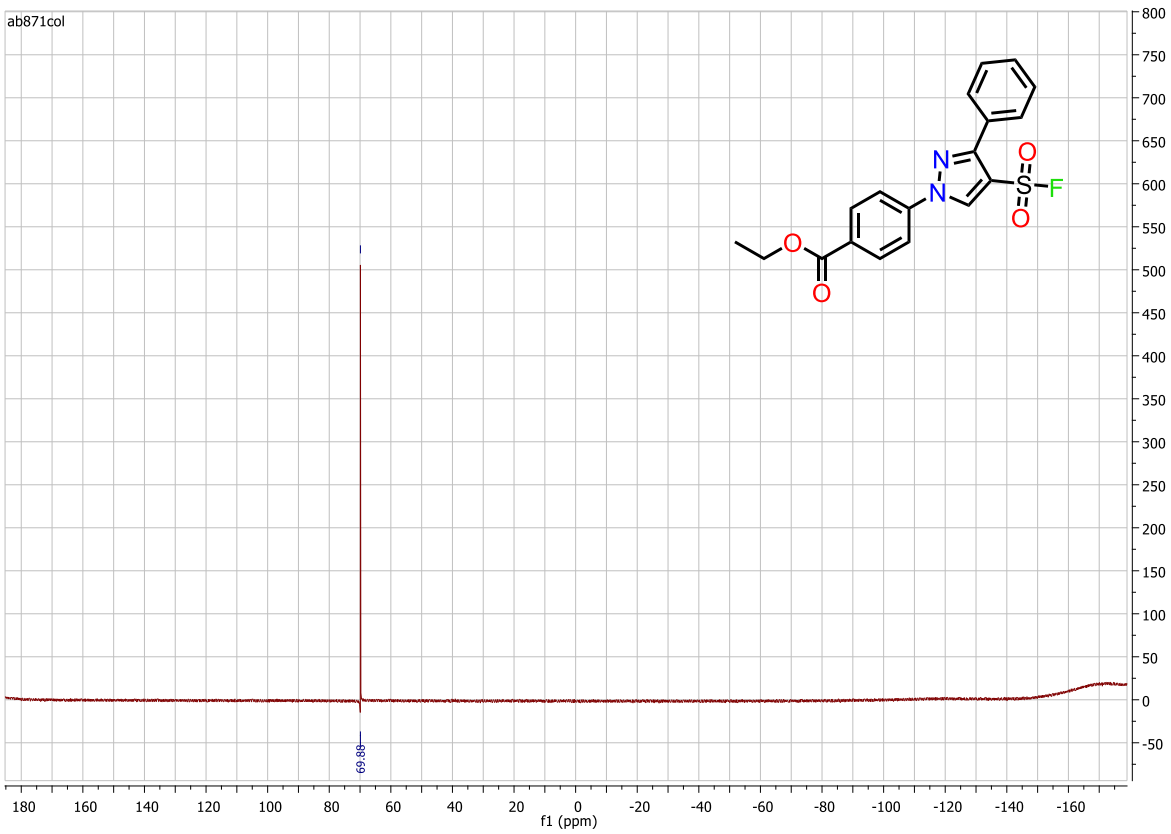


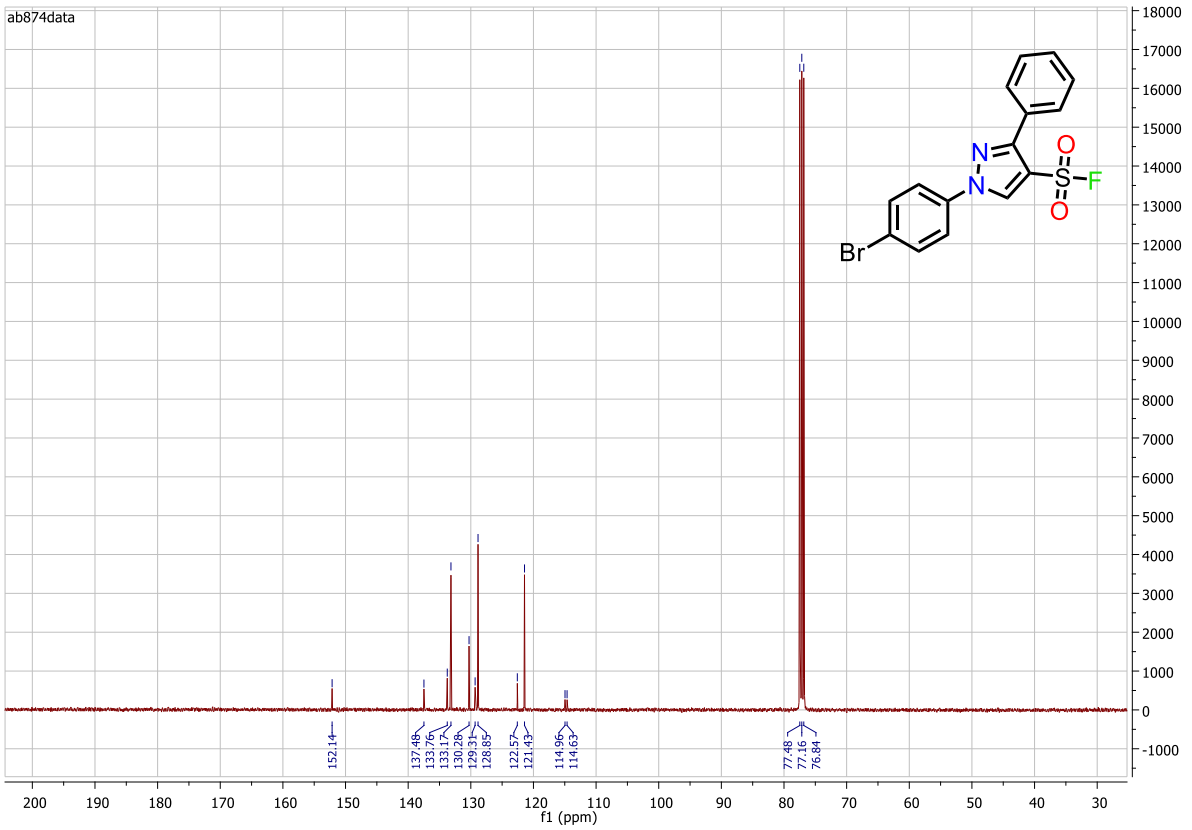
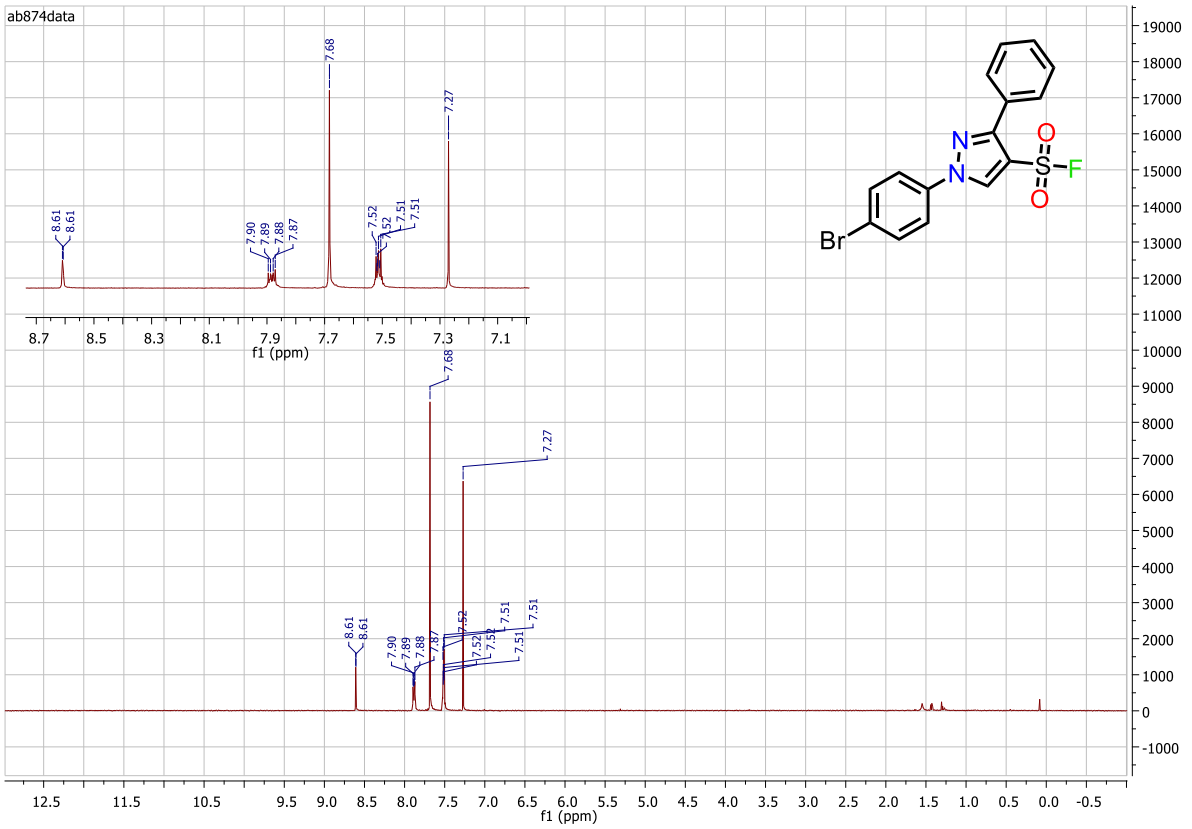
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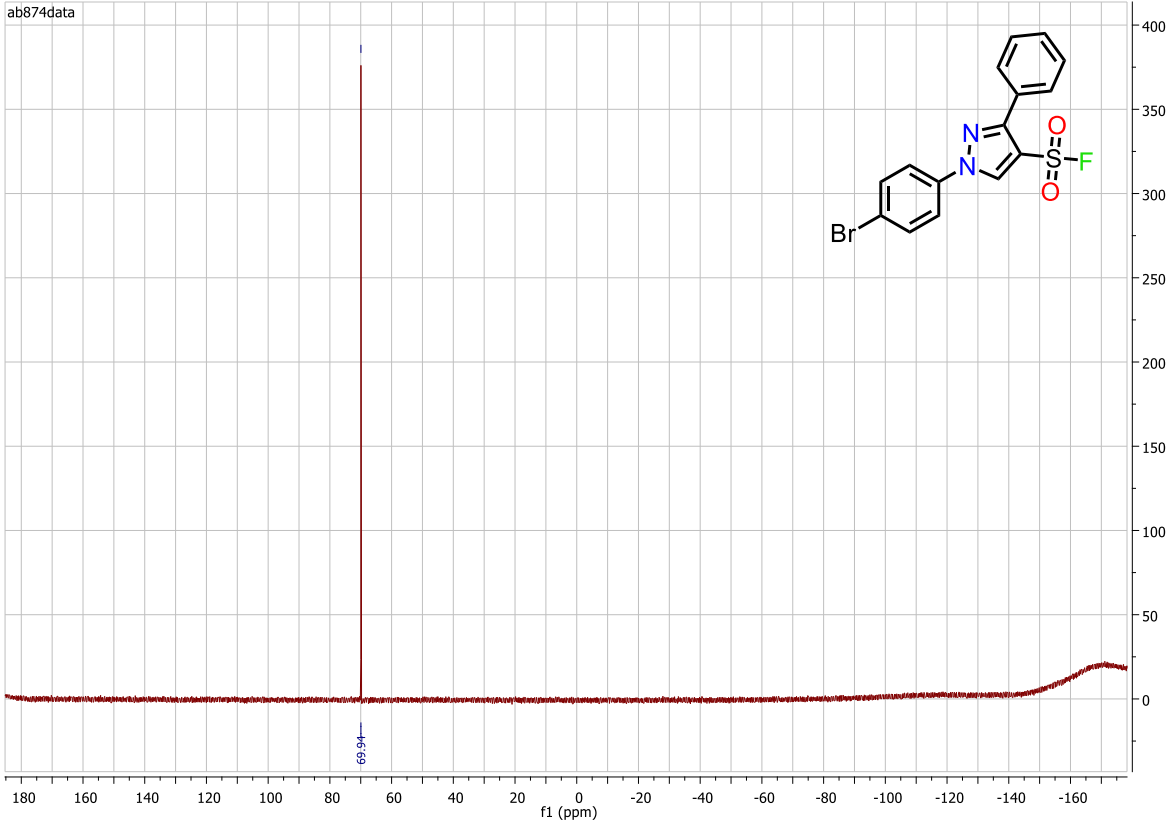


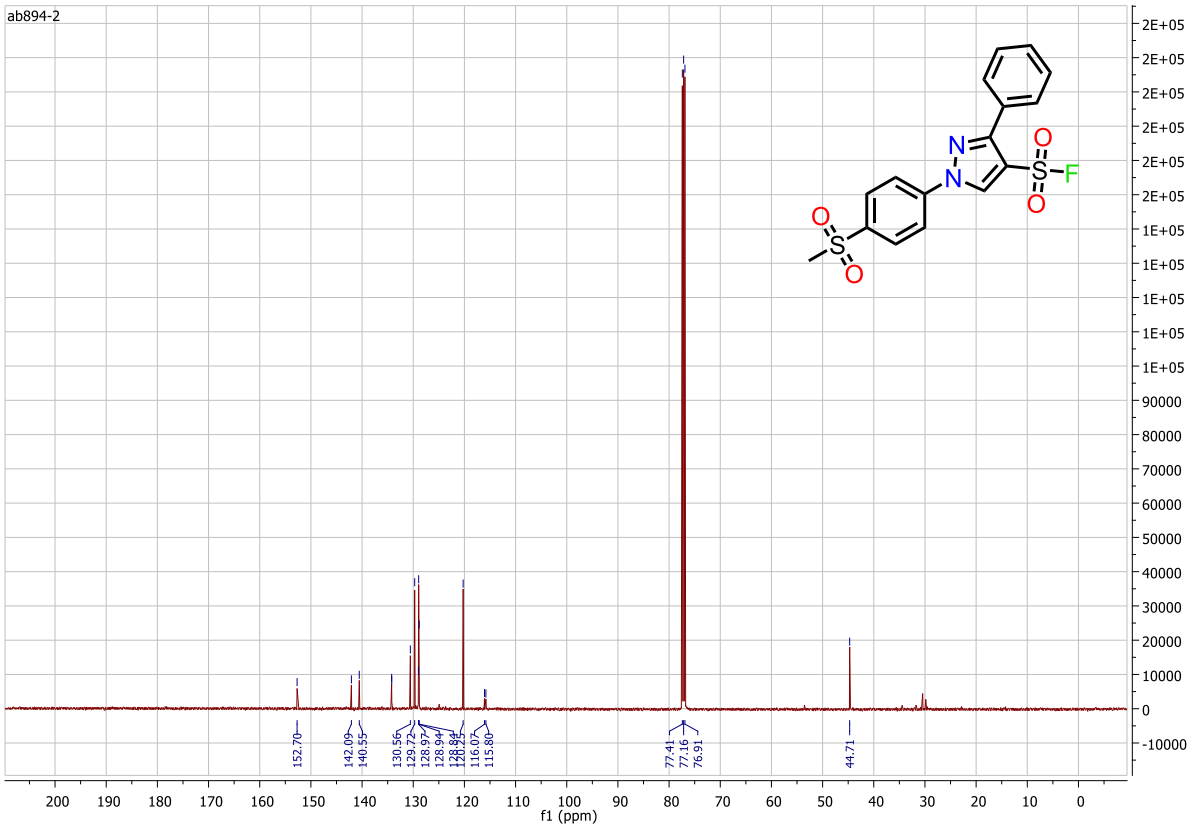
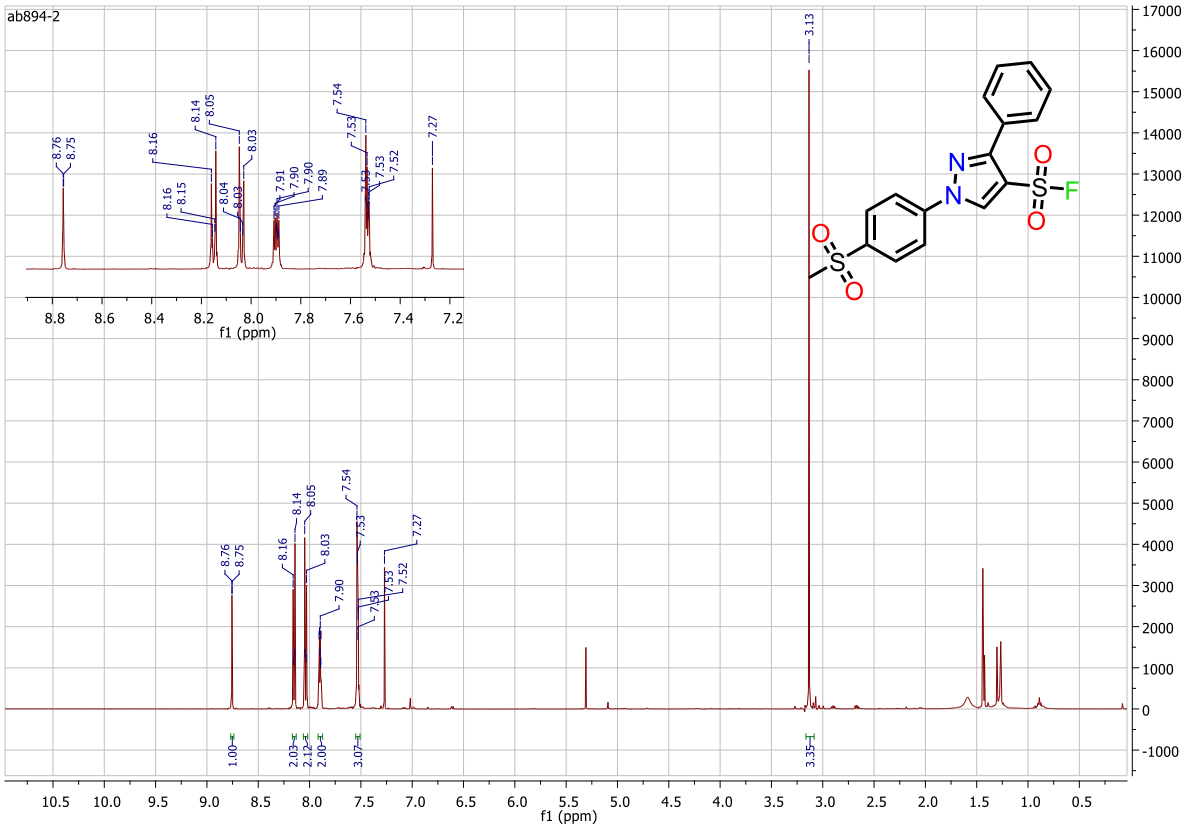
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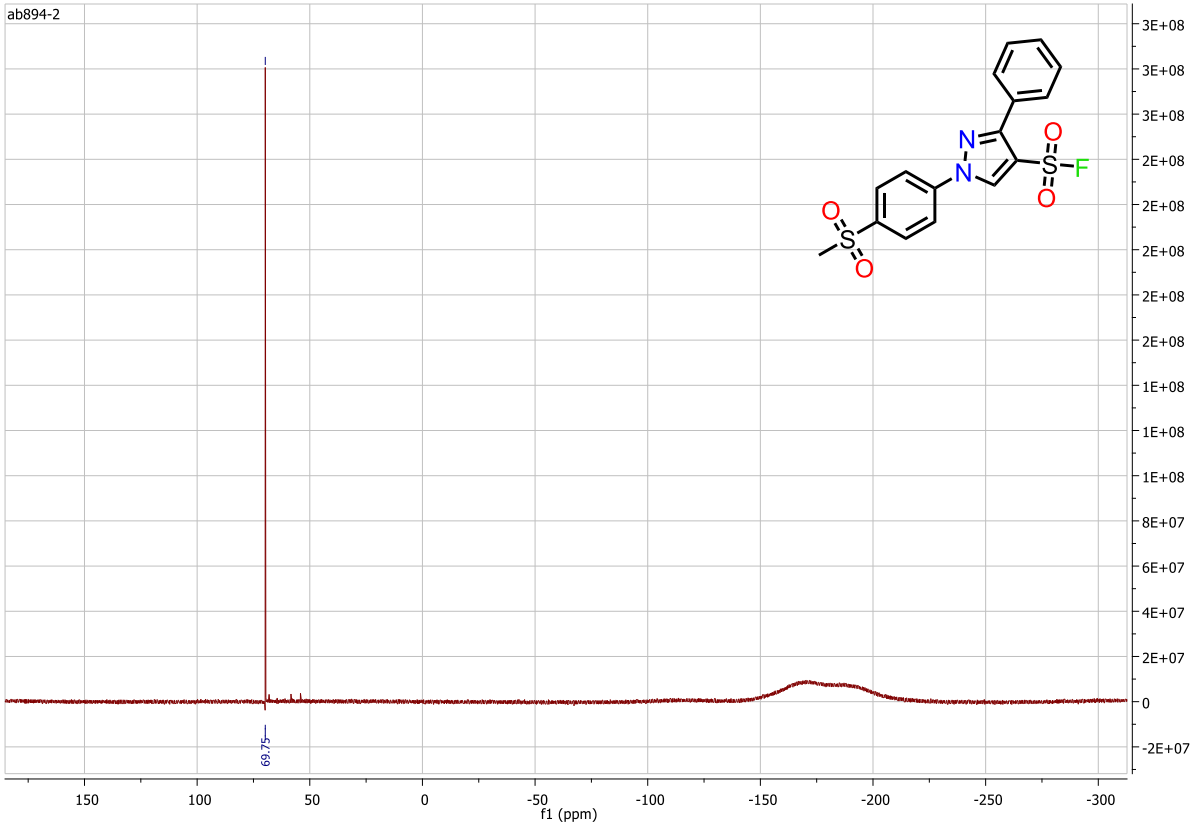


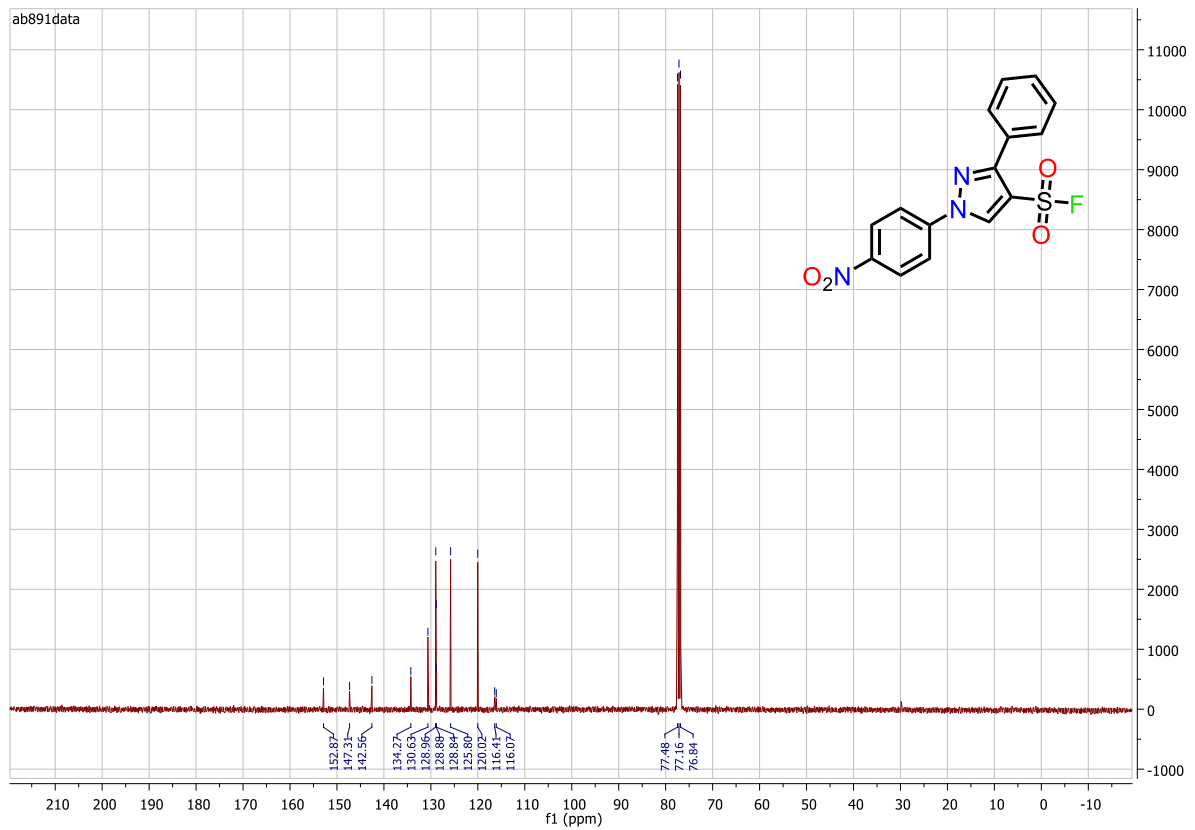
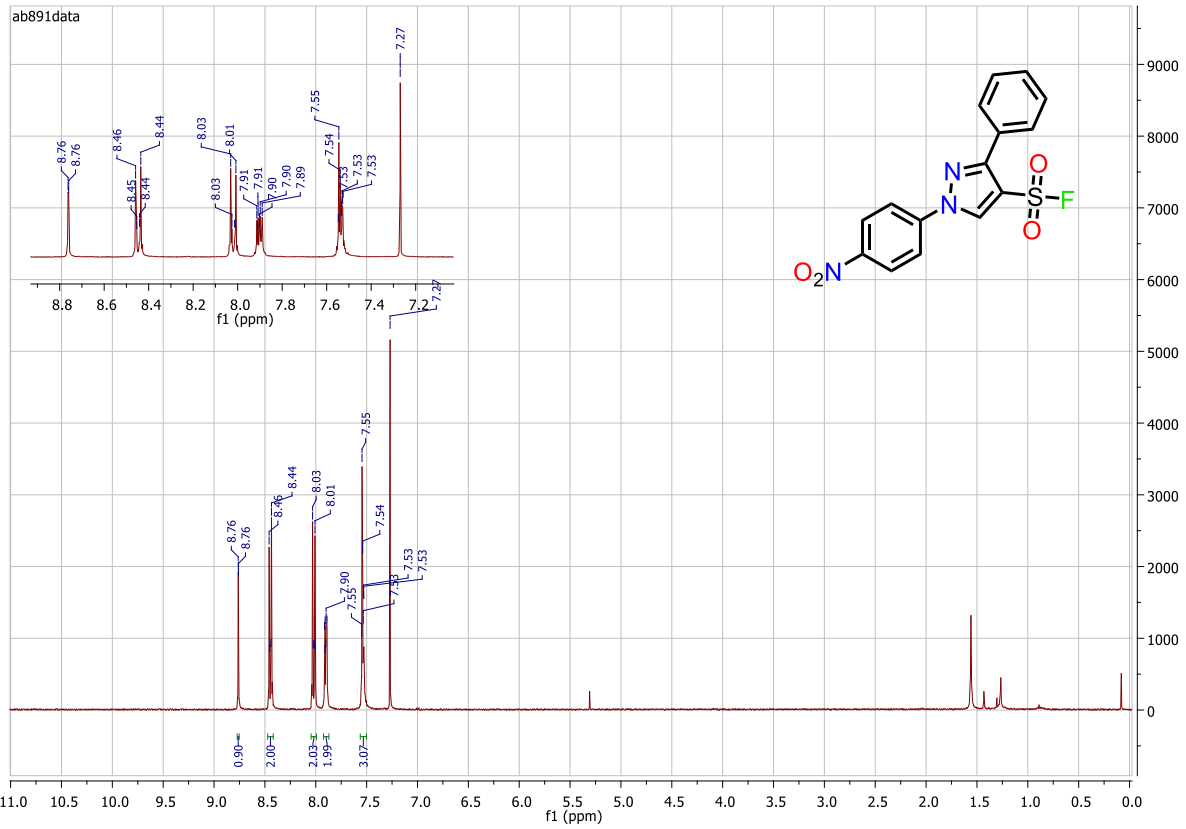
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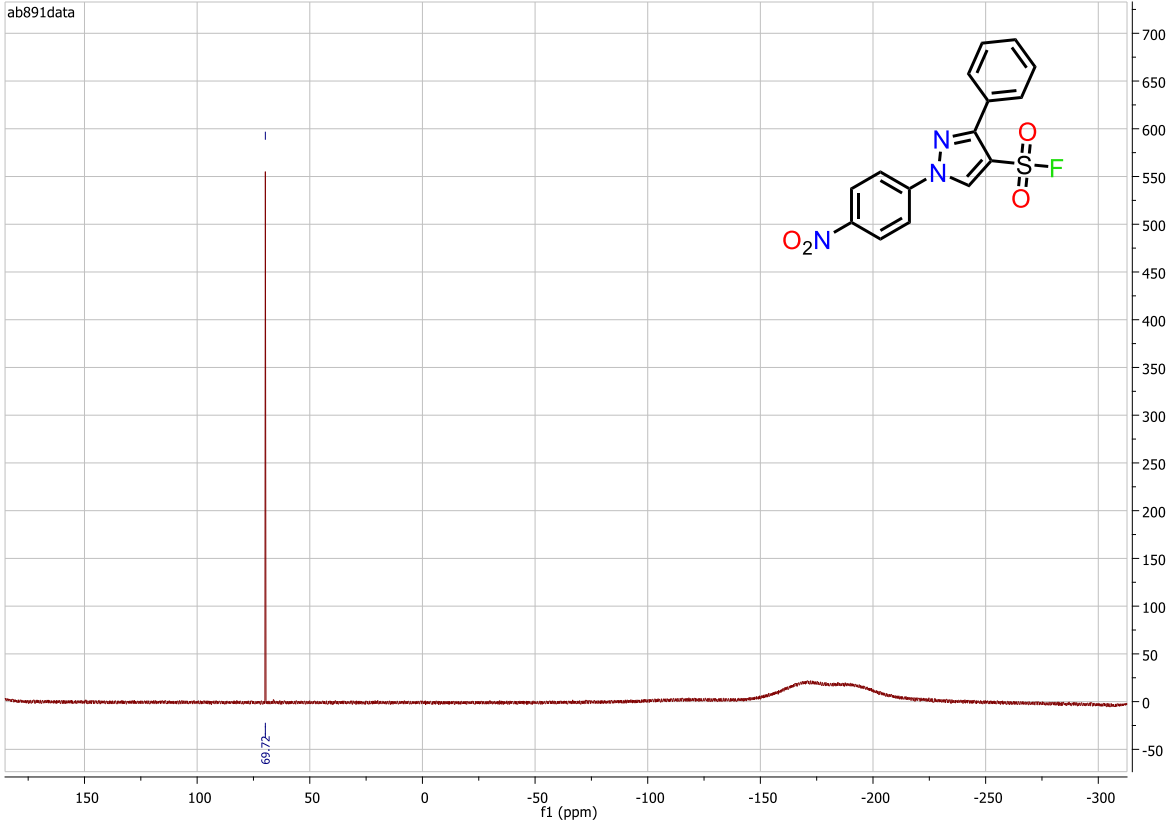


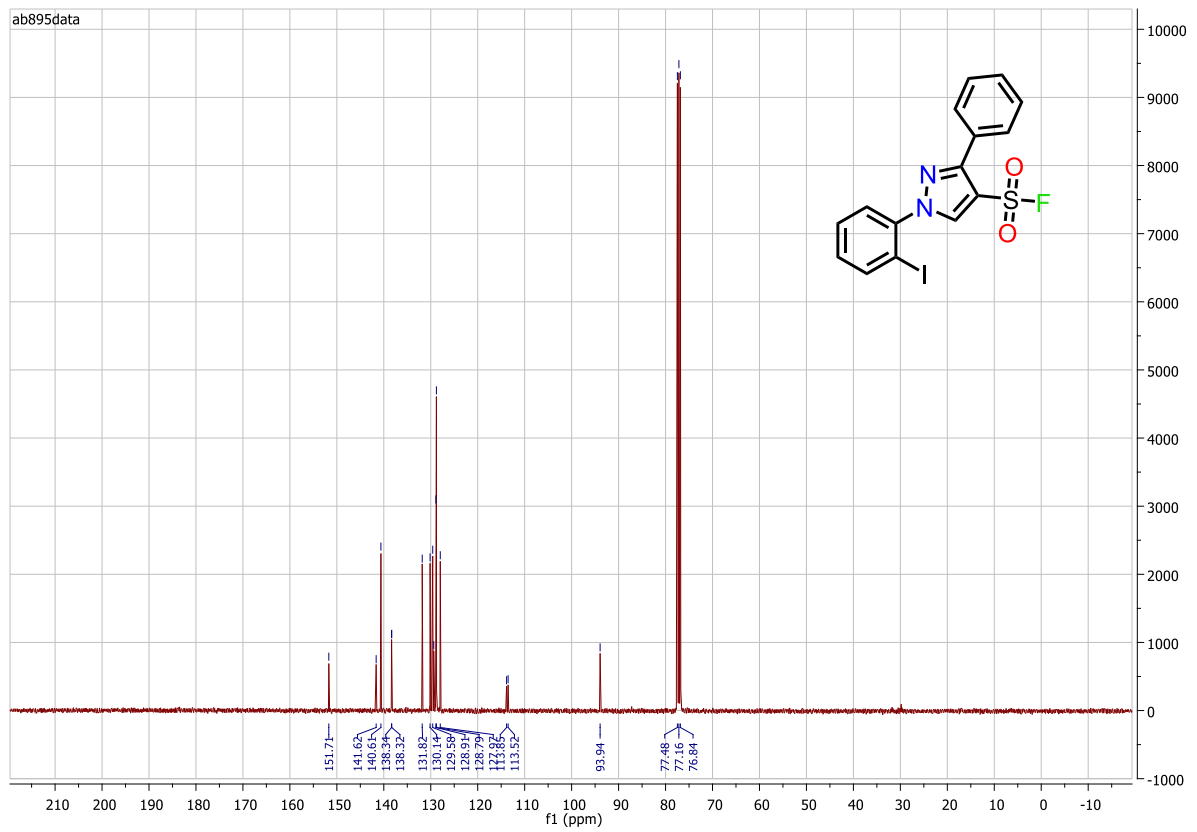
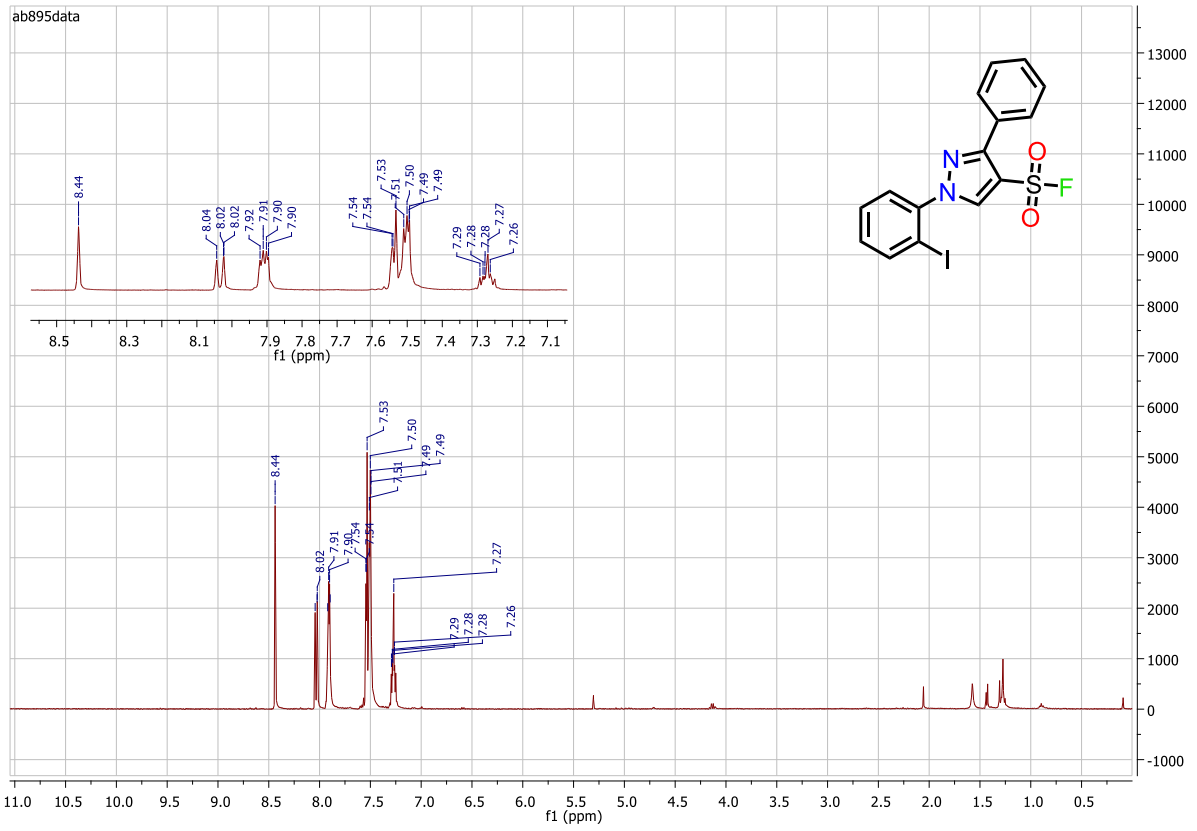
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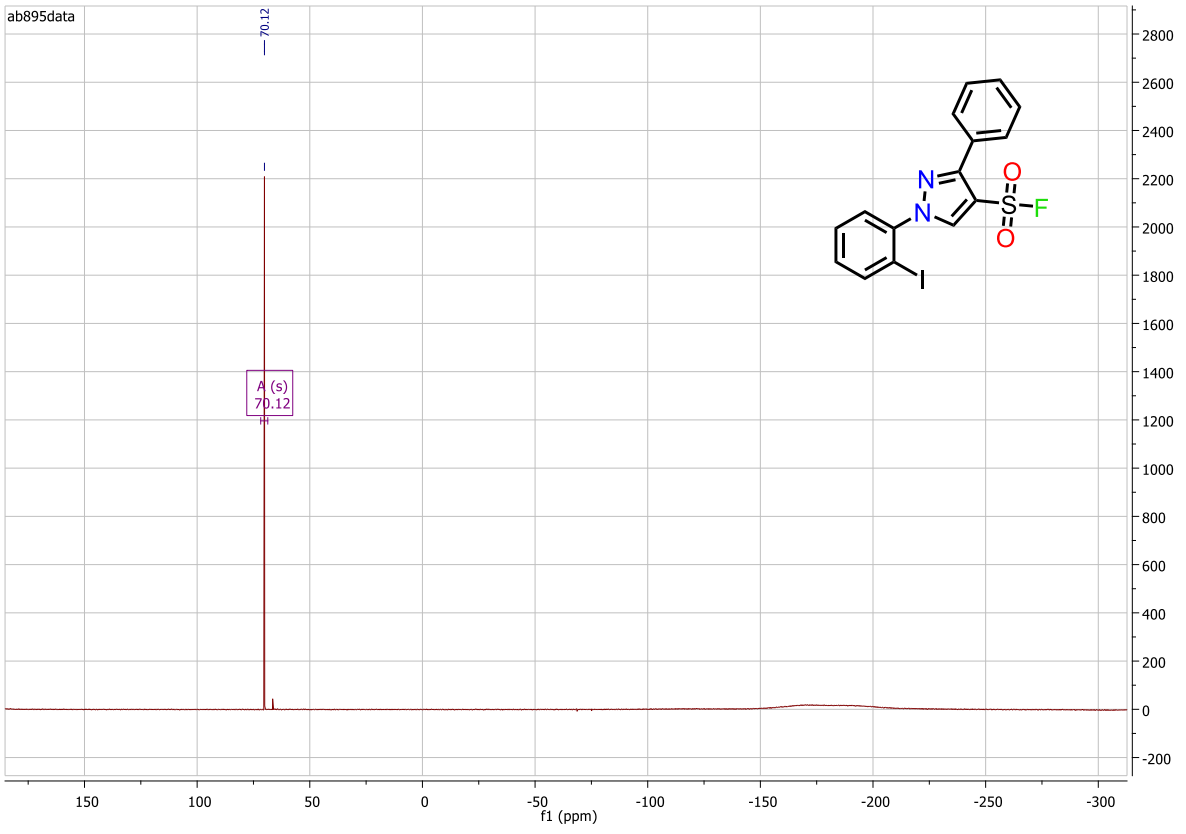


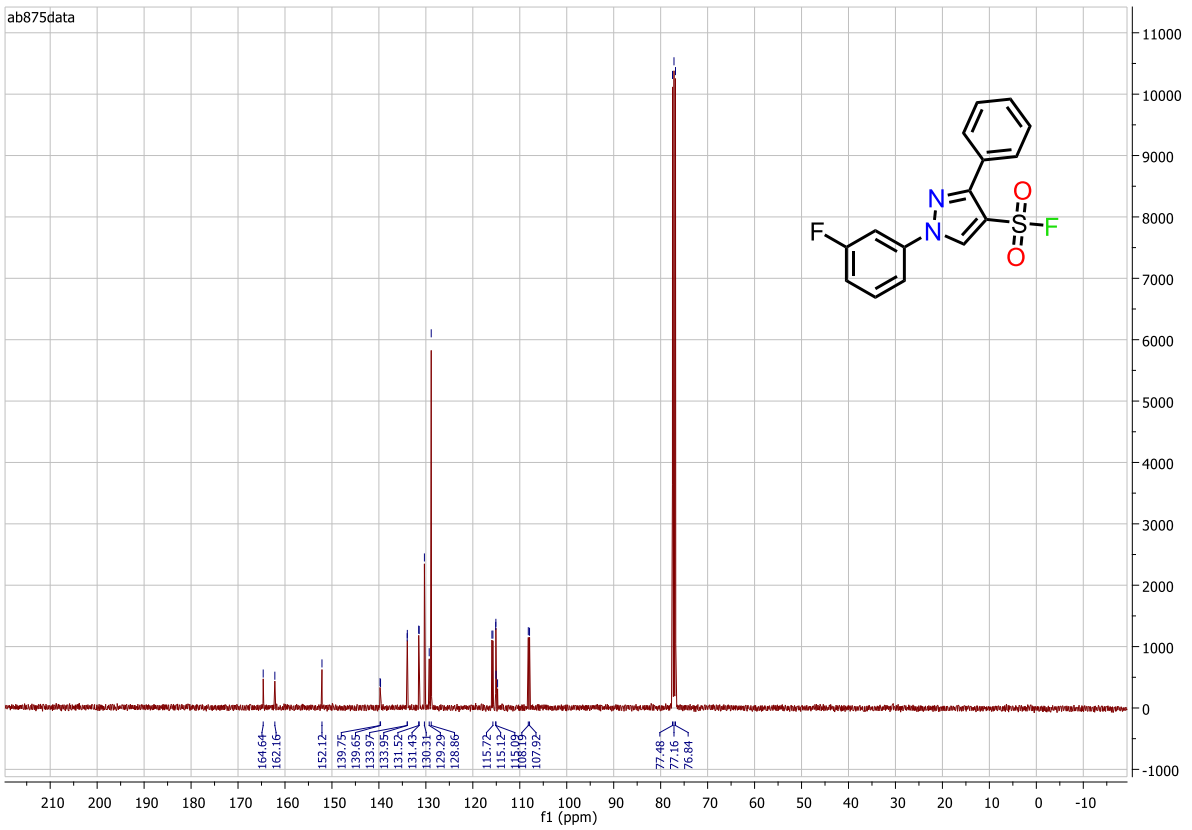
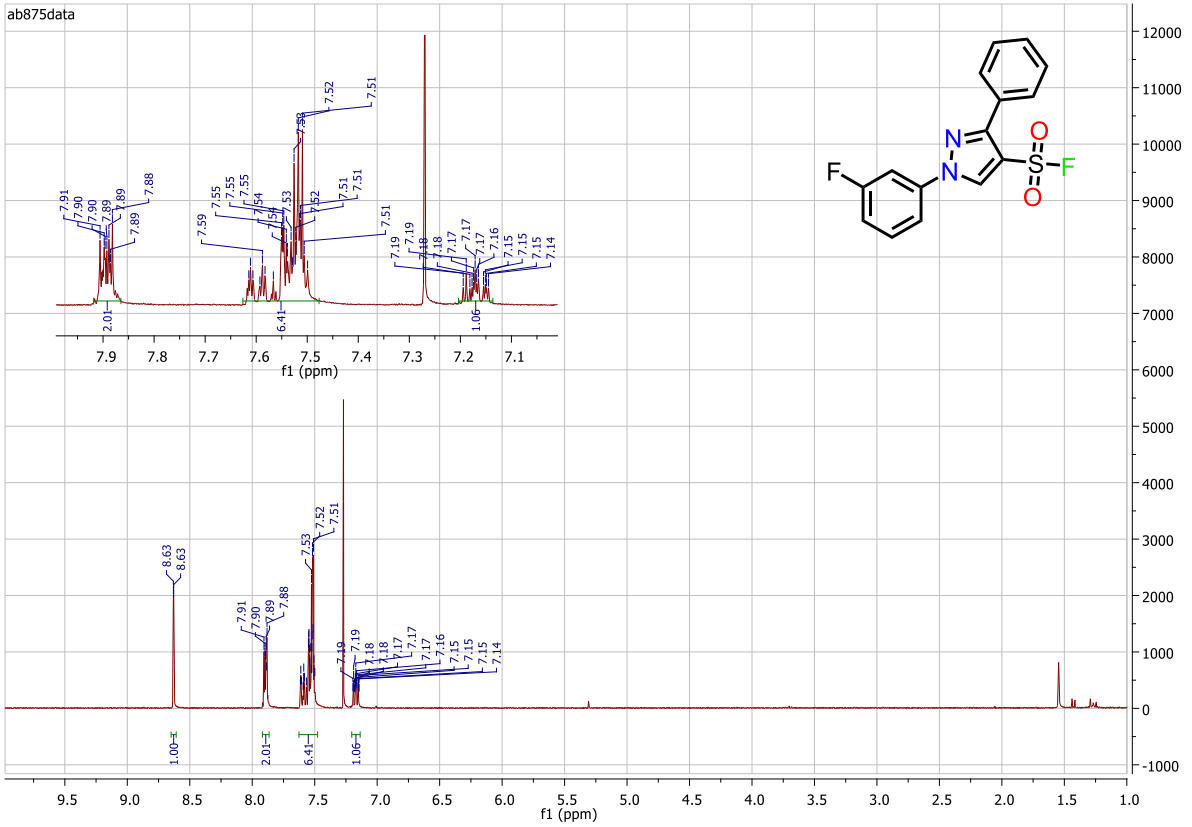


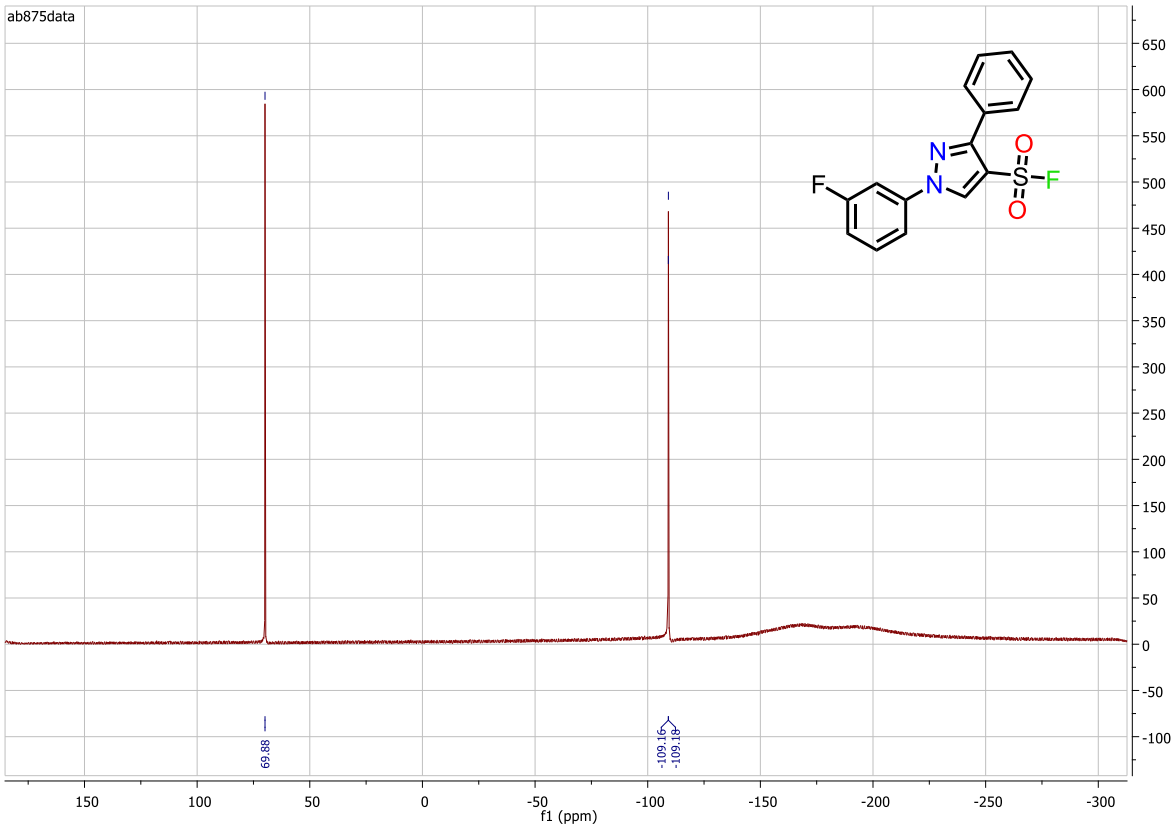
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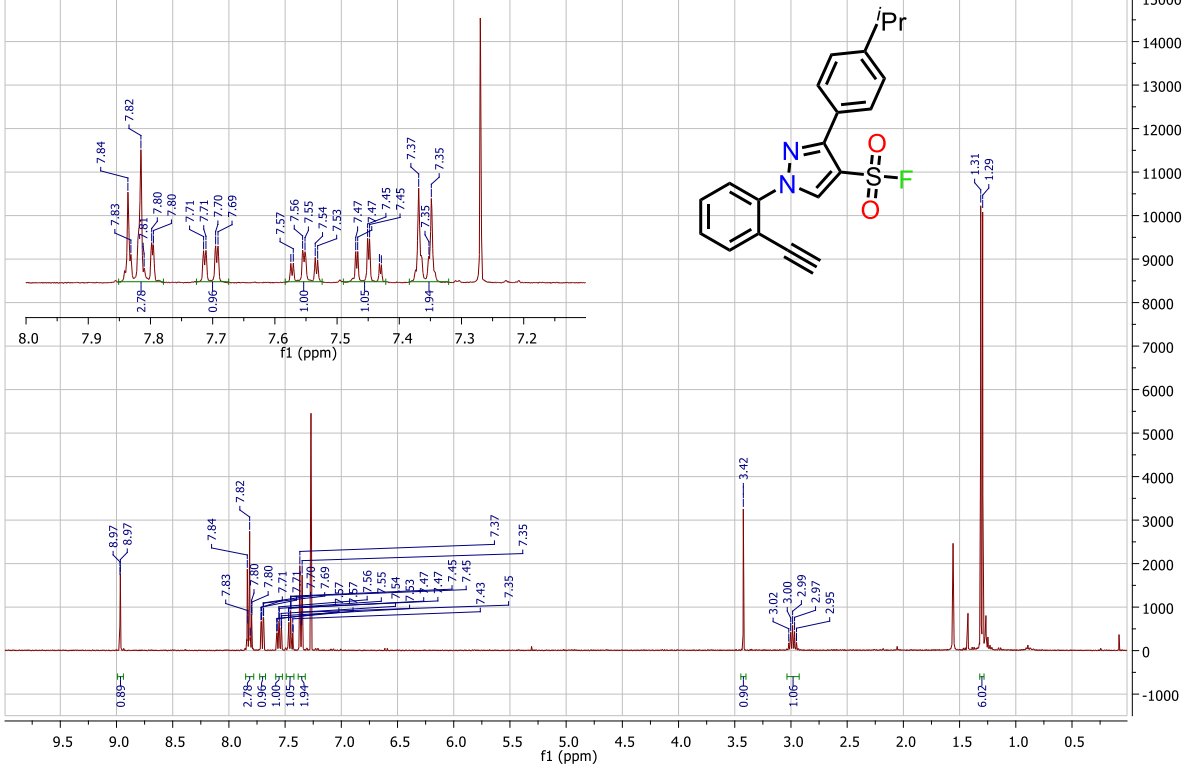




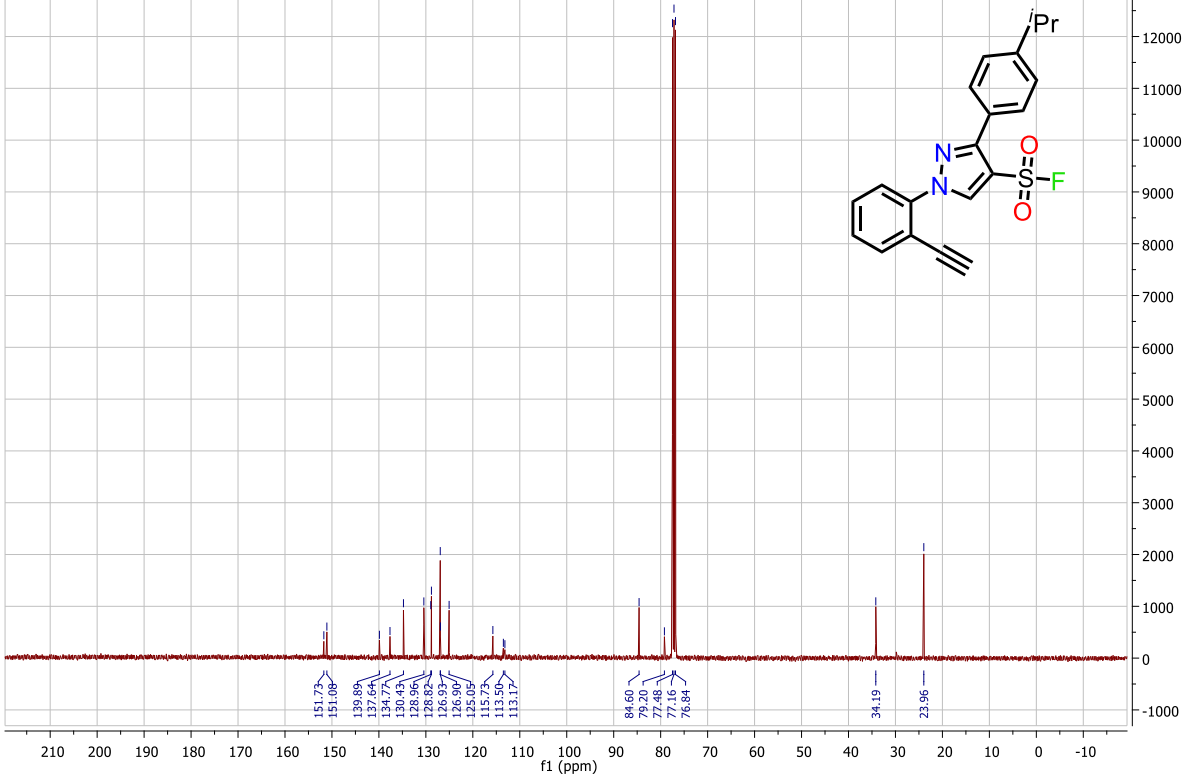




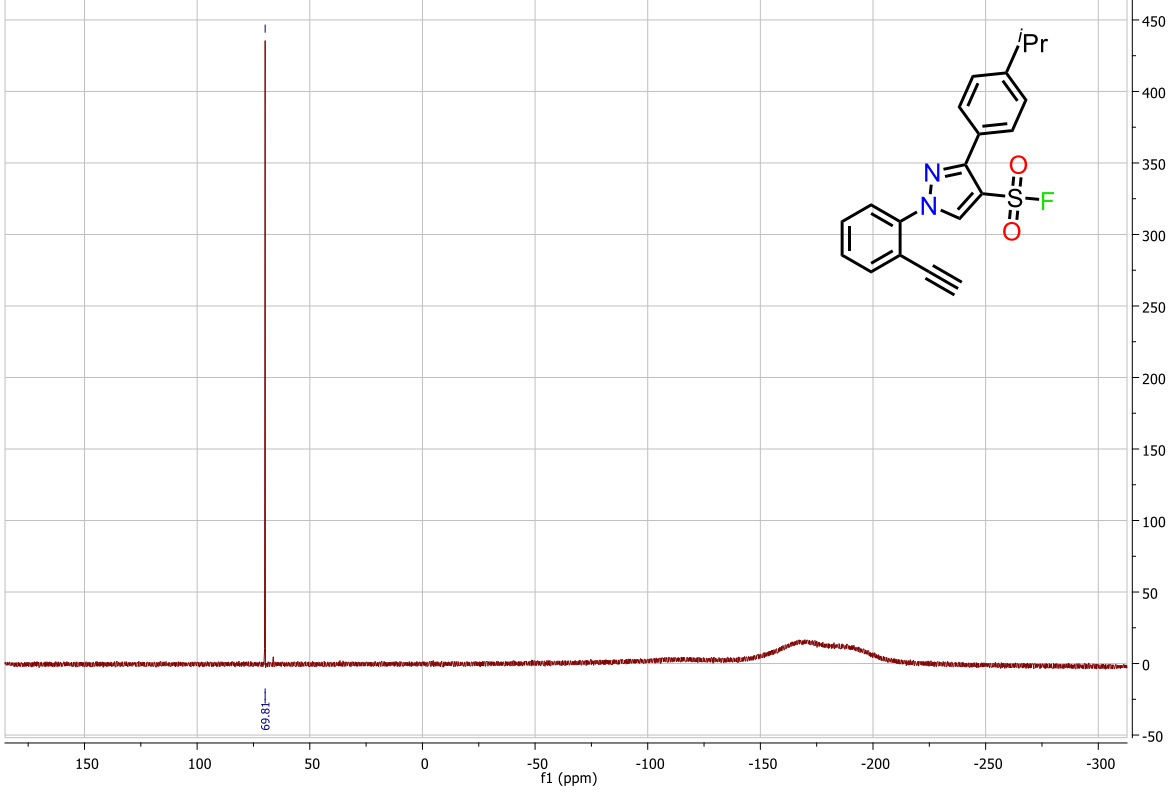
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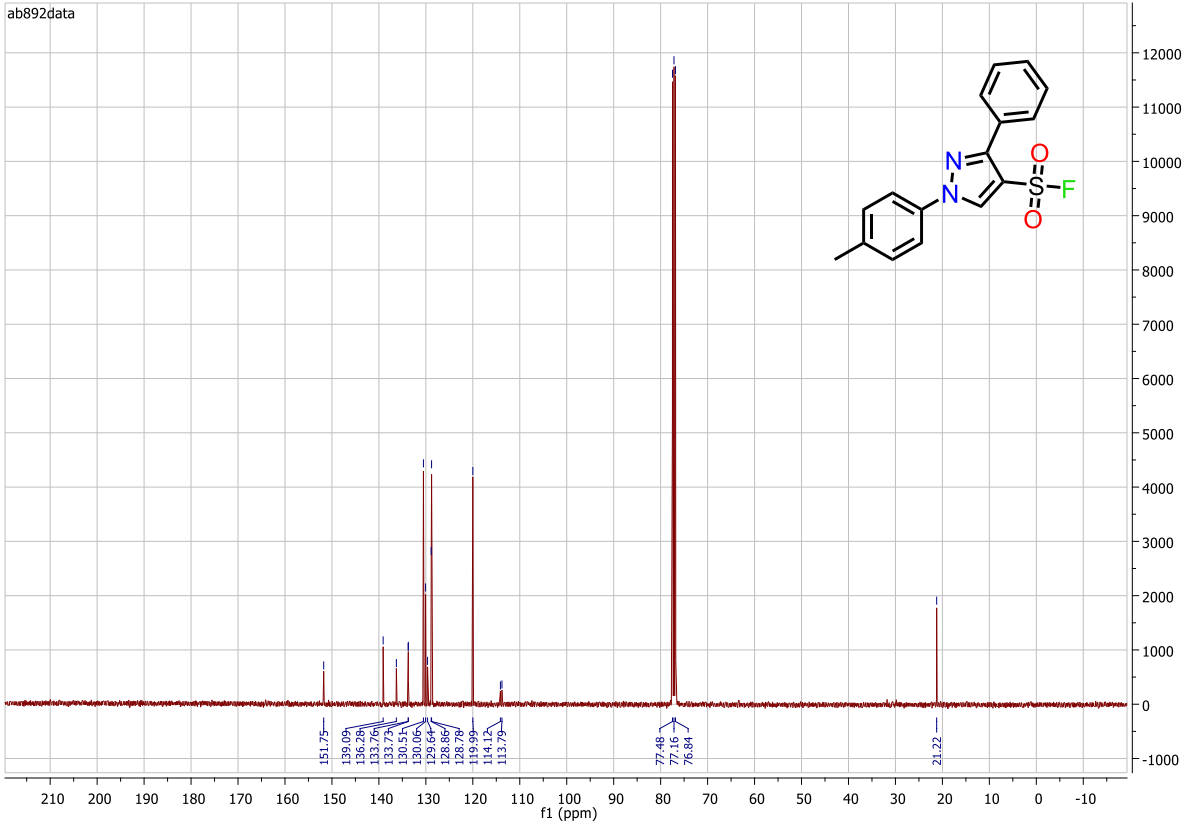
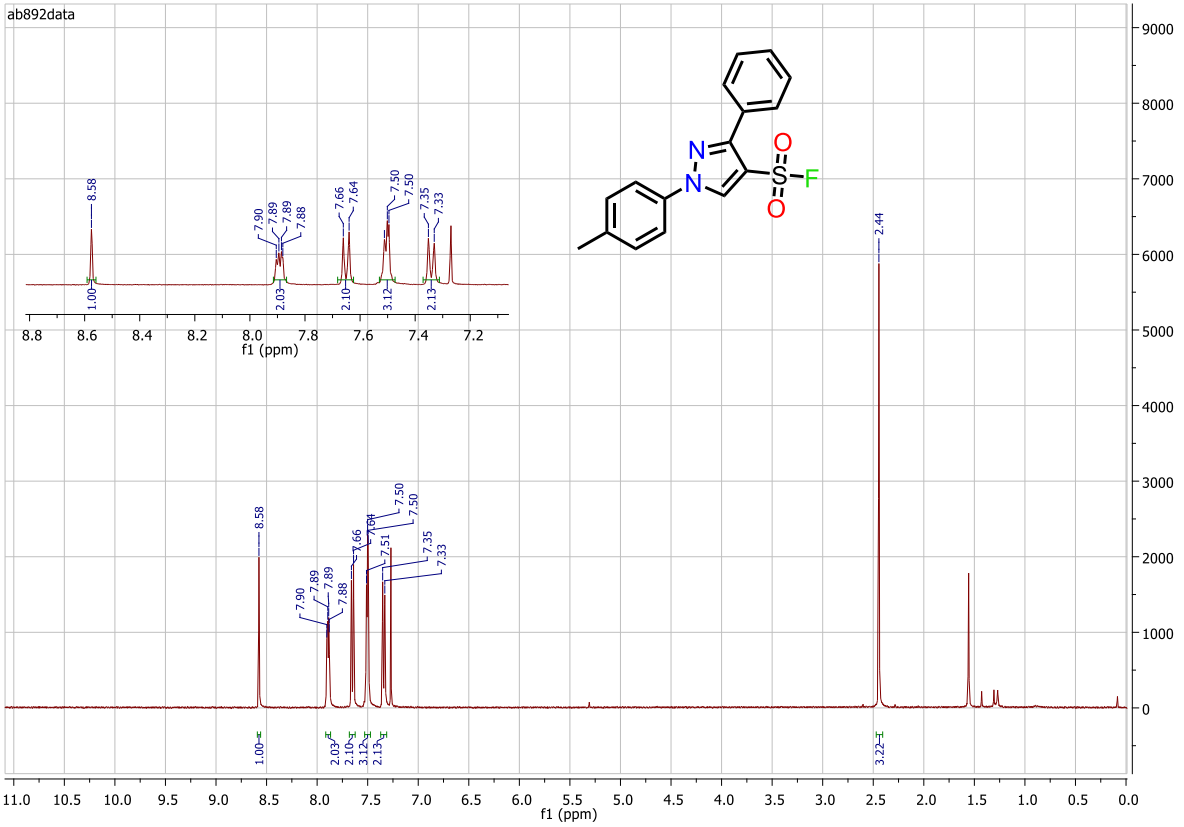


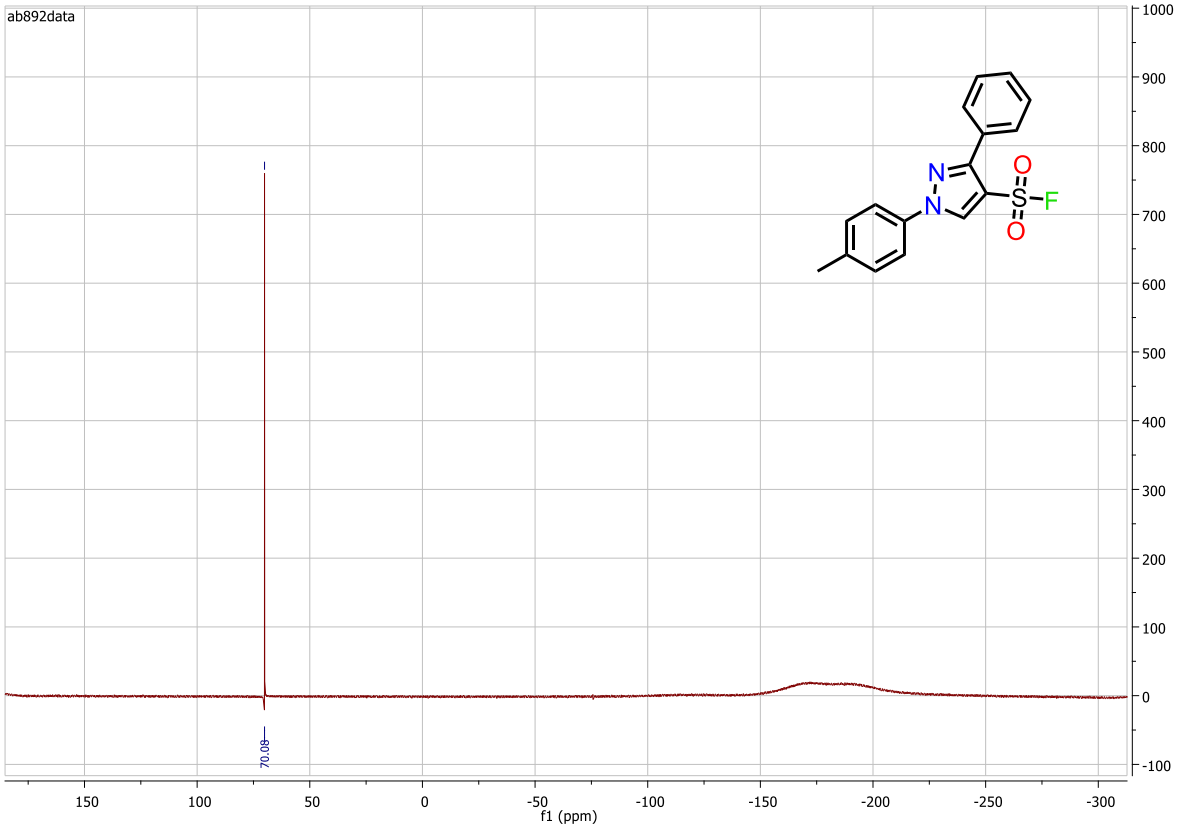
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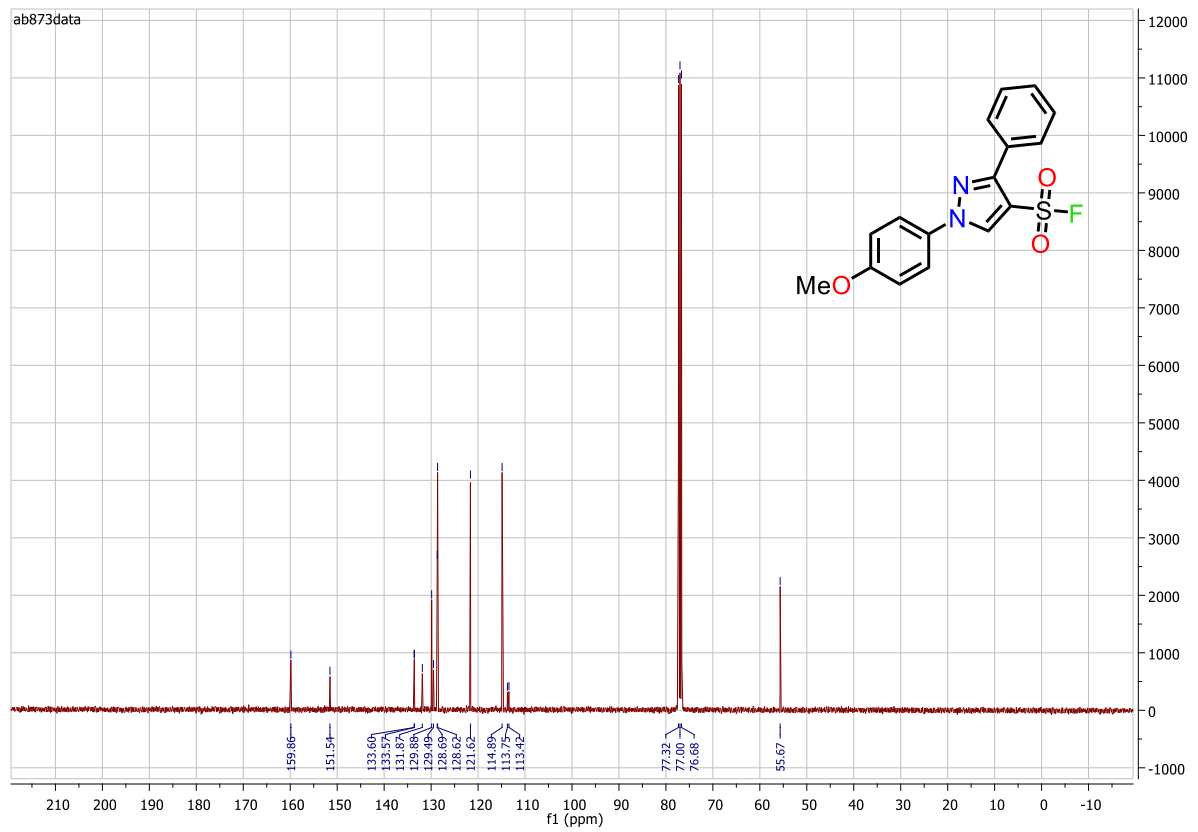
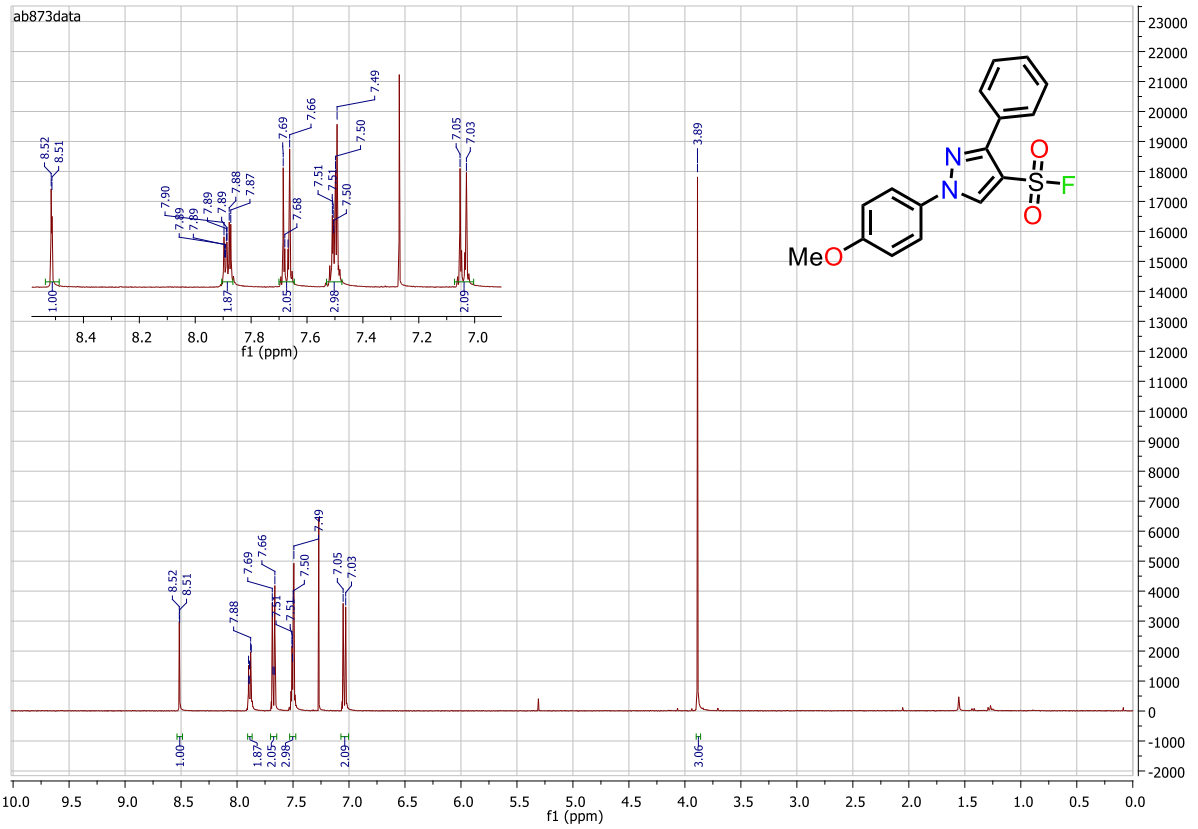


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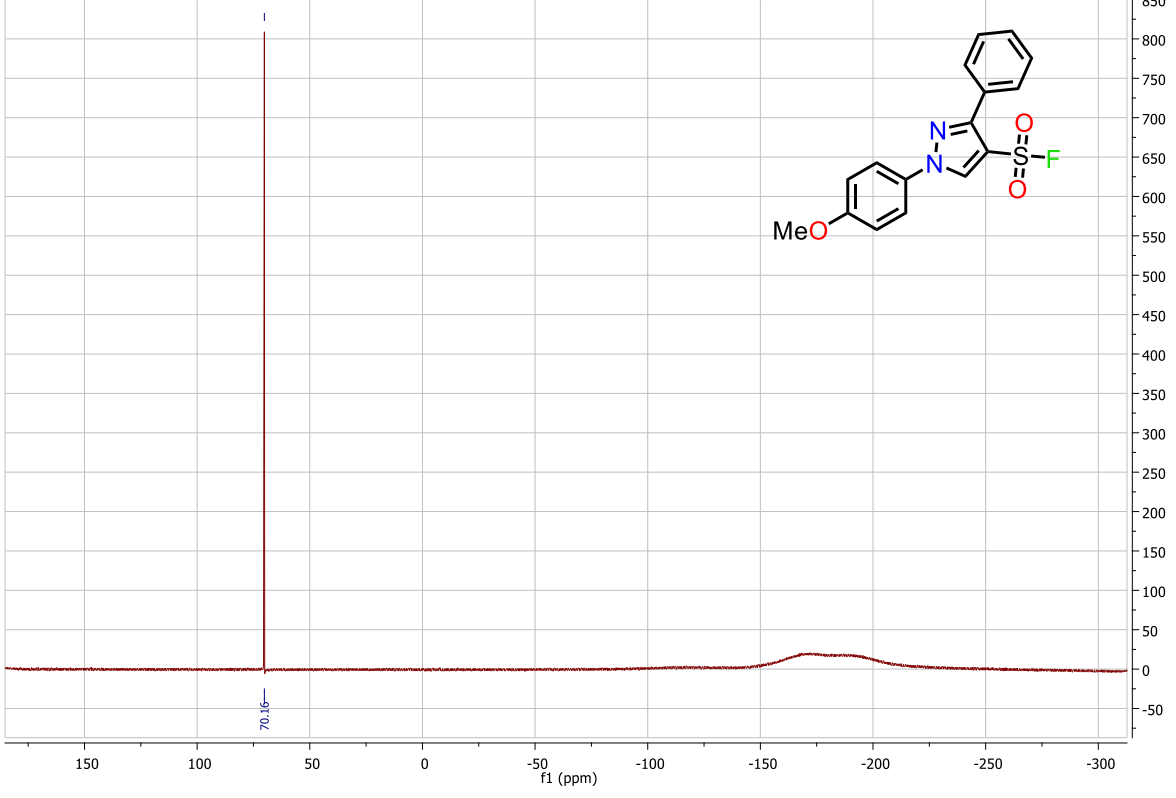


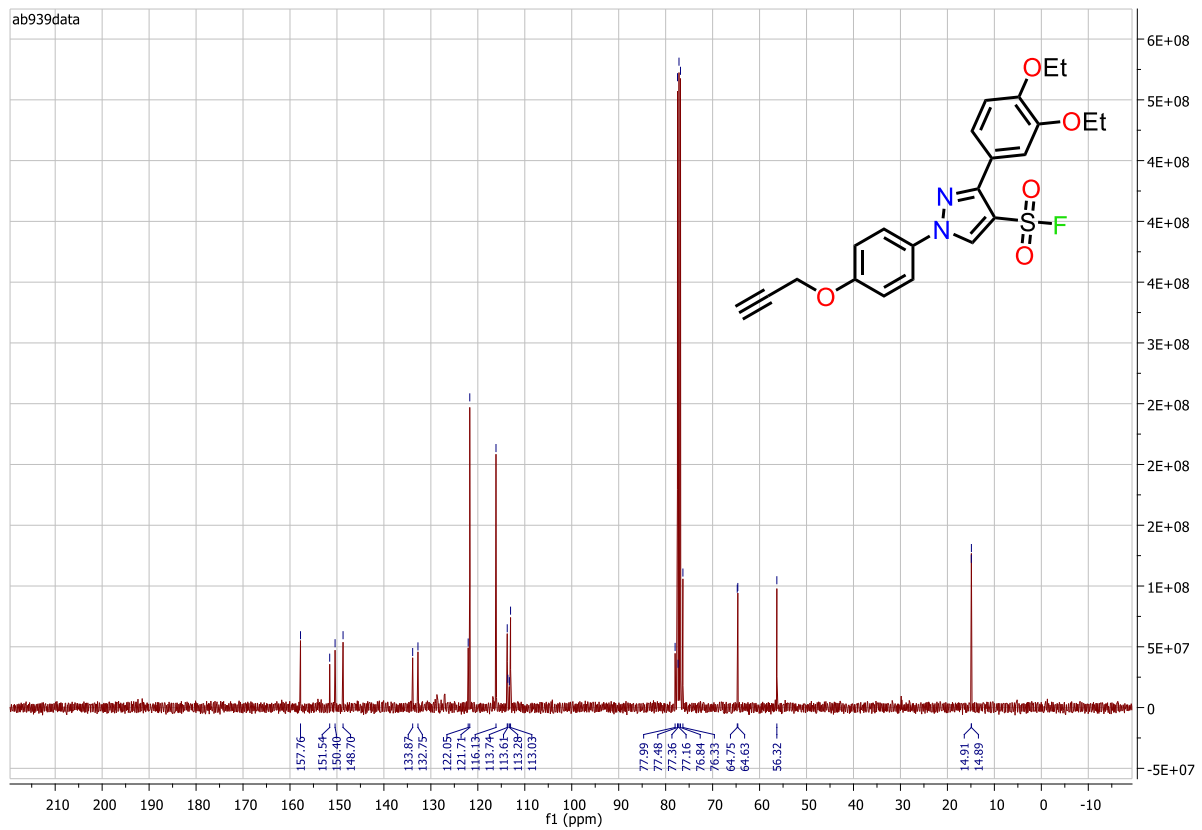
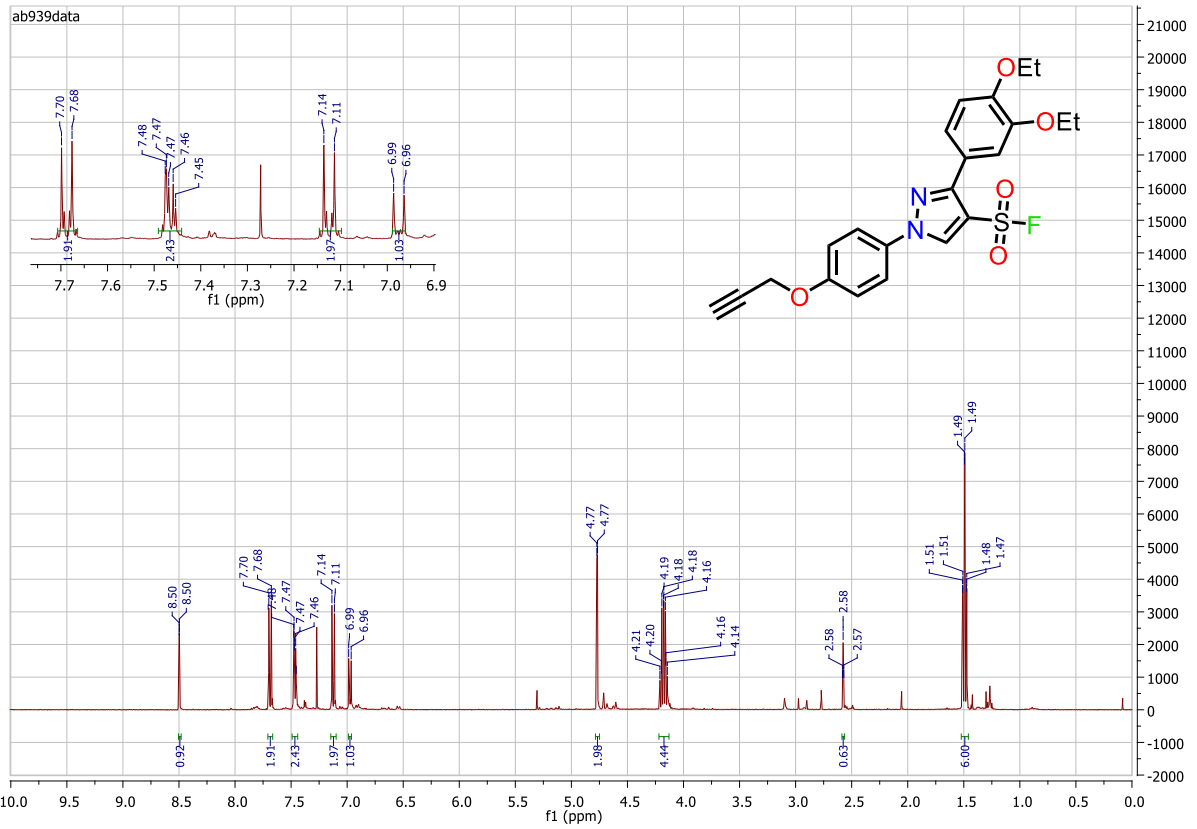


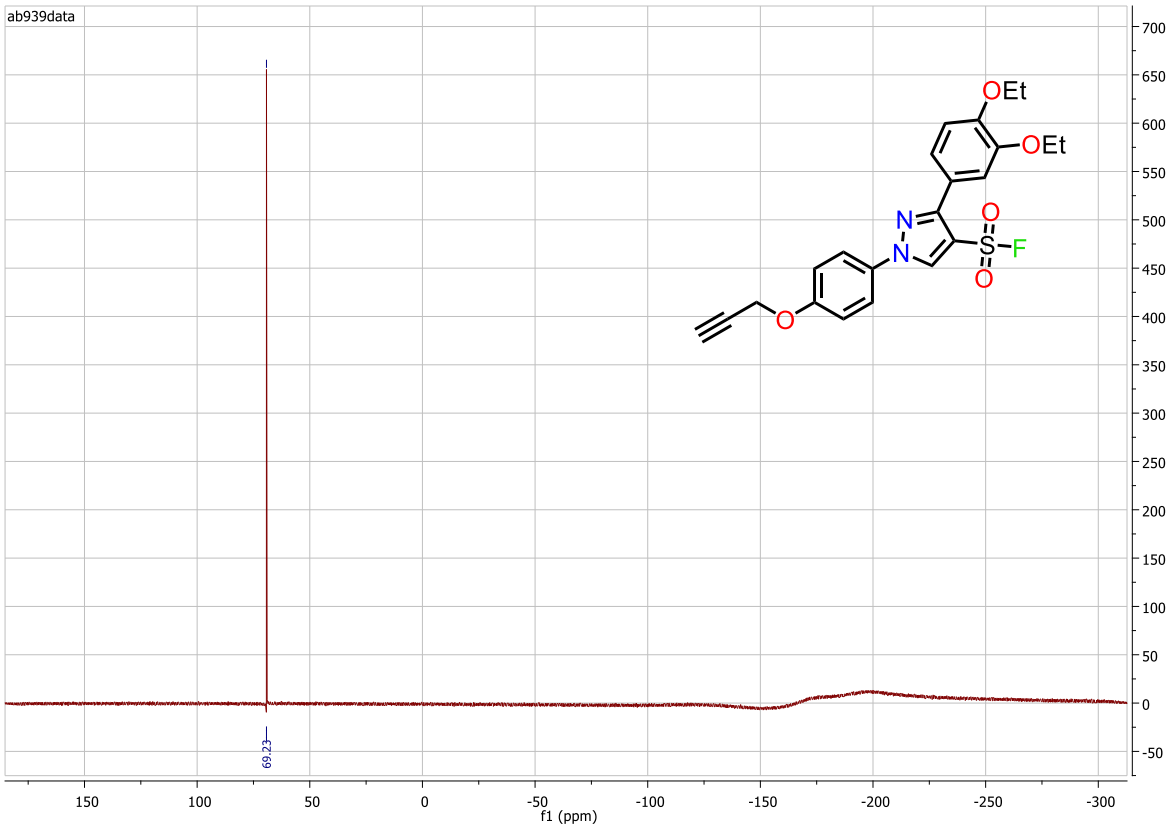


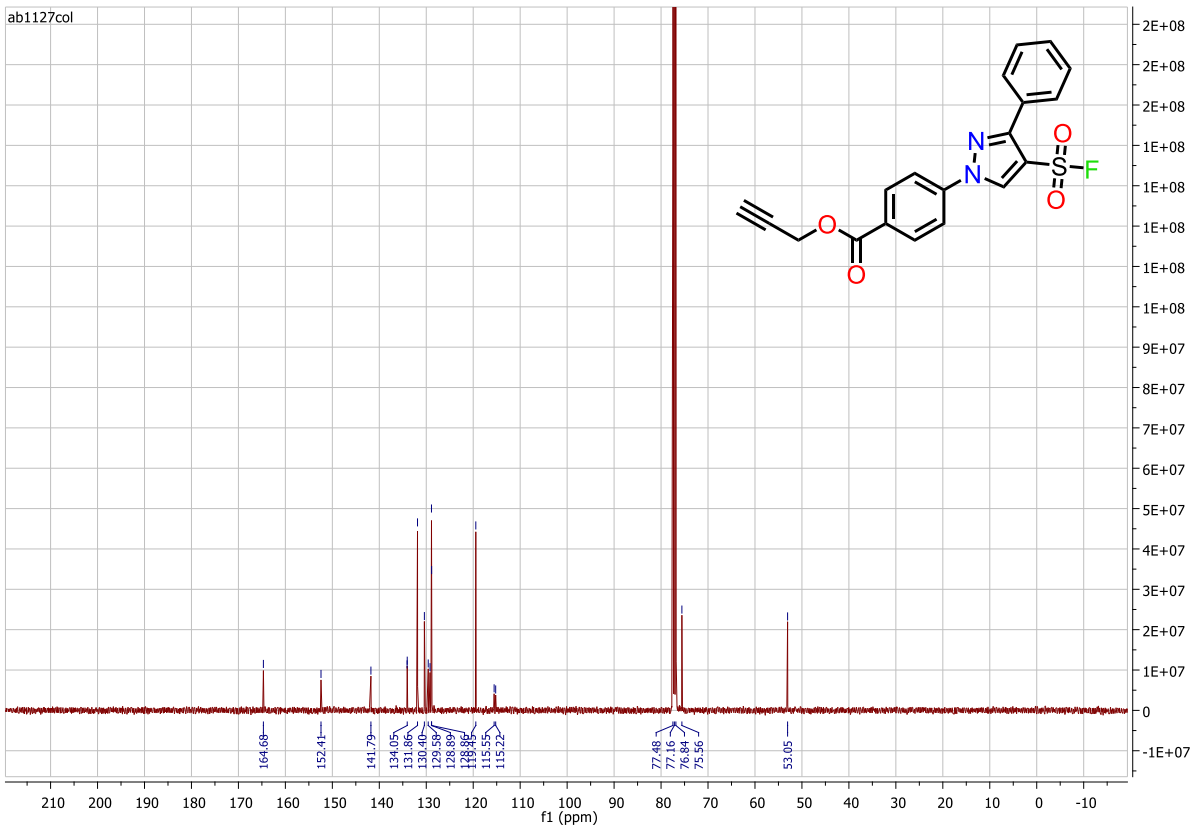
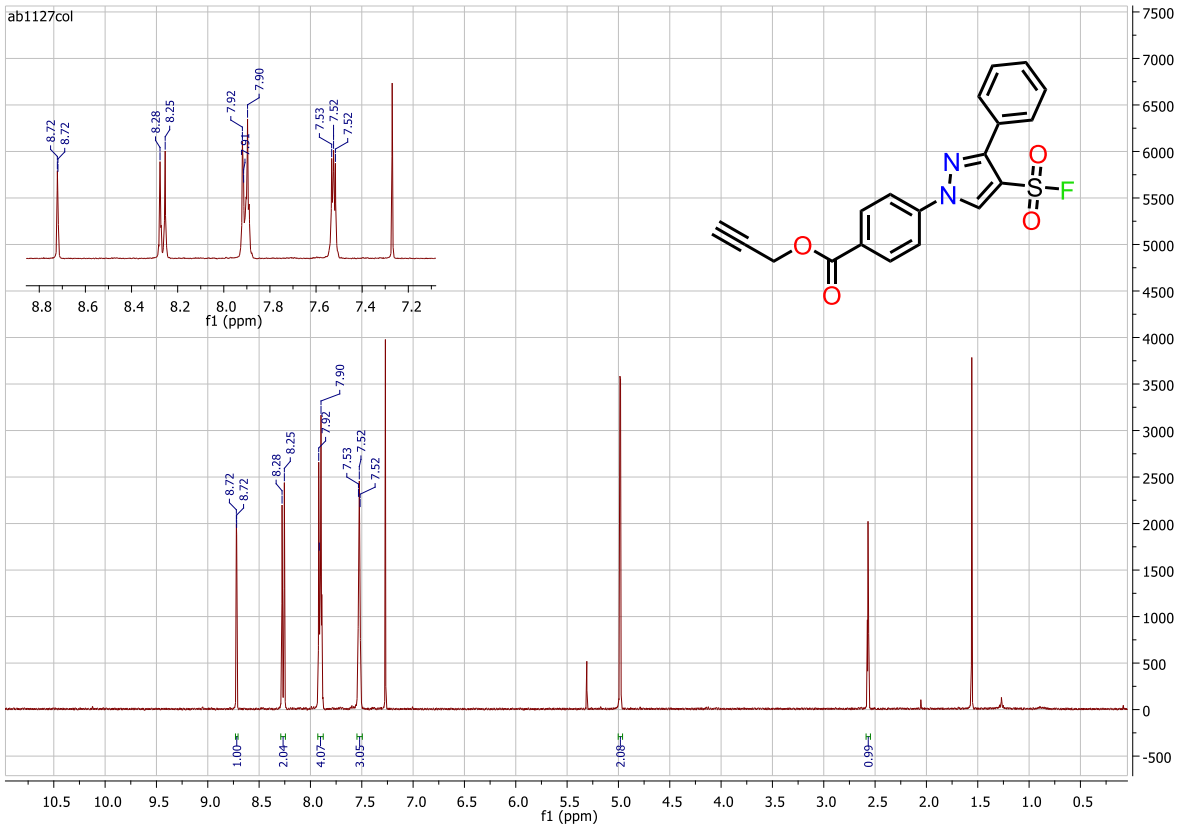


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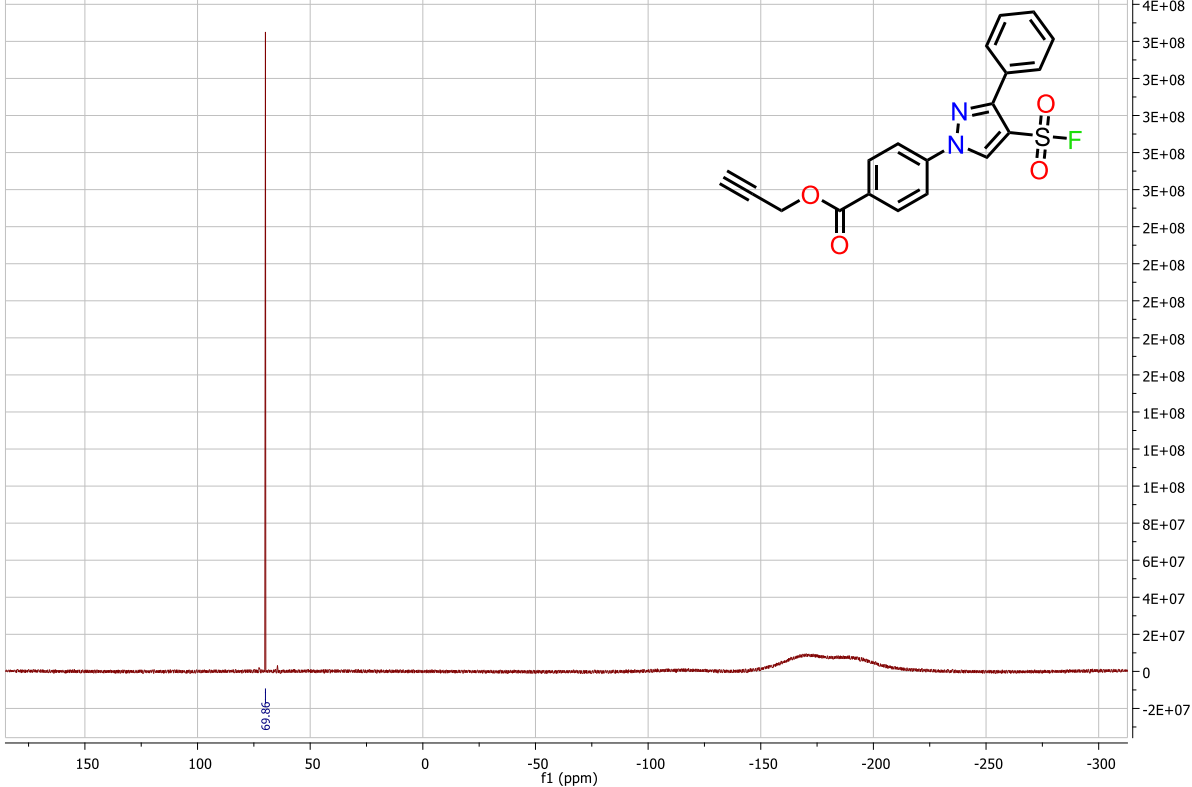


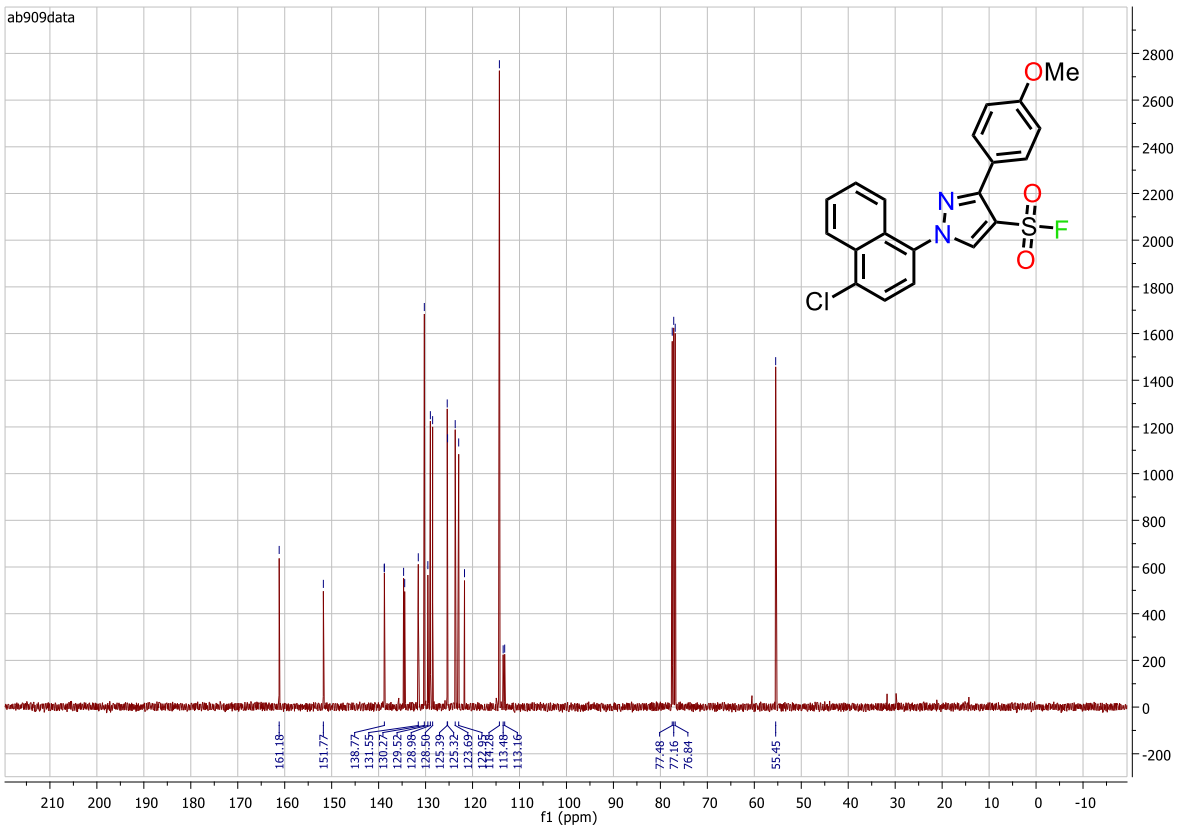
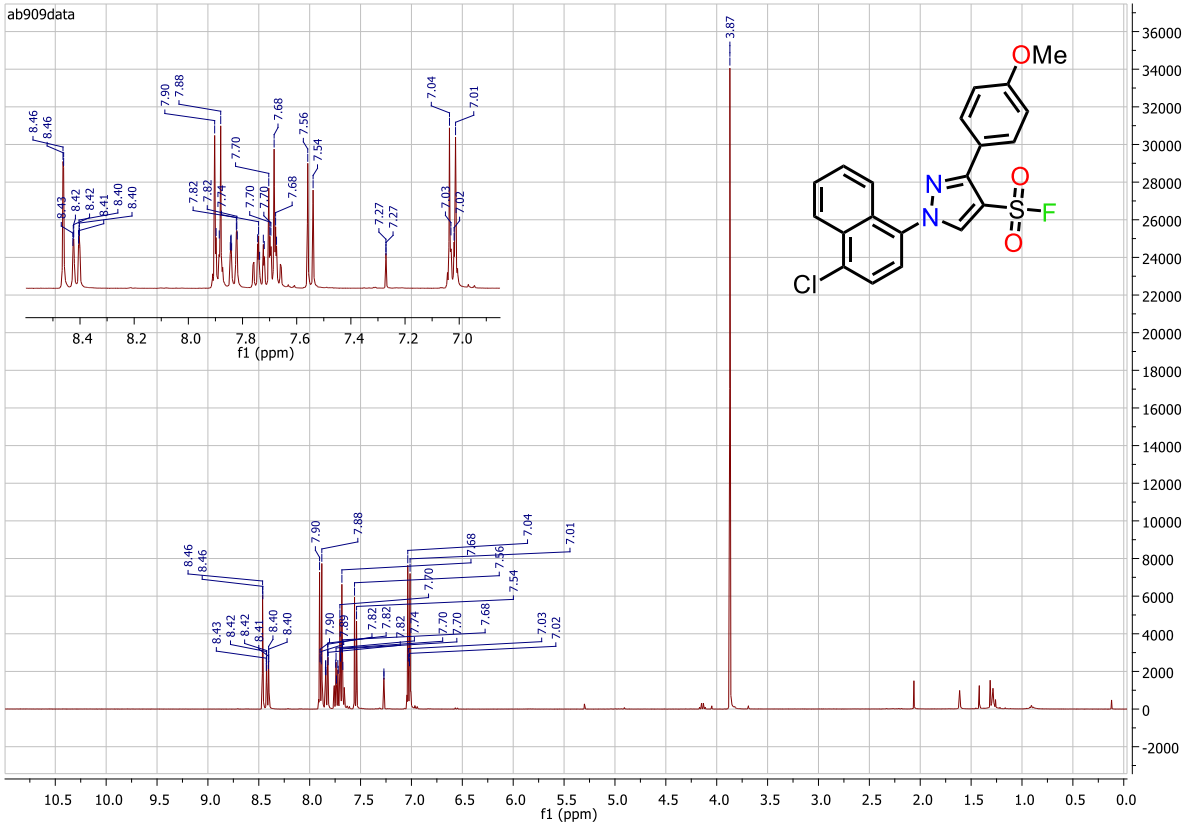




