Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

Remote C(sp^3)−H heteroarylation of *N*-fluoroarylsulfonamides *via* a silyl radical process under visible light irradiation

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Appendix I

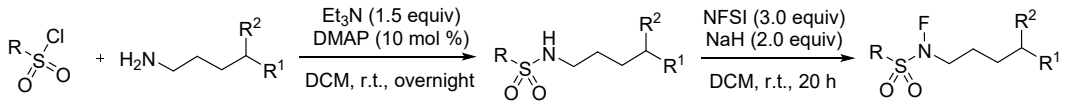
Copies of Relevant ^{1}H-, $^{13}C\{^{1}H\}$- and ^{19}F-NMR Spectra

I. General Methods and Materials.

Unless otherwise specified, proton (^{1}H) and proton-decoupled carbon [^{13}C{^{1}H}] NMR spectra were recorded at room temperature in base-filtered $CDCl_3$ on a spectrometer operating at 500 MHz or 300 MHz for proton and 126 MHz or 75 MHz for carbon nuclei. For ^{1}H NMR spectra, signals arising from the residual protio-forms of the solvent were used as the internal standards. ^{1}H NMR data are recorded as follows: chemical shift (δ) [multiplicity, coupling constant(s) *J* (Hz), relative integral] where multiplicity is defined as: s = singlet; d = doublet; t = triplet; q = quartet; m = multiplet or combinations of the above. The signal due to residual $CHCl_3$ appearing at δ_H 7.26 and the central resonance of the $CDCl_3$ "triplet" appearing at δ_C 77.0 were used to reference ^{1}H and ^{13}C{^{1}H} NMR spectra, respectively. Infrared spectra were recorded, as thin films or solids, on a Nicolet iS50 FT-IR spectrometer fitted with a Smart iTX sampling module and only major absorptions are reported (in cm^{-1}). High-resolution ESI mass spectra were recorded on a time-of-flight instrument. Melting points were measured on an automated melting point system and are uncorrected. Analytical thin layer chromatography (TLC) was performed with silica gel GF_{254} plates. Eluted plates were visualized using a 254 nm UV lamp and/or by treatment with a suitable dip followed by heating. These dips included phosphomolybdic acid: ceric sulfate: sulfuric acid (conc.): water (37.5 g : 7.5 g : 37.5 g : 720 mL) or potassium permanganate : potassium carbonate : 5% sodium hydroxide aqueous solution : water (3 g : 20 g: 5 mL : 300 mL). For column chromatography, 200-300 mesh silica gel was employed. Reagents and inorganic salts as well as dried solvents were generally available from commercial sources and used as supplied. Unless indicated otherwise, reactions were performed under a nitrogen atmosphere.

II. Procedures for the Synthesis of Substrates 1a-w and 2a-r

General procedure for the synthesis of N-F sulfonamides

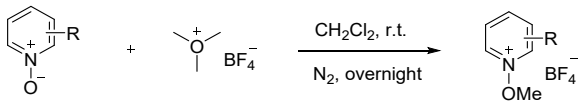


These compounds were prepared using minor modifications of a reported procedure[1].

Step i: A clean, dry round-bottomed flask was charged with a magnetic stir bar and relevant primary amine (1.0 equiv). While being maintained under a nitrogen atmosphere at ambient temperatures the amine was dissolved in DCM (0.2 M) and the resulting solution was treated with freshly distilled triethylamine (1.5 equiv), 4-(*N*,*N*-dimethylamino)pyridine (10 mol %) and the appropriate sulfonyl chloride (1.0 equiv) then added. The ensuing mixture was allowed to stir at ambient temperatures for 16 h then quenched with water (50 mL) and the separated aqueous layer was extracted with DCM (3 ⨱ 50 mL). The combined organic phases were washed with brine (1 ⨱ 50 mL) before being dried (Na_2SO_4), filtered and concentrated under reduced pressure. The residue thus obtained was purified by column chromatography (petroleum ether/ethyl acetate elution) to afford the relevant N-H sulfonamides.

Step ii: A oven-dried rounded bottom flask was charged with a solution of the relevant N-H sulfonamide (1.0 equiv) in dry DCM (0.2 M) and the resulting solution coold to 0 °C (ice-bath) then slowly treated with NaH (2.0 equiv). The ensuing mixture was allowed to warm to ambient tempertures and stirring continued for a further 0.5 h after which time it was treated, in portions, with NFSI (3.0 equiv). The resulting slurry was stirred for 20 h then quenched with NH_4Cl (50 mL of a saturated aqueous solution) and the precipitate so-formed removed by filtration. The filtrate was extracted with DCM (3 ⨱ 50 mL) and the combined organic phases was washed with water (1 ⨱ 50 mL) and brine (1 ⨱ 50 mL) before being dried (Na_2SO_4), filtered and concentrated under reduced pressure. The residue thus obtained was purified by column chromatography (petroleum ether/ethyl acetate elution) to afford the relevant N-F sulfonamides.

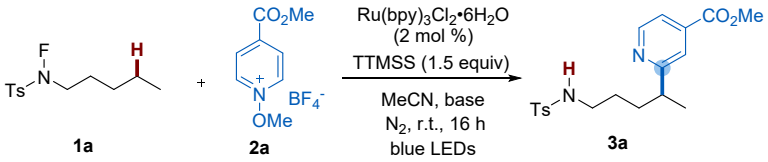
General procedure for the synthesis of *N*-heteroarenium salts



Following a protocol previously reported by our group[2], a solution of the relevant pyridine *N*-oxide (5 mmol) and trimethyloxonium tetrafluoroborate (6 mmol, 1.2 equiv.) in DCM (25 mL) maintained at ambient temperatures under nitrogen was stirred for 16 h then concentrated under reduced pressure. The solid thus obtained was recrystallized (twice) from a mixture of DCM (6 mL) and diethyl ether (60 mL) stored at –20 °C.

III. Optimization of the Reaction Conditions

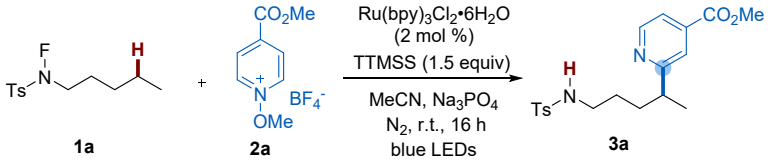
Table S1. Screening of bases.[a]



Entry	Base	Yield (%)[b]
1	—	15
2	$NaHCO_3$	37
3	NaOAc	36
4	NaO^tBu	trace
5	NEt_3	0
6	K_3PO_4	35
7	Na_2HPO_4	30
8	Na_3PO_4	40

[a] Reactions were performed using a mixture of **1a** (0.1 mmol, 1.0 equiv), **2a** (0.2 mmol, 2.0 equiv), base (0.2 mmol, 2.0 equiv) and TTMSS (0.15 mmol, 1.5 equiv) in MeCN (1.0 mL) at room temperature under irradiation with 40W Kessil blue LED (100% intensity, 456 nm) for 16 h. [b] Yields of isolated products are given.

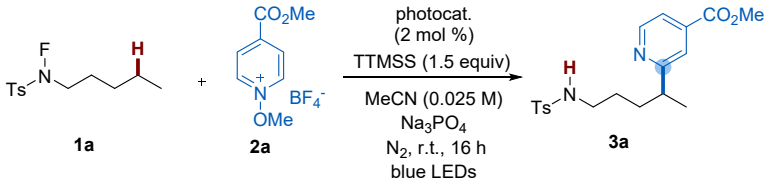
Table S2. Screening of solvent concentration.[a]



Entry	concentration	Yield (%)[b]
1	0.1 M	40
2	0.05 M	61
3	0.025 M	69

[a] Reactions were performed using a mixture of **1a** (0.1 mmol, 1.0 equiv), **2a** (0.2 mmol, 2.0 equiv), Na_3PO_4 (0.2 mmol, 2.0 equiv), and TTMSS (0.15 mmol, 1.5 equiv) in MeCN at room temperature under irradiation with 40W Kessil blue LED (100% intensity, 456 nm) for 16 h. [b] Yields of isolated products are given.

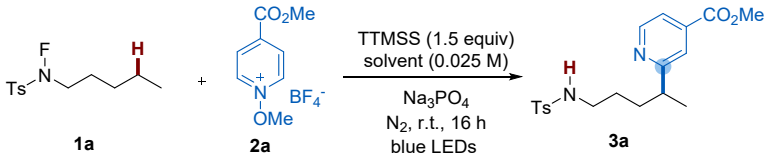
Table S3. Screening of photocatalysts.[a]



Entry	Photocatalyst	Yield (%)[b]
1	$Ru(bpy)_3Cl_2.6H_2O$	69
2	Mes-Acr$^+$	10
3	Eosin Y	12
4	4CzIPN	64
5	*fac*-Ir(ppy)$_3$	15
6	[Ir{dFCF$_3$ppy}$_2$(bpy)]PF$_6$	63
7[c]	—	68

[a] Reactions were performed using a mixture of **1a** (0.1 mmol, 1.0 equiv), **2a** (0.2 mmol, 2.0 equiv), Na_3PO_4 (0.2 mmol, 2.0 equiv), and TTMSS (0.15 mmol, 1.5 equiv) in MeCN (4.0 mL) at room temperature under irradiation with 40W Kessil blue LED (100% intensity, 456 nm) for 16 h. [b] Yields of isolated products are given. [c] Without photocatalyst.

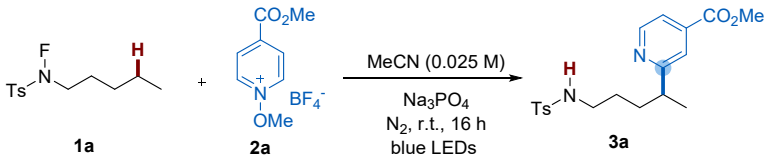
Table S4. Screening of solvents.[a]



Entry	Base	Yield (%)[b]
1	MeCN	68
2	Benzene	0
3	EA	trace
4	DCM	12
5	DCE	25
6	Toluene	trace
7	DMSO	trace

[a] Reactions were performed using a mixture of **1a** (0.1 mmol, 1.0 equiv), **2a** (0.2 mmol, 2.0 equiv), Na_3PO_4 (0.2 mmol, 2.0 equiv), and TTMSS (0.15 mmol, 1.5 equiv) in solvent (4.0 mL) at room temperature under irradiation with 40W Kessil blue LED (100% intensity, 456 nm) for 16 h. [b] Yields of isolated products are given.

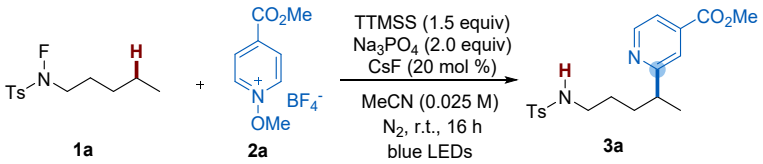
Table S5. Screening of silane sources.[a]



Entry	Silane	Yield (%)[b]
1	TTMSS	68
2	Et_3SiH	0
3	$(MeO)_3SiH$	trace
4	$Ph(Me)_2SiH$	0
5	Ph_3SiH	0

[a] Reactions were performed using a mixture of **1a** (0.1 mmol, 1.0 equiv), **2a** (0.2 mmol, 2.0 equiv), Na_3PO_4 (0.2 mmol, 2.0 equiv), and silane (0.15 mmol, 1.5 equiv) in MeCN (4.0 mL) at room temperature under irradiation with 40W Kessil blue LED (100% intensity, 456 nm) for 16 h. [b] Yields of isolated products are given.

Table S6. Reaction condition screening.[a]

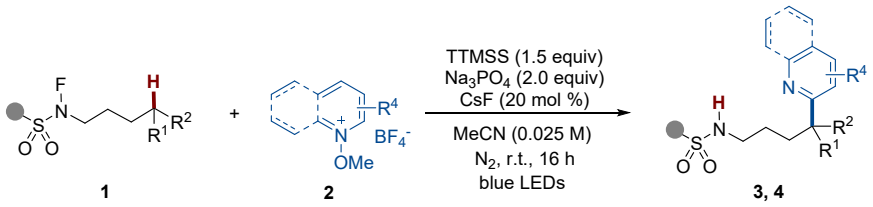


Entry	Deviation from standard conditions	Yield (%)[b]
1	none	77
2	without CsF	68
3	$NaHCO_3$ instead of Na_3PO_4	63
4	Et_3N instead of Na_3PO_4	0
5	without Na_3PO_4	20
6[c]	without Na_3PO_4	64
7	1,2-DCE instead of MeCN	27
8	DMSO instead of MeCN	trace
9	MeCN (0.1 M)	42
10	without TTMSS	0
11	Et_3SiH instead of $(TMS)_3SiH$	0
12	$(MeO)_3SiH$ instead of $(TMS)_3SiH$	trace
13	390/525 nm instead of 456 nm	68/52
14	in dark	0

[a] Reactions were performed using a mixture of **1a** (0.1 mmol, 1.0 equiv), **2a** (0.2 mmol, 2.0 equiv), Na_3PO_4 (0.2 mmol, 2.0 equiv), CsF (20 mol %), and TTMSS (0.15 mmol, 1.5 equiv) in MeCN (4.0 mL) at room temperature under irradiation with 40W Kessil blue LED (100% intensity, 456 nm) for 16 h. [b] Yields of isolated products are given. [c] With 2.0 equiv of CsF.

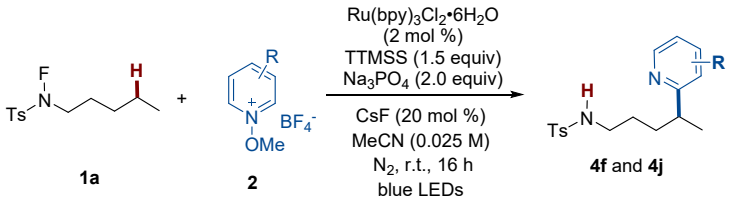
IV. Procedure for the Synthesis of Compounds 3a-z, 3aa-ap, 4a-t.

General procedure for the synthesis of compounds 3a-z, 3aa-ap, 4a-e, 4g-i and 4k-t.



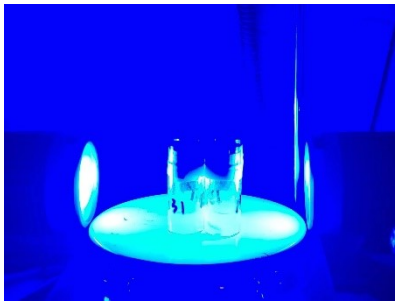
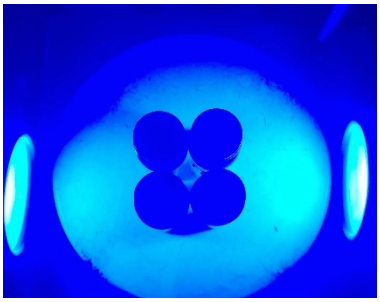
An oven-dried reaction tube equipped with a magnetic stirring bar was placed inside a glove box then charged with the relevant *N*-fluorosulfonamide **1** (0.1 mmol, 1.0 equiv), the relevant *N*-methoxyheteroarenium salt **2** (0.2 mmol, 2.0 equiv), Na_3PO_4 (0.2 mmol, 2.0 equiv), CsF (20 mol %), and tris(trimethylsilyl)silane (TTMS) (0.15 mmol, 1.5 equiv). MeCN (4.0 mL, 0.025 M) was then added to the reaction mixture *via* syringe and the ensuing mixture was taken outside the glove box and stirred magentically at ambient temperatures for 16 h while being irradiated, throughout this time, with two Kessil blue LED lamps (456 nm, 40 W, 100% intensity). Thereafter, solvent was removed from the reaction mixture under reduced pressure and the residue thus obtained was purified by column chromatography (petroleum ether/ethyl acetate elution) to afford the relevant product **3a-z**, **3aa-ap**, **4a-e**, **4g-i** and **4k-t**.

General procedure for the synthesis of compounds 4f and 4j.



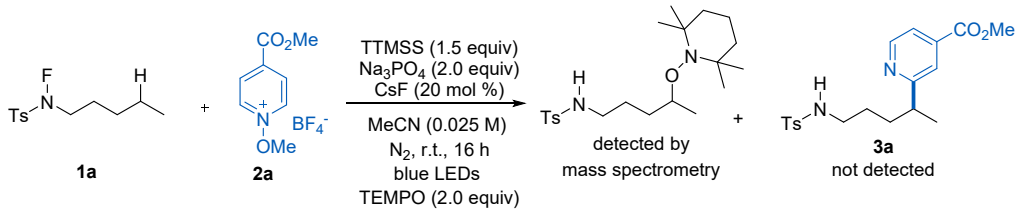
An oven-dried reaction tube equipped with a magnetic stirring bar was charged with the relevant *N*-fluorosulfonamide **1a** (0.1 mmol, 1.0 equiv), the relevant *N*-methoxyheteroarenium salt **2** (0.2 mmol, 2.0 equiv), Na_3PO_4 (0.2 mmol, 2.0 equiv), CsF (20 mol %), tris(trimethylsilyl)silane (TTMS) (0.15 mmol, 1.5 equiv), and $Ru(bpy)_3Cl_2•6H_2O$ (2 mol %) inside a glove box. MeCN (4.0 mL, 0.025 M) was then added to the reaction mixture *via* syringe. The reaction tube was taken outside the glove box and the resulting solution was stirred at room temperature for 16 hours while being irradiated, throughout this time, with two Kessil blue LED lamps (456 nm, 40 W, 100% intensity). After 16 hours, the entire solvent was evaporated under reduced pressure, and the residue thus obtained was purified by column

chromatography (petroleum ether/ethyl acetate elution) to afford the relevant product **4f** and **4j**.


V. Control Experiments

Radical Trapping Experiment Using TEMPO



An oven-dried reaction tube kept inside a glove box equipped with a magnetic stirring bar was charged with the relevant *N*-fluorosulfonamide **1a** (0.1 mmol, 1.0 equiv), the relevant *N*-methoxyheteroarenium salt **2a** (0.2 mmol, 2.0 equiv), Na_3PO_4 (0.2 mmol, 2.0 equiv), CsF (20 mol %), tris(trimethylsilyl)silane (TTMS) (0.15 mmol, 1.5 equiv) and TEMPO (31.2 mg, 0.2 mmol). MeCN (4.0 mL, 0.025 M) was then added, *via* syringe, to the reaction mixture and the reaction tube was removed from the glove box and the resulting solution was stirred at ambient temperatures for 16 h while being irradiated, throughout this time, with two Kessil blue LED lamps (456 nm, 40 W, 100% intensity). Thereafter the reaction mixture was treated with water (30 mL) and then extracted with ethyl acetate (3 ✕ 25 mL). The combined organic phases was washed with water (1 ✕ 30 mL) and brine (1 ✕ 30 mL) before being dried (Na_2SO_4), filtered and concentrated under reduced pressure. The residue thus obtained was subjected to ESI mass spectral analysis and the derived spectrum (shown immediately below) displayed a molecular-associated ion at *m/z* 397.2 consistent with the presence of the anticipated TEMPO trapping product. No evidence for the formation of pyridine **3a** was obtained.

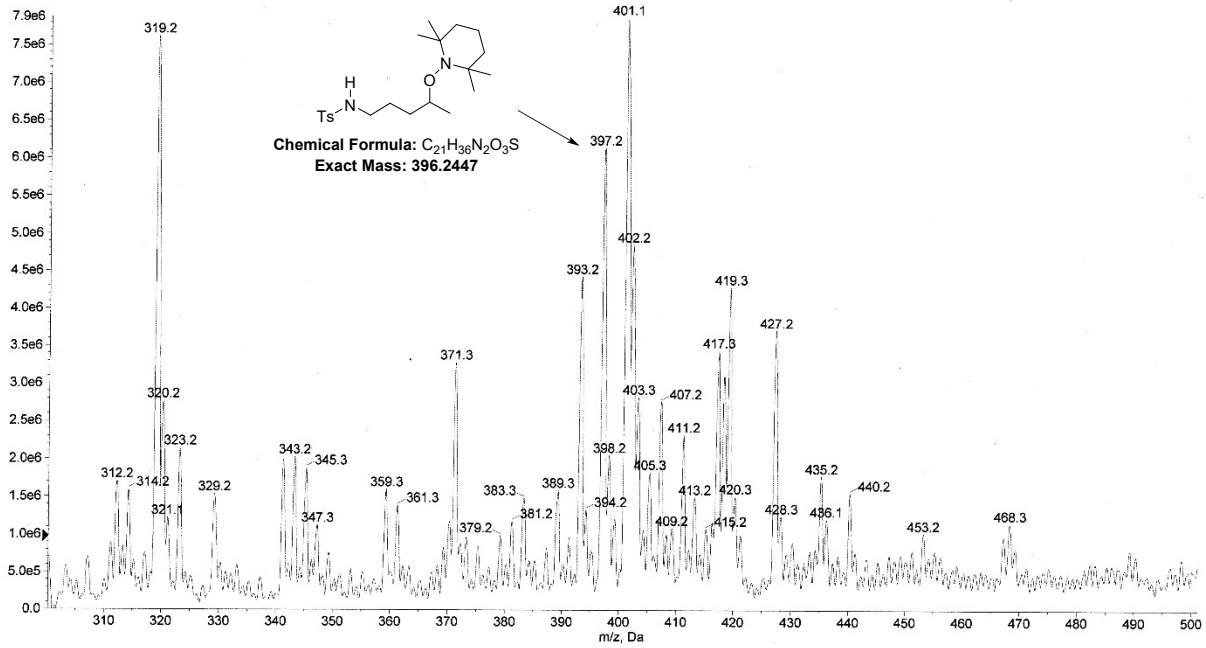
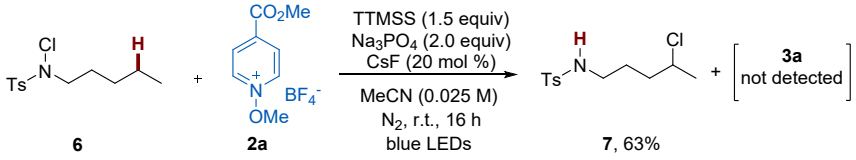


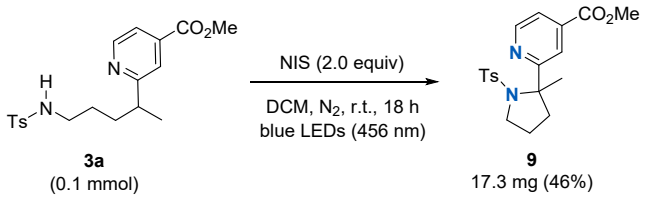
Figure S1. ESI Mass spectrum arising from TEMPO-trapping experiment.

Remote C(sp^3)−H chlorination of sulfonamide



An oven-dried reaction tube contained in a glove box was charged with a magnetic stirring bar, *N*-chlorosulfonamide **6** (0.1 mmol, 1.0 equiv), *N*-methoxyheteroarenium salt **2a** (0.2 mmol, 2.0 equiv), Na_3PO_4 (0.2 mmol, 2.0 equiv), CsF (20 mol %), and tris(trimethylsilyl)silane (TTMS) (0.15 mmol, 1.5 equiv). MeCN (4.0 mL, 0.025 M) was then added, *via* syringe, to the reaction mixture. The reaction tube was then removed from the glove box and the solution contained therein was stirred at room temperature for 16 h while being irradiated, throughout this time, with two Kessil blue LED lamps (456 nm, 40 W, 100% intensity). Thereafter, the reaction mixture was concentrated under reduced pressure and the residue thus obtained was purified by column chromatography on silica gel (4:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.3 in 4:1 v/v petroleum ether/ethyl acetate) to afford compound **7** as a clear, colorless oil (17.5 mg, 63% yield).

Conversion of compound 3a into cyclic derivative 9



An oven-dried reaction tube contained in a glove box and equipped with a magnetic stirring bar was charged with the pyrinidylation product **3a** (0.1 mmol, 1.0 equiv) and *N*-iodosuccinimide (NIS, 0.2 mmol, 2.0 equiv). DCM (2.0 mL, 0.05 M) was then added to the reaction tube *via* syringe then the tube was removed from the glove box and the solution was stirred at ambient temperatures for 18 h while being irradiated, throughout this time, with two Kessil blue LED lamps (456 nm, 10 W, 25% intensity). Thereafter, the reaction mixture was concentrated under reduced pressure and the residue thus obtained was purified by column chromatography on silica gel (3:1 v/v petroleum ether/ethyl acetate elution, $R_f = 0.5$ in 2:1 v/v petroleum ether/ethyl acetate) to afford the unsymmetrically linked *bis*-heterocycle **9** (17.3 mg, 46% yield) as a clear, colorless oil.

VI. Absorption Spectra and Stern–Volmer Quenching of 2a

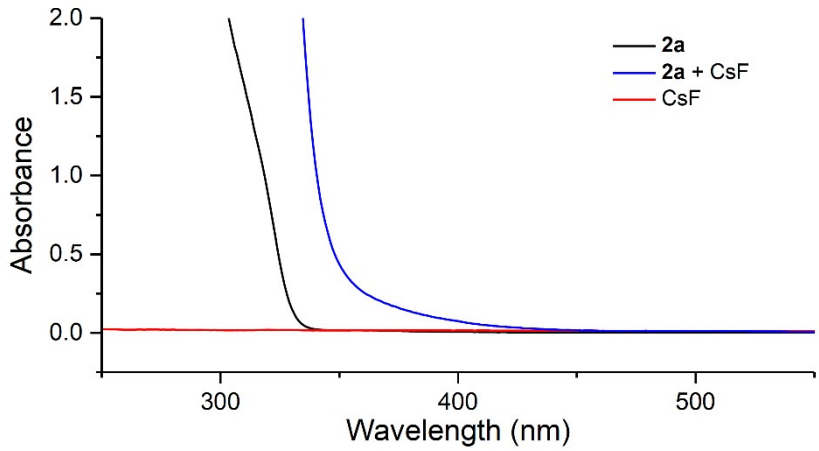


Figure S2. Asorption spectra for compound **2a** in the presence and absence of caesium fluoride (based on 0.025 M of **2a** in MeCN).

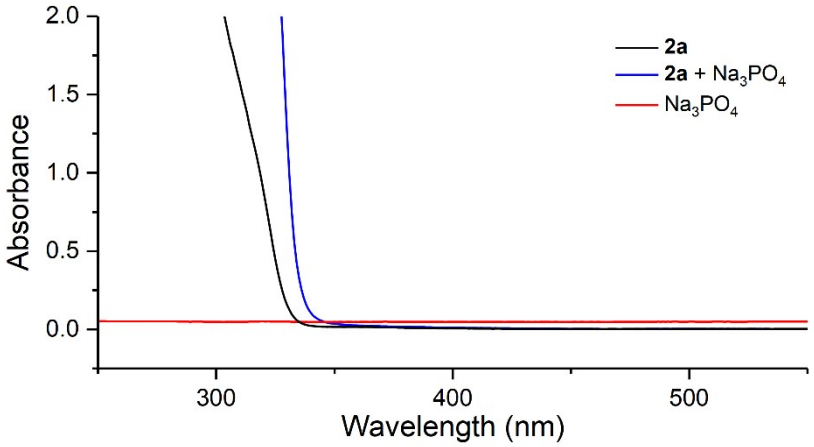


Figure S3. Asorption spectra for compound **2a** in the presence and absence of trisodium phosphate (based on 0.025 M of **2a** in MeCN).

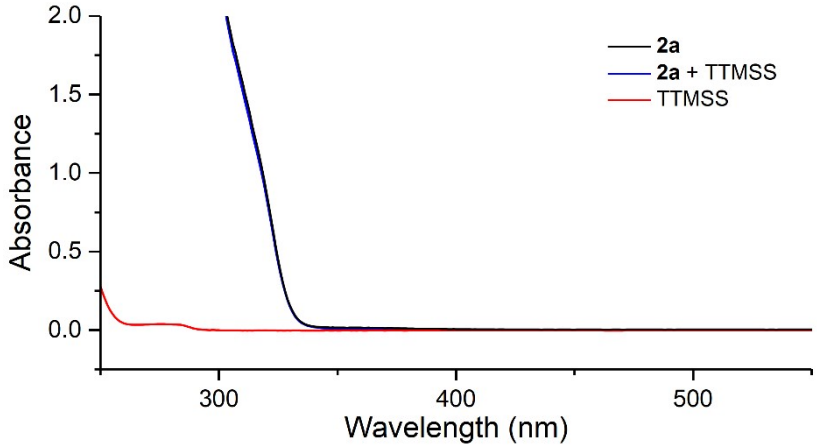


Figure S4. Asorption spectra for **2a** with tris(trimethylsilyl)silane (based on 0.025 M of **2a** in MeCN).

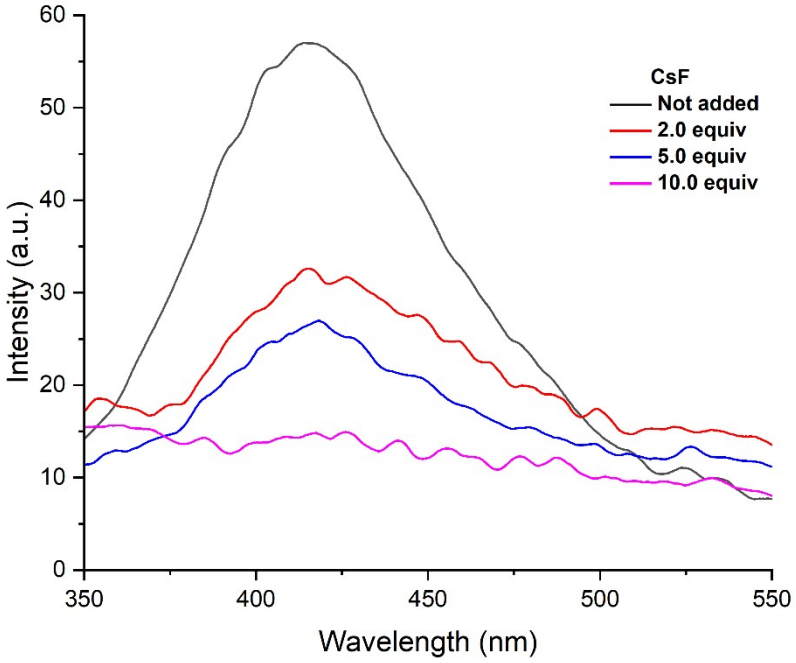


Figure S5. Quenching of the *N*-methoxypyridylinium salt **2a** emission (0.005 M in Me CN) in the presence of increasing amounts of CsF. Excitation wavelength : 290 nm, Bandwidth : Ex 15 nm, Em 20 nm.

VII. Quantum Yield Measurements

Determination of the light intensity at 456 nm.

A Kessil LED lamp (λ_{max} = 456 nm) was used at 100% intensity for the measurement of quantum yield. So, following the procedure of Yoon,[3] the photon flux of the LED (λ_{max} = 456 nm) was determined by standard ferrioxalate actinometry. Specifically, a 0.15 M solution of ferrioxalate was prepared by dissolving potassium ferrioxalate hydrate (0.737 g) in H_2SO_4 (10 mL of a 0.05 M solution) while a buffered solution of 1,10-phenanthroline was prepared by dissolving 1,10-phenanthroline (5.0 mg) and sodium acetate (1.13 g) in H_2SO_4 (5.0 mL of a 0.5 M solution). Both solutions were stored in the dark. To determine the photon flux of the LED, the ferrioxalate solution (2.0 mL) was placed in a cuvette and irradiated for 90 seconds at λ_{max} = 456 nm. After irradiation, the phenanthroline solution (0.35 mL) was added to the cuvette and the resulting mixture was allowed to stir in the dark for 1 h so as to permit the ferrous ions to completely coordinate to the phenanthroline. The absorbance of the resulting solution was measured at 510 nm. A non-irradiated sample was also prepared and the absorbance at 510 nm was measured. Conversion was calculated using eq 1

	Non-irrad	Irrad 01	Irrad 02	Irrad 03
A_{510nm}	0.552	1.086	1.164	1.121
		Average $A_{510\,nm}$ of irradiation samples		1.124

$$mol\ of\ Fe^{2+} = \frac{V \cdot \Delta A_{510\,nm}}{l \cdot \epsilon} = \frac{(0.00235\ L) \cdot (0.572)}{(1.00\ cm) \cdot (11{,}100 \frac{L}{mol} \cdot cm)} = 1.21 \times 10^{-7}\ mol \quad (1)$$

V is the total volume (0.00235 L) of the solution after addition of phenanthroline, ΔA is the difference in absorbance at 510 nm between the irradiated and non-irradiated solutions, l is the path length (1.00 cm), and ε is the molar absorptivity of the ferrioxalate actinometer at 510 nm (11,100 $Lmol^{-1}\ cm^{-1}$).[4] The photon flux was calculated using eq 2:

$$Photon\ flux = \frac{mol\ of\ Fe^{2+}}{\emptyset \cdot t \cdot f} = \frac{1.21 \times 10^{-7} mol}{(0.84) \cdot (90\ s) \cdot (0.945)} = 1.69 \times 10^{-9}\ einstein/s \quad (2)$$

where Φ is the quantum yield for the ferrioxalate actinometer (0.84 at λ = 456 nm),[5] t is the irradiation time (90 s), and f is the fraction of light absorbed at 456 nm by the ferrioxalate actinometer. This value is calculated using eq 3 where $A_{456\ nm}$ is the absorbance of the ferrioxalate solution at 456 nm. An absorption spectrum gave an $A_{456\ nm}$ value of 1.260, indicating that the fraction of absorbed light (f) is 0.945.

$$f = 1 - 10^{-A_{456\,nm}} \quad (3)$$

The photon flux was thus calculated to be 1.69×10^{-9} Einsteins s^{-1} (average of three experiments).

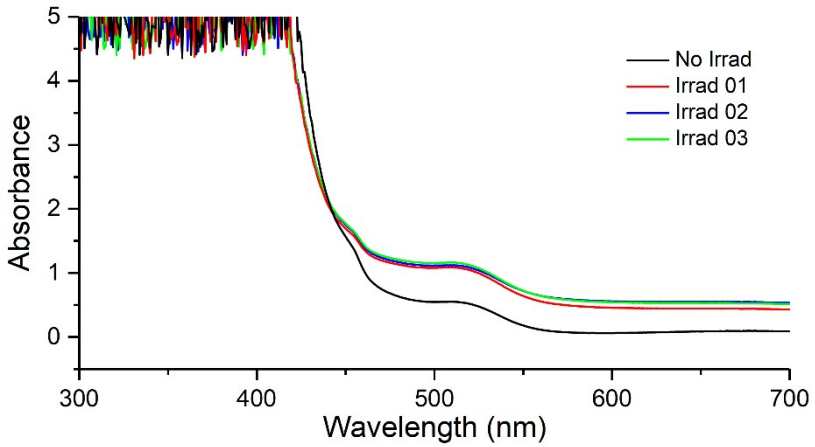


Figure S6. Absorption spectra derived from three irradiation experiments and one non-irradiation experiment.

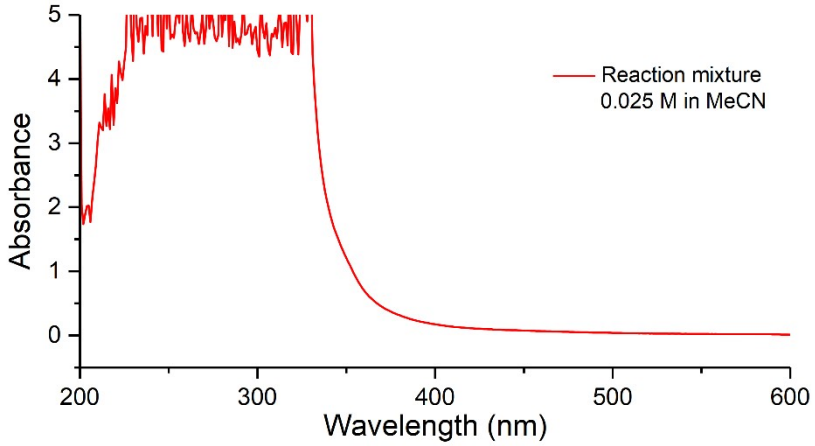
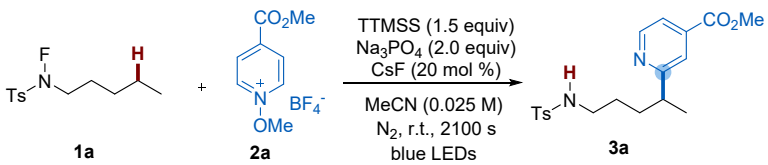


Figure S7. Absorption spectrum of a 0.025 M solution of the reaction mixture in MeCN.

Determination of the reaction quantum yield.



An oven-dried reaction tube contained in a glove box and equipped with a magnetic stirring bar was charged with *N*-fluorosulfonamide **1a** (0.1 mmol, 1.0 equiv), *N*-methoxyheteroarenium

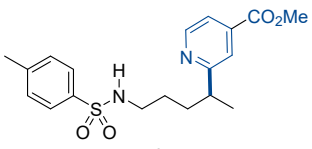
salt **2a** (0.2 mmol, 2.0 equiv), Na_3PO_4 (0.2 mmol, 2.0 equiv), CsF (20 mol %), and tris(trimethylsilyl)silane (TTMS) (0.15 mmol, 1.5 equiv). MeCN (4.0 mL, 0.025 M) was then added, *via* syringe, to the reaction mixture. The reaction tube was then removed from the glove box and reaction mixture was stirred at ambient temperatures for 2100 s while being irradiated, throughout this time, with two Kessil blue LED lamps (456 nm, 40 W, 100% intensity). After this time the reaction mixture was concentrated under reduced pressure and the residue thus obtained was purified by column chromatography on silica gel (2:1 v/v petroleum ether/ethyl acetate elution) gave compound **3a** (7.7 mg, 20%) (2.05×10^{-5} mol of **3a**). The reaction quantum yield (Φ) was determined using eq 4 where the photon flux is 1.69×10^{-9} Einsteins s^{-1} (determined by actinometry as described above), t is the reaction time (2100 s) and f is the fraction of incident light absorbed by the catalyst, determined using eq 3. An absorption spectrum of the reaction (0.025 M) gave an absorbance value of 0.072 at 456 nm (Figure S7), indicating that the fraction of light absorbed by the reaction (f) is 0.153.

$$\Phi = \frac{mol\ of\ product}{flux \cdot t \cdot f} \qquad (4)$$

$$\Phi = \frac{2.05 \times 10^{-5}\ mol}{1.69 \times 10^{-9}\ einstein\ s^{-1} \cdot 2100\ s \cdot 0.153} = 37.8$$

The reaction quantum yield (Φ) was calculated to be 37.8

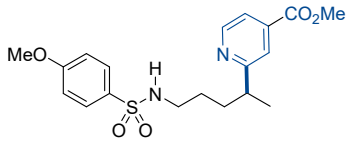
VIII. Compound Characterization and Related Data



3a

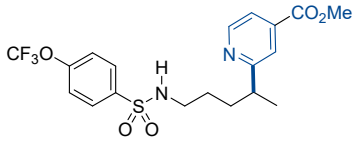
Methyl-2-(5-((4-methylphenyl)sulfonamido)pentan-2-yl)isonicotinate (3a). Reaction of *N*-fluoro-4-methyl-*N*-pentylbenzenesulfonamide **1a** (25.9 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (2:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.3 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **3a** (28.9 mg, 77%) as a light-yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.66 (d, *J* = 5.1 Hz, 1H), 7.71 (d, *J* = 8.3 Hz, 2H),

7.66 – 7.65 (m, 2H), 7.27 (d, J = 8.1 Hz, 2H), 4.91 (s, 1H), 3.95 (s, 3H), 2.89 (dd, J = 6.4, 2.8 Hz, 3H), 2.41 (s, 3H), 1.76 – 1.71 (m, 1H), 1.61 – 1.56 (m, 1H), 1.46 – 1.41 (m, 1H), 1.34 – 1.30 (m, 1H), 1.24 (d, J = 6.9 Hz, 3H); $^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$) δ 166.9, 165.8, 149.8, 143.2, 137.9, 136.9, 129.6, 127.0, 120.9, 120.5, 52.7, 43.1, 41.3, 33.6, 27.4, 21.4, 20.7; IR (ATR) ν_{max} 2924, 1731, 1438, 1276, 1158, 751, 551 cm^{-1}; HRMS (ESI) Calcd for $C_{19}H_{24}N_2O_4S$: $[M+H]^+$ = 377.1530. Found: 377.1512.



3b

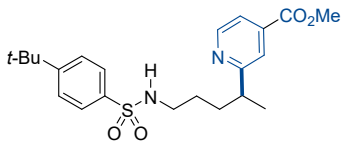
Methyl-2-(5-((4-methoxyphenyl)sulfonamido)pentan-2-yl)isonicotinate (3b). Reaction of *N*-fluoro-4-methoxy-*N*-pentylbenzenesulfonamide **1b** (27.5 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (2:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.2 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **3b** (30.1 mg, 77%) as a light-yellow oil. ^1H NMR (500 MHz, $CDCl_3$) δ 8.66 (d, J = 6.0 Hz, 1H), 7.76 (d, J = 8.9 Hz, 2H), 7.66 – 7.65(m, 2H), 6.95 (d, J = 8.9 Hz, 2H), 4.74 (t, J = 6.1 Hz, 1H), 3.96 (s, 3H), 3.86 (s, 3H), 2.91 – 2.87 (m, 3H), 1.74 – 1.72 (m, 1H), 1.62 – 1.58 (m, 1H), 1.46 – 1.41 (m, 1H), 1.32 – 1.29 (m, 1H), 1.26 (d, J = 6.9 Hz, 3H); $^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$) δ 166.8, 165.8, 162.8, 149.9, 137.8, 131.5, 129.1, 120.9, 120.5, 114.2, 55.6, 52.7, 43.1, 41.3, 33.6, 27.4, 20.8; IR (ATR) ν_{max} 2921, 2851, 1730, 1597, 1298, 1154, 834, 561 cm^{-1}; HRMS (ESI) Calcd for $C_{19}H_{24}N_2O_5S$: $[M+H]^+$ = 393.1479. Found: 393.1488.



3c

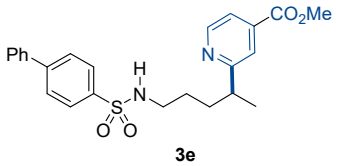
Methyl-2-(5-((4-(trifluoromethoxy)phenyl)sulfonamido)pentan-2-yl)isonicotinate (3c). Reaction of *N*-fluoro-*N*-pentyl-4-(trifluoromethoxy)benzenesulfonamide **1c** (32.9 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (3:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.5 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **3c** (31.6 mg, 71%) as a light-yellow oil. ^1H NMR (500 MHz, $CDCl_3$) δ 8.69 (s, 1H), 7.88 (d, J = 8.8 Hz, 2H), 7.69 – 7.68 (m, 2H), 7.30 (d, J = 8.4 Hz, 2H), 5.11 (s, 1H), 3.96 (s, 3H), 2.97 – 2.92 (m,

3H), 1.78 – 1.73 (m, 1H), 1.63 – 1.57 (m, 1H), 1.50 – 1.45 (m, 1H), 1.36 – 1.32 (m, 1H), 1.25 (d, J = 6.9 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$) δ 166.7, 165.8, 152.0, 149.9, 138.5, 137.9, 129.13, 121.0, 120.9, 120.6, 120.2 (q, J = 259.6 Hz), 52.7, 43.1, 41.2, 33.6, 27.4, 20.8; ^{19}F NMR (471 MHz, $CDCl_3$) δ -57.7; IR (ATR) ν_{max} 2930, 1731, 1600, 1438, 1253, 1155, 763, 601 cm^{-1}; HRMS (ESI) Calcd for $C_{19}H_{21}F_3N_2O_5S$: $[M+H]^+$ = 447.1196. Found: 447.1198.



3d

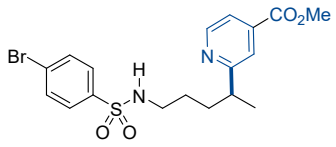
Methyl-2-(5-((4-(tert-butyl)phenyl)sulfonamido)pentan-2-yl)isonicotinate (3d). Reaction of 4-(*tert*-butyl)-*N*-fluoro-*N*-pentylbenzenesulfonamide **1d** (30.1 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (3:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.5 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **3d** (30.9 mg, 74%) as a light-yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.64 (d, J = 4.8 Hz, 1H), 7.74 (d, J = 8.6 Hz, 2H), 7.64 – 7.63 (m, 2H), 7.48 (d, J = 8.6 Hz, 2H), 4.87 (t, J = 6.1 Hz, 1H), 3.94 (s, 3H), 2.96 – 2.87 (m, 3H), 1.76 – 1.72 (m, 1H), 1.61 – 1.58 (m, 1H), 1.48 – 1.44 (m, 1H), 1.34 – 1.31 (m, 10H), 1.23 (d, J = 6.9 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$) δ 166.9, 165.8, 156.2, 149.9, 137.8, 136.9, 126.8, 126.0, 120.9, 120.5, 52.6, 43.1, 41.3, 35.1, 33.6, 31.0, 27.4, 20.8; IR (ATR) ν_{max} 2959, 2869, 1732, 1598, 1292, 1088, 763, 628 cm^{-1}; HRMS (ESI) Calcd for $C_{22}H_{30}N_2O_4S$: $[M+H]^+$ = 419.1999. Found: 419.2000.



3e

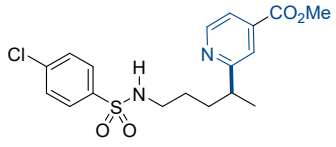
Methyl-2-(5-([1,1'-biphenyl]-4-sulfonamido)pentan-2-yl)isonicotinate (3e). Reaction of *N*-fluoro-*N*-pentyl-[1,1'-biphenyl]-4-sulfonamide **1e** (32.1 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (2:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.4 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **3e** (39.0 mg, 89%) as a light-yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.65 (s, 1H), 7.89 (d, J = 8.5 Hz, 2H), 7.69 – 7.66 (m, 3H), 7.63 (d, J = 5.1 Hz, 1H), 7.58 (d, J = 7.2 Hz, 2H), 7.47 (t, J = 7.5 Hz, 2H), 7.41 (t, J = 7.3 Hz, 1H), 5.04 (t, J = 6.2 Hz, 1H), 3.93 (s, 3H), 2.99 – 2.94 (m, 3H), 1.79 – 1.74 (m, 1H),

1.63 – 1.59 (m, 1H), 1.51 – 1.46 (m, 1H), 1.37 – 1.32 (m, 1H), 1.25 (d, J = 6.9 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$) δ 166.8, 165.7, 149.8, 145.4, 139.3, 138.5, 137.9, 129.0, 128.4, 127.6, 127.5, 127.3, 120.9, 120.6, 52.7, 43.1, 41.2, 33.6, 27.4, 20.8; IR (ATR) ν_{max} 2922, 2852, 1730, 1597, 1294, 1158, 763, 293 cm^{-1}; HRMS (ESI) Calcd for $C_{24}H_{26}N_2O_4S$: $[M+H]^+$ = 439.1686. Found: 439.1692.



3f

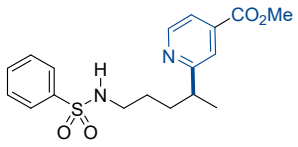
Methyl-2-(5-((4-bromophenyl)sulfonamido)pentan-2-yl)isonicotinate (3f). Reaction of 4-bromo-*N*-fluoro-*N*-pentylbenzenesulfonamide **1f** (32.3 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (3:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.2 in 5:1 v/v petroleum ether/ethyl acetate) gave compound **3f** (32.5 mg, 74%) as a light-yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.64 (dd, J = 4.8, 1.4 Hz, 1H), 7.68 (d, J = 8.6 Hz, 2H), 7.65 (d, J = 4.8 Hz, 2H), 7.60 (d, J = 8.6 Hz, 2H), 5.15 (t, J = 6.0 Hz, 1H), 3.95 (s, 3H), 2.93 – 2.89 (m, 3H), 1.75 – 1.72 (m, 1H), 1.61 – 1.56 (m, 1H), 1.46 – 1.42 (m, 1H), 1.33 – 1.30 (m, 1H), 1.24 (d, J = 6.9 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$) δ 166.7, 165.8, 149.8, 139.1, 137.9, 132.3, 128.5, 127.4, 120.9, 120.6, 52.7, 43.1, 41.2, 33.6, 27.3, 20.8; IR (ATR) ν_{max} 2925, 1729, 1574, 1436, 1292, 1158, 737, 603 cm^{-1}; HRMS (ESI) Calcd for $C_{18}H_{21}BrN_2O_4S$: $[M+H]^+$ = 441.0478. Found: 441.0484.



3g

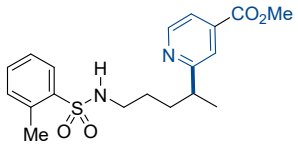
Methyl-2-(5-((4-chlorophenyl)sulfonamido)pentan-2-yl)isonicotinate (3g). Reaction of 4-chloro-*N*-fluoro-*N*-pentylbenzenesulfonamide **1g** (27.9 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (3:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.2 in 3:1 v/v petroleum ether/ethyl acetate) gave compound **3g** (31.2 mg, 79%) as a light-yellow oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.65 (d, J = 5.0 Hz, 1H), 7.75 (d, J = 8.6 Hz, 2H), 7.65 (d, J = 5.3 Hz, 2H), 7.44 (d, J = 8.6 Hz, 2H), 5.07 (t, J = 6.0 Hz, 1H), 3.95 (s, 3H), 2.92 – 2.91 (m, 3H), 1.75 – 1.72 (m, 1H), 1.61 – 1.57 (m, 1H), 1.46 – 1.42 (m, 1H), 1.32 – 1.30 (m,

1H), 1.25 (d, J = 6.9 Hz, 3H); $^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) δ 166.7, 165.8, 149.9, 138.9, 138.6, 137.9, 129.3, 128.5, 120.9, 120.6, 52.7, 43.1, 41.2, 33.6, 27.3, 20.8; IR (ATR) ν_{max} 2926, 1729, 1562, 1436, 1292, 1158, 753, 619 cm^{-1}; HRMS (ESI) Calcd for $C_{18}H_{21}ClN_2O_4S$: $[M+H]^+$ = 397.0983. Found: 397.0971.



3h

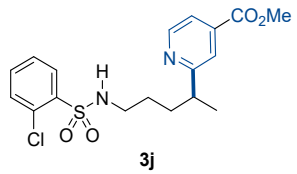
Methyl-2-(5-(phenylsulfonamido)pentan-2-yl)isonicotinate (3h). Reaction of *N*-fluoro-*N*-pentylbenzenesulfonamide **1h** (24.5 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (2:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.3 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **3h** (30.5 mg, 84%) as a light-yellow oil. ^1H NMR (600 MHz, $CDCl_3$) δ 8.66 (d, J = 5.0 Hz, 1H), 7.82 (d, J = 7.1 Hz, 2H), 7.66 – 7.65 (m, 2H), 7.54 (t, J = 7.4 Hz, 1H), 7.47 (t, J = 7.6 Hz, 2H), 5.00 (t, J = 5.9 Hz, 1H), 3.95 (s, 3H), 2.92 – 2.90 (m, 3H), 1.75 – 1.71 (m, 1H), 1.60 – 1.56 (m, 1H), 1.46 – 1.43 (m, 1H), 1.32 – 1.29 (m, 1H), 1.23 (d, J = 6.9 Hz, 3H); $^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) δ 166.9, 165.7, 149.8, 139.9, 138.0, 132.5, 129.0, 127.0, 121.0, 120.6, 52.7, 43.1, 41.2, 33.6, 27.4, 20.7; IR (ATR) ν_{max} 2925, 2854, 1729, 1561, 1291, 1157, 757, 585 cm^{-1}; HRMS (ESI) Calcd for $C_{18}H_{22}N_2O_4S$: $[M+H]^+$ = 363.1373. Found: 363.1371.



3i

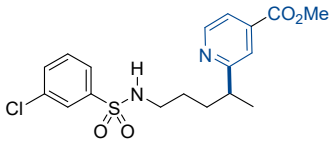
Methyl-2-(5-((2-methylphenyl)sulfonamido)pentan-2-yl)isonicotinate (3i). Reaction of *N*-fluoro-2-methyl-*N*-pentylbenzenesulfonamide **1i** (25.9 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (2:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.4 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **3i** (24.8 mg, 66%) as a light-yellow oil. ^1H NMR (500 MHz, $CDCl_3$) δ 8.61 (d, J = 4.9 Hz, 1H), 7.89 (d, J = 9.3 Hz, 1H), 7.62 – 7.60 (m, 2H), 7.39 (t, J = 8.0 Hz, 1H), 7.24 (d, J = 7.5 Hz, 2H), 4.89 (t, J = 5.9 Hz, 1H), 3.92 (s, 3H), 2.86 (q, J = 6.7 Hz, 3H), 2.58 (s, 3H), 1.71 – 1.65 (m, 1H), 1.56 – 1.51 (m, 1H), 1.41 – 1.38 (m, 1H), 1.29 – 1.25 (m, 1H), 1.20 (d, J = 7.0 Hz, 3H); $^{13}C\{^1H\}$ NMR (126 MHz,

$CDCl_3$) δ 166.8, 165.8, 149.9, 137.9, 137.8, 136.9, 132.6, 132.5, 129.4, 126.1, 120.90, 120.5, 52.7, 42.9, 41.2, 33.6, 27.5, 20.8, 20.2; IR (ATR) ν_{max} 2923, 2853, 1731, 1562, 1438, 1294, 1158, 595 cm^{-1}; HRMS (ESI) Calcd for $C_{19}H_{24}N_2O_4S$: $[M+H]^+$ = 377.1530. Found: 377.1539.



3j

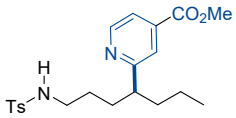
Methyl-2-(5-((2-chlorophenyl)sulfonamido)pentan-2-yl)isonicotinate (3j). Reaction of 2-chloro-*N*-fluoro-*N*-pentylbenzenesulfonamide **1j** (27.9 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (2:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.3 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **3j** (28.2 mg, 71%) as a light-yellow oil. ^1H NMR (500 MHz, $CDCl_3$) δ 8.64 (d, *J* = 5.0 Hz, 1H), 8.05 (d, *J* = 9.1 Hz, 1H), 7.64 – 7.63 (m, 2H), 7.48 – 7.47 (m, 2H), 7.40 – 7.37 (m, 1H), 5.26 (t, *J* = 6.2 Hz, 1H), 3.94 (s, 3H), 2.88 (q, *J* = 6.8 Hz, 3H), 1.76 – 1.71 (m, 1H), 1.63 – 1.57 (m, 1H), 1.46 – 1.40 (m, 1H), 1.32 – 1.29 (m, 1H), 1.24 (d, *J* = 6.9 Hz, 3H); $^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$) δ 166.7, 165.8, 149.9, 137.8, 137.2, 133.6, 131.5, 131.3, 131.2, 127.2, 120.8, 120.5, 52.6, 43.2, 41.3, 33.6, 27.4, 20.7; IR (ATR) ν_{max} 2926, 1727, 1562, 1453, 1291, 1159, 761, 586 cm^{-1}; HRMS (ESI) Calcd for $C_{18}H_{21}ClN_2O_4S$: $[M+H]^+$ = 397.0983. Found: 397.0971.



3k

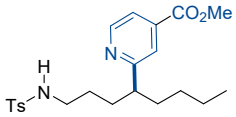
Methyl-2-(5-((3-chlorophenyl)sulfonamido)pentan-2-yl)isonicotinate (3k). Reaction of 3-chloro-*N*-fluoro-*N*-pentylbenzenesulfonamide **1k** (27.9 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (2:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.3 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **3k** (30.5 mg, 77%) as a light-yellow oil. ^1H NMR (500 MHz, $CDCl_3$) δ 8.66 (d, *J* = 5.9 Hz, 1H), 7.82 (t, *J* = 1.9 Hz, 1H), 7.72 (d, *J* = 7.8 Hz, 1H), 7.68 – 7.66 (m, 2H), 7.52 (d, *J* = 11.1 Hz, 1H), 7.43 (t, *J* = 7.9 Hz, 1H), 5.14 (t, *J* = 6.0 Hz, 1H), 3.96 (s, 3H), 2.97 – 2.92 (m, 3H), 1.78 – 1.73 (m, 1H), 1.63 – 1.59 (m, 1H), 1.50 – 1.45 (m, 1H), 1.35 – 1.31 (m, 1H), 1.27 (d, *J* = 6.9 Hz, 3H); $^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$) δ 166.7, 165.8, 149.8, 141.8, 138.0, 135.2, 132.6, 130.3, 127.1, 125.1,

121.0, 120.6, 52.7, 43.2, 41.2, 33.6, 27.4, 20.8; IR (ATR) ν_{max} 2922, 2852, 1716, 1631, 1457, 1259, 1013, 789 cm^{-1}; HRMS (ESI) Calcd for $C_{18}H_{21}ClN_2O_4S$: $[M+H]^+$ = 397.0983. Found: 397.0979.



3l

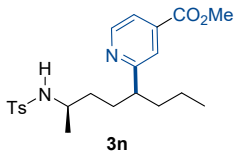
Methyl-2-(1-((4-methylphenyl)sulfonamido)heptan-4-yl)isonicotinate (3l). Reaction of *N*-fluoro-*N*-heptyl-4-methylbenzenesulfonamide **1l** (28.7 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (2:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.3 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **3l** (31.6 mg, 78%) as a light-yellow oil. ^1H NMR (500 MHz, $CDCl_3$) δ 8.59 (d, *J* = 5.0 Hz, 1H), 7.63 (d, *J* = 8.2 Hz, 2H), 7.58 (d, *J* = 5.0 Hz, 1H), 7.54 (s, 1H), 7.20 (d, *J* = 7.2 Hz, 2H), 4.79 (t, *J* = 5.9 Hz, 1H), 3.89 (s, 3H), 2.81 – 2.77 (m, 2H), 2.70 – 2.67 (m, 1H), 2.34 (s, 3H), 1.62 – 1.58 (m, 3H), 1.51 – 1.48 (m, 1H), 1.31 – 1.28 (m, 1H), 1.13 – 1.01 (m, 2H), 1.00 – 0.97 (m, 1H), 0.75 (t, *J* = 7.3 Hz, 3H); $^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$) δ 165.8, 165.7, 149.8, 143.2, 137.7, 136.9, 129.6, 127.0, 121.8, 120.5, 52.7, 47.0, 43.1, 37.8, 32.2, 27.4, 21.4, 20.5, 14.0; IR (ATR) ν_{max} 2926, 2857, 1731, 1438, 1290, 1158, 763, 551 cm^{-1}; HRMS (ESI) Calcd for $C_{21}H_{28}N_2O_4S$: $[M+H]^+$ = 405.1843. Found: 405.1834.



3m

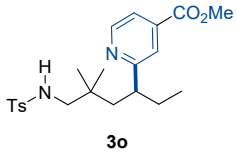
Methyl-2-(1-((4-methylphenyl)sulfonamido)octan-4-yl)isonicotinate (3m). Reaction of *N*-fluoro-4-methyl-*N*-octylbenzenesulfonamide **1m** (30.1 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (2:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.4 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **3m** (31.8 mg, 76%) as a light-yellow oil. ^1H NMR (500 MHz, $CDCl_3$) δ 8.65 (d, *J* = 5.0 Hz, 1H), 7.69 (d, *J* = 8.2 Hz, 2H), 7.64 (d, *J* = 5.1 Hz, 1H), 7.59 (s, 1H), 7.26 (d, *J* = 8.0 Hz, 2H), 4.92 (t, *J* = 6.0 Hz, 1H), 3.95 (s, 3H), 2.85 (q, *J* = 6.7 Hz, 2H), 2.73 – 2.69 (m, 1H), 2.40 (s, 3H), 1.67 – 1.64 (m, 3H), 1.58 – 1.56 (m, 1H), 1.37 – 1.34 (m, 1H), 1.23 – 1.18 (m, 3H), 1.12 – 1.10 (m, 1H), 0.98 – 0.96 (m,

1H), 0.79 (t, J = 7.3 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$) δ 165.9, 165.8, 149.9, 143.2, 137.6, 136.9, 129.6, 127.0, 121.8, 120.4, 52.6, 47.3, 43.1, 35.4, 32.3, 29.5, 27.4, 22.6, 21.4, 13.9; IR (ATR) ν_{max} 2924, 2854, 1732, 1438, 1290, 1158, 763, 561 cm^{-1}; HRMS (ESI) Calcd for $C_{22}H_{30}N_2O_4S$: $[M+H]^+$ = 419.1999. Found: 419.2000.



3n

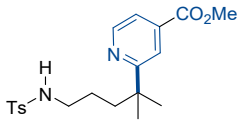
Methyl 2-((7R)-7-((4-methylphenyl)sulfonamido)octan-4-yl)isonicotinate (3n). Reaction of (R)-*N*-fluoro-4-methyl-*N*-(octan-2-yl)benzenesulfonamide **1n** (30.1 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (3:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.5 in 3:1 v/v petroleum ether/ethyl acetate) gave compound **3n** (33.5 mg, 80%, d.r. = 1:1) as a light-yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.69 (d, J = 5.1 Hz, 1H), 7.71 (dd, J = 17.2, 8.3 Hz, 2H), 7.65 (d, J = 5.0 Hz, 1H), 7.58 (s, 1H), 7.26 – 7.24 (m, 2H), 4.74 – 4.64 (m, 1H), 3.95 – 3.95 (m, 3H), 3.25 – 3.19 (m, 1H), 2.69 (s, 1H), 2.40 (d, J = 3.4 Hz, 3H), 1.61 – 1.47 (m, 4H), 1.30 – 1.24 (m, 1H), 1.14 – 1.03 (m, 1H), 0.96 (t, J = 6.9 Hz, 3H), 0.81 – 0.80 (m, 3H); $^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$) δ 166.0 (165.9), 165.9 (165.9), 150.0 (143.0), 138.2 (138.1), 137.5 (137.5), 129.5 (129.5), 127.0 (126.9), 121.9 (121.8), 120.4 (120.3), 52.6 (52.6), 50.2 (49.9), 47.3 (47.0), 38.1 (37.8), 35.2 (34.9), 31.2 (30.6), 21.9 (21.7), 21.4 (21.4), 20.5 (20.4), 14.0 (14.0); IR (ATR) ν_{max} 2927, 2869, 1730, 1436, 1289, 1158, 763, 551 cm^{-1}; HRMS (ESI) Calcd for $C_{22}H_{30}N_2O_4S$: $[M+H]^+$ = 419.1999. Found: 419.1990.



3o

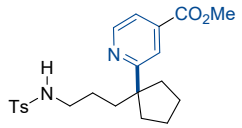
Methyl-2-(5,5-dimethyl-6-((4-methylphenyl)sulfonamido)hexan-3-yl)isonicotinate (3o). Reaction of *N*-(2,2-dimethylhexyl)-*N*-fluoro-4-methylbenzenesulfonamide **1o** (30.1 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (3:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.2 in 5:1 v/v petroleum ether/ethyl acetate) gave compound **3o** (29.6 mg, 71%) as a light-yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.60 (d, J = 5.0 Hz, 1H), 7.62 (s, 1H), 7.59 (d, J = 8.3 Hz, 2H), 7.56 (d, J = 5.1 Hz, 1H), 7.24 (d, J = 8.0 Hz, 2H), 5.17 (dd, J =

9.0, 5.0 Hz, 1H), 3.96 (s, 3H), 2.76 (q, J = 9.1 Hz, 1H), 2.54 (dd, J = 12.5, 9.0 Hz, 1H), 2.40 (s, 3H), 2.17 (dd, J = 12.5, 5.1 Hz, 1H), 2.04 (dd, J = 14.6, 9.8 Hz, 1H), 1.61 – 1.57 (m, 2H), 1.43 (dd, J = 14.6, 2.7 Hz, 1H), 0.83 (s, 3H), 0.75 (s, 3H), 0.70 (t, J = 7.4 Hz, 3H); $^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$) δ 166.5, 165.5, 149.7, 143.0, 137.7, 137.1, 129.5, 126.9, 122.2, 120.6, 52.7, 52.3, 45.0, 43.3, 34.4, 31.2, 26.7, 25.2, 21.4, 11.8; IR (ATR) ν_{max} 2959, 2926, 1731, 1437, 1288, 1160, 814, 551 cm^{-1}; HRMS (ESI) Calcd for $C_{22}H_{30}N_2O_4S$: $[M+H]^+$ = 419.1999. Found: 419.1992.



3p

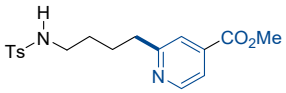
Methyl 2-(2-methyl-5-((4-methylphenyl)sulfonamido)pentan-2-yl)isonicotinate (3p). Reaction of *N*-fluoro-4-methyl-*N*-(4-methylpentyl)benzenesulfonamide **1p** (27.3 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (2:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.4 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **3p** (9.9 mg, 25%) as a light-yellow oil. ^1H NMR (500 MHz, $CDCl_3$) δ 8.63 (d, J = 5.7 Hz, 1H), 7.75 (s, 1H), 7.62 (d, J = 8.3 Hz, 2H), 7.59 (dd, J = 5.0, 1.5 Hz, 1H), 7.19 (d, J = 7.3 Hz, 2H), 4.94 (t, J = 5.9 Hz, 1H), 3.89 (s, 3H), 2.76 (q, J = 6.5 Hz, 2H), 2.34 (s, 3H), 1.66 – 1.63 (m, 2H), 1.29 – 1.21 (m, 8H); $^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$) δ 168.7, 165.8, 149.2, 143.2, 137.9, 136.9, 129.6, 127.0, 120.2, 119.5, 52.7, 43.5, 40.4, 39.2, 27.9, 24.8, 21.4; IR (ATR) ν_{max} 2925, 2855, 1731, 1437, 1302, 1158, 763, 551 cm^{-1}; HRMS (ESI) Calcd for $C_{20}H_{26}N_2O_4S$: $[M+H]^+$ = 391.1686. Found: 391.1678.



3q

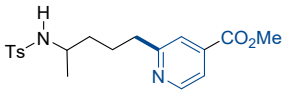
Methyl 2-(1-(3-((4-methylphenyl)sulfonamido)propyl)cyclopentyl)isonicotinate (3q). Reaction of *N*-(3-cyclopentylpropyl)-*N*-fluoro-4-methylbenzenesulfonamide **1q** (29.9 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (3:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.3 in 3:1 v/v petroleum ether/ethyl acetate) gave compound **3q** (8.9 mg, 21%) as a light-yellow oil. ^1H NMR (500 MHz, $CDCl_3$) δ 8.66 (d, J = 5.6 Hz, 1H), 7.75 (s,

1H), 7.66 (d, J = 8.3 Hz, 2H), 7.61 (d, J = 5.0 Hz, 1H), 7.26 – 7.24 (m, 2H), 4.64 (t, J = 5.9 Hz, 1H), 3.95 (s, 3H), 2.78 (q, J = 6.8 Hz, 2H), 2.40 (s, 3H), 2.11 – 2.07 (m, 2H), 1.74 – 1.67 (m, 6H), 1.64 – 1.56 (m, 2H), 1.16 – 1.10 (m, 2H); $^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$) δ 168.4, 166.0, 149.4, 143.2, 137.5, 136.9, 129.6, 127.0, 120.2, 119.9, 53.3, 52.7, 43.5, 37.8, 37.5, 25.5, 23.9, 21.5; IR (ATR) ν_{max} 2951, 2869, 1730, 1436, 1297, 1156, 762, 549 cm^{-1}; HRMS (ESI) Calcd for $C_{22}H_{28}N_2O_4S$: $[M+H]^+$ = 417.1843. Found: 417.1859.



3r

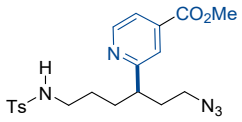
Methyl 2-(4-((4-methylphenyl)sulfonamido)butyl)isonicotinate (3r). Reaction of *N*-butyl-*N*-fluoro-4-methylbenzenesulfonamide **1r** (24.5 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (1:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.1 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **3r** (14.8 mg, 41%) as a light-yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.65 (d, J = 6.0 Hz, 1H), 7.73 (d, J = 8.1 Hz, 2H), 7.68 (d, J = 4.6 Hz, 2H), 7.29 (d, J = 8.0 Hz, 2H), 5.02 (t, J = 6.2 Hz, 1H), 3.96 (s, 3H), 2.97 (q, J = 6.6 Hz, 2H), 2.83 (t, J = 7.6 Hz, 2H), 2.41 (s, 3H), 1.79 – 1.73 (m, 2H), 1.57 – 1.51 (m, 2H); $^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$) δ 165.6, 162.5, 149.6, 143.3, 138.0, 137.0, 129.6, 127.0, 122.2, 120.5, 52.7, 42.8, 37.0, 28.9, 26.3, 21.5; IR (ATR) ν_{max} 2955, 2869, 1685, 1460, 1328, 1152, 733, 580 cm^{-1}; HRMS (ESI) Calcd for $C_{18}H_{22}N_2O_4S$: $[M+H]^+$ = 363.1373. Found: 363.1366.



3s

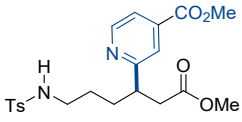
Methyl 2-(4-((4-methylphenyl)sulfonamido)pentyl)isonicotinate (3s). Reaction of *N*-butyl-*N*-fluoro-4-methylbenzenesulfonamide **1s** (24.5 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (1:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.2 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **3s** (22.9 mg, 61%) as a light-yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.67 (dd, J = 5.0, 0.9 Hz, 1H), 7.74 (d, J = 8.4 Hz, 2H), 7.71 – 7.70 (m, 2H), 7.25 (d, J = 8.0 Hz, 2H), 5.05 (d, J = 7.9 Hz, 1H), 3.96 (s, 3H), 3.33 – 3.30 (m, 1H), 2.81 (t, J = 7.6 Hz, 2H), 2.38 (s, 3H), 1.72 – 1.66 (m, 2H), 1.44 (q, J = 7.5 Hz, 2H), 1.01 (d, J = 6.5 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$) δ 165.4, 162.4, 149.0, 143.0, 138.4, 138.2,

129.5, 127.0, 122.5, 120.6, 52.8, 49.8, 36.9, 36.6, 25.2, 21.6, 21.4; IR (ATR) ν_{max} 2925, 2856, 1730, 1437, 1291, 1157, 665, 551 cm^{-1}; HRMS (ESI) Calcd for $C_{19}H_{24}N_2O_4S$: $[M+H]^+$ = 377.1530. Found: 377.1536.



3t

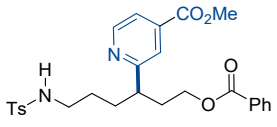
Methyl-2-(1-azido-6-((4-methylphenyl)sulfonamido)hexan-3-yl)isonicotinate (3t). Reaction of *N*-(6-azidohexyl)-*N*-fluoro-4-methylbenzenesulfonamide **1t** (31.4 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (2:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.4 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **3t** (27.6 mg, 64%) as a light-yellow oil. ^1H NMR (500 MHz, $CDCl_3$) δ 8.69 (d, *J* = 5.0 Hz, 1H), 7.70 – 7.67 (m, 3H), 7.62 (s, 1H), 7.28 – 7.26 (m, 2H), 4.66 (t, *J* = 6.2 Hz, 1H), 3.96 (s, 3H), 3.17 – 3.12 (m, 1H), 3.03 – 2.98 (m, 1H), 2.88 – 2.84 (m, 3H), 2.41 (s, 3H), 2.02 – 1.98 (m, 1H), 1.90 – 1.85 (m, 1H), 1.76 – 1.72 (m, 1H), 1.67 – 1.63 (m, 1H), 1.41 – 1.36 (m, 1H), 1.24 – 1.19 (m, 1H); $^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$) δ 165.6, 164.0, 150.5, 143.3, 137.9, 136.9, 129.6, 127.0, 122.2, 121.0, 52.7, 49.3, 44.3, 43.0, 34.3, 32.2, 27.3, 21.5; IR (ATR) ν_{max} 2931, 2095, 1730, 1437, 1290, 1156, 815, 551 cm^{-1}; HRMS (ESI) Calcd for $C_{20}H_{25}N_5O_4S$: $[M+H]^+$ = 432.1700. Found: 432.1689.



