

Metal-free C-H Amination of Arene with *N*-Fluorobenzenesulfonimide Catalysed by Nitroxyl Radical at Room Temperature

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Supporting Information

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General Remarks

All commercially available compounds were purchased from TCI, Alfa-Aesar, Acros, J&K Chemicals, and Adamas-beta. TEMPO was purchased from TCI (98.0% purity, CAS No. 2564-83-2). *N*-fluorobenzenesulfonimide (NFSI) (97.0% purity, CAS No. 133745-75-2) and Deuterated water (D₂O) (99.90% purity, CAS No. 7789-20-0) were purchased from Adamas-beta. Ethyl acetate (EtOAc) (99.8%, SafeDry, water < 50 ppm), 1,4-dioxane (99.7%, SafeDry, water < 50 ppm) and acetonitrile (MeCN) (99.9%, SafeDry, water < 50 ppm) were also purchased from Adamas-beta. 1,2-Dichloroethane (DCE) (99.5%, SuperDry) were purchased from J&K chemicals. Unless otherwise noted, materials obtained from commercial suppliers were used without further purification. Oxazole and thiazole substrates **1** were prepared from corresponding nitriles by our previous reported methods.^[1] Furans, thiophenes, flavone and mesitylene were acquired from Adamas-beta and TCI, and were employed directly. Products were purified by flash chromatography on silica gel using petroleum ether and ethyl acetate as the effluents. ¹H-NMR spectra were recorded on Bruker AVANCE III-400 spectrometers. Chemical shifts (in ppm) were referenced with TMS in CDCl₃ or DMSO (0 ppm). ¹³C-NMR spectra were obtained by using the same NMR spectrometers and were calibrated with CDCl₃ ($\delta = 77.00$ ppm) or DMSO ($\delta = 40.00$ ppm). High resolution mass spectra were obtained from an Agilent 6520B Q-TOF mass spectrometer with electron spray ionization (ESI) as the ion source.

References

- [1] For the preparation of various oxazole-4-carboxylic derivative substrates, see (a) Wang, Y.; Li, Z.; Huang, Y.; Tang, C.; Wu, X.; Xu, J.; Yao, H. *Tetrahedron* **2011**, *67*, 7406; (b) Li, Z.; Ma, L.; Xu, J.; Kong, L.; Wu, X.; Yao, H. *Chem. Commun.* **2012**, *48*, 3763; (c) Wang, X.; Lei, B.; Ma, L.; Jiao, H.; Xing, H.; Chen, J.; Li, Z. *Adv. Synth. Catal.* **2017**, *359*, 4284; (d) Lei, B.; Wang, X.; Ma, L.; Jiao, H.; Zhu, L.; Li, Z. *Org. Biomol. Chem.* **2017**, *15*, 6084; (e) Wang, X.; Lei, B.; Ma, L.; Zhu, L.; Zhang, X.; Zuo, H.; Zhuang, D.; Li, Z. *Chem. Asian J.* **2017**, *12*, 2799.

Optimization of the Reaction Conditions**Table S1** Optimization of the reaction conditions^a

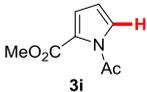
Entry	TEMPO (mol%)	Solvent (2 mL)	Time (h)	Yield (%) ^b
1	40	TCE	12	84
2	20	TCE	12	62
3	20	DCE	12	58
4	20	MeCN	12	trace
5	20	EtOAc	12	52
6	20	TCE	24	81
7	20	MeCN	24	48
8	20	EtOAc	24	88
9	15	EtOAc	24	90
10	10	EtOAc	24	59
11	0	EtOAc	24	0
12^c	15	EtOAc	24	76

^a The reaction was performed with **1a** (0.3 mmol), NFSI (1.2 mmol) and TEMPO in solvent (2 mL) at 25 °C under argon (1 atm) for 12 h or 24 h. ^b Isolated yields of **3a** after column chromatography on silica gel. ^c Loading of NFSI was decline to 0.9 mmol.

Preparation of Pyrrole and Indole Substrates 3i-r

Typical Procedure: To a solution of *N*-unsubstituted pyrrole or indole (5 mmol) in DCE (25 mL) was added DMAP (1 mmol, 0.2 eq) and TEA (10 mmol, 2 eq) at 0 °C. After stirring for 25 minutes, acetic anhydride (Ac₂O, 20 mmol, 4 eq) was added slowly, and then the reaction mixture was stirred at 60 °C overnight. The mixture was then cooled to room temperature, quenched with saturated NH₄Cl (aq.), extracted with EtOAc, washed with water, and dried over Na₂SO₄. After removal of Na₂SO₄ by filtration, the organic phase was concentrated *in vacuo* to give dark residue, which was purified by flash chromatography on silica gel, using petroleum ether and ethyl acetate as the effluent, to afford the corresponding pyrrole and indole substrates **3i-r**.

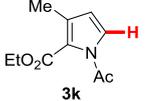
Methyl 1-Acetyl-1*H*-pyrrole-2-carboxylate (**3i**):

 Light yellow oil. ¹H NMR (CDCl₃, 400 MHz): δ = 7.34-7.33 (m, 1H), 6.96-6.95 (m, 1H), 6.23-6.21 (m, 1H), 3.85 (s, 3H), 2.60 (s, 3H) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ = 169.01, 161.39, 126.17, 124.73, 122.57, 110.79, 51.91, 24.60 ppm; HRMS *m/z* (ESI) calcd for [C₈H₉NO₃+Na]⁺ 190.0475, found 190.0475.

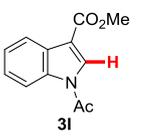
Ethyl 1-Acetyl-1*H*-pyrrole-2-carboxylate (**3j**):

 Light yellow oil. ¹H NMR (CDCl₃, 400 MHz): δ = 7.34-7.33 (m, 1H), 6.96-6.94 (m, 1H), 6.23-6.21 (m, 1H), 4.32 (q, 2H, *J* = 7.1 Hz), 2.60 (s, 3H), 1.35 (t, 3H, *J* = 7.1 Hz) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ = 169.16, 161.13, 126.12, 125.23, 122.44, 115.08, 110.85, 61.02, 24.73, 14.17 ppm; HRMS *m/z* (ESI) calcd for [C₉H₁₁NO₃+Na]⁺ 204.0631, found 204.0635.

Ethyl 1-Acetyl-3-methyl-1*H*-pyrrole-2-carboxylate (**3k**):

 Colorless oil. ¹H NMR (CDCl₃, 400 MHz): δ = 7.14 (d, 1H, *J* = 3.2 Hz), 6.08 (d, 1H, *J* = 3.2 Hz), 4.33 (q, 2H, *J* = 7.1 Hz), 2.51 (s, 3H), 2.22 (s, 3H), 1.35 (t, 3H, *J* = 7.1 Hz) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ = 168.46, 162.32, 132.51, 124.03, 114.33, 61.10, 24.14, 14.33, 12.50 ppm; HRMS *m/z* (ESI) calcd for [C₁₀H₁₃NO₃+Na]⁺ 218.0788, found 218.0791.

Methyl 1-Acetyl-1*H*-indole-3-carboxylate (**3l**):

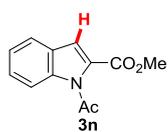
 White solid, m.p. 112-113 °C. ¹H NMR (CDCl₃, 400 MHz): δ = 8.44-8.41 (m, 1H), 8.15-8.11 (m, 2H), 7.41-7.37 (m, 2H), 3.95 (s, 3H), 2.68 (s, 3H) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ = 168.79, 164.40, 135.90, 131.28, 127.28, 125.97, 124.83, 121.54, 116.51, 113.69, 51.68, 23.90 ppm; HRMS *m/z* (ESI) calcd for [C₁₂H₁₁NO₃+Na]⁺ 240.0631, found 240.0632.

Ethyl 1-Acetyl-1H-indole-3-carboxylate (3m):



White solid, m.p. 82-83 °C. ¹H NMR (CDCl₃, 400 MHz): δ = 8.45-8.43 (m, 1H), 8.16-8.12 (m, 2H), 7.43-7.36 (m, 2H), 4.43 (q, 2H, *J* = 7.1 Hz), 2.70 (s, 3H), 1.45 (t, 3H, *J* = 7.1 Hz) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ = 168.87, 164.12, 136.02, 131.25, 127.42, 126.00, 124.86, 121.67, 116.57, 114.17, 60.69, 24.00, 14.55 ppm; HRMS *m/z* (ESI) calcd for [C₁₃H₁₃NO₃+Na]⁺ 254.0788, found 254.0793.

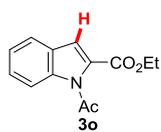
Methyl 1-Acetyl-1H-indole-2-carboxylate (3n):



White solid, m.p. 48-49 °C. ¹H NMR (CDCl₃, 400 MHz): δ = 8.12 (d, 1H, *J* = 8.5 Hz), 7.63 (d, 1H, *J* = 7.9 Hz), 7.47-7.43 (m, 1H), 7.32-7.27 (m, 2H), 3.94 (s, 3H), 2.61 (s, 3H) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ = 171.06, 162.21, 138.52, 129.44, 128.08, 127.20, 123.90, 122.57, 118.47, 115.30, 52.67, 27.27 ppm;

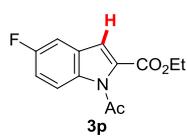
HRMS *m/z* (ESI) calcd for [C₁₂H₁₁NO₃+H]⁺ 218.0812, found 218.0819.

Ethyl 1-Acetyl-1H-indole-2-carboxylate (3o):



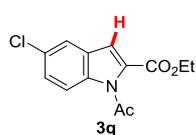
White solid, m.p. 49-50 °C. ¹H NMR (CDCl₃, 400 MHz): δ = 8.11 (d, 1H, *J* = 8.5 Hz), 7.58 (d, 1H, *J* = 7.8 Hz), 7.43-7.39 (m, 1H), 7.28-7.23 (m, 2H), 4.38 (q, 2H, *J* = 7.1 Hz), 2.59 (s, 3H), 1.39 (t, 3H, *J* = 7.1 Hz) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ = 171.10, 161.81, 138.51, 129.86, 127.99, 127.22, 123.86, 122.50, 118.28, 115.32, 61.79, 27.31, 14.34 ppm; HRMS *m/z* (ESI) calcd for [C₁₃H₁₃NO₃+H]⁺ 254.0788, found 254.0793.

Ethyl 1-Acetyl-5-fluoro-1H-indole-2-carboxylate (3p):



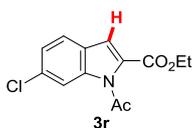
White solid, m.p. 50-51 °C. ¹H NMR (CDCl₃, 400 MHz): δ = 8.13 (dd, 1H, *J* = 9.2 Hz, 4.5 Hz), 7.27-7.25 (m, 2H), 7.17 (td, 1H, *J* = 9.2 Hz, 2.6 Hz), 4.41 (q, 2H, *J* = 7.1 Hz), 2.61 (s, 3H), 1.42 (t, 3H, *J* = 7.1 Hz) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ = 170.89, 161.53, 160.80, 158.40, 134.97, 131.21, 127.94, 127.83, 117.58, 117.54, 116.85, 116.77, 116.28, 116.03, 107.52, 107.29, 62.00, 27.21, 14.32 ppm; HRMS *m/z* (ESI) calcd for [C₁₃H₁₂FNO₃+H]⁺ 272.0693, found 272.0696.

Ethyl 1-Acetyl-5-chloro-1H-indole-2-carboxylate (3q):



White solid, m.p. 68-69 °C. ¹H NMR (CDCl₃, 400 MHz): δ = 8.08 (d, 1H, *J* = 9.0 Hz), 7.58 (d, 1H, *J* = 2.0 Hz), 7.38 (dd, 1H, *J* = 9.0 Hz, 2.0 Hz), 7.23 (s, 1H), 4.41 (q, 2H, *J* = 7.1 Hz), 2.60 (s, 3H), 1.42 (t, 3H, *J* = 7.1 Hz) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ = 170.93, 161.50, 136.84, 130.97, 129.47, 128.27, 128.21, 121.79, 117.07, 116.66, 62.08, 27.27, 14.35 ppm; HRMS *m/z* (ESI) calcd for [C₁₃H₁₂ClNO₃+H]⁺ 288.0398, found 288.0399.

Ethyl 1-Acetyl-6-chloro-1H-indole-2-carboxylate (3r):

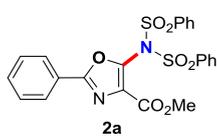


Light yellow solid, m.p. 38-39 °C. ¹H NMR (CDCl₃, 400 MHz): δ = 8.19-8.18 (m, 1H), 7.52 (d, 1H, *J* = 8.4 Hz), 7.28 (s, 1H), 7.25 (dd, 1H, *J* = 8.4 Hz, 1.8 Hz), 4.40 (q, 2H, *J* = 7.1 Hz), 2.60 (s, 3H), 1.41 (t, 3H, *J* = 7.1 Hz) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ = 171.01, 161.45, 138.81, 134.10, 130.30, 125.57, 124.72, 123.20, 117.91, 115.65, 61.96, 27.27, 14.34 ppm; HRMS *m/z* (ESI) calcd for [C₁₃H₁₂ClNO₃+H]⁺ 288.0398, found 288.0398.

Experimental Procedure and Characterization Data

Typical Procedure: To a reaction tube charged with NFSI (378 mg, 1.2 mmol) was added a solution of arene substrate **1** or **3** (0.3 mmol) in solvent (1 mL) under argon (1 atm). The reaction mixture was stirred at 25 °C, and then a solution of TEMPO (7.0 mg, 0.045 mmol, 15 mol%) in solvent (1 mL) was added. After stirring at 25 °C for 24 hours, the mixture was concentrated *in vacuo* to give dark residue, which was purified by flash chromatography on silica gel, using petroleum ether and ethyl acetate as the effluent, to afford the corresponding aminated arene products **2** or **4**.

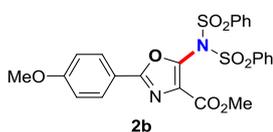
Methyl 2-Phenyl-5-(*N,N*-diphenylsulfonylamino)oxazole-4-carboxylate (**2a**):



The reaction of 0.3 mmol of methyl 2-phenyloxazole-4-carboxylate (**1a**) with 1.2 mmol of NFSI in 2 mL of EtOAc afforded 90% of **2a** after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 3:1, *v/v*) as the effluent. White solid, m.p. 191-193 °C. ¹H NMR (CDCl₃, 400

MHz): δ = 8.09-8.06 (m, 6H), 7.75 (t, 2H, *J* = 7.5 Hz), 7.61 (t, 4H, *J* = 7.5 Hz), 7.56-7.49 (m, 3H), 3.46 (s, 3H) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ = 161.25, 159.74, 140.86, 138.90, 134.66, 132.11, 132.02, 129.16, 129.05, 128.98, 127.12, 125.84, 52.04 ppm; HRMS *m/z* (ESI) calcd for [C₂₃H₁₈N₂O₇S₂+Na]⁺ 521.0448, found 521.0454.

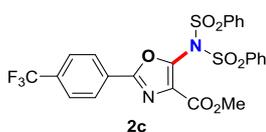
Methyl 2-(4-Methoxyphenyl)-5-(*N,N*-diphenylsulfonylamino)oxazole-4-carboxylate (**2b**):



The reaction of 0.3 mmol of methyl 2-(4-methoxyphenyl)oxazole-4-carboxylate (**1b**) with 1.2 mmol of NFSI in 2 mL of EtOAc afforded 95% of **2b** after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 3:1, *v/v*) as the effluent. White solid, m.p. 182-184

°C. ¹H NMR (CDCl₃, 400 MHz): δ = 8.05-7.97 (m, 6H), 7.72 (t, 2H, *J* = 7.5 Hz), 7.61-7.57 (m, 4H), 6.98 (d, 2H, *J* = 8.9 Hz), 3.86 (s, 3H), 3.42 (s, 3H) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ = 162.53, 161.25, 159.72, 140.11, 138.76, 134.49, 131.88, 128.99, 128.87, 128.79, 118.26, 114.27, 55.36, 51.85 ppm; HRMS *m/z* (ESI) calcd for [C₂₄H₂₀N₂O₈S₂+Na]⁺ 551.0553, found 551.0551.

Methyl 2-(4-Trifluoromethylphenyl)-5-(*N,N*-diphenylsulfonylamino)oxazole-4-carboxylate (**2c**):



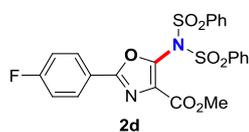
The reaction of 0.3 mmol of methyl 2-(4-trifluoromethylphenyl)oxazole-4-carboxylate (**1c**) with 1.2 mmol of NFSI in 2 mL of EtOAc afforded 51% of **2c** after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 3:1, *v/v*) as the effluent. White solid, m.p. 192-193

°C. ¹H NMR (CDCl₃, 400 MHz): δ = 8.18 (d, 2H, *J* = 8.1 Hz), 8.06-8.03 (m, 4H), 7.77-7.73 (m, 4H), 7.63-7.59 (m, 4H), 3.44 (s, 3H) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ = 159.67, 159.42, 141.53, 138.71, 134.72, 133.46 (q, *J* = 32.7 Hz),

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132.23, 129.14, 129.00, 128.91, 127.37, 125.98 (q, $J = 3.7$ Hz), 123.52 (q, $J = 270.8$ Hz), 52.09 ppm; **HRMS** m/z (ESI) calcd for $[C_{24}H_{17}F_3N_2O_7S_2+Na]^+$ 589.0321, found 589.0325.

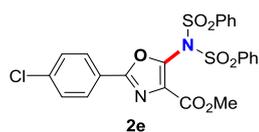
Methyl 2-(4-Fluorophenyl)-5-(*N,N*-diphenylsulfonylamino)oxazole-4-carboxylate (**2d**):



The reaction of 0.3 mmol of methyl 2-(4-fluorophenyl)oxazole-4-carboxylate (**1d**) with 1.2 mmol of NFSI in 2 mL of EtOAc afforded 79% of **2d** after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 3:1, v/v) as the effluent. White solid, m.p. 194-196 °C. **¹H**

NMR (CDCl₃, 400 MHz): $\delta = 8.06$ -8.03 (m, 6H), 7.75-7.71 (m, 2H), 7.62-7.58 (m, 4H), 7.18 (t, 2H, $J = 8.7$ Hz), 3.42 (s, 3H) ppm; **¹³C NMR (CDCl₃, 100 MHz):** $\delta = 164.93$ (d, $J = 252.3$ Hz), 160.29, 159.55, 140.80, 138.72, 134.60, 131.99, 129.34 (d, $J = 9.0$ Hz), 129.06, 128.92, 122.08 (d, $J = 3.1$ Hz), 116.22 (d, $J = 22.1$ Hz), 51.96 ppm; **HRMS** m/z (ESI) calcd for $[C_{23}H_{17}FN_2O_7S_2+Na]^+$ 539.0353, found 539.0356.

