BEILSTEIN JOURNAL OF ORGANIC CHEMISTRY

Supporting Information

for

Installation of -SO_2F groups onto primary amides

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Experimental part

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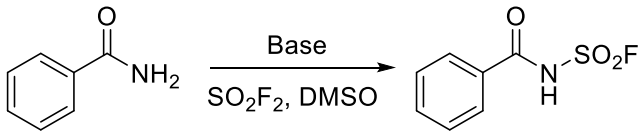
1. General information

All reactions were carried out in dried glassware. All reagents were purchased from commercial sources and used without further purification. Unless otherwise specified, NMR spectra were recorded in $CDCl_3$ or DMSO-d_6 on a 500 MHz (for ^1H), 471 MHz (for ^{19}F), 126 MHz (for ^{13}C) spectrometer. All chemical shifts were reported in ppm relative to TMS (^1H NMR, 0 ppm) as internal standards. Some of the amides was prepared according to literature. Melting points of the products were measured on a micro melting point apparatus (SGW X-4) and are uncorrected. HRMS experiments were performed on a TOF-Q ESI or CI/EI instrument. The coupling constants are reported in Hertz (Hz). The following abbreviations are used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet.

2. Screening the optimized reaction conditions

Table S1 Screening the base[a]

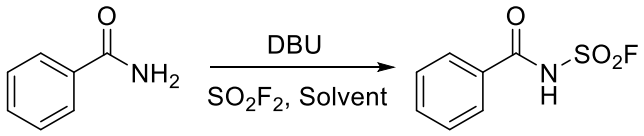


1a **2a**

Entry	Base	Solvent	Temperature (^oC)	Yield (**2a**, %)[b]
1	Cs_2CO_3	DMSO	50	25
2	K_2CO_3	DMSO	50	13
3	KOH	DMSO	50	19
4	NaOH	DMSO	50	15
5	**DBU**	**DMSO**	**50**	**99**
6	Et_3N	DMSO	50	ND [c]
7	DIPEA	DMSO	50	ND

[a] Reaction conditions: Benzamide (**1a**, 1 mmol, 1 equiv), base (5 equiv), and DMSO (1 mL) stirred with a SO_2F_2 balloon at 50 °C for 12 h. [b] Isolated yields. [c] ND = Not Detected.

Table S2 Screening the solvents [a]

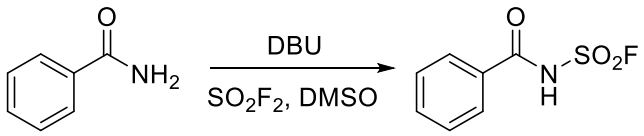


1a **2a**

Entry	Base	Solvent	Temperature (^oC)	Yield (**2a**, %)[b]
1	**DBU**	**DMSO**	**50**	**99**
2	DBU	NMP	50	81
3	DBU	MeCN	50	75
4	DBU	Toluene	50	87
5	DBU	dioxane	50	60
6	DBU	THF	50	79

[a] Reaction conditions: Benzamide (**1a**, 1 mmol, 1 equiv), DBU (5 equiv), and solvent (1 mL) stirred with a SO_2F_2 balloon at 50 °C for 12 h. [b] Isolated yields.

Table S3: Screening the reaction temperature and DBU loading [a]



1a **2a**

Entry	Base	Solvent	Temperature (^{o}C)	Yield (**2a**, %)[b]
1	**DBU (5 equiv)**	**DMSO**	**50**	**99**
2	DBU (5 equiv)	DMSO	40	82
3	DBU (5 equiv)	DMSO	R.T.	51
4	DBU (4 equiv)	DMSO	50	69

[a] Reaction conditions: Benzamide (**1a**, 1 mmol, 1 equiv), DBU (5 equiv), and DMSO (1 mL) stirred with a SO_2F_2 balloon for 12 h. [b] Isolated yields.

3. General procedure for the synthesis of different amides.

The amides of **1a**–**e**, **1j**, **1k**, **1m**, **1n**, **1m**, and **1x** – **z** were purchased from commercial sources and used without further purification. The amides of **1f**, **1g**, **1h**, **1i**, **1l**, and **1o** – **w** were prepared according to the literatures. [1, 2, 3, 4] All the homemade starting materials are identical to those reported regarding the ^{1}H and ^{13}C NMR and melting points (if applicable).

4. Procedure for the synthesis of 2a – z.

A mixture of amides **1** (1 mmol, 1 eq.), DBU (5 mmol, 5 equiv, 761.2 mg, 0.75 mL), and DMSO (1 mL) was allowed to stir at 50 °C under the atmosphere of a SO_2F_2 balloon overnight (monitored by TLC until complete consumption of amide starting materials).

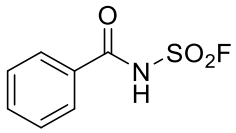
The reaction mixture was quenched by 2 M HCl (10 mL). Then the reaction mixture was extracted with ethyl acetate (3 × 20 mL) and the extracts were washed with water, and concentrated to dryness under reduced pressure. The residue was purified by column chromatography on silica gel using pure ethyl acetate as eluent to give the desired product (**2**).

5. Procedure for the preparation of the crystal 4e

A mixture of amides **1e** (1 mmol, 1 equiv), DBU (5 mmol, 5 equiv, 761.2 mg, 0.75 mL), and DMSO (1 mL) was allowed to stir at 50 °C under the atmosphere of a SO_2F_2 balloon overnight (monitored by TLC until complete consumption of amide starting

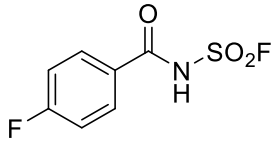
materials). The reaction was quenched with 2 M HCl (10 mL), the resulting mixture was extracted with ethyl acetate (3 × 20 mL) and the extracts were washed with water. Then anhydrous Na_2SO_4 (10 mmol, 10 equiv) was added to the organic phase, which was filtered after being drying for 5 minutes, and the filtrate was concentrated to dryness under reduced pressure. The residue was purified by column chromatography on silica gel using pure ethyl acetate as eluent to give the desired product.

6. Characterization of substrates



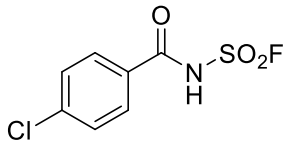
2a

Benzoylsulfamoyl fluoride (2a). Light orange oil (200.0 mg, isolated yield 99%). ^{1}H NMR (500 MHz, DMSO-d_6) δ 7.94 (d, J = 8.4 Hz, 2H), 7.46 (t, J = 7.4 Hz, 1H), 7.38 (t, J = 7.5 Hz, 2H). ^{19}F NMR (471 MHz, DMSO-d_6) δ 50.7 (s, 1F). ^{13}C NMR (126 MHz, DMSO-d_6) δ 170.4, 137.6, 130.9, 128.5, 127.8. HRMS ESI (m/z): $[M+H]^+$ calcd for $C_7H_7FNO_3S$: 204.0125, found: 204.0121.



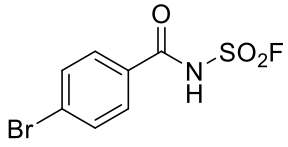
2b

(4-Fluorobenzoyl)sulfamoyl fluoride (2b). Light orange oil (216.8 mg, isolated yield 98%). ^{1}H NMR (500 MHz, DMSO-d_6) δ 7.99 (dd, J = 5.8, J = 8.8 Hz, 2H), 7.17 (t, J = 9.0 Hz, 2H). ^{19}F NMR (471 MHz, DMSO-d_6) δ 50.6 (s, 1F), －110.4-－110.5 (m, 1F). ^{13}C NMR (126 MHz, DMSO-d_6) δ 169.4, 164.0 (d, J = 248.0 Hz), 134.1, 131.1 (d, J = 9.1 Hz), 114.6 (d, J = 21.8 Hz). HRMS ESI (m/z): $[M+H]^+$ calcd for $C_7H_6F_2NO_3S$: 222,0031, found: 222.0028.



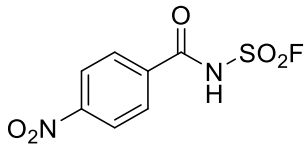
2c

(4-Chlorobenzoyl)sulfamoyl fluoride (2c). Yellow soild (181.1 mg, isolated yield 76%). ^{1}H NMR (500 MHz, DMSO-d_6) δ 7.94 (dd, J = 1.2, J = 8.4 Hz, 2H), 7.43 (d, J = 8.5 Hz, 2H). ^{19}F NMR (471 MHz, DMSO-d_6) δ 50.6 (s, 1F). ^{13}C NMR (126 MHz, DMSO-d_6) δ 167.0, 135.9, 133.1, 129.5, 128.4. Mp 138-139 °C. HRMS ESI (m/z): $[M+H]^+$ calcd for $C_7H_6ClFNO_3S$: 237.9735, found: 237.9731.



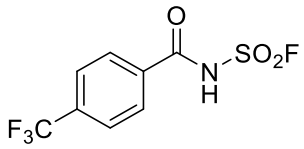
2d

(4-Bromobenzoyl)sulfamoyl fluoride (2d). Orange oil (236.0 mg, isolated yield 84%). ^{1}H NMR (500 MHz, DMSO-d_6) δ 7.87 (d, J = 8.5 Hz, 2H), 7.57 (d, J = 8.6 Hz, 2H). ^{19}F NMR (471 MHz, DMSO-d_6) δ 50.6 (s, 1F). ^{13}C NMR (126 MHz, DMSO-d_6) δ 169.6, 136.8 (d, J = 4.6 Hz), 130.9, 130.7, 124.8. HRMS ESI (m/z): $[M+H]^+$ calcd for $C_7H_6BrFNO_3S$: 281.9230, found: 281.9228.



2e

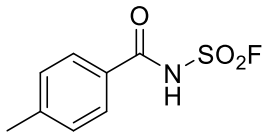
(4-Nitrobenzoyl)sulfamoyl fluoride (2e). Yellow solid (245.5 mg, isolated yield 99%). ^{1}H NMR (500 MHz, DMSO-d_6) δ 8.23 (d, J = 8.9 Hz, 2H), 8.15 (d, J = 9.0 Hz, 2H). ^{19}F NMR (471 MHz, DMSO-d_6) δ 50.4 (s, 1F). ^{13}C NMR (126 MHz, DMSO-d_6) δ 168.3, 148.9, 143.3 (d, J = 3.6 Hz), 129.7, 123.1. Mp 117-119 °C. HRMS ESI (m/z): $[M+H]^+$ calcd for $C_7H_6FN_2O_5S$: 248.9976, found: 248.9972.



2f

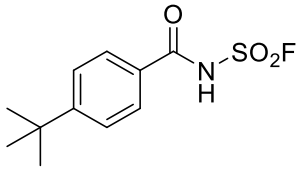
(4-(Trifluoromethyl)benzoyl)sulfamoyl fluoride (2f). Light orange oil (236.9 mg, isolated yield 87%). ^{1}H NMR (500 MHz, DMSO-d_6) δ 8.14 (d, J = 8.1 Hz, 2H), 7.73 (d, J = 8.2 Hz, 2H). ^{19}F NMR (471 MHz, DMSO-d_6) δ 50.4 (s, 1F), ‑61.4 (s, 3F). ^{13}C NMR (126 MHz, DMSO-d_6) δ 169.2, 141.4 (d, J = 3.6 Hz), 131.0 (q, J = 31.8 Hz),

129.3, 124.9 (q, J = 3.7 Hz), 124.3 (q, J = 272.5 Hz), 125.0-124.9 (m). HRMS ESI (m/z): $[M+H]^+$ calcd for $C_8H_6F_4NO_3S$: 271.9999, found: 271.9993.



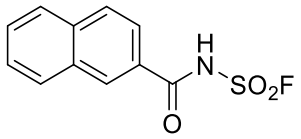
2g

(4-Methylbenzoyl)sulfamoyl fluoride (2g). Light orange oil (136.0 mg, isolated yield 63%). ^{1}H NMR (500 MHz, DMSO-d_6) δ 7.84 (d, J = 8.1 Hz, 2H), 7.17 (d, J = 8.1 Hz, 2H), 2.31 (s, 3H). ^{19}F NMR (471 MHz, DMSO-d_6) δ 50.8 (s, 1F). ^{13}C NMR (126 MHz, DMSO-d_6) δ 170.4 (d, J = 2.7 Hz), 140.5, 132.1, 130.1, 128.5 (d, J = 33.7 Hz), 21.0. HRMS ESI (m/z): $[M+H]^+$ calcd for $C_8H_9FNO_3S$: 218.0282, found: 218.0275.



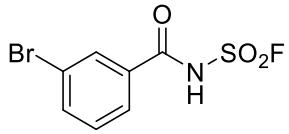
2h

(4-(*tert*-Butyl)benzoyl)sulfamoyl fluoride (2h). Light orange oil (206.2 mg, isolated yield 80%). ^{1}H NMR (500 MHz, DMSO-d_6) δ 7.88 (d, J = 8.2 Hz, 2H), 7.38 (d, J = 8.4 Hz, 2H), 1.28 (s, 9H). ^{19}F NMR (471 MHz, DMSO-d_6) δ 50.9 (s, 1F). ^{13}C NMR (126 MHz, DMSO-d_6) δ 170.4, 153.6, 135.0 (d, J = 4.5 Hz), 128.5, 124.5, 34.5, 31.1. HRMS ESI (m/z): $[M+H]^+$ calcd for $C_{11}H_{15}FNO_3S$: 260.0751, found: 260.0746.



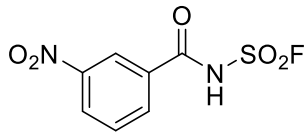
2i

(2-Naphthoyl)sulfamoyl fluoride (2i). Light orange oil (237.8 mg, isolated yield 94%). ^{1}H NMR (500 MHz, DMSO-d_6) δ 8.56 (s, 1H), 8.07-7.89 (m, 4H), 7.57-7.54 (m, 2H). ^{19}F NMR (471 MHz, DMSO-d_6) δ 51.1 (s, 1F). ^{13}C NMR (126 MHz, DMSO-d_6) δ 168.3, 134.5-134.4 (m), 132.3, 131.7, 128.9, 128.0-127.9 (m), 127.7-127.6 (m), 127.4-127.3 (m), 126.7, 126.34-126.32 (m), 124.5. HRMS ESI (m/z): $[M+H]^+$ calcd for $C_{11}H_9FNO_3S$: 254.0282, found: 254.0277.



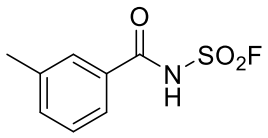
2j

(3-Bromobenzoyl)sulfamoyl fluoride (2j). Orange oil (215.1 mg, isolated yield 76%). ^{1}H NMR (500 MHz, DMSO-d$_6$) δ 8.01 (s, 1H), 7.88 (d, J = 7.6 Hz, 1H), 7.61 (d, J = 1.1 Hz, 1H), 7.33 (t, J = 7.9 Hz, 1H). ^{19}F NMR (471 MHz, DMSO-d$_6$) δ 50.5 (s, 1F). ^{13}C NMR (126 MHz, DMSO-d$_6$) δ 168.9, 139.9 (d, J = 4.6 Hz), 133.6, 131.2, 130.2, 127.5, 121.3. HRMS ESI (m/z): [M+H]$^+$ calcd for $C_7H_6BrFNO_3S$: 281.9230, found: 281.9228.



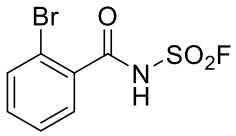
2k

(3-Nitrobenzoyl)sulfamoyl fluoride (2k). Yellow soild (160.6 mg, isolated yield 65%). ^{1}H NMR (500 MHz, DMSO-d$_6$) δ 8.67 (s, 1H), 8.33 (t, J = 7.8 Hz, 2H), 7.70 (t, J = 8.0 Hz, 1H). ^{19}F NMR (471 MHz, DMSO-d$_6$) δ 50.5 (s, 1F). ^{13}C NMR (126 MHz, DMSO-d$_6$) δ 167.9, 147.6, 139.2 (d, J = 4.5 Hz), 134.6, 129.7, 125.4, 122.9. Mp 94-96 °C. HRMS ESI (m/z): [M+H]$^+$ calcd for $C_7H_6FN_2O_5S$: 248.9976, found: 248.9972.



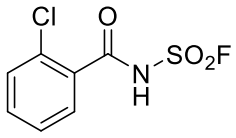
2l

(3-Methylbenzoyl)sulfamoyl fluoride (2l). Light orange oil (210.5 mg, isolated yield 97%). ^{1}H NMR (500 MHz, DMSO-d$_6$) δ 7.79 (s, 1H), 7.76-7.75 (m, 1H), 7.27-7.24 (m, 2H), 2.31 (s, 3H). ^{19}F NMR (471 MHz, DMSO-d$_6$) δ 50.8 (s, 1F). ^{13}C NMR (126 MHz, DMSO-d$_6$) δ 170.9, 137.7 (d, J = 3.6 Hz), 137.0, 131.6, 129.3, 127.8, 125.9, 20.8. HRMS ESI (m/z): [M+H]$^+$ calcd for $C_8H_9FNO_3S$: 218.0282, found: 218.0275.



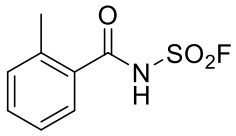
2m

(2-Bromobenzoyl)sulfamoyl fluoride (2m). Light orange oil (279.2 mg, isolated yield 99%). ^{1}H NMR (500 MHz, DMSO-d_6) δ 7.54 (d, J = 7.9 Hz, 1H), 7.51 (dd, J = 7.6, J = 1.5 Hz, 1H), 7.34 (t, J=7.4, 1H), 7.24 (td, J = 7.7, J = 1.5 Hz, 1H). ^{19}F NMR (471 MHz, DMSO-d_6) δ 50.5 (s, 1F). ^{13}C NMR (126 MHz, DMSO-d_6) δ 172.0, 141.1 (d, J = 4.5 Hz), 133.1, 130.3, 129.6, 127.3, 119.1. HRMS ESI (m/z): $[M+H]^+$ calcd for $C_7H_6BrFNO_3S$: 281.9230, found: 281.9228.



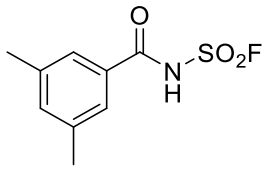
2n

(2-Chlorobenzoyl)sulfamoyl fluoride (2n). Light orange oil (169.1 mg, isolated yield 71%). ^{1}H NMR (500 MHz, DMSO-d_6) δ 7.54 (dd, J = 7.3, J = 1.6 Hz, 1H), 7.39-7.37 (m, 1H), 7.35-7.28 (m, 2H). ^{19}F NMR (471 MHz, DMSO-d_6) δ 50.5 (s, 1F). ^{13}C NMR (126 MHz, DMSO-d_6) δ 171.1, 139.1 (d, J = 4.5 Hz), 130.3, 130.1, 129.9, 129.6, 126.7. HRMS ESI (m/z): $[M+H]^+$ calcd for $C_7H_6ClFNO_3S$: 237.9735, found: 237.9731.



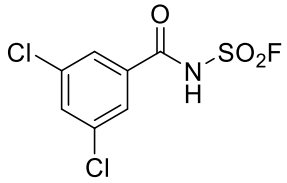
2o

(2-Methylbenzoyl)sulfamoyl fluoride (2o). Light orange oil (130.2 mg, isolated yield 60%). ^{1}H NMR (500 MHz, DMSO-d_6) δ 7.63 (d, J = 7.4 Hz, 1H), 7.24-7.22 (m, 1H), 7.16-7.13 (m, 2H), 2.44 (s, 3H). ^{19}F NMR (471 MHz, DMSO-d_6) δ 50.5 (s, 1F). ^{13}C NMR (126 MHz, DMSO-d_6) δ 173.5, 138.6 (d, J = 4.5 Hz), 136.6, 130.8, 129.1, 129.0, 125.2, 20.8. HRMS ESI (m/z): $[M+H]^+$ calcd for $C_8H_9FNO_3S$: 218.0282, found: 218.0275.