3u

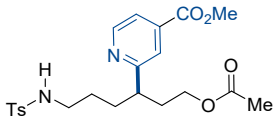
Methyl-2-(1-methoxy-6-((4-methylphenyl)sulfonamido)-1-oxohexan-3-yl)isonicotinate (3u). Reaction of methyl 6-((*N*-fluoro-4-methylphenyl)sulfonamido)hexanoate **1u** (31.7 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (1.5:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.2 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **3u** (29.9 mg, 69%) as a light-yellow oil. ^1H NMR (500 MHz, $CDCl_3$) δ 8.59 (d, *J* = 5.8 Hz, 1H), 7.62 (d, *J* = 8.3 Hz, 2H), 7.60 – 7.59 (m, 2H), 7.20 (d, *J* = 8.0 Hz, 2H), 4.65 (t, *J* = 6.1 Hz, 1H), 3.88 (s, 3H), 3.51 (s, 3H), 3.22 – 3.17 (m, 1H), 2.81 (q, *J* = 6.8 Hz, 2H), 2.73 (dd, *J* = 16.2, 8.2 Hz, 1H), 2.54 (dd, *J* = 16.2, 6.4 Hz, 1H), 2.34 (s, 3H), 1.70 – 1.67 (m, 1H), 1.62 –

1.56 (m, 1H), 1.34 – 1.29 (m, 1H), 1.20 – 1.15 (m, 1H); $^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$) δ 172.6, 165.6, 163.7, 150.2, 143.3, 137.7, 136.9, 129.6, 127.0, 122.3, 120.9, 52.7, 51.6, 42.9, 42.8, 39.3, 31.8, 27.1, 21.5; IR (ATR) ν_{max} 2920, 2851, 1732, 1437, 1292, 1159, 763, 550 cm^{-1}; HRMS (ESI) Calcd for $C_{21}H_{26}N_2O_6S$: $[M+H]^+$ = 435.1584. Found: 435.1572.



3v

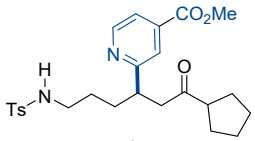
Methyl-2-(1-(benzoyloxy)-6-((4-methylphenyl)sulfonamido)hexan-3-yl)isonicotinate (3v). Reaction of 6-((*N*-fluoro-4-methylphenyl)sulfonamido)hexyl benzoate **1v** (39.3 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (1.5:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.2 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **3v** (37.8 mg, 74%) as a light-yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.66 (d, *J* = 5.8 Hz, 1H), 7.89 (d, *J* = 7.1 Hz, 2H), 7.68 (d, *J* = 8.2 Hz, 2H), 7.63 – 7.62 (m, 2H), 7.53 (t, *J* = 7.4 Hz, 1H), 7.39 (t, *J* = 7.8 Hz, 2H), 7.26 – 7.24 (m, 2H), 4.79 (t, *J* = 6.2 Hz, 1H), 4.23 – 4.19 (m, 1H), 4.11 – 4.07 (m, 1H), 3.91 (s, 3H), 2.98 – 2.94 (m, 1H), 2.86 (q, *J* = 6.7 Hz, 2H), 2.39 (s, 3H), 2.24 – 2.19 (m, 1H), 2.09 – 2.05 (m, 1H), 1.78 – 1.69 (m, 2H), 1.42 – 1.37 (m, 1H), 1.24 – 1.19 (m, 1H); $^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$) δ 166.4, 165.5, 164.4, 150.3, 143.3, 137.8, 136.9, 132.9, 130.0, 129.6, 129.4, 128.2, 127.0, 122.1, 120.8, 63.0, 52.6, 44.2, 43.0, 34.1, 32.3, 27.3, 21.4; IR (ATR) ν_{max} 2925, 1716, 1600, 1437, 1273, 1156, 714, 551 cm^{-1}; HRMS (ESI) Calcd for $C_{27}H_{30}N_2O_6S$: $[M+H]^+$ = 511.1897. Found: 511.1875.



3w

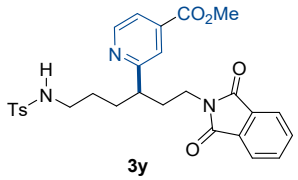
Methyl-2-(1-acetoxy-6-((4-methylphenyl)sulfonamido)hexan-3-yl)isonicotinate (3w). Reaction of 6-((*N*-fluoro-4-methylphenyl)sulfonamido)hexyl acetate **1w** (33.1 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (1.5:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.2 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **3w** (31.4 mg, 70%) as a light-yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.66 (d, *J* = 5.0 Hz, 1H), 7.68 (d, *J* = 8.3 Hz, 2H), 7.65 (d, *J* = 5.0 Hz, 1H), 7.60 (s, 1H), 7.27 – 7.25 (m, 2H), 4.73 (t, *J* = 6.2 Hz,

1H), 3.94 – 3.91 (m, 4H), 3.82 – 3.79 (m, 1H), 2.86 – 2.83 (m, 3H), 2.40 (s, 3H), 2.07 – 2.03 (m, 1H), 1.95 – 1.91 (m, 4H), 1.74 – 1.71 (m, 1H), 1.67 – 1.64 (m, 1H), 1.39 – 1.35 (m, 1H), 1.24 – 1.20 (m, 1H); $^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$) δ 171.0, 165.6, 164.4, 150.3, 143.3, 137.7, 136.9, 129.6, 127.0, 122.1, 120.8, 62.4, 52.7, 43.9, 43.0, 34.0, 32.2, 27.3, 21.4, 20.8; IR (ATR) ν_{max} 2926, 1729, 1561, 1437, 1289, 1156, 762, 550 cm^{-1}; HRMS (ESI) Calcd for $C_{22}H_{28}N_2O_6S$: $[M+H]^+$ = 449.1741. Found: 449.1761.



3x

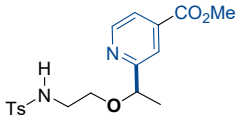
Methyl-2-(1-cyclopentyl-6-((4-methylphenyl)sulfonamido)-1-oxohexan-3-yl)isonicotinate (3x). Reaction of *N*-(6-cyclopentyl-6-oxohexyl)-*N*-fluoro-4-methylbenzenesulfonamide **1x** (35.5 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (2:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.3 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **3x** (31.2 mg, 66%) as a light-yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.62 (dd, *J* = 5.0, 0.9 Hz, 1H), 7.70 – 7.66 (m, 2H), 7.65 (t, *J* = 1.2 Hz, 1H), 7.63 (dd, *J* = 5.0, 1.6 Hz, 1H), 7.27 – 7.25 (m, 2H), 4.75 (t, *J* = 6.2 Hz, 1H), 3.93 (s, 3H), 3.32 – 3.29 (m, 1H), 3.01 (dd, *J* = 17.6, 7.7 Hz, 1H), 2.86 (q, *J* = 6.7 Hz, 2H), 2.76 – 2.69 (m, 2H), 2.40 (s, 3H), 1.73 – 1.67 (m, 4H), 1.59 – 1.52 (m, 6H), 1.39 – 1.35 (m, 1H), 1.24 – 1.20 (m, 1H); $^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$) δ 211.7, 165.7, 164.5, 150.1, 143.2, 137.6, 136.9, 129.6, 127.0, 122.7, 120.7, 52.6, 51.6, 46.8, 42.8, 41.6, 31.9, 28.7, 28.6, 27.2, 25.9, 25.8, 21.5; IR (ATR) ν_{max} 2951, 2868, 1731, 1438, 1291, 1158, 816, 551 cm^{-1}; HRMS (ESI) Calcd for $C_{25}H_{32}N_2O_5S$: $[M+H]^+$ = 473.2105. Found: 473.2091.



3y

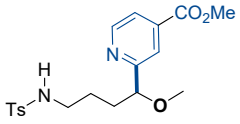
Methyl-2-(1-(1,3-dioxoisoindolin-2-yl)-6-((4-methylphenyl)sulfonamido)hexan-3-yl)isonicotinate (3y). Reaction of *N*-(6-(1,3-dioxoisoindolin-2-yl)hexyl)-*N*-fluoro-4-methylbenzenesulfonamide **1y** (41.8 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (1.5:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.3 in 2:1

v/v petroleum ether/ethyl acetate) gave compound **3y** (37.9 mg, 71%) as a light-yellow oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.60 (d, *J* = 5.0 Hz, 1H), 7.76 (dd, *J* = 5.4, 3.0 Hz, 2H), 7.69 – 7.66 (m, 4H), 7.61 (s, 1H), 7.57 (d, *J* = 4.9 Hz, 1H), 7.25 (d, *J* = 8.3 Hz, 2H), 4.82 (t, *J* = 5.5 Hz, 1H), 3.93 (s, 3H), 3.56 (t, *J* = 7.3 Hz, 2H), 2.85 (q, *J* = 6.7 Hz, 2H), 2.78 (t, *J* = 8.7 Hz, 1H), 2.39 (s, 3H), 2.19 – 2.15 (m, 1H), 1.98 – 1.95 (m, 1H), 1.74 – 1.70 (m, 1H), 1.34 – 1.30 (m, 1H), 1.25 – 1.22 (m, 1H); ^{13}C{^{1}H} NMR (151 MHz, $CDCl_3$) δ 168.2, 165.6, 164.3, 150.2, 143.2, 137.6, 136.9, 133.9, 131.9, 129.6, 127.0, 123.1, 122.0, 120.7, 52.6, 44.7, 42.8, 36.1, 33.6, 31.9, 27.1, 21.4; IR (ATR) ν_{max} 2926, 1706, 1599, 1397, 1288, 1156, 721, 550 cm^{-1}; HRMS (ESI) Calcd for $C_{28}H_{29}N_3O_6S$: $[M+H]^+$ = 536.1850. Found: 536.1860.



3z

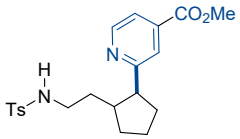
Methyl-2-(1-(2-((4-methylphenyl)sulfonamido)ethoxy)ethyl)isonicotinate (3z). Reaction of *N*-(2-ethoxyethyl)-*N*-fluoro-4-methylbenzenesulfonamid*e* **1z** (26.1 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (1.5:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.2 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **3z** (19.3 mg, 51%) as a light-yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.65 (d, *J* = 5.8 Hz, 1H), 7.76 (s, 1H), 7.69 – 7.66 (m, 3H), 7.21 (d, *J* = 8.6 Hz, 2H), 5.50 (t, *J* = 5.7 Hz, 1H), 4.46 (q, *J* = 6.6 Hz, 1H), 3.90 (s, 3H), 3.4 – 3.44 (m, 1H), 3.31 – 3.27 (m, 1H), 3.10 – 3.04 (m, 2H), 2.35 (s, 3H), 1.36 (d, *J* = 6.6 Hz, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 165.5, 163.5, 150.0, 143.3, 138.3, 137.0, 129.6, 127.0, 121.8, 119.6, 78.8, 67.4, 52.8, 43.1, 22.0, 21.5; IR (ATR) ν_{max} 2924, 1730, 1600, 1438, 1292, 1158, 764, 551 cm^{-1}; HRMS (ESI) Calcd for $C_{18}H_{22}N_2O_5S$: $[M+H]^+$ = 379.1322. Found: 379.1333.



3aa

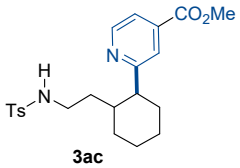
Methyl-2-(1-methoxy-4-((4-methylphenyl)sulfonamido)butyl)isonicotinate (3aa). Reaction of *N*-fluoro-*N*-(4-methoxybutyl)-4-methylbenzenesulfonamide **1aa** (27.5 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (1.5:1 v/v petroleum

ether/ethyl acetate elution, R_f = 0.2 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **3aa** (20.3 mg, 52%) as a light-yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.69 (d, *J* = 5.0 Hz, 1H), 7.89 (s, 1H), 7.75 (dd, *J* = 5.0, 1.5 Hz, 1H), 7.72 (d, *J* = 8.3 Hz, 2H), 7.28 (d, *J* = 7.5 Hz, 2H), 4.88 (t, *J* = 6.1 Hz, 1H), 4.29 (t, *J* = 6.3 Hz, 1H), 3.97 (s, 3H), 3.27 (s, 3H), 2.98 – 2.94 (m, 2H), 2.41 (s, 3H), 1.77 – 1.74 (m, 2H), 1.60 – 1.54 (m, 2H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 165.6, 163.0, 149.9, 143.3, 138.4, 137.0, 129.6, 127.1, 121.8, 119.6, 83.9, 57.4, 52.8, 43.0, 33.6, 25.6, 21.5; IR (ATR) ν_{max} 2923, 1729, 1451, 1330, 1156, 663, 546 cm^{-1}; HRMS (ESI) Calcd for $C_{19}H_{24}N_2O_5S$: $[M+H]^+$ = 393.1479. Found: 393.1477.



3ab

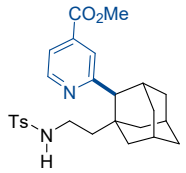
Methyl 2-(2-(2-((4-methylphenyl)sulfonamido)ethyl)cyclopentyl)isonicotinate (3ab). Reaction of *N*-(2-cyclopentylethyl)-*N*-fluoro-4-methylbenzenesulfonamide **1ab** (28.5 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (2:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.4 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **3ab** (29.0 mg, 72%, d.r. > 20:1) as a light-yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.64 (d, *J* = 5.0 Hz, 1H), 7.66 – 7.63 (m, 4H), 7.23 (d, *J* = 8.0 Hz, 2H), 5.28 (t, *J* = 5.9 Hz, 1H), 3.95 (s, 3H), 2.87 – 2.80 (m, 3H), 2.39 (s, 3H), 2.20 (q, *J* = 8.9 Hz, 1H), 2.12 – 2.06 (m, 1H), 1.94 – 1.90 (m, 1H), 1.78 – 1.70 (m, 3H), 1.52 – 1.48 (m, 2H), 1.29 – 1.26 (m, 2H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 165.8, 165.6, 149.9, 143.1, 137.8, 137.0, 129.5, 127.0, 121.5, 120.5, 53.5, 52.7, 43.4, 42.0, 34.4, 34.1, 32.7, 24.3, 21.4; IR (ATR) ν_{max} 2951, 2868, 1729, 1436, 1288, 1155, 762, 549 cm^{-1}; HRMS (ESI) Calcd for $C_{21}H_{26}N_2O_4S$: $[M+H]^+$ = 403.1686. Found: 403.1694.



3ac

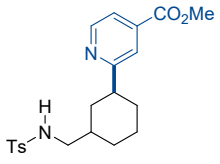
Methyl 2-(2-(2-((4-methylphenyl)sulfonamido)ethyl)cyclohexyl)isonicotinate (3ac). Reaction of *N*-(2-cyclohexylethyl)-*N*-fluoro-4-methylbenzenesulfonamide **1ac** (29.9 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (2:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.4 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **3ac** (32.9 mg,

79%, d.r. > 20:1) as a light-yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.65 (d, *J* = 5.9 Hz, 1H), 7.66 – 7.62 (m, 4H), 7.24 (d, *J* = 8.0 Hz, 2H), 5.21 (t, *J* = 5.4 Hz, 1H), 3.94 (s, 3H), 2.85 – 2.80 (m, 1H), 2.74 – 2.72 (m, 1H), 2.46 (t, *J* = 11.2 Hz, 1H), 2.39 (s, 3H), 1.80 – 1.70 (m, 5H), 1.46 – 1.39 (m, 1H), 1.30 – 1.25 (m, 2H), 1.16 – 1.12 (m, 2H), 1.02 – 0.94 (m, 1H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 166.2, 165.7, 149.9, 143.0, 137.9, 136.8, 129.5, 127.0, 122.0, 120.5, 52.7, 51.8, 40.7, 37.9, 34.8, 33.9, 32.0, 26.2, 25.9, 21.4; IR (ATR) ν_{max} 2925, 2854, 1730, 1438, 1291, 1159, 763, 551 cm^{-1}; HRMS (ESI) Calcd for $C_{22}H_{28}N_2O_4S$: $[M+H]^+$ = 417.1843. Found: 417.1836.



3ad

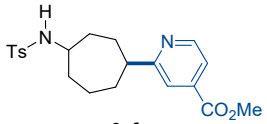
Methyl 2-((1r,3s,5R,7S)-1-(2-((4-methylphenyl)sulfonamido)ethyl)adamantan-2-yl)isonicotinate (3ad). Reaction of *N*-(2-((3r,5r,7r)-adamantan-1-yl)ethyl)-*N*-fluoro-4-methylbenzenesulfonamide **1ad** (35.1 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (3:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.2 in 5:1 v/v petroleum ether/ethyl acetate) gave compound **3ad** (28.5 mg, 61%, d.r. > 20:1) as a light-yellow oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.62 (d, *J* = 5.0 Hz, 1H), 7.69 (t, *J* = 1.2 Hz, 1H), 7.61 – 7.59 (m, 3H), 7.23 (d, *J* = 8.0 Hz, 2H), 4.41 (t, *J* = 5.9 Hz, 1H), 3.95 (s, 3H), 2.93 (s, 1H), 2.85 – 2.80 (m, 2H), 2.71 (d, *J* = 12.2 Hz, 1H), 2.41 (s, 3H), 2.12 (d, *J* = 12.3 Hz, 1H), 2.01 (d, *J* = 4.9 Hz, 2H), 1.91 (s, 1H), 1.84 (s, 2H), 1.72 (d, *J* = 12.2 Hz, 1H), 1.64 (d, *J* = 11.8 Hz, 2H), 1.51 (d, *J* = 14.9 Hz, 1H), 1.41 (d, *J* = 12.6 Hz, 1H), 1.35 – 1.34 (m, 1H), 1.29 – 1.26 (m, 2H), 1.14 – 1.10 (m, 1H); ^{13}C{^{1}H} NMR (151 MHz, $CDCl_3$) δ 165.9, 165.4, 148.9, 143.1, 137.3, 136.9, 129.5, 127.0, 123.4, 119.8, 55.7, 52.6, 44.0, 40.1, 39.4, 38.2, 37.6, 37.3, 35.0, 35.0, 30.4, 28.8, 28.1, 21.5; IR (ATR) ν_{max} 2904, 2850, 1730, 1437, 1294, 1158, 762, 550 cm^{-1}; HRMS (ESI) Calcd for $C_{26}H_{32}N_2O_4S$: $[M+H]^+$ = 469.2156. Found: 469.2158.



3ae

Methyl 2-(3-(((4-methylphenyl)sulfonamido)methyl)cyclohexyl)isonicotinate (3ae).

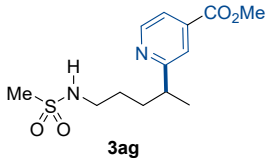
Reaction of *N*-(cyclohexylmethyl)-*N*-fluoro-4-methylbenzenesulfonamide **1ae** (28.5 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (2:1 v/v petroleum ether/ethyl acetate elution,) gave compound **3ae** (16.1 mg, 40%, d.r. = 2.4:1). (major, 11.4 mg, R_f = 0.2 in 2:1 v/v petroleum ether/ethyl acetateas a colorless oil). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.66 (d, *J* = 5.0 Hz, 1H), 7.72 (d, *J* = 8.3 Hz, 2H), 7.67 – 7.65 (m, 2H), 7.27 (d, *J* = 8.1 Hz, 2H), 4.69 (t, *J* = 6.5 Hz, 1H), 3.94 (s, 3H), 2.84 – 2.76 (m, 3H), 2.40 (s, 3H), 1.91 (dd, *J* = 27.3, 11.1 Hz, 4H), 1.77 (d, *J* = 12.9 Hz, 1H), 1.46 – 1.37 (m, 2H), 1.22 – 1.14 (m, 1H), 0.97 – 0.89 (m, 1H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 166.6, 165.8, 149.8, 143.3, 137.0, 129.6, 127.0, 120.5, 52.6, 49.3, 45.6, 37.9, 36.3, 32.4, 29.8, 25.5, 21.5. HRMS (ESI) Calcd for $C_{21}H_{26}N_2O_4S$: $[M+H]^+$ = 403.1686. Found: 403.1674. (minor, 4.7 mg, R_f = 0.3 in 2:1 v/v petroleum ether/ethyl acetateas a colorless oil). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.67 (d, *J* = 5.1 Hz, 1H), 7.76 (d, *J* = 8.3 Hz, 2H), 7.72 (s, 1H), 7.66 (d, *J* = 4.5 Hz, 1H), 7.29 (d, *J* = 8.0 Hz, 2H), 4.76 (s, 1H), 3.95 (s, 3H), 3.03 (t, *J* = 7.1 Hz, 2H), 2.95 (s, 1H), 2.41 (s, 3H), 1.96 – 1.95 (m, 1H), 1.87 – 1.85 (m, 2H), 1.79 – 1.76 (m, 1H), 1.72 – 1.66 (m, 1H), 1.56 – 1.53 (m, 3H), 1.49 – 1.44 (m, 1H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 166.5, 165.8, 149.6, 143.3, 138.0, 137.1, 129.7, 127.1, 120. 8, 120.5, 52.7, 45.6, 40.1, 33.6, 33.4, 31.7, 27.7, 21.5, 20.8. IR (ATR) ν_{max} 2923, 2854, 1729, 1437, 1294, 1156, 661, 550 cm^{-1}; HRMS (ESI) Calcd for $C_{21}H_{26}N_2O_4S$: $[M+H]^+$ = 403.1686. Found: 403.1679.



3af

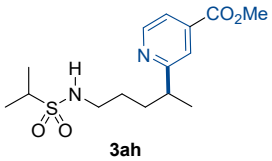
Methyl 2-(4-((4-methylphenyl)sulfonamido)cycloheptyl)isonicotinate (3af). Reaction of *N*-cycloheptyl-*N*-fluoro-4-methylbenzenesulfonamide **1af** (28.5 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (2:1 v/v petroleum ether/ethyl acetate elution,) gave compound **3af** (17.1 mg, 42%, d.r. = 1.1:1). (major, 9.1 mg, R_f = 0.3 in 2:1 v/v petroleum ether/ethyl acetateas a colorless oil). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.70 (d, *J* = 5.0 Hz, 1H), 7.77 (d, *J* = 8.3 Hz, 2H), 7.66 (d, *J* = 8.0 Hz, 2H), 7.38 – 7.26 (m, 2H), 5.95 (d, *J* = 8.4 Hz, 1H), 3.94 (s, 3H), 3.62 – 3.59 (m, 1H), 3.07 – 3.03 (m, 1H), 2.40 (s, 3H), 1.97 – 1.93 (m, 2H), 1.83 – 1.72 (m, 5H), 1.64 – 1.60 (m, 1H), 1.56 – 1.52 (m, 1H), 1.40 – 1.33 (m, 1H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 167.7, 165.7, 149.5, 142.9, 138.6, 138.0, 129.6, 126.9, 121.1, 120.5,

53.4, 52.7, 46.4, 36.3, 35.0, 32.8, 27.7, 22.5, 21.5. HRMS (ESI) Calcd for $C_{21}H_{26}N_2O_4S$: $[M+H]^+$ = 403.1686. Found: 403.1678. (minor, 8.0 mg, R_f = 0.3 in 2:1 v/v petroleum ether/ethyl acetateas a colorless oil). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.65 (d, J = 4.6 Hz, 1H), 7.77 (d, J = 8.4 Hz, 2H), 7.64 – 7.63 (m, 2H), 7.30 (d, J = 8.0 Hz, 2H), 4.60 (d, J = 7.7 Hz, 1H), 3.94 (s, 3H), 3.44 – 3.42 (m, 1H), 2.93 (s, 1H), 2.42 (s, 3H), 2.01 – 1.94 (m, 2H), 1.88 – 1.84 (m, 2H), 1.70 – 1.63 (m, 5H), 1.53 – 1.46 (m, 1H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 167.9, 165.8, 149.8, 143.2, 138.1, 137.9, 129.7, 127.0, 120.5, 120.4, 54.8, 52.7, 48.4, 35.5, 35.1, 34.2, 30.6, 22.0, 21.5; IR (ATR) ν_{max} 2925, 2857, 1729, 1437, 1293, 1155, 665, 550 cm^{-1}; HRMS (ESI) Calcd for $C_{21}H_{26}N_2O_4S$: $[M+H]^+$ = 403.1686. Found: 403.1674.



3ag

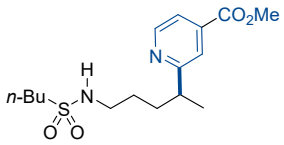
Methyl 2-(5-(methylsulfonamido)pentan-2-yl)isonicotinate (3ag). Reaction of *N*-fluoro-*N*-pentylmethanesulfonamide **1ag** (18.3 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (50:1 v/v dichloromethane/methanol elution, R_f = 0.3 in 40:1 v/v dichloromethane/methanol) gave compound **3ag** (27.7 mg, 92%) as a light-yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.68 (d, J = 5.0 Hz, 1H), 7.70 (s, 1H), 7.67 (d, J = 5.0 Hz, 1H), 4.57 (s, 1H), 3.96 (s, 3H), 3.10 (q, J = 6.8 Hz, 2H), 3.02 – 2.98 (m, 1H), 2.91 (s, 3H), 1.88 – 1.84 (m, 1H), 1.70 – 1.67 (m, 1H), 1.59 – 1.54 (m, 1H), 1.45 – 1.41 (m, 1H), 1.32 (d, J = 6.9 Hz, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 166.8, 165.8, 149.9, 138.0, 121.0, 120.6, 52.7, 43.2, 41.4, 40.3, 33.6, 28.0, 20.9; IR (ATR) ν_{max} 2927, 1729, 1561, 1437, 1294, 1147, 763, 521 cm^{-1}; HRMS (ESI) Calcd for $C_{13}H_{20}N_2O_4S$: $[M+H]^+$ = 301.1217. Found: 301.1218.



3ah

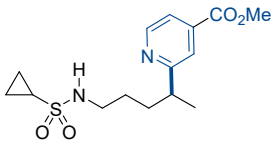
Methyl 2-(5-((1-methylethyl)sulfonamido)pentan-2-yl)isonicotinate (3ah). Reaction of *N*-fluoro-*N*-pentylpropane-2-sulfonamide **1ah** (21.1 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (1.5:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.2 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **3ah** (26.6 mg, 81%) as a

light-yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δδ 8.68 (d, J = 5.1 Hz, 1H), 7.69 (s, 1H), 7.66 (d, J = 5.1 Hz, 1H), 4.50 – 4.48 (m, 1H), 3.94 (s, 3H), 3.12 – 3.07 (m, 3H), 3.00 (q, J = 6.9 Hz, 1H), 1.85 – 1.80 (m, 1H), 1.70 – 1.65 (m, 1H), 1.55 – 1.50 (m, 1H), 1.41 (d, J = 6.8 Hz, 1H), 1.33 – 1.29 (m, 9H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 166.9, 165.8, 149.9, 137.8, 120.9, 120.5, 53.2, 52.6, 43.5, 41.4, 33.6, 28.5, 20.8, 16.6, 16.6; IR (ATR) ν_{max} 2934, 1729, 1561, 1437, 1292, 1131, 763, 512 cm^{-1}; HRMS (ESI) Calcd for $C_{15}H_{24}N_2O_4S$: $[M+H]^+$ = 329.1530. Found: 329.1522.



3ai

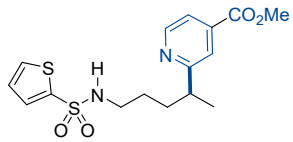
Methyl 2-(5-(butylsulfonamido)pentan-2-yl)isonicotinate (3ai). Reaction of *N*-fluoro-*N*-pentylbutane-1-sulfonamide **1ai** (22.5 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (2:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.3 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **3ai** (20.4 mg, 60%) as a light-yellow oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.68 (dd, J = 5.0, 0.9 Hz, 1H), 7.69 (s, 1H), 7.67 (dd, J = 5.0, 1.6 Hz, 1H), 4.48 (t, J = 5.9 Hz, 1H), 3.95 (s, 3H), 3.07 (q, J = 6.8 Hz, 2H), 3.00 – 2.95 (m, 3H), 1.85 – 1.82 (m, 1H), 1.76 – 1.73 (m, 2H), 1.70 – 1.67 (m, 1H), 1.56 – 1.52 (m, 1H), 1.44 – 1.40 (m, 3H), 1.31 (d, J = 6.9 Hz, 3H), 0.92 (t, J = 7.4 Hz, 3H); ^{13}C{^{1}H} NMR (151 MHz, $CDCl_3$) δ 166.8, 165.8, 149.9, 137.9, 120.9, 120.6, 52.7, 52.3, 43.2, 41.4, 33.7, 28.2, 25.6, 21.5, 20.9, 13.6; IR (ATR) ν_{max} 2959, 2873, 1730, 1437, 1293, 1139, 763, 564 cm^{-1}; HRMS (ESI) Calcd for $C_{16}H_{26}N_2O_4S$: $[M+H]^+$ = 343.1686. Found: 343.1703.



3aj

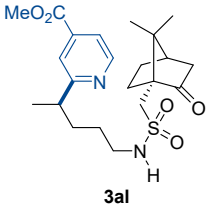
Methyl 2-(5-(cyclopropanesulfonamido)pentan-2-yl)isonicotinate (3aj). Reaction of *N*-fluoro-*N*-pentylcyclopropanesulfonamide **1aj** (20.9 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (1:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.1 in 1:1 v/v petroleum ether/ethyl acetate) gave compound **3aj** (27.1 mg, 83%) as a light-

yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.66 (d, *J* = 5.6 Hz, 1H), 7.68 (s, 1H), 7.65 (dd, *J* = 5.0, 1.5 Hz, 1H), 4.64 (t, *J* = 6.1 Hz, 1H), 3.94 (s, 3H), 3.11 (q, *J* = 6.8 Hz, 2H), 3.00 – 2.96 (m, 1H), 2.37 – 2.32 (m, 1H), 1.85 – 1.81 (m, 1H), 1.69 – 1.66 (m, 1H), 1.55 – 1.52 (m, 1H), 1.43 – 1.40 (m, 1H), 1.30 (d, *J* = 6.9 Hz, 3H), 1.12 – 1.11 (m, 2H), 0.93 (dd, *J* = 7.8, 2.2 Hz, 2H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 166.9, 165.8, 149.9, 137.8, 120.9, 120.5, 52.7, 43.3, 41.4, 33.7, 29.9, 28.1, 20.8, 5.2, 5.2; IR (ATR) ν_{max} 2923, 1729, 1561, 1437, 1296, 1145, 892, 763 cm^{-1}; HRMS (ESI) Calcd for $C_{15}H_{22}N_2O_4S$: $[M+H]^+$ = 327.1373. Found: 327.1368.



3ak

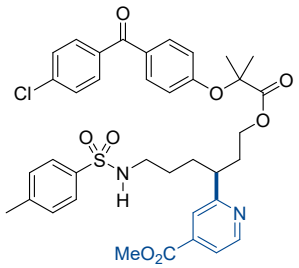
Methyl 2-(5-(thiophene-2-sulfonamido)pentan-2-yl)isonicotinate (3ak). Reaction of *N*-fluoro-*N*-pentylthiophene-2-sulfonamide **1ak** (25.1 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (1.5:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.2 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **3ak** (19.0 mg, 52%) as a light-yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.64 (d, *J* = 4.9 Hz, 1H), 7.64 (d, *J* = 5.1 Hz, 2H), 7.55 – 7.53 (m, 2H), 7.05 – 7.03 (m, 1H), 5.20 (t, *J* = 5.9 Hz, 1H), 3.94 (s, 3H), 3.02 – 2.97 (m, 2H), 2.94 – 2.90 (m, 1H), 1.78 – 1.73 (m, 1H), 1.65 – 1.59 (m, 1H), 1.50 – 1.44 (m, 1H), 1.36 – 1.32 (m, 1H), 1.25 (d, *J* = 6.9 Hz, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 166.8, 165.8, 149.8, 141.0, 137.9, 131.9, 131.57, 127.3, 120.9, 120.5, 52.7, 43.4, 41.2, 33.59, 27.2, 20.8; IR (ATR) ν_{max} 2920, 2851, 1730, 1437, 1295, 1156, 763, 592 cm^{-1}; HRMS (ESI) Calcd for $C_{16}H_{20}N_2O_4S_2$: $[M+H]^+$ = 369.0937. Found: 369.0927.



3al

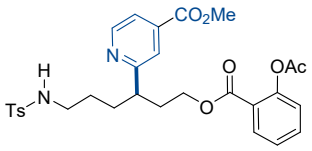
Methyl 2-(5-((((1R,4S)-7,7-dimethyl-2-oxobicyclo[2.2.1]heptan-1-yl)methyl)sulfonamide-o)pentan-2-yl)isonicotinate (3al). Reaction of 1-((1R,4S)-7,7-dimethyl-2-oxobicyclo[2.2.1]-heptan-1-yl)-*N*-fluoro-*N*-pentylmethanesulfonamide **1al** (31.9 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (2:1 v/v petroleum ether/ethyl acetate elution,

R_f = 0.3 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **3al** (24.9 mg, 57%) as a light-yellow oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.71 (s, 1H), 7.71 (s, 1H), 7.68 (d, *J* = 5.0 Hz, 1H), 5.24 – 5.22 (m, 1H), 3.95 (s, 3H), 3.36 (d, *J* = 15.1 Hz, 1H), 3.15 – 3.11 (m, 2H), 2.88 (d, *J* = 15.1 Hz, 1H), 2.37 (d, *J* = 18.6 Hz, 1H), 2.22 (t, *J* = 14.8 Hz, 1H), 2.11 (t, *J* = 4.5 Hz, 1H), 2.04 – 2.01 (m, 1H), 1.94 – 1.92 (m, 2H), 1.85 – 1.81 (m, 2H), 1.73 – 1.71 (m, 1H), 1.60 – 1.56 (m, 1H), 1.47 – 1.43 (m, 2H), 1.32 (d, *J* = 6.9 Hz, 3H), 1.01 (s, 3H), 0.89 (s, 3H); ^{13}C{^{1}H} NMR (151 MHz, $CDCl_3$) δ 217.0, 167.0, 165.8, 149.9, 138.0, 121.0, 120.6, 59.2, 52.7, 49.2, 48.8, 43.6, 42.9, 42.8, 41.5, 33.8, 28.1, 27.0, 26.6, 20.7, 19.9, 19.5; IR (ATR) ν_{max} 2955, 2924, 1730, 1437, 1291, 1145, 763, 568 cm^{-1}; HRMS (ESI) Calcd for $C_{22}H_{32}N_2O_5S$: $[M+H]^+$ = 437.2105. Found: 437.2094.



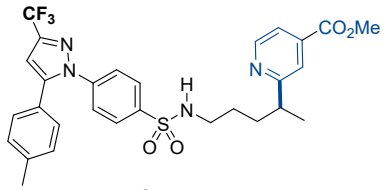
3am

Methyl 2-(1-((2-(4-(4-chlorobenzoyl)phenoxy)-2-methylpropanoyl)oxy)-6-((4-methylphenyl)sulfonamido)hexan-3-yl)isonicotinate (3am). Reaction of 6-((N-fluoro-4-methylphenyl)sulfonamido)hexyl 2-(4-(4-chlorobenzoyl)phenoxy)-2-methylpropanoate **1am** (58.9 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (1.5:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.2 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **3am** (43.6 mg, 62%) as a light-yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.65 (d, *J* = 5.0 Hz, 1H), 7.72 – 7.68 (m, 6H), 7.64 (d, *J* = 6.3 Hz, 1H), 7.48 (s, 1H), 7.43 (d, *J* = 8.5 Hz, 2H), 7.26 – 7.25 (m, 2H), 6.84 (d, *J* = 8.8 Hz, 2H), 4.88 (t, *J* = 6.3 Hz, 1H), 4.10 – 4.06 (m, 1H), 3.92 (s, 3H), 3.89 – 3.85 (m, 1H), 2.80 – 2.76 (m, 2H), 2.62 (s, 1H), 2.40 (s, 3H), 2.02 – 1.99 (m, 1H), 1.90 – 1.85 (m, 1H), 1.64 (d, *J* = 6.9 Hz, 7H), 1.56 – 1.53 (m, 1H), 1.21– 1.17 (m, 1H), 1.11– 1.05 (m, 1H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 194.5, 173.5, 165.5, 164.1, 159.7, 150.4, 143.2, 138.6, 137.8, 137.0, 136.1, 132.0, 131.3, 130.3, 129.6, 128.6, 127.0, 122.2, 120.9, 117.0, 79.3, 63.6, 52.7, 43.7, 43.0, 33.8, 32.1, 27.4, 25.9, 24.9, 21.5; IR (ATR) ν_{max} 2925, 1733, 1651, 1596, 1277, 1140, 750, 550 cm^{-1}; HRMS (ESI) Calcd for $C_{37}H_{39}ClN_2O_8S$: $[M+H]^+$ = 707.2188. Found: 707.2108.



3an

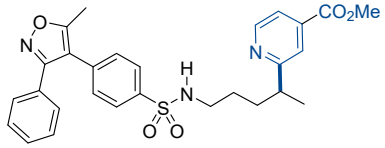
Methyl 2-(1-((2-acetoxybenzoyl)oxy)-6-((4-methylphenyl)sulfonamido)hexan-3-yl)isonicotinate (3an). Reaction of 6-((*N*-fluoro-4-methylphenyl)sulfonamido)hexyl 2-acetoxybenzoate **1an** (45.1 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (1.5:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.2 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **3an** (32.8 mg, 58%) as a light-yellow oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.67 (d, *J* = 5.0 Hz, 1H), 7.86 (d, *J* = 7.8 Hz, 1H), 7.67 (d, *J* = 8.3 Hz, 2H), 7.64 (d, *J* = 5.0 Hz, 1H), 7.62 (s, 1H), 7.55 (t, *J* = 7.8 Hz, 1H), 7.28 (t, *J* = 7.6 Hz, 1H), 7.25 (d, *J* = 8.7 Hz, 2H), 7.08 (d, *J* = 9.0 Hz, 1H), 4.67 (t, *J* = 6.3 Hz, 1H), 4.16 – 4.13 (m, 1H), 4.07 – 4.04 (m, 1H), 3.93 (s, 3H), 2.94 – 2.92 (m, 1H), 2.85 (q, *J* = 6.7 Hz, 2H), 2.40 (s, 3H), 2.32 (s, 3H), 2.21 – 2.17 (m, 1H), 2.07 – 2.04 (m, 1H), 1.78 – 1.75 (m, 1H), 1.71 – 1.69 (m, 1H), 1.39 – 1.35 (m, 1H), 1.25 – 1.23 (m, 1H); ^{13}C{^{1}H} NMR (151 MHz, $CDCl_3$) δ 169.7, 165.6, 164.3, 164.2, 150.6, 150.4, 143.3, 137.8, 136.9, 133.8, 131.6, 129.6, 127.0, 125.9, 123.7, 123.1, 122.2, 120.9, 63.1, 52.7, 44.0, 42.9, 34.1, 32.2, 27.3, 21.5, 21.0; IR (ATR) ν_{max} 2925, 1723, 1605, 1452, 1292, 1157, 756, 550 cm^{-1}; HRMS (ESI) Calcd for $C_{29}H_{32}N_2O_8S$: $[M+H]^+$ = 569.1952. Found: 569.1947.



3ao

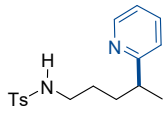
Methyl 2-(5-((4-(5-(p-tolyl)-3-(trifluoromethyl)-1H-pyrazol-1-yl)phenyl)sulfonamido)pentan-2-yl)isonicotinate (3ao). Reaction of *N*-fluoro-*N*-pentyl-4-(5-(p-tolyl)-3-(trifluoromethyl)-1H-pyrazol-1-yl)benzenesulfonamide **1ao** (46.9 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (2:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.3 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **3ao** (41.7 mg, 71%) as a light-yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.67 (d, *J* = 5.1 Hz, 1H), 7.80 (d, *J* = 8.6 Hz, 2H), 7.68 – 7.66 (m, 2H), 7.43 (d, *J* = 8.6 Hz, 2H), 7.16 (d, *J* = 7.8 Hz, 2H), 7.09 (d, *J* = 8.3

Hz, 2H), 6.73 (s, 1H), 5.19 (t, J = 6.0 Hz, 1H), 3.94 (s, 3H), 2.98 – 2.89 (m, 3H), 2.36 (s, 3H), 1.79 – 1.74 (m, 1H), 1.62 – 1.58 (m, 1H), 1.47 – 1.43 (m, 1H), 1.35 – 1.32 (m, 1H), 1.25 (d, J = 6.9 Hz, 3H); $^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$) δ 166.8, 165.6, 149.8, 145.2, 144.0 (q, J = 38.5 Hz), 142.3, 139.7, 139.5, 138.2, 129.7, 128.7, 128.0, 125.6, 125.5, 121.1, 121.0 (q, J = 269.6 Hz), 120.7, 106.2, 52.7, 43.1, 41.1, 33.6, 27.3, 21.3, 20.8; ^{19}F NMR (471 MHz, $CDCl_3$) δ -62.43; IR (ATR) ν_{max} 2926, 1731, 1599, 1471, 1236, 1160, 975, 616 cm^{-1}; HRMS (ESI) Calcd for $C_{29}H_{29}F_3N_4O_4S$: $[M+H]^+$ = 587.1934. Found: 587.1917.



3ap

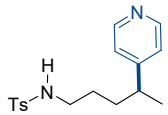
Methyl 2-(5-((4-(5-methyl-3-phenylisoxazol-4-yl)phenyl)sulfonamido)pentan-2-yl)isonicotinate (3ap). Reaction of *N*-fluoro-4-(5-methyl-3-phenylisoxazol-4-yl)-*N*-pentylbenzenesulfonamide **1ap** (40.2 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (1.5:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.2 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **3ao** (38.7 mg, 74%) as a light-yellow oil. ^1H NMR (500 MHz, $CDCl_3$) δ 8.66 (d, J = 5.0 Hz, 1H), 7.82 (d, J = 8.4 Hz, 2H), 7.67 – 7.65 (m, 2H), 7.39 – 7.31 (m, 5H), 7.29 (d, J = 8.4 Hz, 2H), 4.95 (t, J = 6.0 Hz, 1H), 3.94 (s, 3H), 3.00 – 2.93 (m, 3H), 2.48 (s, 3H), 1.80 – 1.74 (m, 1H), 1.65 – 1.61 (m, 1H), 1.52 – 1.46 (m, 1H), 1.38 – 1.35 (m, 1H), 1.27 (d, J = 6.9 Hz, 3H); $^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$) δ 167.2, 166.7, 165.8, 161.1, 149.9, 139.2, 137.9, 135.0, 130.2, 129.7, 128.7, 128.4, 127.4, 120.9, 120.6, 114.5, 52.7, 43.2, 41.2, 33.6, 27.4, 20.9, 11.8; IR (ATR) ν_{max} 2921, 2851, 1729, 1437, 1292, 1158, 740, 607 cm^{-1}; HRMS (ESI) Calcd for $C_{28}H_{29}N_3O_5S$: $[M+H]^+$ = 520.1901. Found: 520.1892.



4a

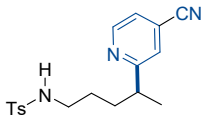
4-Methyl-*N*-(4-(pyridin-2-yl)pentyl)benzenesulfonamide (4a). Reaction of *N*-fluoro-4-methyl-*N*-pentylbenzenesulfonamide **1a** (25.9 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2b** (39.4 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (2:1 ~ 1:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.5 in

1:1 v/v petroleum ether/ethyl acetate) gave compound as a light-yellow oil (21.4 mg, 67% yield; C2 (**4a**, 16.7 mg): C4 (**4a'**, 4.7 mg) = 3.6 : 1). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.41 (d, *J* = 4.6 Hz, 1H), 7.64 (d, *J* = 8.2 Hz, 2H), 7.51 (t, *J* = 7.7 Hz, 1H), 7.19 (d, *J* = 8.0 Hz, 2H), 7.03 – 7.00 (m, 2H), 5.18 – 5.16 (m, 1H), 2.83 – 2.81 (m, 2H), 2.75 – 2.71 (m, 1H), 2.33 (s, 3H), 1.66 – 1.60 (m, 1H), 1.50 – 1.44 (m, 1H), 1.40 – 1.34 (m, 1H), 1.27 – 1.23 (m, 1H), 1.14 (d, *J* = 6.9 Hz, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 165.5, 148.9, 143.1, 137.0, 136.5, 129.5, 127.0, 121.6, 121.2, 43.1, 41.1, 33.7, 27.3, 21.4, 20.8; IR (ATR) ν_{max} 2926, 2867, 1594, 1434, 1323, 1155, 660, 550 cm^{-1}; HRMS (ESI) Calcd for $C_{17}H_{22}N_2O_2S$: $[M+H]^+$ = 319.1475. Found: 319.1488.



4a'

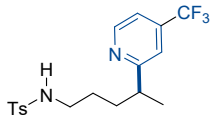
4-Methyl-*N*-(4-(pyridin-4-yl)pentyl)benzenesulfonamide (4a'). Reaction of *N*-fluoro-4-methyl-*N*-pentylbenzenesulfonamide **1a** (25.9 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2b** (39.4 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (2:1 ~ 1:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.2 in 1:1 v/v petroleum ether/ethyl acetate) gave compound as a light-yellow oil (21.4 mg, 67% yield; C2 (**4a**, 16.7 mg): C4 (**4a'**, 4.7 mg) = 3.6 : 1). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.46 (d, *J* = 5.5 Hz, 2H), 7.70 (d, *J* = 8.3 Hz, 2H), 7.28 (d, *J* = 8.0 Hz, 2H), 7.04 (d, *J* = 5.6 Hz, 2H), 4.76 (t, *J* = 6.3 Hz, 1H), 2.89 (q, *J* = 6.7 Hz, 2H), 2.61 (q, *J* = 7.1 Hz, 1H), 2.42 (s, 3H), 1.56 (q, *J* = 7.8 Hz, 2H), 1.43 – 1.38 (m, 1H), 1.31 – 1.27 (m, 1H), 1.19 (d, *J* = 6.9 Hz, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 155.9, 149.6, 143.34, 137.0, 129.7, 127.0, 122.5, 43.0, 39.0, 34.3, 27.5, 21.5, 21.4; IR (ATR) ν_{max} 2925, 2867, 1601, 1417, 1322, 1155, 660, 551 cm^{-1}; HRMS (ESI) Calcd for $C_{17}H_{22}N_2O_2S$: $[M+H]^+$ = 319.1475. Found: 319.1476.



4b

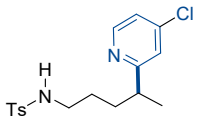
***N*-(4-(4-Cyanopyridin-2-yl)pentyl)-4-methylbenzenesulfonamide (4b).** Reaction of *N*-fluoro-4-methyl-*N*-pentylbenzenesulfonamide **1a** (25.9 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2c** (44.4 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (2:1 v/v petroleum ether/ethyl acetate elution,

R_f = 0.3 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **4b** (25.1 mg, 73%) as a light-yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.68 (d, *J* = 4.9 Hz, 1H), 7.71 (d, *J* = 8.3 Hz, 2H), 7.32 (dd, *J* = 5.0, 1.4 Hz, 1H), 7.30 – 7.27 (m, 3H), 4.86 (t, *J* = 6.1 Hz, 1H), 2.91 – 2.86 (m, 3H), 2.41 (s, 3H), 1.74 – 1.69 (m, 1H), 1.61 – 1.55 (m, 1H), 1.46 – 1.41 (m, 1H), 1.33 – 1.29 (m, 1H), 1.22 (d, *J* = 6.9 Hz, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 167.4, 150.2, 143.3, 136.9, 129.6, 127.0, 123.4, 122.7, 120.7, 116.7, 43.0, 41.3, 33.3, 27.4, 21.5, 20.5; IR (ATR) ν_{max} 2929, 2238, 1595, 1400, 1322, 1155, 660, 549 cm^{-1}; HRMS (ESI) Calcd for $C_{18}H_{21}N_3O_2S$: $[M+H]^+$ = 344.1427. Found: 344.1418.



4c

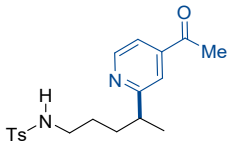
4-Methyl-*N*-(4-(4-(trifluoromethyl)pyridin-2-yl)pentyl)benzenesulfonamide (4c). Reaction of *N*-fluoro-4-methyl-*N*-pentylbenzenesulfonamide **1a** (25.9 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2d** (53.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (3:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.2 in 3:1 v/v petroleum ether/ethyl acetate) gave compound **4c** (28.6 mg, 74%) as a light-yellow oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.66 (d, *J* = 5.1 Hz, 1H), 7.70 (d, *J* = 8.3 Hz, 2H), 7.30 (d, *J* = 5.1 Hz, 1H), 7.27 – 7.25 (m, 3H), 5.03 (t, *J* = 6.1 Hz, 1H), 2.91 – 2.87 (m, 3H), 2.39 (s, 3H), 1.75 – 1.70 (m, 1H), 1.61 – 1.56 (m, 1H), 1.46 – 1.41 (m, 1H), 1.32 – 1.28 (m, 1H), 1.22 (d, *J* = 6.9 Hz, 3H); ^{13}C{^{1}H} NMR (151 MHz, $CDCl_3$) δ 167.3, 150.1, 143.3, 138.6 (q, *J* = 33.7 Hz), 136.9, 129.6, 127.0, 122.8 (q, *J* = 273.2 Hz), 117.2, 117.2, 116.9, 116.9, 43.0, 41.4, 33.4, 27.4, 21.4, 20.6; ^{19}F NMR (565 MHz, Chloroform-d) δ -64.74; IR (ATR) ν_{max} 2929, 1599, 1411, 1324, 1133, 664, 549 cm^{-1}; HRMS (ESI) Calcd for $C_{18}H_{21}F_3N_2O_2S$: $[M+H]^+$ = 387.1349. Found: 387.1350.



4d

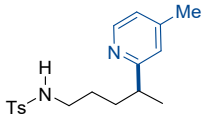
***N*-(4-(4-Chloropyridin-2-yl)pentyl)-4-methylbenzenesulfonamide (4d).** Reaction of *N*-fluoro-4-methyl-*N*-pentylbenzenesulfonamide **1a** (25.9 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2e** (46.2 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (2:1 v/v petroleum ether/ethyl acetate elution,

R_f = 0.2 in 3:1 v/v petroleum ether/ethyl acetate) gave compound **4d** (25.2 mg, 71%) as a light-yellow oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.31 (d, J = 5.3 Hz, 1H), 7.64 (d, J = 8.2 Hz, 2H), 7.20 (d, J = 8.0 Hz, 2H), 7.04 – 7.01 (m, 2H), 5.12 (t, J = 6.1 Hz, 1H), 2.83 – 2.80 (m, 2H), 2.72 – 2.68 (m, 1H), 2.33 (s, 3H), 1.64 – 1.59 (m, 1H), 1.50 – 1.45 (m, 1H), 1.38 – 1.34 (m, 1H), 1.26 – 1.21 (m, 1H), 1.12 (d, J = 6.9 Hz, 3H); ^{13}C{^{1}H} NMR (151 MHz, $CDCl_3$) δ 167.4, 149.9, 144.3, 143.2, 136.9, 129.6, 127.0, 121.9, 121.6, 43.0, 41.1, 33.4, 27.3, 21.4, 20.6; IR (ATR) ν_{max} 2927, 2868, 1575, 1467, 1322, 1154, 659, 549 cm^{-1}; HRMS (ESI) Calcd for $C_{17}H_{21}ClN_2O_2S$: $[M+H]^+$ = 353.1085. Found: 353.1076.



4e

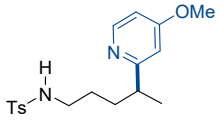
***N*-(4-(4-Acetylpyridin-2-yl)pentyl)-4-methylbenzenesulfonamide (4e).** Reaction of *N*-fluoro-4-methyl-*N*-pentylbenzenesulfonamide **1a** (25.9 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2f** (47.8 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (2:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.2 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **4e** (18.5 mg, 51%) as a light-yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.61 (d, J = 5.7 Hz, 1H), 7.63 (d, J = 8.3 Hz, 2H), 7.45 (dd, J = 3.0, 1.1 Hz, 2H), 7.20 (d, J = 7.9 Hz, 2H), 4.97 (t, J = 6.1 Hz, 1H), 2.82 (q, J = 6.7 Hz, 3H), 2.54 (s, 3H), 2.34 (s, 3H), 1.69 – 1.65 (m, 1H), 1.54 – 1.50 (m, 1H), 1.40 – 1.35 (m, 1H), 1.27 – 1.24 (m, 1H), 1.17 (d, J = 6.9 Hz, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 197.7, 167.2, 150.2, 143.3, 143.2, 136.9, 129.6, 127.0, 119.1, 119.0, 43.0, 41.3, 33.5, 27.4, 26.7, 21.4, 20.8; IR (ATR) ν_{max} 2928, 1693, 1406, 1322, 1154, 659, 549 cm^{-1}; HRMS (ESI) Calcd for $C_{19}H_{24}N_2O_3S$: $[M+H]^+$ = 361.1580. Found: 361.1576.



4f

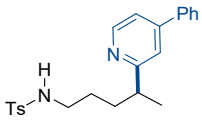
4-Methyl-*N*-(4-(4-methylpyridin-2-yl)pentyl)benzenesulfonamide (4f). Reaction of *N*-fluoro-4-methyl-*N*-pentylbenzenesulfonamide **1a** (25.9 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2g** (42.2 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (2:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.2 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **4f** (16.5 mg, 50%) as a light-

yellow oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.34 (d, J = 4.9 Hz, 1H), 7.71 (d, J = 8.2 Hz, 2H), 7.27 (d, J = 8.3 Hz, 2H), 6.93 – 6.90 (m, 2H), 5.38 (t, J = 6.0 Hz, 1H), 2.89 (q, J = 6.4 Hz, 2H), 2.79 – 2.72 (m, 1H), 2.41 (s, 3H), 2.31 (s, 3H), 1.72 – 1.66 (m, 1H), 1.58 – 1.51 (m, 1H), 1.46 – 1.40 (m, 1H), 1.35 – 1.31 (m, 1H), 1.20 (d, J = 6.9 Hz, 3H); ^{13}C{^{1}H} NMR (75 MHz, $CDCl_3$) δ 165.3, 148.6, 147.6, 143.0, 137.0, 129.5, 127.0, 122.4, 122.3, 43.1, 40.9, 33.7, 27.3, 21.4, 21.0, 20.9; IR (ATR) ν_{max} 2925, 2866, 1606, 1451, 1323, 1155, 659, 549 cm^{-1}; HRMS (ESI) Calcd for $C_{18}H_{24}N_2O_2S$: $[M+H]^+$ = 333.1631. Found: 333.1640.



4g

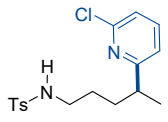
***N*-(4-(4-Methoxypyridin-2-yl)pentyl)-4-methylbenzenesulfonamide (4g).** Reaction of *N*-fluoro-4-methyl-*N*-pentylbenzenesulfonamide **1a** (25.9 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2h** (45.4 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (2:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.2 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **4g** (19.7 mg, 57%) as a light-yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.32 (d, J = 5.8 Hz, 1H), 7.70 (d, J = 8.3 Hz, 2H), 7.27 – 7.25 (m, 2H), 6.64 (dd, J = 5.7, 2.5 Hz, 1H), 6.60 (d, J = 2.5 Hz, 1H), 5.18 (s, 1H), 3.83 (s, 3H), 2.90 – 2.88 (m, 2H), 2.77 – 2.73 (m, 1H), 2.40 (s, 3H), 1.72 – 1.67 (m, 1H), 1.55 – 1.51 (m, 1H), 1.46 – 1.41 (m, 1H), 1.34 – 1.31 (m, 1H), 1.20 (d, J = 6.9 Hz, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 167.2, 166.2, 150.2, 143.1, 137.1, 129.6, 127.0, 107.7, 107.4, 55.0, 43.1, 41.1, 33.7, 27.3, 21.5, 20.9; IR (ATR) ν_{max} 2923, 2853, 1597, 1459, 1305, 1155, 759, 550 cm^{-1}; HRMS (ESI) Calcd for $C_{18}H_{24}N_2O_3S$: $[M+H]^+$ = 349.1580. Found: 349.1573.



4h

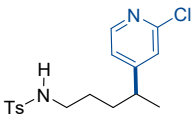
4-Methyl-*N*-(4-(4-phenylpyridin-2-yl)pentyl)benzenesulfonamide (4h). Reaction of *N*-fluoro-4-methyl-*N*-pentylbenzenesulfonamide **1a** (25.9 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2i** (54.6 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (2:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.3 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **4g** (28.0 mg, 71%) as a light-yellow oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.54 (d, J = 5.2 Hz, 1H), 7.70 (d, J = 8.2 Hz, 2H),

7.61 (d, J = 7.0 Hz, 2H), 7.48 (t, J = 7.4 Hz, 2H), 7.43 (t, J = 7.3 Hz, 1H), 7.31 – 7.29 (m, 2H), 7.24 (d, J = 8.0 Hz, 2H), 5.17 (t, J = 6.0 Hz, 1H), 2.93 – 2.87 (m, 3H), 2.38 (s, 3H), 1.79 – 1.75 (m, 1H), 1.63 – 1.58 (m, 1H), 1.50 – 1.45 (m, 1H), 1.37 – 1.34 (m, 1H), 1.26 (d, J = 6.9 Hz, 3H); $^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) δ 166.1, 149.4, 149.0, 143.1, 138.3, 137.0, 129.6, 129.0, 128.9, 127.0, 127.0, 119.6, 119.4, 43.1, 41.3, 33.7, 27.4, 21.4, 21.0; IR (ATR) ν_{max} 2927, 2865, 1598, 1450, 1323, 1155, 762, 550 cm^{-1}; HRMS (ESI) Calcd for $C_{23}H_{26}N_2O_2S$: $[M+H]^+$ = 395.1788. Found: 395.1781.



4i

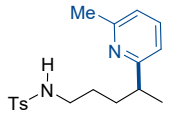
***N*-(4-(6-Chloropyridin-2-yl)pentyl)-4-methylbenzenesulfonamide (4i).** Reaction of *N*-fluoro-4-methyl-*N*-pentylbenzenesulfonamide **1a** (25.9 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2j** (46.2 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (4:1 ~ 2:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.2 in 5:1 v/v petroleum ether/ethyl acetate) gave compound as a light-yellow oil (23.9 mg, 68% yield; C6 (**4i**, 13.6 mg): C4 (**4i'**, 10.3 mg) = 1.3 : 1). ^1H NMR (500 MHz, $CDCl_3$) δ 7.71 (d, J = 8.2 Hz, 2H), 7.54 (t, J = 7.7 Hz, 1H), 7.28 (d, J = 8.1 Hz, 2H), 7.13 (d, J = 7.8 Hz, 1H), 7.00 (d, J = 7.6 Hz, 1H), 4.55 (t, J = 6.2 Hz, 1H), 2.90 (q, J = 6.8 Hz, 2H), 2.81 – 2.76 (m, 1H), 2.42 (s, 3H), 1.73 – 1.69 (m, 1H), 1.56 – 1.53 (m, 1H), 1.47 – 1.43 (m, 1H), 1.34 – 1.32 (m, 1H), 1.21 (d, J = 6.9 Hz, 3H); $^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$) δ 166.9, 150.8, 143.3, 139.0, 136.9, 129.7, 127.1, 121.7, 119.9, 43.1, 41.1, 33.5, 27.5, 21.5, 20.6; IR (ATR) ν_{max} 2924, 1585, 1436, 1323, 1156, 1093, 663, 551 cm^{-1}; HRMS (ESI) Calcd for $C_{17}H_{21}ClN_2O_2S$: $[M+H]^+$ = 353.1085. Found: 353.1081.



4i'

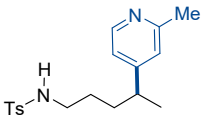
***N*-(4-(2-Chloropyridin-4-yl)pentyl)-4-methylbenzenesulfonamide (4i').** Reaction of *N*-fluoro-4-methyl-*N*-pentylbenzenesulfonamide **1a** (25.9 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2j** (46.2 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (4:1 ~ 2:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.1 in 5:1 v/v petroleum ether/ethyl acetate) gave compound as a light-yellow oil

(23.9 mg, 68% yield; C6 (**4i**, 13.6 mg): C4 (**4i'**, 10.3 mg) = 1.3 : 1). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.25 (d, *J* = 5.1 Hz, 1H), 7.70 (d, *J* = 8.3 Hz, 2H), 7.29 (d, *J* = 8.0 Hz, 2H), 7.07 (s, 1H), 6.97 (d, *J* = 6.4 Hz, 1H), 4.45 (t, *J* = 6.4 Hz, 1H), 2.91 (q, *J* = 6.8 Hz, 2H), 2.62 (q, *J* = 7.0 Hz, 1H), 2.42 (s, 3H), 1.57 – 1.54 (m, 2H), 1.43 – 1.39 (m, 1H), 1.32 – 1.29 (m, 1H), 1.20 (d, *J* = 6.9 Hz, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 159.3, 151.7, 149.7, 143.5, 136.9, 129.7, 127.0, 122.8, 121.2, 43.0, 38.9, 34.1, 27.5, 21.5, 21.2; IR (ATR) ν_{max} 2922, 1593, 1458, 1322, 1155, 1089, 661, 550 cm^{-1}; HRMS (ESI) Calcd for $C_{17}H_{21}ClN_2O_2S$: $[M+H]^+$ = 353.1085. Found: 353.1080.



4j

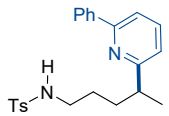
4-Methyl-*N*-(4-(6-methylpyridin-2-yl)pentyl)benzenesulfonamide (4j). Reaction of *N*-fluoro-4-methyl-*N*-pentylbenzenesulfonamide **1a** (25.9 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2k** (42.2 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (2:1 ~ 1:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.3 in 2:1 v/v petroleum ether/ethyl acetate) gave compound as a light-yellow oil (20.9 mg, 63% yield; C6 (**4j**, 15.8 mg): C4 (**4j'**, 5.1 mg) = 3.1 : 1). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.65 (d, *J* = 8.2 Hz, 2H), 7.40 (t, *J* = 7.7 Hz, 1H), 7.21 – 7.19 (m, 2H), 6.88 (d, *J* = 7.6 Hz, 1H), 6.81 (d, *J* = 7.7 Hz, 1H), 5.28 (d, *J* = 5.1 Hz, 1H), 2.87 – 2.80 (m, 2H), 2.72 – 2.68 (m, 1H), 2.45 (s, 3H), 2.34 (s, 3H), 1.62 – 1.59 (m, 1H), 1.48 – 1.43 (m, 1H), 1.35 – 1.27 (m, 2H), 1.12 (d, *J* = 7.0 Hz, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 165.0, 157.5, 143.1, 137.1, 136.7, 129.5, 127.0, 120.7, 117.8, 43.1, 40.9, 34.1, 27.0, 24.4, 21.4, 20.9; IR (ATR) ν_{max} 2925, 2866, 1593, 1453, 1322, 1154, 1092, 660, 549 cm^{-1}; HRMS (ESI) Calcd for $C_{18}H_{24}N_2O_2S$: $[M+H]^+$ = 333.1631. Found: 333.1643.



4j'

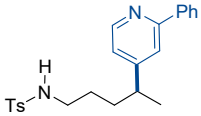
4-Methyl-*N*-(4-(2-methylpyridin-4-yl)pentyl)benzenesulfonamide (4j'). Reaction of *N*-fluoro-4-methyl-*N*-pentylbenzenesulfonamide **1a** (25.9 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2k** (42.2 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (2:1 ~ 1:1 v/v petroleum ether/ethyl acetate

elution, R_f = 0.1 in 2:1 v/v petroleum ether/ethyl acetate) gave compound as a light-yellow oil (20.9 mg, 63% yield; C6 (**4j**, 15.8 mg): C4 (**4j'**, 5.1 mg) = 3.1 : 1). ^{1}H NMR (500 MHz, $CDCl_3$) 8.26 (d, *J* = 5.1 Hz, 1H), 7.63 (d, *J* = 8.1 Hz, 2H), 7.21 (d, *J* = 9.2 Hz, 2H), 6.82 (s, 1H), 6.76 (d, *J* = 5.1 Hz, 1H), 4.73 (t, *J* = 6.4 Hz, 1H), 2.82 (q, *J* = 6.7 Hz, 2H), 2.49 (q, *J* = 7.0 Hz, 1H), 2.43 (s, 3H), 2.35 (s, 3H), 1.47 (q, *J* = 7.7 Hz, 2H), 1.35 – 1.31 (m, 1H), 1.25 – 1.20 (m, 1H), 1.10 (d, *J* = 6.9 Hz, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 158.3, 156.1, 149.0, 143.3, 137.0, 129.6, 127.0, 122.0, 119.5, 43.0, 38.9, 34.2, 27.6, 24.3, 21.5, 21.4; IR (ATR) ν_{max} 2923, 1604, 1454, 1322, 1154, 1092, 659, 550 cm^{-1}; HRMS (ESI) Calcd for $C_{18}H_{24}N_2O_2S$: $[M+H]^+$ = 333.1631. Found: 333.1639.



4k

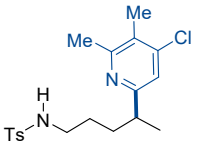
4-Methyl-*N*-(4-(6-phenylpyridin-2-yl)pentyl)benzenesulfonamide (4k). Reaction of *N*-fluoro-4-methyl-*N*-pentylbenzenesulfonamide **1a** (25.9 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2l** (54.6 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (5:1 ~ 3:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.6 in 3:1 v/v petroleum ether/ethyl acetate) gave compound as a light-yellow oil (27.7 mg, 70% yield; C6 (**4k**, 9.2 mg): C4 (**4k'**, 18.5 mg) = 1 : 2). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.01 (dd, *J* = 8.3, 1.3 Hz, 2H), 7.68 (d, *J* = 8.3 Hz, 2H), 7.64 (d, *J* = 7.7 Hz, 1H), 7.54 (d, *J* = 8.6 Hz, 1H), 7.47 (t, *J* = 7.4 Hz, 2H), 7.41 (t, *J* = 7.3 Hz, 1H), 7.24 (d, *J* = 7.9 Hz, 2H), 7.02 (d, *J* = 8.4 Hz, 1H), 4.78 (t, *J* = 6.1 Hz, 1H), 2.94 – 2.90 (m, 3H), 2.39 (s, 3H), 1.83 – 1.79 (m, 1H), 1.64 – 1.59 (m, 1H), 1.48 – 1.44 (m, 1H), 1.40 – 1.37 (m, 1H), 1.28 (d, *J* = 6.9 Hz, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 165.4, 156.4, 143.1, 139.6, 137.0, 137.0, 129.6, 128.7, 128.6, 127.0, 126.9, 119.8, 117.8, 43.2, 41.3, 33.9, 27.4, 21.4, 20.9; IR (ATR) ν_{max} 2924, 2854, 1570, 1445, 1323, 1155, 662, 551 cm^{-1}; HRMS (ESI) Calcd for $C_{23}H_{26}N_2O_2S$: $[M+H]^+$ = 395.1788. Found: 395.1791.



4k'

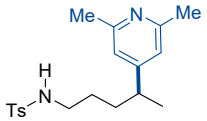
4-Methyl-*N*-(4-(2-phenylpyridin-4-yl)pentyl)benzenesulfonamide (4k'). Reaction of *N*-fluoro-4-methyl-*N*-pentylbenzenesulfonamide **1a** (25.9 mg, 0.1 mmol) with the *N*-

methoxypyridinium tetrafluoroborate **2l** (54.6 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (5:1 ~ 3:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.3 in 3:1 v/v petroleum ether/ethyl acetate) gave compound as a light-yellow oil (27.7 mg, 70% yield; C6 (**4k**, 9.2 mg): C4 (**4k'**, 18.5 mg) = 1 : 2). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.57 (d, *J* = 5.1 Hz, 1H), 7.95 (d, *J* = 8.6 Hz, 2H), 7.69 (d, *J* = 8.3 Hz, 2H), 7.49 – 7.46 (m, 3H), 7.41 (t, *J* = 7.3 Hz, 1H), 7.26 (d, *J* = 2.2 Hz, 2H), 6.99 (d, *J* = 6.6 Hz, 1H), 4.42 (t, *J* = 6.2 Hz, 1H), 2.91 (q, *J* = 6.8 Hz, 2H), 2.69 (q, *J* = 7.1 Hz, 1H), 2.39 (s, 3H), 1.63 – 1.59 (m, 2H), 1.46 – 1.42 (m, 1H), 1.35 – 1.31 (m, 1H), 1.25 (d, *J* = 6.9 Hz, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 157.6, 156.6, 149.7, 143.4, 139.4, 136.9, 129.7, 128.9, 128.7, 127.0, 127.0, 120.9, 119.5, 43.1, 39.3, 34.3, 27.6, 21.5, 21.5; IR (ATR) ν_{max} 2921, 2852, 1599, 1447, 1323, 1155, 661, 551 cm^{-1}; HRMS (ESI) Calcd for $C_{23}H_{26}N_2O_2S$: $[M+H]^+$ = 395.1788. Found: 395.1795.



4l

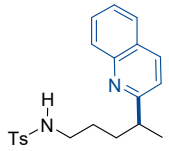
***N*-(4-(4-Chloro-5,6-dimethylpyridin-2-yl)pentyl)-4-methylbenzenesulfonamide (4l).** Reaction of *N*-fluoro-4-methyl-*N*-pentylbenzenesulfonamide **1a** (25.9 mg, 0.1 mmol) with the *N*-methoxypyridinium tetrafluoroborate **2m** (51.8 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (3:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.2 in 5:1 v/v petroleum ether/ethyl acetate) gave compound **4m** (23.7 mg, 62%) as a light-yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.65 (d, *J* = 8.3 Hz, 2H), 7.20 (d, *J* = 8.3 Hz, 2H), 6.86 (s, 1H), 5.24 (t, *J* = 6.0 Hz, 1H), 2.89 – 2.79 (m, 2H), 2.67 – 2.63 (m, 1H), 2.45 (s, 3H), 2.34 (s, 3H), 2.23 (s, 3H), 1.60 – 1.55 (m, 1H), 1.47 – 1.42 (m, 1H), 1.36 – 1.28 (m, 2H), 1.09 (d, *J* = 6.9 Hz, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 162.8, 157.8, 144.5, 143.1, 137.0, 129.6, 127.2, 127.0, 119.4, 43.0, 40.3, 34.0, 27.0, 23.4, 21.5, 20.7, 15.2; IR (ATR) ν_{max} 2924, 2856, 1555, 1453, 1322, 1156, 660, 549 cm^{-1}; HRMS (ESI) Calcd for $C_{19}H_{25}ClN_2O_2S$: $[M+H]^+$ = 381.1398. Found: 381.1390.



4m

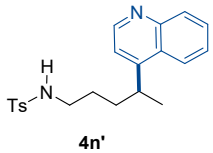
***N*-(4-(2,6-Dimethylpyridin-4-yl)pentyl)-4-methylbenzenesulfonamide (4m).** Reaction of *N*-fluoro-4-methyl-*N*-pentylbenzenesulfonamide **1a** (25.9 mg, 0.1 mmol) with the *N*-

methoxypyridinium tetrafluoroborate **2n** (45.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (2:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.2 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **4m** (22.6 mg, 65%) as a light-yellow oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.70 (d, *J* = 8.3 Hz, 2H), 7.27 (d, *J* = 7.9 Hz, 2H), 6.69 (s, 2H), 4.67 (t, *J* = 5.9 Hz, 1H), 2.88 (q, *J* = 6.8 Hz, 2H), 2.52 – 2.49 (m, 1H), 2.46 (s, 6H), 2.41 (s, 3H), 1.52 (q, *J* = 8.0, 7.6 Hz, 2H), 1.40 – 1.37 (m, 1H), 1.30 – 1.27 (m, 1H), 1.15 (d, *J* = 6.9 Hz, 3H); ^{13}C{^{1}H} NMR (151 MHz, $CDCl_3$) δ 157.6, 156.4, 143.4, 136.9, 129.6, 127.0, 119.0, 43.1, 38.9, 34.2, 27.6, 24.3, 21.5, 21.4; IR (ATR) ν_{max} 2924, 2855, 1608, 1453, 1324, 1157, 661, 551 cm^{-1}; HRMS (ESI) Calcd for $C_{19}H_{26}N_2O_2S$: $[M+H]^+$ = 347.1788. Found: 347.1781.