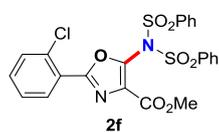
Methyl 2-(4-Chlorophenyl)-5-(*N,N*-diphenylsulfonylamino)oxazole-4-carboxylate (**2e**):



The reaction of 0.3 mmol of methyl 2-(4-chlorophenyl)oxazole-4-carboxylate (**1e**) with 1.2 mmol of NFSI in 2 mL of EtOAc afforded 63% of **2e** after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 3:1, v/v) as the effluent. White solid, m.p. 216-217 °C. **¹H**

NMR (CDCl₃, 400 MHz): $\delta = 8.05$ -8.02 (m, 4H), 7.99 (d, 2H, $J = 8.6$ Hz), 7.73 (t, 2H, $J = 7.5$ Hz), 7.62-7.58 (m, 4H), 7.47 (d, 2H, $J = 8.6$ Hz), 3.43 (s, 3H) ppm; **¹³C NMR (CDCl₃, 100 MHz):** $\delta = 160.24$, 159.52, 140.97, 138.73, 138.30, 134.63, 132.08, 129.30, 129.09, 128.96, 128.29, 124.22, 52.01 ppm; **HRMS** m/z (ESI) calcd for $[C_{23}H_{17}ClN_2O_7S_2+Na]^+$ 555.0058, found 555.0061.

Methyl 2-(2-Chlorophenyl)-5-(*N,N*-diphenylsulfonylamino)oxazole-4-carboxylate (**2f**):

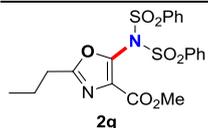


The reaction of 0.3 mmol of methyl 2-(2-chlorophenyl)oxazole-4-carboxylate (**1f**) with 1.2 mmol of NFSI in 2 mL of EtOAc afforded 67% of **2f** after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 3:1, v/v) as the effluent. Light yellow solid, m.p. 137-138 °C. **¹H**

NMR (CDCl₃, 400 MHz): $\delta = 8.11$ (dd, 1H, $J = 7.8$ Hz, 1.5 Hz), 8.07-8.05 (m, 4H), 7.72 (t, 2H, $J = 7.5$ Hz), 7.61-7.57 (m, 4H), 7.52-7.37 (m, 3H), 3.44 (s, 3H) ppm; **¹³C NMR (CDCl₃, 100 MHz):** $\delta = 159.52$, 159.44, 141.20, 138.81, 134.58, 133.00, 132.51, 131.75, 131.22, 129.10, 129.01, 128.93, 126.96, 124.82, 52.02 ppm; **HRMS** m/z (ESI) calcd for $[C_{23}H_{17}ClN_2O_7S_2+Na]^+$ 555.0058, found 555.0060.

Methyl 2-*n*-Propyl-5-(*N,N*-diphenylsulfonylamino)oxazole-4-carboxylate (**2g**):

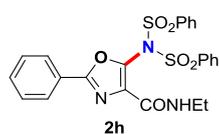
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The reaction of 0.3 mmol of methyl 2-propyloxazole-4-carboxylate (**1g**) with 1.2 mmol of NFSI in 2 mL of EtOAc afforded 66% of **2g** after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 3:1, *v/v*) as the effluent. White solid, m.p. 131-132 °C. **¹H NMR (CDCl₃, 400**

MHz): δ = 8.01-7.99 (m, 4H), 7.72-7.70 (t, 2H, *J* = 7.5 Hz), 7.61-7.57 (m, 4H), 3.40 (s, 3H), 2.80 (t, 2H, *J* = 7.6 Hz), 1.81 (h, 2H, *J* = 7.6 Hz), 1.00 (t, 3H, *J* = 7.6 Hz) ppm; **¹³C NMR (CDCl₃, 150 MHz)**: δ = 165.24, 159.64, 140.70, 138.81, 134.52, 130.69, 129.05, 128.90, 51.86, 30.32, 20.12, 13.45 ppm; **HRMS *m/z* (ESI)** calcd for [C₂₀H₂₀N₂O₇S₂+Na]⁺ 487.0604, found 487.0612.

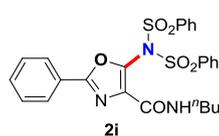
N-Ethyl 2-Phenyl-5-(*N*', *N*'-diphenylsulfonylamino)oxazole-4-formamide (**2h**):



The reaction of 0.3 mmol of *N*-ethyl 2-phenyloxazole-4-formamide (**1h**) with 1.2 mmol of NFSI in 2 mL of EtOAc afforded 77% of **2h** after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 3:1, *v/v*) as the effluent. White solid, m.p. 167-168 °C. **¹H NMR (CDCl₃, 400**

MHz): δ = 8.07-8.05 (m, 4H), 7.96-7.94 (m, 2H), 7.69 (t, 2H, *J* = 7.5 Hz), 7.58-7.44 (m, 7H), 6.96 (t, 1H, *J* = 5.5 Hz), 3.26 (p, 2H, *J* = 7.2 Hz), 1.12 (t, 3H, *J* = 7.2 Hz) ppm; **¹³C NMR (CDCl₃, 100 MHz)**: δ = 160.06, 157.95, 138.55, 137.82, 134.44, 134.26, 131.72, 129.06, 128.91, 128.88, 126.76, 125.92, 33.88, 14.66 ppm; **HRMS *m/z* (ESI)** calcd for [C₂₄H₂₁N₃O₆S₂+Na]⁺ 534.0764, found 534.0766.

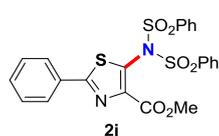
N-*n*-Butyl 2-Phenyl-5-(*N*', *N*'-diphenylsulfonylamino)oxazole-4-formamide (**2i**):



The reaction of 0.3 mmol of *N*-*n*-butyl 2-phenyloxazole-4-formamide (**1i**) with 1.2 mmol of NFSI in 2 mL of EtOAc afforded 81% of **2i** after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 3:1, *v/v*) as the effluent. White solid, m.p. 160-161 °C. **¹H NMR (CDCl₃, 400**

MHz): δ = 8.07-8.05 (m, 4H), 7.96-7.94 (m, 2H), 7.69 (t, 2H, *J* = 7.5 Hz), 7.57-7.44 (m, 7H), 6.97 (t, 1H, *J* = 5.7 Hz), 3.22 (q, 2H, *J* = 7.0 Hz), 1.50-1.43 (m, 2H), 1.36-1.31 (m, 2H), 0.93 (t, 3H, *J* = 7.2 Hz) ppm; **¹³C NMR (CDCl₃, 100 MHz)**: δ = 160.01, 157.99, 138.53, 137.75, 134.39, 134.27, 131.67, 129.02, 128.87, 128.84, 126.73, 125.89, 38.70, 31.40, 19.94, 13.66 ppm; **HRMS *m/z* (ESI)** calcd for [C₂₄H₂₁N₃O₆S₂+H]⁺ 540.1258, found 540.1258.

Methyl 2-Phenyl-5-(*N*, *N*-diphenylsulfonylamino)thiazole-4-carboxylate (**2j**):

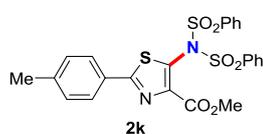


The reaction of 0.3 mmol of methyl 2-phenylthiazole-4-carboxylate (**1j**) with 1.2 mmol of NFSI in 2 mL of EtOAc afforded 65% of **2j** after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 3:1, *v/v*) as the effluent. White solid, m.p. 169-171 °C. **¹H NMR (CDCl₃, 400**

MHz): δ = 8.06-8.00 (m, 6H), 7.74 (t, 2H, *J* = 7.5 Hz), 7.63-7.59 (m, 4H), 7.53-7.47 (m, 3H), 3.44 (s, 3H) ppm; **¹³C NMR (CDCl₃, 100 MHz)**: δ = 168.21, 159.61, 145.71, 138.49, 134.19, 132.11, 131.29, 128.85, 128.80, 128.76, 126.61, 51.66

ppm; **HRMS** m/z (ESI) calcd for $[C_{23}H_{18}N_2O_6S_3+Na]^+$ 537.0219, found 537.0228.

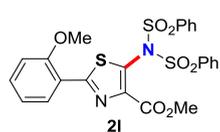
Methyl 2-(4-Methylphenyl)-5-(*N,N*-diphenylsulfonylamino)thiazole-4-carboxylate (2k):



The reaction of 0.3 mmol of methyl 2-(4-methylphenyl)thiazole-4-carboxylate (**1k**) with 1.2 mmol of NFSI in 2 mL of EtOAc afforded 81% of **2k** after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 3:1, v/v) as the effluent. White solid, m.p. 138-139 °C. **¹H**

NMR (CDCl₃, 400 MHz): δ = 8.03-8.01 (m, 4H), 7.86 (d, 2H, J = 8.0 Hz), 7.70 (t, 2H, J = 7.5 Hz), 7.59-7.55 (m, 4H), 7.26 (d, 2H, J = 8.0 Hz), 3.39 (s, 3H), 2.40 (s, 3H) ppm; **¹³C NMR (CDCl₃, 100 MHz):** δ = 168.63, 159.87, 145.81, 142.07, 138.75, 134.35, 133.94, 129.72, 129.68, 129.02, 128.96, 126.74, 51.83, 21.46 ppm; **HRMS** m/z (ESI) calcd for $[C_{24}H_{20}N_2O_6S_3+Na]^+$ 551.0376, found 551.0375.

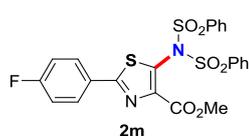
Methyl 2-(2-Methoxyphenyl)-5-(*N,N*-diphenylsulfonylamino)thiazole-4-carboxylate (2l):



The reaction of 0.3 mmol of methyl 2-(2-methoxyphenyl)thiazole-4-carboxylate (**1l**) with 1.2 mmol of NFSI in 2 mL of EtOAc afforded 91% of **2l** after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 3:1, v/v) as the effluent. White solid, m.p. 179-180 °C. **¹H**

NMR (CDCl₃, 400 MHz): δ = 8.51 (dd, 1H, J = 7.9 Hz, 1.5 Hz), 8.02-8.00 (m, 4H), 7.69 (t, 2H, J = 7.5 Hz), 7.58-7.54 (m, 4H), 7.46-7.42 (m, 1H), 7.12-7.08 (m, 1H), 7.02 (d, 1H, J = 8.3 Hz), 3.99 (s, 3H), 3.38 (s, 3H) ppm; **¹³C NMR (CDCl₃, 100 MHz):** δ = 162.07, 160.28, 156.63, 143.84, 138.88, 134.48, 134.17, 132.17, 128.95, 128.90, 128.34, 121.06, 121.02, 111.05, 55.55, 51.65 ppm; **HRMS** m/z (ESI) calcd for $[C_{24}H_{20}N_2O_7S_3+H]^+$ 545.0505, found 545.0504.

Methyl 2-(4-Fluorophenyl)-5-(*N,N*-diphenylsulfonylamino)thiazole-4-carboxylate (2m):



The reaction of 0.3 mmol of methyl 2-(4-fluorophenyl)thiazole-4-carboxylate (**1m**) with 1.2 mmol of NFSI in 2 mL of EtOAc afforded 62% of **2m** after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 3:1, v/v) as the effluent. White solid, m.p. 152-154 °C. **¹H**

NMR (CDCl₃, 400 MHz): δ = 8.03-7.96 (m, 6H), 7.73-7.69 (m, 2H), 7.61-7.56 (m, 4H), 7.18-7.13 (m, 2H), 3.39 (s, 3H) ppm; **¹³C NMR (CDCl₃, 100 MHz):** δ = 167.13, 164.68 (d, J = 251.5 Hz), 159.77, 145.97, 138.70, 134.50, 134.43, 129.08, 128.98, 128.92, 128.72 (d, J = 3.1 Hz), 116.24 (d, J = 22.0 Hz), 51.90 ppm; **HRMS** m/z (ESI) calcd for $[C_{23}H_{17}FN_2O_6S_3+Na]^+$ 555.0125, found 555.0127.

***N*-Ethyl 2-Phenyl-5-(*N,N'*-diphenylsulfonylamino)thiazole-4-formamide (2n):**



The reaction of 0.3 mmol of *N*-ethyl 2-phenylthiazole-4-formamide (**1n**) with 1.2 mmol of NFSI in 2

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mL of EtOAc afforded 61% of **2n** after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 3:1, v/v) as the effluent. Light yellow solid, m.p. 207-208 °C. ¹H NMR (CDCl₃, 400 MHz): δ = 8.05-8.03 (m, 4H), 7.92-7.90 (m, 2H), 7.67 (t, 2H, *J* = 7.5 Hz), 7.56-7.52 (m, 4H), 7.48-7.46 (m, 3H), 7.19 (t, 1H, *J* = 5.4 Hz), 3.17 (p, 2H, *J* = 7.2 Hz), 1.07 (t, 3H, *J* = 7.2 Hz) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ = 167.46, 158.36, 148.07, 138.60, 134.20, 132.41, 131.37, 131.02, 129.13, 129.06, 128.86, 126.57, 33.88, 14.71 ppm; HRMS *m/z* (ESI) calcd for [C₂₄H₂₁N₃O₅S₃+Na]⁺ 550.0536, found 550.0546.

N-(Benzofuran-2-yl)-*N*-(phenylsulfonyl)benzenesulfonamide (**4a**):



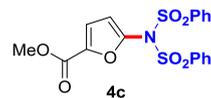
The reaction of 0.3 mmol of 2,3-benzofuran (**3a**) with 0.6 mmol of NFSI in 2 mL of DCE afforded 91% of **4a** after flash chromatography on silica gel using petroleum ether and ethyl acetate (10:1, v/v) as the effluent. Colorless solid, m.p. 144-145 °C. ¹H NMR (CDCl₃, 400 MHz): δ = 8.06-8.03 (m, 4H), 7.72-7.68 (m, 2H), 7.60-7.56 (m, 5H), 7.46 (dd, 1H, *J* = 8.3 Hz, 0.6 Hz), 7.37 (td, 1H, *J* = 8.3 Hz, 1.2 Hz), 7.29-7.24 (m, 1H), 6.59 (d, 1H, *J* = 0.8 Hz) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ = 153.67, 141.65, 138.90, 134.39, 129.13, 128.68, 127.18, 126.27, 123.43, 121.89, 111.77, 109.02 ppm; HRMS *m/z* (ESI) calcd for [C₂₀H₁₅NO₅S₂+Na]⁺ 436.0284, found 436.0290.

N-(5-Acetylfuran-2-yl)-*N*-(phenylsulfonyl)benzenesulfonamide (**4b**):



The reaction of 0.3 mmol of 1-(furan-2-yl)ethan-1-one (**3b**) with 1.2 mmol of NFSI in 2 mL of MeCN afforded 90% of **4b** after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 3:1, v/v) as the effluent. Colorless solid, m.p. 156-157 °C. ¹H NMR (CDCl₃, 400 MHz): δ = 8.00-7.98 (m, 4H), 7.71 (t, 2H, *J* = 7.5 Hz), 7.60-7.56 (m, 4H), 7.15 (d, 1H, *J* = 3.6 Hz), 6.37 (d, 1H, *J* = 3.6 Hz), 2.37 (s, 3H) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ = 186.47, 151.75, 141.73, 138.58, 134.58, 129.17, 128.59, 117.05, 114.10, 25.88 ppm; HRMS *m/z* (ESI) calcd for [C₁₈H₁₅NO₆S₂+Na]⁺ 428.0233, found 428.0239.

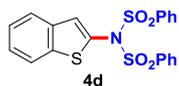
Methyl 5-(*N*-(phenylsulfonyl)phenylsulfonamido)furan-2-carboxylate (**4c**):



The reaction of 0.3 mmol of methyl furan-2-carboxylate (**3c**) with 0.6 mmol of NFSI in 2 mL of MeCN afforded 76% of **4c** after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 3:1, v/v) as the effluent. Colorless solid, m.p. 176-177 °C. ¹H NMR (CDCl₃, 400 MHz): δ = 8.01-7.99 (m, 4H), 7.72-7.68 (m, 2H), 7.60-7.56 (m, 4H), 7.17 (d, 1H, *J* = 3.5 Hz), 6.31 (d, 1H, *J* = 3.5 Hz), 3.88 (s, 3H) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ = 158.18, 144.11, 142.00, 138.60, 134.52, 129.17, 128.68, 118.75, 113.66, 52.14 ppm; HRMS *m/z* (ESI) calcd for [C₁₈H₁₅NO₇S₂+Na]⁺ 444.0182, found 444.0187.

N-(Benzo[*b*]thiophen-2-yl)-*N*-(phenylsulfonyl)benzenesulfonamide (**4d**):

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The reaction of 0.3 mmol of benzo[b]thiophene (**3d**) with 0.6 mmol of NFSI in 2 mL of EtOAc afforded 62% of **4d** after flash chromatography on silica gel using petroleum ether and ethyl acetate (10:1, v/v) as the effluent. Colorless solid, m.p. 111-112 °C. ¹H NMR (CDCl₃, 400 MHz): δ = 8.04-8.02 (m, 4H), 7.75-7.68 (m, 4H), 7.59-7.55 (m, 4H), 7.41-7.34 (m, 2H), 7.02 (s, 1H) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ = 140.27, 138.72, 136.72, 134.32, 134.17, 129.10, 128.79, 128.73, 126.09, 124.76, 124.75, 122.38 ppm; HRMS *m/z* (ESI) calcd for [C₂₀H₁₅NO₄S₃+Na]⁺ 452.0055, found 452.0063.