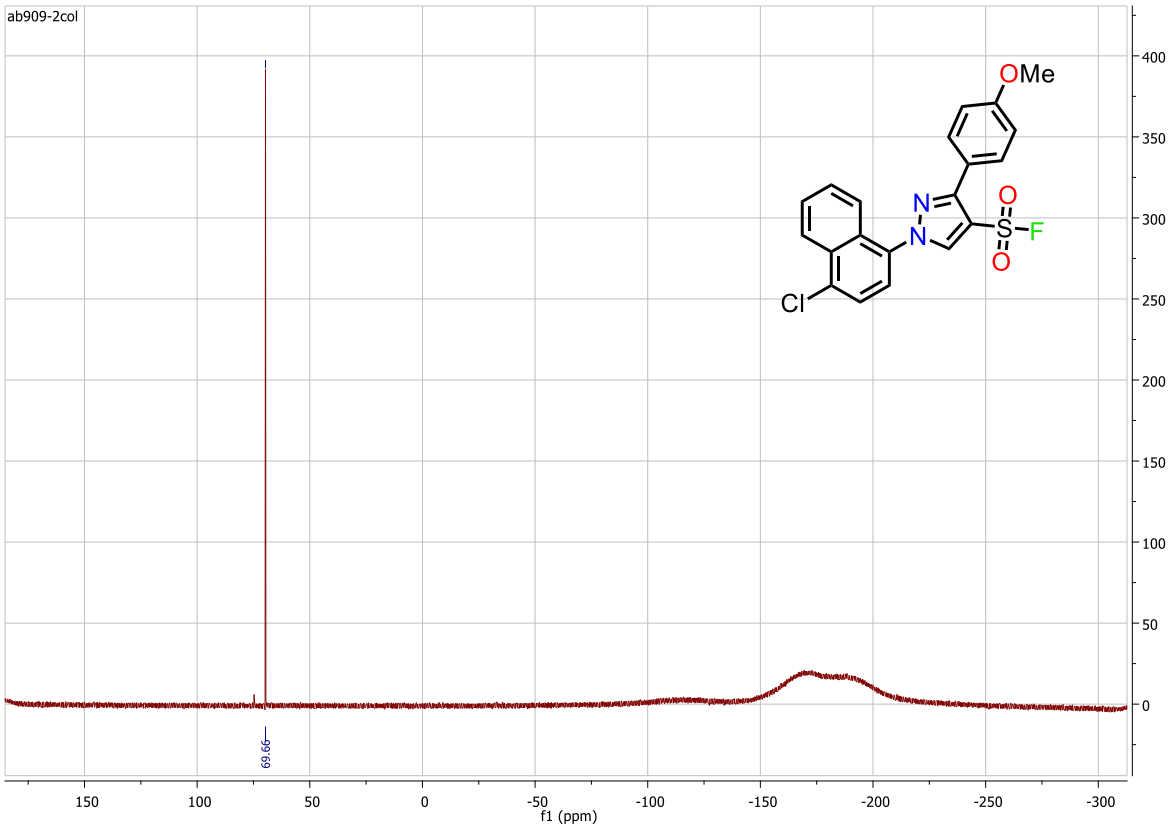


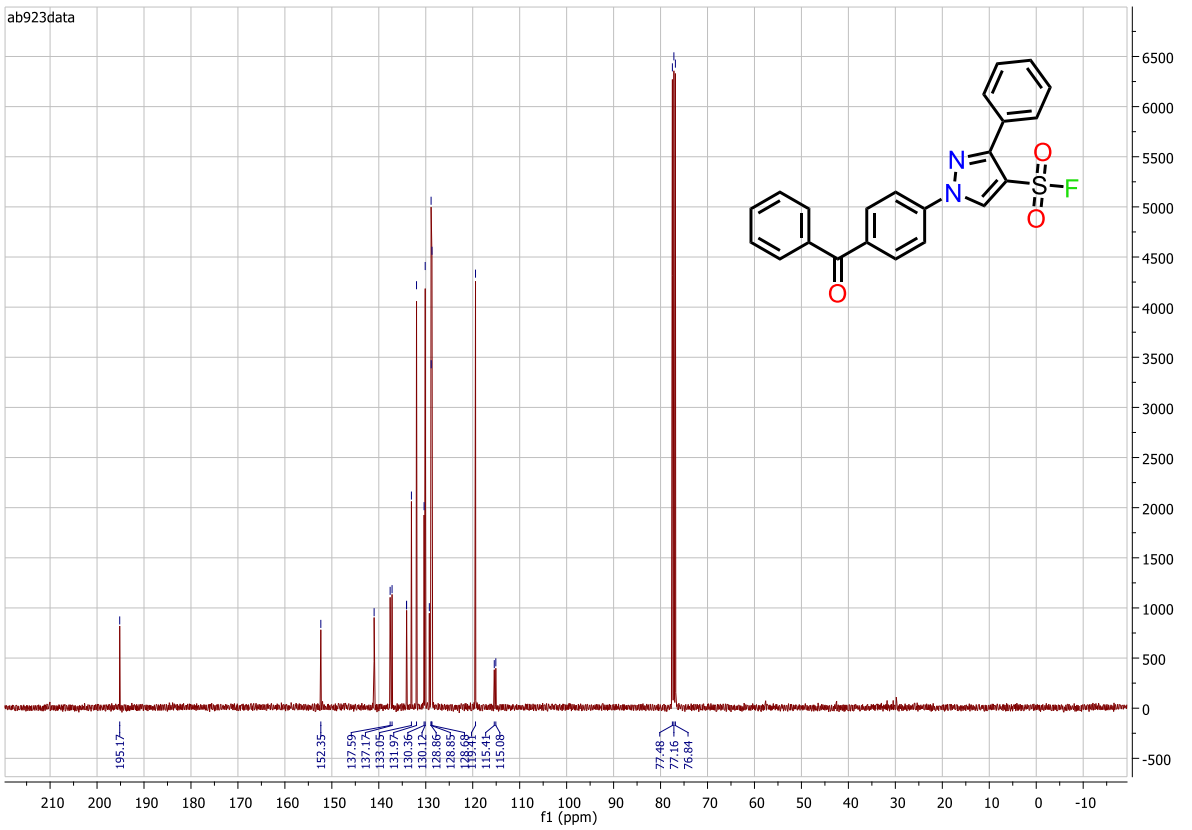
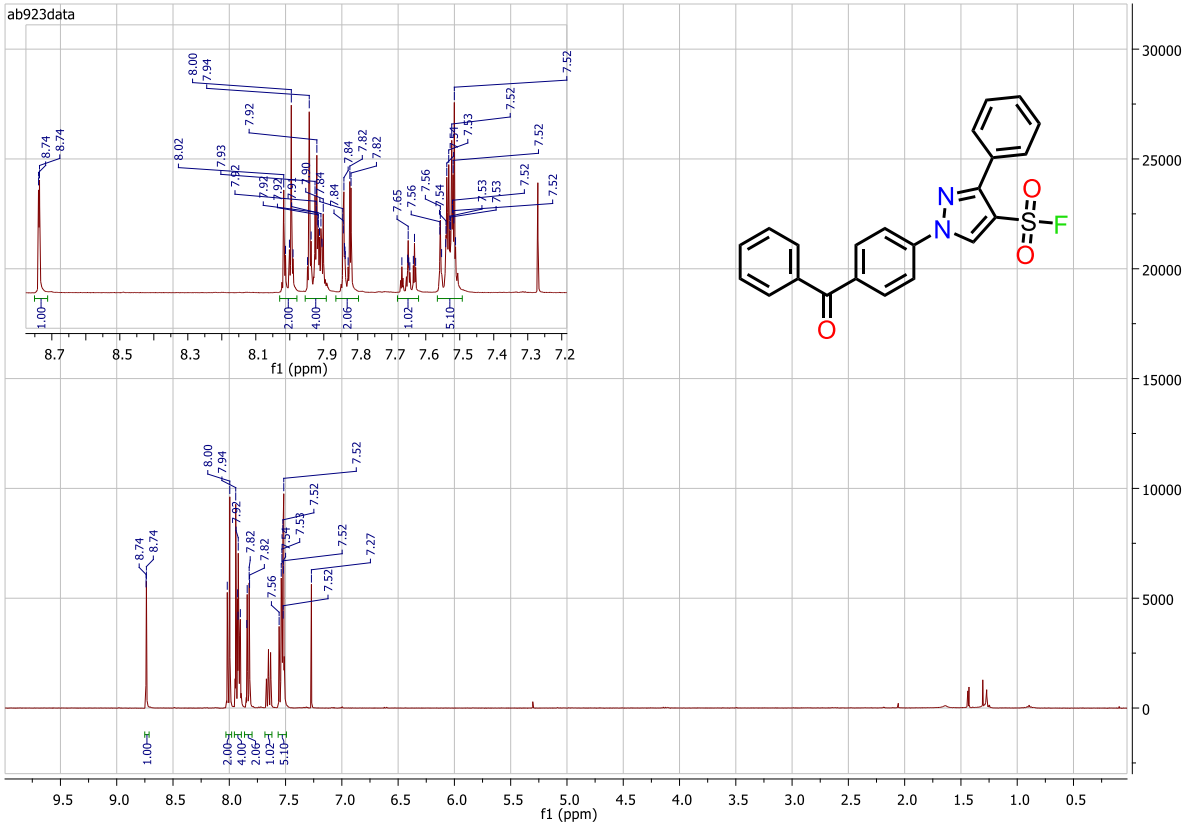
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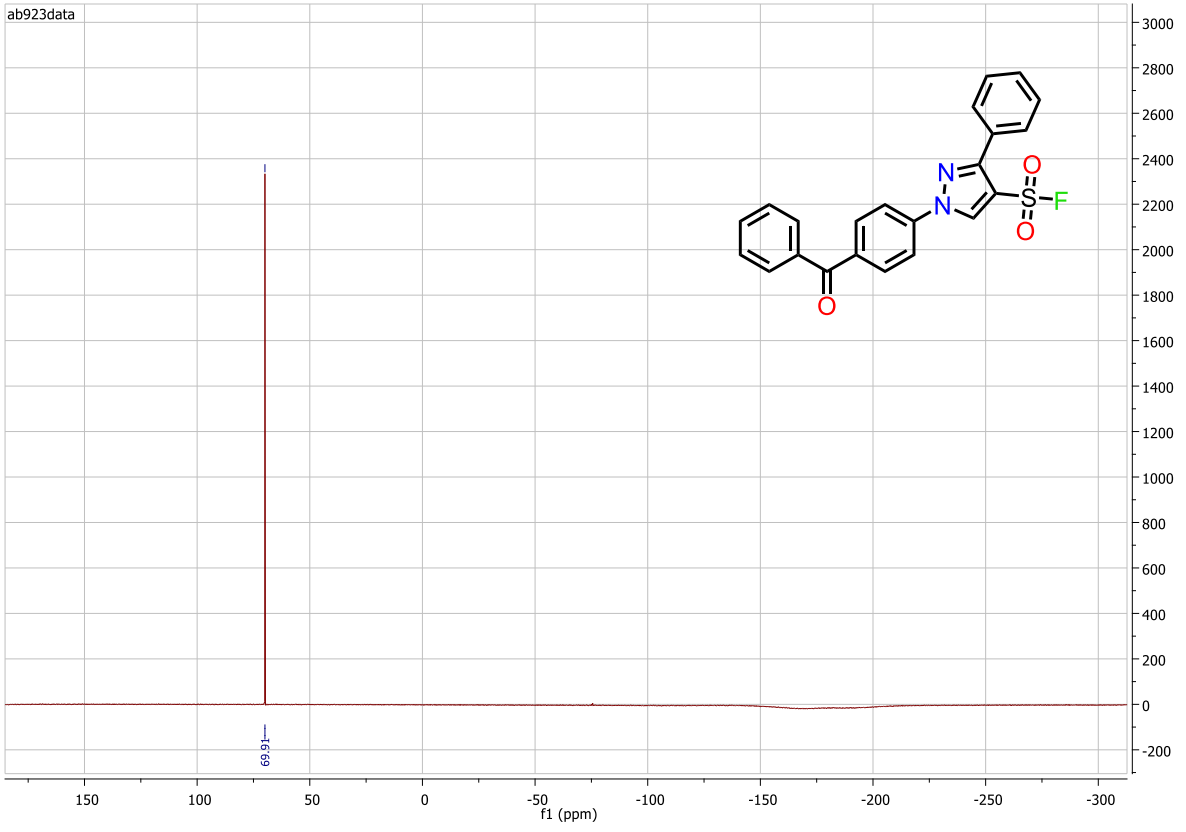


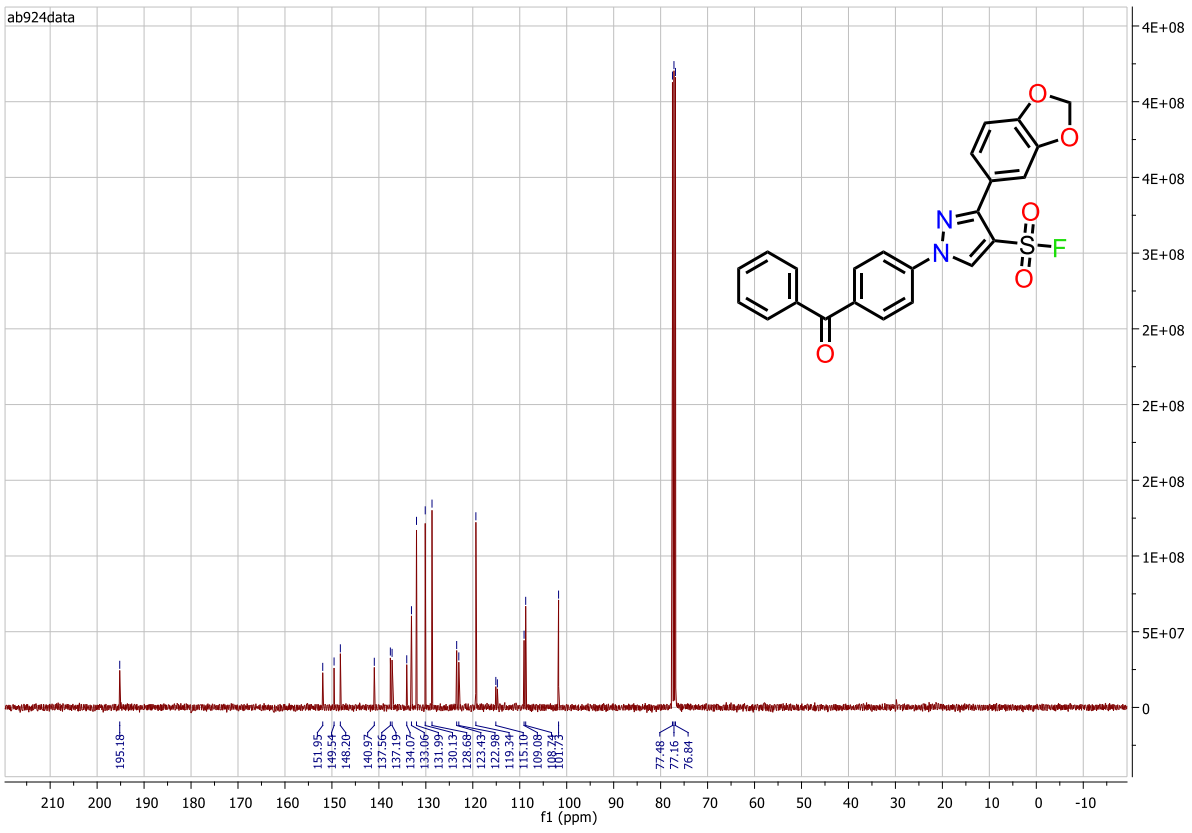
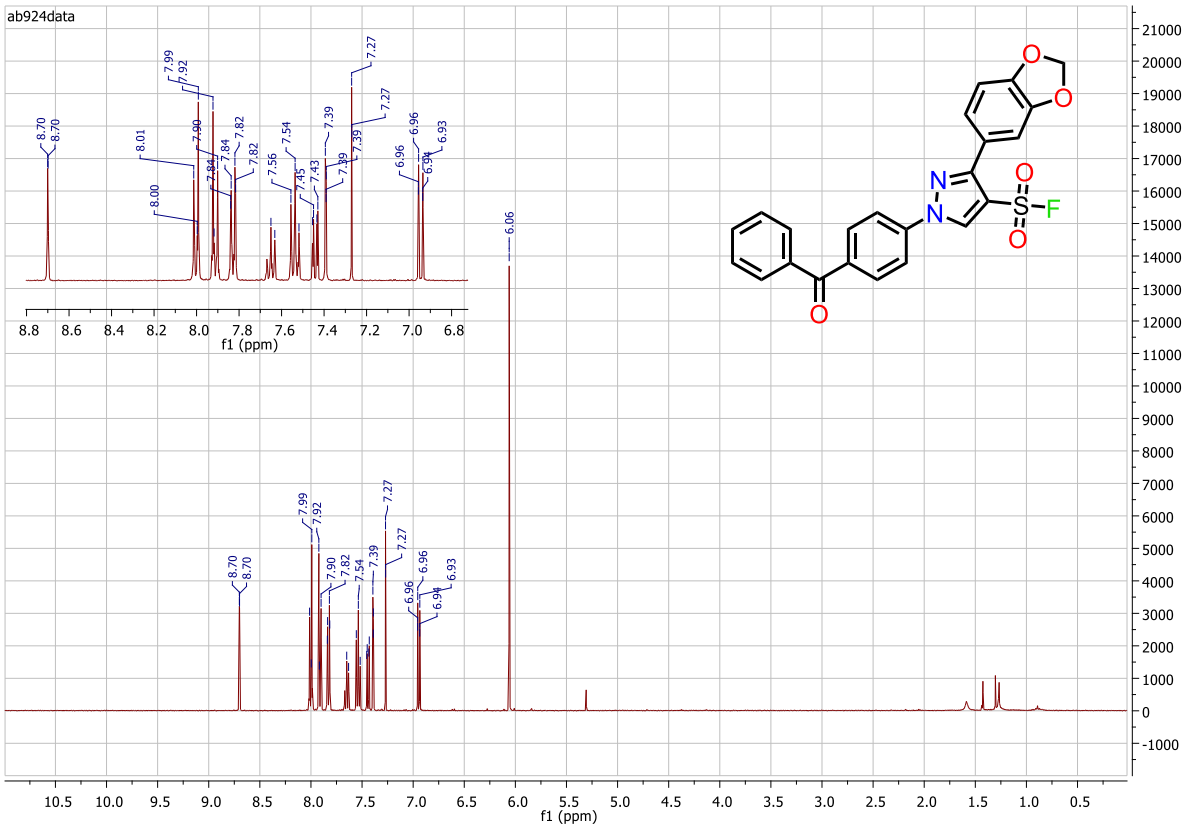
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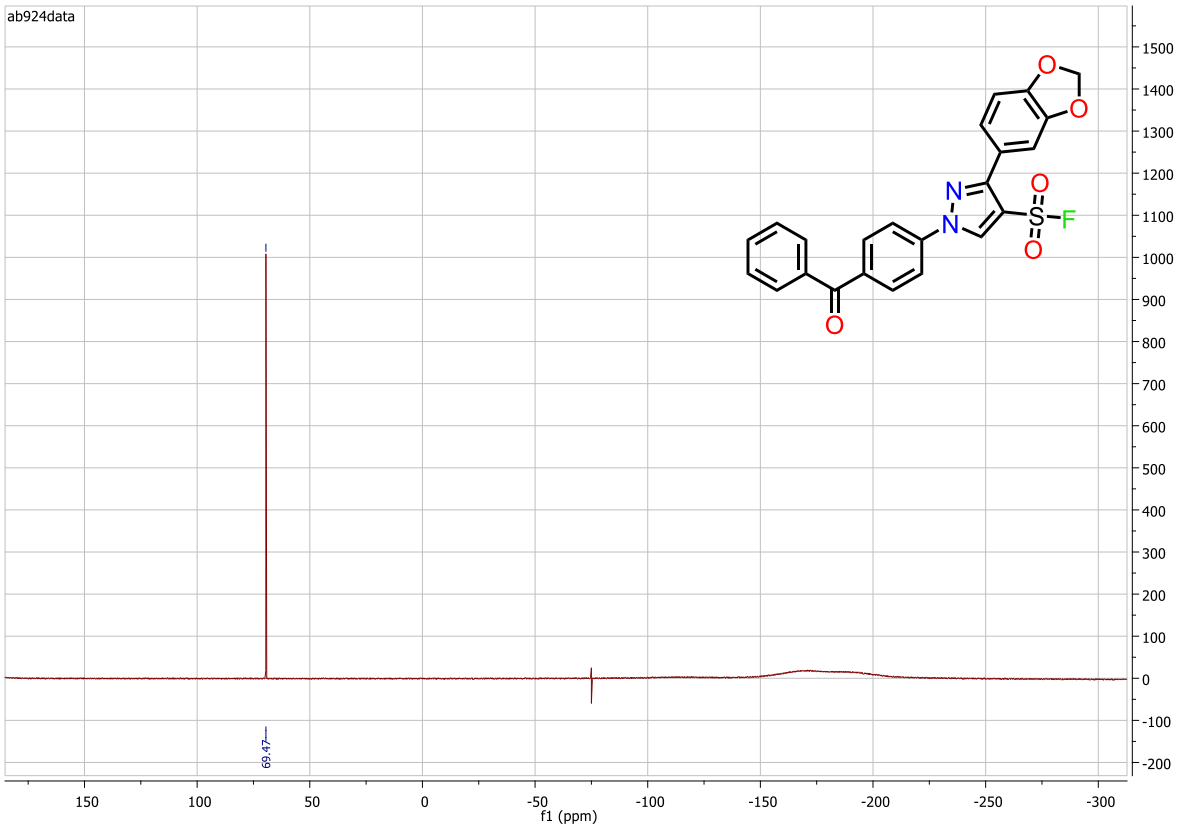


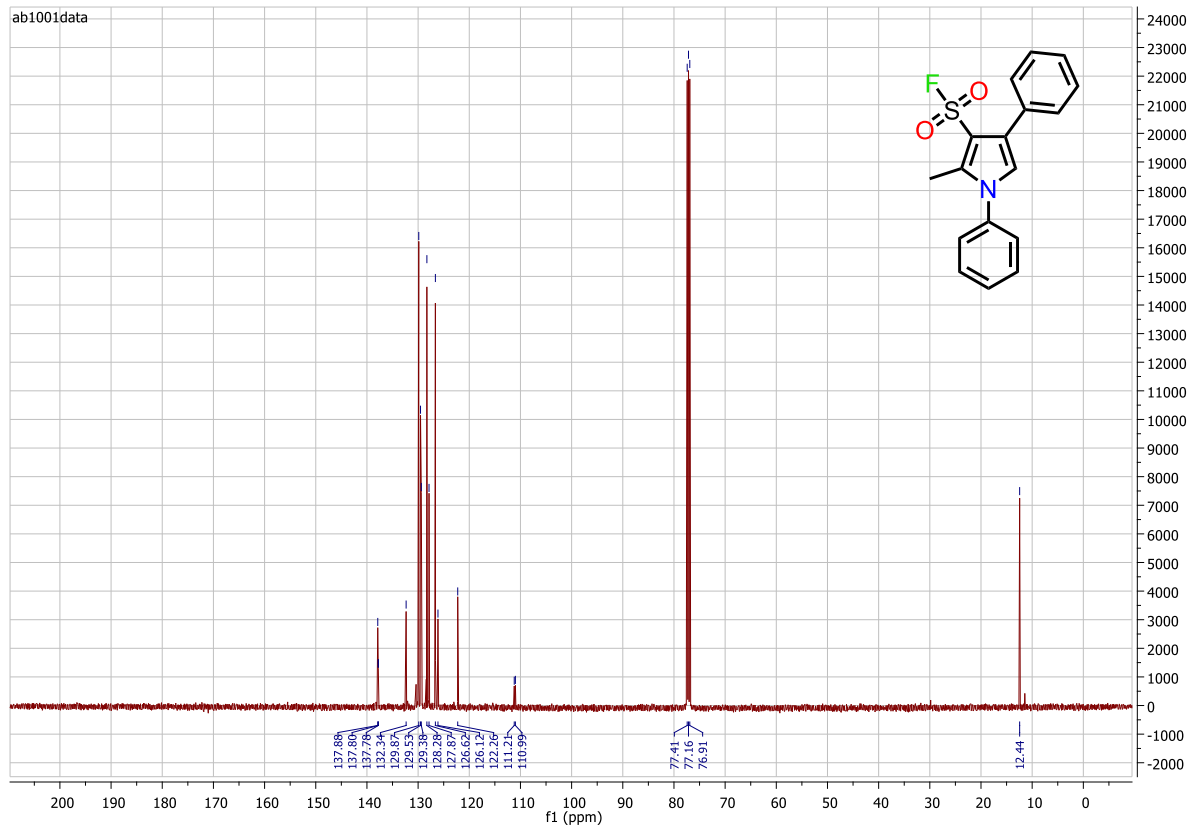
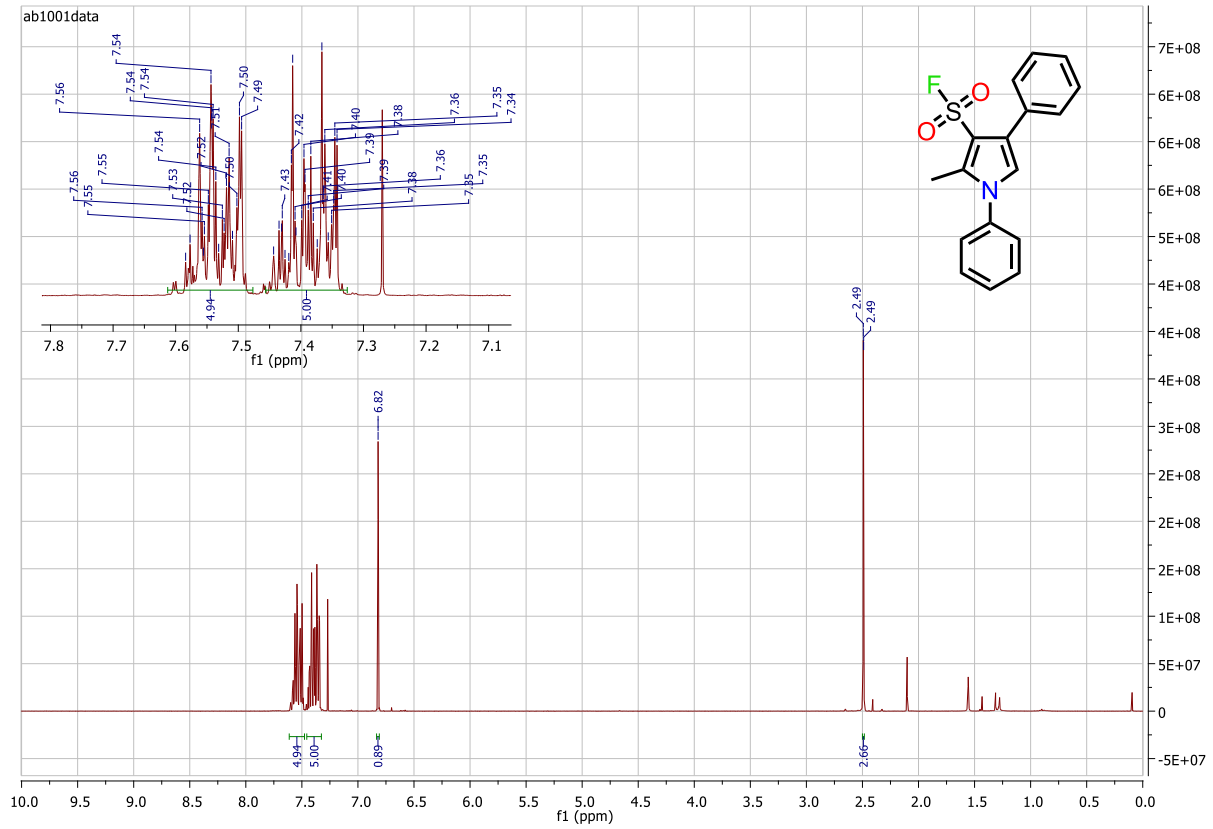
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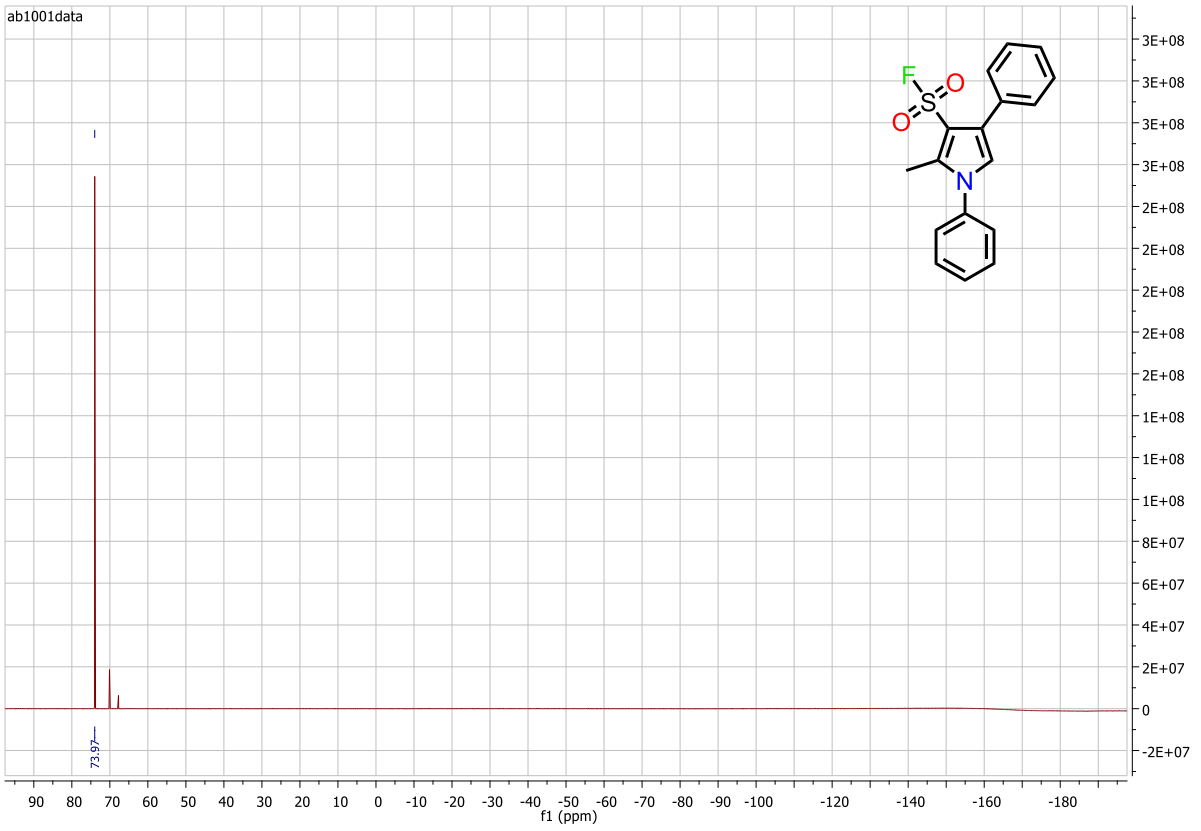


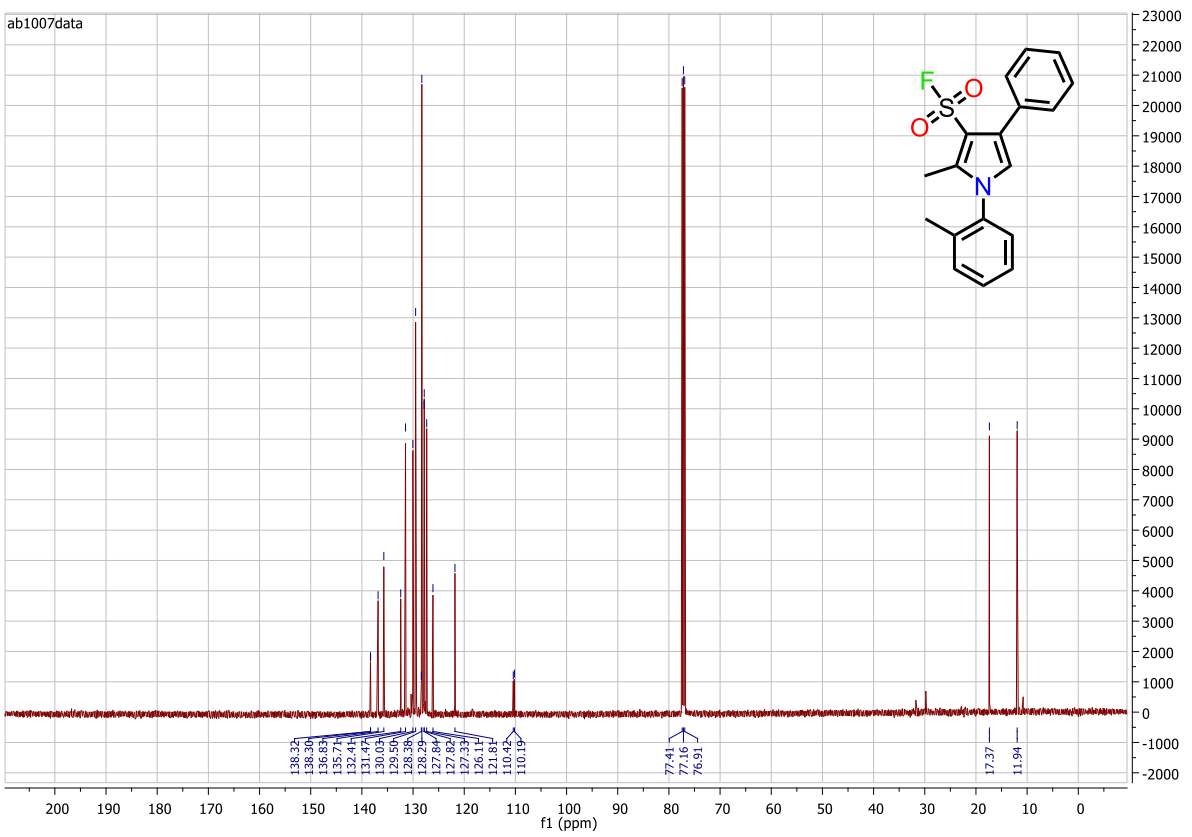
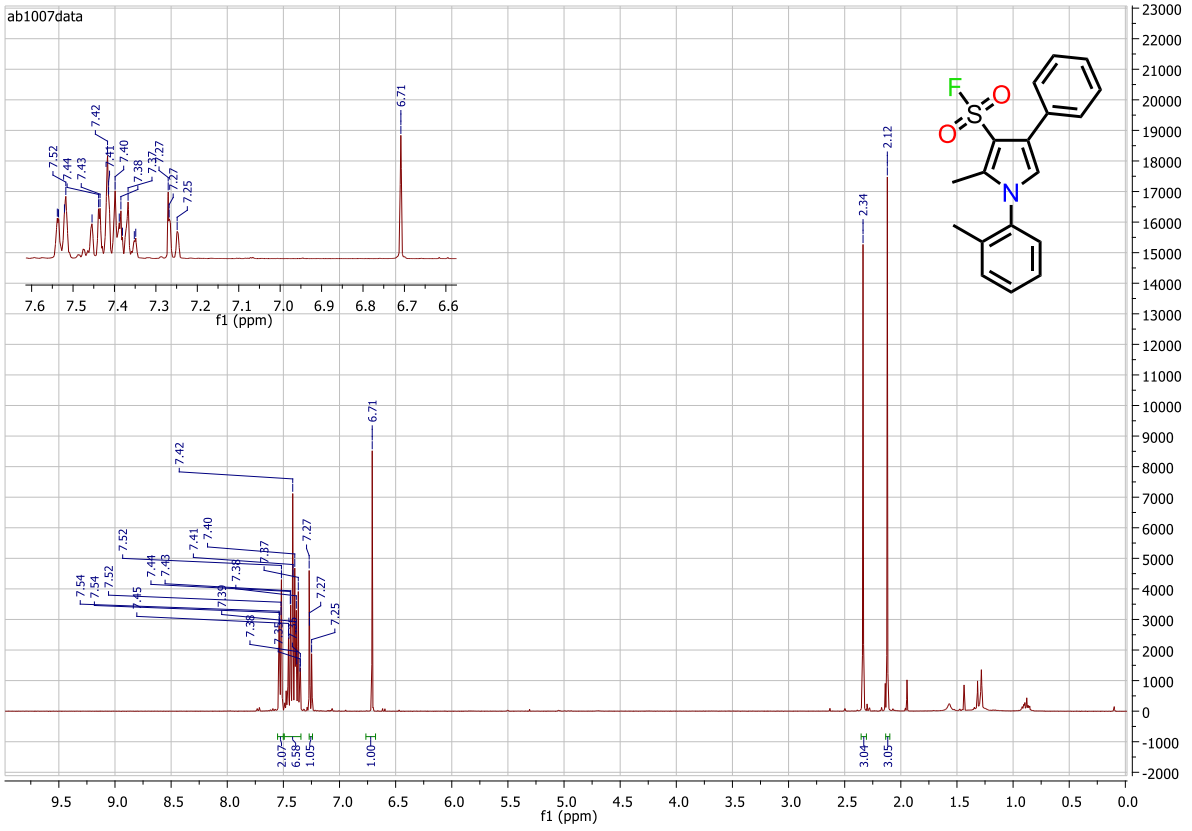
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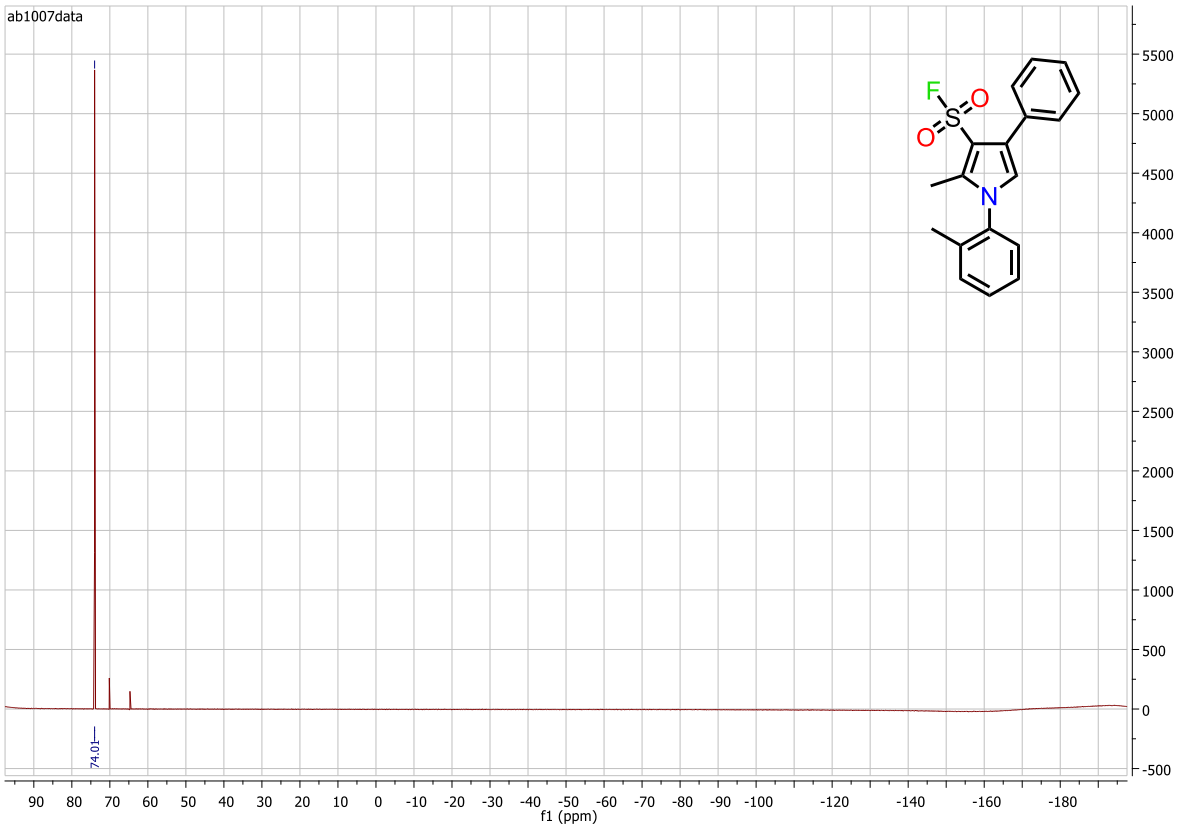


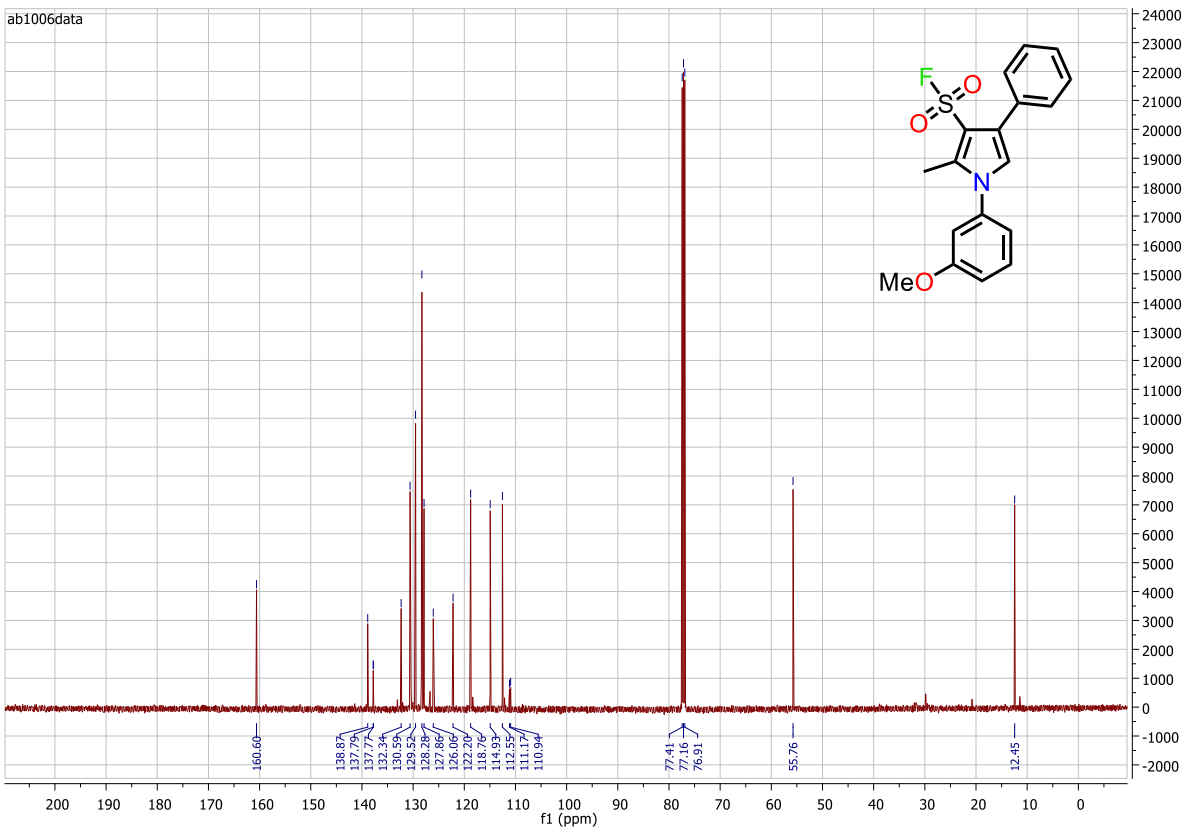
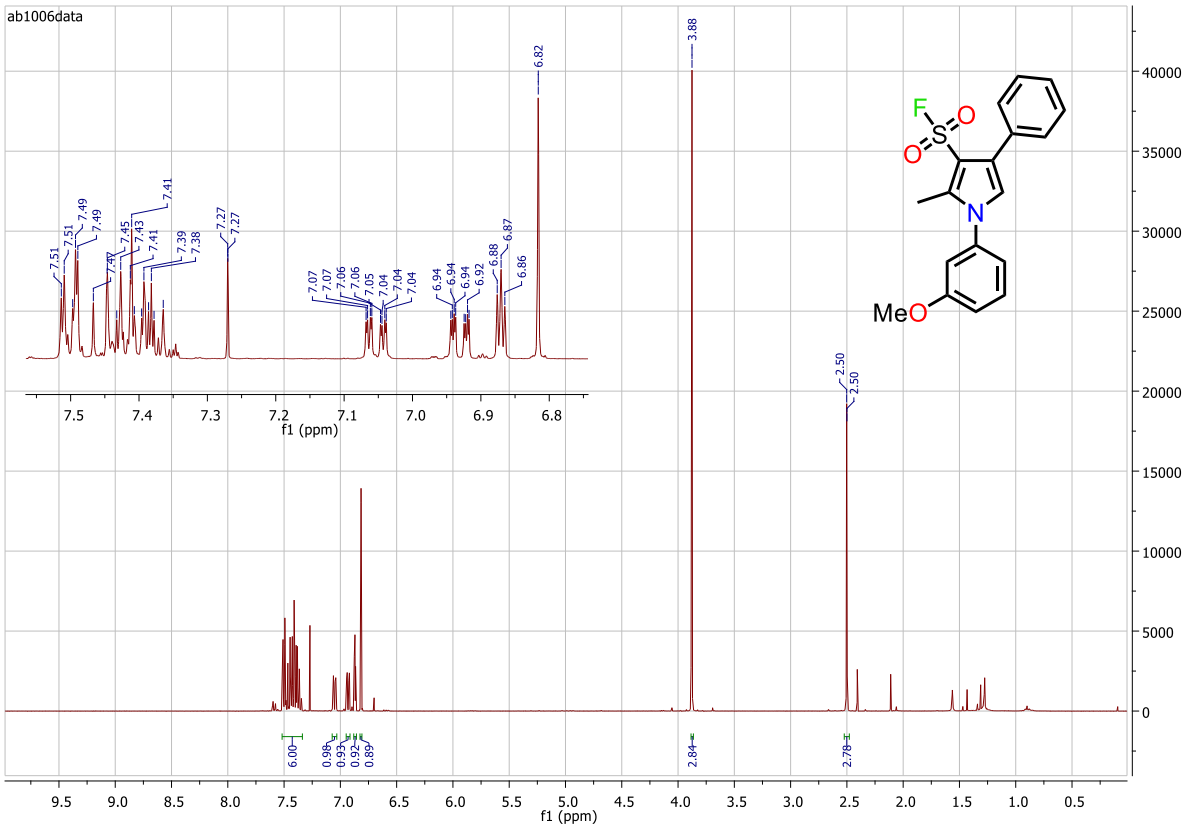


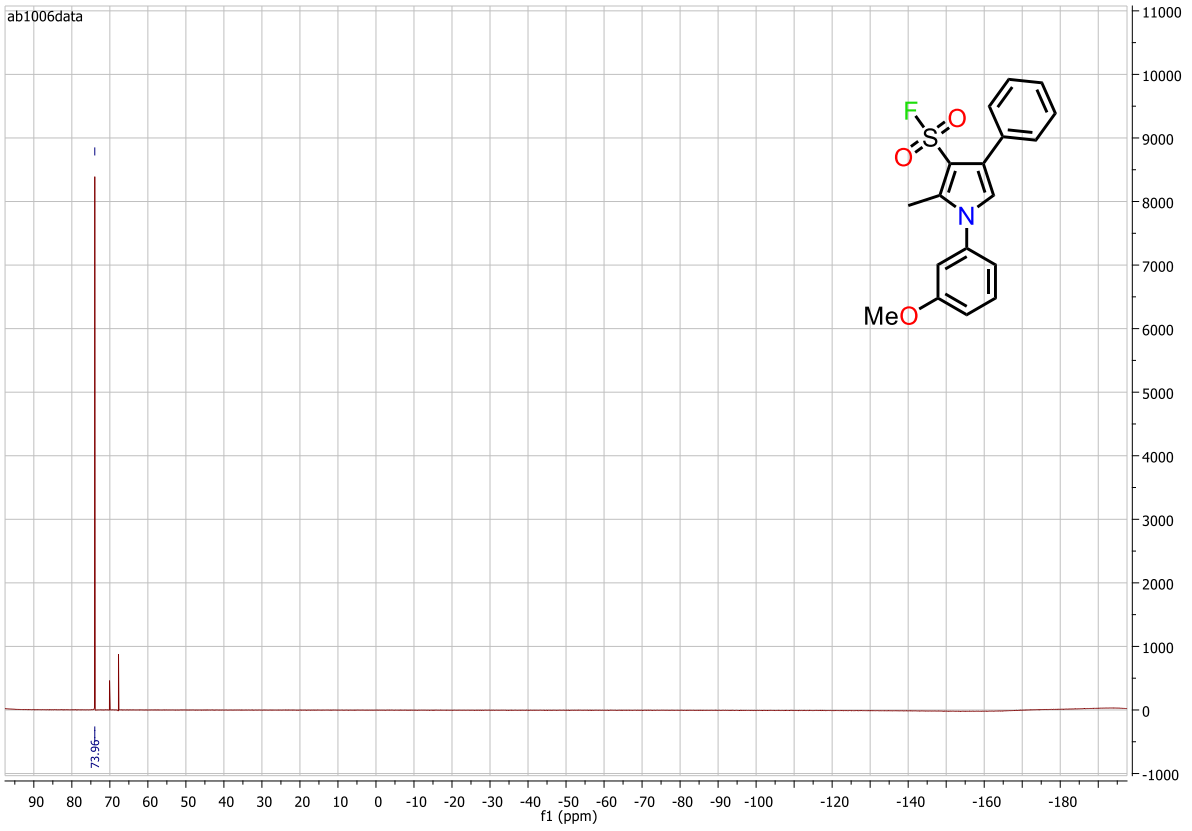
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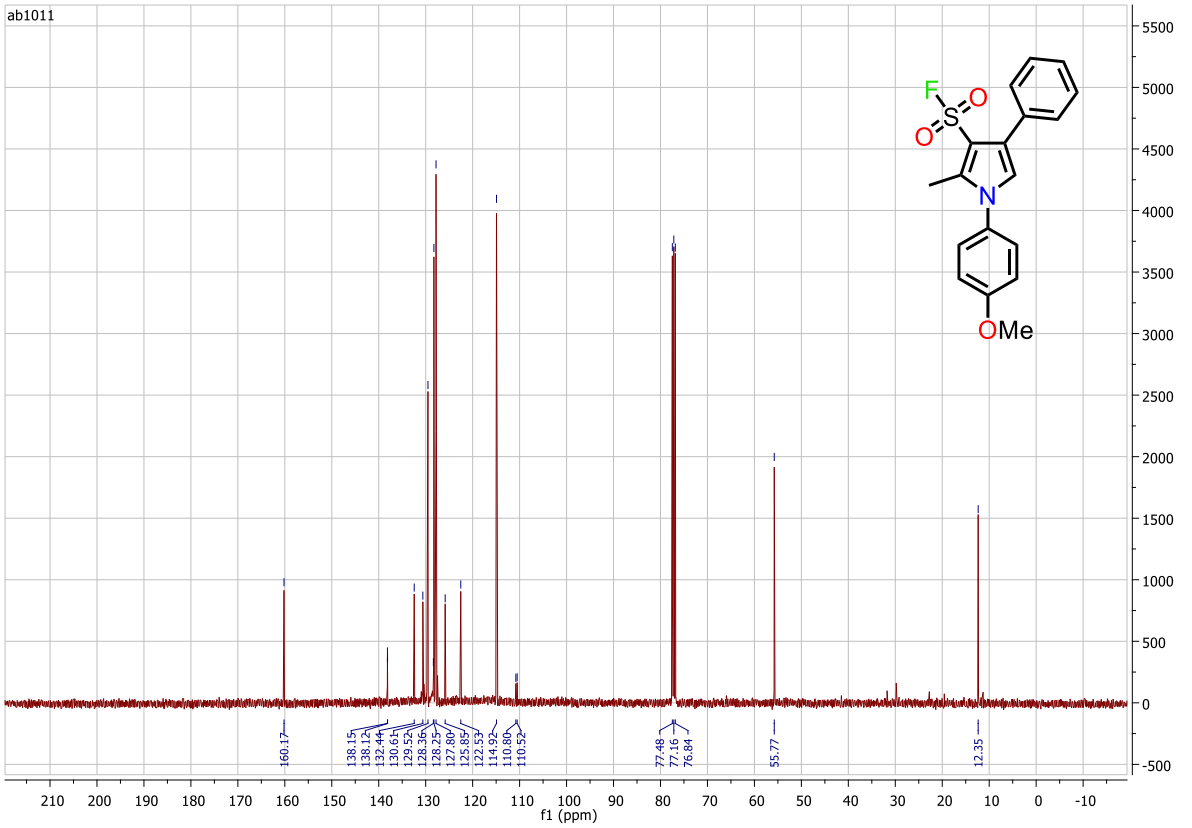
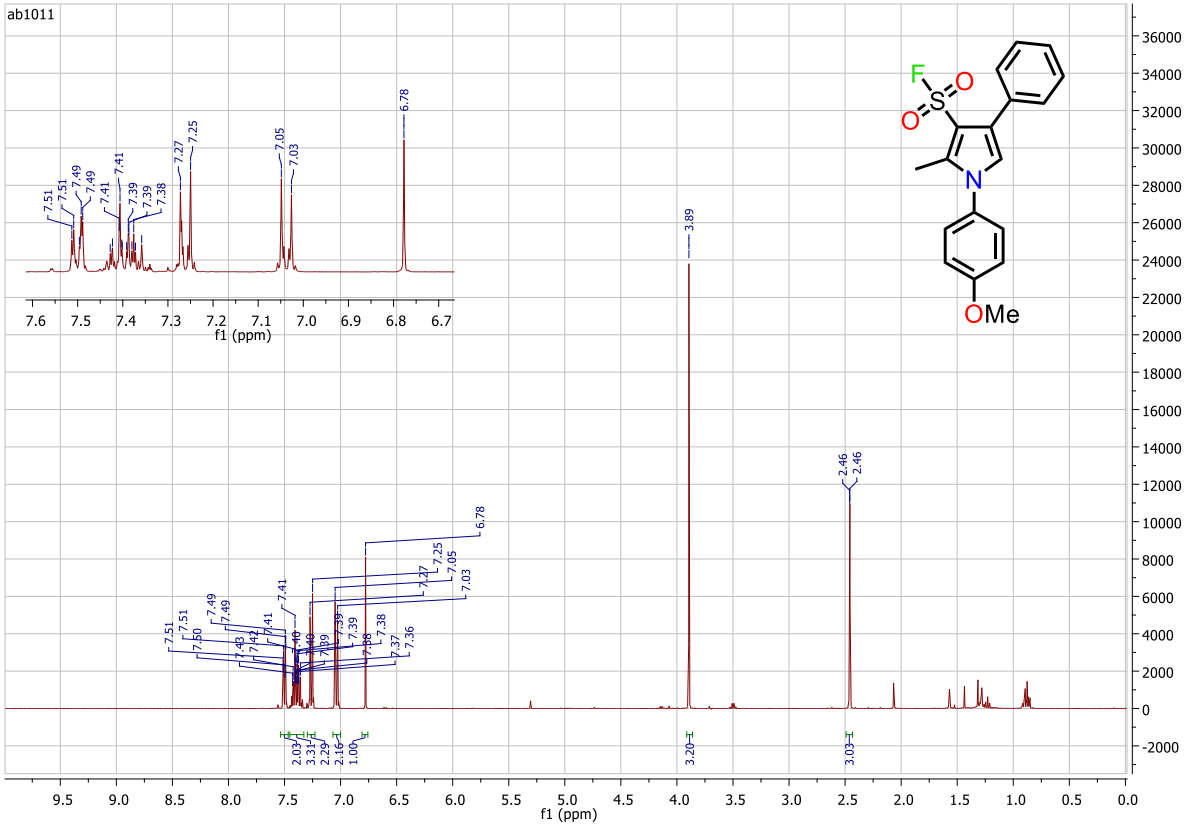




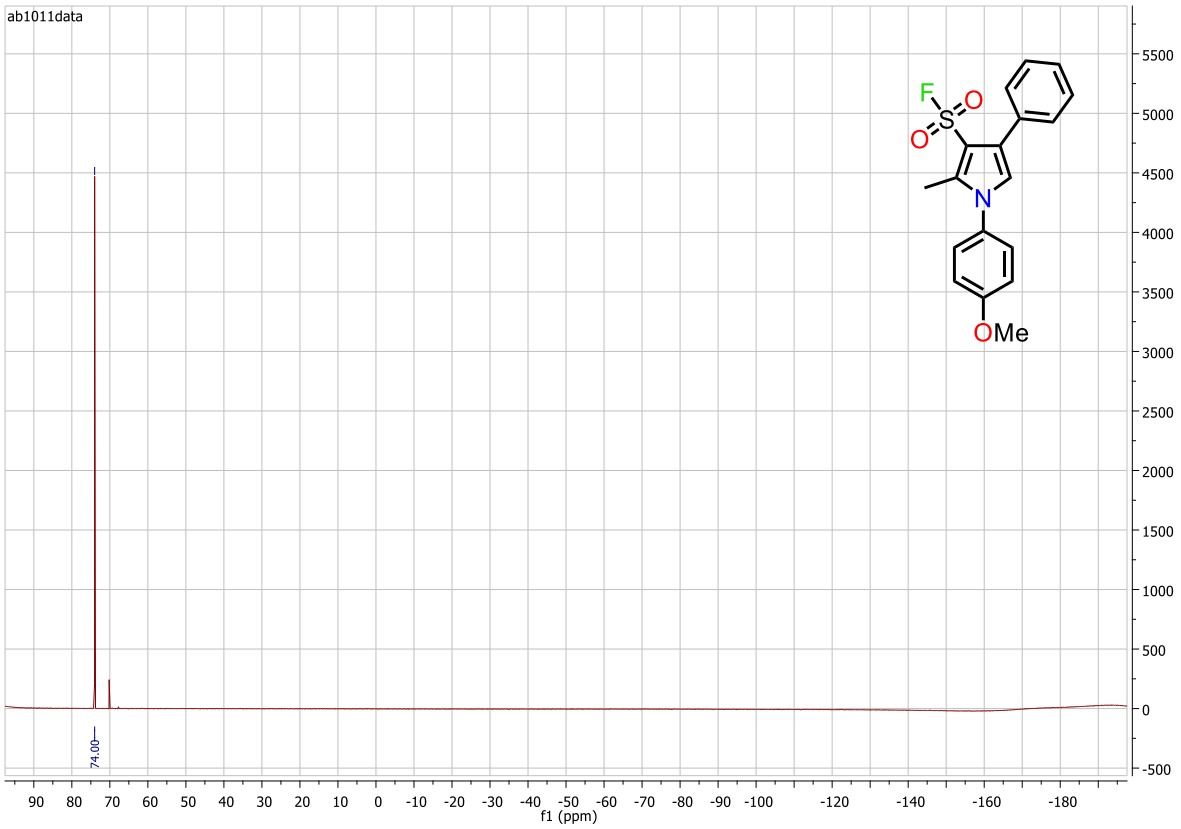


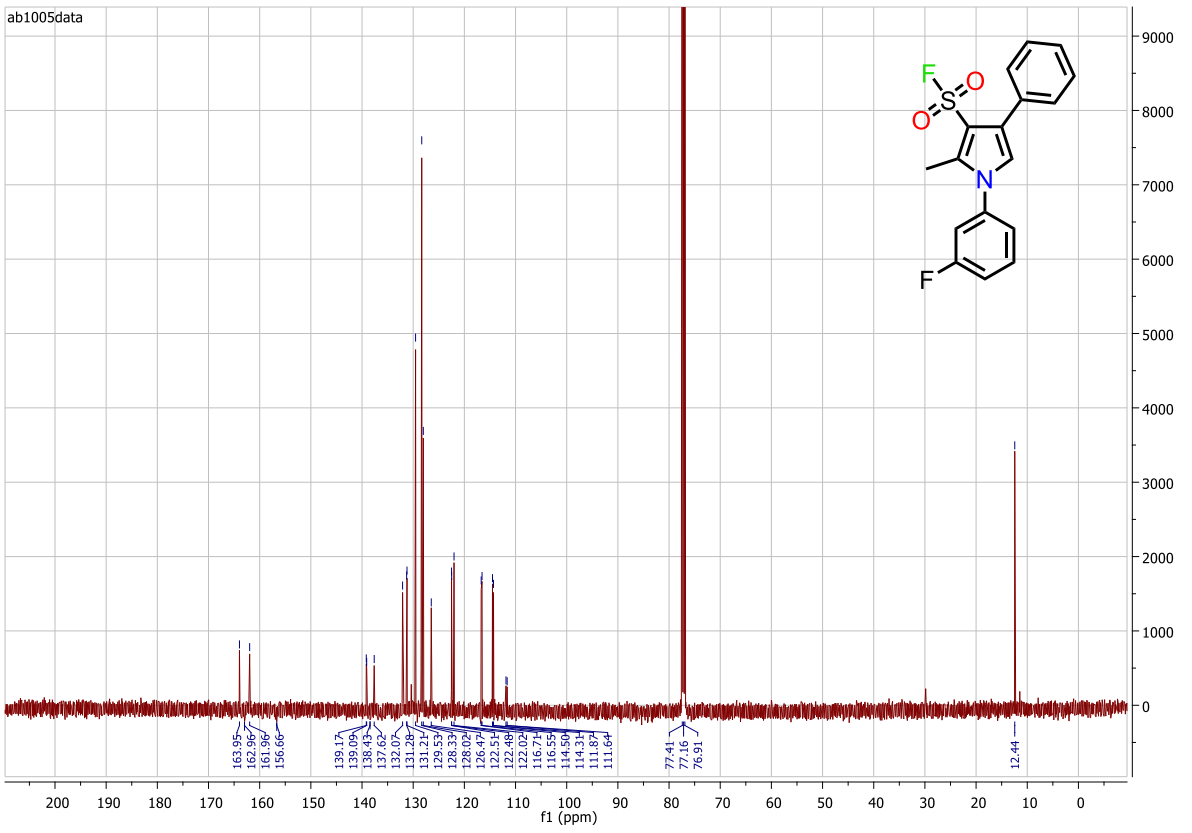
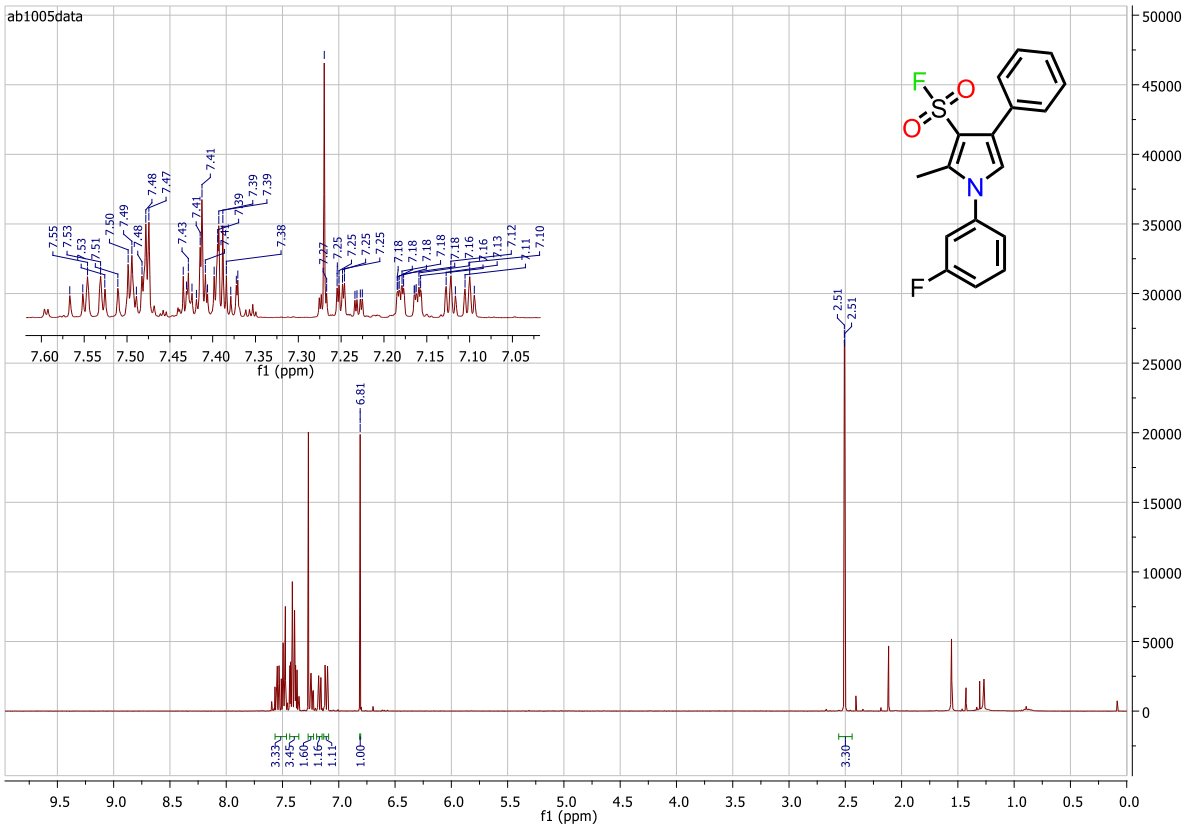




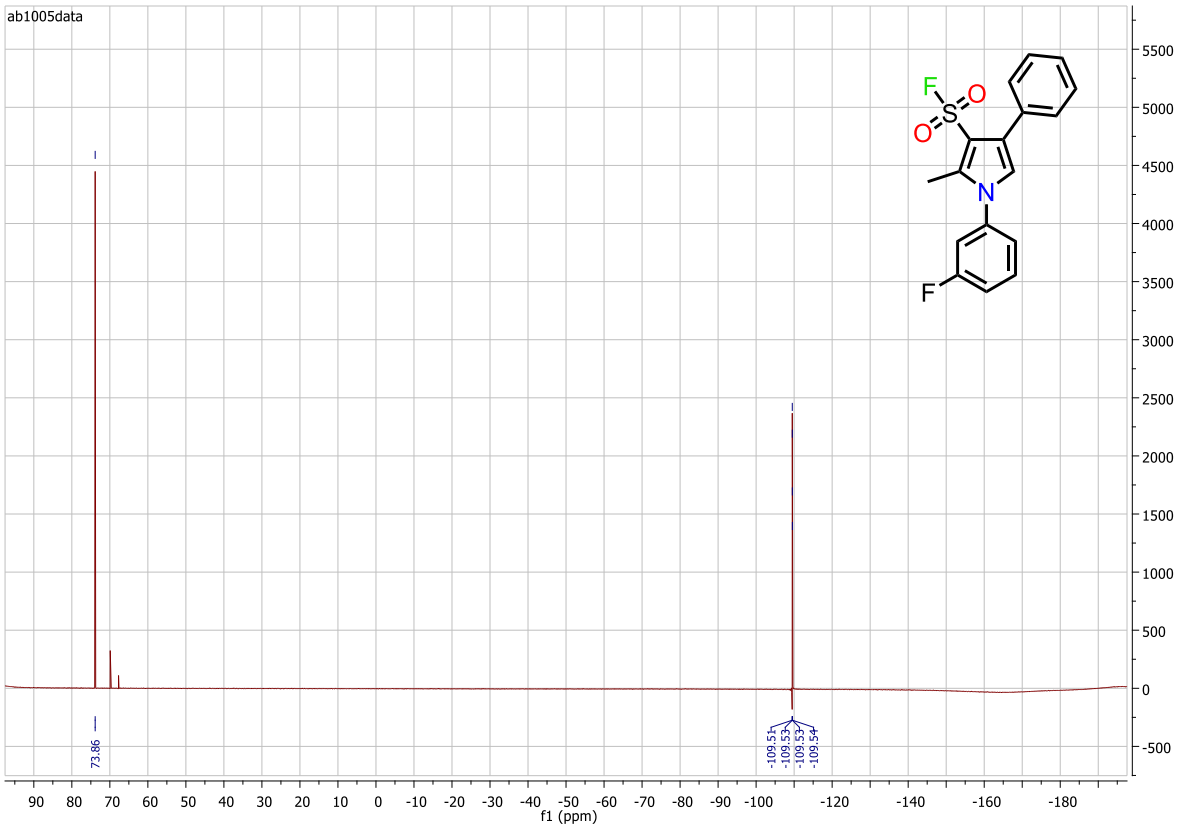


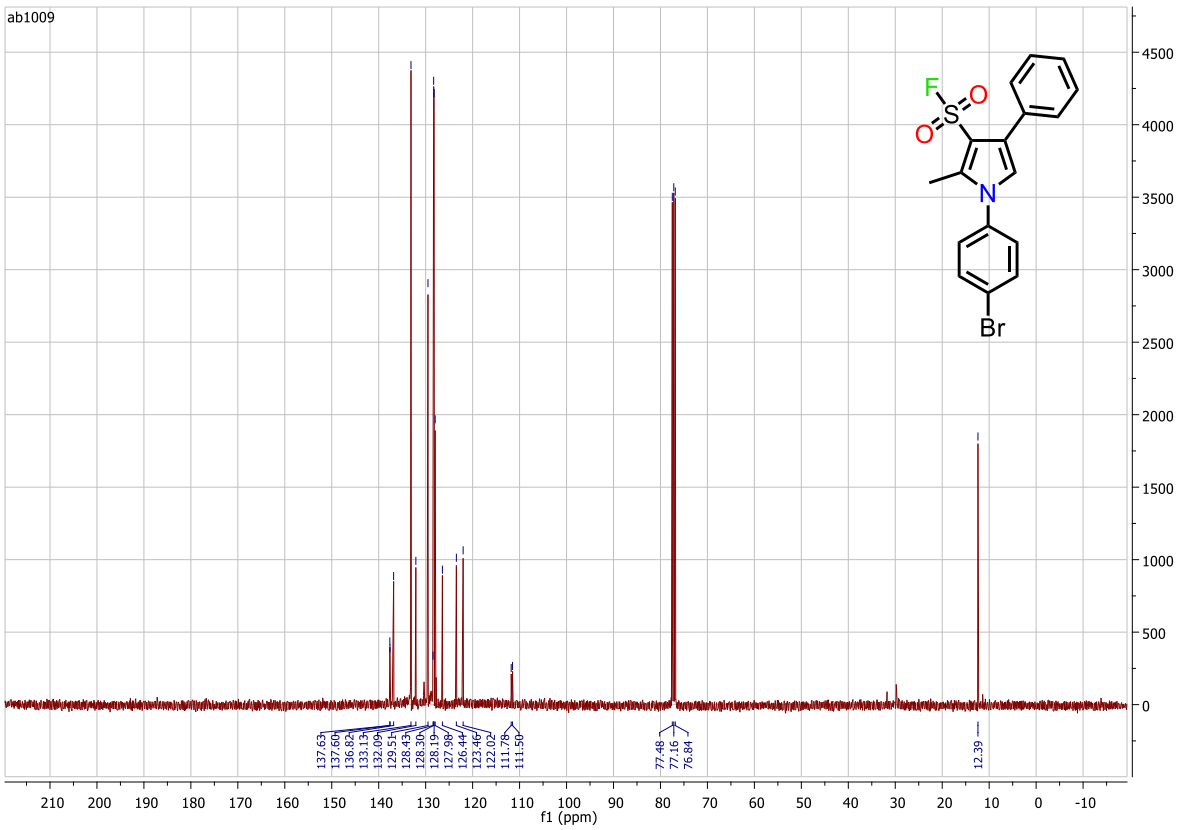
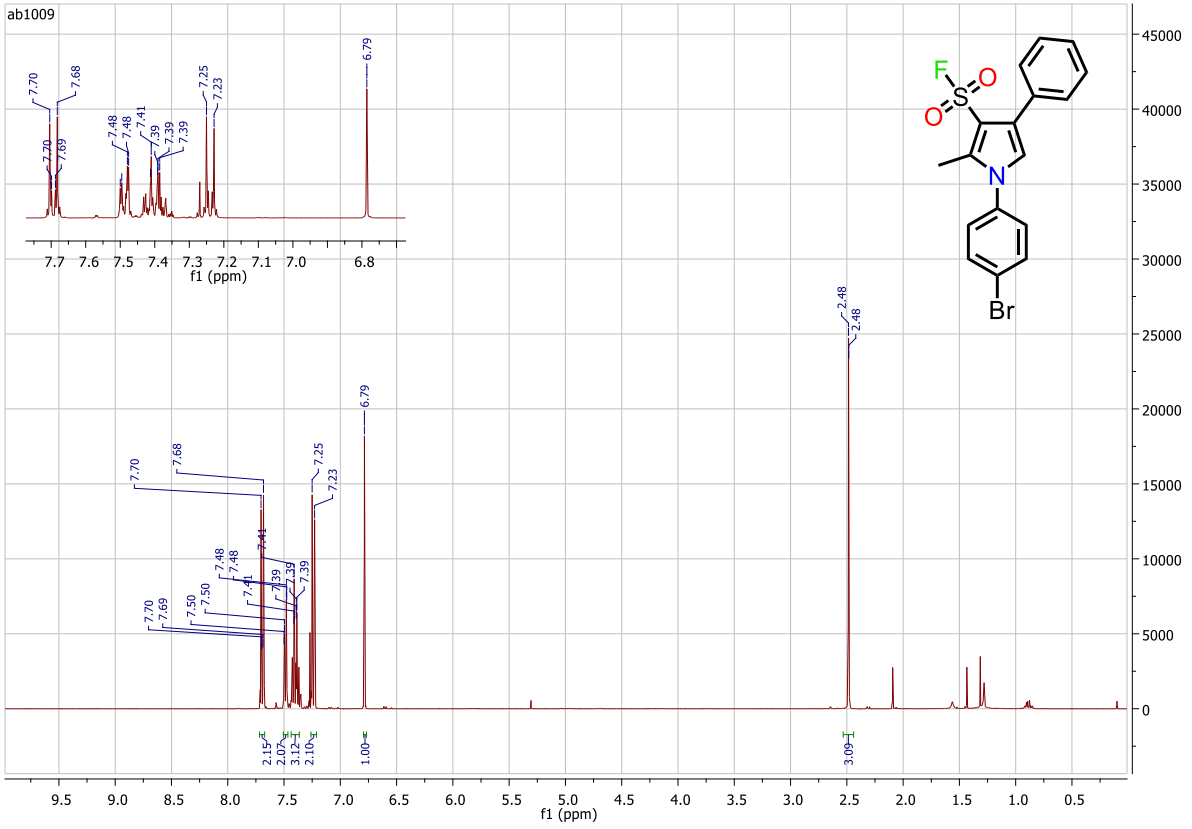
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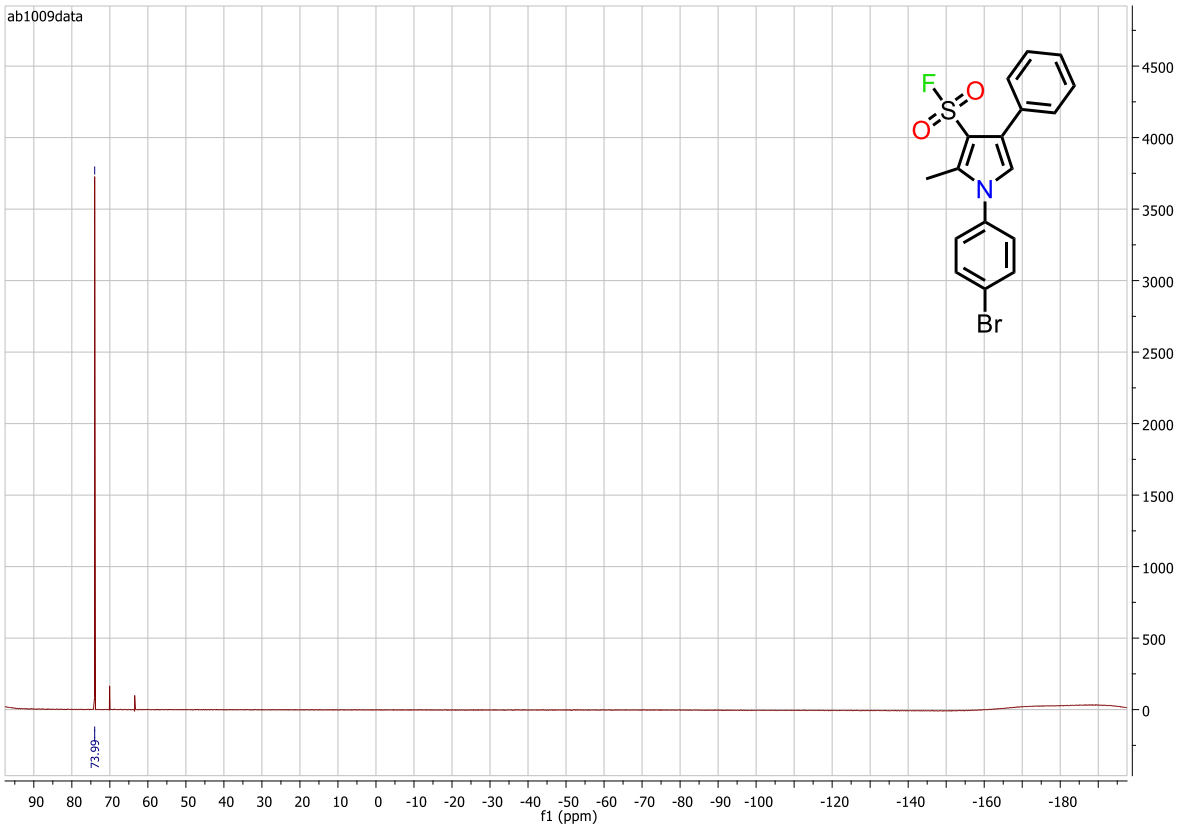


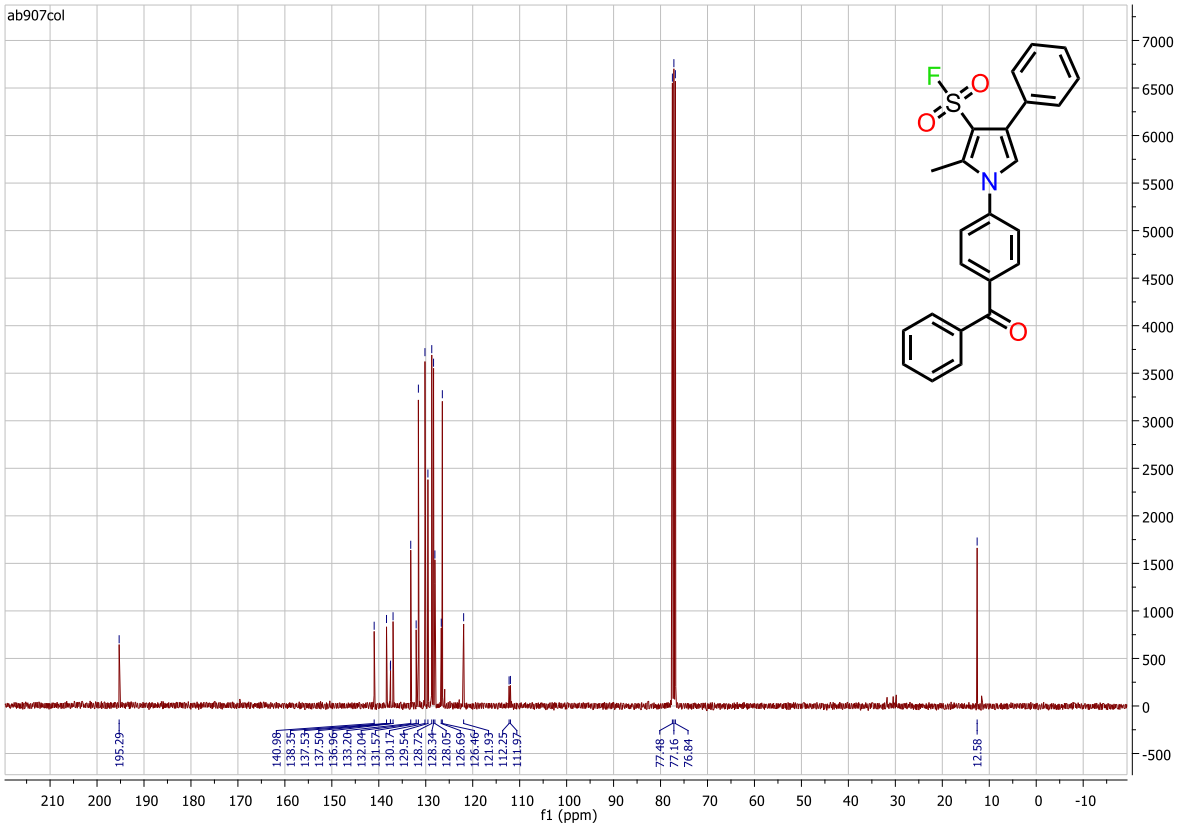
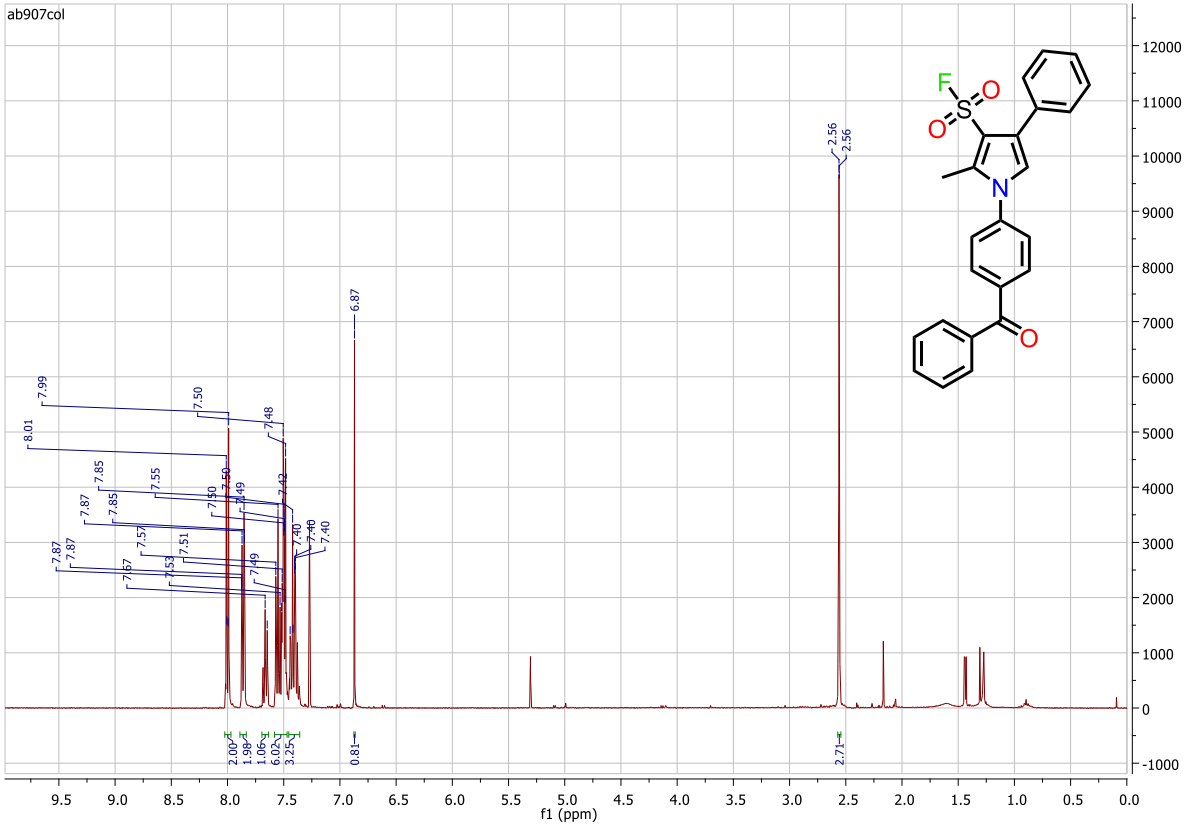
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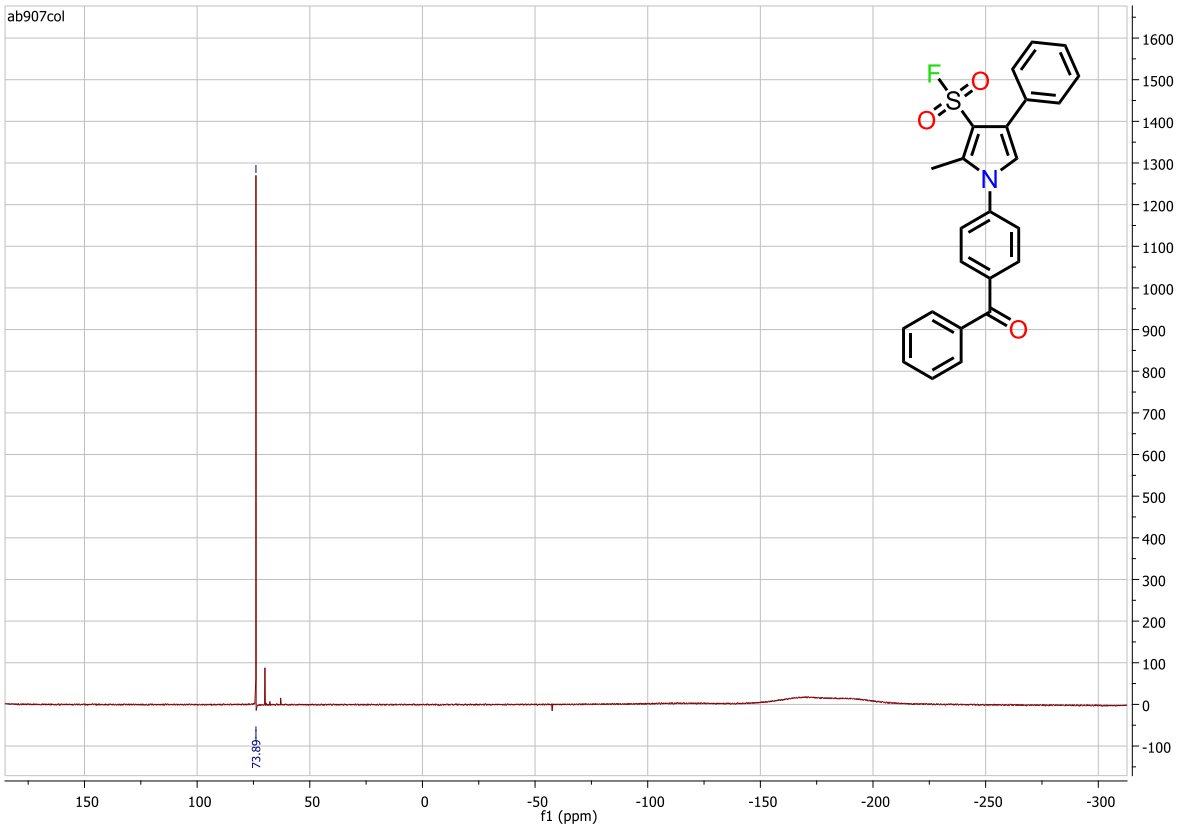


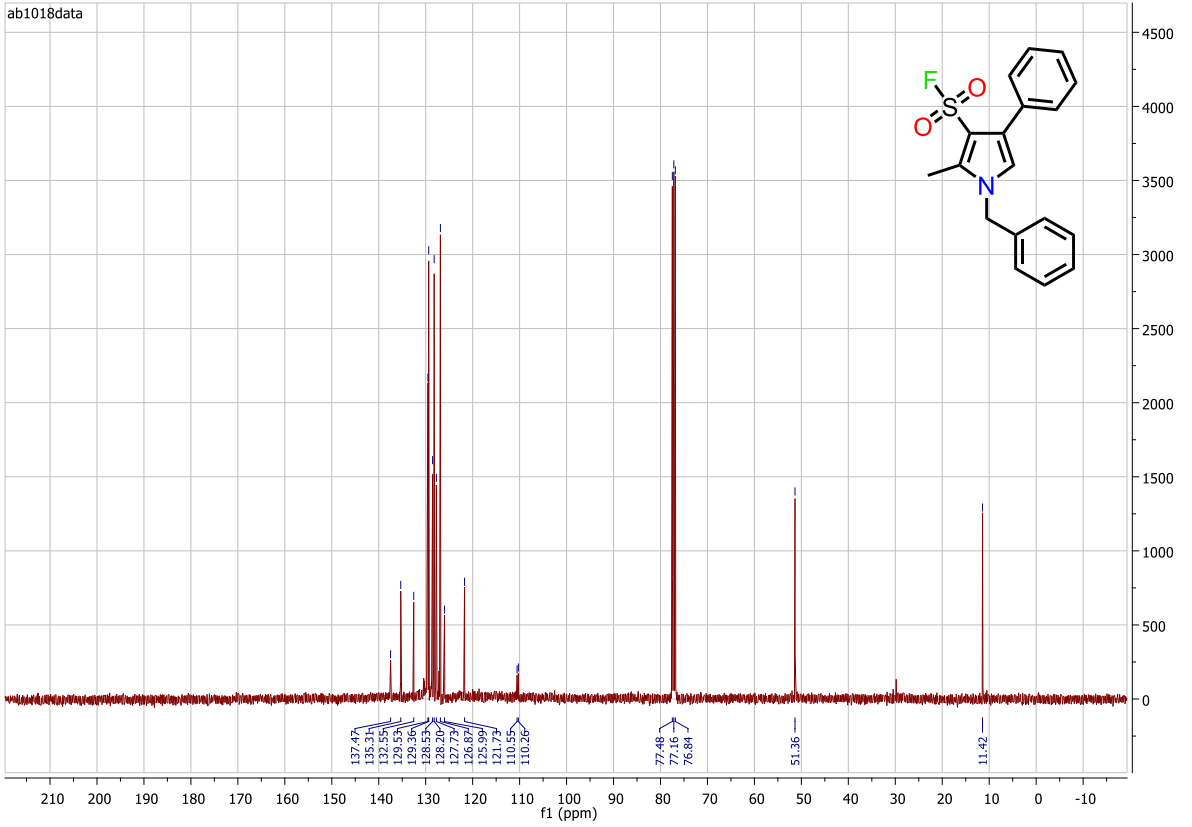
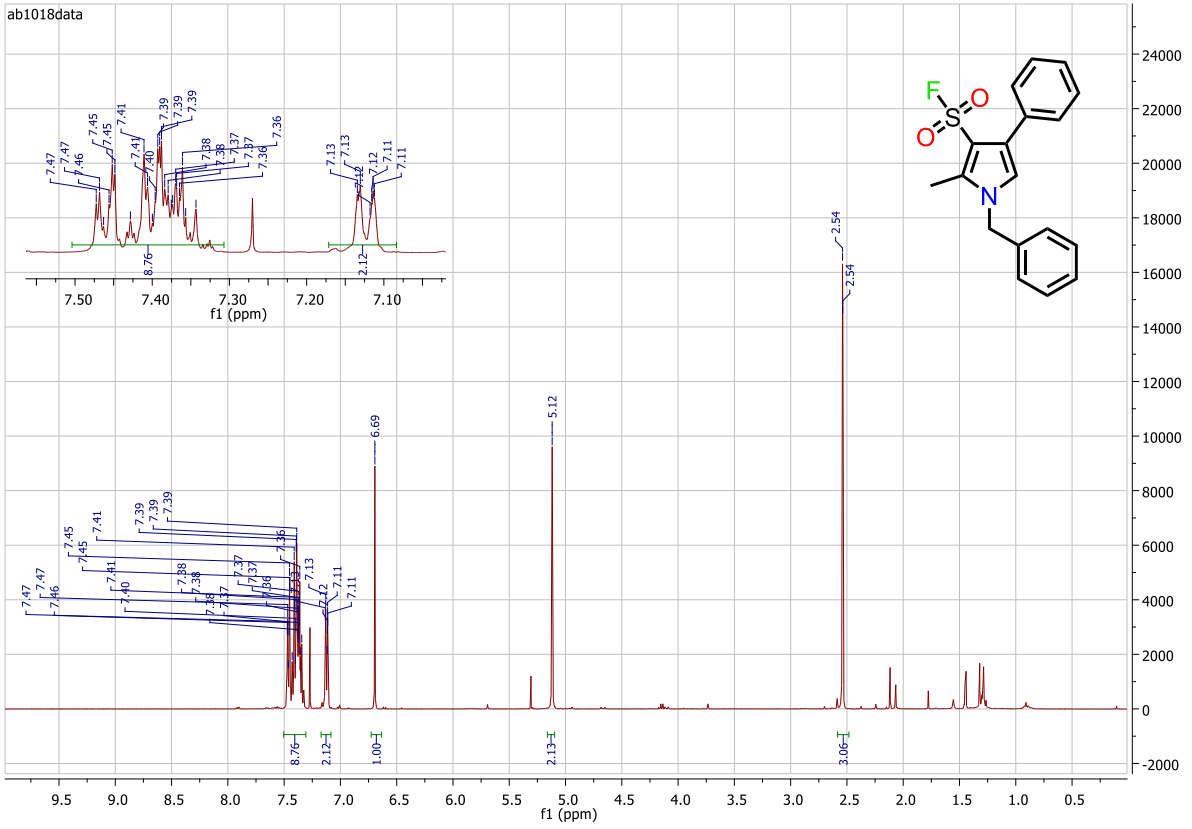
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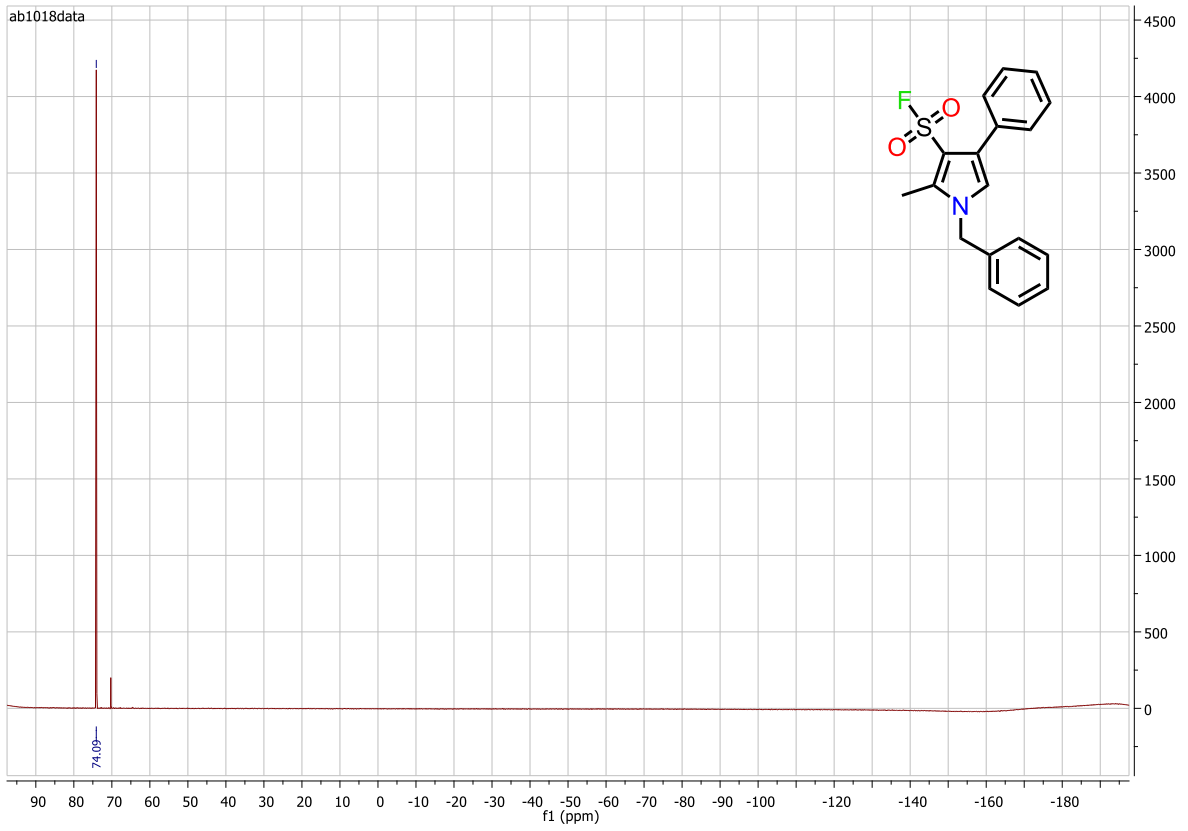


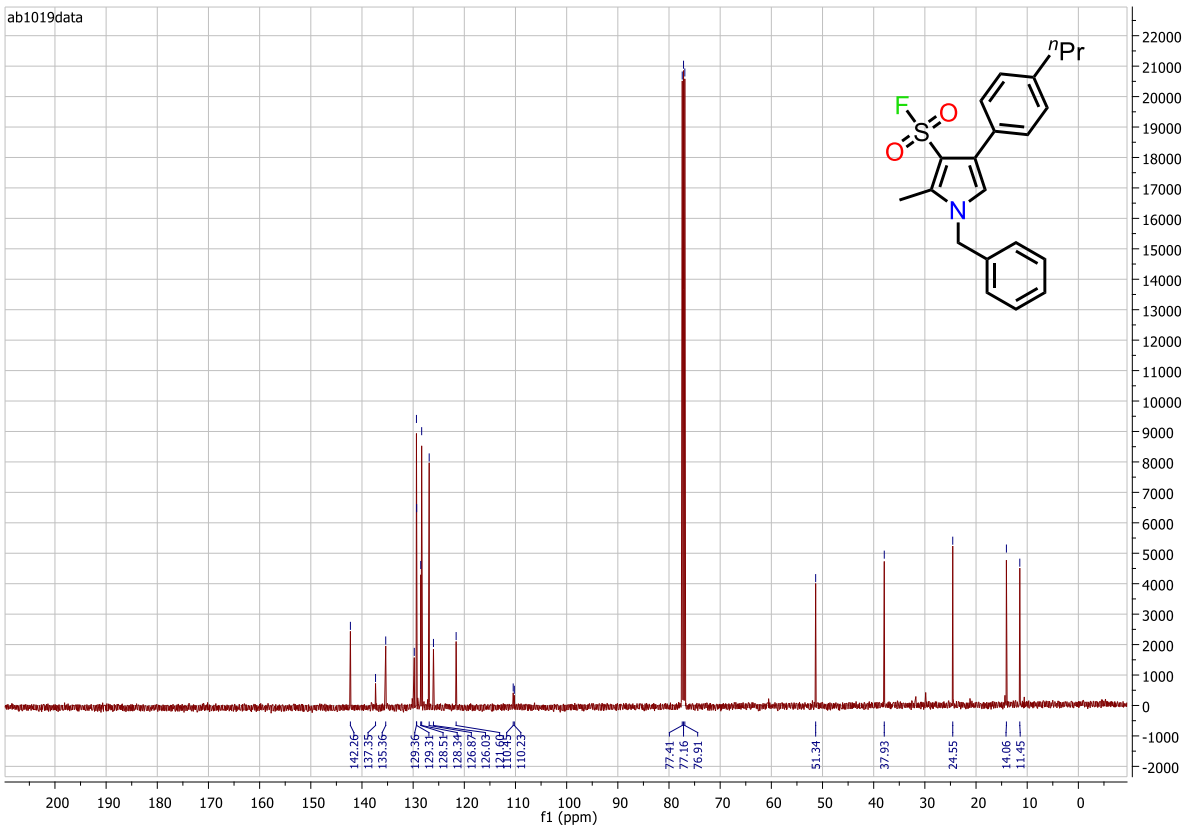
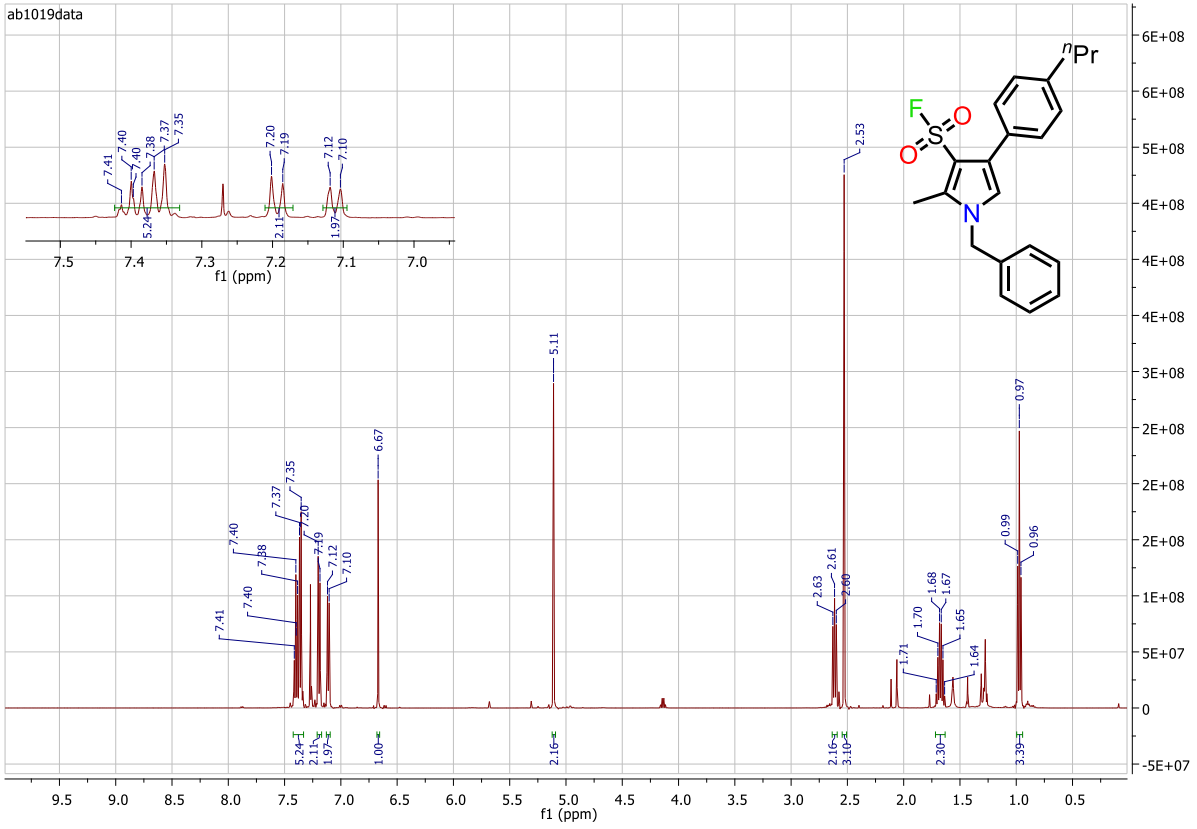


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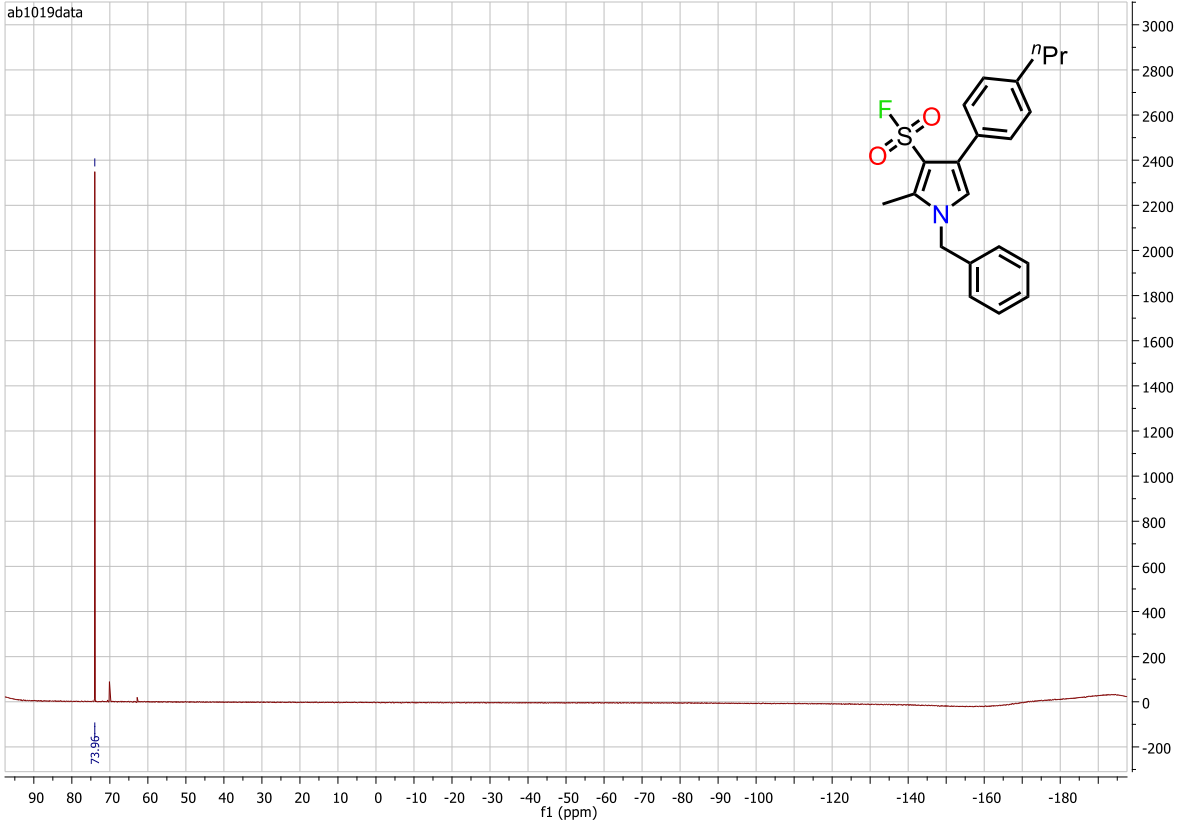


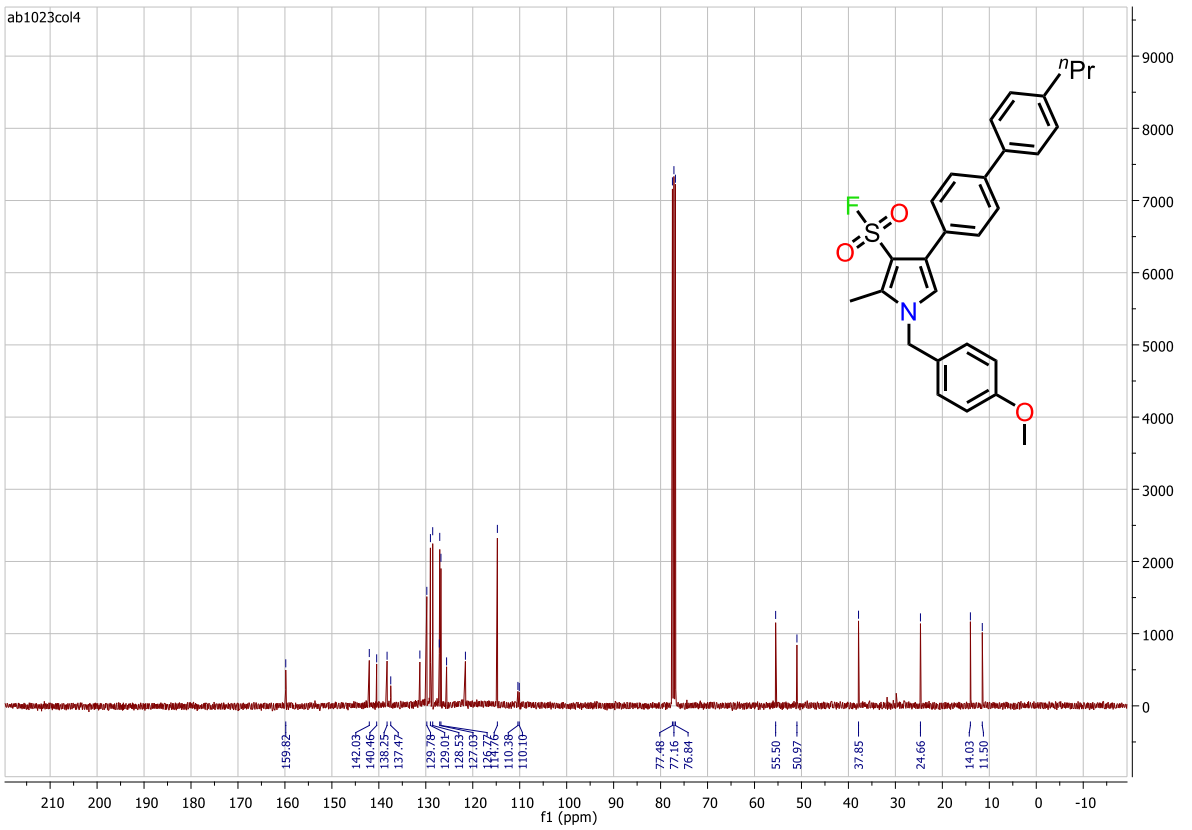
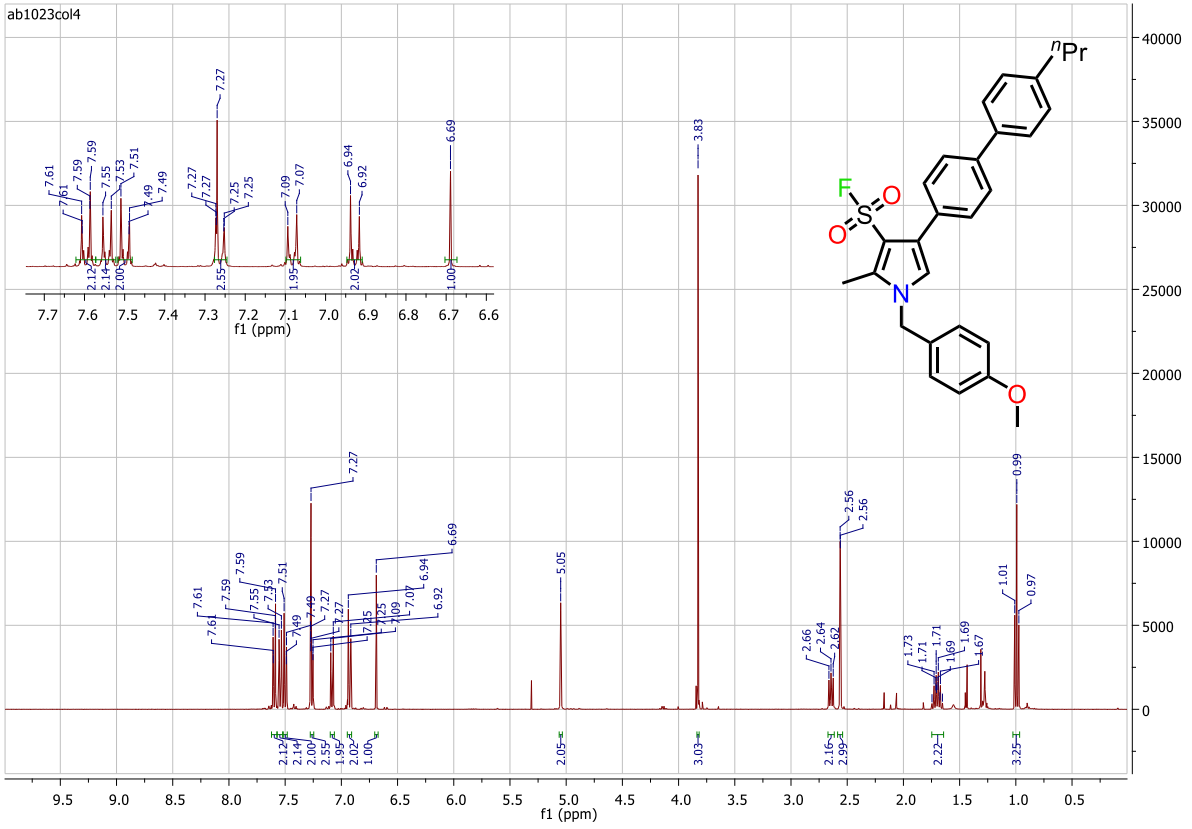