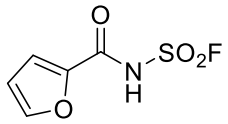
2p

(3,5-Dimethylbenzoyl)sulfamoyl fluoride (2p). Light orange oil (182.6 mg, isolated yield 79%). ^{1}H NMR (500 MHz, DMSO-d$_6$) δ 7.55 (s, 2H), 7.07 (s, 1H), 2.28 (s, 6H). ^{19}F NMR (471 MHz, DMSO-d$_6$) δ 50.8 (s, 1F). ^{13}C NMR (126 MHz, DMSO-d$_6$) δ 170.6, 136.6, 132.1, 126.4, 20.8. HRMS ESI (m/z): $[M+H]^+$ calcd for $C_9H_{11}FNO_3S$: 232.0438, found: 232.0432.



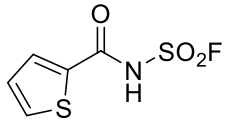
2q

(3,5-Dichlorobenzoyl)sulfamoyl fluoride (2q). Light orange oil (205.6 mg, isolated yield 76%). ^{1}H NMR (500 MHz, DMSO-d$_6$) δ 7.85 (d, J = 1.7 Hz, 2H), 7.61 (s, 1H). ^{19}F NMR (471 MHz, DMSO-d$_6$) δ 50.4 (s, 1F). ^{13}C NMR (126 MHz, DMSO-d$_6$) δ 167.6, 141.2 (d, J = 4.6 Hz), 134.0, 130.2, 127.1. HRMS ESI (m/z): $[M+H]^+$ calcd for $C_7H_5Cl_2FNO_3S$: 271.9346, found: 271.9341.



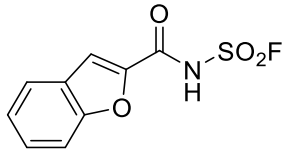
2r

(Furan-2-ylcarbonyl)sulfamoyl fluoride (2r). Light yellow soild (131.7 mg, isolated yield 68%). ^{1}H NMR (500 MHz, DMSO-d$_6$) δ 7.70 (s, 1H), 6.89 (d, J = 3.4 Hz, 1H), 6.50 (q, J = 1.7 Hz, 1H). ^{19}F NMR (471 MHz, DMSO-d$_6$) δ 51.8 (s, 1F). ^{13}C NMR (126 MHz, DMSO-d$_6$) δ 162.3, 145.0, 144.7, 114.0, 111.4. Mp 68-69 °C. HRMS ESI (m/z): $[M+H]^+$ calcd for $C_5H_5FNO_4S$: 193.9918, found: 193.9915.



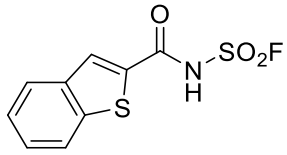
2s

(Thiophene-2-ylcarbonyl)sulfamoyl fluoride (2s). Yellow soild (188.1 mg, isolated yield 90%). ^{1}H NMR (500 MHz, DMSO-d$_6$) δ 7.60 (dd, J = 4.9, J = 1.1 Hz, 1H), 7.52 (d, J = 3.5 Hz, 1H), 7.05 (dd, J = 3.7, J = 4.9 Hz, 1H). ^{19}F NMR (471 MHz, DMSO-d$_6$) δ 56.2 (d, J = 5.7 Hz, 1F). ^{13}C NMR (126 MHz, DMSO-d$_6$) δ 163.0, 140.3, 131.0, 130.0, 128.8. HRMS ESI (m/z): [M+H]$^+$ calcd for $C_5H_5FNO_3S_2$: 209.9689, found: 209.9687.



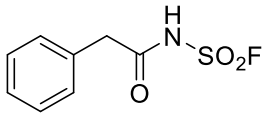
2t

(Benzofuran-2-ylcarbonyl)sulfamoyl fluoride (2t). Light orange oil (136.0 mg, isolated yield 56%). ^{1}H NMR (500 MHz, DMSO-d$_6$) δ 7.71 (d, J = 7.8 Hz, 1H), 7.61 (d, J = 8.4 Hz, 1H), 7.41-7.36 (m, 2H), 7.27 (t, J=7.5, 1H). ^{19}F NMR (471 MHz, DMSO-d$_6$) δ 51.6 (s, 1F). ^{13}C NMR (126 MHz, DMSO-d$_6$) δ 163.2, 154.7, 152.9, 127.6, 126.3, 123.4, 122.6, 111.8, 109.9. HRMS ESI (m/z): [M+H]$^+$ calcd for $C_9H_7FNO_4S$: 244.0074, found: 244.0070.



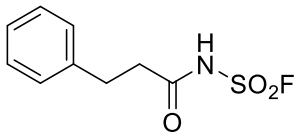
2u

(Benzo[*b*]thiophene-2-ylcarbonyl)sulfamoyl fluoride (2u). Light orange oil (220.4 mg, isolated yield 85%). ^{1}H NMR (500 MHz, DMSO-d$_6$) δ 7.93-7.89 (m, 3H), 7.41-7.35 (m, 2H). ^{19}F NMR (471 MHz, DMSO-d$_6$) δ 51.2 (s, 1F). ^{13}C NMR (126 MHz, DMSO-d$_6$) δ 166.3, 143.7 (d, J = 5.5 Hz), 141.1, 139.4, 126.7, 126.0, 125.2, 124.6, 122.8. HRMS ESI (m/z): [M+H]$^+$ calcd for $C_9H_7FNO_3S_2$: 259.9846, found: 259.9842.



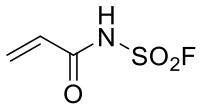
2v

(2-Phenylacetyl)sulfamoyl fluoride (2v). Light orange oil (197.9 mg, isolated yield 91%). ^{1}H NMR (500 MHz, DMSO-d$_6$) δ 7.28-7.25 (m, 2H), 7.22 (d, J = 6.9 Hz, 2H), 7.18 (t, J=7.1 Hz, 1H), 3.36 (s, 2H). ^{19}F NMR (471 MHz, DMSO-d$_6$) δ 50.2 (s, 1F). ^{13}C NMR (126 MHz, DMSO-d$_6$) δ 176.3, 137.4, 129.4, 128.0, 125.9, 46.1 (d, J = 4.5 Hz). HRMS ESI (m/z): $[M+H]^+$ calcd for $C_8H_9FNO_3S$: 218.0282, found: 218.0275.



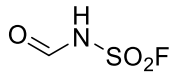
2w

(3-Phenylpropanoyl)sulfamoyl fluoride (2w). Light orange oil (138.6 mg, isolated yield 60%). ^{1}H NMR (500 MHz, DMSO-d$_6$) δ 7.26-7.23 (m, 2H), 7.19 (d, J = 6.9 Hz, 2H), 7.16-7.13 (m, 1H), 2.77 (t, J = 7.9 Hz, 2H), 2.35 (t, J = 7.9 Hz, 2H). ^{19}F NMR (471 MHz, DMSO-d$_6$) δ 50.8 (s, 1F). ^{13}C NMR (126 MHz, DMSO-d$_6$) δ 176.8, 141.8, 128.4 (d, J = 5.5 Hz), 126.9, 126.0, 31.2, 30.7. HRMS ESI (m/z): $[M+H]^+$ calcd for $C_9H_{11}FNO_3S$: 232.0438, found: 232.0432.



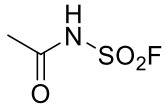
2x

Acryloylsulfamoyl fluoride (2x). Light orange oil (129.6 mg, isolated yield 85%). ^{1}H NMR (500 MHz, DMSO-d$_6$) δ 6.01-6.00 (m, 2H), 5.49 (dd, J = 4.6, J = 7.8 Hz, 1H). ^{19}F NMR (471 MHz, DMSO-d$_6$) δ 50.7 (s, 1F). ^{13}C NMR (126 MHz, DMSO-d$_6$) δ 170.3, 136.8 (d, J = 4.5 Hz), 124.7. HRMS ESI (m/z): $[M+H]^+$ calcd for $C_3H_5FNO_3S$: 153.9969, found:153.9961.



2y

Formylsulfamoyl fluoride (2y). Orange oil (82.5 mg, isolated yield 65%). ^{1}H NMR (500 MHz, DMSO-d_6) δ 8.92 (s, 1H). ^{19}F NMR (471 MHz, DMSO-d_6) δ 59.0 (s, 1F). ^{13}C NMR (126 MHz, DMSO-d_6) δ 170.4. HRMS ESI (m/z): $[M+H]^+$ calcd for CH_3FNO_3S: 127.9812, found: 127.9808.



2z

Acetylsulfamoyl fluoride (2z). Light orange oil (67.7 mg, isolated yield 48%). ^{1}H NMR (500 MHz, DMSO-d_6) δ 1.99 (s, 3H). ^{19}F NMR (471 MHz, DMSO-d_6) δ 51.5 (s, 1F). ^{13}C NMR (126 MHz, DMSO-d_6) δ 171.0, 24.4. HRMS ESI (m/z): $[M+H]^+$ calcd for $C_2H_5FNO_3S$: 141.9969, found: 141.9965.

7. References

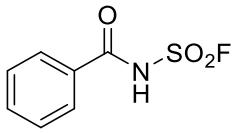
[1] Wu, Y.; Sun, P.; Zhang, K.; Yang, T.; Ya, H.; Lin, A. *J. Org. Chem.,* **2016**, *81*, 2166-2173.

[2] Lei, P.; Xu, Y.; Du, J.; Yang, X.-L.; Yuan, H.-Z.; Xu, G.-F.; Ling, Y. *Bioorg. Med. Chem. Lett*., **2016**, *26*, 2544-2546.

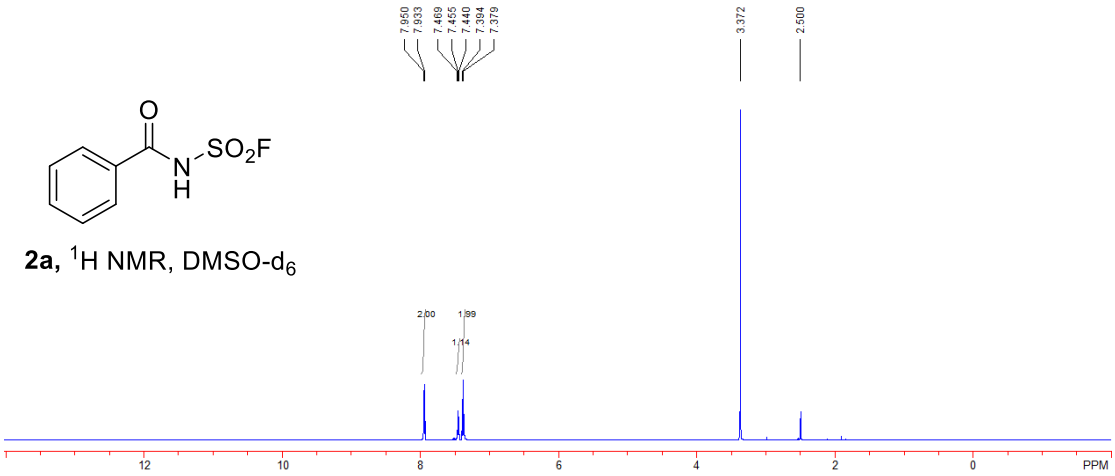
[3] Gaspari, P.; Banerjee, T.; Malachowski, W. P.; Muller, A. J.; Prendergast, G. C.; DuHadaway, J.; Bennett, S.; Donovan, A. M. *J. Med. Chem*., **2006**, *49*, 684-692.

[4] Guo, W.; Huang, J.; Wu, H.; Liu, T.; Luo, Z.; Jian, J.; Zeng, Z. *Org. Chem. Front.*, **2018**, *5*, 2950-2954.

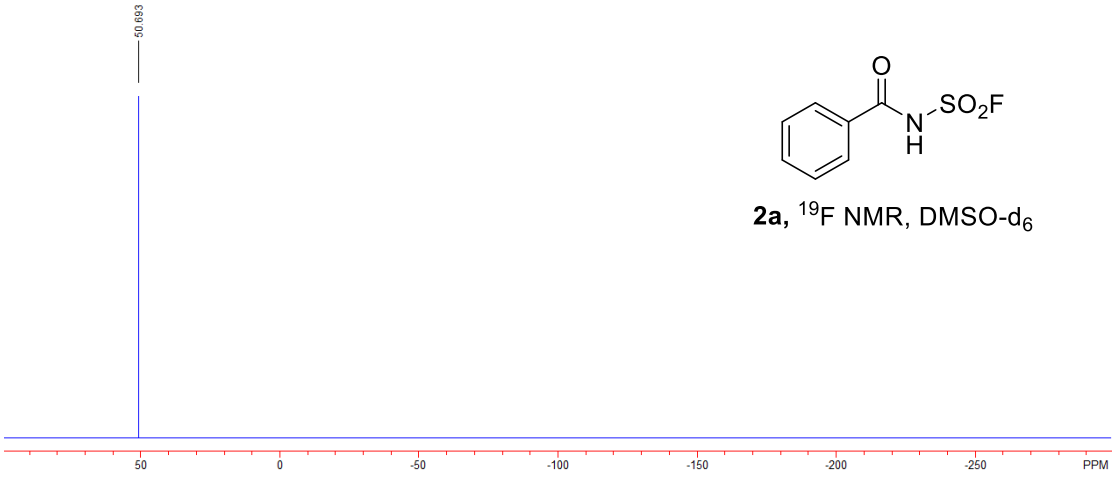
8. ^{1}H, ^{19}F, ^{13}C NMR spectra



2a



2a, ^{1}H NMR, DMSO-d_6



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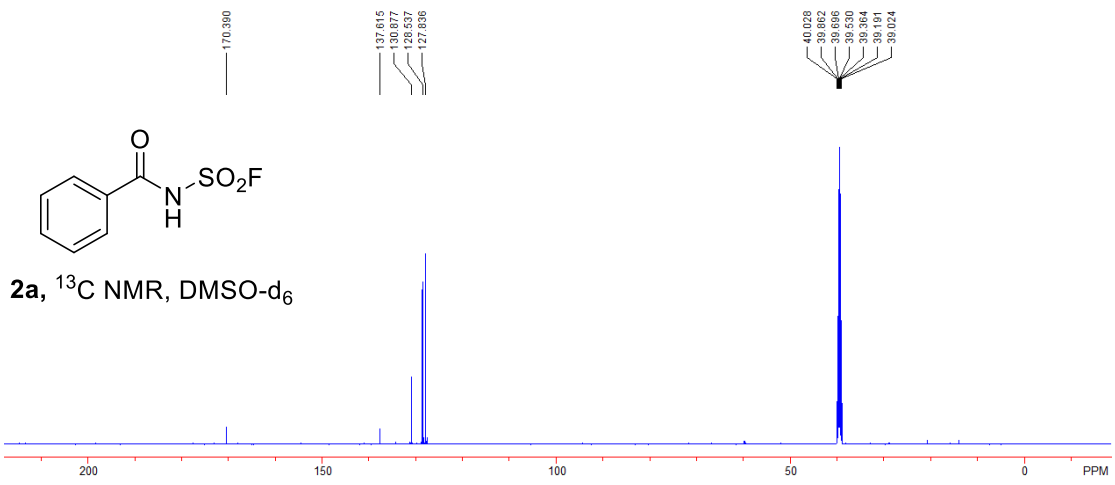

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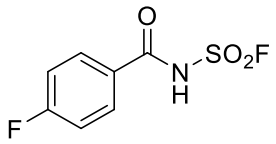
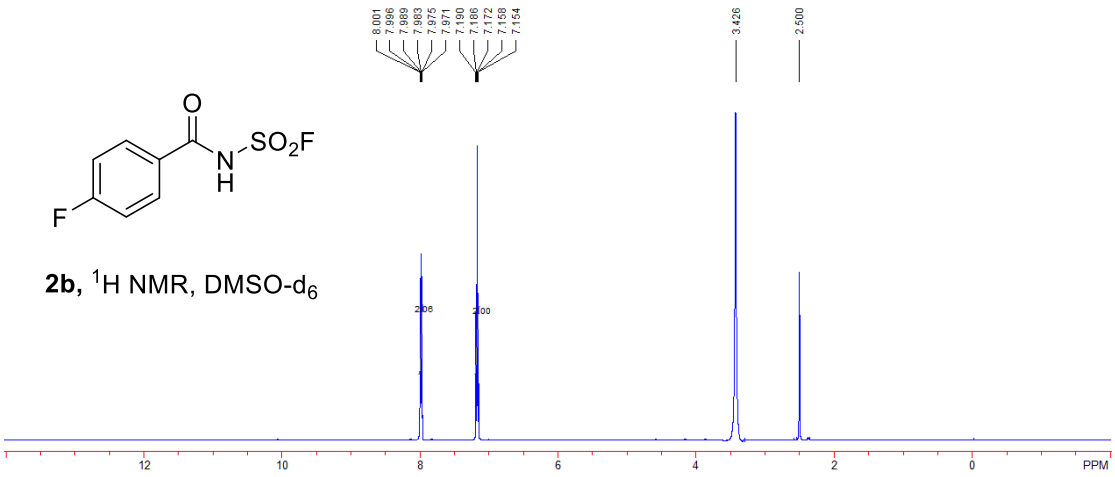
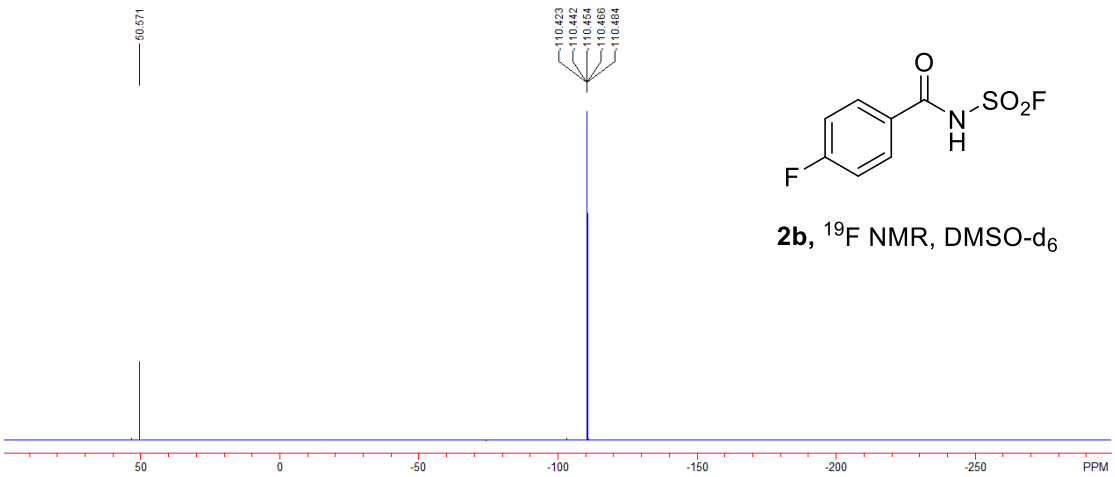


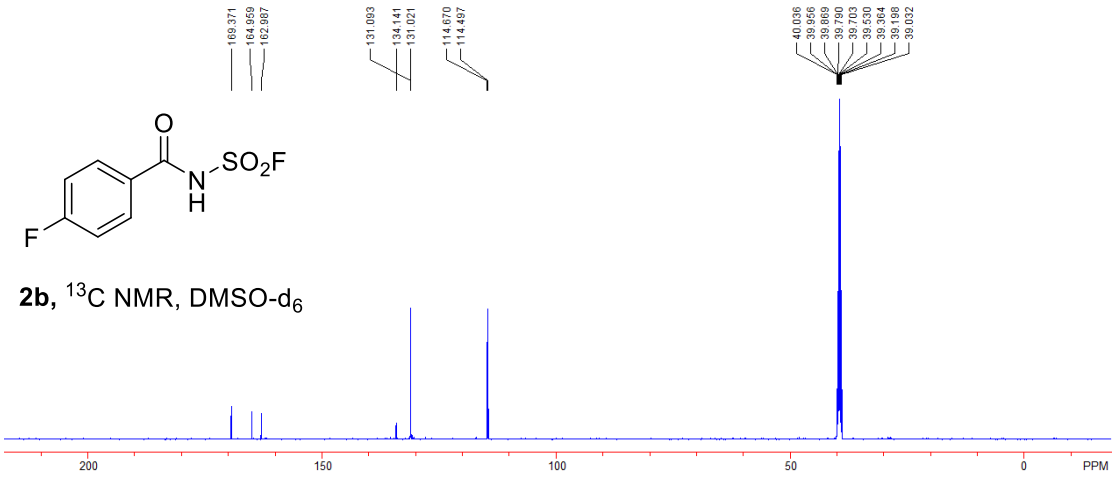
2b



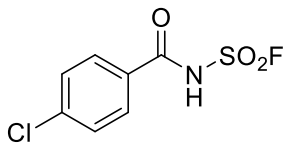
2b, ^{1}H NMR, DMSO-d$_6$



2b, ^{19}F NMR, DMSO-d$_6$



2b, ^{13}C NMR, DMSO-d$_6$



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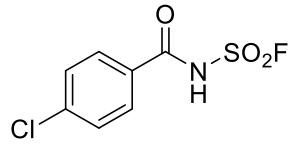