N-(Phenylsulfonyl)-*N*-(5-phenylthiophen-2-yl)benzenesulfonamide (**4e**):



The reaction of 0.3 mmol of 2-phenylthiophene (**3e**) with 0.6 mmol of NFSI in 2 mL of 1,4-dioxane afforded 87% of **4e** after flash chromatography on silica gel using petroleum ether and ethyl acetate (10:1, v/v) as the effluent. Colorless solid, m.p. 108-109 °C. ¹H NMR (CDCl₃, 400 MHz): δ = 8.04-8.02 (m, 4H), 7.69 (t, 2H, *J* = 7.5 Hz), 7.59-7.53 (m, 6H), 7.39-7.30 (m, 3H), 7.12 (d, 1H, *J* = 3.9 Hz), 6.70 (d, 1H, *J* = 3.9 Hz) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ = 147.64, 138.76, 134.23, 133.49, 132.59, 132.45, 129.07, 128.98, 128.75, 128.46, 125.93, 121.36 ppm; HRMS *m/z* (ESI) calcd for [C₂₂H₁₇NO₄S₃+Na]⁺ 478.0212, found 478.0220.

N-(5-Methylthiophen-2-yl)-*N*-(phenylsulfonyl)benzenesulfonamide (**4f**):



The reaction of 0.3 mmol of 2-methylthiophene (**3f**) with 0.6 mmol of NFSI in 2 mL of EtOAc afforded 82% of **4f** after flash chromatography on silica gel using petroleum ether and ethyl acetate (10:1, v/v) as the effluent. Colorless solid, m.p. 111-112 °C. ¹H NMR (CDCl₃, 400 MHz): δ = 8.00-7.98 (m, 4H), 7.69-7.65 (m, 2H), 7.56-7.53 (m, 4H), 6.58 (dd, 1H, *J* = 3.7 Hz, 1.0 Hz), 6.51 (d, 1H, *J* = 3.7 Hz), 2.45 (d, 3H, *J* = 0.7 Hz) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ = 143.78, 138.79, 134.10, 131.67, 130.66, 128.96, 128.65, 123.90, 15.99 ppm; HRMS *m/z* (ESI) calcd for [C₁₇H₁₅NO₄S₃+Na]⁺ 416.0055, found 416.0057.

N-(Phenylsulfonyl)-*N*-(5-(trimethylsilyl)thiophen-2-yl)benzenesulfonamide (**4g**):



The reaction of 0.3 mmol of trimethyl(thiophen-2-yl)silane (**3g**) with 1.2 mmol of NFSI in 2 mL of 1,4-dioxane afforded 72% of **4g** after flash chromatography on silica gel using petroleum ether and ethyl acetate (10:1, v/v) as the effluent. Colorless solid, m.p. 103-104 °C. ¹H NMR (CDCl₃, 400 MHz): δ = 7.99-7.97 (m, 4H), 7.68 (t, 2H, *J* = 7.5 Hz), 7.57-7.53 (m, 4H), 7.04 (d, 1H, *J* = 3.6 Hz), 6.75 (d, 1H, *J* = 3.6 Hz), 0.30 (s, 9H) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ = 145.63, 138.79, 138.16, 134.12, 132.10, 128.97, 128.72, -0.33 ppm; HRMS *m/z* (ESI) calcd for [C₁₉H₂₁NO₄S₃Si+Na]⁺ 474.0294, found 474.0302.

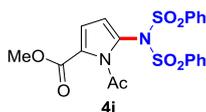
N-(Phenylsulfonyl)-*N*-(5-(trimethylsilyl)thiophen-2-yl)benzenesulfonamide (**4h**):

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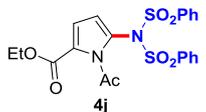
The reaction of 0.3 mmol of 1-(thiophen-2-yl)ethan-1-one (**3h**) with 1.2 mmol of NFSI in 2 mL of EtOAc afforded 57% of **4h** after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 3:1, *v/v*) as the effluent. Colorless solid, m.p. 155-156 °C. ¹H NMR (CDCl₃, 400 MHz): δ = 7.99-7.97 (m, 4H), 7.71 (t, 2H, *J* = 7.5 Hz), 7.60-7.56 (m, 4H), 7.51 (d, 1H, *J* = 4.0 Hz), 6.77 (d, 1H, *J* = 4.0 Hz), 2.53 (s, 3H) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ = 190.27, 146.14, 141.02, 138.34, 134.53, 132.38, 130.26, 129.22, 128.71, 26.39 ppm; HRMS *m/z* (ESI) calcd for [C₁₈H₁₅NO₅S₃+Na]⁺ 444.0005, found 444.0013.

Methyl 1-Acetyl-5-(*N*-(phenylsulfonyl)phenylsulfonamido)-1*H*-pyrrole-2-carboxylate (**4i**):



The reaction of 0.3 mmol of methyl 1-acetyl-1*H*-pyrrole-2-carboxylate (**3i**) with 0.6 mmol of NFSI in 2 mL of EtOAc afforded 78% of **4i** after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 3:1, *v/v*) as the effluent. White solid, m.p. 109-110 °C. ¹H NMR (CDCl₃, 400 MHz): δ = 8.01-7.99 (m, 4H), 7.70 (t, 2H, *J* = 7.5 Hz), 7.58-7.54 (m, 4H), 6.88 (d, 1H, *J* = 3.9 Hz), 5.88 (d, 1H, *J* = 3.9 Hz), 3.86 (s, 3H), 2.48 (s, 3H) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ = 170.62, 160.35, 137.71, 134.47, 129.56, 128.71, 125.17, 124.78, 117.75, 114.28, 52.17, 28.14 ppm; HRMS *m/z* (ESI) calcd for [C₂₀H₁₈N₂O₇S₂+Na]⁺ 485.0448, found 485.0445.

Ethyl 1-Acetyl-5-(*N*-(phenylsulfonyl)phenylsulfonamido)-1*H*-pyrrole-2-carboxylate (**4j**):



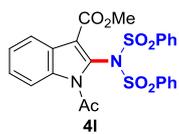
The reaction of 0.3 mmol of ethyl 1-acetyl-1*H*-pyrrole-2-carboxylate (**3j**) with 0.6 mmol of NFSI in 2 mL of EtOAc afforded 82% of **4j** after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1, *v/v*) as the effluent. Colorless solid, m.p. 96-97 °C. ¹H NMR (CDCl₃, 400 MHz): δ = 8.01-7.99 (m, 4H), 7.70 (t, 2H, *J* = 7.5 Hz), 7.57-7.53 (m, 4H), 6.88 (d, 1H, *J* = 3.9 Hz), 5.86 (d, 1H, *J* = 3.9 Hz), 4.32 (q, 2H, *J* = 7.1 Hz), 2.48 (s, 3H), 1.35 (t, 3H, *J* = 7.1 Hz) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ = 170.69, 159.95, 137.72, 135.80, 134.46, 129.59, 128.70, 125.18, 124.98, 117.55, 114.24, 61.32, 28.22, 14.17 ppm; HRMS *m/z* (ESI) calcd for [C₂₁H₂₀N₂O₇S₂+Na]⁺ 499.0604, found 499.0609.

Ethyl 1-Acetyl-3-methyl-5-(*N*-(phenylsulfonyl)phenylsulfonamido)-1*H*-pyrrole-2-carboxylate (**4k**):



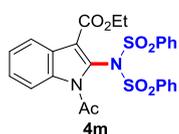
The reaction of 0.3 mmol of ethyl 1-acetyl-3-methyl-1*H*-pyrrole-2-carboxylate (**3k**) with 0.6 mmol of NFSI in 2 mL of EtOAc afforded 90% of **4k** after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1, *v/v*) as the effluent. Colorless solid, m.p. 131-132 °C. ¹H NMR (CDCl₃, 400 MHz): δ = 8.01-7.99 (m, 4H), 7.69 (t, 2H, *J* = 7.5 Hz), 7.57-7.53 (m, 4H), 5.73 (s, 1H), 4.32 (q, 2H, *J* = 7.2 Hz), 2.39 (s, 3H), 2.26 (s, 3H), 1.35 (t, 3H, *J* = 7.2 Hz) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ = 170.47, 160.73, 137.81, 134.41, 129.72, 129.52, 128.66, 123.57, 122.29, 116.71, 61.09, 28.07, 14.16, 13.00 ppm; HRMS *m/z* (ESI) calcd for [C₂₂H₂₂N₂O₇S₂+Na]⁺ 513.0761, found 513.0757.

Methyl 1-Acetyl-2-(*N*-(phenylsulfonyl)phenylsulfonamido)-1*H*-indole-3-carboxylate (4l):



The reaction of 0.3 mmol of methyl 1-acetyl-1*H*-indole-3-carboxylate (**3l**) with 0.6 mmol of NFSI in 2 mL of EtOAc afforded 87% of **4l** after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 3:1, v/v) as the effluent. Light yellow oil. ¹H NMR (CDCl₃, 400 MHz): δ = 8.31 (d, 1H, *J* = 7.9 Hz), 8.06-8.04 (m, 4H), 7.77 (d, 1H, *J* = 8.4 Hz), 7.68 (t, 2H, *J* = 7.5 Hz), 7.56-7.52 (m, 4H), 7.46-7.36 (m, 2H), 3.19 (s, 3H), 2.46 (s, 3H) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ = 169.46, 162.33, 138.92, 134.25, 134.04, 129.92, 128.76, 128.60, 126.69, 125.47, 124.17, 122.82, 114.28, 113.27, 50.64, 26.66 ppm; HRMS *m/z* (ESI) calcd for [C₂₄H₂₀N₂O₇S₂+Na]⁺ 535.0604, found 535.0604.

Ethyl 1-Acetyl-2-(*N*-(phenylsulfonyl)phenylsulfonamido)-1*H*-indole-3-carboxylate (4m):



The reaction of 0.3 mmol of ethyl 1-acetyl-1*H*-indole-3-carboxylate (**3m**) with 0.6 mmol of NFSI in 2 mL of EtOAc afforded 95% of **4m** after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 3:1, v/v) as the effluent. Colorless oil. ¹H NMR (CDCl₃, 400 MHz): δ = 8.30 (d, 1H, *J* = 7.9 Hz), 8.06-8.04 (m, 4H), 7.76 (d, 1H, *J* = 8.4 Hz), 7.68 (t, 2H, *J* = 7.5 Hz), 7.56-7.53 (m, 4H), 7.46-7.36 (m, 2H), 3.73 (q, 2H, *J* = 7.1 Hz), 2.46 (s, 3H), 1.09 (t, 3H, *J* = 7.1 Hz) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ = 169.46, 162.02, 139.03, 134.21, 134.03, 129.90, 128.75, 128.60, 126.63, 125.53, 124.10, 123.00, 114.26, 113.72, 60.48, 26.71, 13.88 ppm; HRMS *m/z* (ESI) calcd for [C₂₅H₂₂N₂O₇S₂+Na]⁺ 549.0761, found 549.0756.

Methyl 1-Acetyl-3-(*N*-(phenylsulfonyl)phenylsulfonamido)-1*H*-indole-2-carboxylate (4n):



The reaction of 0.3 mmol of methyl 1-acetyl-1*H*-indole-2-carboxylate (**3n**) with 1.2 mmol of NFSI in 2 mL of EtOAc afforded 71% of **4n** after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 3:1, v/v) as the effluent. Light yellow solid, m.p. 158-159 °C. ¹H NMR (CDCl₃, 400 MHz): δ = 8.05-7.98 (m, 5H), 7.66 (t, 2H, *J* = 7.5 Hz), 7.53-7.49 (m, 4H), 7.40 (t, 1H, *J* = 7.5 Hz), 7.10 (t, 1H, *J* = 7.5 Hz), 6.99 (d, 1H, *J* = 8.0 Hz), 3.44 (s, 3H), 2.59 (s, 3H) ppm; ¹³C NMR (CDCl₃, 100 MHz): δ = 170.04, 160.08, 139.21, 135.41, 134.12, 129.16, 129.11, 128.76, 128.16, 125.89, 124.09, 120.75, 120.57, 114.76, 52.39, 27.01 ppm; HRMS *m/z* (ESI) calcd for [C₂₄H₂₀N₂O₇S₂+Na]⁺ 535.0604, found 535.0604.

Ethyl 1-Acetyl-3-(*N*-(phenylsulfonyl)phenylsulfonamido)-1*H*-indole-2-carboxylate (4o):

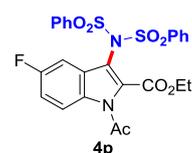


The reaction of 0.3 mmol of ethyl 1-acetyl-1*H*-indole-2-carboxylate (**3o**) with 1.2 mmol of NFSI in 2 mL of EtOAc afforded 83% of **4o** after flash chromatography on silica gel using petroleum ether and ethyl acetate

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(6:1 to 3:1, v/v) as the effluent. Light yellow solid, m.p. 145-146 °C. $^1\text{H NMR}$ (CDCl_3 , 400 MHz): δ = 8.02-7.98 (m, 5H), 7.65 (t, 2H, J = 7.5 Hz), 7.51-7.47 (m, 4H), 7.39-7.35 (m, 1H), 7.07 (t, 1H, J = 7.5 Hz), 6.92 (d, 1H, J = 8.0 Hz), 3.96 (q, 2H, J = 7.2 Hz), 2.59 (s, 3H), 1.08 (t, 3H, J = 7.2 Hz) ppm; $^{13}\text{C NMR}$ (CDCl_3 , 100 MHz): δ = 170.04, 159.86, 139.25, 135.23, 134.09, 129.65, 129.14, 128.73, 127.95, 125.88, 123.95, 120.73, 120.07, 114.68, 62.26, 27.00, 13.67 ppm; **HRMS** m/z (ESI) calcd for $[\text{C}_{25}\text{H}_{22}\text{N}_2\text{O}_7\text{S}_2+\text{Na}]^+$ 549.0761, found 549.0773.

Ethyl 1-Acetyl-5-fluoro-3-(*N*-(phenylsulfonyl)phenylsulfonamido)-1*H*-indole-2-carboxylate (**4p**):



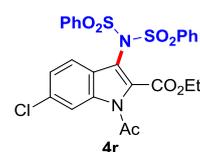
The reaction of 0.3 mmol of ethyl 1-acetyl-5-fluoro-1*H*-indole-2-carboxylate (**3p**) with 0.6 mmol of NFSI in 2 mL of EtOAc afforded 62% of **4p** after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1, v/v) as the effluent. Light yellow solid, m.p. 142-143 °C. $^1\text{H NMR}$ (CDCl_3 , 400 MHz): δ = 8.05 (dd, 1H, J = 9.0 Hz, 4.2 Hz), 8.01-7.98 (m, 4H), 7.68 (t, 2H, J = 7.5 Hz), 7.55-7.51 (m, 4H), 7.11 (td, 1H, J = 9.0 Hz, 2.5 Hz), 6.52 (dd, 1H, J = 8.3 Hz, 2.5 Hz), 3.98 (q, 2H, J = 7.2 Hz), 2.58 (s, 3H), 1.09 (t, 3H, J = 7.2 Hz) ppm; $^{13}\text{C NMR}$ (CDCl_3 , 100 MHz): δ = 169.89, 159.66, 159.59 (d, J = 241.8 Hz), 139.17, 134.30, 131.36 (d, J = 68.4 Hz), 129.19, 128.85, 126.96 (d, J = 9.9 Hz), 119.68 (d, J = 4.7 Hz), 116.54 (d, J = 25.5 Hz), 116.53 (d, J = 8.8 Hz), 105.81 (d, J = 24.9 Hz), 62.54, 26.92, 13.72 ppm; **HRMS** m/z (ESI) calcd for $[\text{C}_{25}\text{H}_{21}\text{FN}_2\text{O}_7\text{S}_2+\text{Na}]^+$ 567.0666, found 567.0662.

Ethyl 1-Acetyl-5-chloro-3-(*N*-(phenylsulfonyl)phenylsulfonamido)-1*H*-indole-2-carboxylate (**4q**):



The reaction of 0.3 mmol of ethyl 1-acetyl-5-chloro-1*H*-indole-2-carboxylate (**3q**) with 0.6 mmol of NFSI in 2 mL of EtOAc afforded 63% of **4q** after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1, v/v) as the effluent. Light yellow solid, m.p. 143-144 °C. $^1\text{H NMR}$ (CDCl_3 , 400 MHz): δ = 8.00-7.98 (m, 5H), 7.69 (t, 2H, J = 7.5 Hz), 7.55-7.51 (m, 4H), 7.31 (dd, 1H, J = 9.0 Hz, 2.1 Hz), 6.74 (d, 1H, J = 1.7 Hz), 3.99 (q, 2H, J = 7.2 Hz), 2.58 (s, 3H), 1.09 (t, 3H, J = 7.2 Hz) ppm; $^{13}\text{C NMR}$ (CDCl_3 , 100 MHz): δ = 169.84, 159.60, 139.06, 135.79, 134.36, 133.59, 130.76, 129.98, 129.21, 128.83, 128.33, 127.00, 120.03, 119.20, 116.21, 62.57, 26.92, 13.71 ppm; **HRMS** m/z (ESI) calcd for $[\text{C}_{25}\text{H}_{21}\text{ClN}_2\text{O}_7\text{S}_2+\text{Na}]^+$ 583.0371, found 583.0364.

Ethyl 1-Acetyl-6-chloro-3-(*N*-(phenylsulfonyl)phenylsulfonamido)-1*H*-indole-2-carboxylate (**4r**):

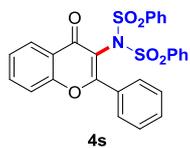


The reaction of 0.3 mmol of ethyl 1-acetyl-6-chloro-1*H*-indole-2-carboxylate (**3r**) with 1.2 mmol of NFSI in 2 mL of EtOAc afforded 76% of **4r** after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1, v/v) as the effluent. White solid, m.p. 167-168 °C. $^1\text{H NMR}$ (CDCl_3 , 400 MHz): δ = 8.13 (d, 1H, J = 1.6 Hz), 8.00-7.98 (m, 4H), 7.68 (t, 2H, J = 7.5 Hz), 7.54-7.50 (m, 4H), 7.06 (dd, 1H, J = 8.5 Hz, 1.6 Hz), 6.82 (d, 1H, J = 8.5 Hz), 3.96 (q, 2H, J = 7.2 Hz), 2.57 (s, 3H), 1.08 (t, 3H, J = 7.2 Hz) ppm; $^{13}\text{C NMR}$ (CDCl_3 , 100 MHz): δ = 169.97, 159.63, 139.18, 135.80, 135.60, 134.25, 129.99, 129.17, 128.87, 124.97, 124.40, 121.62, 120.05,

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115.15, 62.52, 27.01, 13.71 ppm; **HRMS** m/z (ESI) calcd for $[C_{25}H_{21}ClN_2O_7S_2+Na]^+$ 583.0371, found 583.0370.