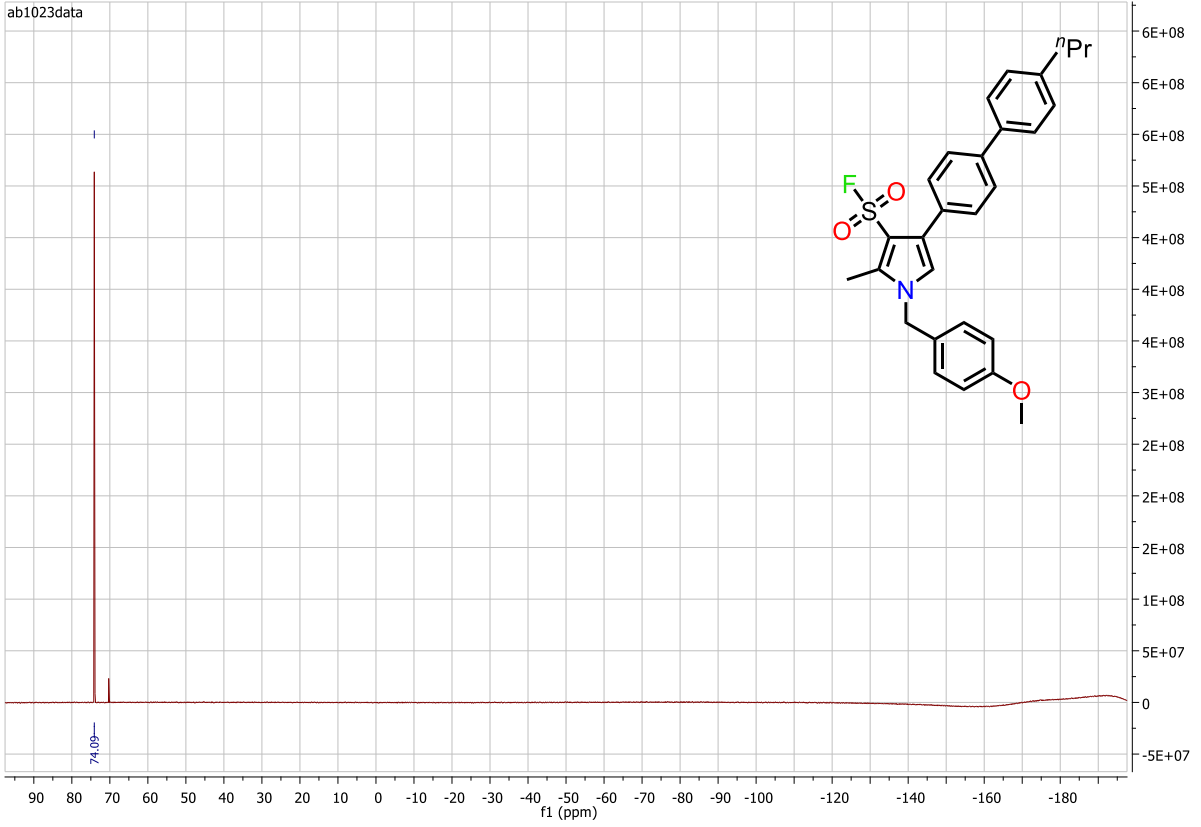


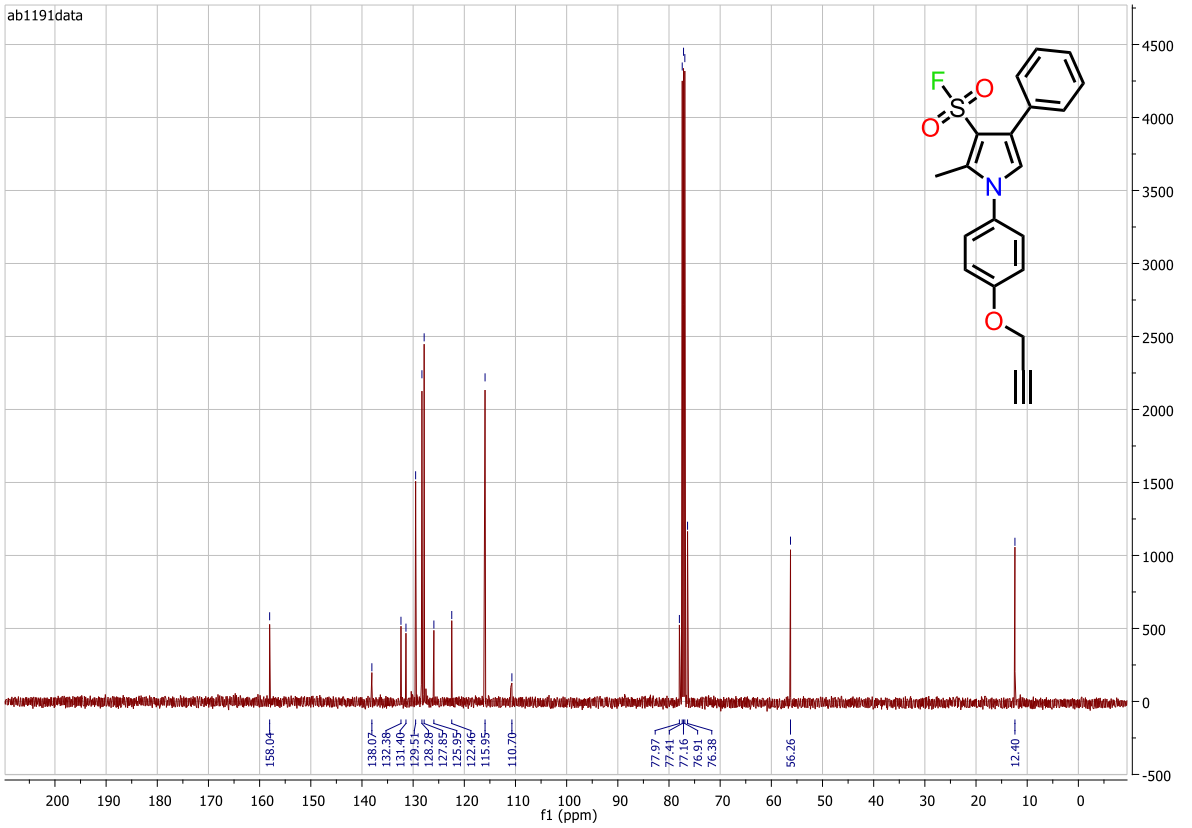
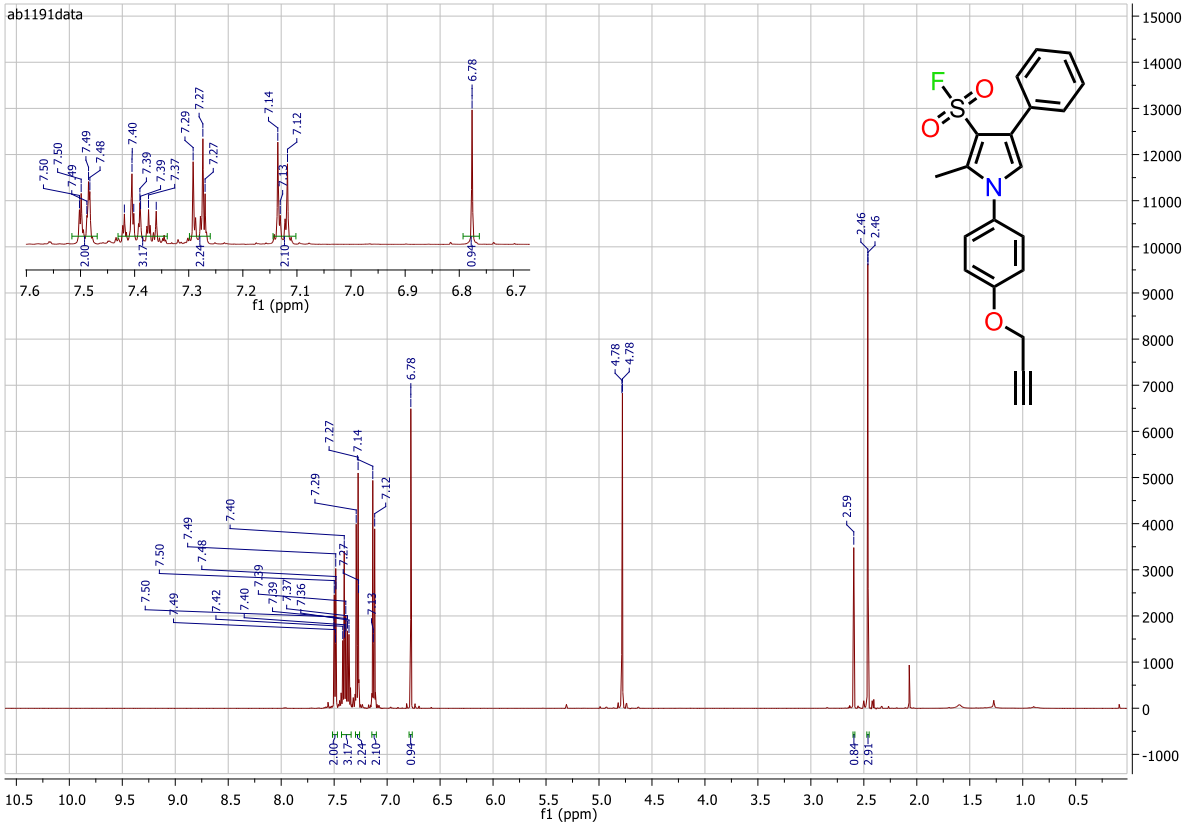
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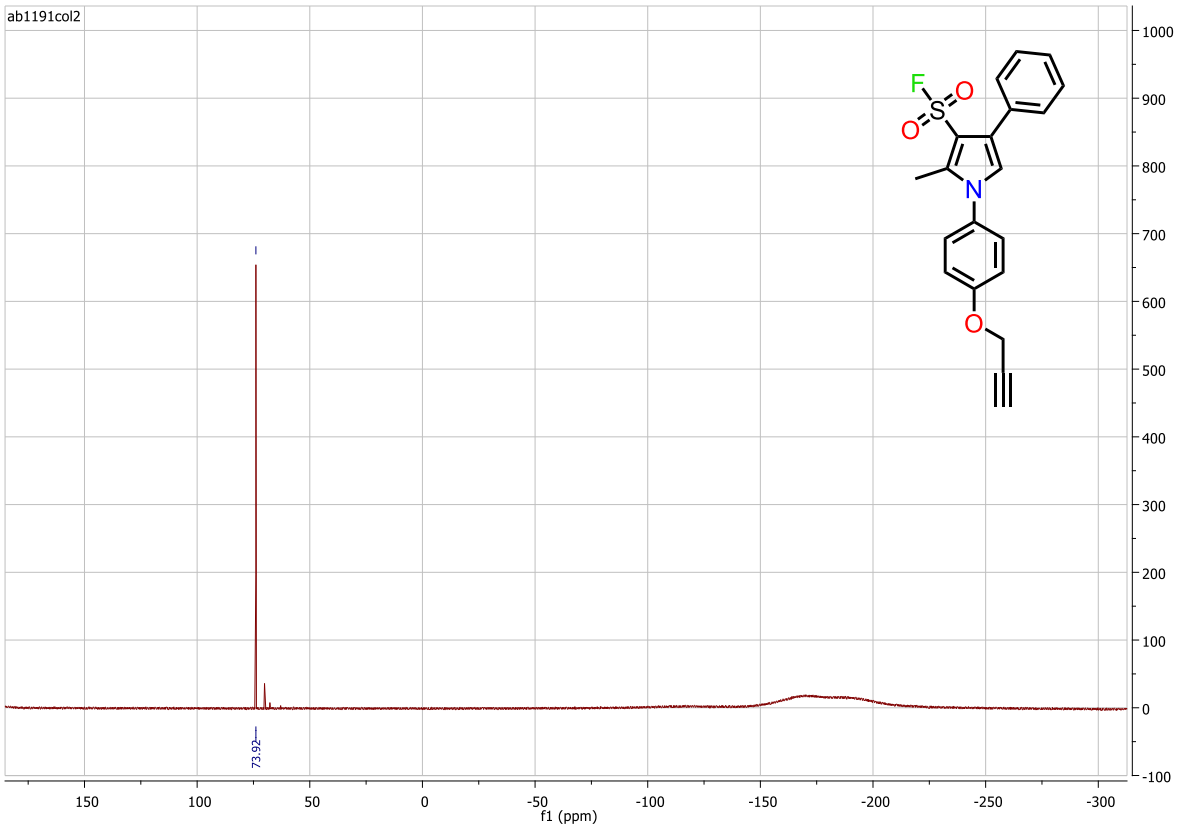


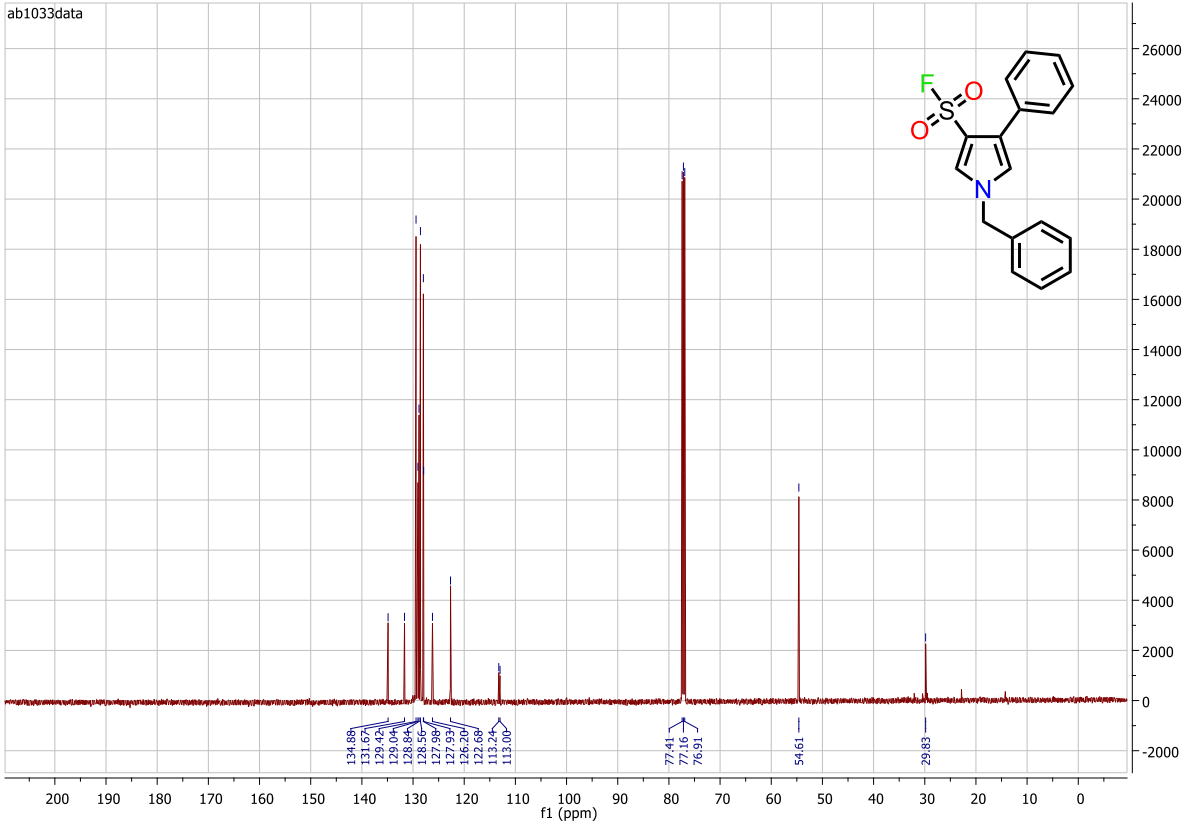
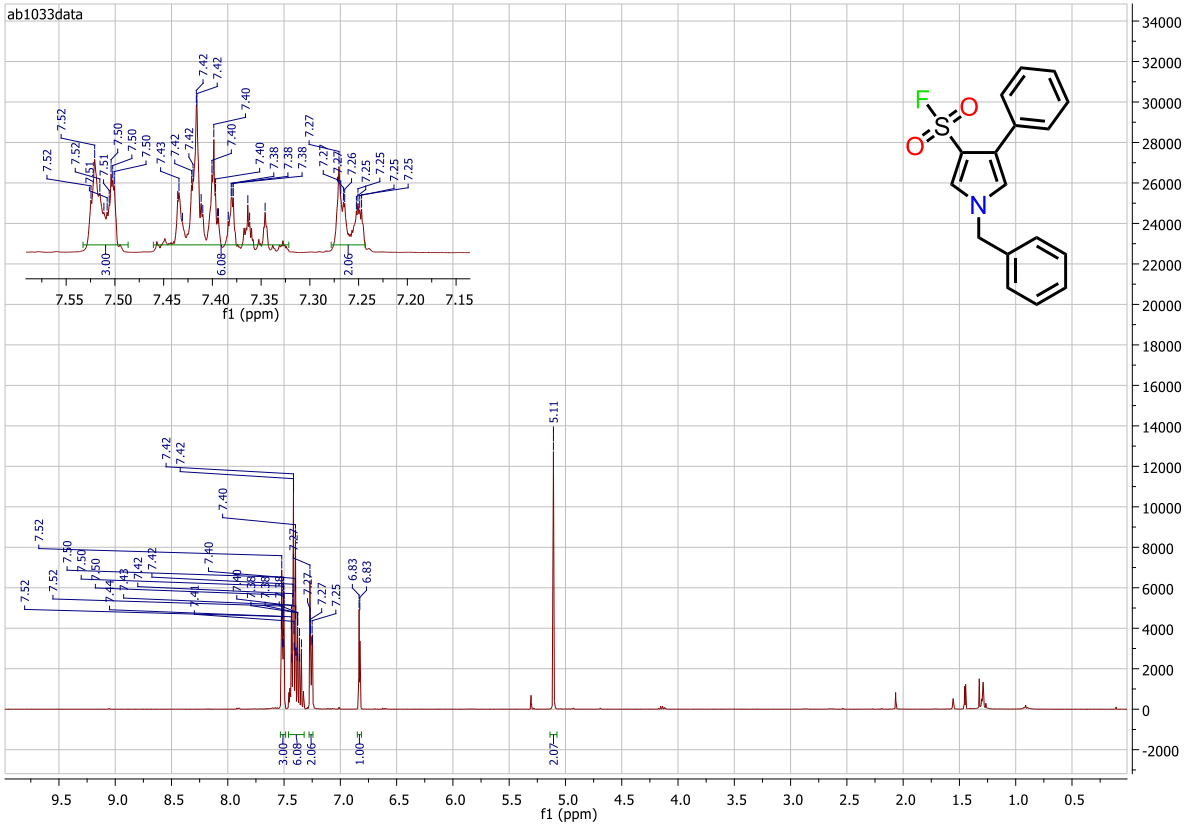


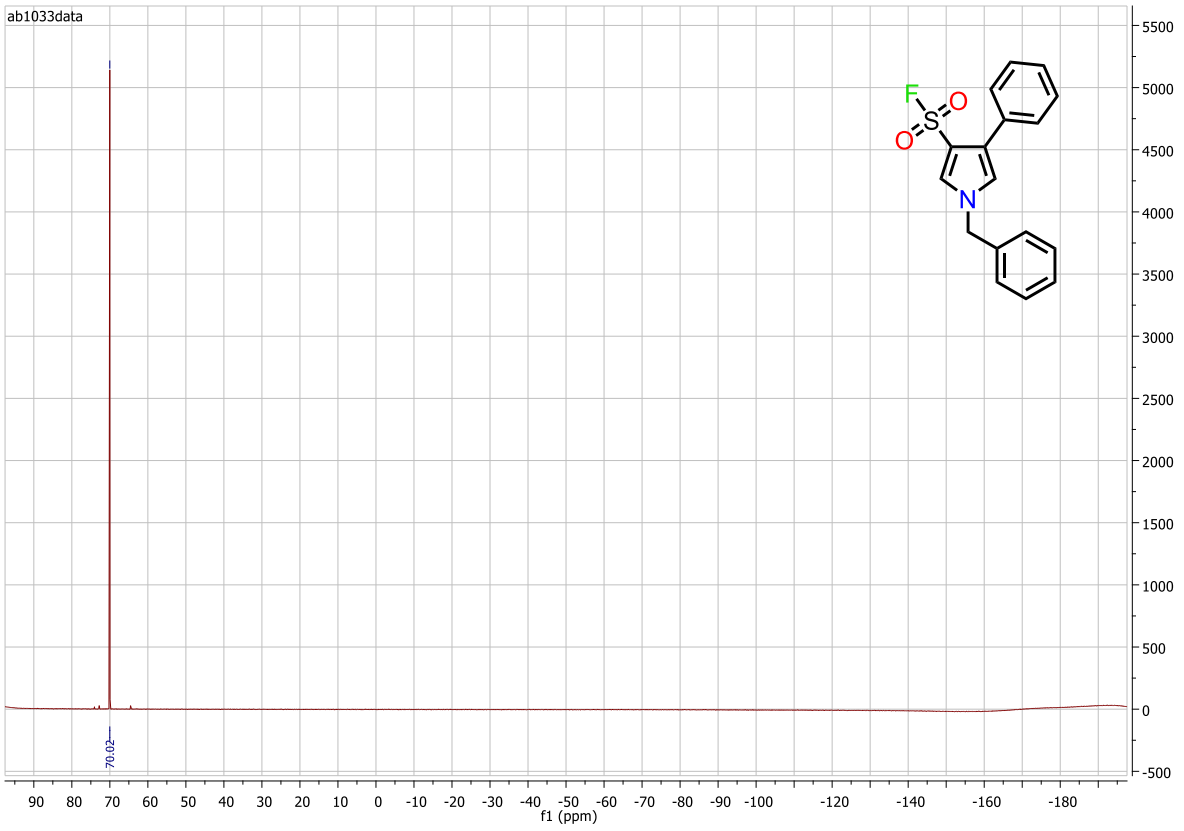
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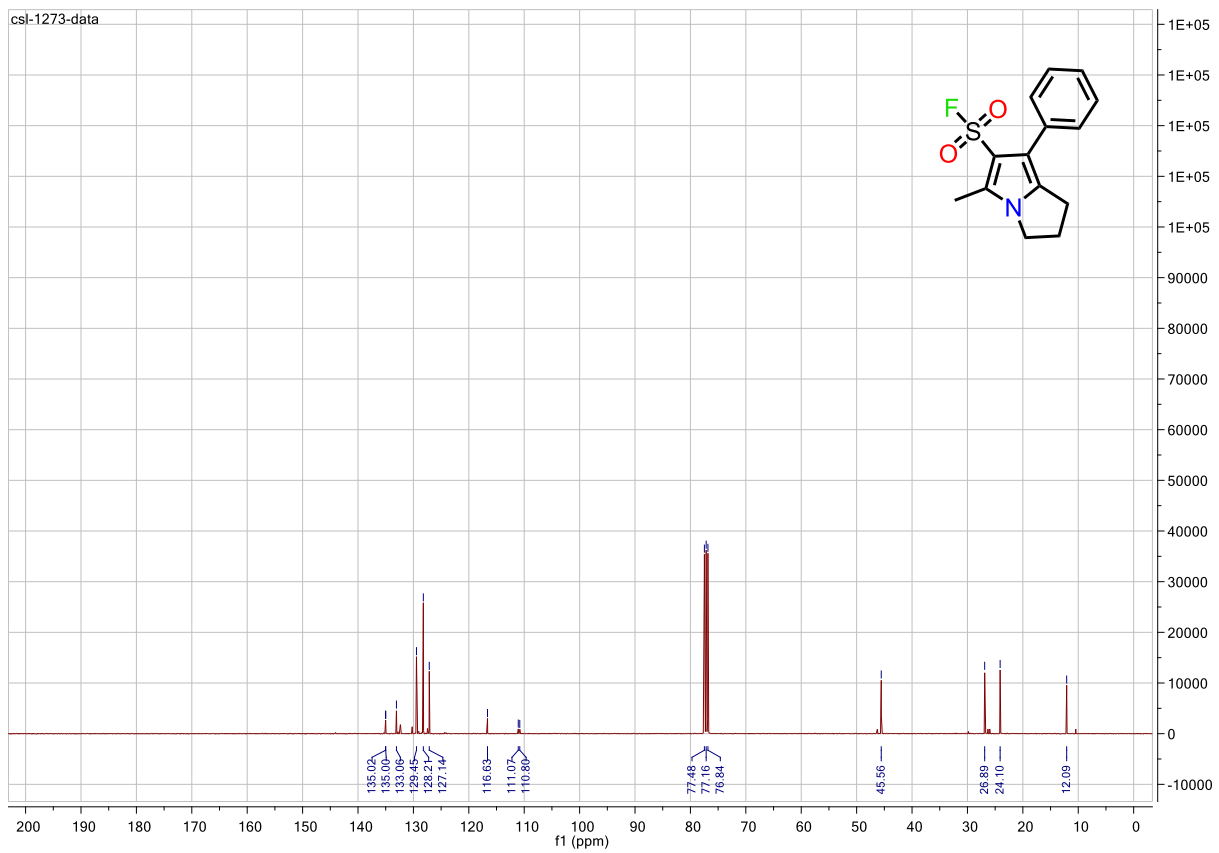
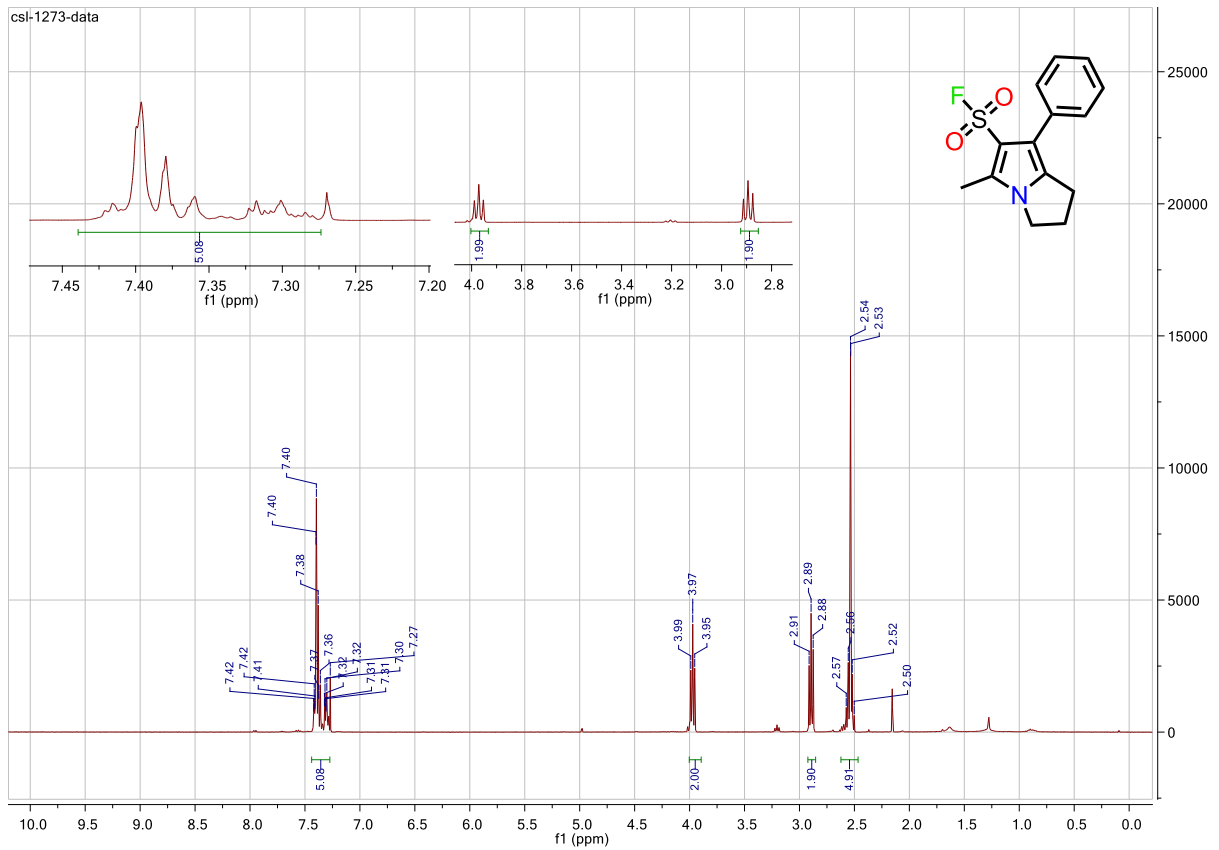


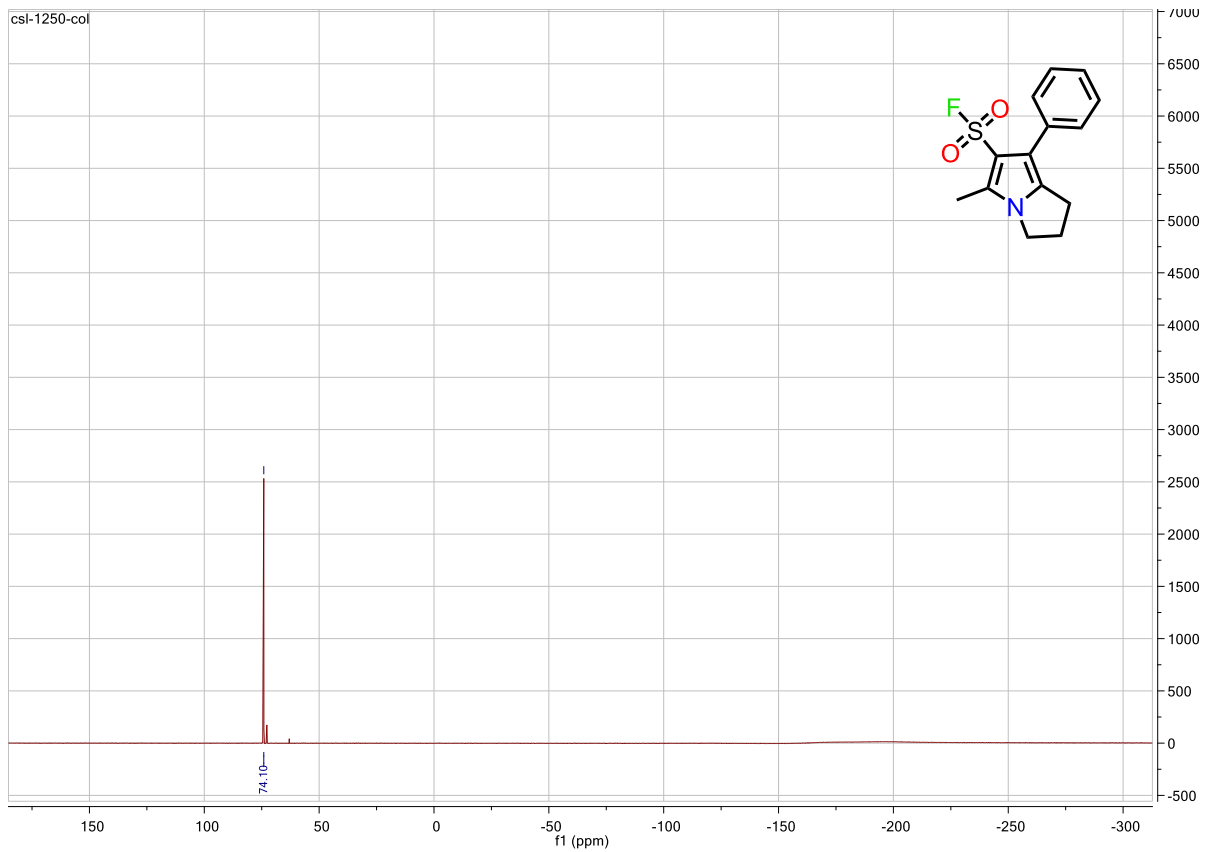


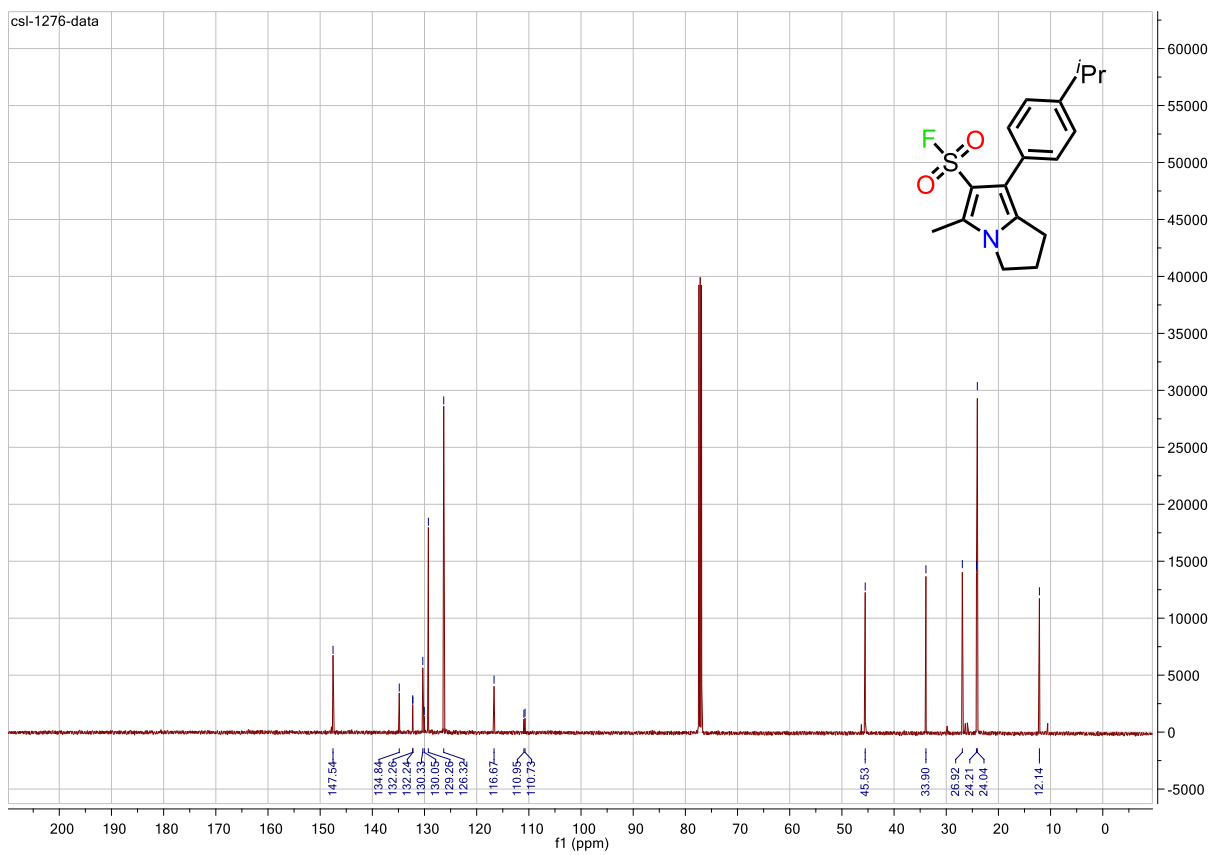
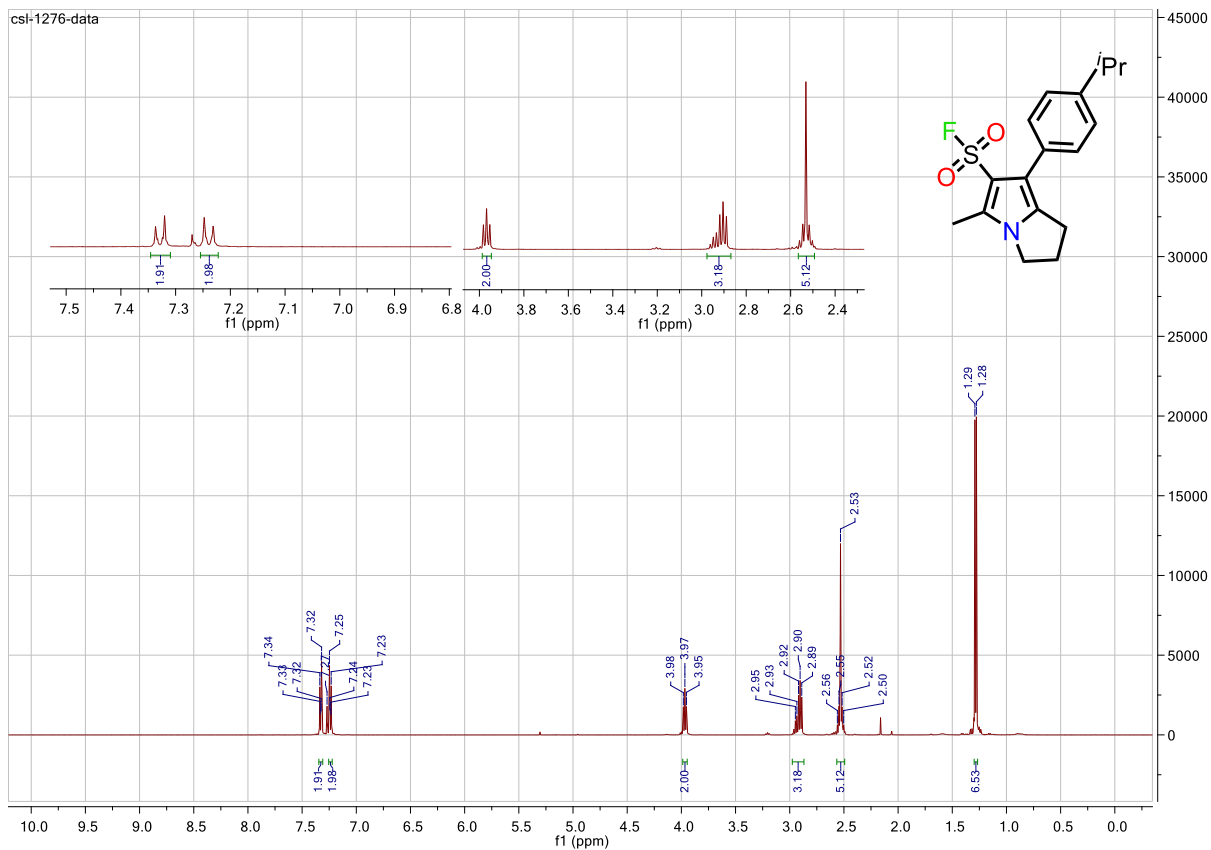


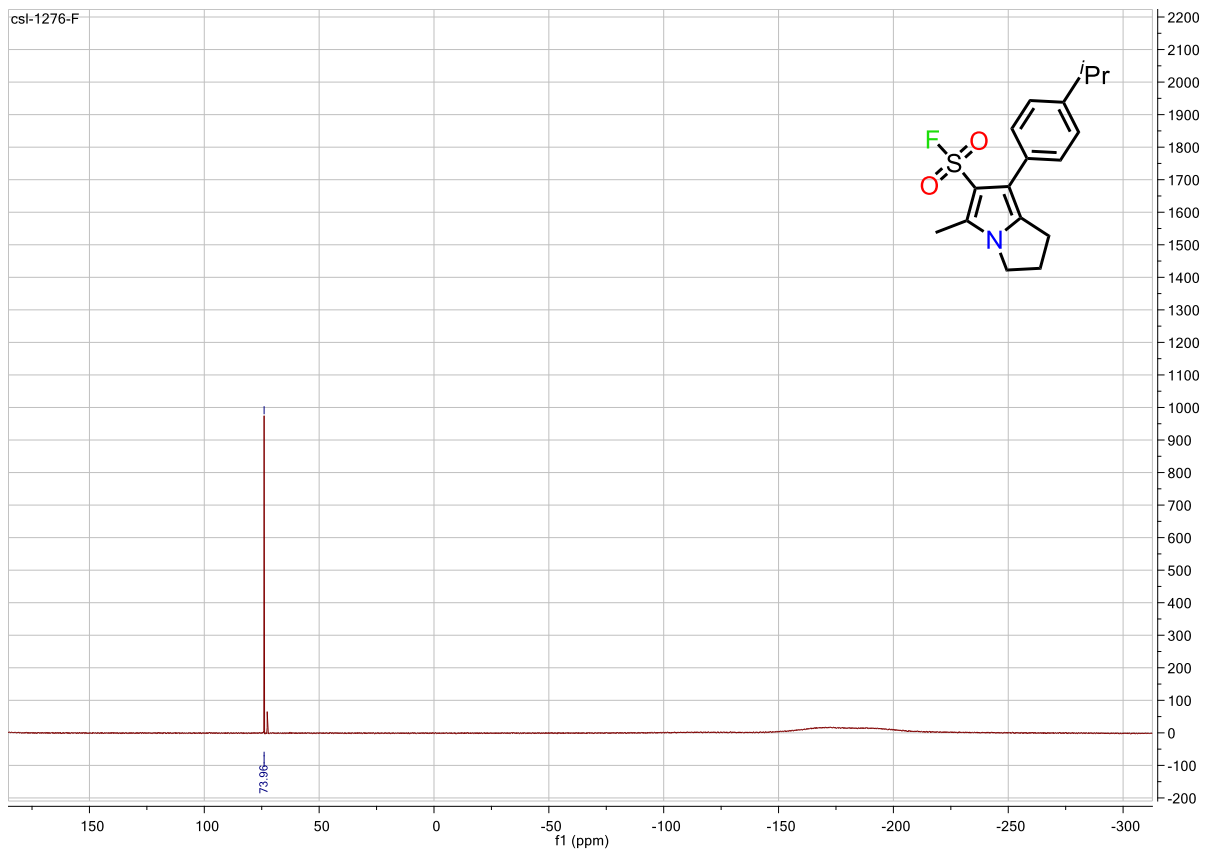


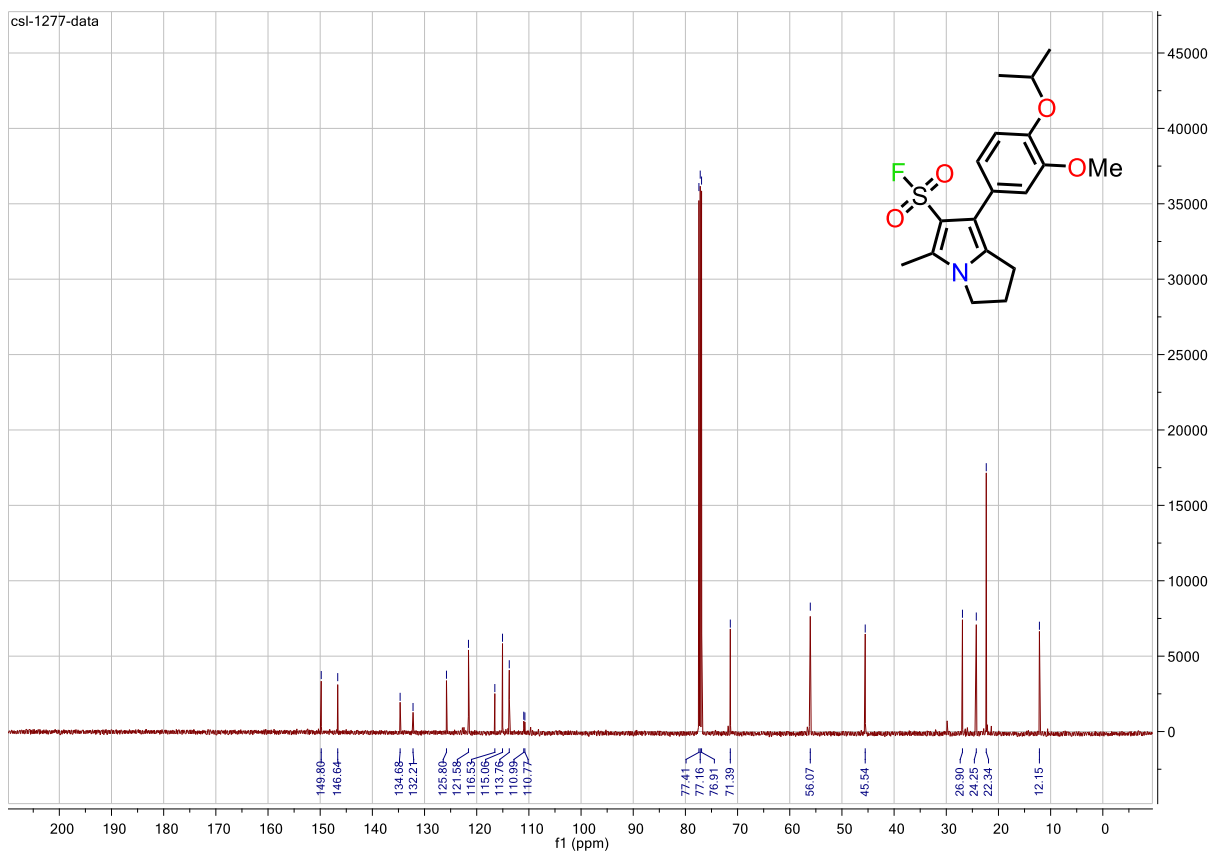
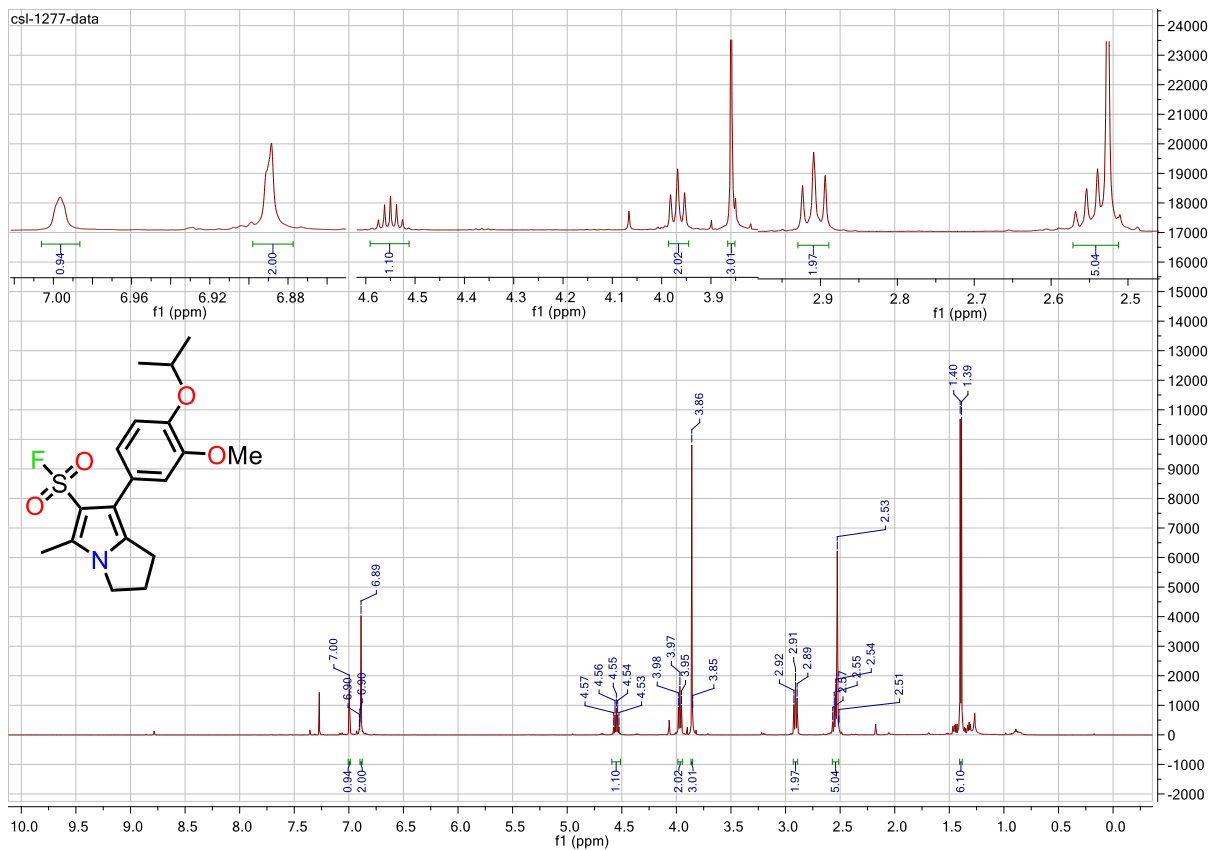


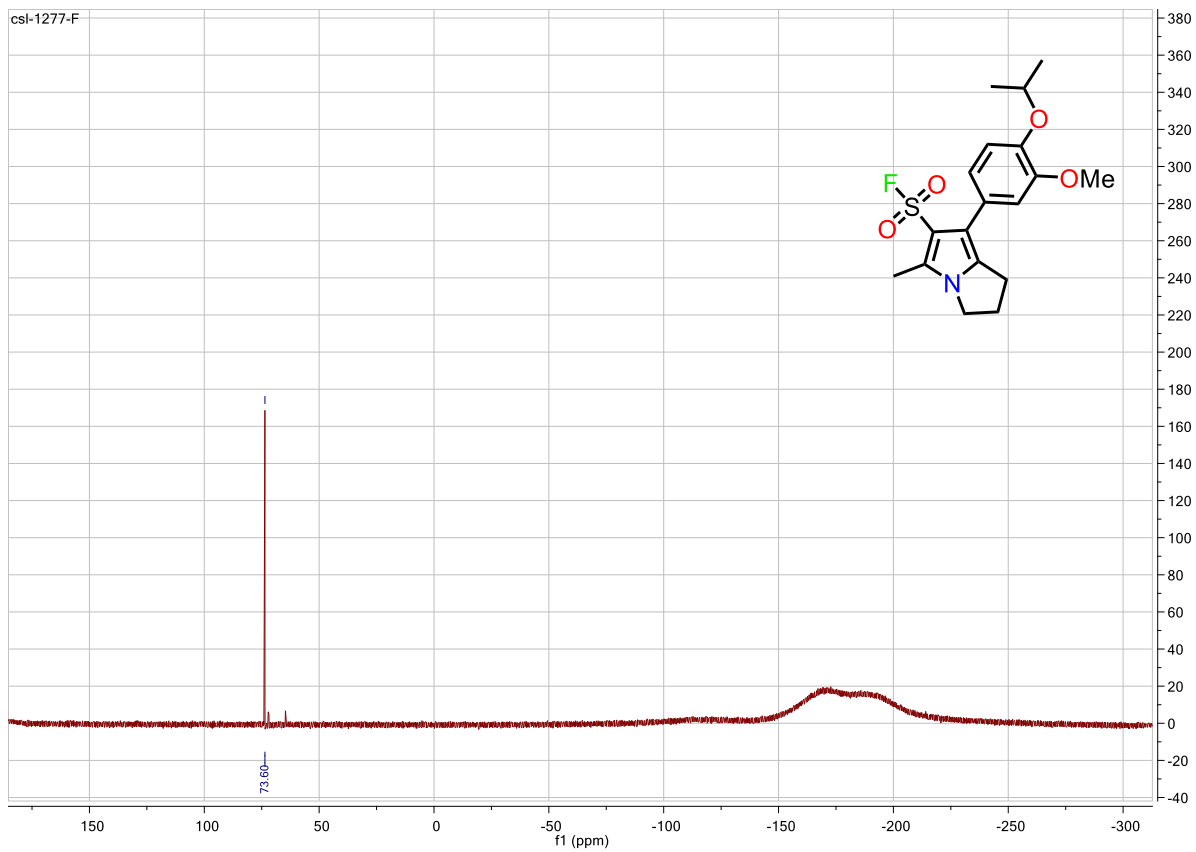


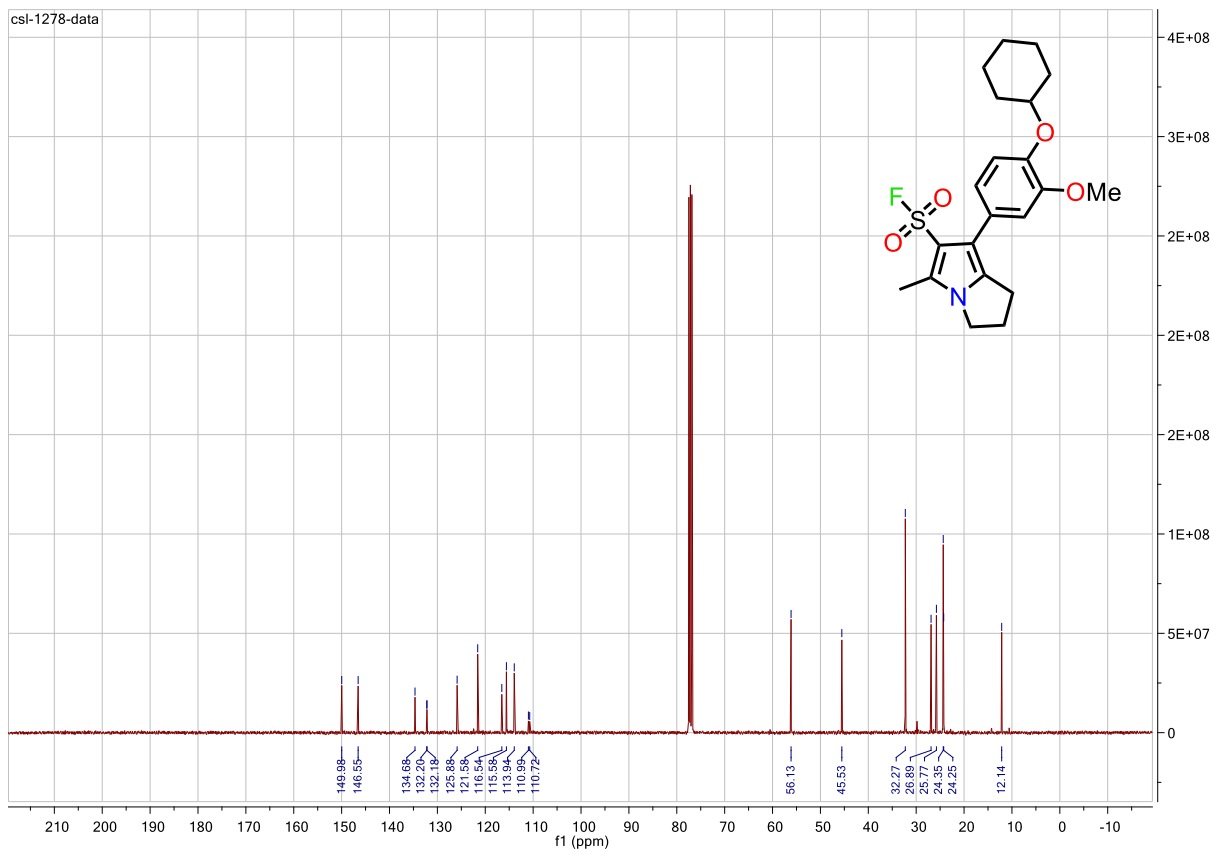
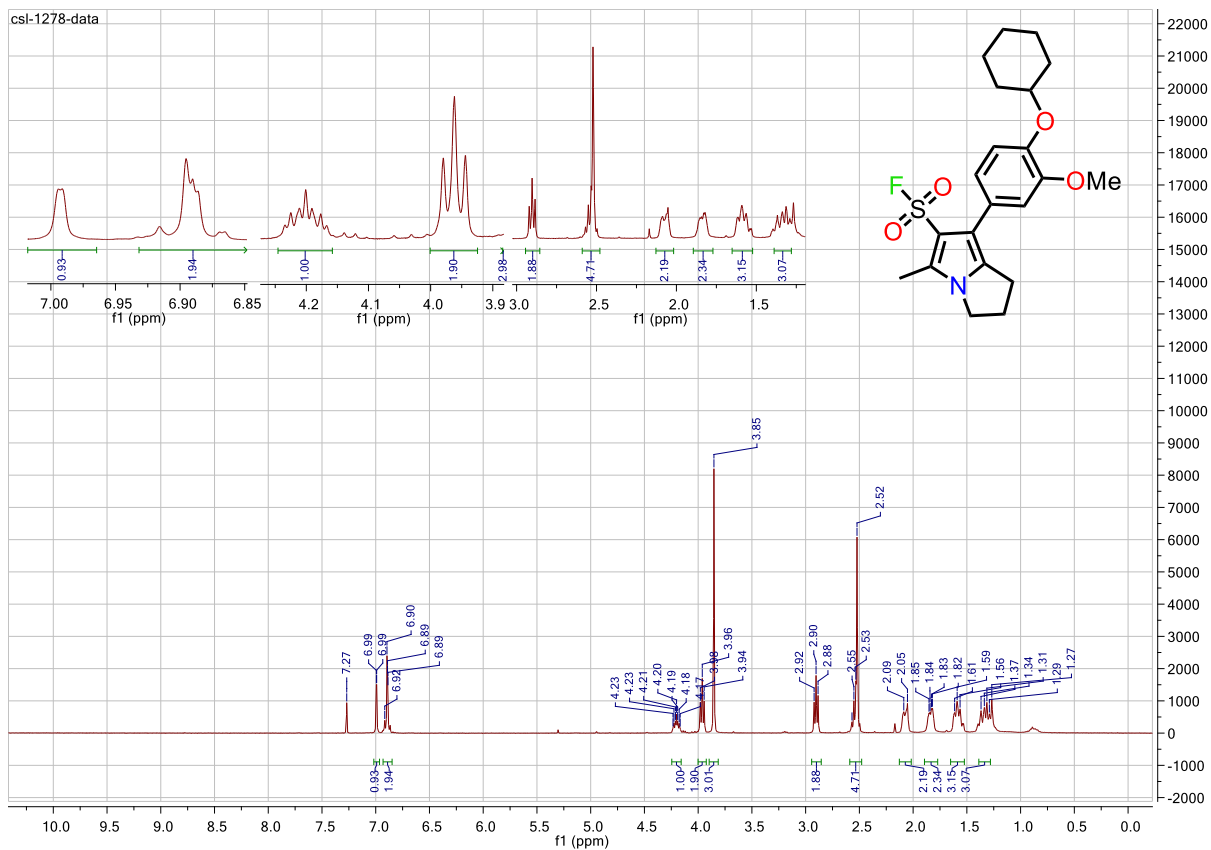


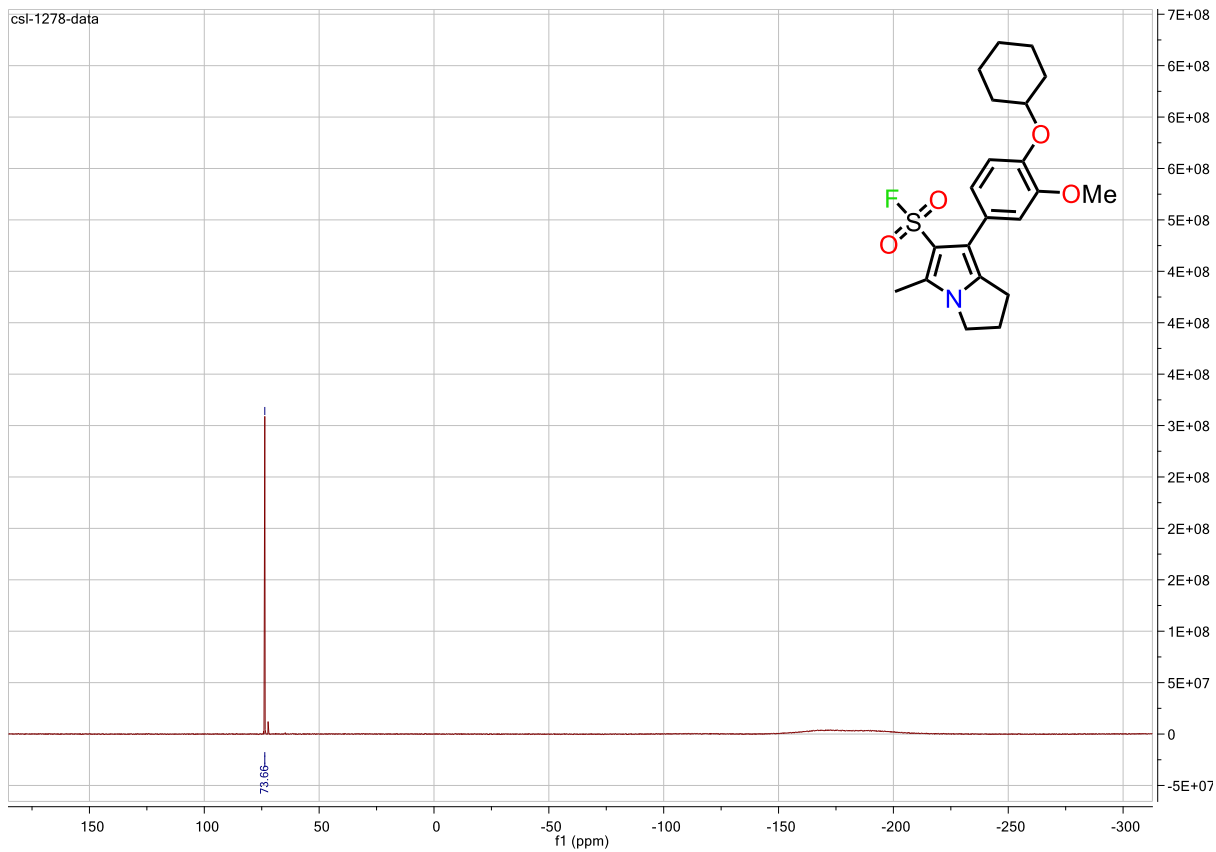


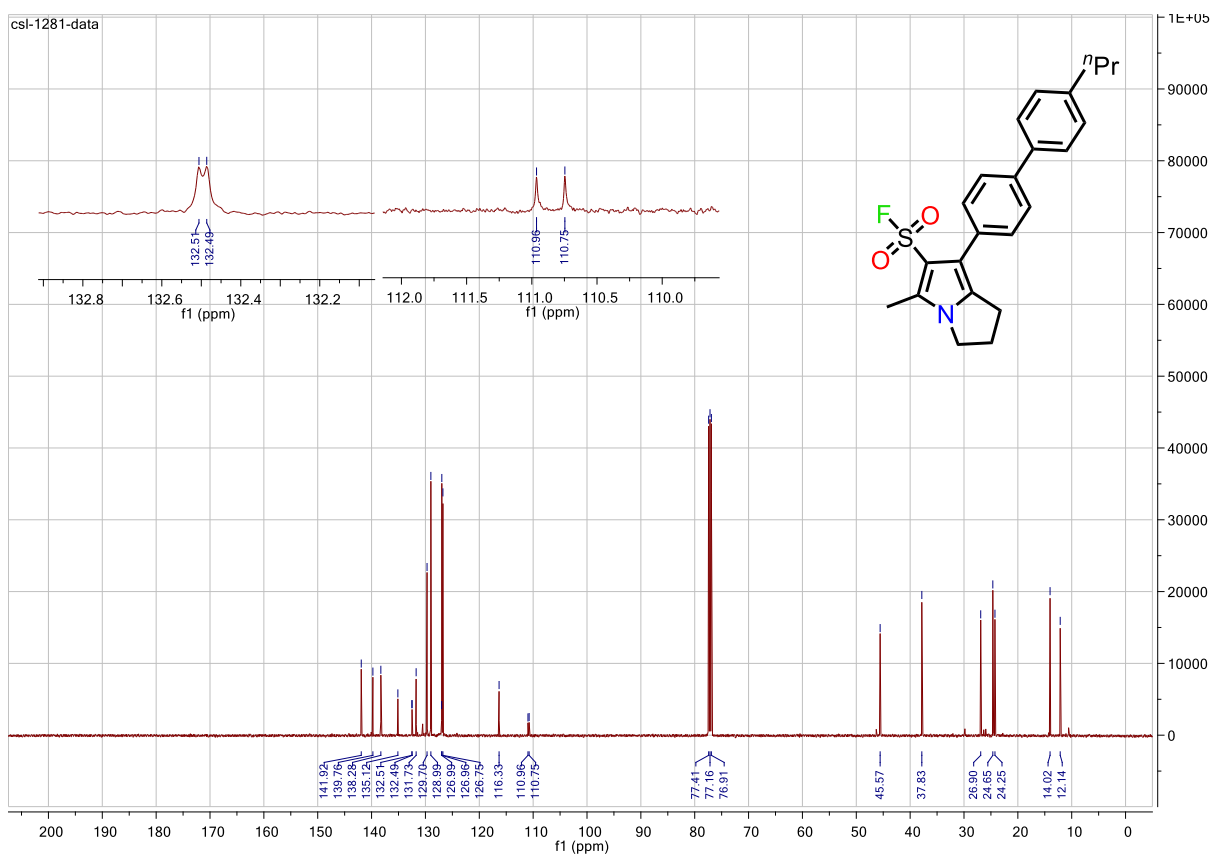
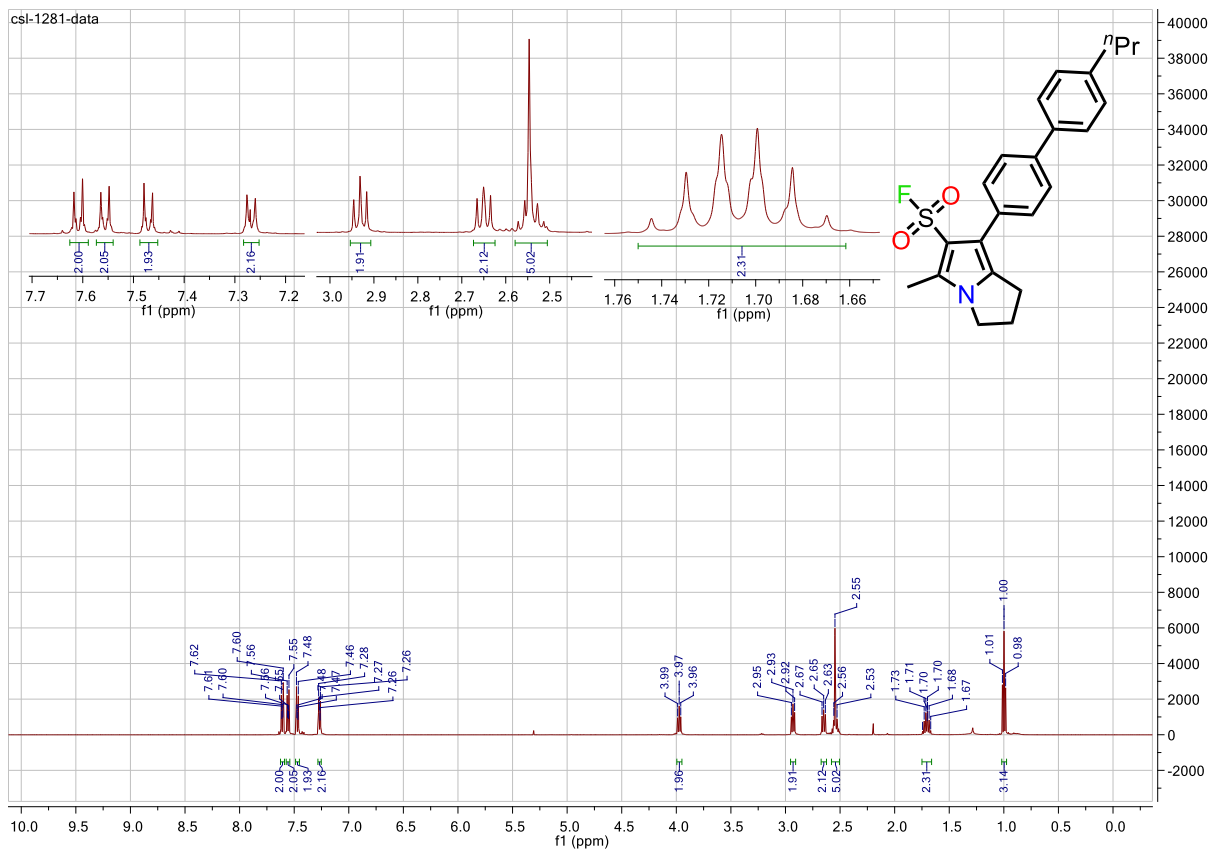


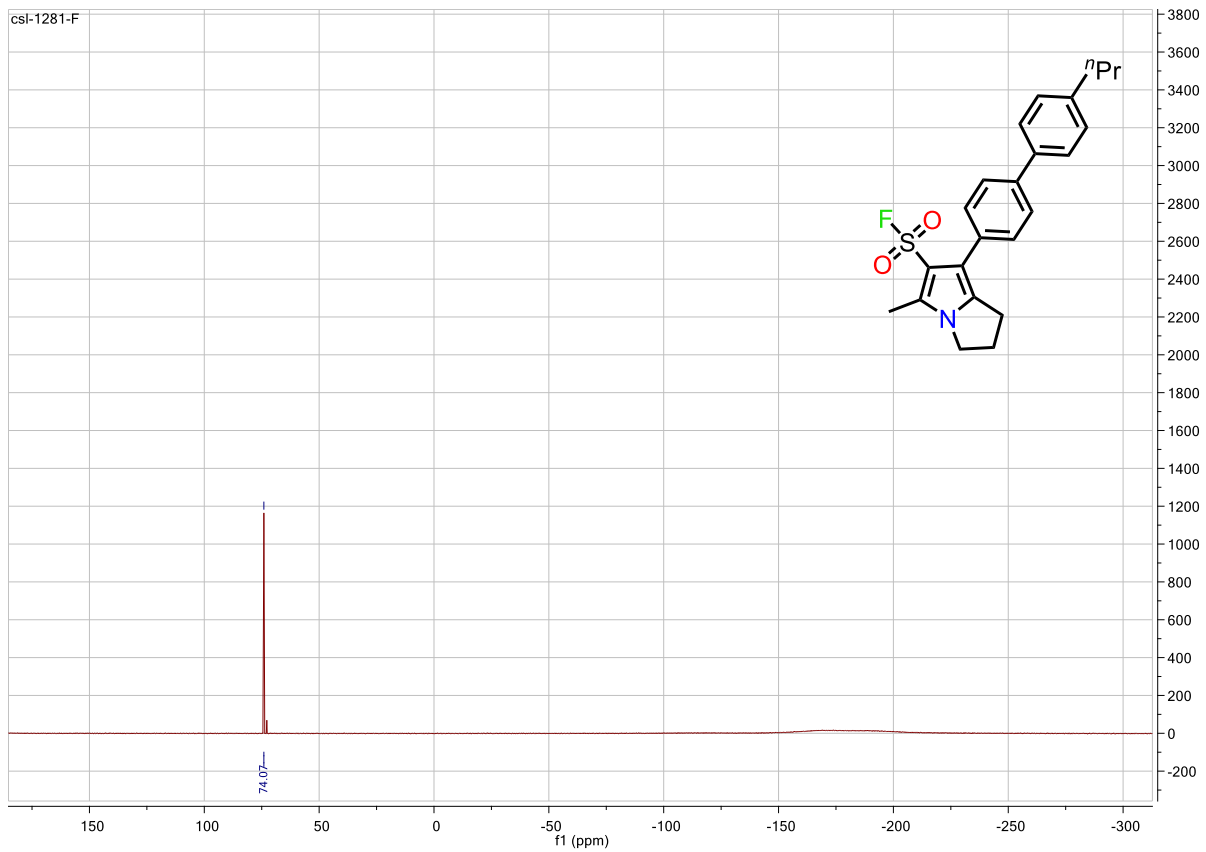


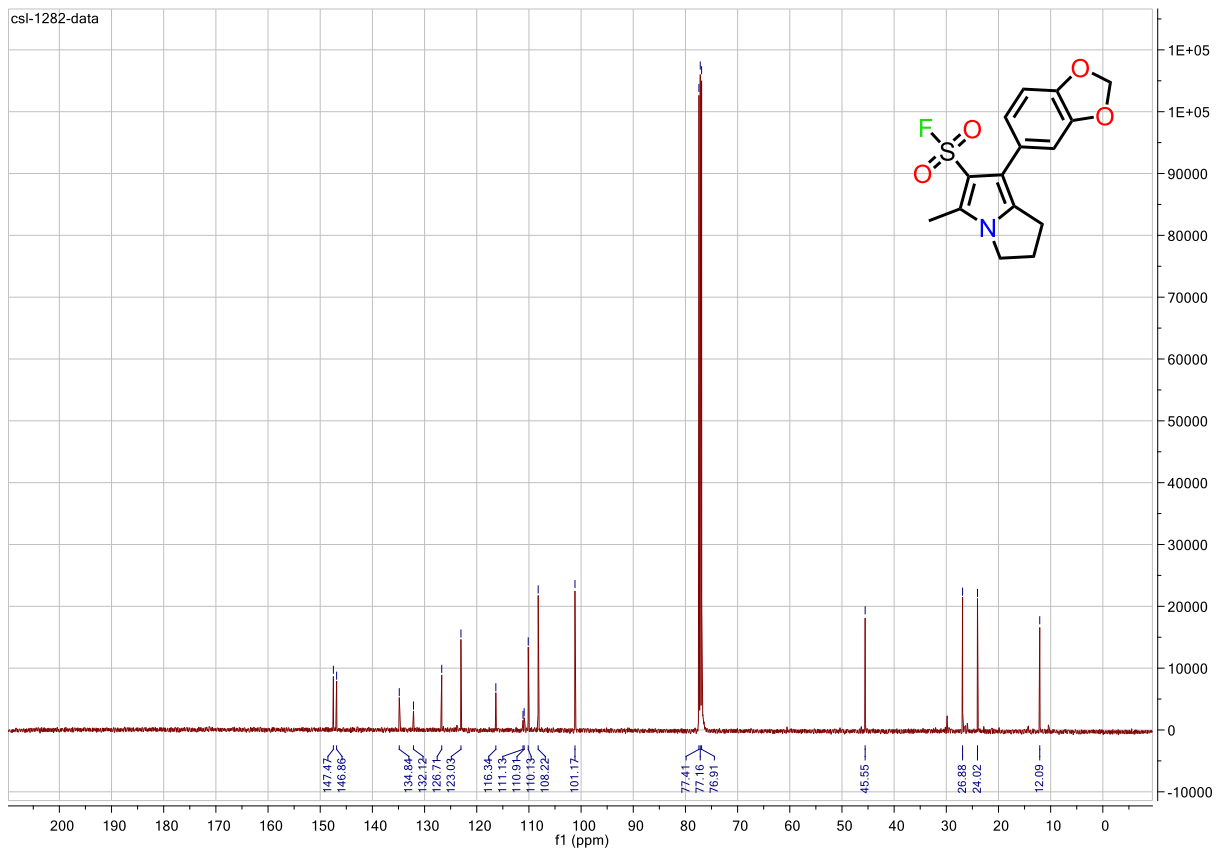
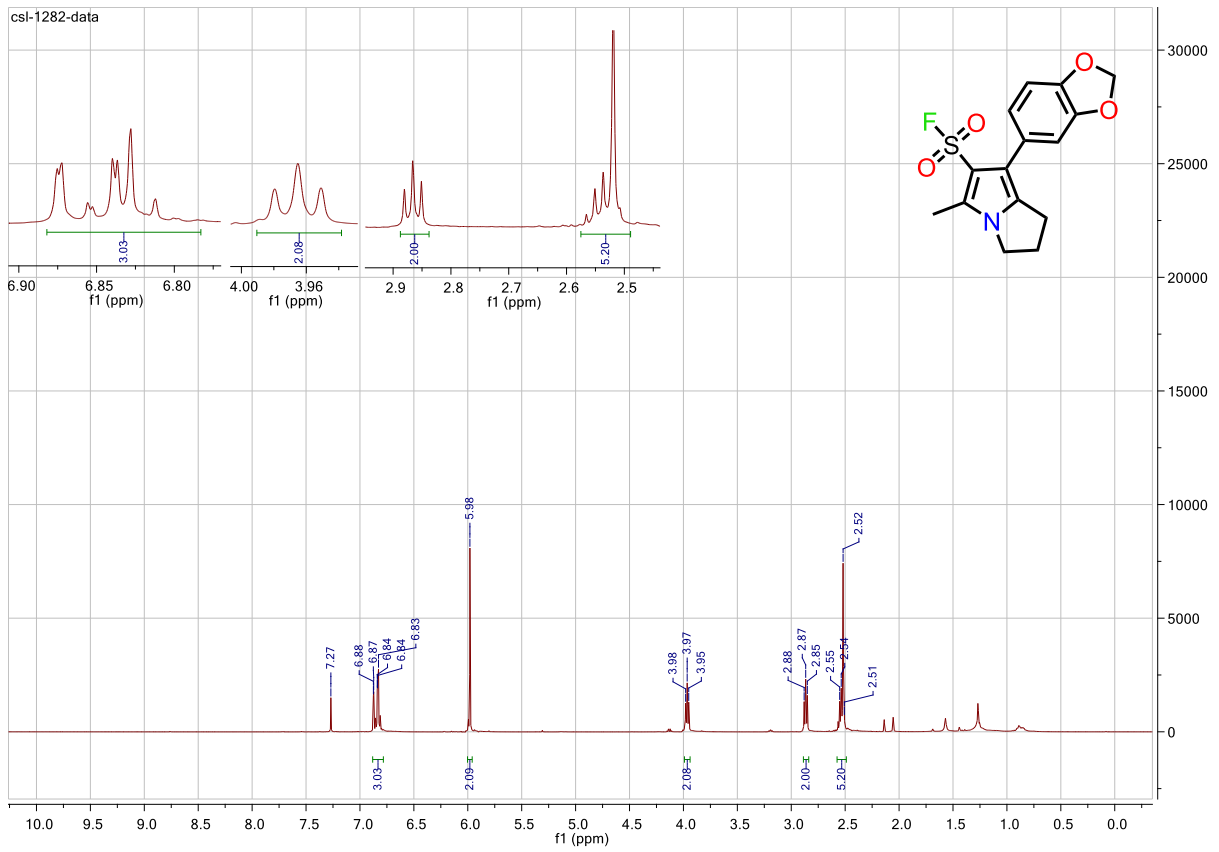


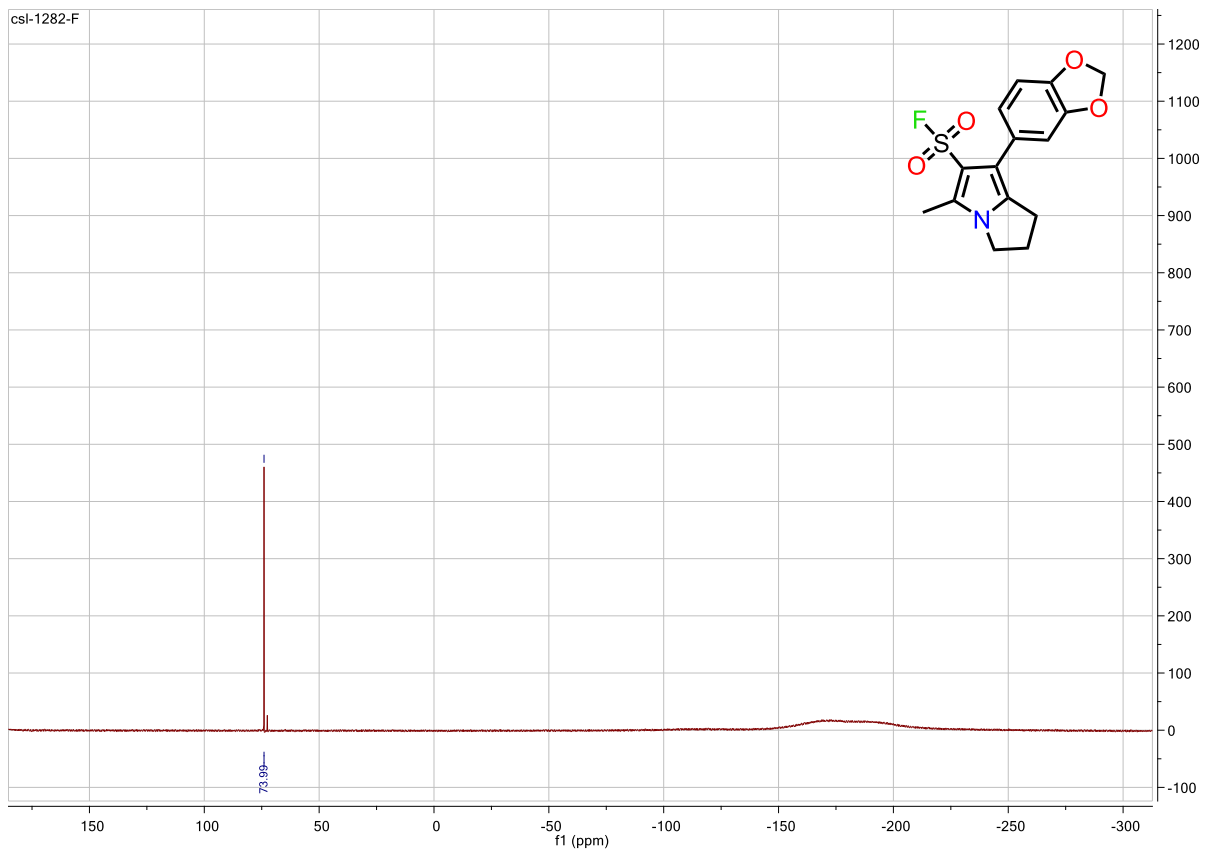


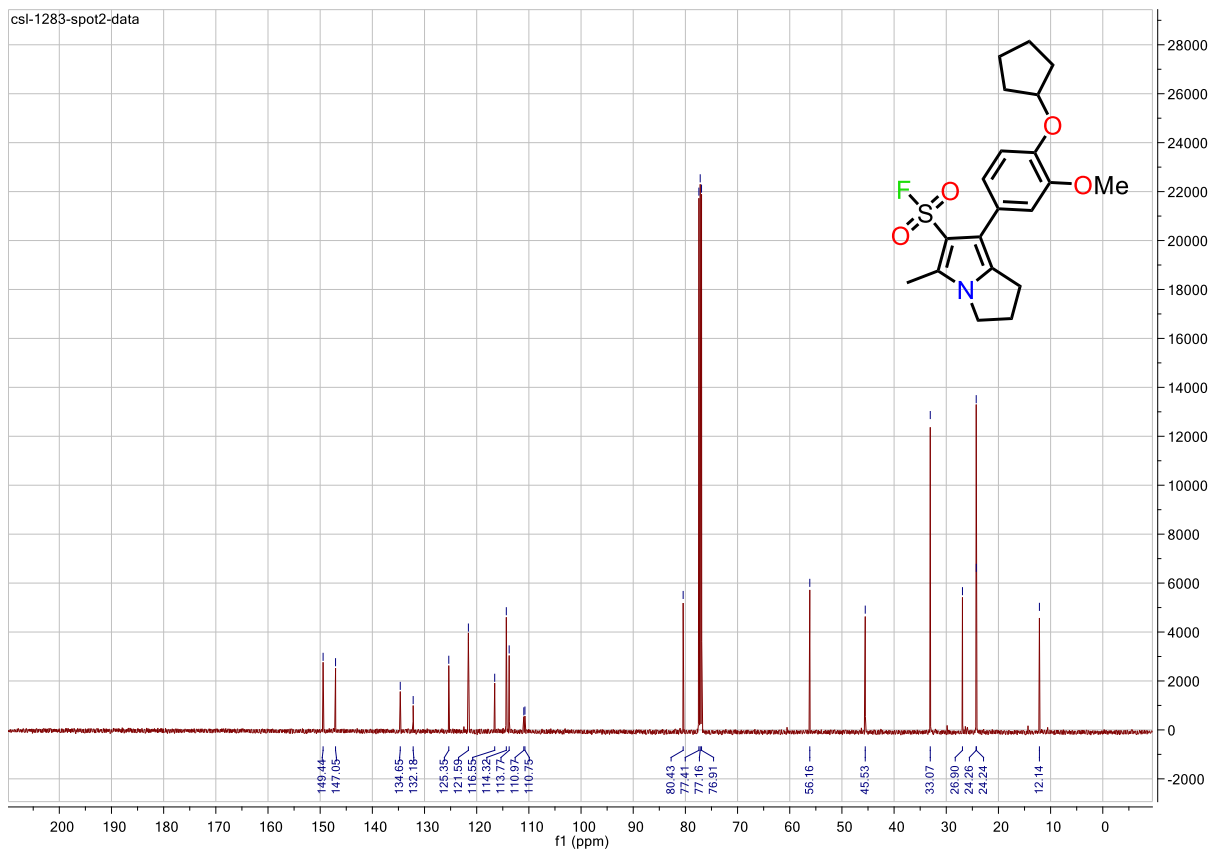
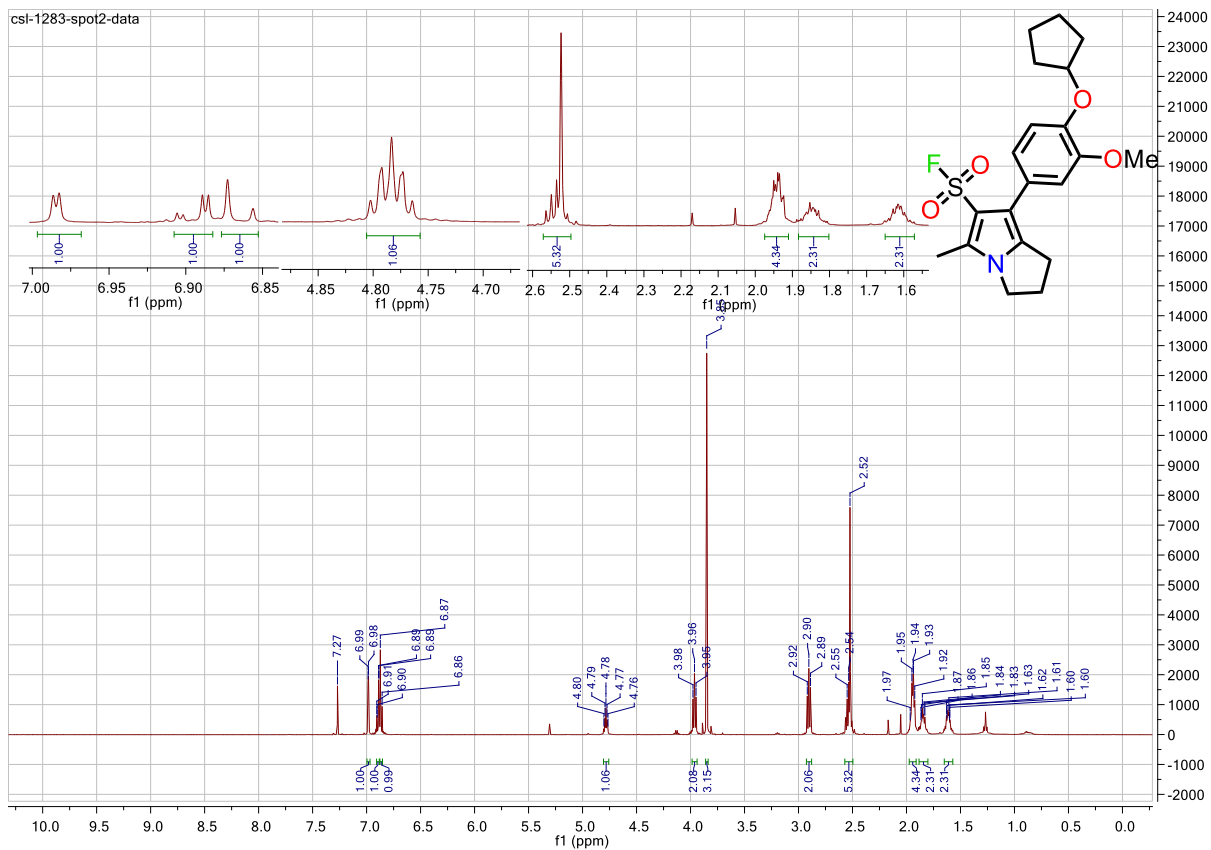


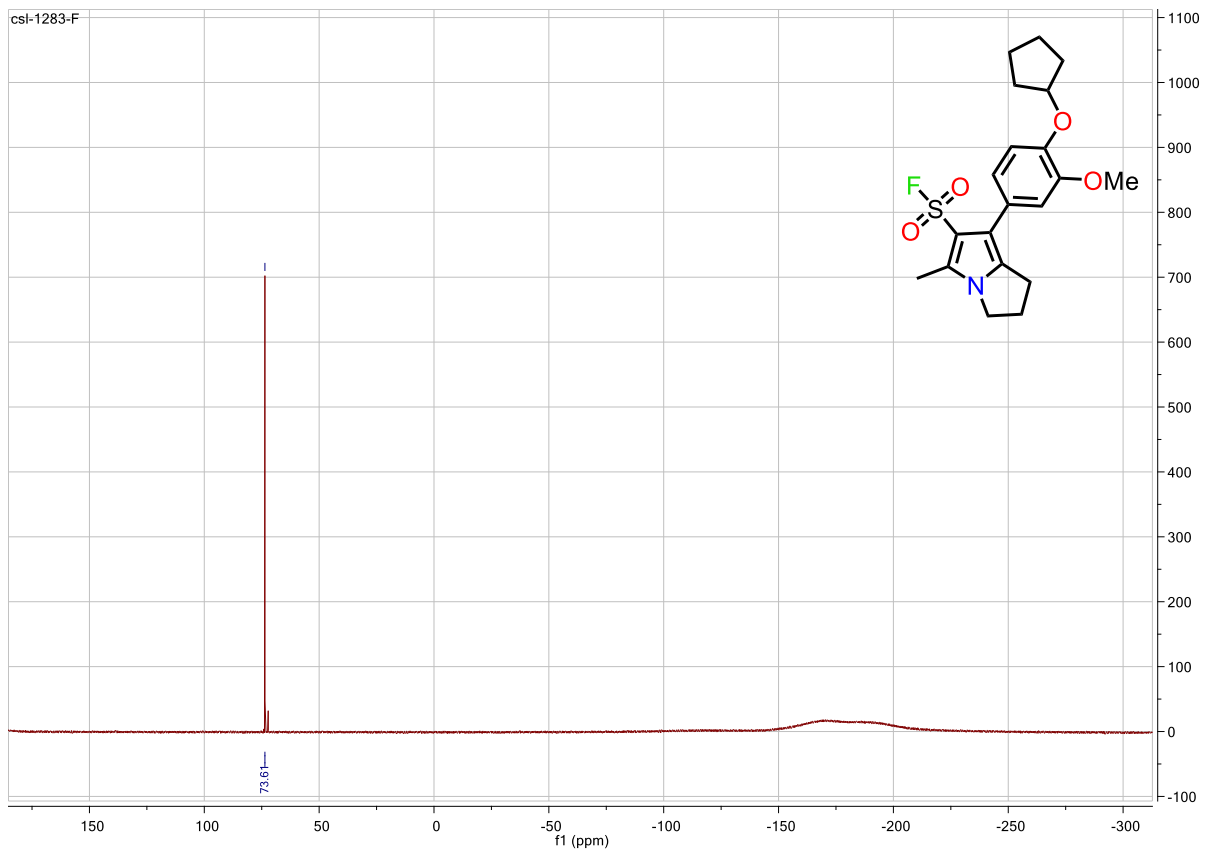


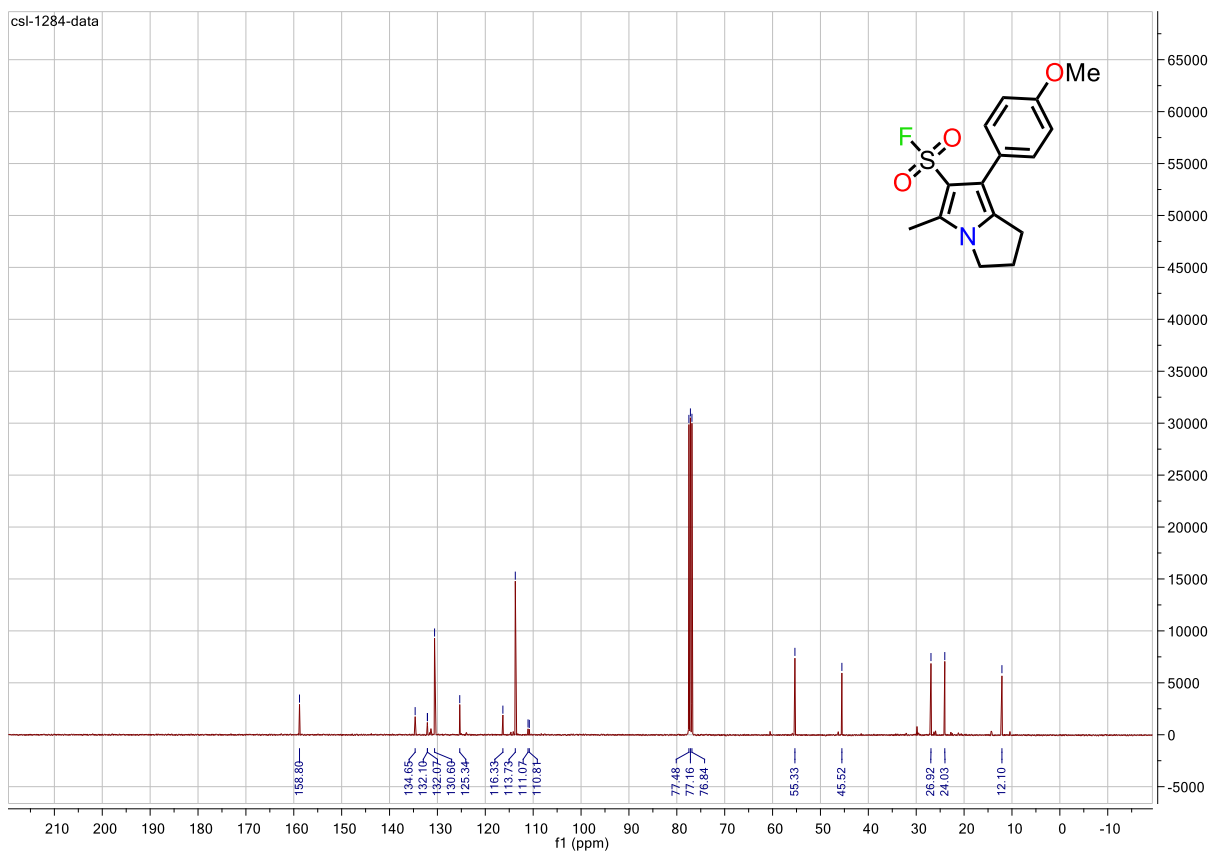
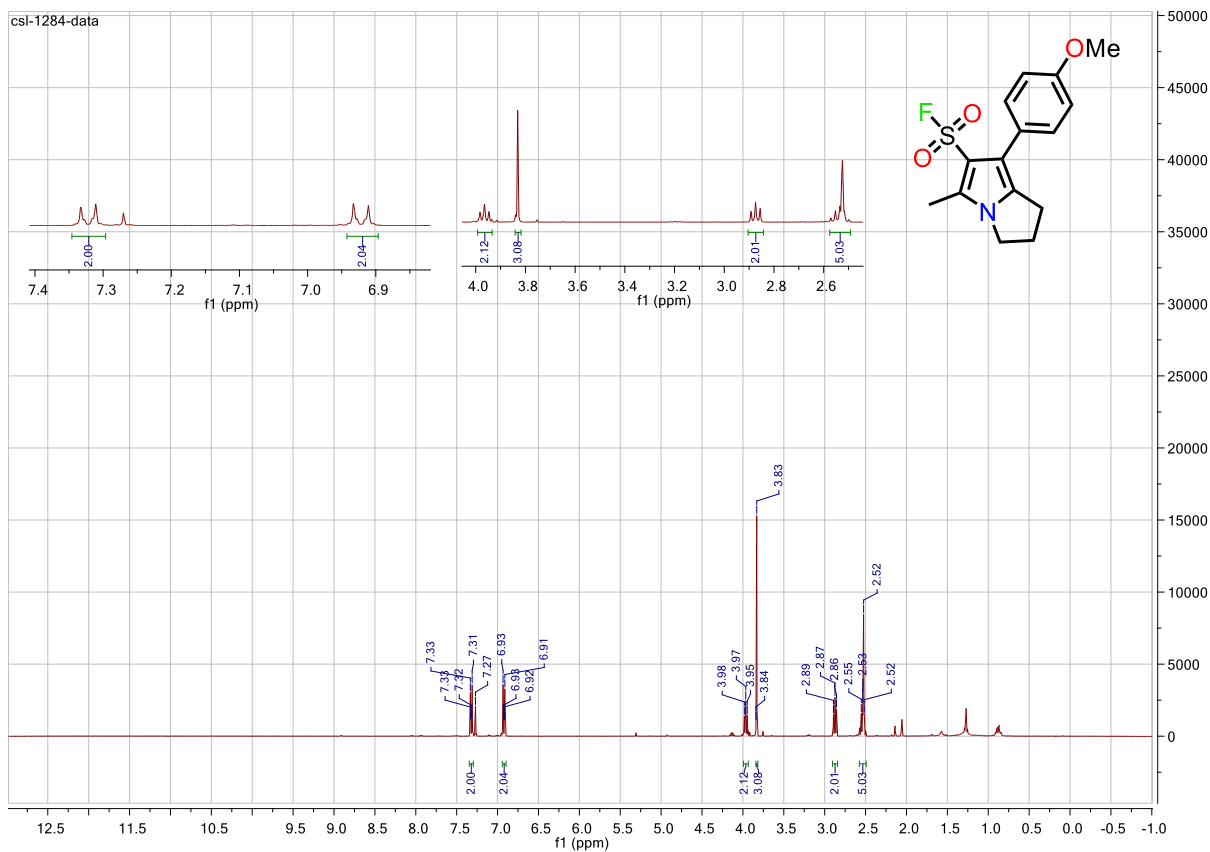


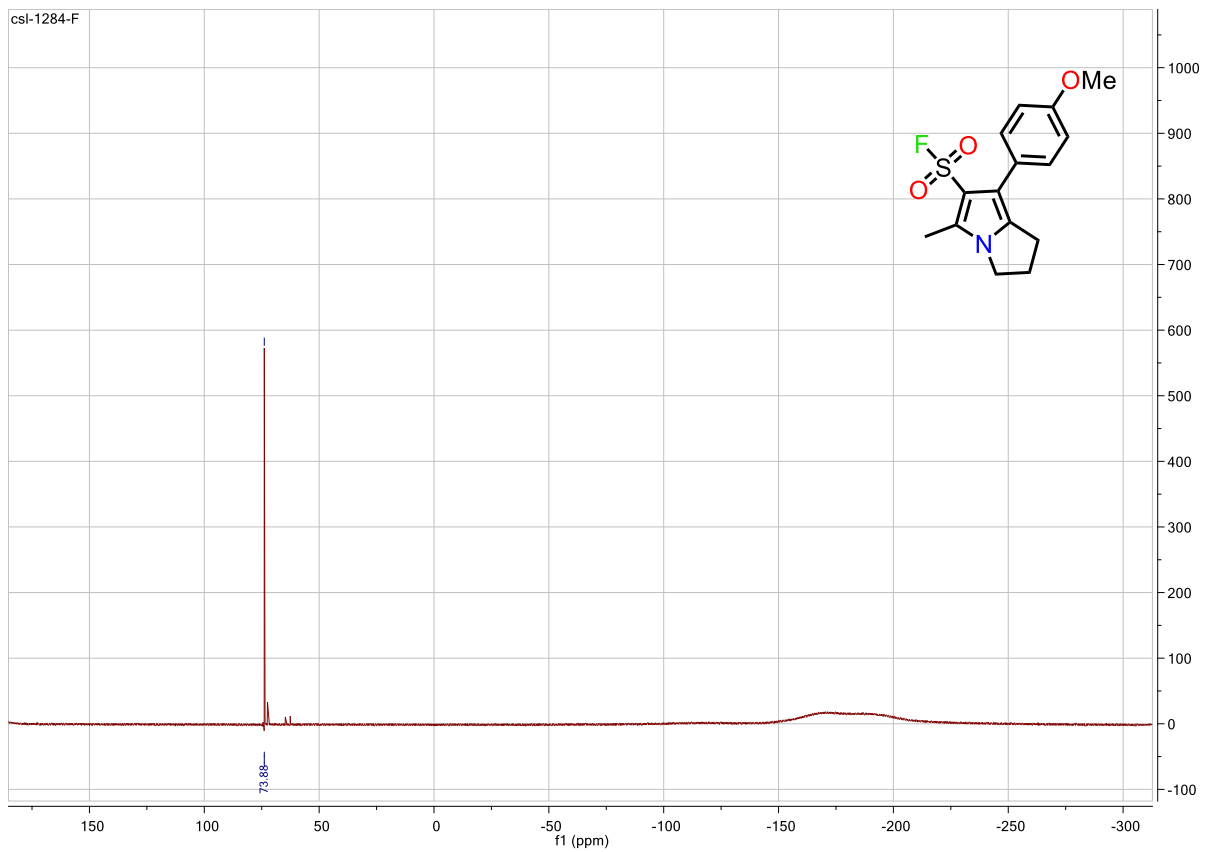


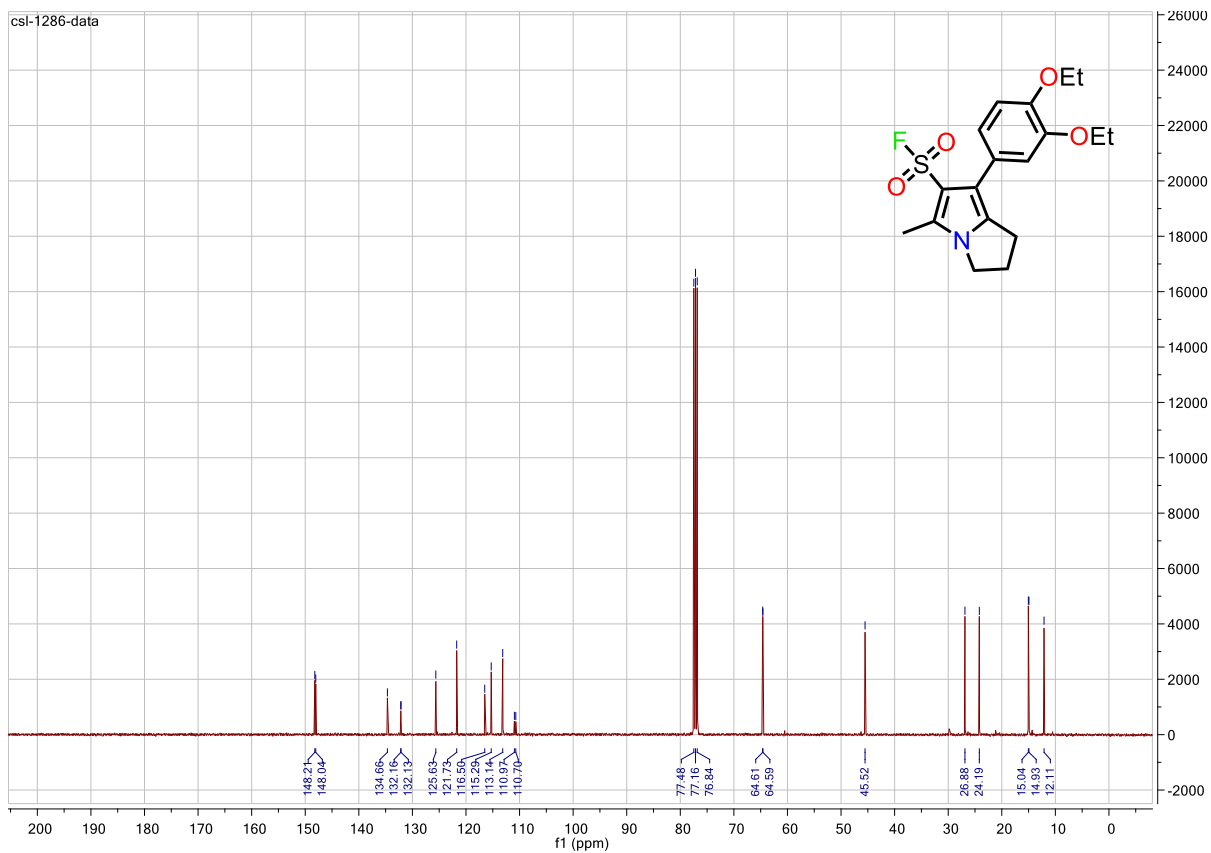
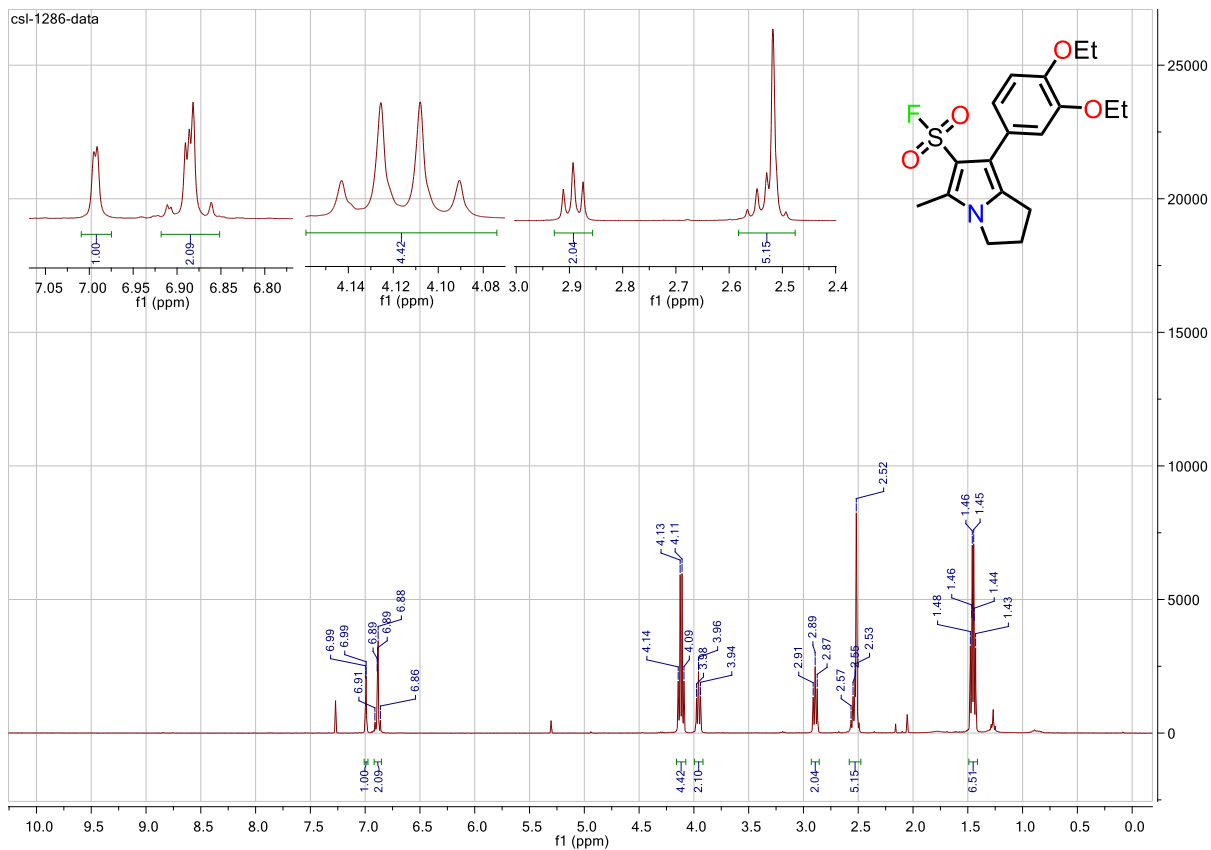