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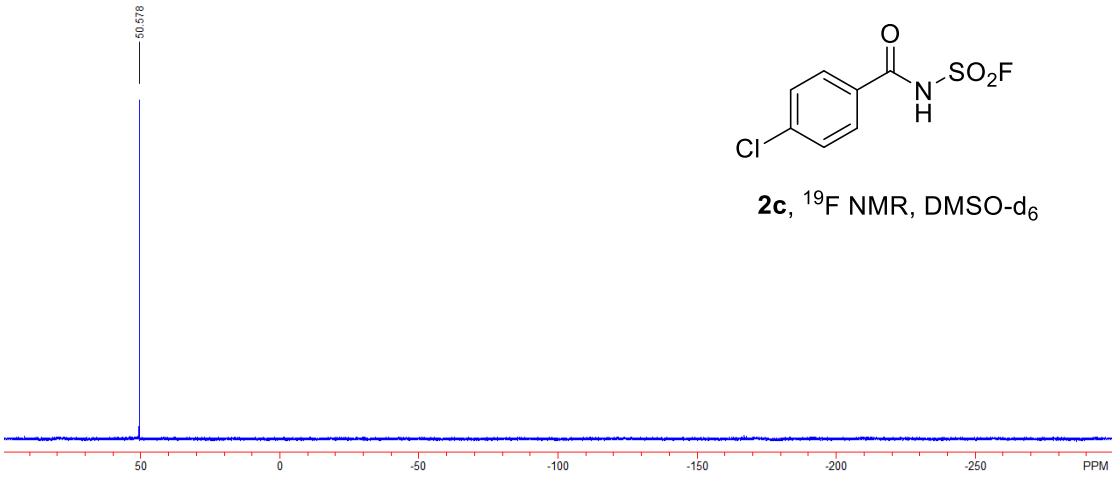

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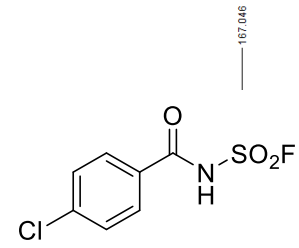

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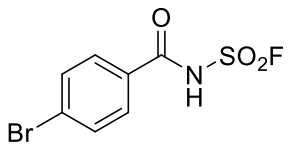

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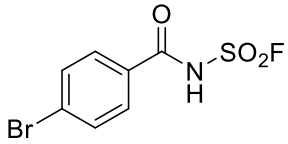

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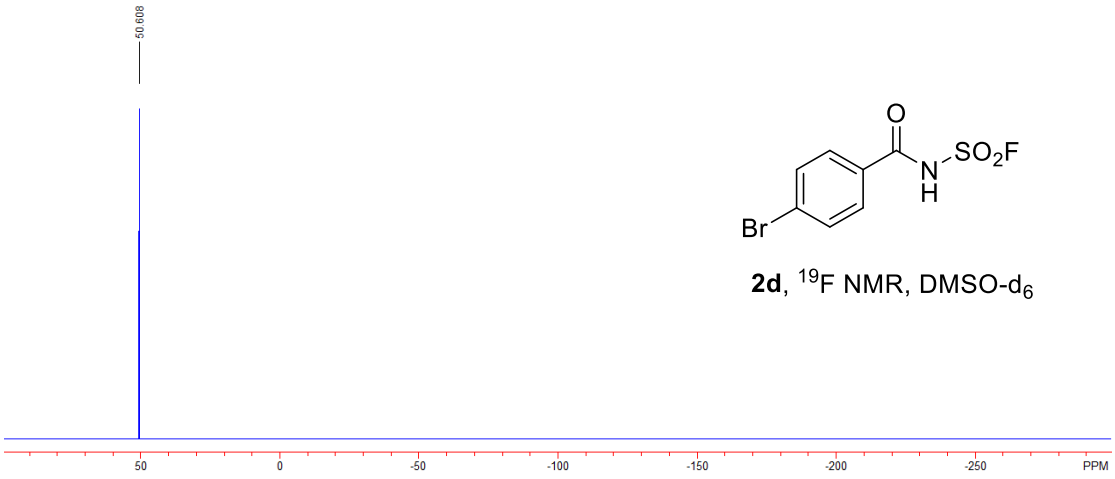

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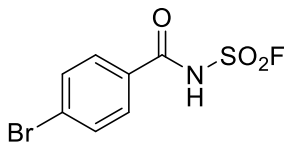

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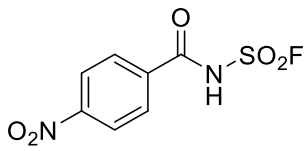

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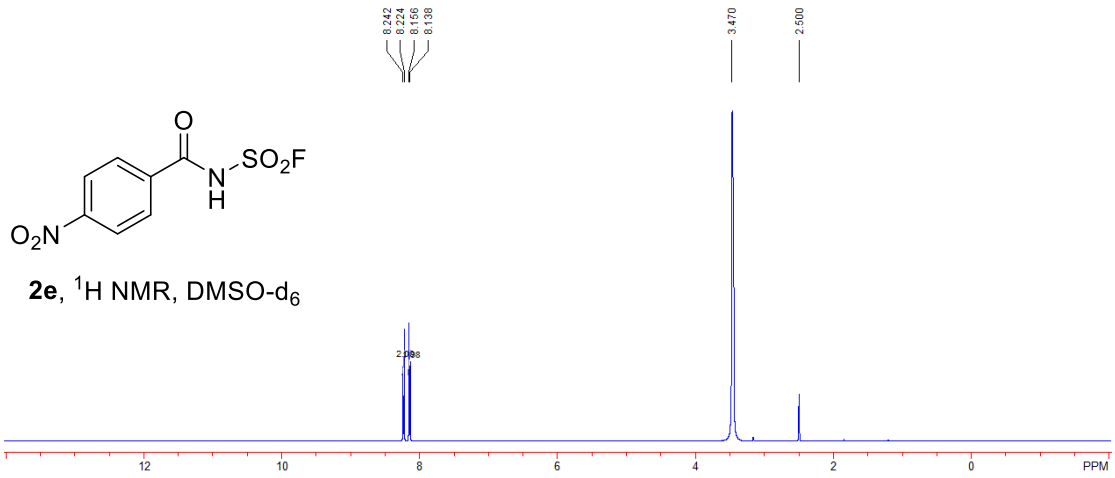
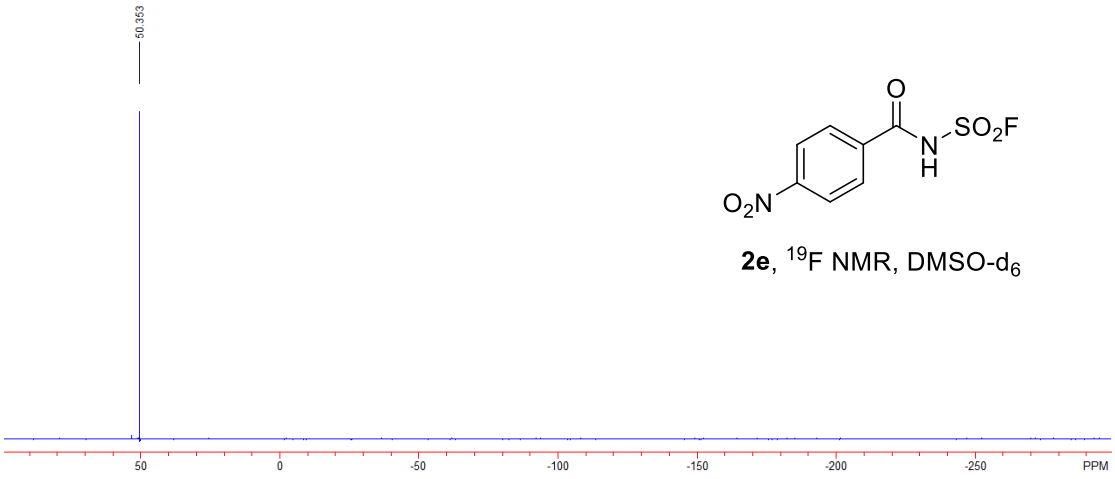
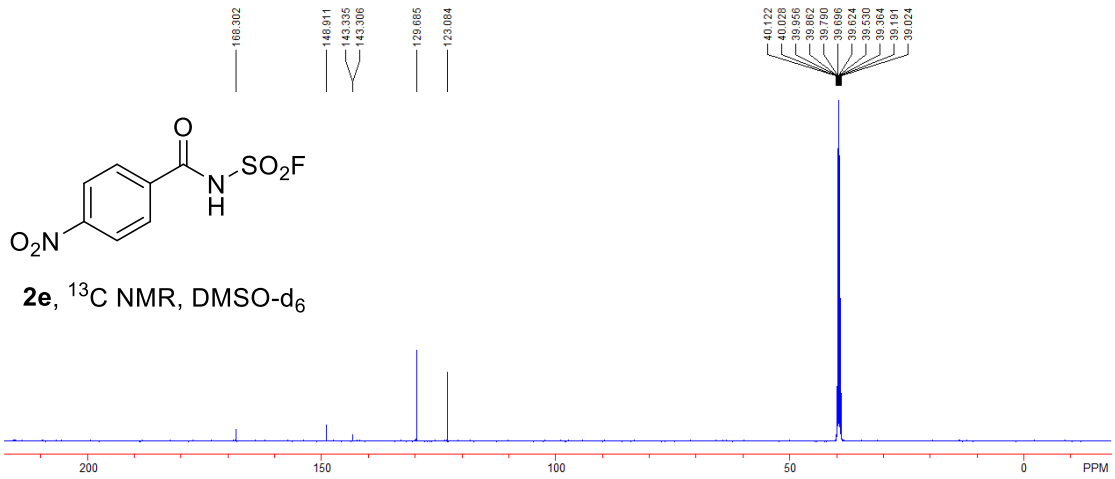


2e, ^{1}H NMR, DMSO-d$_6$



2e, ^{19}F NMR, DMSO-d$_6$



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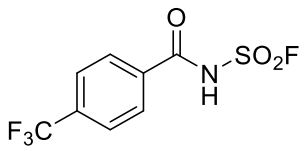

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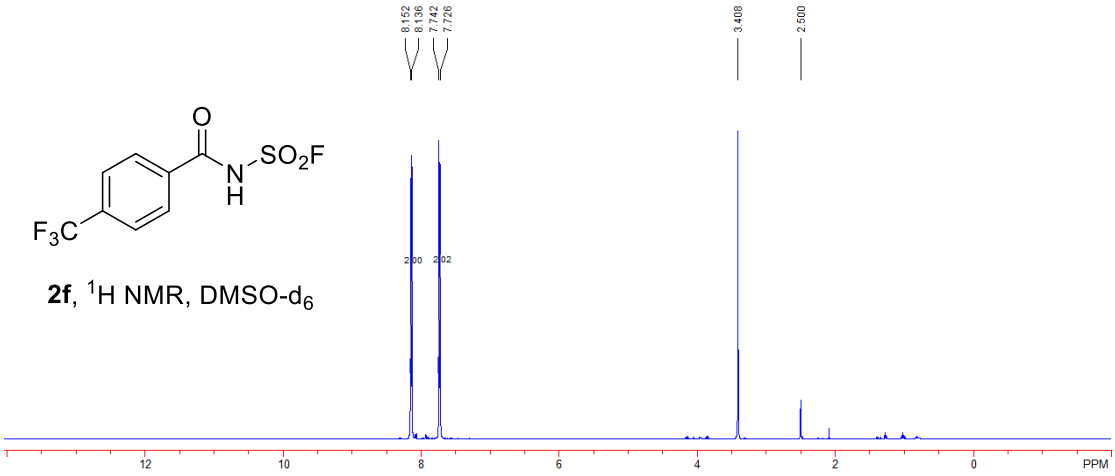

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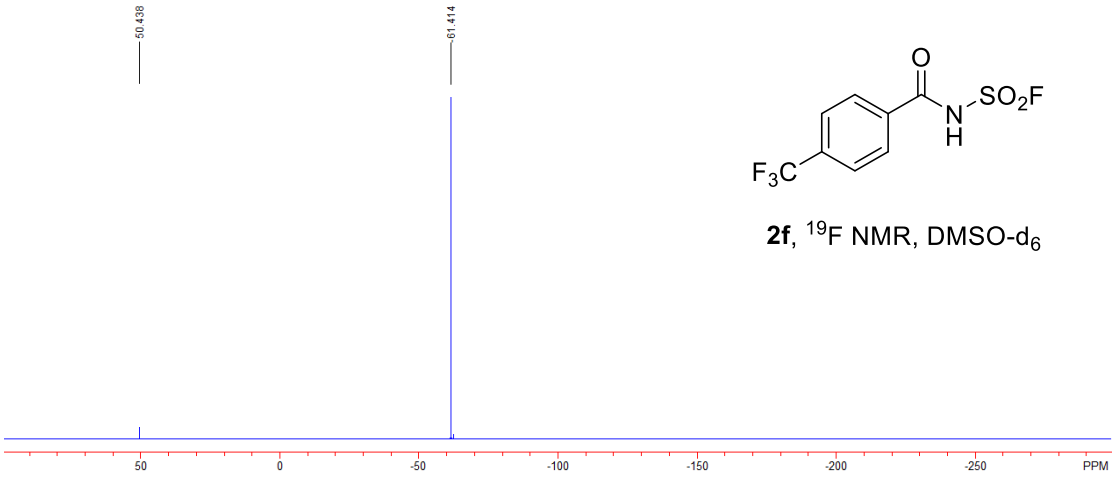

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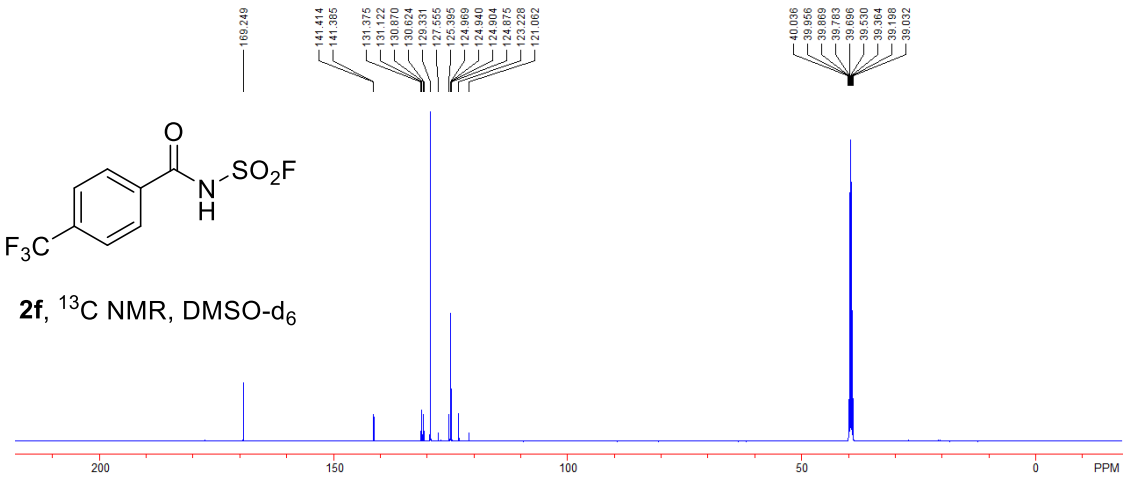

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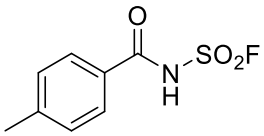

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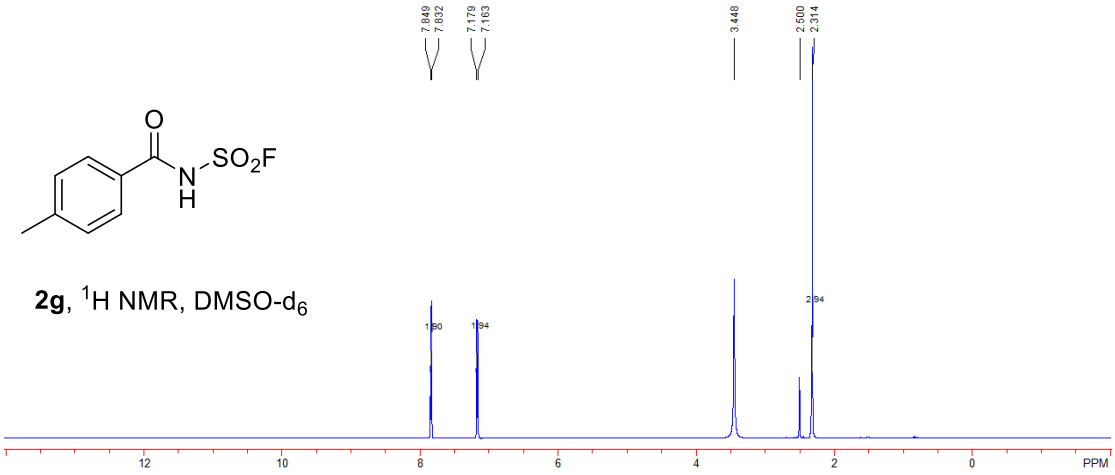

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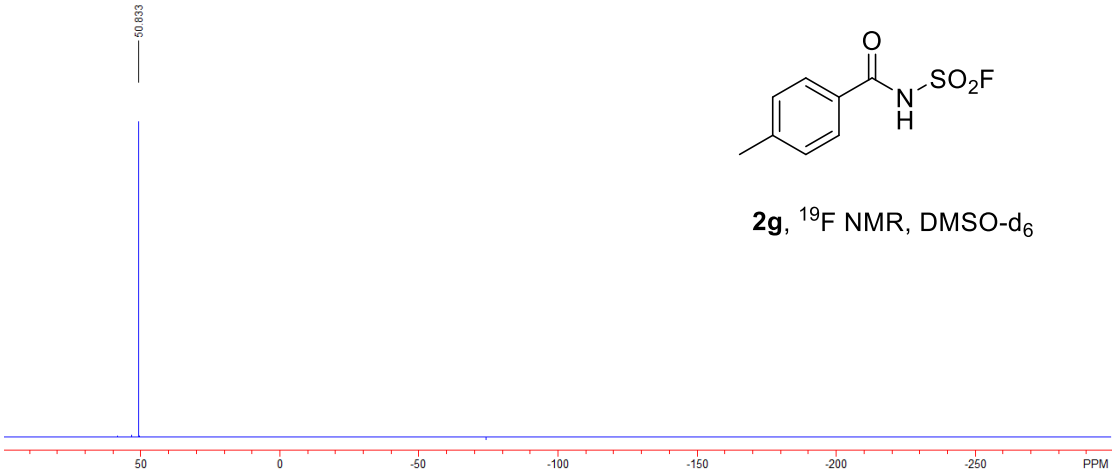

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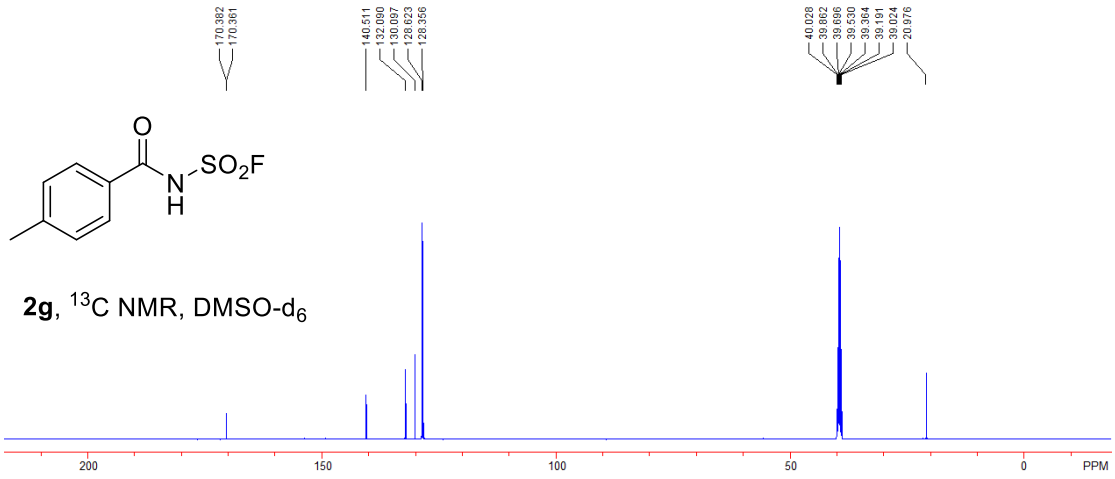