N-(4-Oxo-2-phenyl-4*H*-chromen-3-yl)-*N*-(phenylsulfonyl)benzenesulfonamide (**4s**):



The reaction of 0.3 mmol of 2-phenyl-4*H*-chromen-4-one (**3s**) with 0.6 mmol of NFSI in 2 mL of DCE afforded 58% of **4s** after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 3:1, v/v) as the effluent. Colorless solid, m.p. 205-206 °C. **¹H NMR** ($CDCl_3$, 400 MHz): δ = 8.14 (dd, 1H, J = 7.9 Hz, 1.3 Hz), 7.91-7.89 (m, 2H), 7.84-7.81 (m, 4H), 7.74-7.70 (m, 1H), 7.55-7.51 (m, 3H), 7.43 (t, 2H, J = 7.5

Hz), 7.37-7.32 (m, 6H) ppm; **¹³C NMR** ($CDCl_3$, 100 MHz): δ = 174.53, 168.24, 155.54, 139.18, 134.41, 133.74, 131.37, 130.85, 129.58, 129.02, 128.33, 128.23, 126.37, 125.81, 123.47, 119.25, 118.05 ppm; **HRMS** m/z (ESI) calcd for $[C_{27}H_{19}NO_6S_2+Na]^+$ 540.0546, found 540.0548.

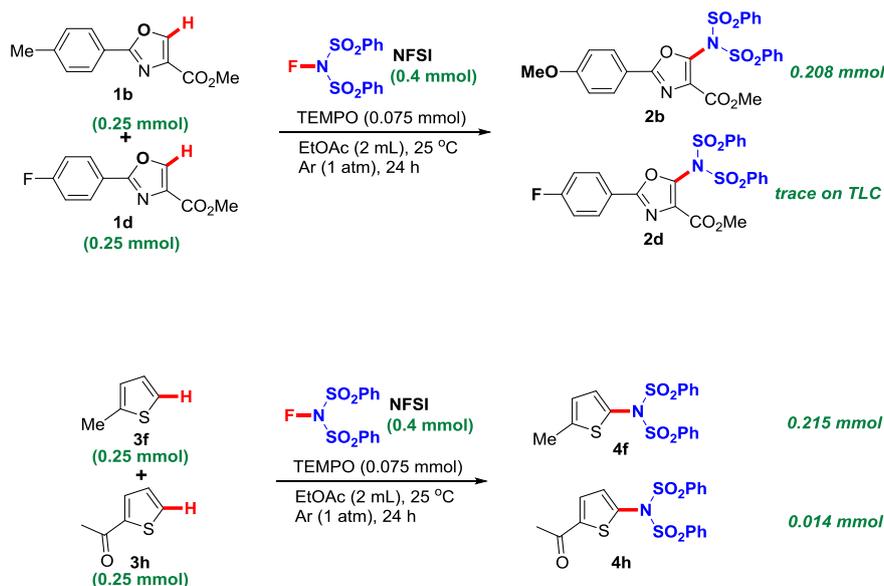
N-Mesityl-*N*-(phenylsulfonyl)benzenesulfonamide (**4t**):



The reaction of 0.3 mmol of mesitylene (**3t**) with 0.6 mmol of NFSI in 2 mL of EtOAc afforded 41% of **4t** after flash chromatography on silica gel using petroleum ether and ethyl acetate (10:1, v/v) as the

effluent. White solid, m.p. 165-166 °C. **¹H NMR** ($CDCl_3$, 400 MHz): δ = 8.05-8.03 (m, 4H), 7.68-7.65 (m, 2H), 7.56-7.52 (m, 4H), 6.87 (s, 2H), 2.28 (s, 3H), 1.81 (s, 6H) ppm; **¹³C NMR** ($CDCl_3$, 100 MHz): δ = 140.38, 139.81, 139.74, 133.98, 130.00, 129.29, 128.84, 128.11, 126.62, 21.04, 19.04 ppm; **HRMS** m/z (ESI) calcd for $[C_{21}H_{21}NO_4S_2+Na]^+$ 438.0804, found 438.0808.

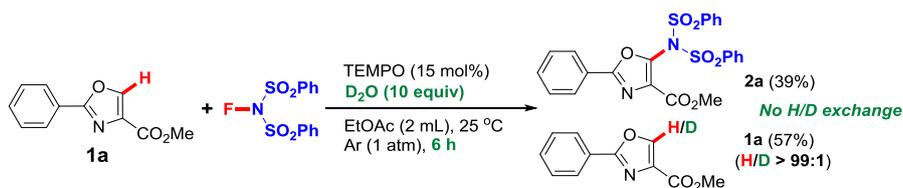
Competition Experiments between Electron-rich/deficient Arenes



Scheme S1. Competitive experiments

Typical Procedure: To a reaction tube charged with NFSI (126 mg, 0.4 mmol) was added a solution of oxazole **1b** (0.25 mmol) and **1d** (0.25 mmol) in EtOAc (1 mL) under argon (1 atm). The reaction mixture was stirred at 25 °C, and then a solution of TEMPO (11.7 mg, 0.075 mmol, 15 mol%) in EtOAc (1 mL) was added. After stirring at 25 °C for 24 hours, the electron-rich product **2b** was generated predominantly, while only trace **2d** was observed on TLC. Then the mixture was concentrated *in vacuo* to give dark residue, which was purified by flash chromatography using petroleum ether and ethyl acetate as the effluent (6:1 to 3:1, *v/v*) on silica gel to afford 109.7 mg of aminated oxazole **2b** (0.208 mmol, 83.2%), and its structure was proved by ¹H NMR, ¹³C NMR and HRMS.

To a reaction tube charged with NFSI (126 mg, 0.4 mmol) was added a solution of thiophene **3f** (0.25 mmol) and **3h** (0.25 mmol) in EtOAc (1 mL) under argon (1 atm). The reaction mixture was stirred at 25 °C, and then a solution of TEMPO (11.7 mg, 0.075 mmol, 15 mol%) in EtOAc (1 mL) was added. After stirring at 25 °C for 24 hours, the electron-rich product **4f** was generated predominantly, while only trace **4h** was observed on TLC. Then the mixture was concentrated *in vacuo* to give dark residue, which was purified by flash chromatography using petroleum ether and ethyl acetate as the effluent (15:1 to 6:1 to 3:1, *v/v*) on silica gel to afford 84.4 mg of aminated thiophene **4f** (0.215 mmol, 86.0%) and 5.9 mg of aminated thiophene **4h** (0.014 mmol, 5.6%). Their structures were proved by ¹H NMR, ¹³C NMR and HRMS.

Deuterium Exchange Experiment**Scheme S2.** Deuterium exchange experiment on oxazole **1a**

Typical Procedure: To a reaction tube charged with NFSI (378 mg, 1.2 mmol) was added a solution of oxazole **1a** (0.3 mmol) and deuterated water D₂O (60 mg, 3 mmol) in EtOAc (1 mL) under argon (1 atm). The reaction mixture was stirred at 25 °C, and then a solution of TEMPO (7.0 mg, 0.045 mmol, 15 mol%) in EtOAc (1 mL) was added. After stirring at 25 °C for 6 hours, the mixture was concentrated *in vacuo* to give dark residue, which was purified by flash chromatography using petroleum ether and ethyl acetate as the effluent (6:1 to 3:1, *v/v*) on silica gel to afford 58.5 mg of aminated oxazole **2a** (0.117 mmol, 39.2%), and its structure was proved by ¹H NMR, ¹³C NMR and HRMS. Meanwhile, 34.4 mg of unreacted oxazole **1a** (0.169 mmol, 56.5%) was recovered, and its ¹H NMR spectrum suggested that no H/D exchange occurred.

Independent Kinetic Isotope Effect (KIE) Experiments



Scheme S3. Parallel KIE experiments between benzofuran **3a** and C2-deuterated benzofuran **3a-d**

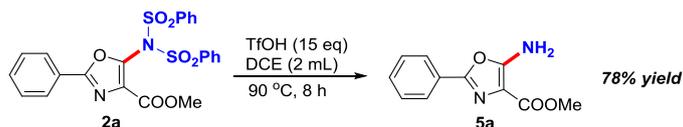
Typical Procedure: To a reaction tube charged with NFSI (378 mg, 1.2 mmol) was added a solution of benzofuran **3a** or C2-deuterated benzofuran **3a-d** (0.3 mmol) in EtOAc (1 mL) under argon (1 atm). The reaction mixture was stirred at 25 °C, and then a solution of TEMPO (7.0mg, 0.045 mmol, 15 mol%) in EtOAc (1 mL) was added. After stirring at 25 °C for indicate time as shown in Table S1, the mixture was concentrated *in vacuo* to give dark residue, which was analyzed with ¹H NMR in CDCl₃ to determine the yields of aminated benzofuran **4a** for each reaction, using CH₂Br₂ as an internal standard. Thus, the corresponding slope, intercept and R² were calculated as shown in Table S2. Accordingly, the equations for these two reactions were acquired, and the KIE were determined to be 1.92.

Table S2. Results of parallel KIE experiments

Time (min)	30	60	90	120	150	180	Slope	Intercept	RSQ
Yield of 4a from 3a (%)	13.23	20.19	26.06	32.10	41.03	46.50	0.2237	6.3607	0.9964
Yield of 4a from 3a-d (%)	7.38	11.92	14.34	18.84	21.87	25.02	0.1167	4.3067	0.9947
Equation for 4a from 3a :	y = 0.2237x+6.3607					R ² = 0.9964	y = yield (%)		
Equation for [D]-4a from 3a-d :	y = 0.1167x+4.3067					R ² = 0.9947	x = time (min)		

$$k_H/k_D = 0.2237 / 0.1167 = 1.9169$$

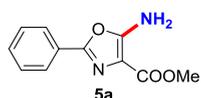
Deprotection Experiment



Scheme S4. Deprotection experiment of **2a**

Typical Procedure: To a reaction tube charged with a solution of aminated oxazole **2a** (149.4 mg, 0.3 mmol) in DCE (1 mL) was slowly added a solution of TfOH (4.5 mmol) in DCE (1 mL) at room temperature. The reaction mixture was stirred at 90 °C for 8 hours, the mixture was cooled to room temperature, quenched with saturated NaHCO₃ (aq.), and concentrated *in vacuo* to give dark residue, which was purified by flash chromatography using petroleum ether and ethyl acetate as the effluent (3:1, *v/v*) on silica gel to afford 51.2 mg of 5-amino oxazole **5a** (0.235 mmol, 78.3%).

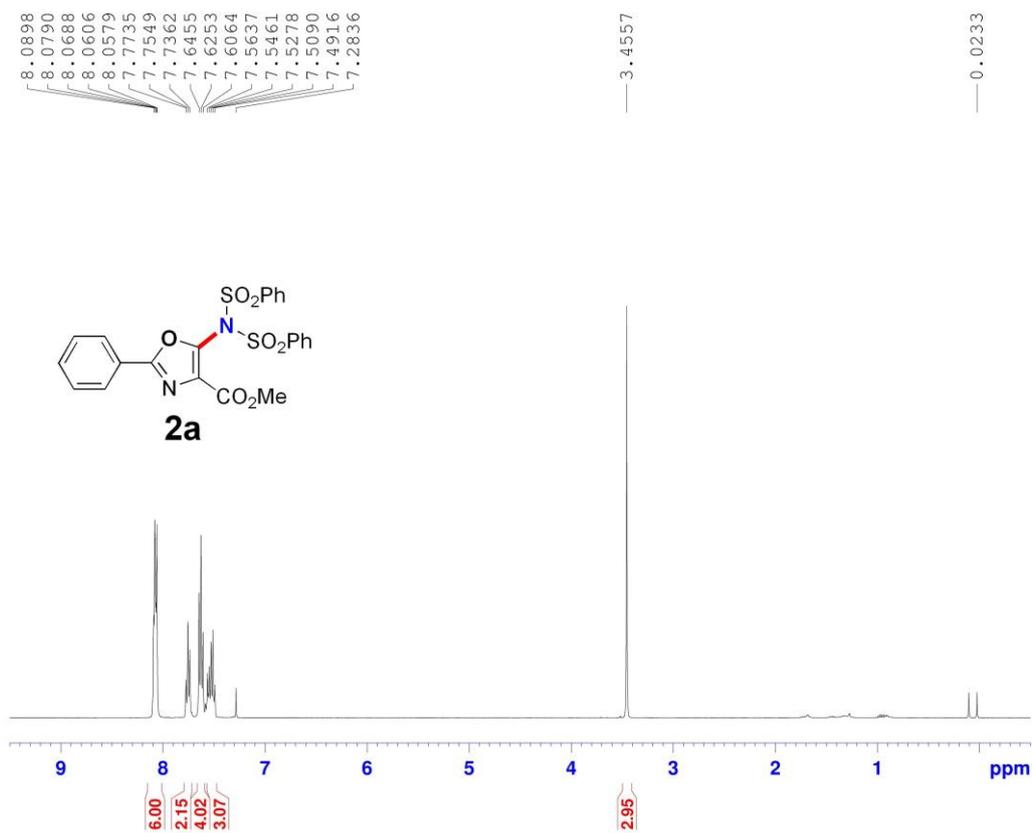
Methyl 5-amino-2-phenyloxazole-4-carboxylate (**5a**):



White solid, m.p. 185-186 °C. ¹H NMR (DMSO, 400 MHz): δ = 7.81-7.79 (m, 2H), 7.52-7.43 (m, 5H), 3.75 (s, 3H) ppm; ¹³C NMR (DMSO, 100 MHz): δ = 163.23, 161.07, 149.05, 130.02, 129.51, 127.10,

125.28, 103.80, 50.94 ppm; HRMS *m/z* (ESI) calcd for [C₁₁H₁₀N₂O₃+H]⁺ 219.0764, found 219.0766.

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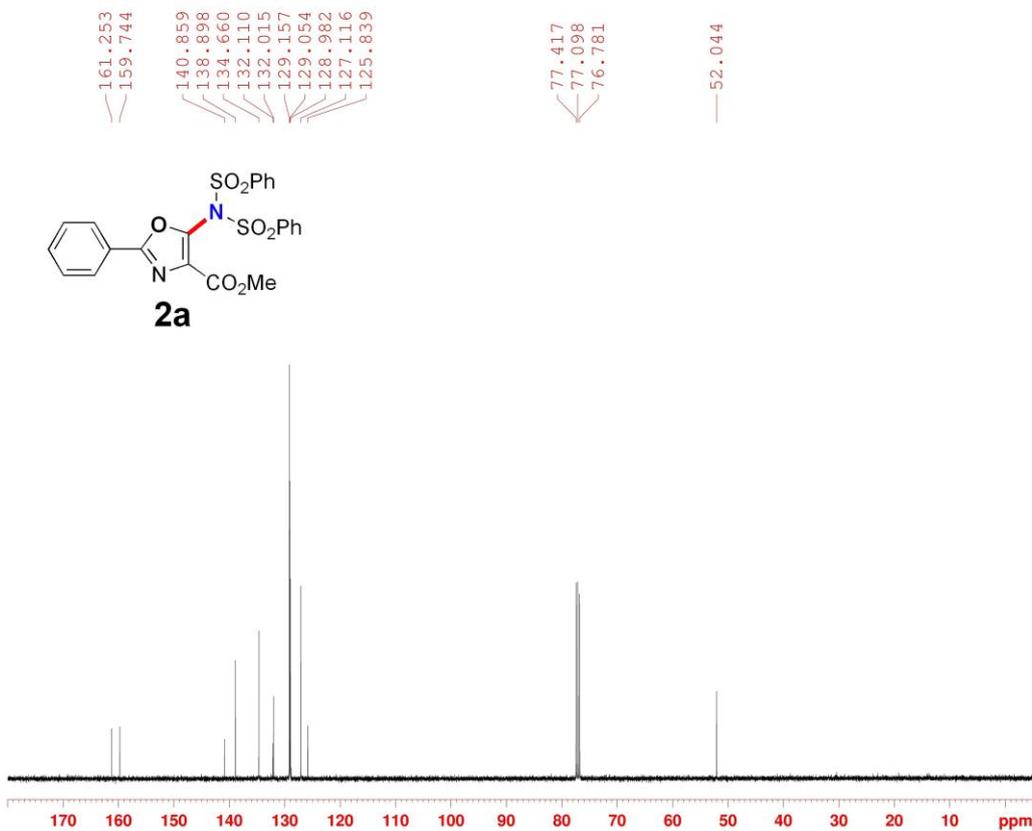


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FIDRES     0.122266 Hz
AQ         4.0894966 sec
RG         47.33
DW         62.400 usec
DE         6.50 usec
TE         300.2 K
D1         1.00000000 sec
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TDO        1
    