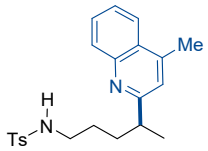
4n

4-Methyl-*N*-(4-(quinolin-2-yl)pentyl)benzenesulfonamide (4n). Reaction of *N*-fluoro-4-methyl-*N*-pentylbenzenesulfonamide **1a** (25.9 mg, 0.1 mmol) with the *N*-methoxyquinolinium tetrafluoroborate **2o** (49.4 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (4:1 ~ 2:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.5 in 2:1 v/v petroleum ether/ethyl acetate) gave compound as a light-yellow oil (22.9 mg, 62% yield; C2 (**4n**, 13.7 mg): C4 (**4n'**, 9.2 mg) = 1.5 : 1). ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.08 (dd, *J* = 12.1, 8.5 Hz, 2H), 7.77 (d, *J* = 8.1 Hz, 1H), 7.70 – 7.67 (m, 3H), 7.50 (t, *J* = 7.9 Hz, 1H), 7.24 (d, *J* = 8.5 Hz, 1H), 7.20 (d, *J* = 8.0 Hz, 2H), 5.44 (t, *J* = 5.8 Hz, 1H), 3.03 – 3.00 (m, 1H), 2.97 – 2.94 (m, 1H), 2.90 – 2.87 (m, 1H), 2.36 (s, 3H), 1.85 – 1.81 (m, 1H), 1.66 – 1.62 (m, 1H), 1.47 – 1.43 (m, 1H), 1.37 – 1.34 (m, 1H), 1.30 (d, *J* = 7.0 Hz, 3H); ^{13}C{^{1}H} NMR (151 MHz, $CDCl_3$) δ 165.9, 147.5, 143.0, 137.0, 136.9, 129.5, 129.5, 128.8, 127.4, 127.0, 126.9, 125.9, 119.6, 43.1, 41.8, 33.7, 27.1, 21.4, 21.0; IR (ATR) ν_{max} 2923, 2854, 1599, 1454, 1321, 1154, 661, 550 cm^{-1}; HRMS (ESI) Calcd for $C_{21}H_{24}N_2O_2S$: $[M+H]^+$ = 369.1631. Found: 369.1626.



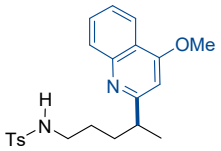
4n'

4-Methyl-*N*-(4-(quinolin-4-yl)pentyl)benzenesulfonamide (4n'). Reaction of *N*-fluoro-4-methyl-*N*-pentylbenzenesulfonamide **1a** (25.9 mg, 0.1 mmol) with the *N*-methoxyquinolinium tetrafluoroborate **2o** (49.4 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (4:1 ~ 2:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.2 in 2:1 v/v petroleum ether/ethyl acetate) gave compound as a light-yellow oil (22.9 mg, 62% yield; C2 (**4n**, 13.7 mg): C4 (**4n'**, 9.2 mg) = 1.5 : 1). ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.80 (d, *J* = 4.6 Hz, 1H), 8.11 (d, *J* = 8.1 Hz, 1H), 8.03 (d, *J* = 8.4 Hz, 1H), 7.71 – 7.67 (m, 3H), 7.55 (t, *J* = 7.6 Hz, 1H), 7.24 (d, *J* = 8.0 Hz, 2H), 7.19 (d, *J* = 4.6 Hz, 1H), 4.61 (t, *J* = 6.3 Hz, 1H), 3.55 (q, *J* = 7.0 Hz, 1H), 2.92 (q, *J* = 6.7 Hz, 2H), 2.39 (s, 3H), 1.80 – 1.76 (m, 1H), 1.72 – 1.69 (m, 1H), 1.49 – 1.45 (m, 1H), 1.42 – 1.39 (m, 1H), 1.32 (d, *J* = 6.9 Hz, 3H); ^{13}C{^{1}H} NMR (151 MHz, $CDCl_3$) δ 152.9, 150.2, 148.3, 143.4, 136.9, 130.3, 129.7, 129.0, 127.1, 127.0, 126.4, 122.8, 117.5, 43.1, 34.0, 32.9, 27.6, 21.5, 21.3; IR (ATR) ν_{max} 2922, 2853, 1589, 1457, 1323, 1155, 762, 550 cm^{-1}; HRMS (ESI) Calcd for $C_{21}H_{24}N_2O_2S$: $[M+H]^+$ = 369.1631. Found: 369.1621.



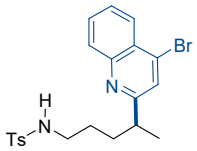
4o

4-Methyl-*N*-(4-(4-methylquinolin-2-yl)pentyl)benzenesulfonamide (4o). Reaction of *N*-fluoro-4-methyl-*N*-pentylbenzenesulfonamide **1a** (25.9 mg, 0.1 mmol) with the *N*-methoxyquinolinium tetrafluoroborate **2p** (52.2 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (2:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.3 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **4o** (25.7 mg, 67%) as a light-yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.13 (d, *J* = 8.4 Hz, 1H), 7.95 (d, *J* = 8.3 Hz, 1H), 7.71 – 7.67 (m, 3H), 7.53 (t, *J* = 7.6 Hz, 1H), 7.20 (d, *J* = 8.0 Hz, 2H), 7.10 (s, 1H), 5.60 (t, *J* = 6.0 Hz, 1H), 3.03 – 2.94 (m, 2H), 2.91 – 2.86 (m, 1H), 2.68 (s, 3H), 2.36 (s, 3H), 1.85 – 1.80 (m, 1H), 1.67 – 1.61 (m, 1H), 1.48 – 1.44 (m, 1H), 1.39 – 1.35 (m, 1H), 1.29 (d, *J* = 7.0 Hz, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 165.5, 146.8, 145.3, 143.0, 137.0, 129.5, 129.4, 129.0, 127.0, 127.0, 125.8, 123.6, 120.2, 43.0, 41.4, 33.7, 27.0, 21.4, 21.0, 18.9; IR (ATR) ν_{max} 2925, 2855, 1602, 1450, 1323, 1157, 761, 551 cm^{-1}; HRMS (ESI) Calcd for $C_{22}H_{26}N_2O_2S$: $[M+H]^+$ = 383.1788. Found: 383.1795.



4p

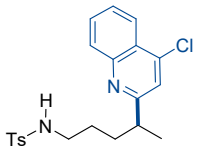
***N*-(4-(4-Methoxyquinolin-2-yl)pentyl)-4-methylbenzenesulfonamide (4p).** Reaction of *N*-fluoro-4-methyl-*N*-pentylbenzenesulfonamide **1a** (25.9 mg, 0.1 mmol) with the *N*-methoxyquinolinium tetrafluoroborate **2q** (55.4 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (2:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.3 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **4p** (22.0 mg, 55%) as a light-yellow oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.14 (d, *J* = 9.3 Hz, 1H), 8.03 (d, *J* = 8.2 Hz, 1H), 7.68 – 7.66 (m, 3H), 7.48 – 7.43 (m, 1H), 7.18 (d, *J* = 7.9 Hz, 2H), 6.57 (s, 1H), 5.71 (t, *J* = 5.9 Hz, 1H), 4.03 (s, 3H), 2.99 – 2.95 (m, 2H), 2.90 – 2.86 (m, 1H), 2.36 (s, 3H), 1.86 – 1.82 (m, 1H), 1.66 – 1.62 (m, 1H), 1.48 – 1.44 (m, 1H), 1.40 – 1.36 (m, 1H), 1.29 (d, *J* = 7.0 Hz, 3H); ^{13}C{^{1}H} NMR (151 MHz, $CDCl_3$) δ 167.2, 162.9, 148.16, 143.0, 137.0, 129.9, 129.5, 128.2, 127.0, 125.1, 121.6, 120.2, 98.0, 55.6, 43.0, 42.2, 33.8, 27.0, 21.4, 21.1; IR (ATR) ν_{max} 2923, 2853, 1596, 1457, 1322, 1157, 662, 551 cm^{-1}; HRMS (ESI) Calcd for $C_{22}H_{26}N_2O_3S$: $[M+H]^+$ = 399.1737. Found: 399.1730.



4q

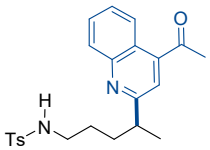
***N*-(4-(4-Bromoquinolin-2-yl)pentyl)-4-methylbenzenesulfonamide (4q).** Reaction of *N*-fluoro-4-methyl-*N*-pentylbenzenesulfonamide **1a** (25.9 mg, 0.1 mmol) with the *N*-methoxyquinolinium tetrafluoroborate **2r** (64.8 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (3:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.3 in 3:1 v/v petroleum ether/ethyl acetate) gave compound **4q** (22.6 mg, 51%) as a light-yellow oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.12 (d, *J* = 9.3 Hz, 1H), 8.05 (d, *J* = 8.4 Hz, 1H), 7.73 (t, *J* = 8.3 Hz, 1H), 7.67 (d, *J* = 8.3 Hz, 2H), 7.58 (t, *J* = 8.2 Hz, 1H), 7.52 (s, 1H), 7.20 (d, *J* = 7.9 Hz, 2H), 5.31 (t, *J* = 5.9 Hz, 1H), 2.97 – 2.94 (m, 2H), 2.91 – 2.88 (m, 1H), 2.36 (s, 3H), 1.83 – 1.79 (m, 1H), 1.66 – 1.61 (m, 1H), 1.48 – 1.43 (m, 1H), 1.38 – 1.34 (m, 1H), 1.28 (d, *J* = 7.0 Hz, 3H); ^{13}C{^{1}H} NMR (151 MHz, $CDCl_3$) δ 165.9, 148.2, 143.1, 136.9, 134.5, 130.4, 129.5, 129.3, 127.1, 127.0, 126.5, 126.4, 123.7, 43.0, 41.6, 33.4, 27.1, 21.4, 20.8; IR

(ATR) ν_{max} 2924, 1584, 1491, 1322, 1155, 1092, 761, 549 cm^{-1}; HRMS (ESI) Calcd for $C_{21}H_{23}BrN_2O_2S$: $[M+H]^+$ = 447.0736. Found: 447.0730.



4r

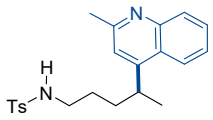
***N*-(4-(4-Chloroquinolin-2-yl)pentyl)-4-methylbenzenesulfonamide (4r).** Reaction of *N*-fluoro-4-methyl-*N*-pentylbenzenesulfonamide **1a** (25.9 mg, 0.1 mmol) with the *N*-methoxyquinolinium tetrafluoroborate **2s** (56.2 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (3:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.3 in 3:1 v/v petroleum ether/ethyl acetate) gave compound **4r** (21.4 mg, 53%) as a light-yellow oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.16 (d, *J* = 9.3 Hz, 1H), 8.07 (d, *J* = 8.4 Hz, 1H), 7.73 (t, *J* = 8.4 Hz, 1H), 7.68 (d, *J* = 8.3 Hz, 2H), 7.58 (t, *J* = 8.2 Hz, 1H), 7.31 (s, 1H), 7.19 (d, *J* = 7.9 Hz, 2H), 5.39 (t, *J* = 5.9 Hz, 1H), 2.97 – 2.94 (m, 2H), 2.91 – 2.87 (m, 1H), 2.35 (s, 3H), 1.83 – 1.78 (m, 1H), 1.65 – 1.61 (m, 1H), 1.47 – 1.42 (m, 1H), 1.37 – 1.34 (m, 1H), 1.28 (d, *J* = 7.0 Hz, 3H); ^{13}C{^{1}H} NMR (151 MHz, $CDCl_3$) δ 166.0, 148.3, 143.1, 142.9, 136.9, 130.4, 129.5, 129.2, 127.0, 126.8, 125.0, 123.8, 119.8, 43.0, 41.8, 33.4, 27.1, 21.4, 20.8; IR (ATR) ν_{max} 2926, 1589, 1494, 1322, 1155, 1092, 762, 549 cm^{-1}; HRMS (ESI) Calcd for $C_{21}H_{23}ClN_2O_2S$: $[M+H]^+$ = 403.1242. Found: 403.1236.



4s

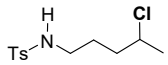
***N*-(4-(4-acetylquinolin-2-yl)pentyl)-4-methylbenzenesulfonamide (4s).** Reaction of *N*-fluoro-4-methyl-*N*-pentylbenzenesulfonamide **1a** (25.9 mg, 0.1 mmol) with the *N*-methoxyquinolinium tetrafluoroborate **2t** (57.8 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (3:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.4 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **4r** (20.6 mg, 50%) as a light-yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.34 (d, *J* = 9.4 Hz, 1H), 8.11 (d, *J* = 8.5 Hz, 1H), 7.73 (t, *J* = 8.4 Hz, 1H), 7.66 (d, *J* = 8.3 Hz, 2H), 7.57 (t, *J* = 8.3 Hz, 1H), 7.43 (s, 1H), 7.20 (d, *J* = 8.0 Hz, 2H), 5.15 (t, *J* = 5.5 Hz, 1H), 3.09 – 3.04 (m, 1H), 2.95 – 2.89 (m, 2H), 2.73 (s, 3H), 2.36 (s, 3H), 1.90 – 1.87 (m, 1H), 1.71 – 1.66 (m, 1H), 1.51 – 1.47 (m, 1H), 1.41 – 1.37

(m, 1H), 1.33 (d, J = 7.0 Hz, 3H); $^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$) δ 201.7, 165.4, 148.6, 143.8, 143.2, 136.9, 129.9, 129.6, 129.5, 127.5, 127.0, 125.2, 122.3, 118.6, 43.0, 41.9, 33.4, 30.2, 27.2, 21.4, 20.9; IR (ATR) ν_{max} 2925, 1690, 1594, 1454, 1323, 1155, 763, 549 cm^{-1}; HRMS (ESI) Calcd for $C_{23}H_{26}N_2O_3S$: $[M+H]^+$ = 411.1737. Found: 411.1738.



4t

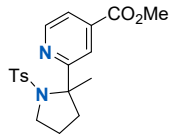
4-Methyl-*N*-(4-(2-methylquinolin-4-yl)pentyl)benzenesulfonamide (4t). Reaction of *N*-fluoro-4-methyl-*N*-pentylbenzenesulfonamide **1a** (25.9 mg, 0.1 mmol) with the *N*-methoxyquinolinium tetrafluoroborate **2u** (52.2 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (2:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.2 in 2:1 v/v petroleum ether/ethyl acetate) gave compound **4r** (27.0 mg, 71%) as a light-yellow oil. ^1H NMR (500 MHz, $CDCl_3$) δ 8.02 (d, J = 8.0 Hz, 1H), 7.95 (d, J = 8.3 Hz, 1H), 7.68 – 7.66 (m, 2H), 7.63 (d, J = 8.3 Hz, 1H), 7.47 (t, J = 7.6 Hz, 1H), 7.22 (d, J = 8.0 Hz, 2H), 7.09 (s, 1H), 4.76 (t, J = 6.3 Hz, 1H), 3.48 (q, J = 7.0 Hz, 1H), 2.91 (q, J = 6.8 Hz, 2H), 2.69 (s, 3H), 2.38 (s, 3H), 1.77 – 1.73 (m, 1H), 1.70 – 1.66 (m, 1H), 1.49 – 1.46 (m, 1H), 1.43 – 1.39 (m, 1H), 1.30 (d, J = 6.9 Hz, 3H); $^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$) δ 158.6, 152.9, 148.0, 143.4, 136.8, 129.6, 129.4, 129.0, 127.0, 125.5, 125.3, 122.6, 118.4, 43.1, 34.0, 32.8, 27.6, 25.4, 21.4, 21.2; IR (ATR) ν_{max} 2921, 2852, 1599, 1458, 1325, 1157, 762, 551 cm^{-1}; HRMS (ESI) Calcd for $C_{22}H_{26}N_2O_2S$: $[M+H]^+$ = 383.1788. Found: 383.1785.



7

***N*-(4-chloropentyl)-4-methylbenzenesulfonamide (7).** Reaction of *N*-chlorosulfonimide **6** (27.5 mg, 0.1 mmol) with the *N*-methoxyheteroarenium salt **2a** (51.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (4:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.3 in 4:1 v/v petroleum ether/ethyl acetate) gave compound **7** (17.5 mg, 63%) as a colorless oil. The spectral data obtained was in accord with the assigned structure and matched the reported in the literature.[7] ^1H NMR (600 MHz, $CDCl_3$) δ 7.74 (d, J = 8.3 Hz, 2H), 7.31 (d, J = 7.5 Hz, 2H), 4.63 (t, J = 6.3 Hz, 1H), 3.97 – 3.95 (m, 1H), 2.96 (q, J = 6.5 Hz, 2H), 2.43 (s, 3H), 1.72 – 1.61 (m, 4H), 1.46 (d, J = 6.5 Hz, 3H); $^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) δ 143.5, 136.8, 129.7, 127.1, 58.0, 42.6, 37.0, 26.7, 25.3, 21.5. (Known

compounds, HRMS data detailed in Ref.)



9

Methyl 2-(2-methyl-1-tosylpyrrolidin-2-yl)isonicotinate (9). Reaction of pyridine-based product **3a** (37.6 mg, 0.1 mmol) with the relevant *N*-iodosuccinimide (45.0 mg, 0.2 mmol) followed by standard work-up and flash column chromatography on silica gel (3:1 v/v petroleum ether/ethyl acetate elution, R_f = 0.5 in 2:1 v/v petroleum ether/ethyl acetate) gave the unsymmetrically linked *bis*-heterocycle **9** (17.3 mg, 46%) as a colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.60 (d, *J* = 5.0 Hz, 1H), 8.17 (s, 1H), 7.72 (d, *J* = 5.9 Hz, 1H), 7.56 (d, *J* = 8.3 Hz, 2H), 7.22 (d, *J* = 8.0 Hz, 2H), 3.98 (s, 3H), 3.75 – 3.70 (m, 1H), 3.63 – 3.58 (m, 1H), 2.41 (s, 3H), 2.40 – 2.36 (m, 1H), 2.03 – 1.99 (m, 1H), 1.96 (s, 3H), 1.93 – 1.88 (m, 2H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 165.7, 165.7, 149.0, 142.8, 138.0, 137.9, 129.3, 127.2, 121.1, 120.3, 70.5, 52.8, 50.0, 44.1, 25.3, 22.9, 21.5; IR (ATR) ν_{max} 2948, 2363, 1730, 1445, 1337, 1152, 1001, 664 cm^{-1}; HRMS (ESI) Calcd for $C_{19}H_{22}N_2O_4S$: $[M+H]^+$ = 375.1373. Found: 375.1362.

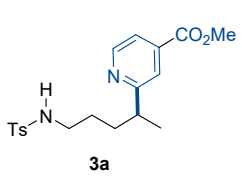
IX. X-ray Crystallographic Data

All single-crystal diffraction data collection were performed on a Rigaku XtaLAB Pro diffractometer using CuKα (λ = 1.5418 Å) radiation. The initial structure elucidation and the subsequent refinement used the same method as the literature.[6] Briefly, the structures were solved by direct method using ShelXT-2014. In the structure refinements, non-H atoms were refined anisotropically. H-Atoms bonded to carbons were placed on geometrically ideal positions using the riding model. H-Atoms bonded to oxygen were located using the difference Fourier map and were included in the calculation of structure factors with isotropic temperature factors. Crystallographic data for the molecular structures of PSCC@**3a,** PSCC@**3r,** PSCC@**3ab**, PSCC@**4c**, PSCC@**4m,** PSCC@**4o** and PSCC@**4t** reported in this paper have been deposited with the Cambridge Crystallographic Data Centre, can be accessed via www.ccdc.cam.ac.uk/data_request/cif.

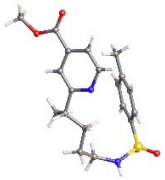
MOF EF @ Guest

Items	MOF EF⊃ 3a-BTHH	MOF EF⊃ 3r-BTHH	MOF EF⊃ 3ab-BTHH	MOF EF⊃ 4c-BTHH	MOF EF⊃ 4m-BTHH	MOF EF⊃ 4o-BTHH	MOF EF⊃ 4t-BTHH
Asymmetric Formula	$CuIC_{23}H_{14}N_2\supset$	$CuIC_{23}H_{14}N_2\supset$	$CuIC_{23}H_{14}N_2\supset$	$CuIC_{23}H_{14}N_2\supset$	$CuIC_{23}H_{14}N_2\supset$	$CuIC_{23}H_{14}N_2\supset$	$CuIC_{23}H_{14}N_2\supset$
	$0.5*(C_{19}H_{24}N_2O_4S)$	$0.5*(C_{18}H_{22}N_2O_4S)$	$0.5*(C_{21}H_{26}N_2O_4S)$	$0.5*(C_{18}H_{21}F_3N_2O_2S)$	$0.25*(C_{19}H_{26}N_2O_3S)$	$0.5*(C_{22}H_{26}N_2O_2S)$	$0.5*(C_{22}H_{26}N_2O_3S)$
Mw [g mol^{-1}]	697.06	690.04	710.08	702.04	599.44	700.08	708.08
System	orthorhombic	orthorhombic	orthorhombic	orthorhombic	orthorhombic	orthorhombic	orthorhombic
Space group	Pccn	Pccn	Pccn	Pccn	Pccn	Pccn	Pccn
a(Å)	19.4830(3)	19.7563(4)	21.1353(3)	19.5675(6)	21.7880(2)	21.7171(6)	21.6760(4)
b(Å)	21.8147(3)	21.3754(5)	19.9080(3)	21.5385(7)	19.38730(10)	19.5864(5)	19.3535(4)
c(Å)	15.0660(2)	15.1394(3)	15.09944(20)	15.1075(4)	15.04640(10)	14.9909(4)	15.1099(3)
α(°)	90	90	90	90	90	90	90
β(°)	90	90	90	90	90	90	90
γ(°)	90	90	90	90	90	90	90
V(Å)	6403.29(16)	6393.4(2)	6353.23(15)	6367.1(3)	6355.76(8)	6376.5(3)	6338.7(2)
Z	4	4	4	4	4	4	4
dcalcd[mg cm−3]	1.434	1.420	1.471	1.452	1.240	1.446	1.455
R_{int}	0.0646	0.1246	0.0342	0.1083	0.1368	0.1142	0.0834
GooF	1.074	1.161	1.063	1.141	1.141	1.100	1.050
Flack	None	None	None	None	None	None	None
Temperature (K)	99.97(12)	99.97(13)	99.95(18)	100.01(10)	100.0(3)	99.9(3)	99.98(14)
R1 [I > 2σ(I)]	0.0996	0.1290	0.0608	0.1371	0.0599	0.0973	0.0915
wR2(all data)	0.2869	0.3087	0.1689	0.3486	0.1719	0.2413	0.2394
F(000)	2760	2724	2812.0	2768.0	2348	2776	2776
CCDC No.	2341937	2341936	2341938	2341940	2341941	2341939	2341942

Crystallographic Data for 3a (CCDC : 2341937)



3a



Datablock: ef-hyt-1-bthh

Bond precision: C-C = 0.0129 A Wavelength=1.54184

Cell: a=19.4830(3) b=21.8147(3) c=15.0660(2)

alpha=90 beta=90 gamma=90

Temperature: 100 K

	Calculated	Reported
Volume	6403.29(16)	6403.29(16)
Space group	P c c n	P c c n
Hall group	-P 2ab 2ac	-P 2ab 2ac
Moiety formula	C45 H28 Cu2 I2 N4, C19 H24 N2 O4 S	C64 H52 Cu2 I2 N6 O4 S
Sum formula	C64 H52 Cu2 I2 N6 O4 S	C64 H52 Cu2 I2 N6 O4 S
Mr	1382.08	1382.05
Dx,g cm-3	1.434	1.434
Z	4	4
Mu (mm-1)	9.080	9.080
F000	2760.0	2760.0
F000'	2749.48	
h,k,lmax	24,27,18	23,27,18
Nref	6543	6347
Tmin,Tmax	0.387,0.484	0.331,1.000
Tmin'	0.223	

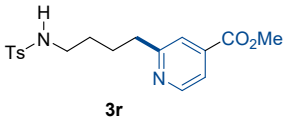
Correction method= # Reported T Limits: Tmin=0.331 Tmax=1.000 AbsCorr = MULTI-SCAN

Data completeness= 0.970 Theta(max)= 74.324

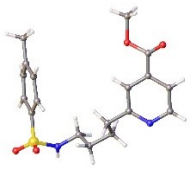
R(reflections)= 0.0942(5610) wR2(reflections)= 0.2869(6347)

S = 1.074 Npar= 451

Crystallographic Data for 3r (CCDC : 2341936)



3r



Datablock: ef-hyt-241-bthh

Bond precision: C-C = 0.0183 A Wavelength=1.54184

Cell: a=19.7563(4) b=21.3754(5) c=15.1394(3)

alpha=90 beta=90 gamma=90

Temperature: 100 K

	Calculated	Reported
Volume	6393.4(2)	6393.4(2)
Space group	P c c n	P c c n
Hall group	-P 2ab 2ac	-P 2ab 2ac
Moiety formula	C45 H28 Cu2 I2 N4, C18 H22 N2 O4 S	C45 H28 Cu2 I2 N4, C18 H22 N2 O4 S
Sum formula	C63 H50 Cu2 I2 N6 O4 S	C63 H49 Cu2 I2 N6 O4 S
Mr	1368.05	1367.02
Dx,g cm-3	1.421	1.420
Z	4	4
Mu (mm-1)	9.088	9.088
F000	2728.0	2724.0
F000'	2717.42	
h,k,lmax	24,26,18	24,25,18
Nref	6252	6199
Tmin,Tmax	0.386,0.441	0.113,1.000
Tmin'	0.292	

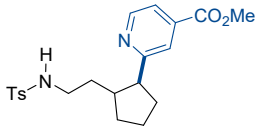
Correction method= # Reported T Limits: Tmin=0.113 Tmax=1.000 AbsCorr = MULTI-SCAN

Data completeness= 0.992 Theta(max)= 71.579

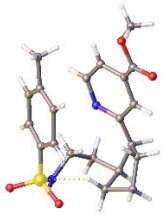
R(reflections)= 0.1228(5664) wR2(reflections)= 0.3070(6199)

S = 1.162 Npar= 456

Crystallographic Data for 3ab (CCDC : 2341938)



3ab



Datablock: ef-hyt-152-bthh-red

Bond precision: C-C = 0.0064 A Wavelength=1.54184

Cell: a=21.1353(3) b=19.9080(3) c=15.0994(2)

alpha=90 beta=90 gamma=90

Temperature: 100 K

	Calculated	Reported
Volume	6353.26(16)	6353.23(15)
Space group	P c c n	P c c n
Hall group	-P 2ab 2ac	-P 2ab 2ac
Moiety formula	C45 H28 Cu2 I2 N4, C21 H26 N2 O4 S	C45 H28 Cu2 I2 N4, C21 H26 N2 O4 S
Sum formula	C66 H54 Cu2 I2 N6 O4 S	C66 H53 Cu2 I2 N6 O4 S
Mr	1408.11	1407.08
Dx,g cm-3	1.472	1.471
Z	4	4
Mu (mm-1)	9.163	9.163
F000	2816.0	2812.0
F000'	2805.61	
h,k,lmax	26,24,18	25,24,18
Nref	6461	6288
Tmin,Tmax	0.240,0.160	0.260,1.000
Tmin'	0.107	

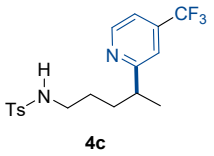
Correction method= # Reported T Limits: Tmin=0.260 Tmax=1.000 AbsCorr = MULTI-SCAN

Data completeness= 0.973 Theta(max)= 74.082

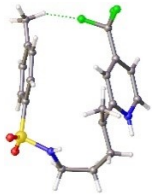
R(reflections)= 0.0533(5410) wR2(reflections)= 0.1685(6288)

S = 1.060 Npar= 481

Crystallographic Data for 4c (CCDC : 2341940)



4c



Datablock: ef-hyt-288-bthh

Bond precision: C-C = 0.0196 A Wavelength=1.54184

Cell: a=19.5675(6) b=21.5385(7) c=15.1075(4)

alpha=90 beta=90 gamma=90

Temperature: 100 K

	Calculated	Reported
Volume	6367.1(3)	6367.1(3)
Space group	P c c n	P c c n
Hall group	-P 2ab 2ac	-P 2ab 2ac
Moiety formula	C45 H28 Cu2 I2 N4, C18 H21 F3 N2 O2 S	C45 H28 Cu2 I2 N4, C18 H21 F3 N2 O2 S
Sum formula	C63 H49 Cu2 F3 I2 N6 O2 S	C63 H49 Cu2 F3 I2 N6 O2 S
Mr	1392.04	1392.02
Dx,g cm-3	1.452	1.452
Z	4	4
Mu (mm-1)	9.181	9.181
F000	2768.0	2768.0
F000'	2757.89	
h,k,lmax	23,25,17	23,25,17
Nref	5622	5614
Tmin,Tmax	0.318,0.399	0.154,1.000
Tmin'	0.240	

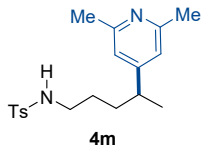
Correction method= # Reported T Limits: Tmin=0.154 Tmax=1.000 AbsCorr = MULTI-SCAN

Data completeness= 0.999 Theta(max)= 66.599

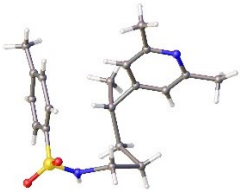
R(reflections)= 0.1271(4929) wR2(reflections)= 0.3486(5614)

S = 1.141 Npar= 452

Crystallographic Data for 4m (CCDC : 2341941)



4m



Datablock: ef-hyt-300-bthh2

Bond precision: C-C = 0.0068 A Wavelength=1.54184

Cell: a=21.7880(2) b=19.3873(1) c=15.0464(1)

alpha=90 beta=90 gamma=90

Temperature: 100 K

	Calculated	Reported
Volume	6355.76(8)	6355.76(8)
Space group	P c c n	P c c n
Hall group	-P 2ab 2ac	-P 2ab 2ac
Moiety formula	2(C45 H28 Cu2 I2 N4), C7 H8 N O2 S, 4(C2 H2.25 N0.25), 4(C0.25	C90 H56 Cu4 I4 N8, C19 H26 N2 O2 S
Sum formula	C109 H82 Cu4 I4 N10 O2 S	C109 H82 Cu4 I4 N10 O3 S
Mr	2357.71	2373.66
Dx,g cm-3	1.232	1.240
Z	2	2
Mu (mm-1)	8.869	8.878
F000	2332.0	2348.0
F000'	2319.56	
h,k,lmax	27,24,18	26,23,18
Nref	6476	6375
Tmin,Tmax	0.837,0.974	
Tmin'	0.837	

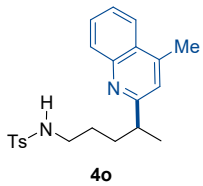
Correction method= Not given

Data completeness= 0.984 Theta(max)= 74.225

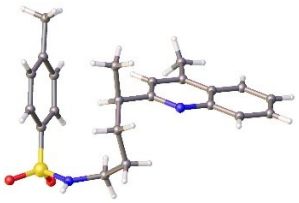
R(reflections)= 0.0591(6231) wR2(reflections)= 0.1719(6375)

S = 1.141 Npar= 446

Crystallographic Data for 4o (CCDC : 2341939)



4o



Datablock: ef-hyt-245-1-bthh

Bond precision: C-C = 0.0122 A Wavelength=1.54184

Cell: a=21.7171(6) b=19.5864(5) c=14.9909(4)

alpha=90 beta=90 gamma=90

Temperature: 100 K

	Calculated	Reported
Volume	6376.5(3)	6376.5(3)
Space group	P c c n	P c c n
Hall group	-P 2ab 2ac	-P 2ab 2ac
Moiety formula	C45 H28 Cu2 I2 N4, C22 H26 N2 O2 S	C45 H28 Cu2 I2 N4, C22 H26 N2 O2 S
Sum formula	C67 H54 Cu2 I2 N6 O2 S	C67 H54 Cu2 I2 N6 O2 S
Mr	1388.12	1388.10
Dx,g cm-3	1.446	1.446
Z	4	4
Mu (mm-1)	9.097	9.097
F000	2776.0	2776.0
F000'	2765.29	
h,k,lmax	27,24,18	26,24,18
Nref	6603	6378
Tmin,Tmax	0.463,0.403	0.329,1.000
Tmin'	0.350	

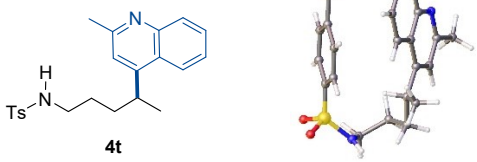
Correction method= # Reported T Limits: Tmin=0.329 Tmax=1.000 AbsCorr = MULTI-SCAN

Data completeness= 0.966 Theta(max)= 75.218

R(reflections)= 0.0909(5628) wR2(reflections)= 0.2413(6378)

S = 1.100 Npar= 462

Crystallographic Data for 4t (CCDC : 2341942)



4t

Datablock: ef-hyt-305-bthh

Bond precision: C-C = 0.0092 A Wavelength=1.54184

Cell: a=21.6760(4) b=19.3535(4) c=15.1099(3)

alpha=90 beta=90 gamma=90

Temperature: 100 K

	Calculated	Reported
Volume	6338.7(2)	6338.7(2)
Space group	P c c n	P c c n
Hall group	-P 2ab 2ac	-P 2ab 2ac
Moiety formula	C45 H28 Cu2 I2 N4, C22 H26 N2 O2 S	C45 H28 Cu2 I2 N4, C22 H26 N2 O2 S
Sum formula	C67 H54 Cu2 I2 N6 O2 S	C67 H54 Cu2 I2 N6 O2 S
Mr	1388.12	1388.10
Dx,g cm-3	1.455	1.455
Z	4	4
Mu (mm-1)	9.151	9.151
F000	2776.0	2776.0
F000'	2765.29	
h,k,lmax	25,23,17	25,23,17
Nref	5600	5596
Tmin,Tmax	0.308,0.400	0.130,1.000
Tmin'	0.220	

Correction method= # Reported T Limits: Tmin=0.130 Tmax=1.000 AbsCorr = MULTI-SCAN

Data completeness= 0.999 Theta(max)= 66.585

R(reflections)= 0.0793(4775) wR2(reflections)= 0.2295(5596)

S = 1.065 Npar= 448

X. References

[1] H. Zhang, Y. Zhou, P. Tian, C. Jiang, *Org. Lett.* **2019**, *21*, 1921-1925.

[2] Y. Chen, G.-Y. Zhang, C. Guo, P. Lan, M. G. Banwell, Y.-T. He, *Chem. Eur. J.* **2022**, *28*, e202104627.

[3] M. A. Cismesia, T. P. Yoon, *Chem. Sci.* **2015**, *6*, 5426-5434.

[4] H. J. Kuhn, S. E. Braslavsky, R. Schmidt, *Pure Appl. Chem.* **2004**, *76*, 2105-2146.

[5] J. N. Demas, W. D. Bowman, E. F. Zalewski, R. A. Velapoldi, *J. Phys. Chem.* **1981**, *85*, 2766-2771.

[6] S.-Q. Qin, W. Xu, Q.-Q. Wang, R.-Y. Chen, D.-Z. Yang, Y. Lu, W.-C. Ye, R.-W. Jiang, *Org. Chem. Front.* **2024**, *11*, 194-204.

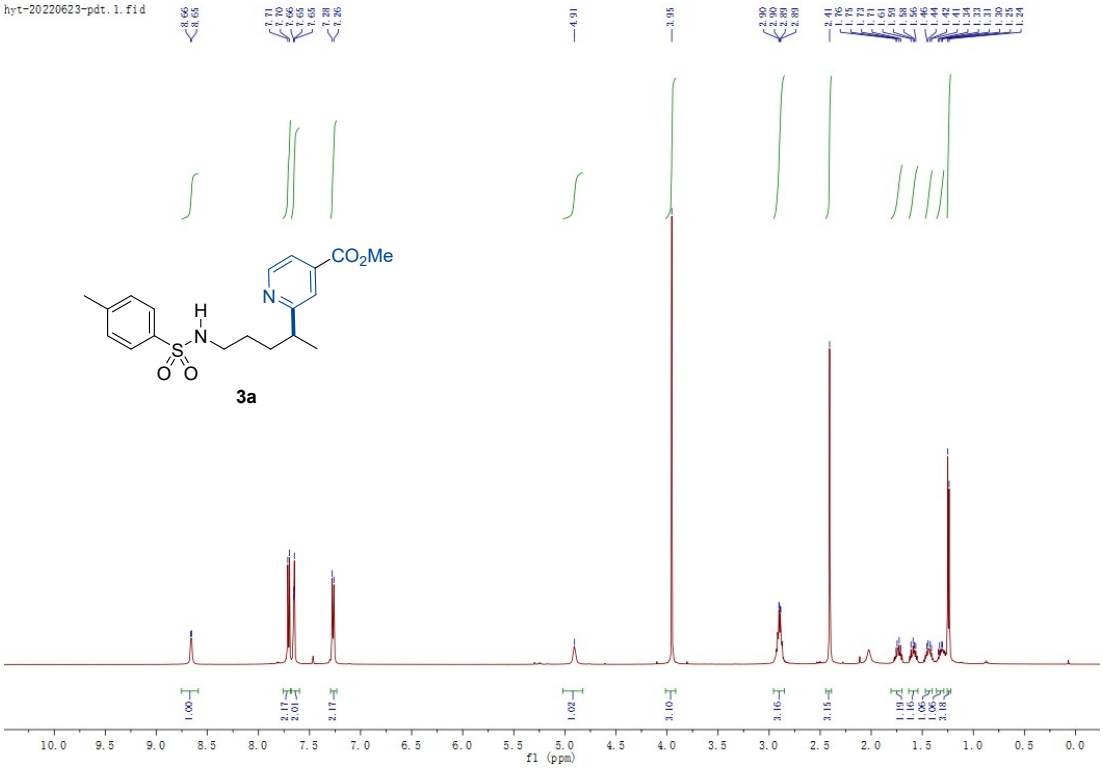
[7] C. Wang, C. Tan, Y. Zhu, G. Liu, Y. Huang, R. Liu, *ACS Catal.* **2023**, *13*, 13896-13901.

Appendix I

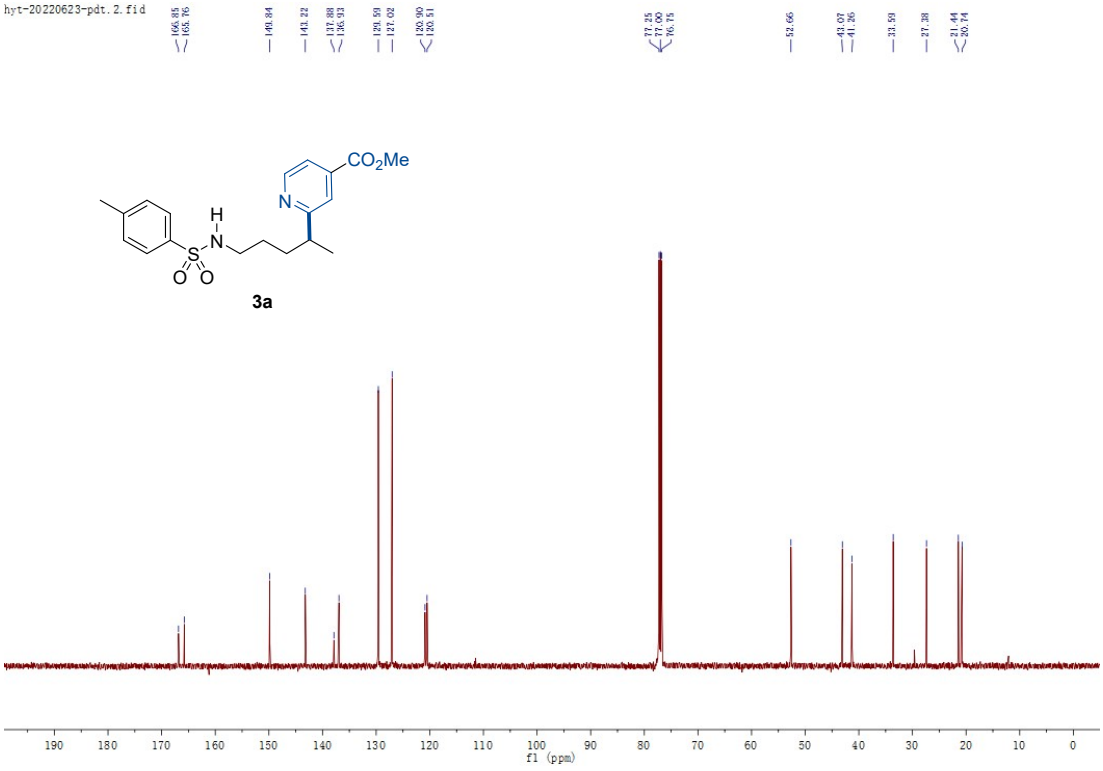
Copies of Relevant ^{1}H-, $^{13}C\{^{1}H\}$- and ^{19}F-NMR Spectra

Methyl-2-(5-((4-methylphenyl)sulfonamido)pentan-2-yl)isonicotinate (3a).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

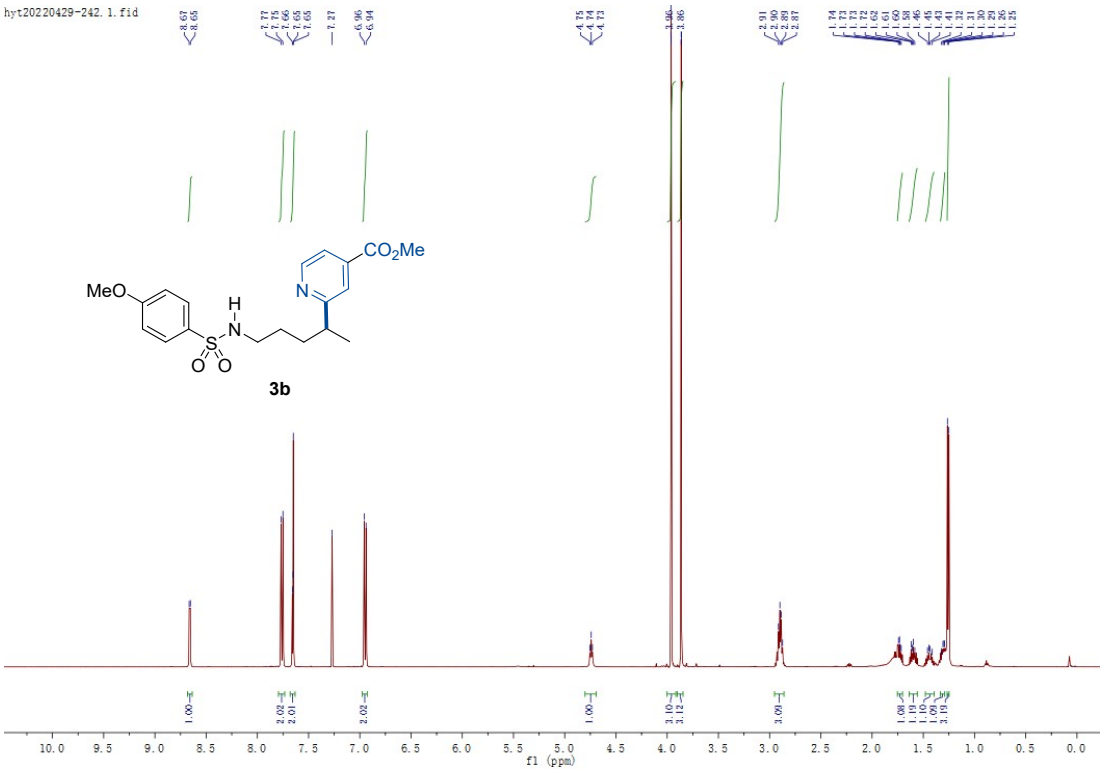


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

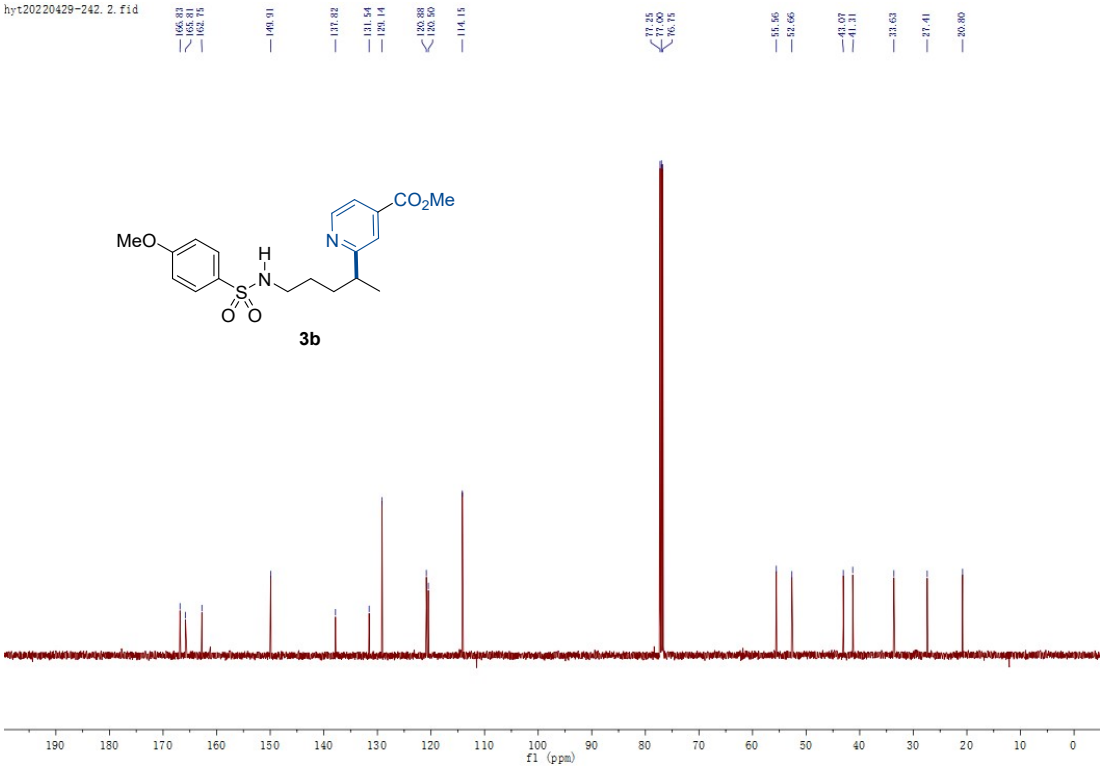


Methyl-2-(5-((4-methoxyphenyl)sulfonamido)pentan-2-yl)isonicotinate (3b).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

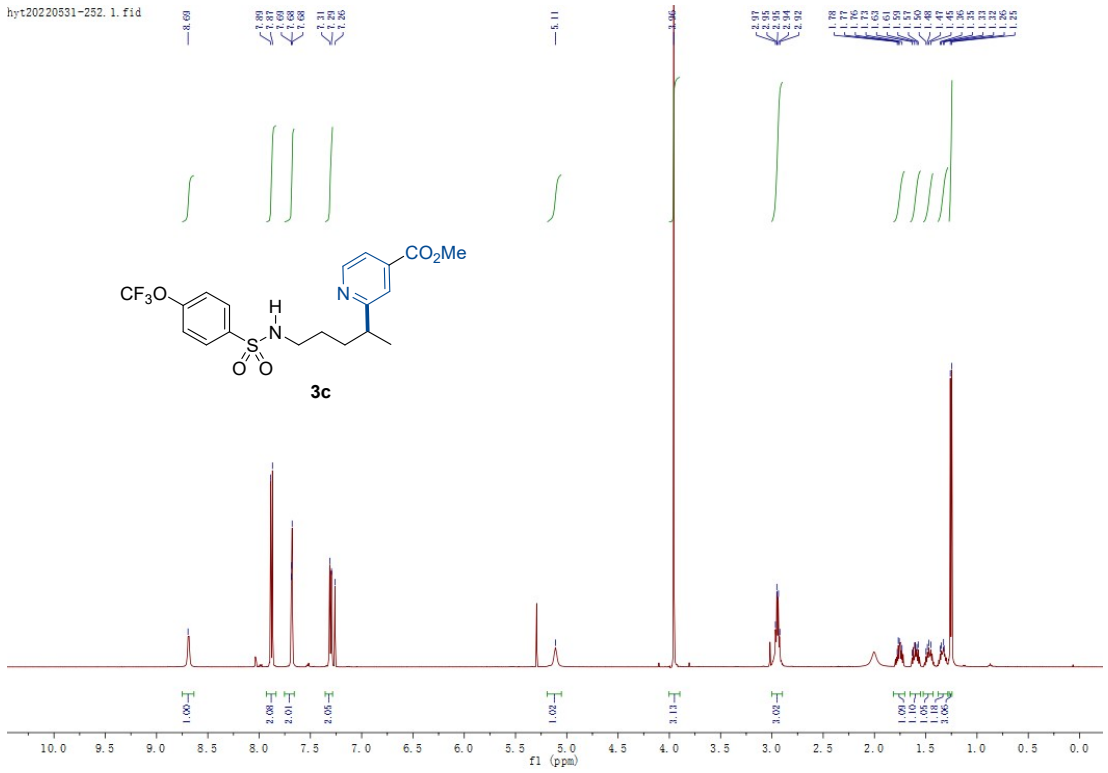


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

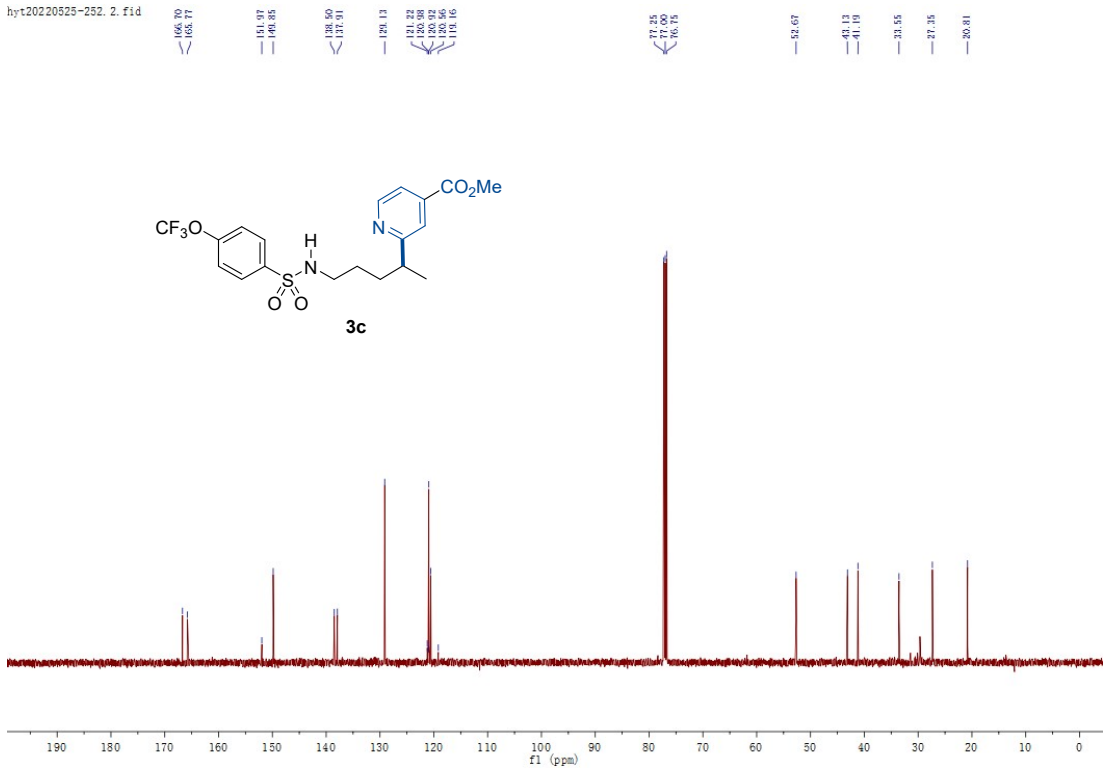


Methyl-2-(5-((4-(trifluoromethoxy)phenyl)sulfonamido)pentan-2-yl)isonicotinate (3c).

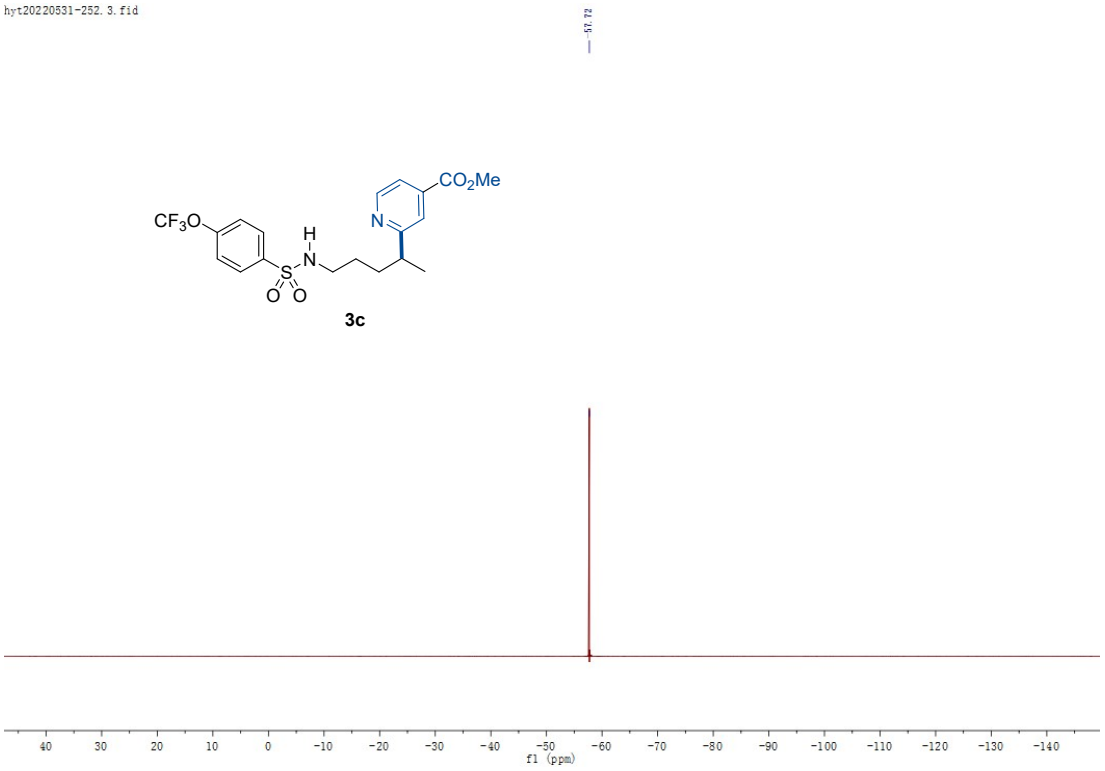
500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)



126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

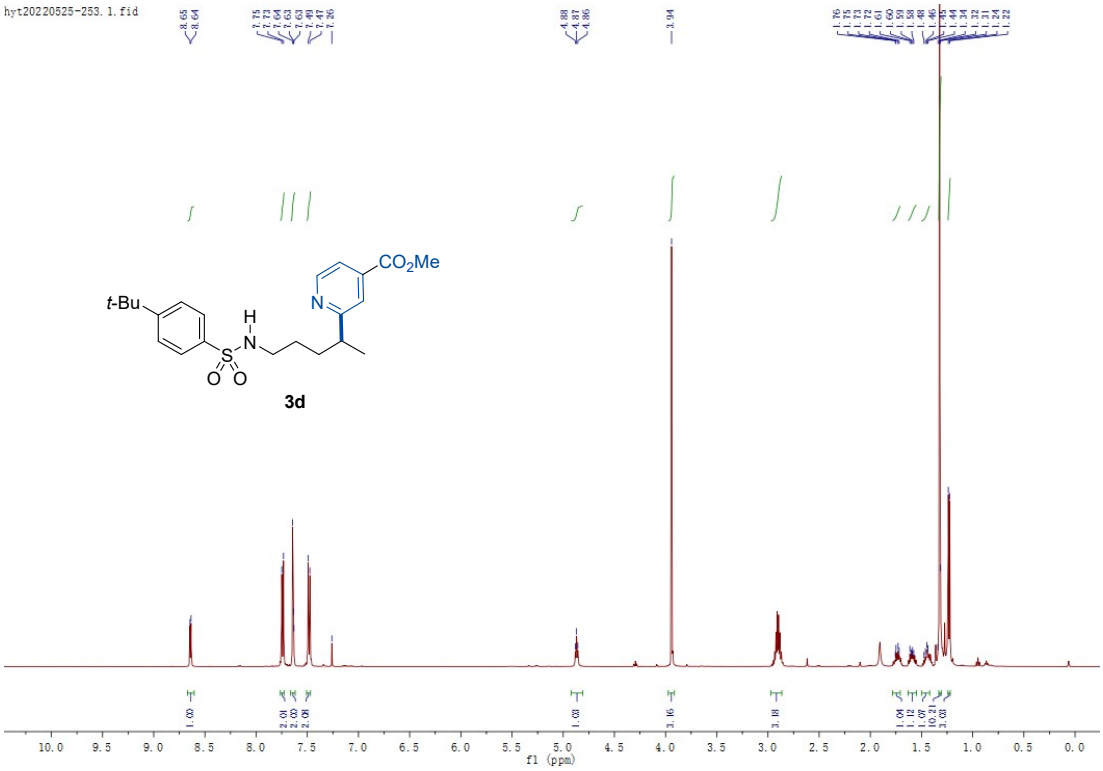


471 MHz ^{19}F NMR Spectrum (recorded in $CDCl_3$)

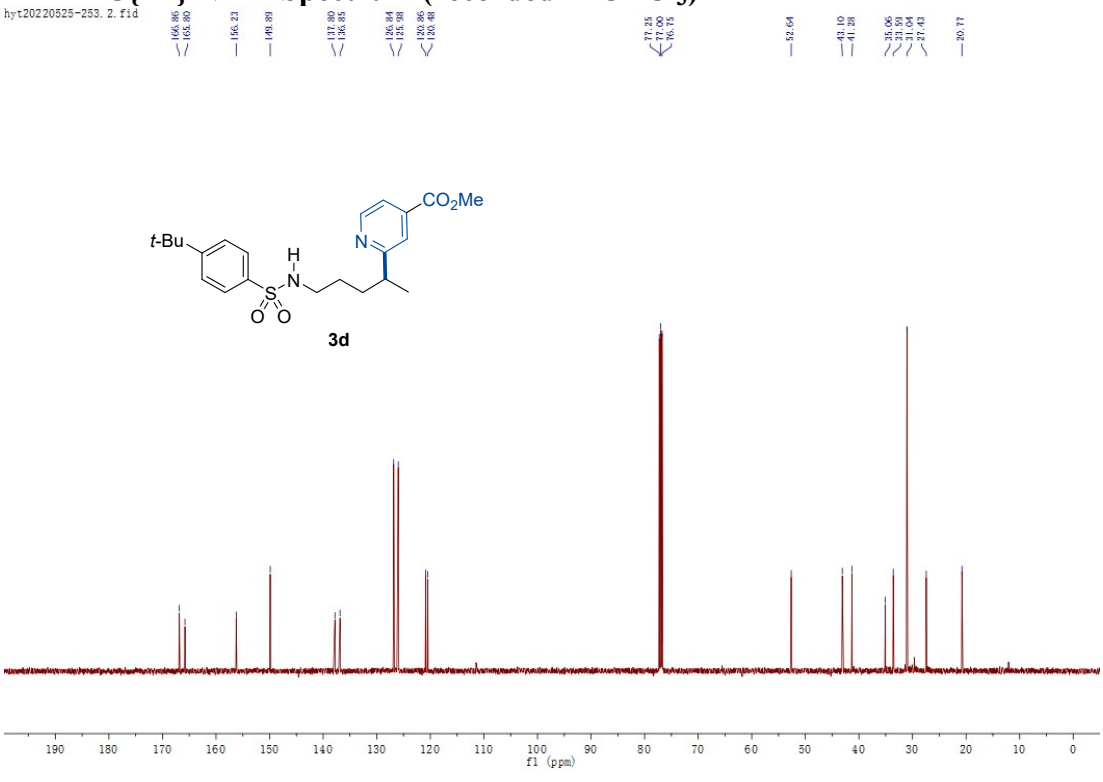


Methyl-2-(5-((4-(tert-butyl)phenyl)sulfonamido)pentan-2-yl)isonicotinate (3d).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

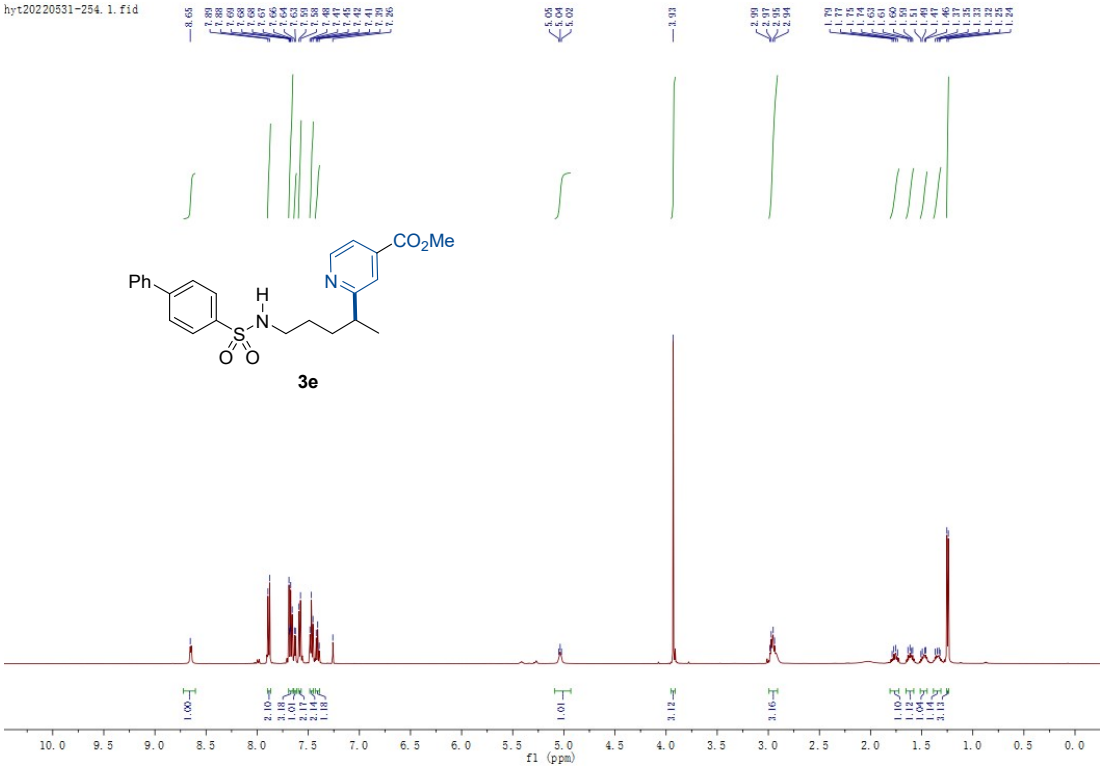


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

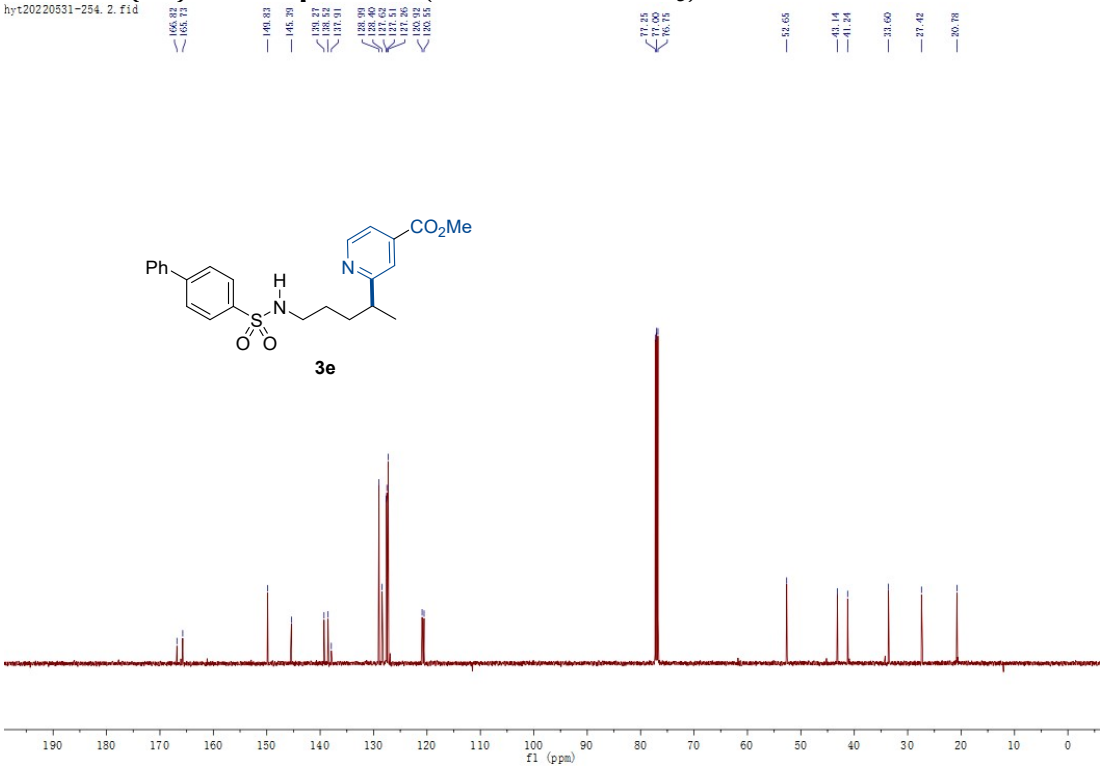


Methyl-2-(5-([1,1'-biphenyl]-4-sulfonamido)pentan-2-yl)isonicotinate (3e).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

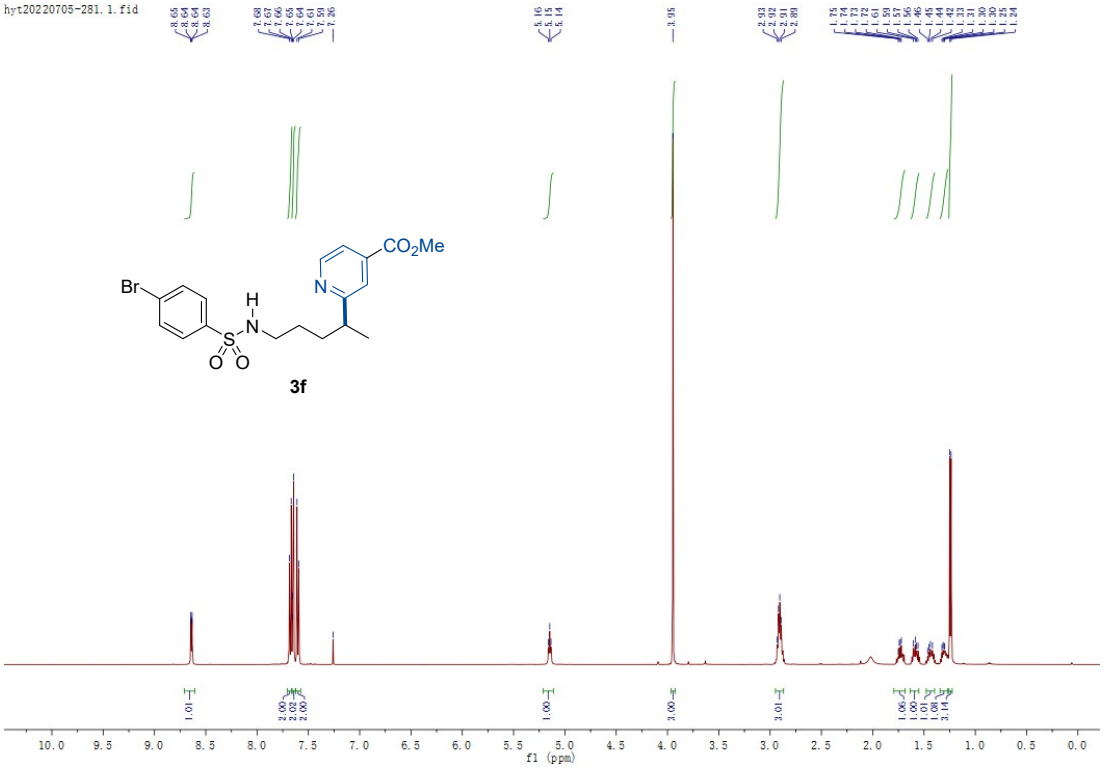


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

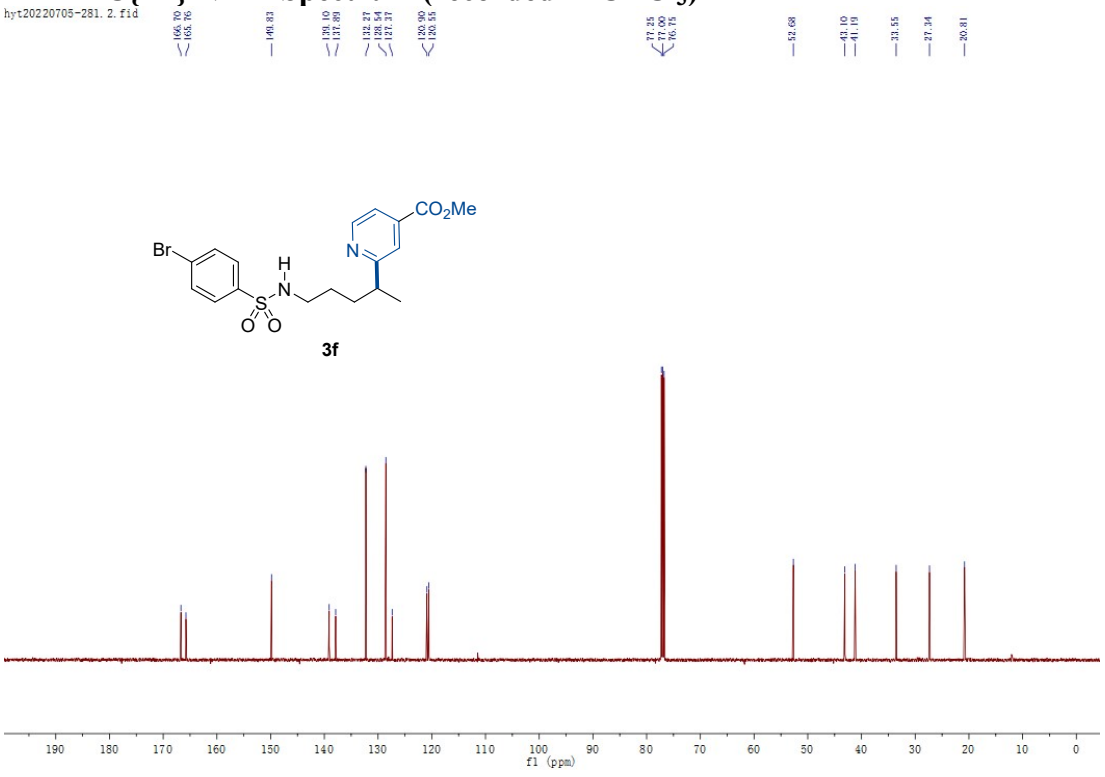


Methyl-2-(5-((4-bromophenyl)sulfonamido)pentan-2-yl)isonicotinate (3f).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

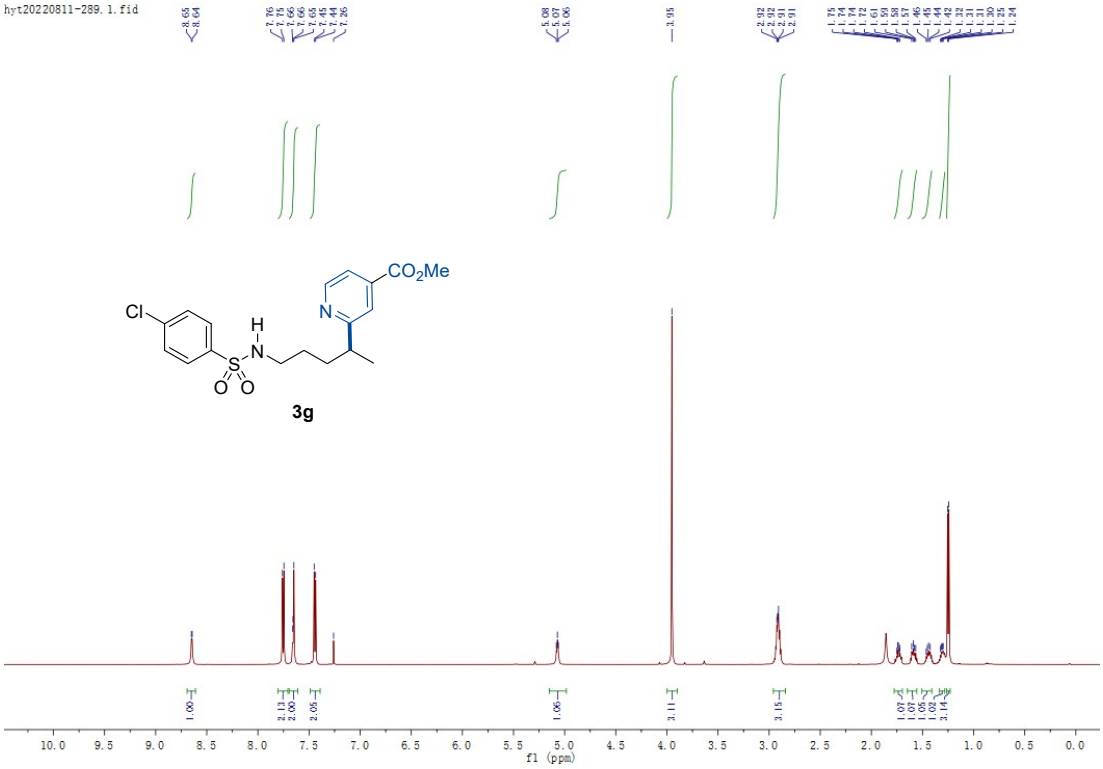


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

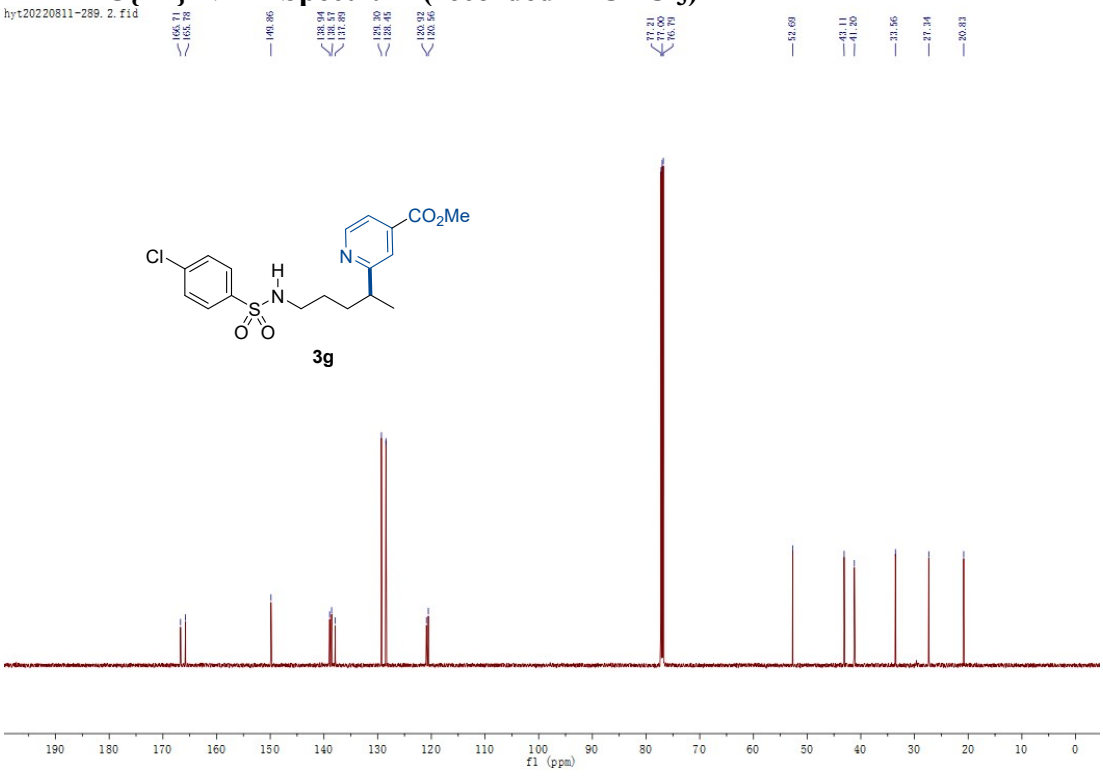


Methyl-2-(5-((4-chlorophenyl)sulfonamido)pentan-2-yl)isonicotinate (3g).