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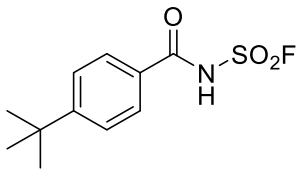
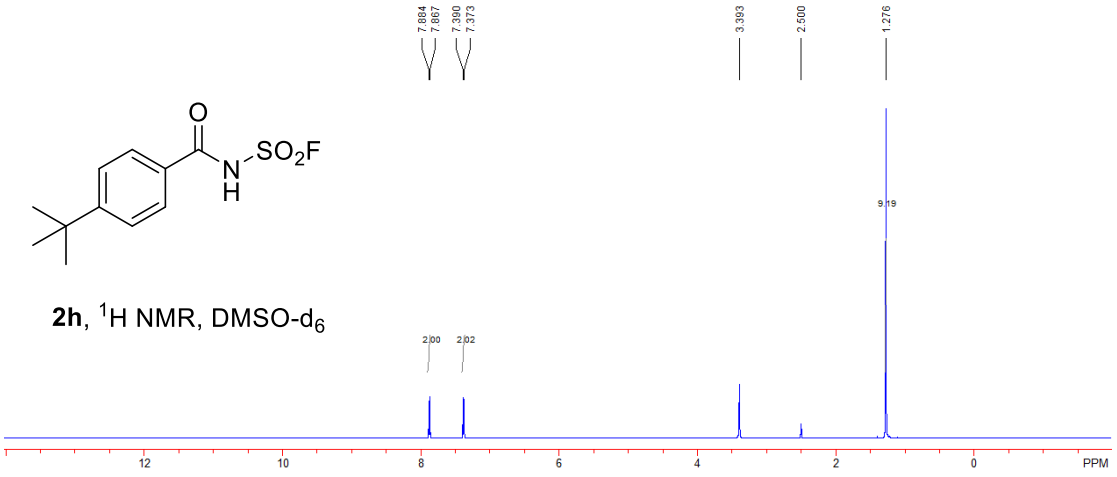
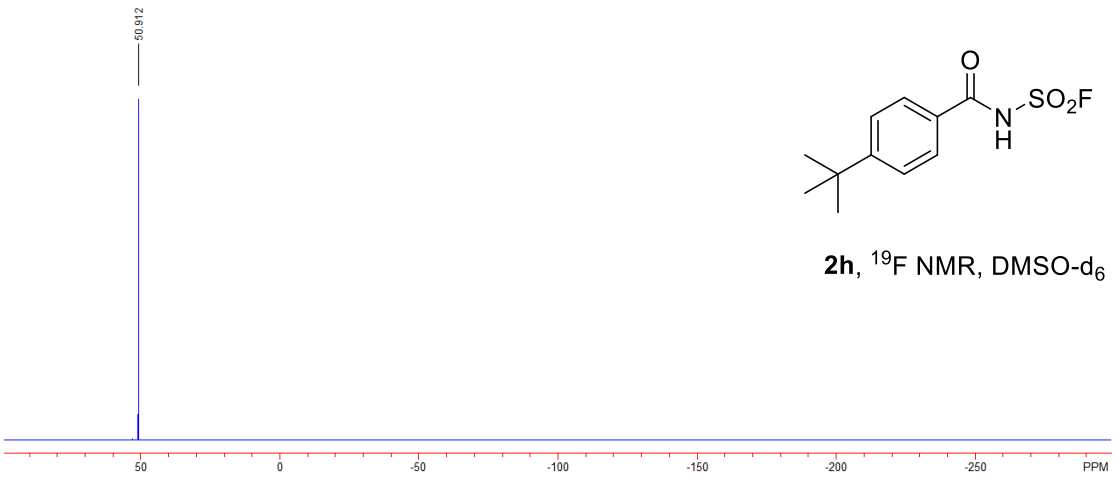


2h



2h, ^{1}H NMR, DMSO-d$_6$



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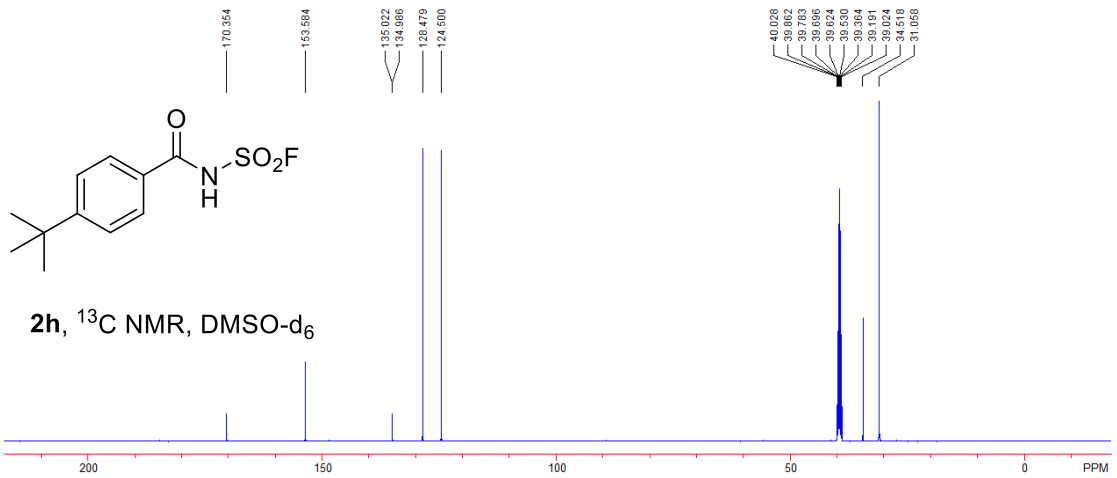

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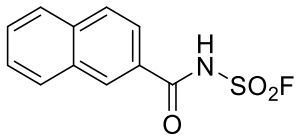

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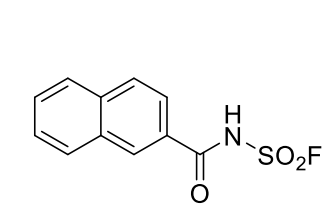
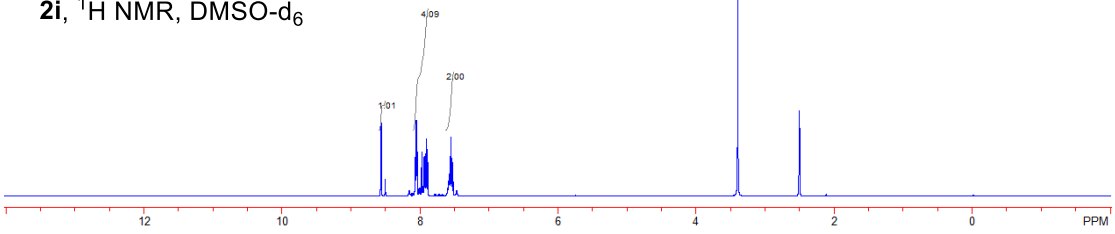
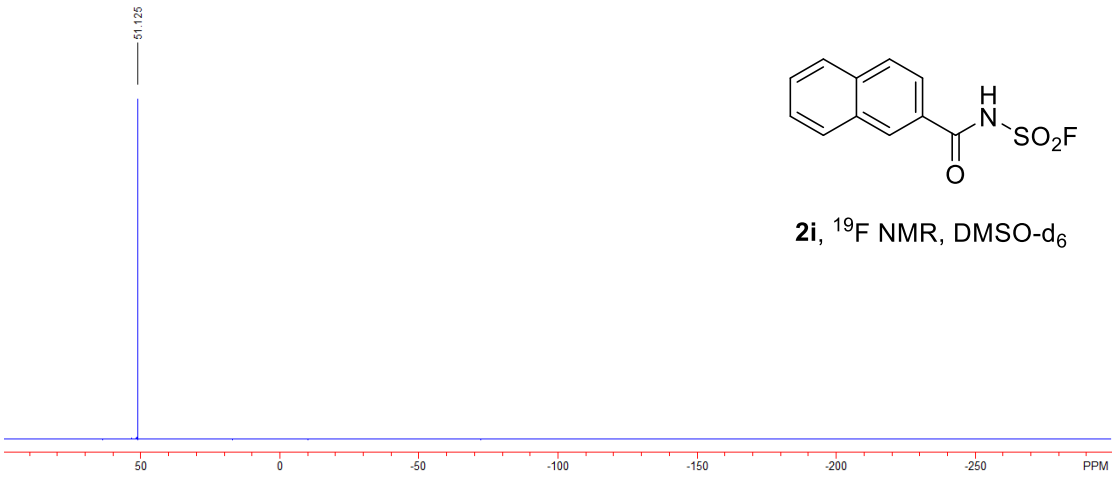


2i, ^{1}H NMR, DMSO-d$_6$





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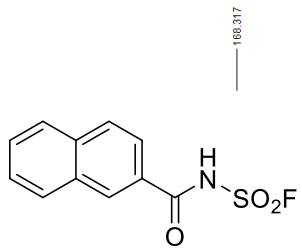
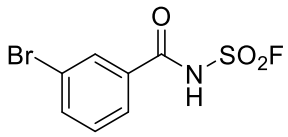
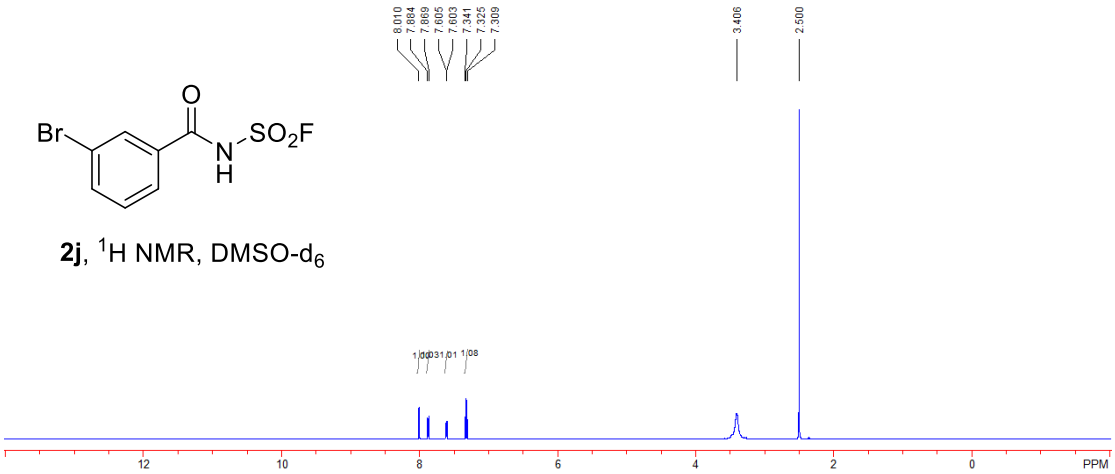


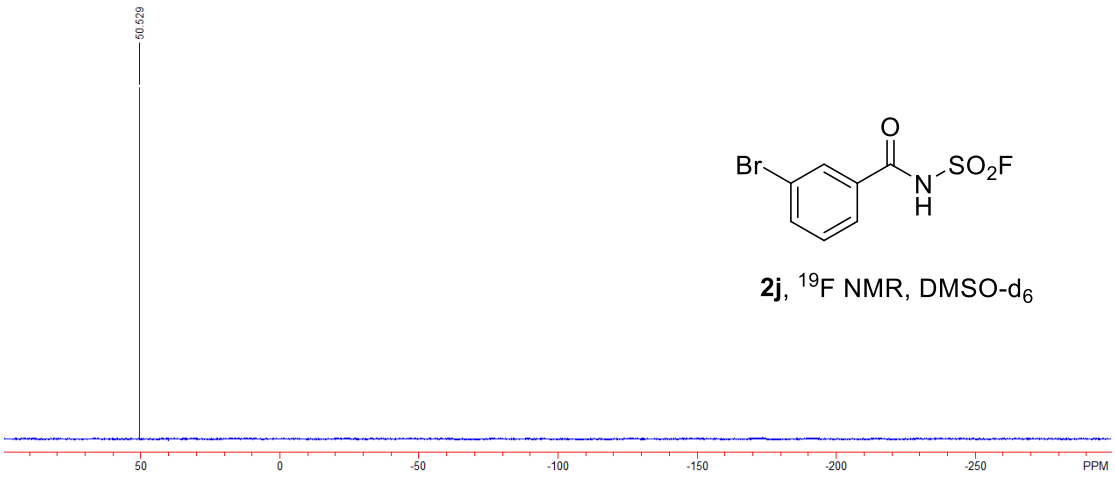
2i, ^{13}C NMR, DMSO-d$_6$



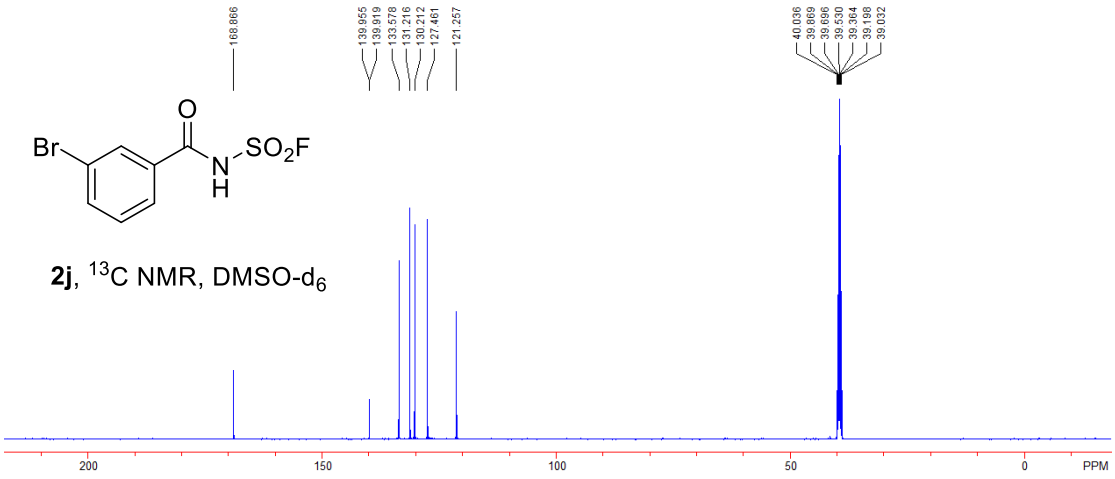
2j



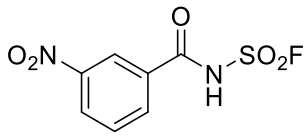
2j, ^{1}H NMR, DMSO-d$_6$



2j, ^{19}F NMR, DMSO-d$_6$



2j, ^{13}C NMR, DMSO-d$_6$



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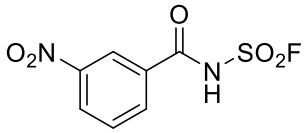
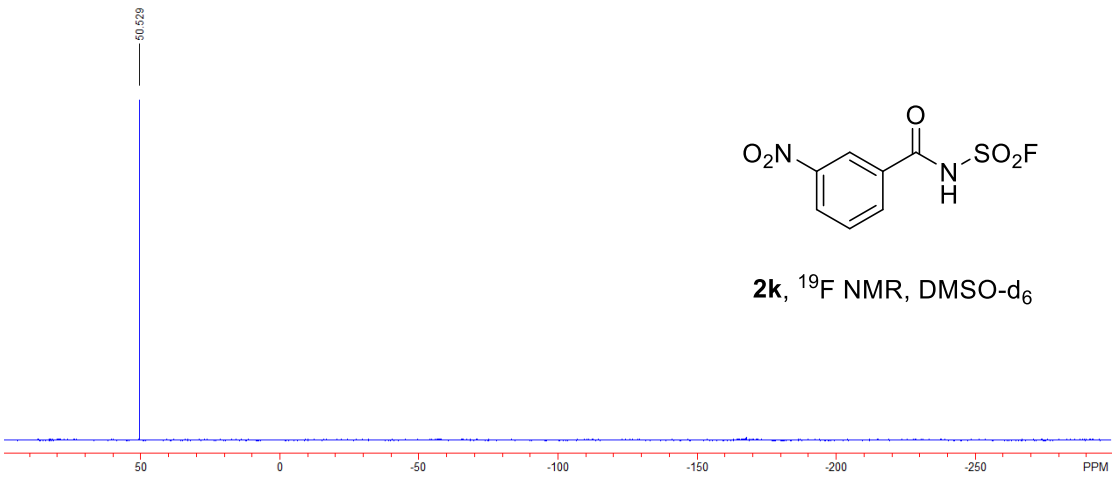


2k, ^{1}H NMR, DMSO-d$_6$



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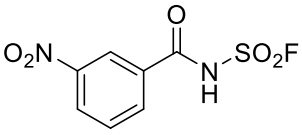

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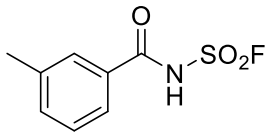

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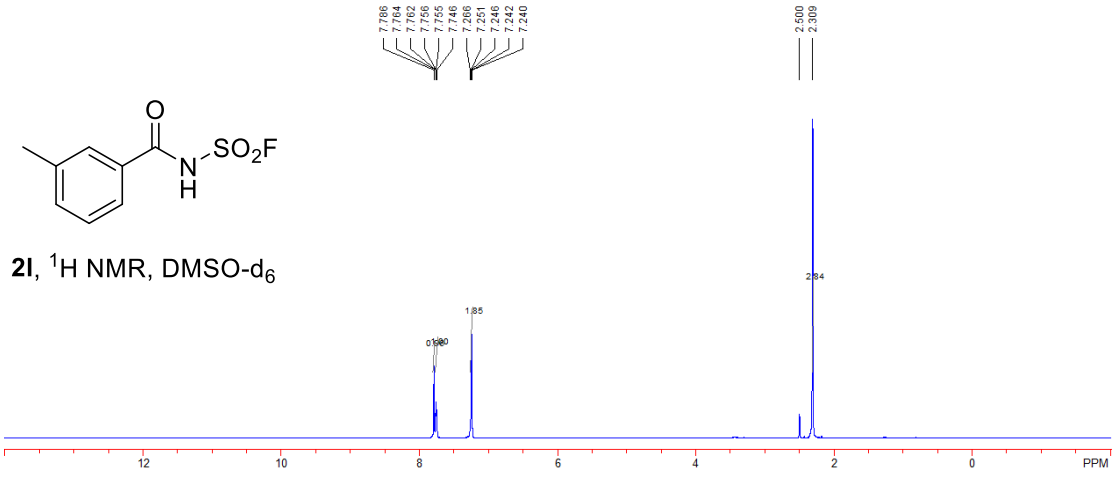

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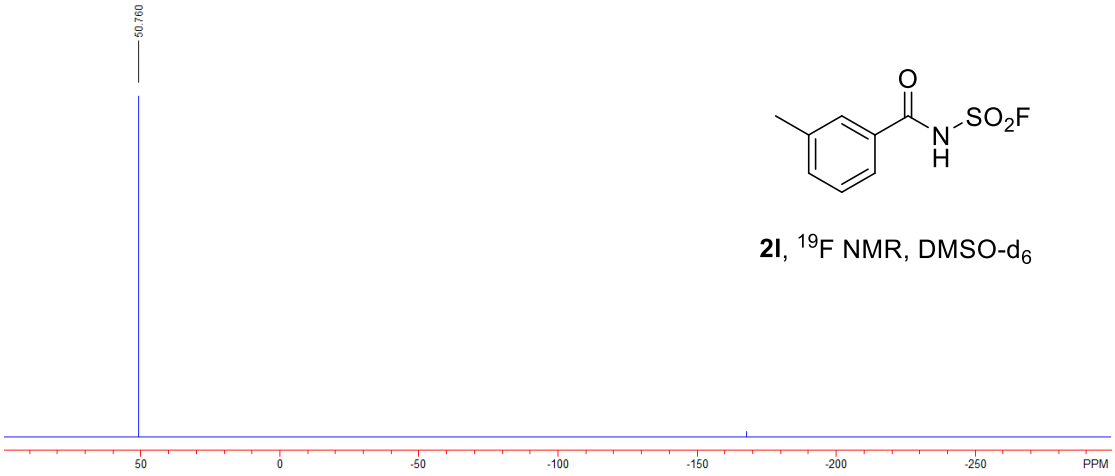