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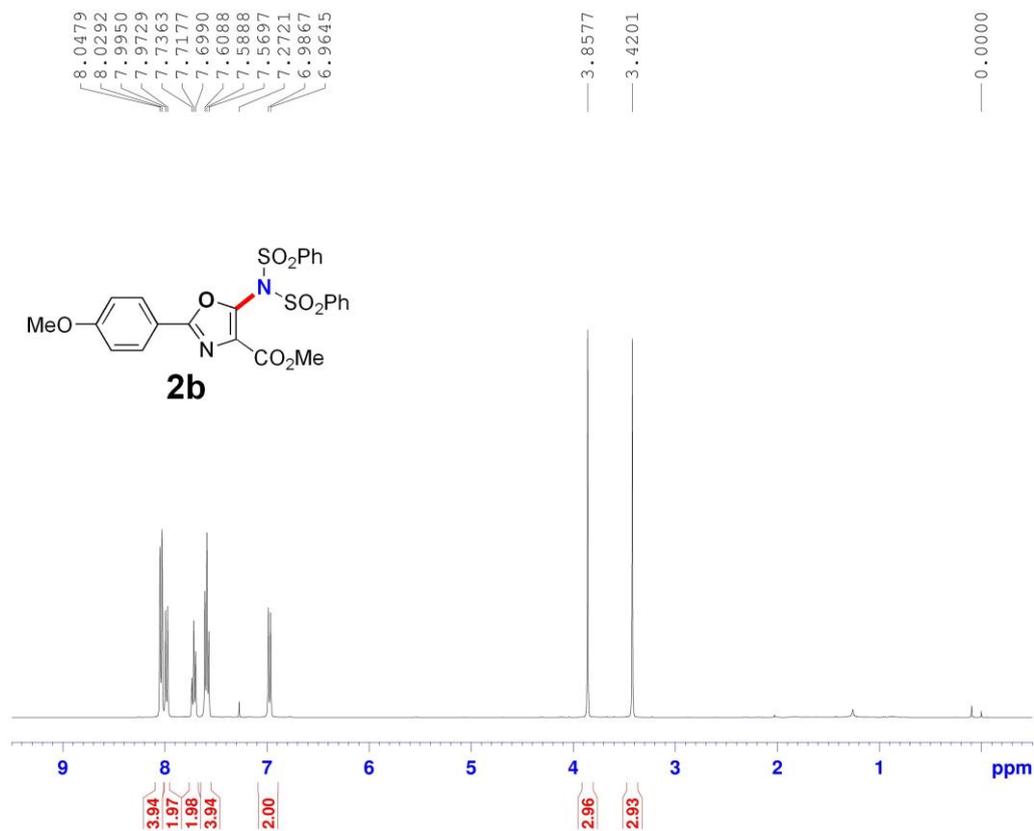
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Electronic Supplementary Information

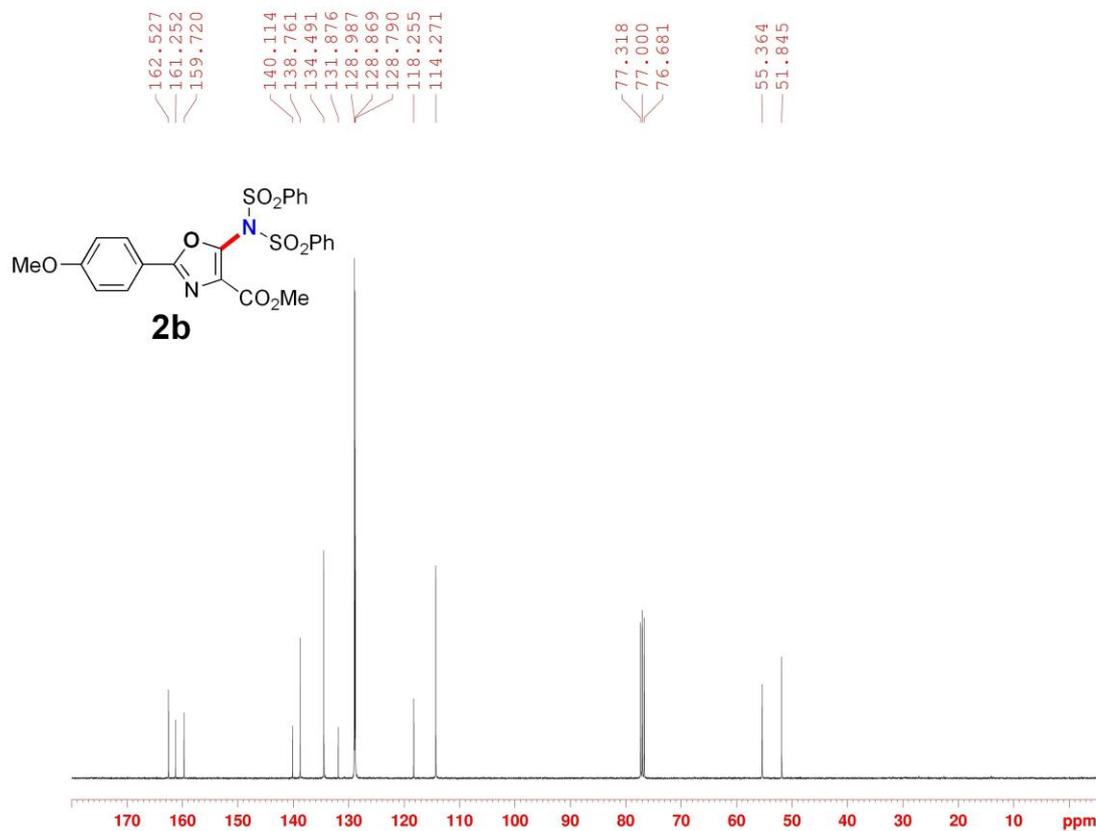


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AQ        4.0894866 sec
RG        18.6
DW        62.400 usec
DE        6.50 usec
TE        300.2 K
D1        1.00000000 sec
D11       0.23000000 sec
TDO       1
    
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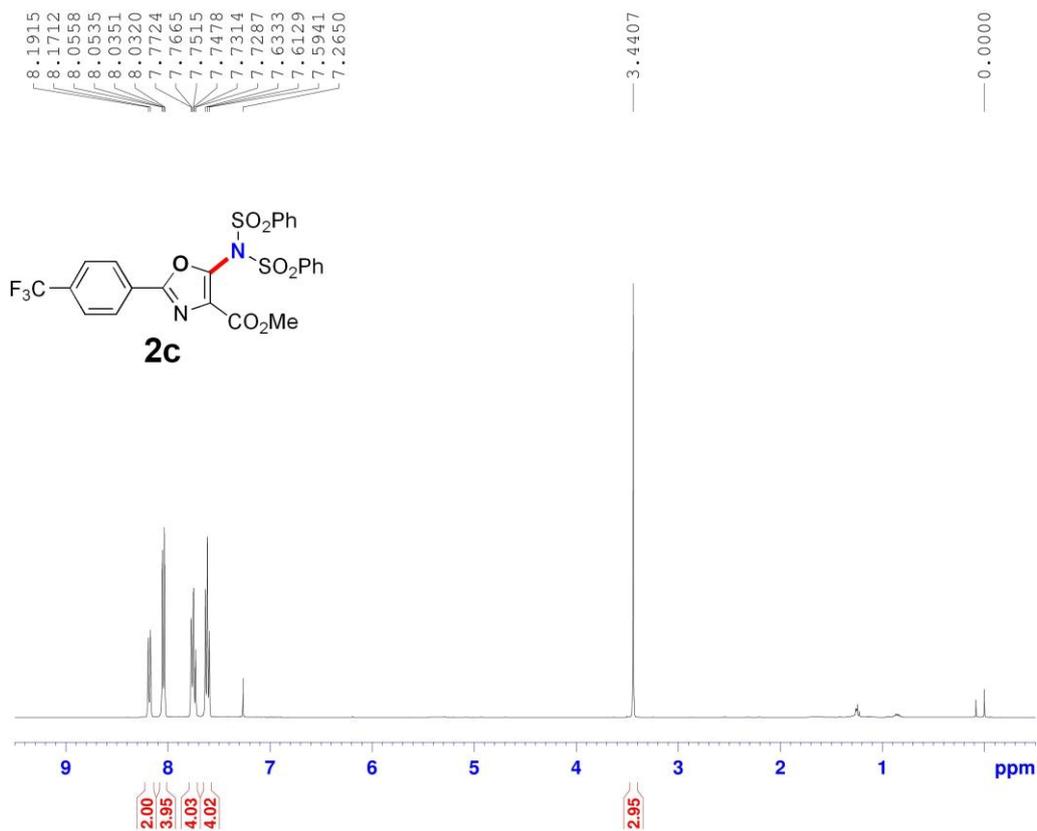
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FIDRES    0.366798 Hz
AQ        1.361988 sec
RG        194.26
DW        20.800 usec
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TE        300.1 K
D1        2.00000000 sec
D11       0.23000000 sec
TDO       1
    
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===== CHANNEL f1 =====
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Electronic Supplementary Information

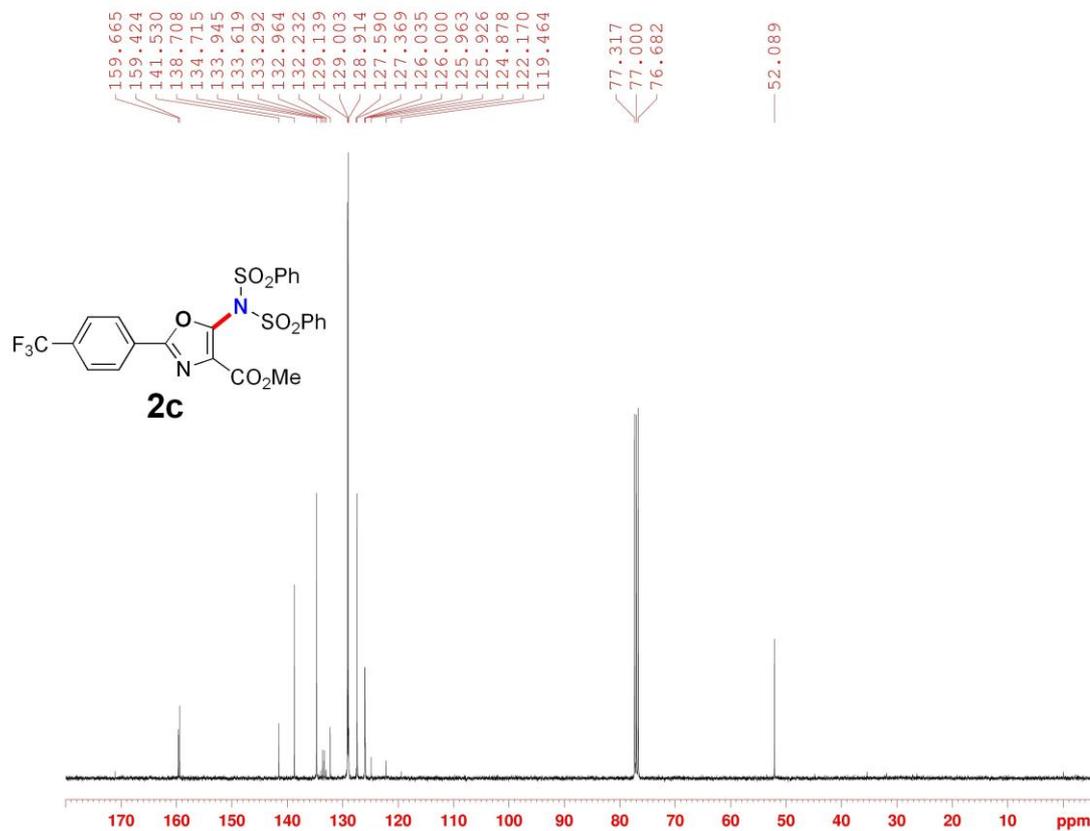


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FIDRES     0.122266 Hz
AQ         4.0894966 sec
RG         47.33
DW         62.400 usec
DE         6.50 usec
TE         300.1 K
D1         1.00000000 sec
TDO        1
    
```

```

----- CHANNEL f1 -----
SFO1     400.1324710 MHz
NUC1      1H
P1        8.04 usec
SI        65536
SF        400.1300074 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
    
```



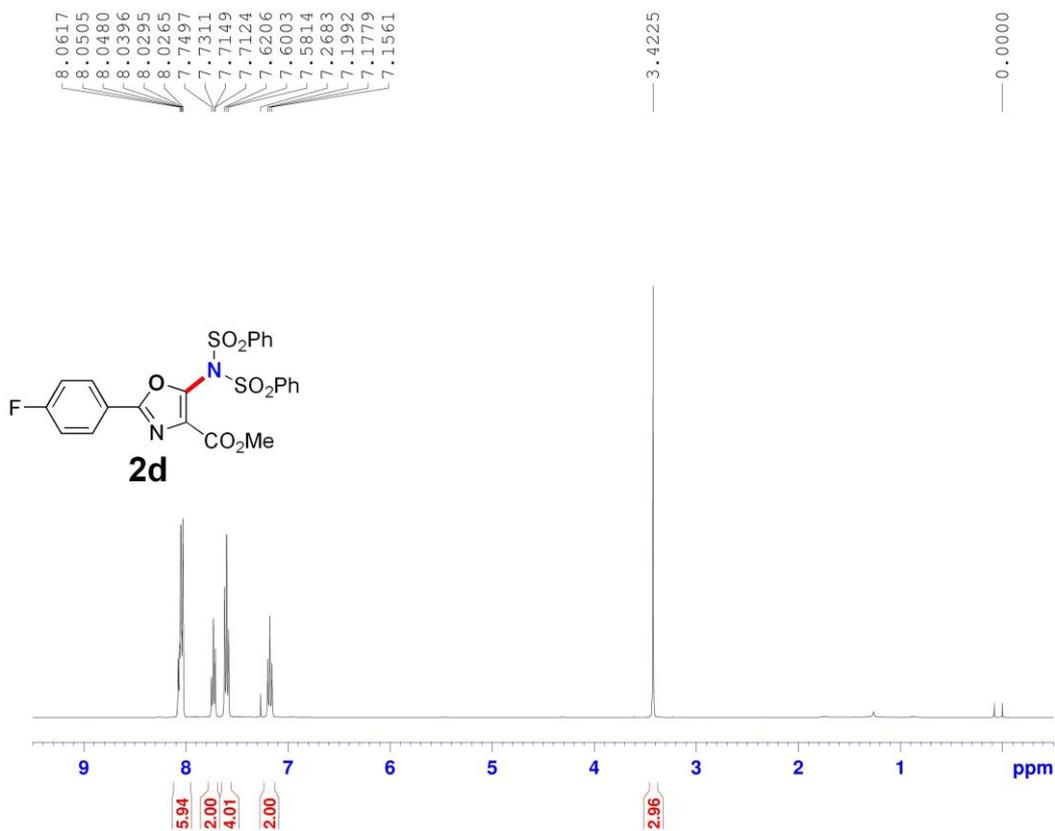
```

NAME      CLJ-WL-M201
EXPNO    2
PROCNO    1
Date_     20190105
Time      6.26
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         512
DS         0
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.361988 sec
RG         194.26
DW         20.800 usec
DE         6.50 usec
TE         300.1 K
D1         2.00000000 sec
D11       0.23000000 sec
TDO        1
    
```

```

----- CHANNEL f1 -----
SFO1     100.6228293 MHz
NUC1      13C
P1        8.54 usec
SI        32768
SF        100.6127744 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
    
```

Electronic Supplementary Information

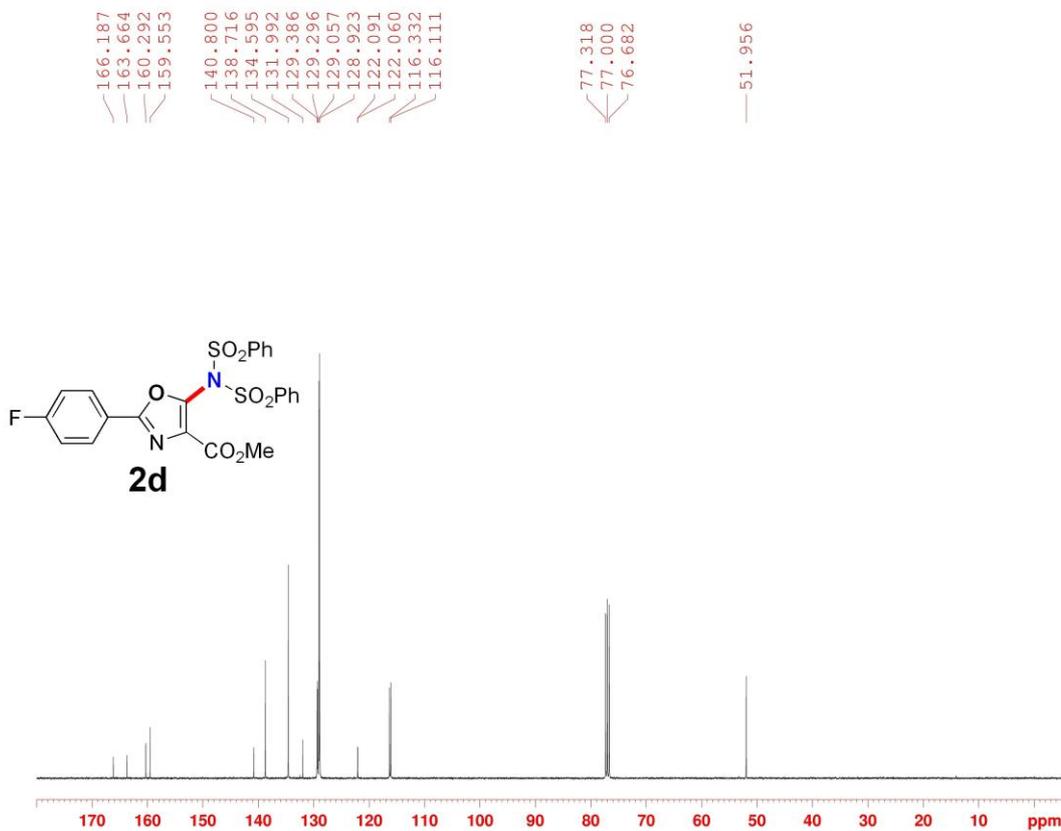


```

NAME      CLJ-WL-M204
EXPNO    1
PROCNO    1
Date_     20190104
Time      4.09
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         12
DS         0
SWH        8012.820 Hz
FIDRES     0.122266 Hz
AQ          4.0894866 sec
RG          36.4
DW          62.400 usec
DE          6.50 usec
TE          300.1 K
D1          1.00000000 sec
TDO         1
    
```

```

----- CHANNEL f1 -----
SFO1      400.1324710 MHz
NUC1       1H
P1         8.04 usec
SI         65536
SF         400.1300059 MHz
WDW        EM
SSB         0
LB          0.30 Hz
GB          0
PC          1.00
    