600 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

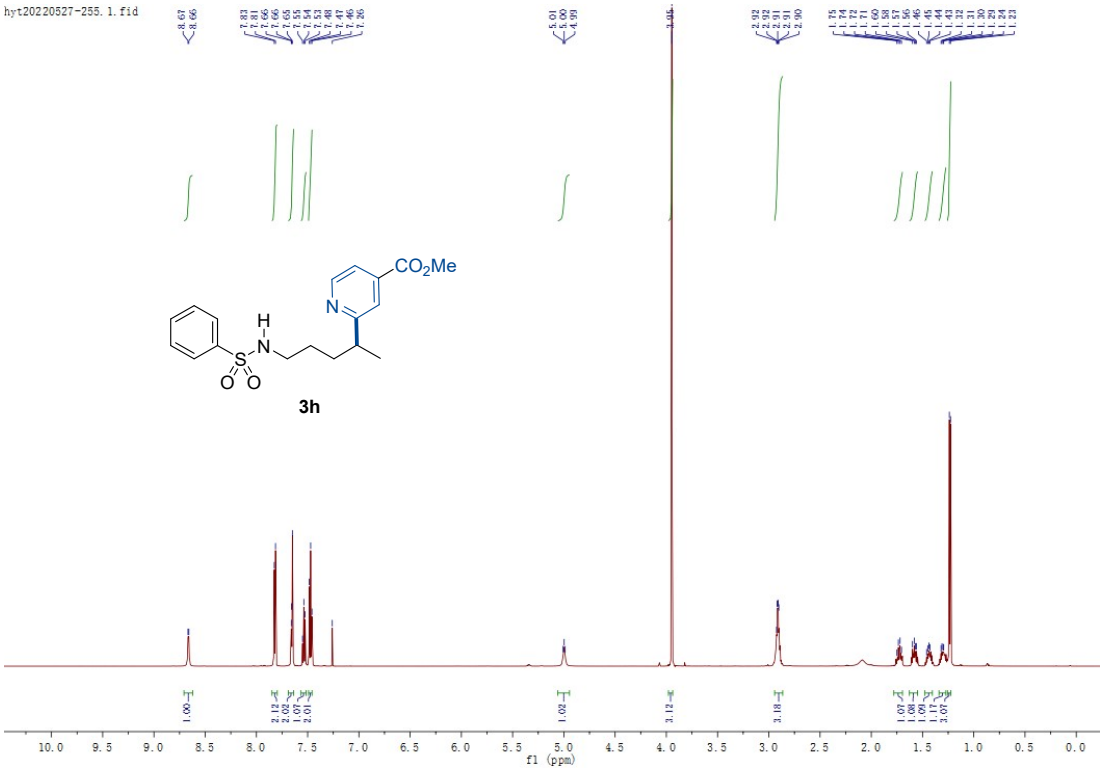


151 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

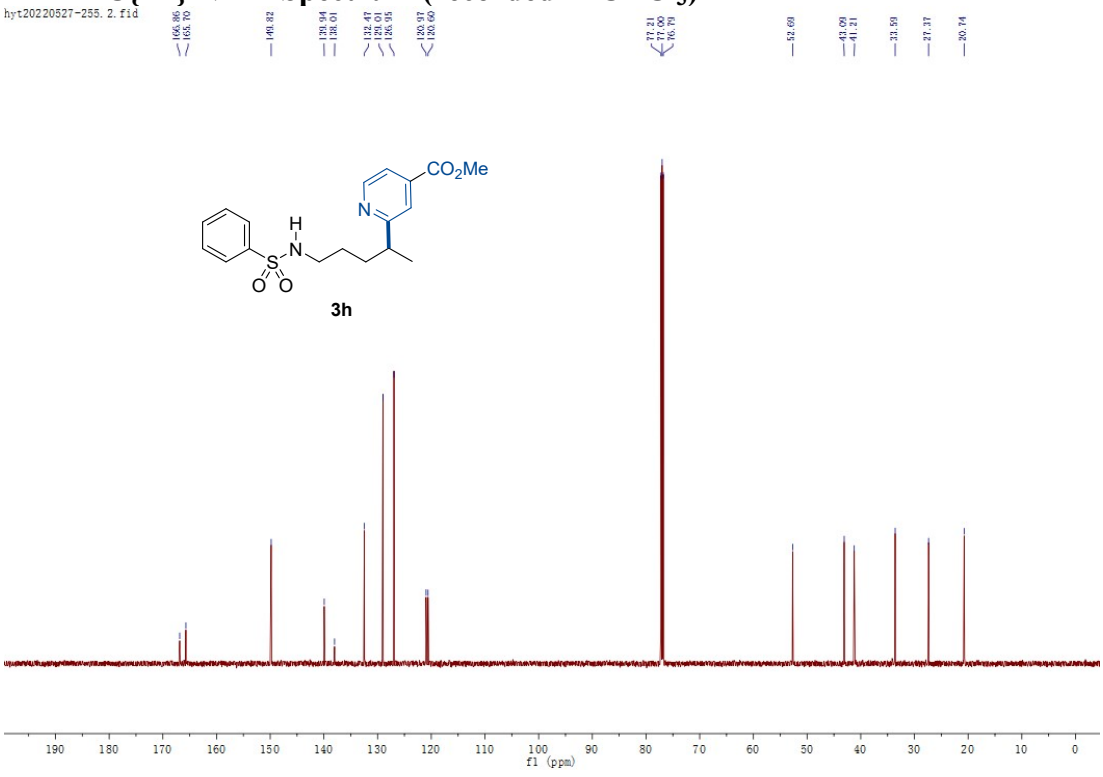


Methyl-2-(5-(phenylsulfonamido)pentan-2-yl)isonicotinate (3h).

600 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

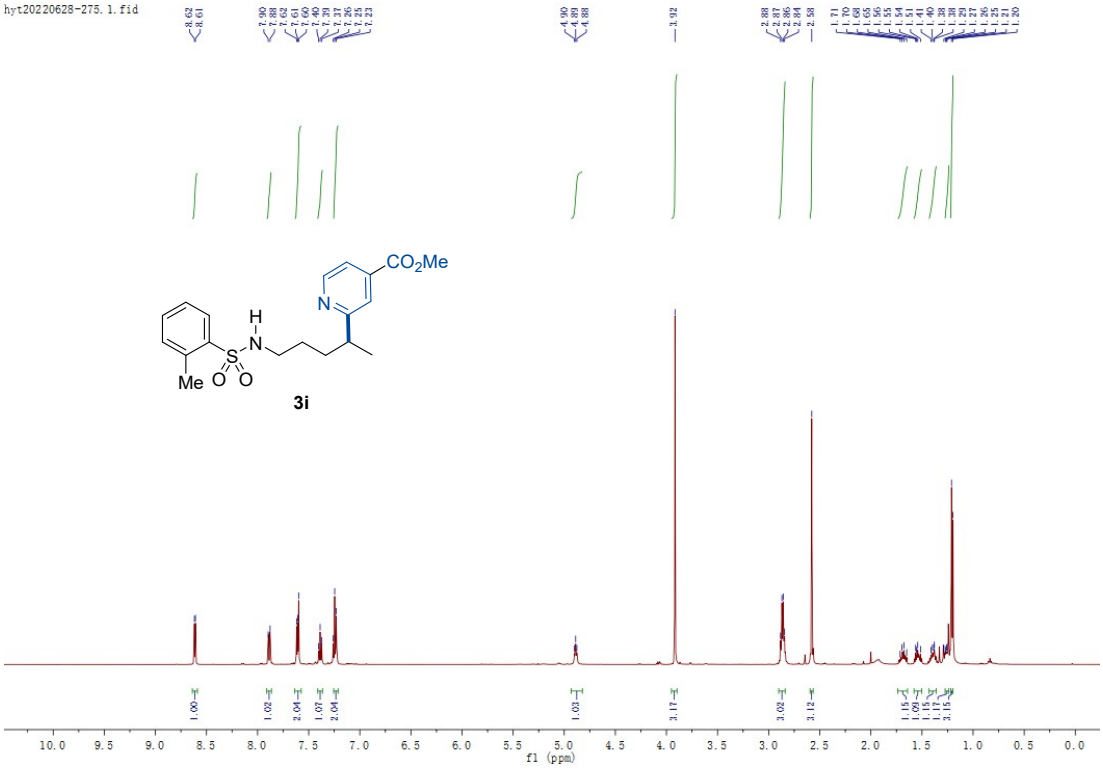


151 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

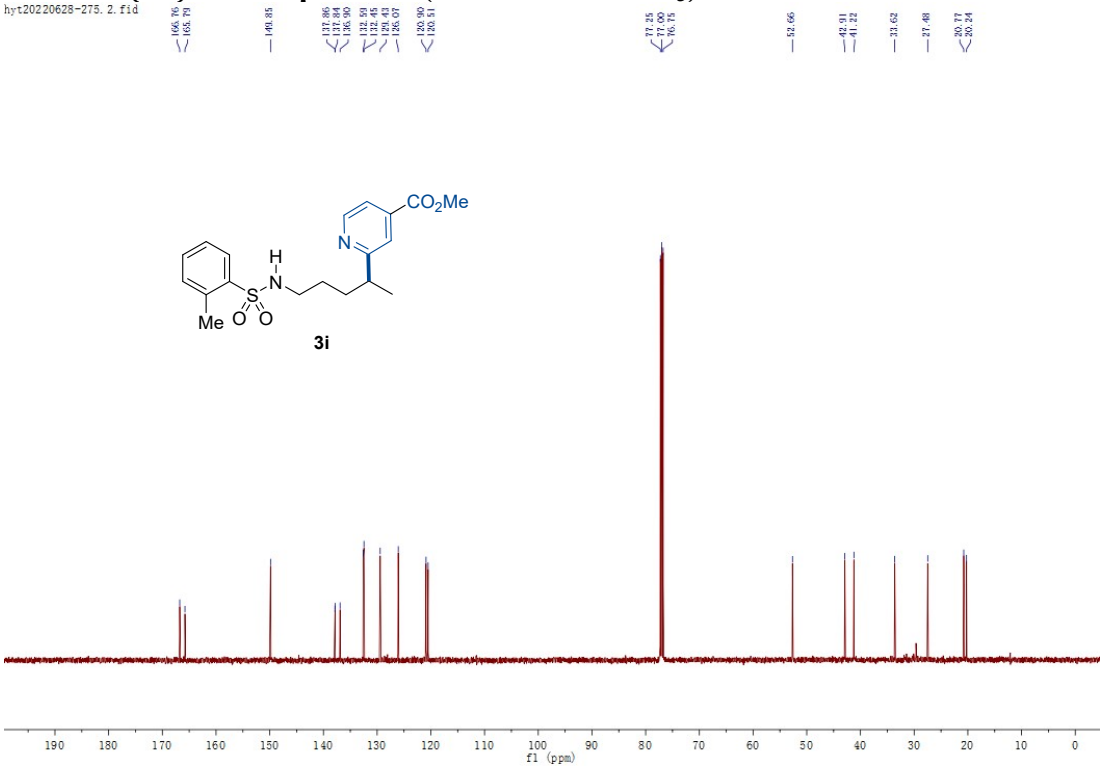


Methyl-2-(5-((2-methylphenyl)sulfonamido)pentan-2-yl)isonicotinate (3i).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

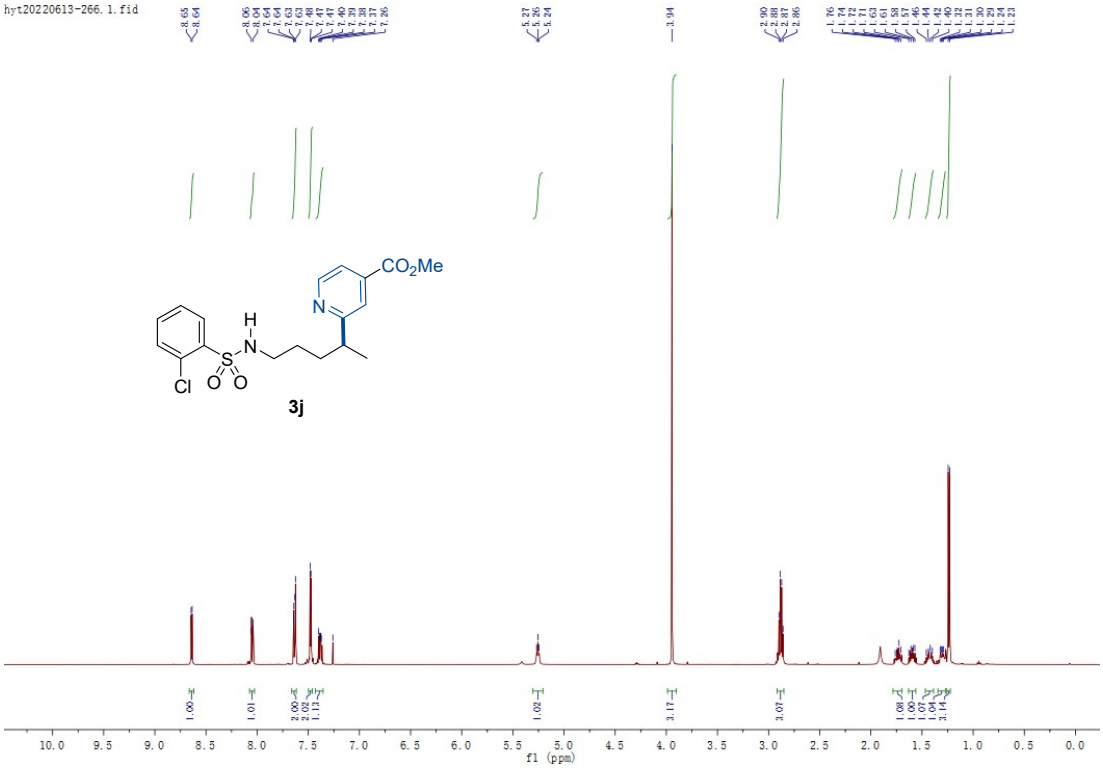


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

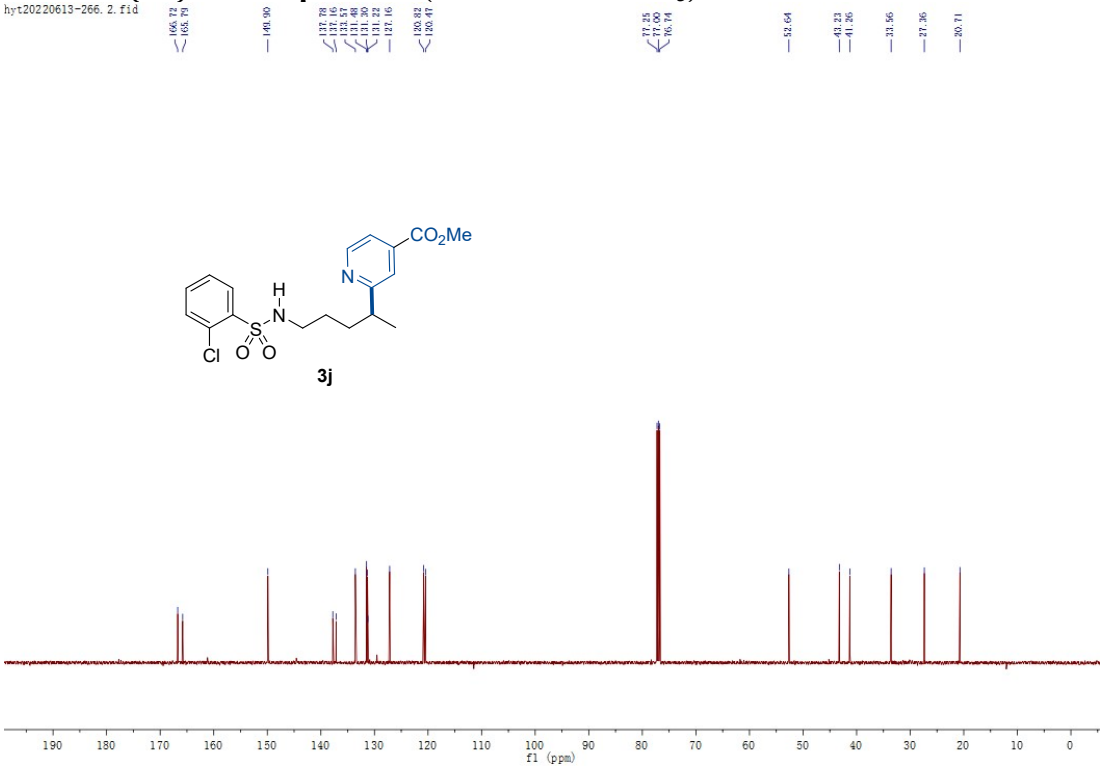


Methyl-2-(5-((2-chlorophenyl)sulfonamido)pentan-2-yl)isonicotinate (3j).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

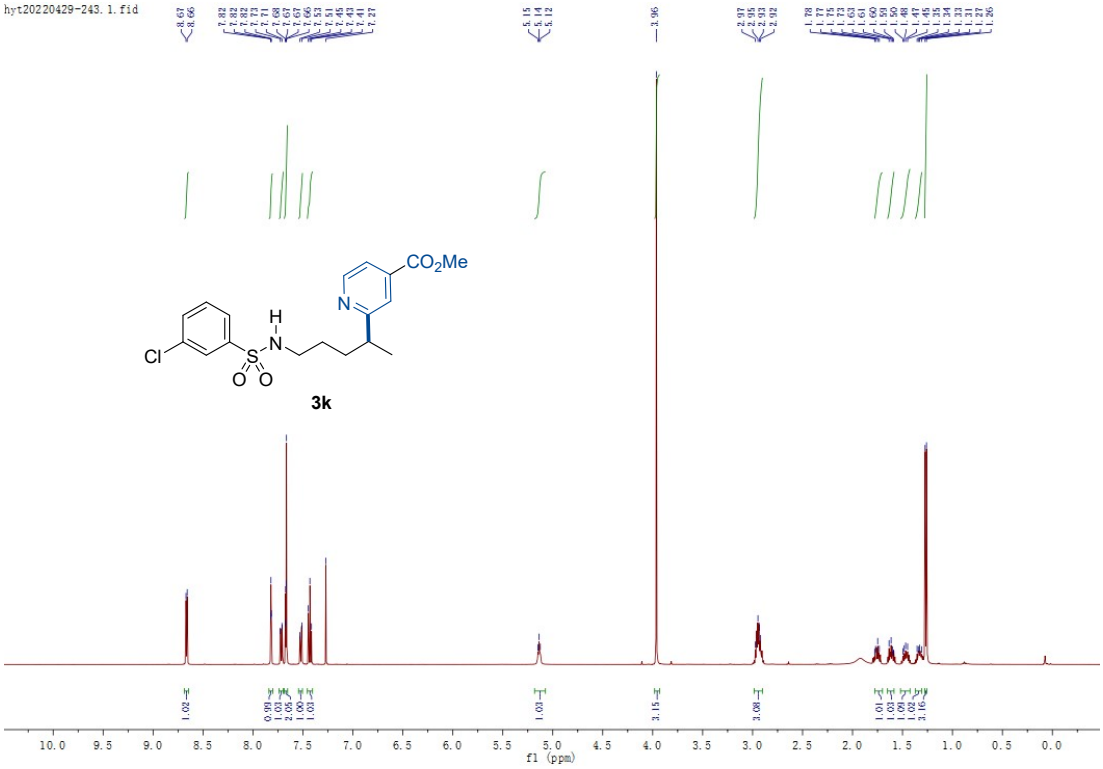


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

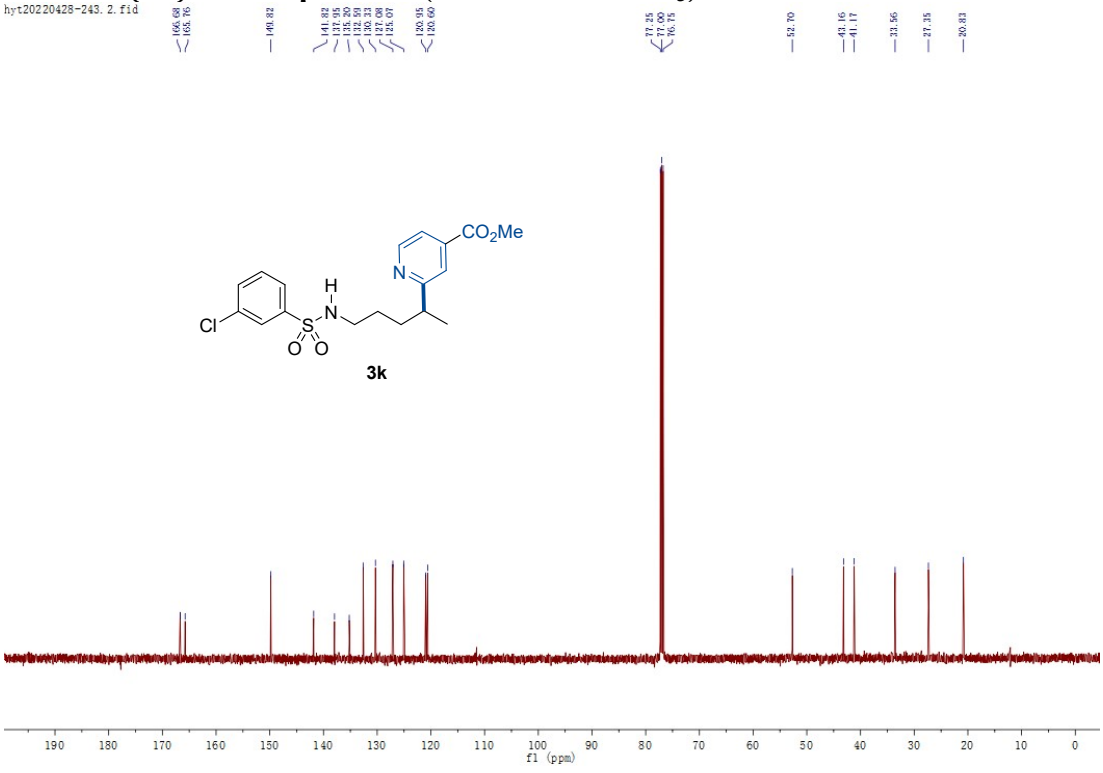


Methyl-2-(5-((3-chlorophenyl)sulfonamido)pentan-2-yl)isonicotinate (3k).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

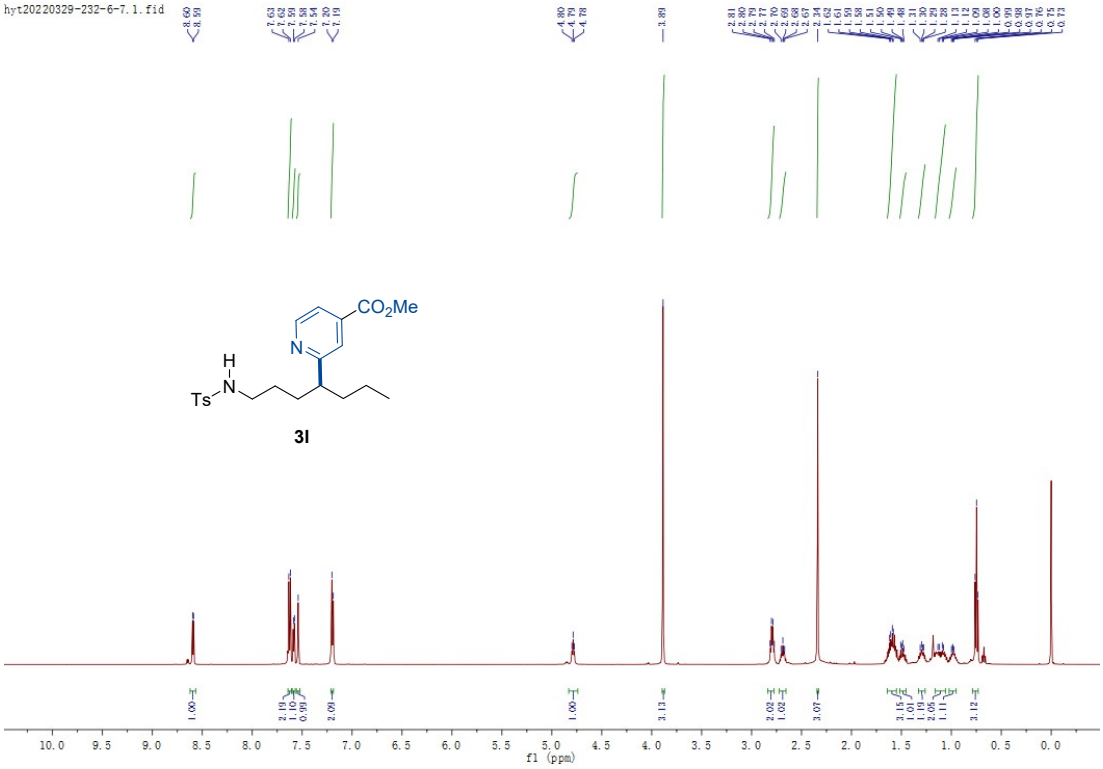


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

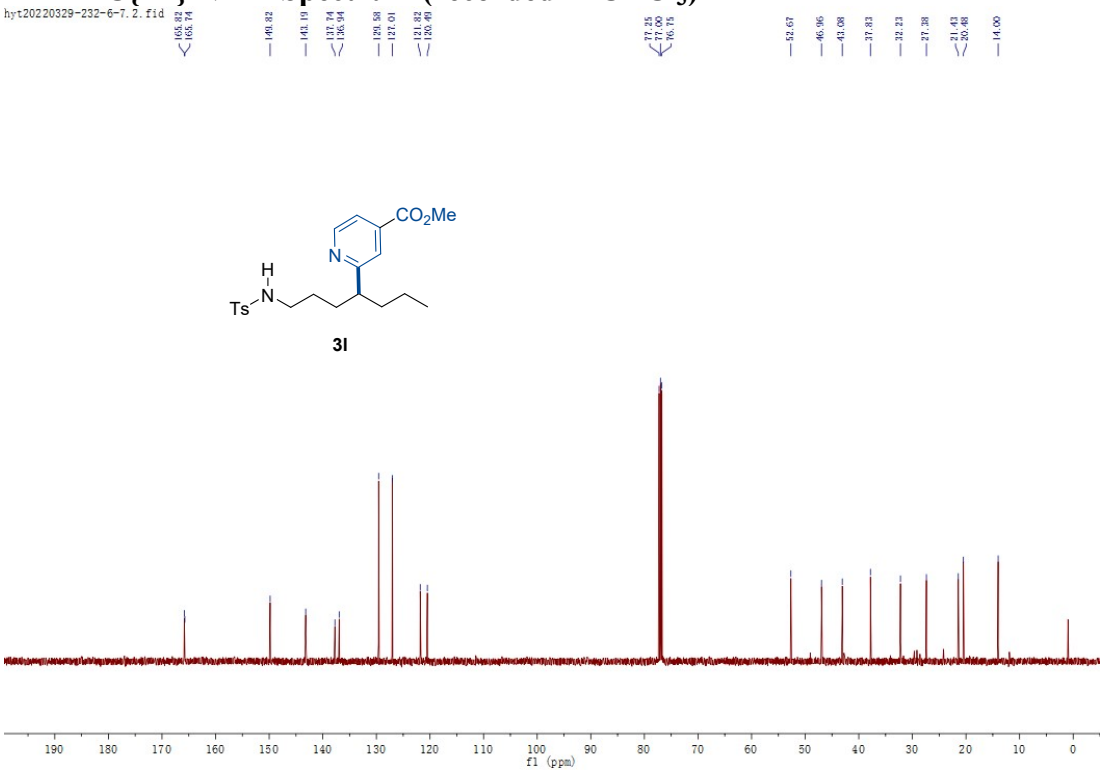


Methyl-2-(1-((4-methylphenyl)sulfonamido)heptan-4-yl)isonicotinate (3l).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

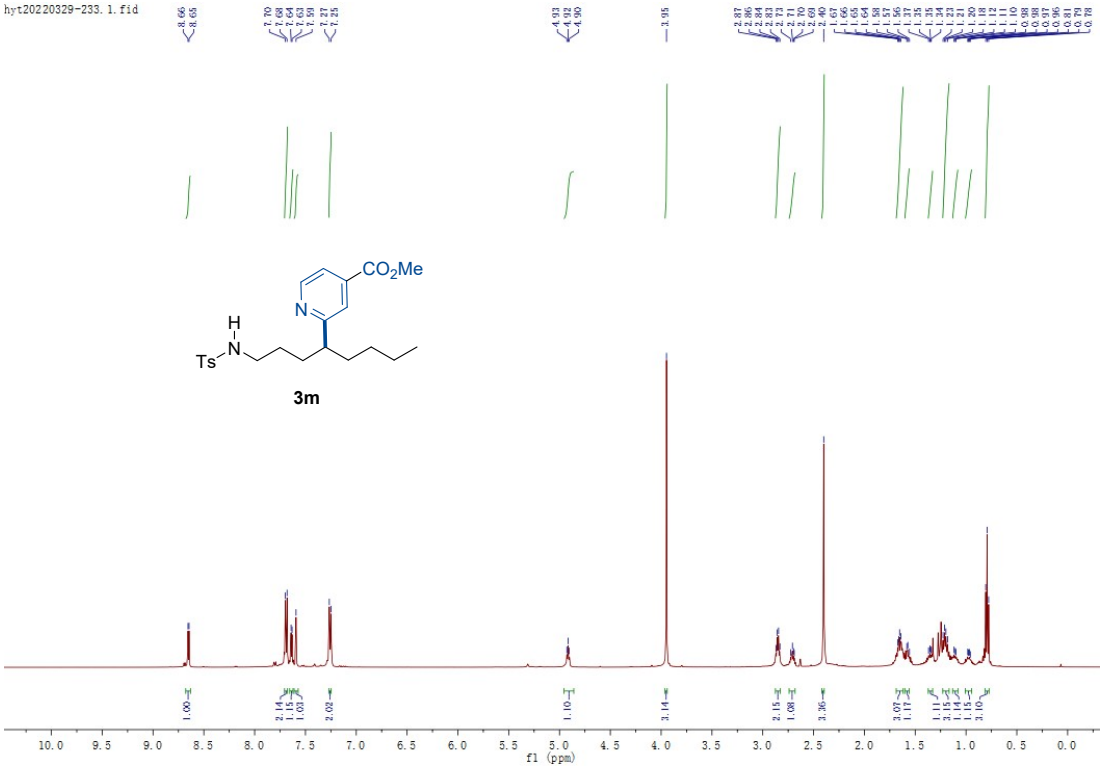


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

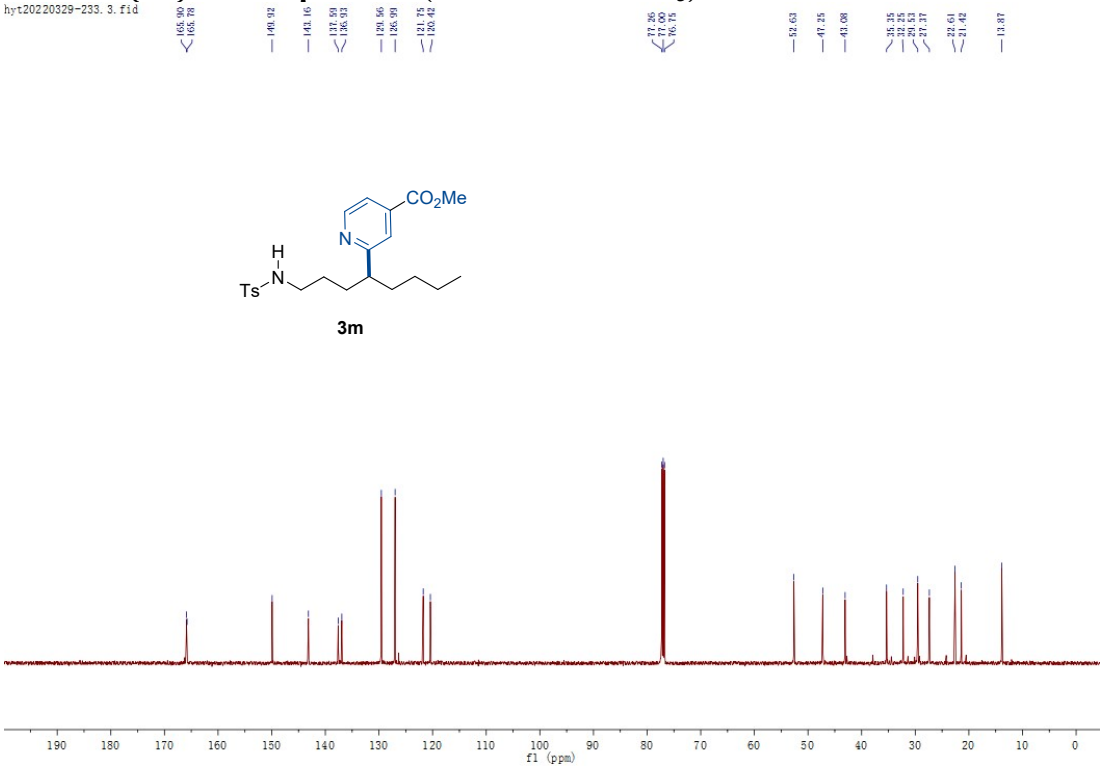


Methyl-2-(1-((4-methylphenyl)sulfonamido)octan-4-yl)isonicotinate (3m).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

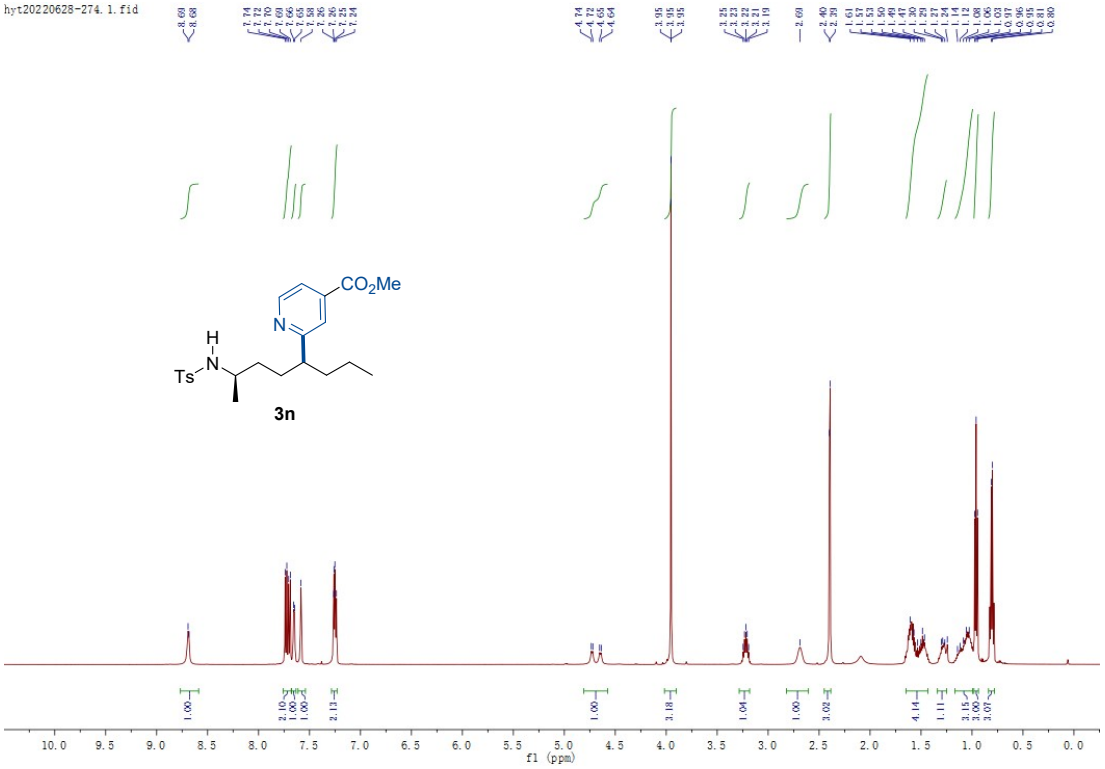


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

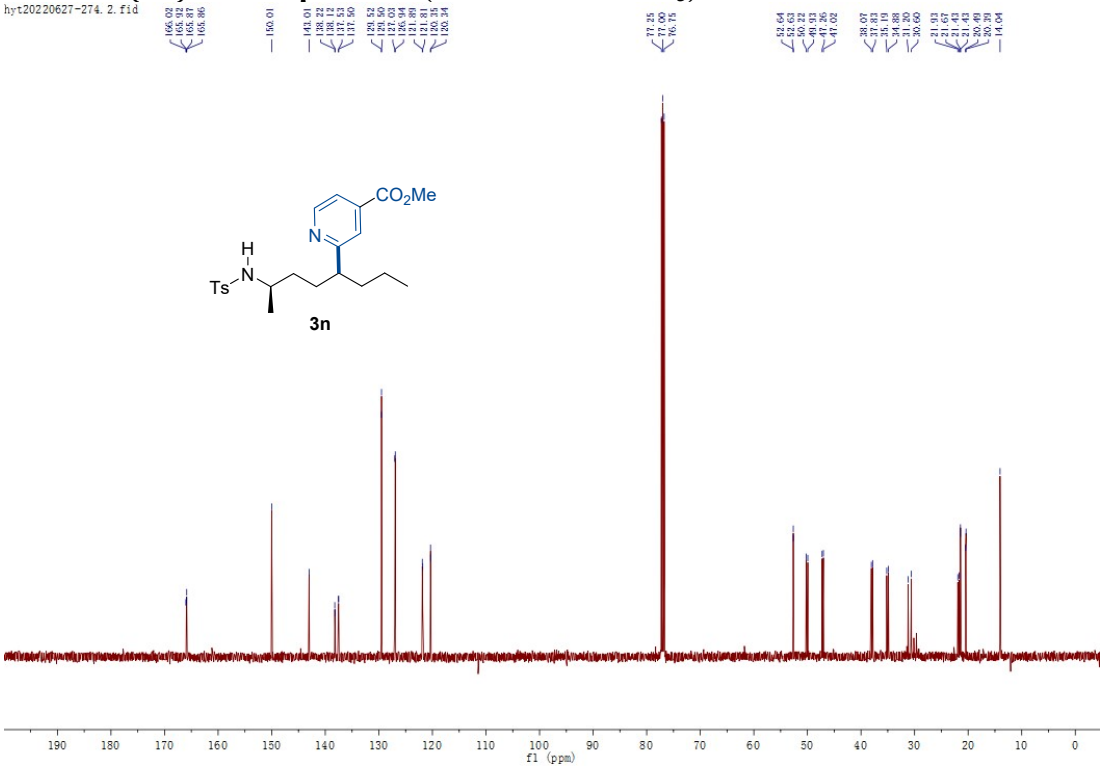


Methyl 2-((7R)-7-((4-methylphenyl)sulfonamido)octan-4-yl)isonicotinate (3n).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

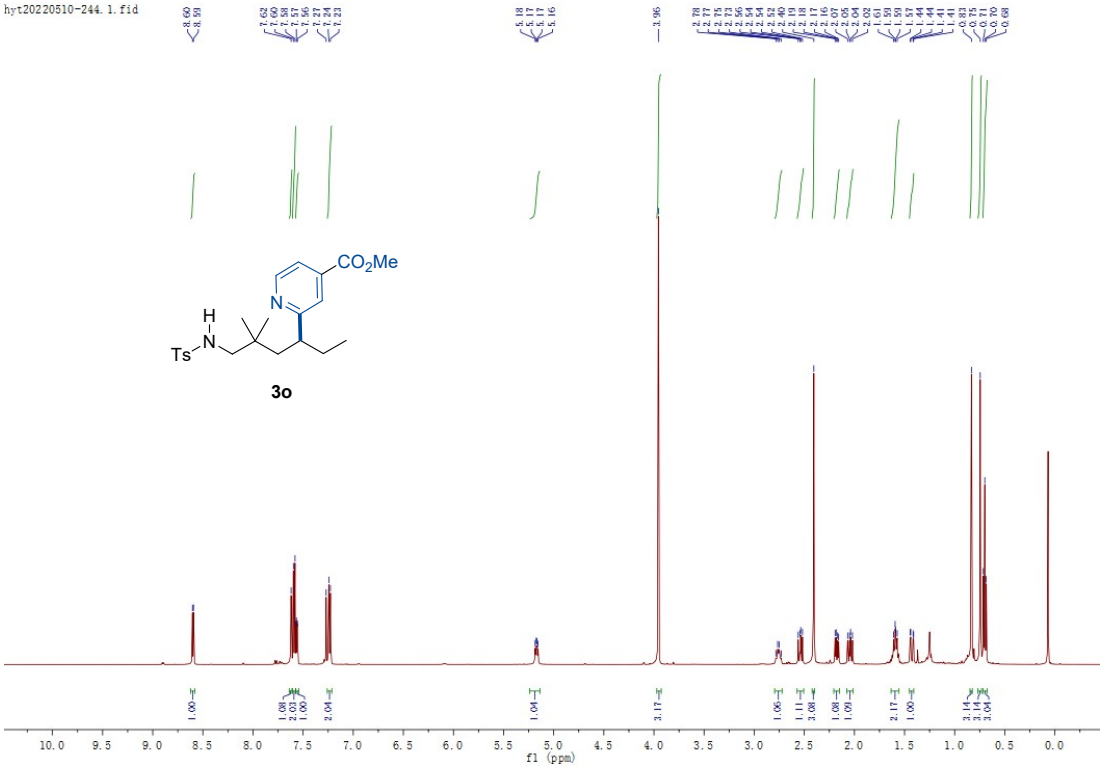


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

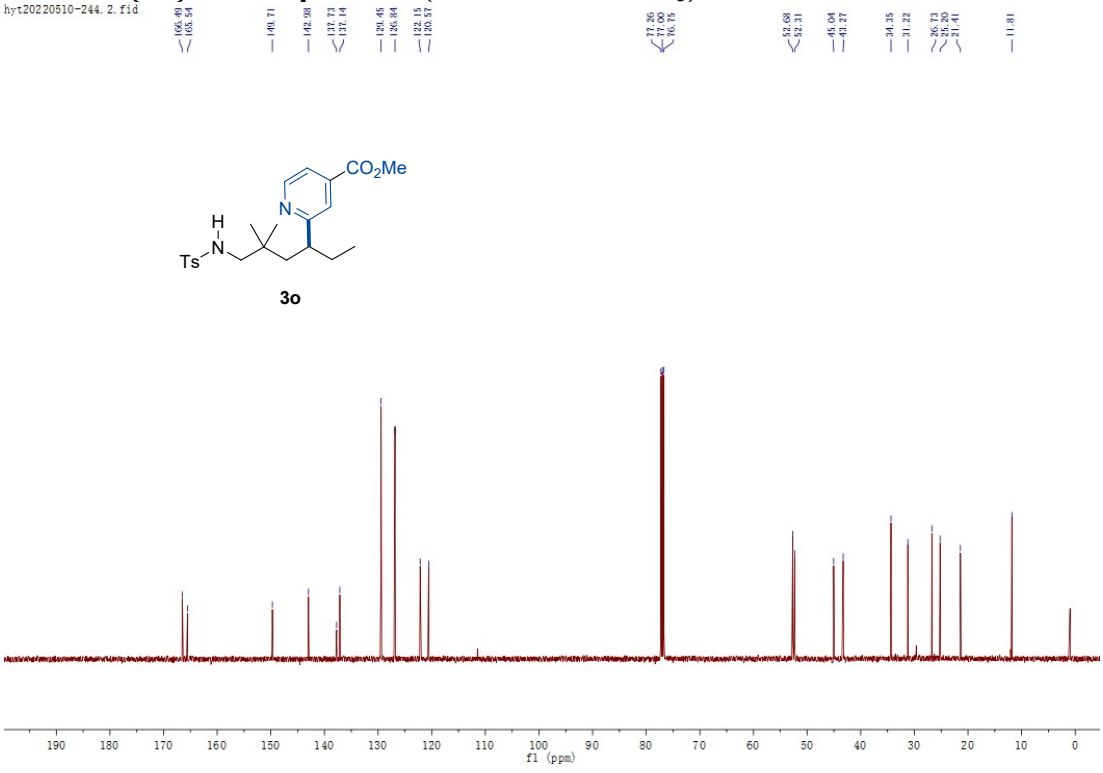


Methyl-2-(5,5-dimethyl-6-((4-methylphenyl)sulfonamido)hexan-3-yl)isonicotinate (3o).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

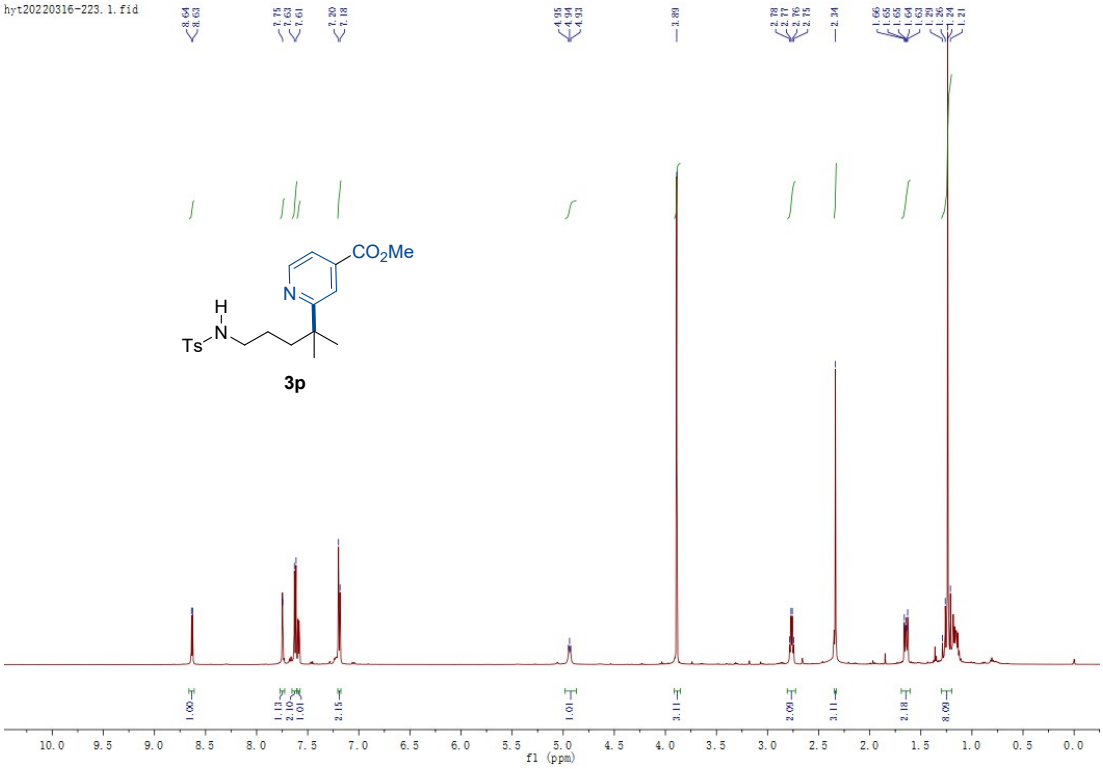


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

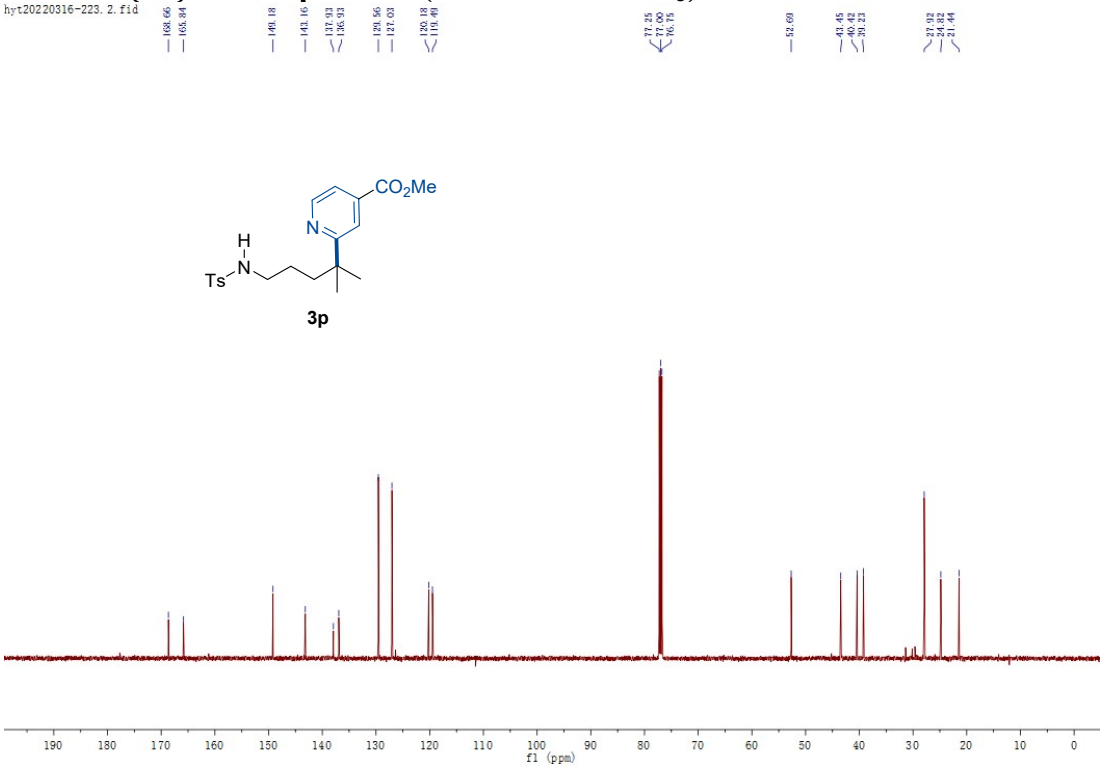


Methyl 2-(2-methyl-5-((4-methylphenyl)sulfonamido)pentan-2-yl)isonicotinate (3p).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

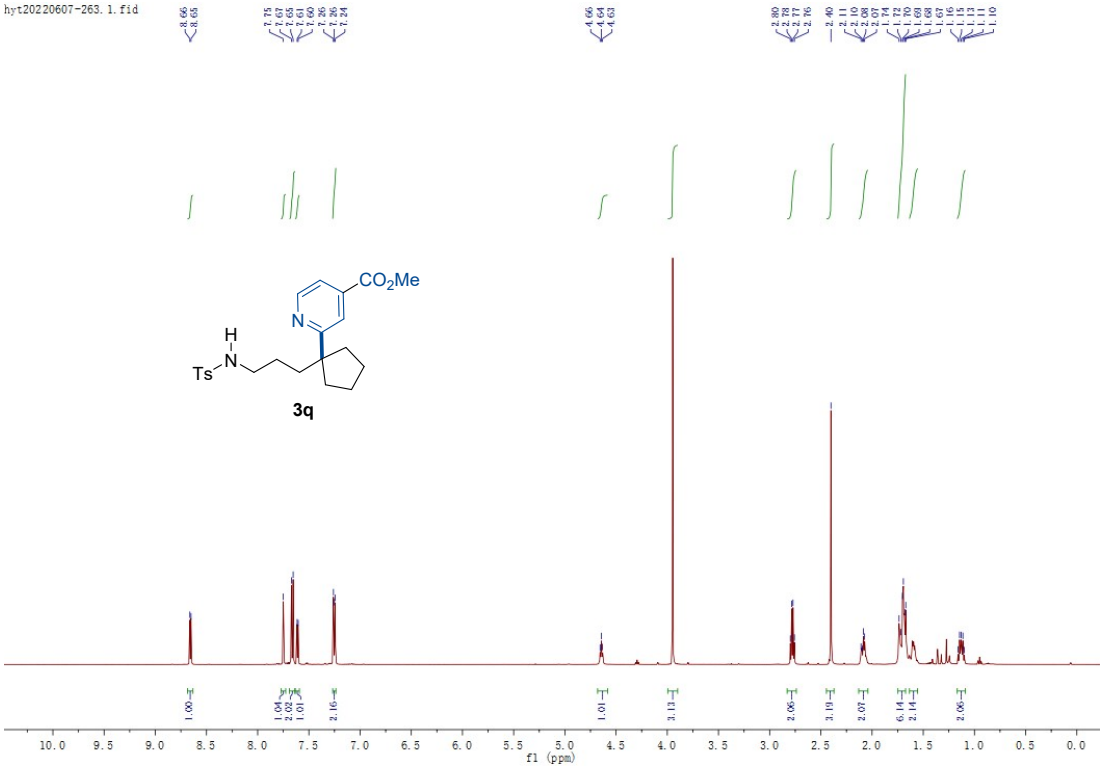


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

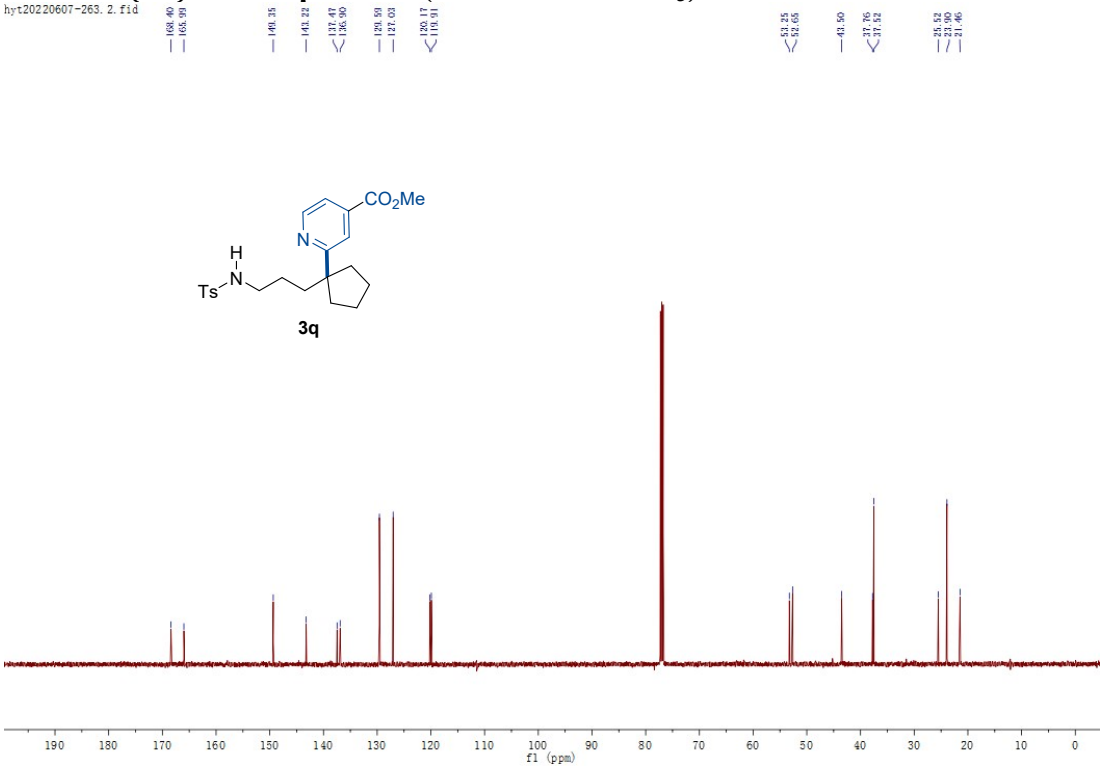


Methyl 2-(1-(3-((4-methylphenyl)sulfonamido)propyl)cyclopentyl)isonicotinate (3q).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

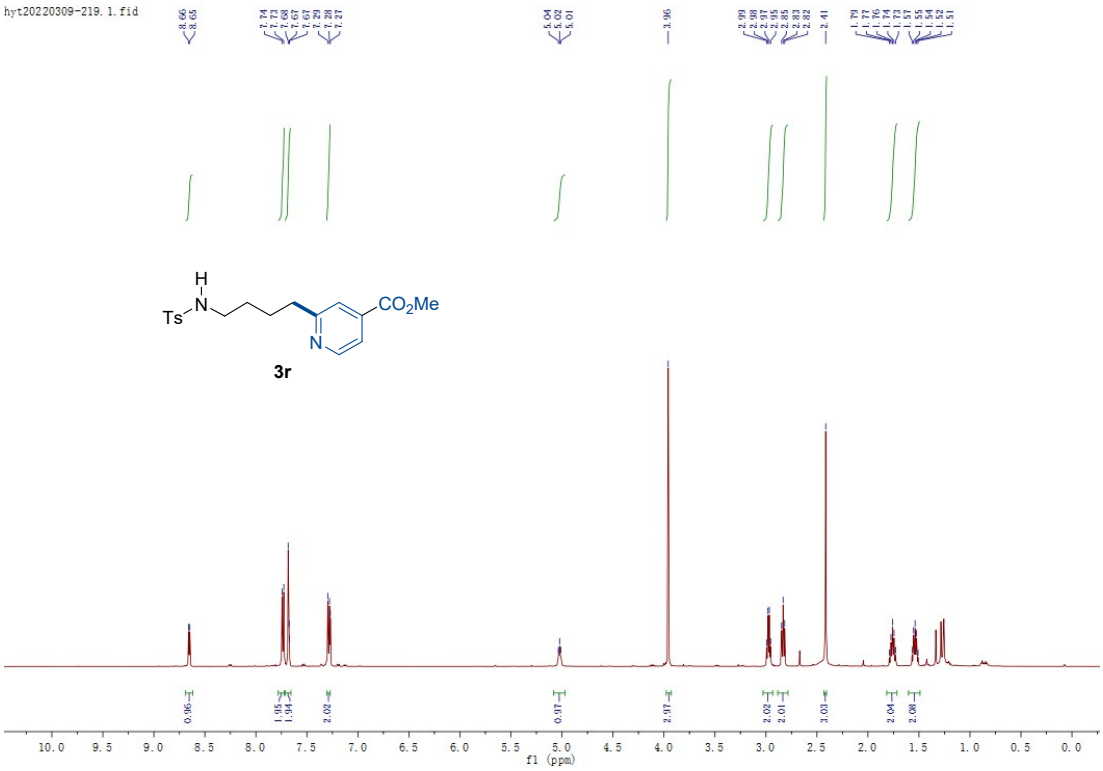


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

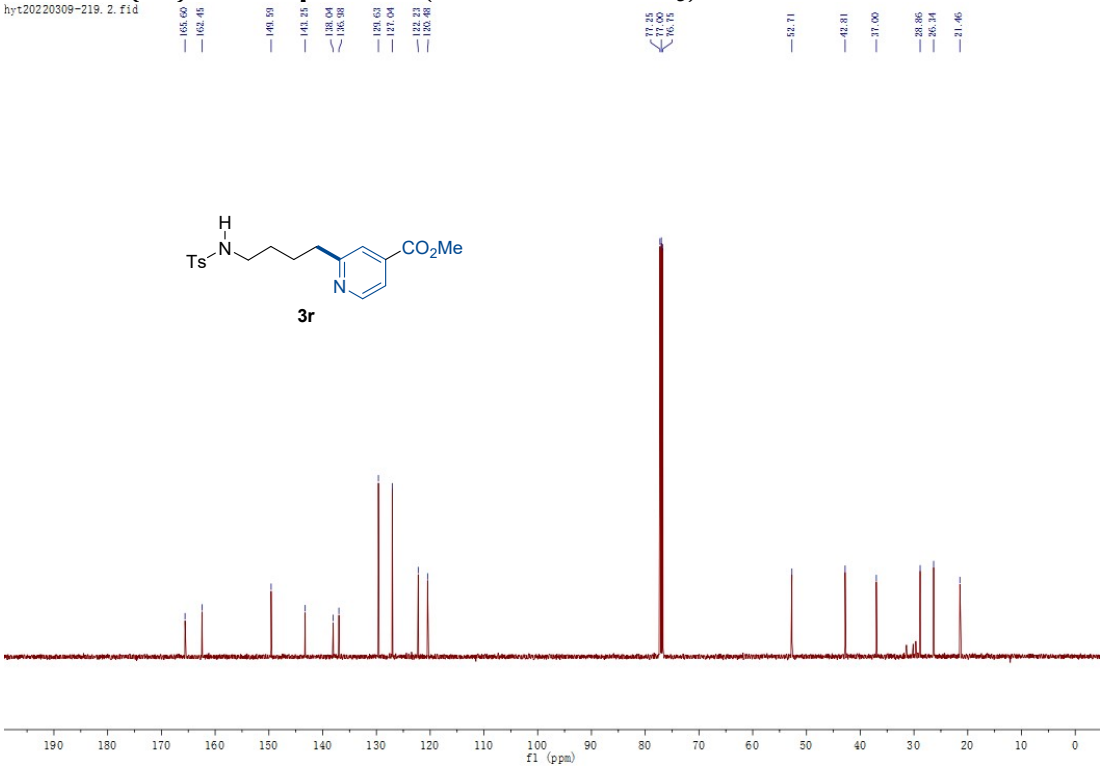


Methyl 2-(4-((4-methylphenyl)sulfonamido)butyl)isonicotinate (3r).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

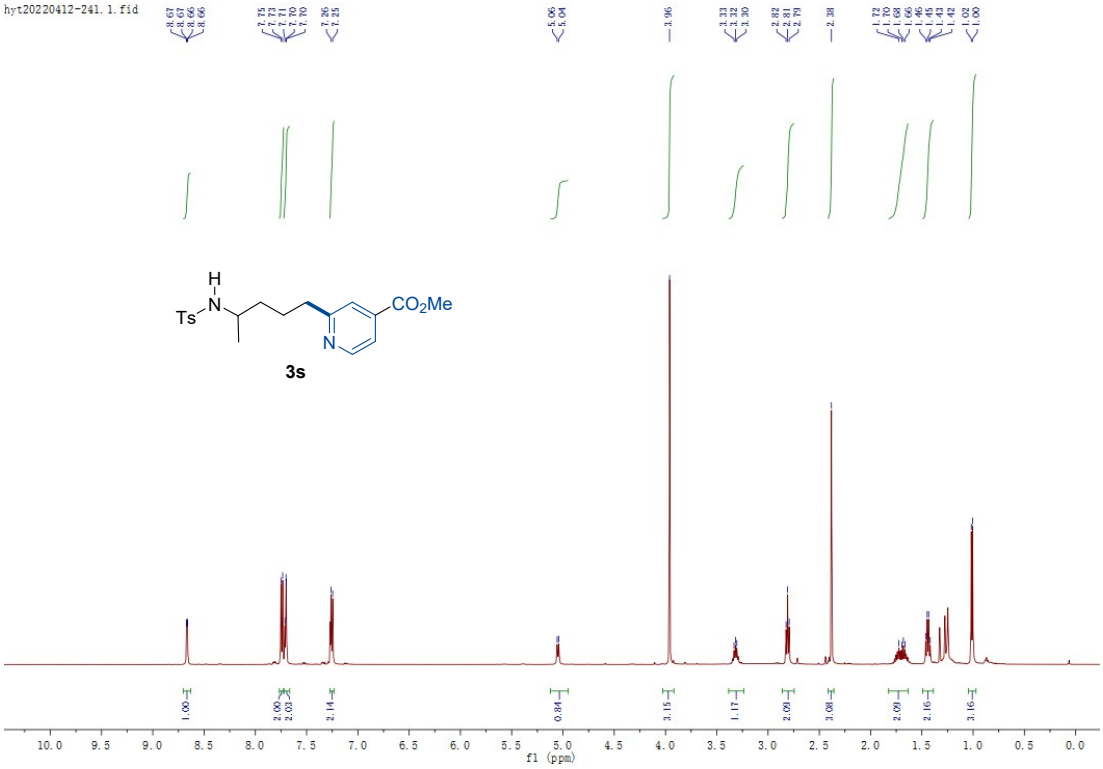


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

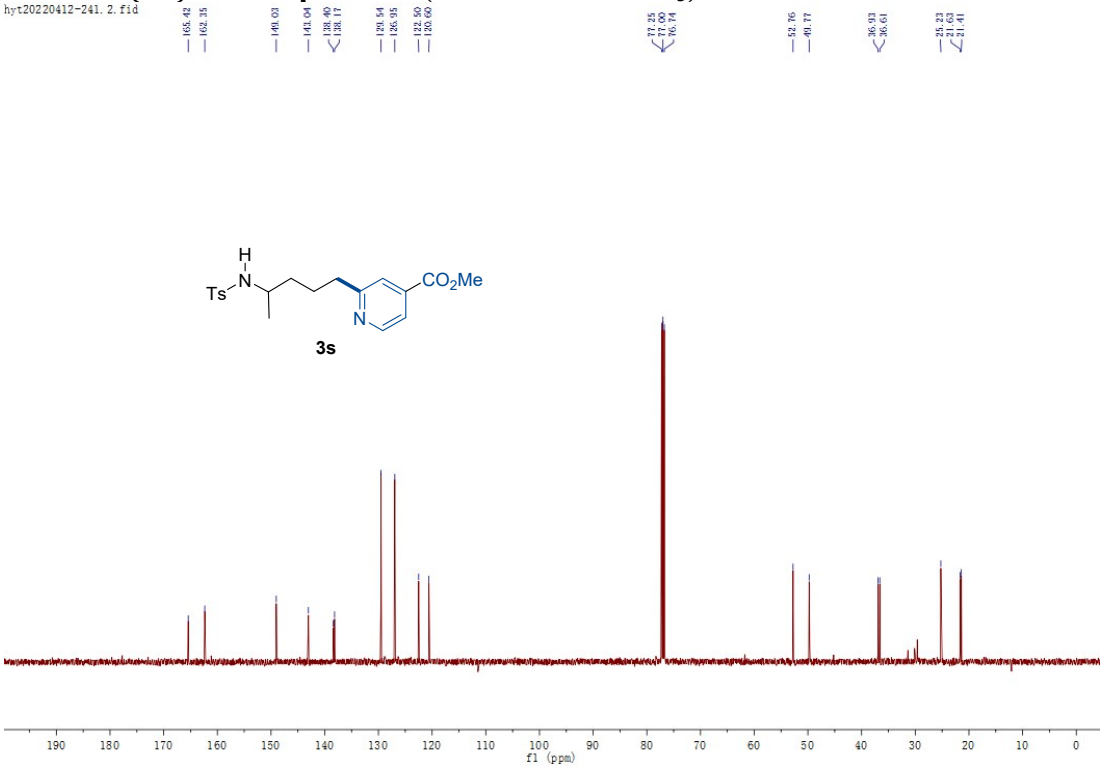


Methyl 2-(4-((4-methylphenyl)sulfonamido)pentyl)isonicotinate (3s).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