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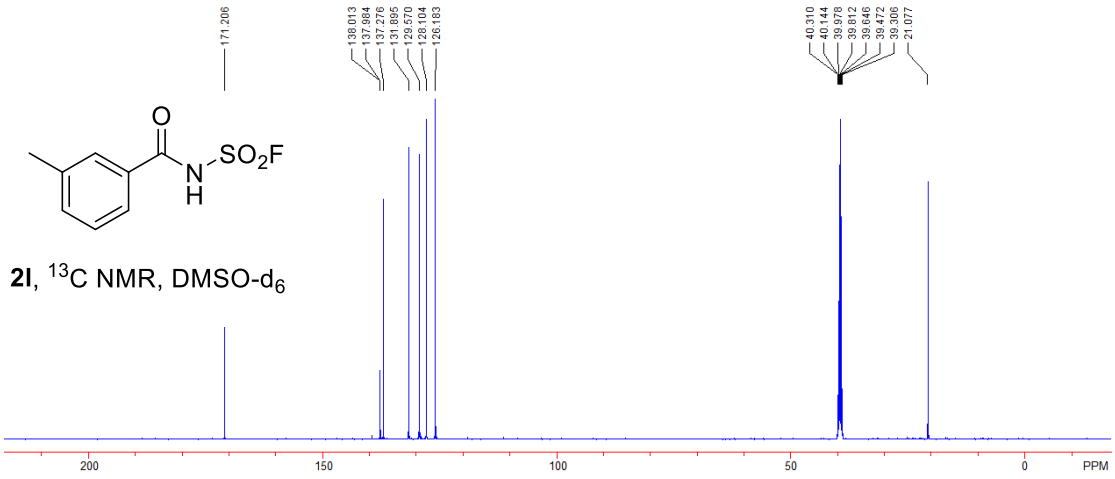
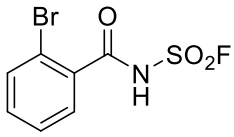


2l, ^{13}C NMR, DMSO-d$_6$



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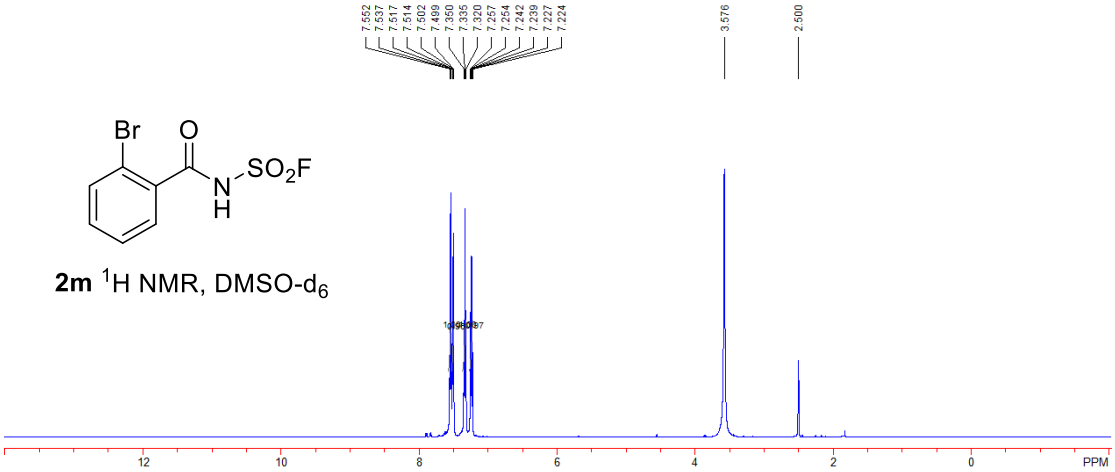
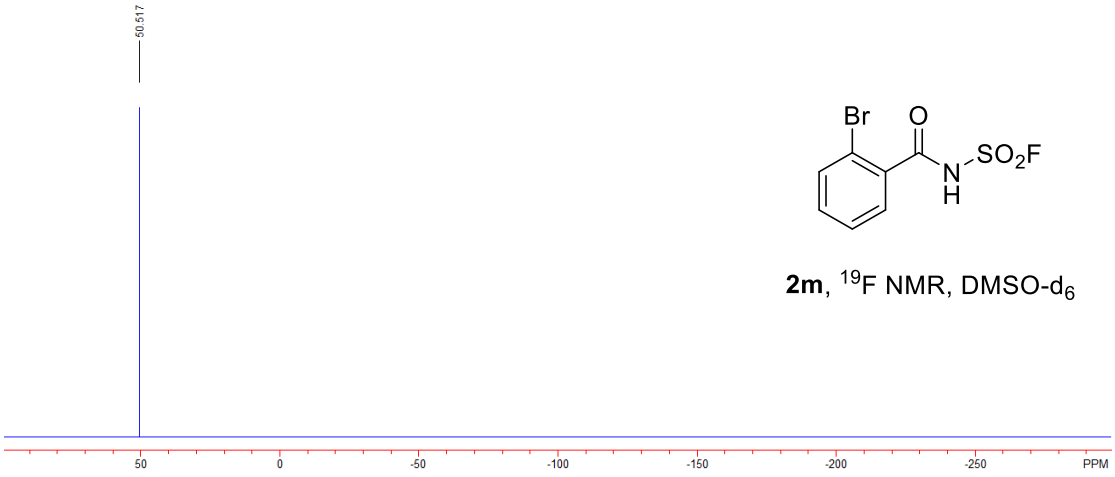


2m ^{1}H NMR, DMSO-d$_6$



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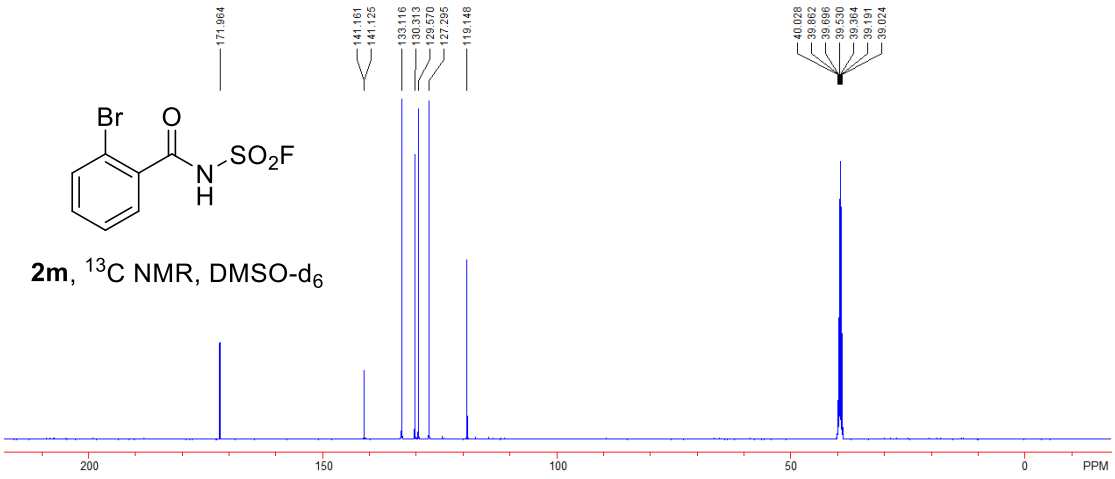
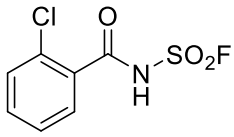


2m, ^{13}C NMR, DMSO-d$_6$



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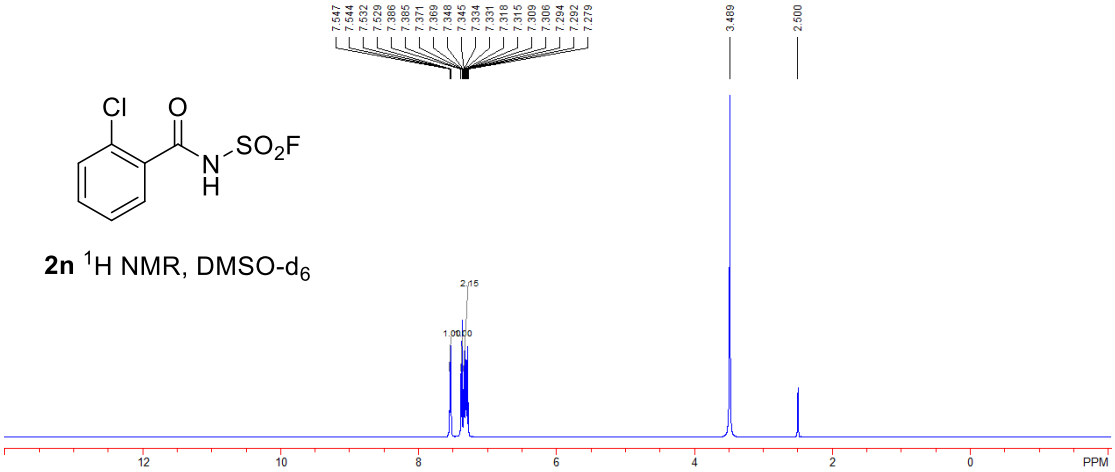
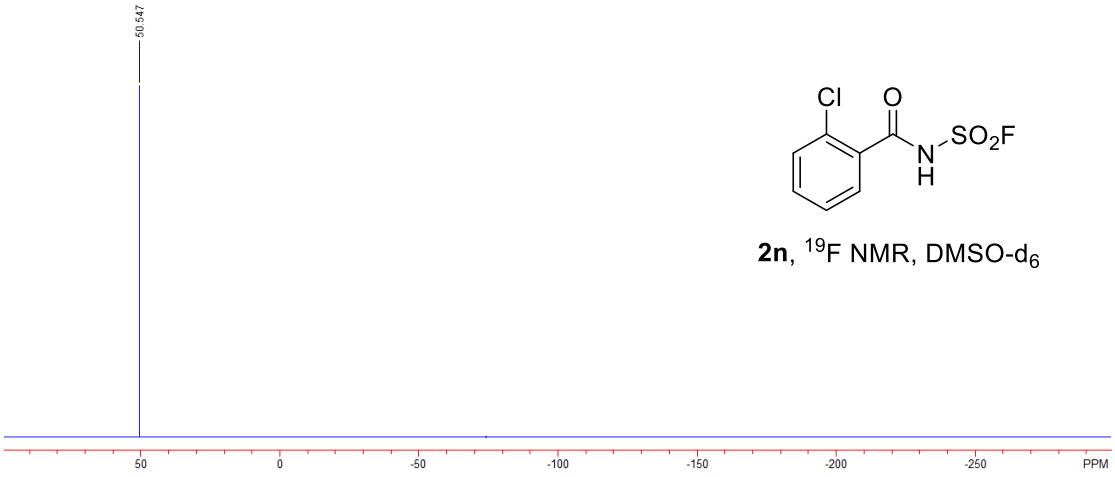


2n ^{1}H NMR, DMSO-d$_6$



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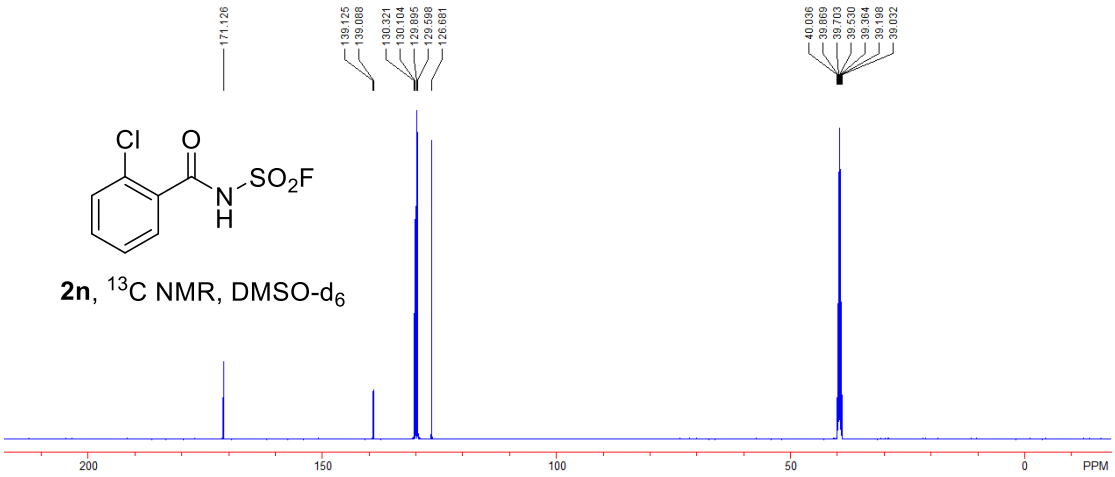
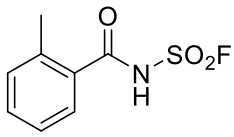


2n, ^{13}C NMR, DMSO-d$_6$



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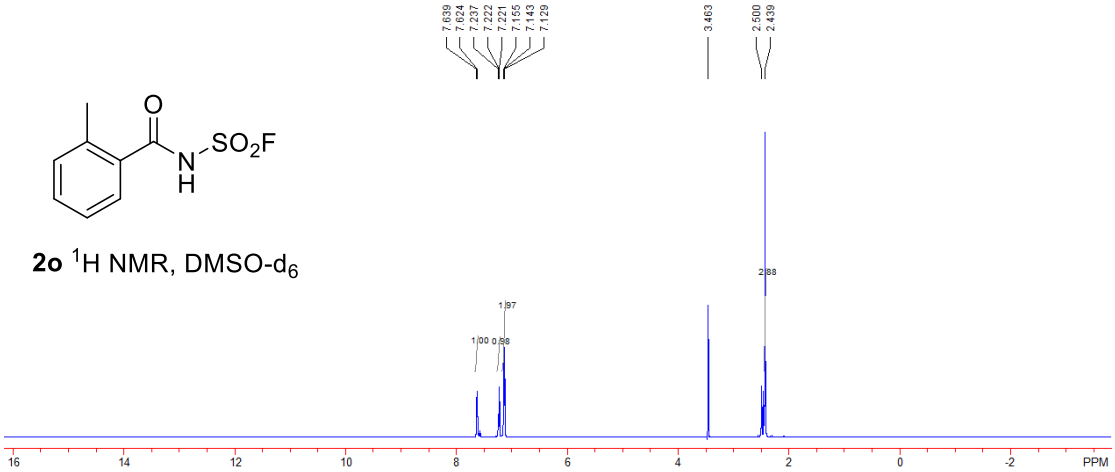
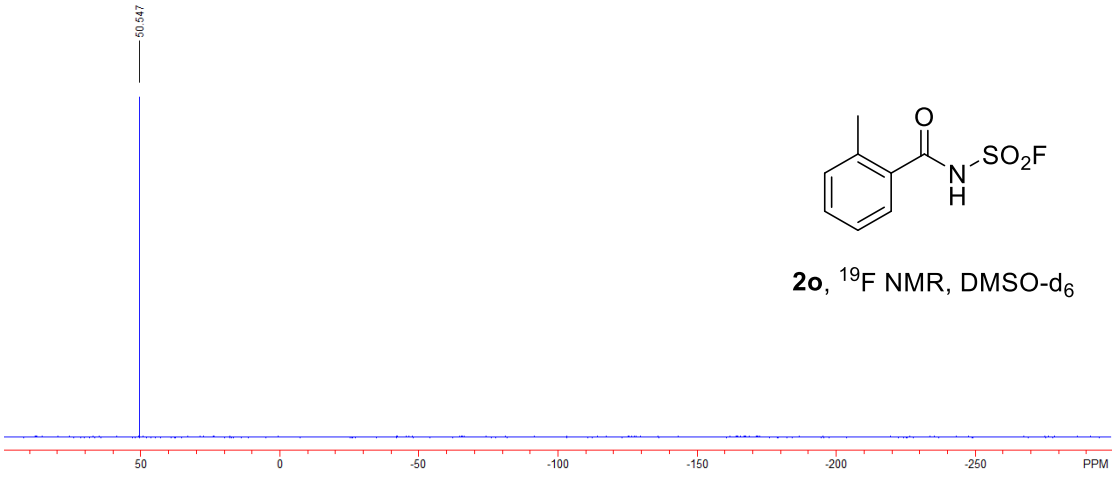


2o ^{1}H NMR, DMSO-d$_6$



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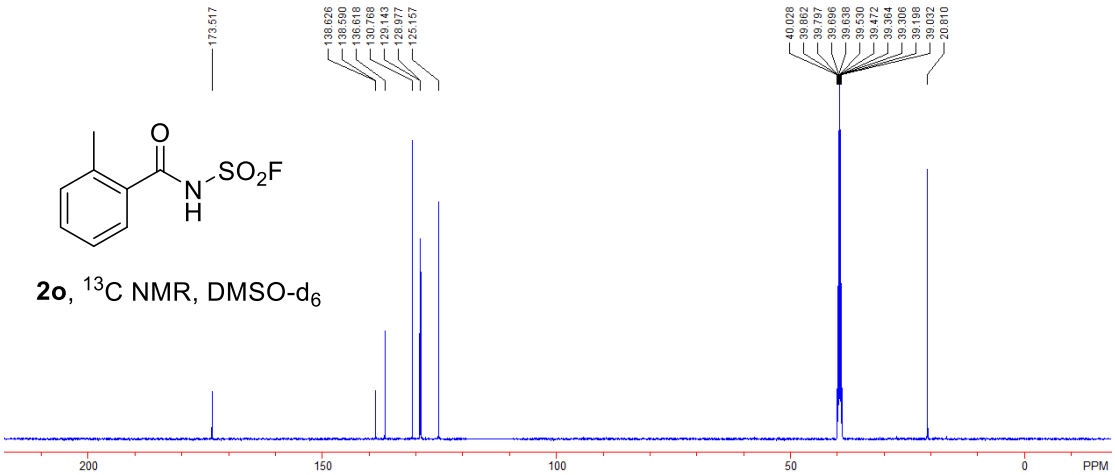

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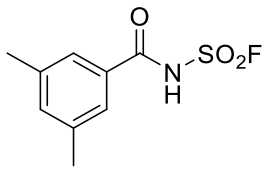

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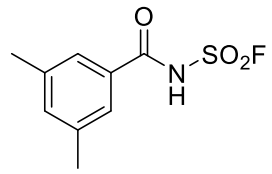
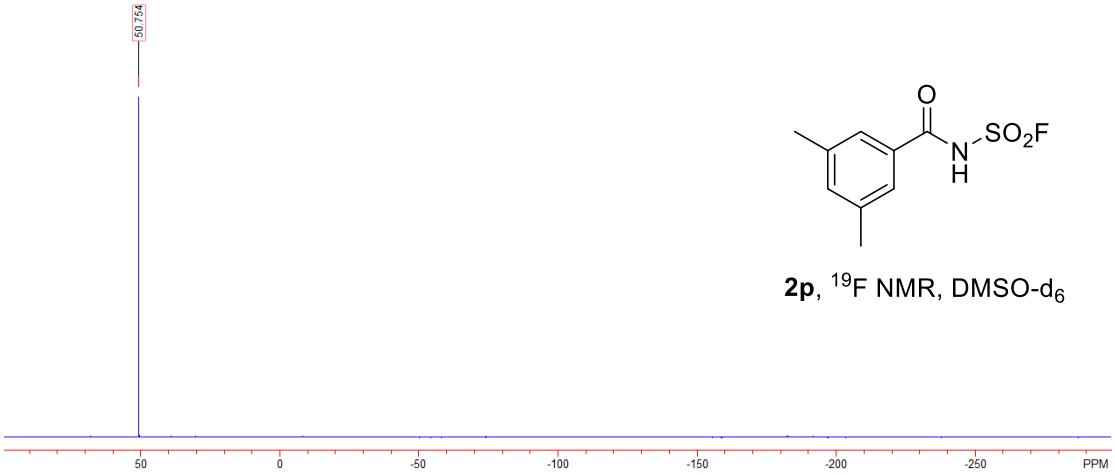