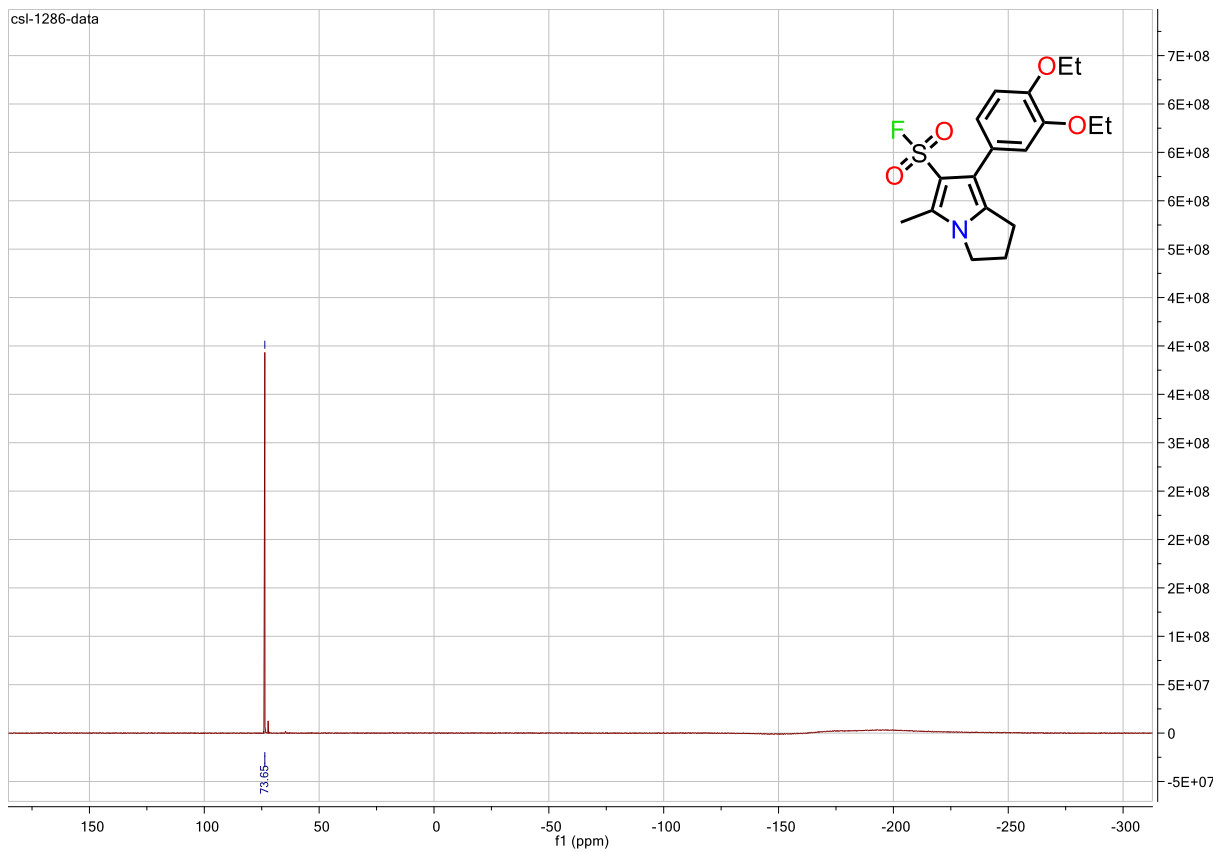


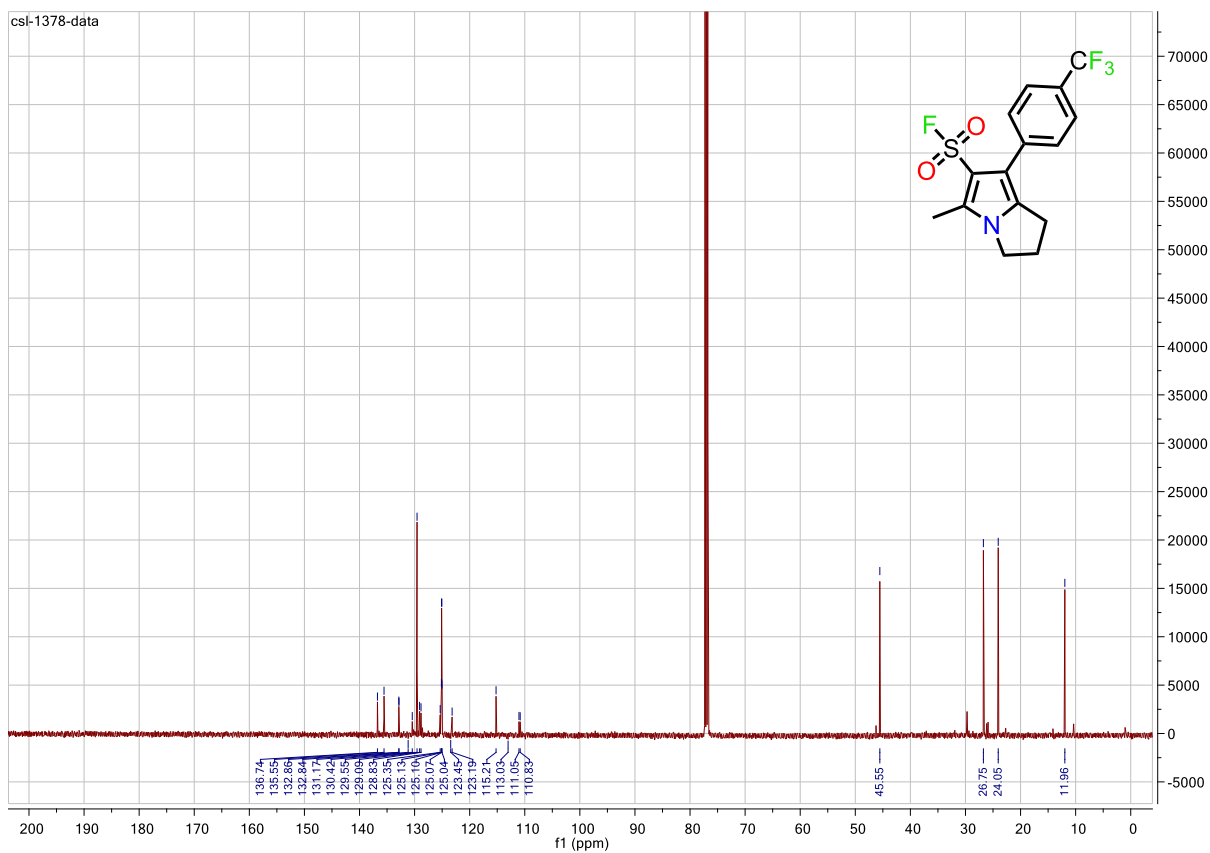
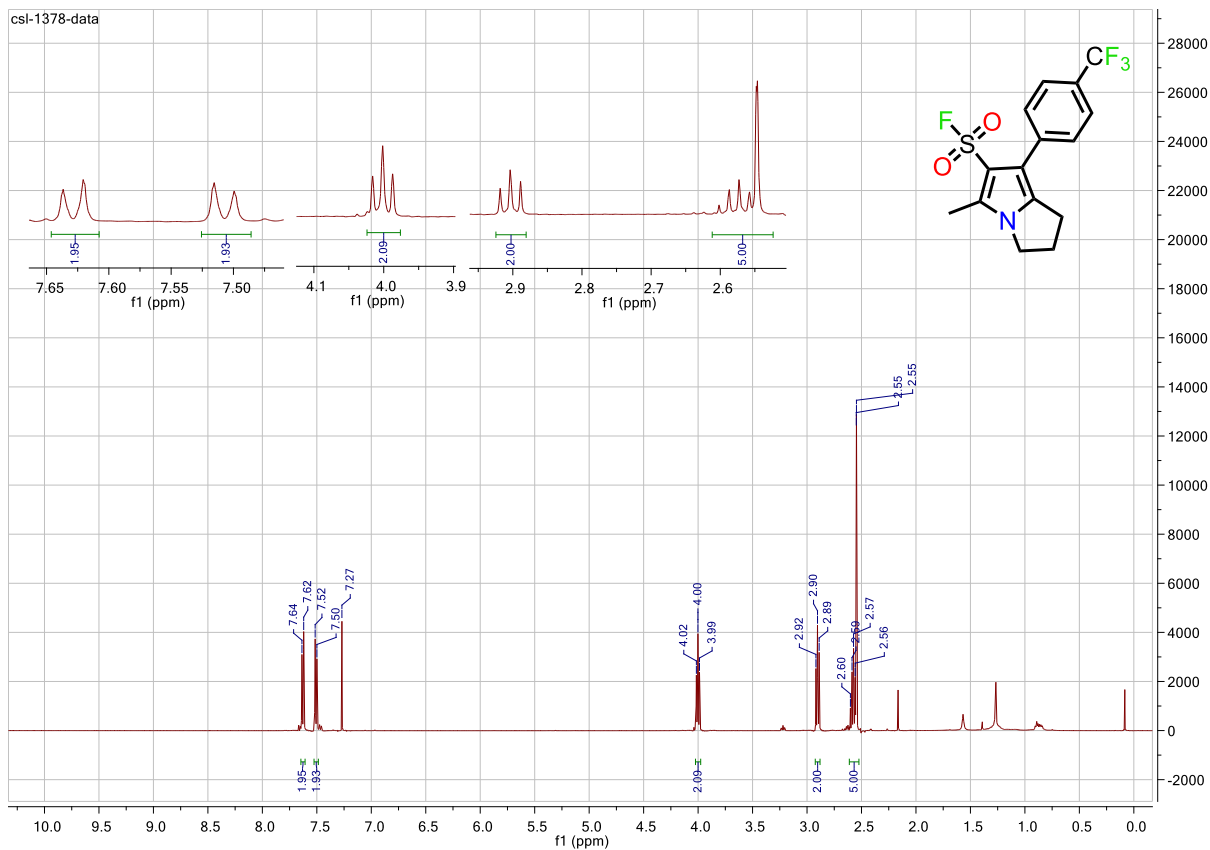


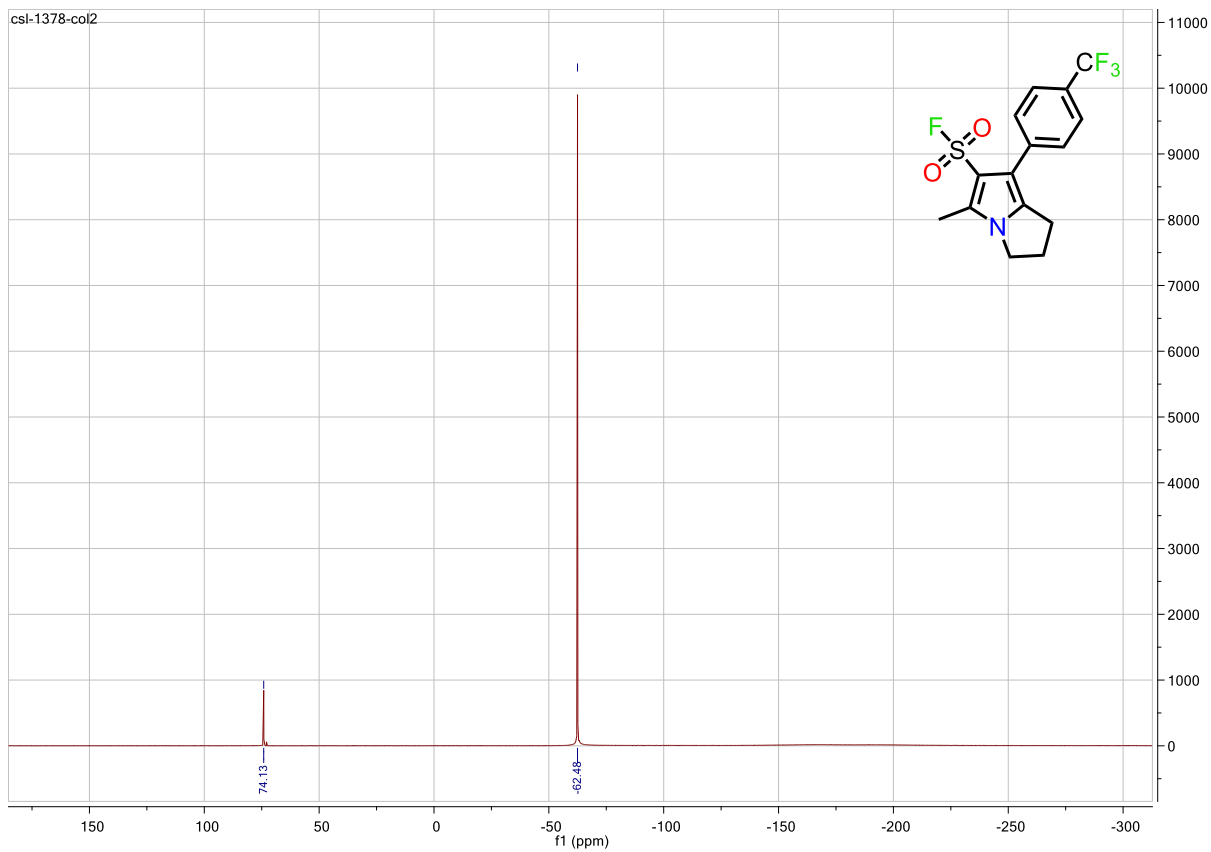


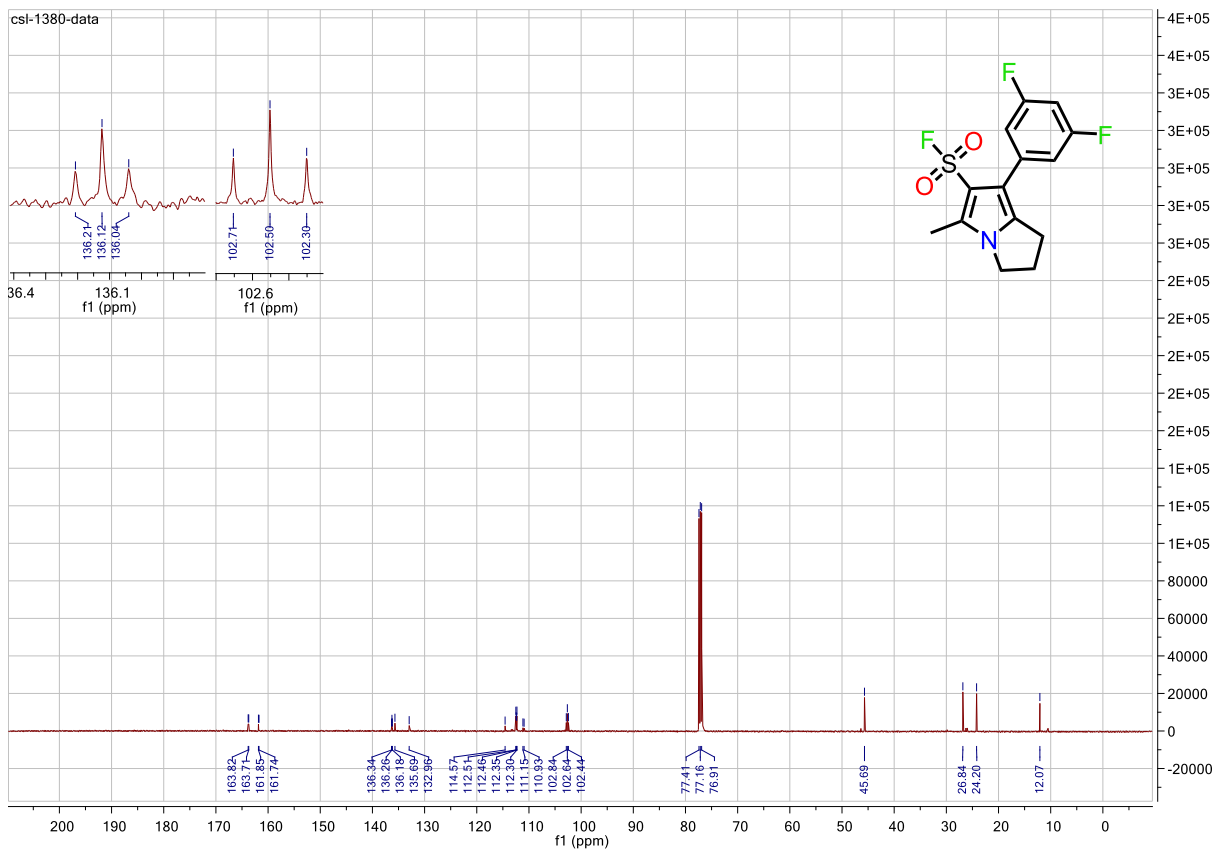
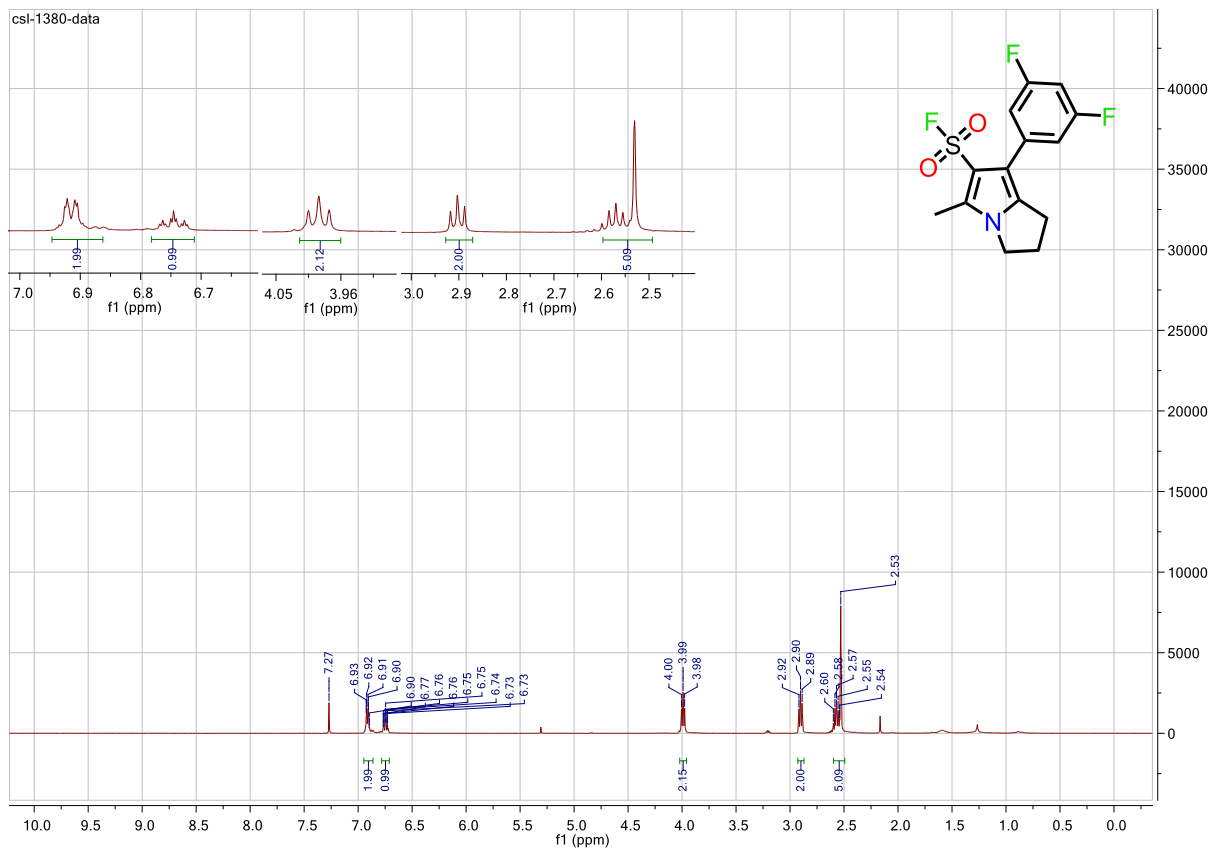


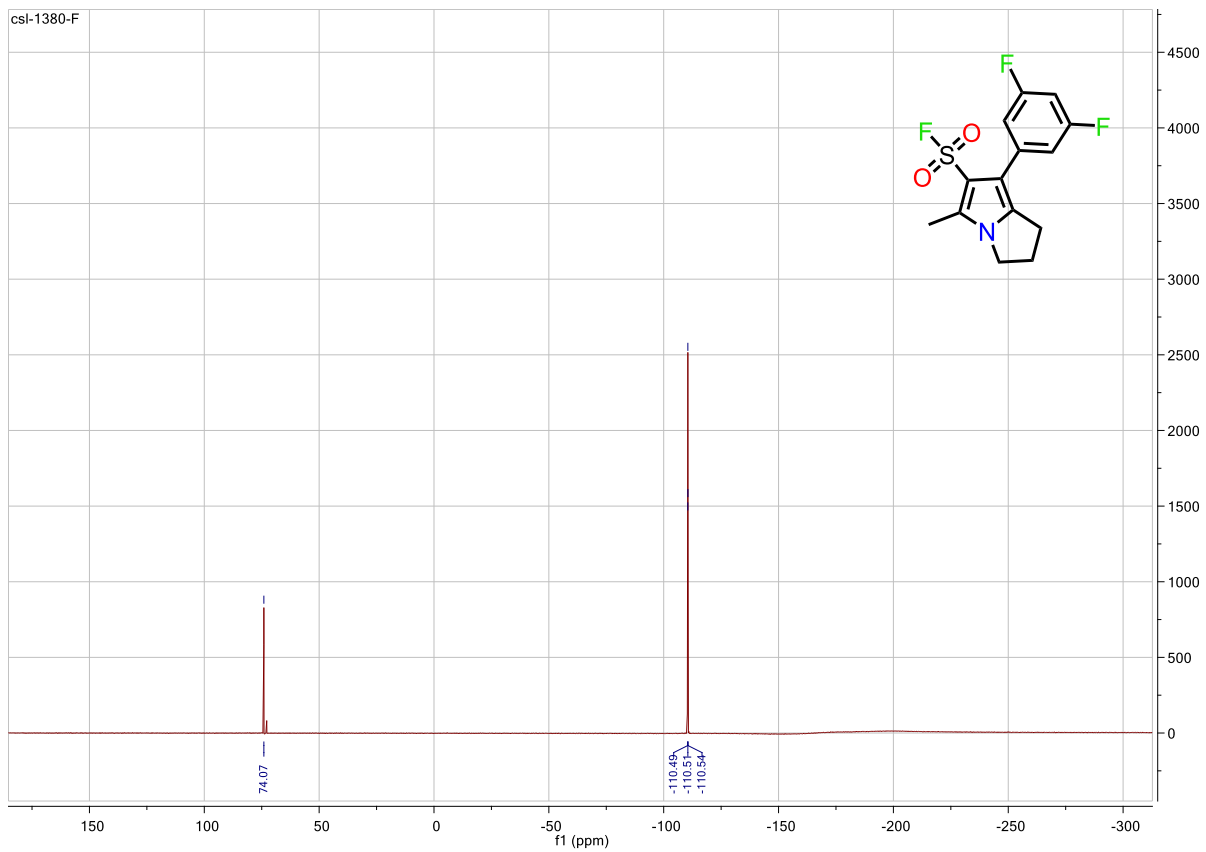


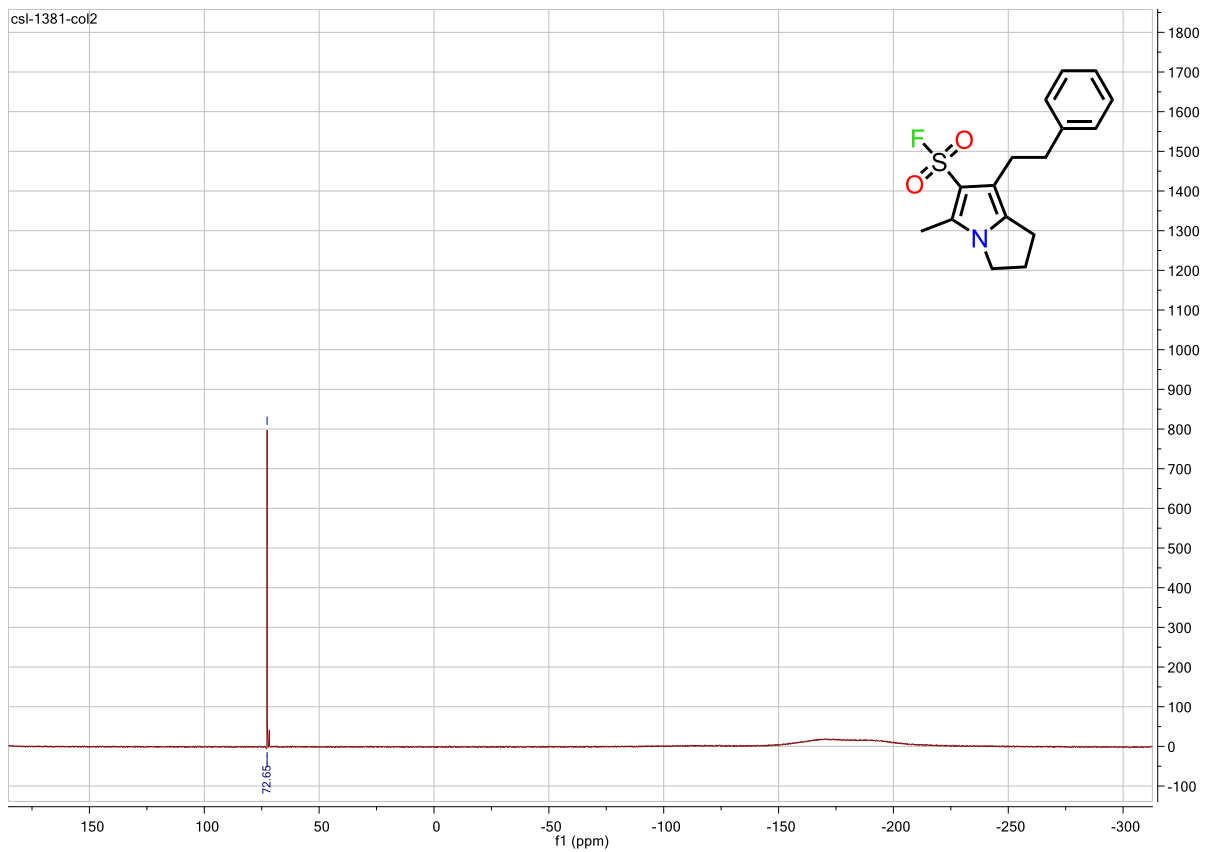


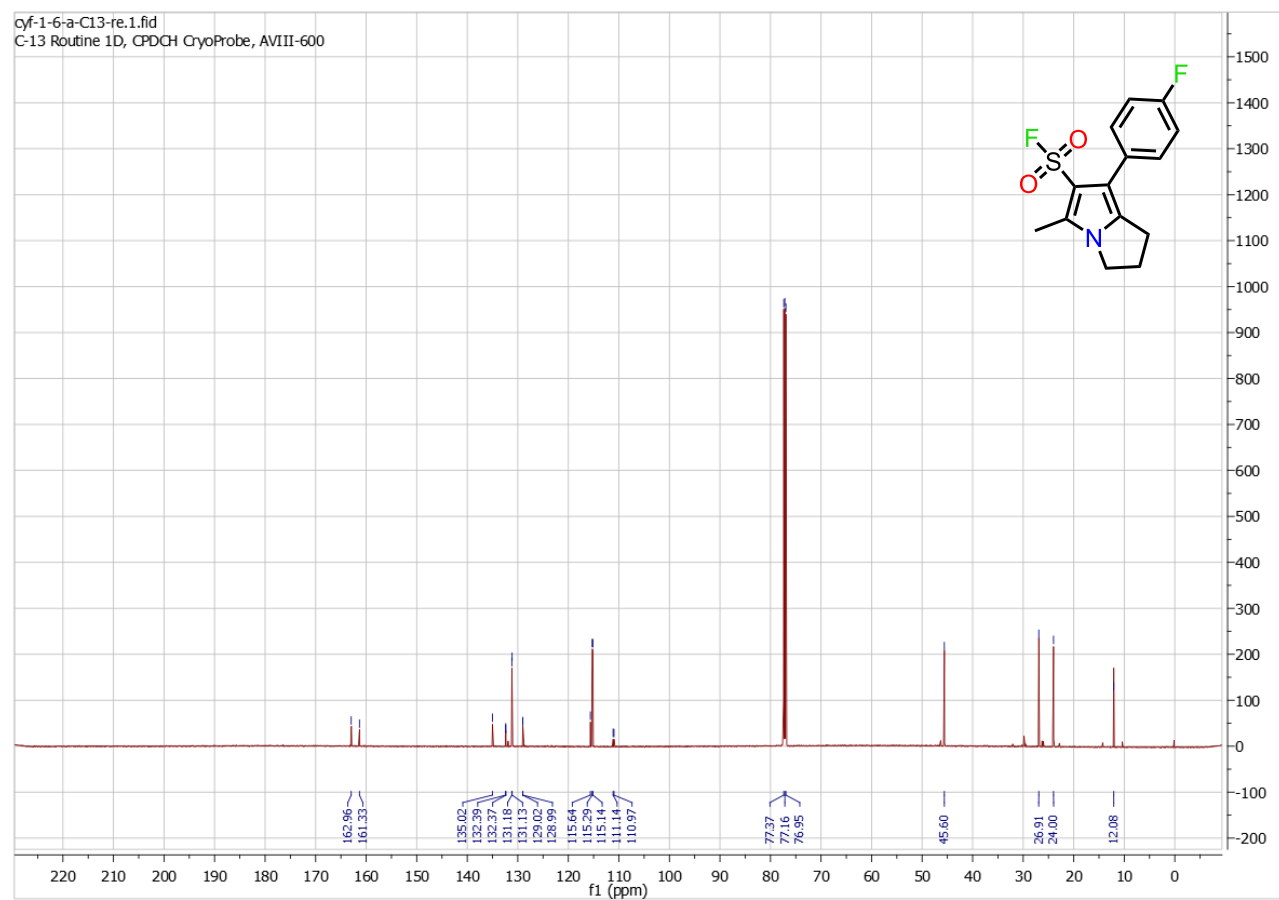
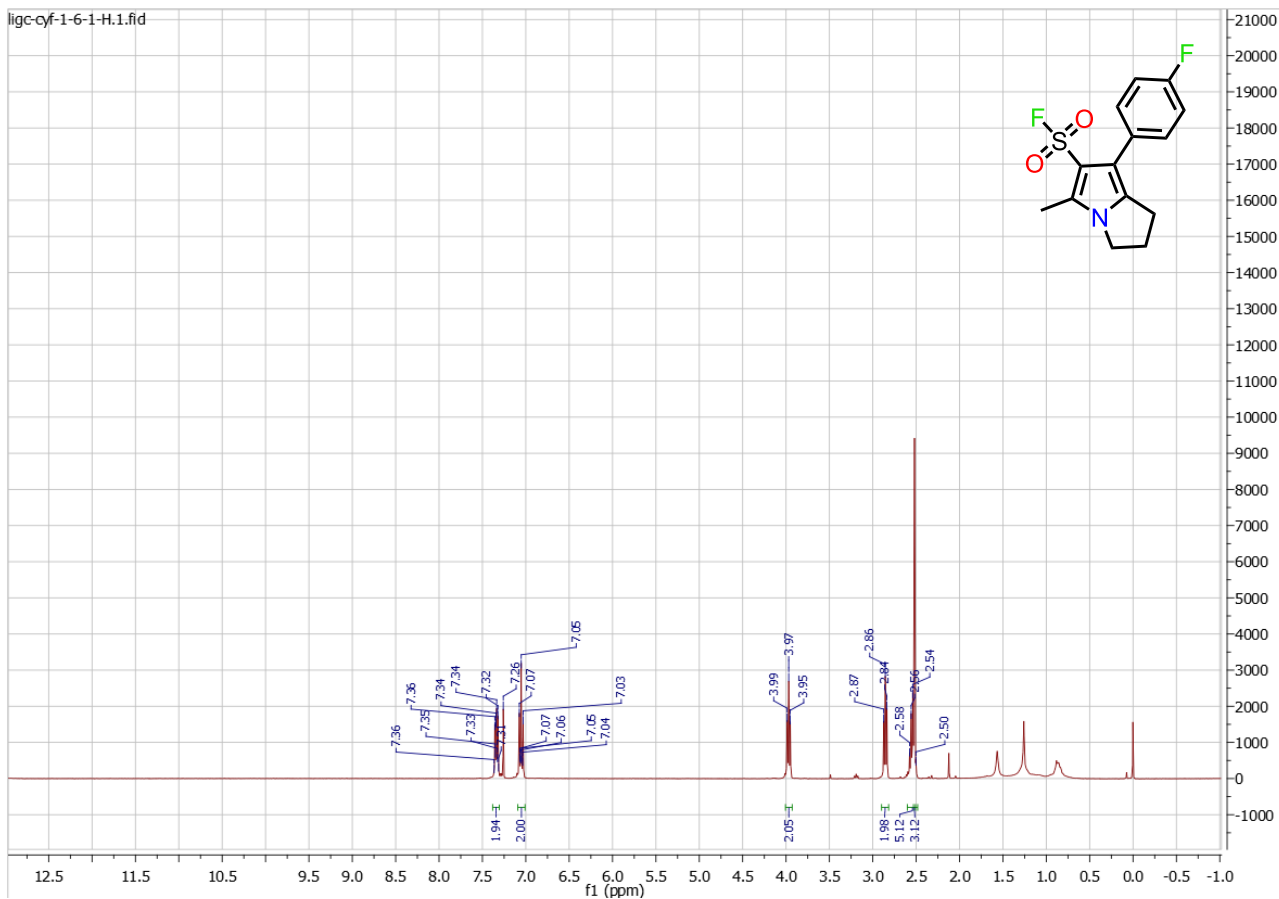




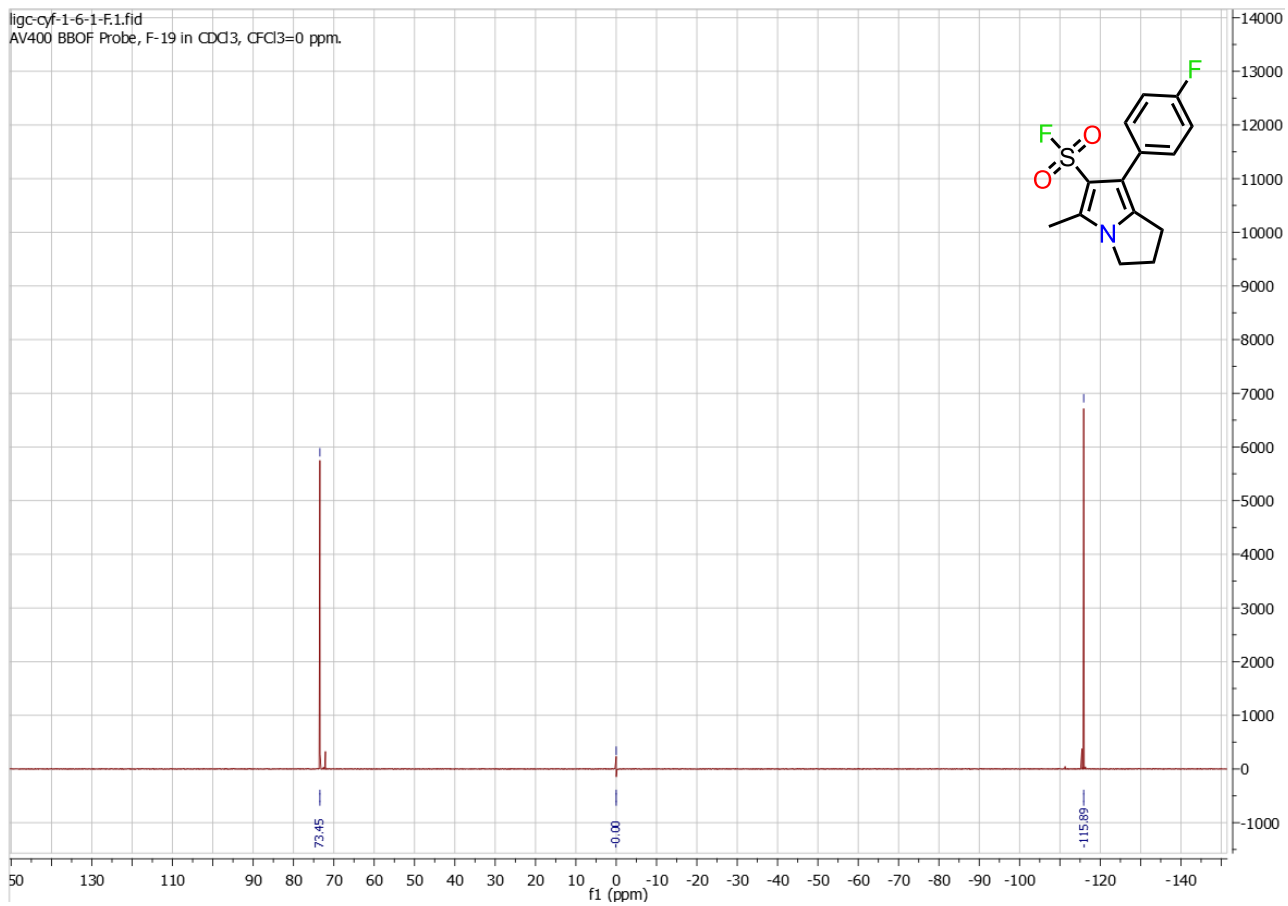


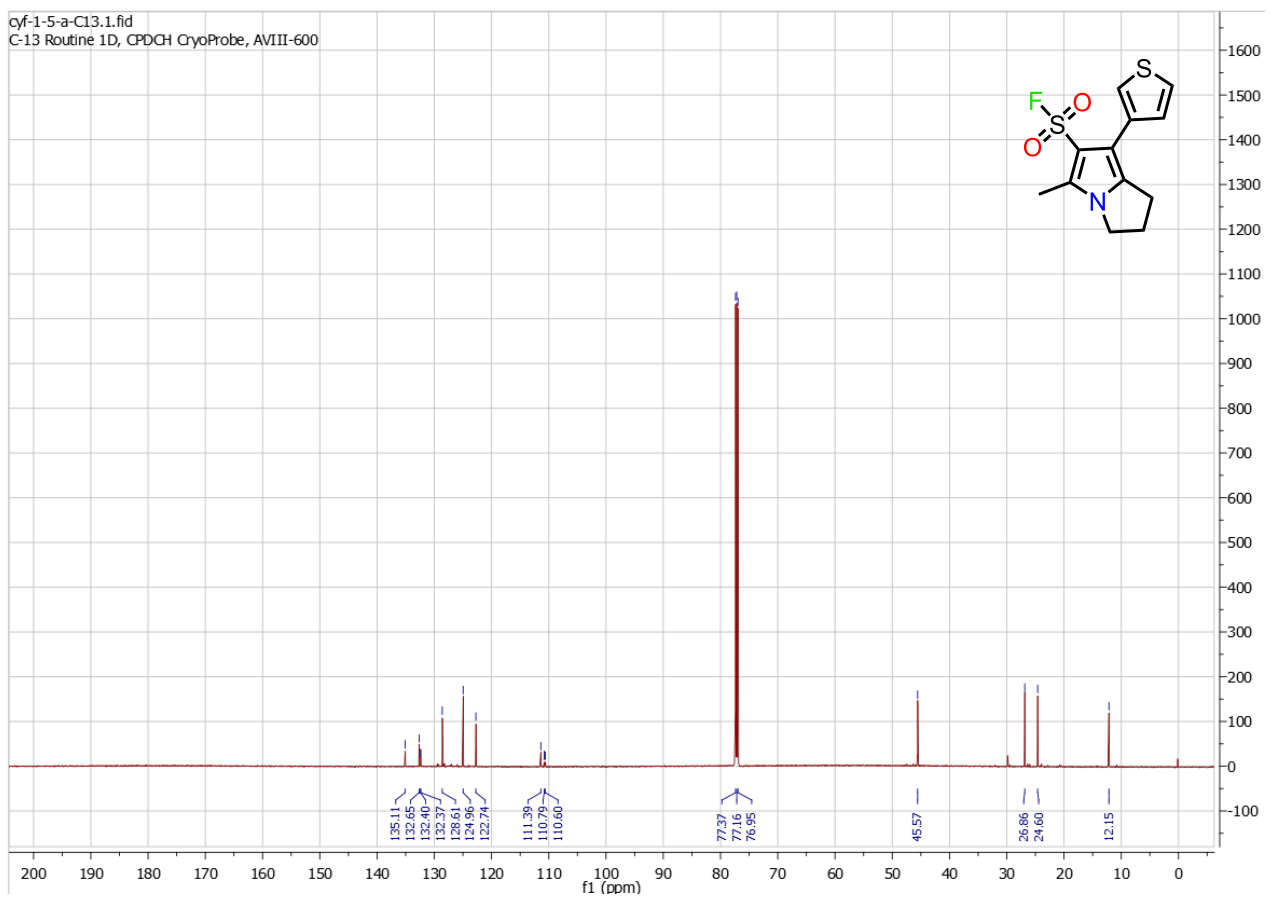
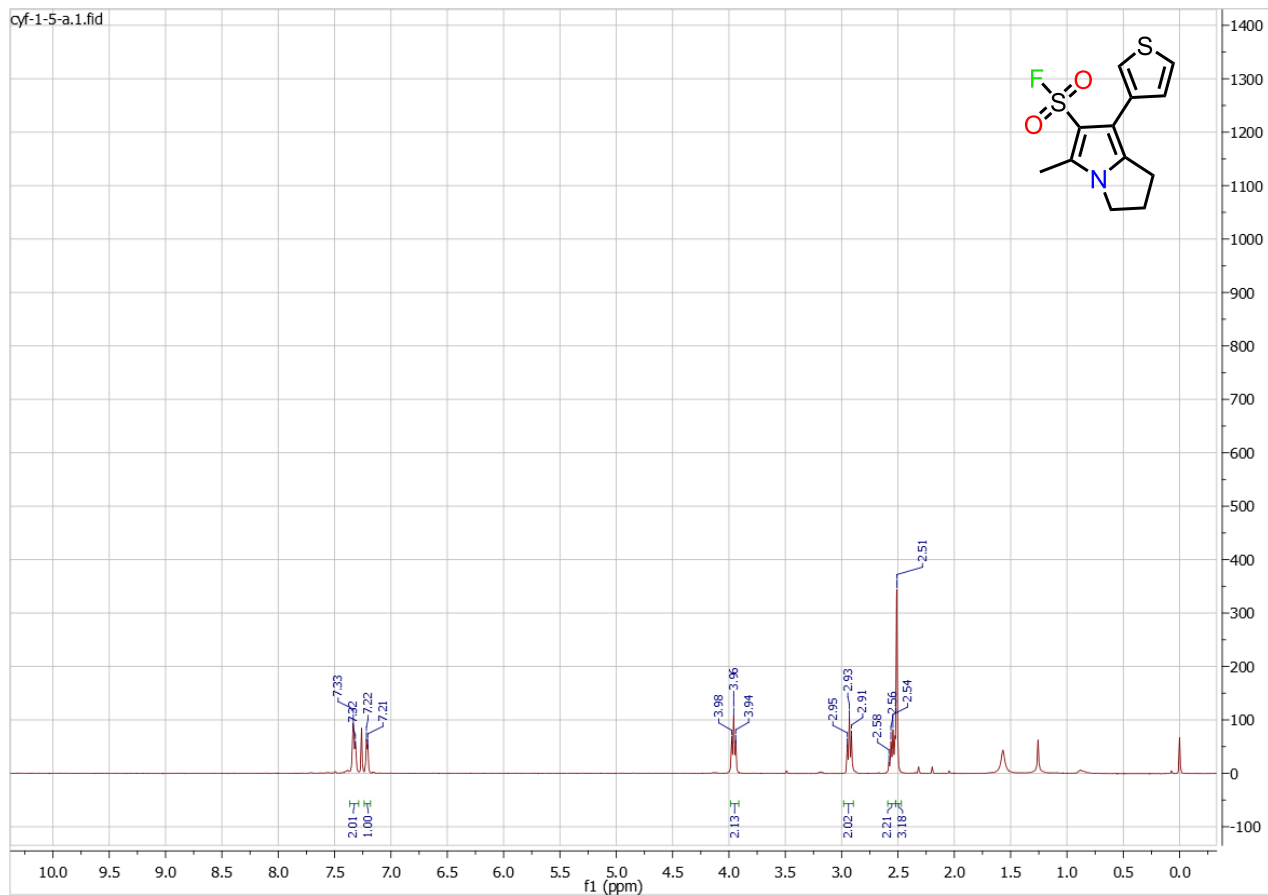




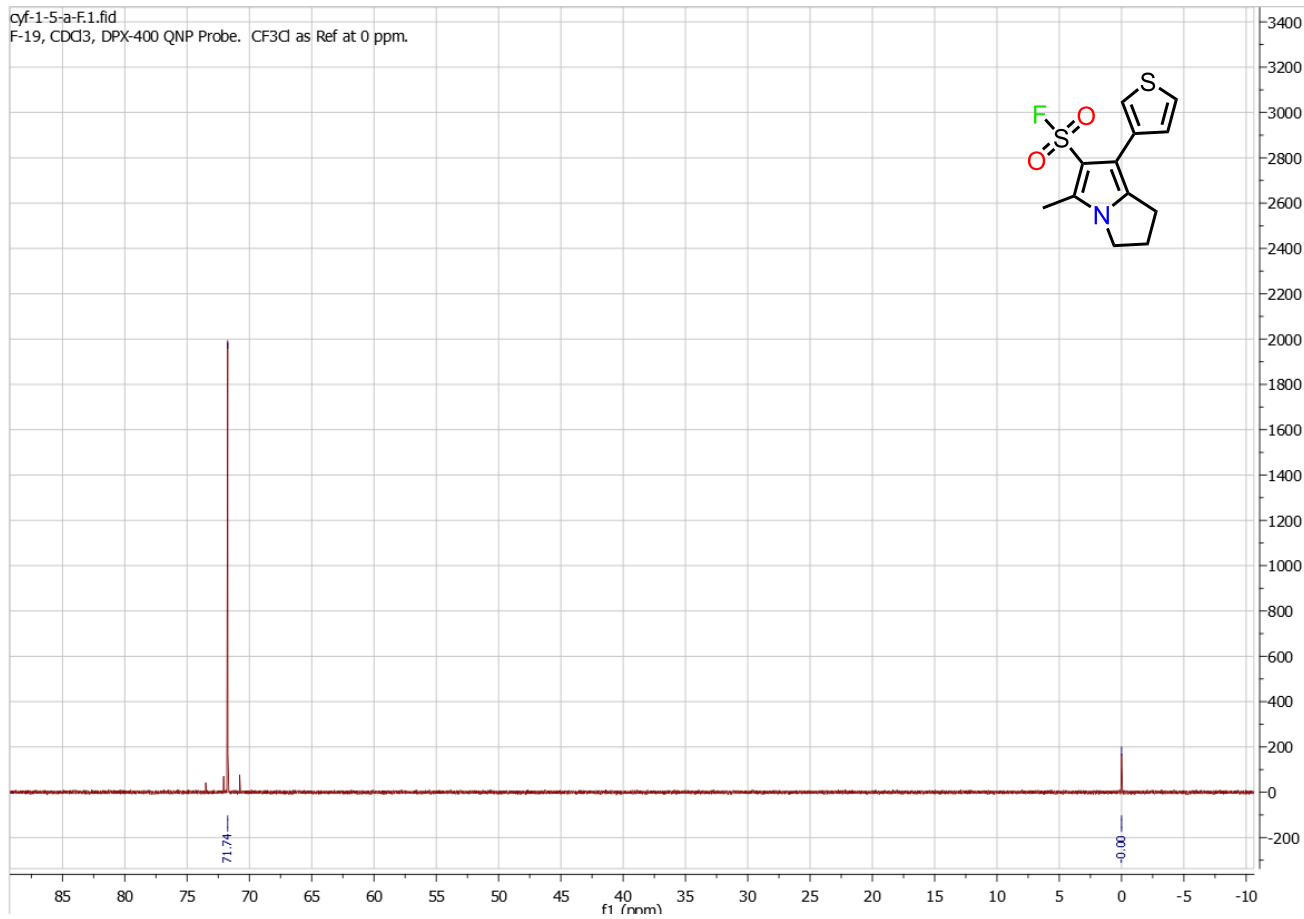


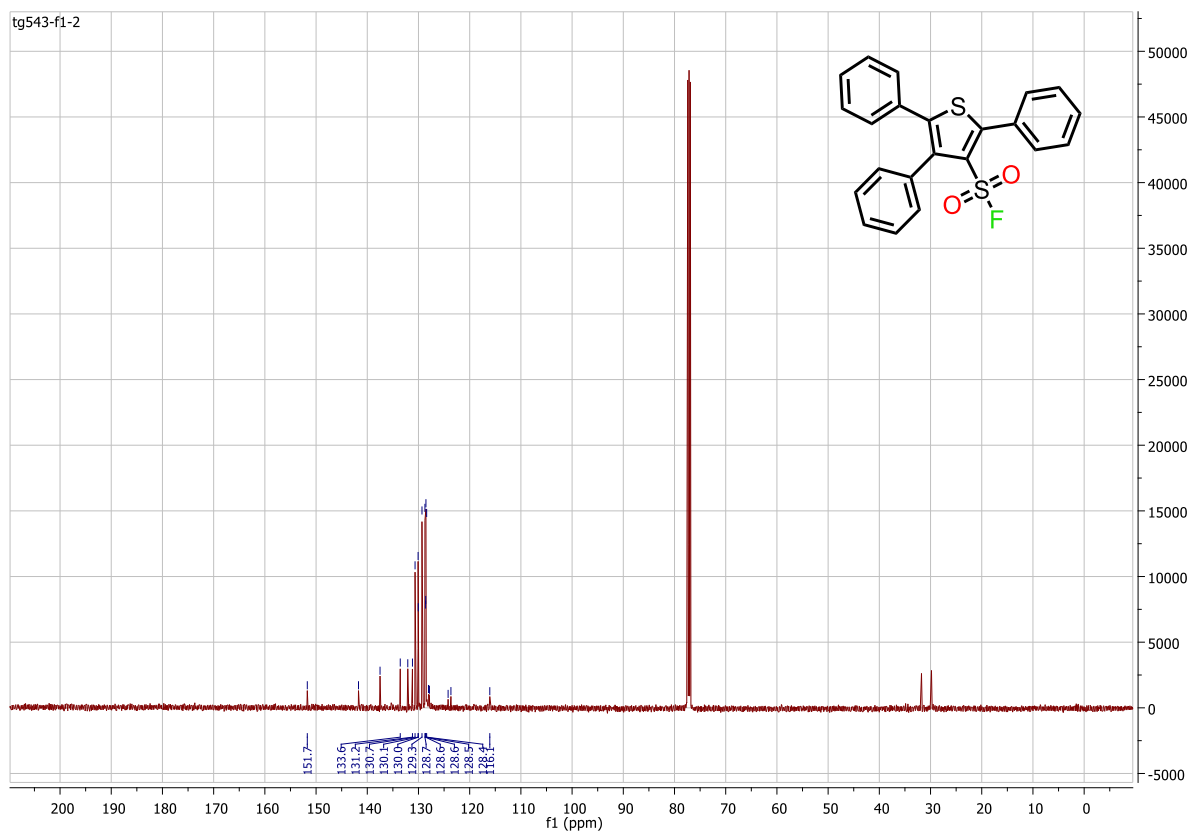
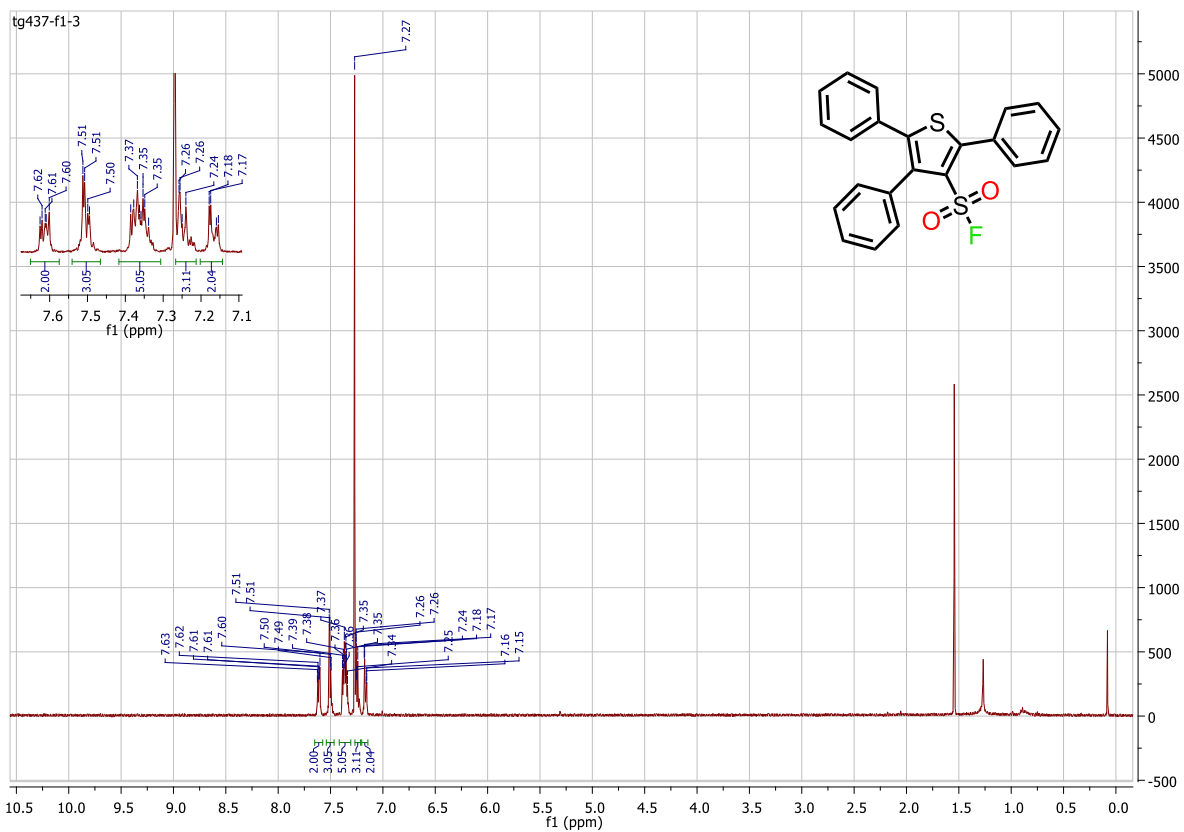
lig-cyf-1-6-1-F.1.fid
AV400 BBOF Probe, F-19 in CDCl₃, CFC13=0 ppm.



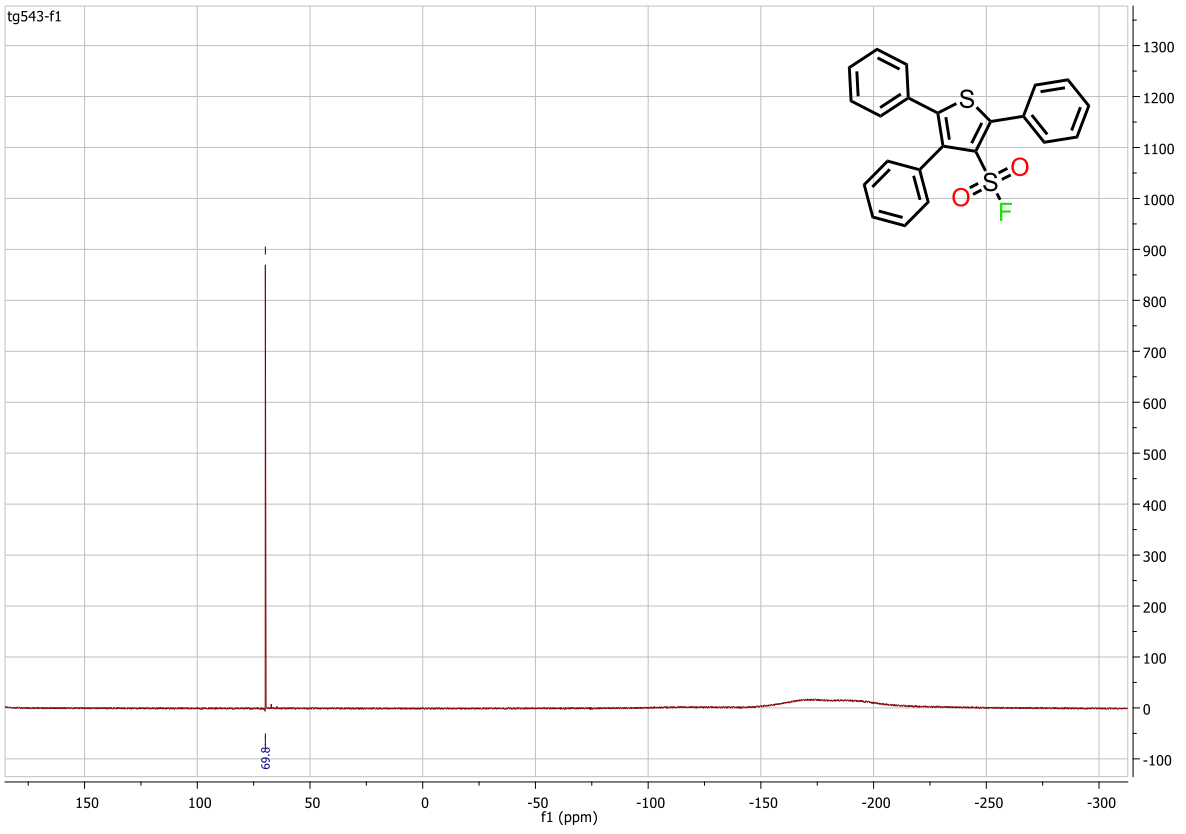


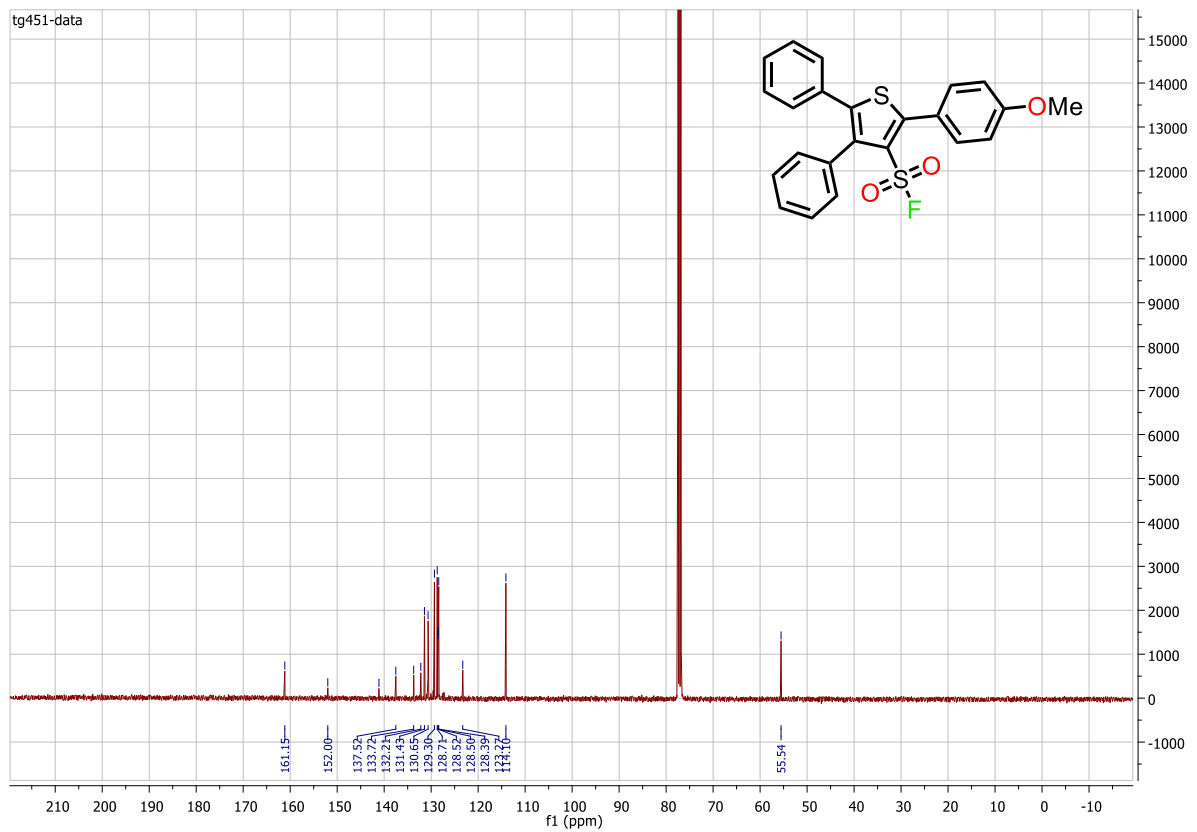
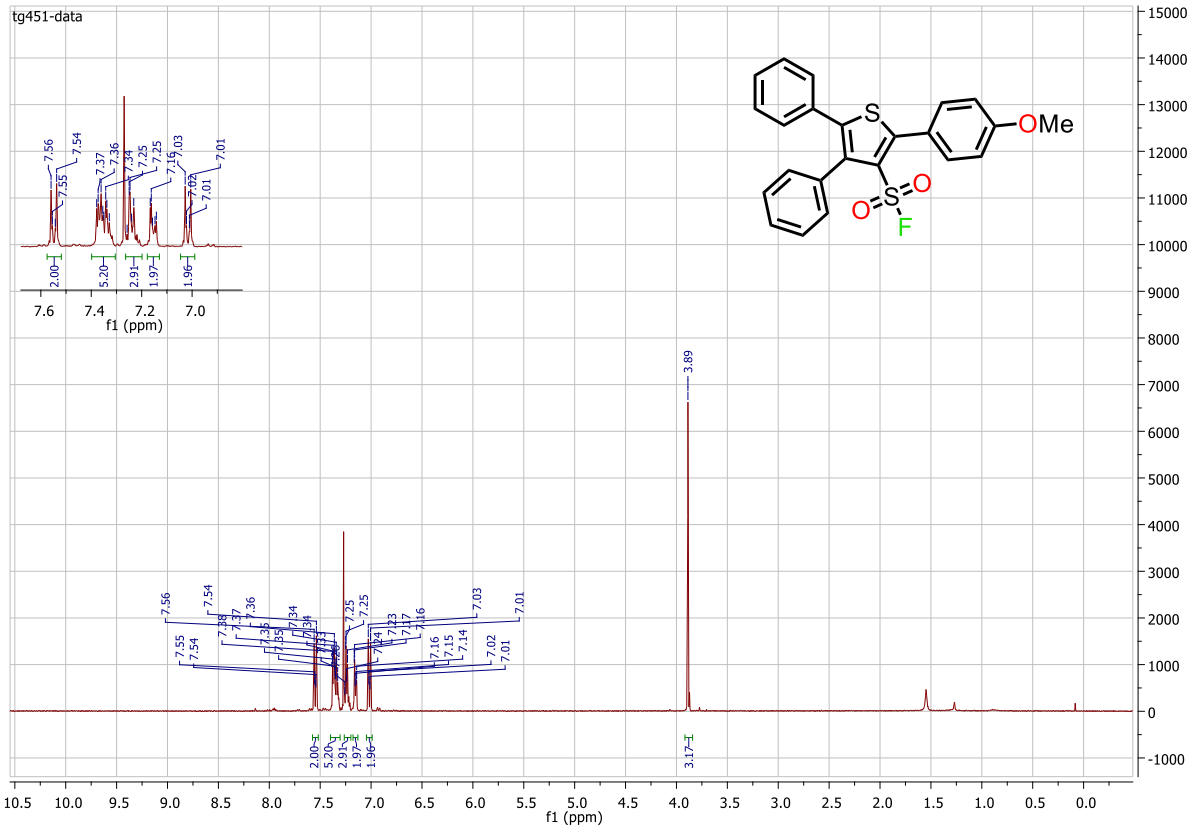
Cyf-1-5-a-F.1.fid
F-19, CDCl₃, DPX-400 QNP Probe. CF₃Cl as Ref at 0 ppm.

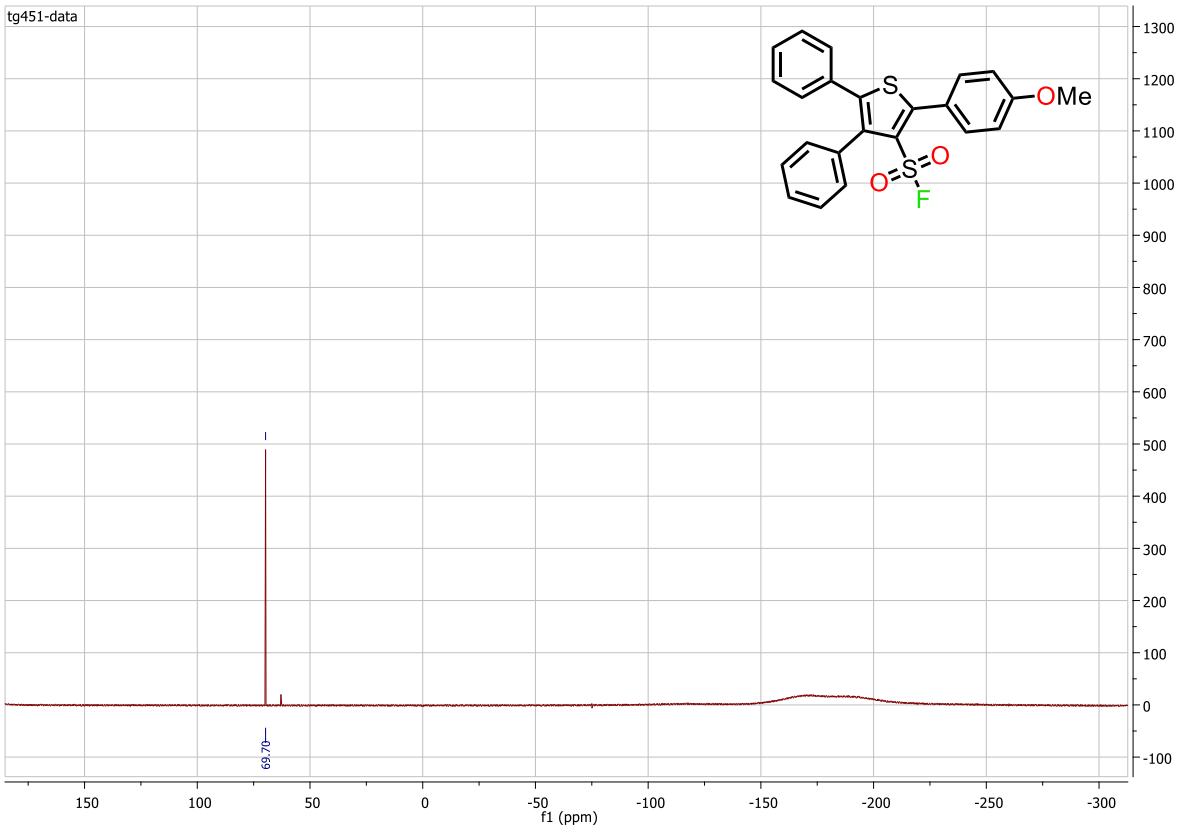


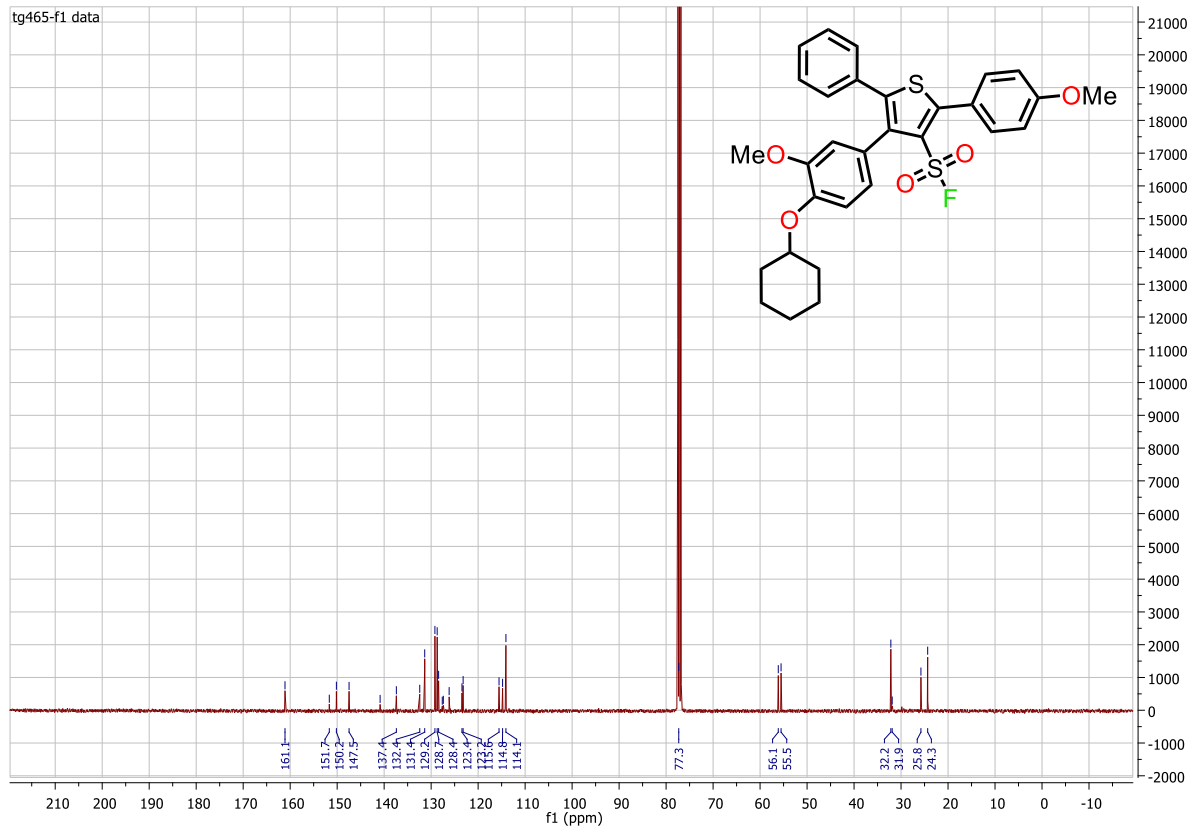
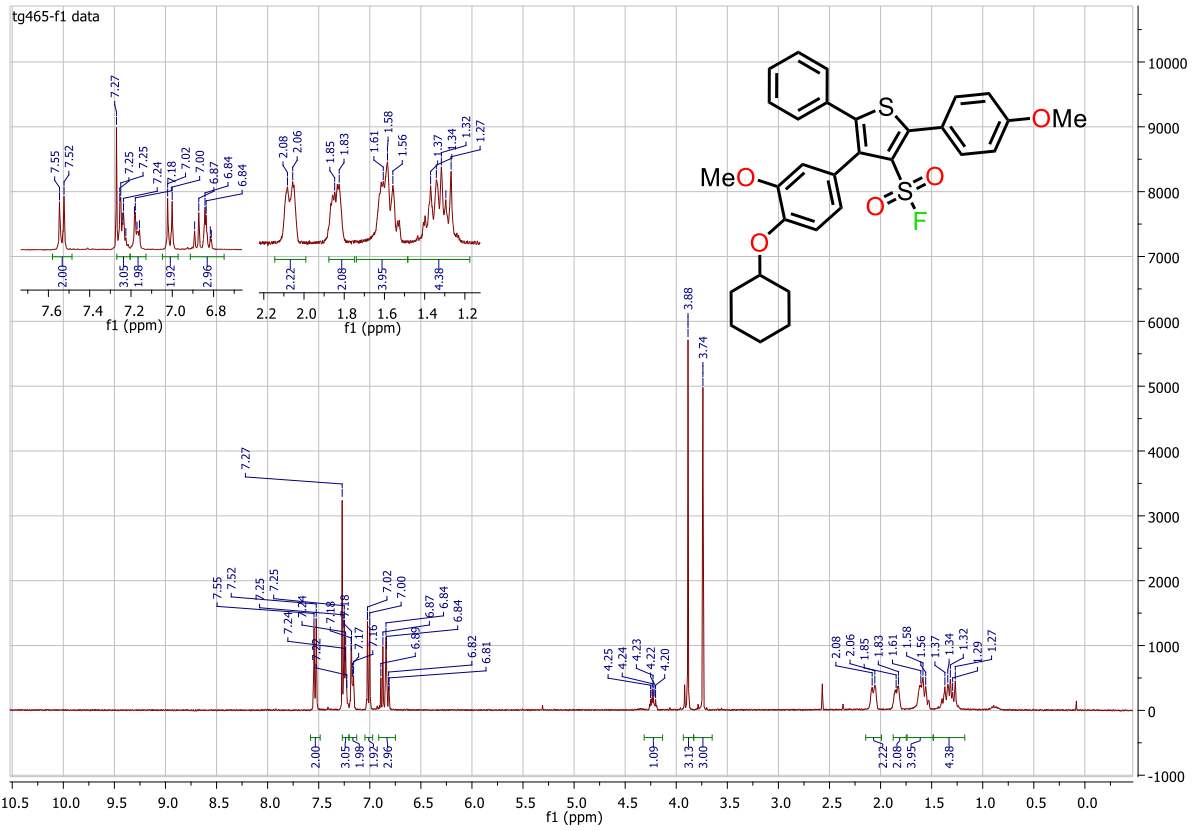


tg543-f1

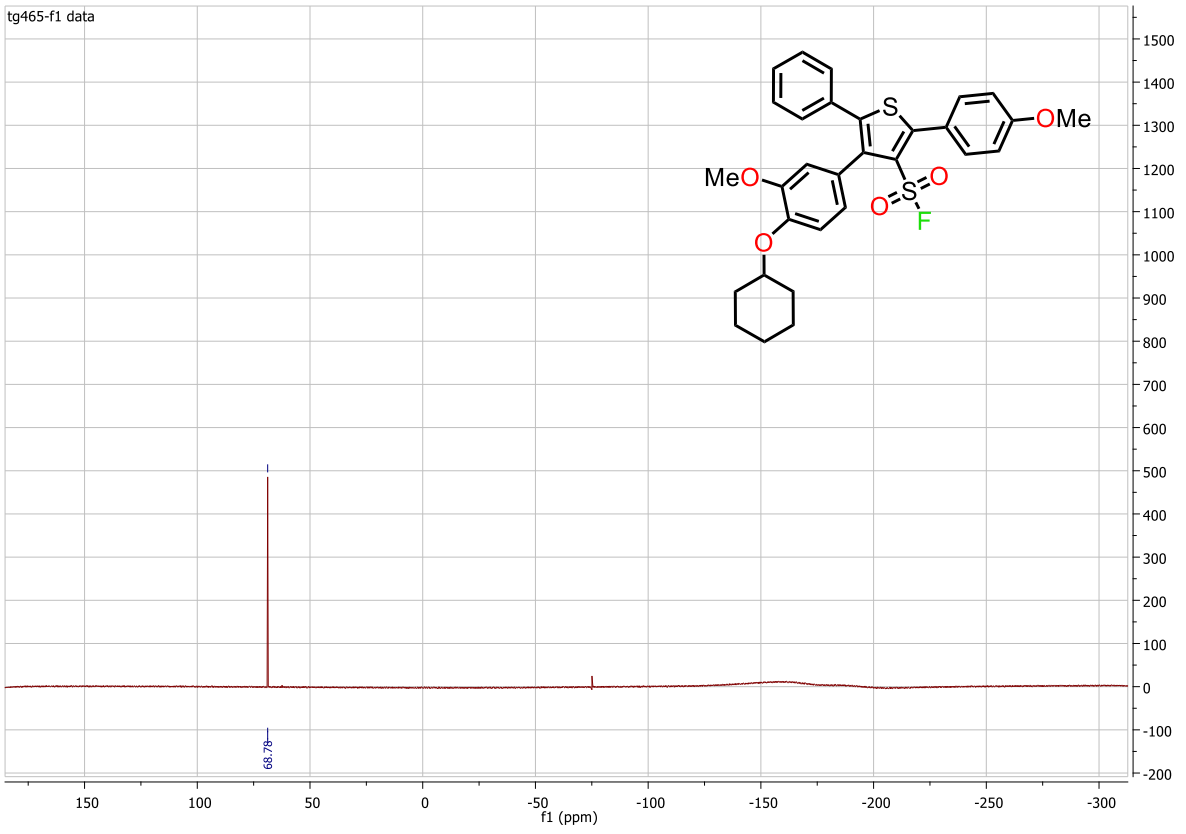


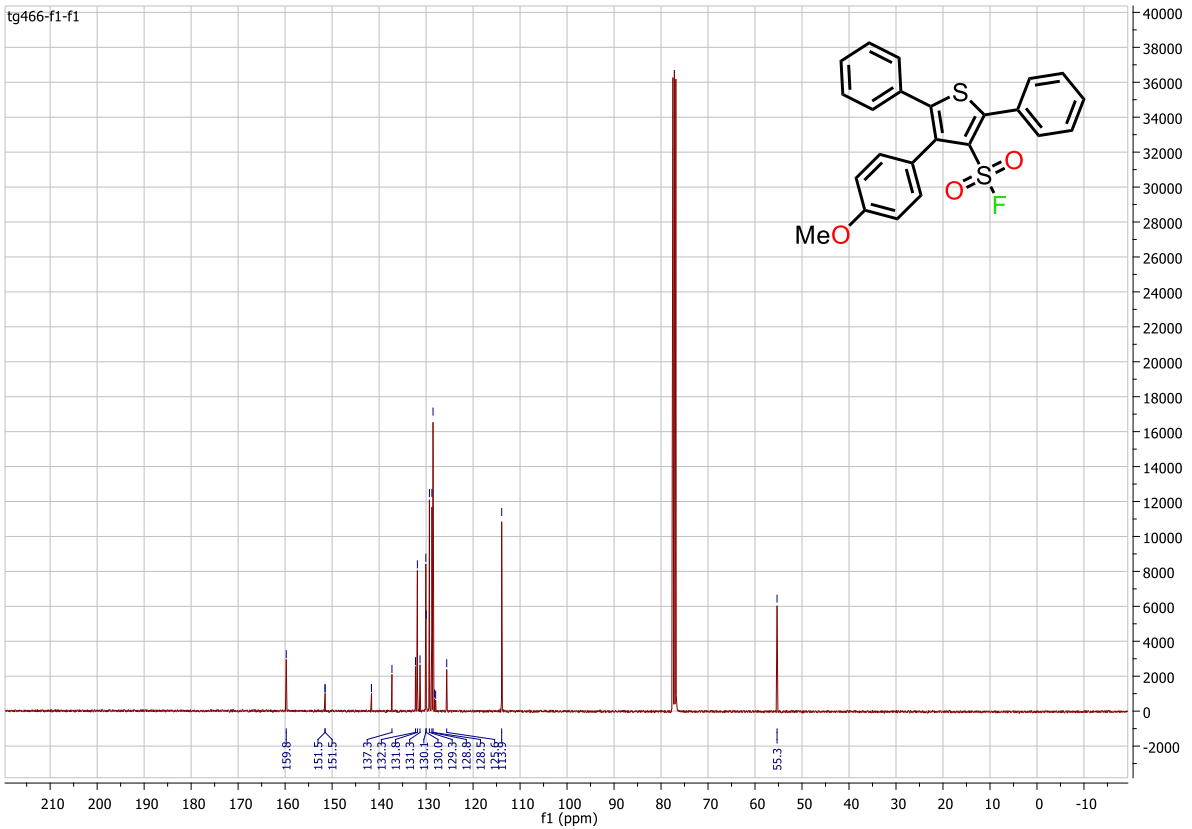
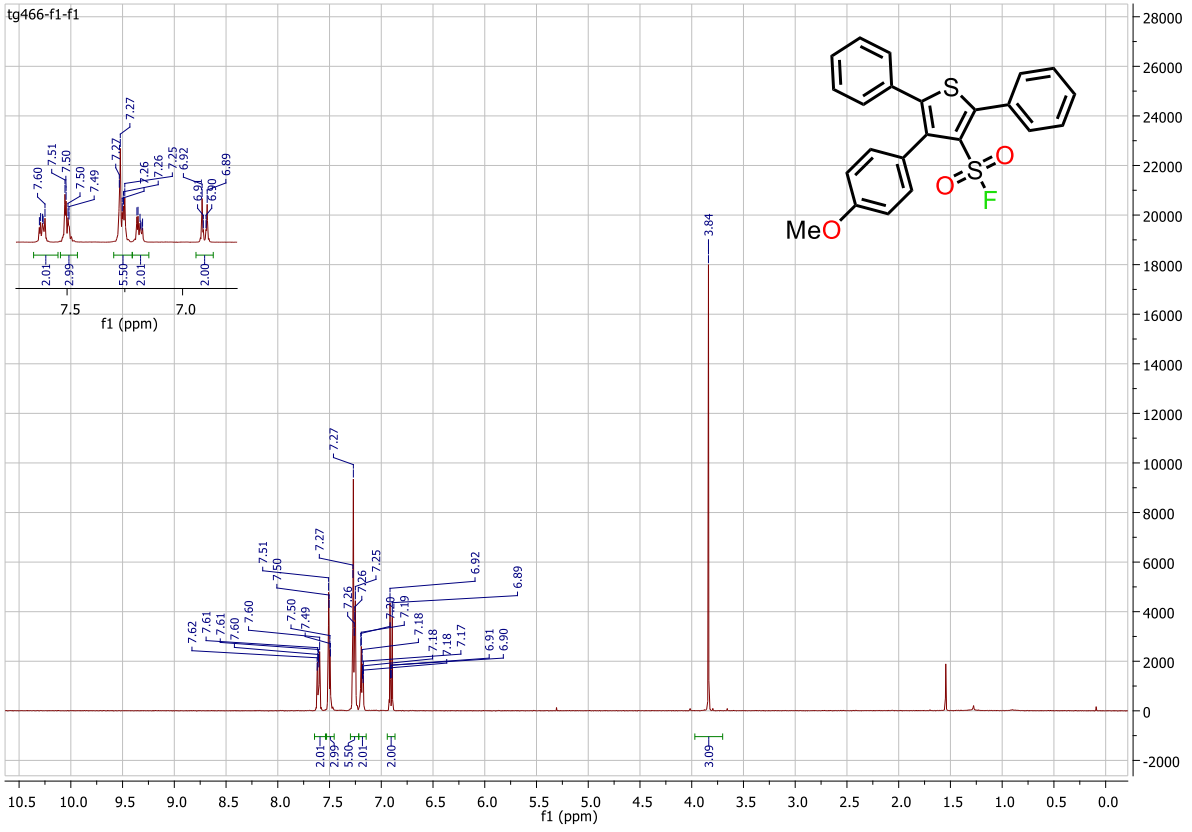


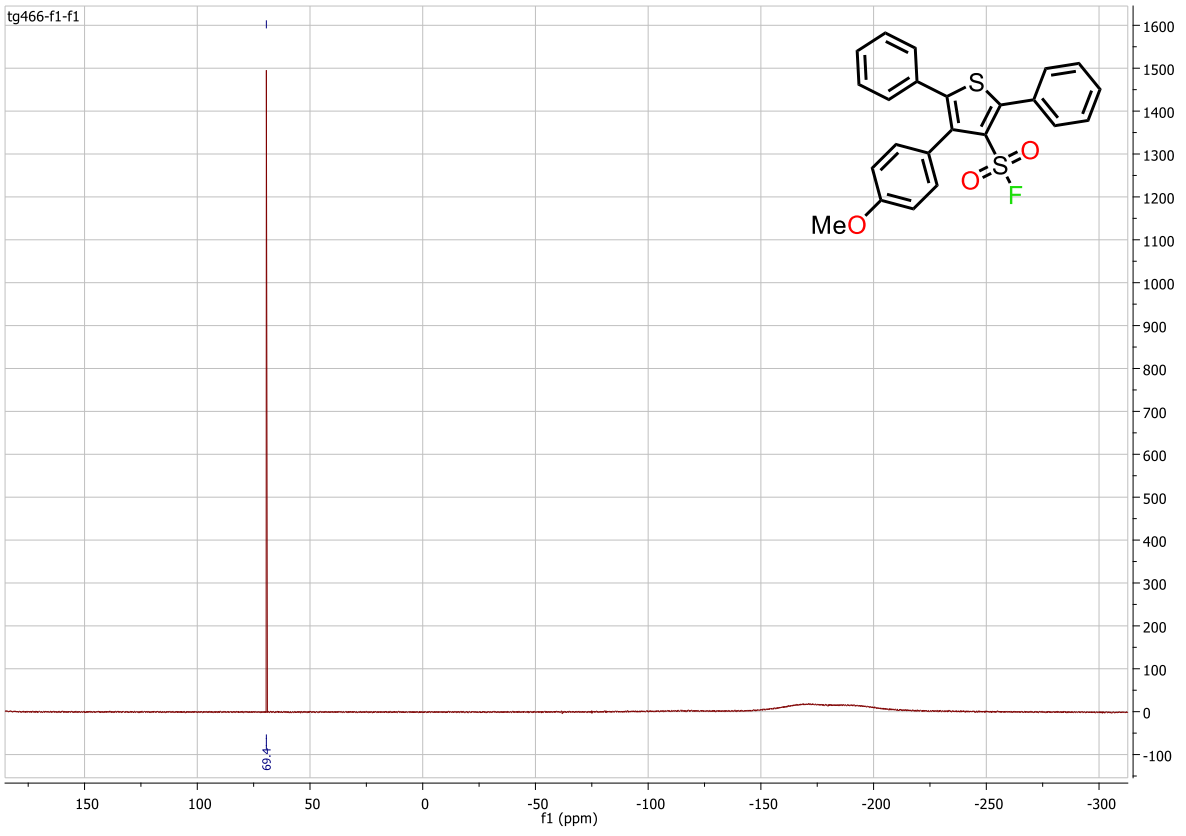


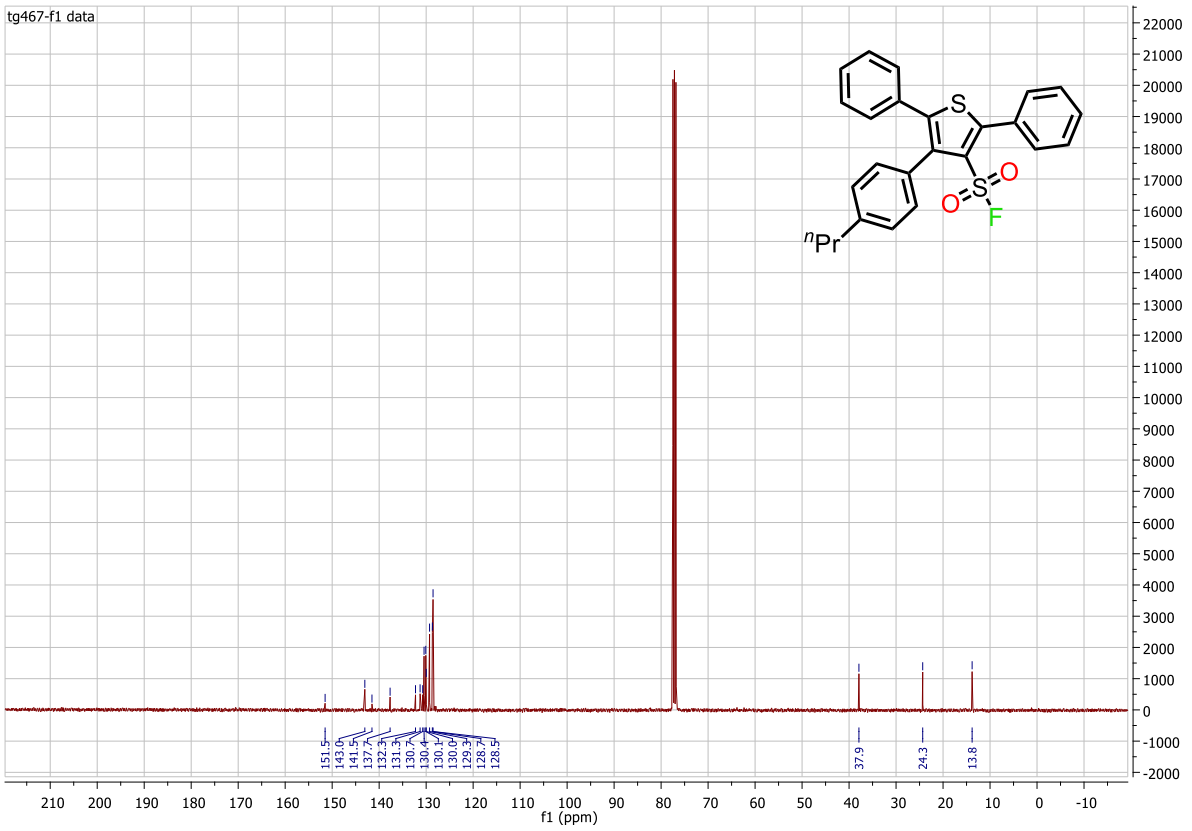
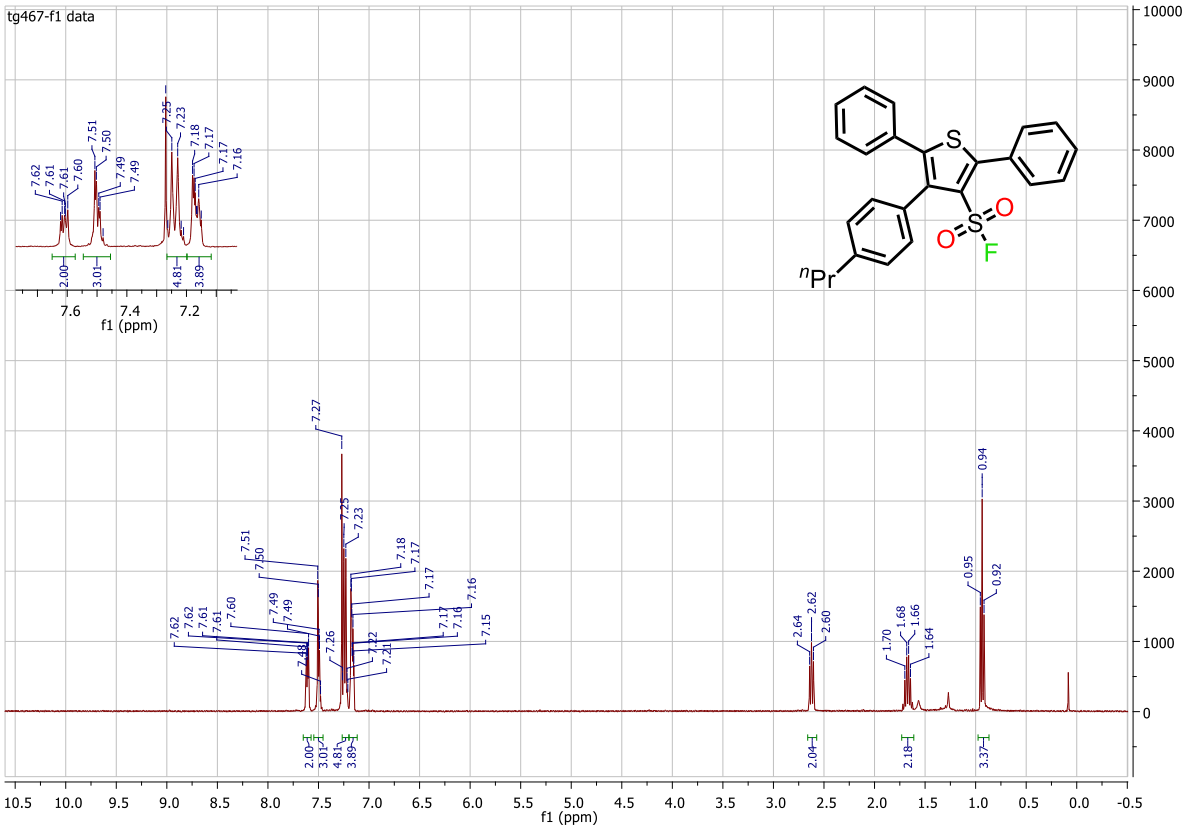


tg465-f1 data

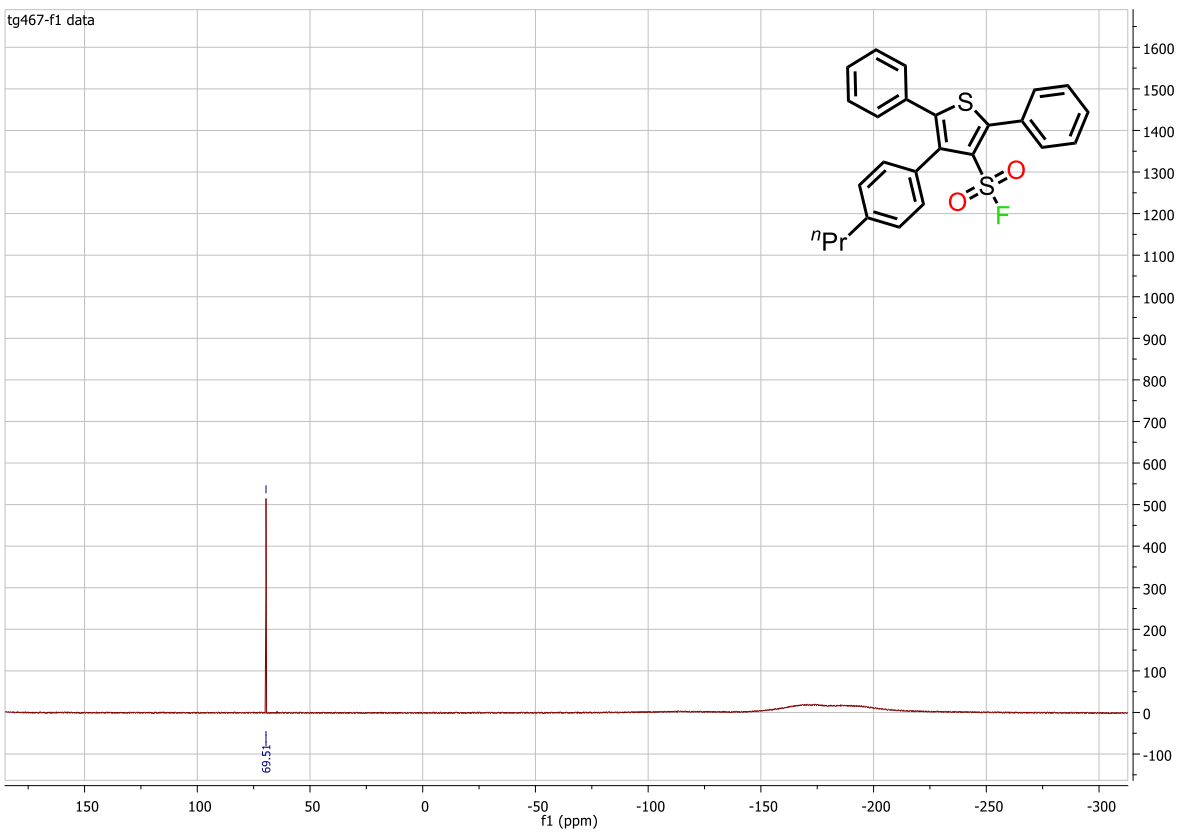


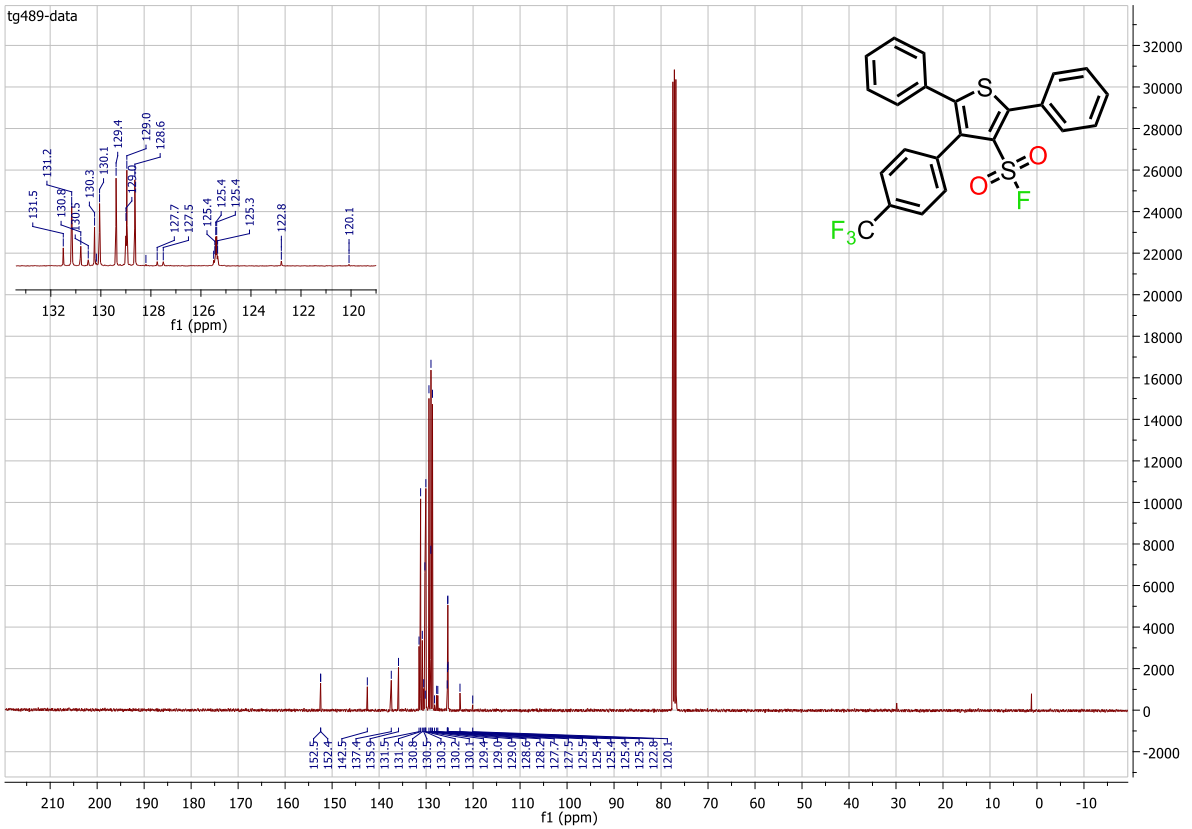
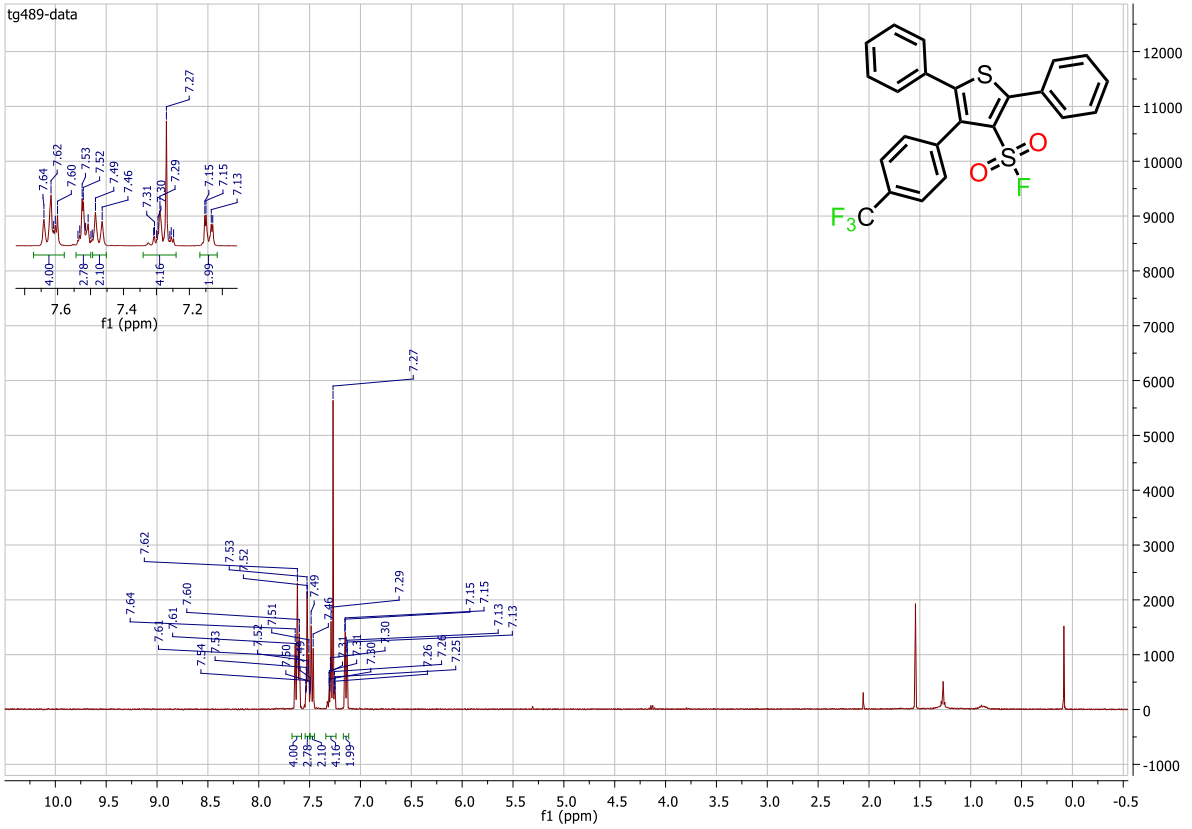


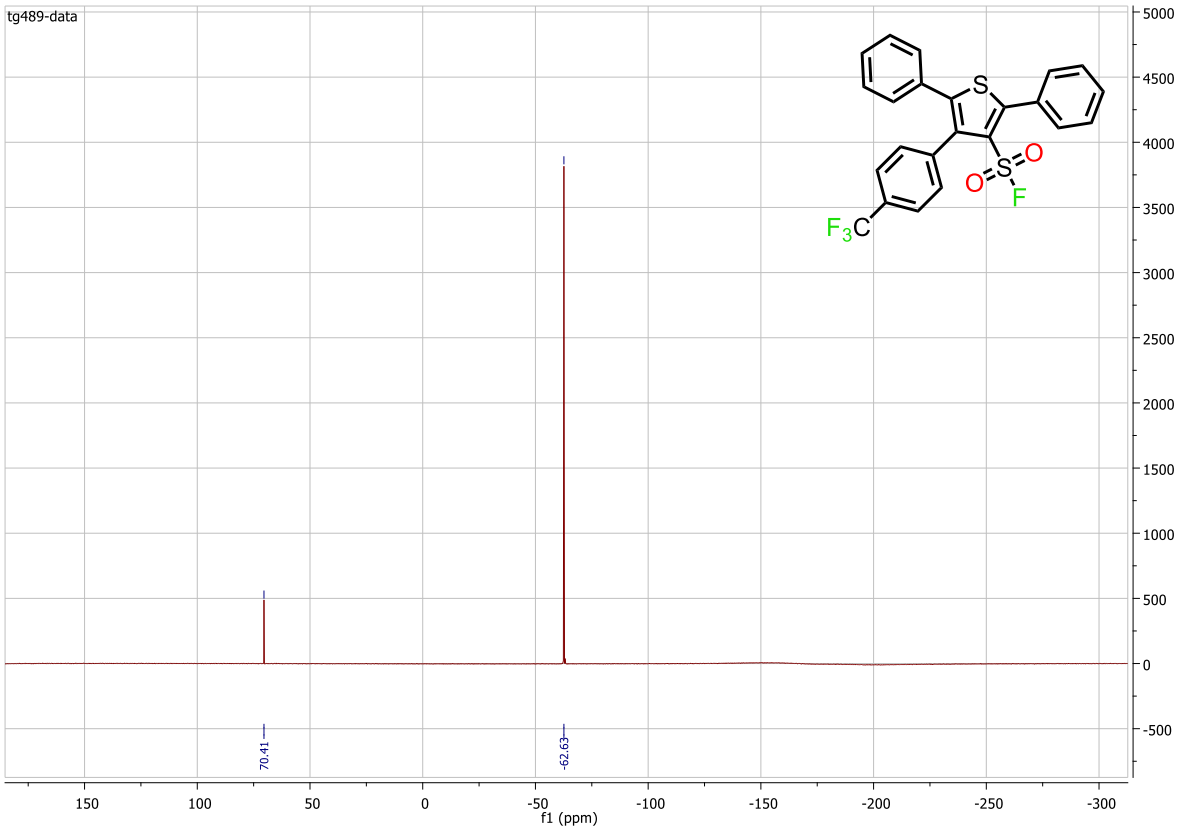


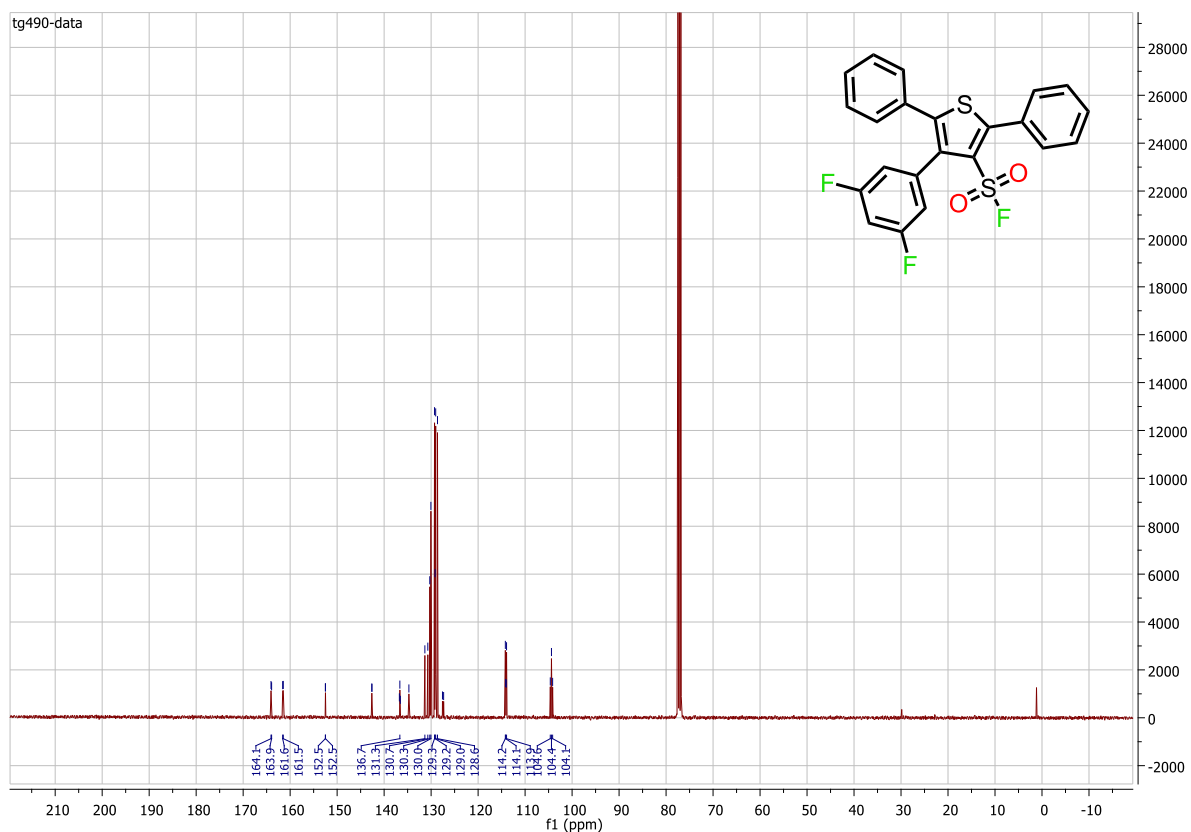
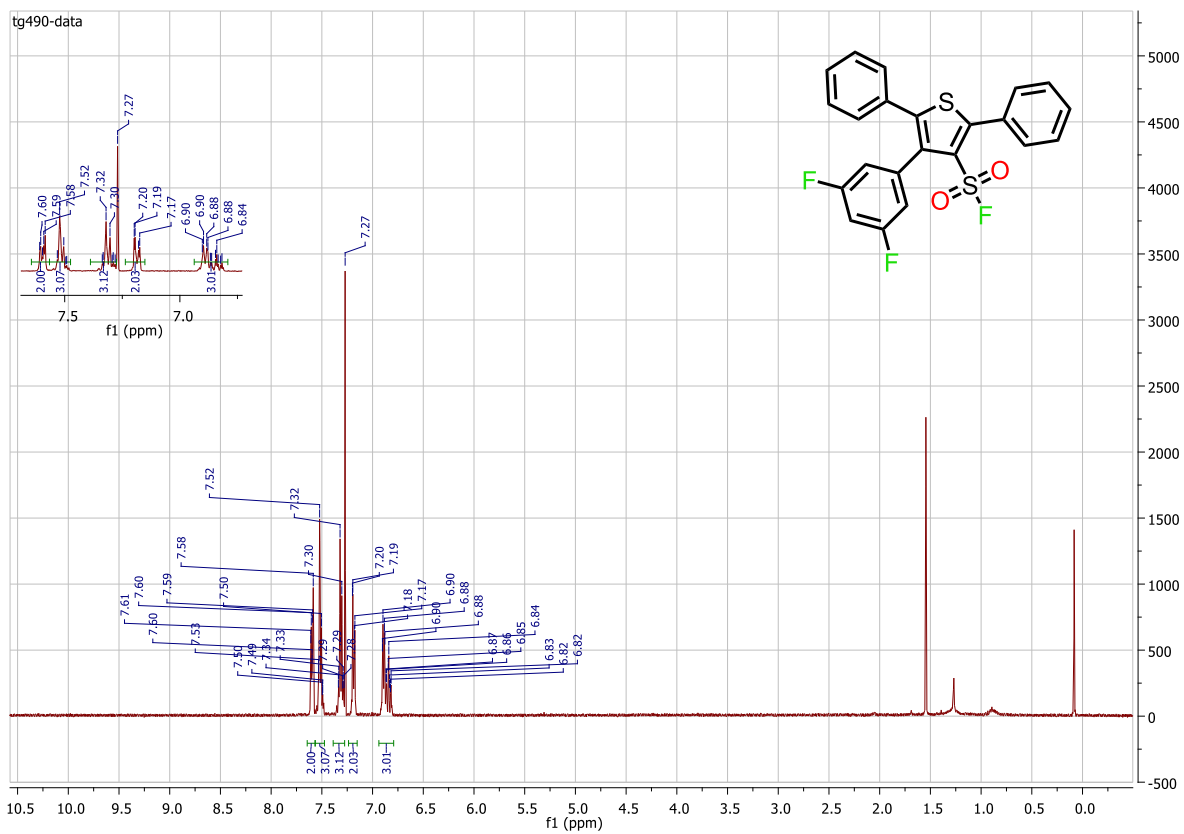