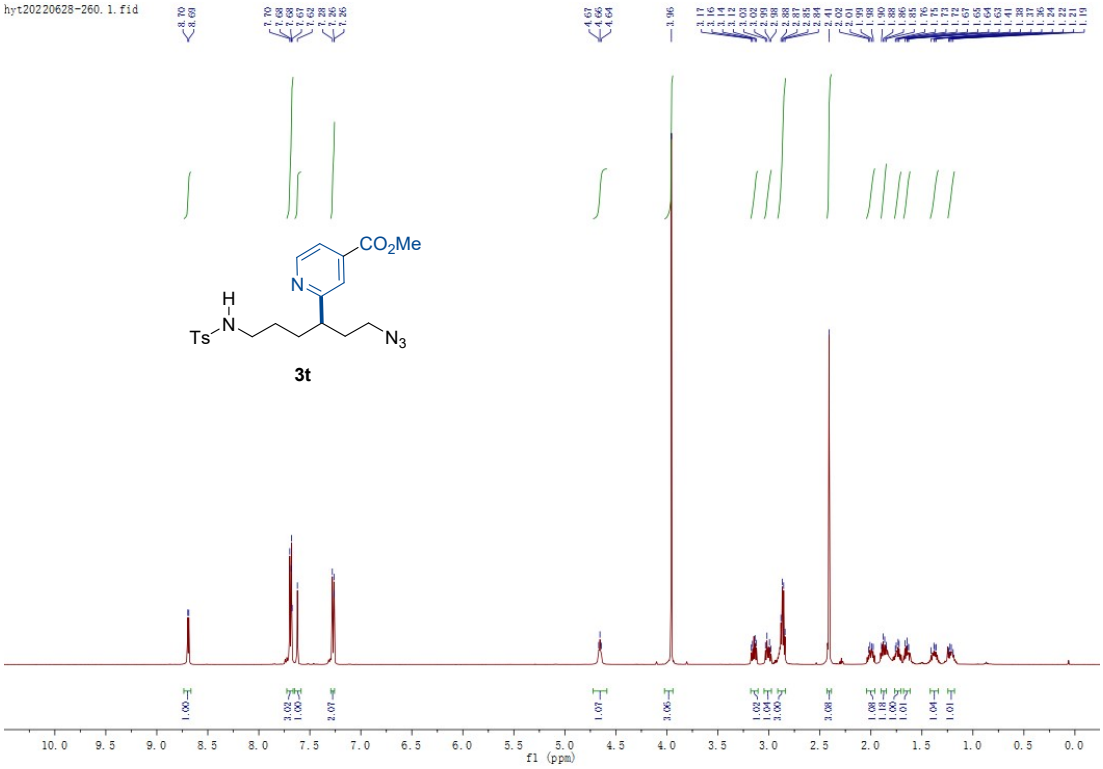


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

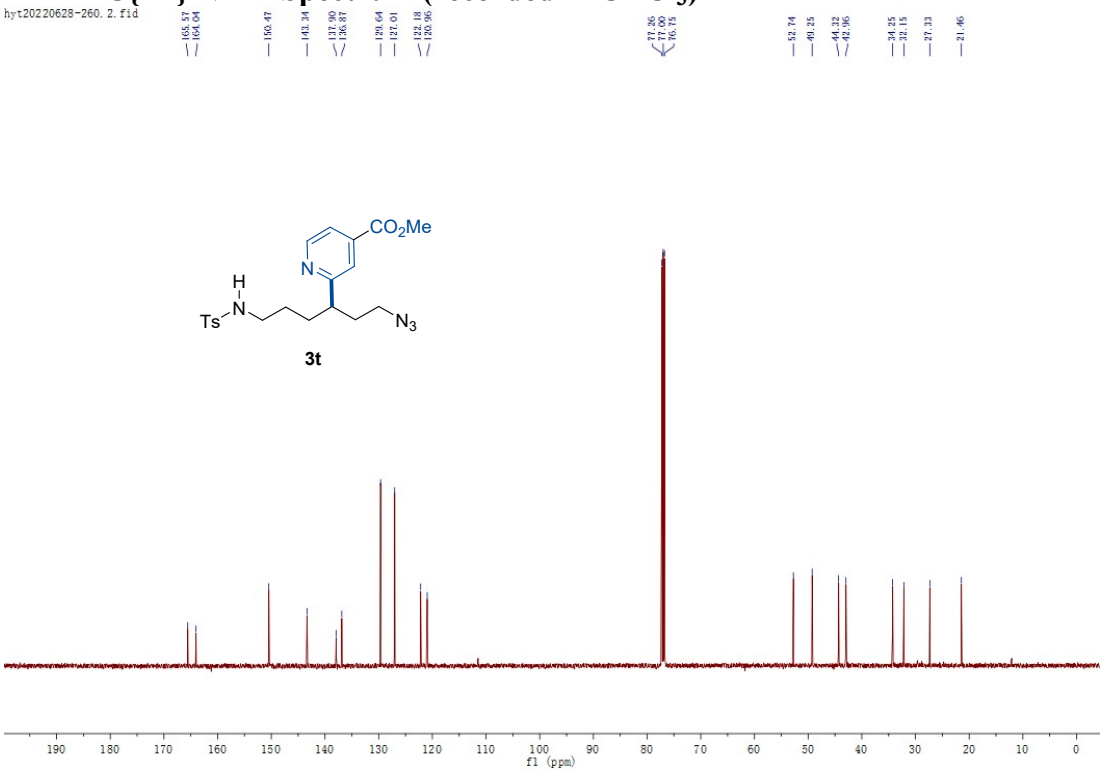


Methyl-2-(1-azido-6-((4-methylphenyl)sulfonamido)hexan-3-yl)isonicotinate (3t).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

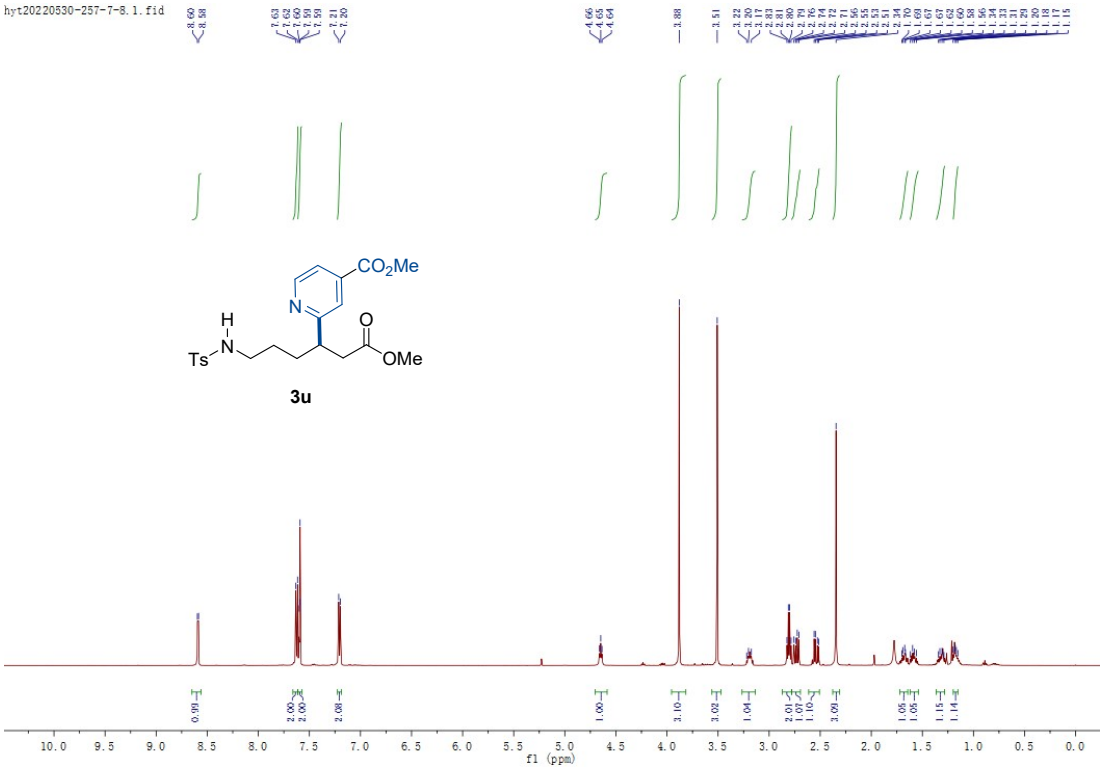


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

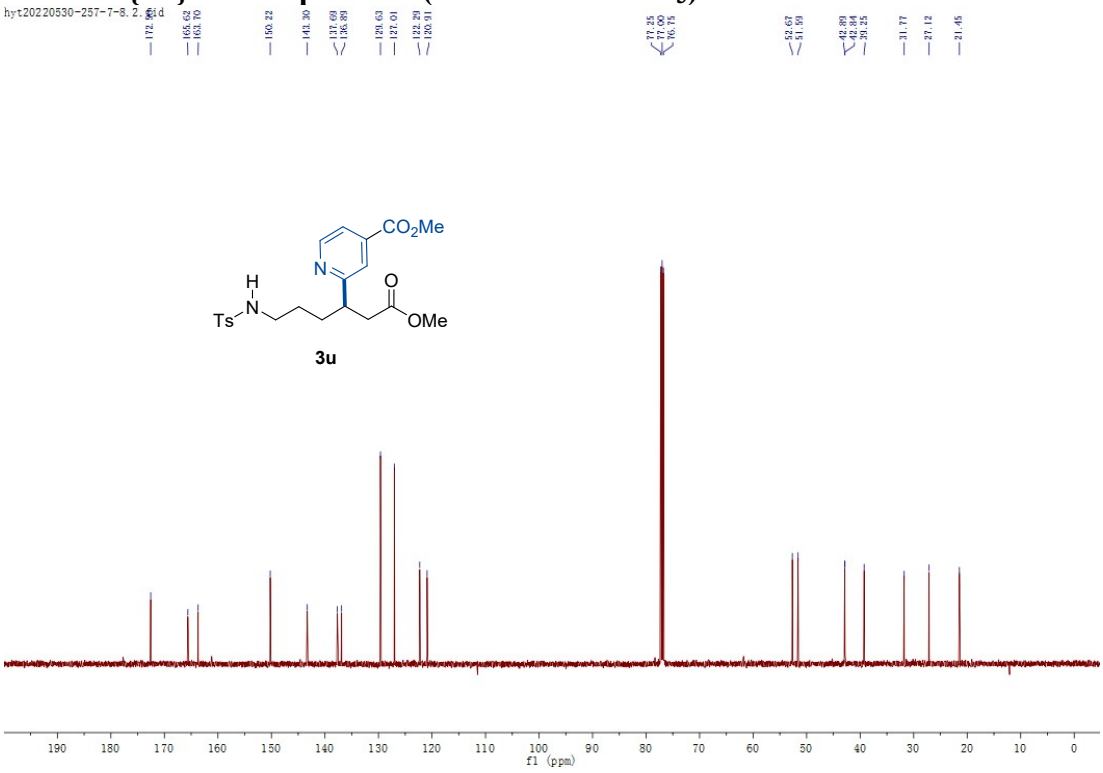


Methyl-2-(1-methoxy-6-((4-methylphenyl)sulfonamido)-1-oxohexan-3-yl)isonicotinate (3u).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

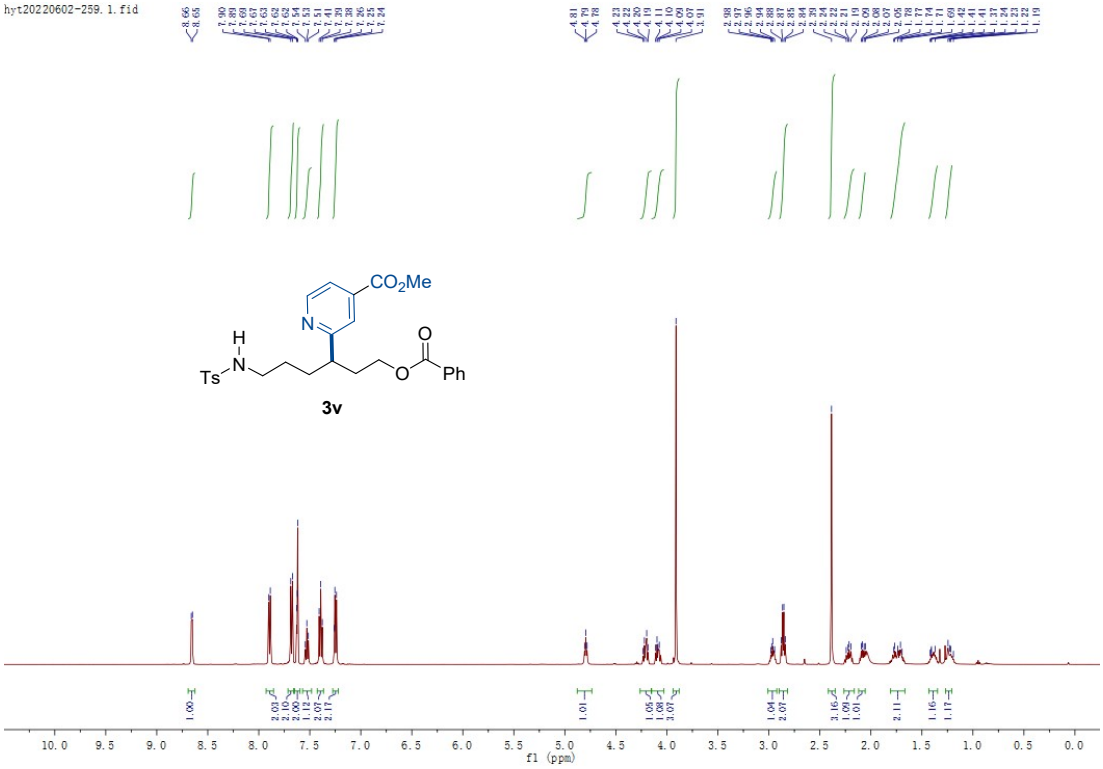


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

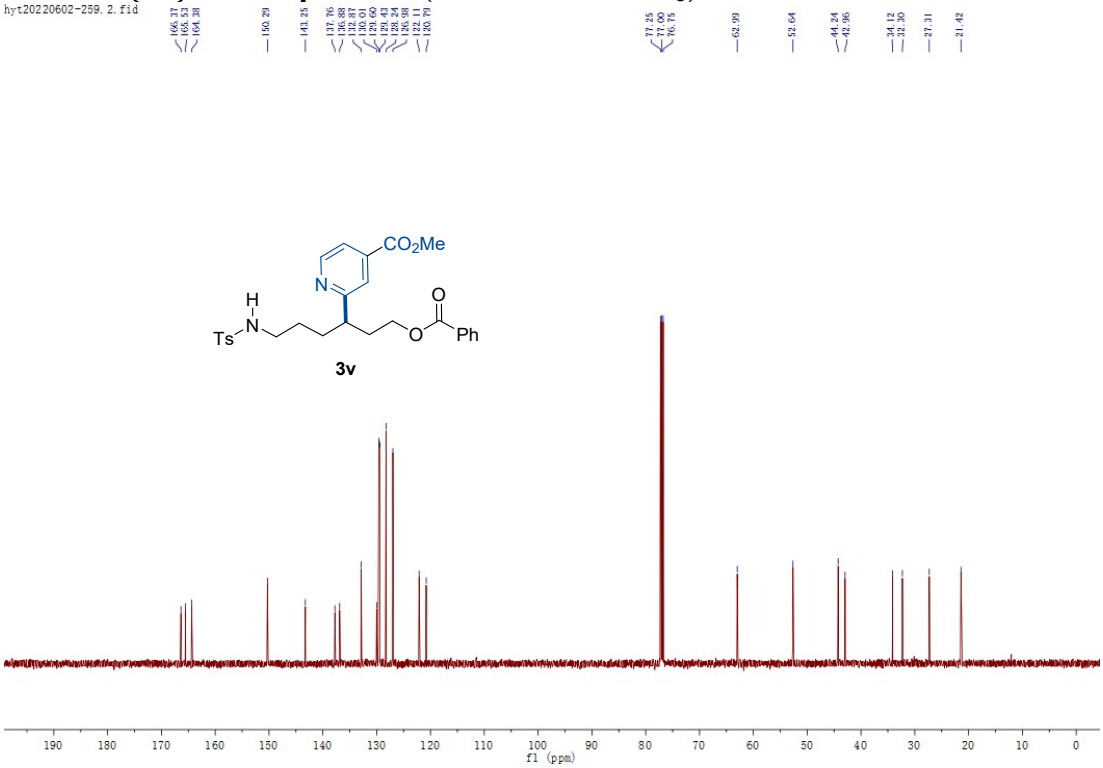


Methyl-2-(1-(benzoyloxy)-6-((4-methylphenyl)sulfonamido)hexan-3-yl)isonicotinate (3v).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

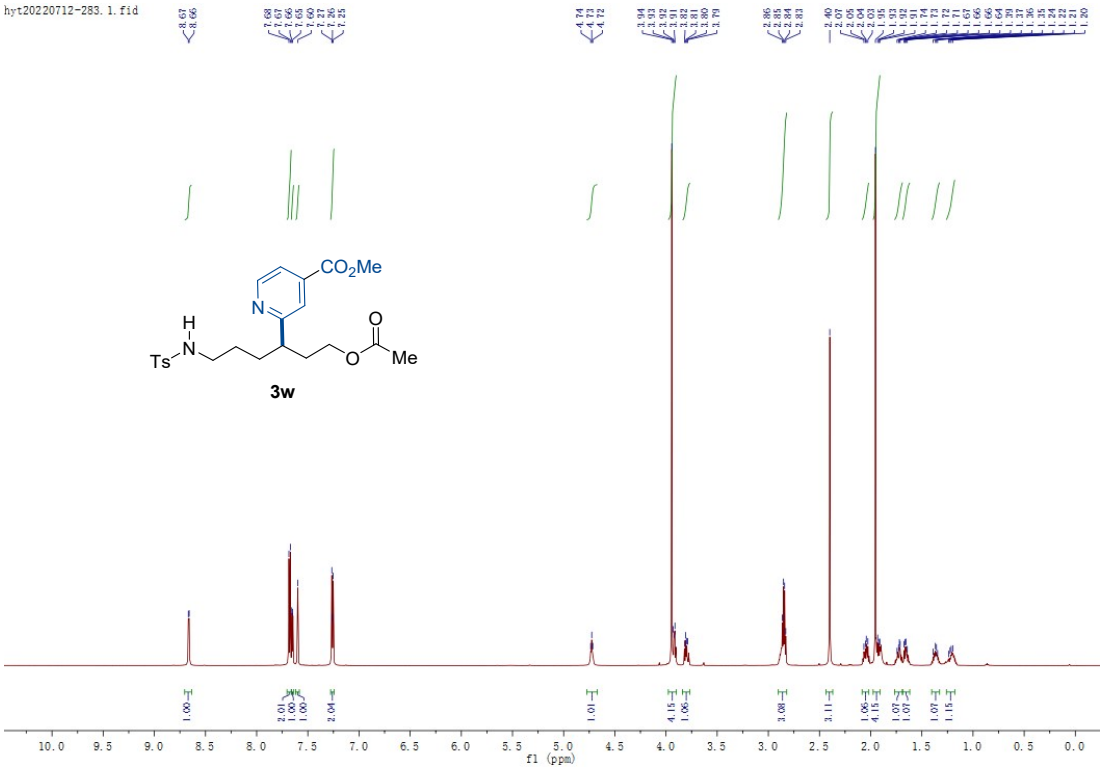


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

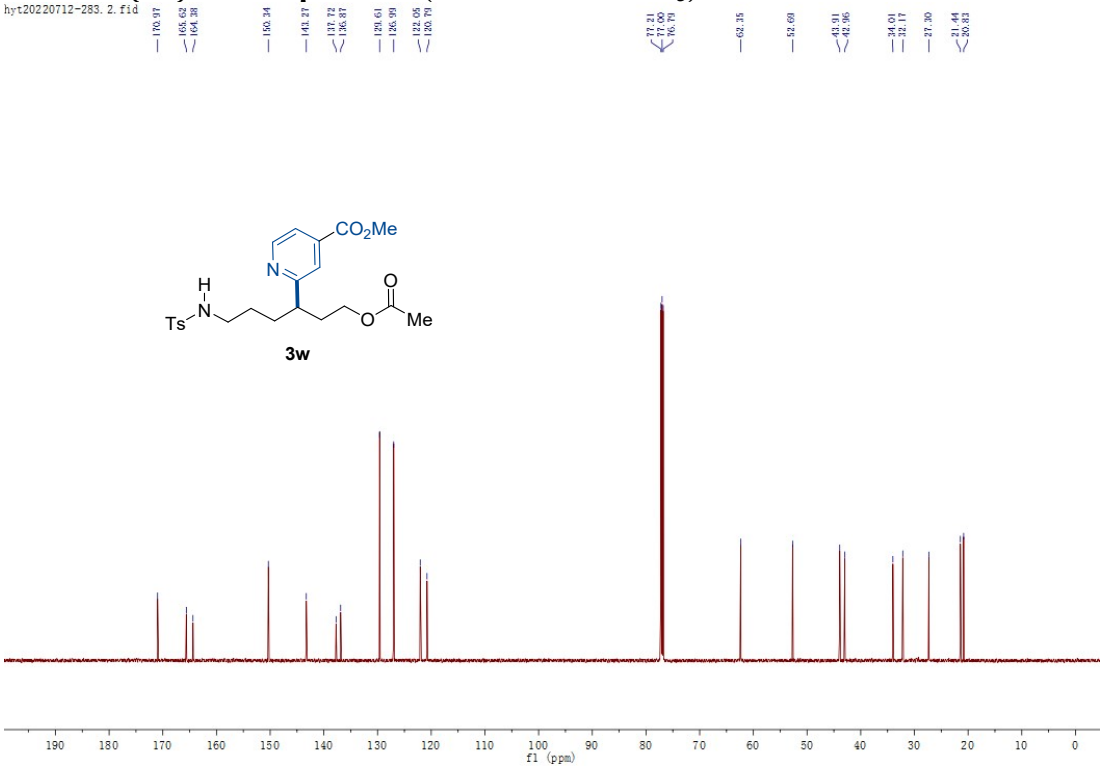


Methyl-2-(1-acetoxy-6-((4-methylphenyl)sulfonamido)hexan-3-yl)isonicotinate (3w).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)



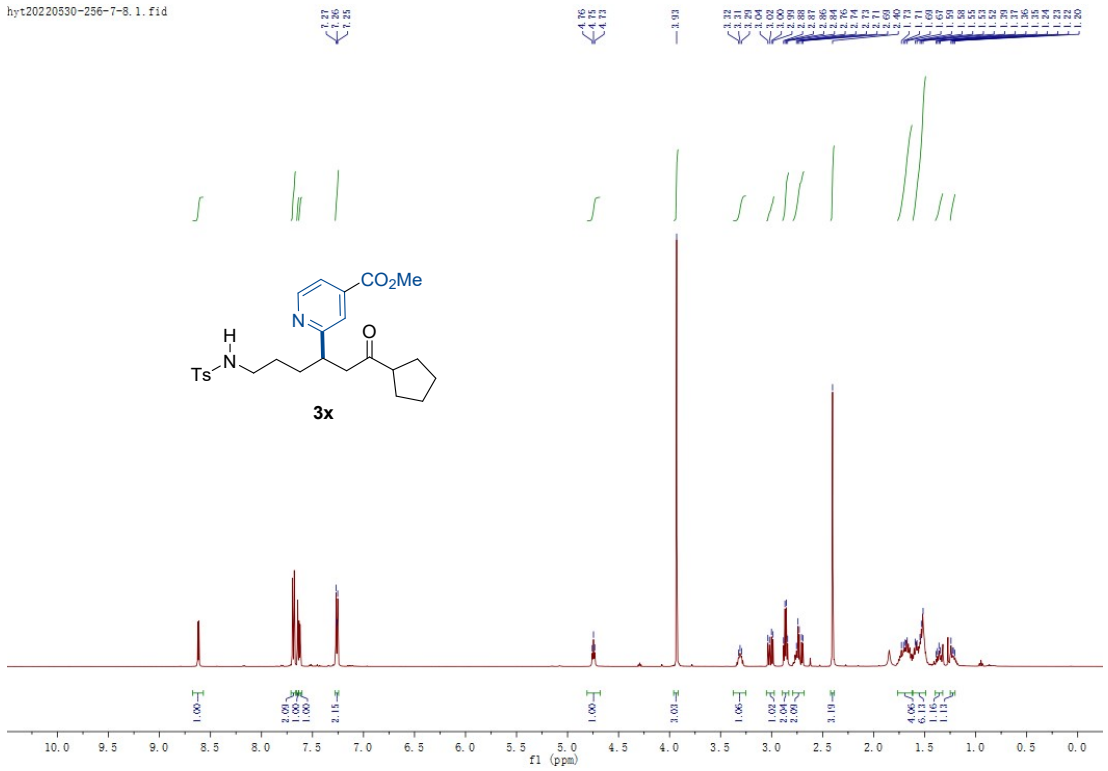
126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)



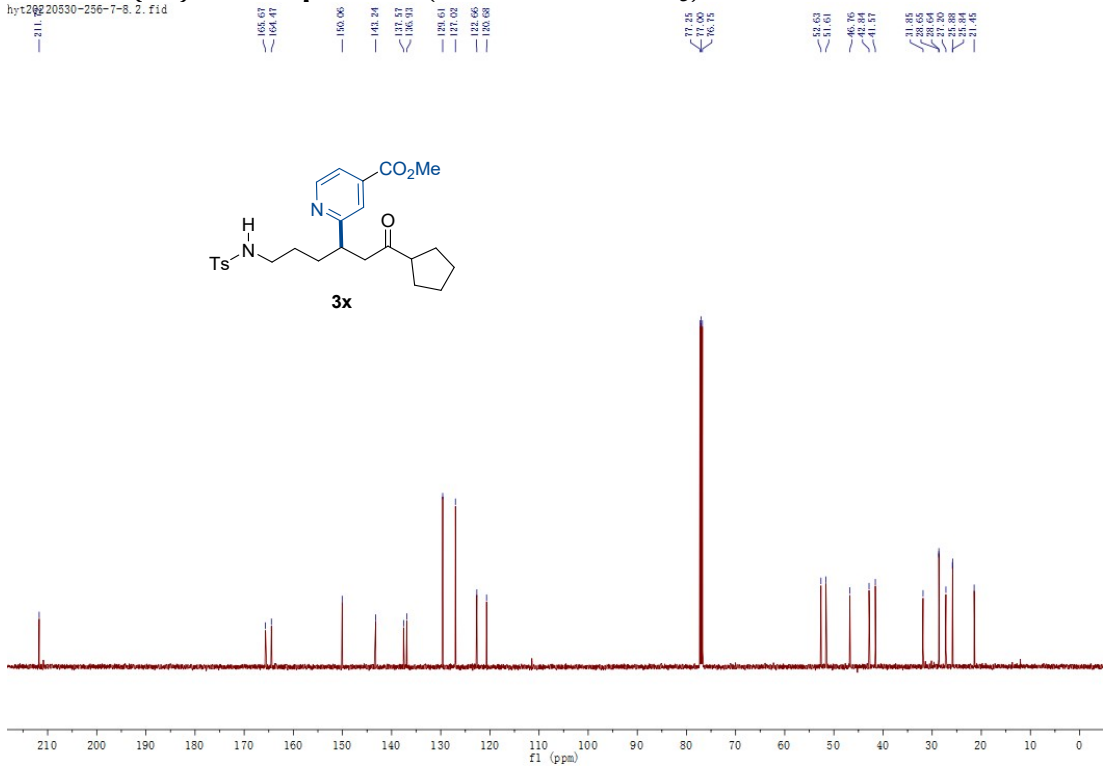
Methyl-2-(1-cyclopentyl-6-((4-methylphenyl)sulfonamido)-1-oxohexan-3-yl)isonicotinate

(3x).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)



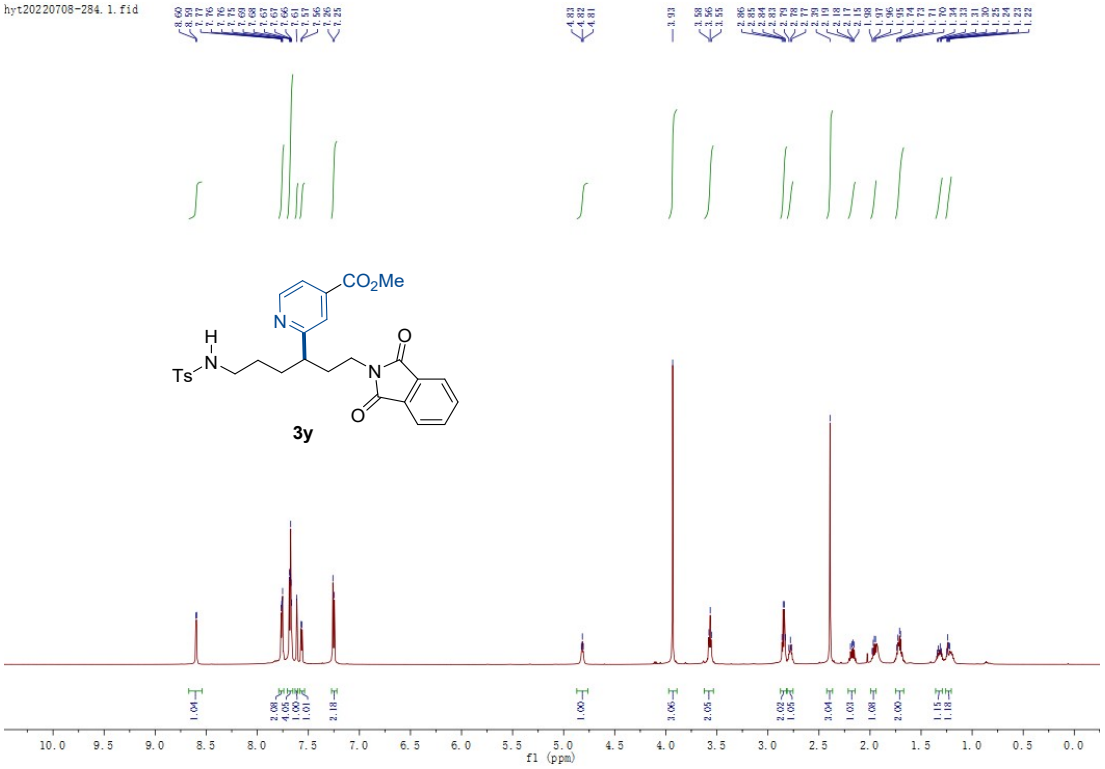
126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)



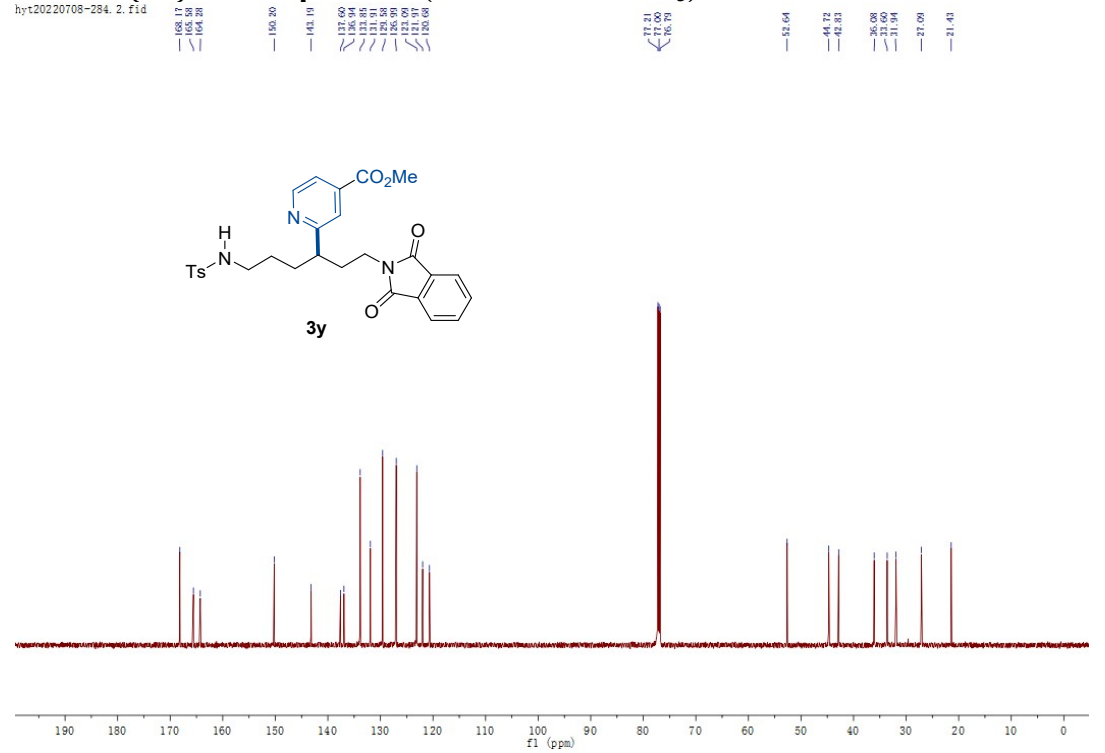
Methyl-2-(1-(1,3-dioxoisoindolin-2-yl)-6-((4-methylphenyl)sulfonamido)hexan-3-

yl)isonicotinate (3y).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

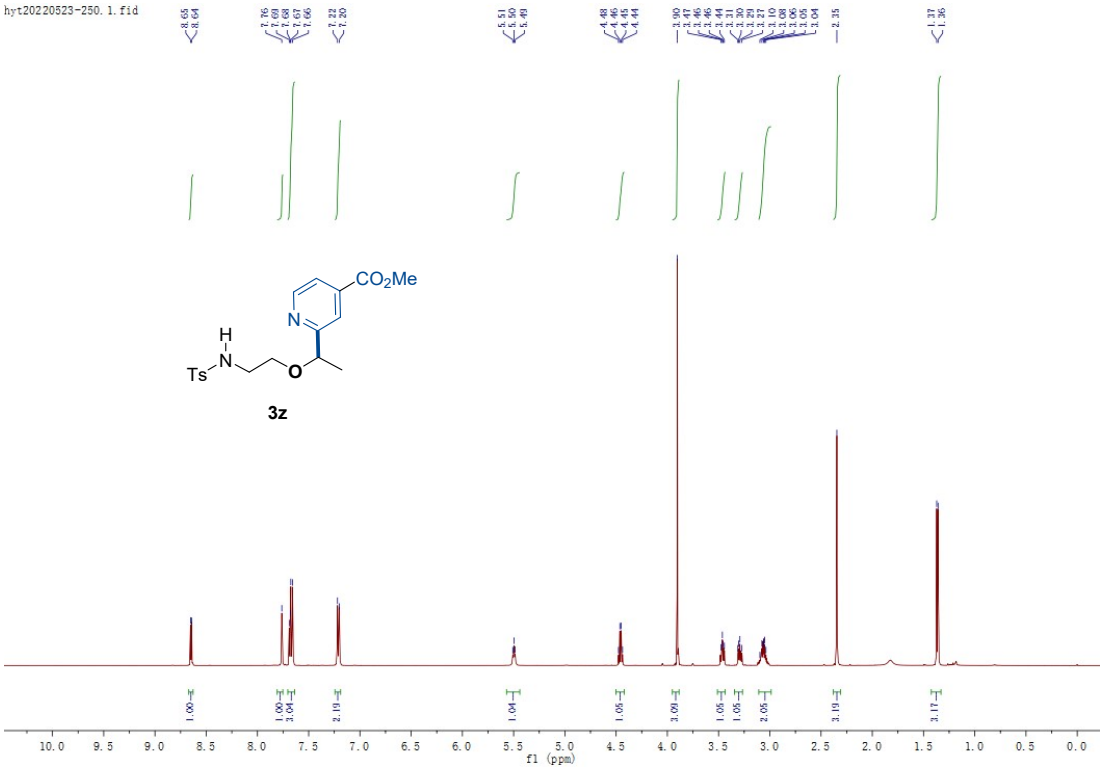


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

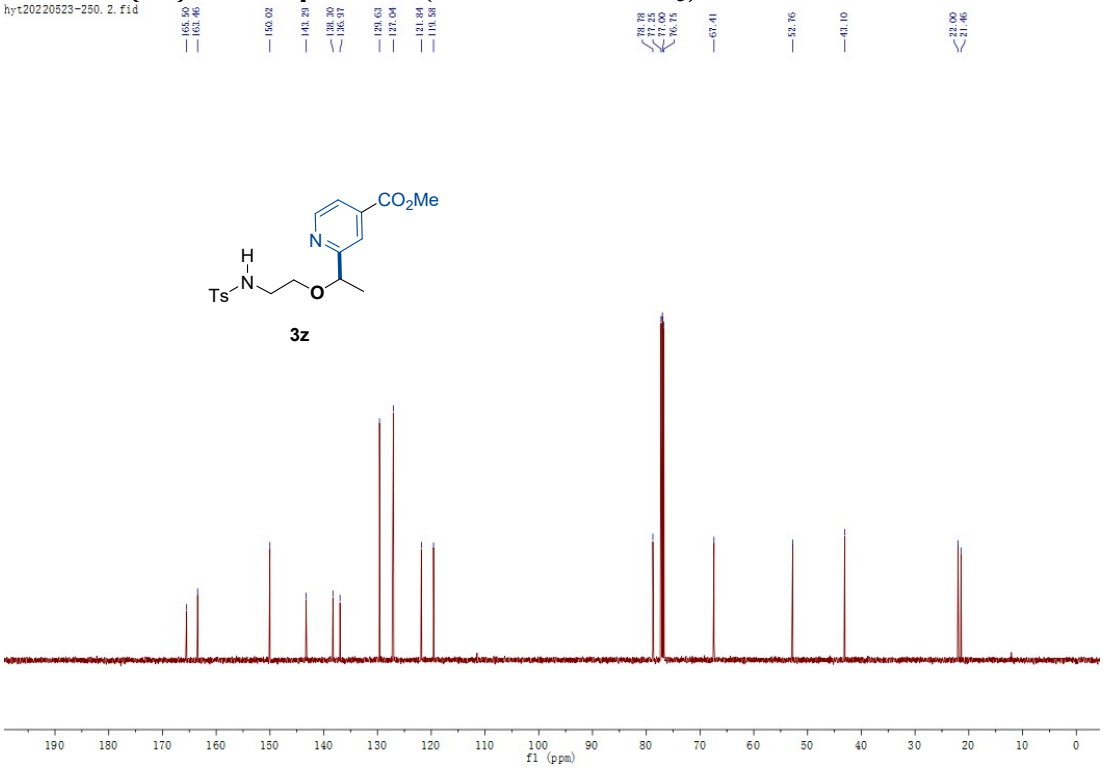


Methyl-2-(1-(2-((4-methylphenyl)sulfonamido)ethoxy)ethyl)isonicotinate (3z).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

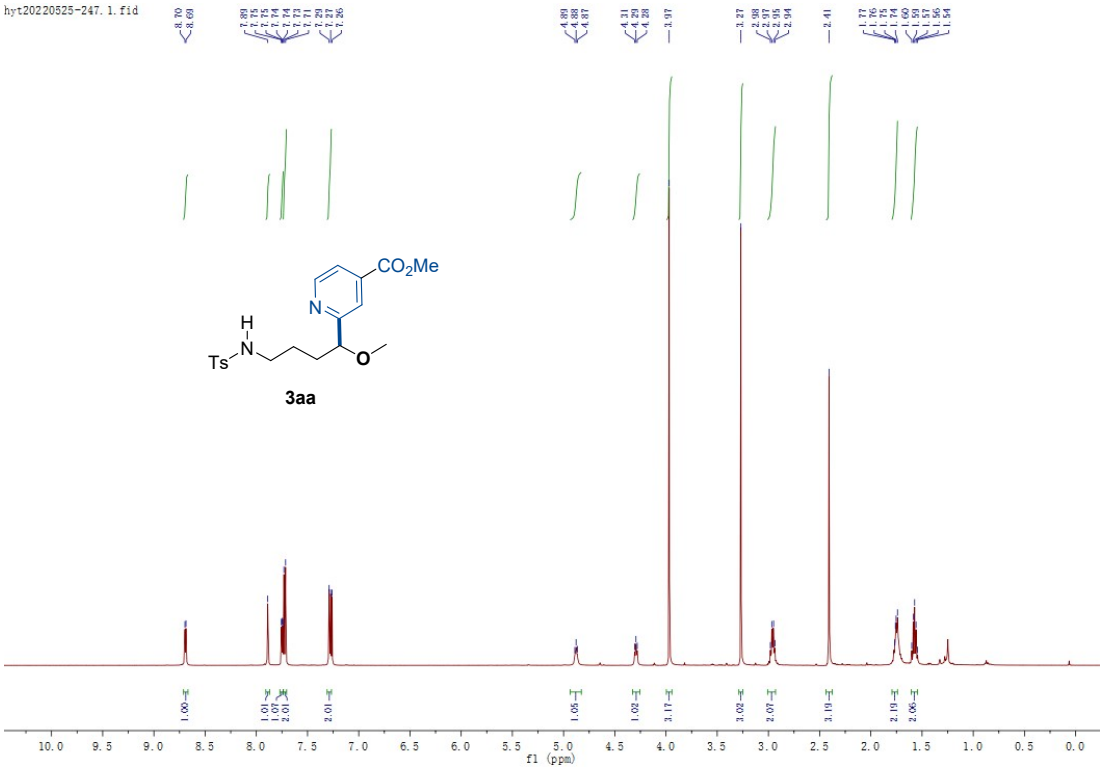


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

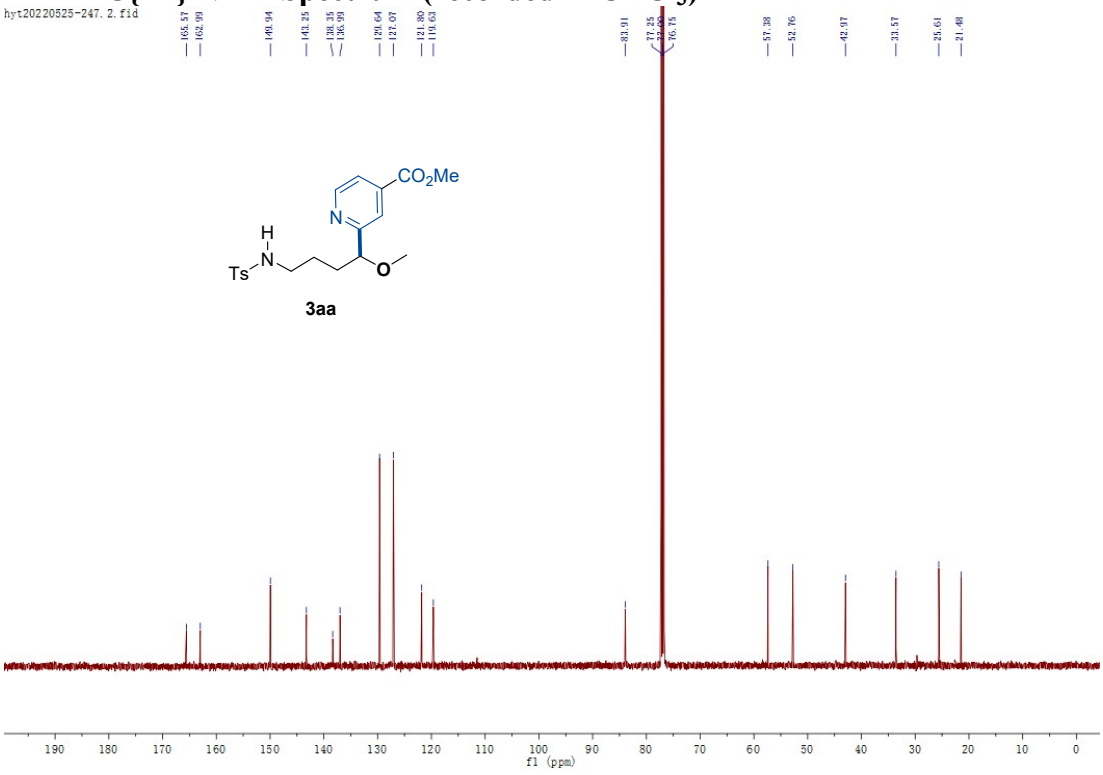


Methyl-2-(1-methoxy-4-((4-methylphenyl)sulfonamido)butyl)isonicotinate (3aa).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

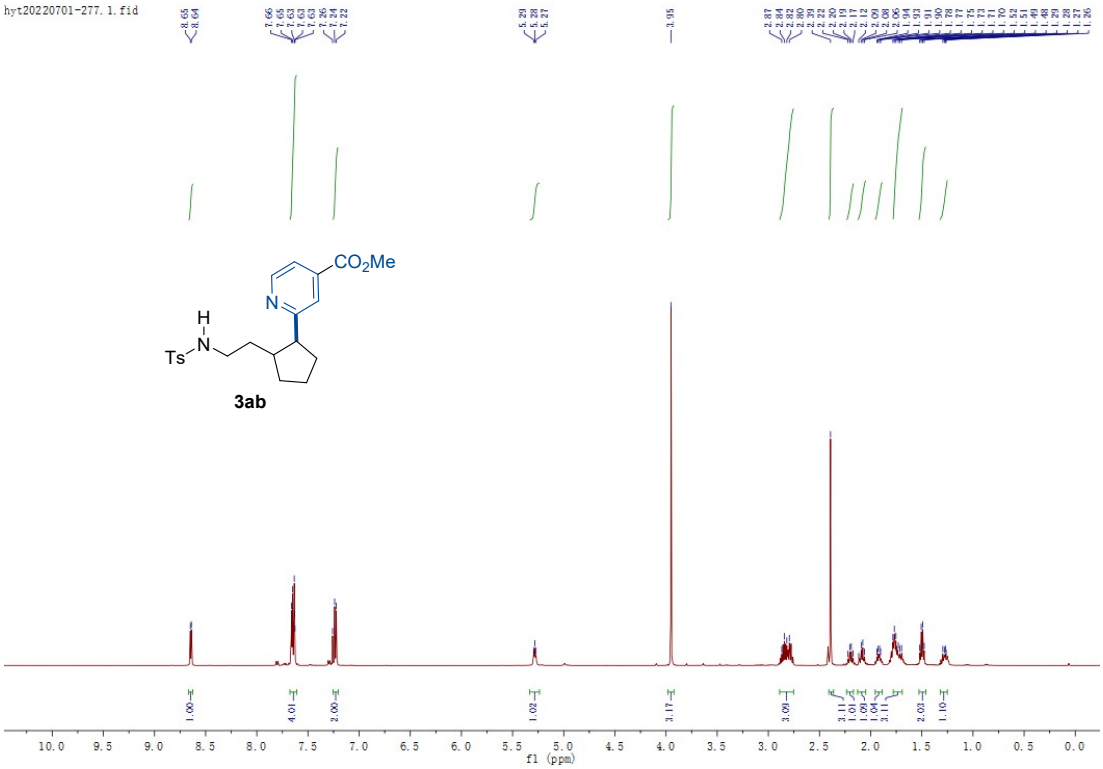


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

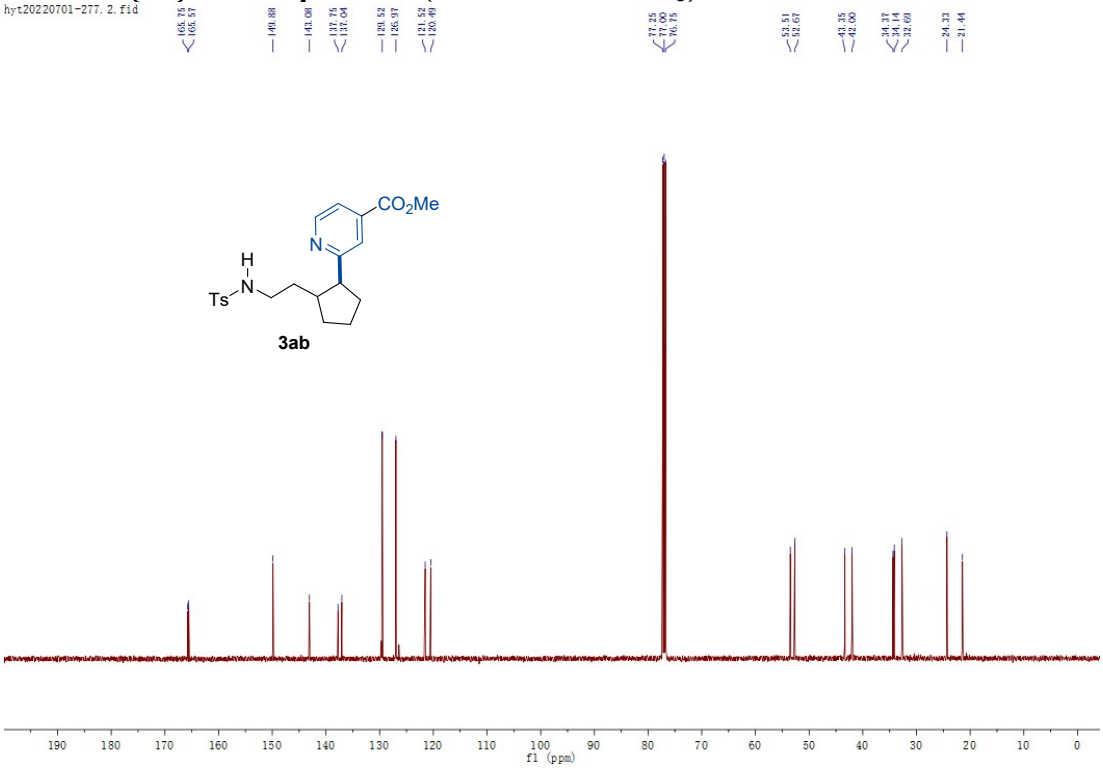


Methyl 2-(2-(2-((4-methylphenyl)sulfonamido)ethyl)cyclopentyl)isonicotinate (3ab).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

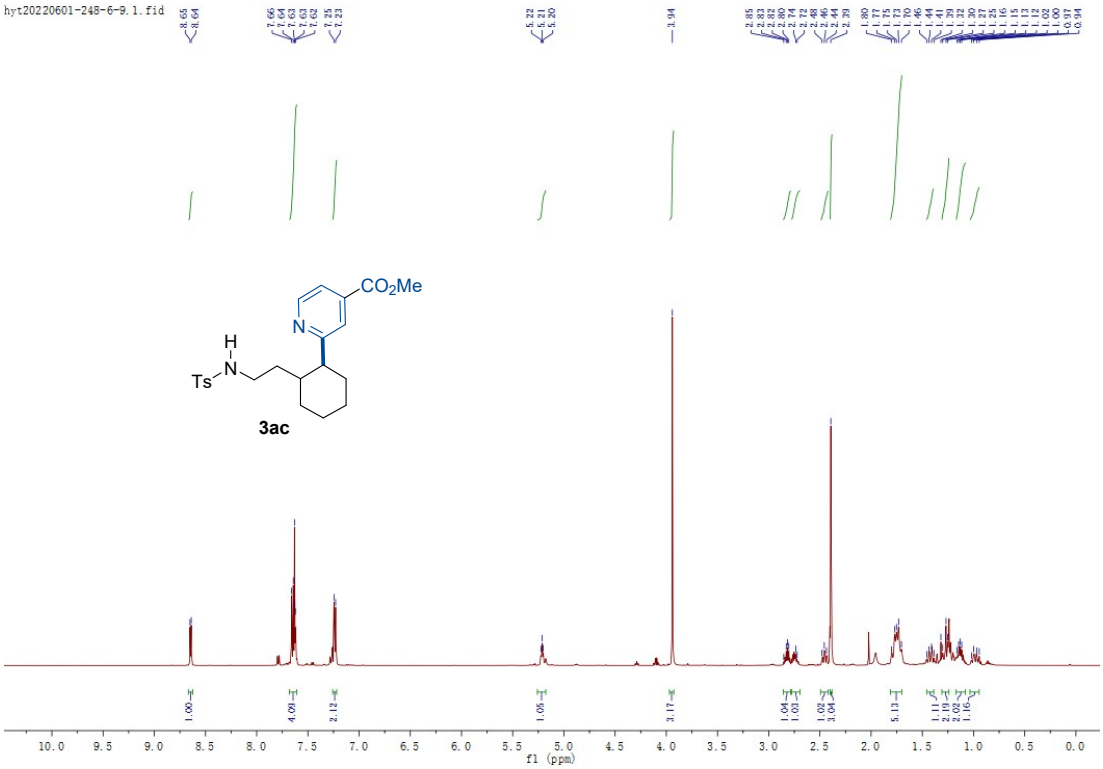


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

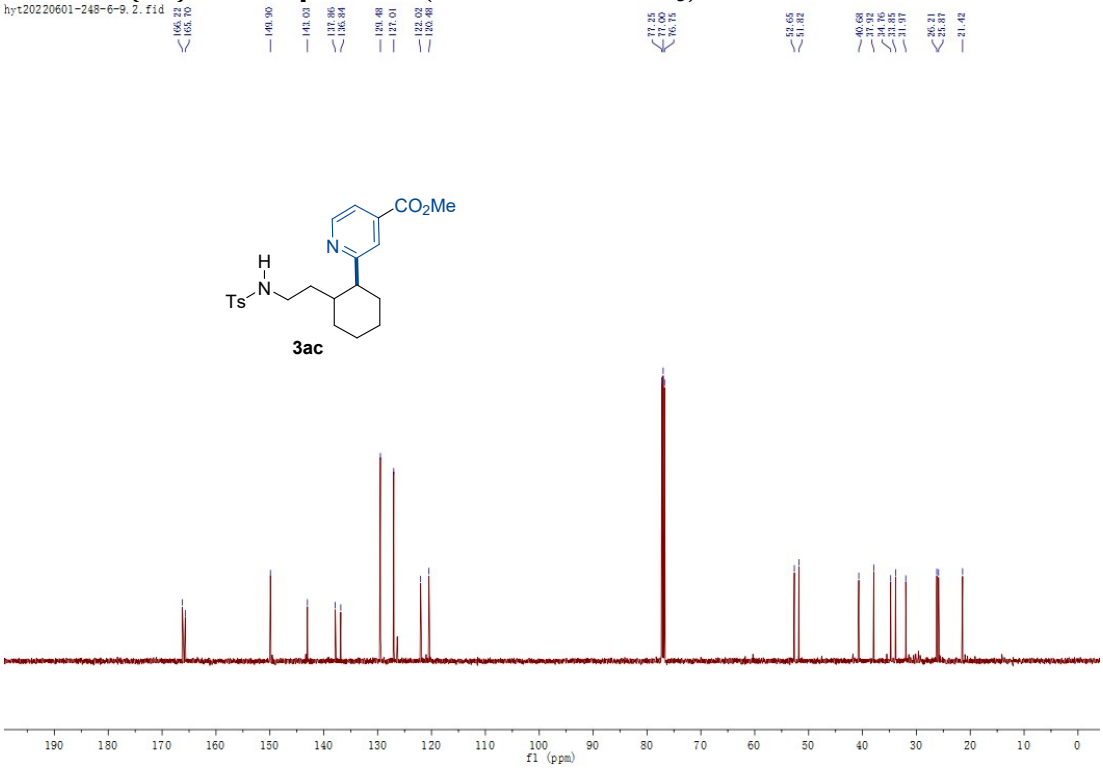


Methyl 2-(2-(2-((4-methylphenyl)sulfonamido)ethyl)cyclohexyl)isonicotinate (3ac).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)



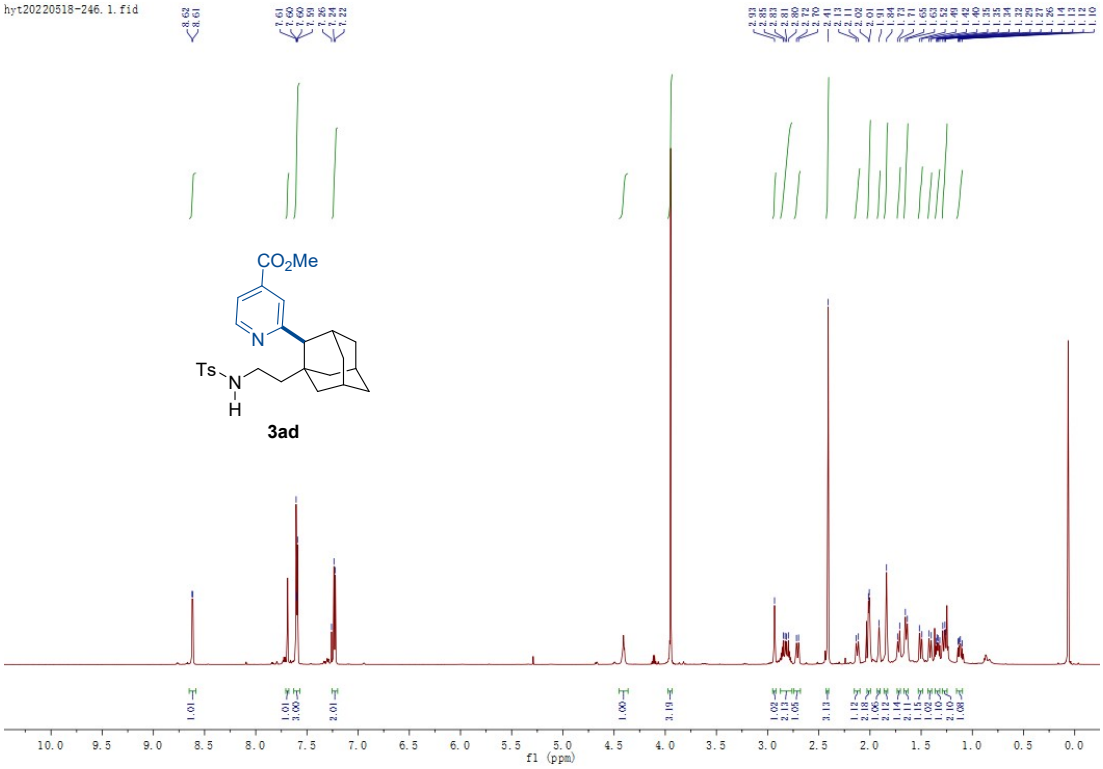
126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)



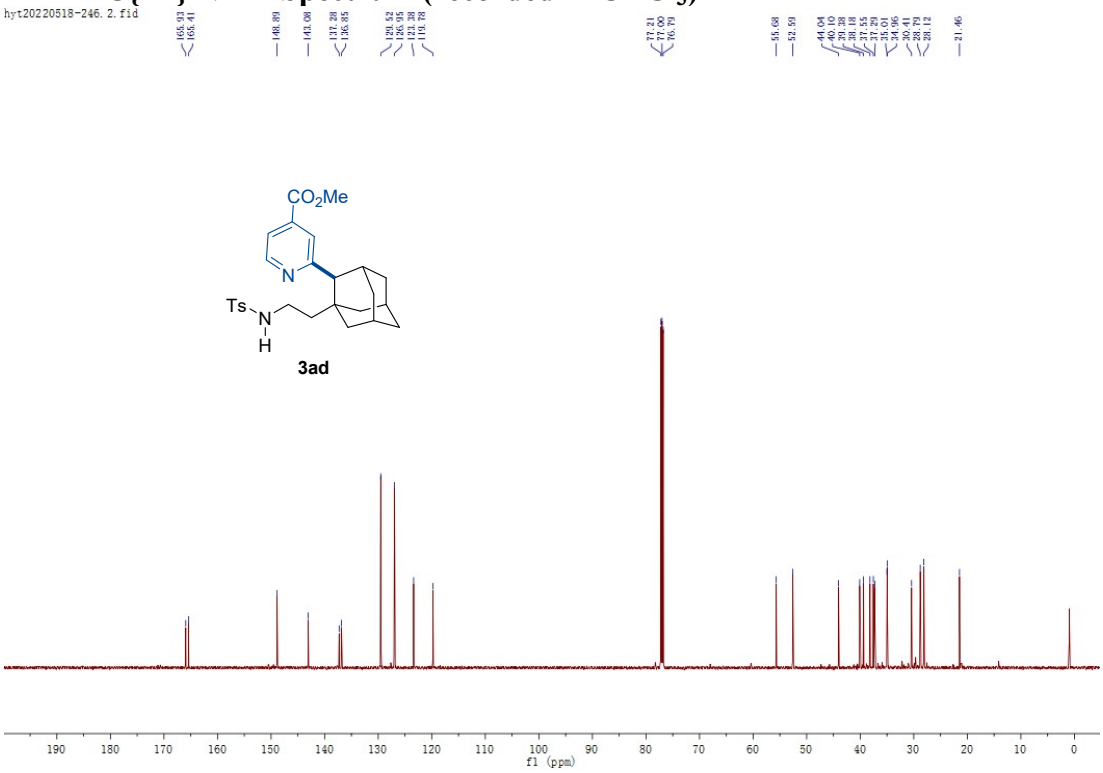
Methyl 2-((1r,3s,5R,7S)-1-(2-((4-methylphenyl)sulfonamido)ethyl)adamantan-2-yl)isoni-

cotinate (3ad).

600 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

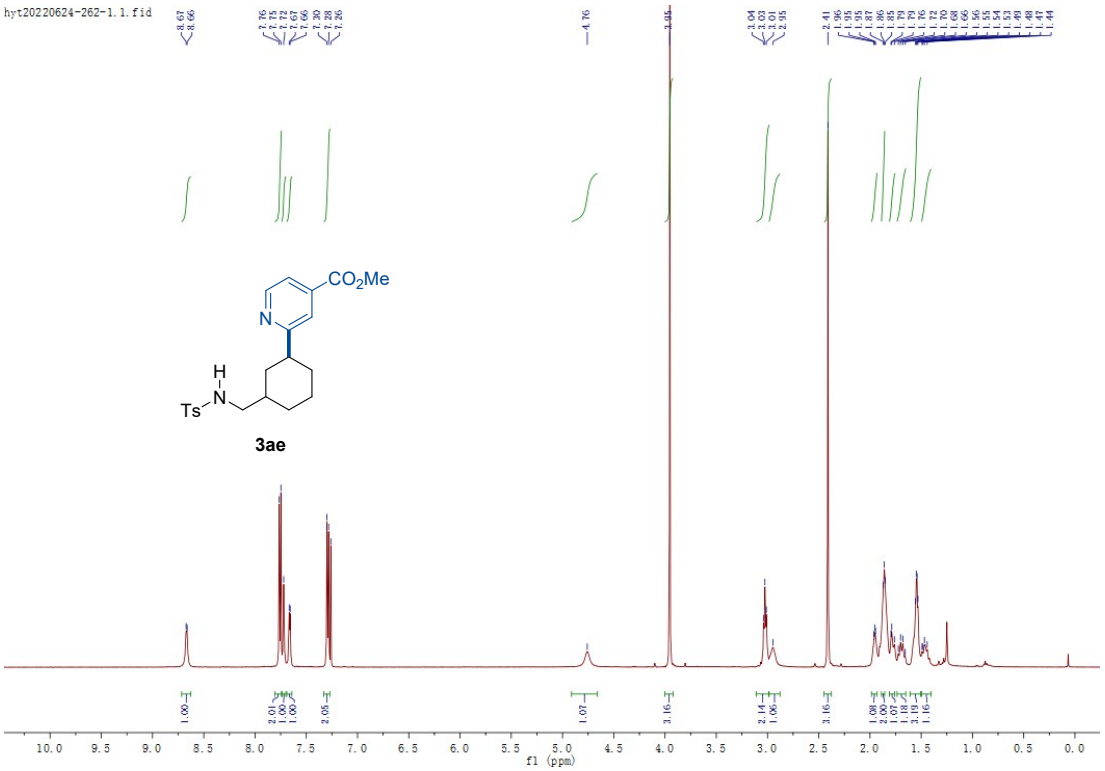


151 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

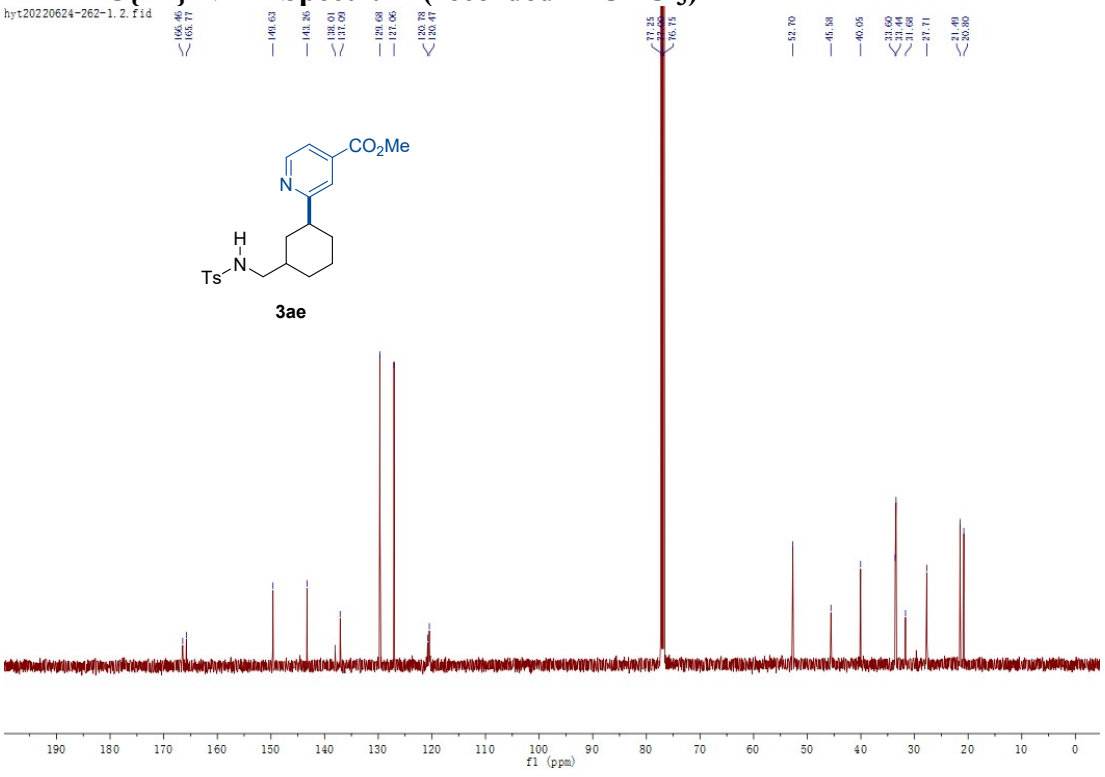


Methyl 2-(3-(((4-methylphenyl)sulfonamido)methyl)cyclohexyl)isonicotinate (3ae).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

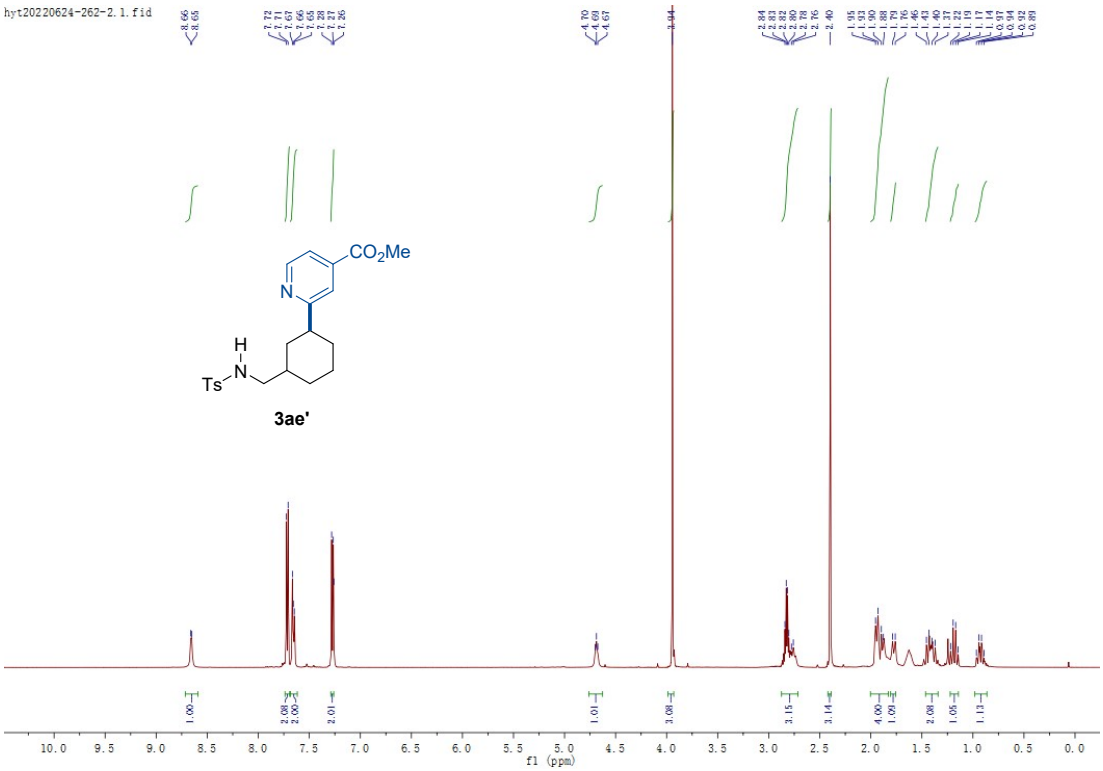


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

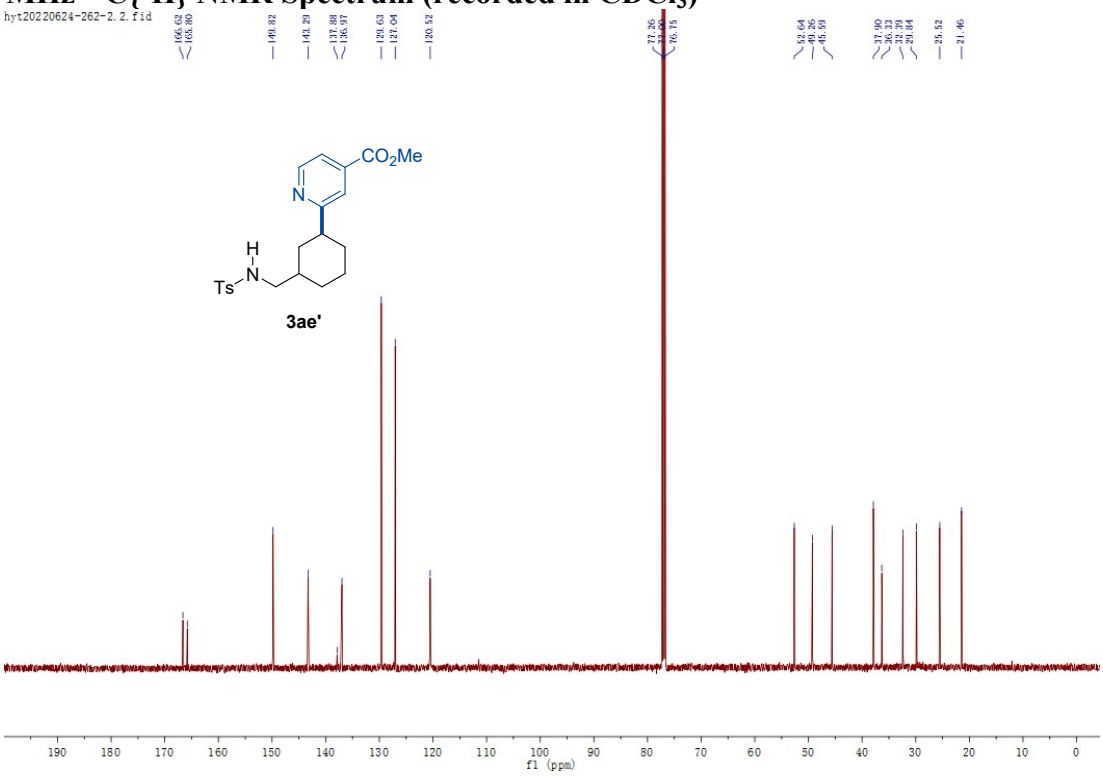


Methyl 2-(3-(((4-methylphenyl)sulfonamido)methyl)cyclohexyl)isonicotinate (3ae').