2p ^{1}H NMR, DMSO-d$_6$



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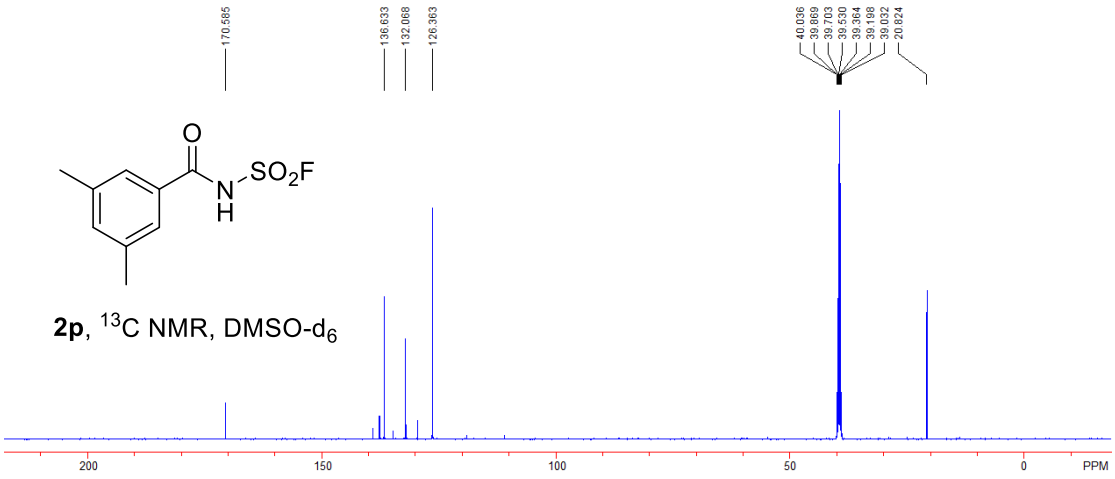

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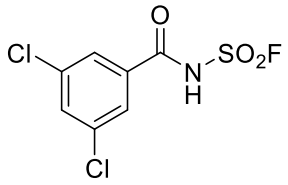

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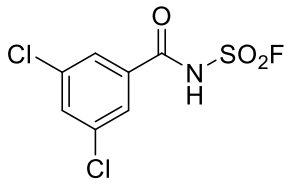
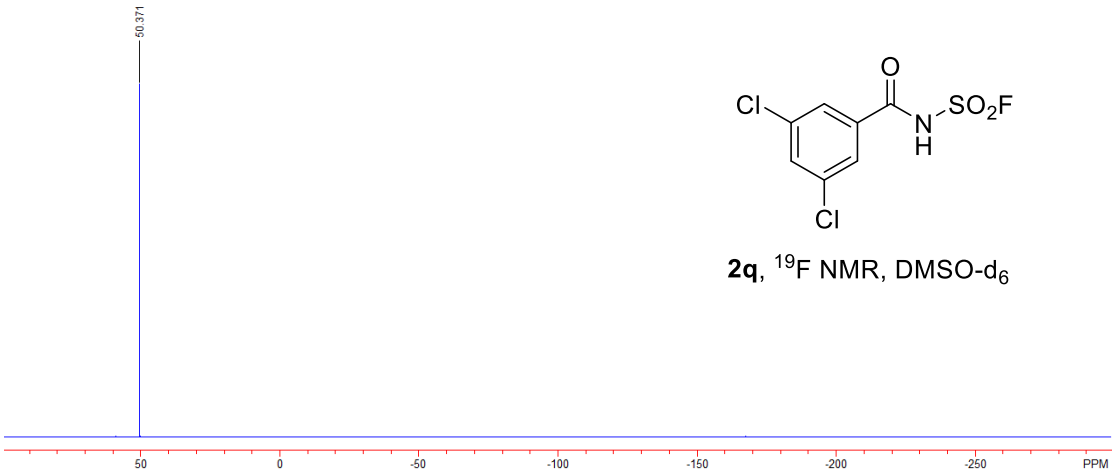


2q ^{1}H NMR, DMSO-d$_6$



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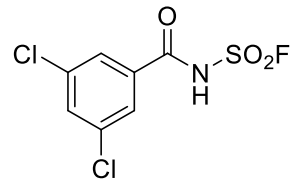

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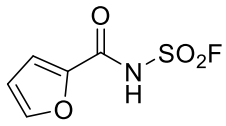

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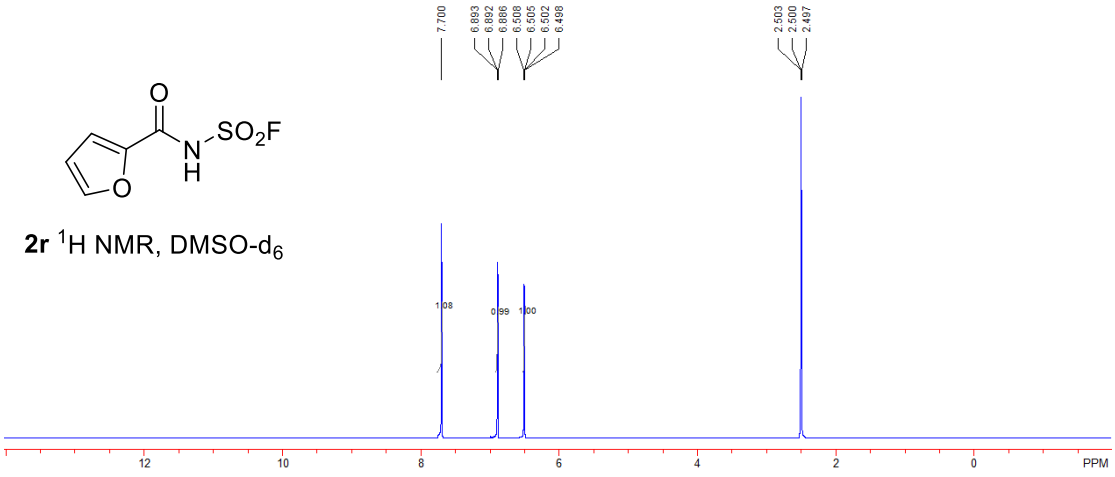
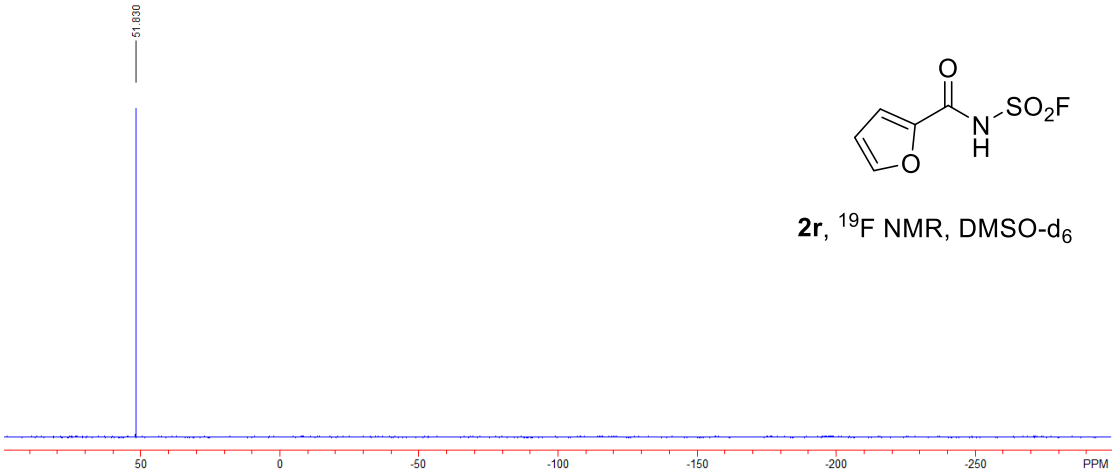


2r ^{1}H NMR, DMSO-d$_6$



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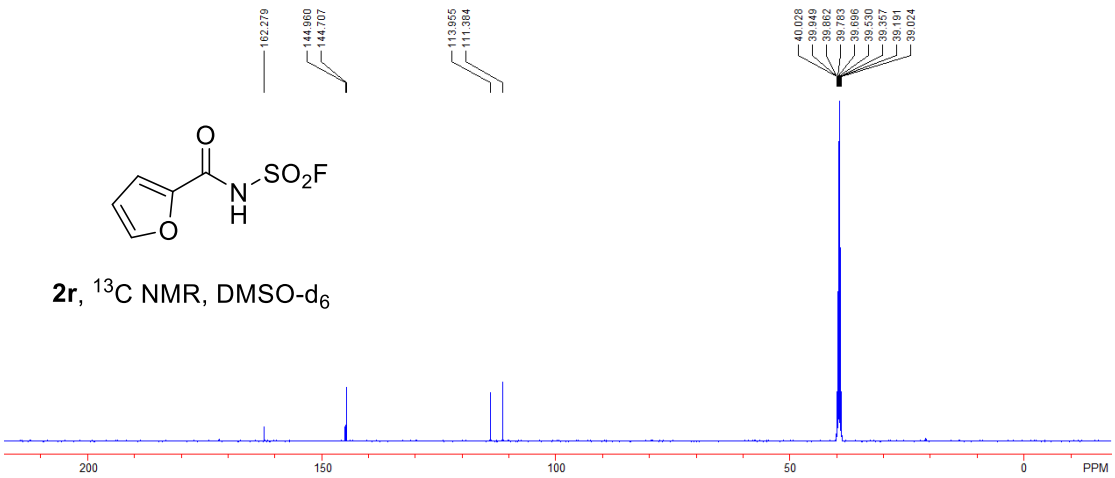
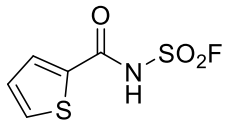


2r, ^{13}C NMR, DMSO-d$_6$



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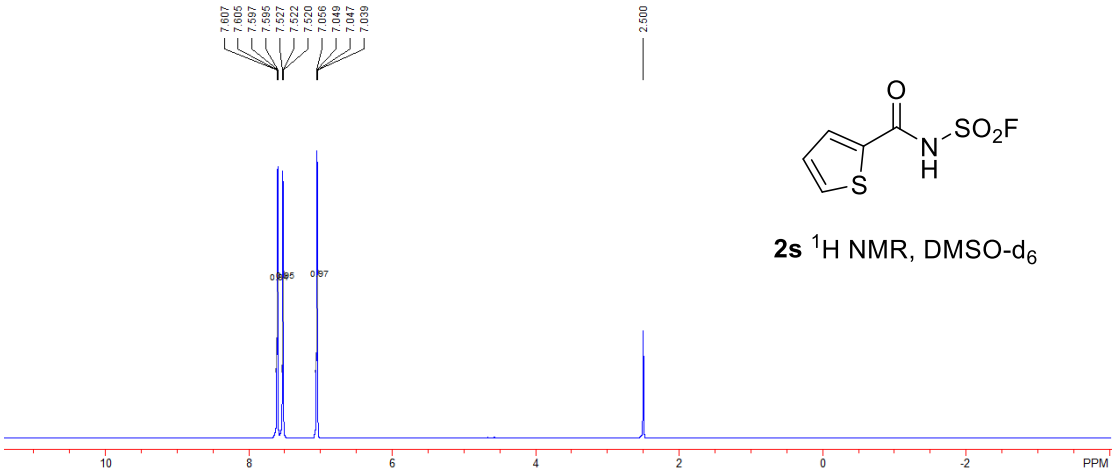
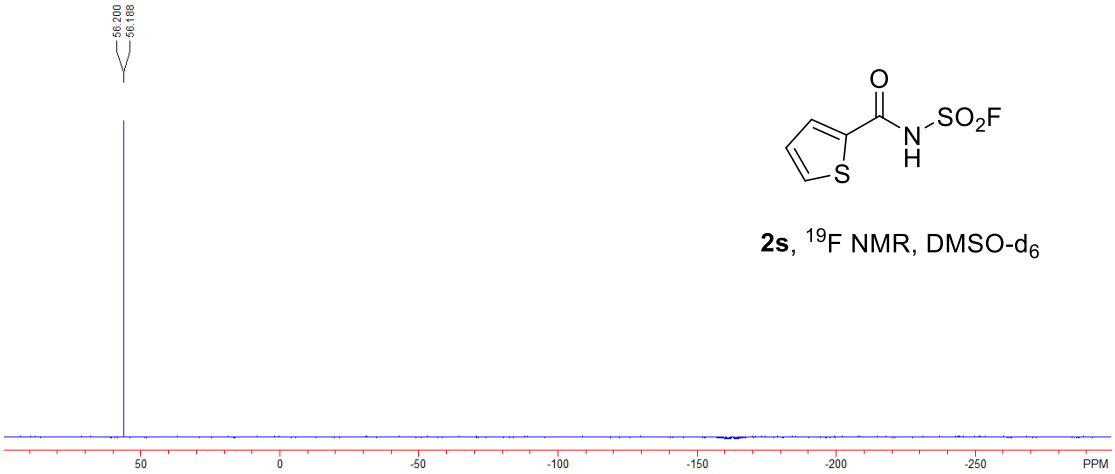


2s ^{1}H NMR, DMSO-d$_6$



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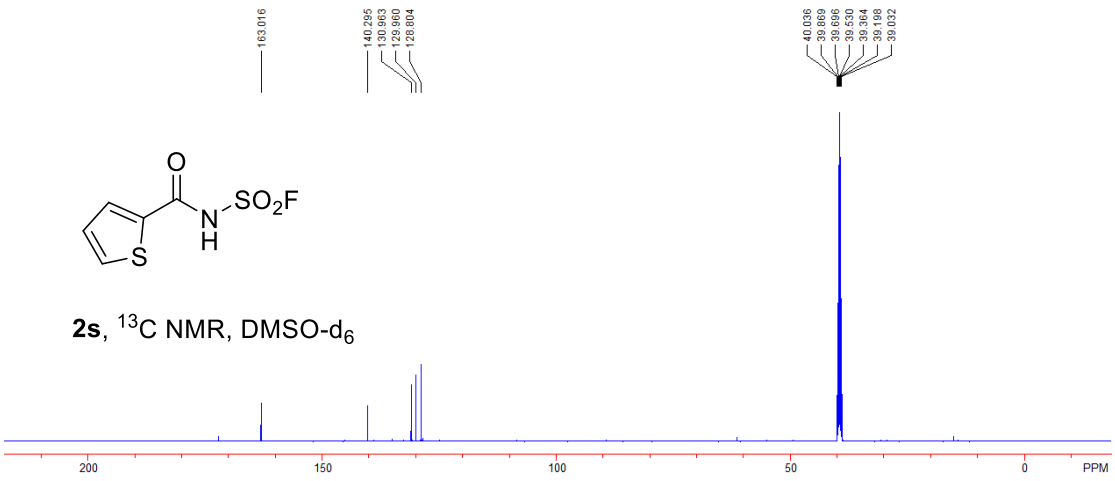
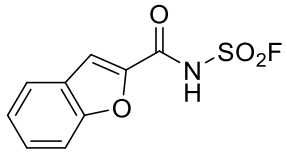


2s, ^{13}C NMR, DMSO-d$_6$



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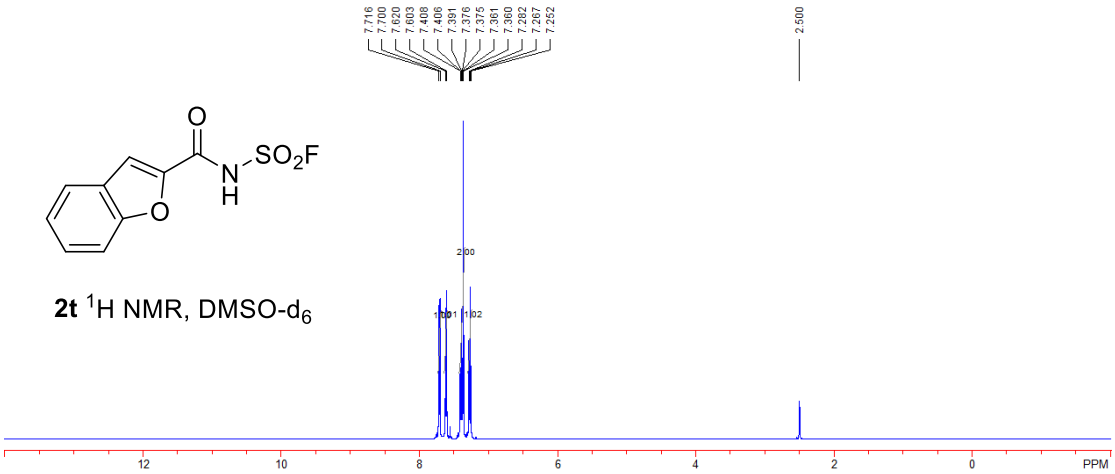
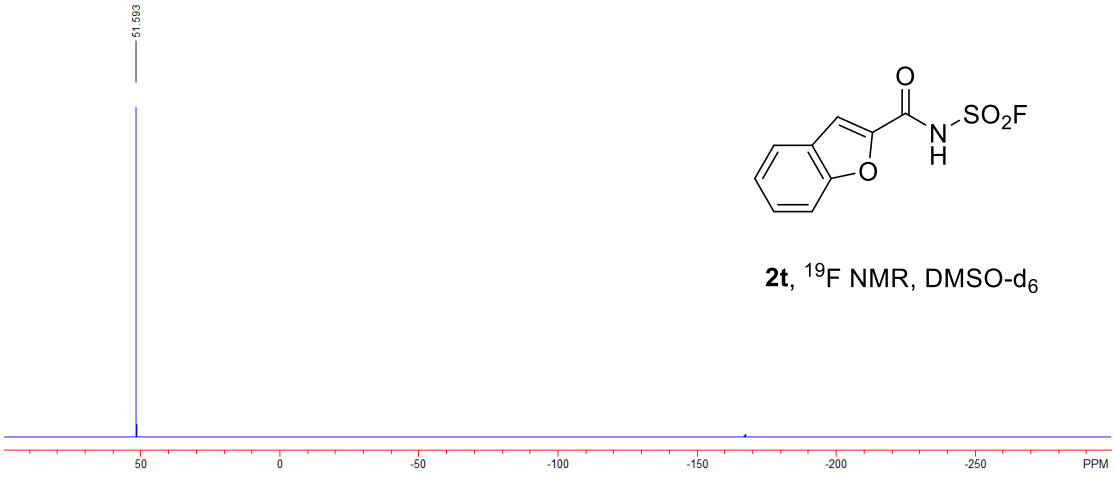


2t ^{1}H NMR, DMSO-d$_6$



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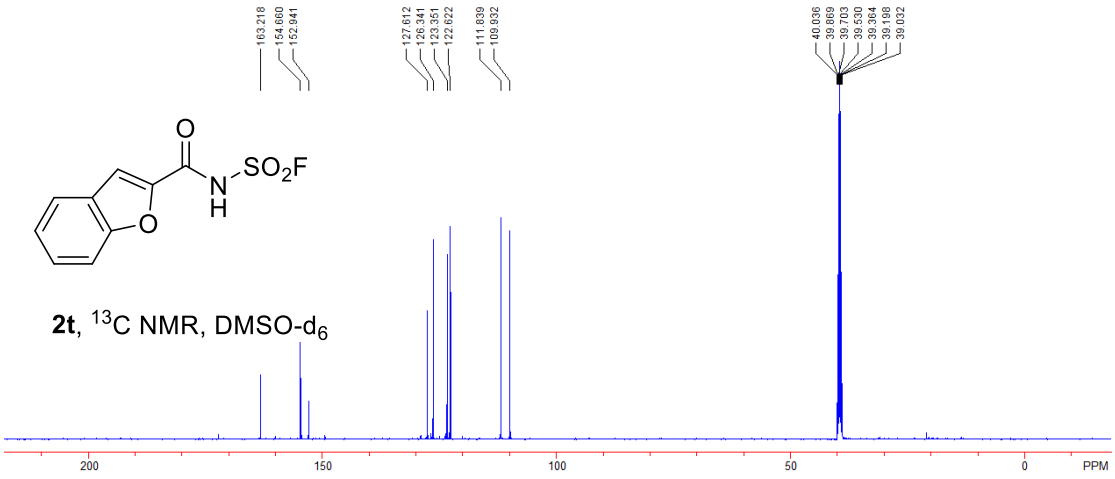
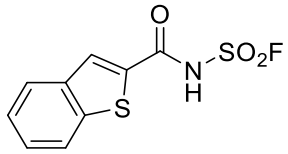