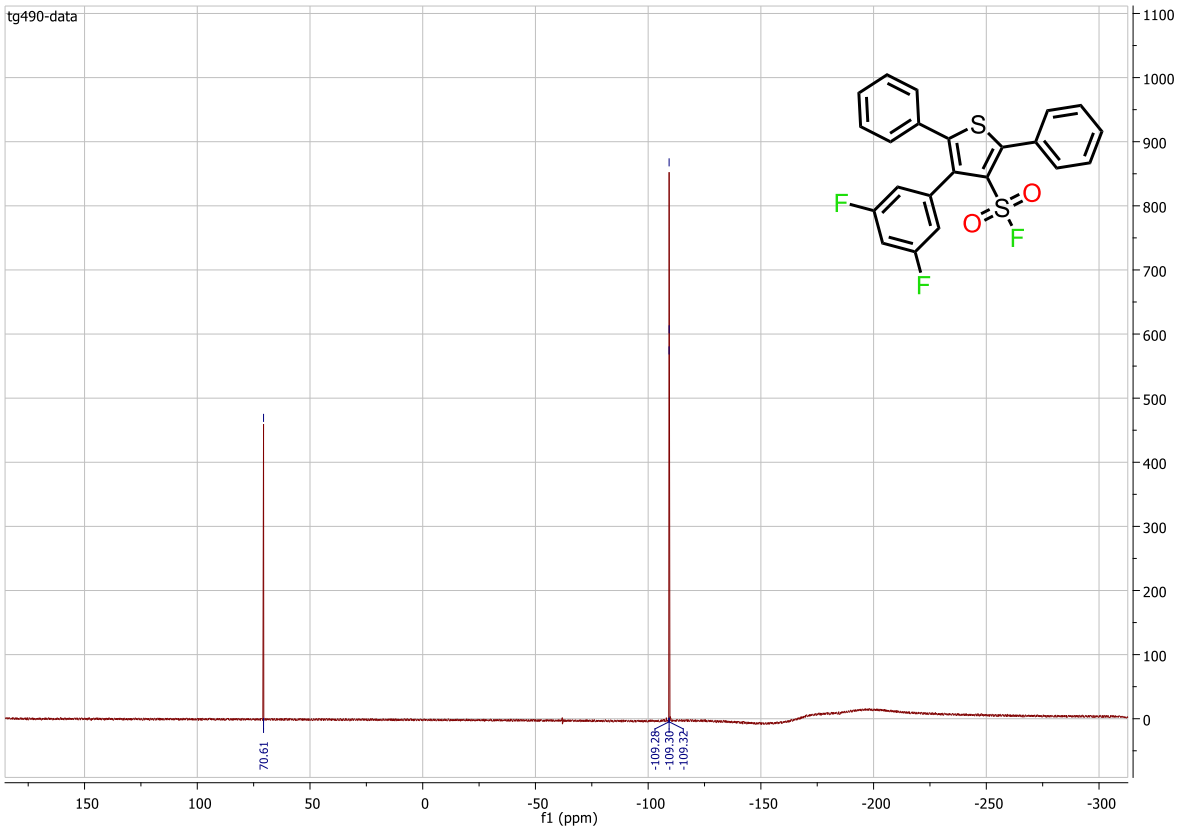
tg467-f1 data

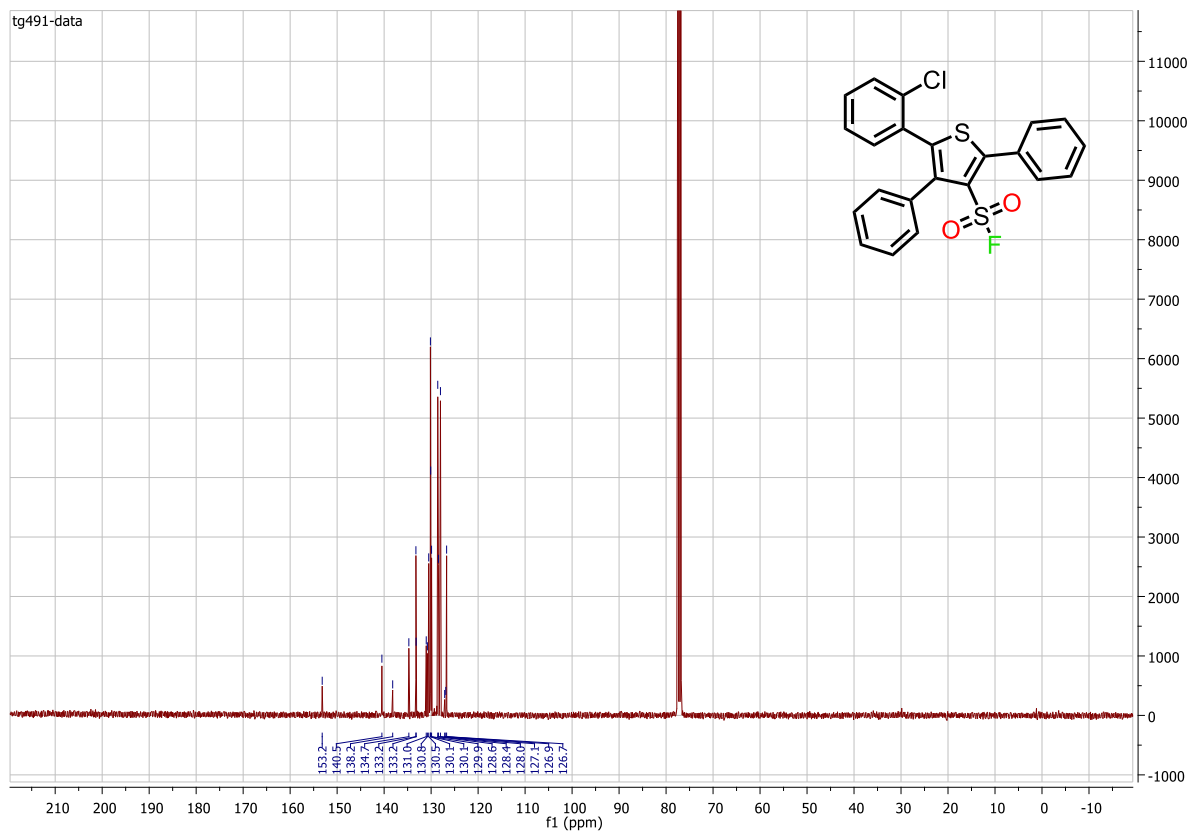
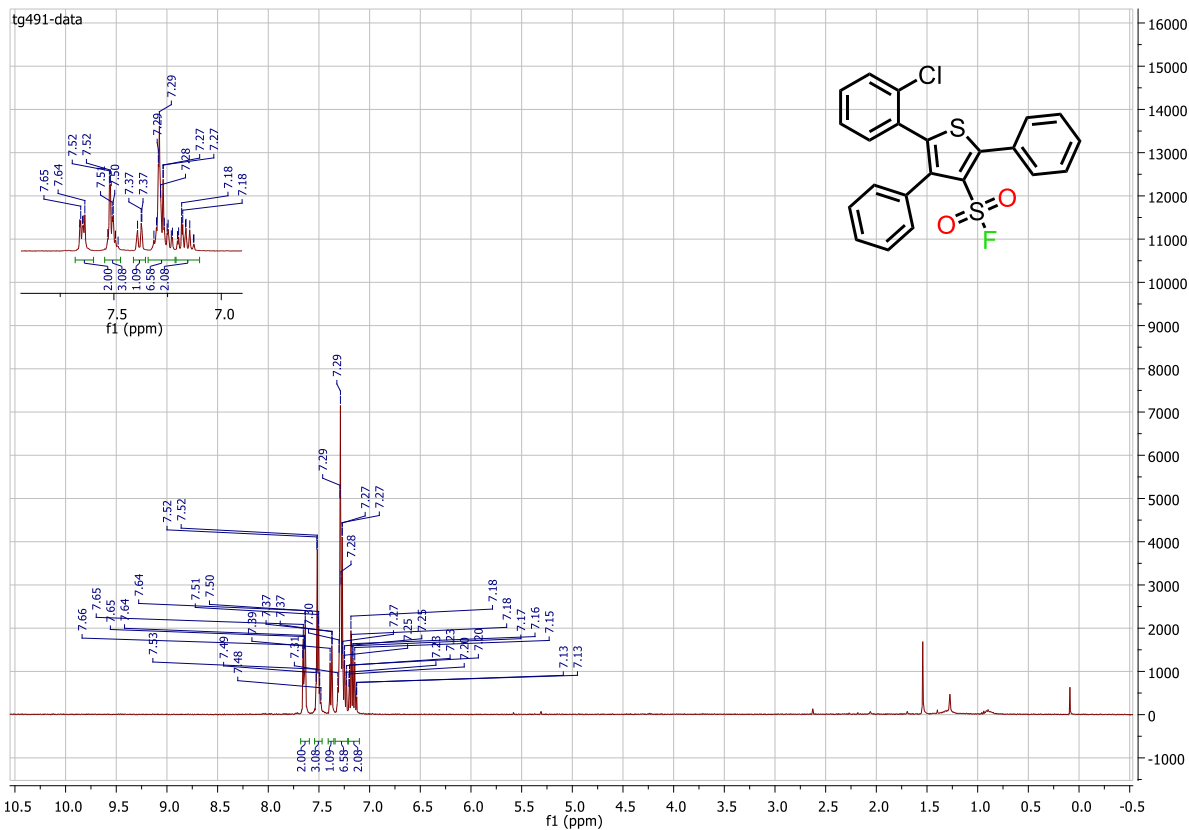


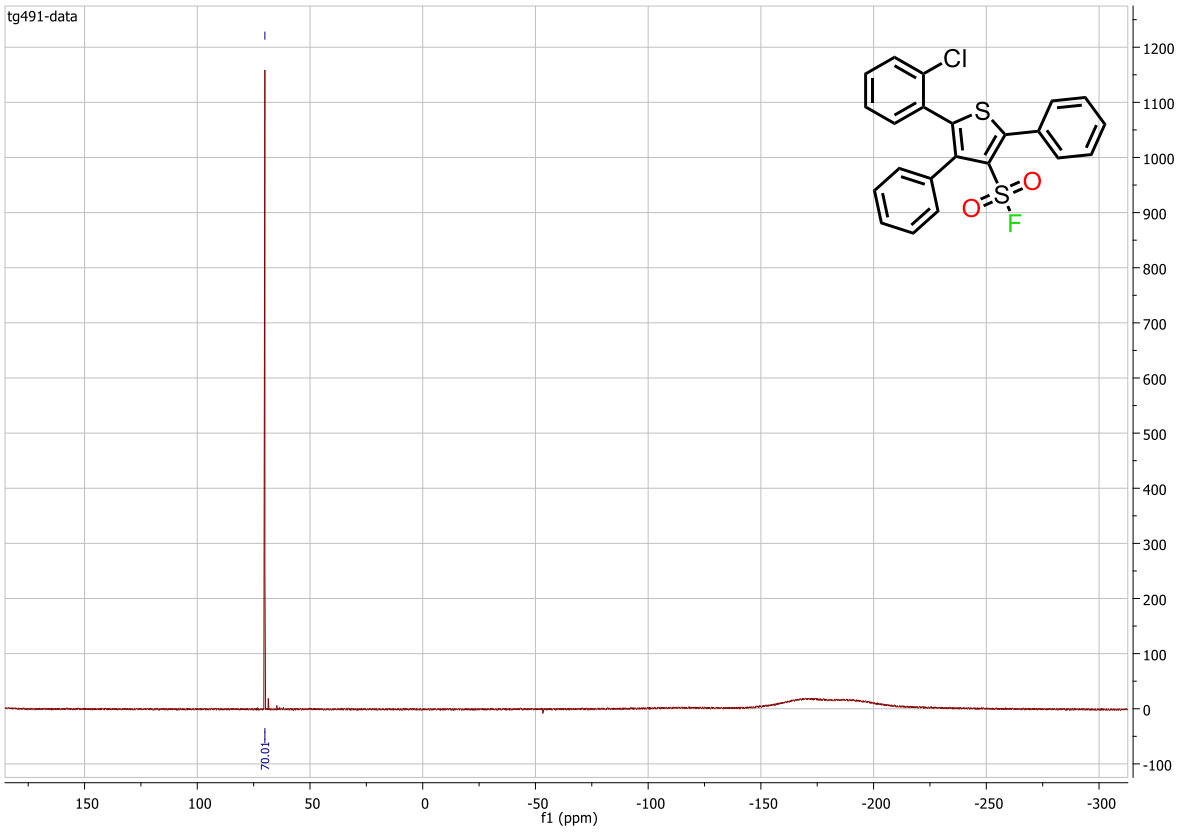


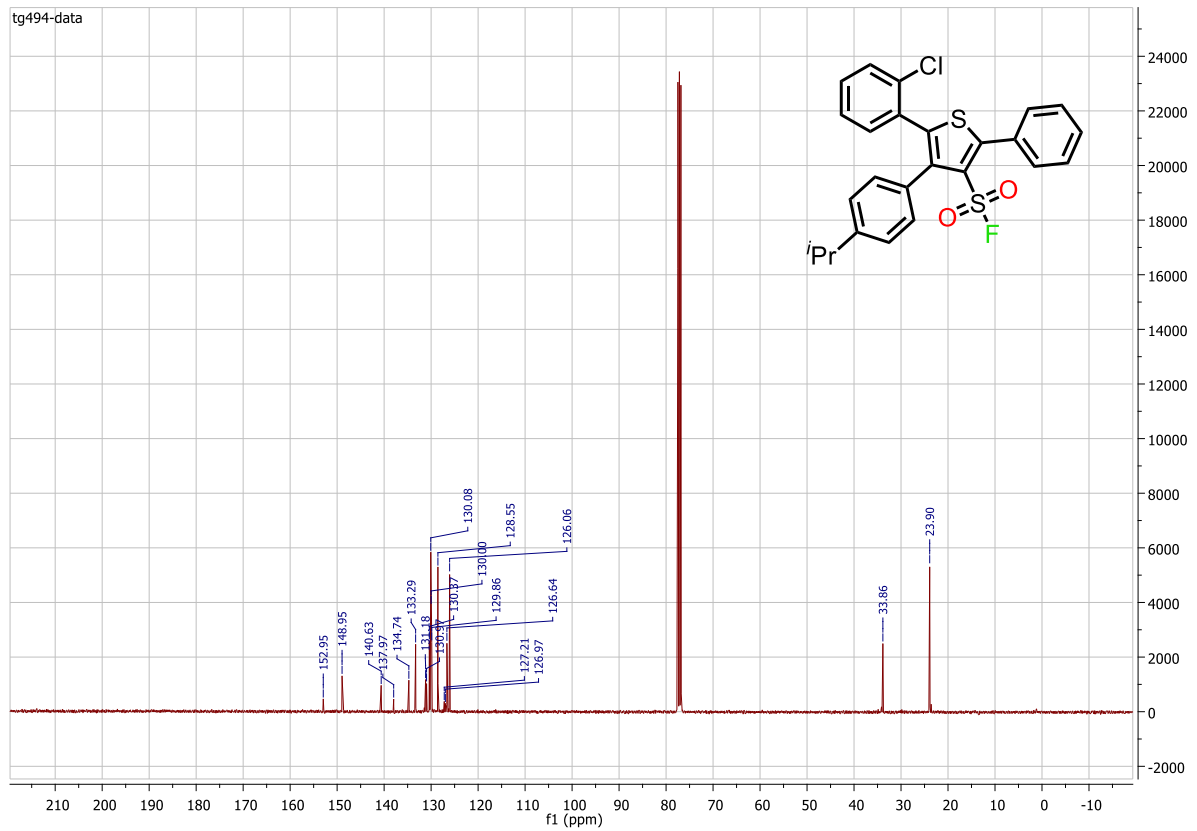
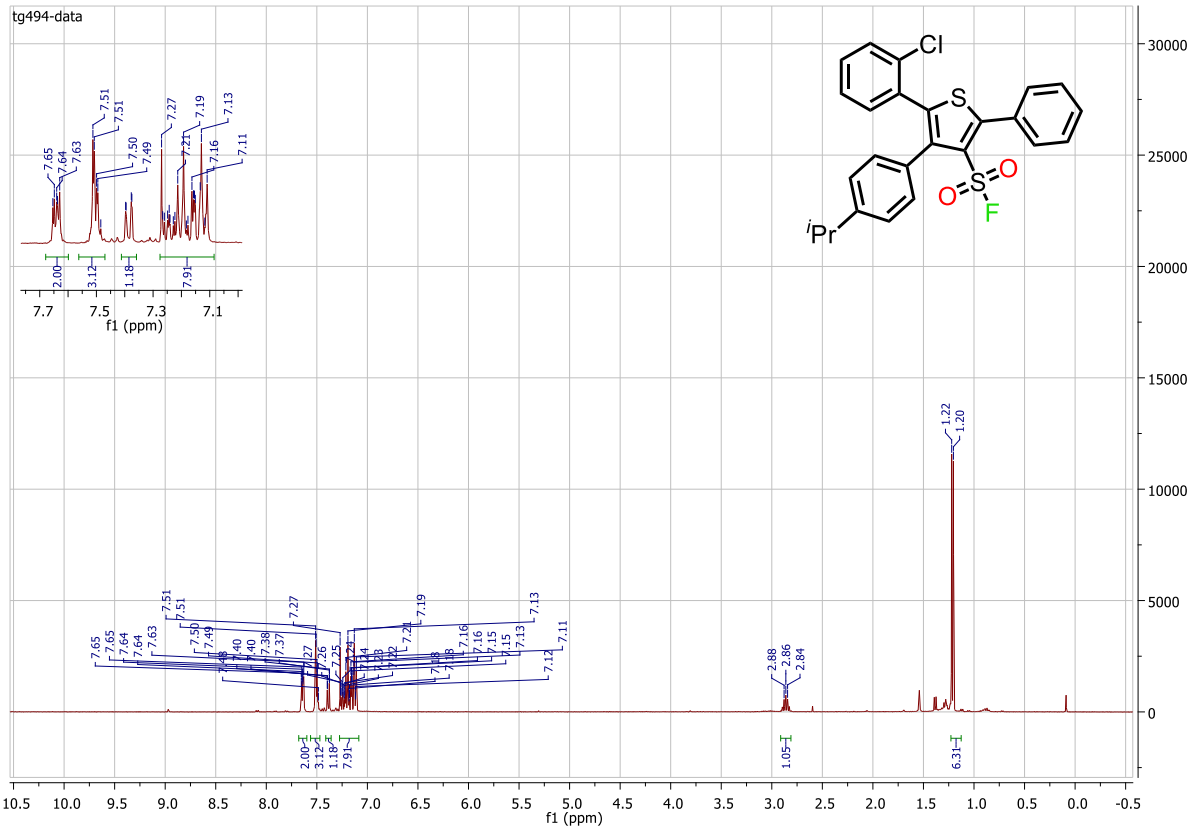




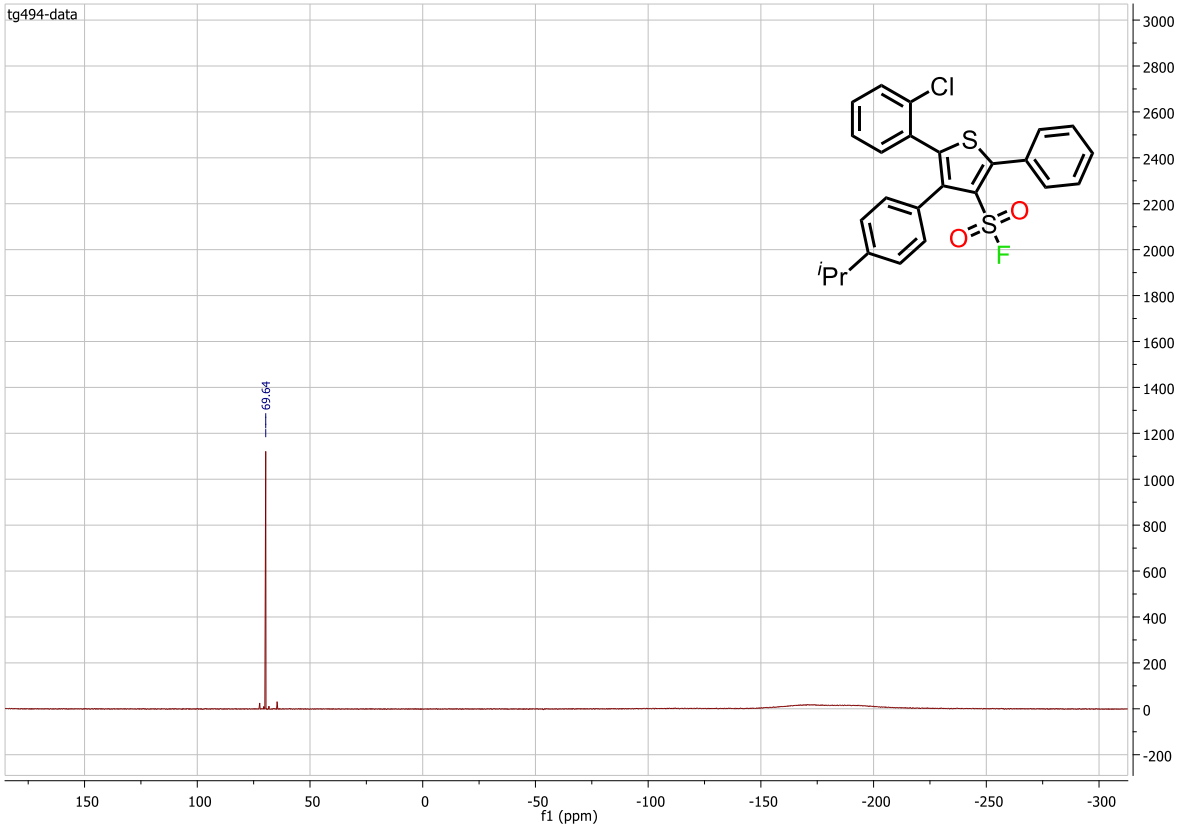


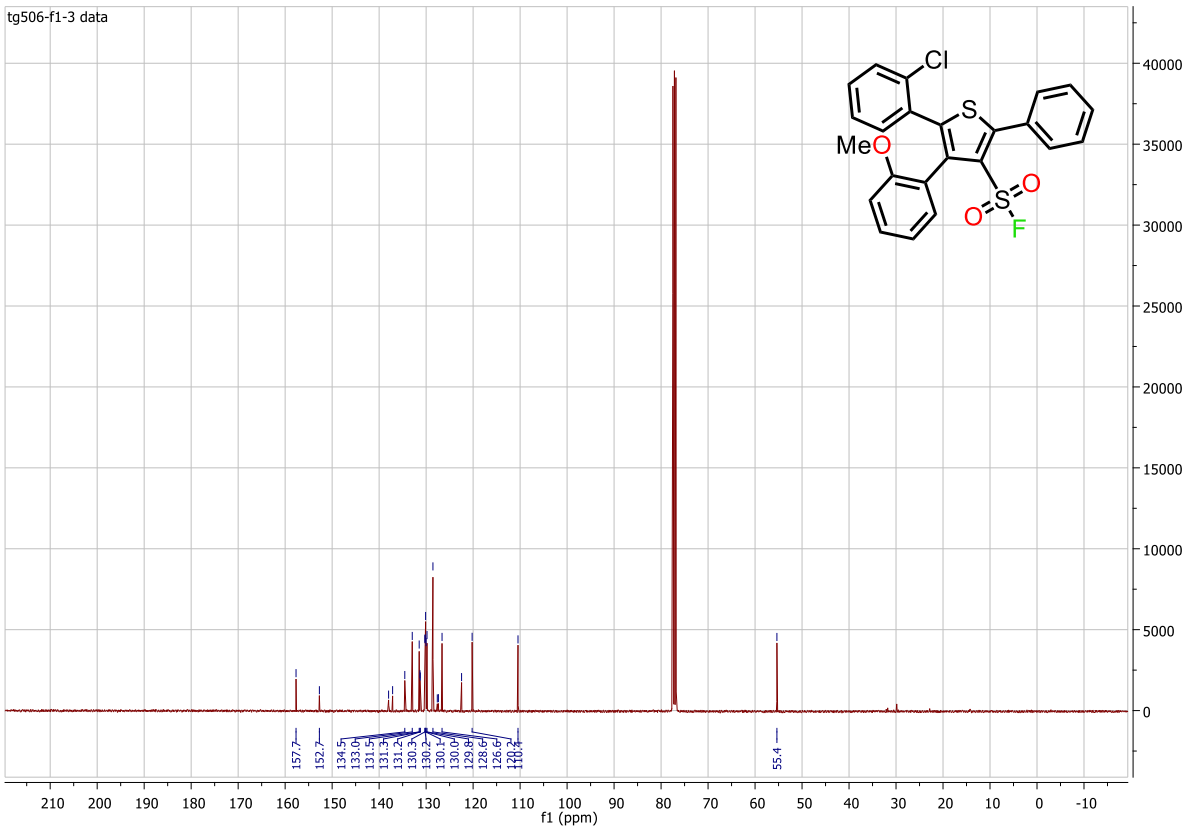
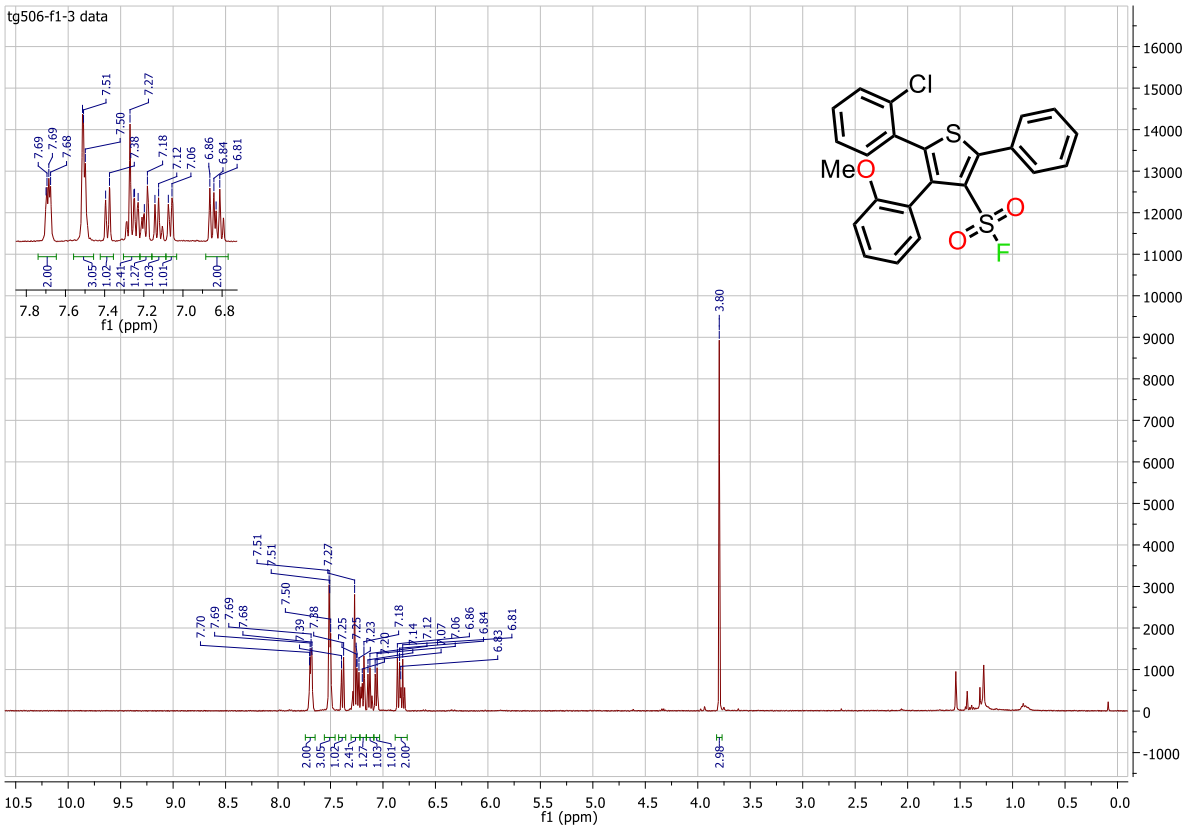




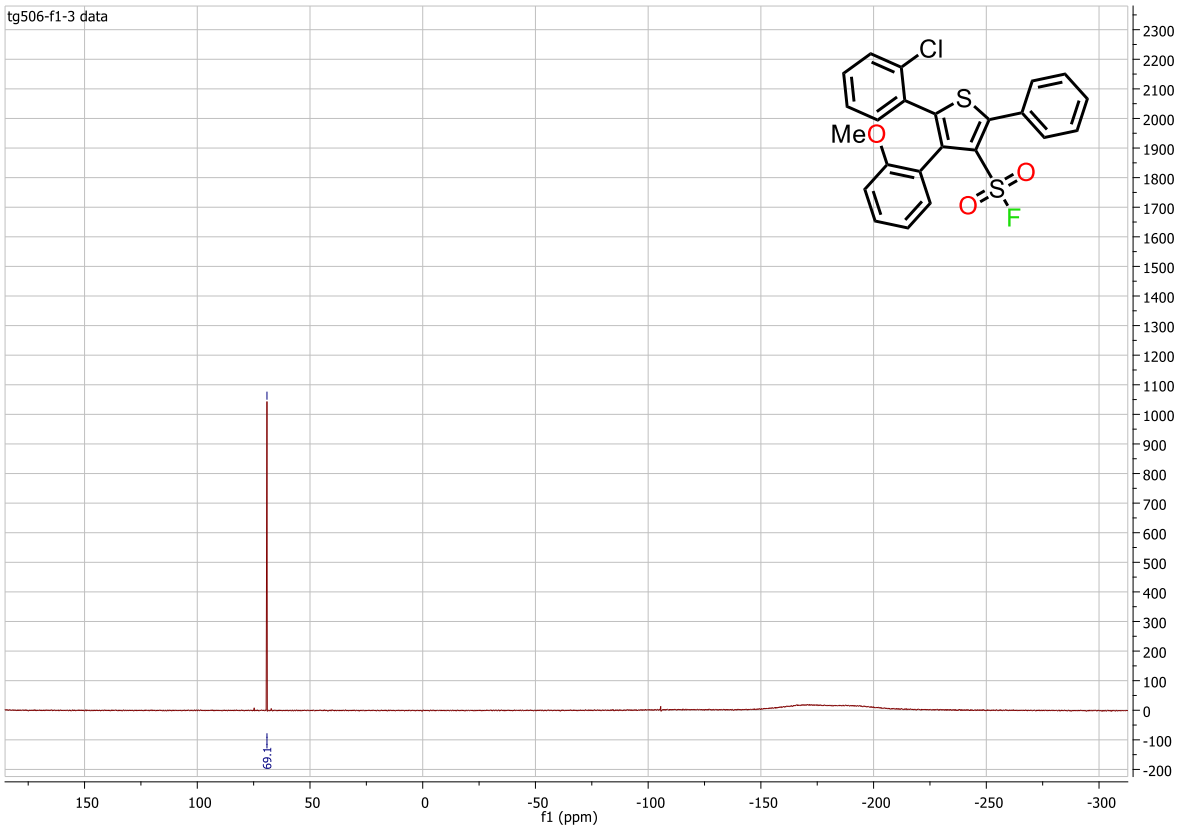


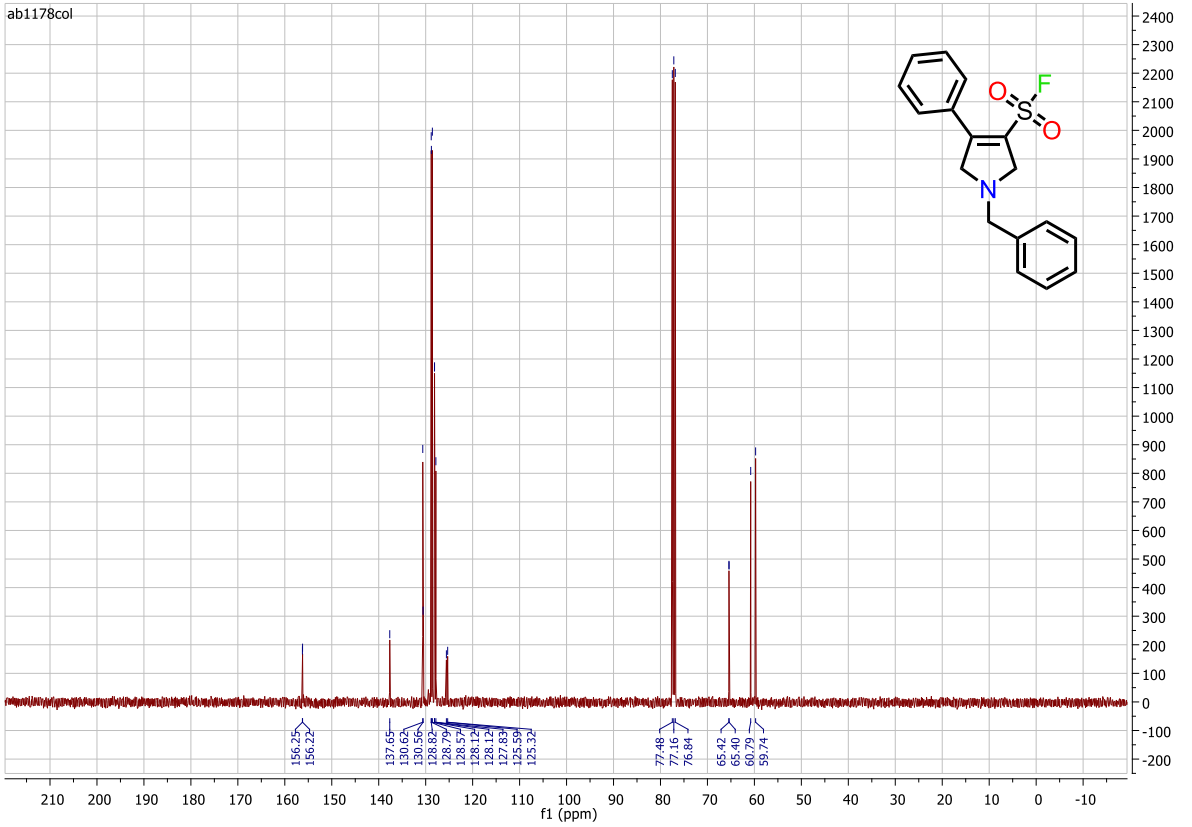
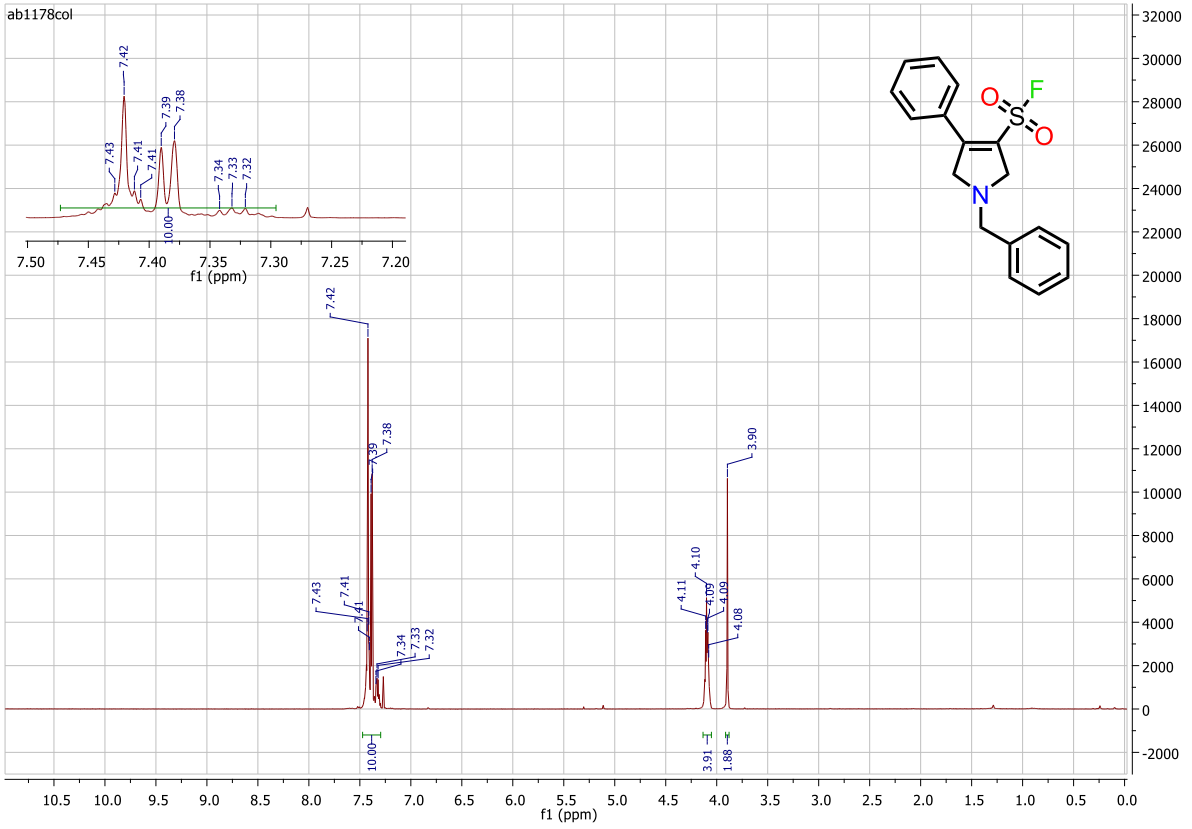
tg494-data

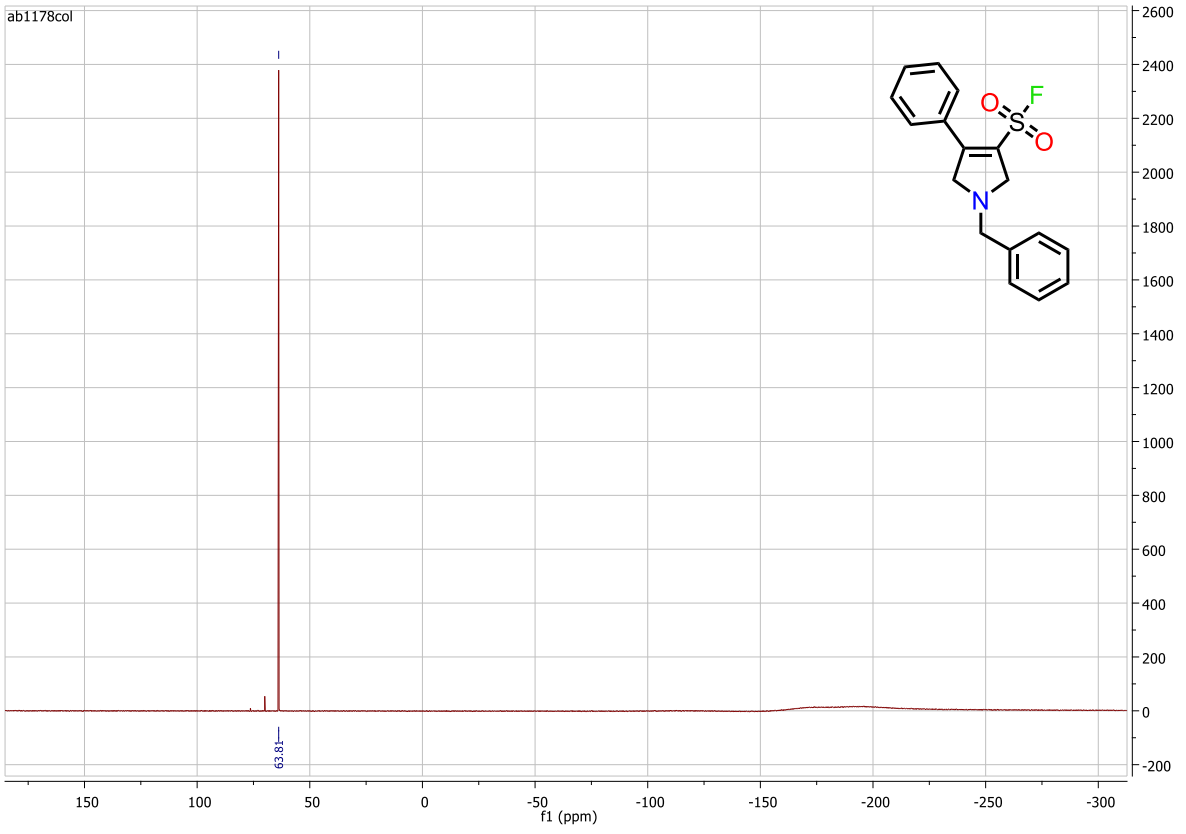


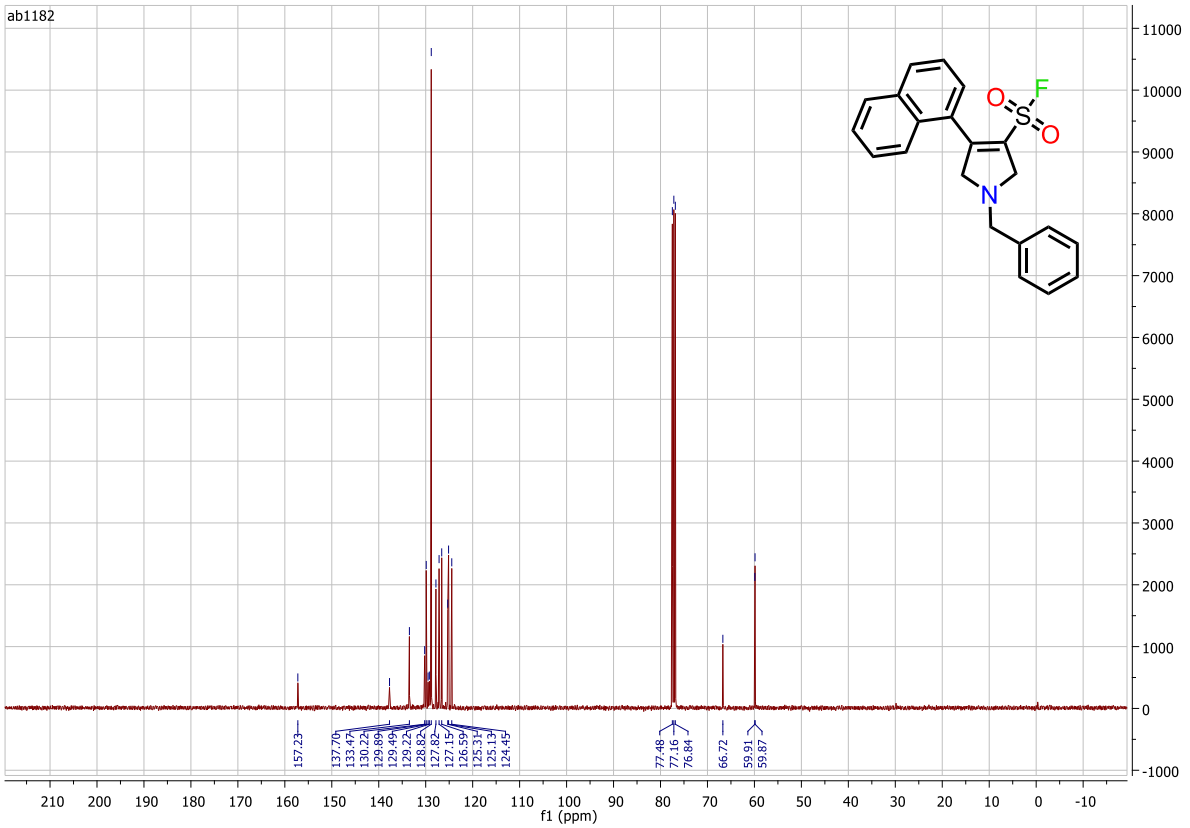
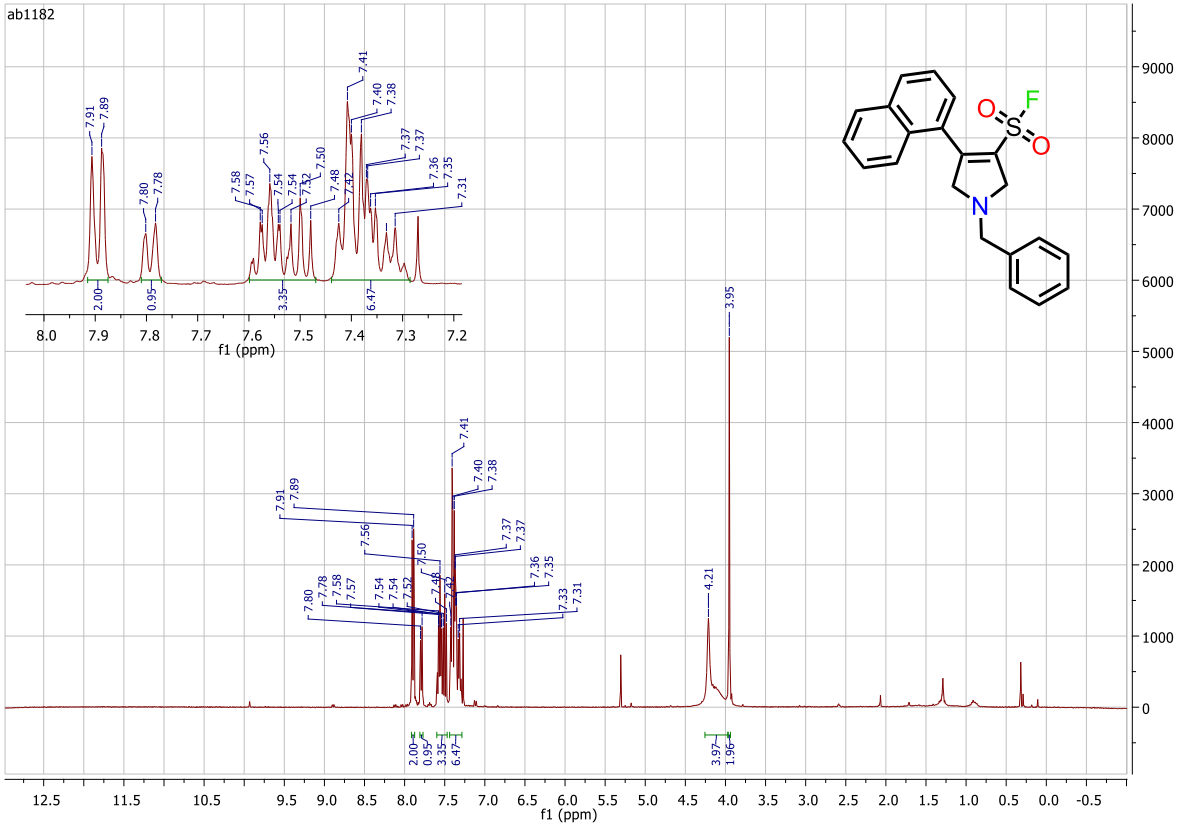


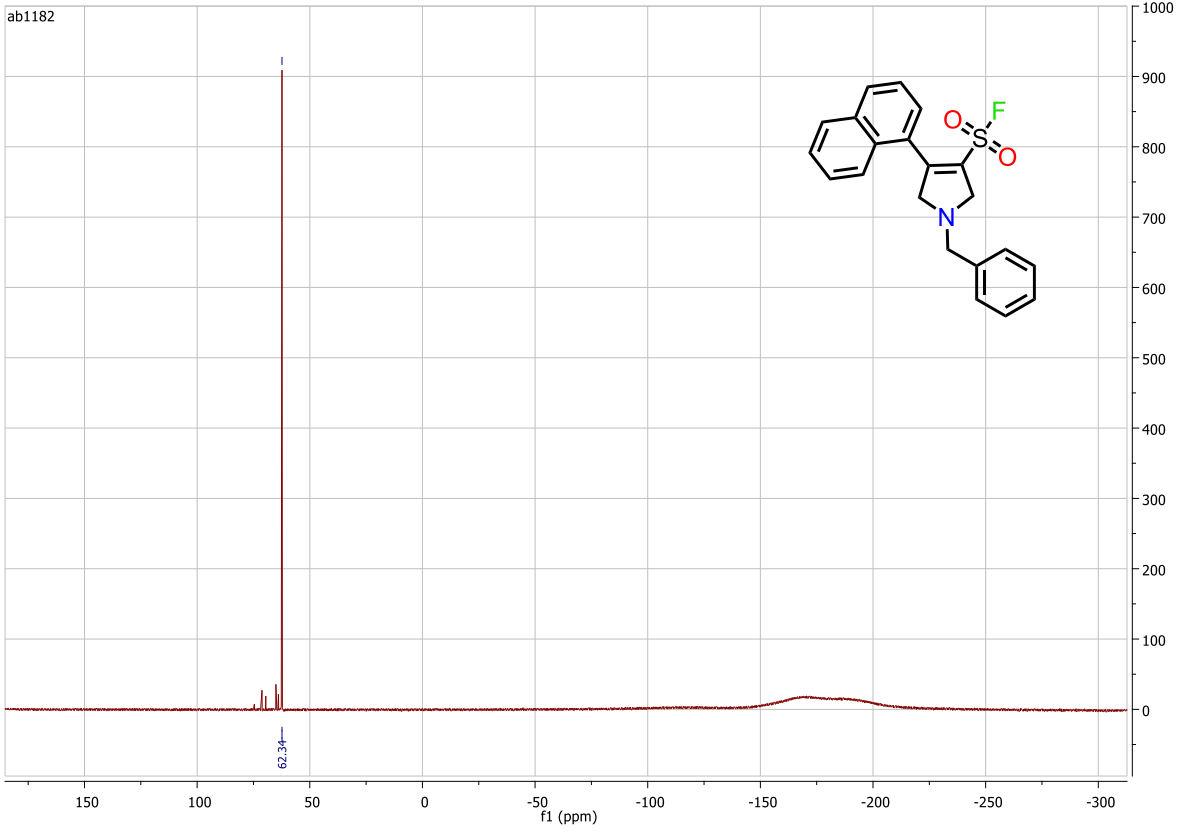
tg506-f1-3 data

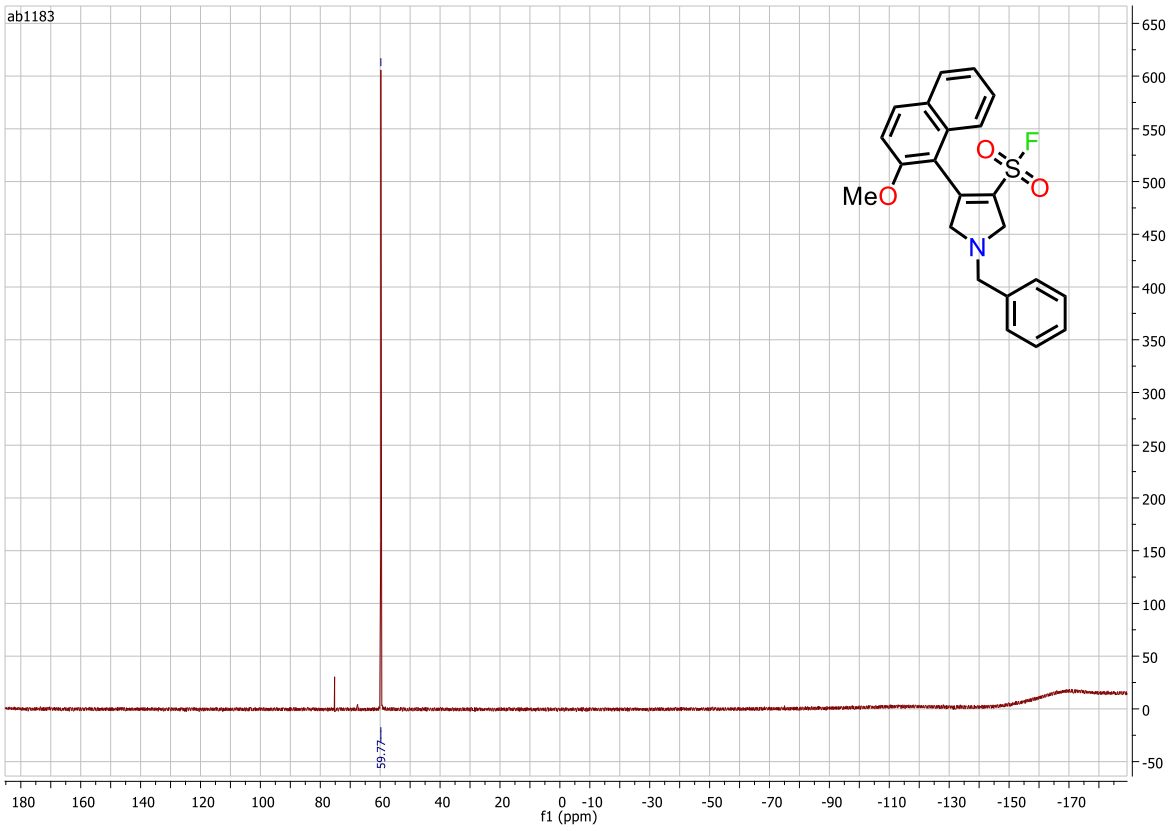


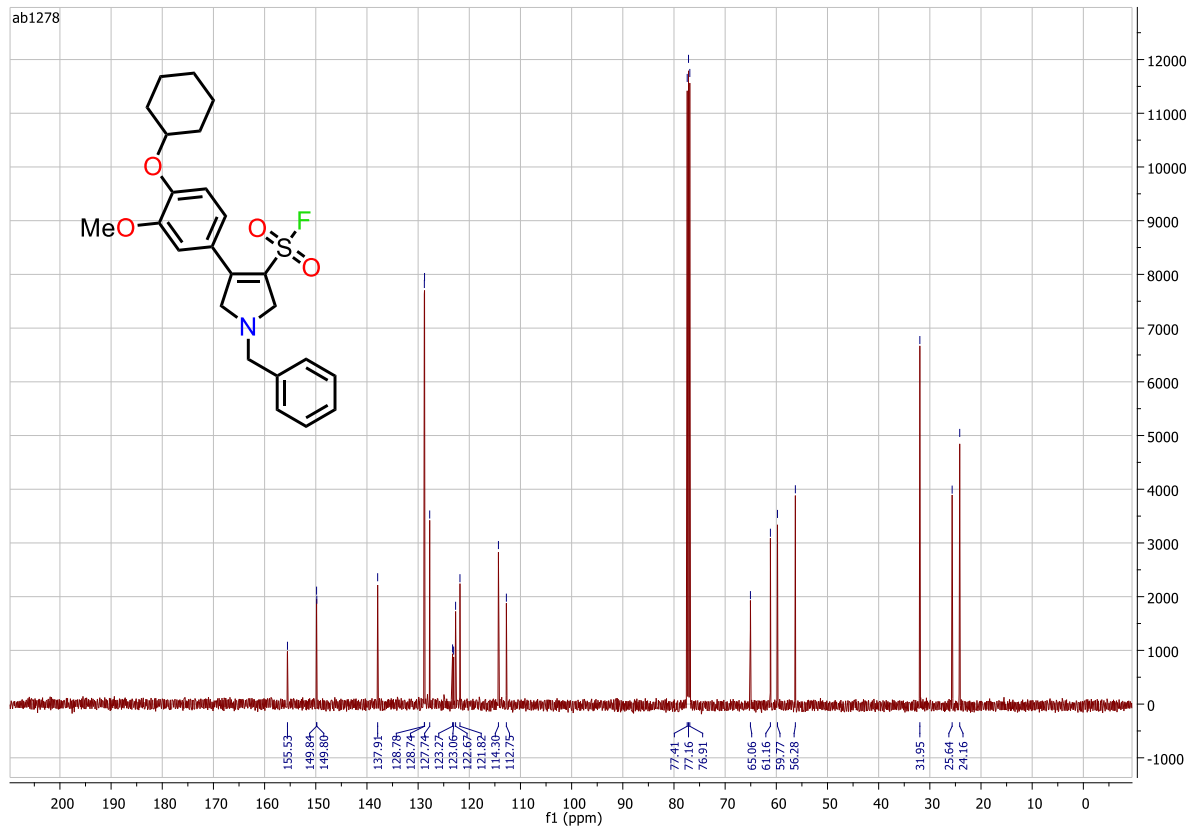
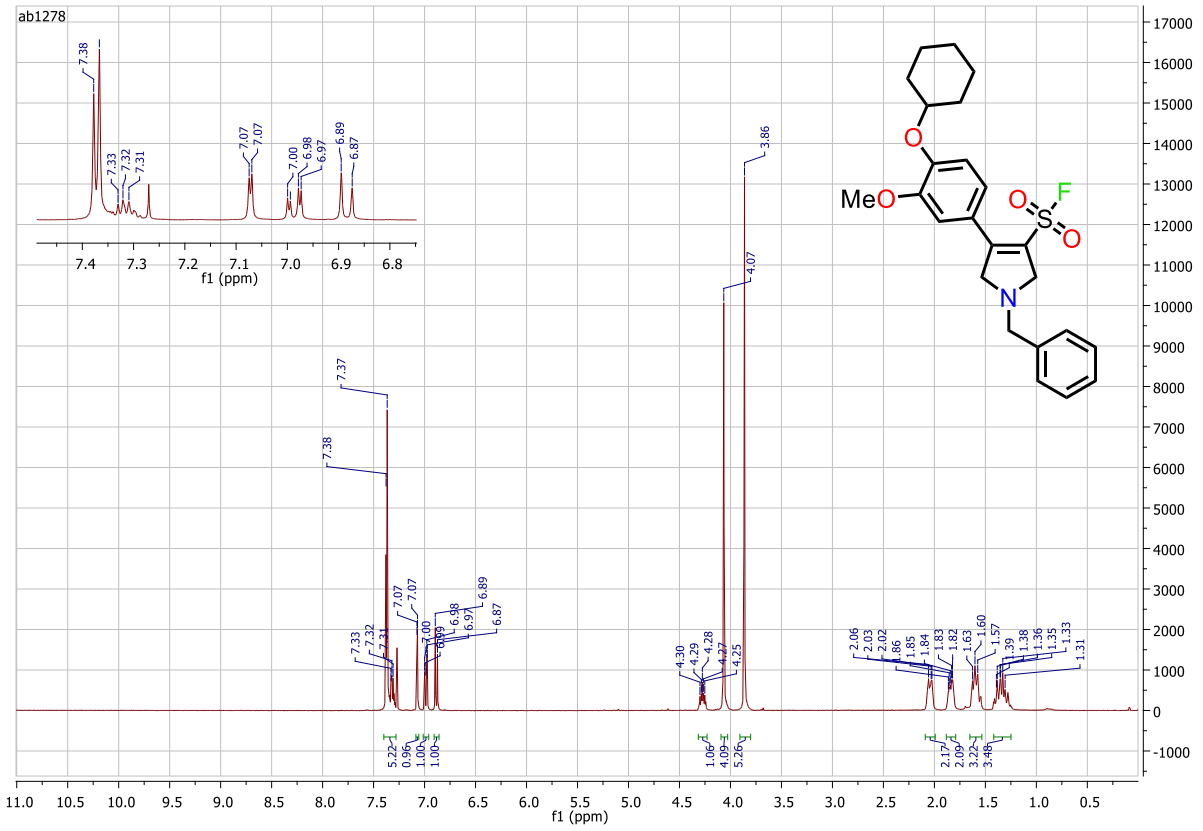


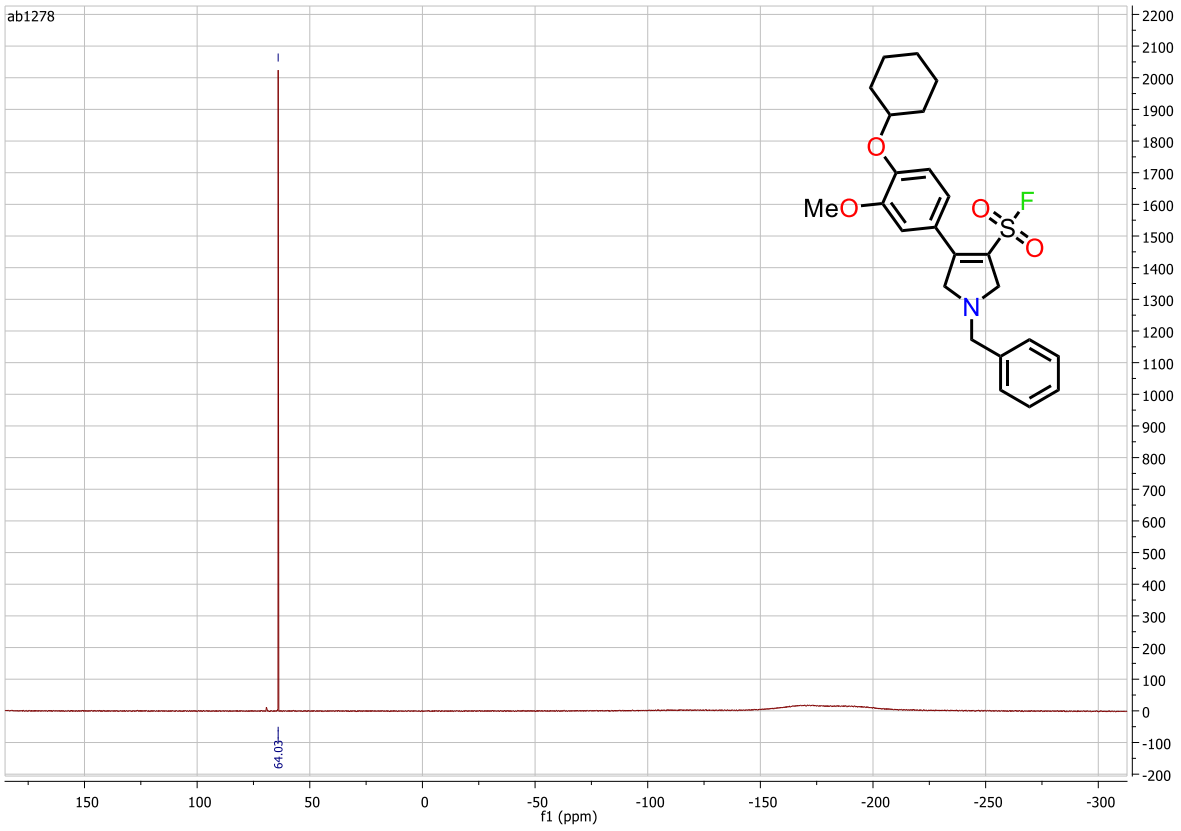


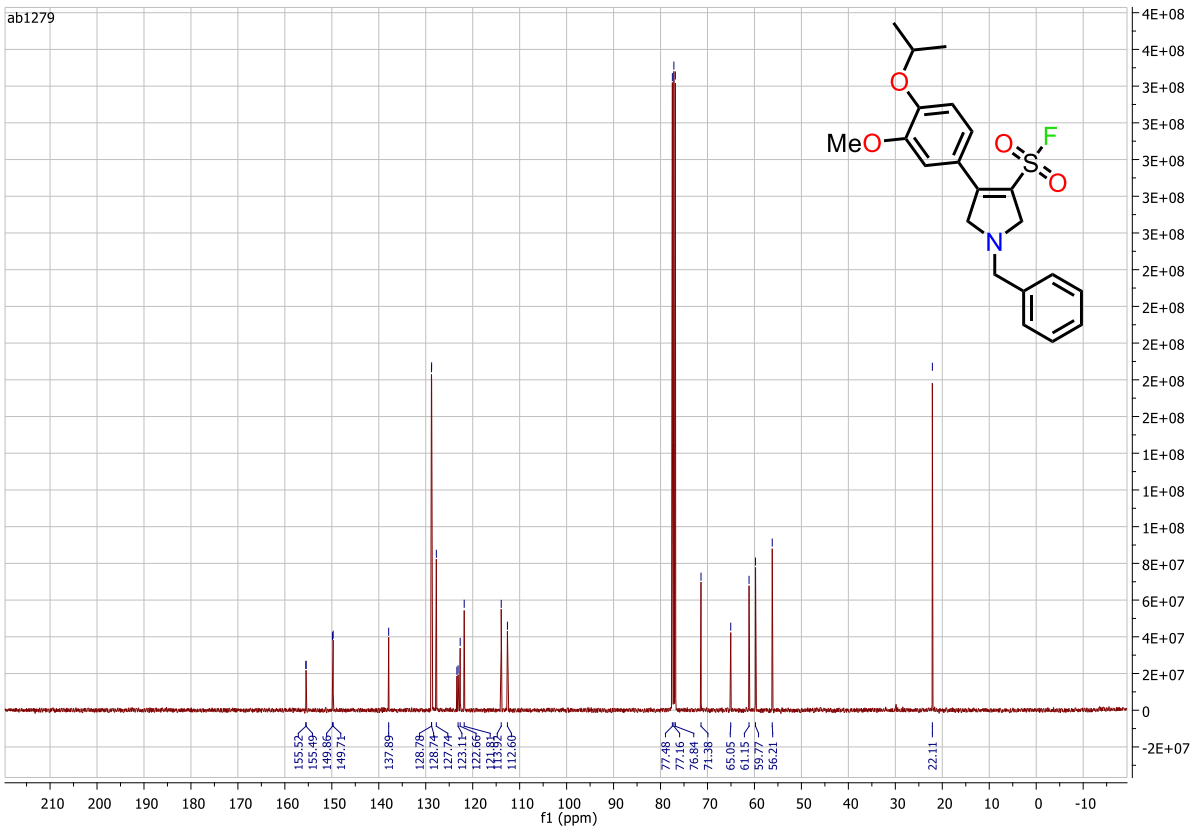
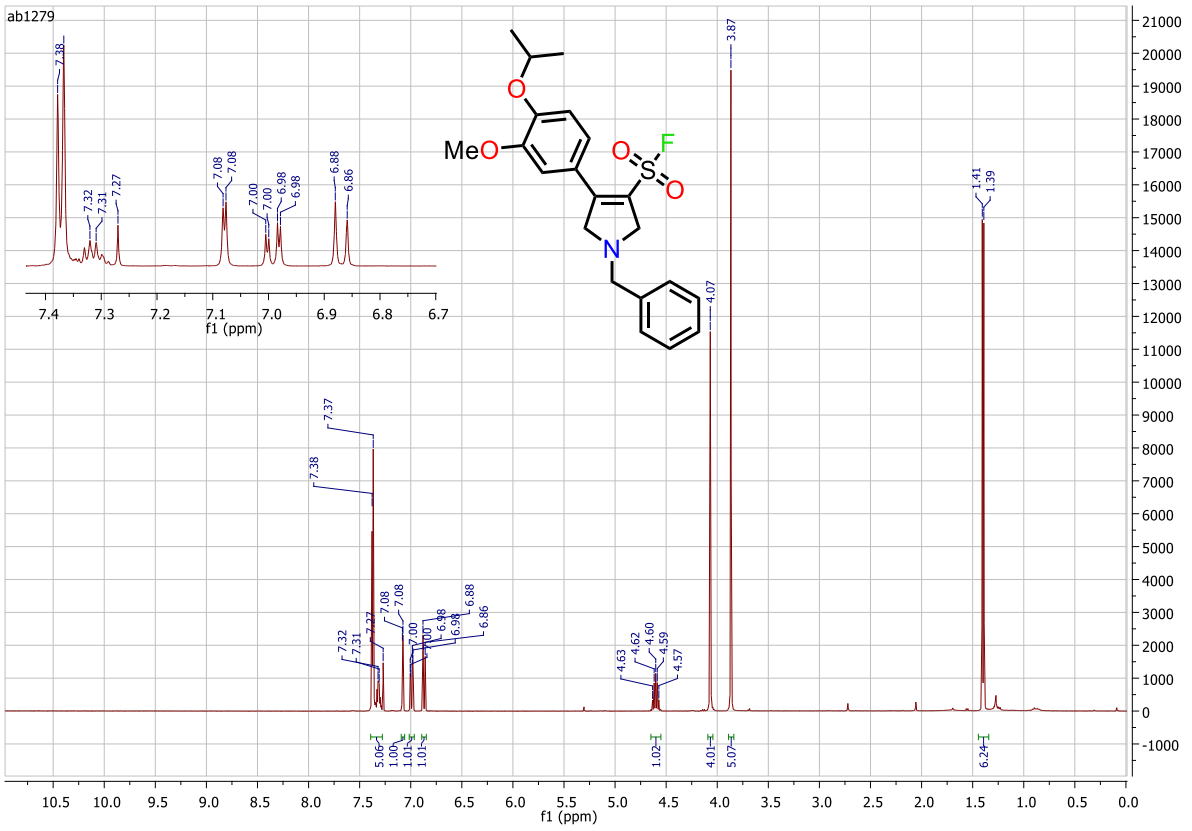




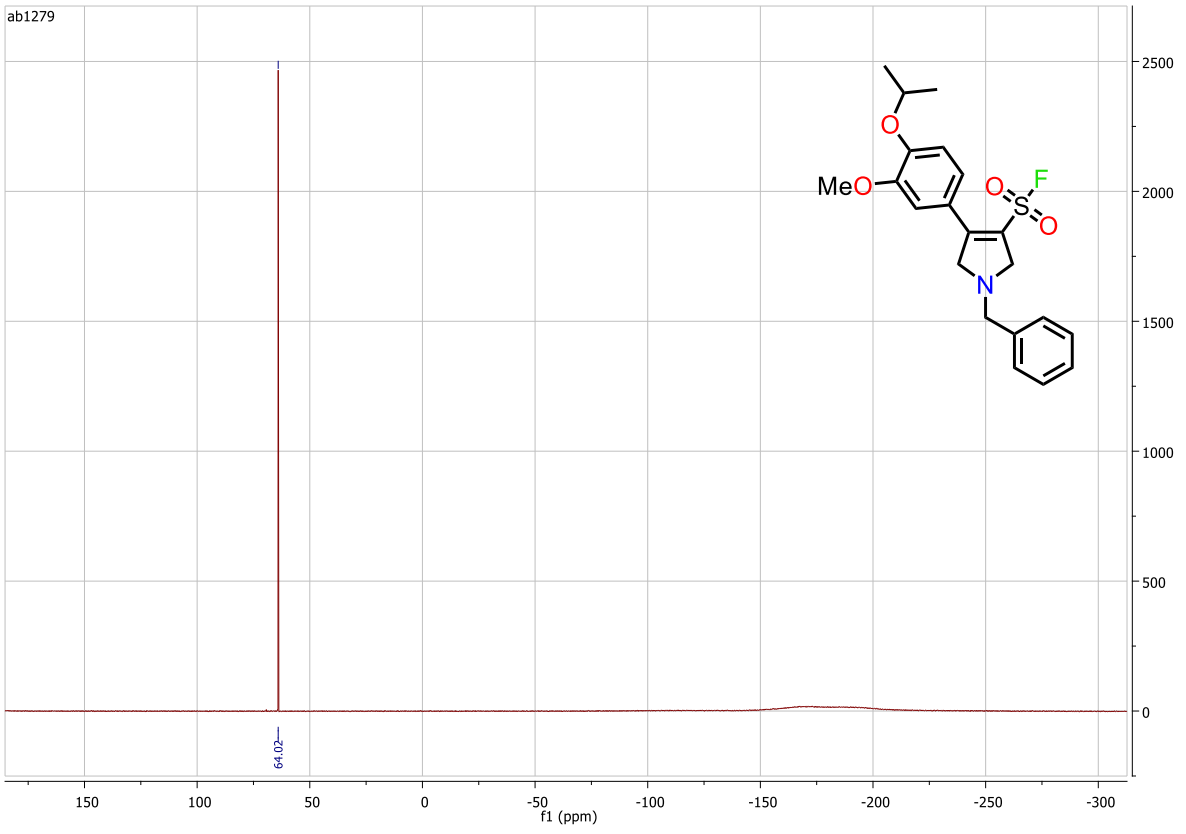


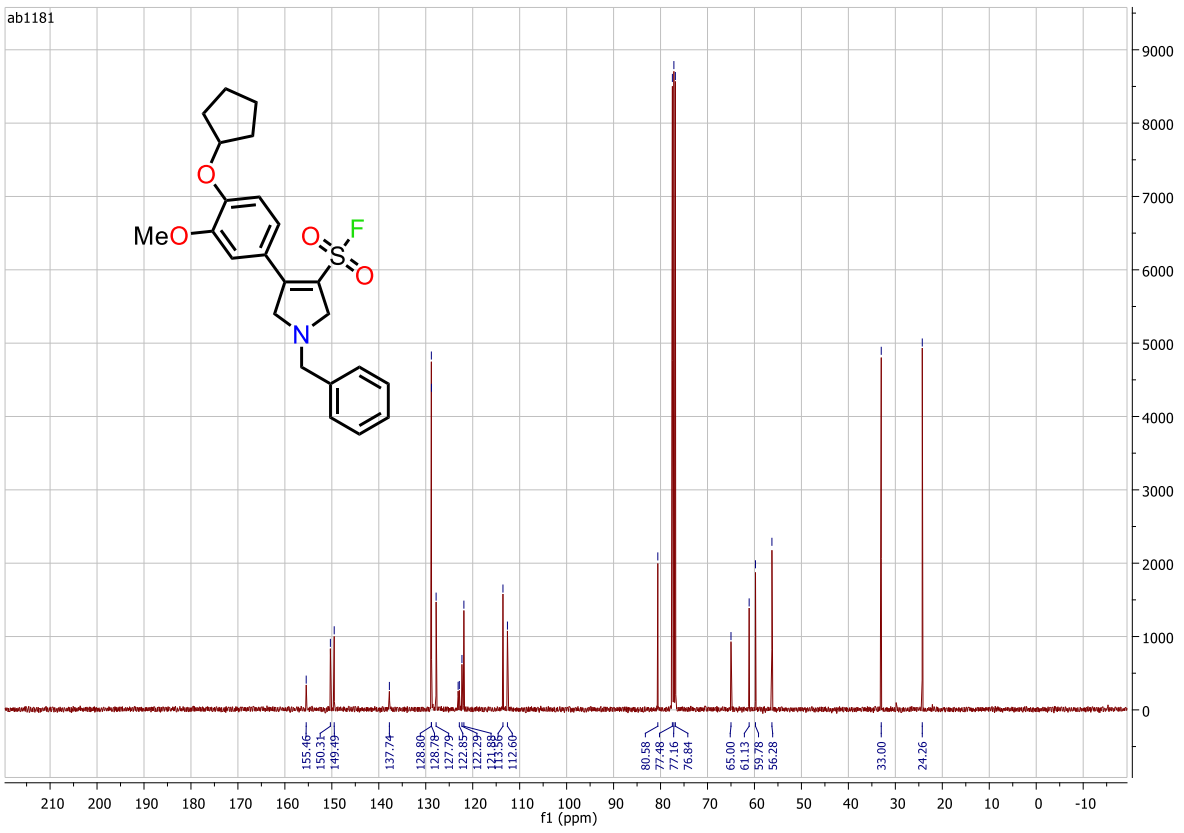
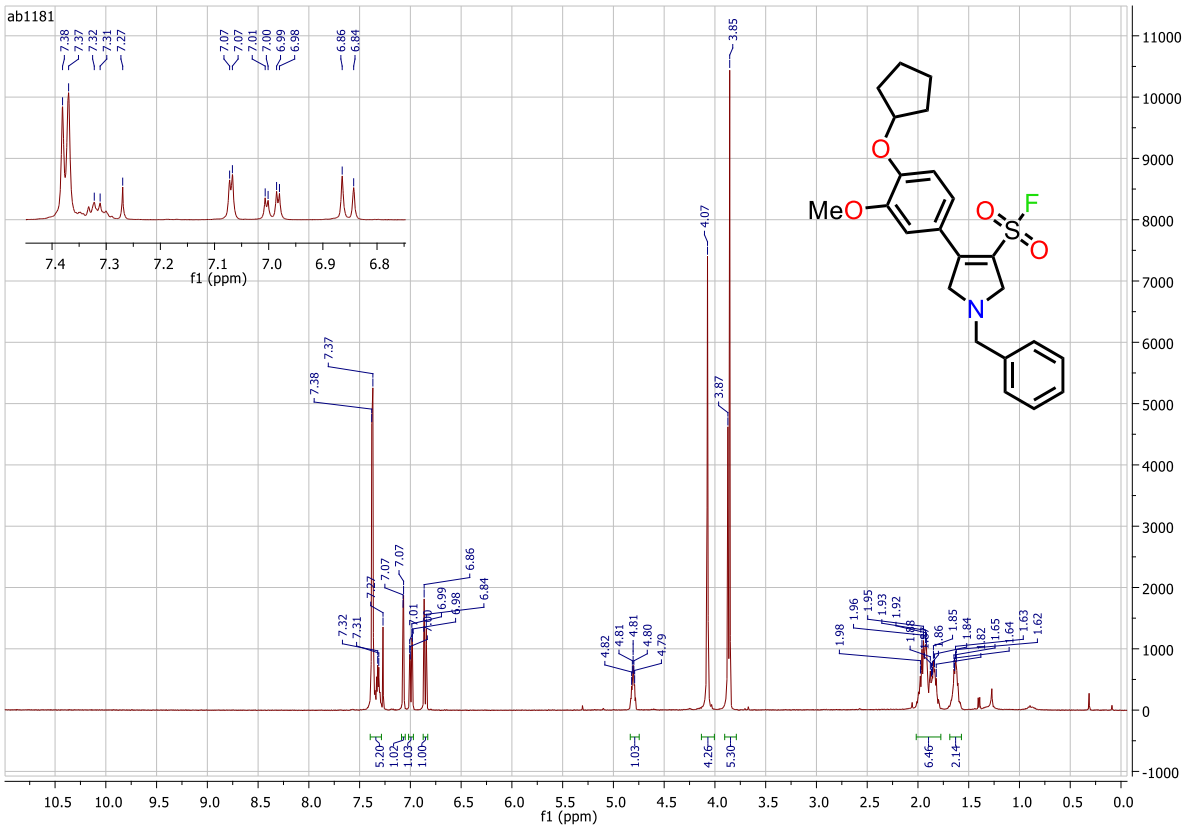


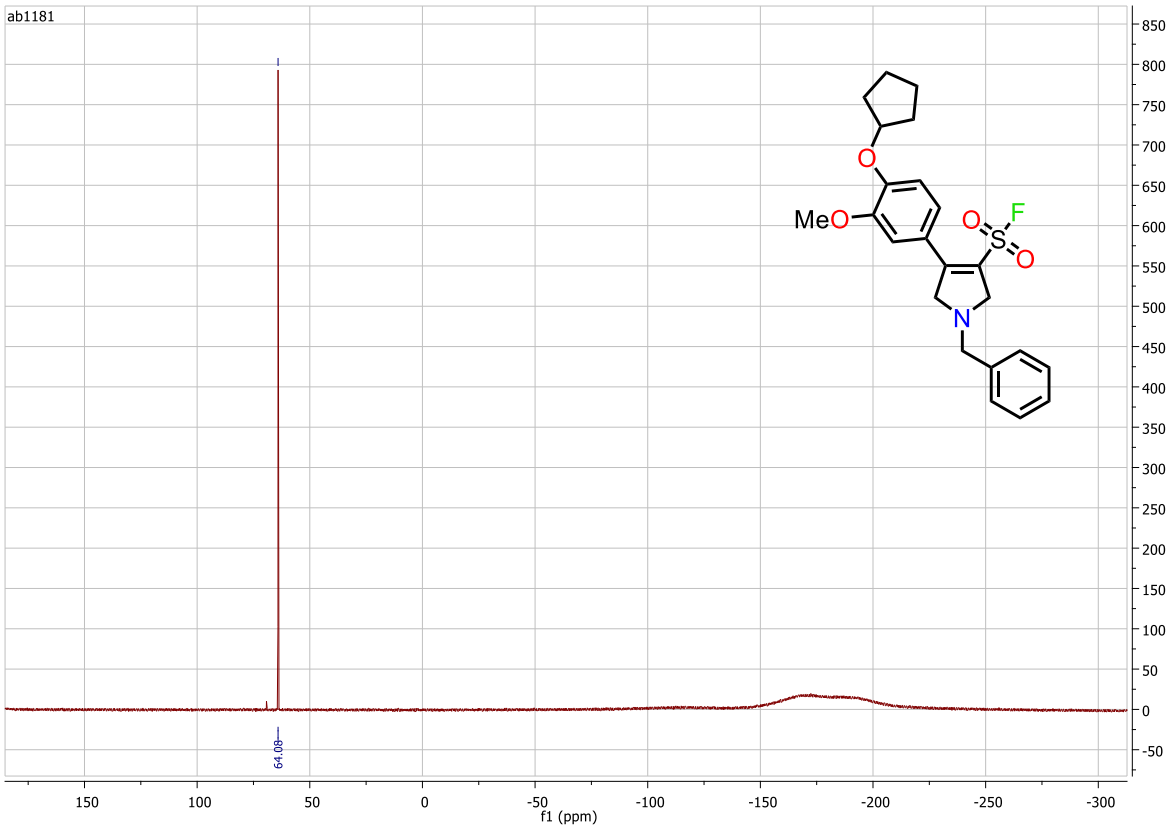


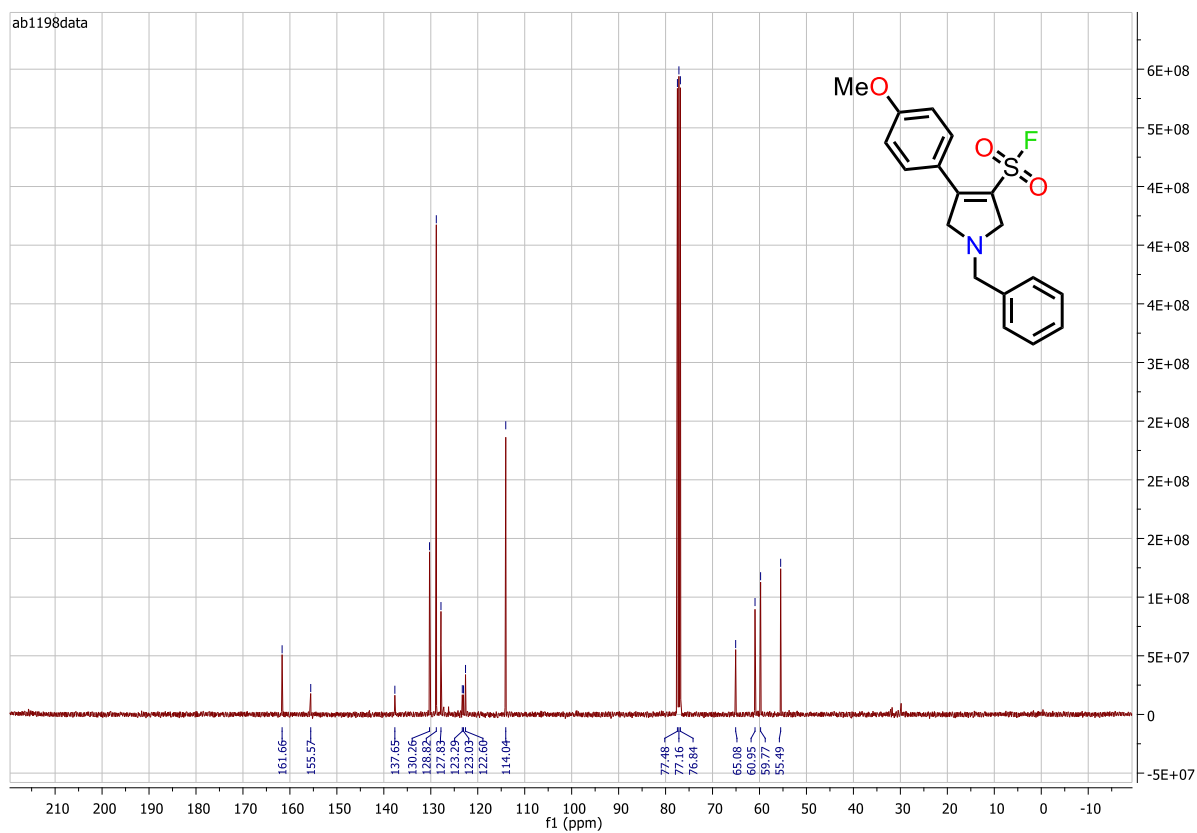
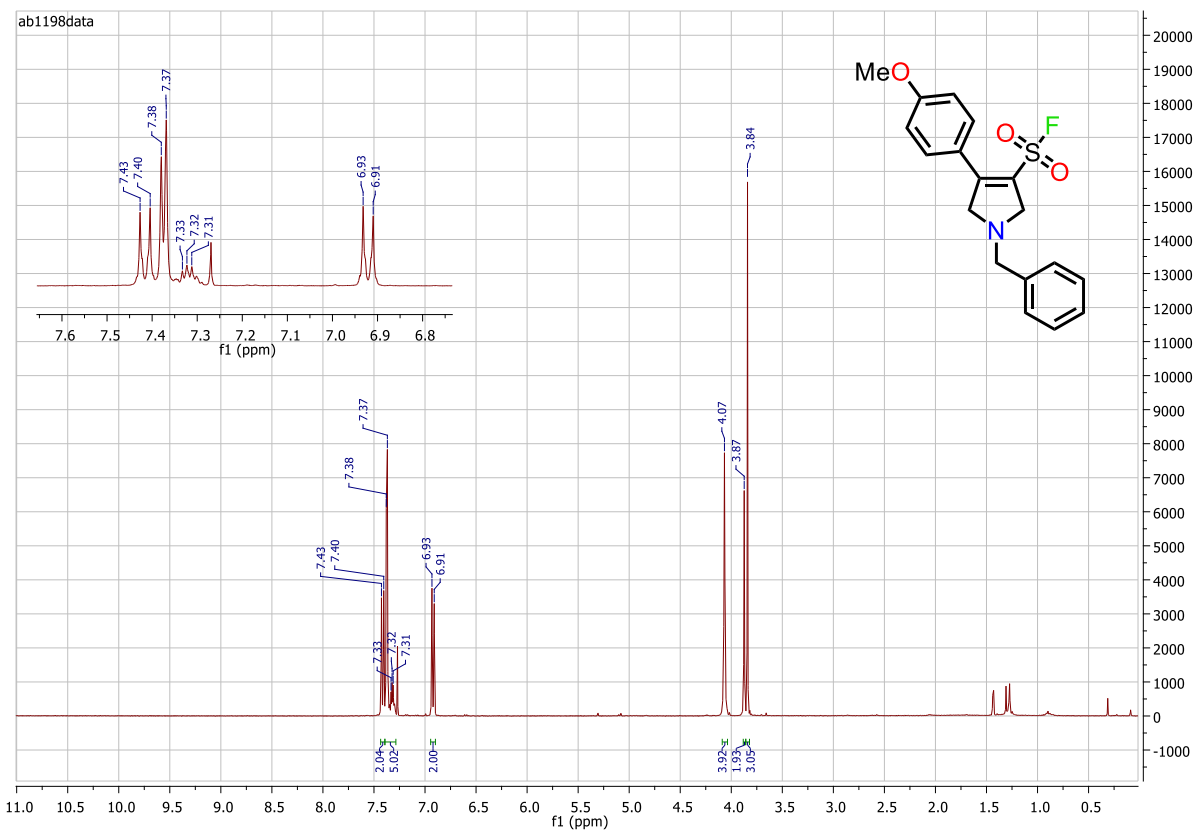


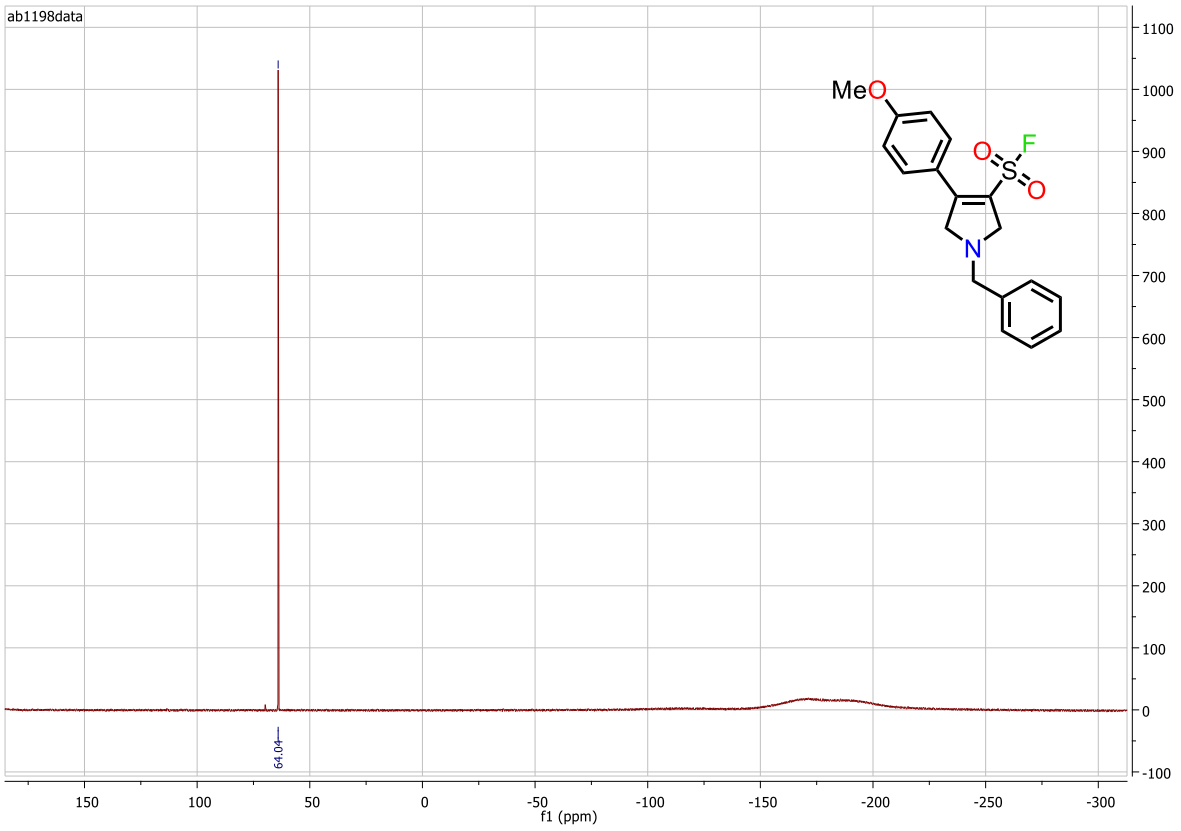
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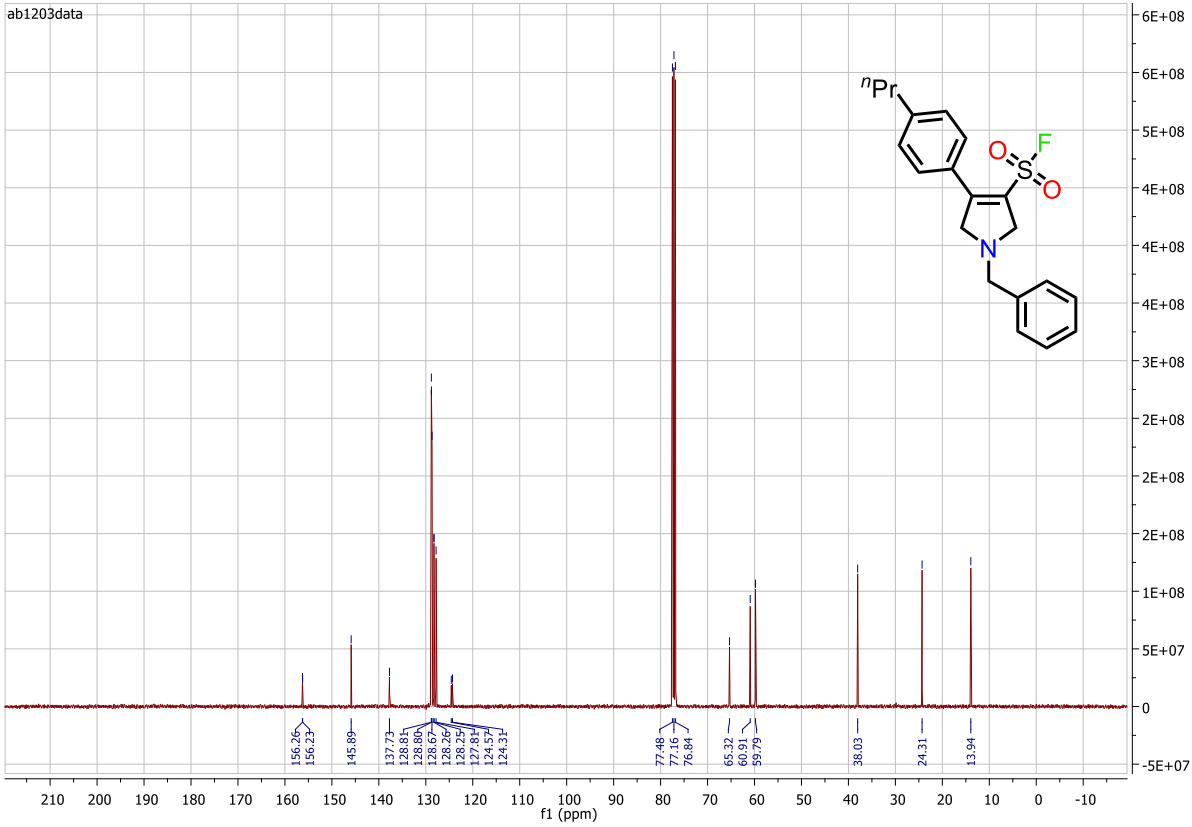
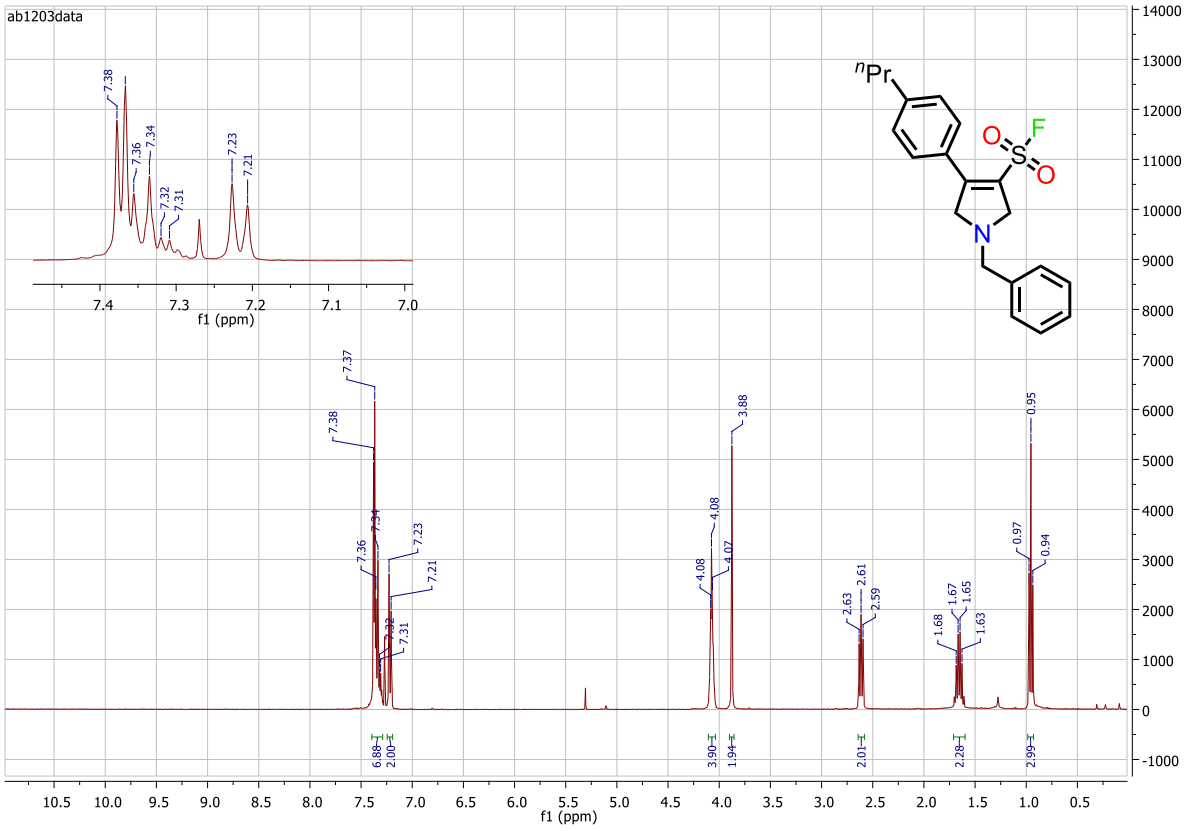


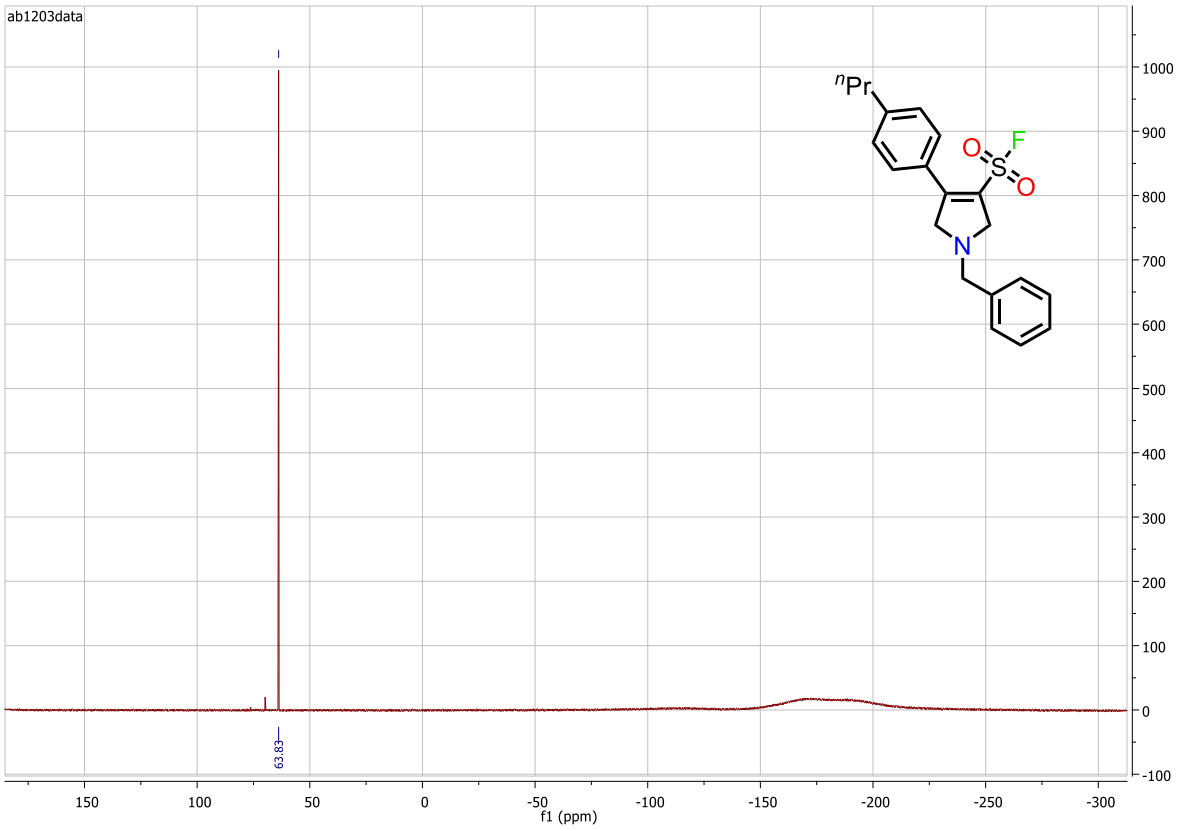


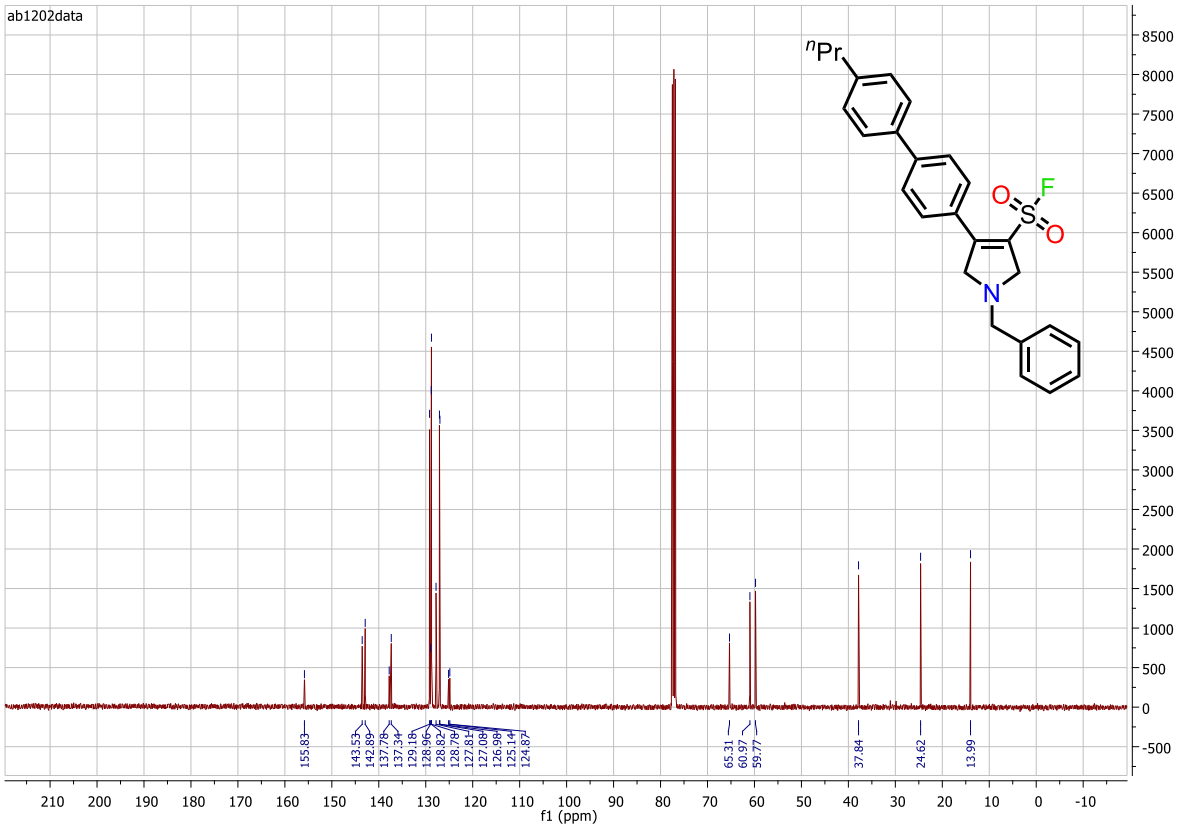
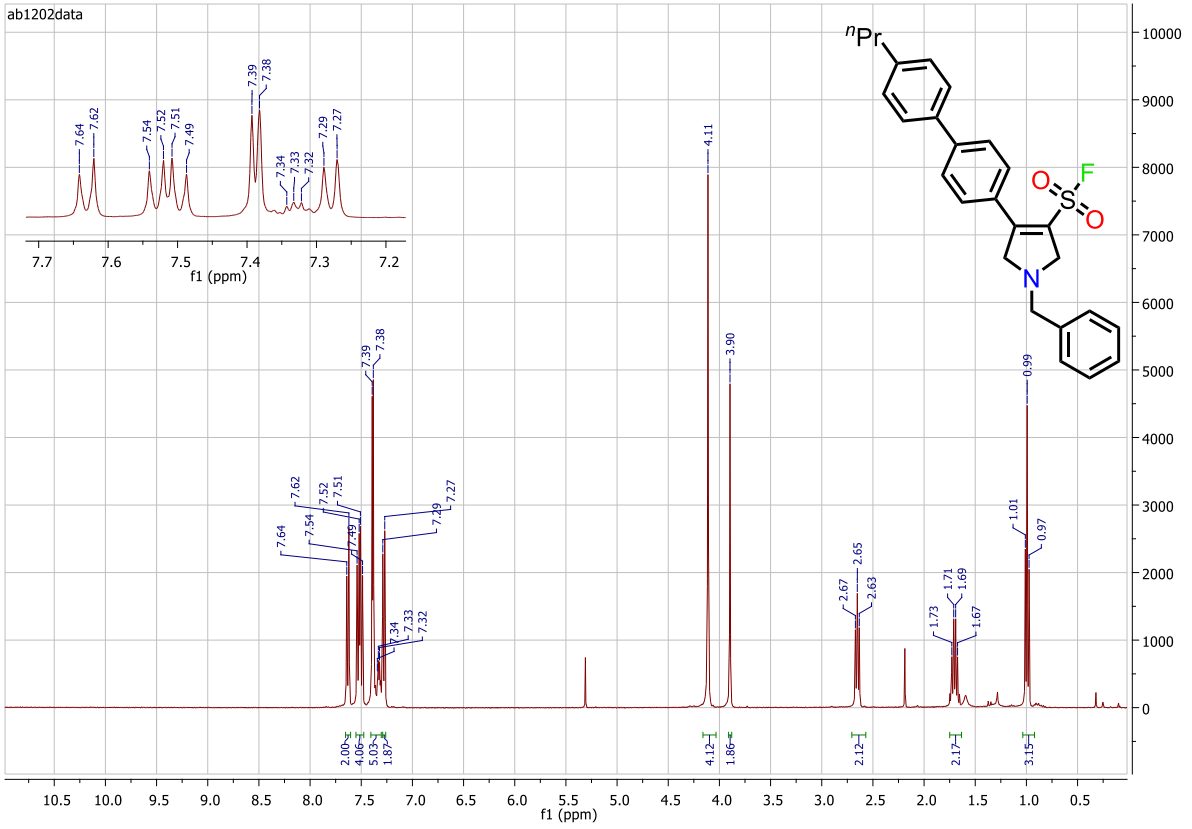