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

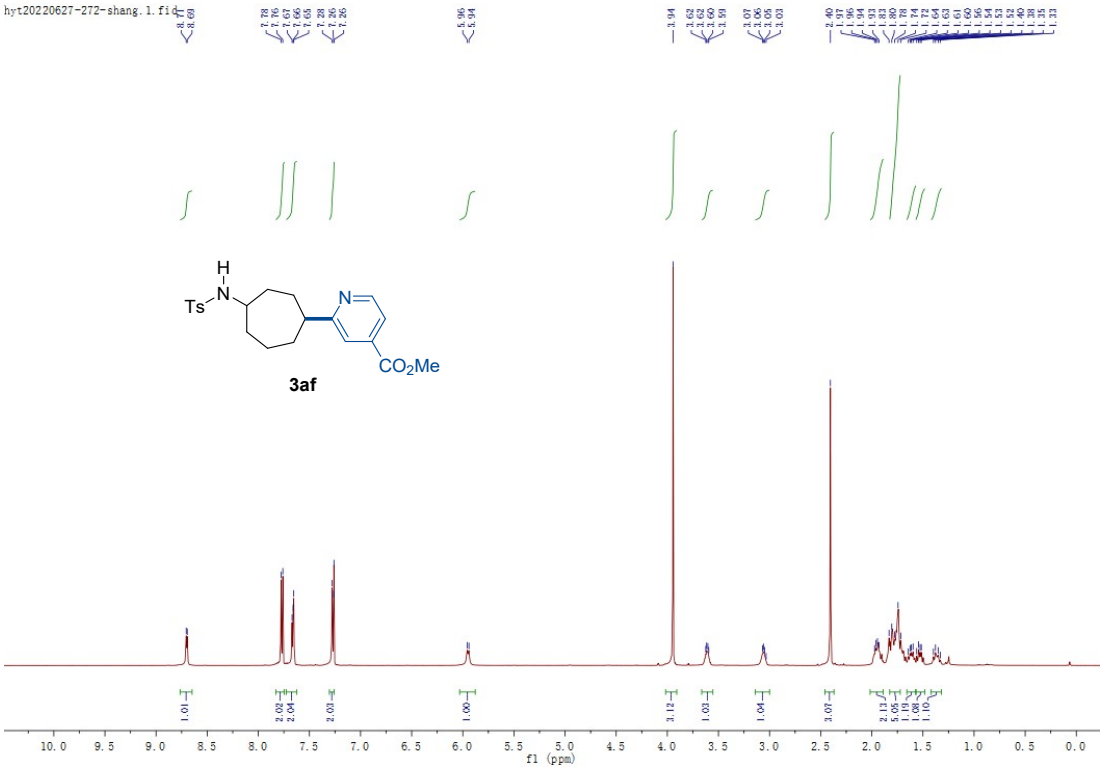


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

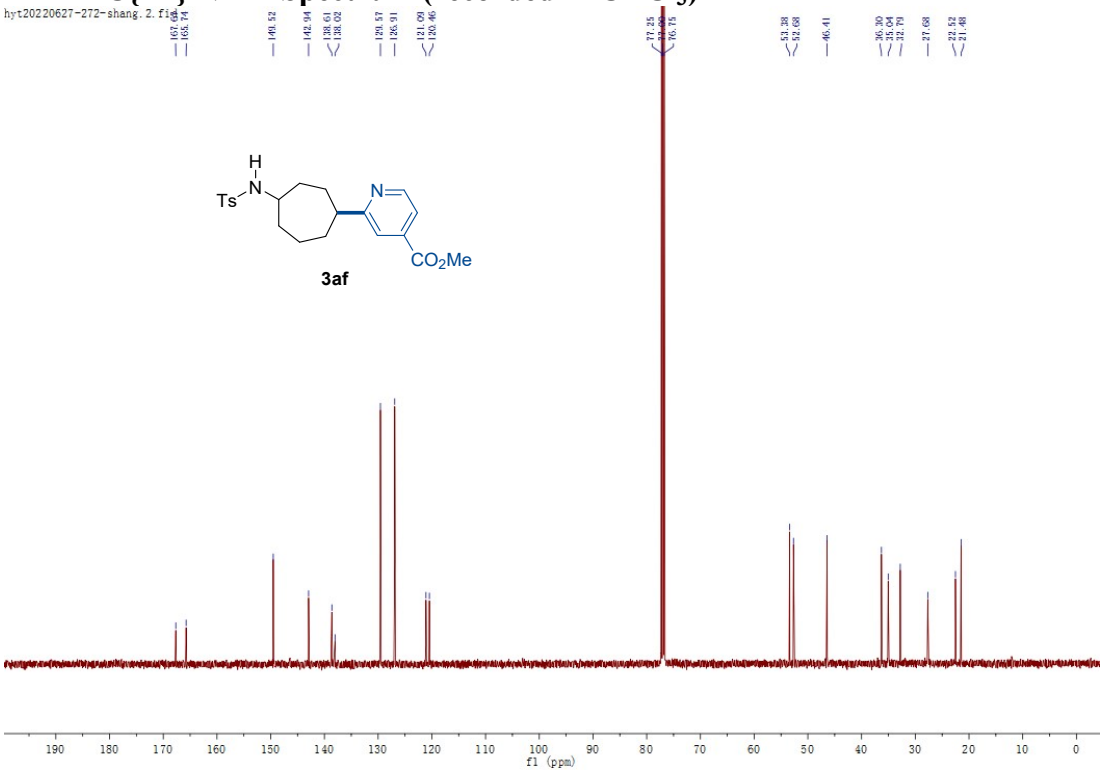


Methyl 2-(4-((4-methylphenyl)sulfonamido)cycloheptyl)isonicotinate (3af).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

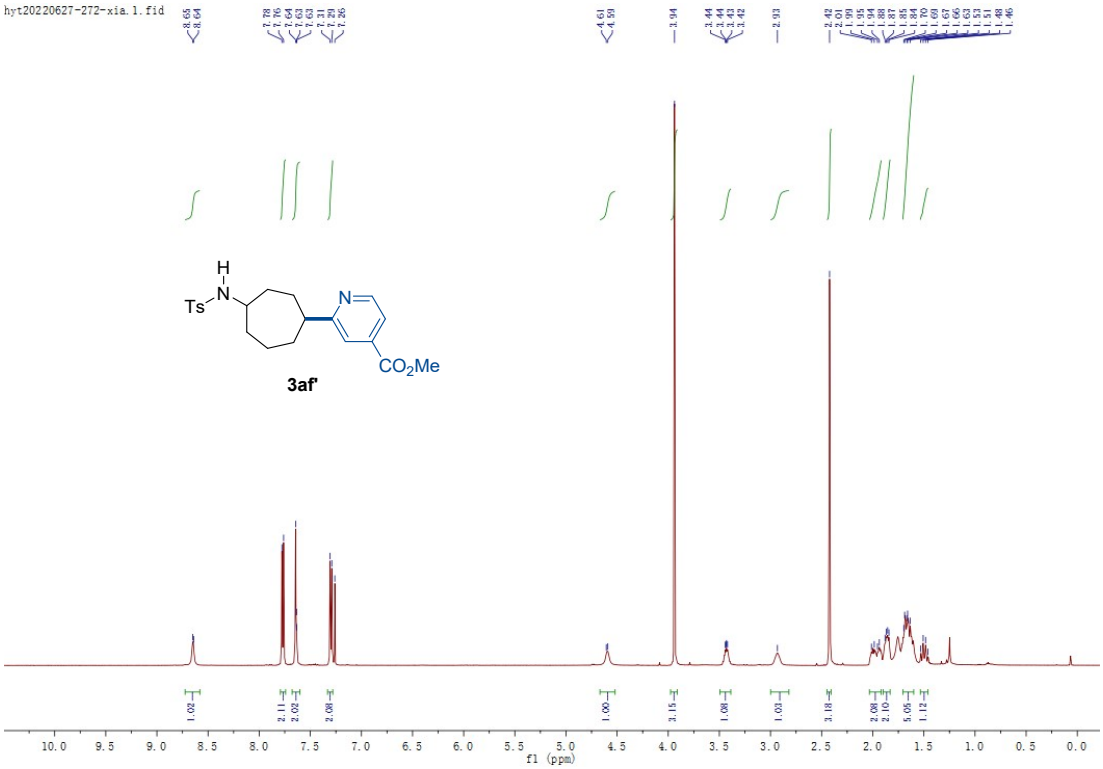


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

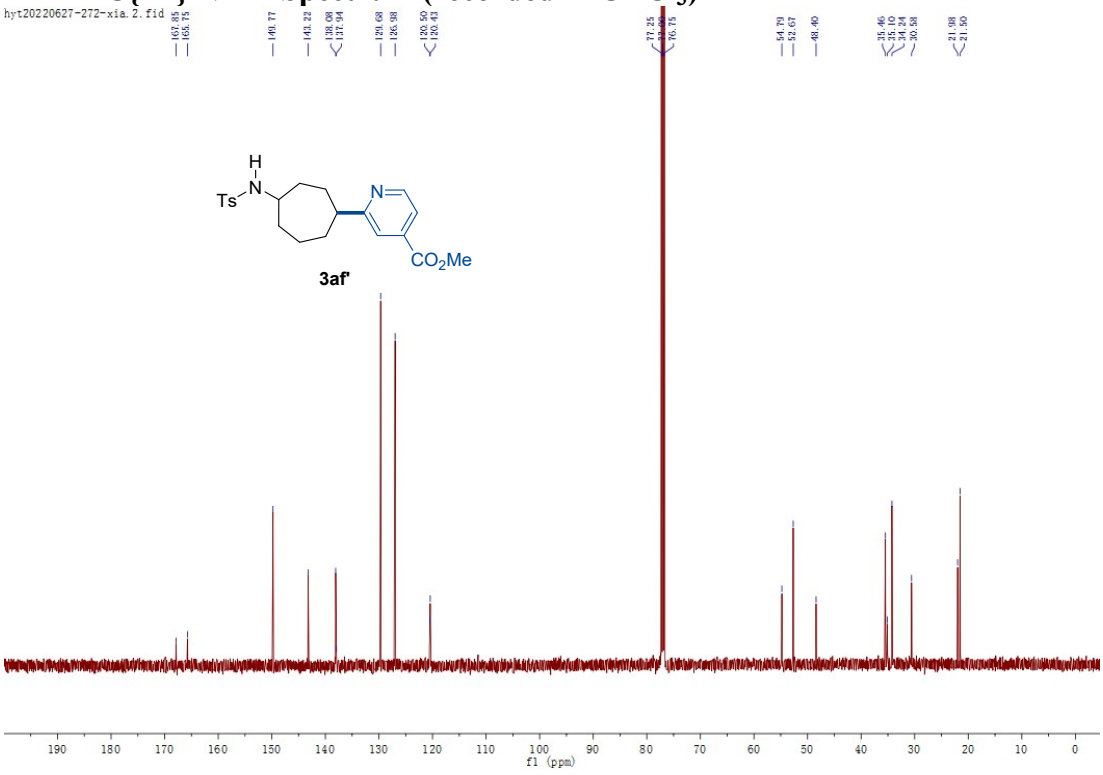


Methyl 2-(4-((4-methylphenyl)sulfonamido)cycloheptyl)isonicotinate (3af').

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

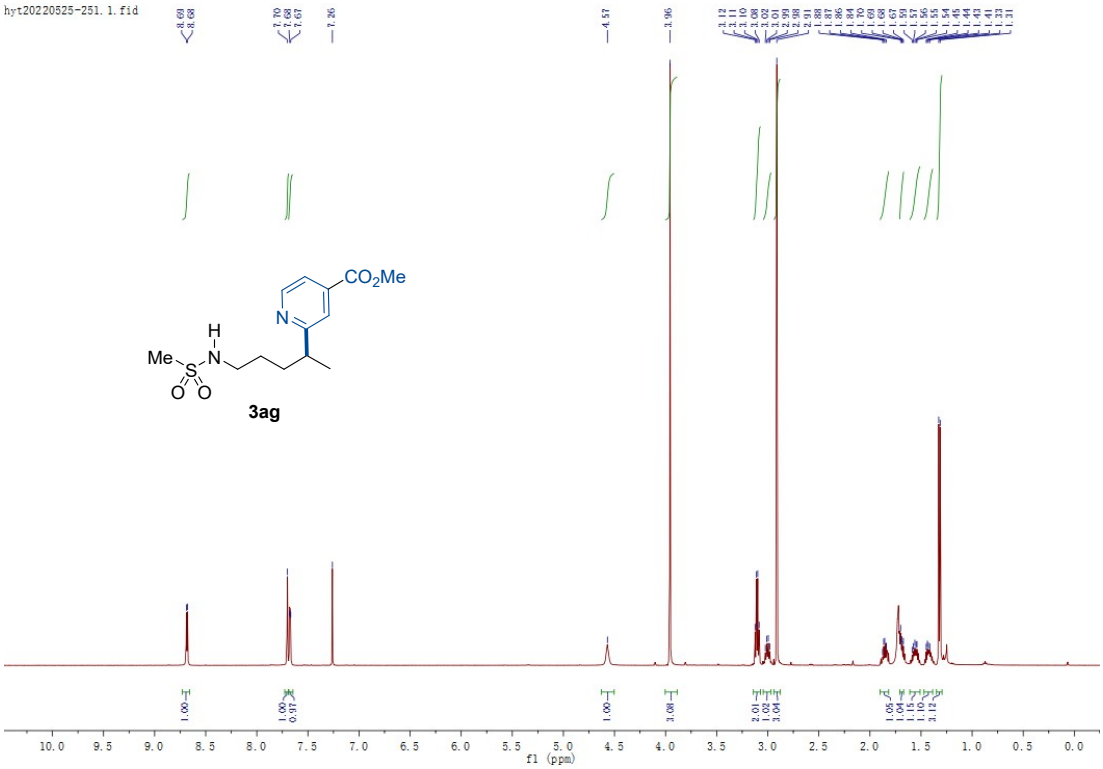


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

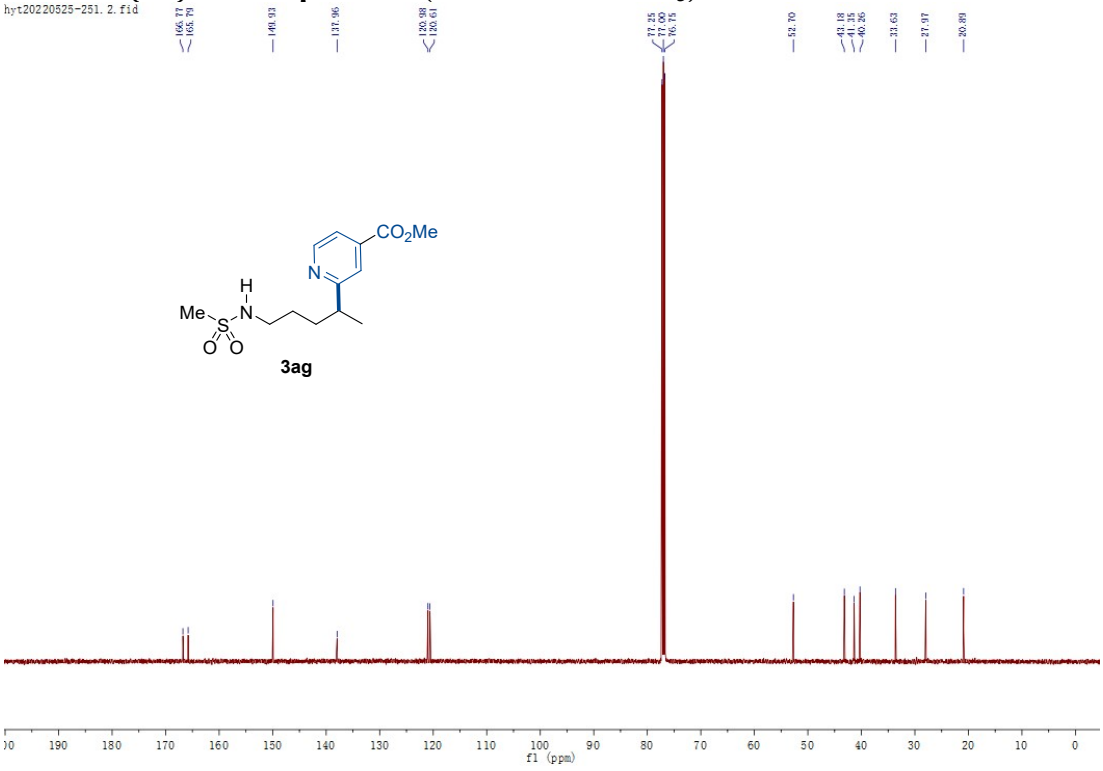


Methyl 2-(5-(methylsulfonamido)pentan-2-yl)isonicotinate (3ag).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

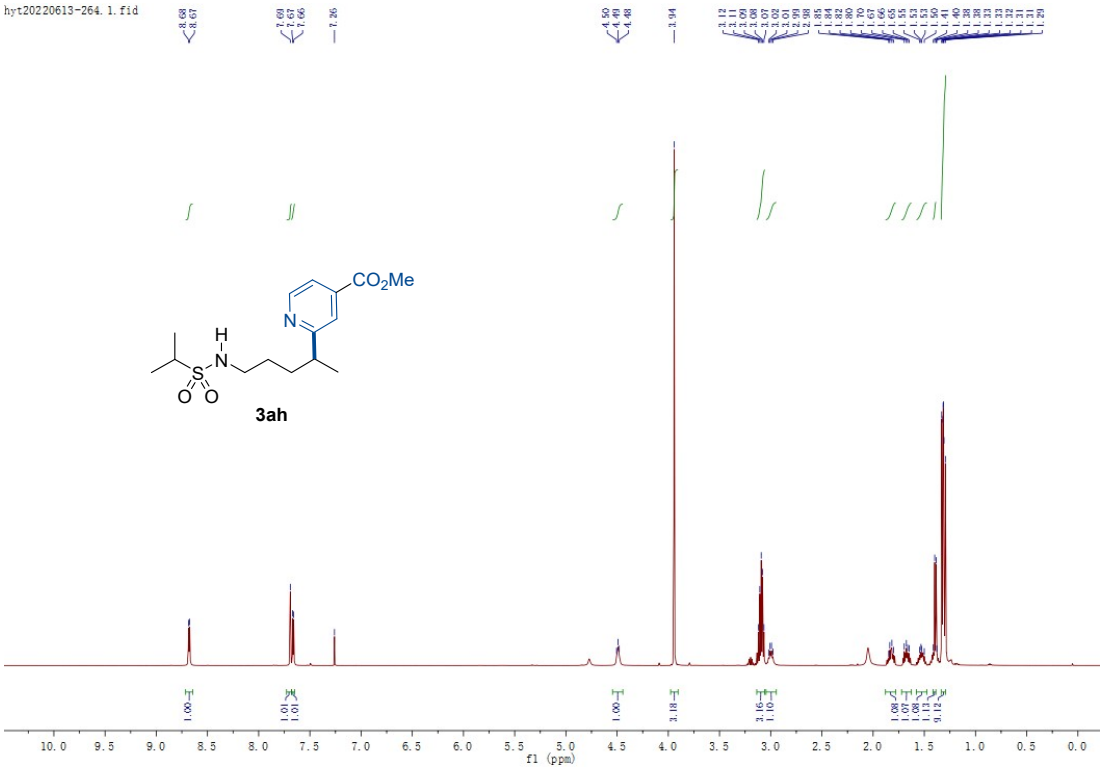


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

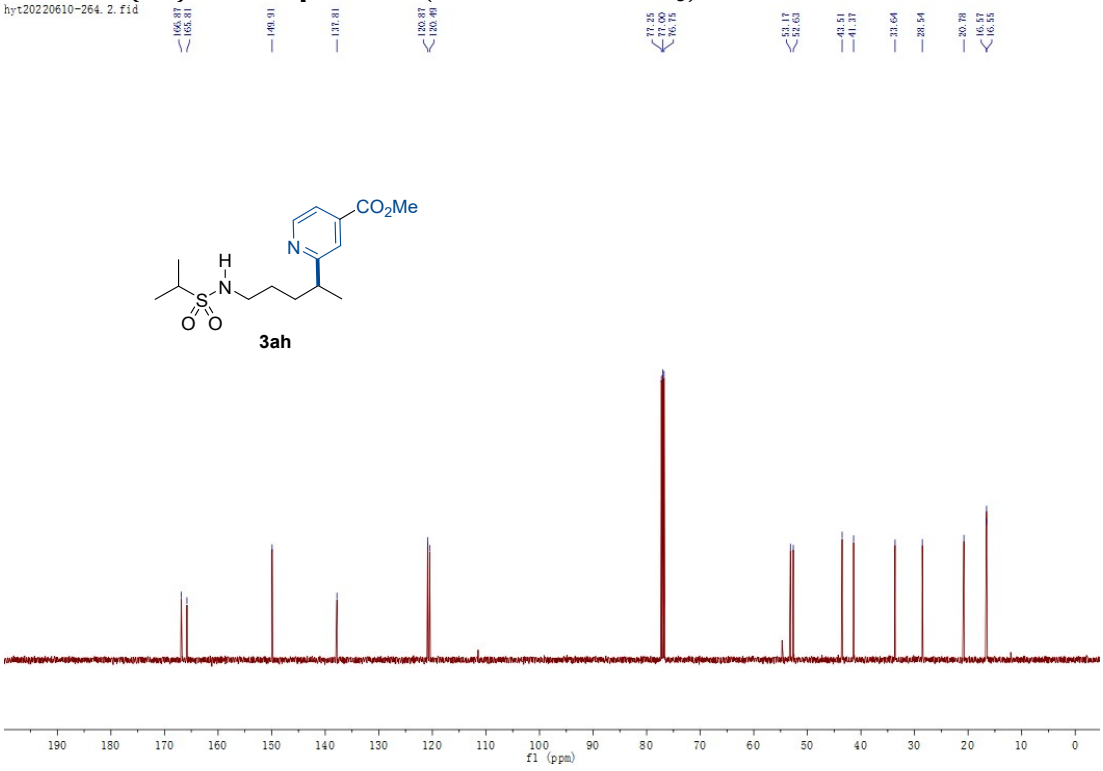


Methyl 2-(5-((1-methylethyl)sulfonamido)pentan-2-yl)isonicotinate (3ah).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

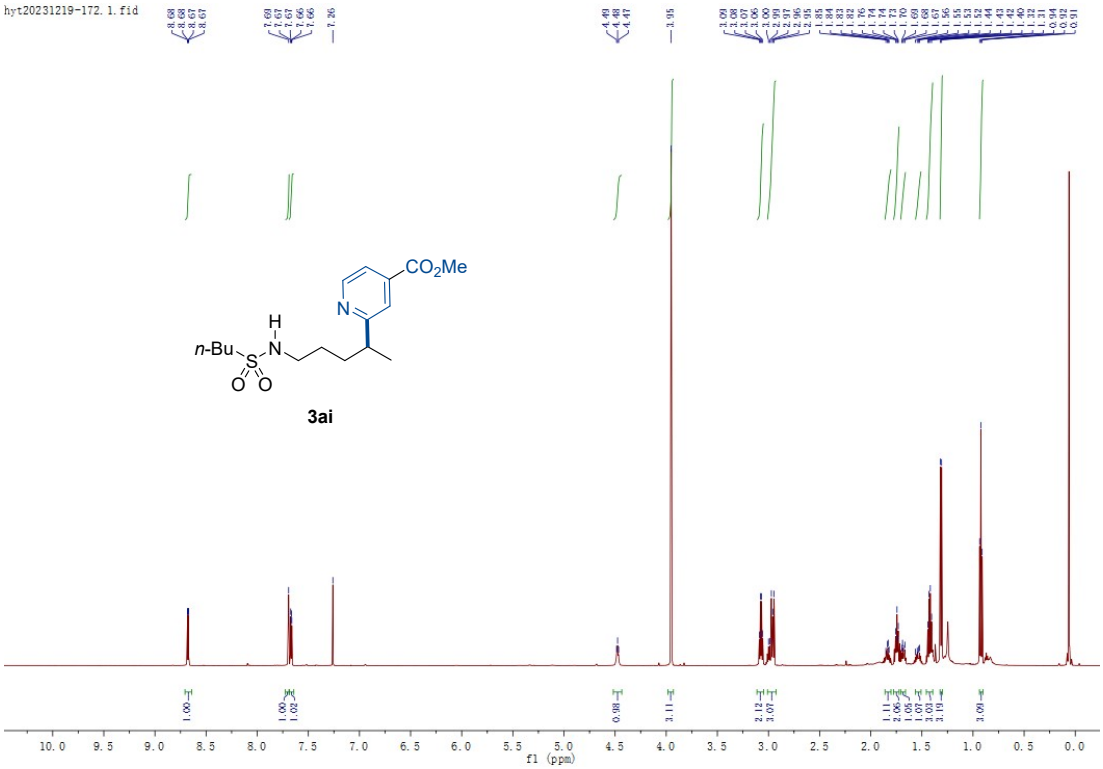


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

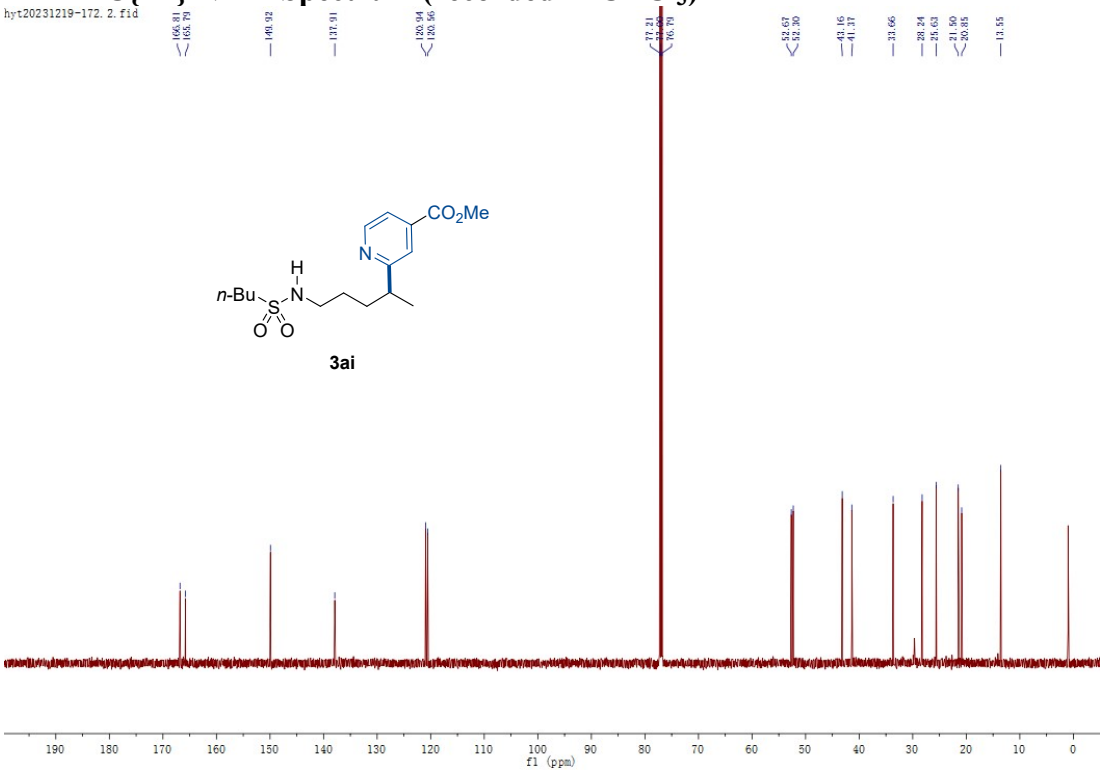


Methyl 2-(5-(butylsulfonamido)pentan-2-yl)isonicotinate (3ai).

600 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

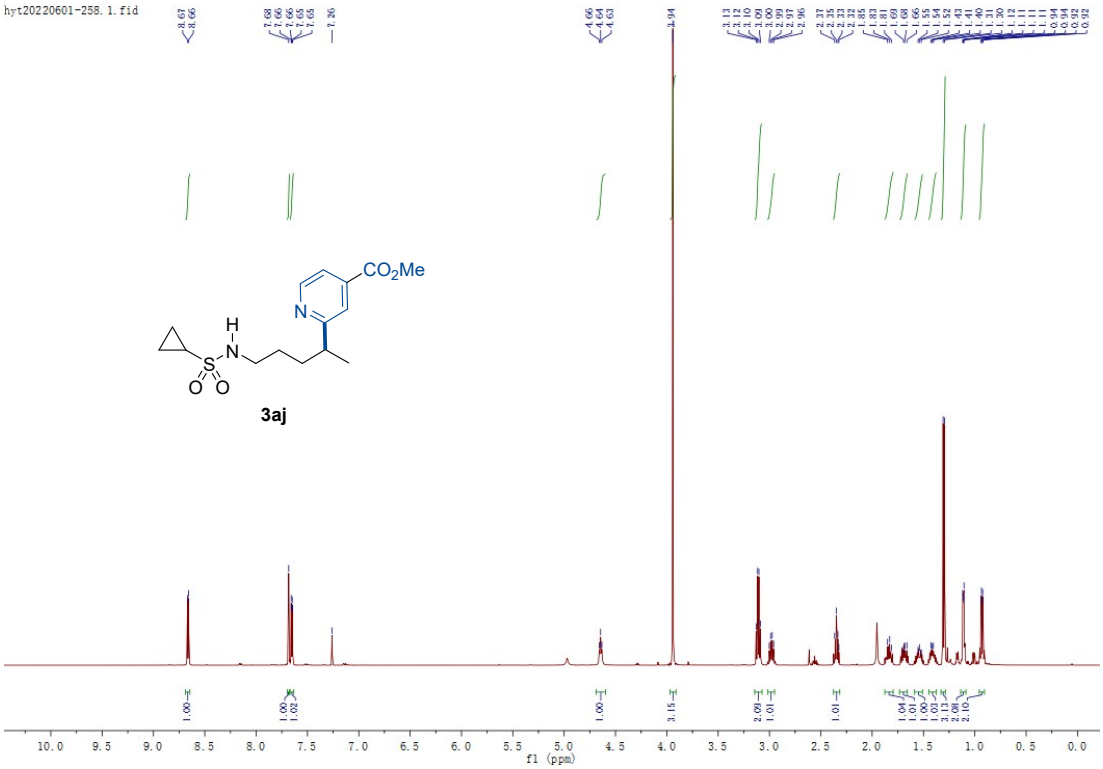


151 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

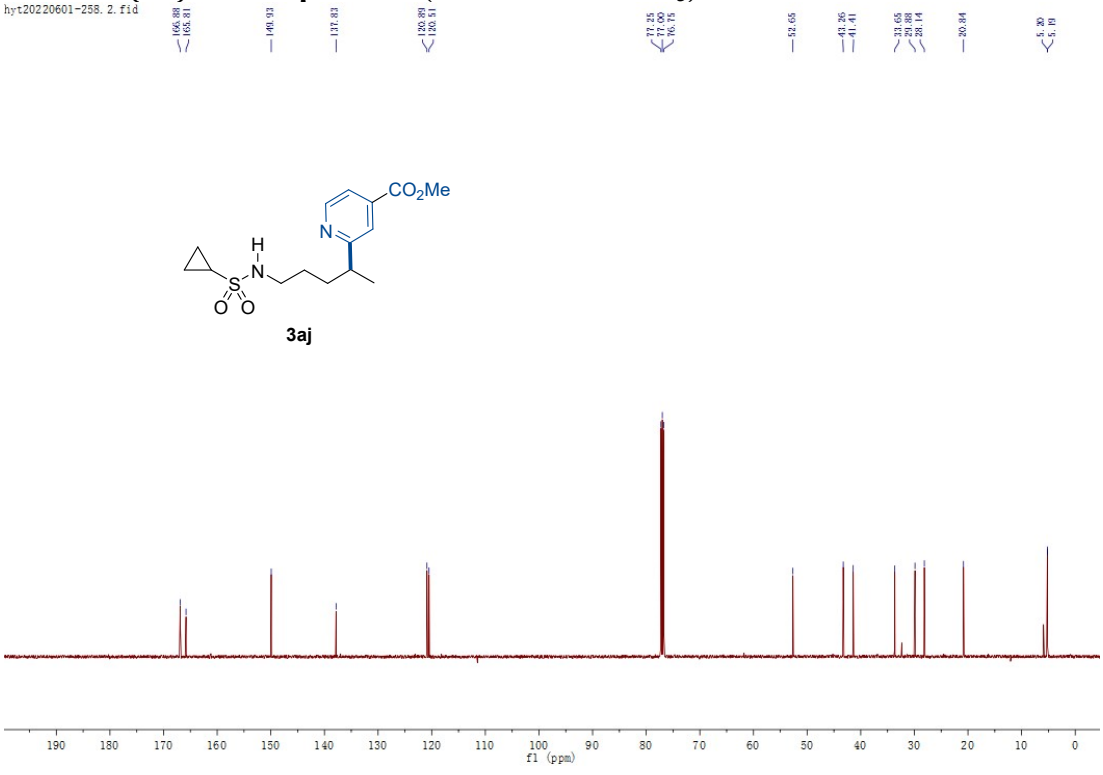


Methyl 2-(5-(cyclopropanesulfonamido)pentan-2-yl)isonicotinate (3aj).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

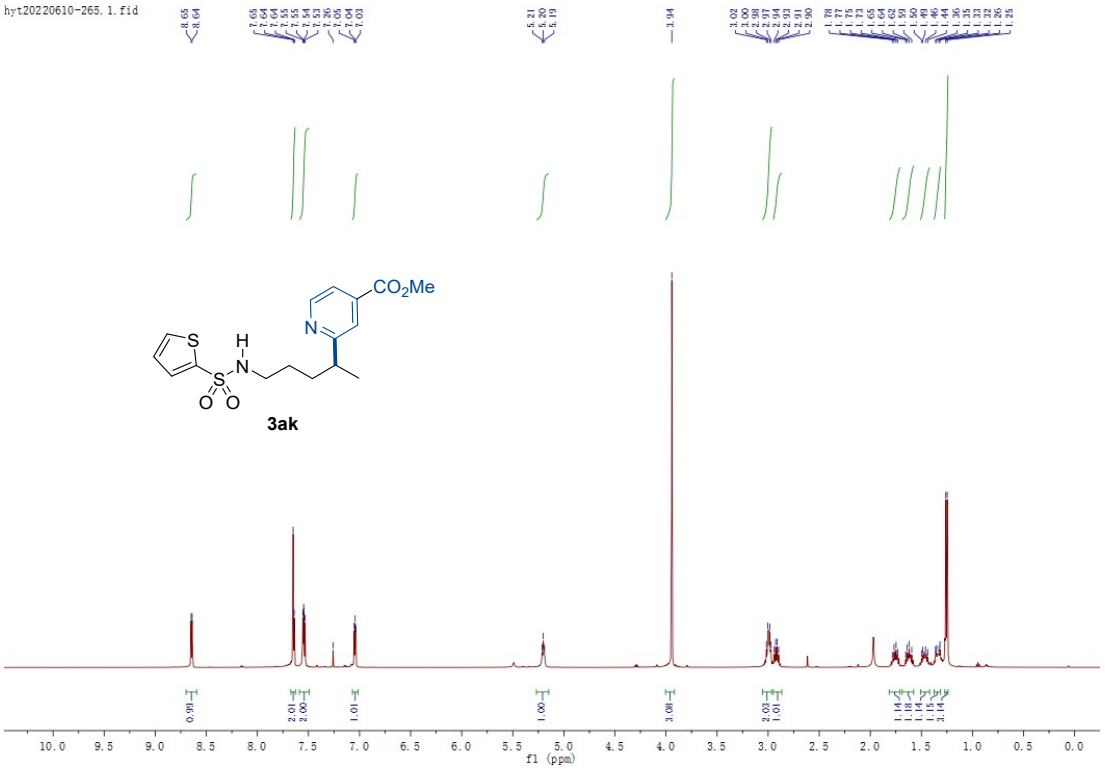


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

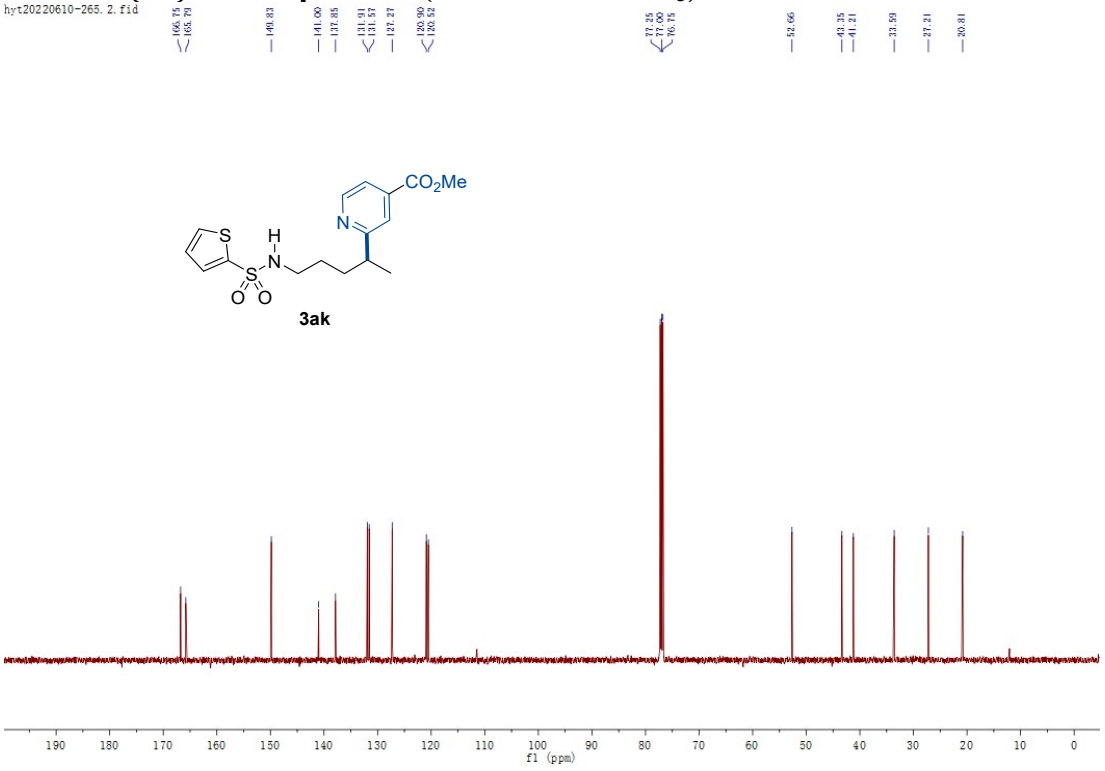


Methyl 2-(5-(thiophene-2-sulfonamido)pentan-2-yl)isonicotinate (3ak).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)



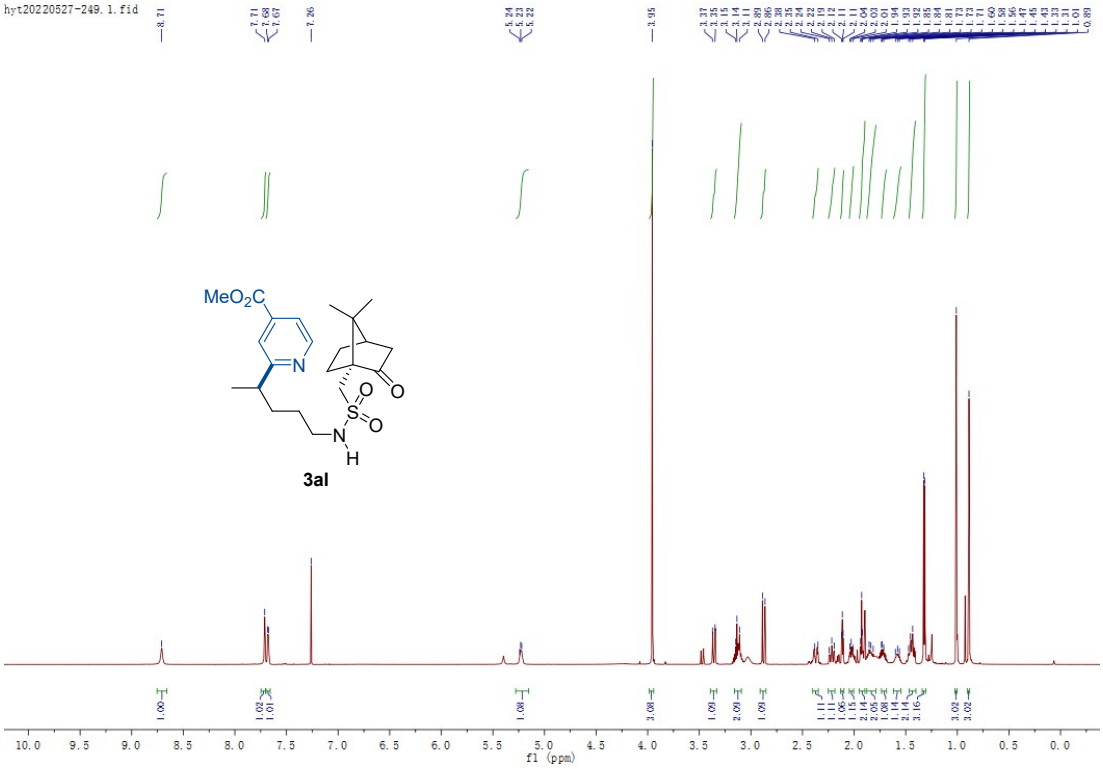
126 MHz $^{13}C\{^{1}H\}$ NMR Spectrum (recorded in $CDCl_3$)



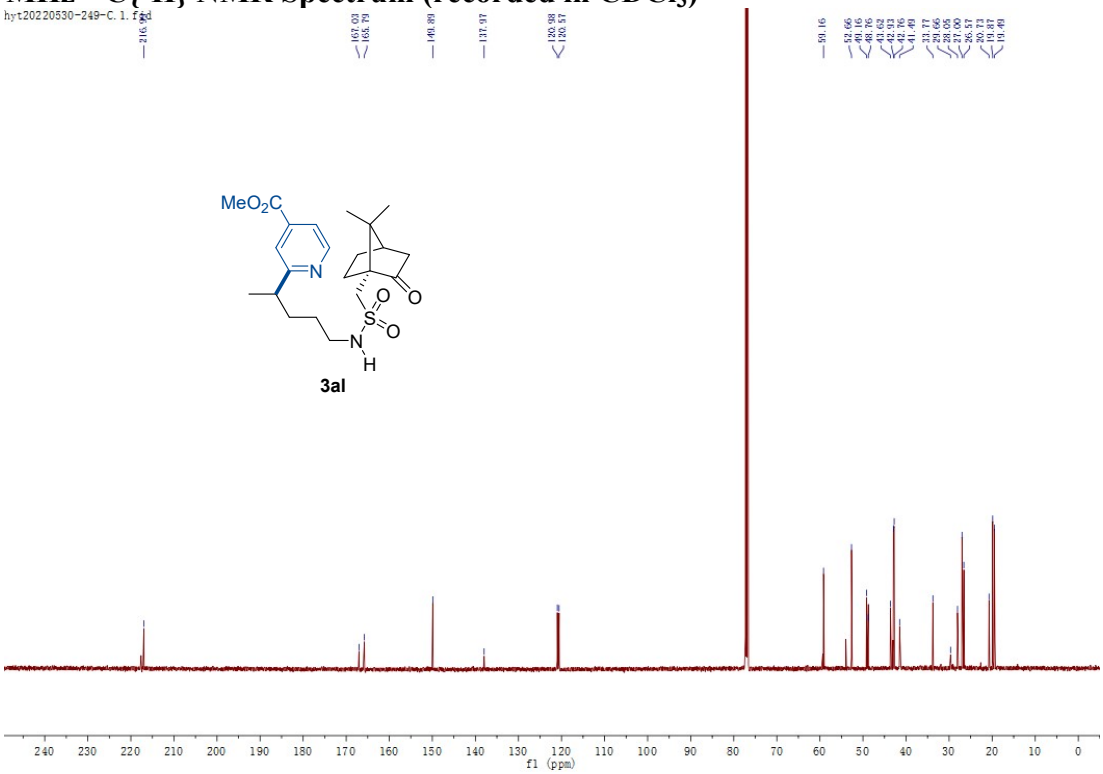
Methyl 2-(5-((((1R,4S)-7,7-dimethyl-2-oxobicyclo[2.2.1]heptan-1-yl)methyl)sulfonamide-

o)pentan-2-yl)isonicotinate (3al).

600 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)



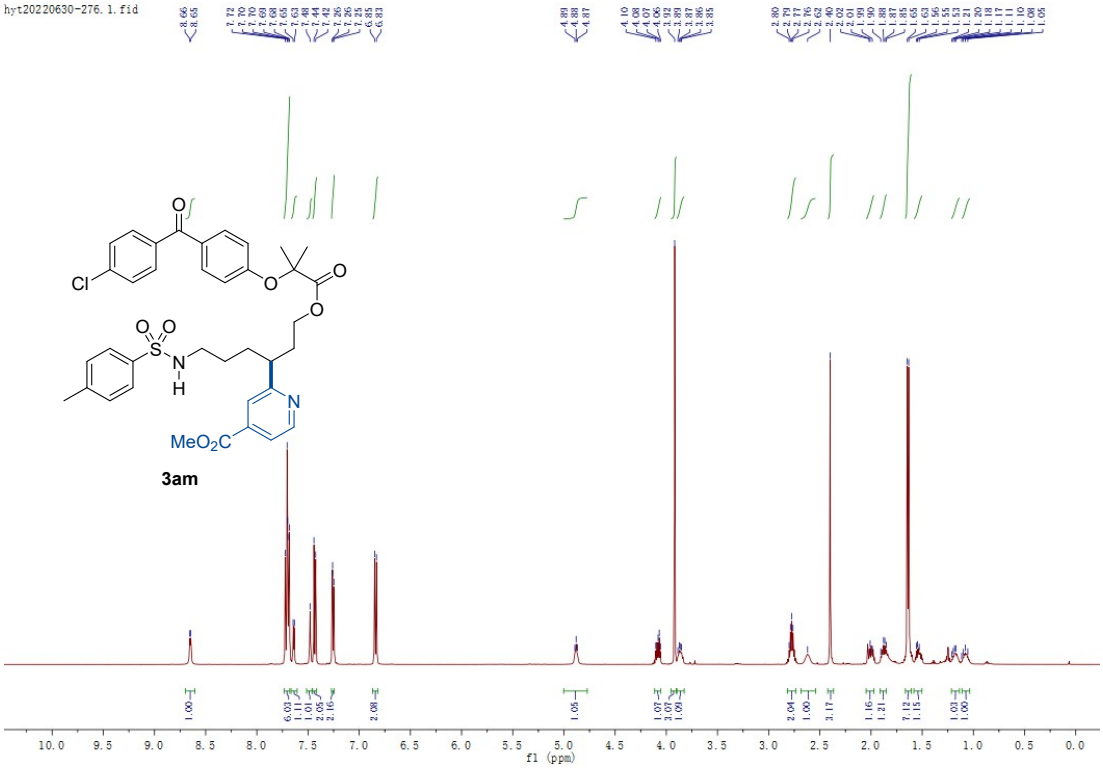
151 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)



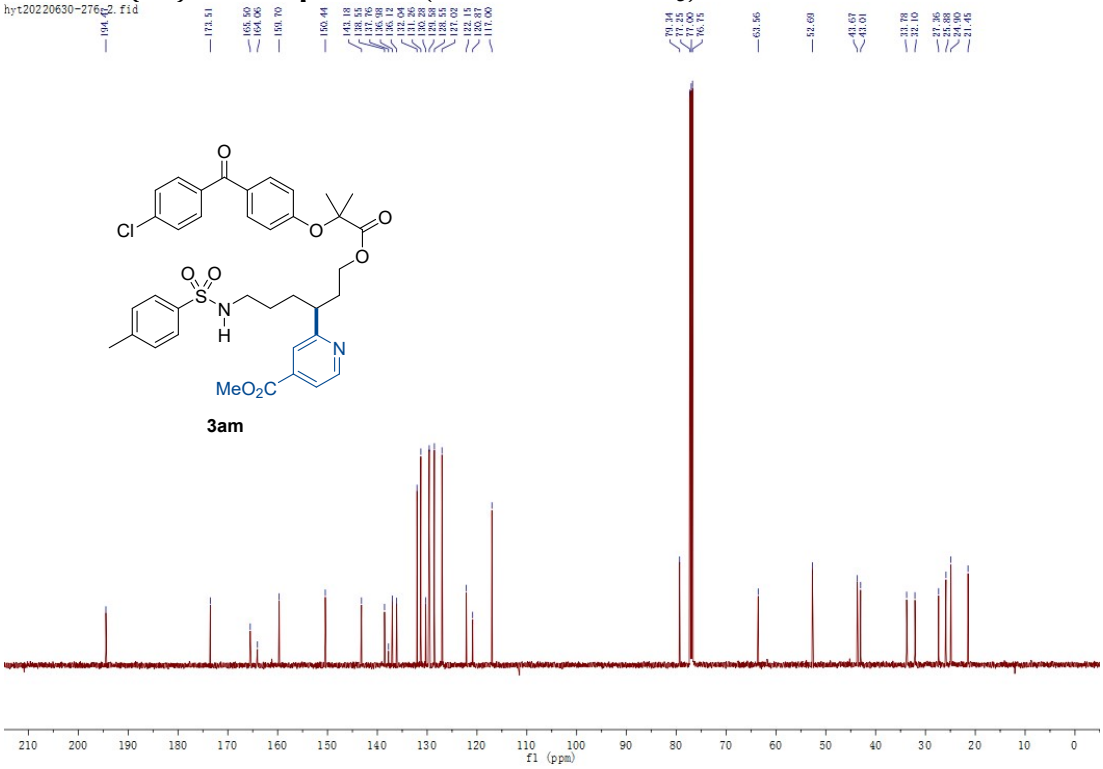
Methyl 2-(1-((2-(4-(4-chlorobenzoyl)phenoxy)-2-methylpropanoyl)oxy)-6-((4-methylphe-

nyl)sulfonamido)hexan-3-yl)isonicotinate (3am).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)



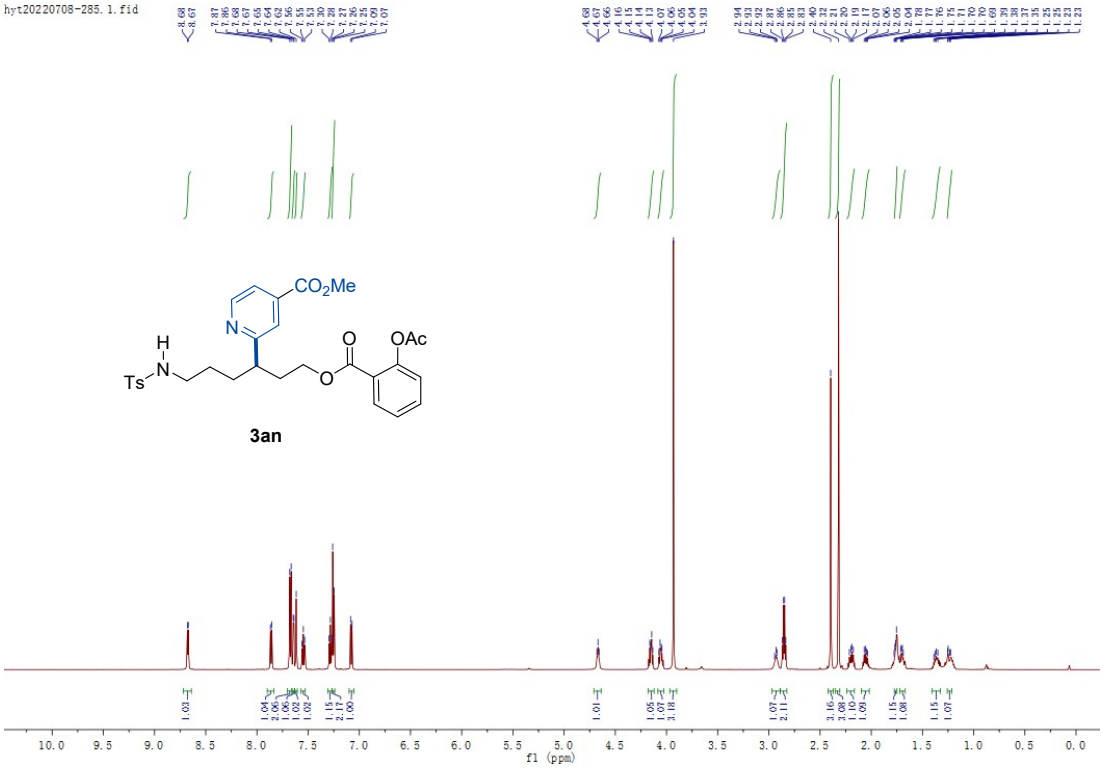
126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)



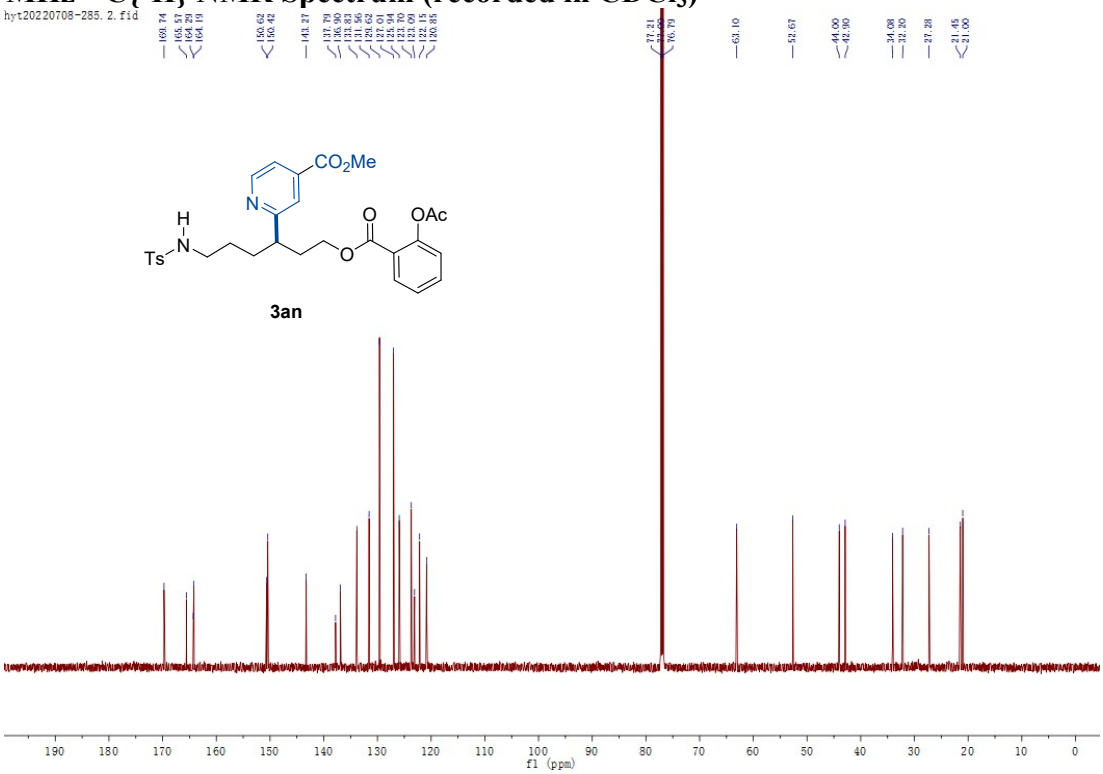
Methyl 2-(1-((2-acetoxybenzoyl)oxy)-6-((4-methylphenyl)sulfonamido)hexan-3-yl)isoni-

cotinate (3an).

600 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)



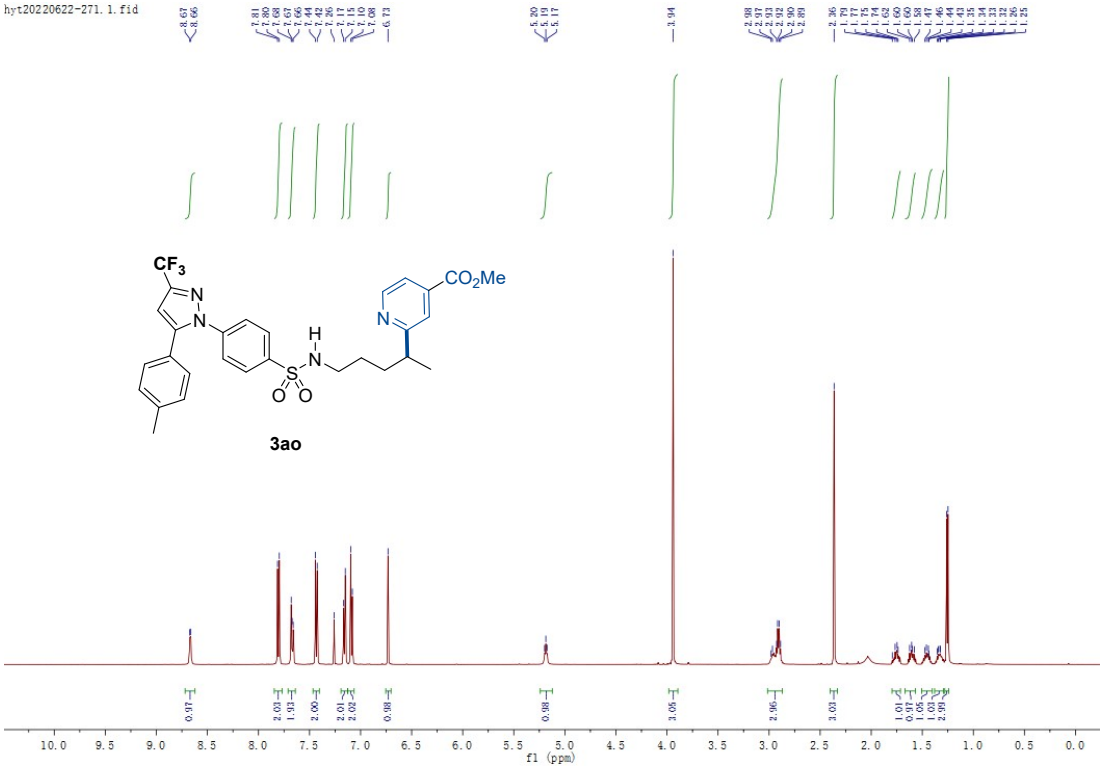
151 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)



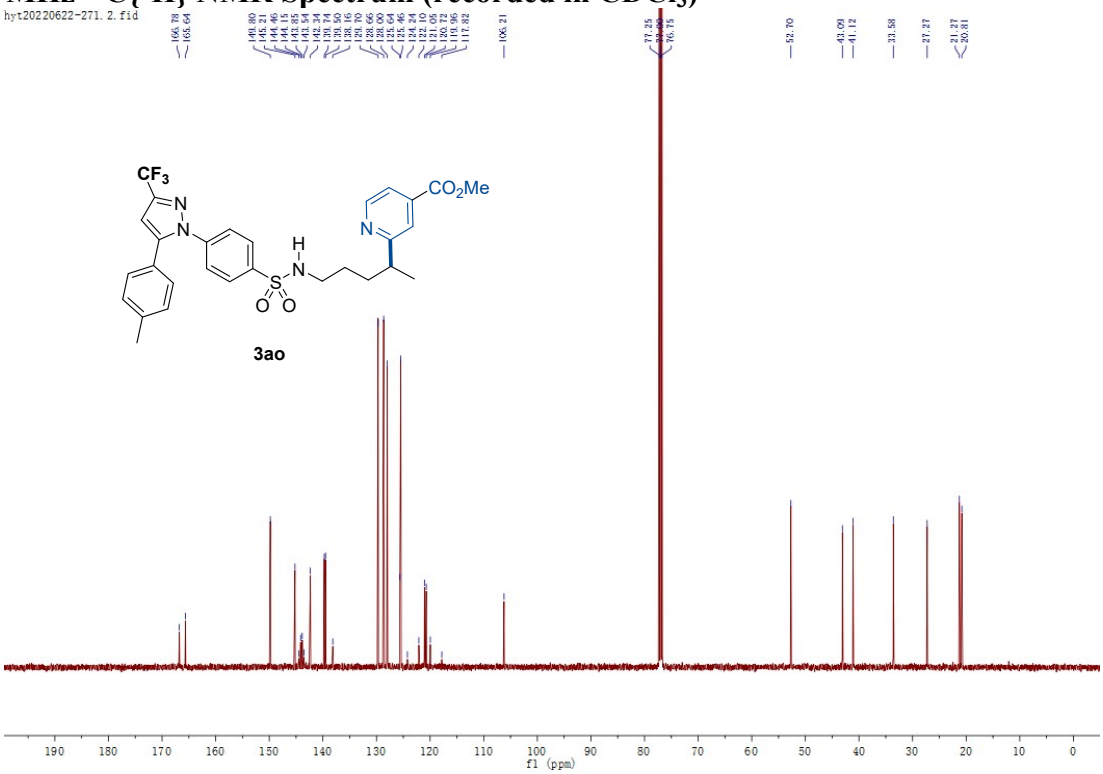
Methyl 2-(5-((4-(5-(p-tolyl)-3-(trifluoromethyl)-1H-pyrazol-1-yl)phenyl)sulfonamido)pe-

ntan-2-yl)isonicotinate (3ao).

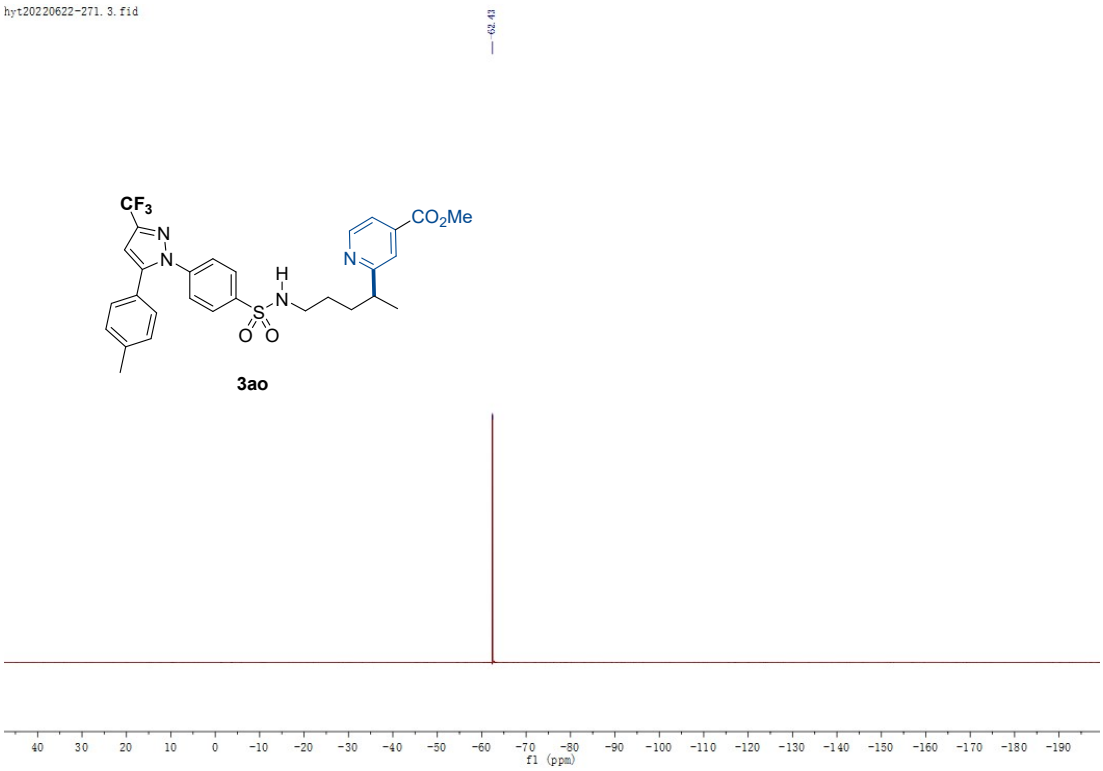
500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)



126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)



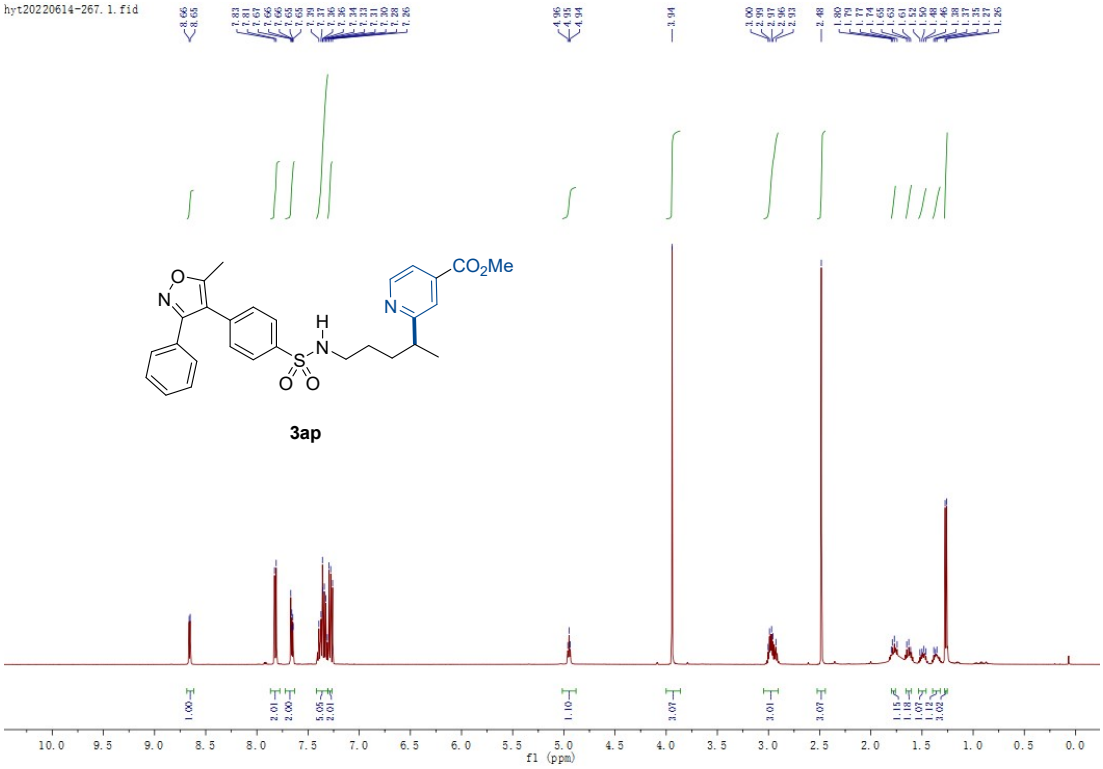
471 MHz ^{19}F NMR Spectrum (recorded in $CDCl_3$)



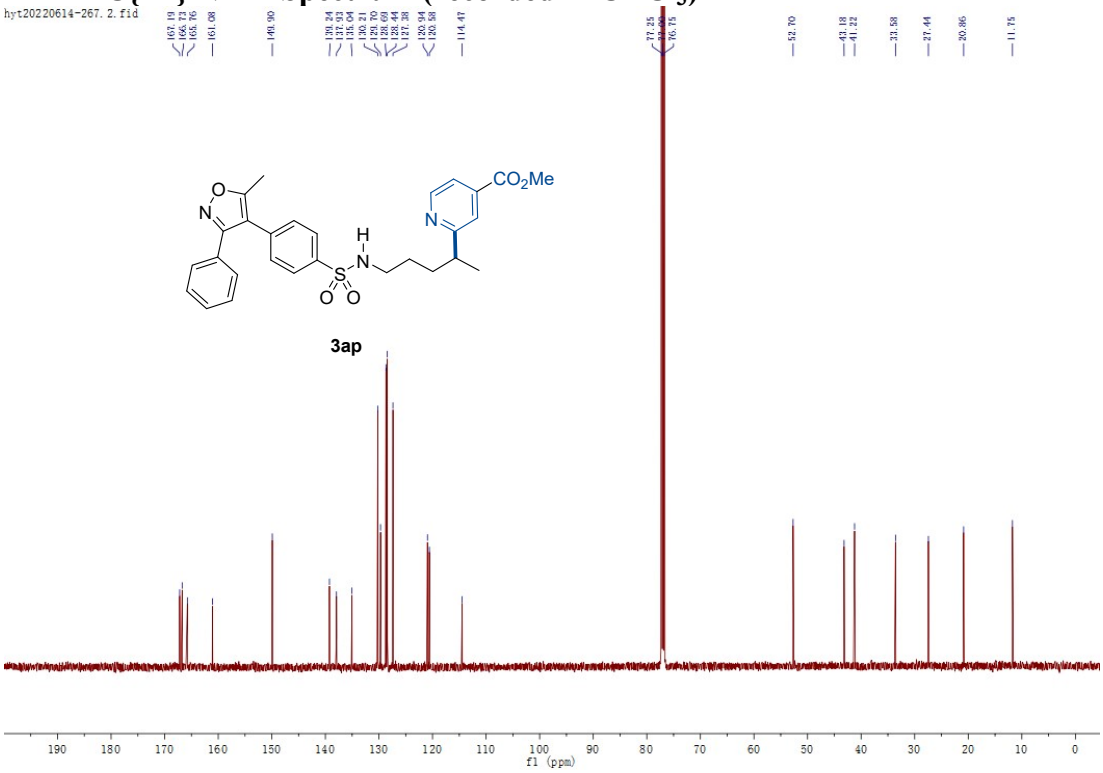
Methyl 2-(5-((4-(5-methyl-3-phenylisoxazol-4-yl)phenyl)sulfonamido)pentan-2-yl)isonic-

otinate (3ap).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

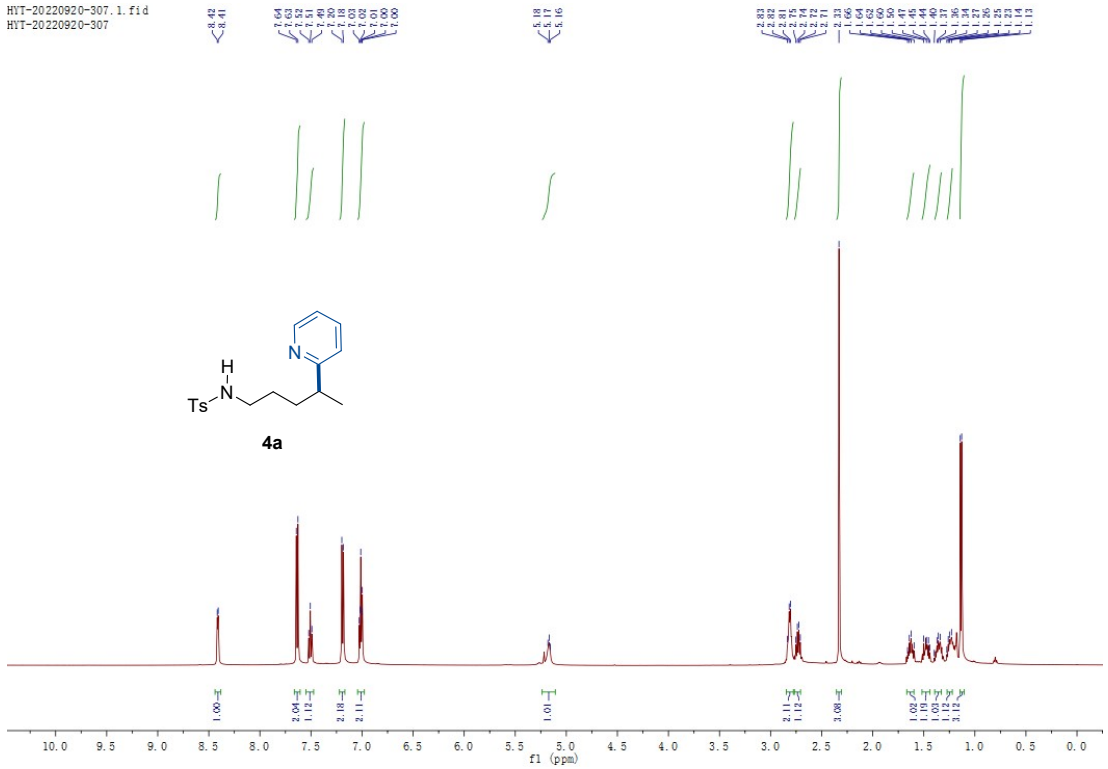


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

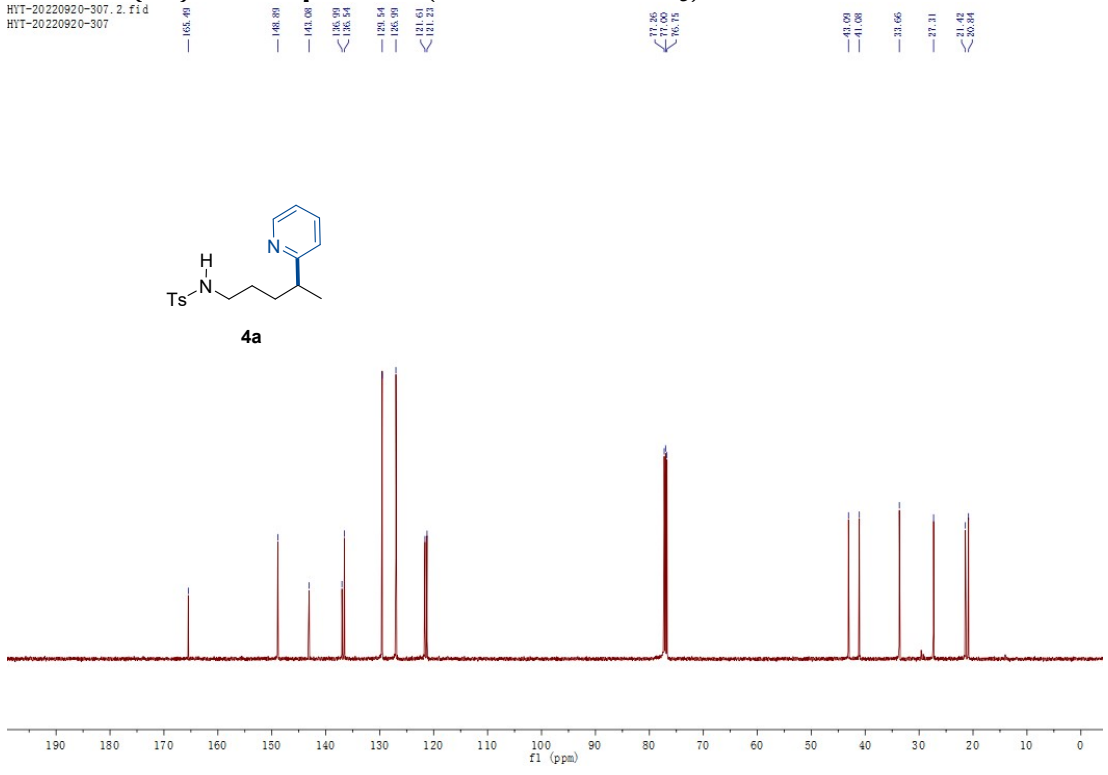


4-Methyl-*N*-(4-(pyridin-2-yl)pentyl)benzenesulfonamide (4a).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

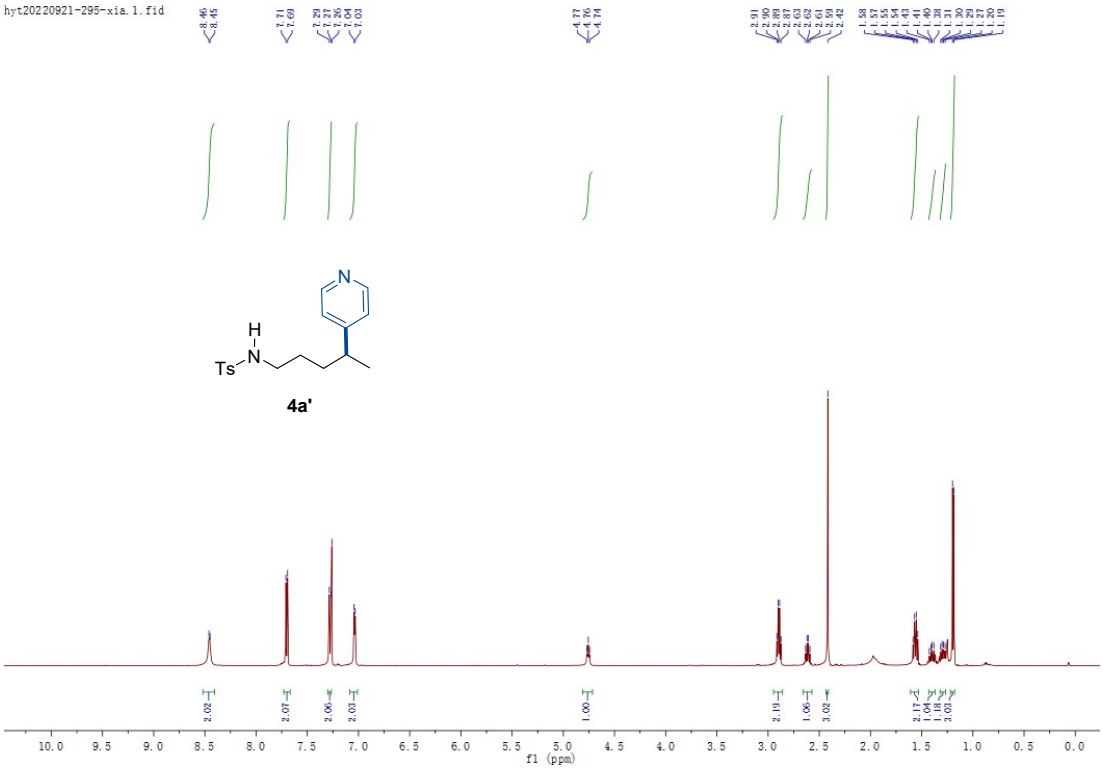


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

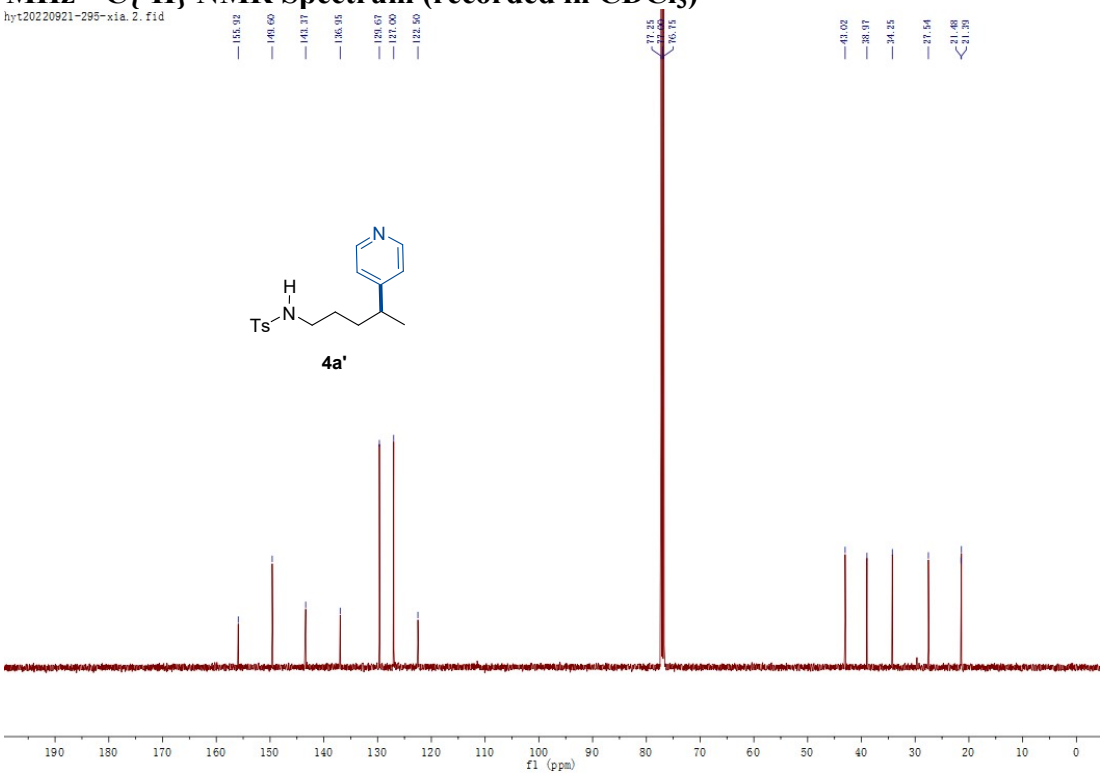


4-Methyl-*N*-(4-(pyridin-4-yl)pentyl)benzenesulfonamide (4a').