```



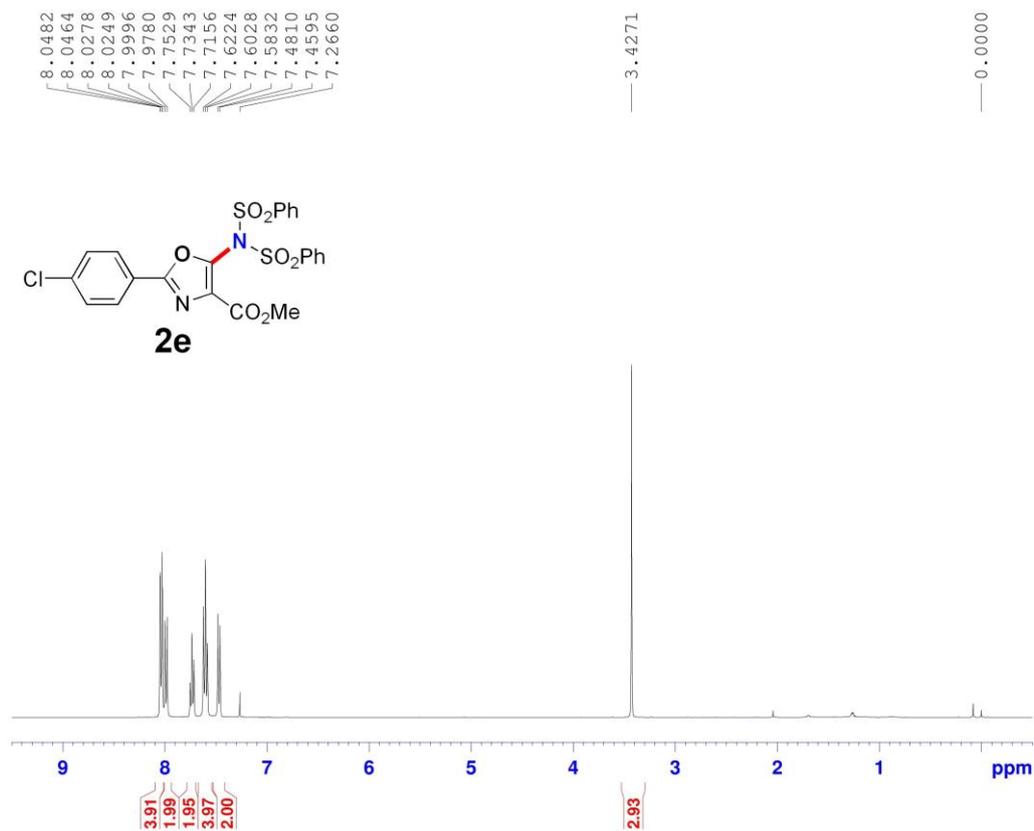
```

NAME      CLJ-WL-M204
EXPNO    2
PROCNO    1
Date_     20190104
Time      4.39
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         512
DS         0
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ          1.361988 sec
RG          194.26
DW          20.800 usec
DE          6.50 usec
TE          300.1 K
D1          2.00000000 sec
D11        0.23000000 sec
TDO         1
    
```

```

----- CHANNEL f1 -----
SFO1      100.6228293 MHz
NUC1       13C
P1         8.54 usec
SI         32768
SF         100.6127797 MHz
WDW        EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40
    
```

Electronic Supplementary Information



```

NAME      CLJ-WL-M199
EXPNO    1
PROCNO   1
Date_    20181231
Time     5.42
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zg30
TD       65536
SOLVENT  CDCl3
NS       12
DS       0
SWH      8012.820 Hz
FIDRES   0.122266 Hz
AQ       4.0894966 sec
RG       41.07
DW       62.400 usec
DE       6.50 usec
TE       300.2 K
D1       1.00000000 sec
D11      0.20000000 sec
TDO      1
    
```

```

===== CHANNEL f1 =====
SF01    400.1324710 MHz
NUC1     1H
P1       8.04 usec
SI       65536
SF       400.1300069 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
    
```

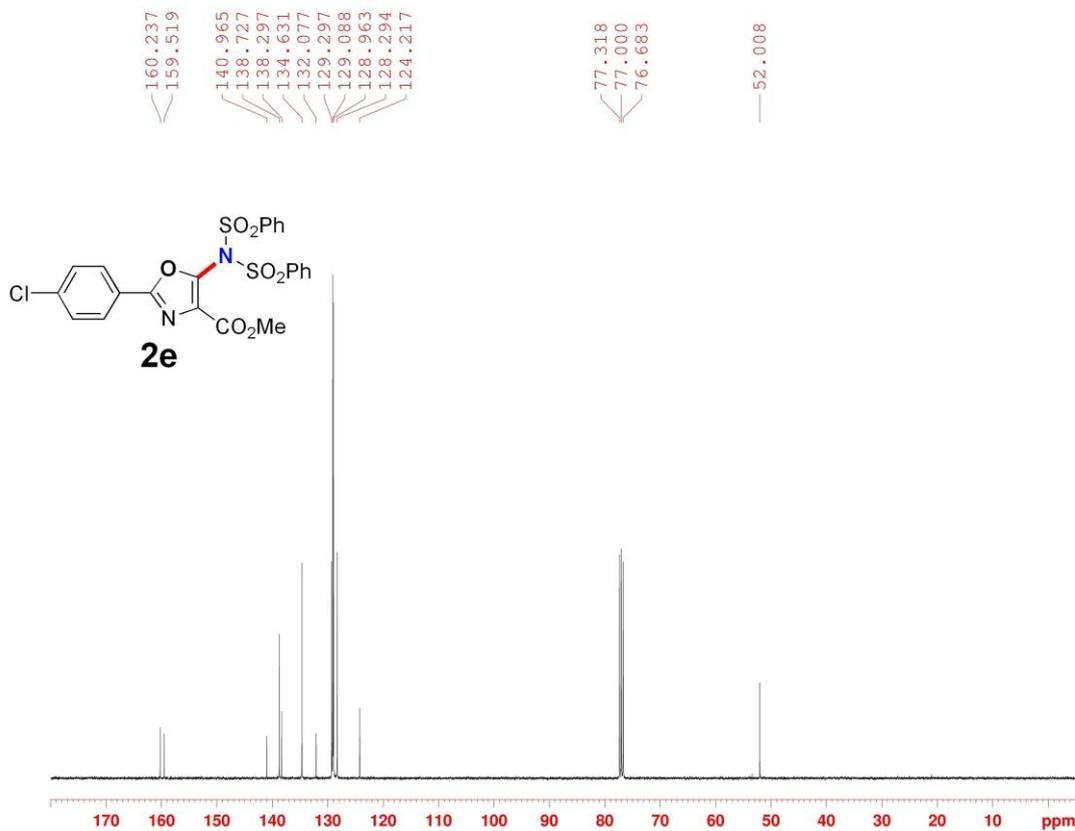


```

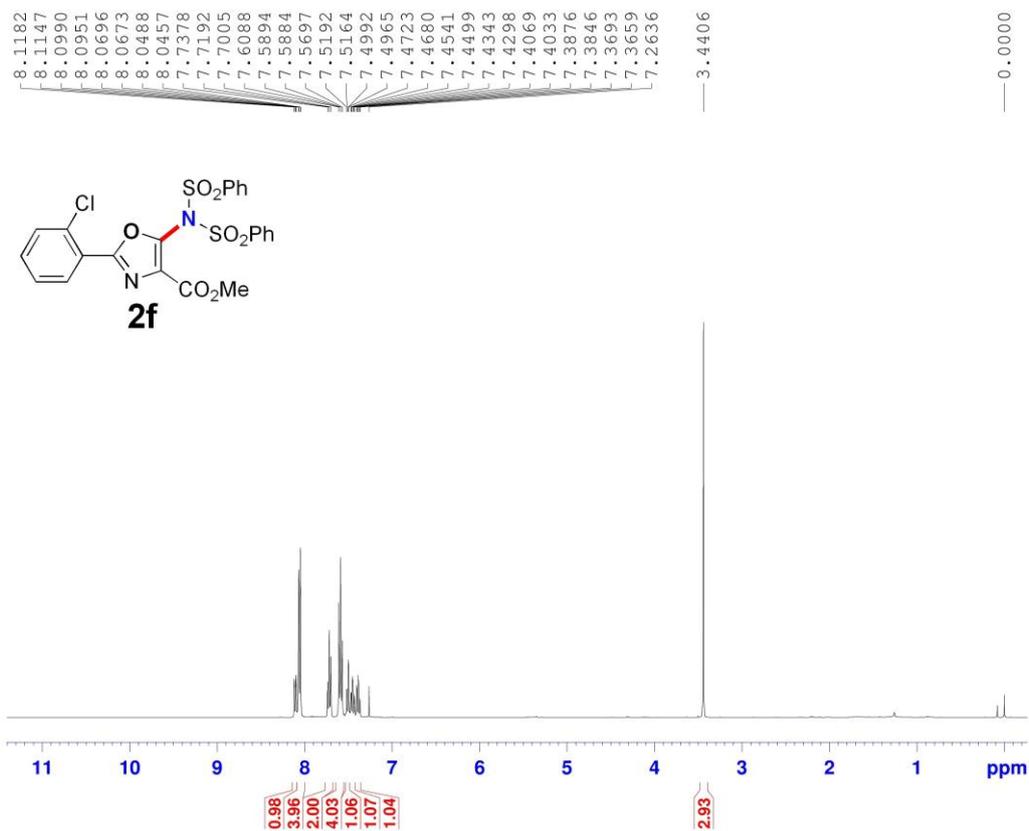
NAME      CLJ-WL-M199
EXPNO    2
PROCNO   1
Date_    20181221
Time     6.12
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       512
DS       0
SWH      24038.461 Hz
FIDRES   0.366798 Hz
AQ       1.361988 sec
RG       194.26
DW       20.800 usec
DE       6.50 usec
TE       300.2 K
D1       2.00000000 sec
D11      0.20000000 sec
TDO      1
    
```

```

===== CHANNEL f1 =====
SF01    100.6228293 MHz
NUC1     13C
P1       8.54 usec
SI       32768
SF       100.6127769 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
    
```



Electronic Supplementary Information

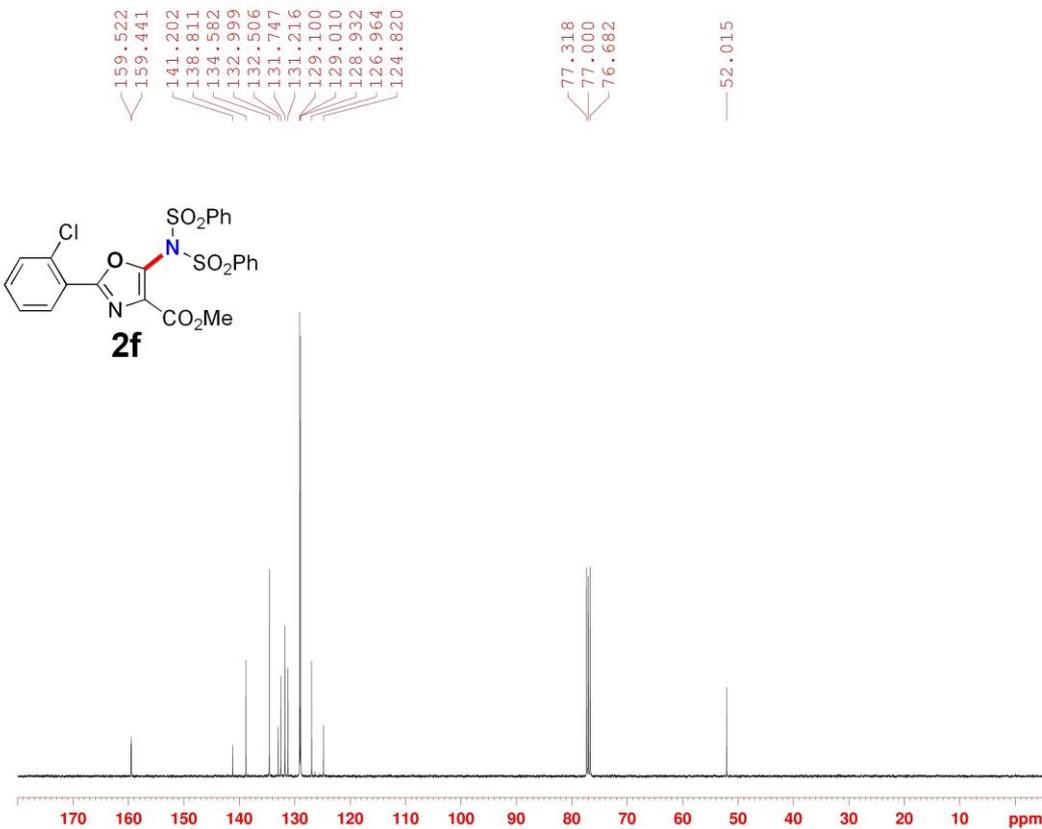


```

NAME      CLJ-WL-M207
EXPNO    1
PROCNO    1
Date_    20190110
Time     5.43
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD        65536
SOLVENT   CDCl3
NS        12
DS        0
SWH       8012.820 Hz
FIDRES    0.122266 Hz
AQ        4.0894966 sec
RG        51.19
DW        62.400 usec
DE        6.50 usec
TE        300.1 K
D1        1.00000000 sec
D11       1
TDO       1
    
```

```

===== CHANNEL f1 =====
SF01    400.1324710 MHz
NUC1     1H
P1       8.04 usec
SI       65536
SF       400.1300080 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
    
```



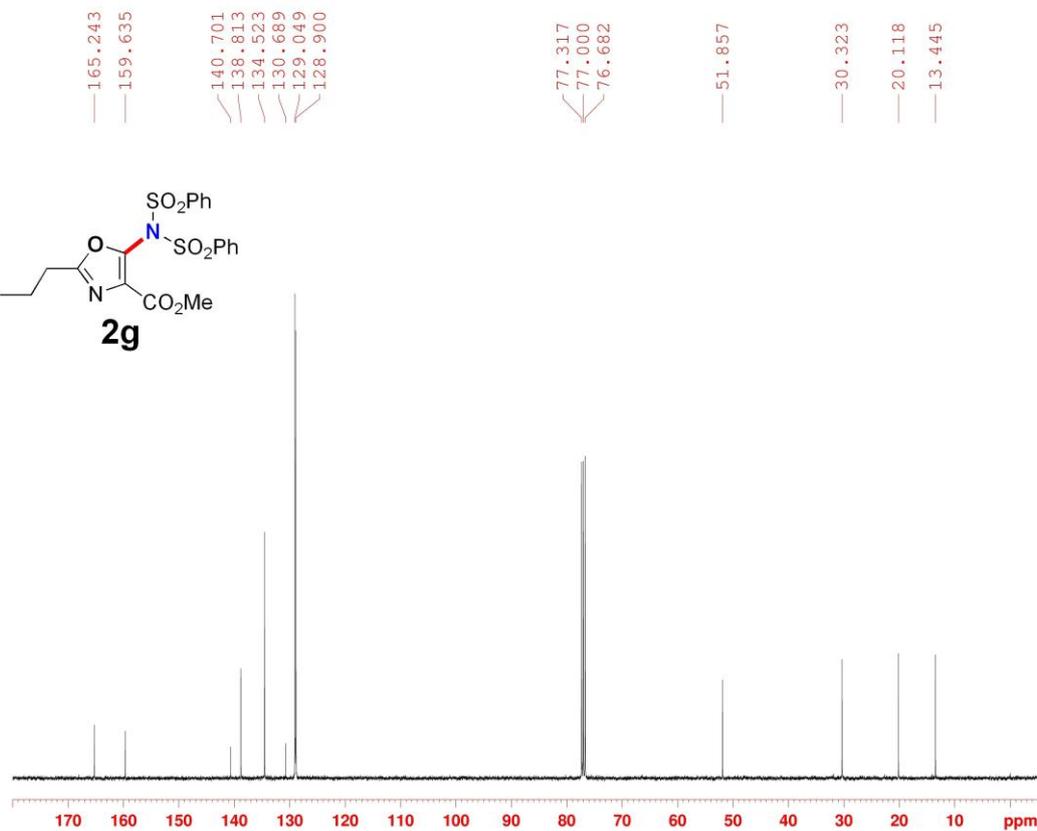
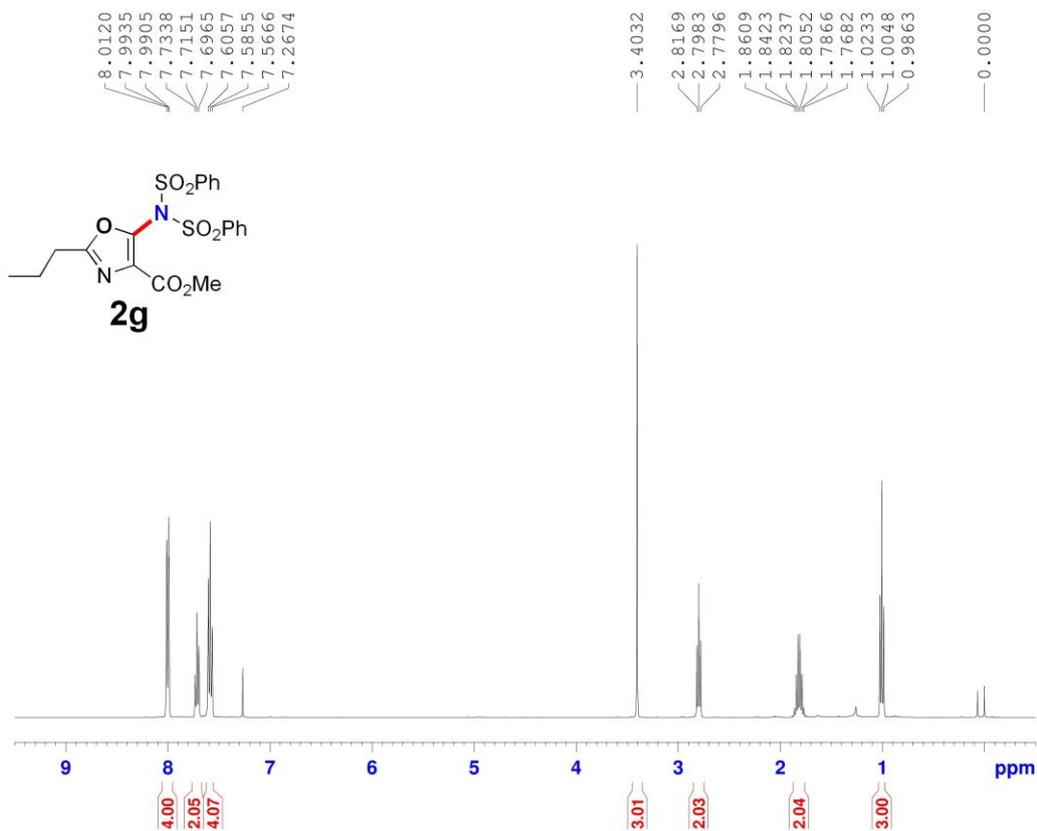
```