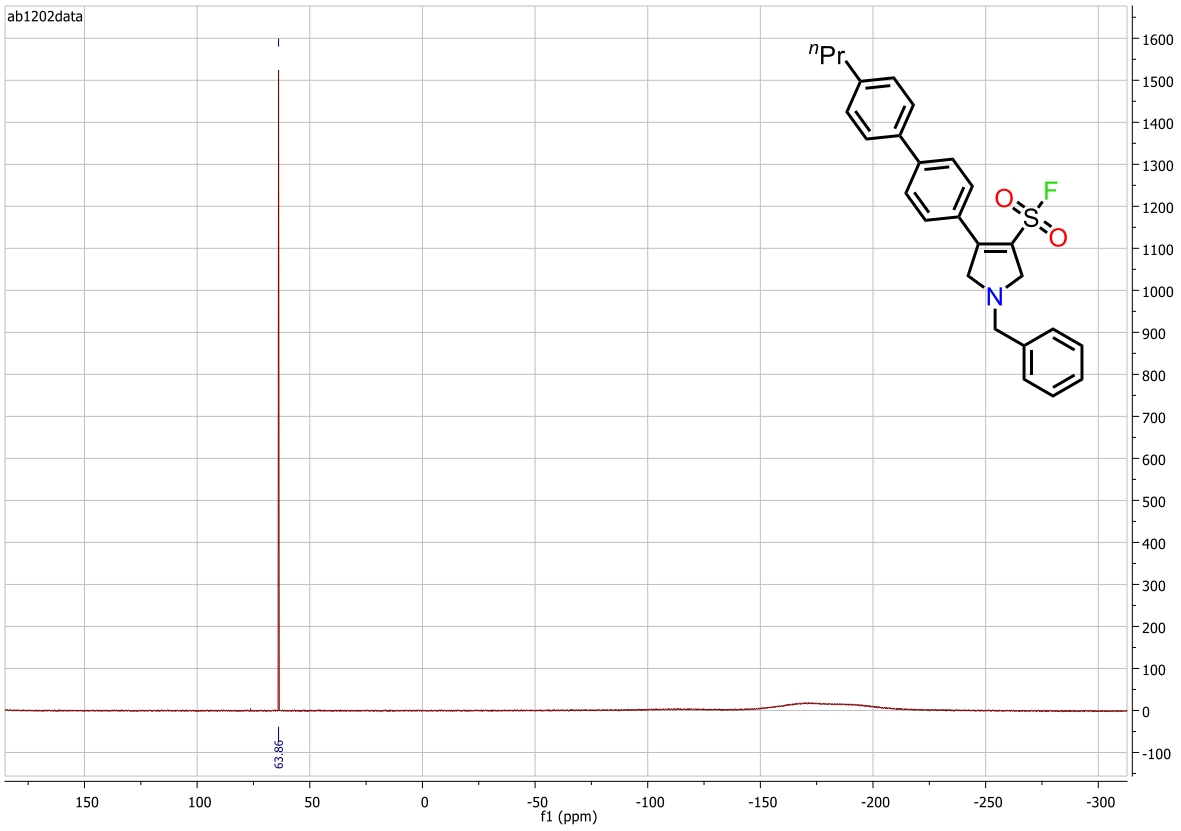


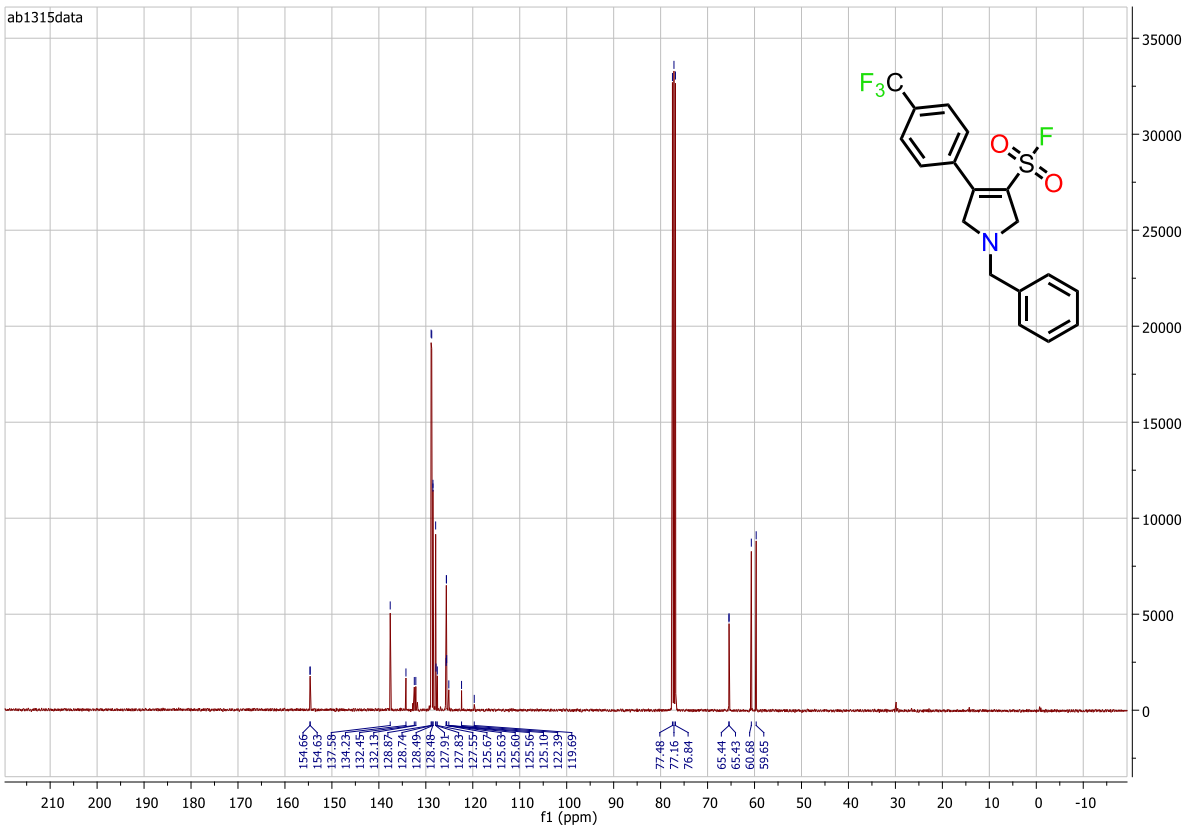
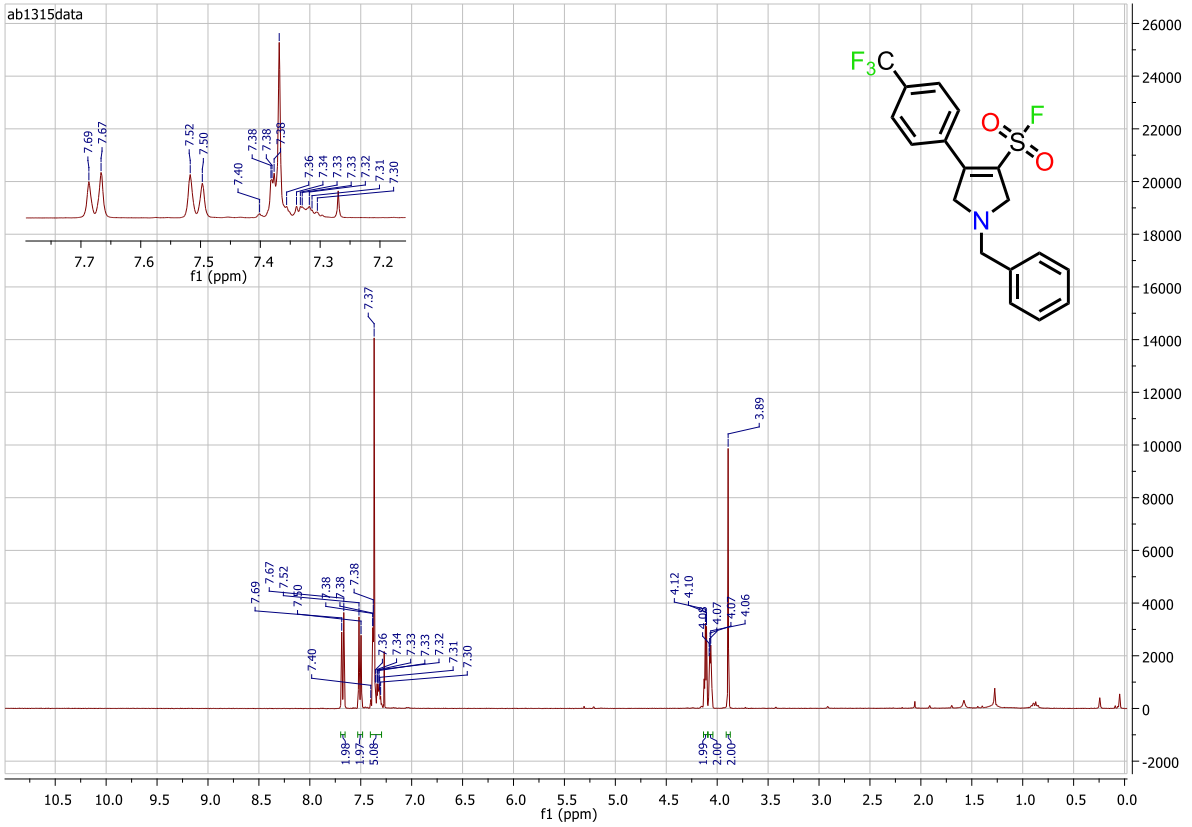


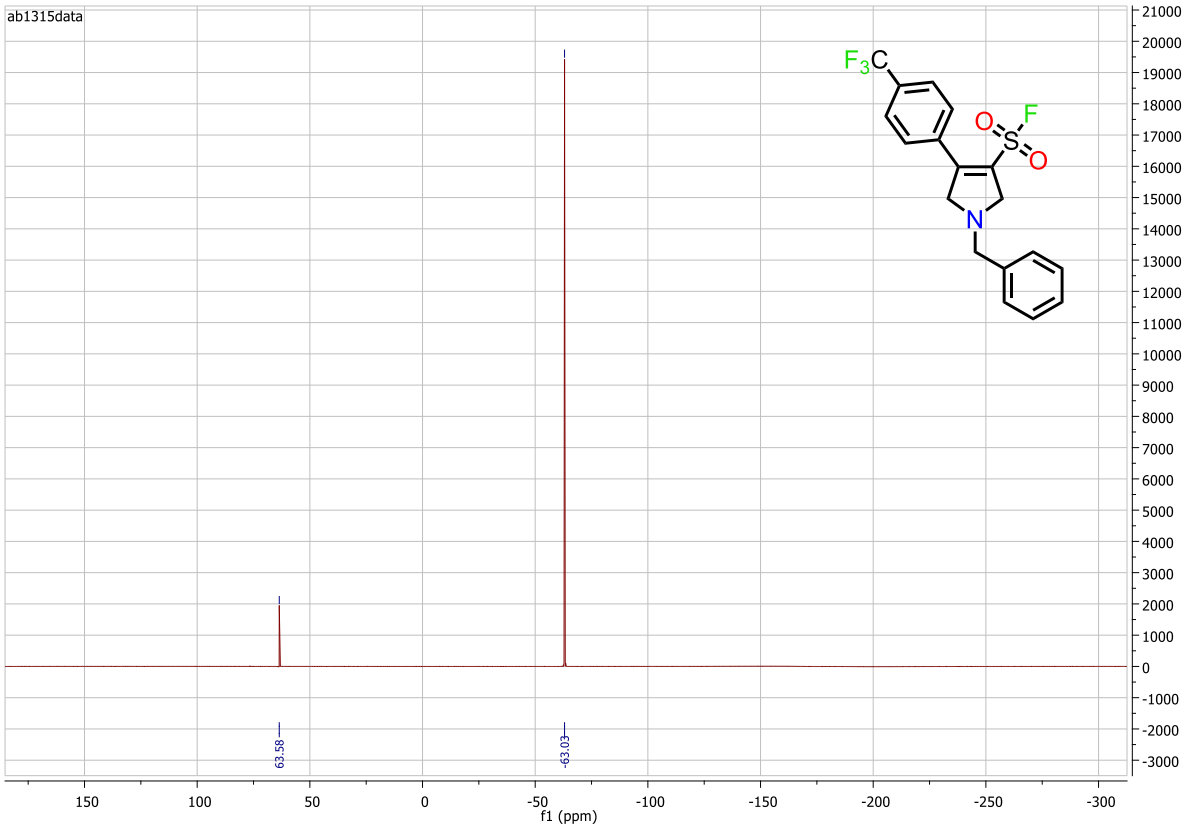


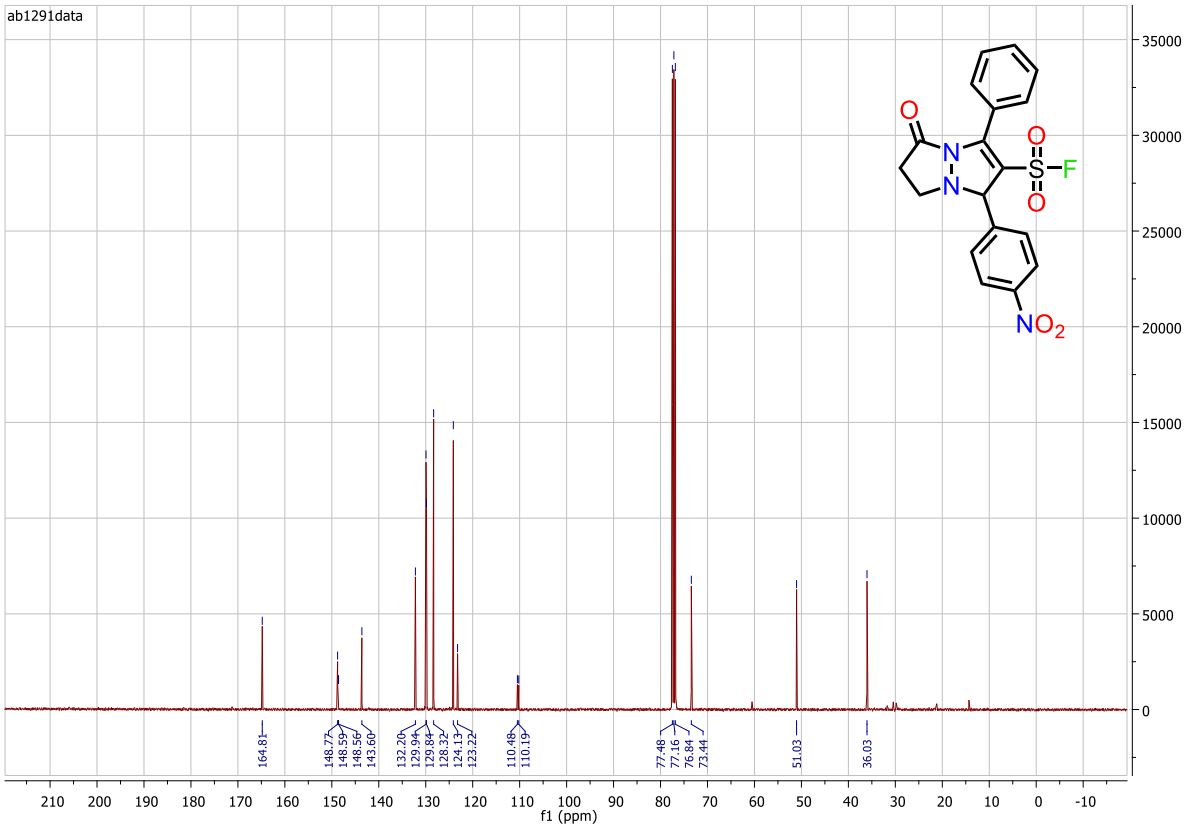
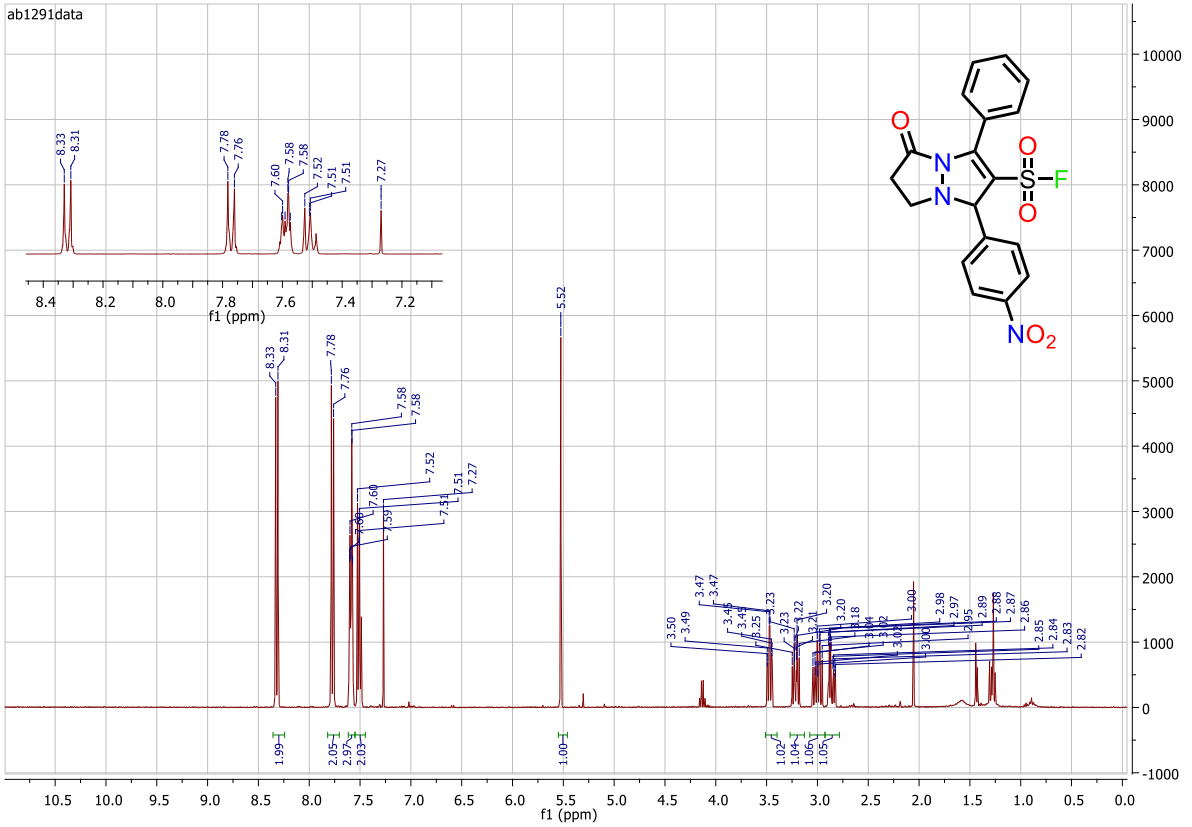


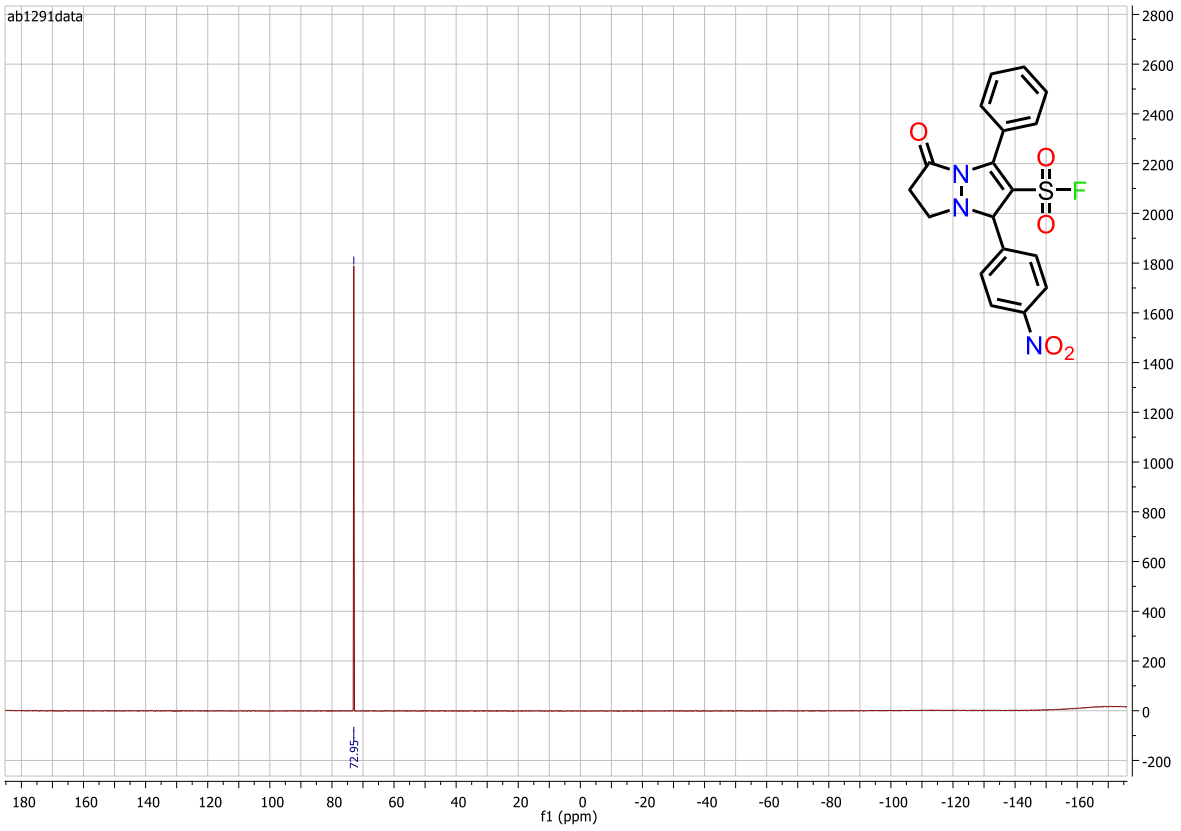


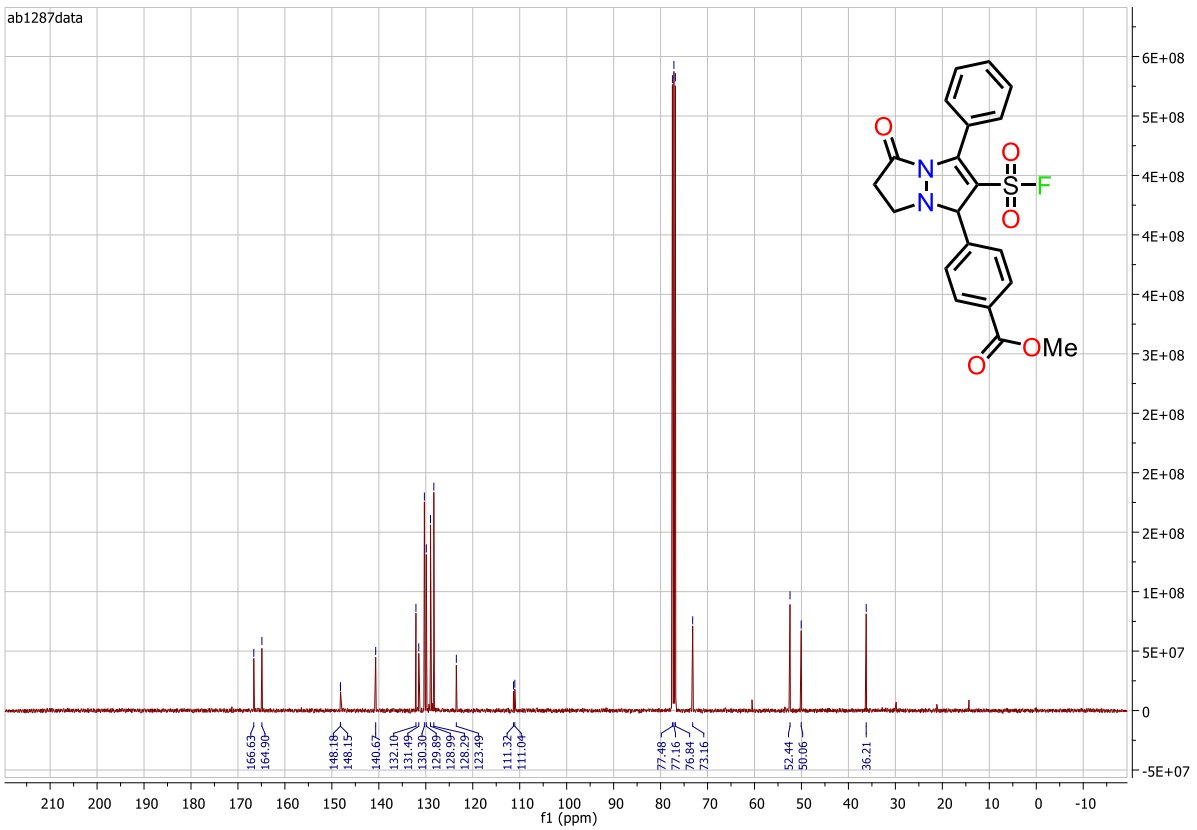
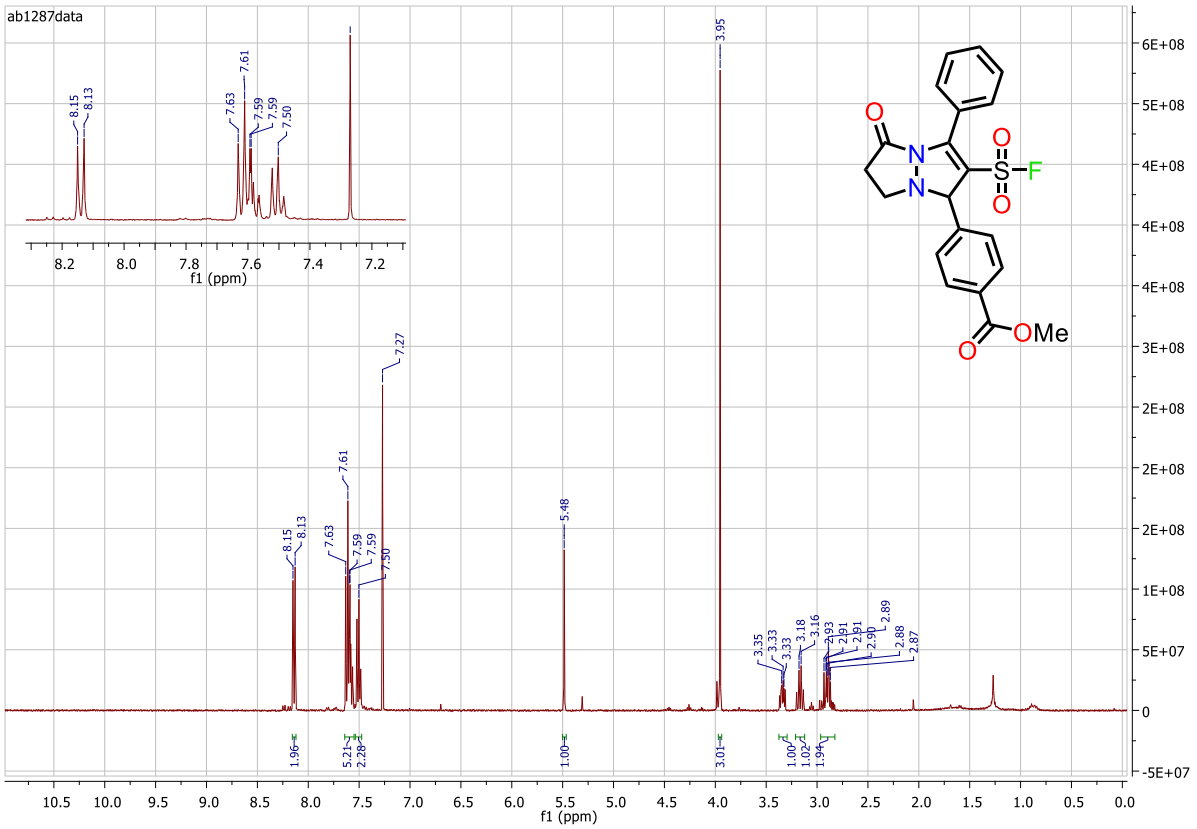


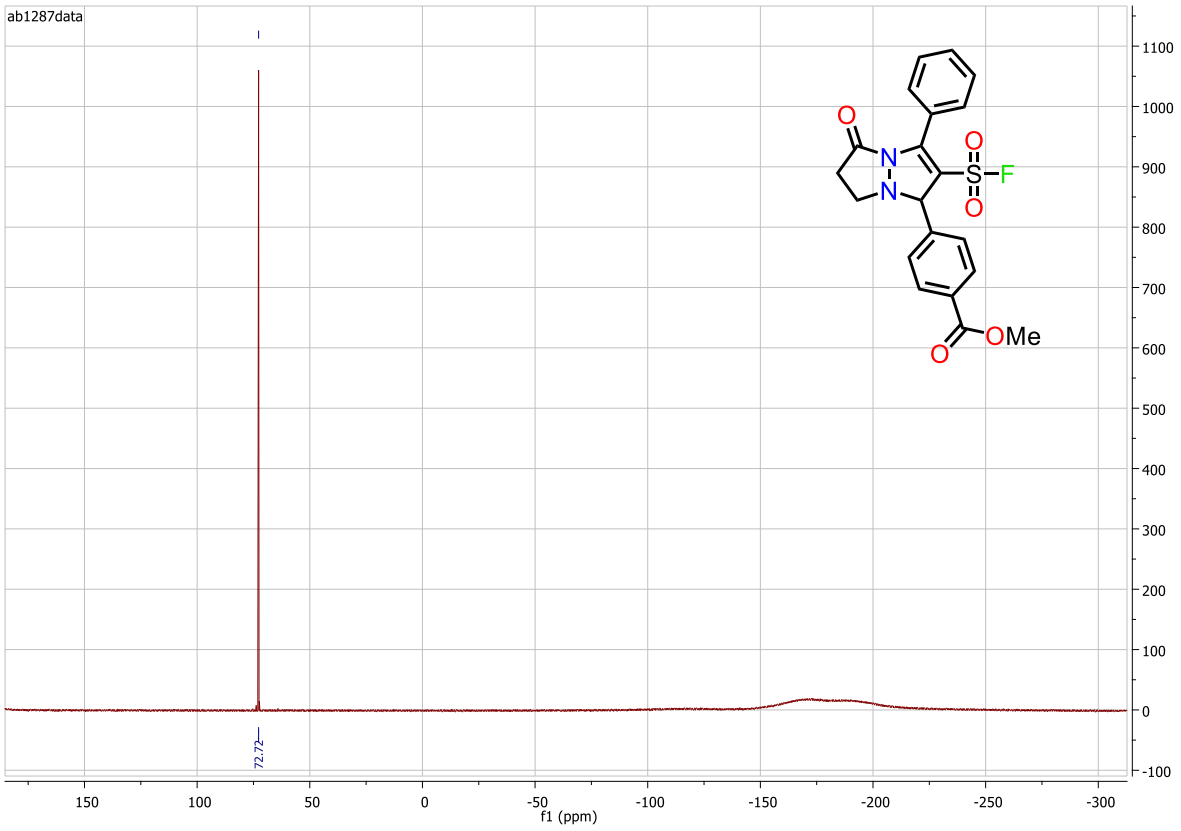


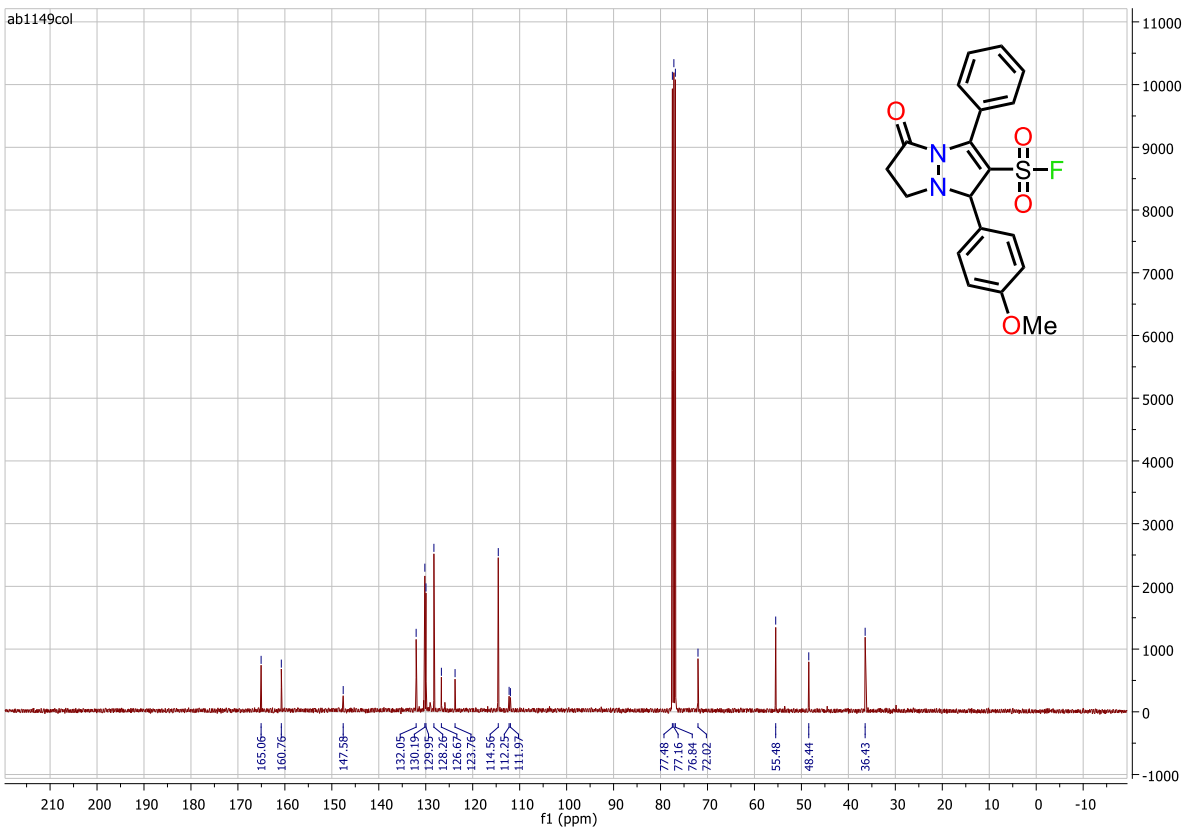
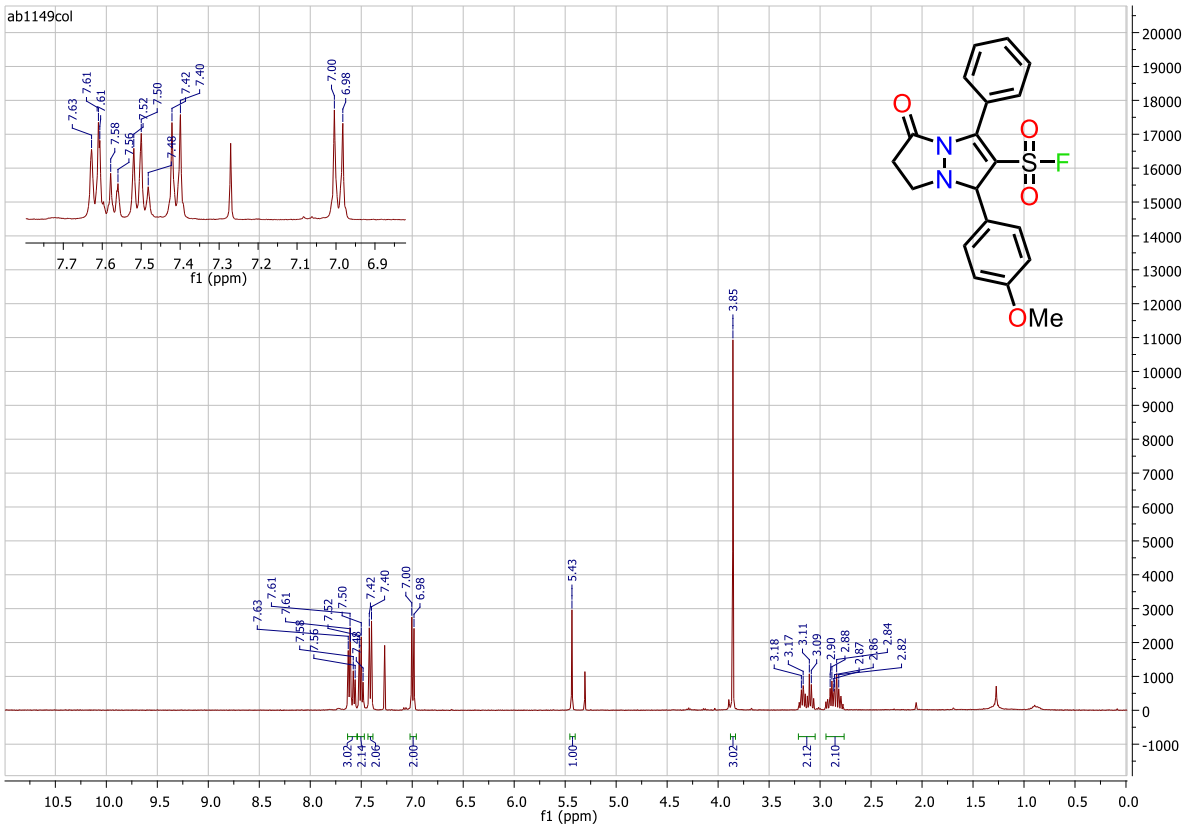




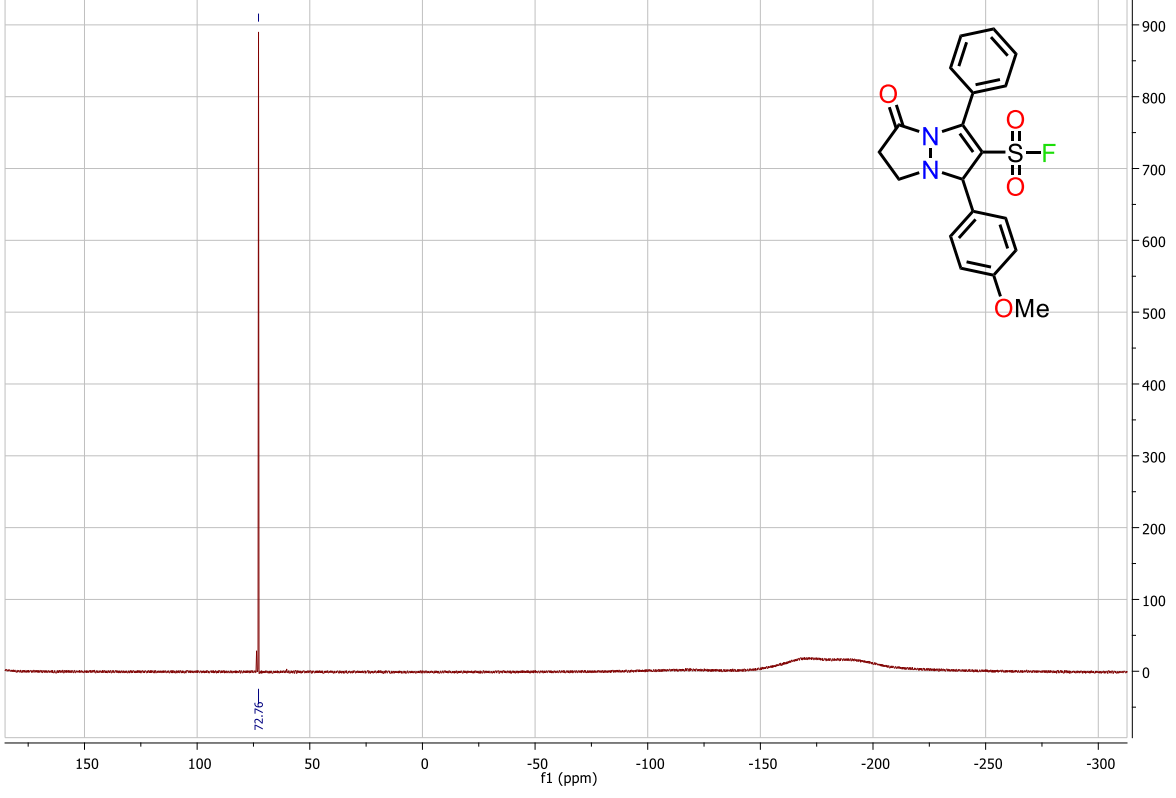


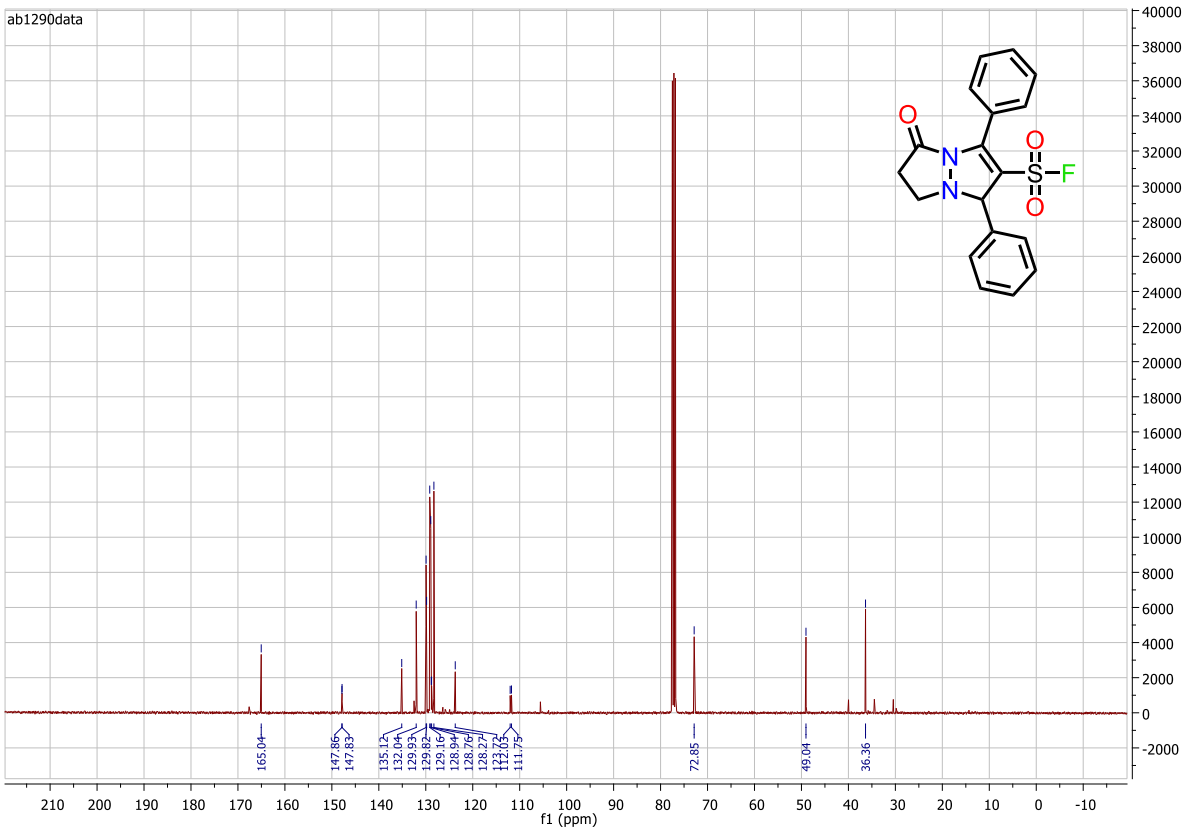
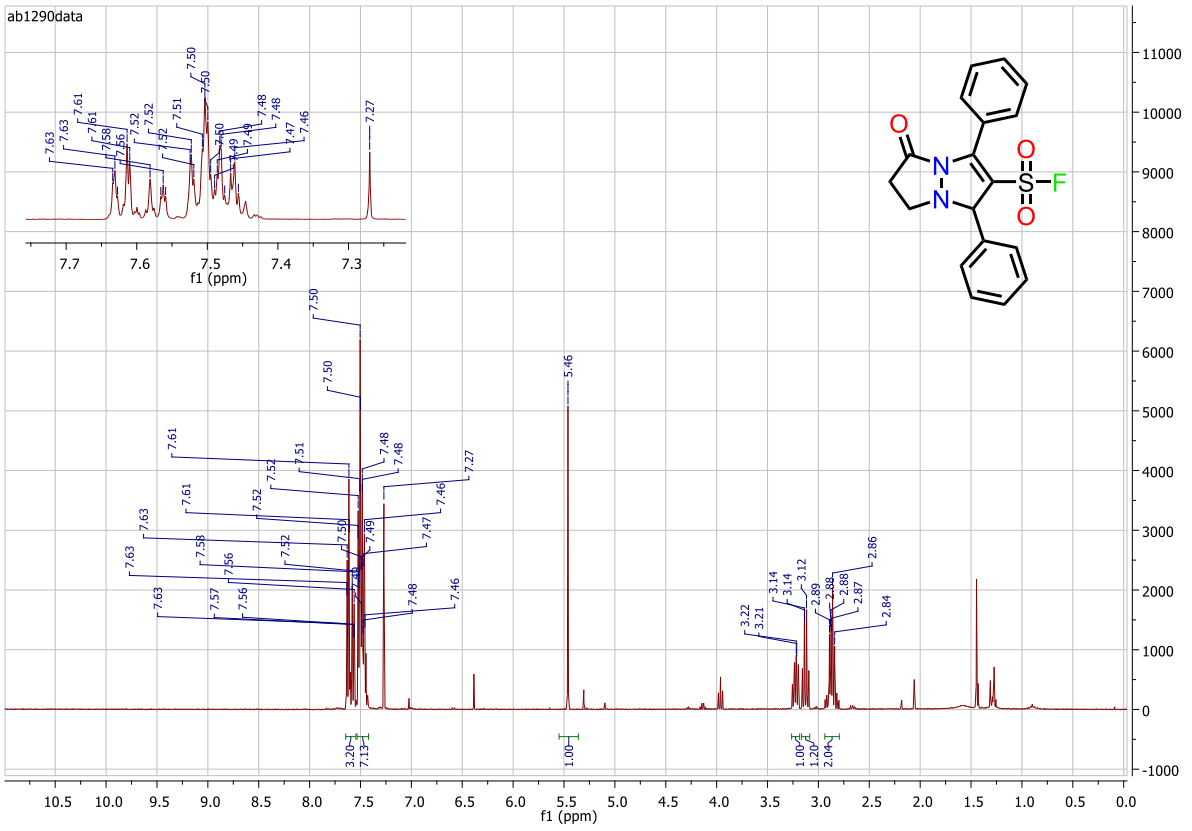




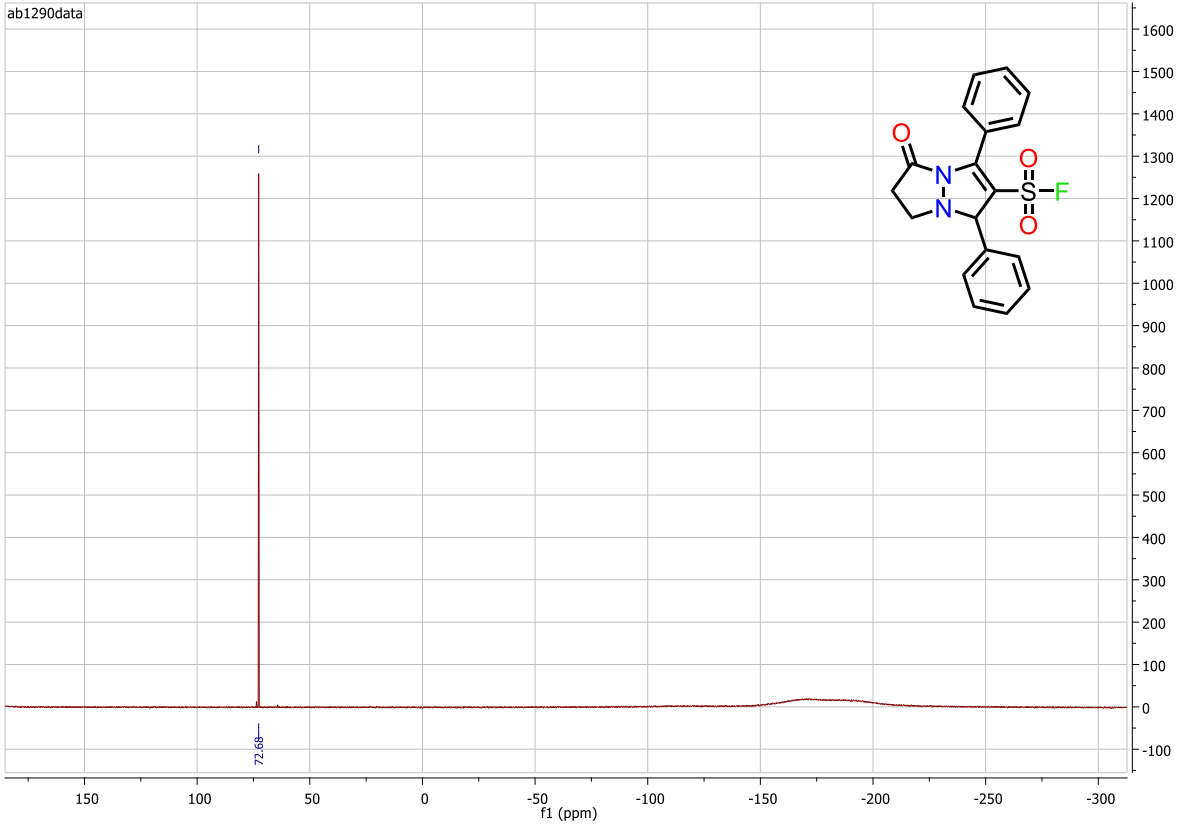


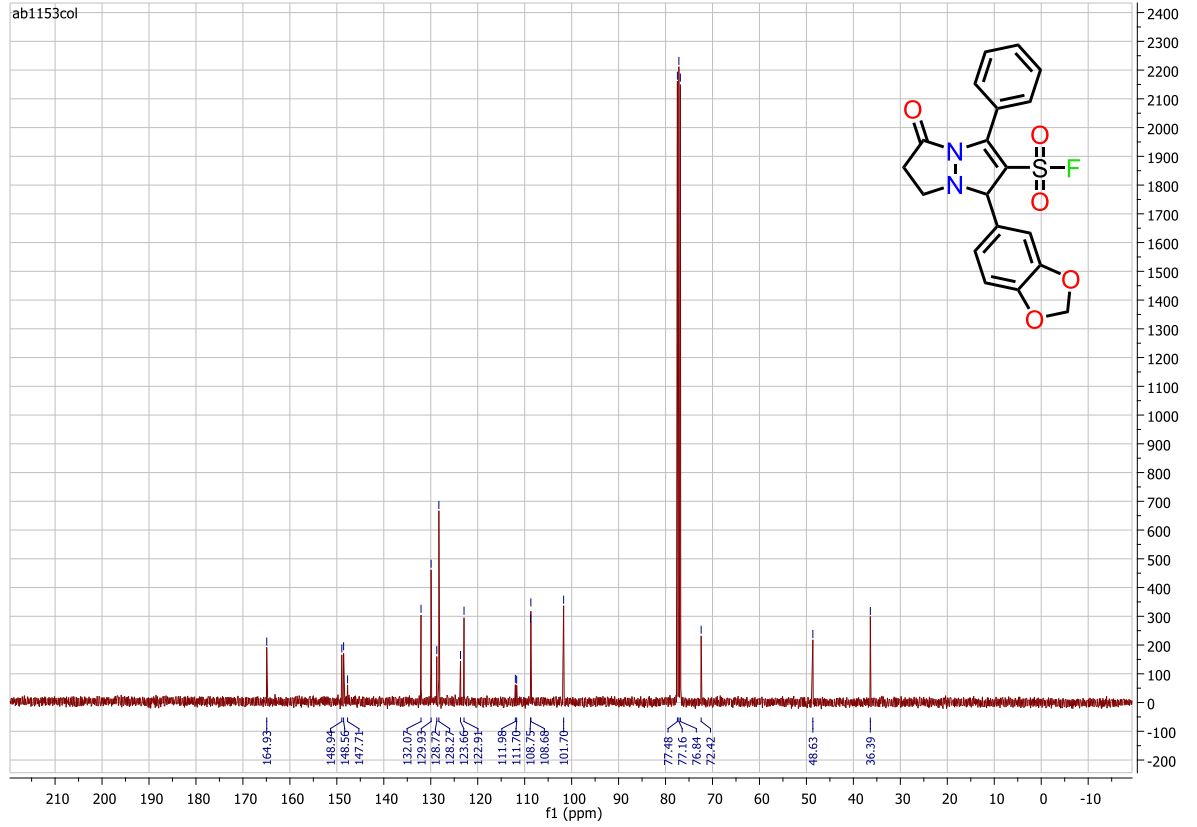
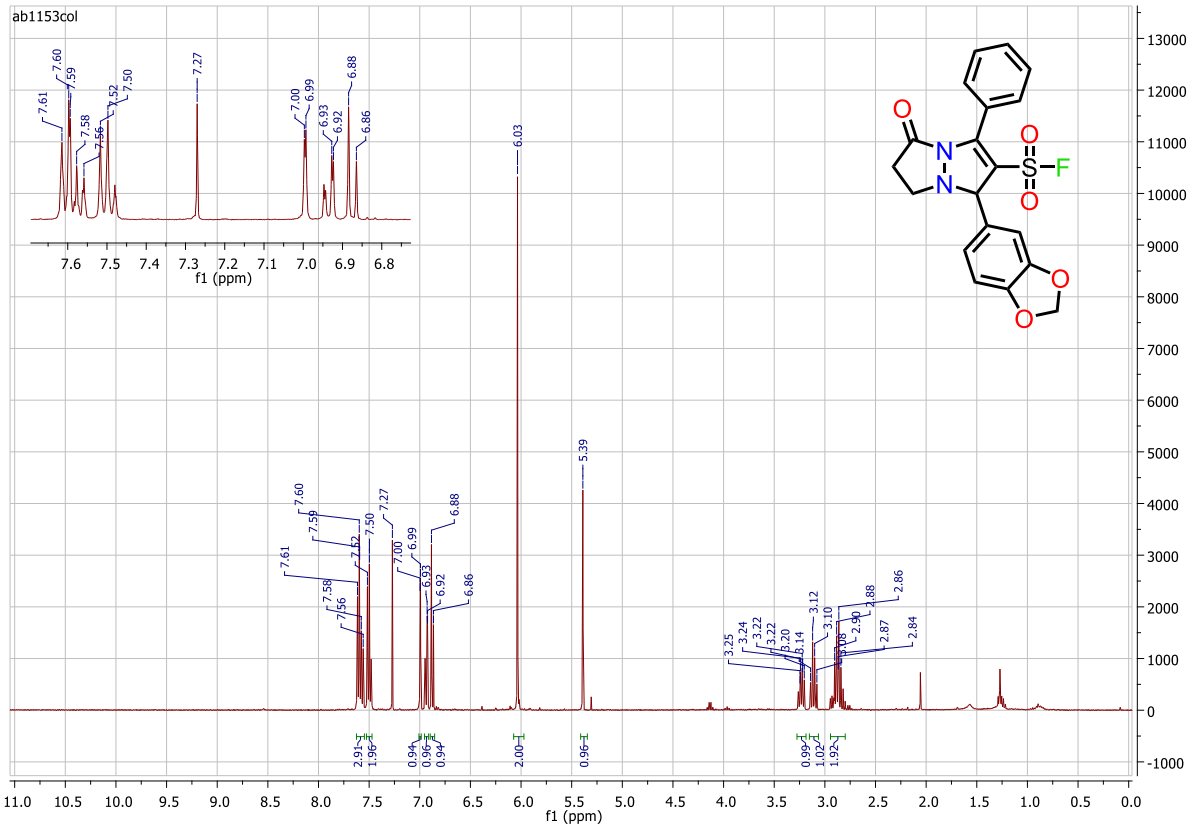
ab1149col



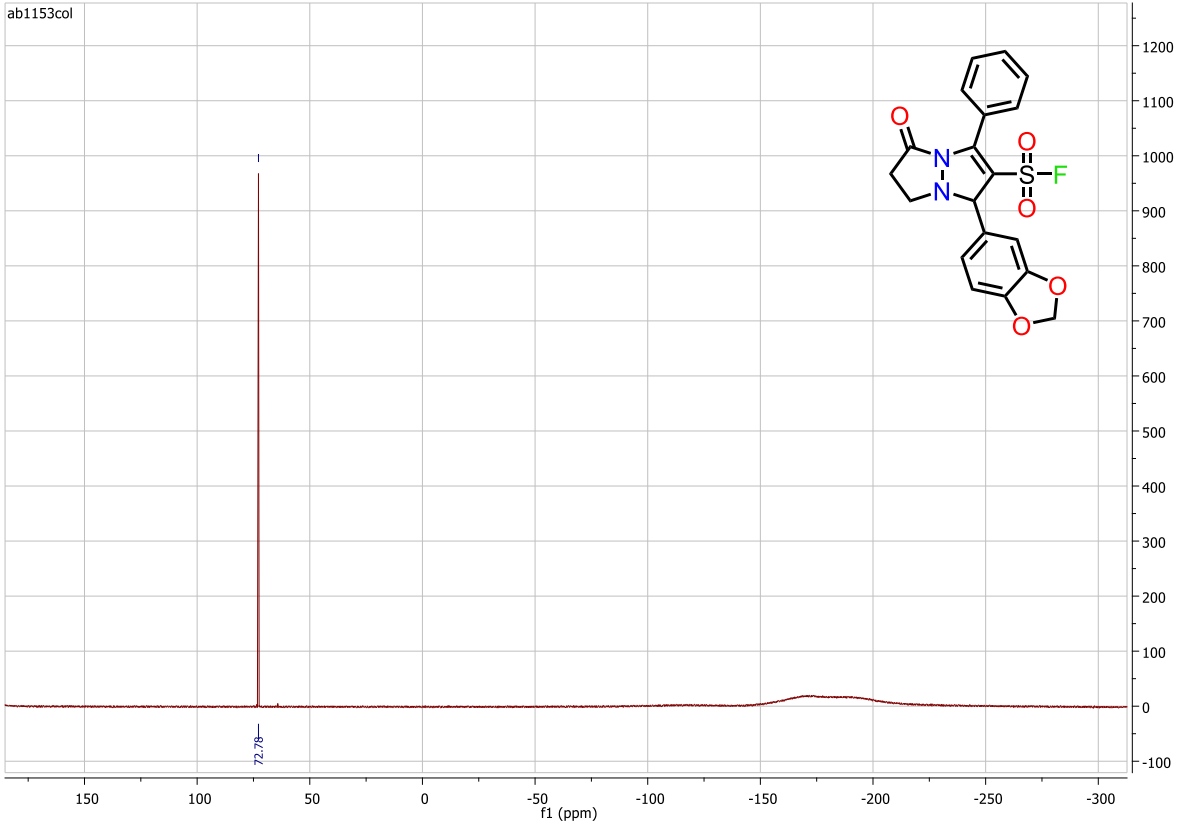


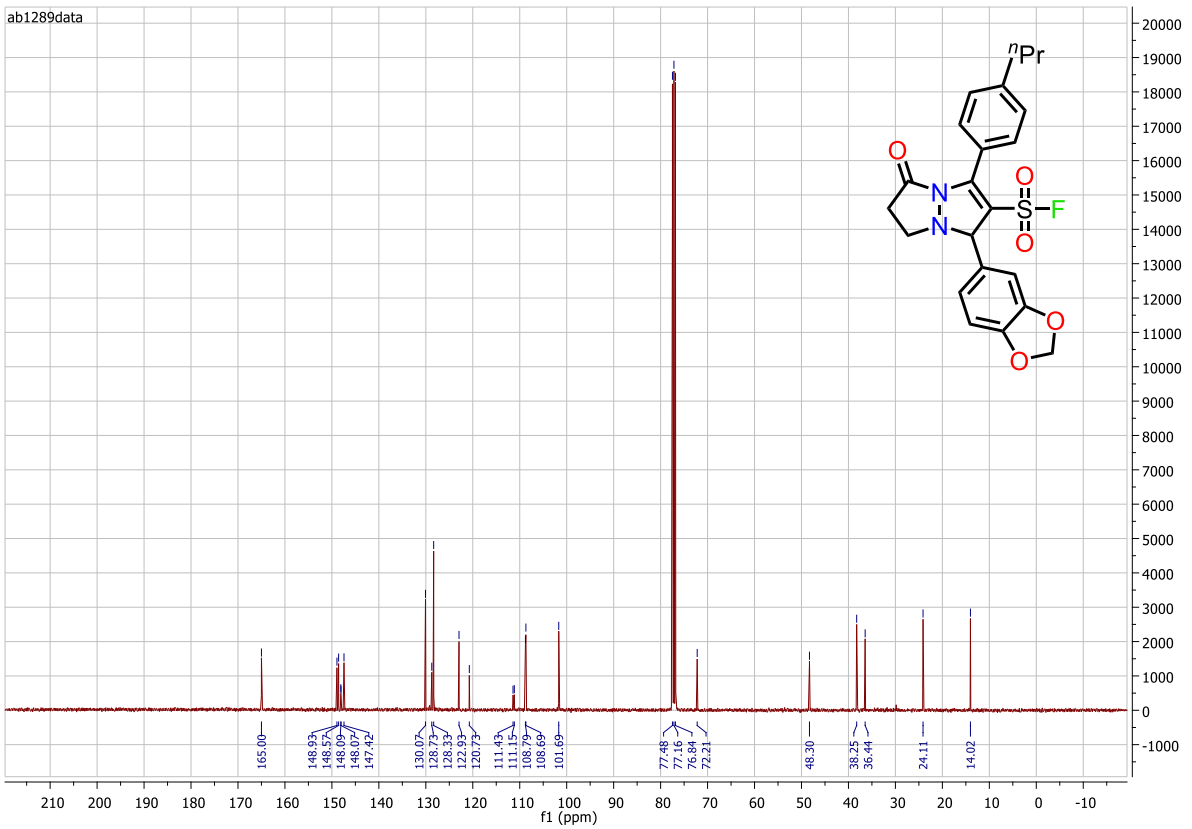
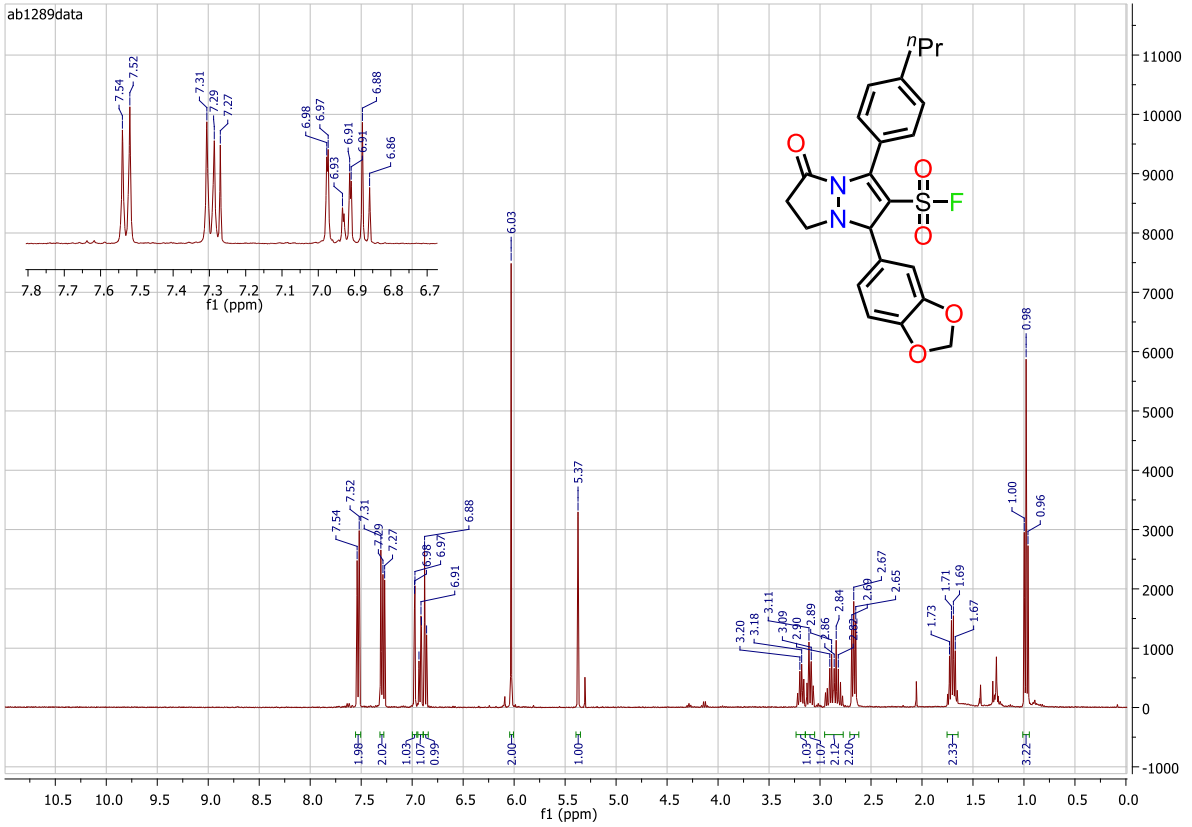
ab1290data



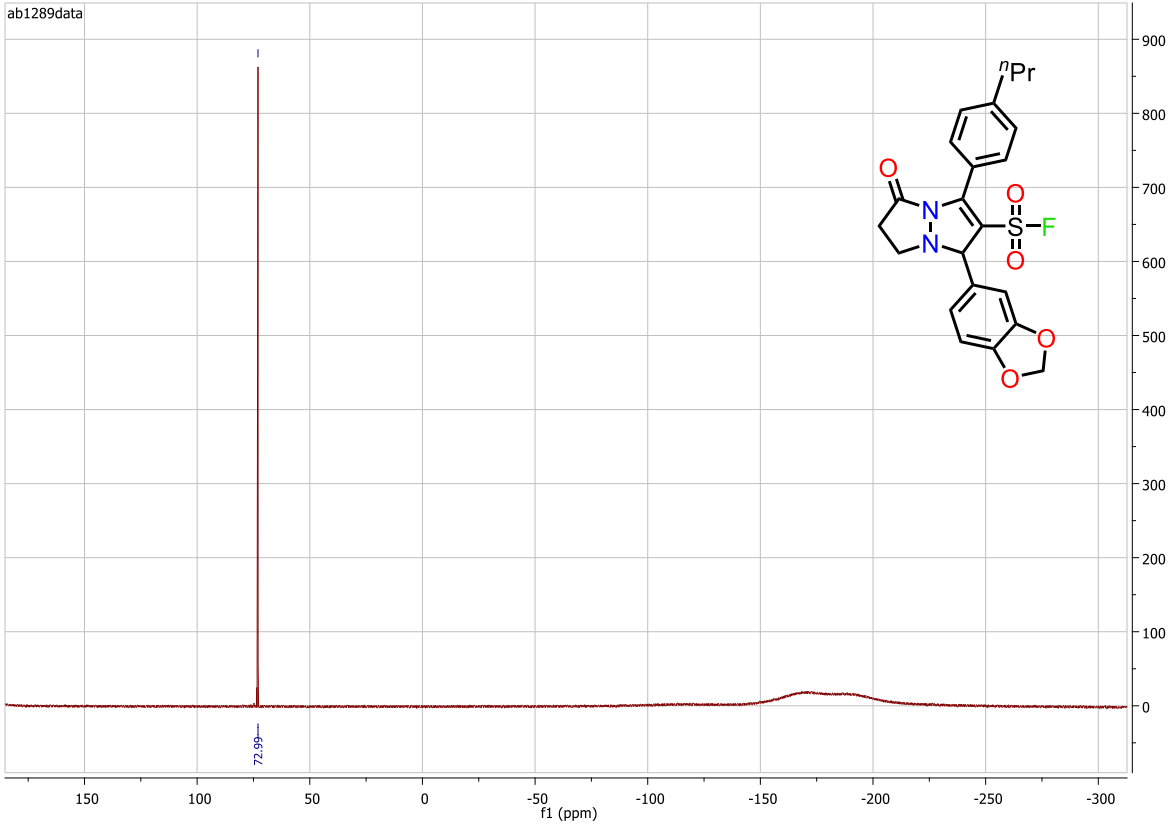


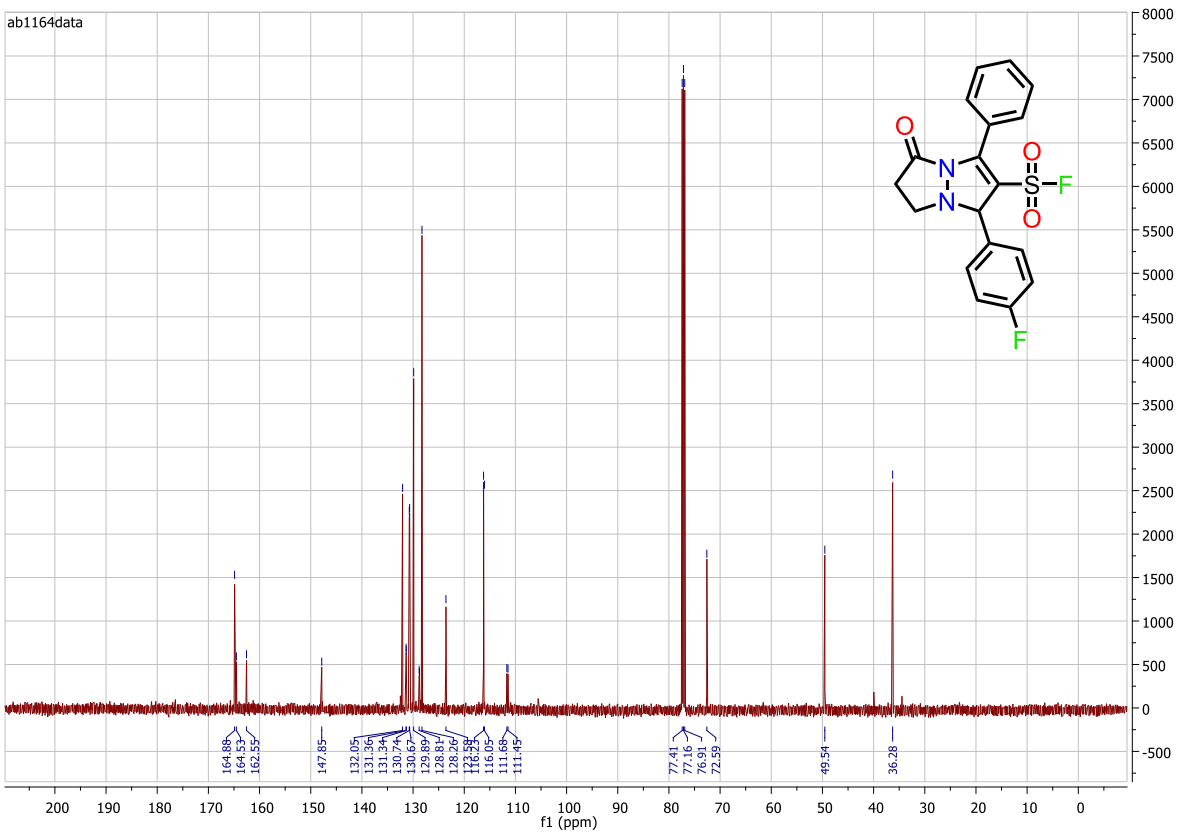
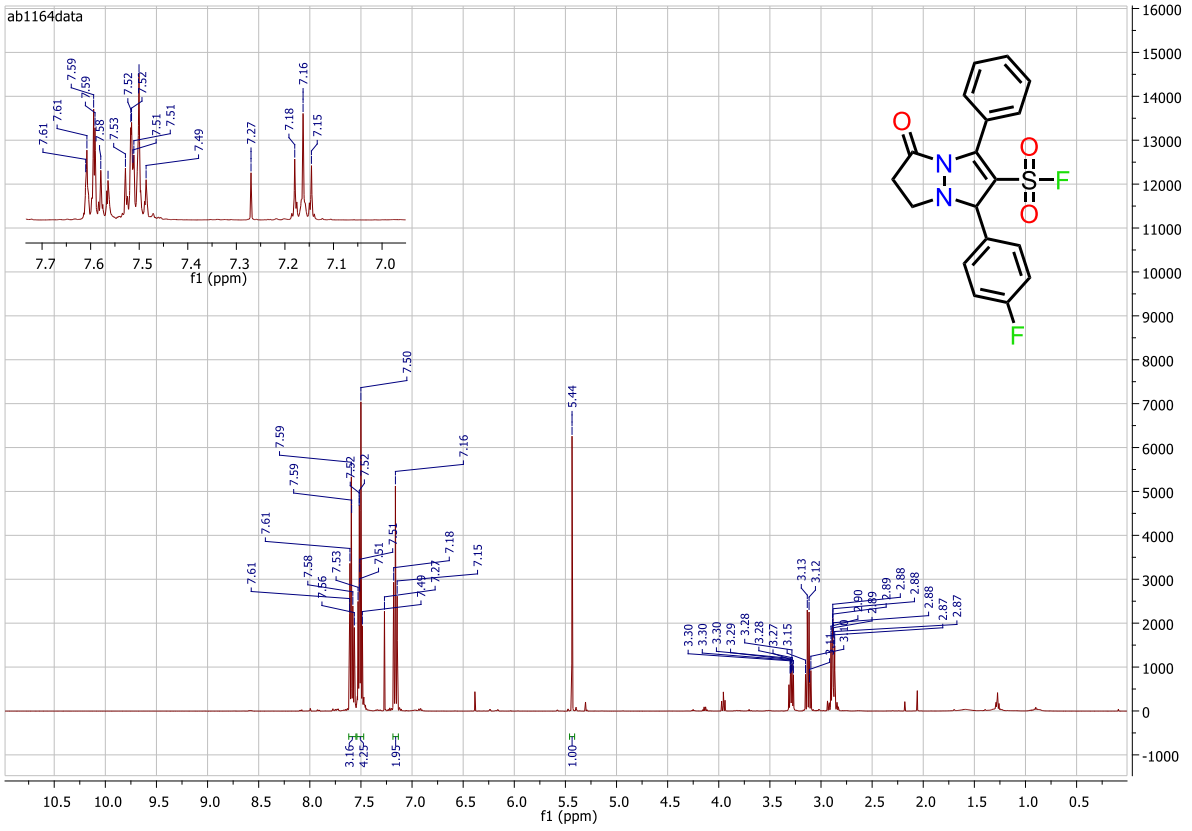
ab1153col

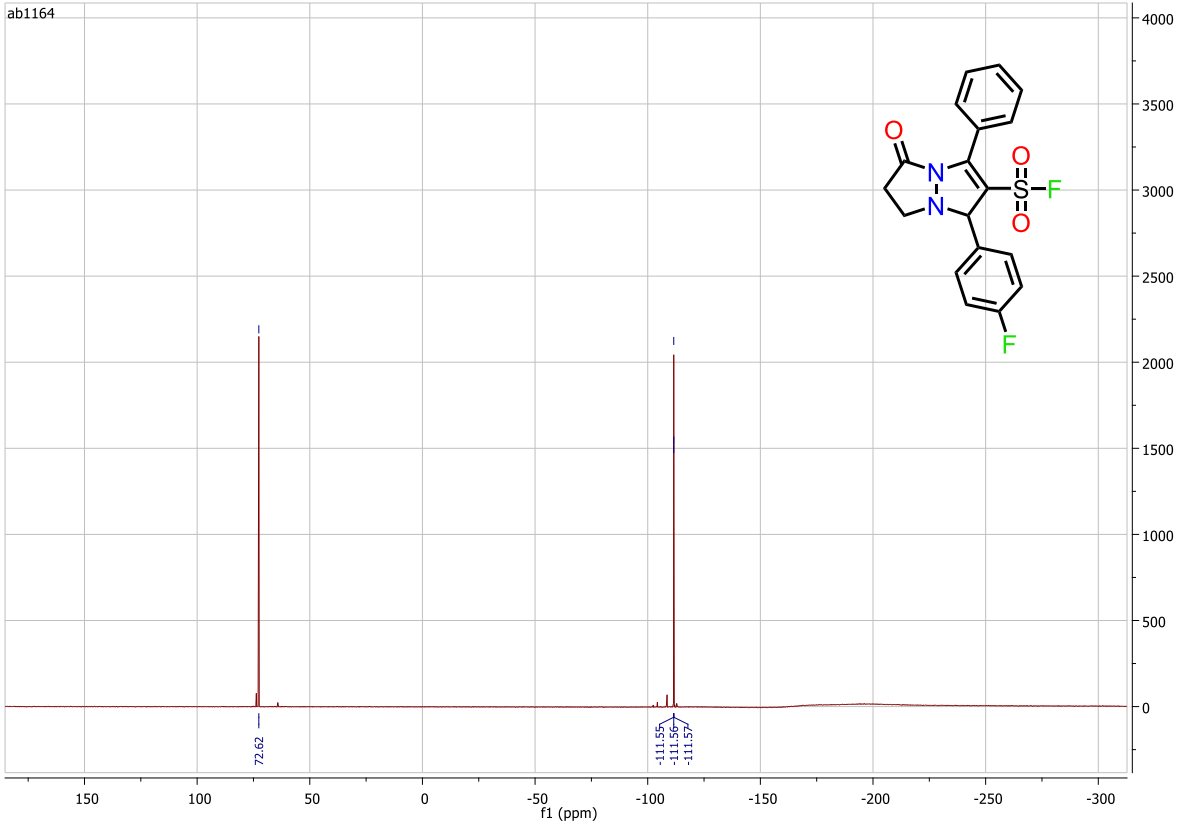


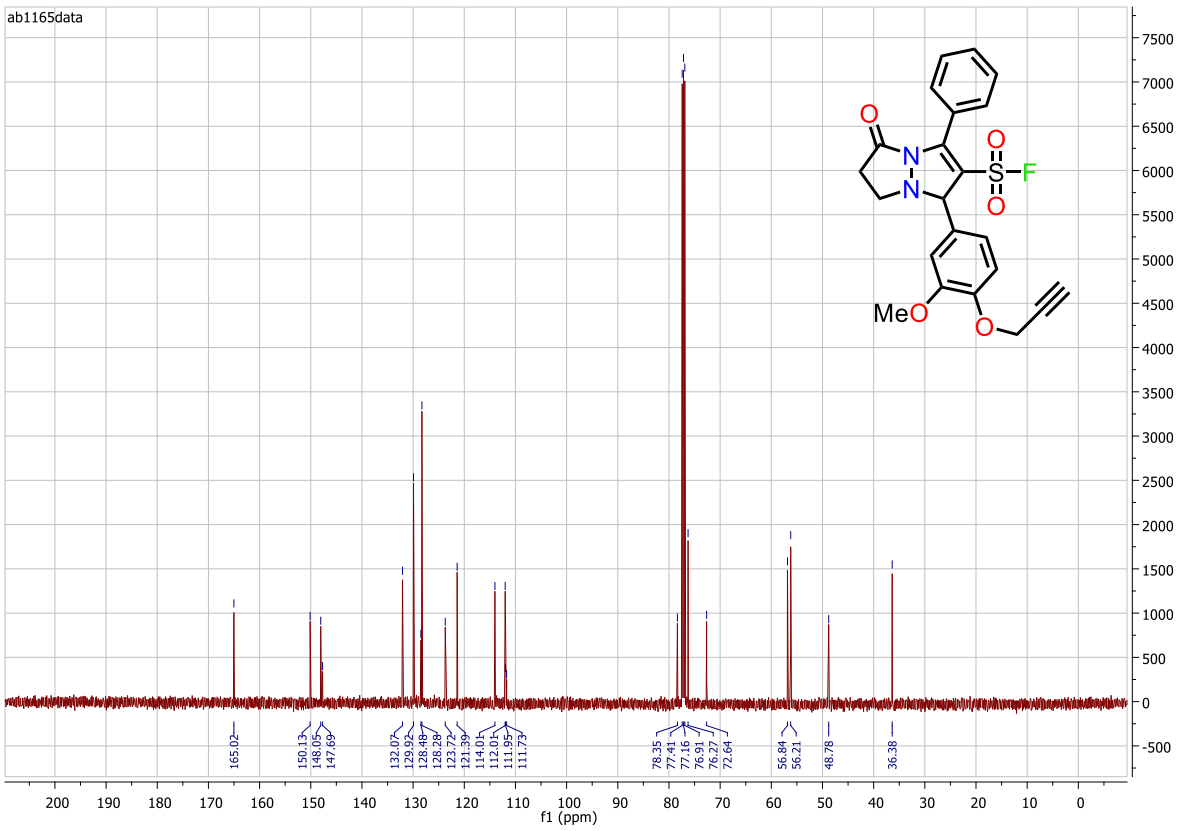
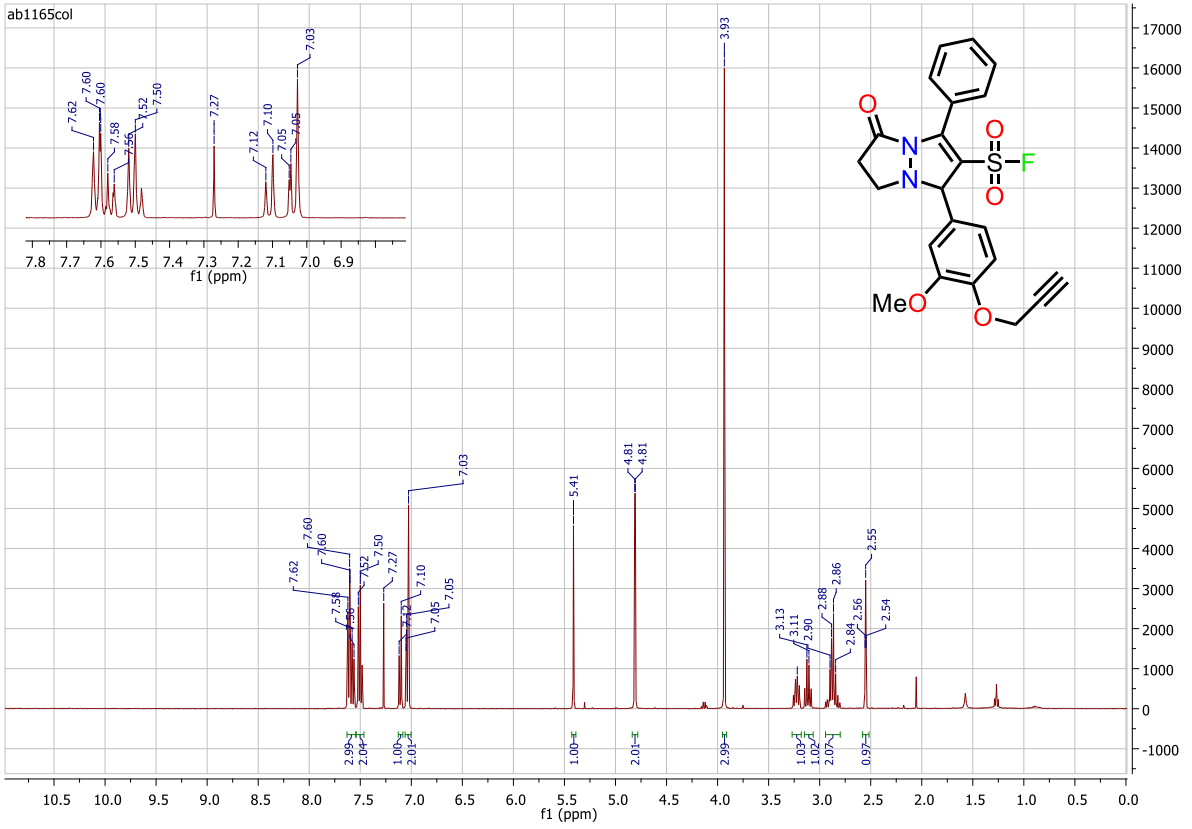


ab1289data

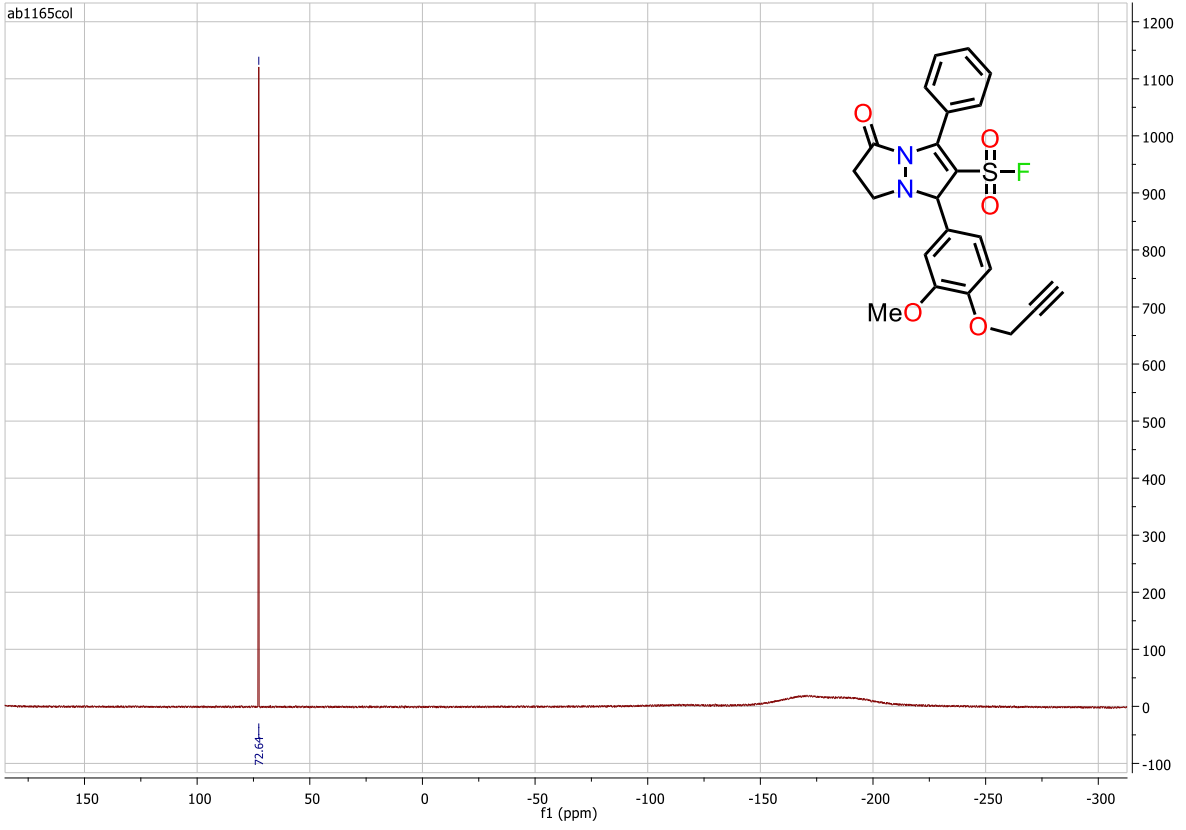


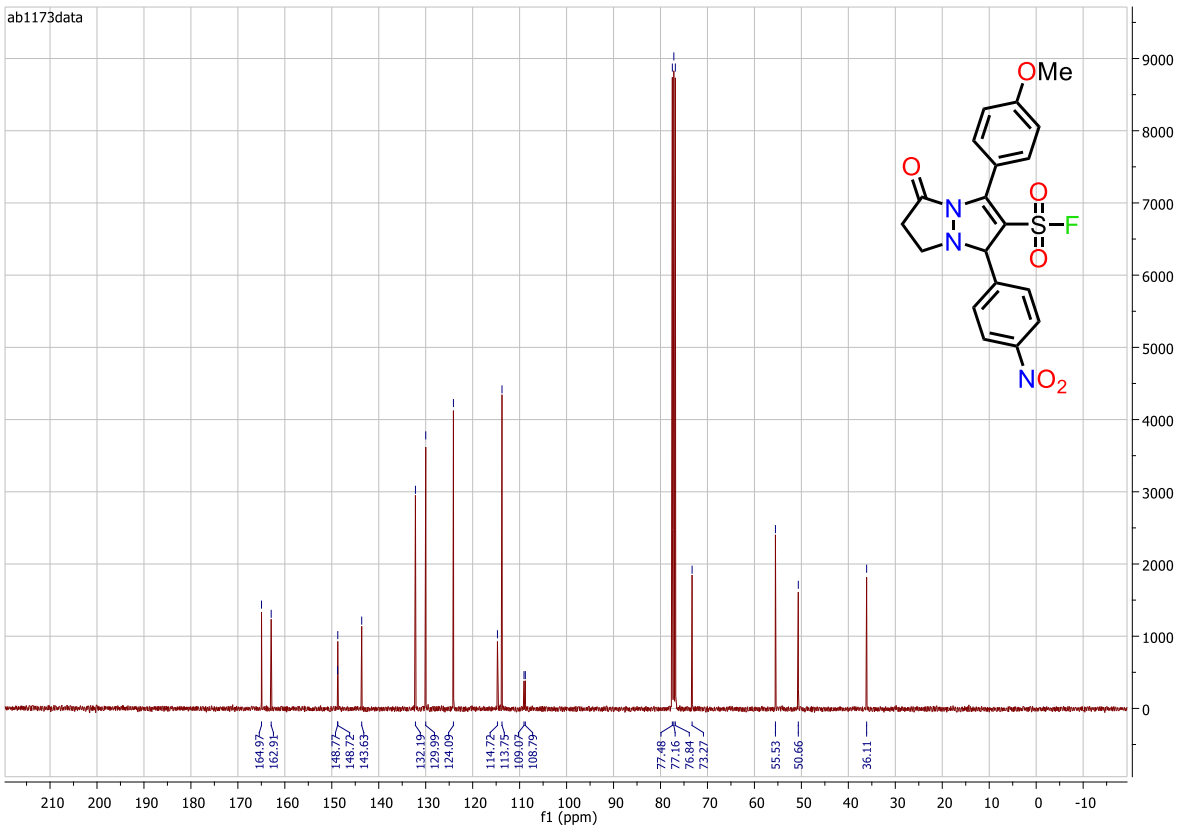
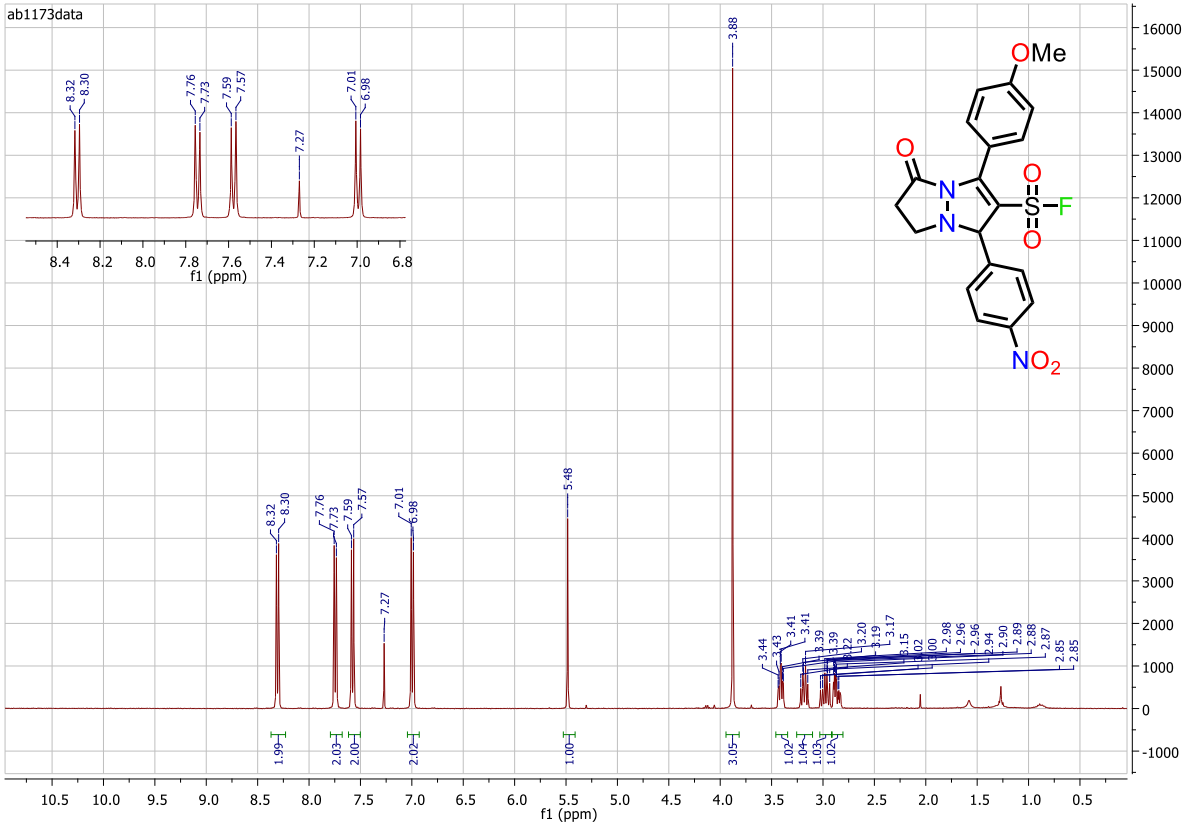




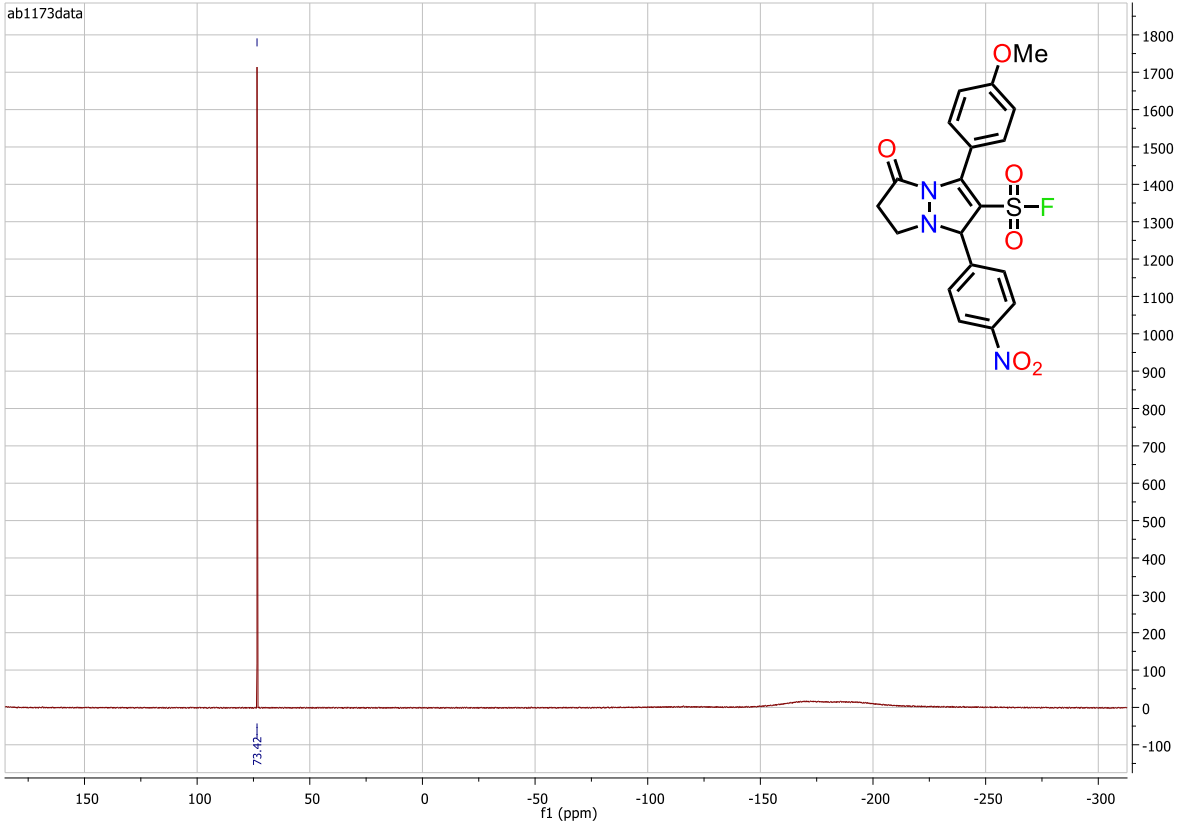


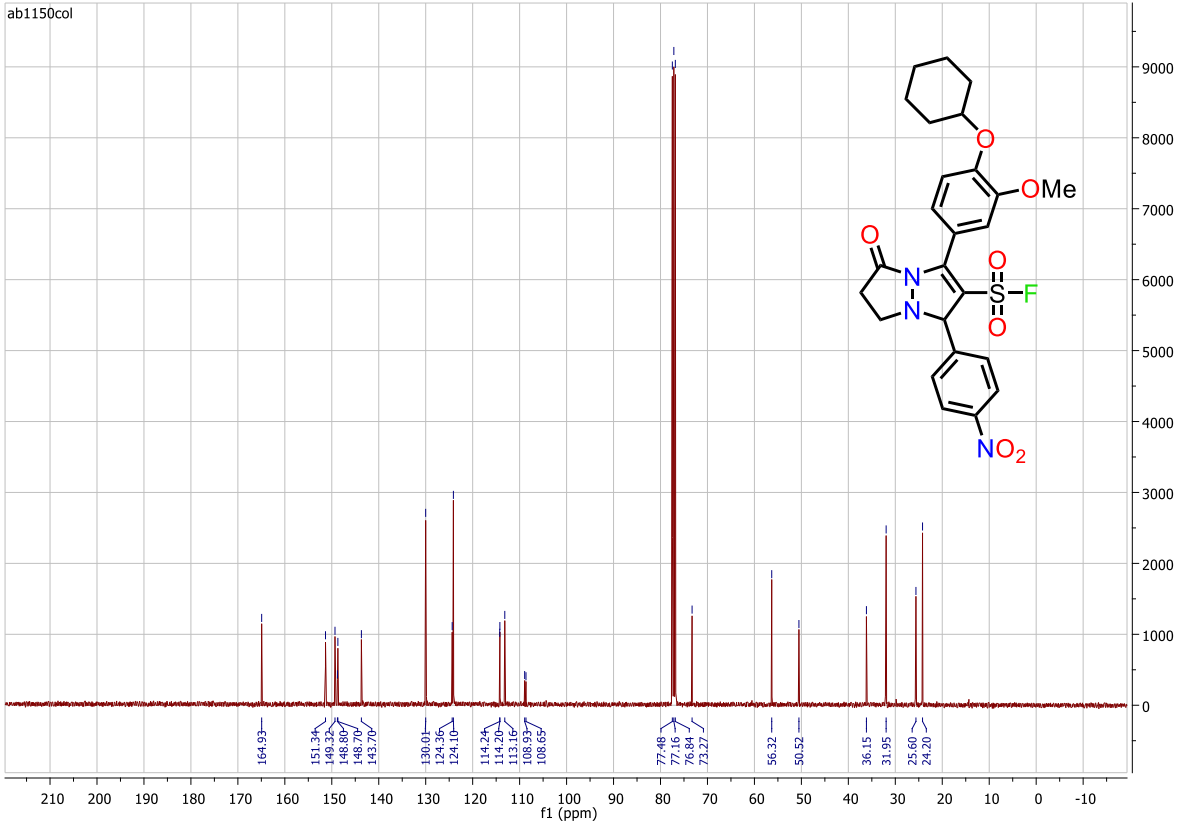
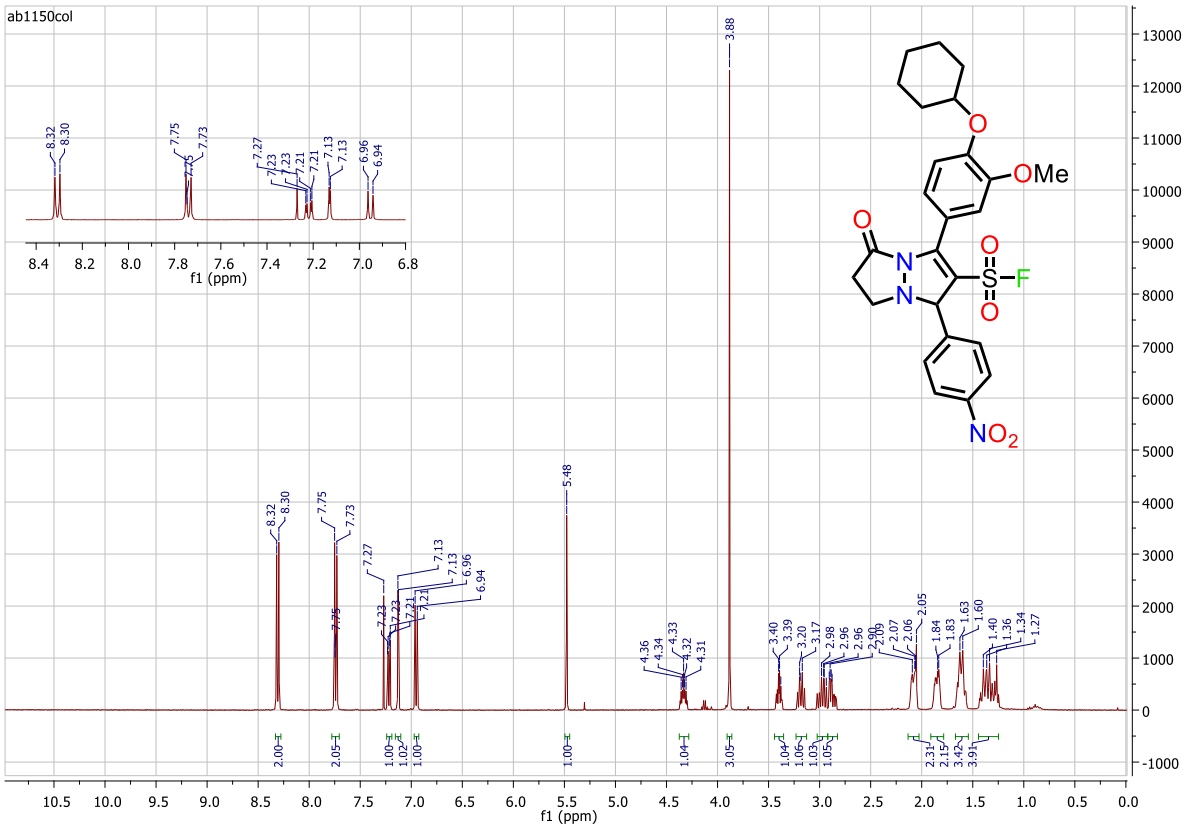
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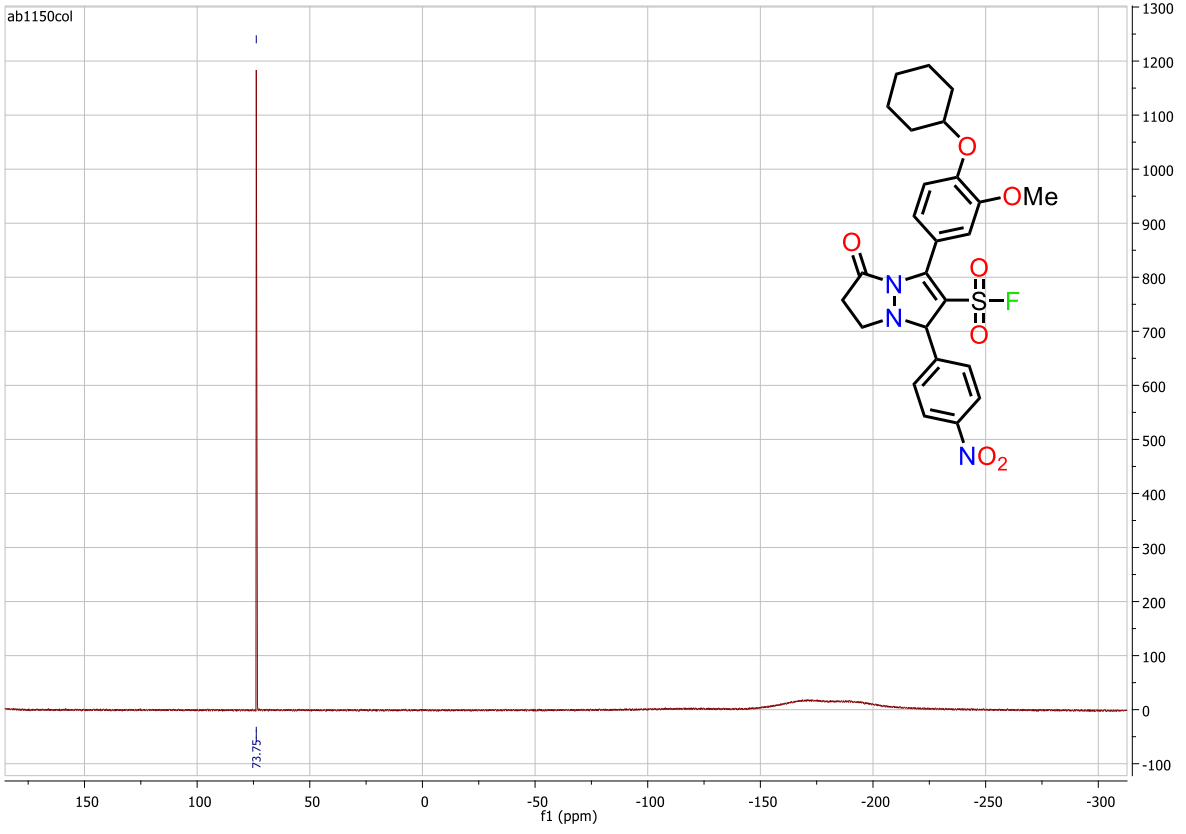