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

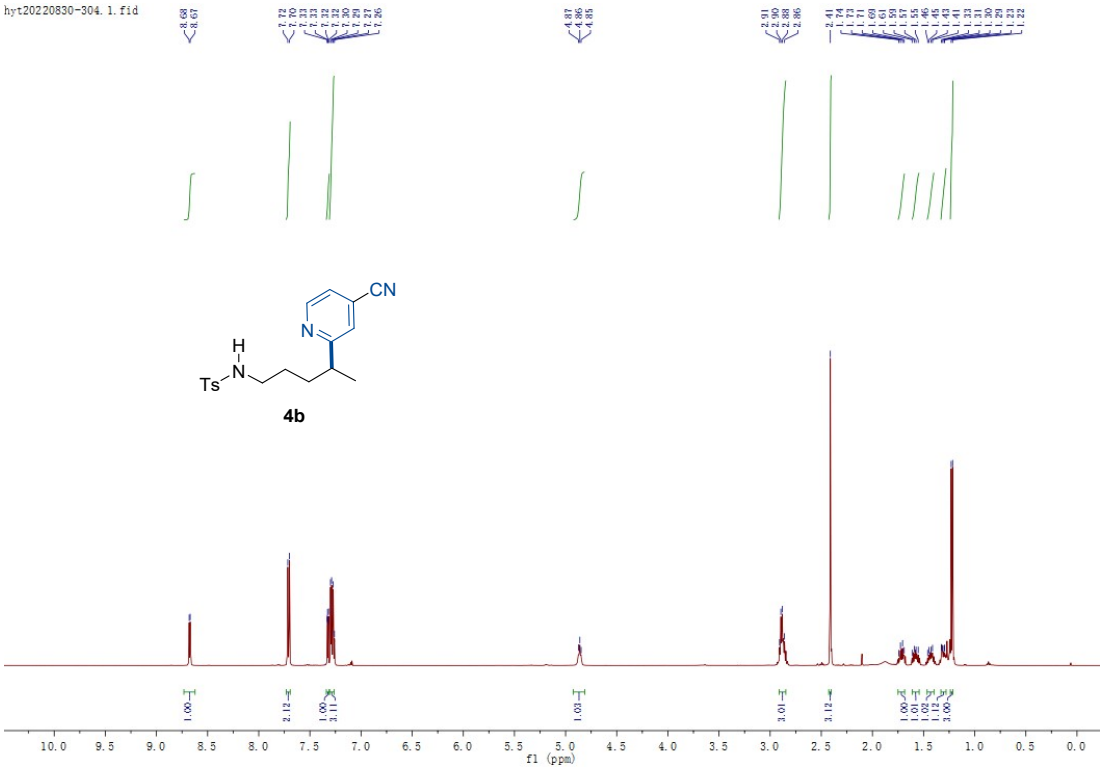


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

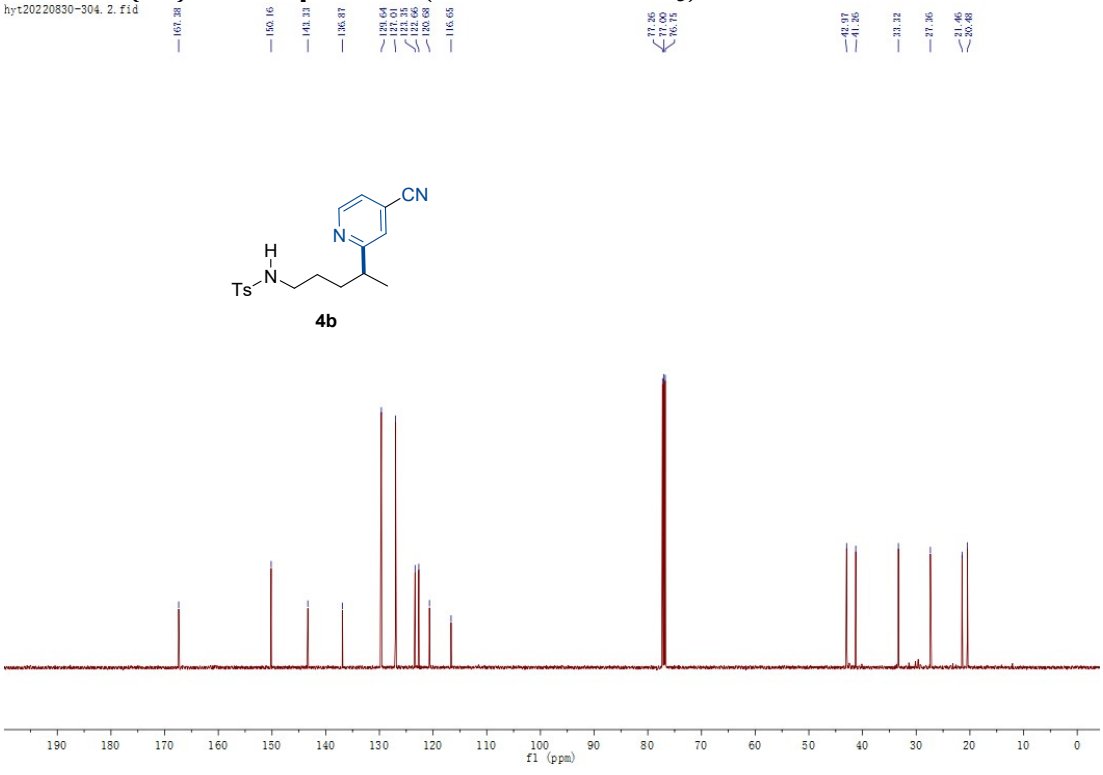


N-(4-(4-Cyanopyridin-2-yl)pentyl)-4-methylbenzenesulfonamide (4b).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

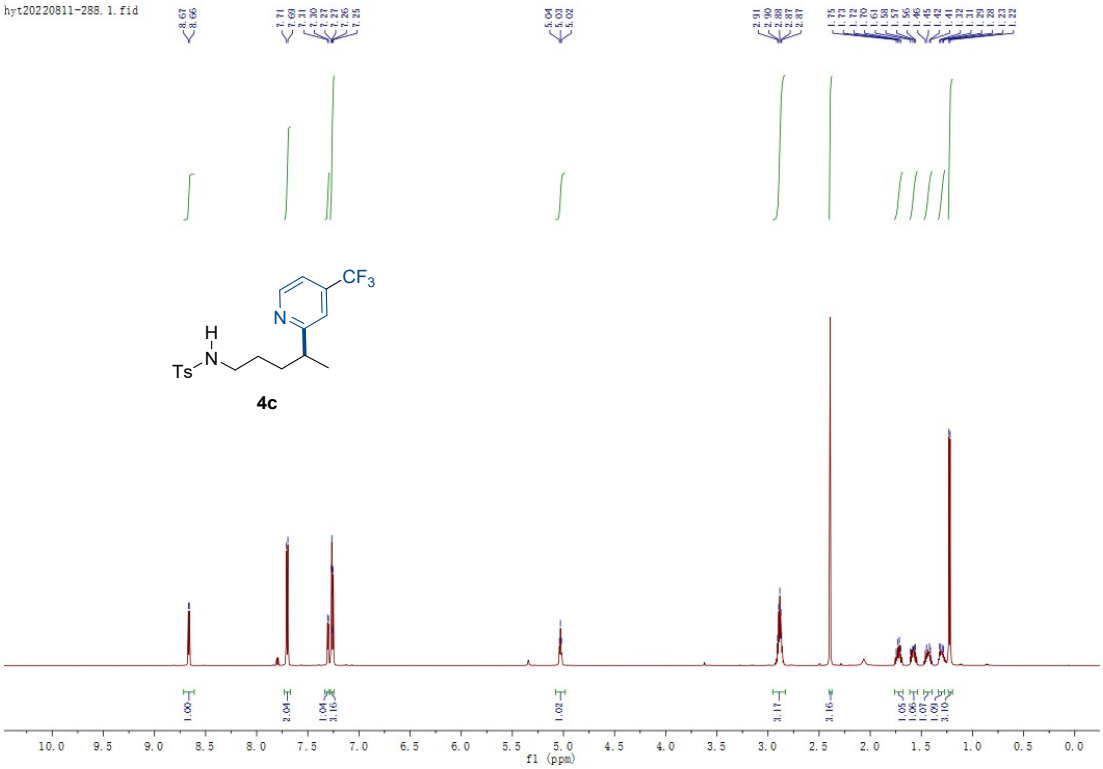


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

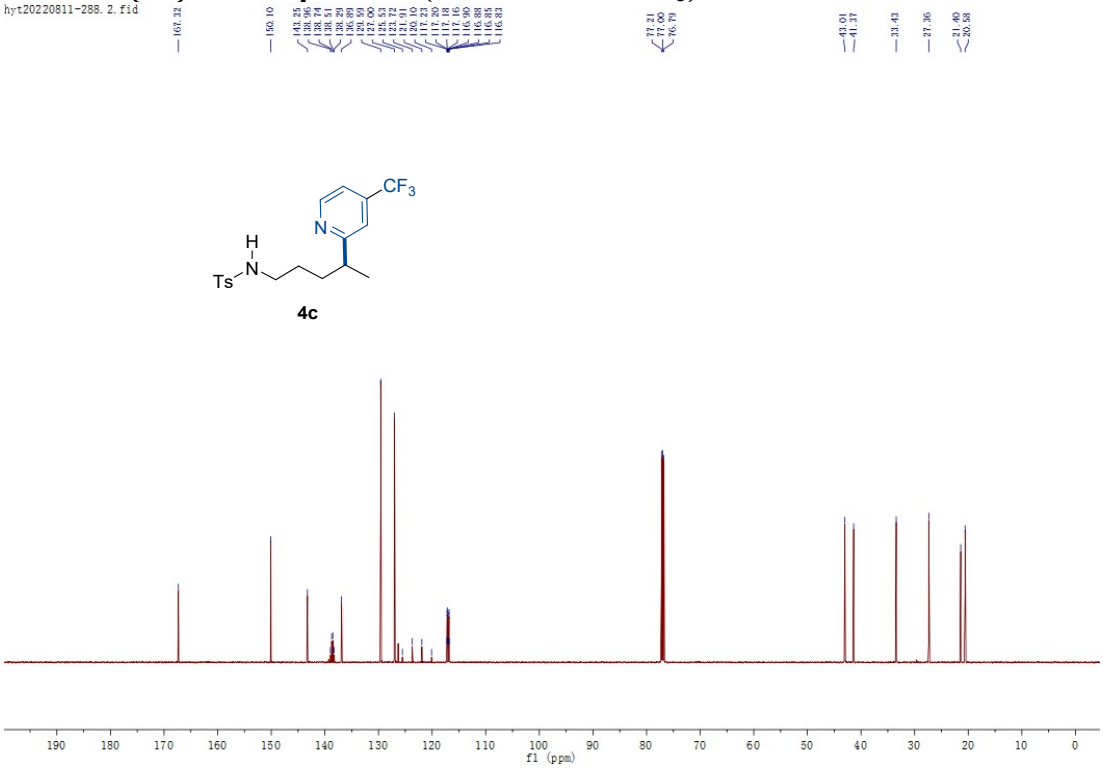


4-Methyl-*N*-(4-(4-(trifluoromethyl)pyridin-2-yl)pentyl)benzenesulfonamide (4c).

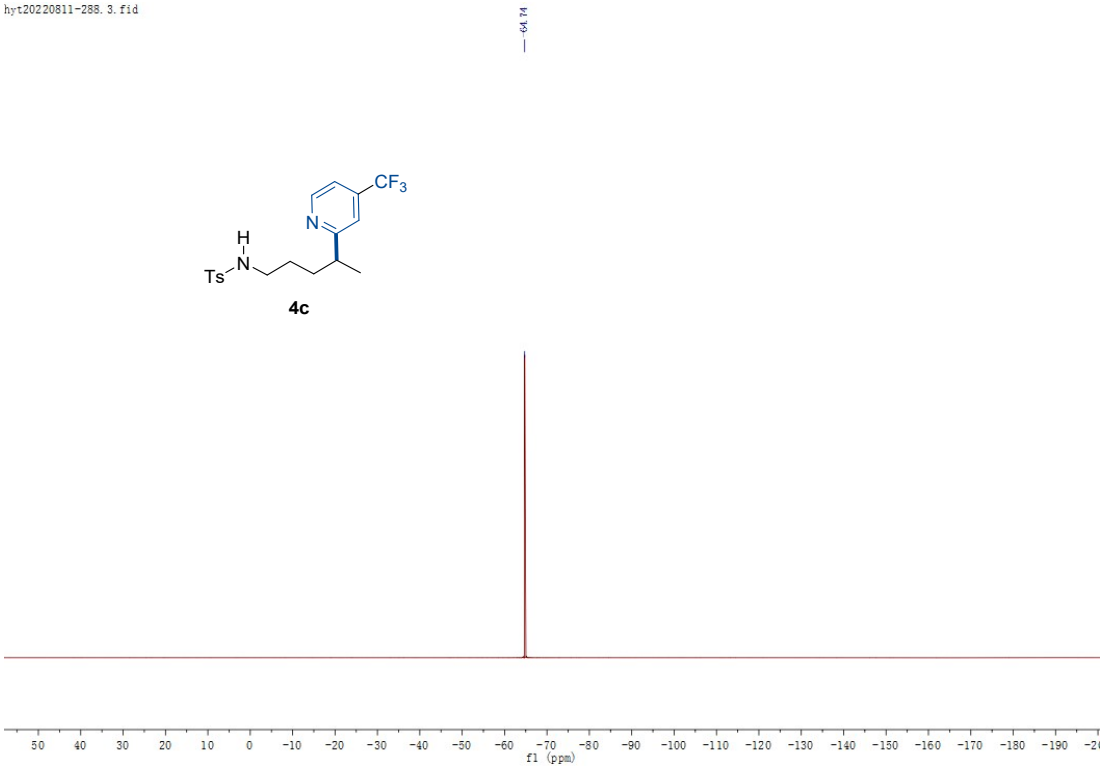
600 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)



151 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

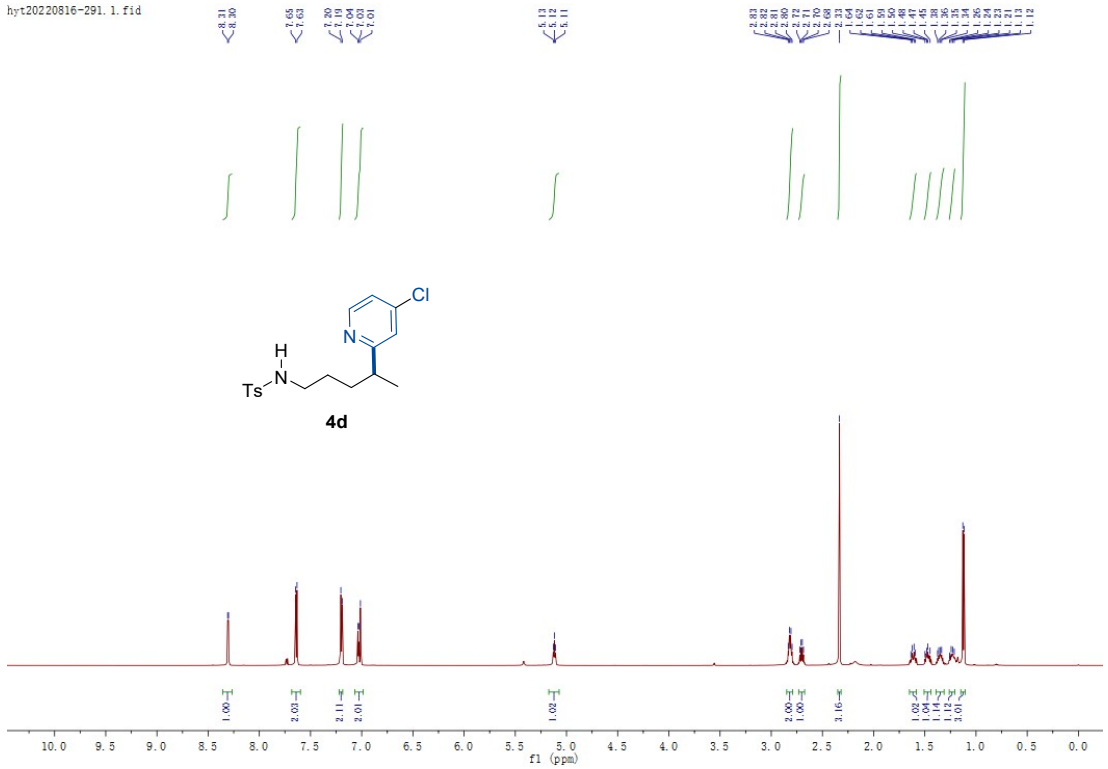


565 MHz ^{19}F NMR Spectrum (recorded in $CDCl_3$)

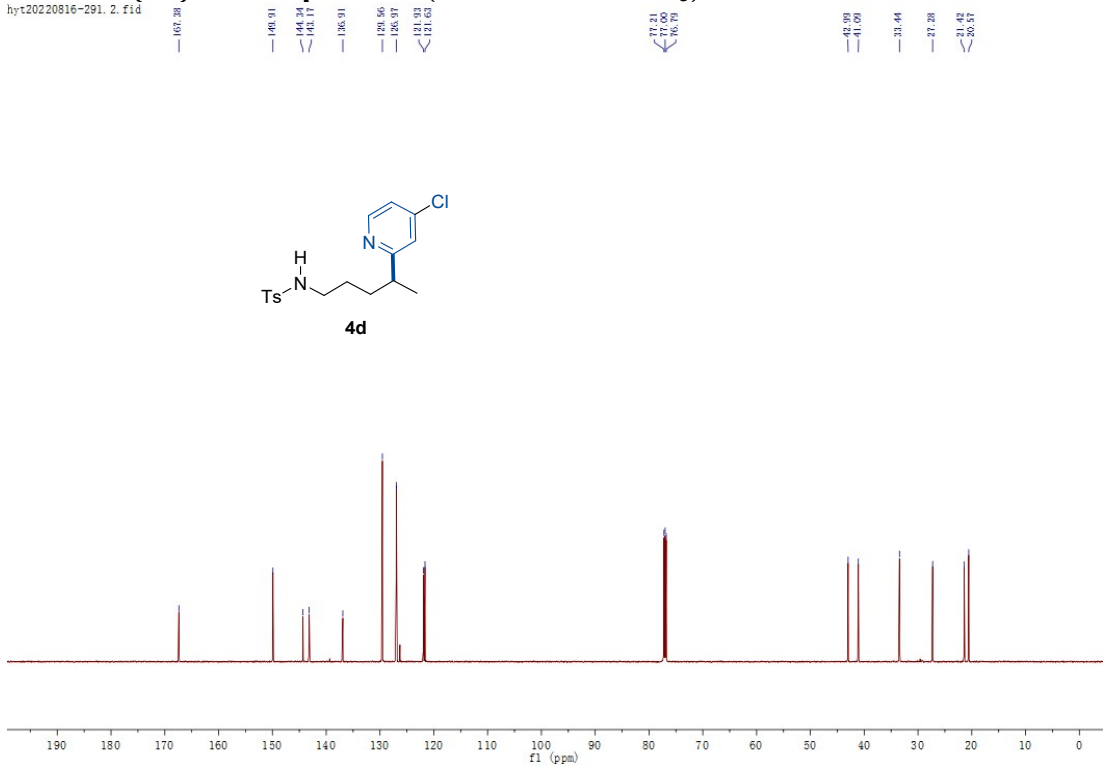


***N*-(4-(4-Chloropyridin-2-yl)pentyl)-4-methylbenzenesulfonamide (4d).**

600 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

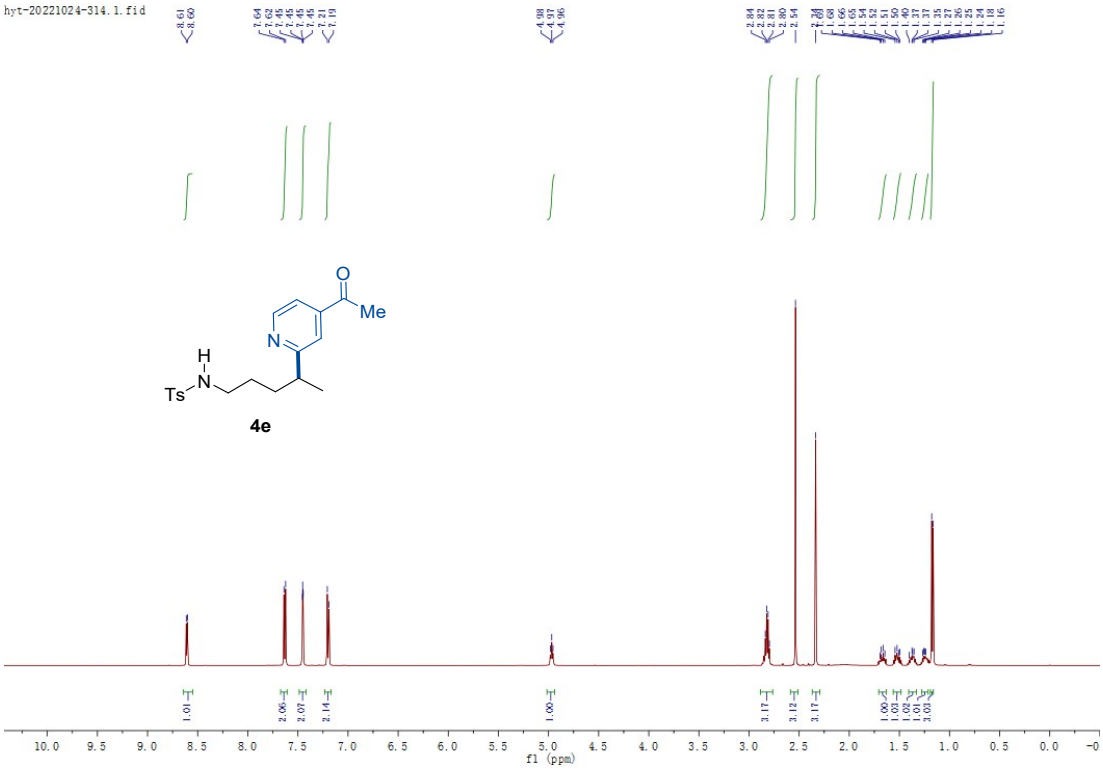


151 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

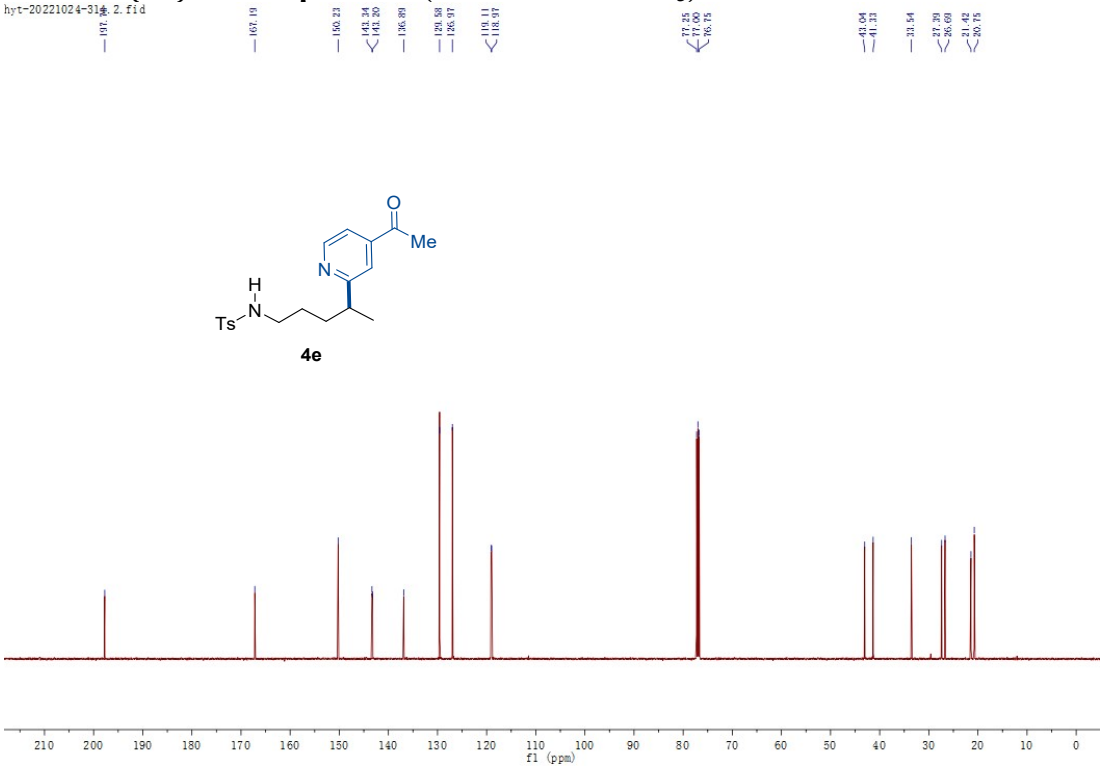


N-(4-(4-Acetylpyridin-2-yl)pentyl)-4-methylbenzenesulfonamide (4e).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

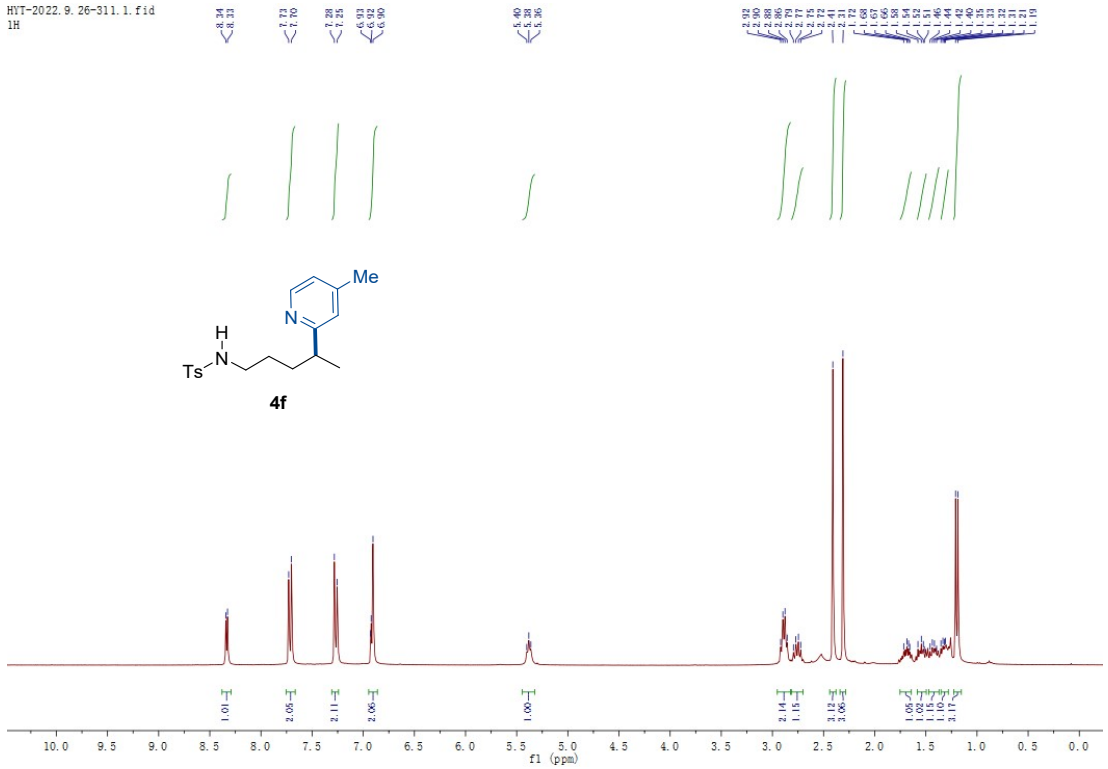


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

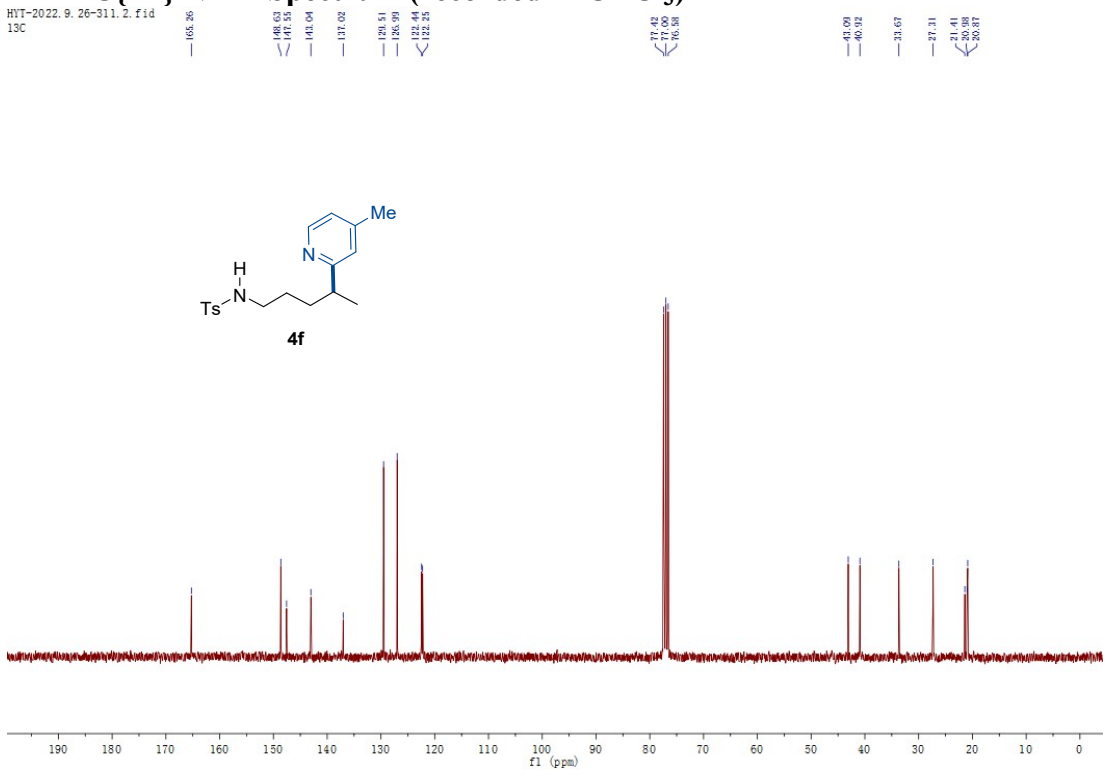


4-Methyl-*N*-(4-(4-methylpyridin-2-yl)pentyl)benzenesulfonamide (4f).

300 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

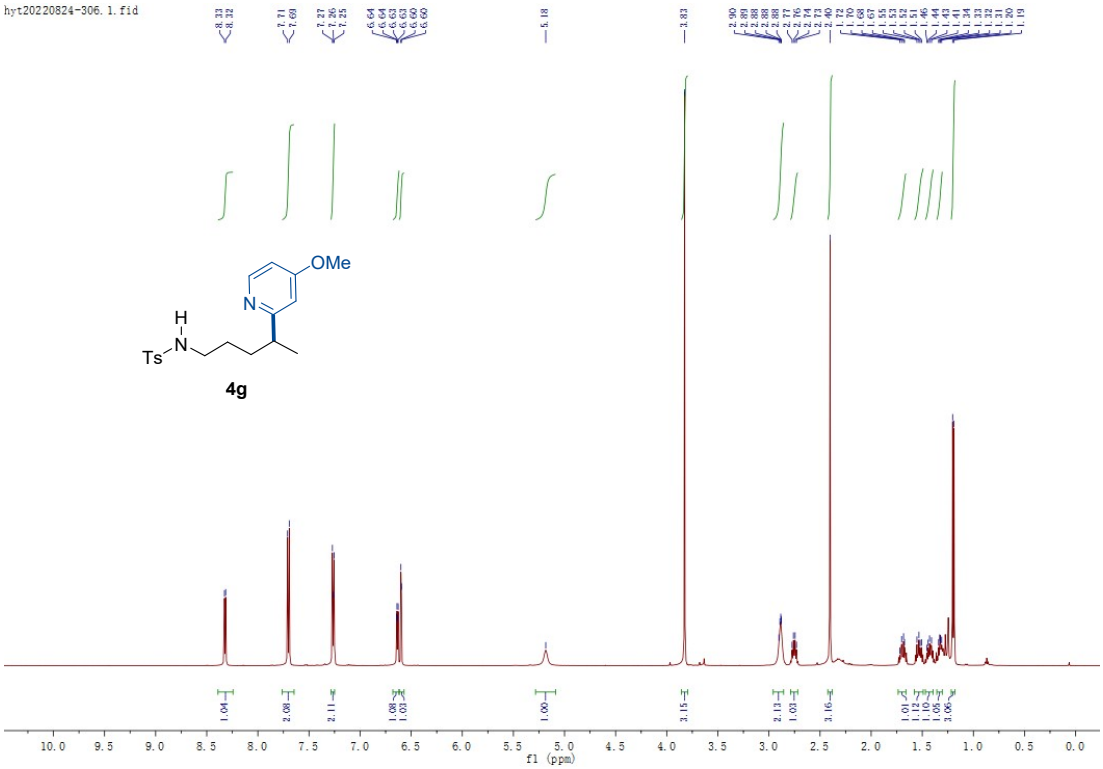


75 MHz $^{13}C\{^{1}H\}$ NMR Spectrum (recorded in $CDCl_3$)

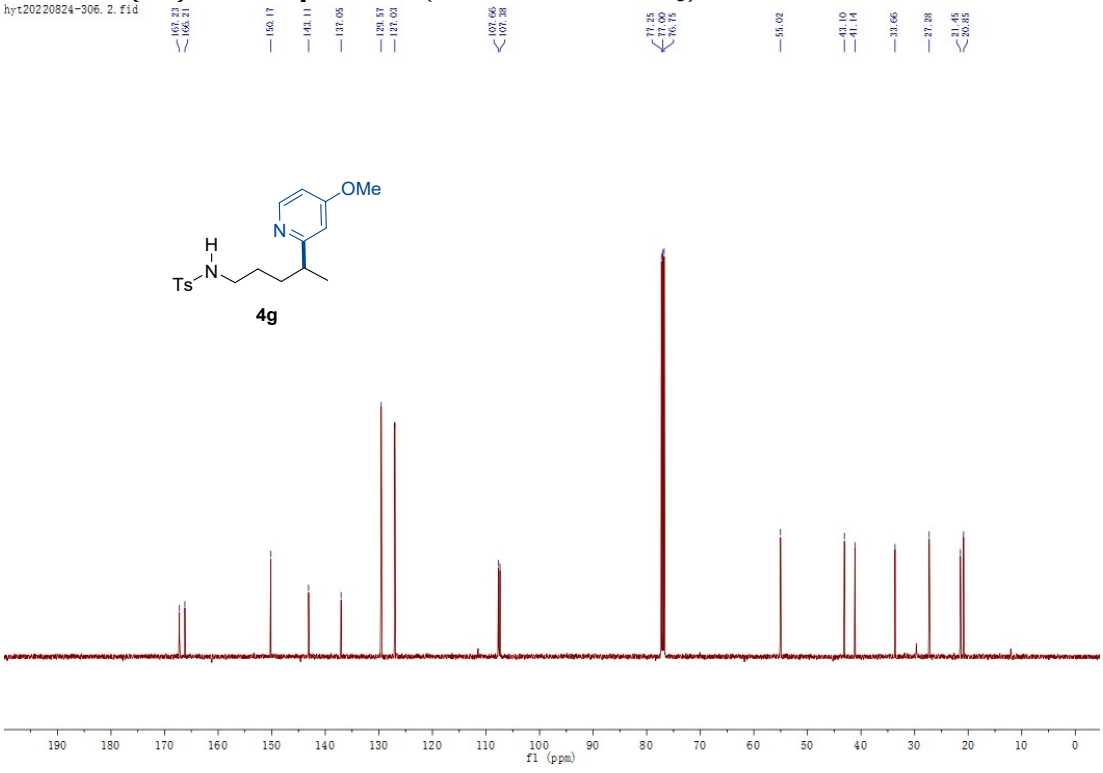


N-(4-(4-Methoxypyridin-2-yl)pentyl)-4-methylbenzenesulfonamide (4g).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

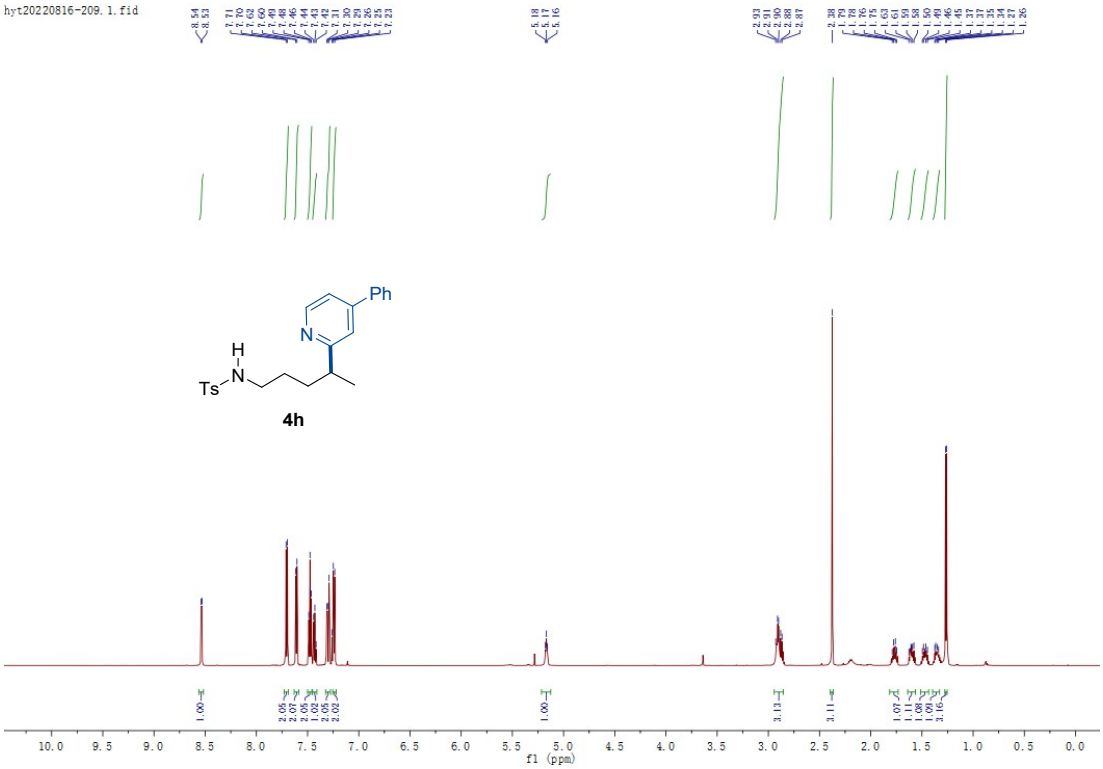


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

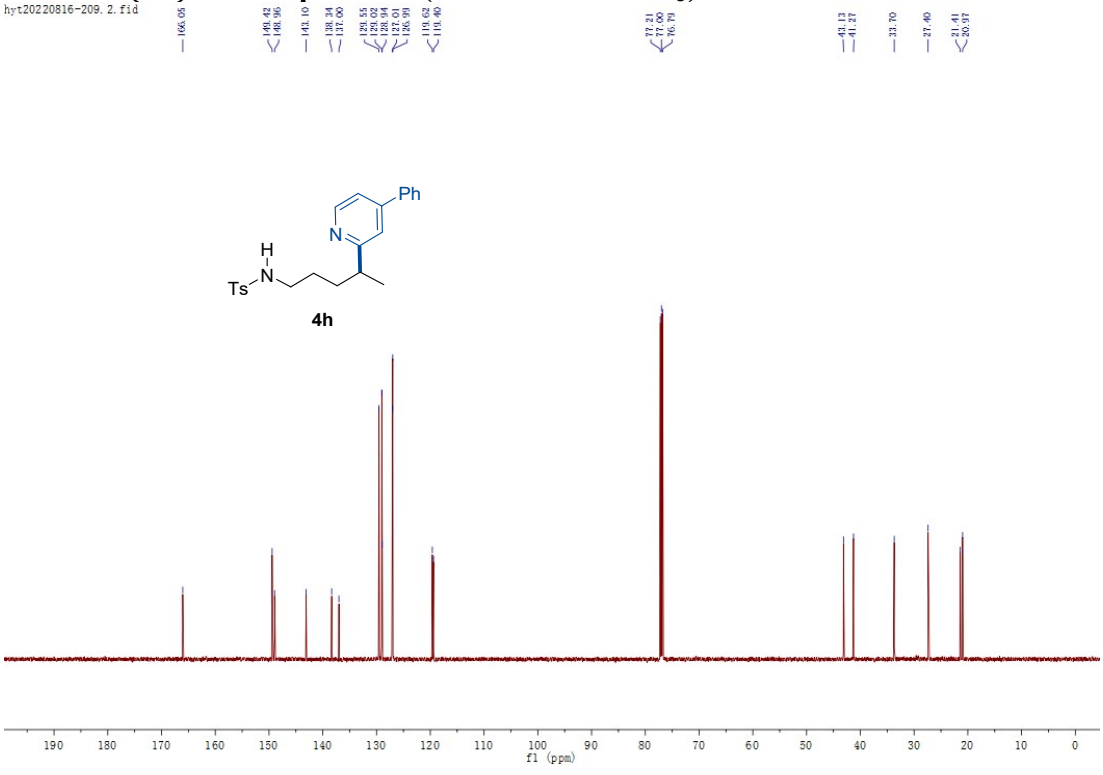


4-Methyl-*N*-(4-(4-phenylpyridin-2-yl)pentyl)benzenesulfonamide (4h).

600 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

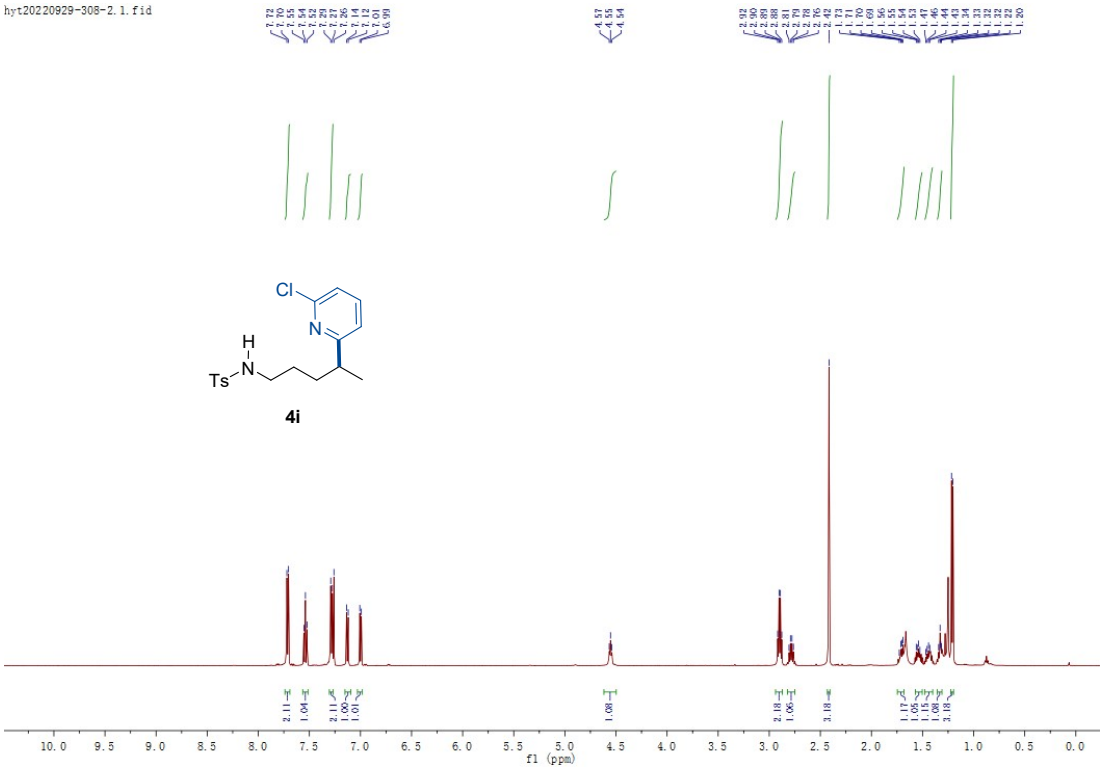


151 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

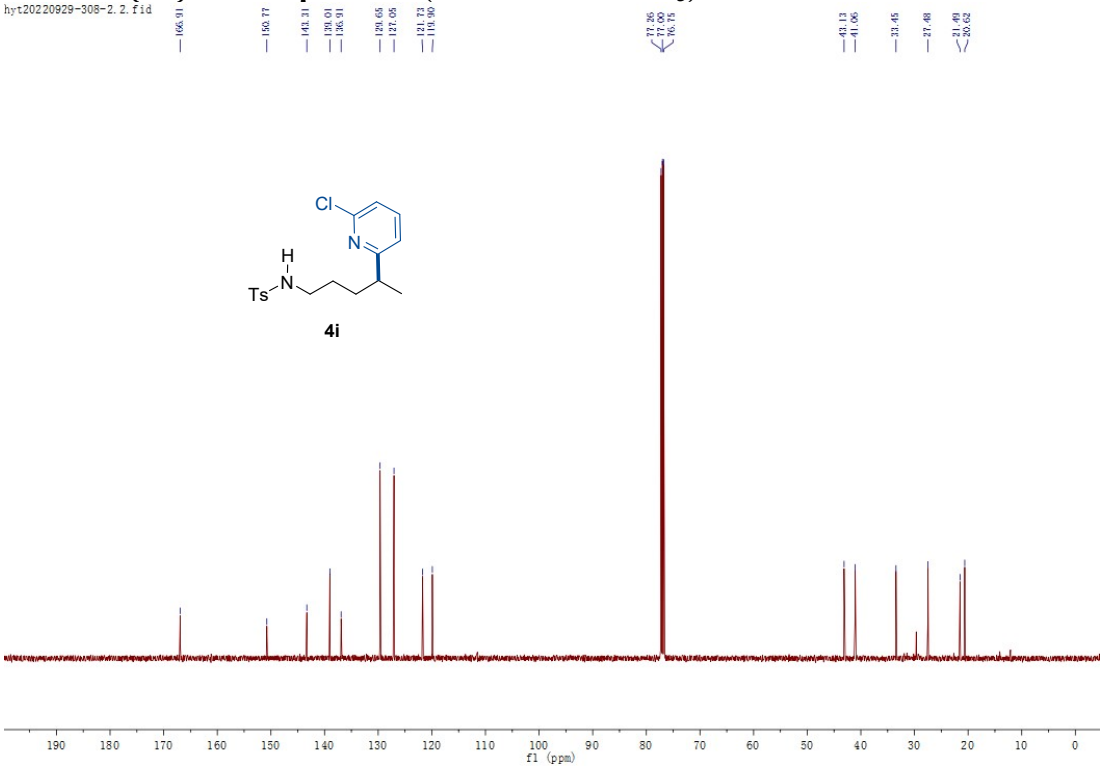


N-(4-(6-Chloropyridin-2-yl)pentyl)-4-methylbenzenesulfonamide (4i).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

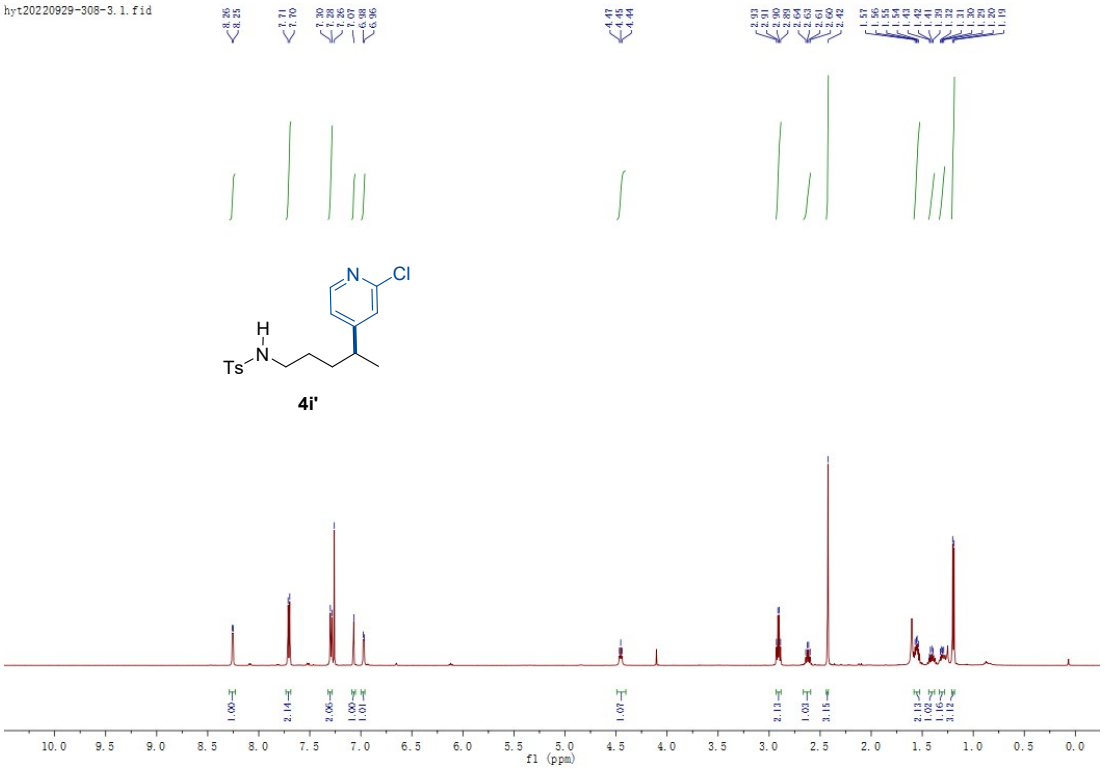


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

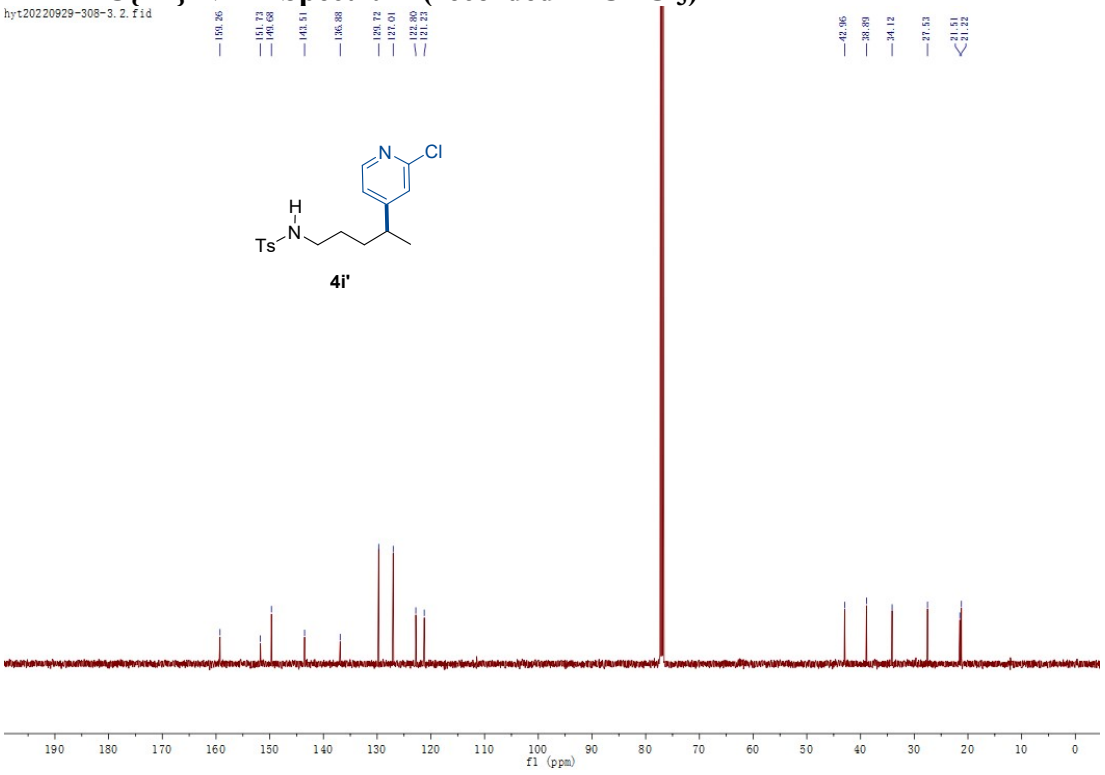


***N*-(4-(2-Chloropyridin-4-yl)pentyl)-4-methylbenzenesulfonamide (4i').**

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

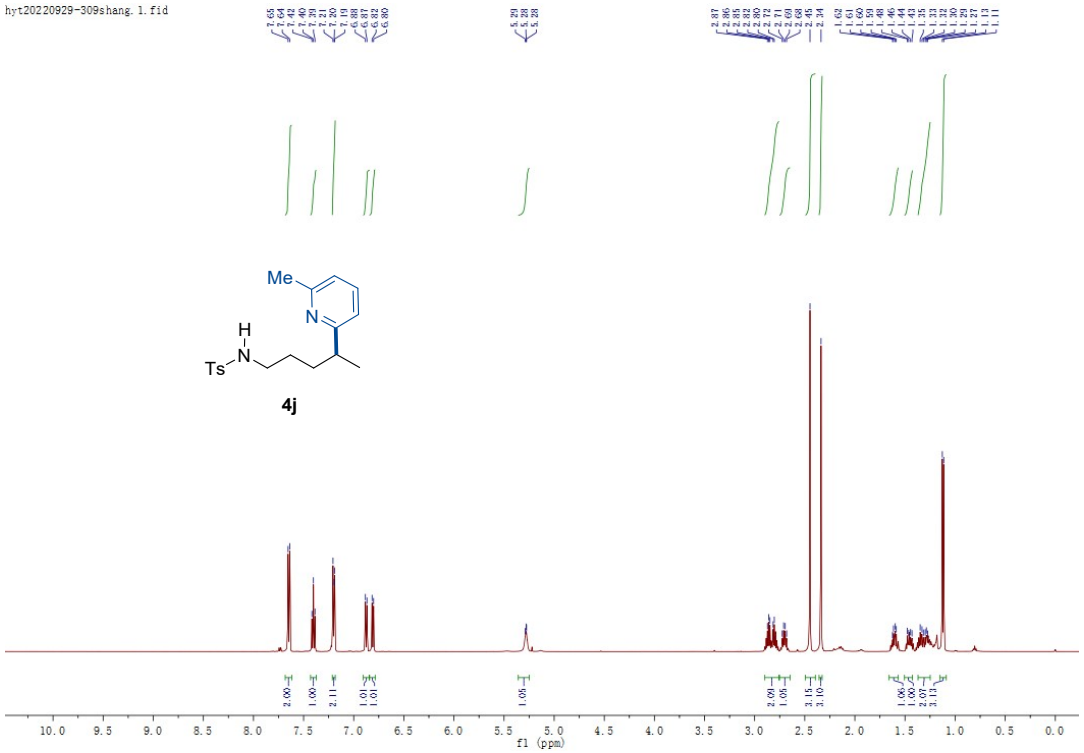


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

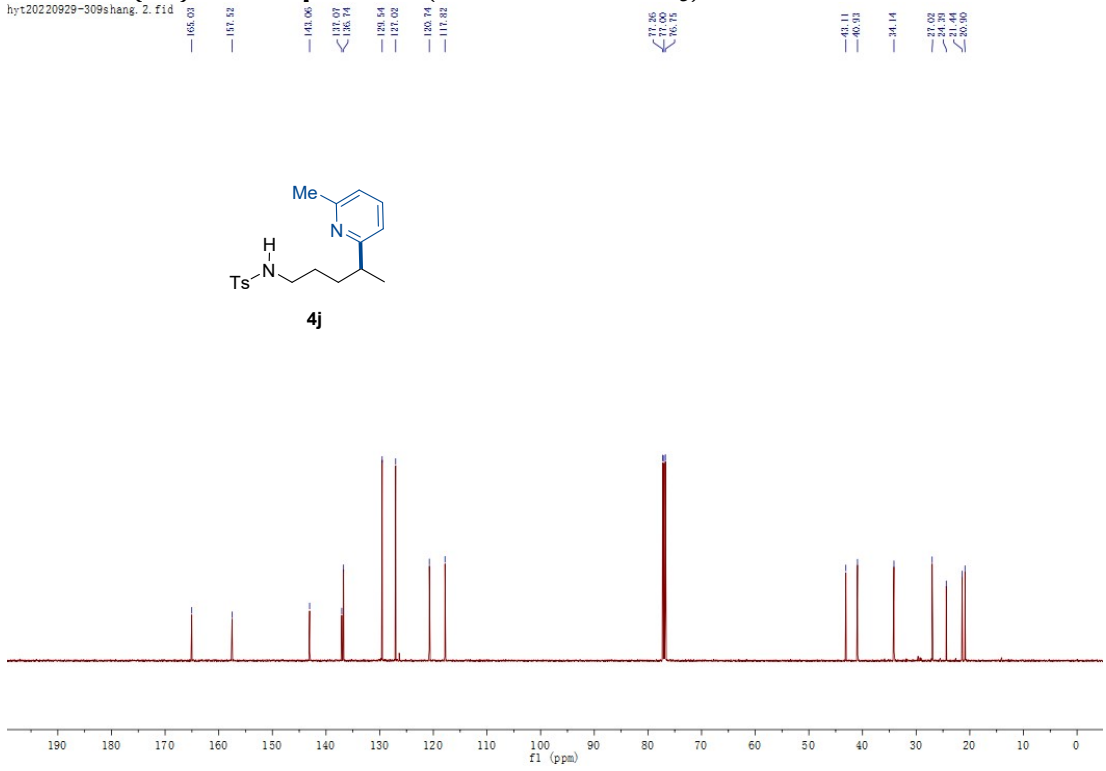


4-Methyl-*N*-(4-(6-methylpyridin-2-yl)pentyl)benzenesulfonamide (4j).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

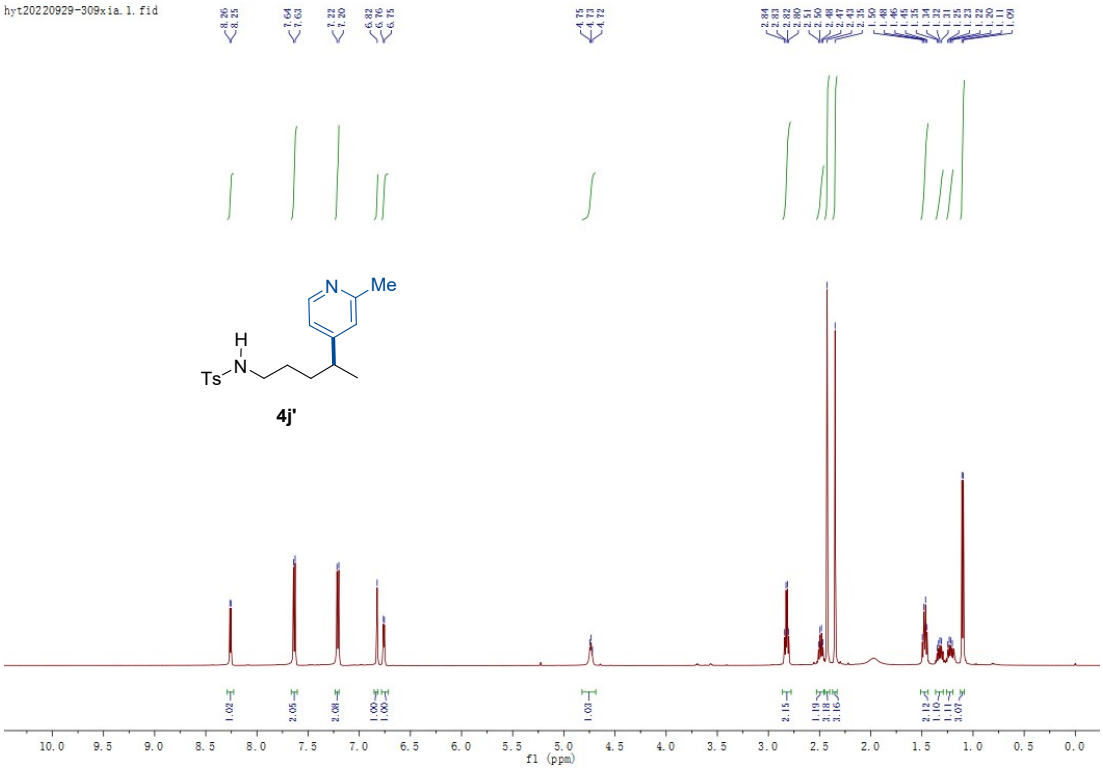


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

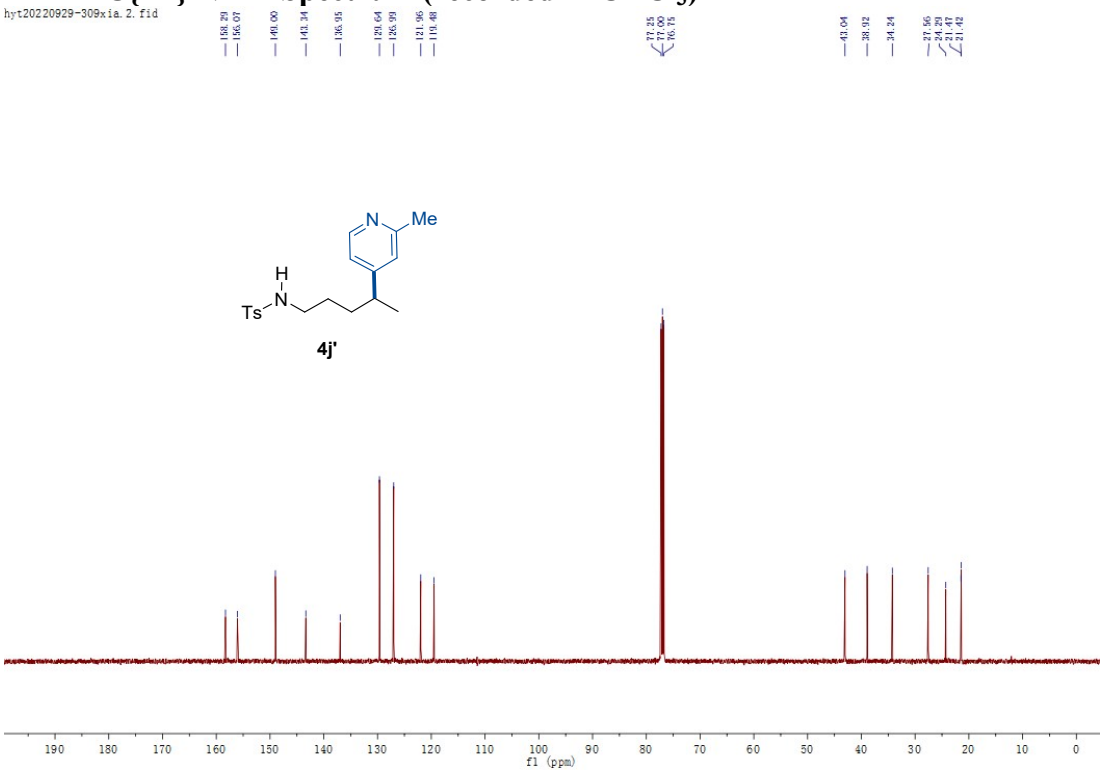


4-Methyl-*N*-(4-(2-methylpyridin-4-yl)pentyl)benzenesulfonamide (4j').