NAME      CLJ-WL-M207
EXPNO    2
PROCNO    1
Date_    20190110
Time     5.41
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS        512
DS        0
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ        1.361988 sec
RG        194.26
DW        20.800 usec
DE        6.50 usec
TE        300.1 K
D1        2.00000000 sec
D11       0.23000000 sec
TDO       1
    
```

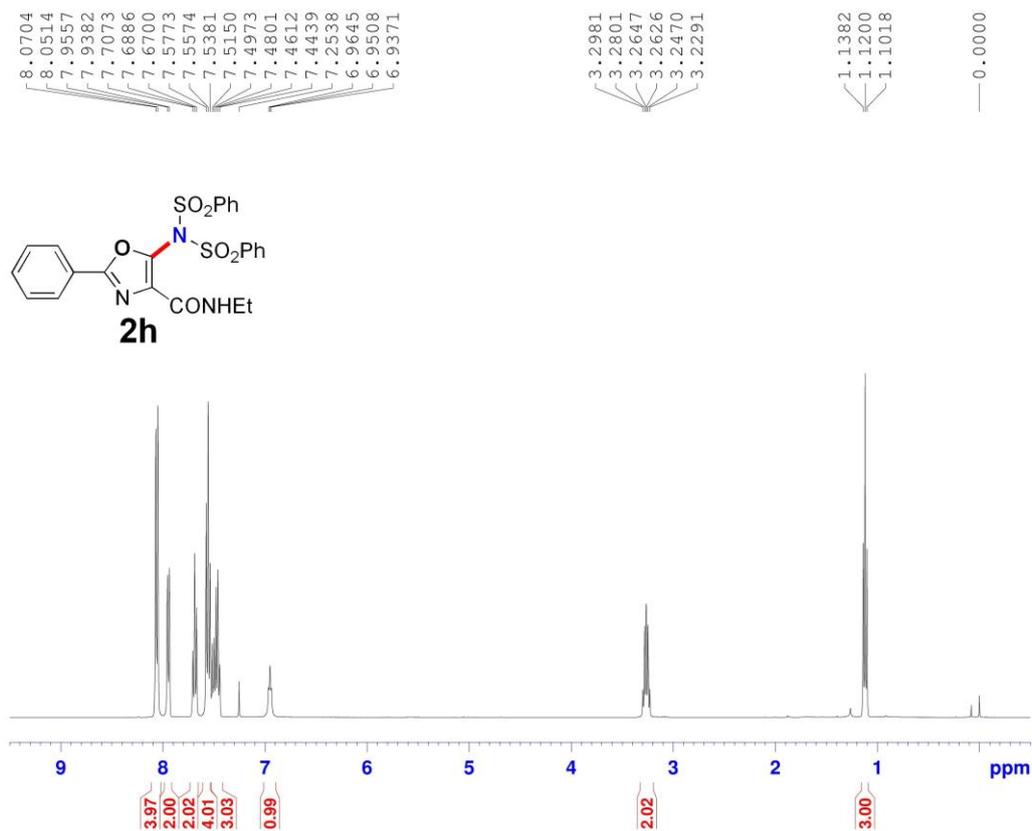
```

===== CHANNEL f1 =====
SF01    100.6228293 MHz
NUC1     13C
P1       8.54 usec
SI       32768
SF       100.6127757 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
    
```

Electronic Supplementary Information



Electronic Supplementary Information

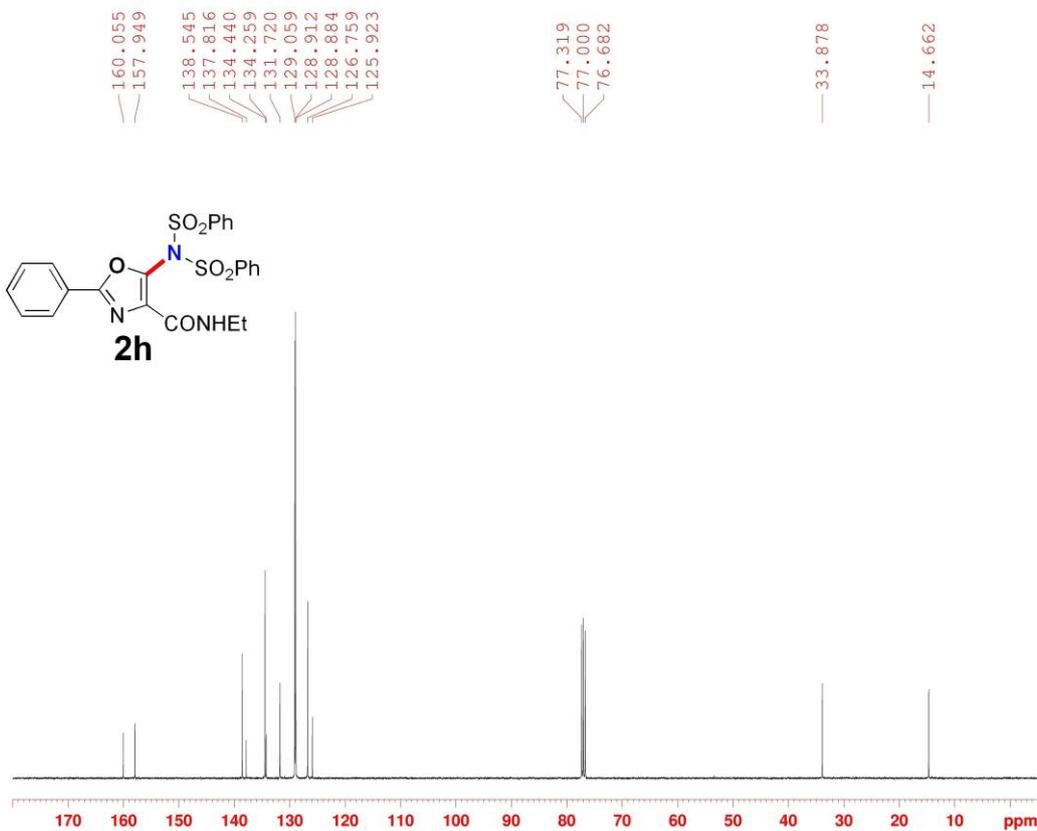


```

NAME      CLJ-WL-M205
EXPNO    2
PROCNO    1
Date_     20190106
Time      7.49
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         12
DS         0
SWH        8012.820 Hz
FIDRES     0.122266 Hz
AQ         4.0894966 sec
RG         23.9
DW         62.400 usec
DE         6.50 usec
TE         300.1 K
D1         1.00000000 sec
TDO        1
    
```

```

===== CHANNEL f1 =====
SFO1     400.1324710 MHz
NUC1      1H
P1        8.04 usec
SI        65536
SF        400.1300119 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
    
```



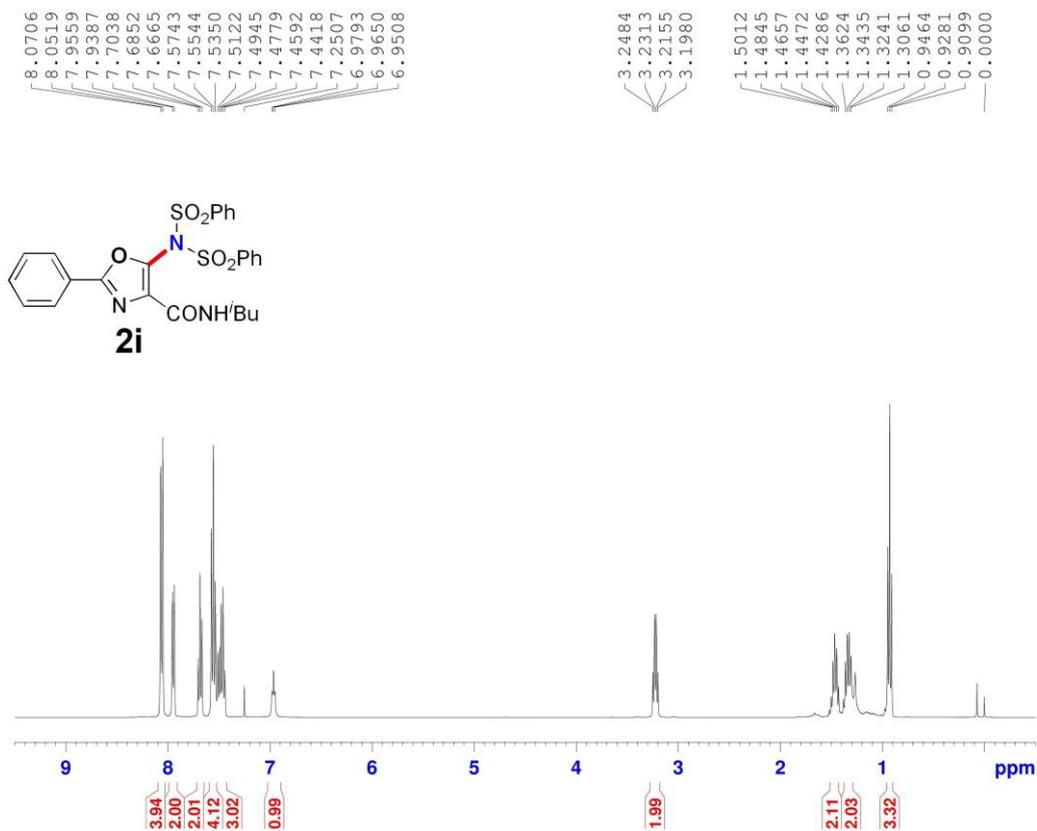
```

NAME      CLJ-WL-M205
EXPNO    1
PROCNO    1
Date_     20190106
Time      7.48
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         512
DS         0
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.361988 sec
RG         194.26
DW         20.800 usec
DE         6.50 usec
TE         300.2 K
D1         2.00000000 sec
D11        0.20000000 sec
TDO        1
    
```

```

===== CHANNEL f1 =====
SFO1     100.6228293 MHz
NUC1      13C
P1        8.54 usec
SI        32768
SF        100.6127791 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
    
```

Electronic Supplementary Information

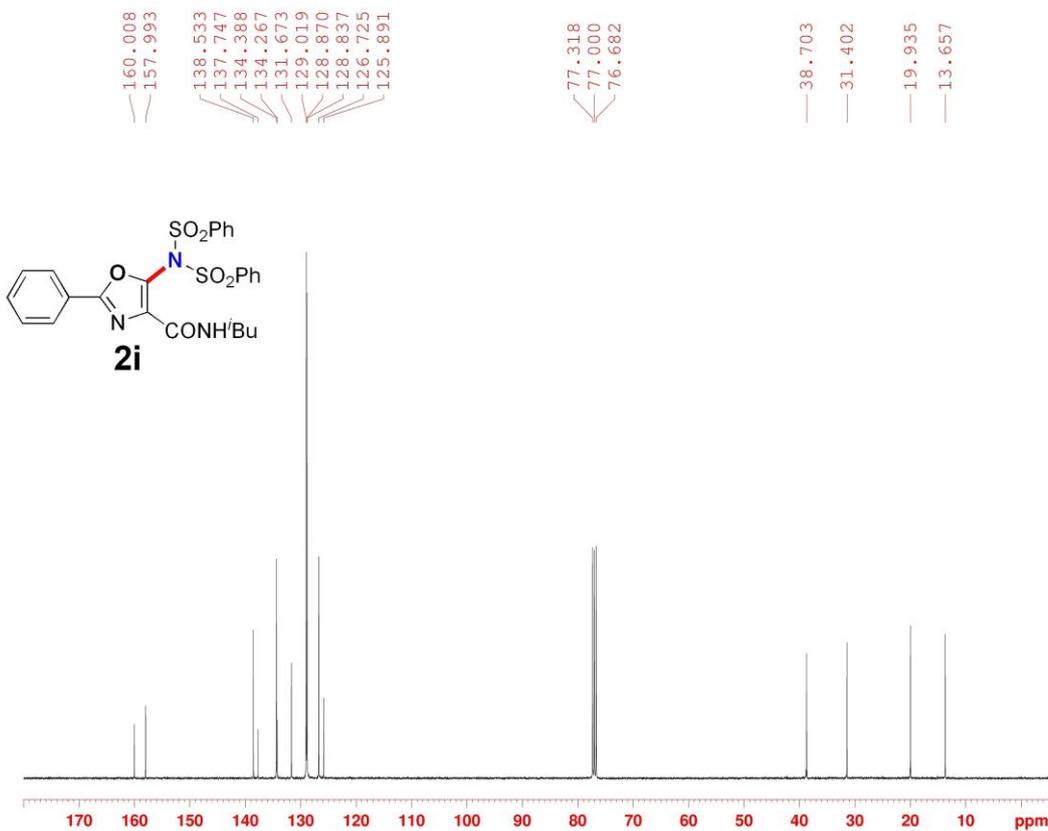


```

NAME      CLJ-WL-M206
EXPNO    2
PROCNO    1
Date_     20190106
Time      8.24
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         12
DS         0
SWH        8012.820 Hz
FIDRES     0.122266 Hz
AQ         4.0894866 sec
RG         18.6
DW         62.400 usec
DE         6.50 usec
TE         300.1 K
D1         1.00000000 sec
TD0        1
    
```

```

----- CHANNEL f1 -----
SFO1     400.1324710 MHz
NUC1      1H
P1        8.04 usec
SI        65536
SF        400.1300132 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
    
```



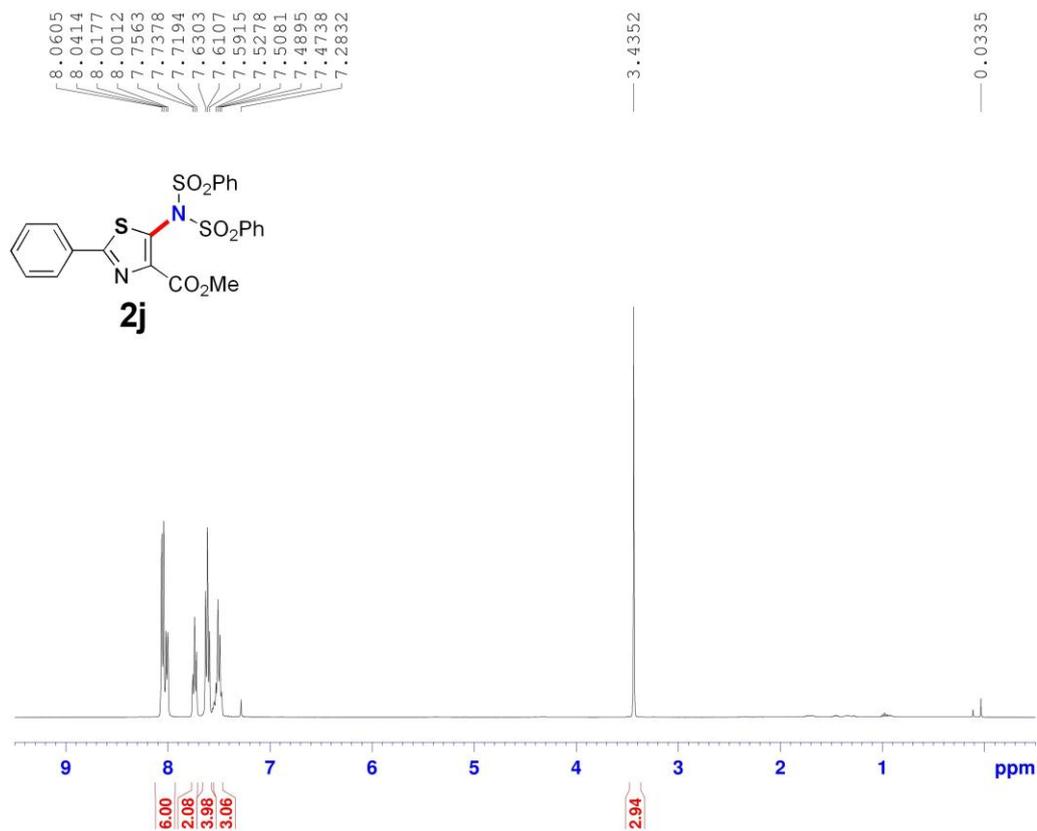
```

NAME      CLJ-WL-M206
EXPNO    1
PROCNO    1
Date_     20190106
Time      8.22
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         512
DS         0
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.361988 sec
RG         194.26
DW         20.800 usec
DE         6.50 usec
TE         300.1 K
D1         2.00000000 sec
D11        0.23000000 sec
TD0        1
    
```

```

----- CHANNEL f1 -----
SFO1     100.6228293 MHz
NUC1      13C
P1        8.54 usec
SI        32768
SF        100.6127811 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
    
```

Electronic Supplementary Information

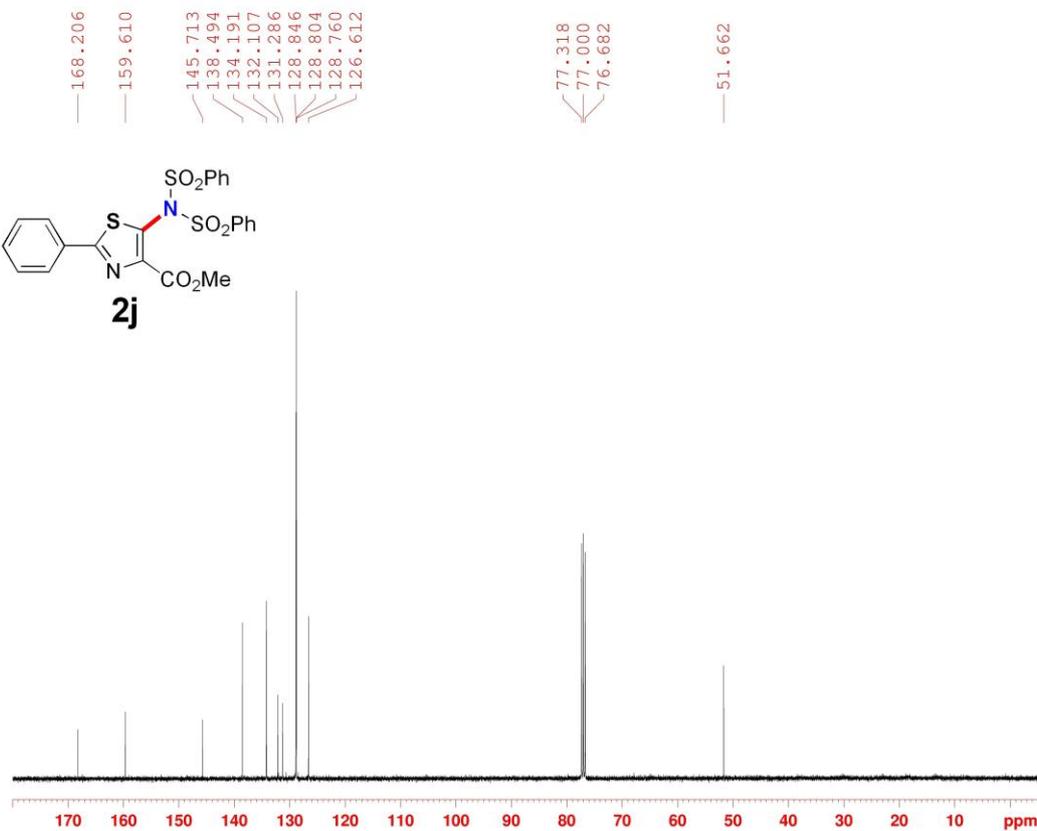