2t, ^{13}C NMR, DMSO-d$_6$



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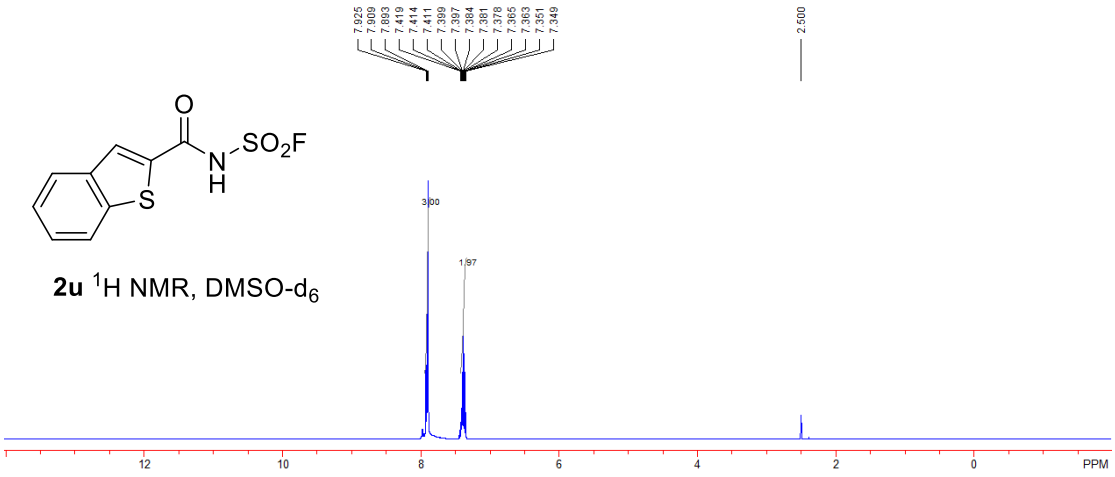
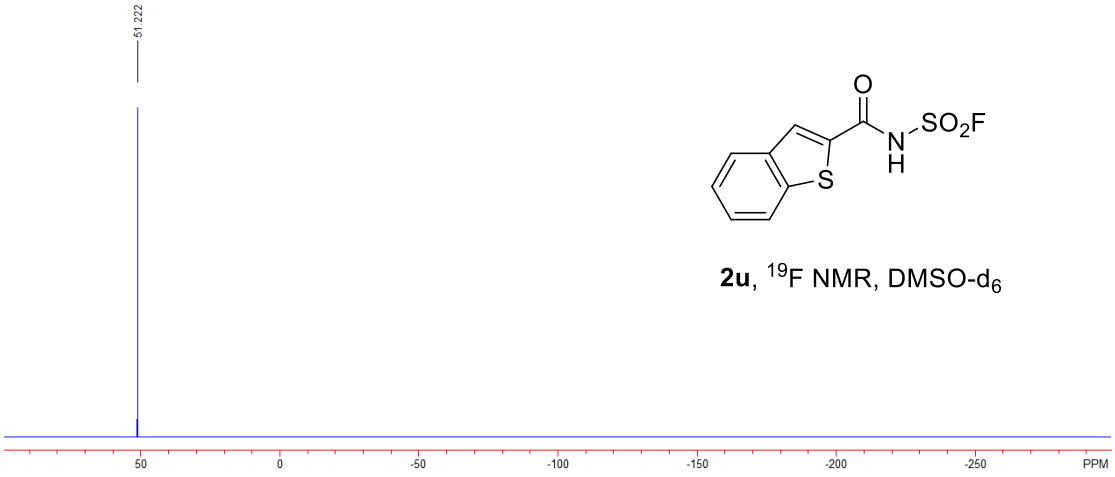
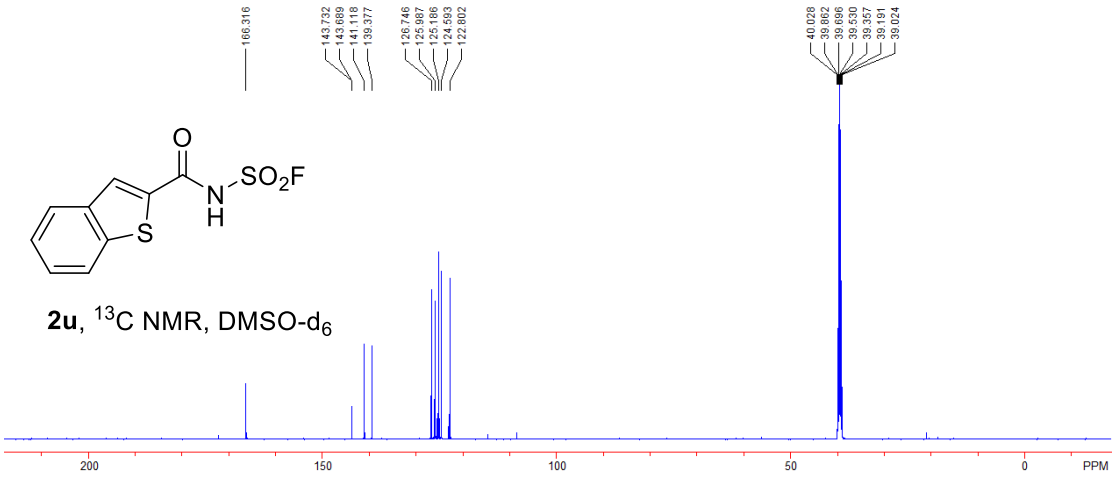


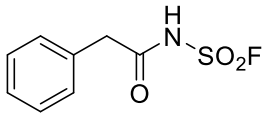
2u ^{1}H NMR, DMSO-d$_6$



2u, ^{19}F NMR, DMSO-d$_6$



2u, ^{13}C NMR, DMSO-d$_6$



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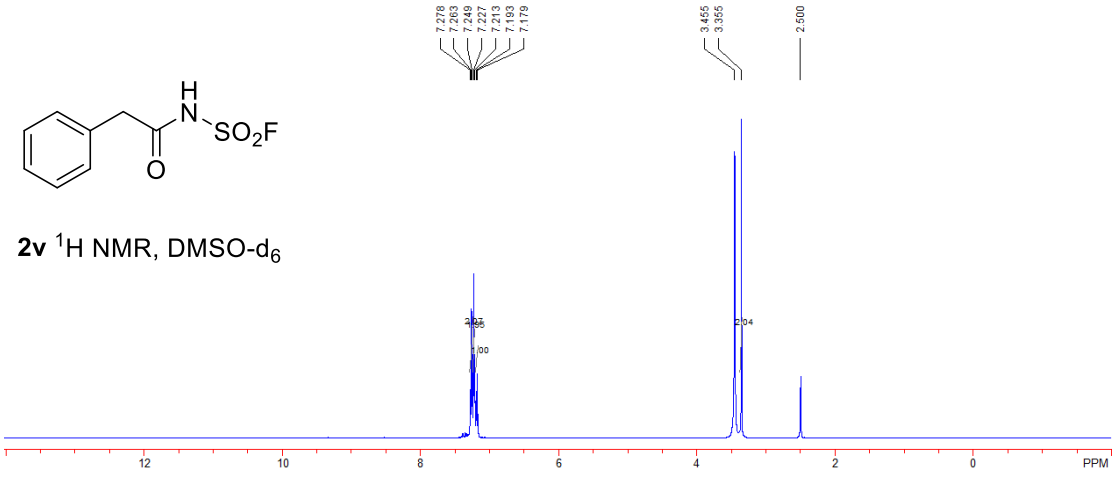

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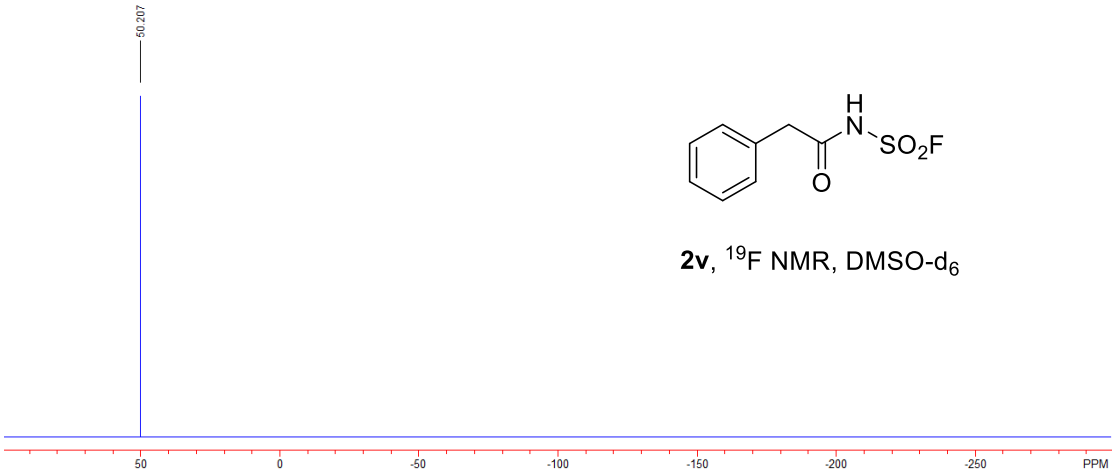

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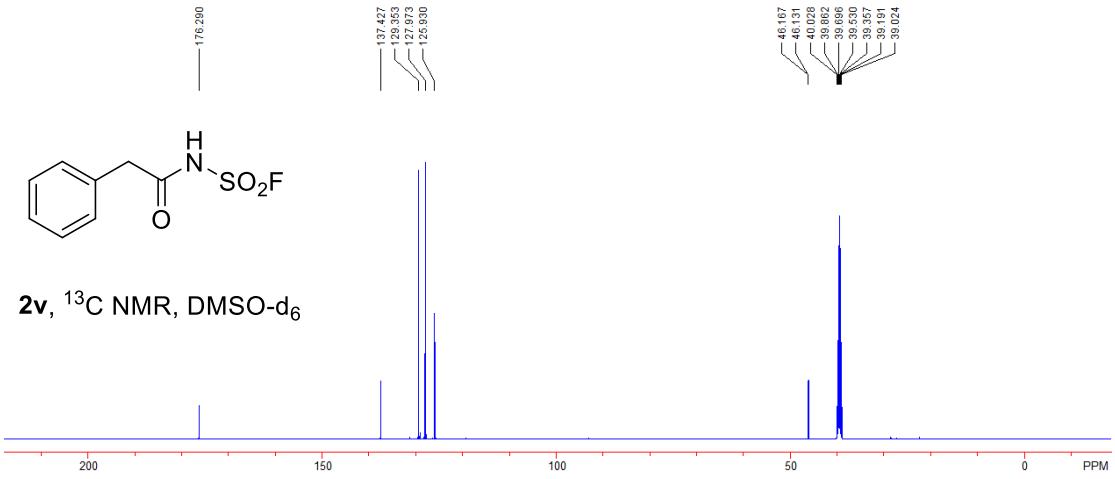
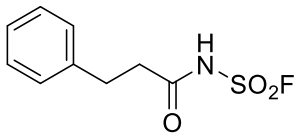
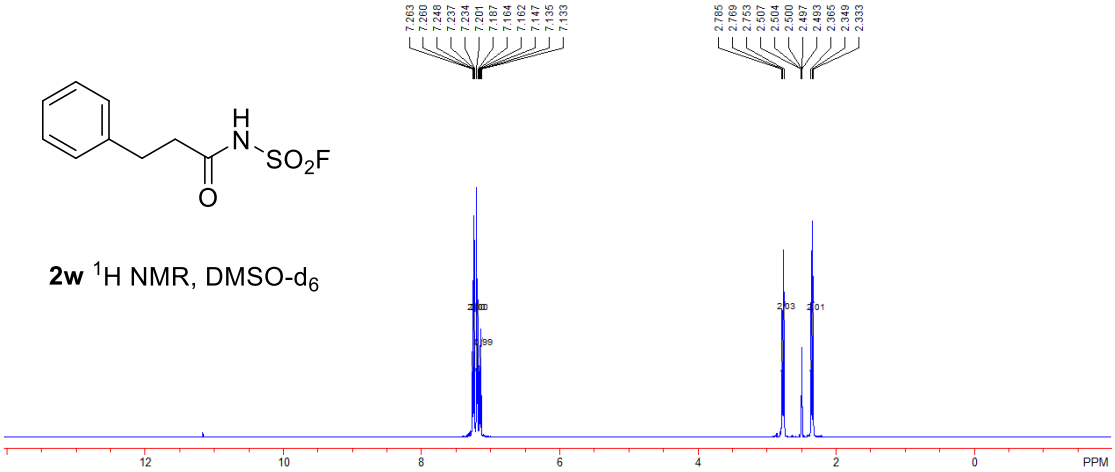