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

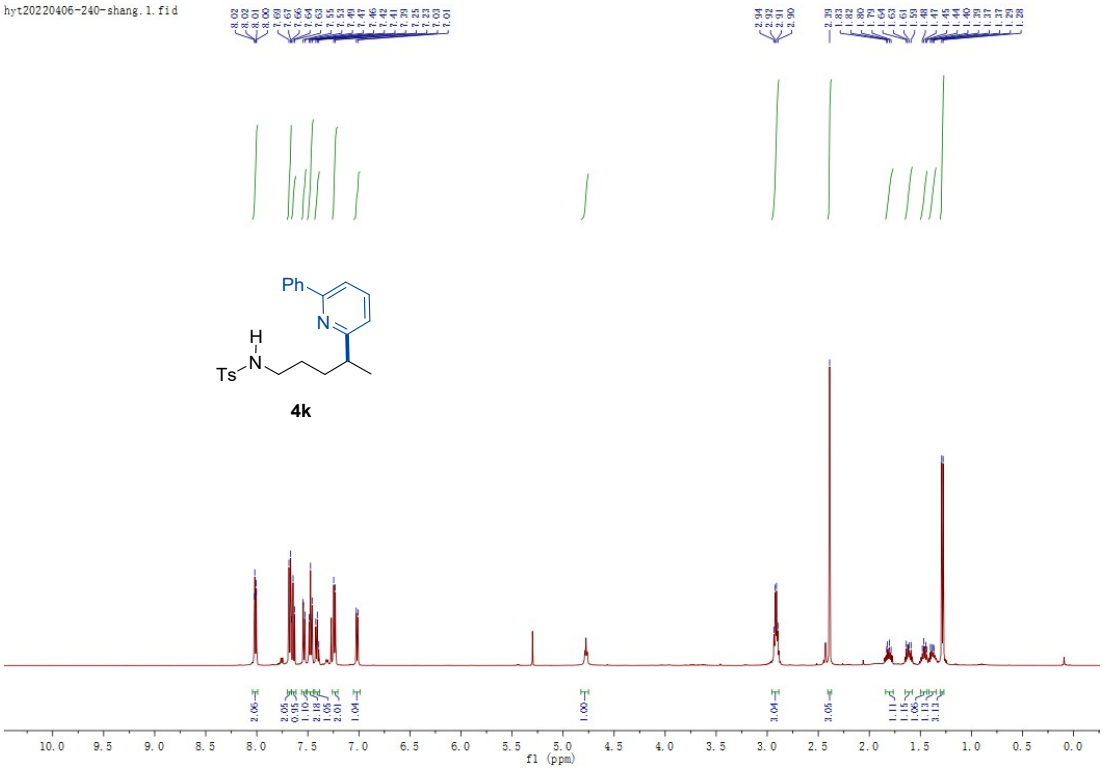


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

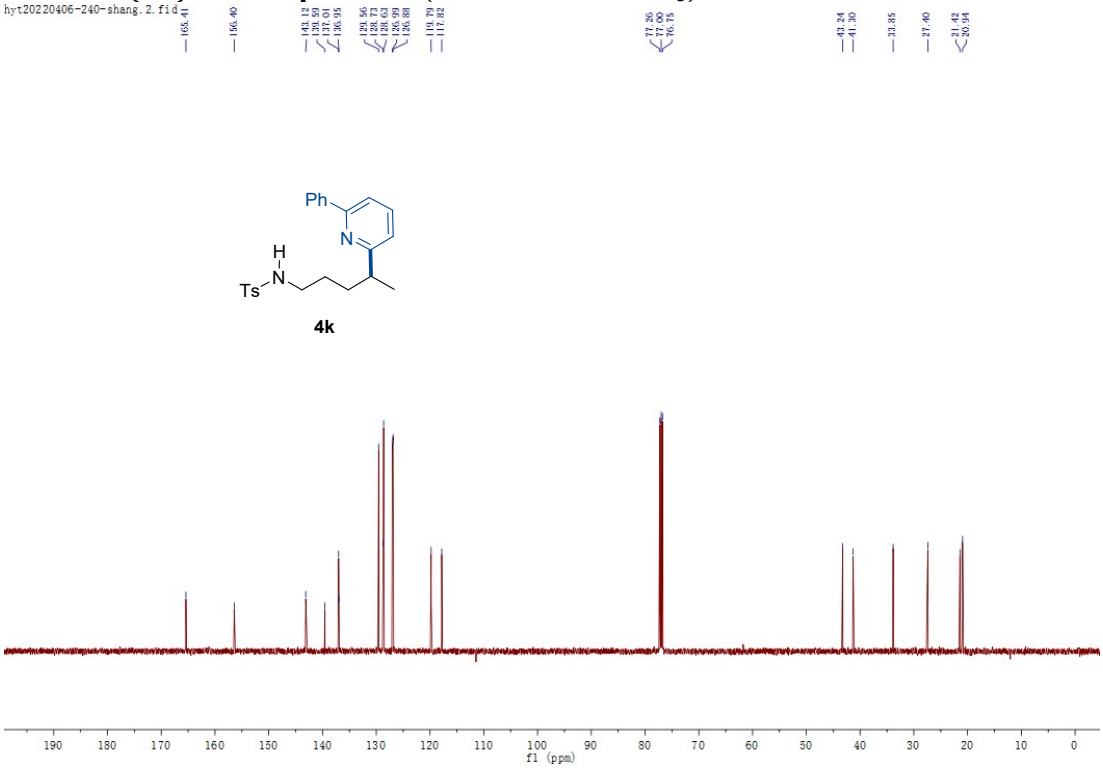


4-Methyl-*N*-(4-(6-phenylpyridin-2-yl)pentyl)benzenesulfonamide (4k).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

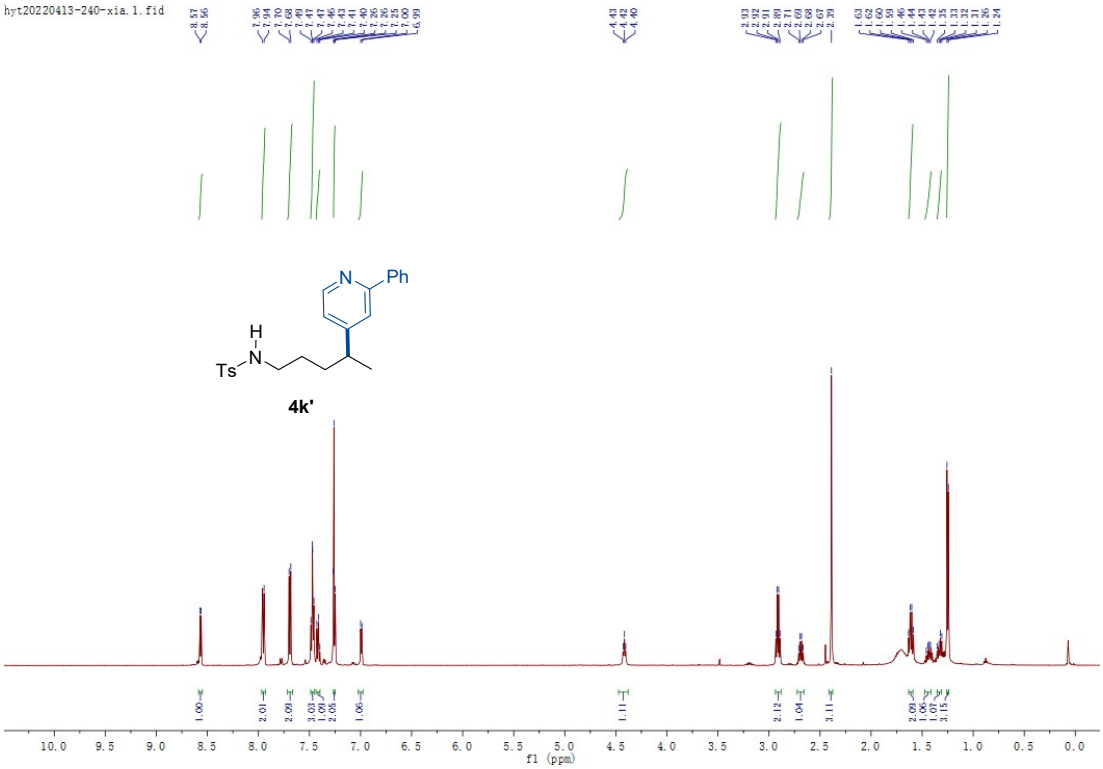


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

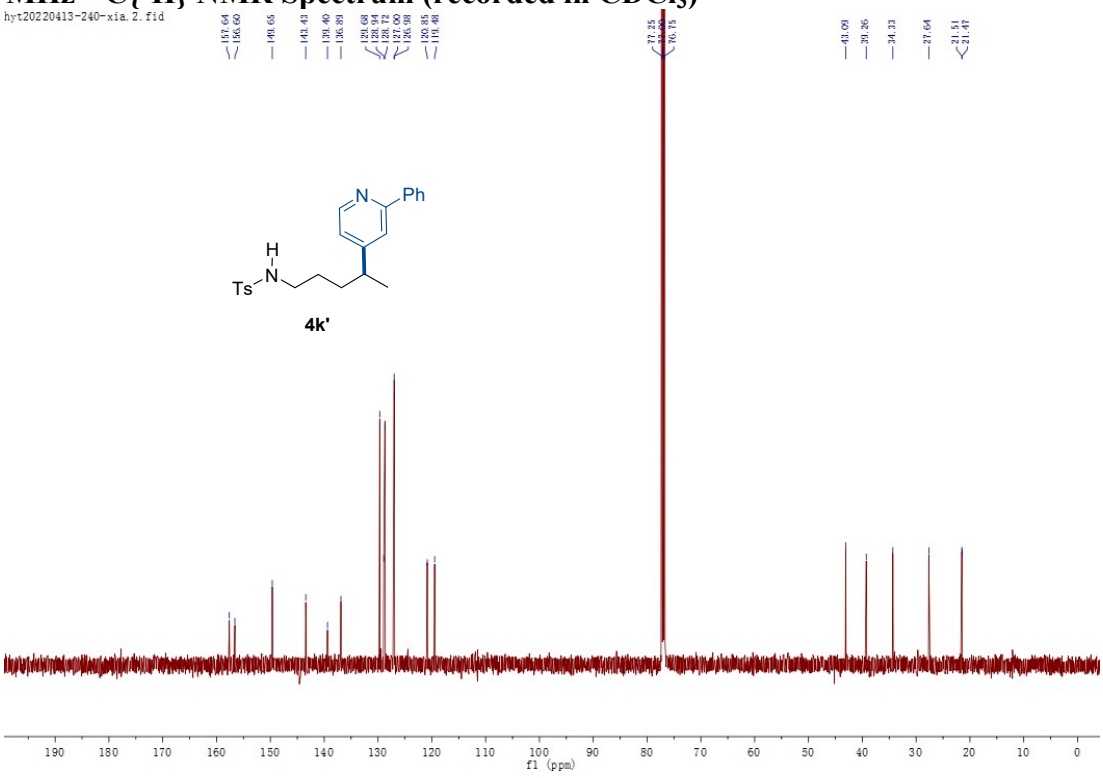


4-Methyl-*N*-(4-(2-phenylpyridin-4-yl)pentyl)benzenesulfonamide (4k').

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

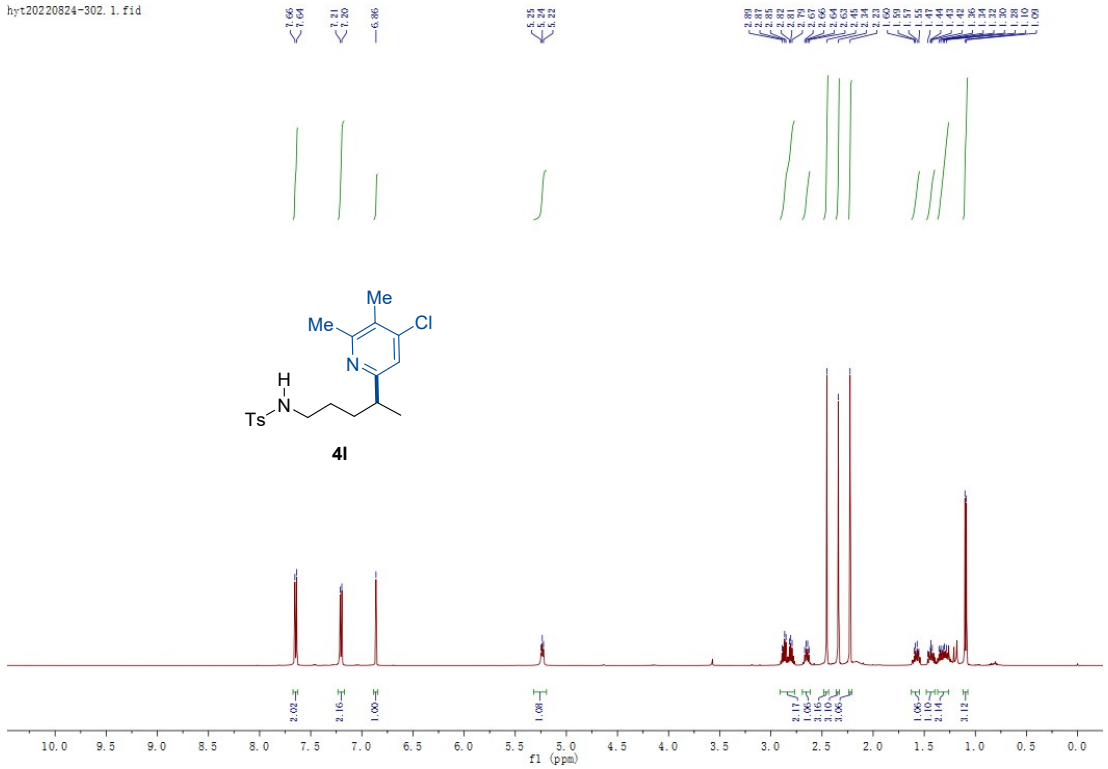


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

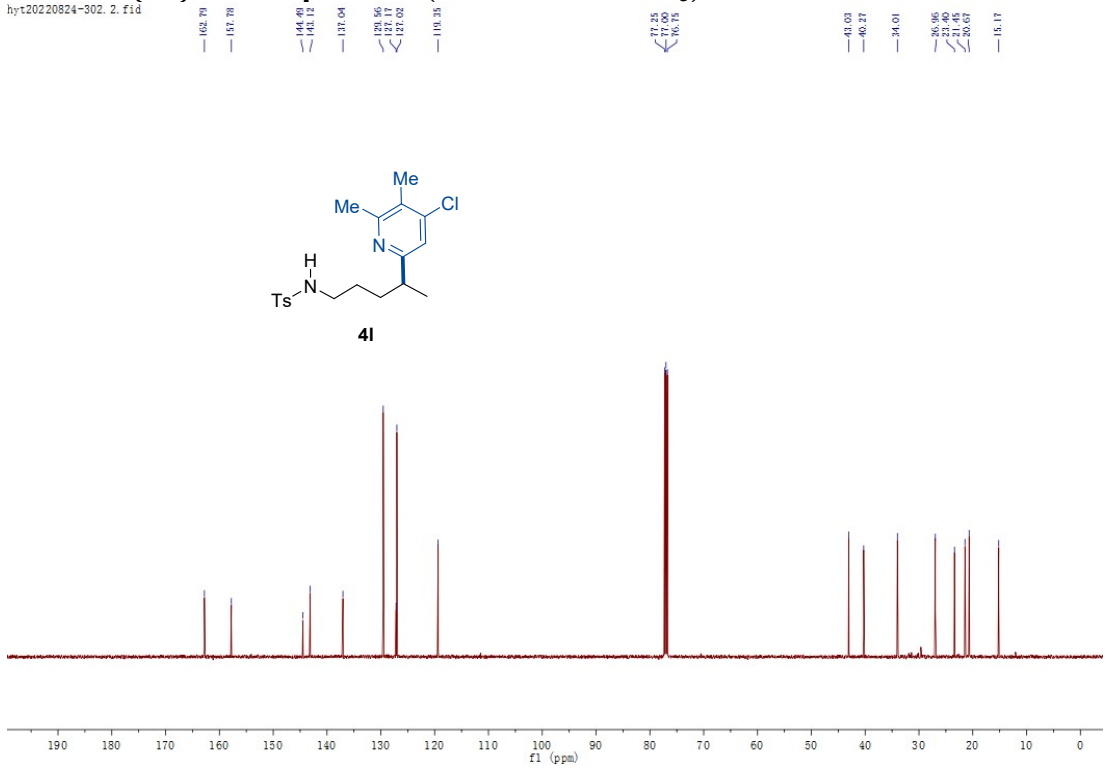


***N*-(4-(4-Chloro-5,6-dimethylpyridin-2-yl)pentyl)-4-methylbenzenesulfonamide (4l).**

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

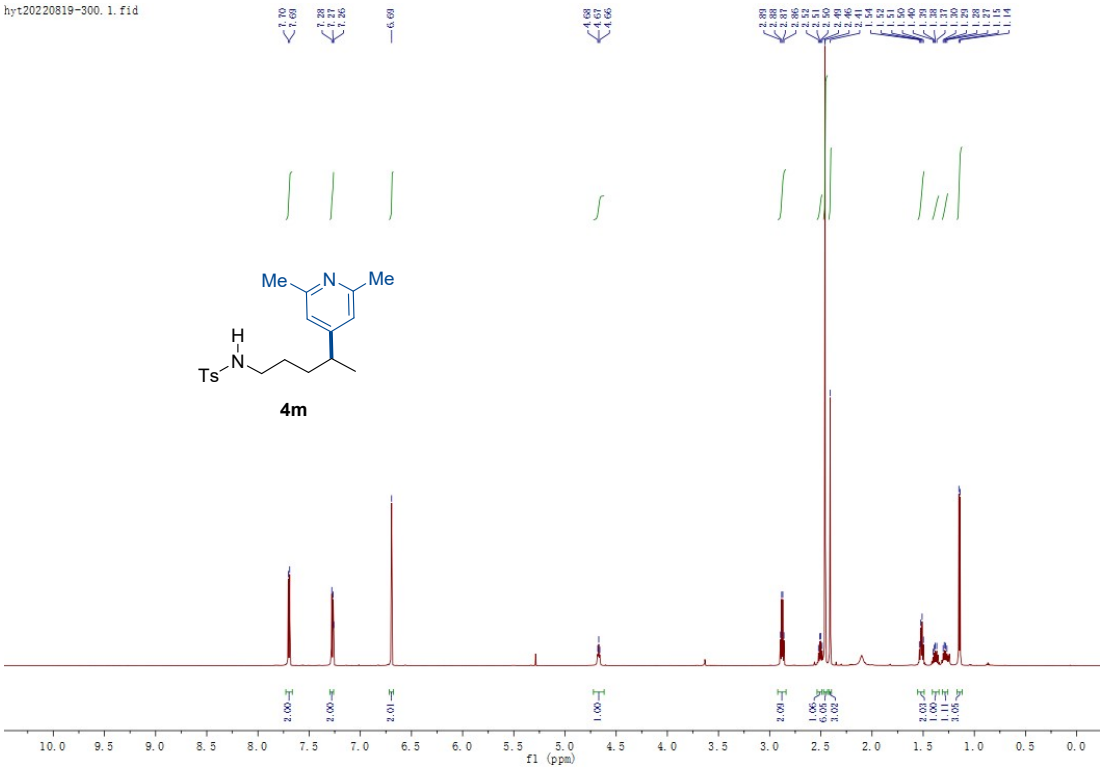


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

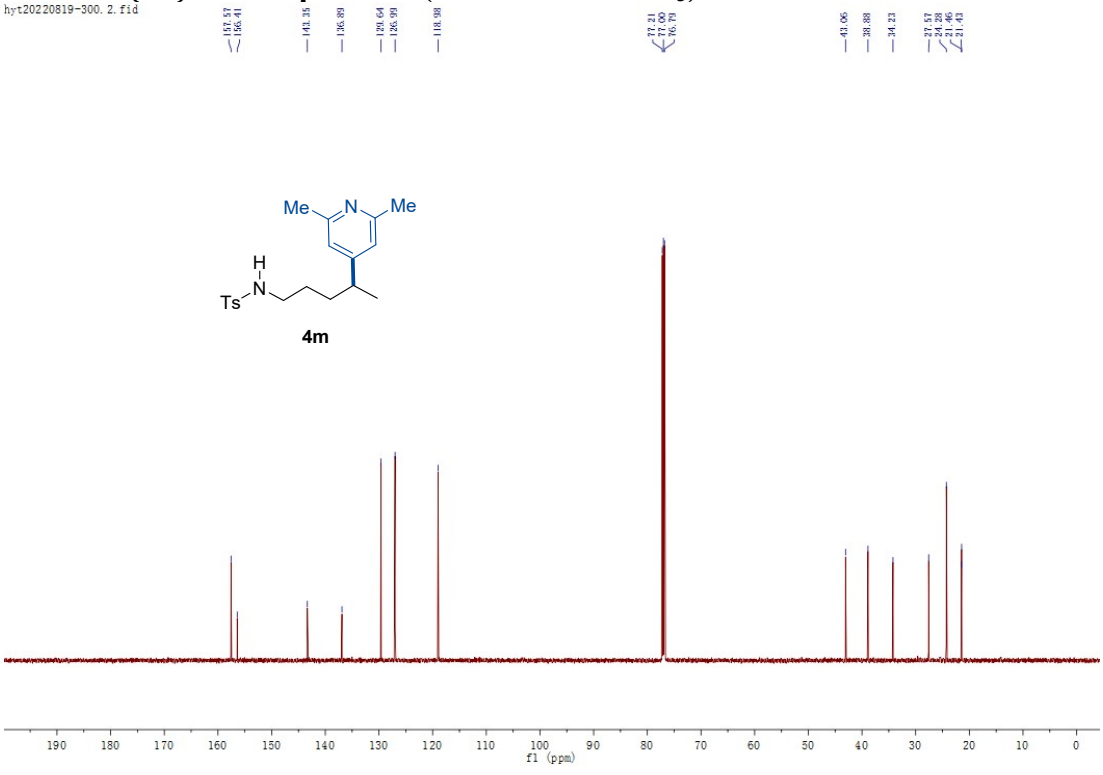


N-(4-(2,6-Dimethylpyridin-4-yl)pentyl)-4-methylbenzenesulfonamide (4m).

600 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

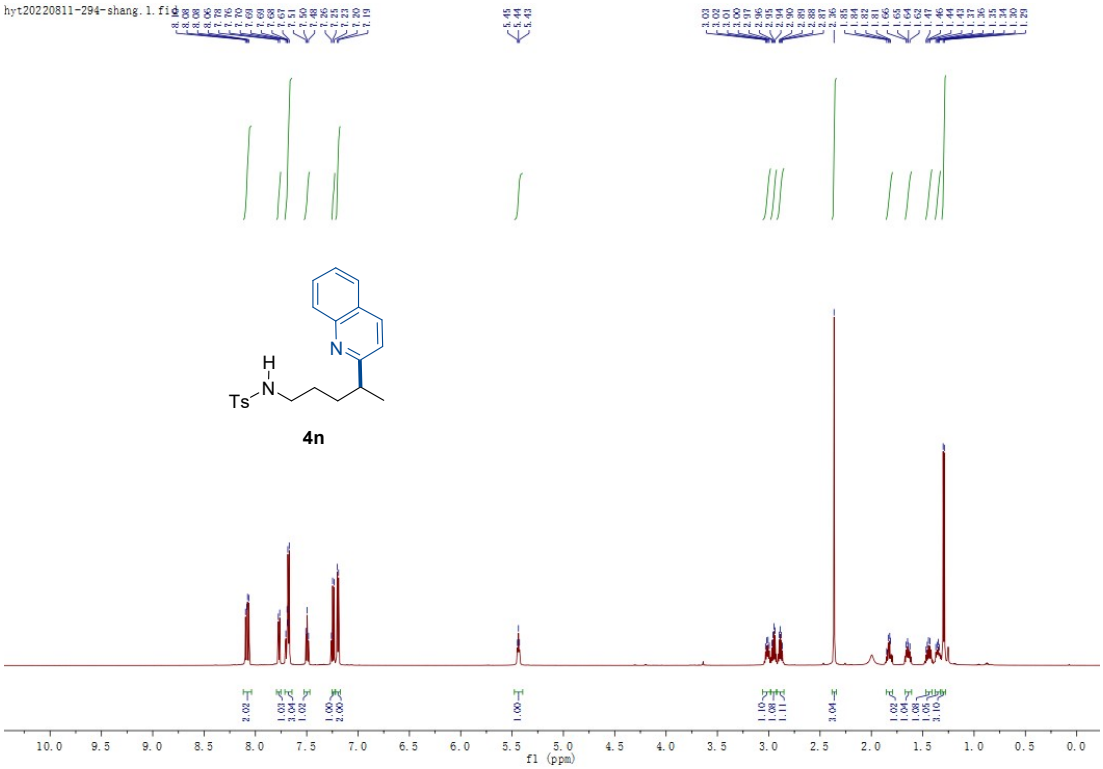


151 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

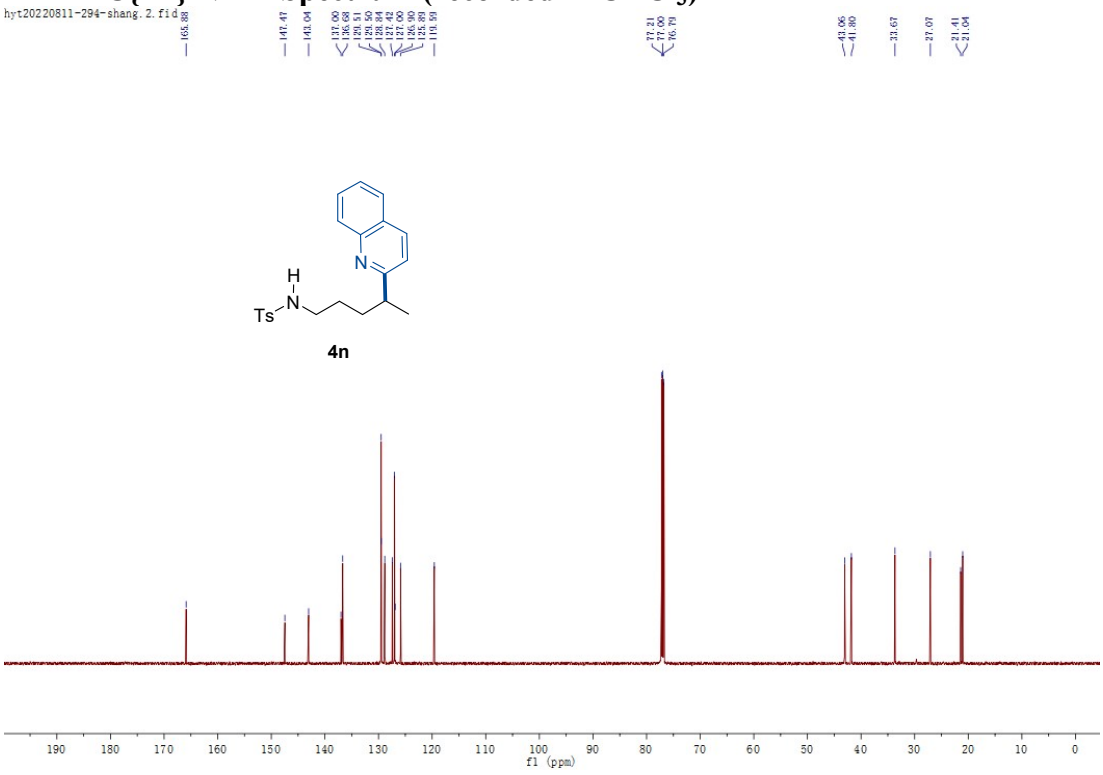


4-Methyl-*N*-(4-(quinolin-2-yl)pentyl)benzenesulfonamide (4n).

600 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

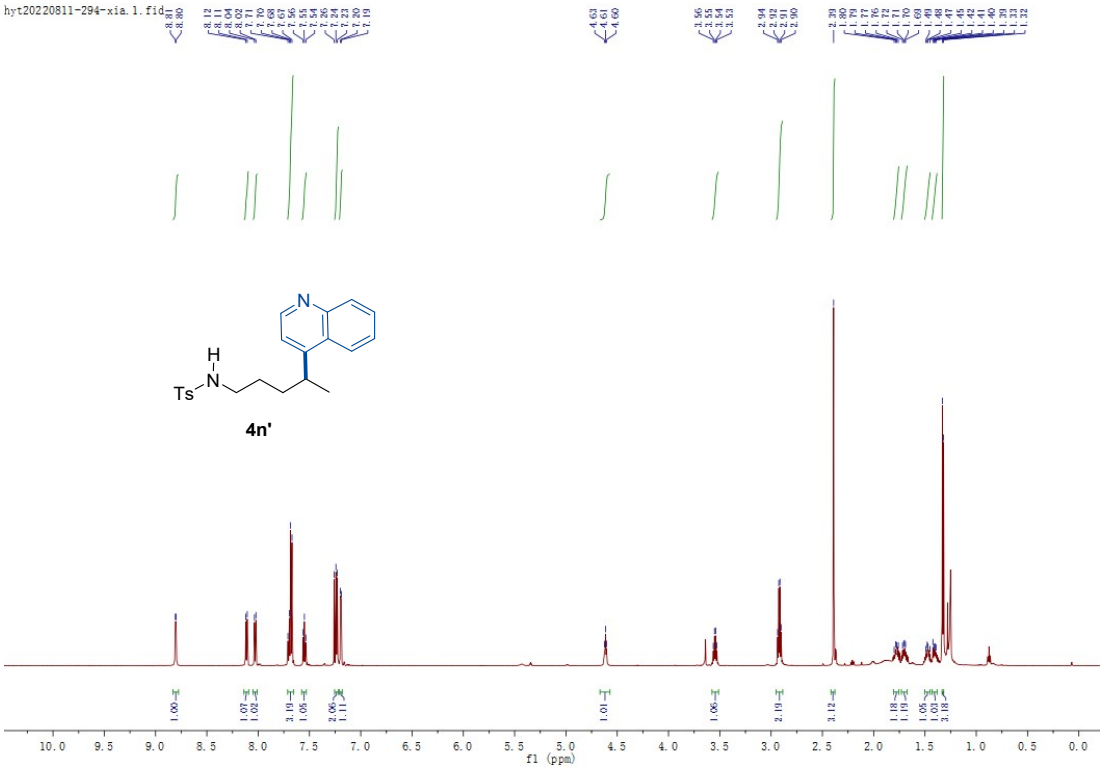


151 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

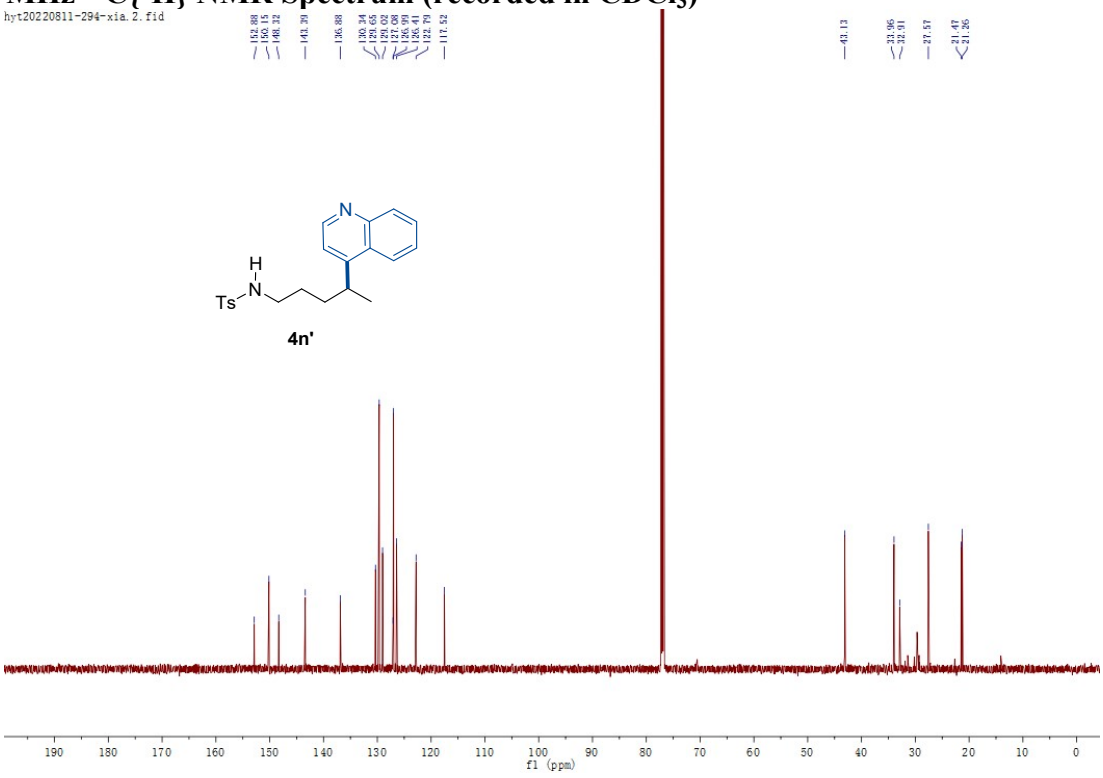


4-Methyl-*N*-(4-(quinolin-4-yl)pentyl)benzenesulfonamide (4n').

600 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

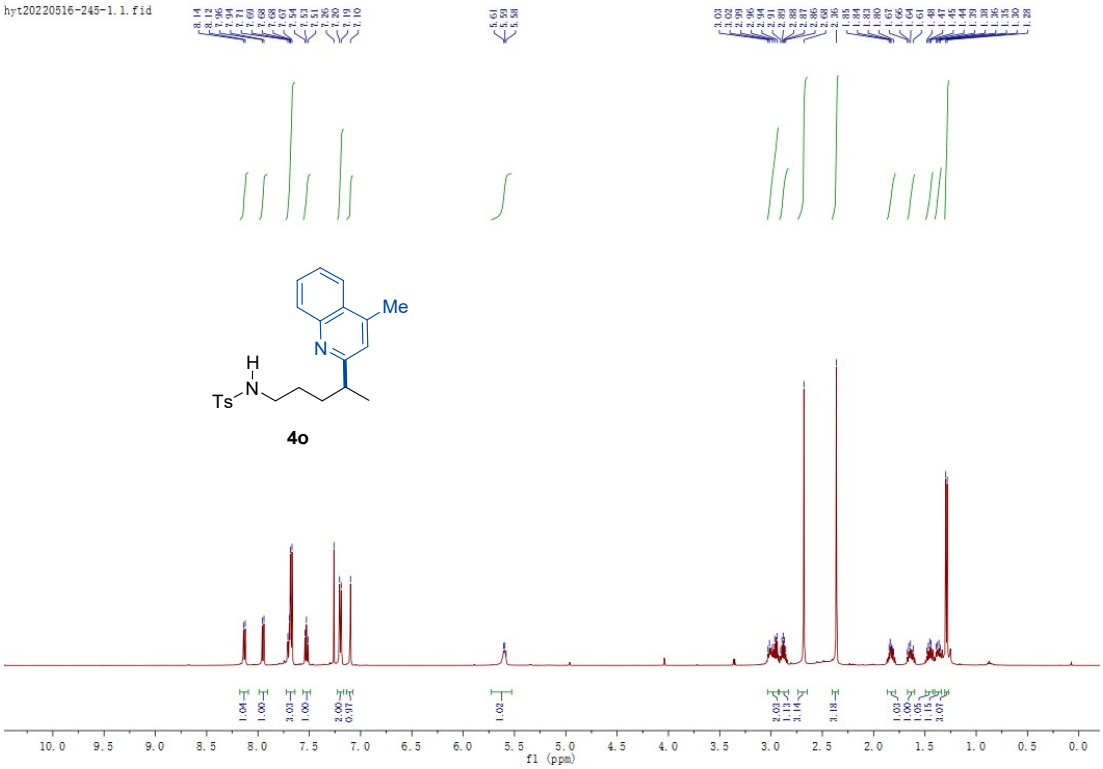


151 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

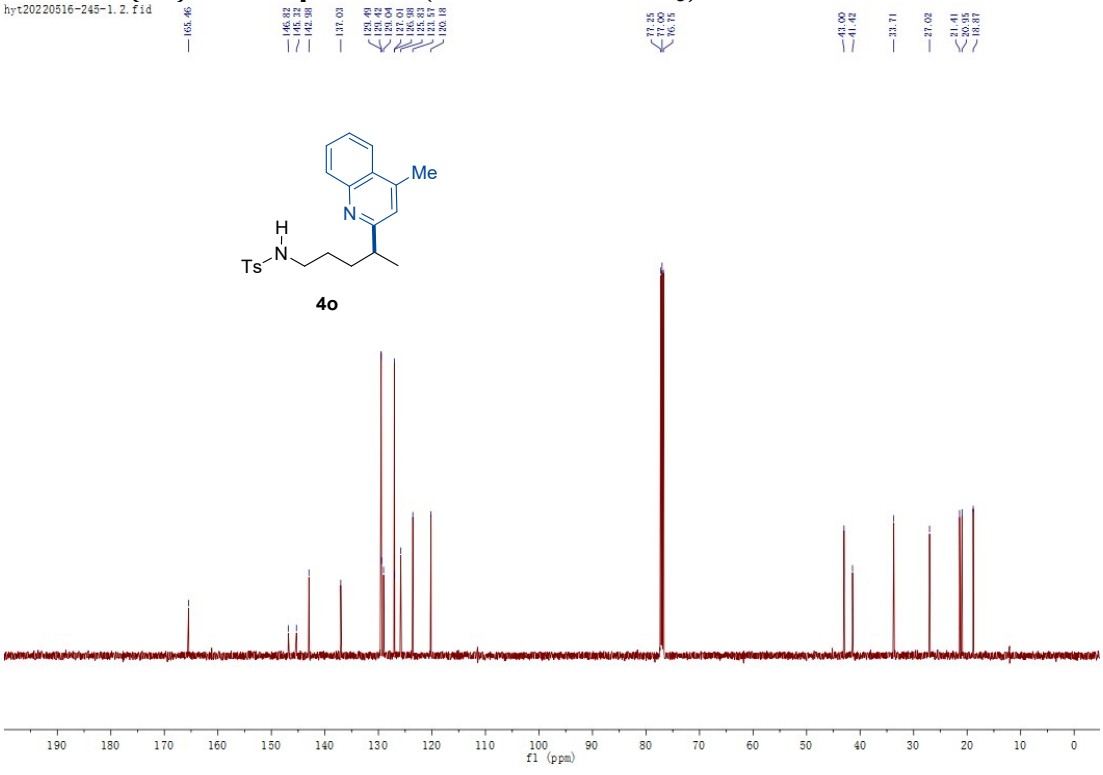


4-Methyl-*N*-(4-(4-methylquinolin-2-yl)pentyl)benzenesulfonamide (4o).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

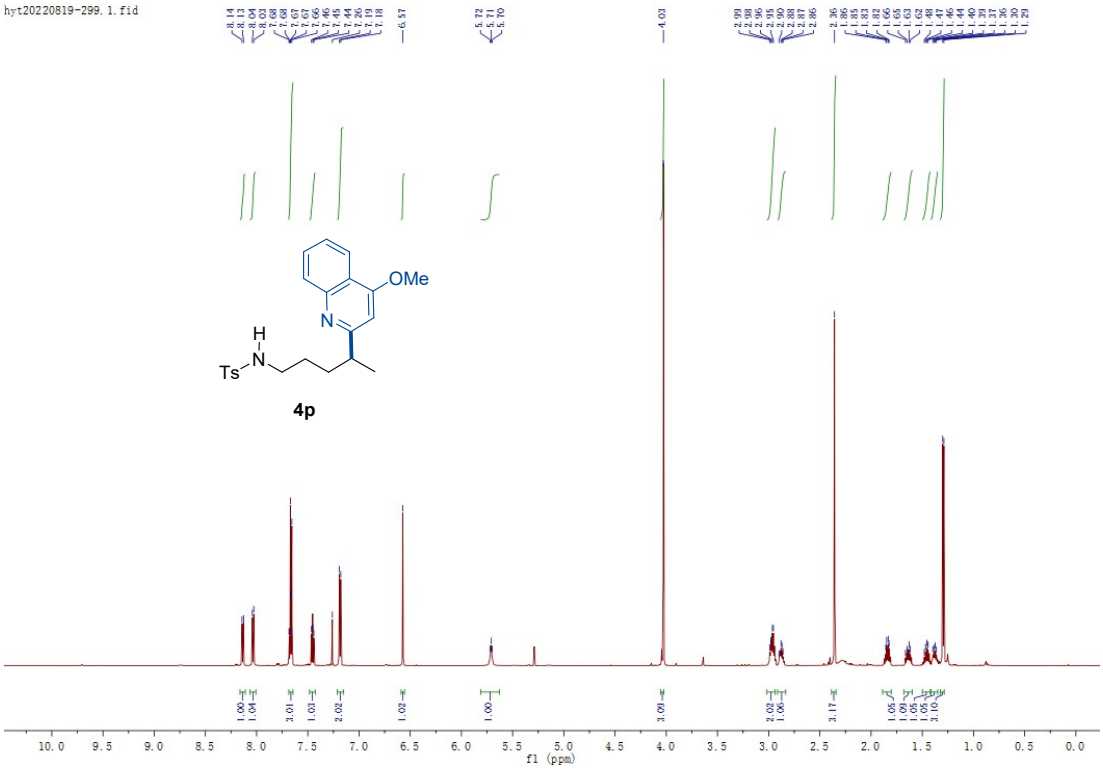


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

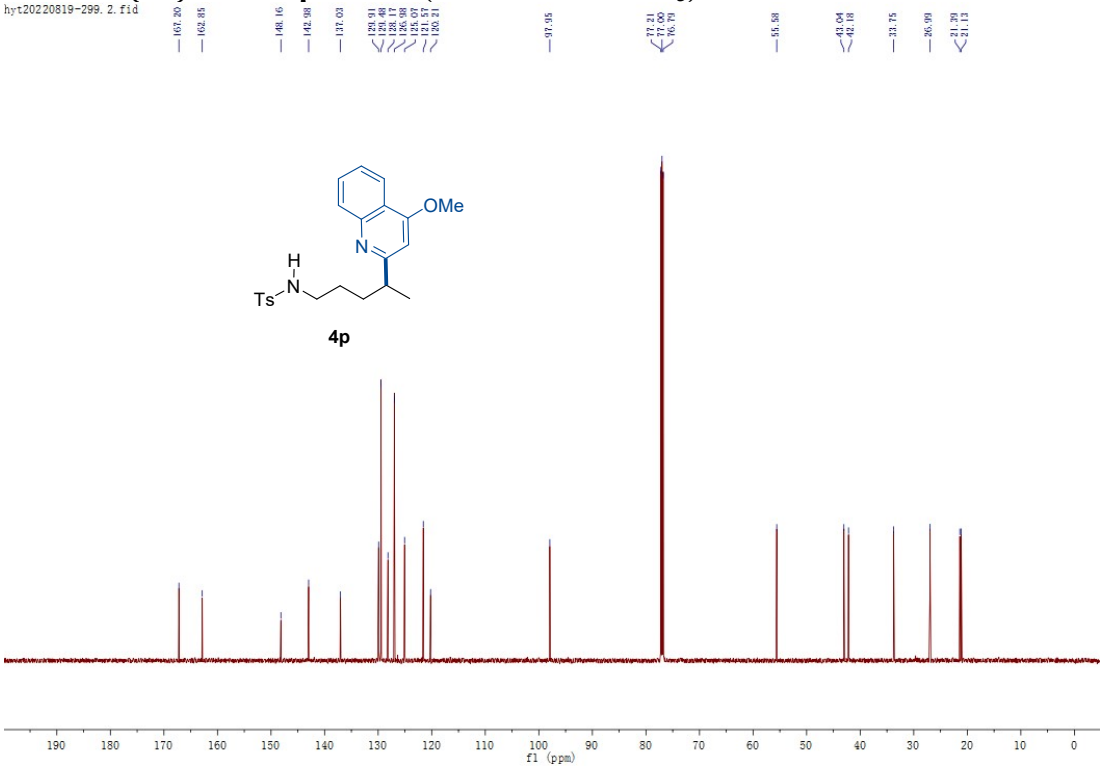


***N*-(4-(4-Methoxyquinolin-2-yl)pentyl)-4-methylbenzenesulfonamide (4p).**

600 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

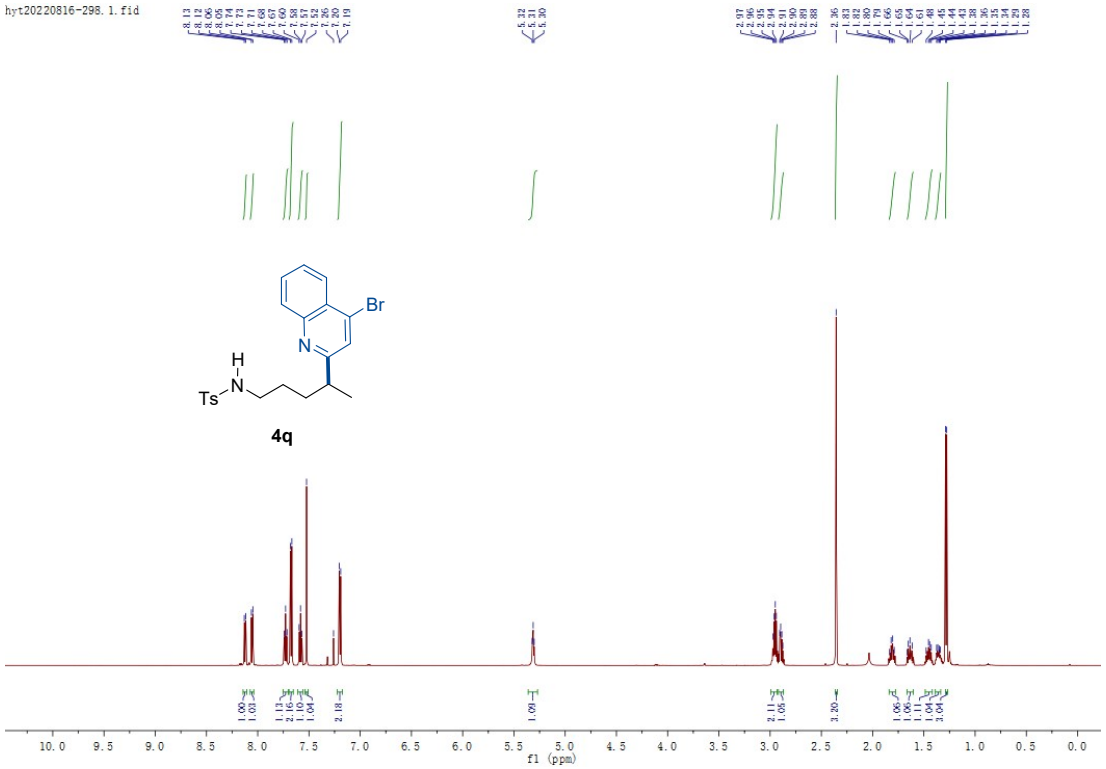


151 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

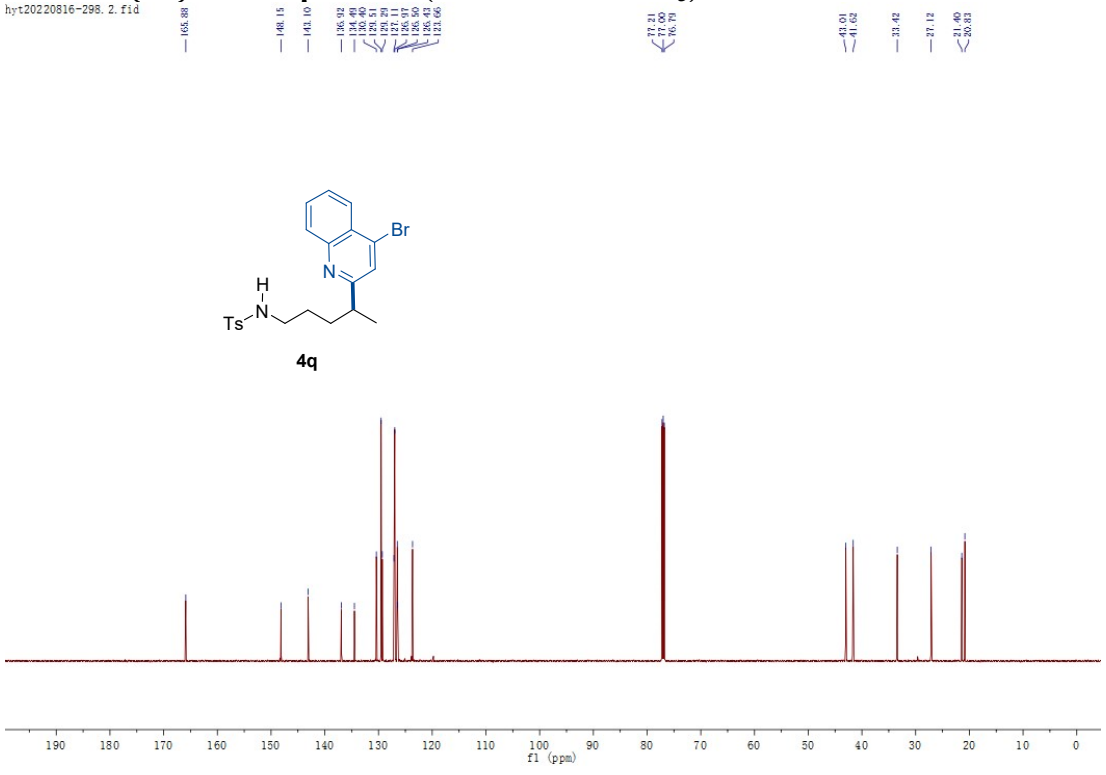


N-(4-(4-Bromoquinolin-2-yl)pentyl)-4-methylbenzenesulfonamide (4q).

600 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

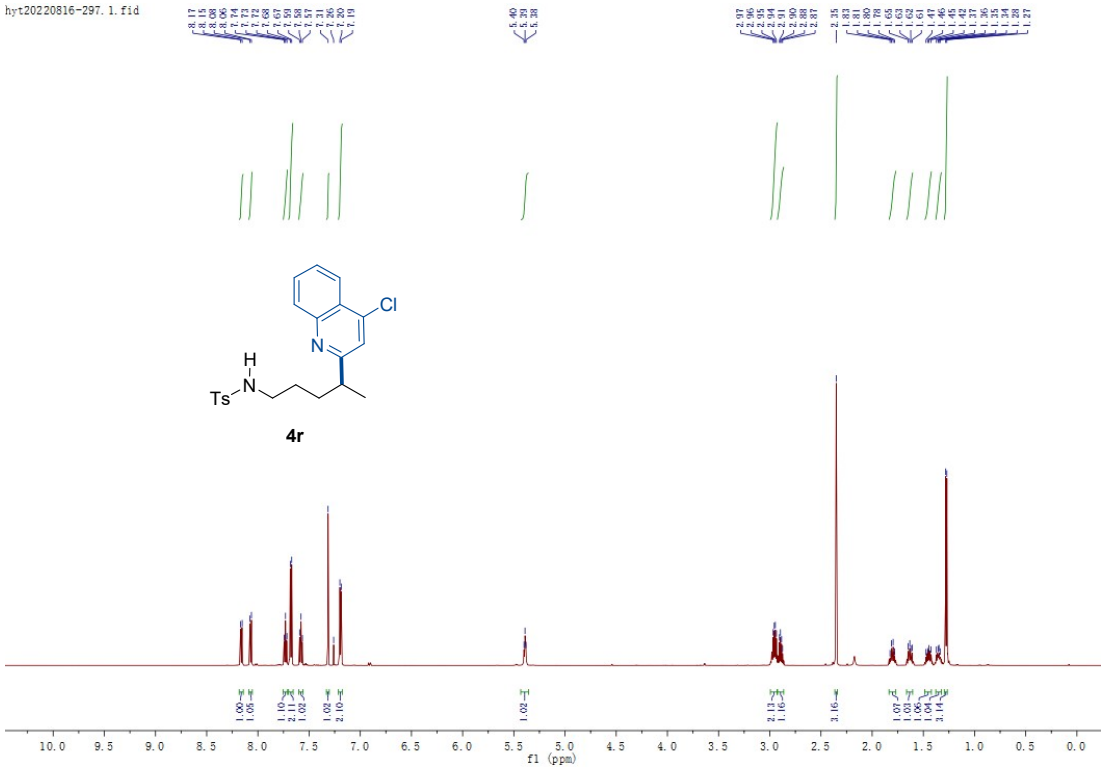


151 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

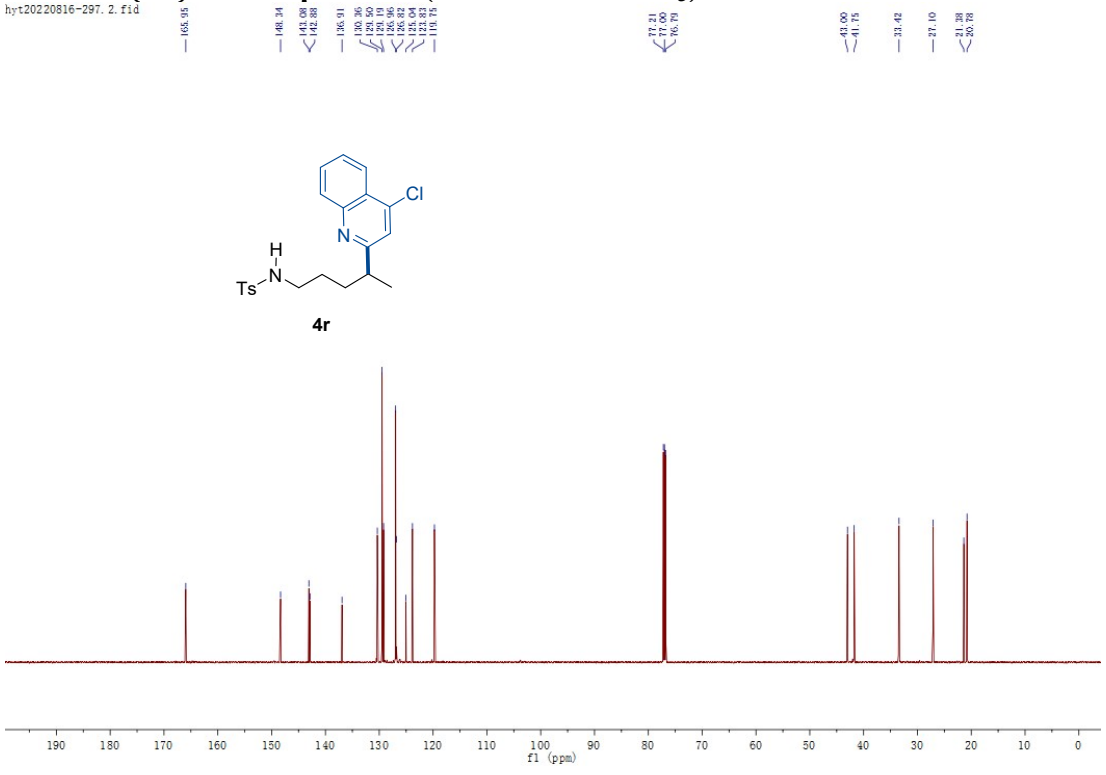


***N*-(4-(4-Chloroquinolin-2-yl)pentyl)-4-methylbenzenesulfonamide (4r).**

600 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

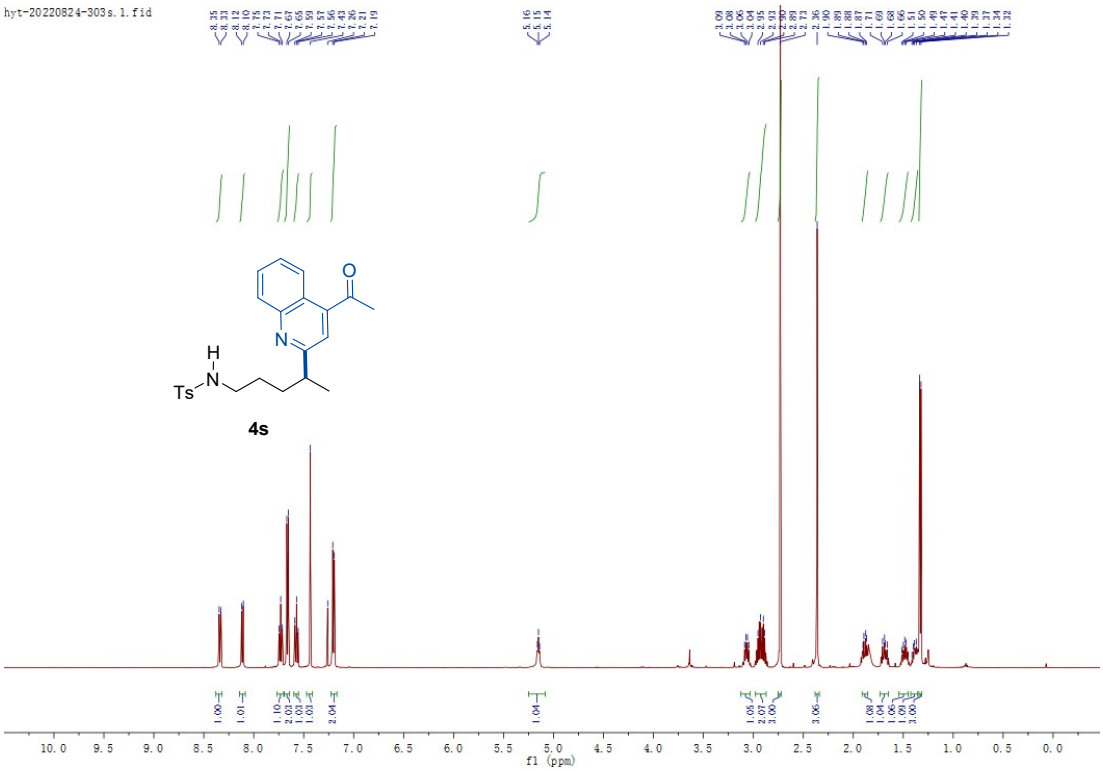


151 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

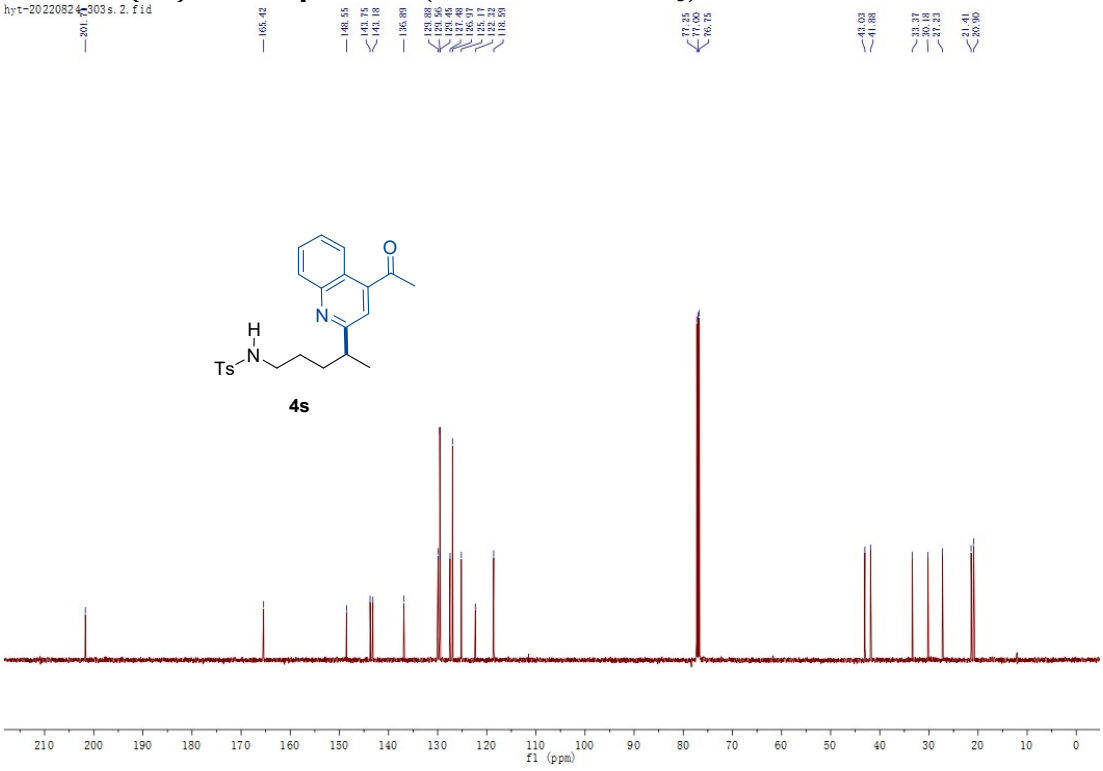


***N*-(4-(4-acetylquinolin-2-yl)pentyl)-4-methylbenzenesulfonamide (4s).**

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

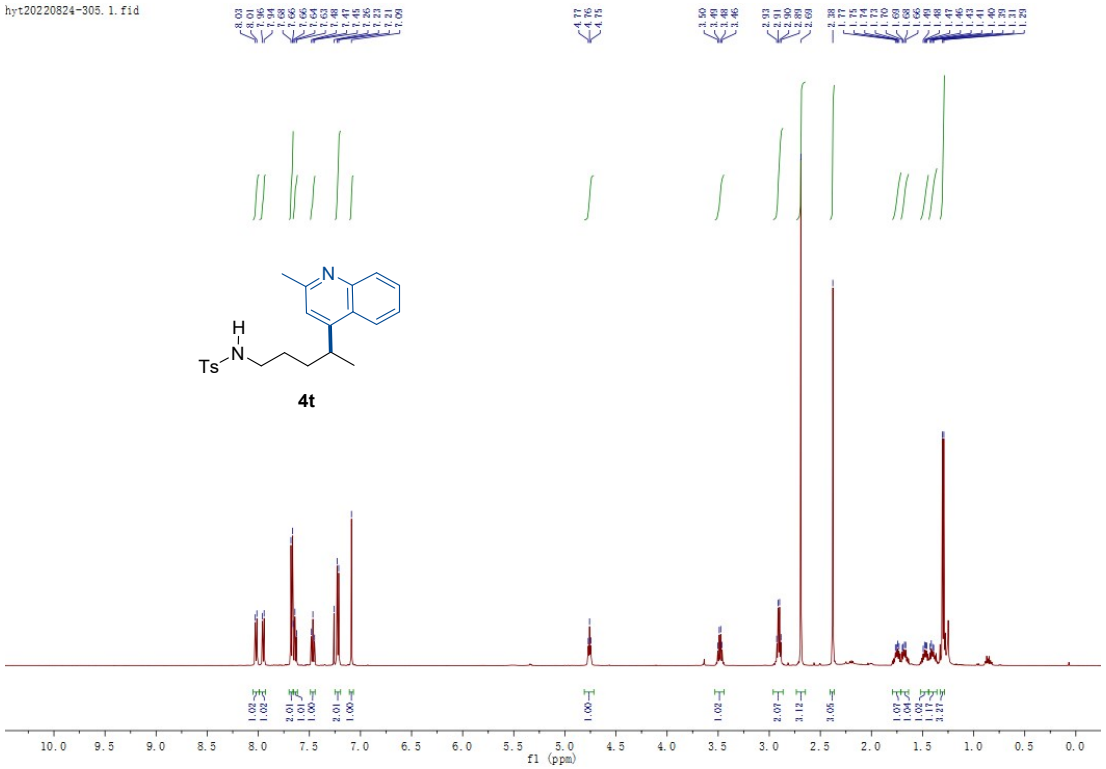


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

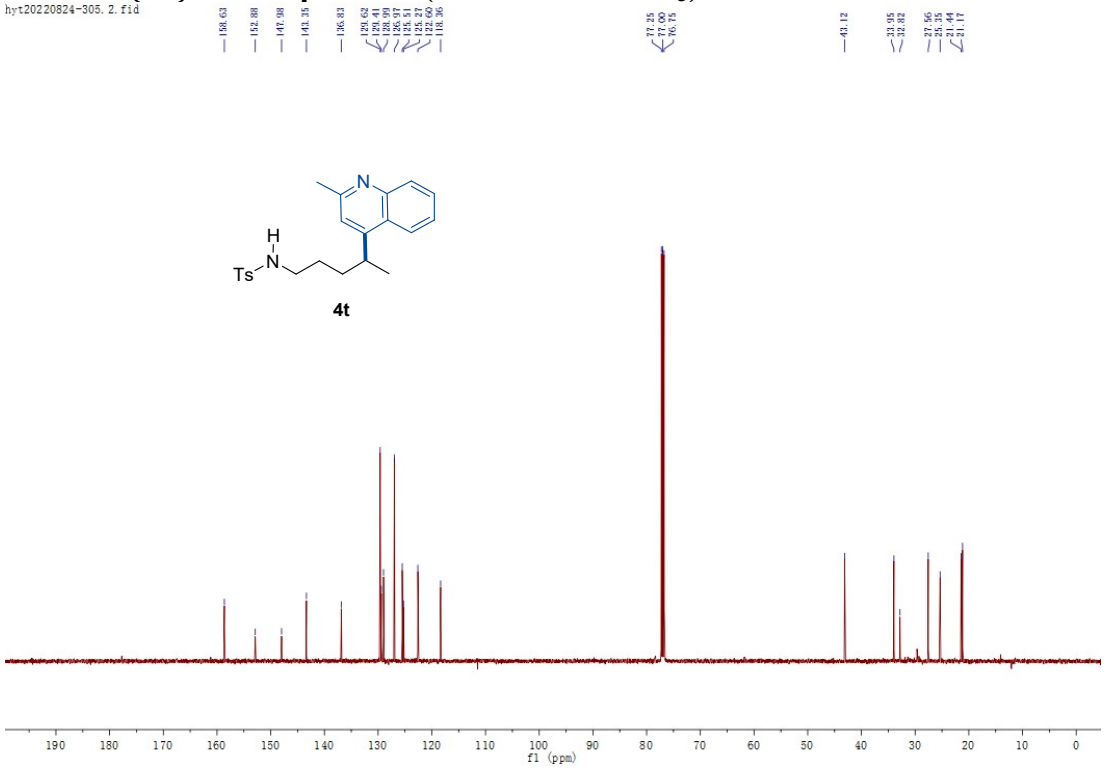


4-Methyl-*N*-(4-(2-methylquinolin-4-yl)pentyl)benzenesulfonamide (4t).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

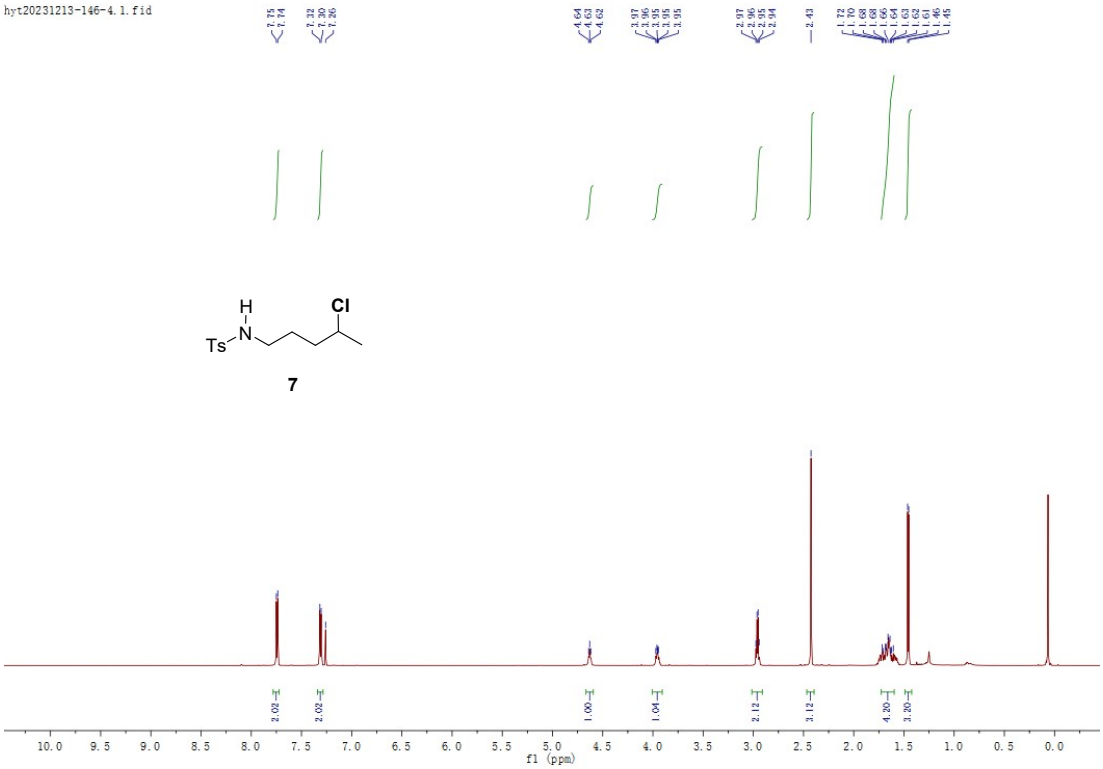


126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)

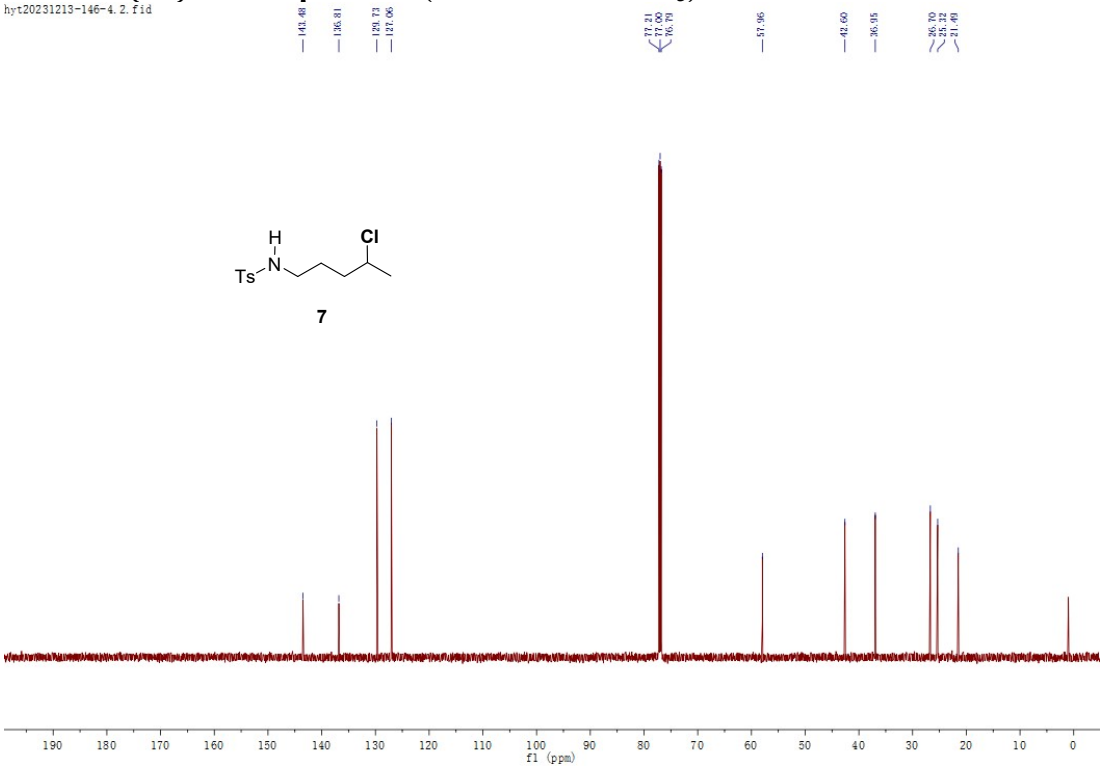


N-(4-chloropentyl)-4-methylbenzenesulfonamide (7).

600 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)

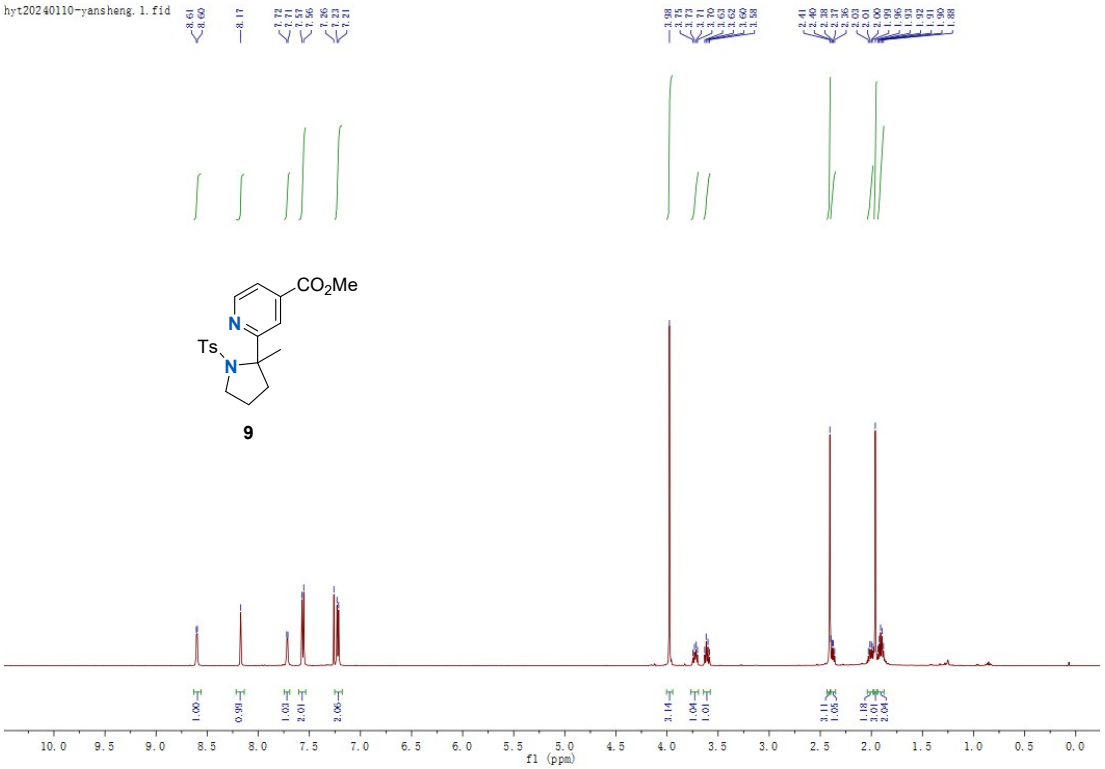


151 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)



Methyl 2-(2-methyl-1-tosylpyrrolidin-2-yl)isonicotinate (9).

500 MHz ^{1}H NMR Spectrum (recorded in $CDCl_3$)



126 MHz ^{13}C{^{1}H} NMR Spectrum (recorded in $CDCl_3$)