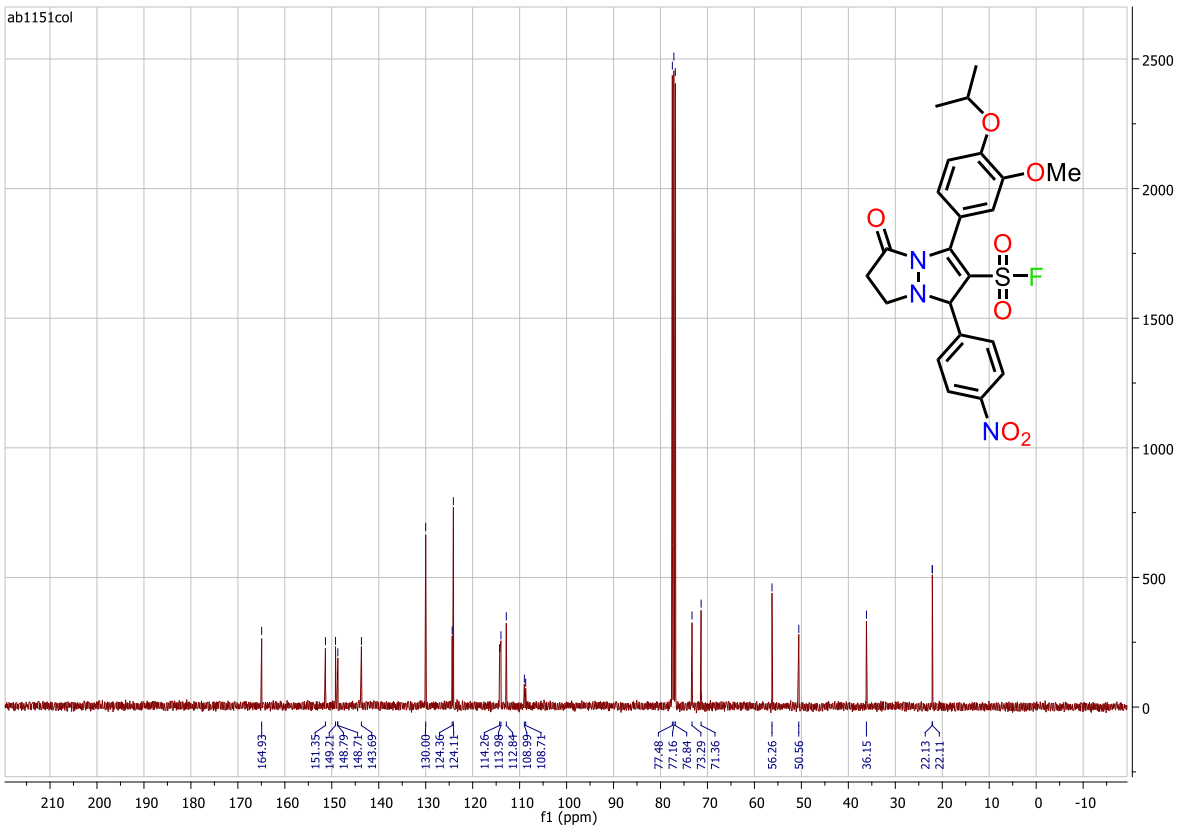
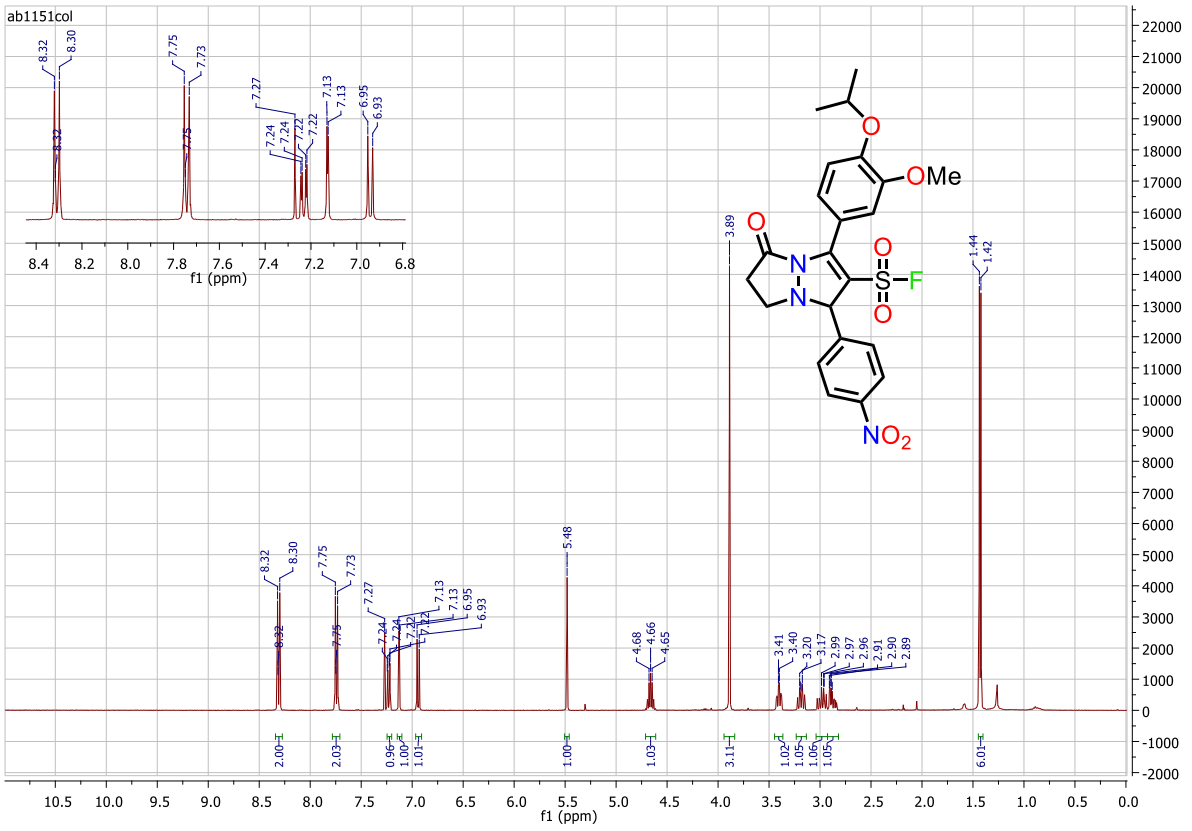


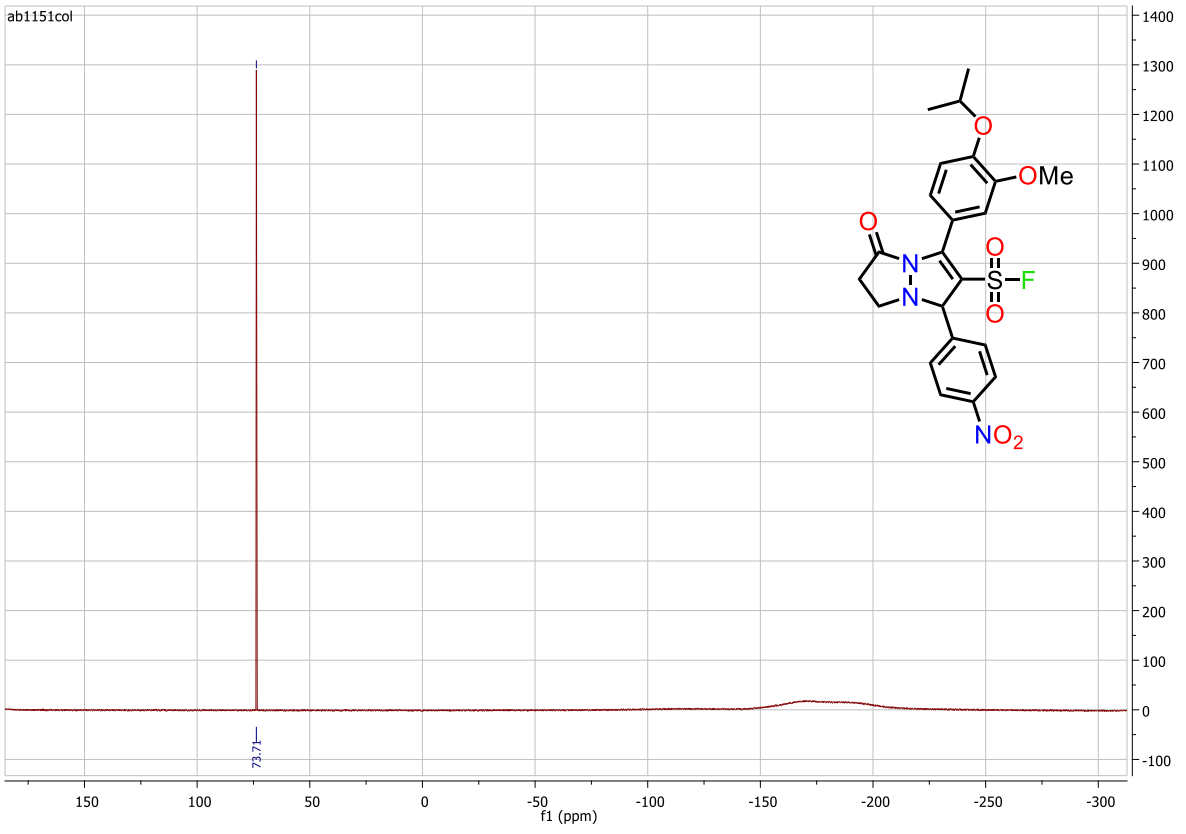
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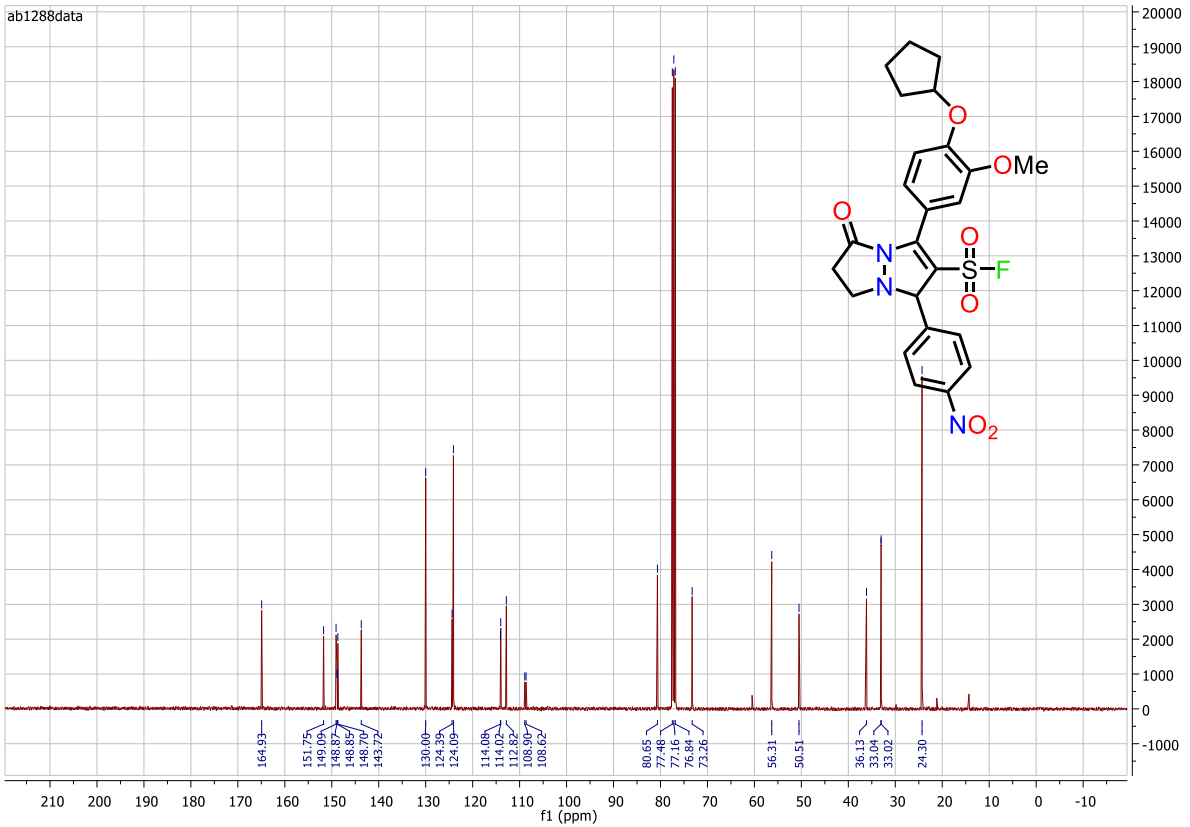
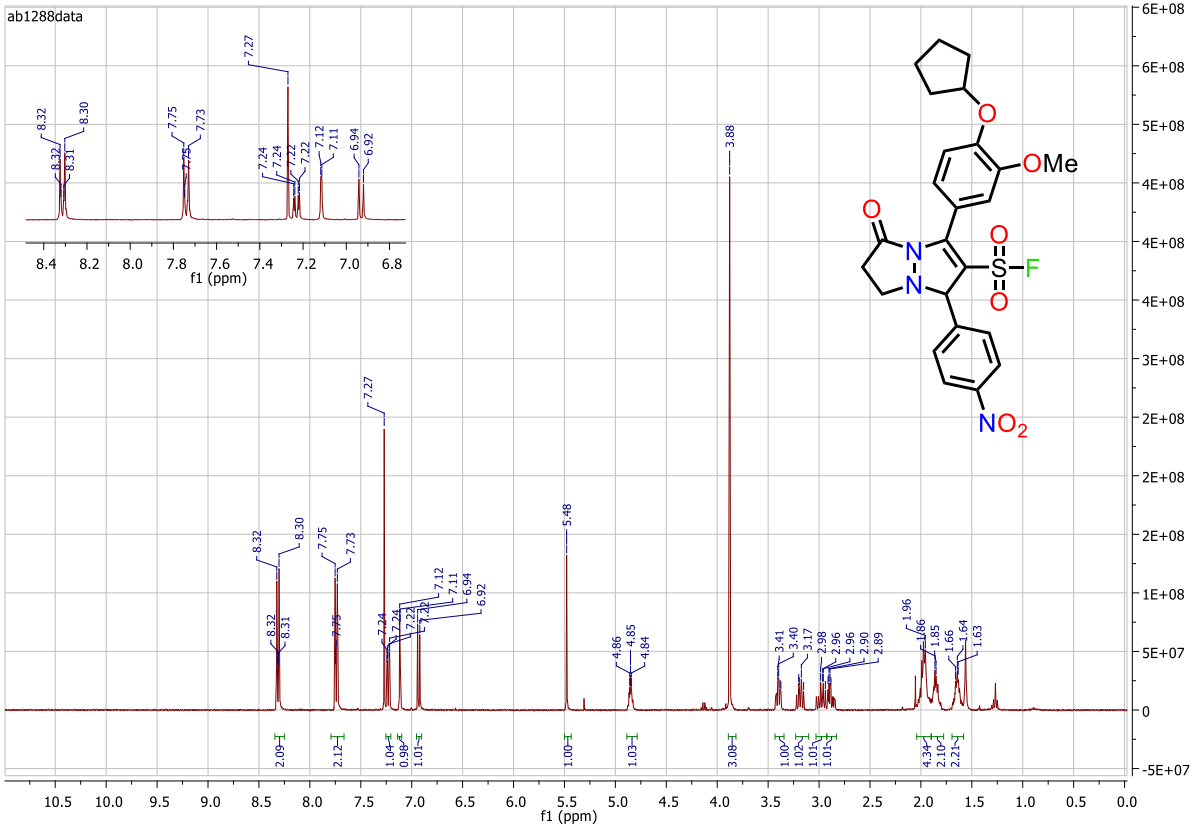


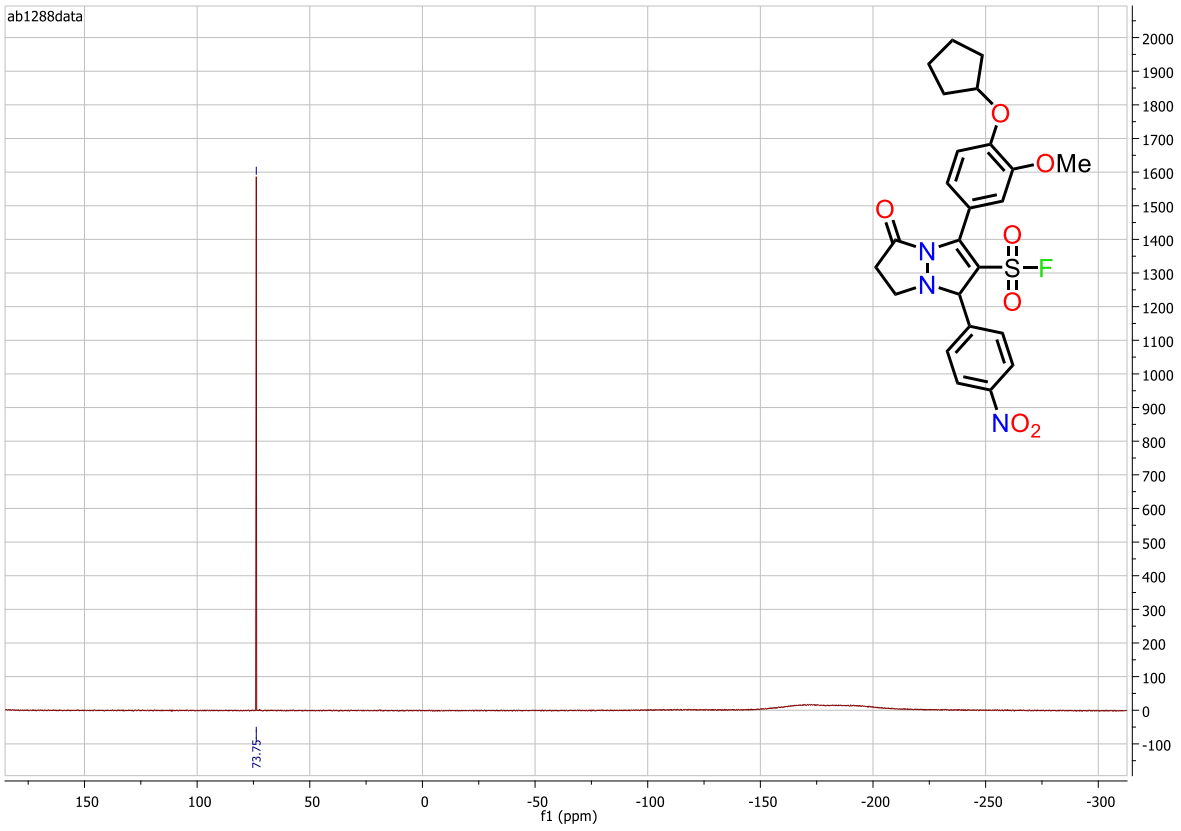


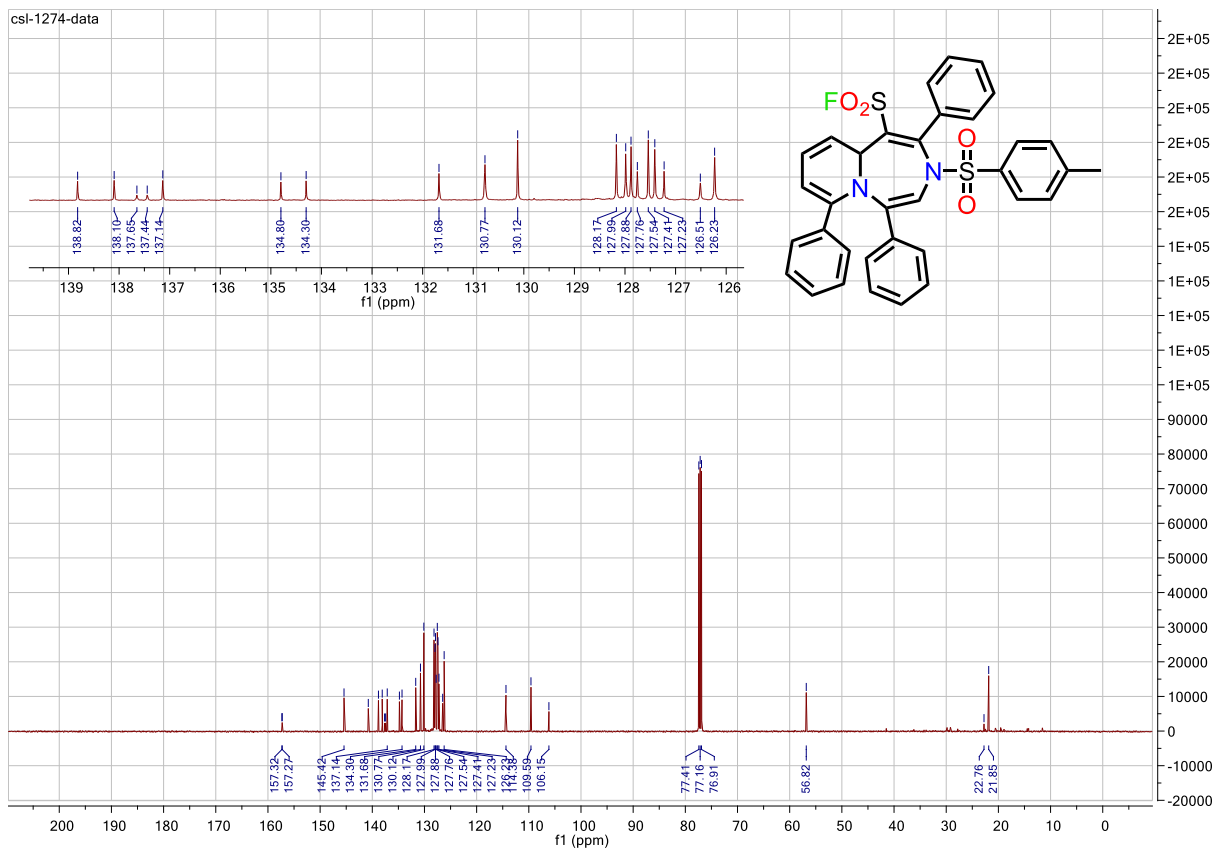
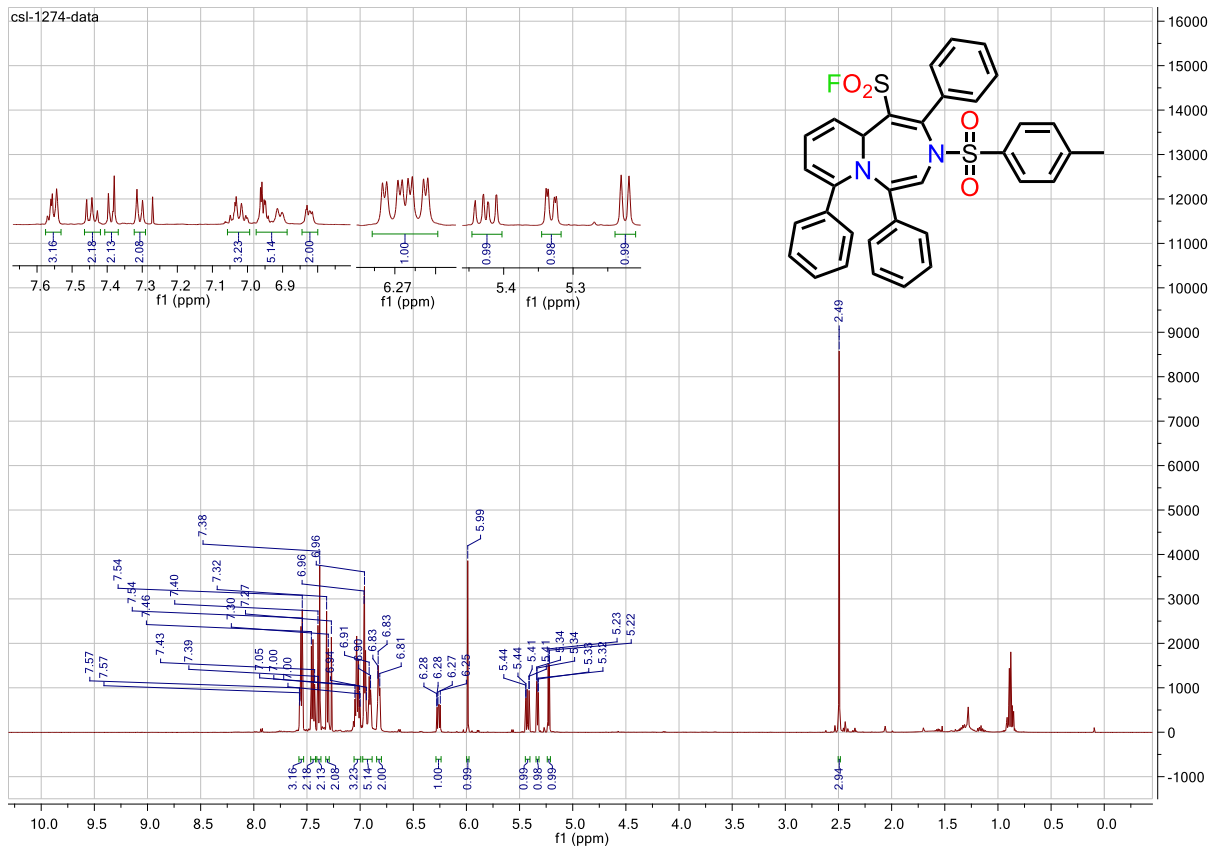


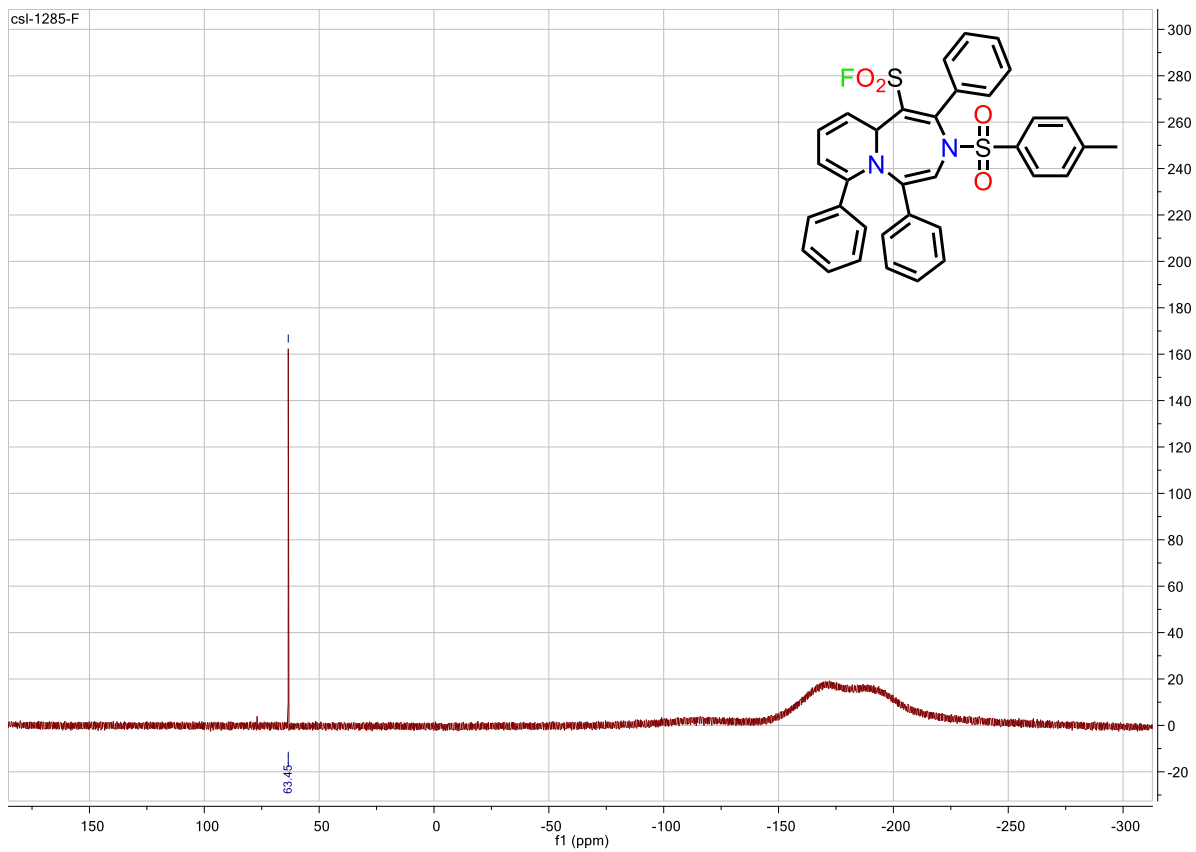


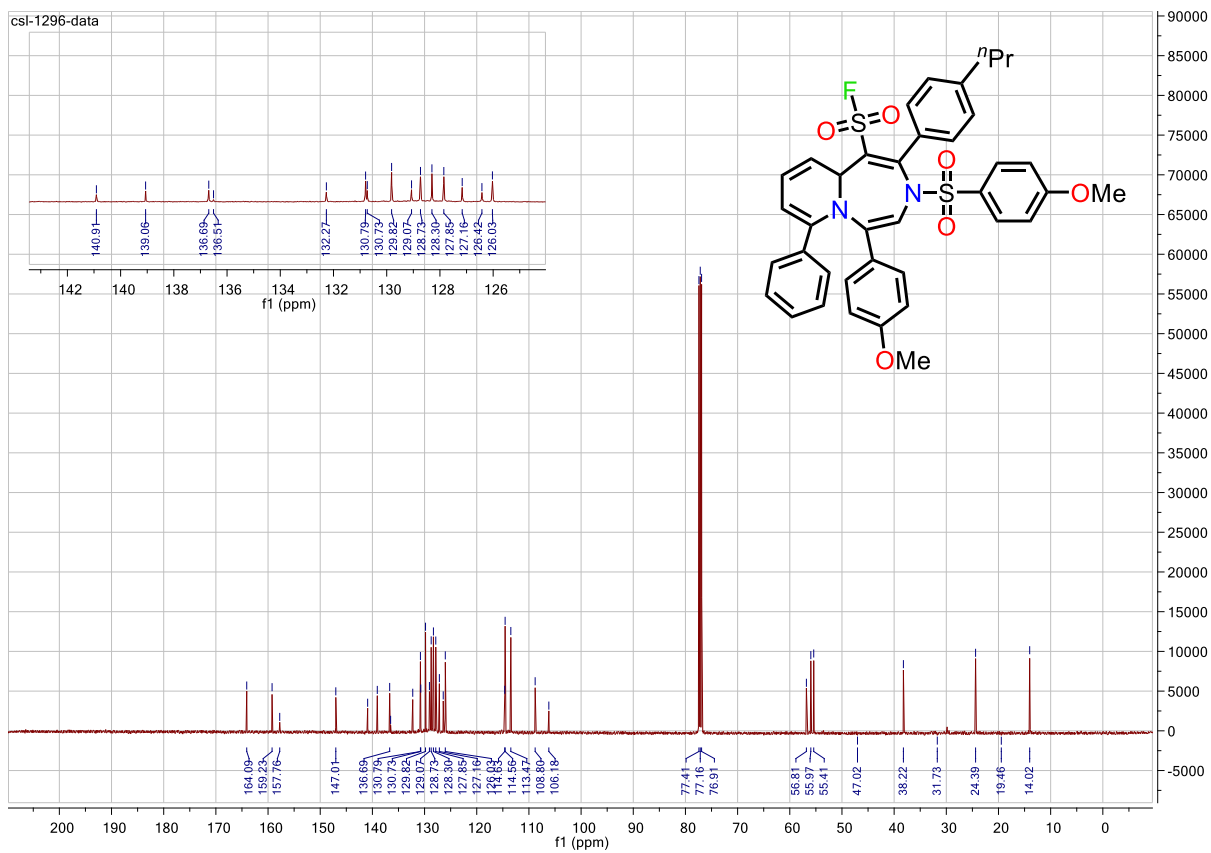
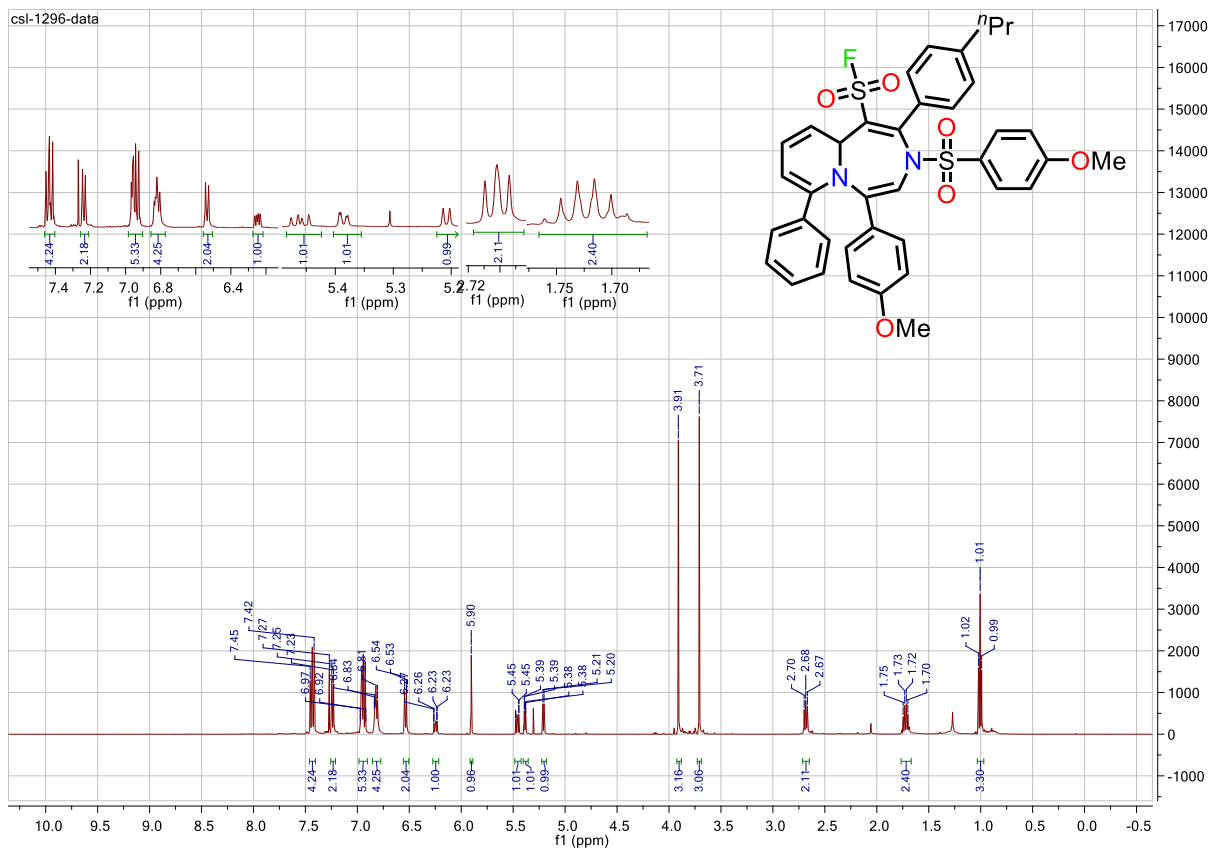


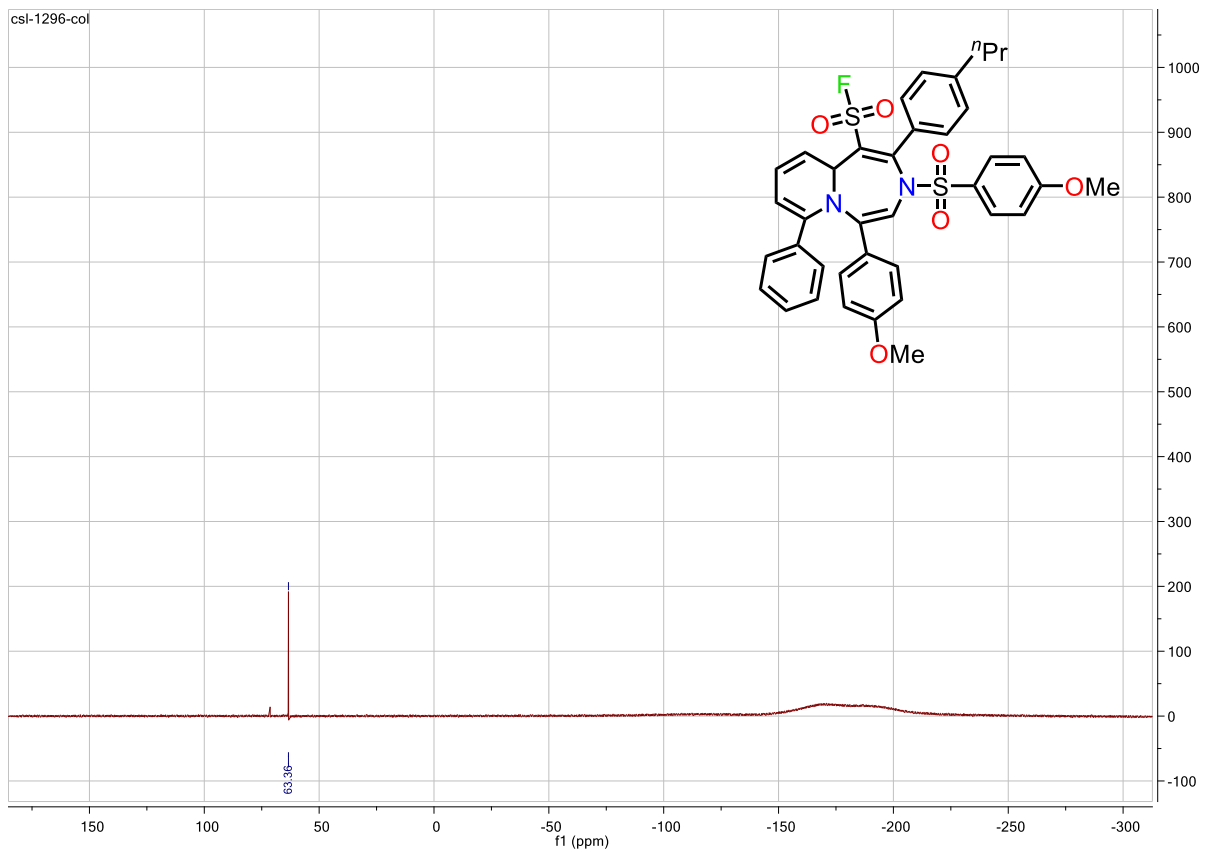


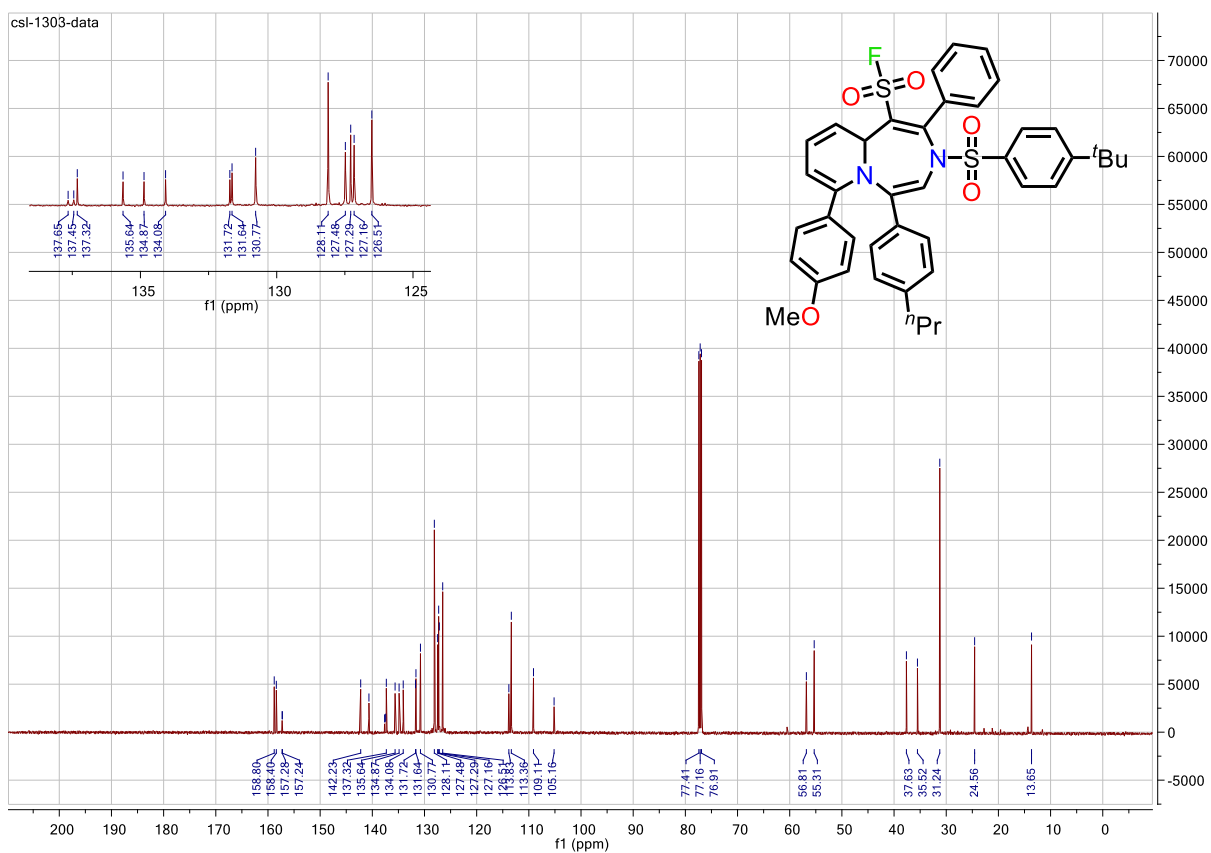
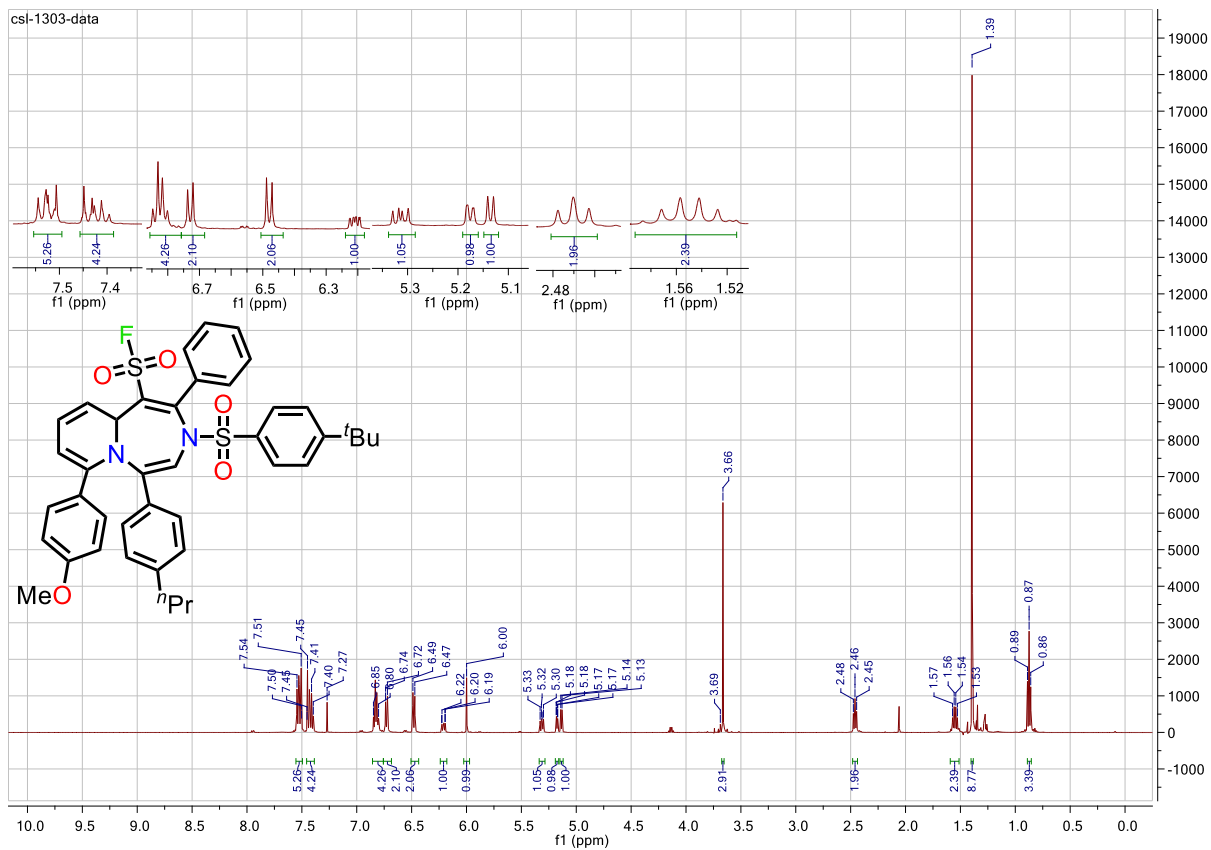


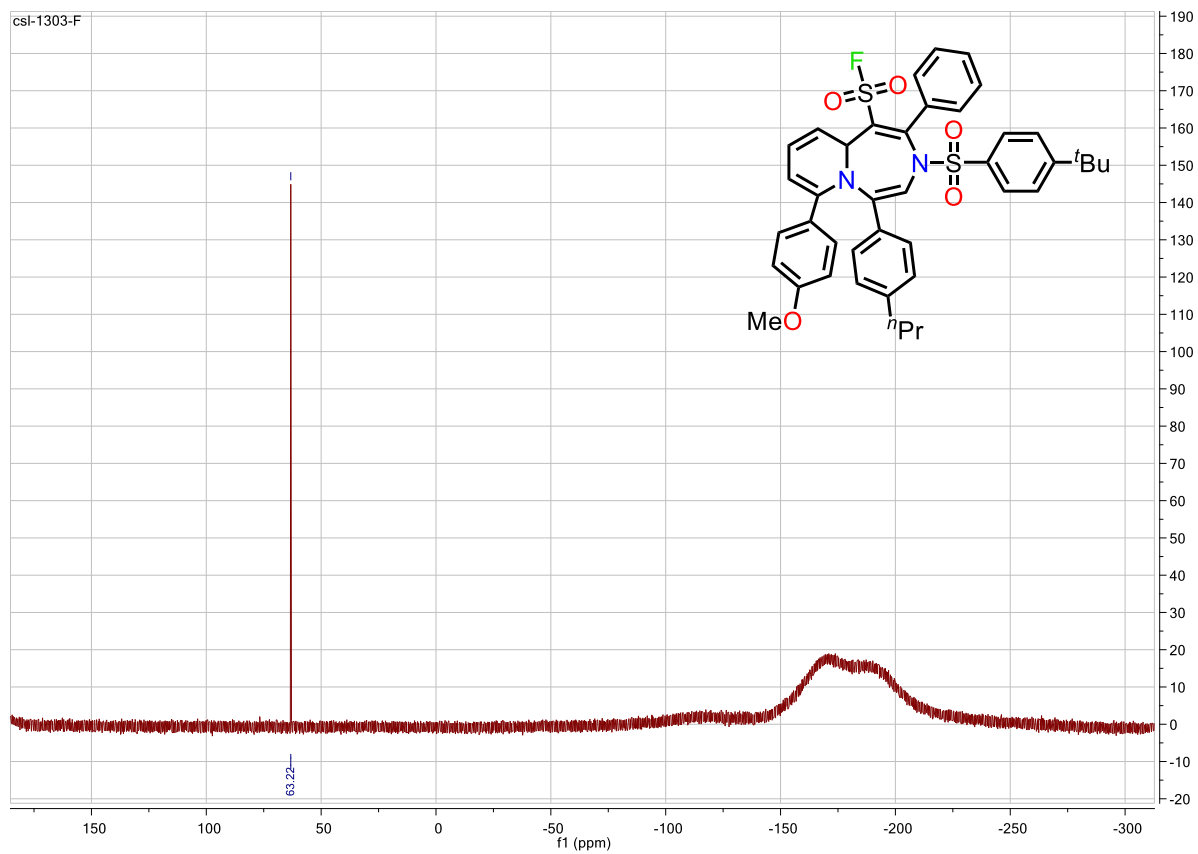


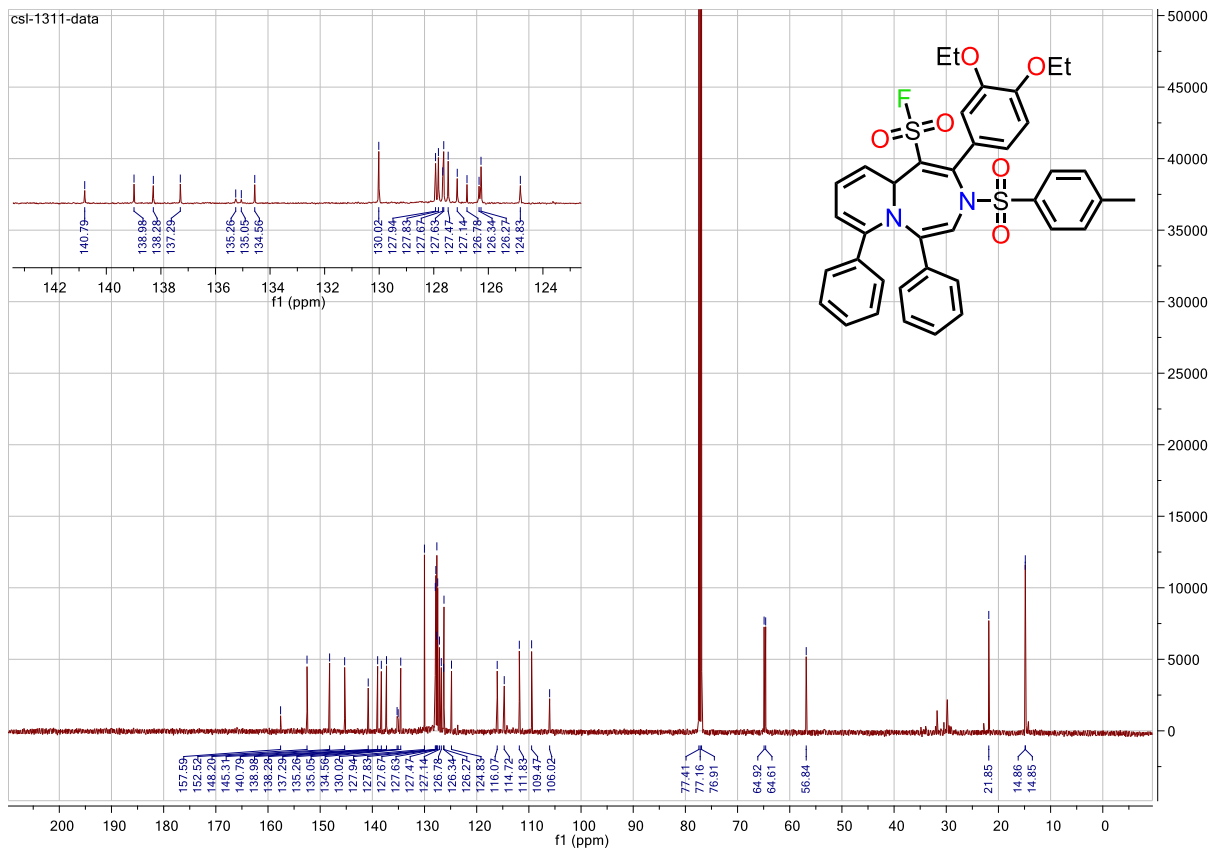
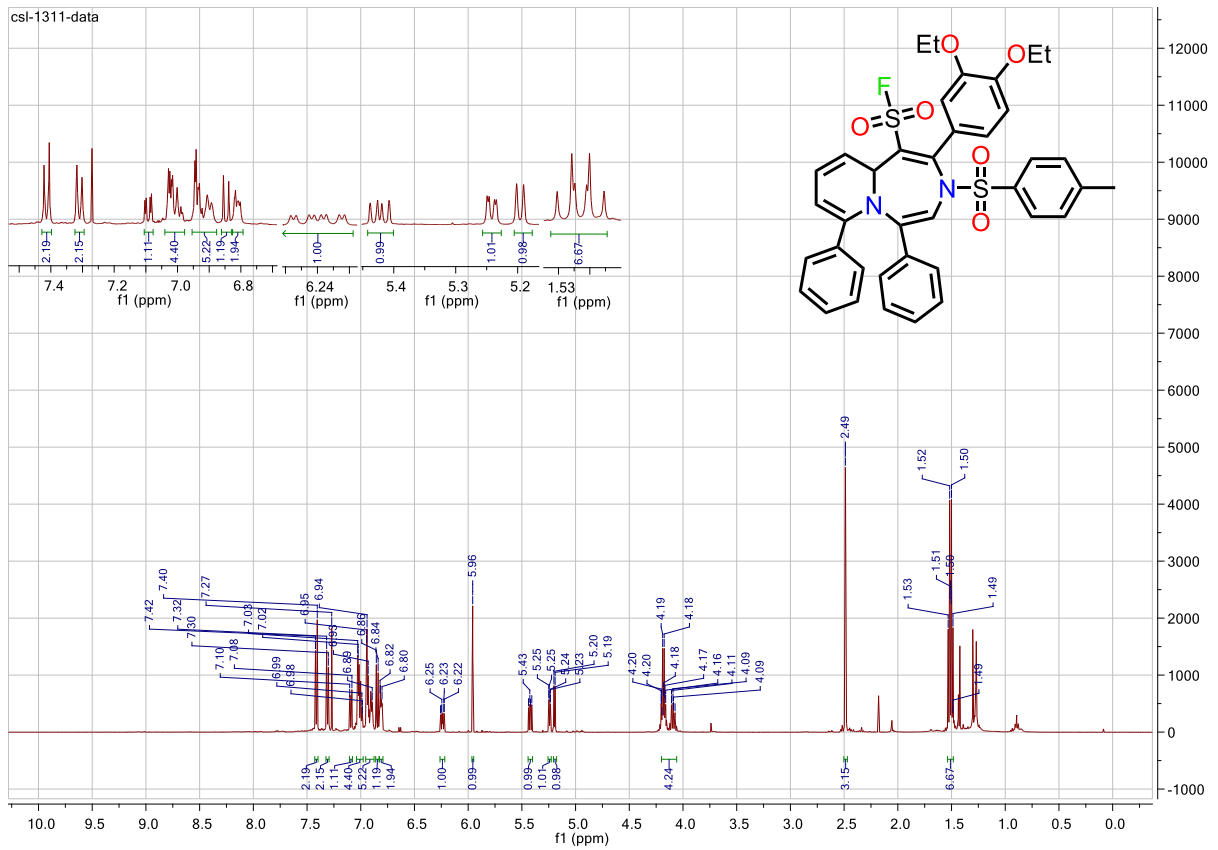


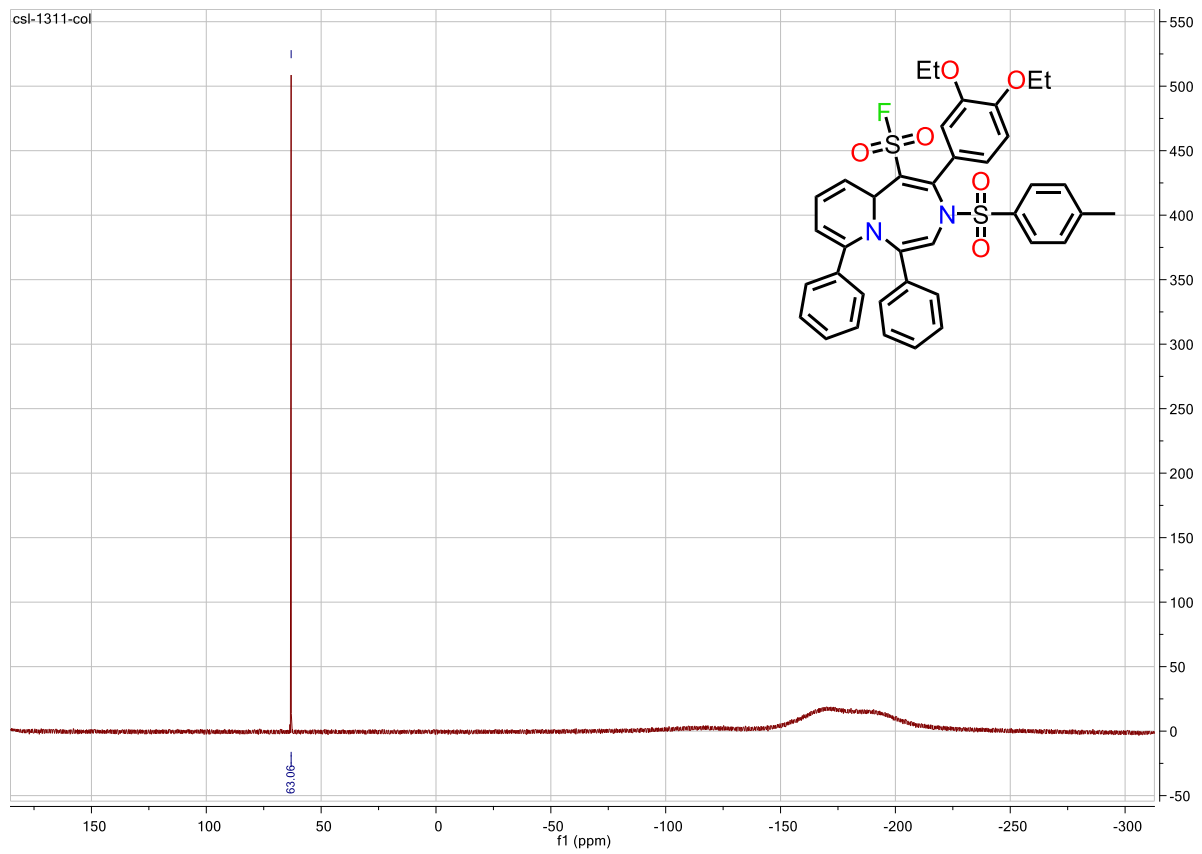


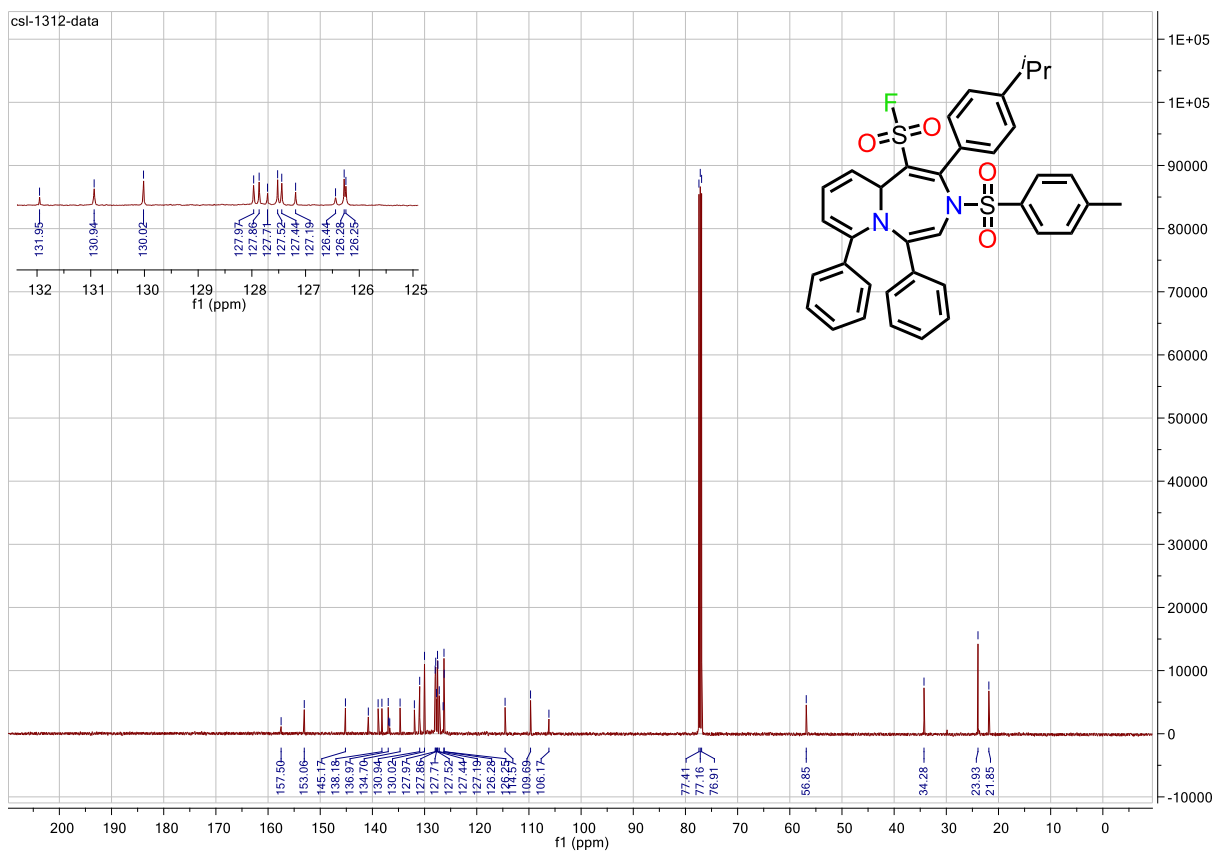
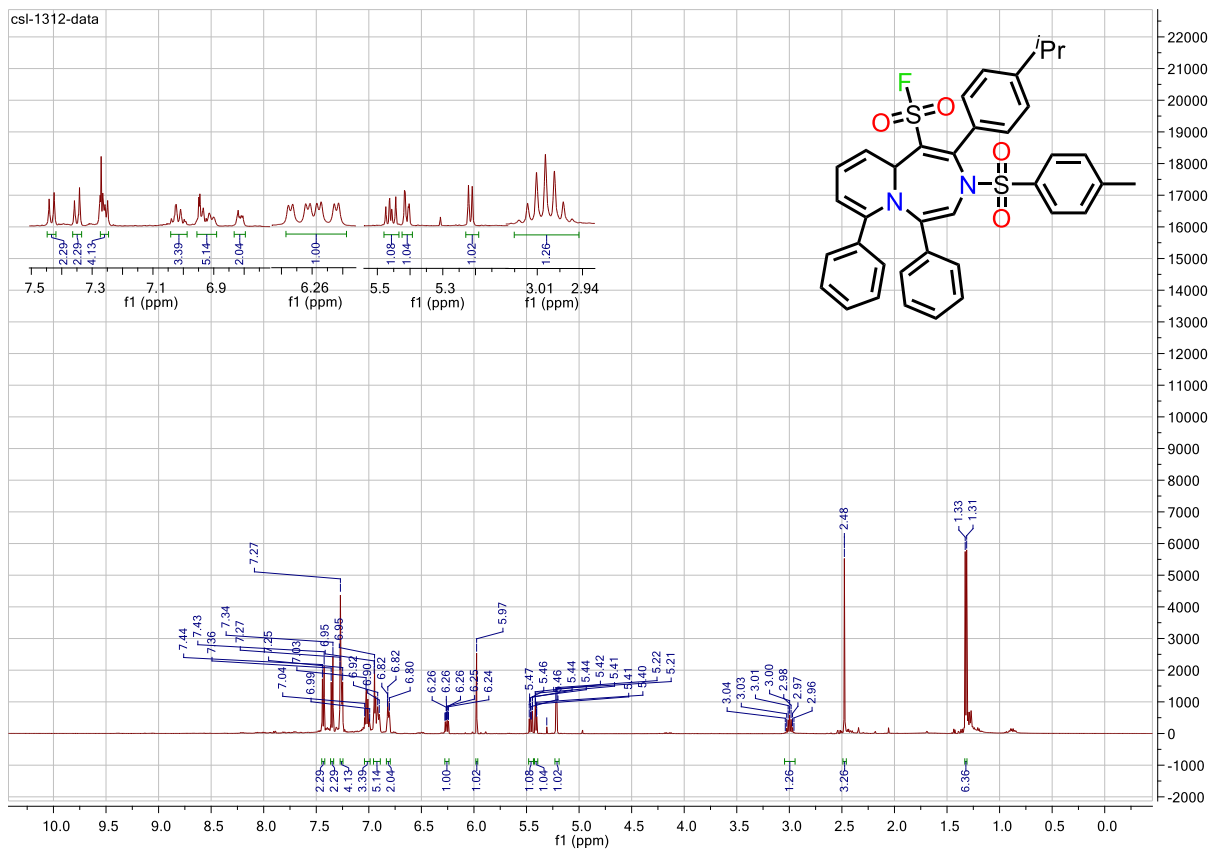


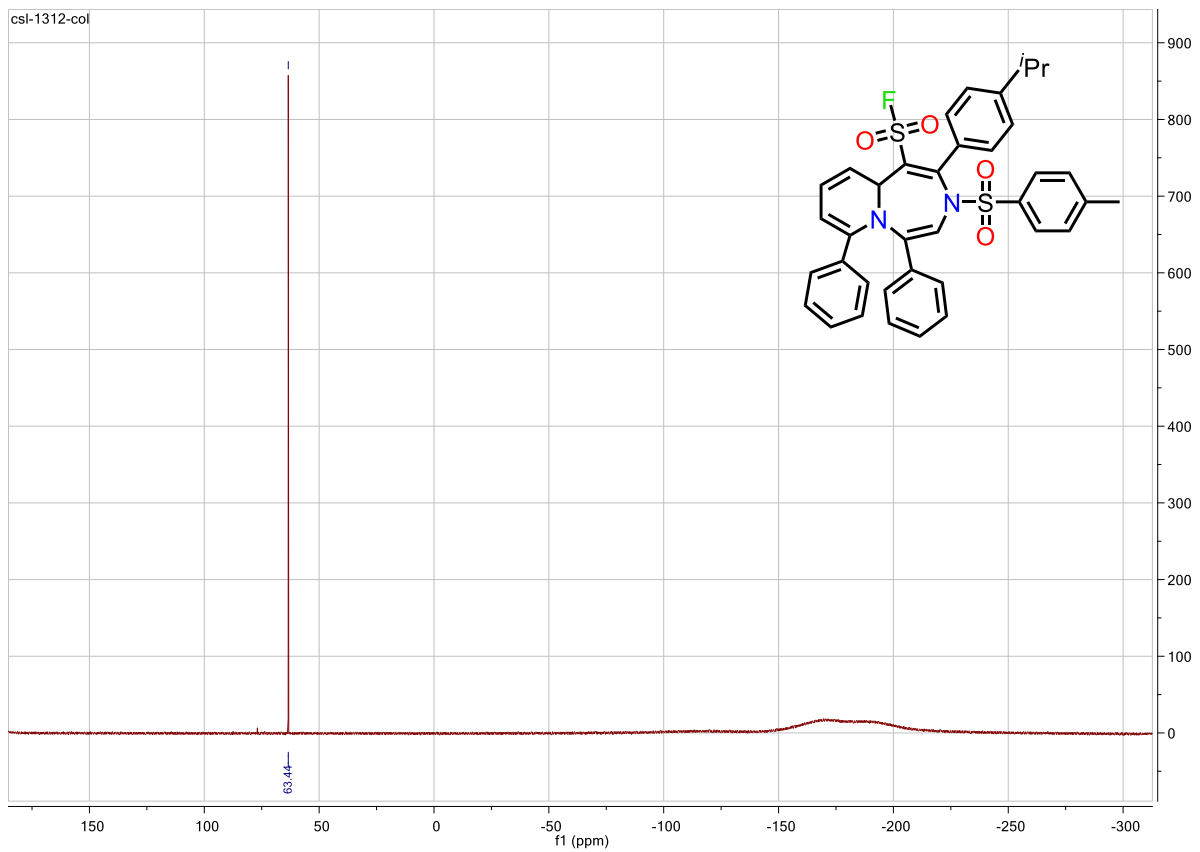


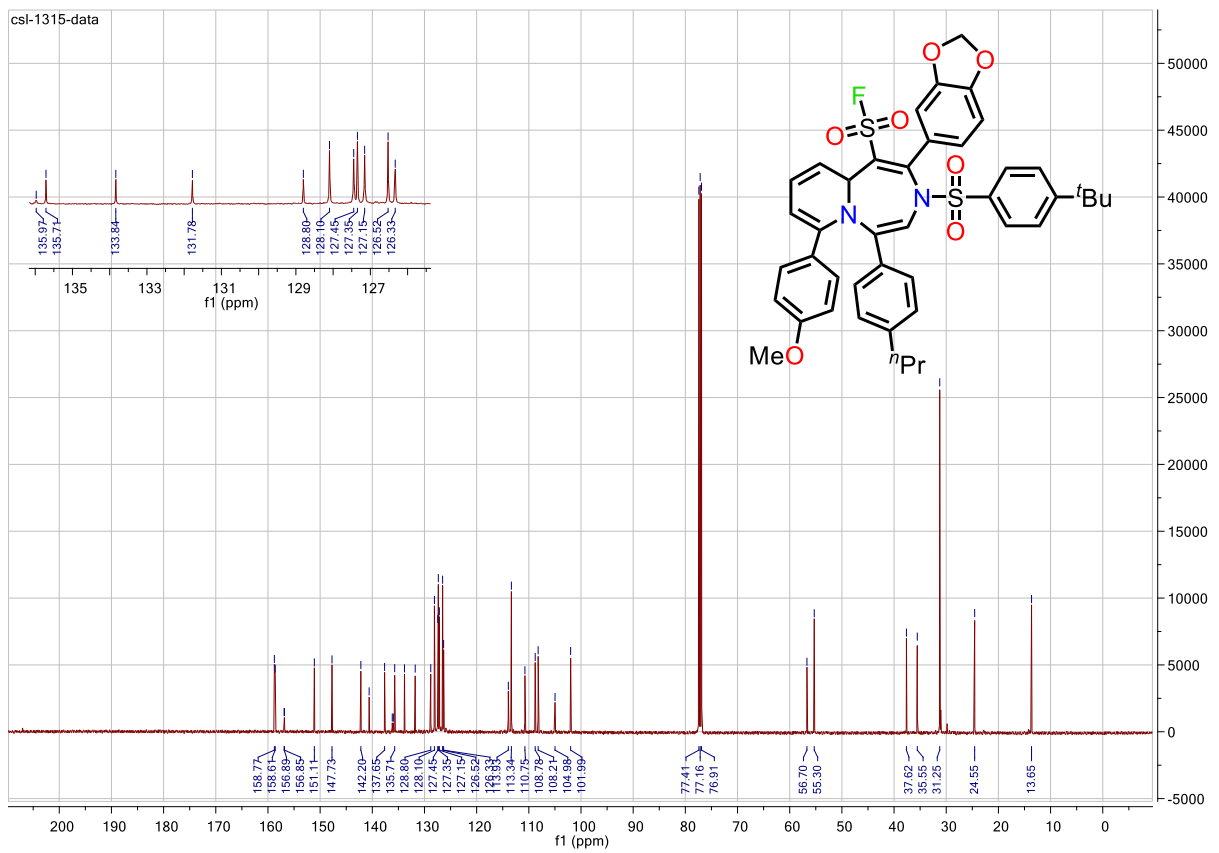
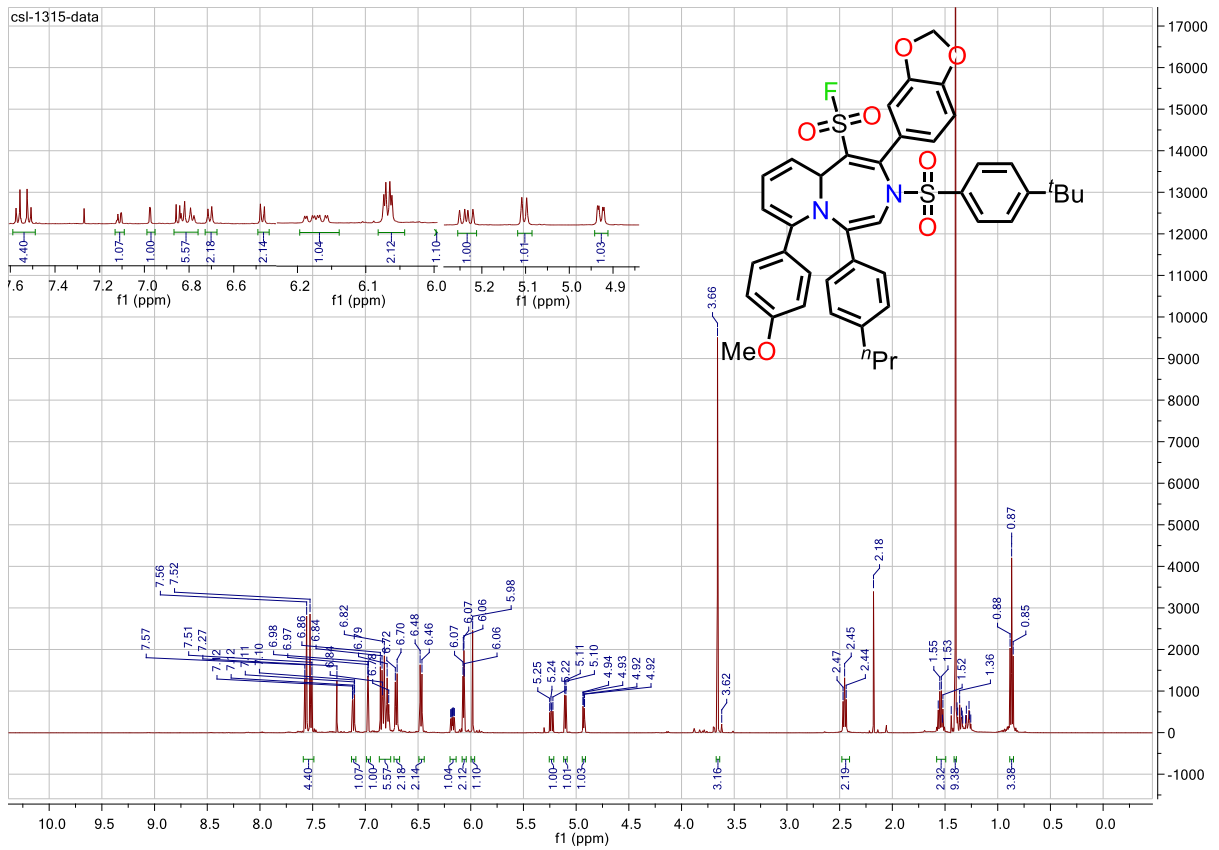


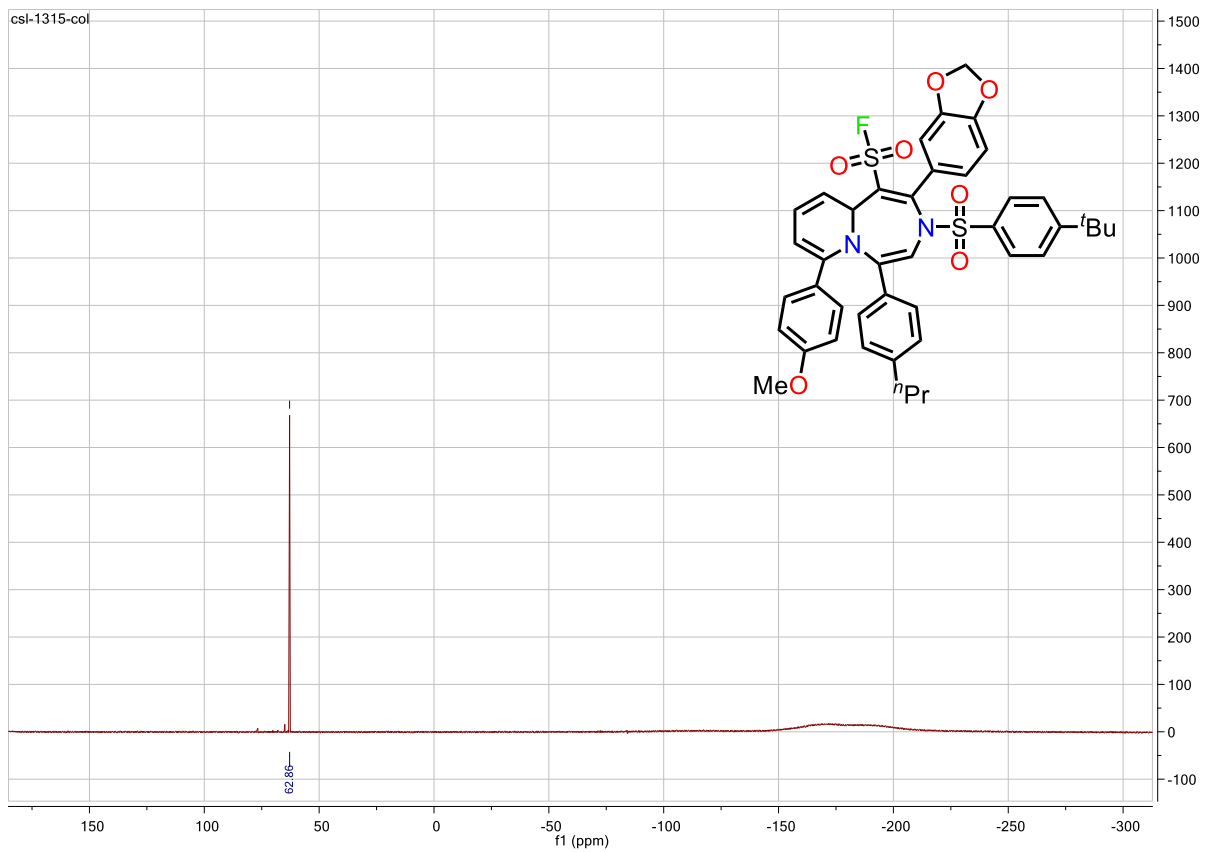


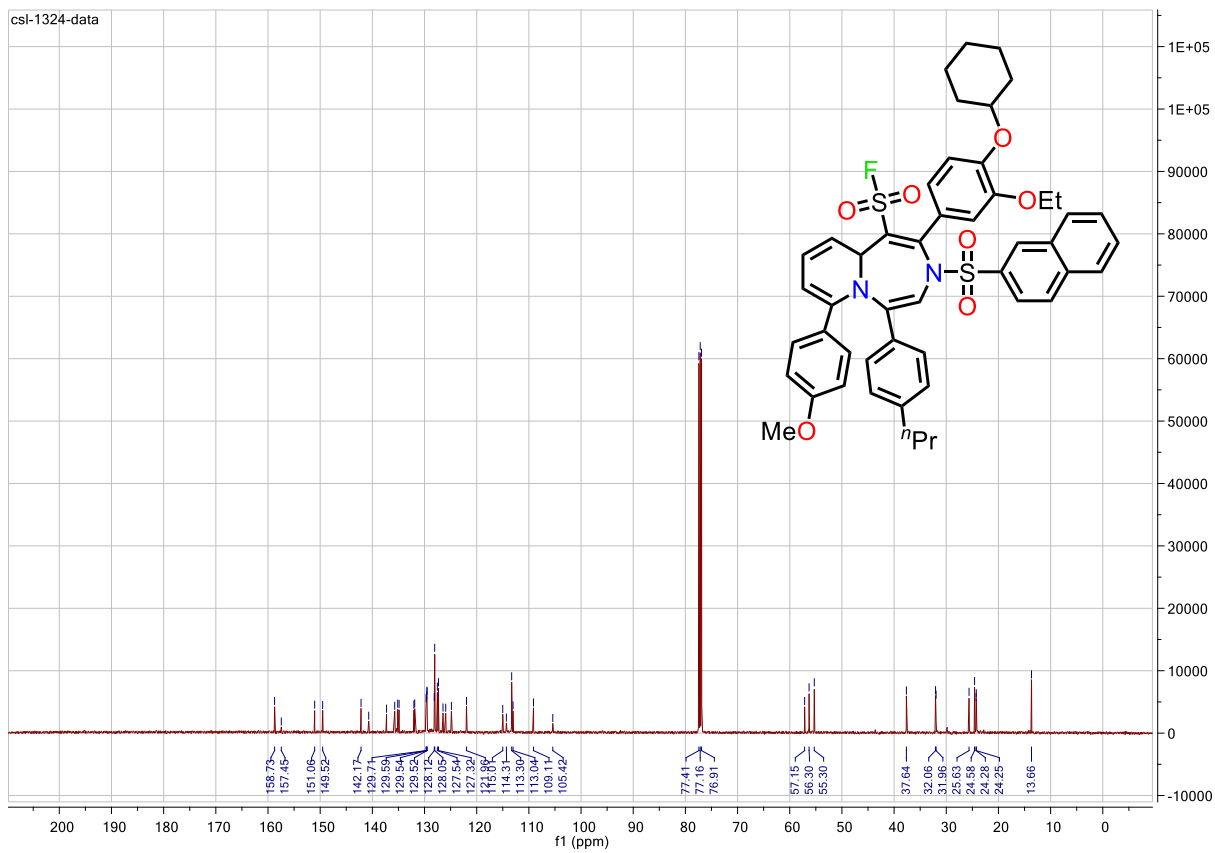
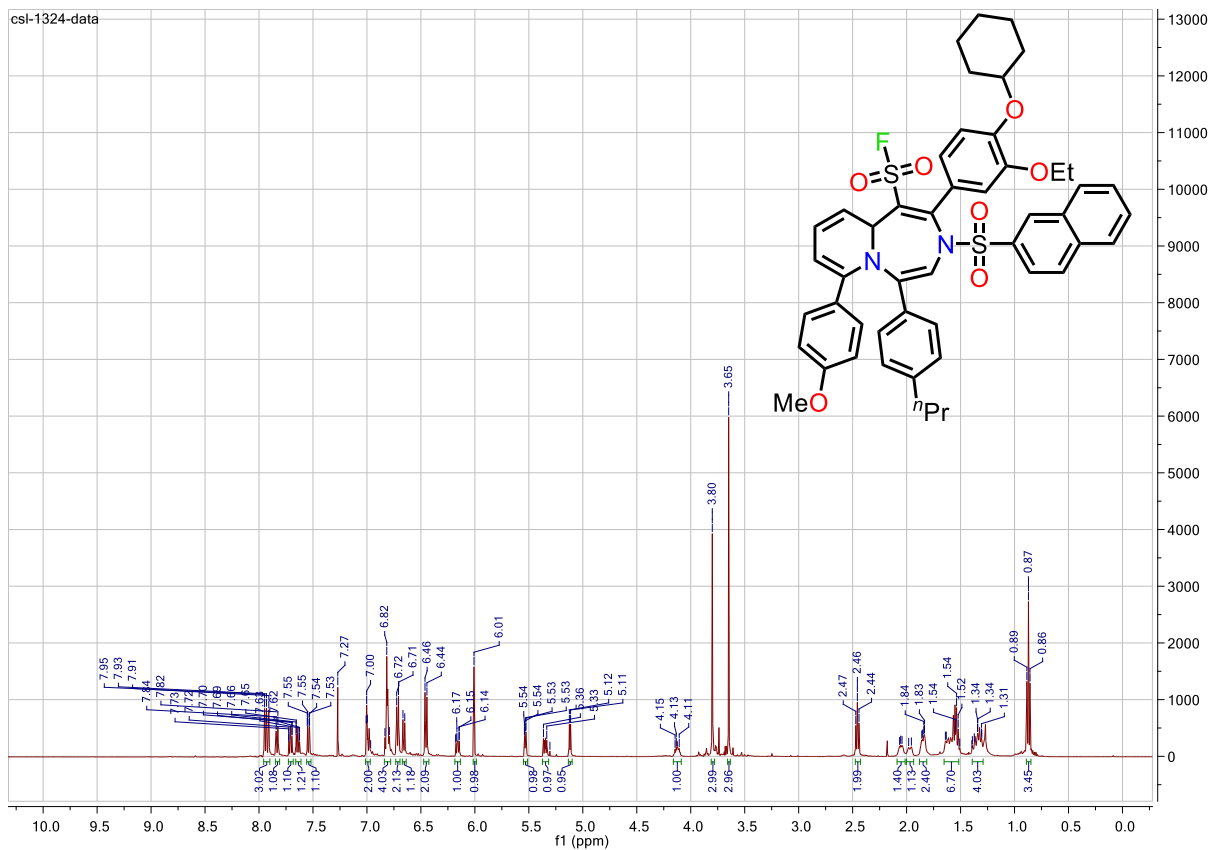


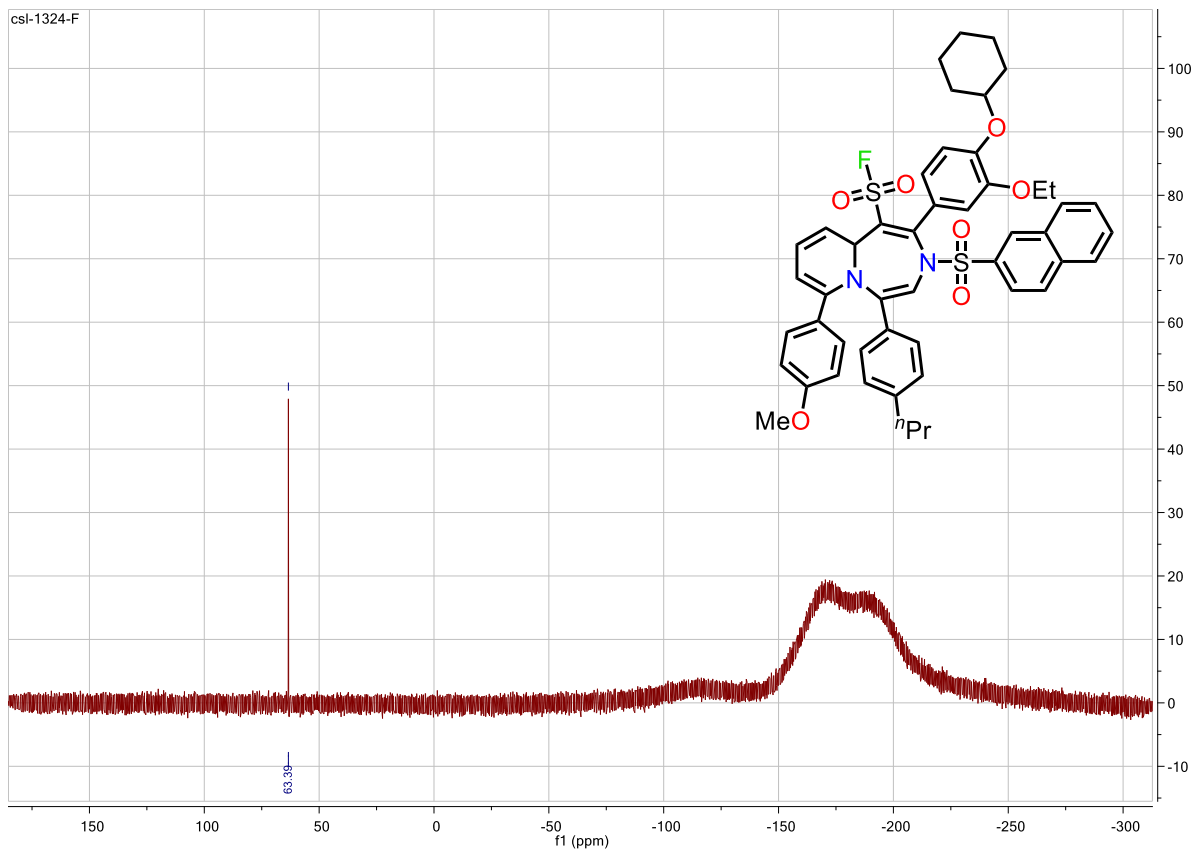


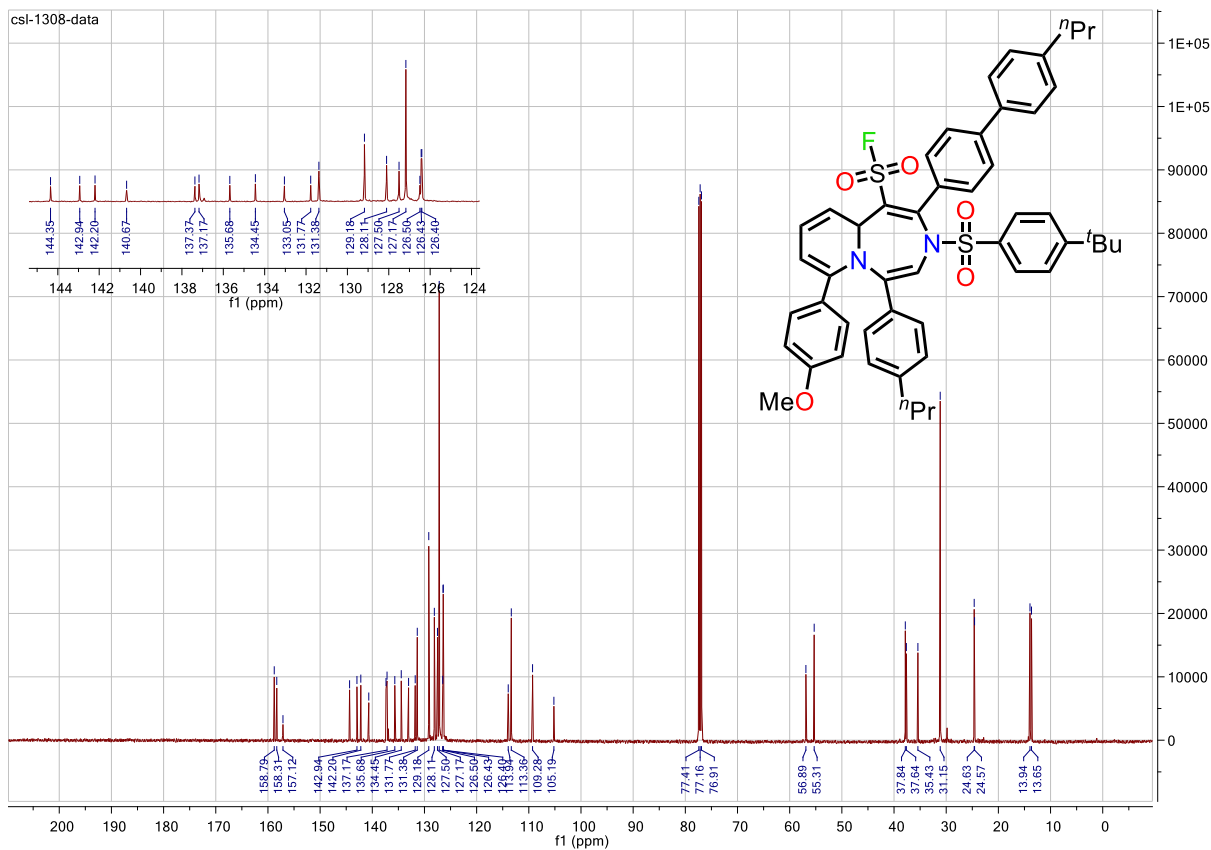
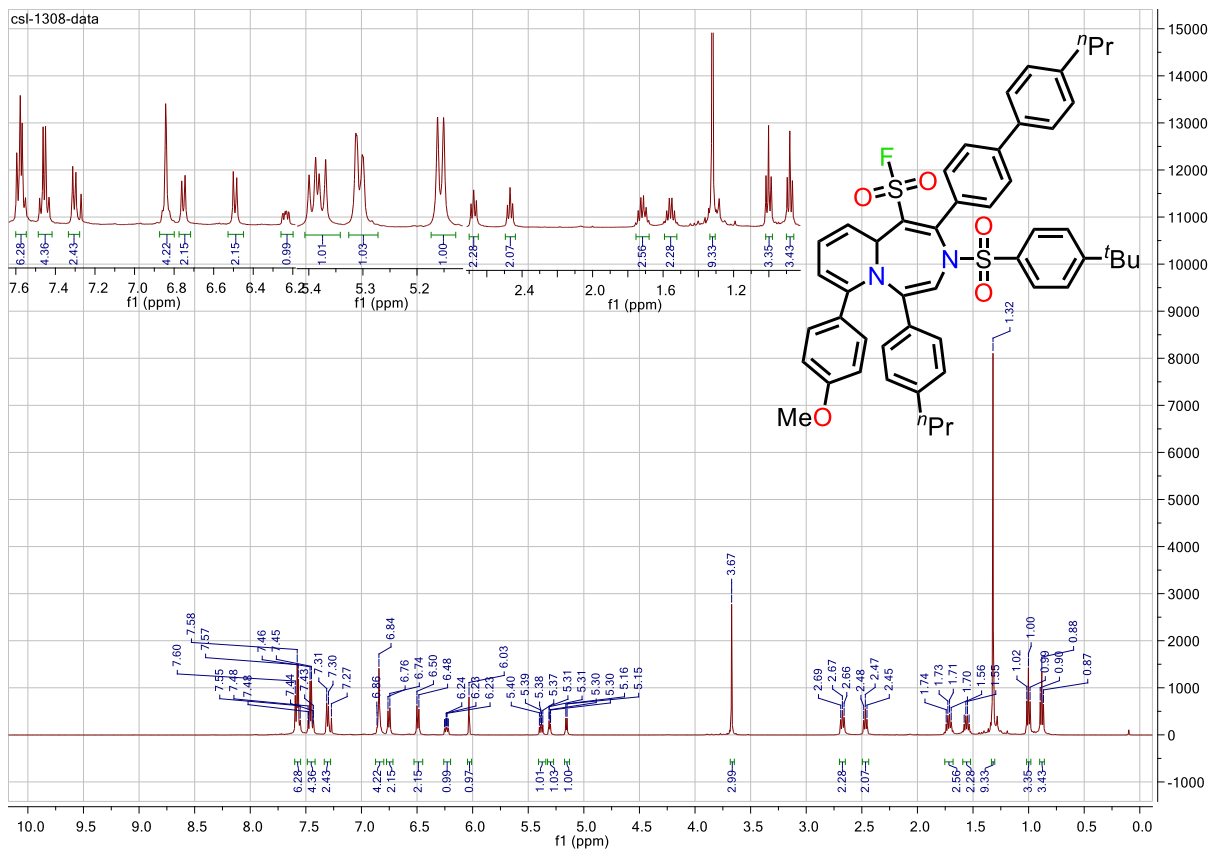


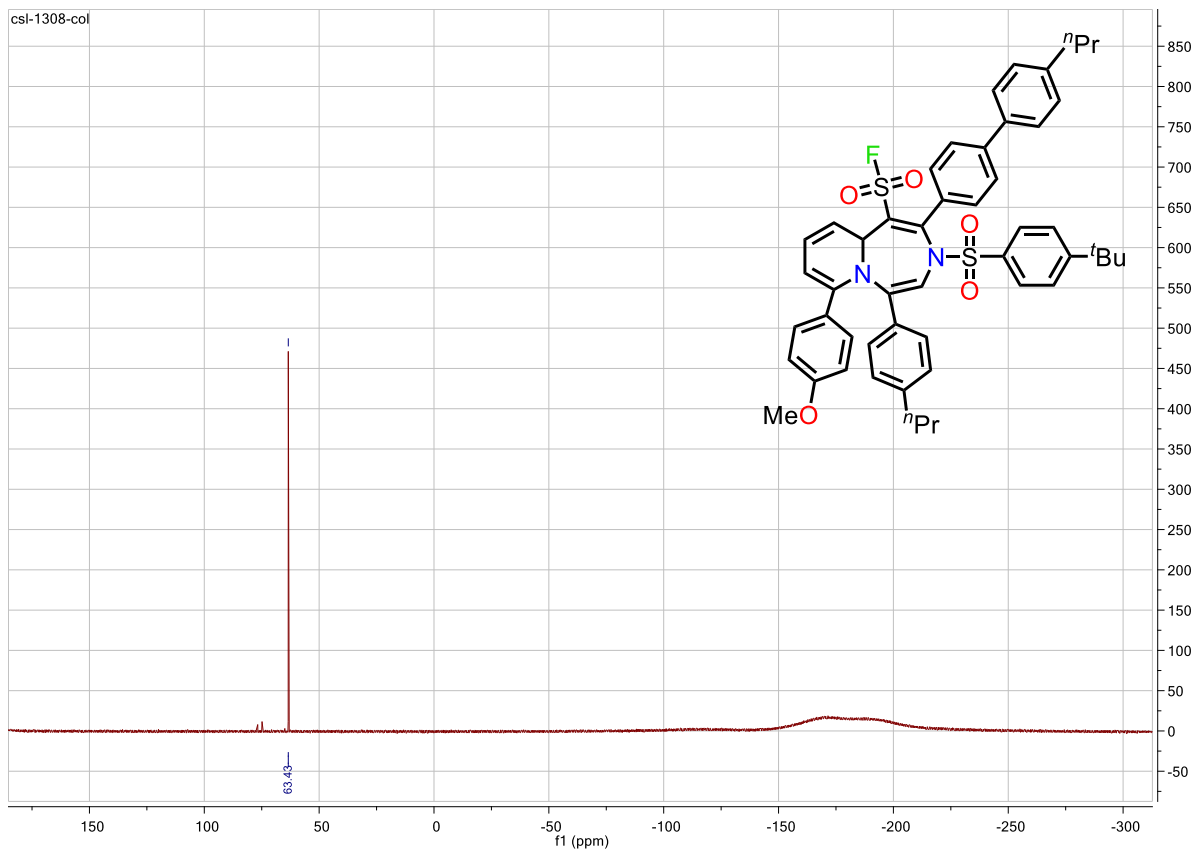


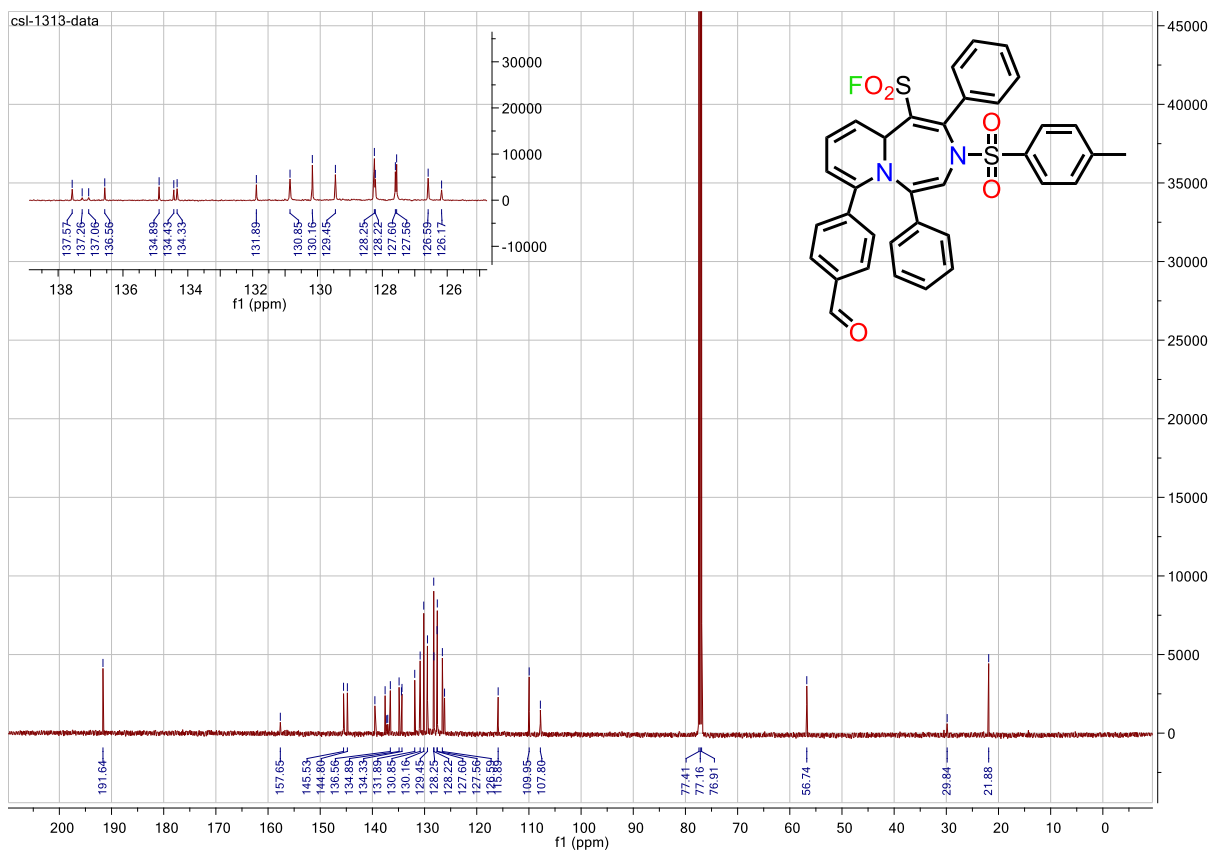
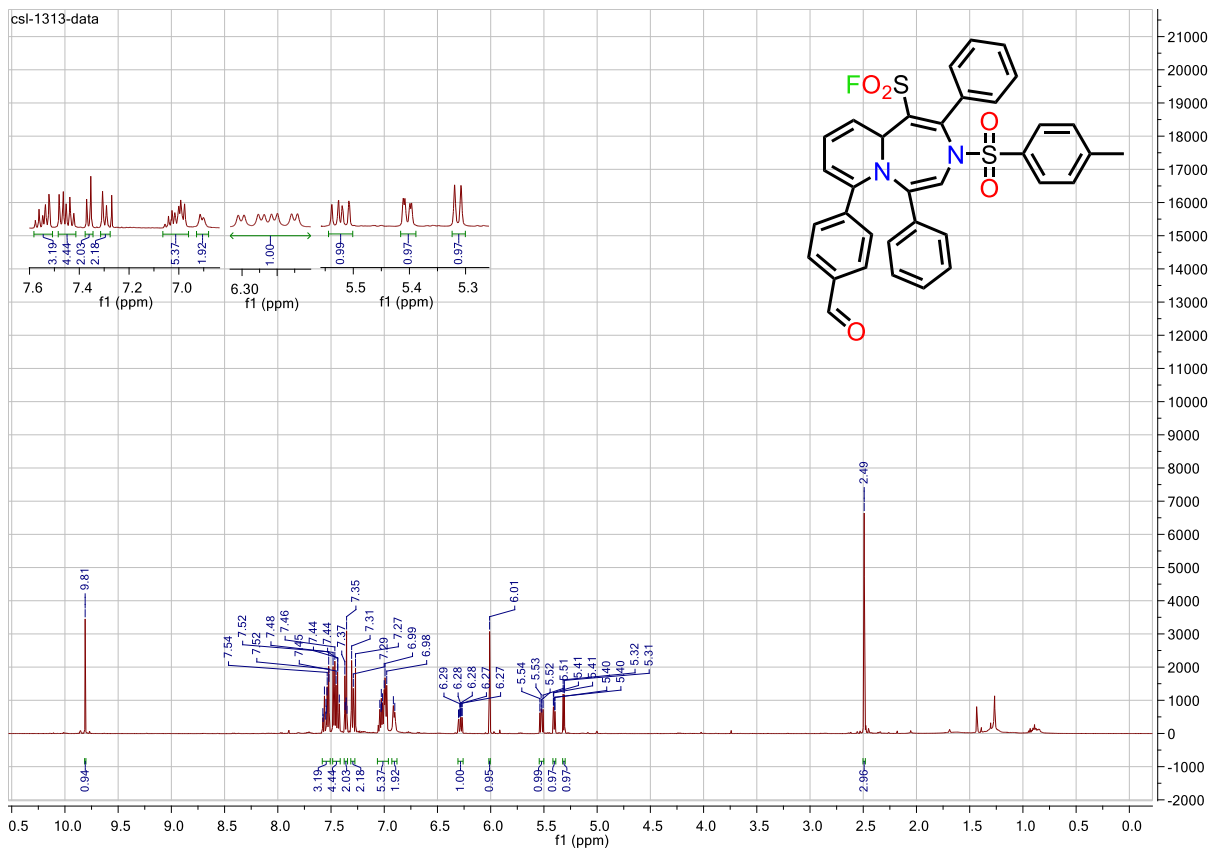


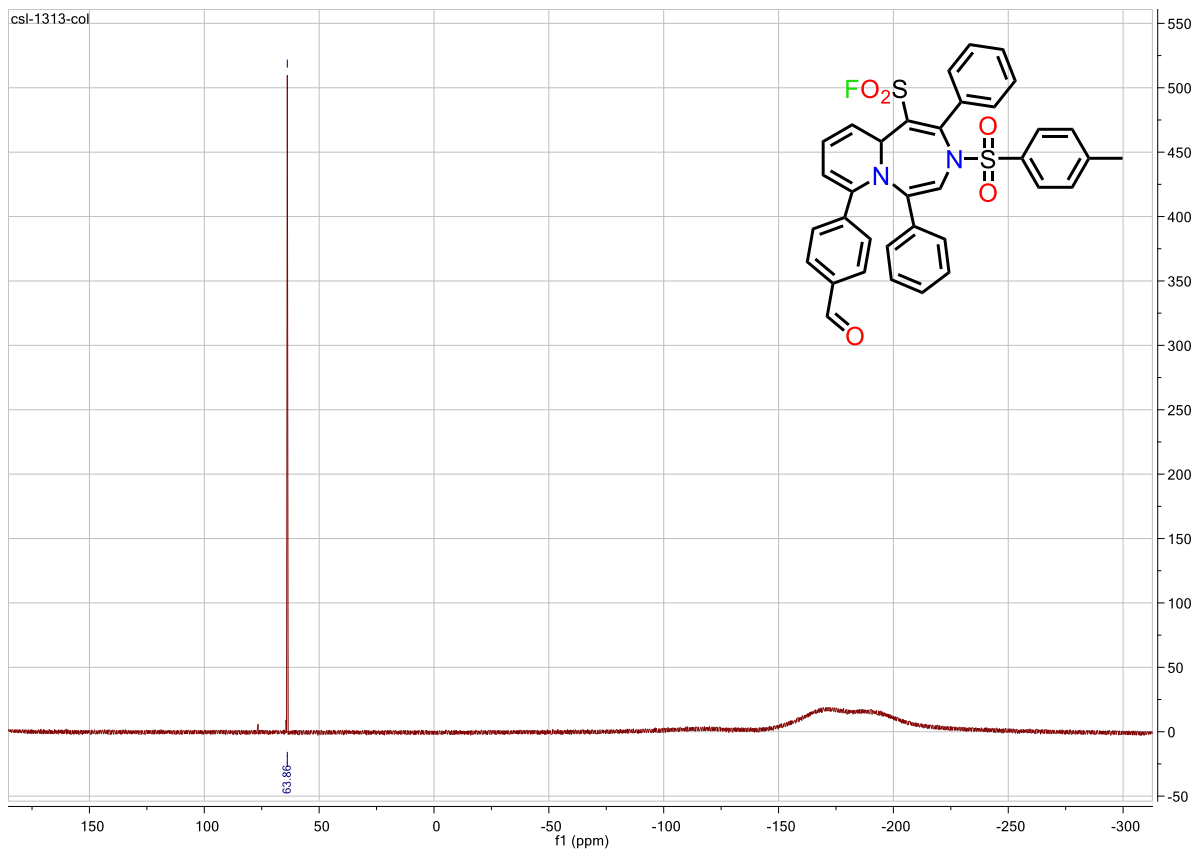


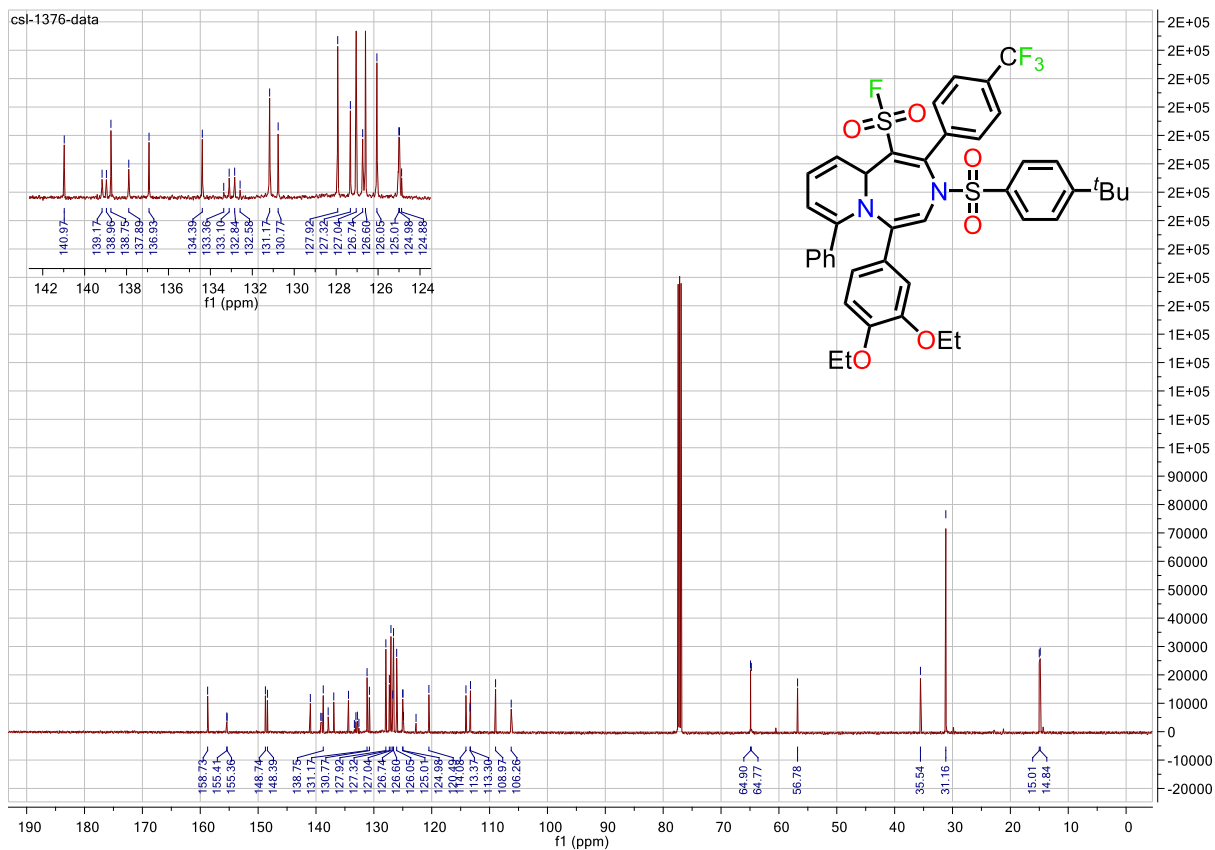
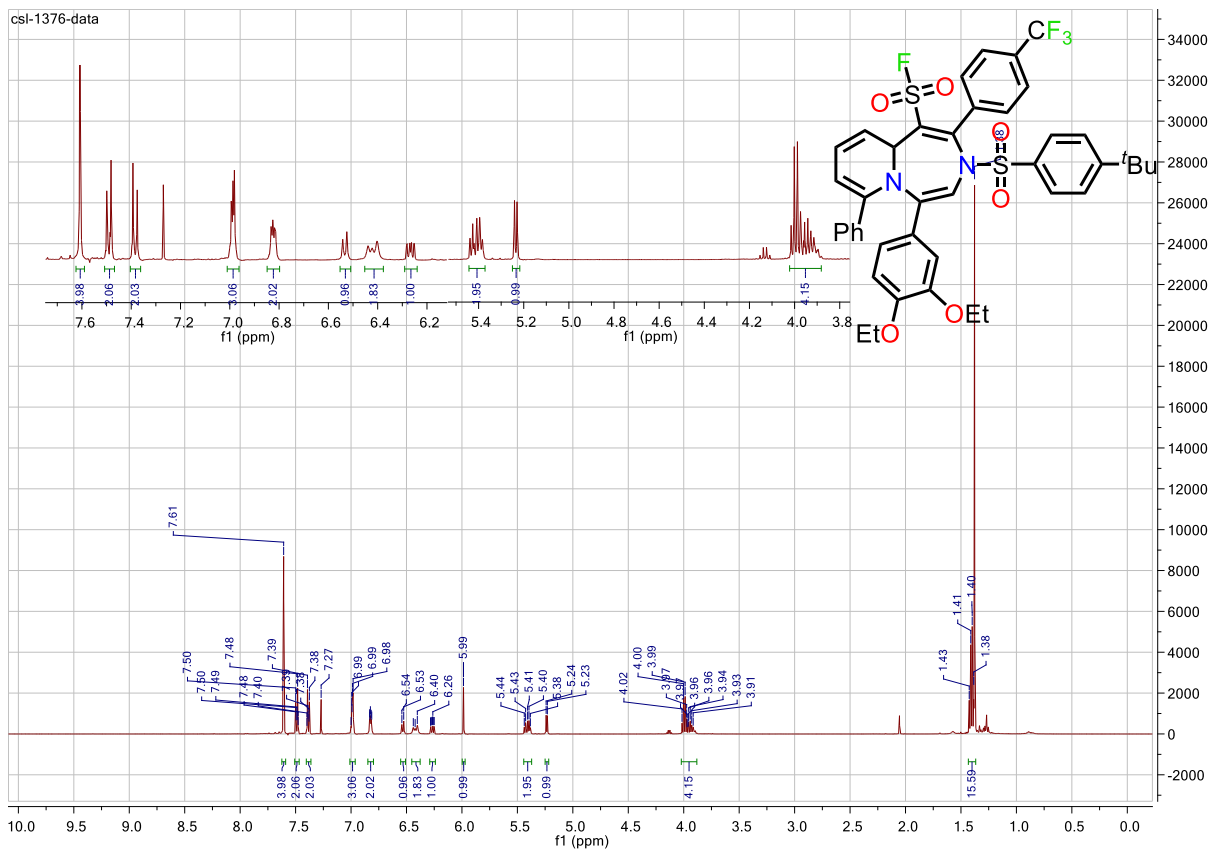