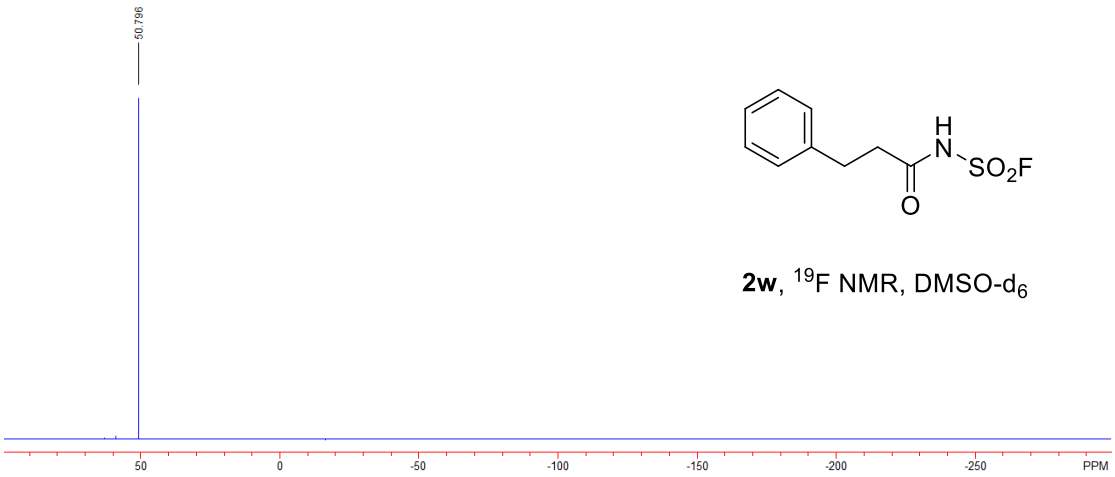
2v, ^{13}C NMR, DMSO-d$_6$



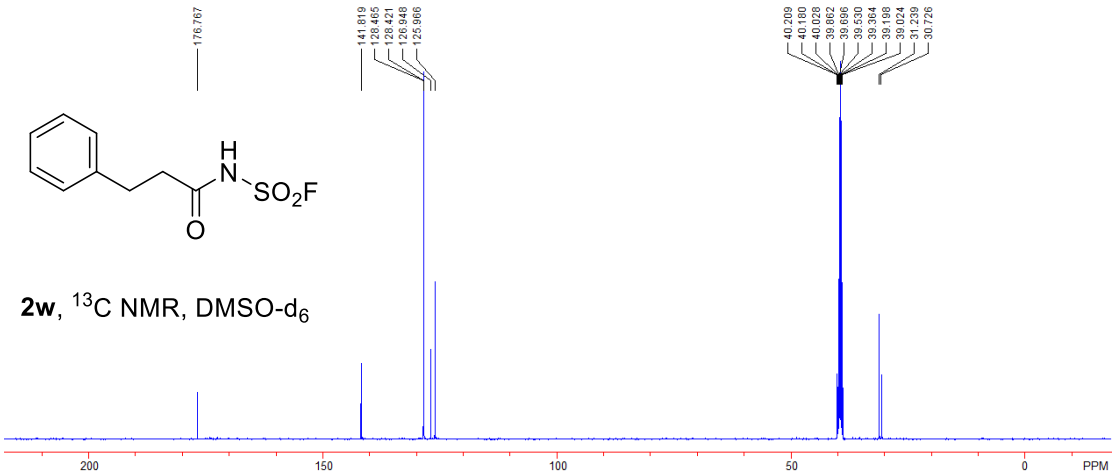
2w



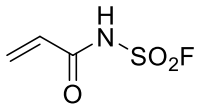
2w ^{1}H NMR, DMSO-d$_6$



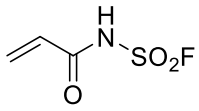
2w, ^{19}F NMR, DMSO-d$_6$



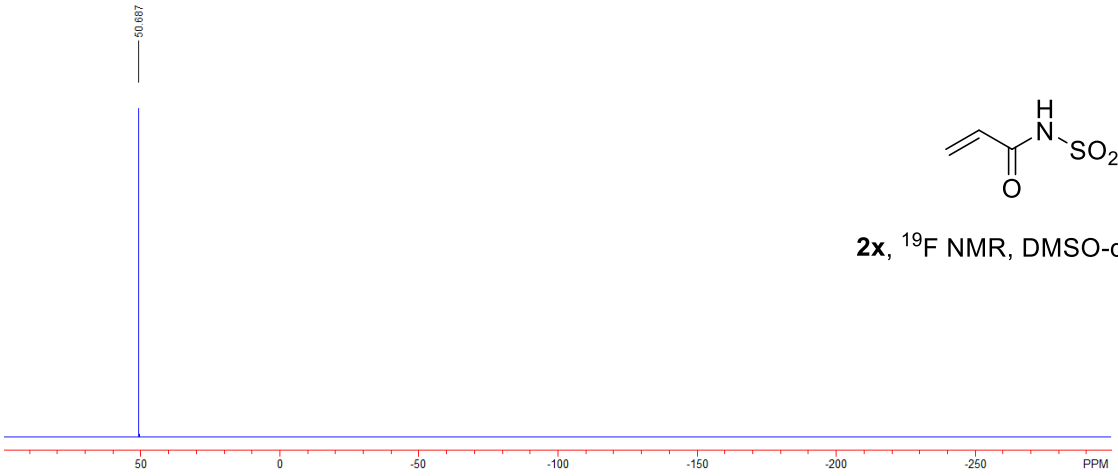
2w, ^{13}C NMR, DMSO-d$_6$



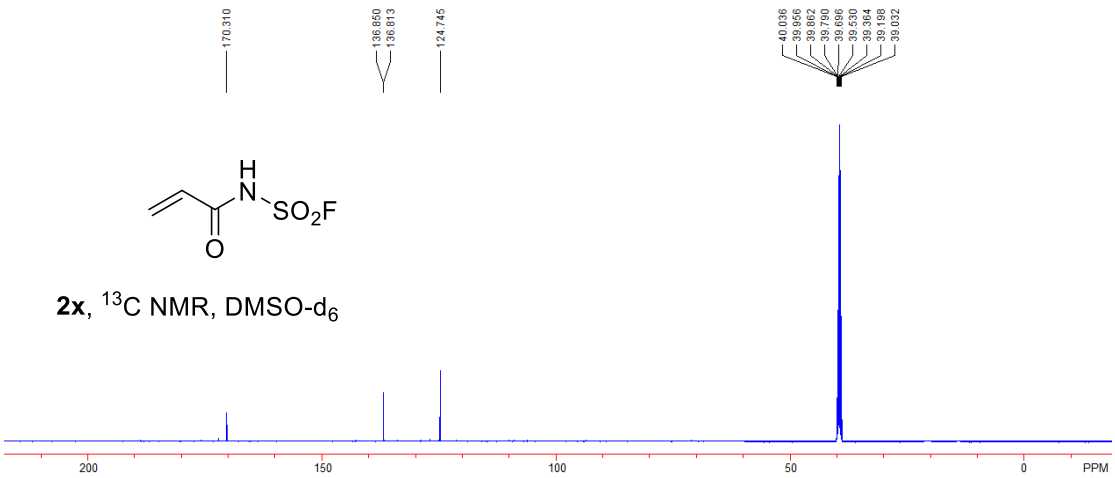
2x



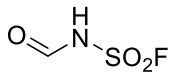
2x ^{1}H NMR, DMSO-d_6



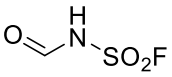
2x, ^{19}F NMR, DMSO-d_6



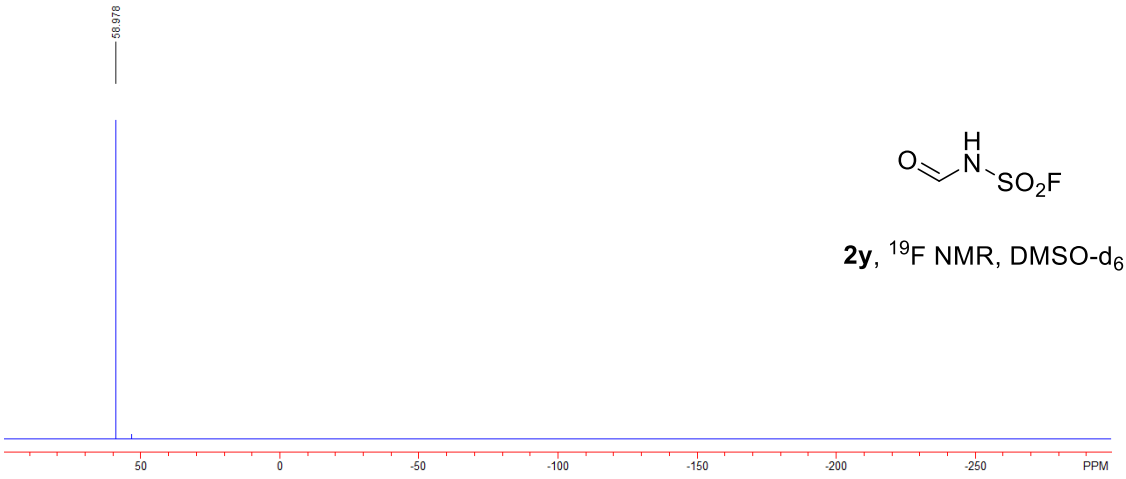
2x, ^{13}C NMR, DMSO-d_6



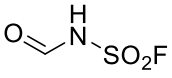
2y



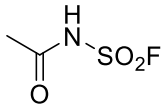
2y ^{1}H NMR, DMSO-d$_6$



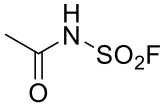
2y, ^{19}F NMR, DMSO-d$_6$



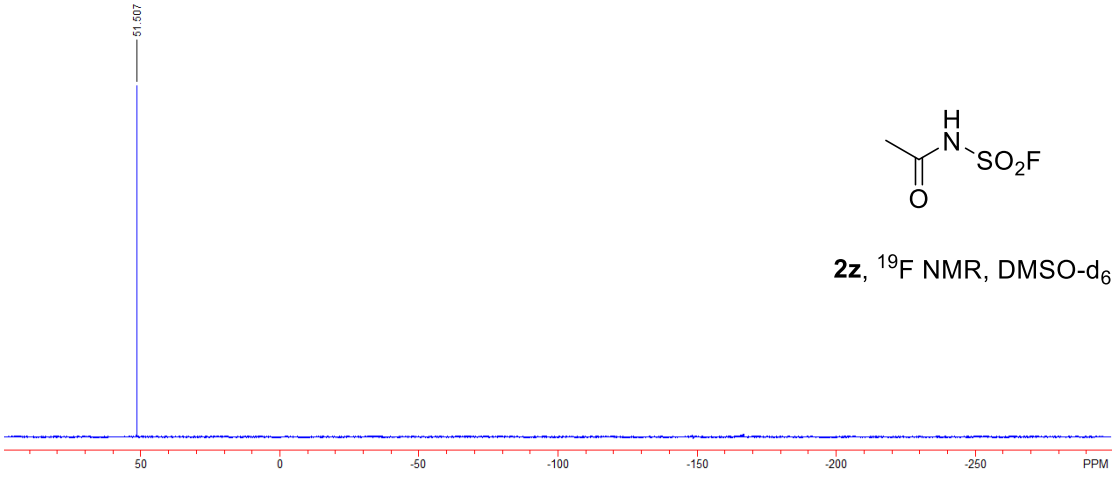
2y, ^{13}C NMR, DMSO-d$_6$



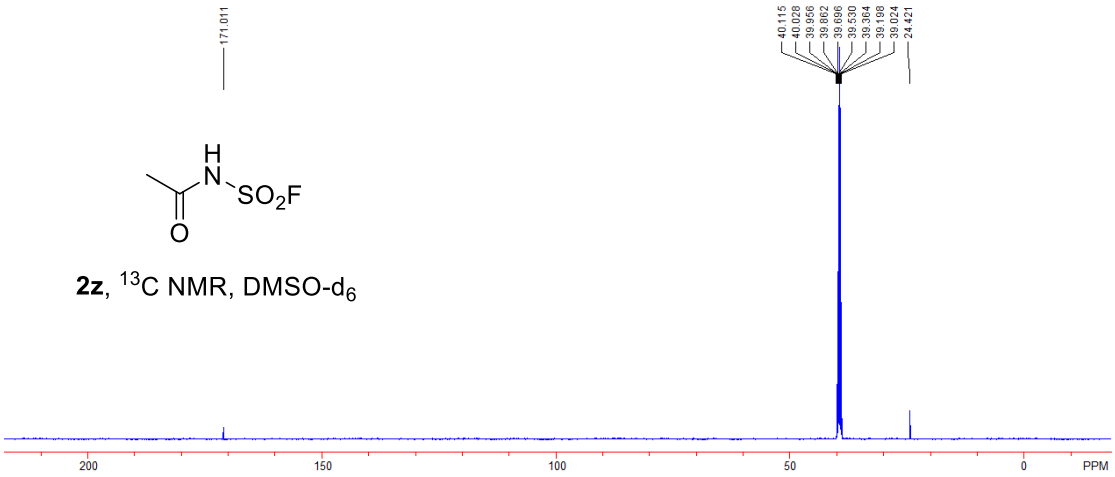
2z



2z ^{1}H NMR, DMSO-d$_6$



2z, ^{19}F NMR, DMSO-d$_6$



2z, ^{13}C NMR, DMSO-d$_6$

9. Single crystal data of 4e.

Table 4. Crystal data and structure refinement for 181108c.

Identification code	181108c
Empirical formula	C7 H8 F N2 Na O7 S
Formula weight	306.20
Temperature	293(2) K
Wavelength	0.71073 A
Crystal system, space group	Triclinic, P-1
Unit cell dimensions	a = 5.7233(5) A alpha = 93.5580(10) deg. b = 7.9155(7) A beta = 96.119(2) deg. c = 12.9158(11) A gamma = 91.6400(10) deg.
Volume	580.30(9) A^3
Z, Calculated density	2, 1.752 Mg/m^3
Absorption coefficient	0.362 mm^-1
F(000)	312
Crystal size	0.47 x 0.35 x 0.18 mm
Theta range for data collection	2.58 to 25.00 deg.
Limiting indices	-6<=h<=6, -9<=k<=9, -15<=l<=12
Reflections collected / unique	2959 / 2018 [R(int) = 0.0477]
Completeness to theta = 25.00	98.4 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9376 and 0.8481
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2018 / 0 / 172
Goodness-of-fit on F^2	1.106
Final R indices [I>2sigma(I)]	R1 = 0.0782, wR2 = 0.1947
R indices (all data)	R1 = 0.0929, wR2 = 0.2058
Largest diff. peak and hole	0.595 and -0.616 e.A^-3

Table 5. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for 181108c.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
Na(1)	7313(3)	8827(2)	4447(1)	45(1)
F(1)	2803(5)	4668(4)	4095(2)	66(1)
N(1)	1721(6)	5657(5)	2329(3)	44(1)
N(2)	6653(7)	8298(5)	-1527(3)	51(1)
O(1)	-96(6)	6673(4)	3955(3)	58(1)
O(2)	-859(7)	3811(5)	3164(3)	67(1)
O(3)	4261(5)	7789(4)	3147(2)	52(1)
O(4)	5341(7)	7984(6)	-2323(3)	80(1)
O(5)	8572(7)	8957(5)	-1527(3)	73(1)
O(6)	10555(5)	10375(4)	3820(2)	50(1)
O(7)	4097(5)	8327(4)	5452(2)	46(1)
S(1)	674(2)	5267(1)	3358(1)	42(1)
C(1)	3437(7)	6902(5)	2374(3)	40(1)
C(2)	4266(7)	7195(5)	1327(3)	38(1)
C(3)	2945(7)	6645(5)	395(3)	44(1)
C(4)	3687(8)	7005(6)	-552(3)	45(1)
C(5)	5820(7)	7859(5)	-534(3)	40(1)
C(6)	7204(8)	8378(6)	376(3)	45(1)
C(7)	6396(7)	8030(6)	1311(3)	45(1)

Table 6. Bond lengths [A] and angles [deg] for 181108c.

Na(1)-O(3)	2.380(3)
Na(1)-O(1)#1	2.392(3)
Na(1)-O(7)	2.400(3)
Na(1)-O(7)#2	2.415(3)
Na(1)-O(6)	2.432(3)
Na(1)-O(6)#3	2.470(3)
Na(1)-Na(1)#2	3.641(3)
Na(1)-Na(1)#3	3.655(3)
F(1)-S(1)	1.567(3)
N(1)-C(1)	1.365(5)
N(1)-S(1)	1.560(4)
N(2)-O(5)	1.203(5)
N(2)-O(4)	1.215(5)
N(2)-C(5)	1.472(5)
O(1)-S(1)	1.422(3)
O(1)-Na(1)#4	2.392(3)
O(2)-S(1)	1.421(3)
O(3)-C(1)	1.228(5)
O(6)-Na(1)#3	2.470(3)
O(6)-H(6A)	0.8500
O(6)-H(6B)	0.8499
O(7)-Na(1)#2	2.415(3)
O(7)-H(7A)	0.8500
O(7)-H(7B)	0.8500
C(1)-C(2)	1.508(6)
C(2)-C(7)	1.373(6)
C(2)-C(3)	1.391(5)
C(3)-C(4)	1.380(6)
C(3)-H(3)	0.9300
C(4)-C(5)	1.376(6)
C(4)-H(4)	0.9300
C(5)-C(6)	1.379(6)
C(6)-C(7)	1.379(6)
C(6)-H(6)	0.9300
C(7)-H(7)	0.9300
O(3)-Na(1)-O(1)#1	92.37(13)
O(3)-Na(1)-O(7)	77.52(11)
O(1)#1-Na(1)-O(7)	123.41(13)
O(3)-Na(1)-O(7)#2	94.46(12)
O(1)#1-Na(1)-O(7)#2	154.82(13)

O(7)-Na(1)-O(7)#2	81.75(12)
O(3)-Na(1)-O(6)	115.75(12)
O(1)#1-Na(1)-O(6)	75.73(11)
O(7)-Na(1)-O(6)	157.70(12)
O(7)#2-Na(1)-O(6)	79.47(11)
O(3)-Na(1)-O(6)#3	160.29(13)
O(1)#1-Na(1)-O(6)#3	96.48(12)
O(7)-Na(1)-O(6)#3	82.90(11)
O(7)#2-Na(1)-O(6)#3	84.95(11)
O(6)-Na(1)-O(6)#3	83.57(11)
O(3)-Na(1)-Na(1)#2	84.79(10)
O(1)#1-Na(1)-Na(1)#2	164.42(12)
O(7)-Na(1)-Na(1)#2	41.02(8)
O(7)#2-Na(1)-Na(1)#2	40.72(7)
O(6)-Na(1)-Na(1)#2	119.26(11)
O(6)#3-Na(1)-Na(1)#2	81.96(9)
O(3)-Na(1)-Na(1)#3	157.71(11)
O(1)#1-Na(1)-Na(1)#3	84.97(10)
O(7)-Na(1)-Na(1)#3	122.15(10)
O(7)#2-Na(1)-Na(1)#3	79.57(9)
O(6)-Na(1)-Na(1)#3	42.18(8)
O(6)#3-Na(1)-Na(1)#3	41.39(7)
Na(1)#2-Na(1)-Na(1)#3	103.34(8)
C(1)-N(1)-S(1)	118.2(3)
O(5)-N(2)-O(4)	122.3(4)
O(5)-N(2)-C(5)	119.7(4)
O(4)-N(2)-C(5)	117.9(4)
S(1)-O(1)-Na(1)#4	157.2(2)
C(1)-O(3)-Na(1)	154.5(3)
Na(1)-O(6)-Na(1)#3	96.43(11)
Na(1)-O(6)-H(6A)	112.4
Na(1)#3-O(6)-H(6A)	112.6
Na(1)-O(6)-H(6B)	112.4
Na(1)#3-O(6)-H(6B)	112.4
H(6A)-O(6)-H(6B)	110.1
Na(1)-O(7)-Na(1)#2	98.25(11)
Na(1)-O(7)-H(7A)	112.2
Na(1)#2-O(7)-H(7A)	112.1
Na(1)-O(7)-H(7B)	112.1
Na(1)#2-O(7)-H(7B)	112.1
H(7A)-O(7)-H(7B)	109.8
O(2)-S(1)-O(1)	117.8(2)
O(2)-S(1)-N(1)	109.0(2)
O(1)-S(1)-N(1)	116.6(2)

O(2)-S(1)-F(1)	104.3(2)
O(1)-S(1)-F(1)	102.28(18)
N(1)-S(1)-F(1)	104.93(18)
O(3)-C(1)-N(1)	127.0(4)
O(3)-C(1)-C(2)	119.6(4)
N(1)-C(1)-C(2)	113.4(3)
C(7)-C(2)-C(3)	119.9(4)
C(7)-C(2)-C(1)	118.1(3)
C(3)-C(2)-C(1)	122.0(4)
C(4)-C(3)-C(2)	120.8(4)
C(4)-C(3)-H(3)	119.6
C(2)-C(3)-H(3)	119.6
C(5)-C(4)-C(3)	117.4(4)
C(5)-C(4)-H(4)	121.3
C(3)-C(4)-H(4)	121.3
C(4)-C(5)-C(6)	123.2(4)
C(4)-C(5)-N(2)	119.1(4)
C(6)-C(5)-N(2)	117.7(4)
C(7)-C(6)-C(5)	118.1(4)
C(7)-C(6)-H(6)	120.9
C(5)-C(6)-H(6)	120.9
C(2)-C(7)-C(6)	120.5(4)
C(2)-C(7)-H(7)	119.7
C(6)-C(7)-H(7)	119.7

Symmetry transformations used to generate equivalent atoms:
#1 x+1,y,z #2 -x+1,-y+2,-z+1 #3 -x+2,-y+2,-z+1
#4 x-1,y,z

Table 7. Anisotropic displacement parameters (A^2 x 10^3) for 181108c.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
Na(1)	40(1)	46(1)	48(1)	2(1)	5(1)	2(1)
F(1)	70(2)	73(2)	58(2)	27(1)	-4(1)	20(2)
N(1)	47(2)	45(2)	40(2)	6(2)	2(2)	-10(2)
N(2)	60(2)	51(2)	44(2)	9(2)	6(2)	7(2)
O(1)	61(2)	47(2)	70(2)	8(2)	24(2)	9(2)
O(2)	77(2)	59(2)	65(2)	5(2)	12(2)	-27(2)
O(3)	54(2)	63(2)	37(2)	0(1)	1(1)	-17(2)
O(4)	89(3)	114(3)	37(2)	17(2)	1(2)	-6(2)
O(5)	63(2)	95(3)	61(2)	12(2)	17(2)	-19(2)
O(6)	49(2)	46(2)	58(2)	16(1)	11(1)	1(1)
O(7)	49(2)	46(2)	45(2)	11(1)	2(1)	1(1)
S(1)	46(1)	36(1)	46(1)	9(1)	5(1)	0(1)
C(1)	36(2)	41(2)	42(2)	5(2)	1(2)	4(2)
C(2)	39(2)	33(2)	41(2)	5(2)	-1(2)	3(2)
C(3)	39(2)	45(3)	48(2)	9(2)	3(2)	-2(2)
C(4)	49(2)	48(3)	37(2)	1(2)	-4(2)	1(2)
C(5)	44(2)	40(2)	37(2)	10(2)	6(2)	6(2)
C(6)	42(2)	47(3)	48(2)	11(2)	4(2)	-5(2)
C(7)	39(2)	55(3)	38(2)	5(2)	-3(2)	-2(2)

Table 8. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for 181108c.

	x	y	z	U(eq)
H(6A)	10103	11230	3496	60
H(6B)	11349	9747	3441	60
H(7A)	4555	7994	6051	56
H(7B)	3072	7625	5135	56
H(3)	1543	6026	409	53
H(4)	2781	6683	-1178	55
H(6)	8643	8947	360	55
H(7)	7300	8364	1936	54

Table 9. Torsion angles [deg] for 181108c.

O(1)#1-Na(1)-O(3)-C(1)	-10.7(7)
O(7)-Na(1)-O(3)-C(1)	-134.4(7)
O(7)#2-Na(1)-O(3)-C(1)	145.1(7)
O(6)-Na(1)-O(3)-C(1)	64.6(7)
O(6)#3-Na(1)-O(3)-C(1)	-127.4(7)
Na(1)#2-Na(1)-O(3)-C(1)	-175.3(7)
Na(1)#3-Na(1)-O(3)-C(1)	71.8(8)
O(3)-Na(1)-O(6)-Na(1)#3	175.93(14)
O(1)#1-Na(1)-O(6)-Na(1)#3	-98.39(13)
O(7)-Na(1)-O(6)-Na(1)#3	53.0(3)
O(7)#2-Na(1)-O(6)-Na(1)#3	86.03(12)
O(6)#3-Na(1)-O(6)-Na(1)#3	0.0
Na(1)#2-Na(1)-O(6)-Na(1)#3	77.03(12)
O(3)-Na(1)-O(7)-Na(1)#2	-96.47(13)
O(1)#1-Na(1)-O(7)-Na(1)#2	179.00(14)
O(7)#2-Na(1)-O(7)-Na(1)#2	0.0
O(6)-Na(1)-O(7)-Na(1)#2	32.8(3)
O(6)#3-Na(1)-O(7)-Na(1)#2	85.90(12)
Na(1)#3-Na(1)-O(7)-Na(1)#2	72.11(13)
Na(1)#4-O(1)-S(1)-O(2)	-51.3(6)
Na(1)#4-O(1)-S(1)-N(1)	81.2(5)
Na(1)#4-O(1)-S(1)-F(1)	-165.0(5)
C(1)-N(1)-S(1)-O(2)	-173.1(3)
C(1)-N(1)-S(1)-O(1)	50.5(4)
C(1)-N(1)-S(1)-F(1)	-61.8(4)
Na(1)-O(3)-C(1)-N(1)	116.1(6)
Na(1)-O(3)-C(1)-C(2)	-66.4(8)
S(1)-N(1)-C(1)-O(3)	-0.9(6)
S(1)-N(1)-C(1)-C(2)	-178.5(3)
O(3)-C(1)-C(2)-C(7)	20.8(6)
N(1)-C(1)-C(2)-C(7)	-161.3(4)
O(3)-C(1)-C(2)-C(3)	-159.3(4)
N(1)-C(1)-C(2)-C(3)	18.6(6)
C(7)-C(2)-C(3)-C(4)	-3.4(6)
C(1)-C(2)-C(3)-C(4)	176.7(4)
C(2)-C(3)-C(4)-C(5)	2.7(6)
C(3)-C(4)-C(5)-C(6)	-0.8(7)
C(3)-C(4)-C(5)-N(2)	-178.8(4)
O(5)-N(2)-C(5)-C(4)	-175.7(4)
O(4)-N(2)-C(5)-C(4)	4.8(6)
O(5)-N(2)-C(5)-C(6)	6.2(6)

O(4)-N(2)-C(5)-C(6)	-173.2(4)
C(4)-C(5)-C(6)-C(7)	-0.4(7)
N(2)-C(5)-C(6)-C(7)	177.6(4)
C(3)-C(2)-C(7)-C(6)	2.1(7)
C(1)-C(2)-C(7)-C(6)	-178.0(4)
C(5)-C(6)-C(7)-C(2)	-0.2(7)

Symmetry transformations used to generate equivalent atoms:
#1 x+1,y,z #2 -x+1,-y+2,-z+1 #3 -x+2,-y+2,-z+1
#4 x-1,y,z

Table 10. Hydrogen bonds for 181108c [A and deg.].

D-H	d(D-H)	d(H..A)	<DHA	d(D..A)	A
O6-H6A	0.850	2.187	161.73	3.006	O2 [x+1, y+1, z]
O6-H6B	0.850	2.353	154.45	3.141	O3 [x+1, y, z]
O7-H7A	0.850	2.101	160.82	2.917	O4 [x, y, z+1]
O7-H7A	0.850	2.637	111.27	3.050	F1 [-x+1, -y+1, -z+1]
O7-H7B	0.850	2.317	157.46	3.119	O1
O7-H7B	0.850	2.618	139.96	3.314	F1