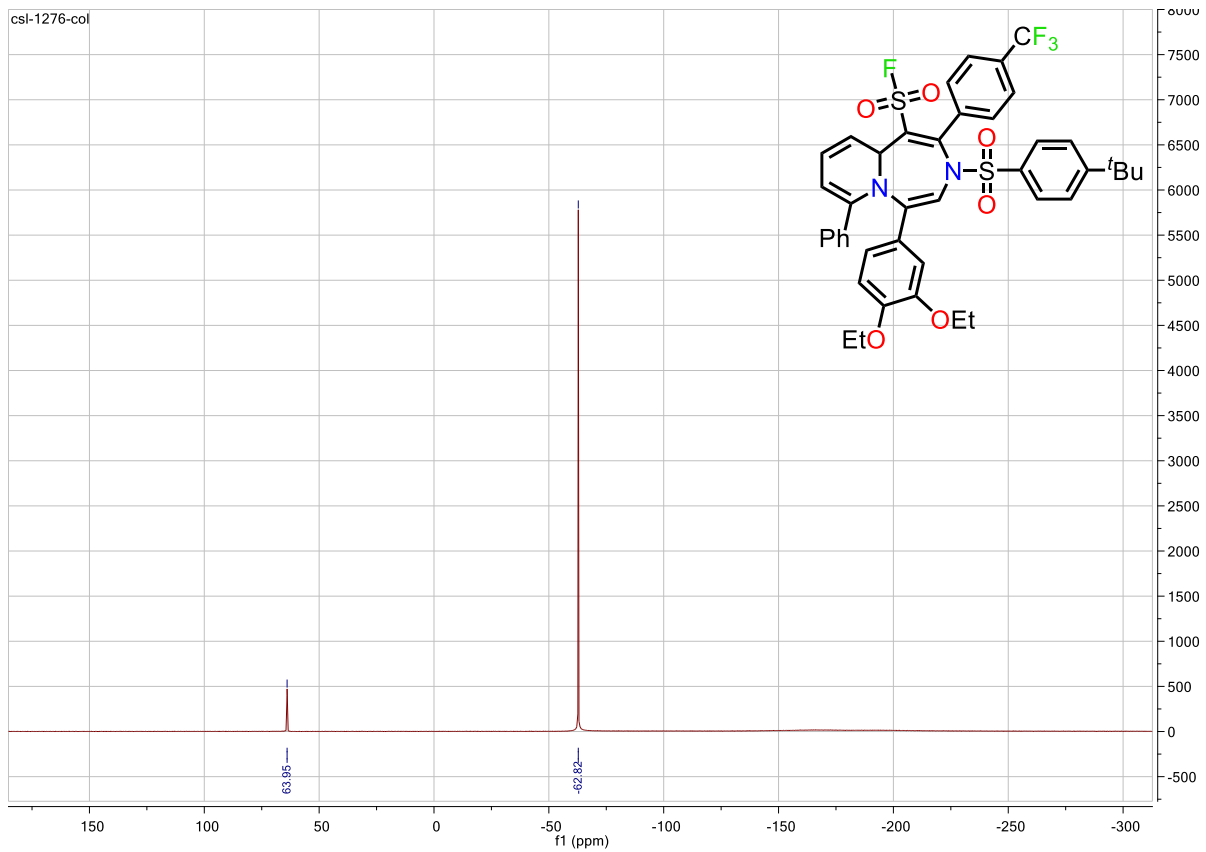


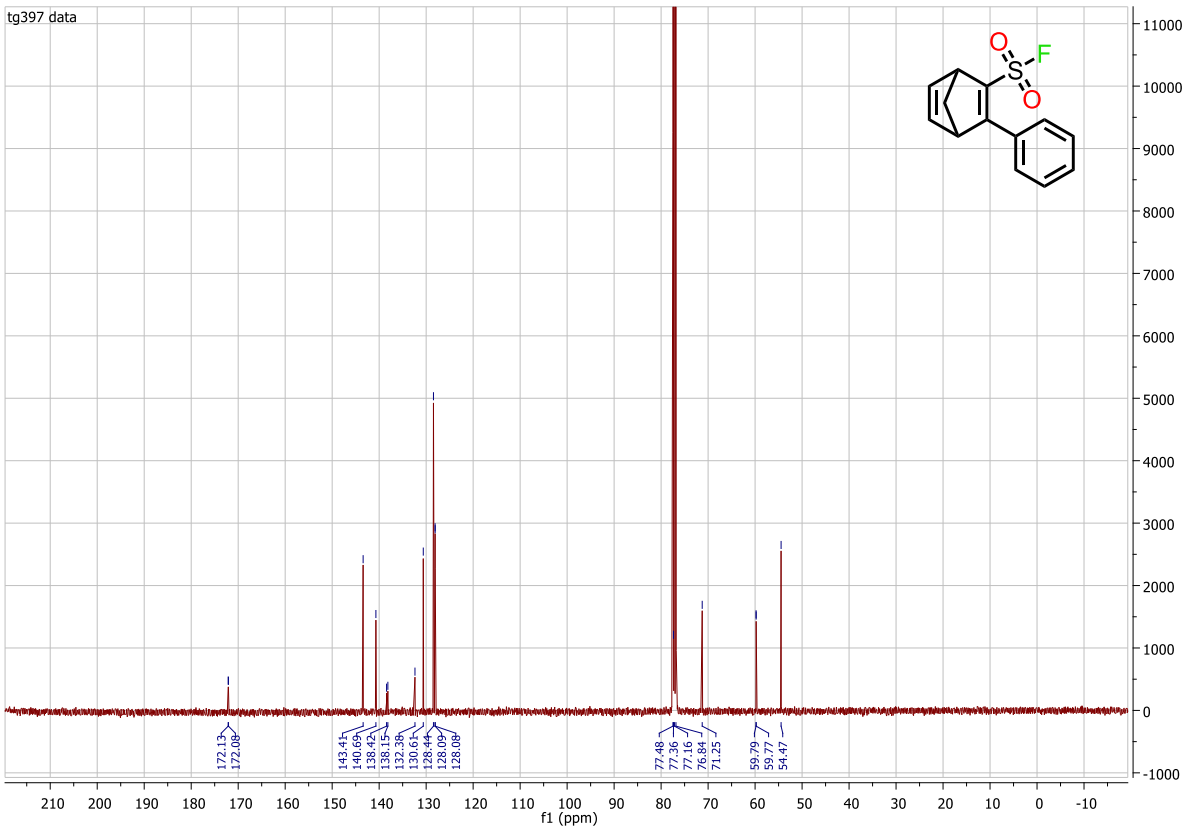
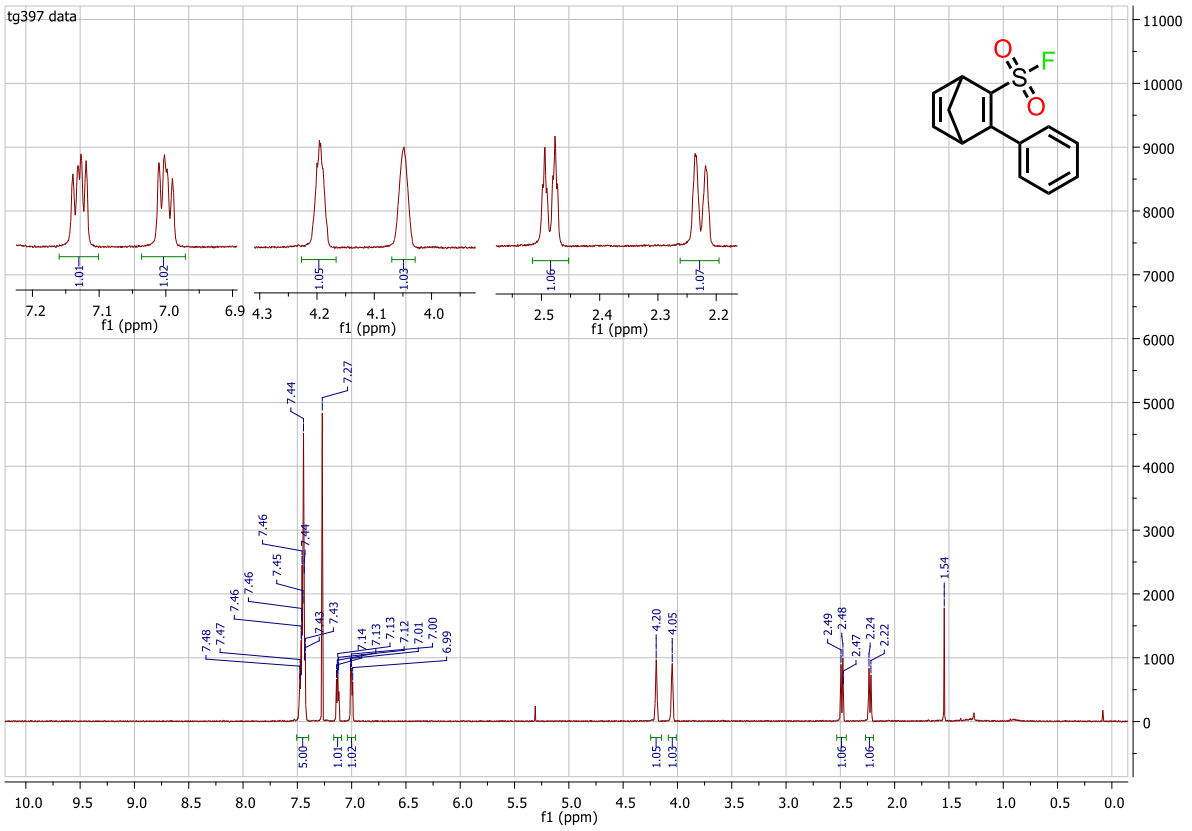


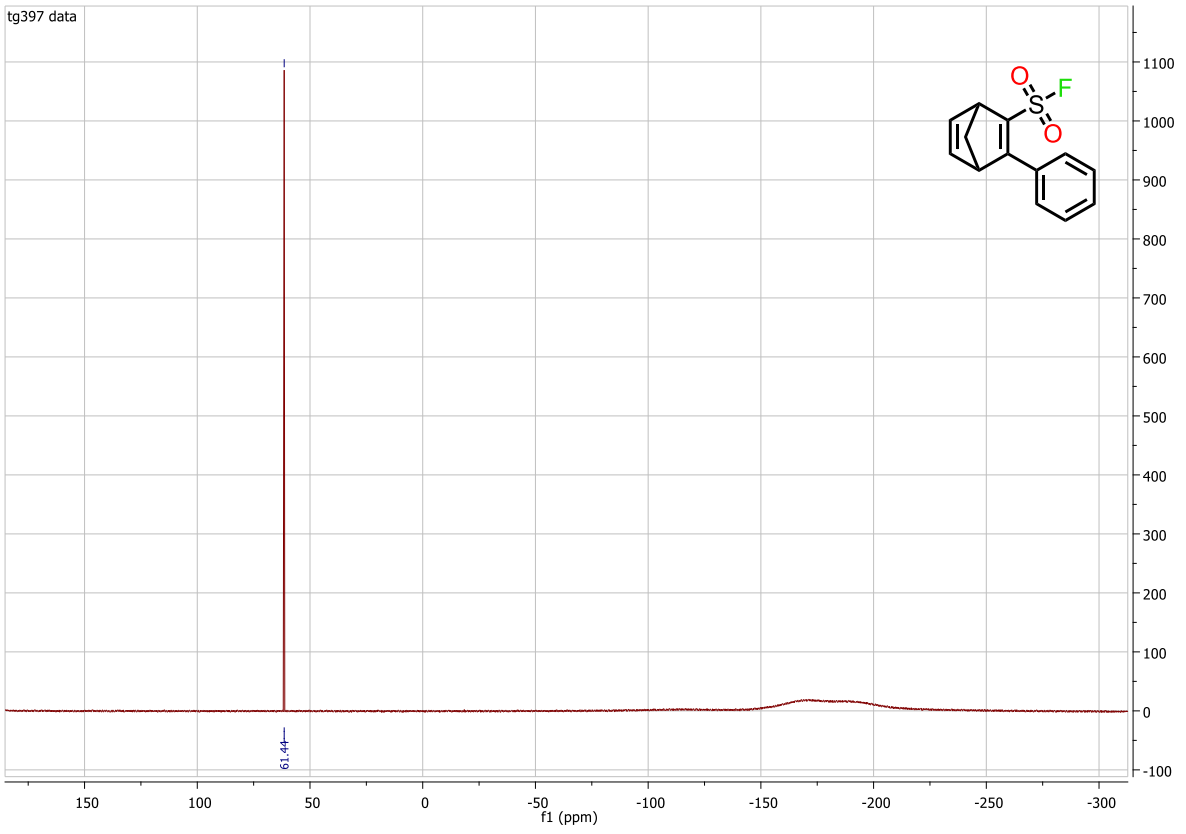


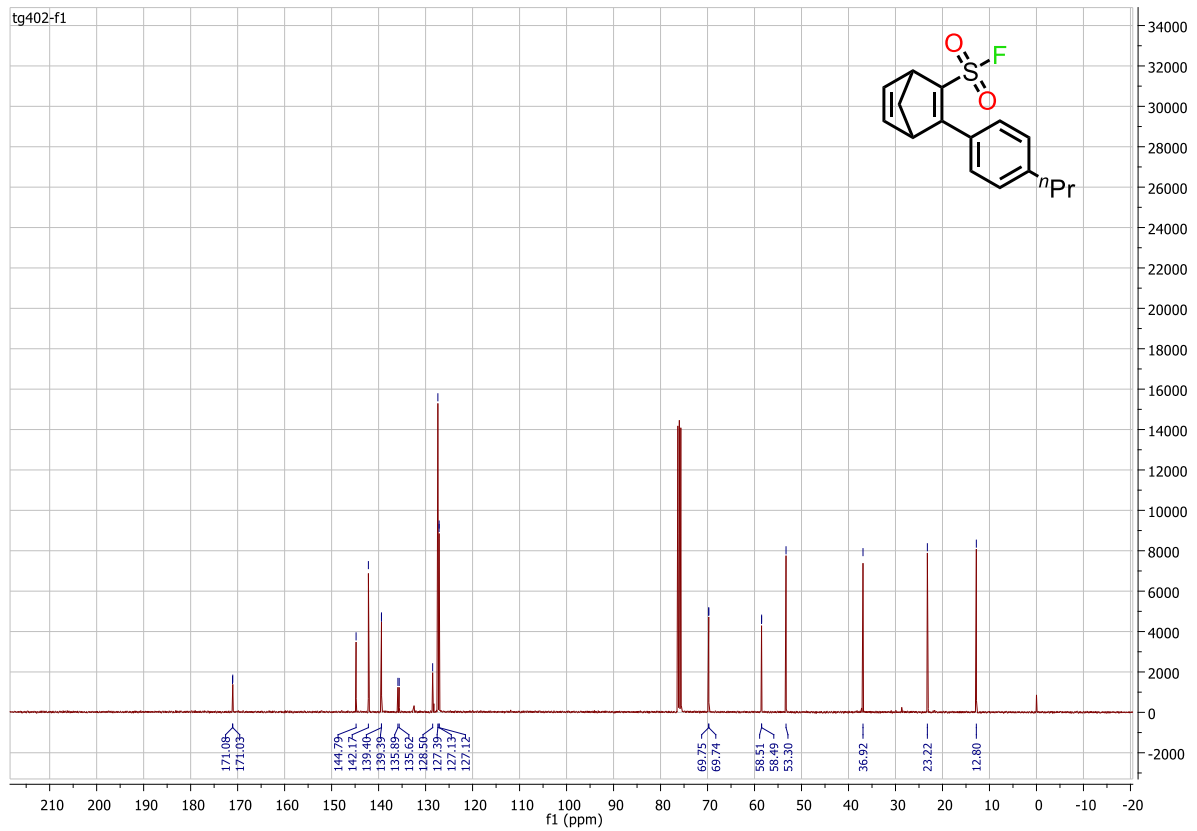
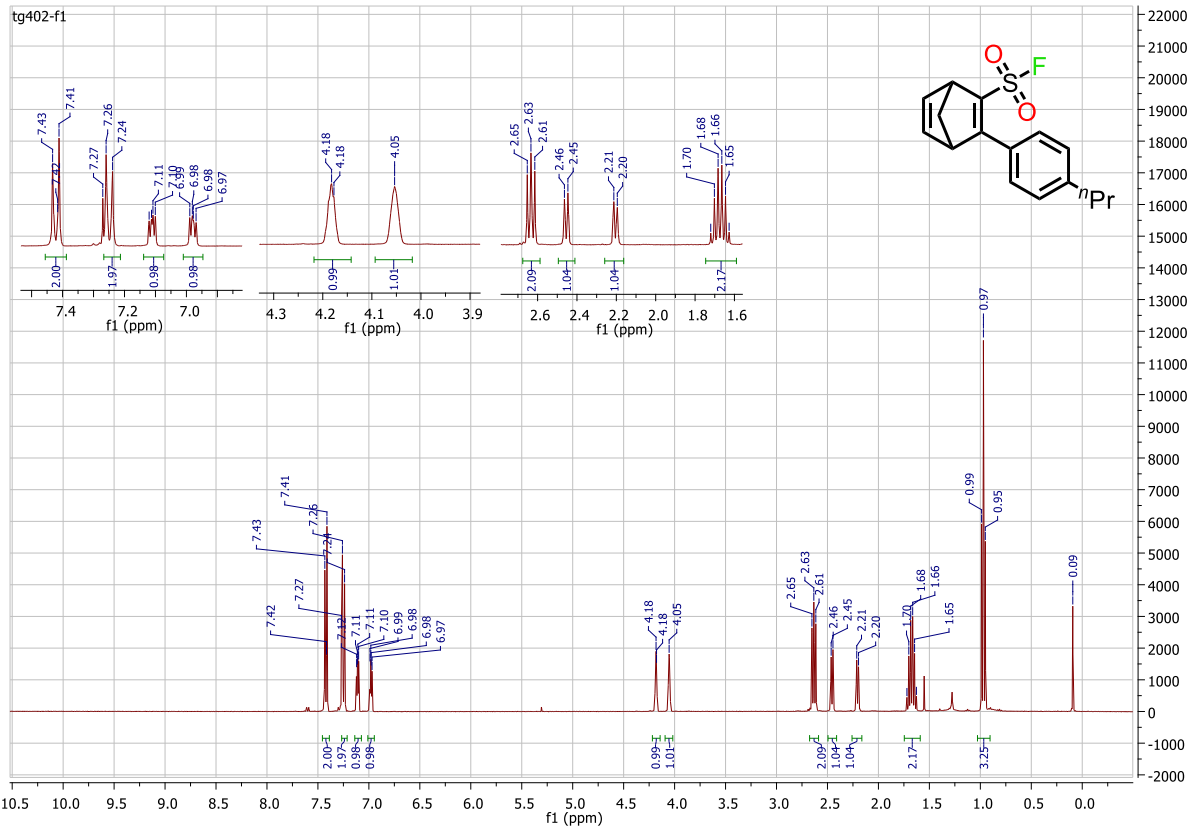


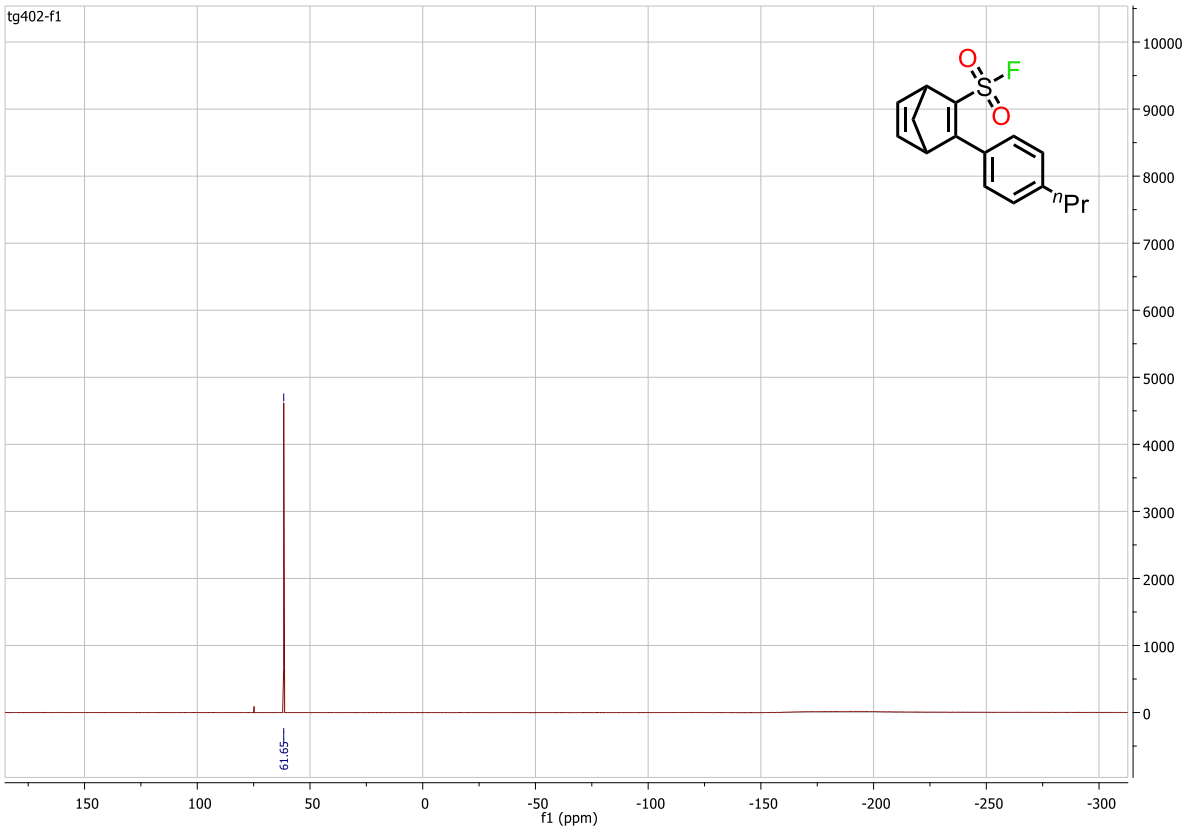


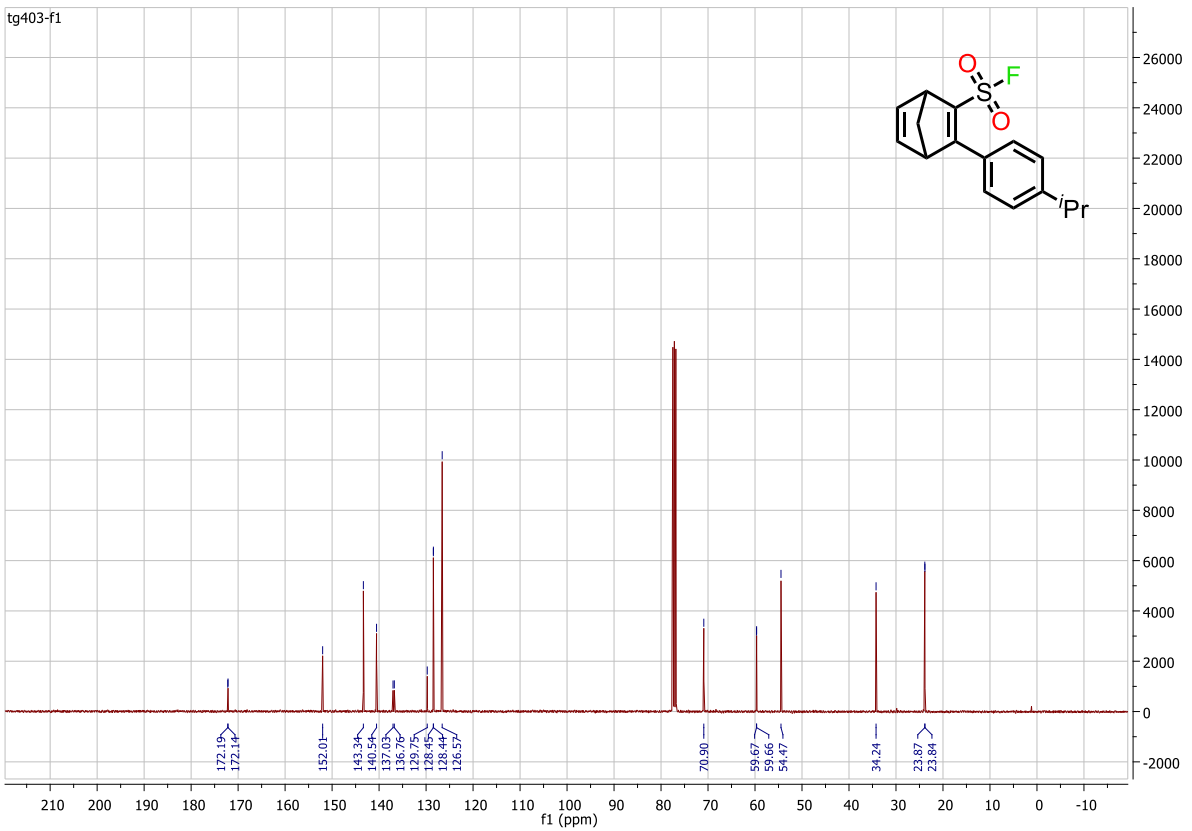
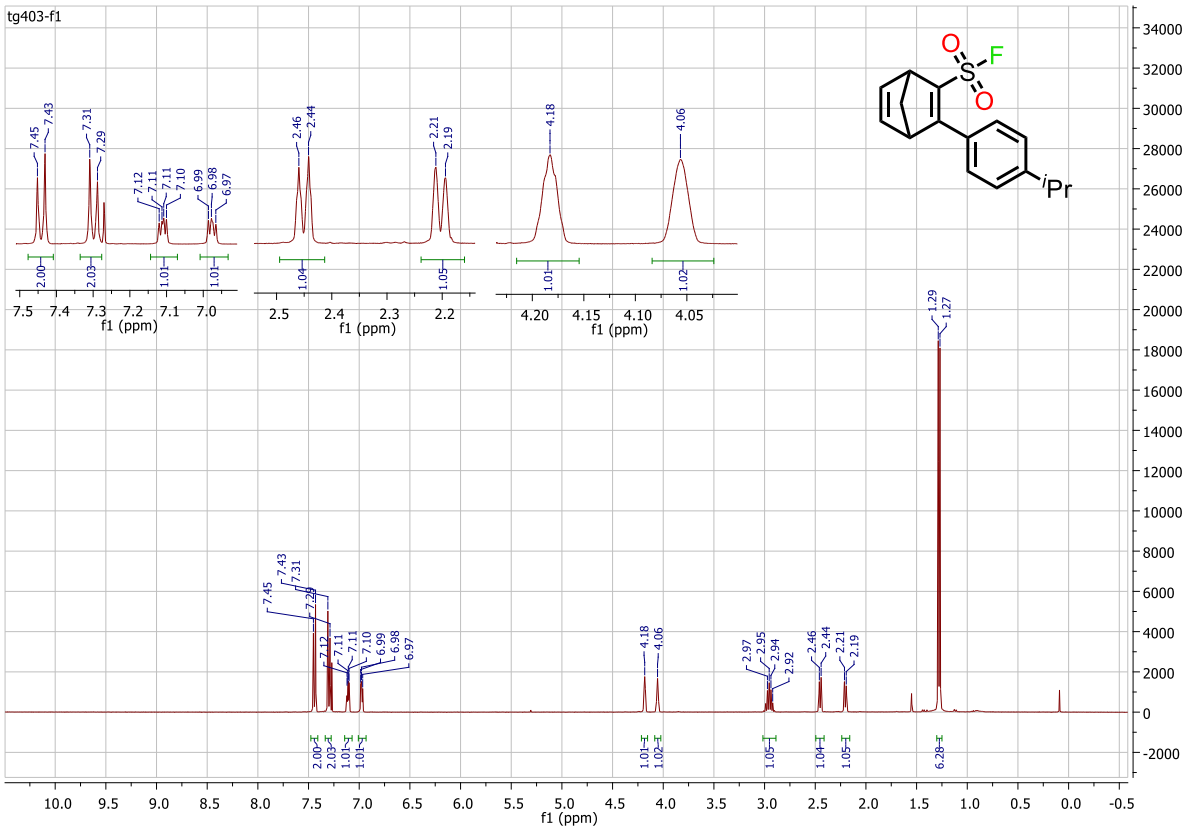


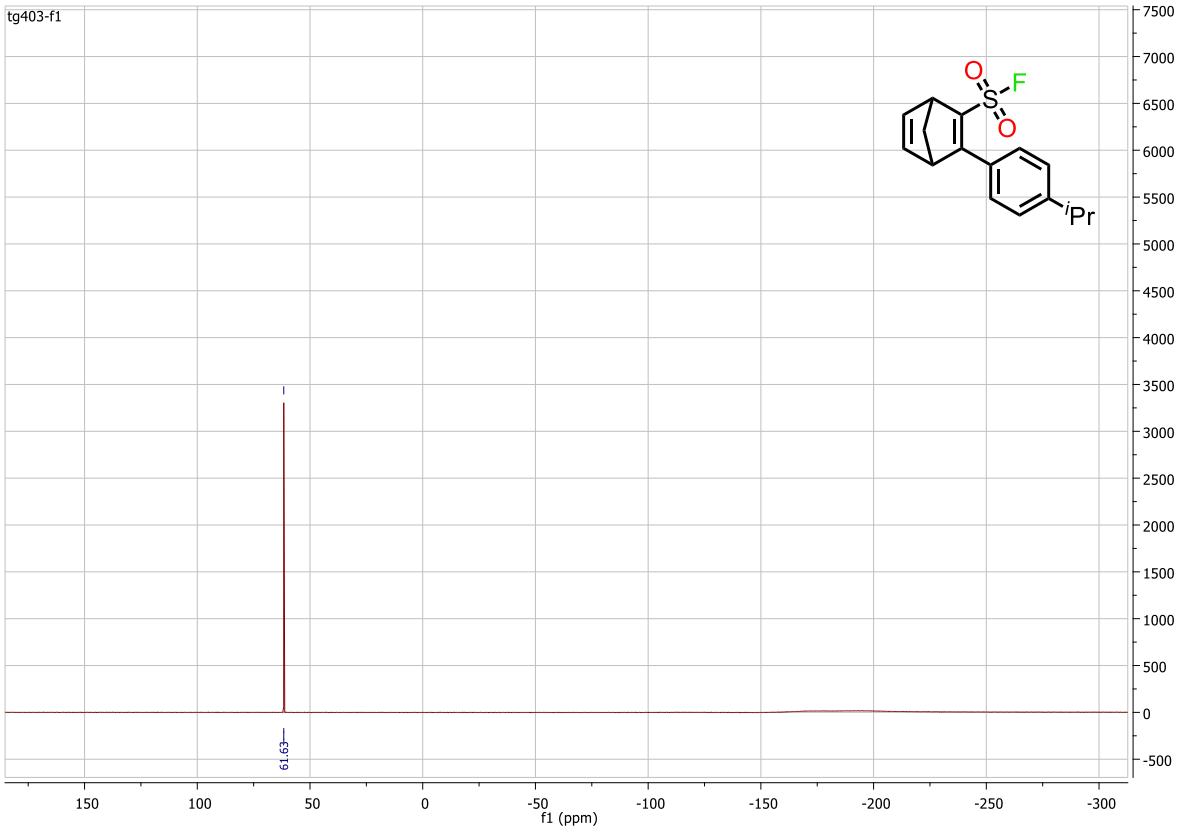




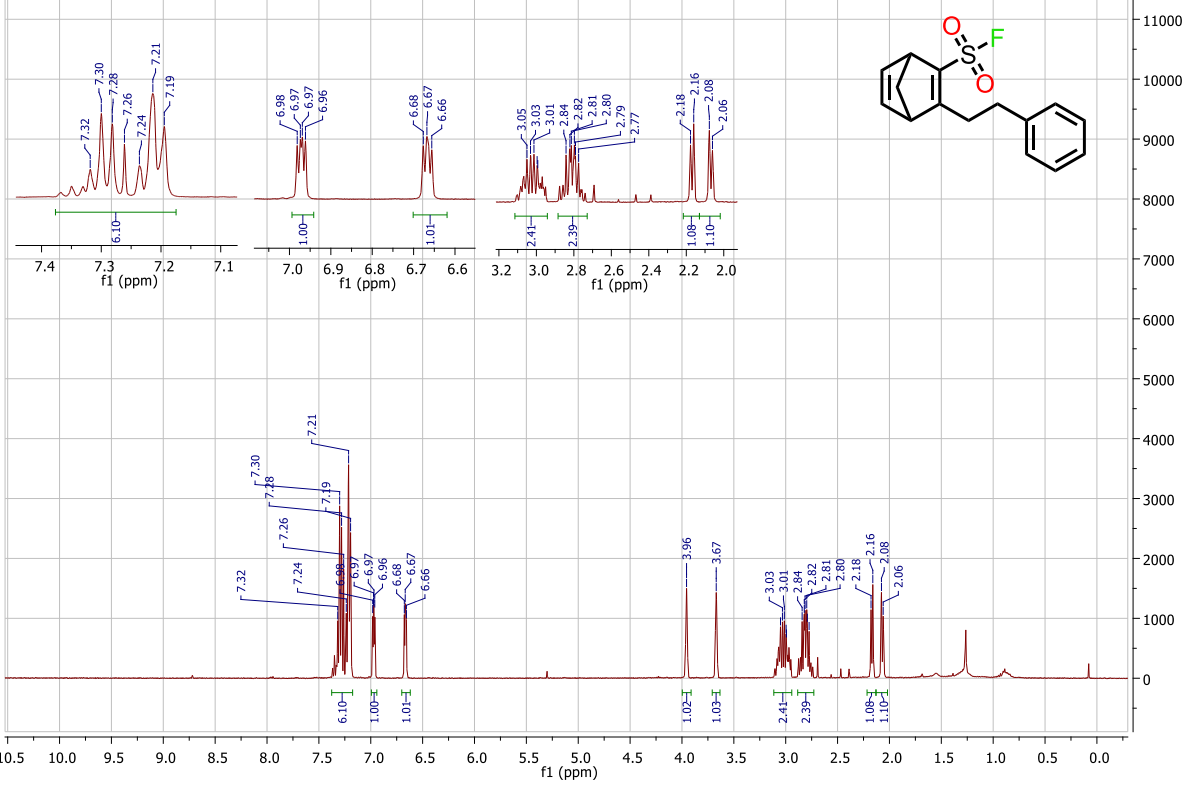


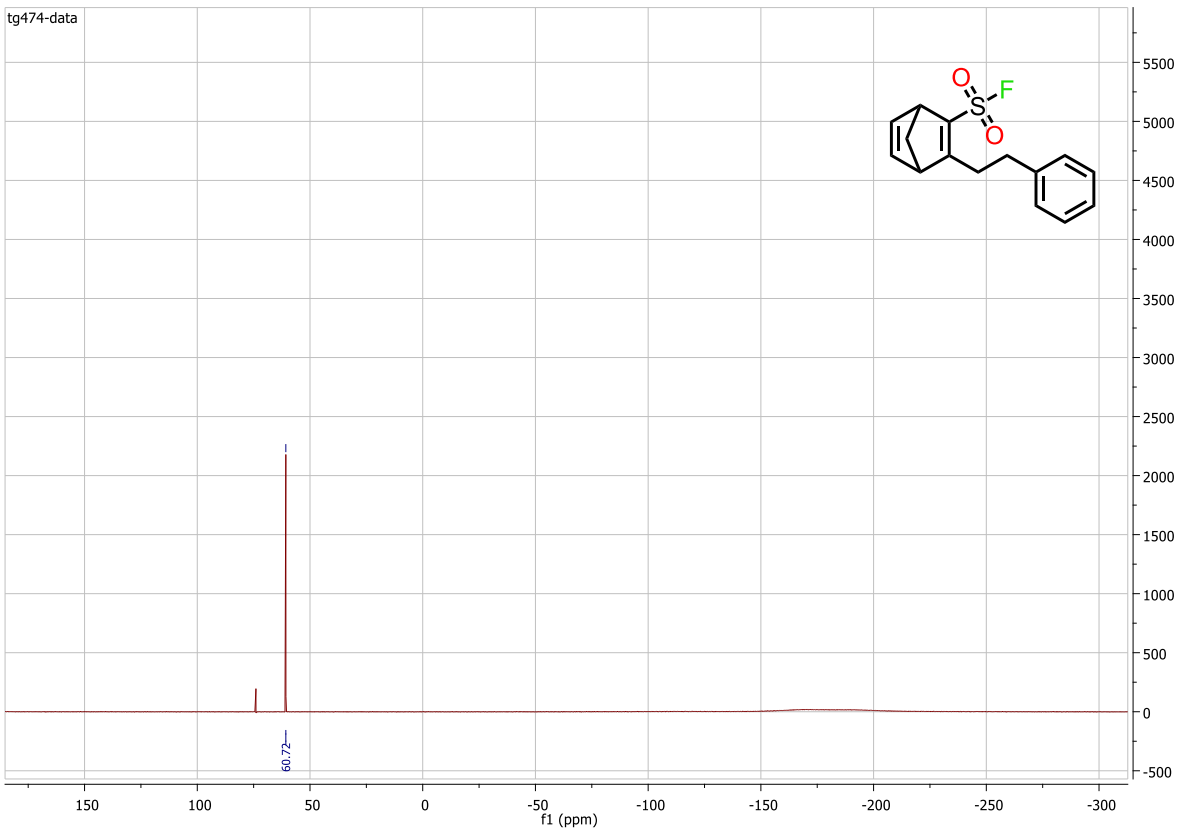
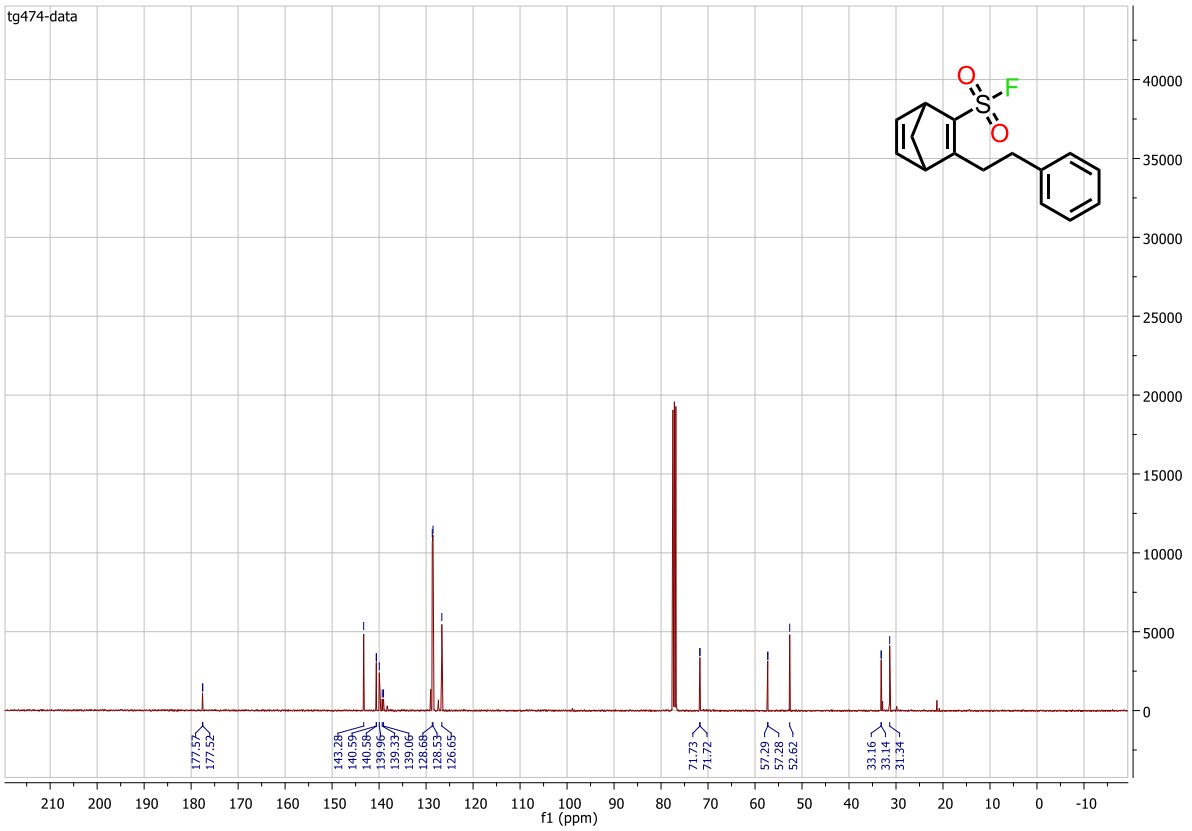


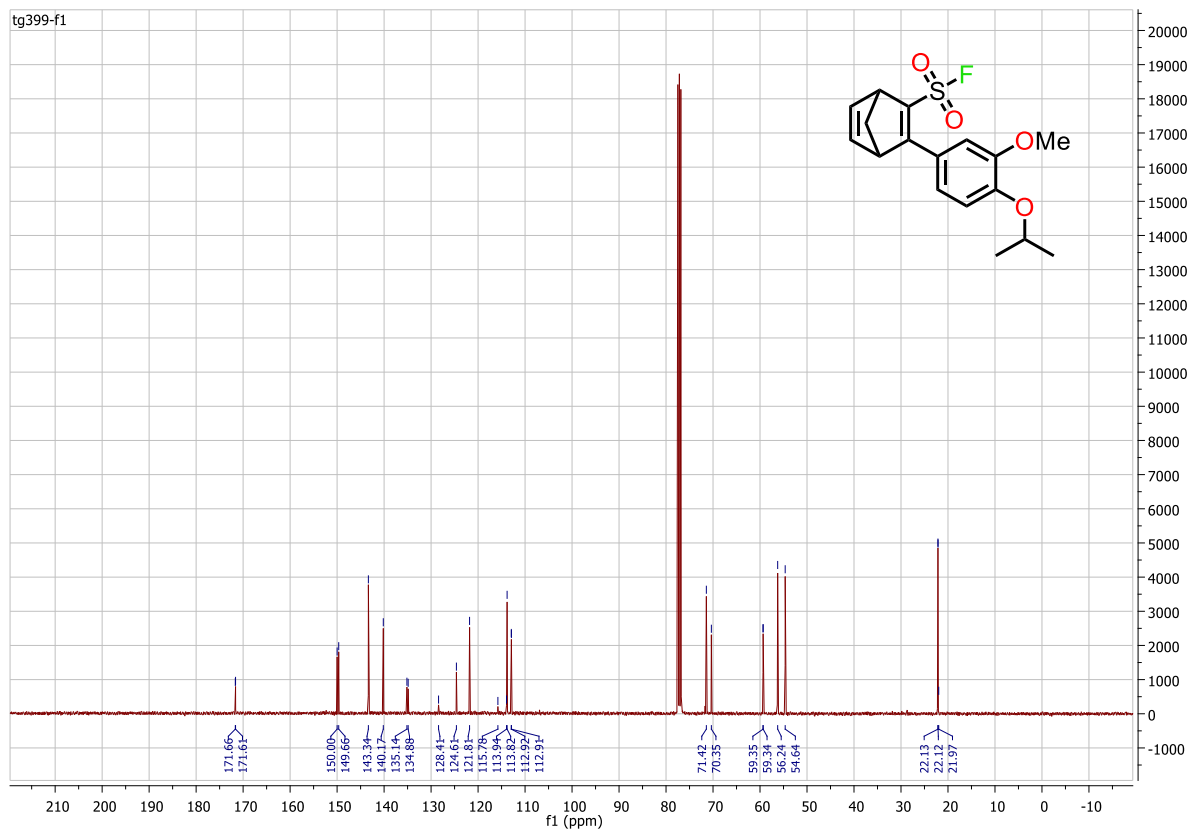
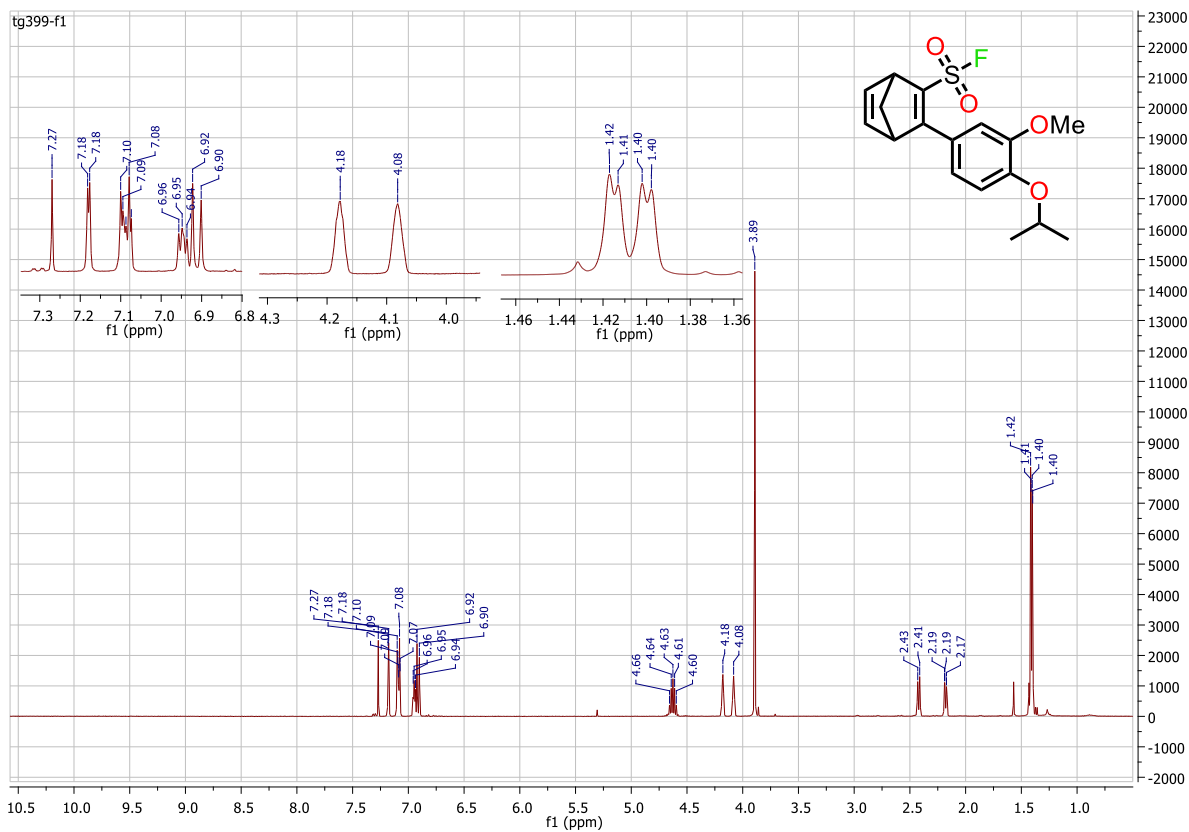


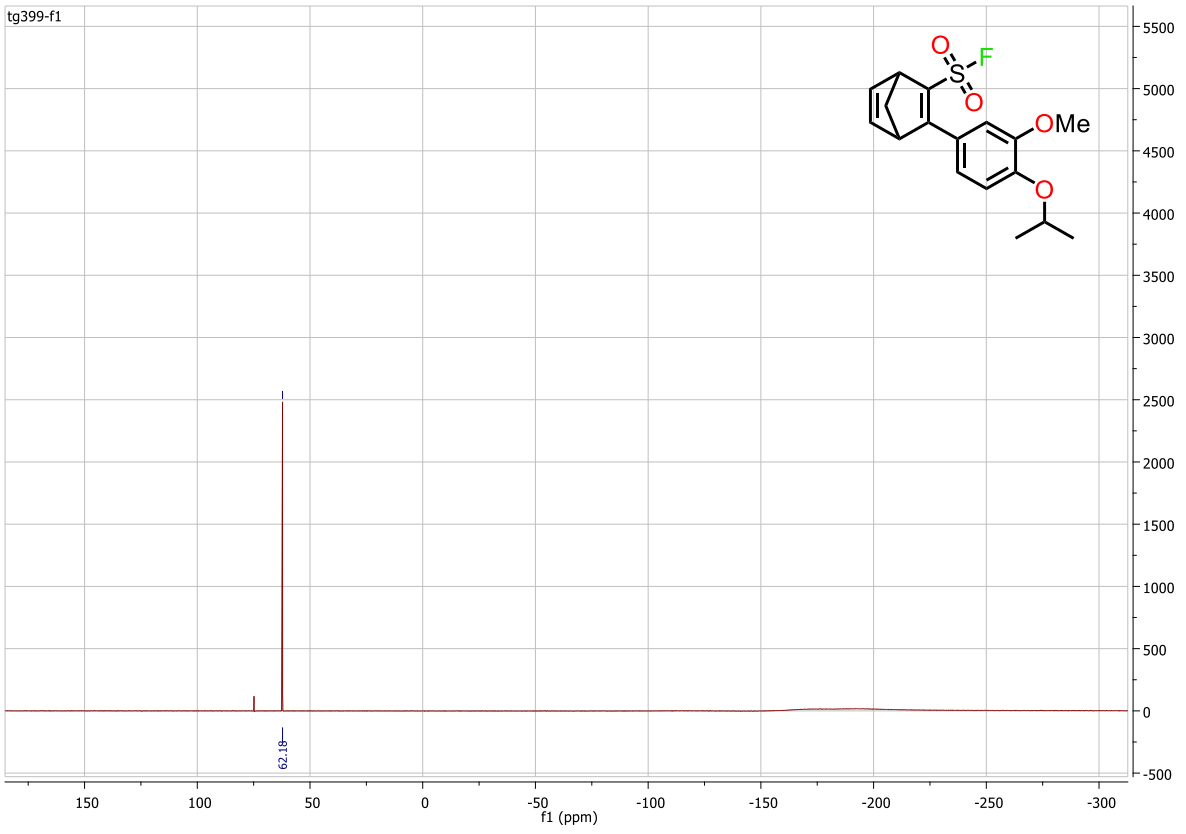


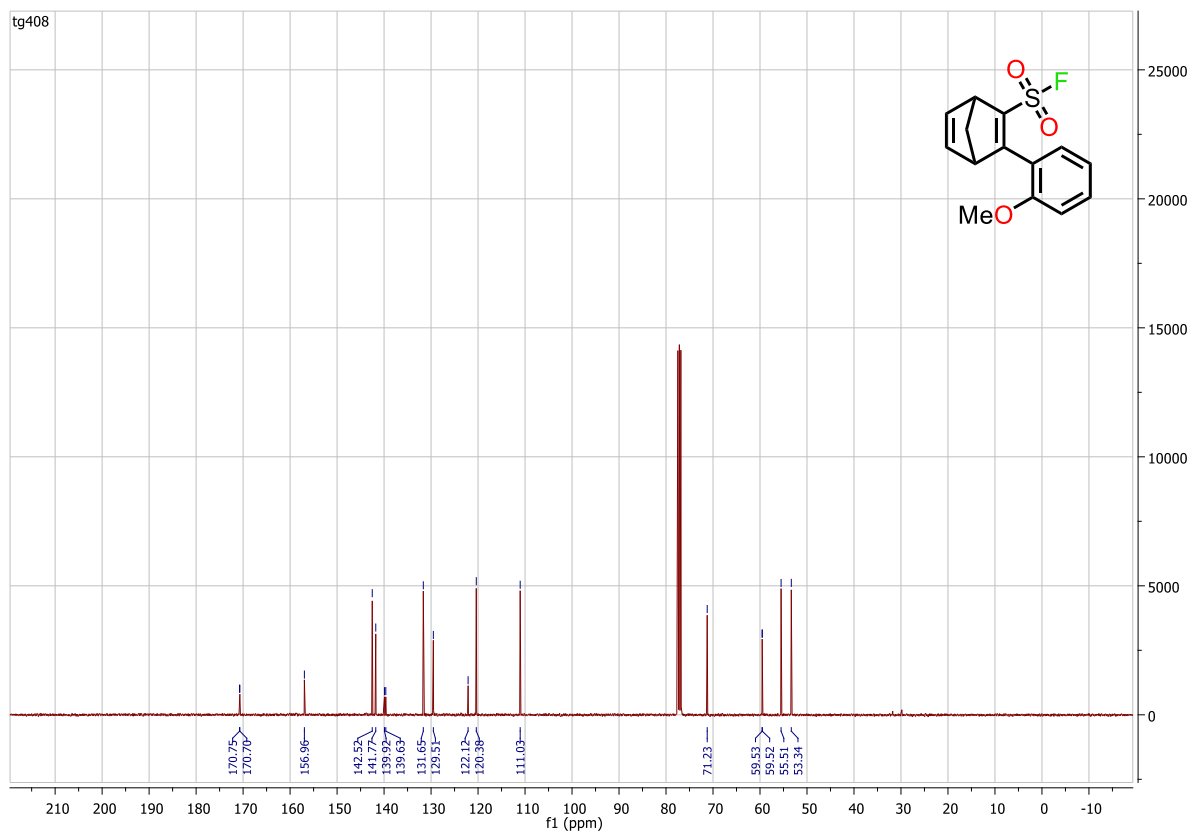
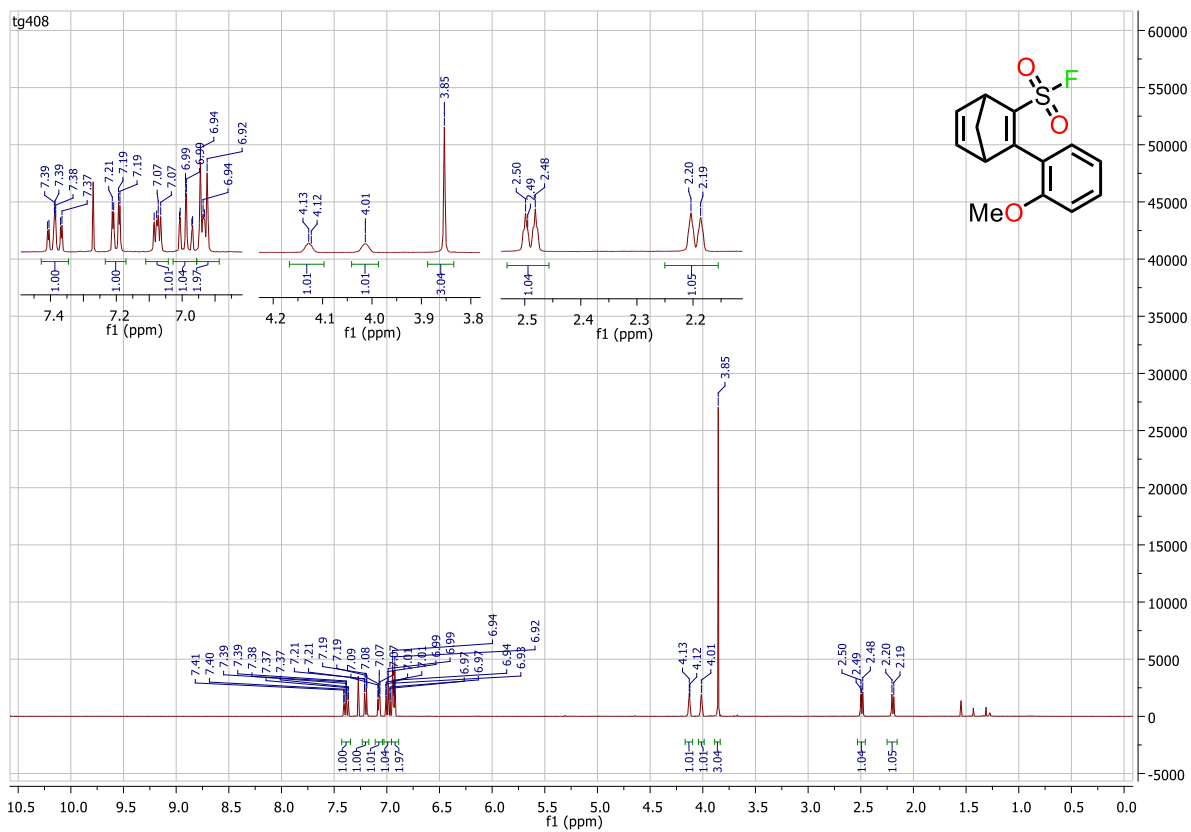
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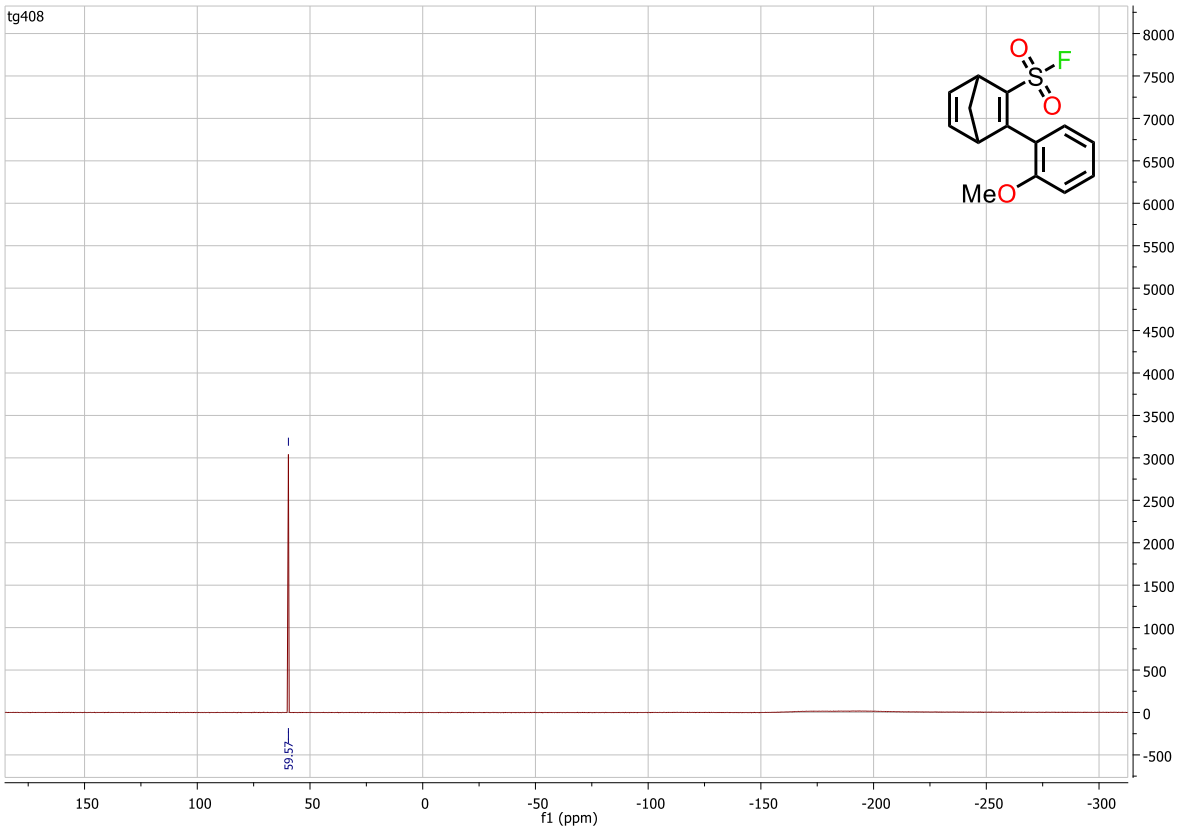


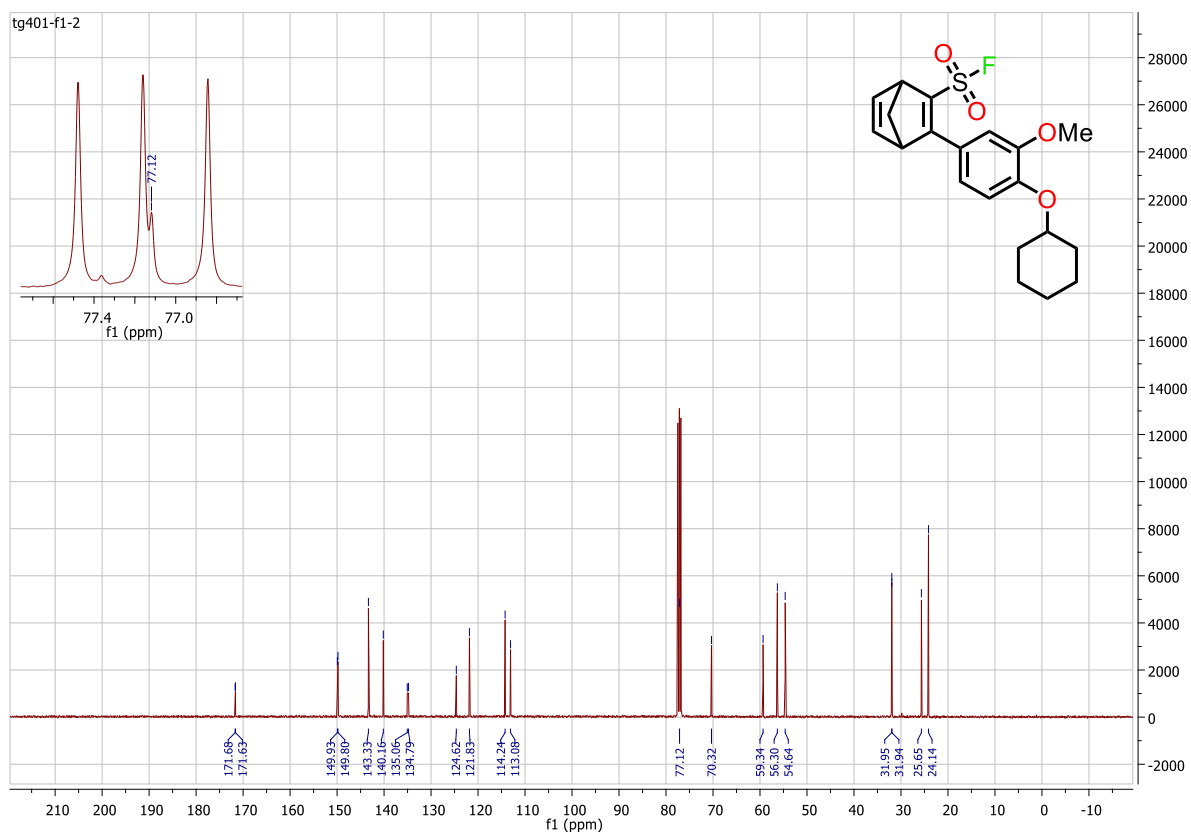
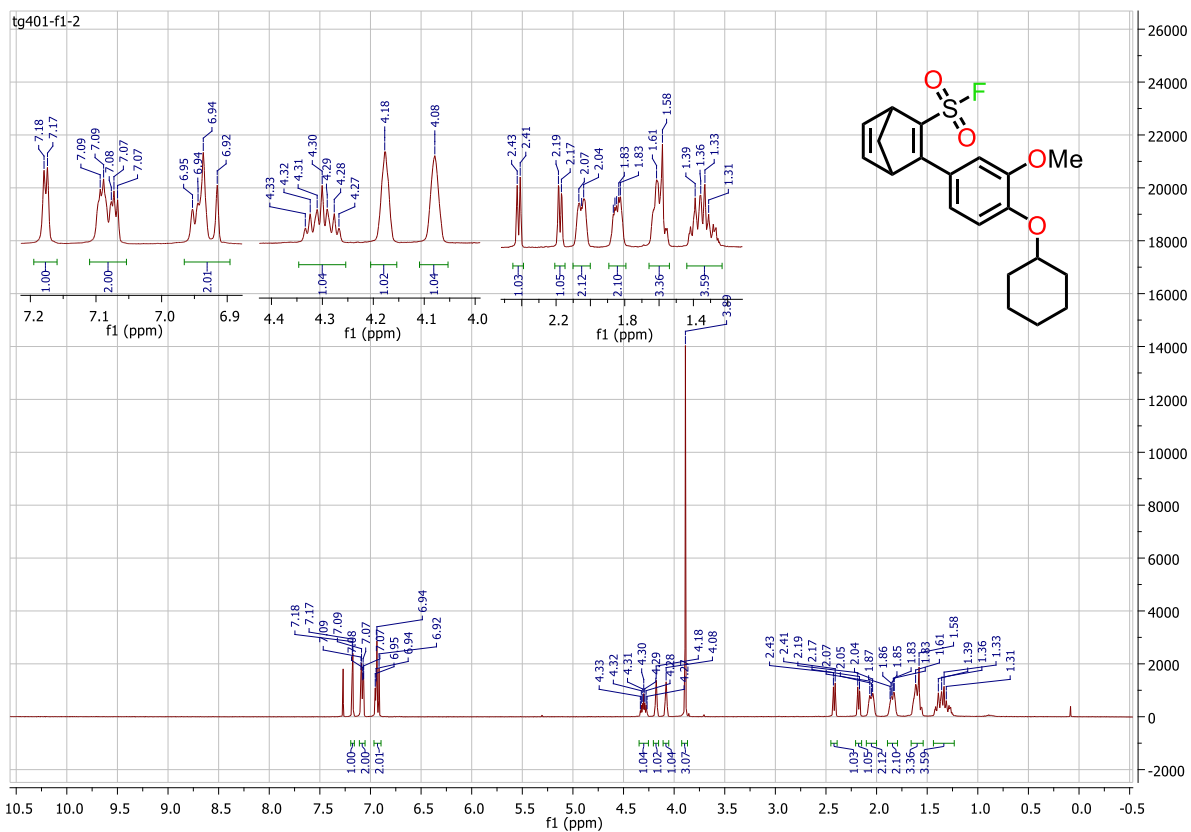


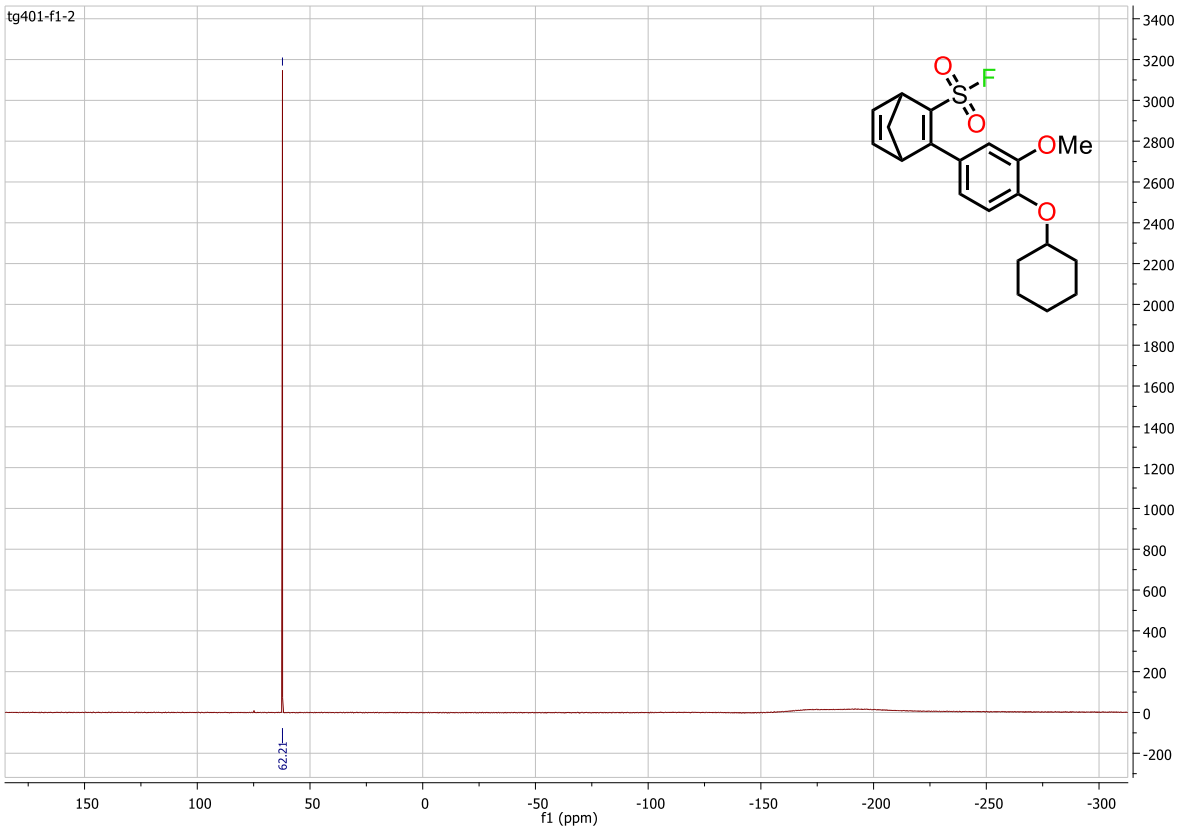


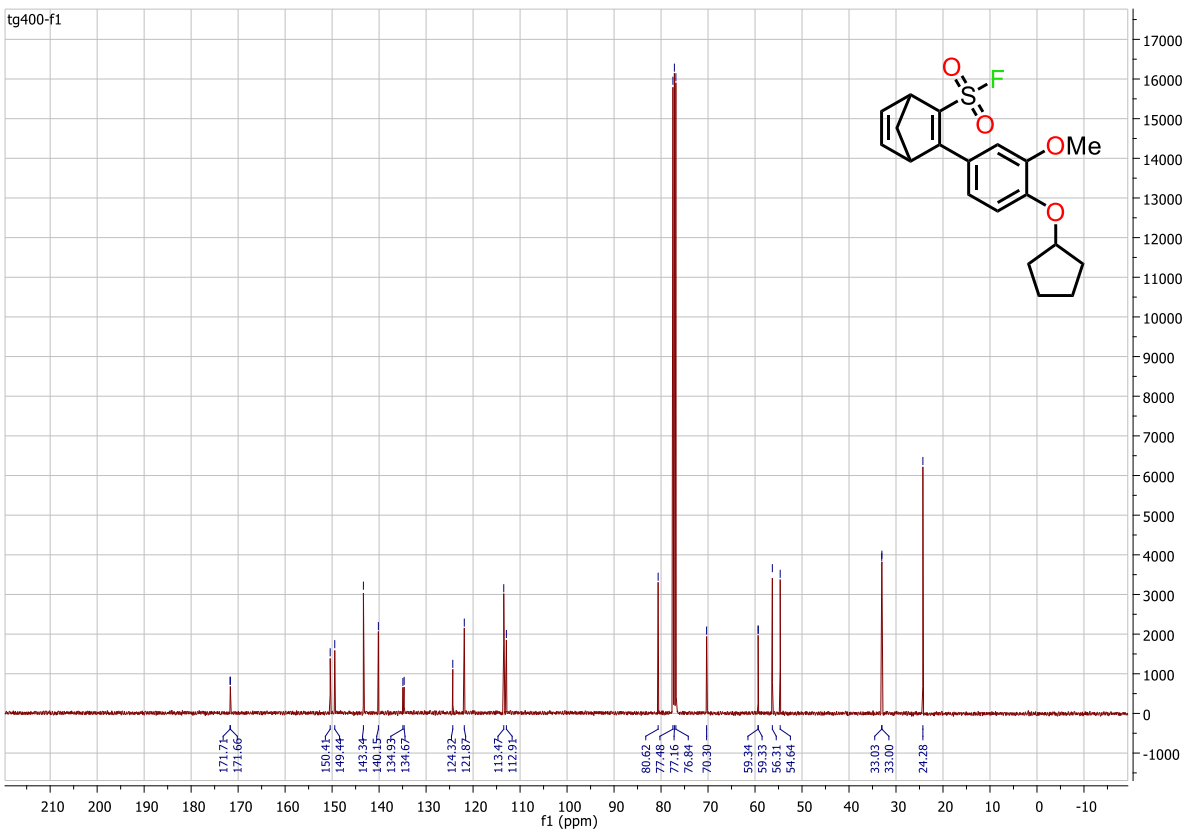
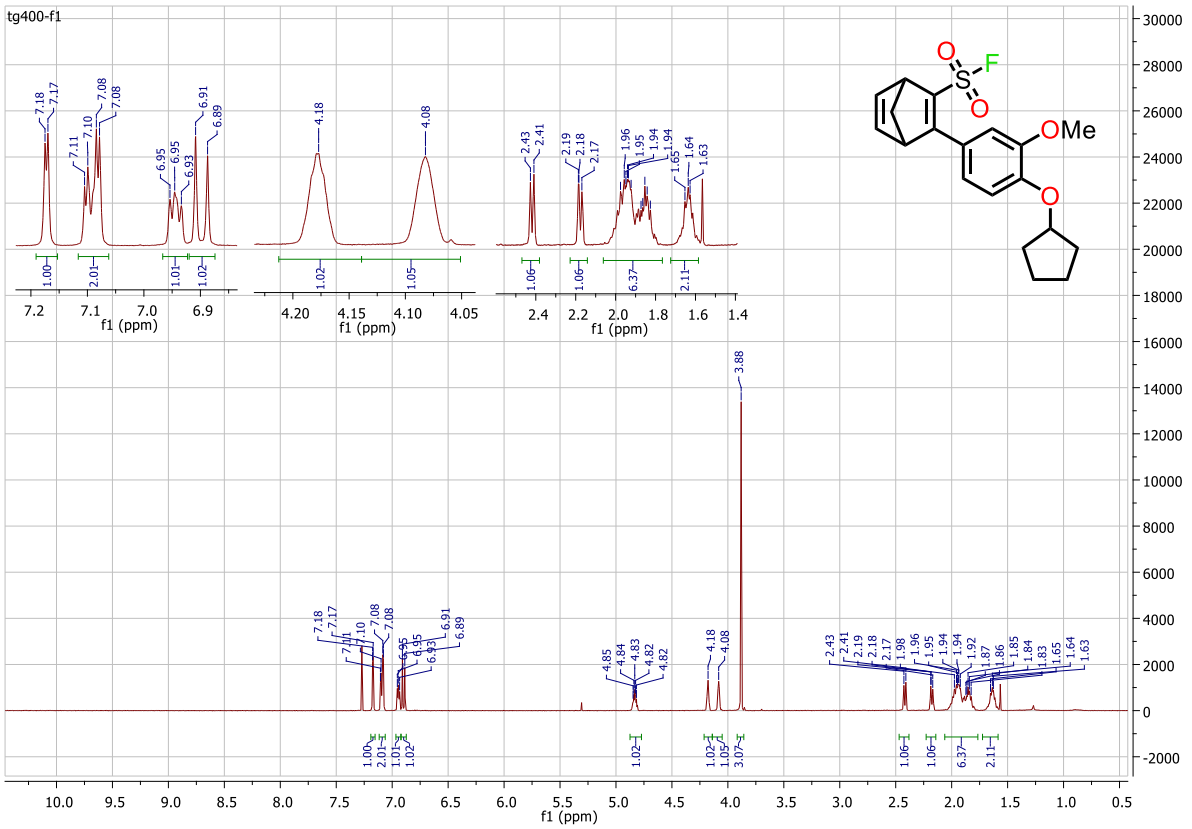


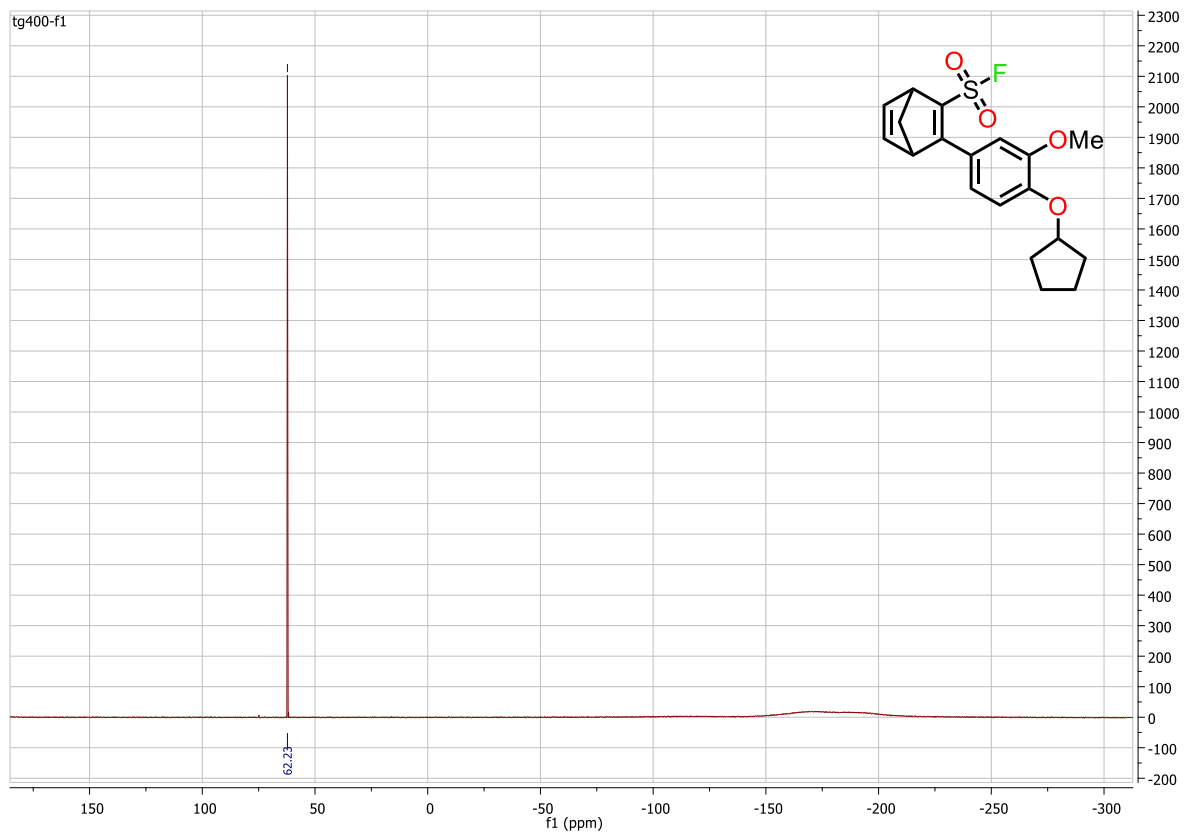


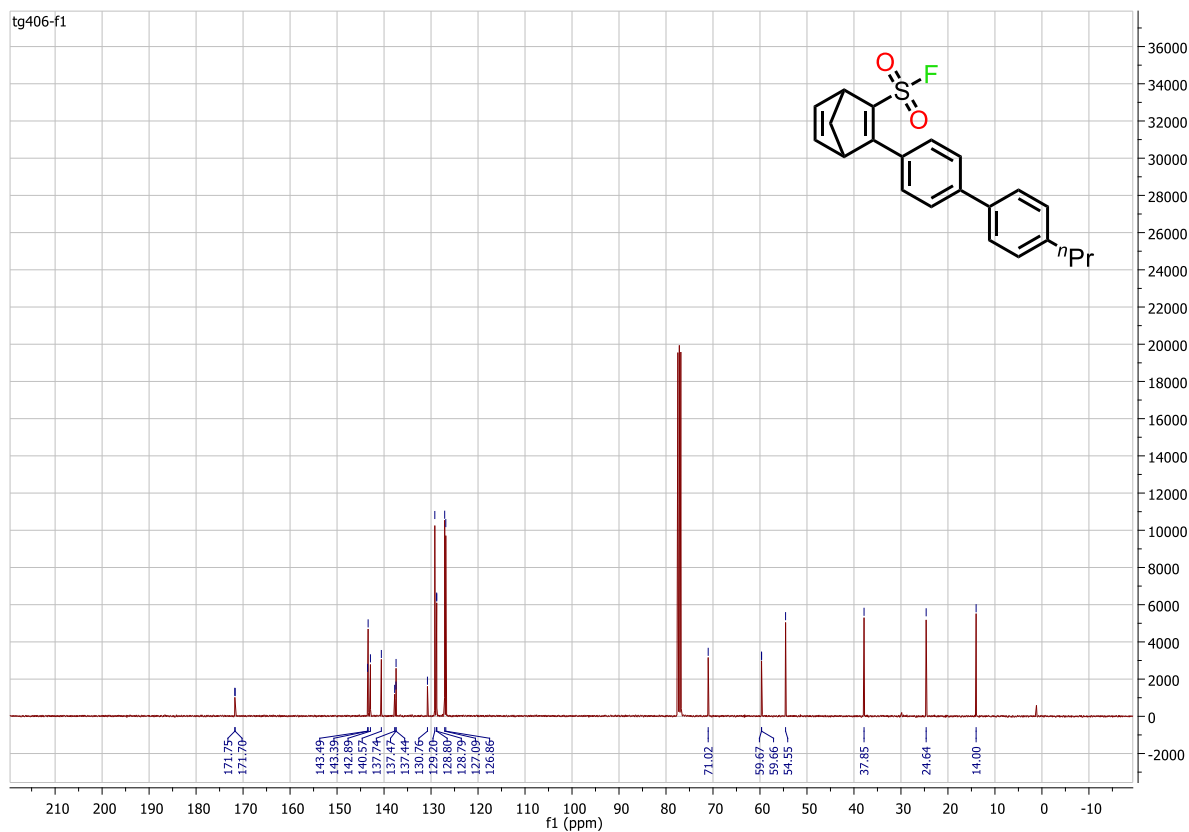
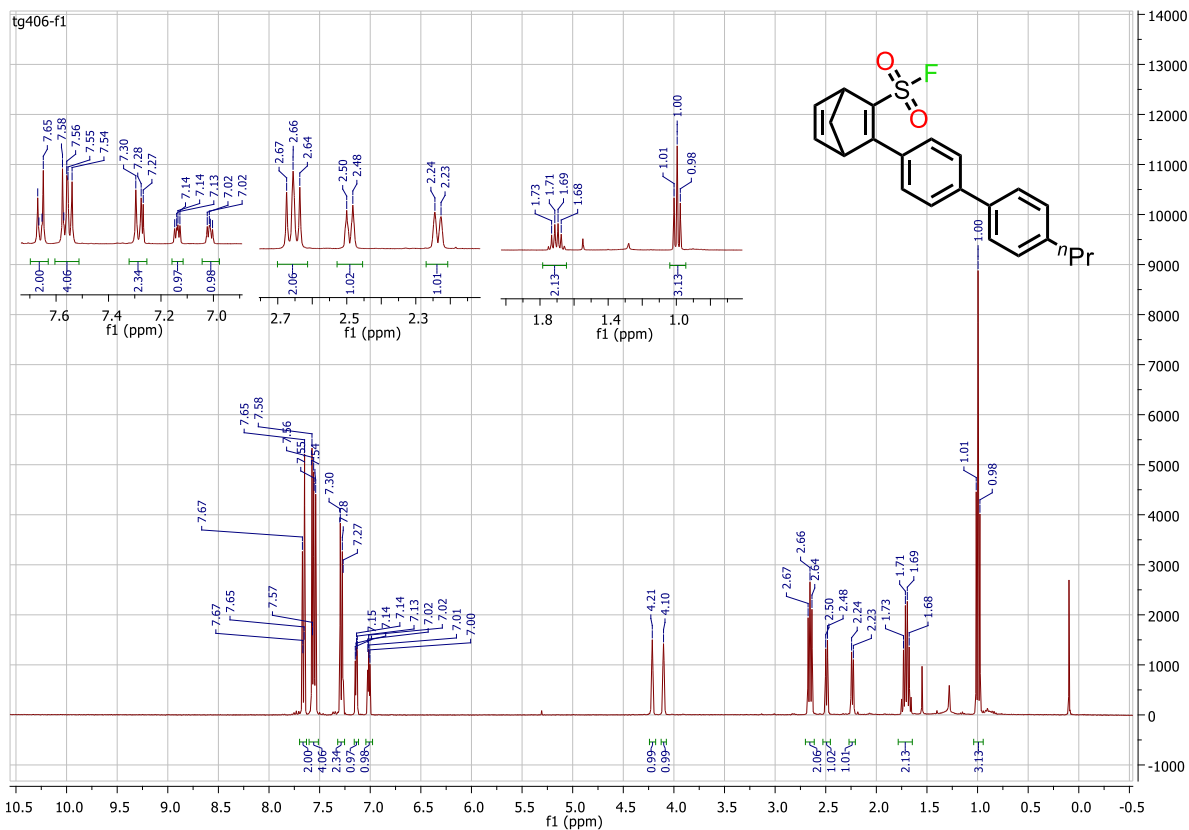


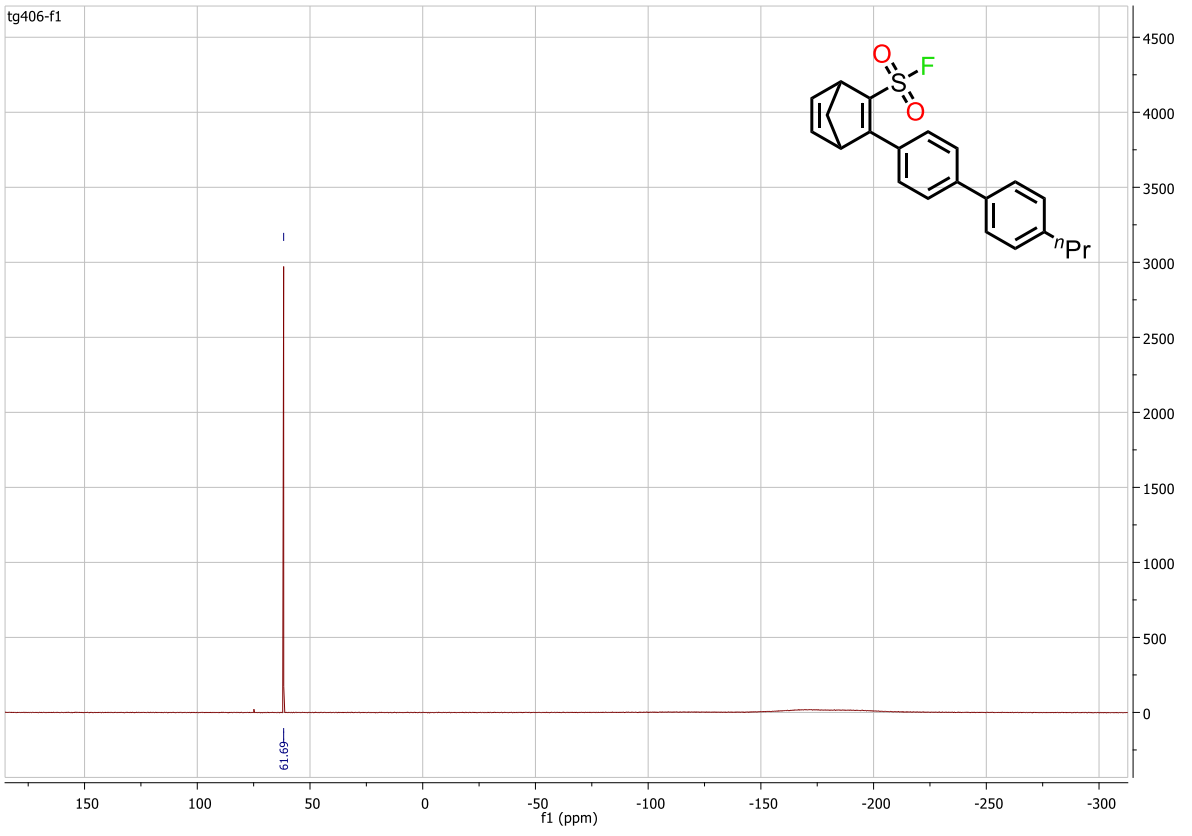


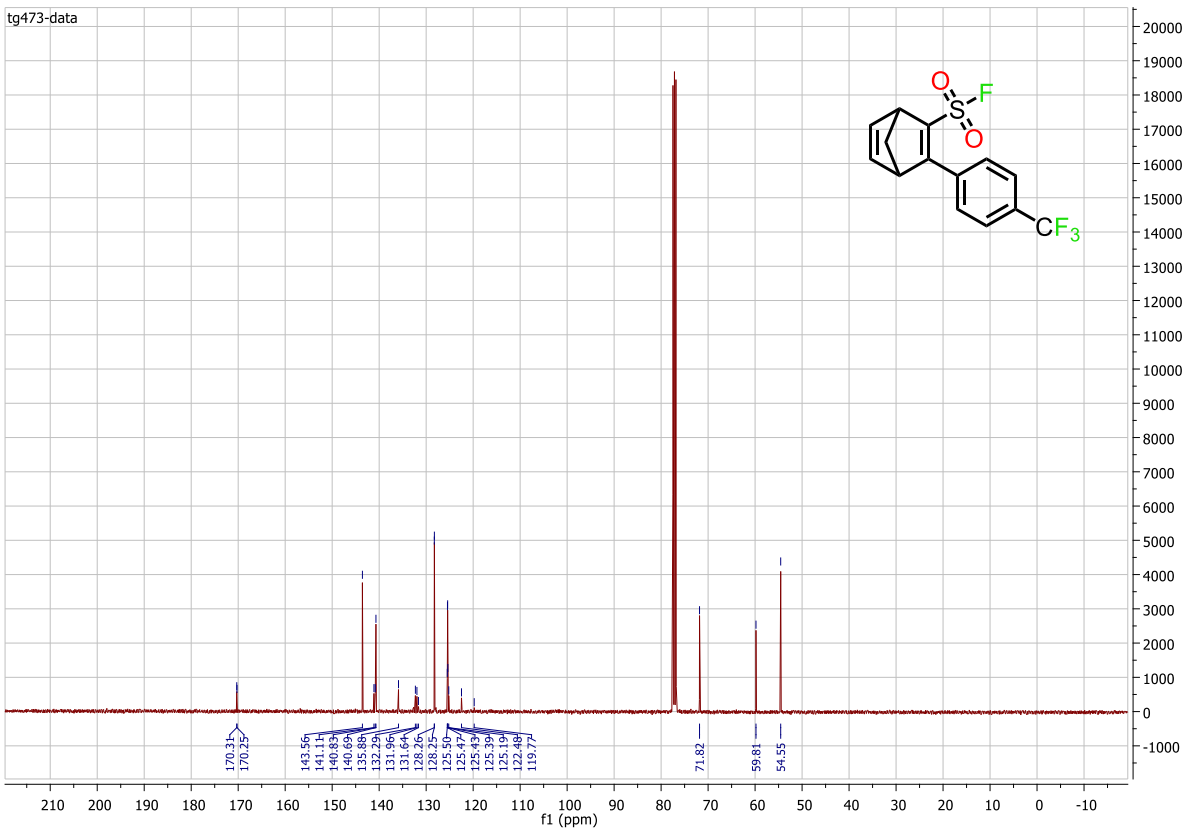
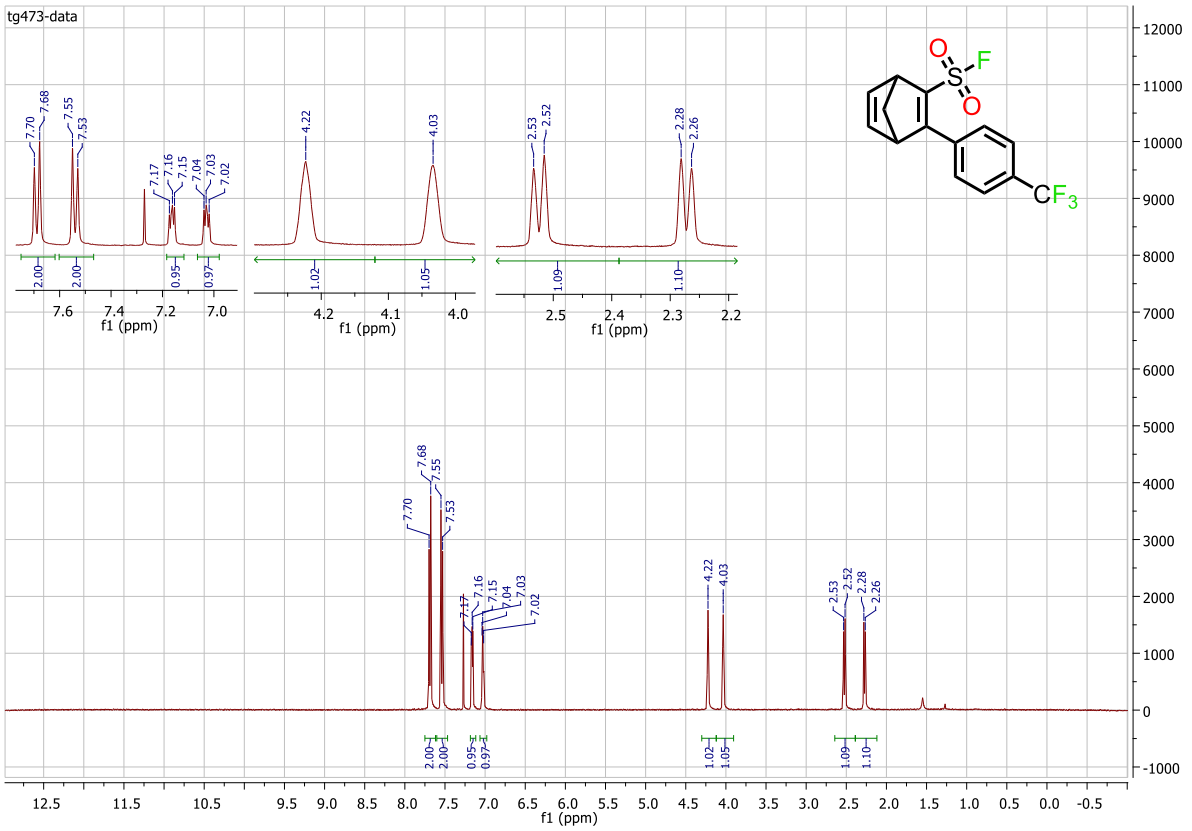


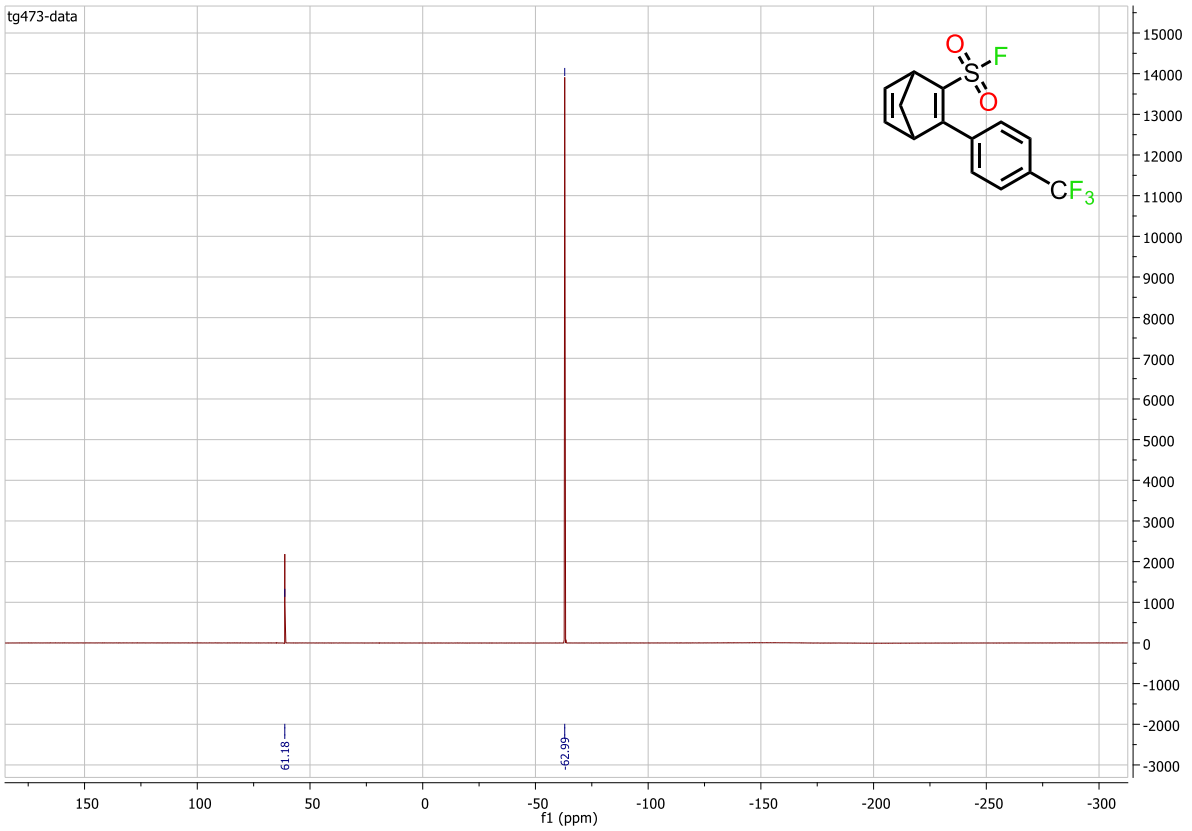


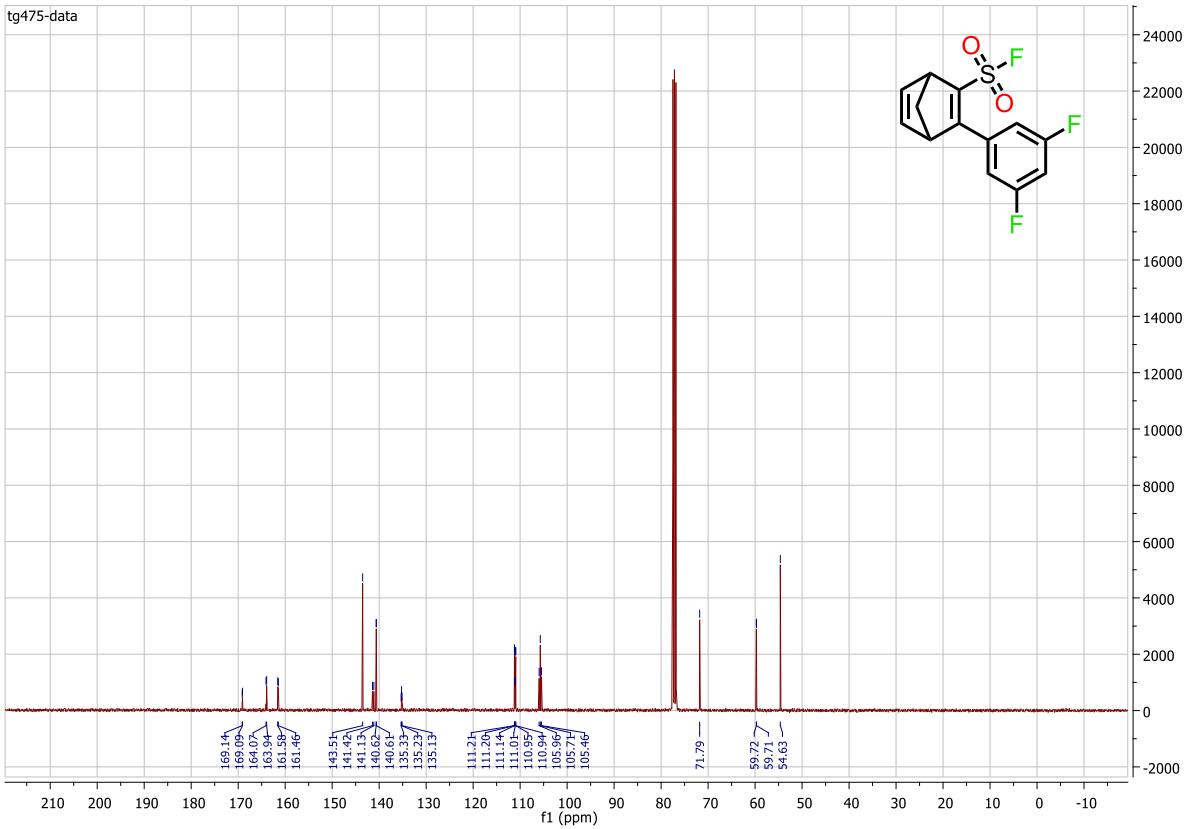
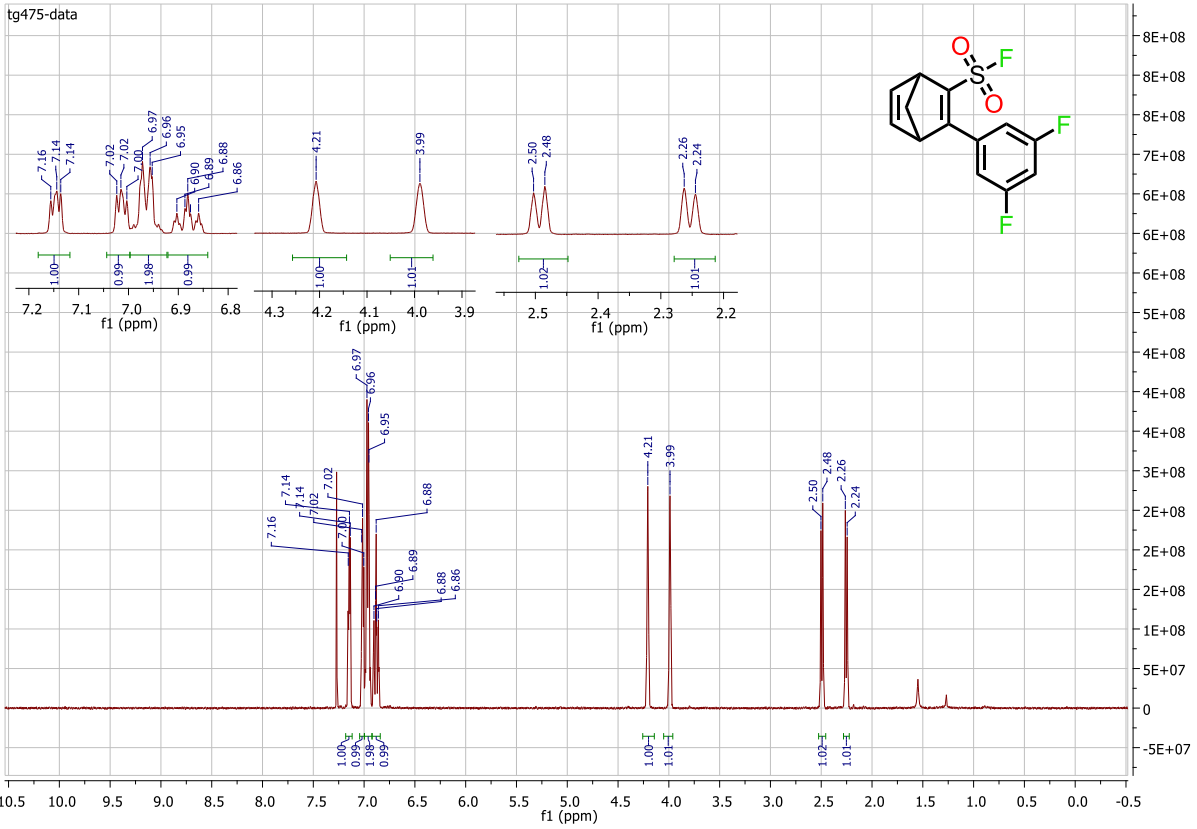












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