```

NAME      CLJ-WL-M159
EXPNO     1
PROCNO    1
Date_     20181119
Time      12.46
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         12
DS         0
SWH        8012.820 Hz
FIDRES     0.122266 Hz
AQ         4.0894866 sec
RG         36.4
DW         62.400 usec
DE         6.50 usec
TE         300.2 K
D1         1.00000000 sec
TDO        1
    
```

```

===== CHANNEL f1 =====
SFO1     400.1324710 MHz
NUC1      1H
P1        8.04 usec
SI        65536
SF        400.1300000 MHz
WDW        no
SSB        0
LB         0.00 Hz
GB         0
PC         1.00
    
```



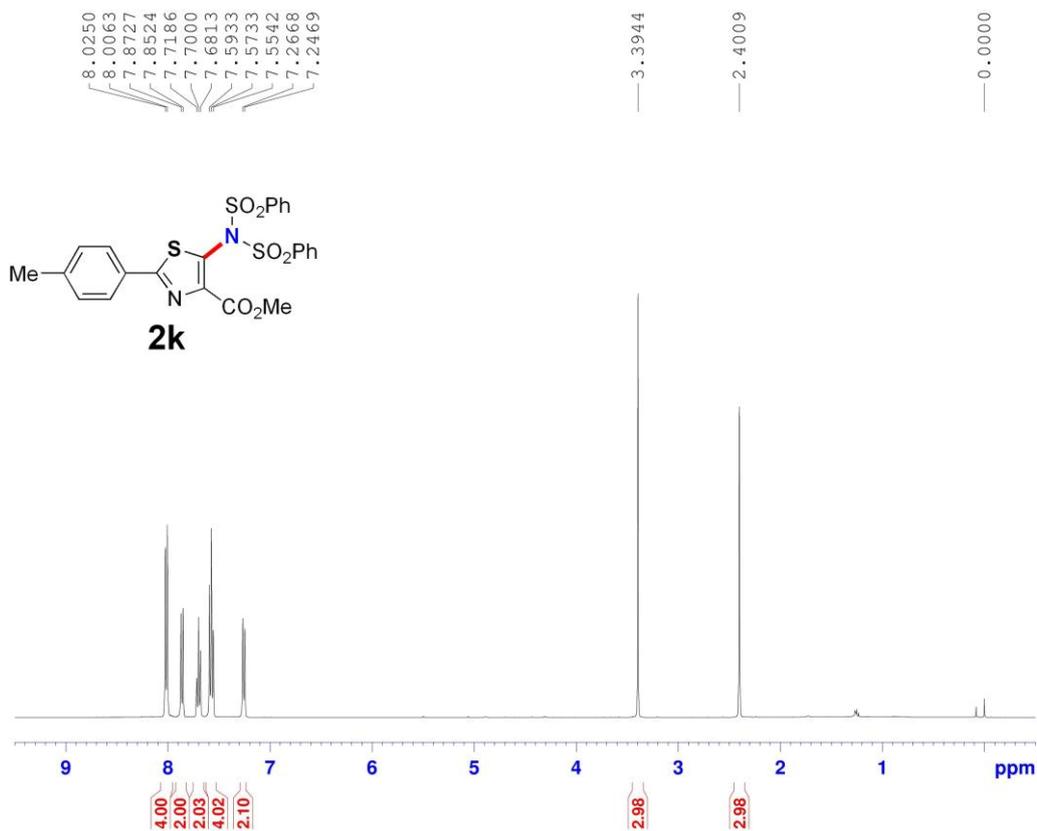
```

NAME      CLJ-WL-M159
EXPNO     2
PROCNO    1
Date_     20181119
Time      13.02
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         256
DS         0
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.361988 sec
RG         194.26
DW         20.800 usec
DE         6.50 usec
TE         300.1 K
D1         2.00000000 sec
D11        0.20000000 sec
TDO        1
    
```

```

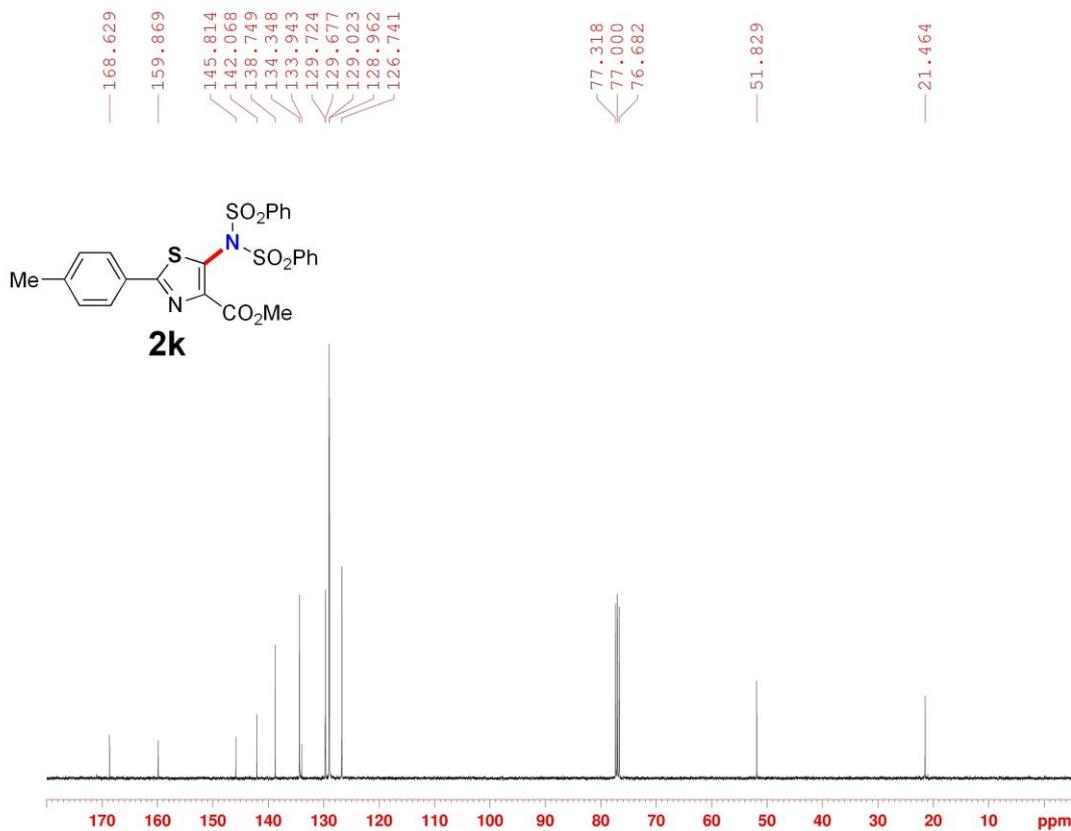
===== CHANNEL f1 =====
SFO1     100.6228293 MHz
NUC1      13C
P1        8.54 usec
SI        32768
SF        100.6128011 MHz
WDW        no
SSB        0
LB         0.00 Hz
GB         0
PC         1.40
    
```

Electronic Supplementary Information



```

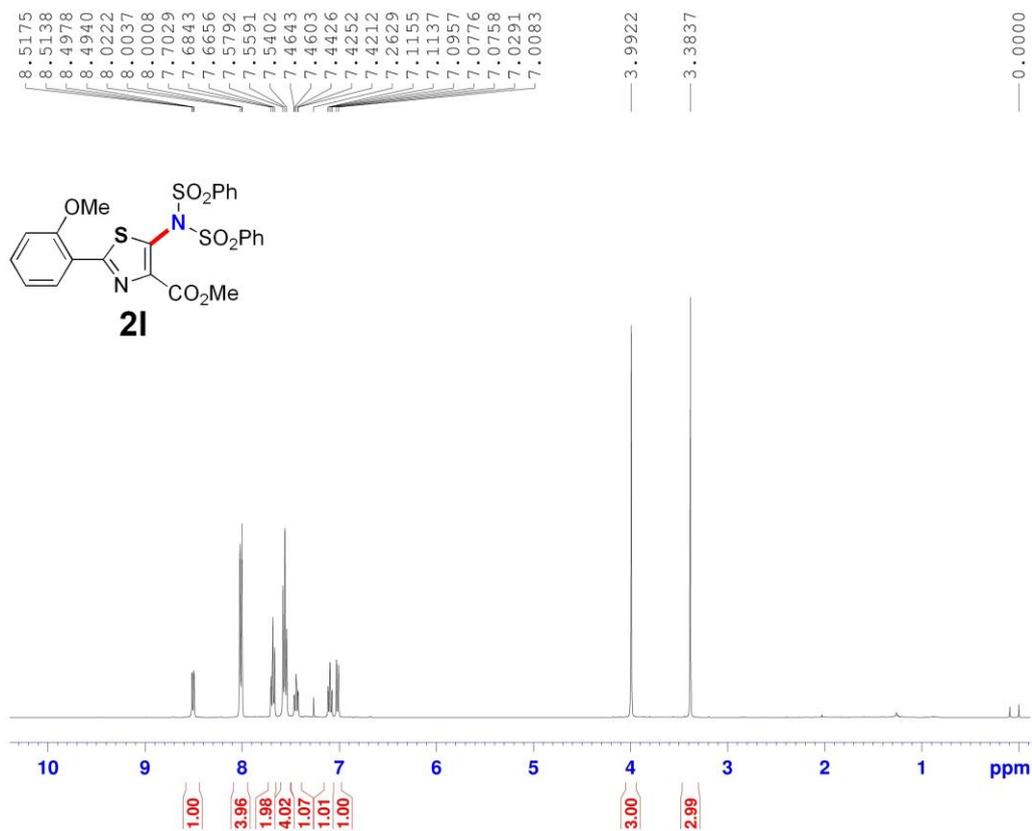
NAME      CLJ-WL-M213
EXPNO    1
PROCNO    1
Date_    20190108
Time     14.19
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD        65536
SOLVENT   CDCl3
NS        12
DS        0
SWH       8012.820 Hz
FIDRES    0.122266 Hz
AQ        4.0894966 sec
RG        23.9
DW        62.400 usec
DE        6.50 usec
TE        300.1 K
D1        1.00000000 sec
TDO       1
----- CHANNEL f1 -----
SFO1     400.1324710 MHz
NUC1     1H
P1       8.04 usec
SI       65536
SF       400.1300085 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
    
```



```

NAME      CLJ-WL-M213
EXPNO    2
PROCNO    1
Date_    20190108
Time     14.30
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS        200
DS        0
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ        1.361988 sec
RG        194.26
DW        20.800 usec
DE        6.50 usec
TE        300.1 K
D1        2.00000000 sec
D11      0.20000000 sec
TDO       1
----- CHANNEL f1 -----
SFO1     100.6228293 MHz
NUC1     13C
P1       8.54 usec
SI       32768
SF       100.6127783 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
    
```

Electronic Supplementary Information



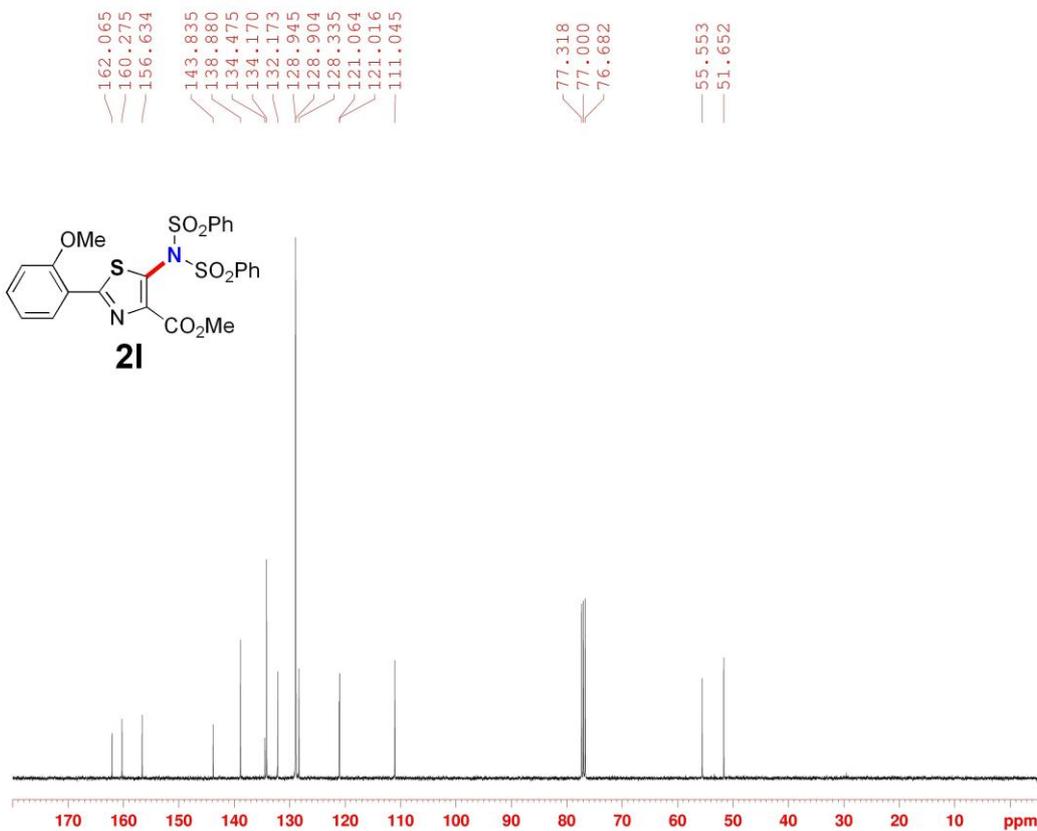
BRUKER

```

NAME      CLJ-WL-M210
EXPNO    1
PROCNO   1
Date_    20190109
Time     9.03
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zg30
TD       65536
SOLVENT  CDCl3
NS       12
DS       0
SWH      8012.820 MHz
FIDRES   0.122266 MHz
AQ       4.0894966 sec
RG       21.11
DW       62.400 usec
DE       6.50 usec
TE       300.1 K
D1       1.00000000 sec
TDO      1
    
```

```

===== CHANNEL f1 =====
SF01   400.1324710 MHz
NUC1    1H
P1      8.04 usec
SI      65536
SF      400.1300083 MHz
WDW     EM
SSB     0
LB      0.30 MHz
GB      0
PC      1.00
    
```



BRUKER

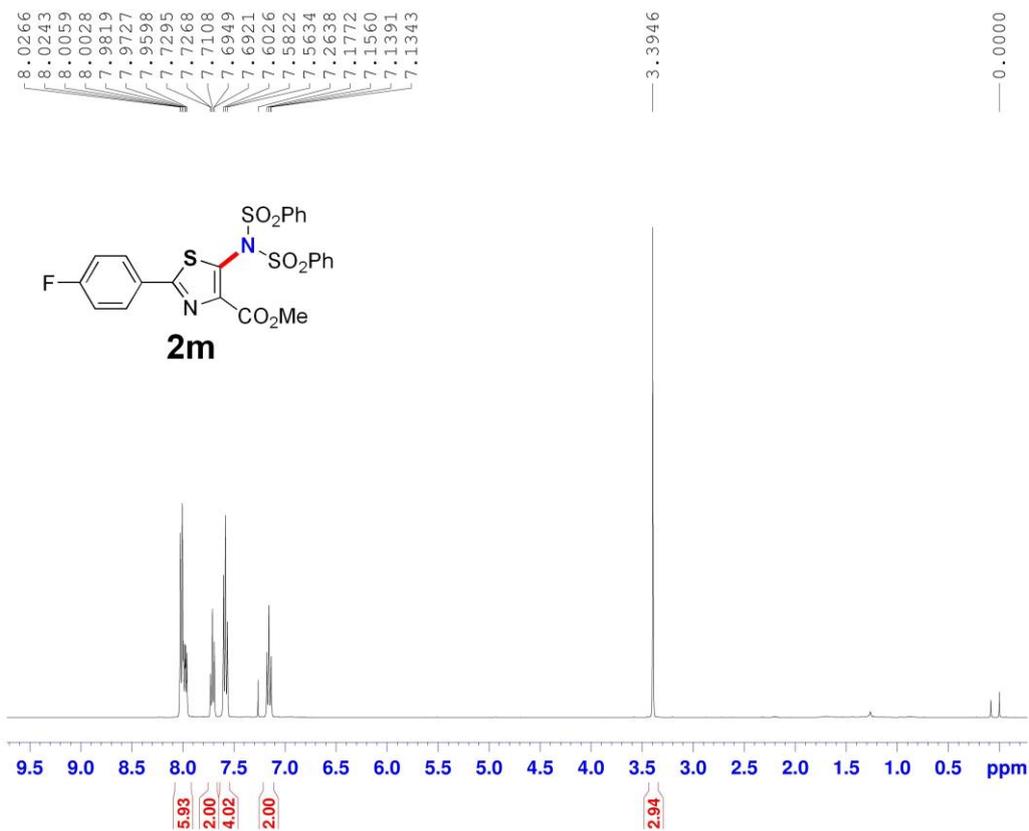
```

NAME      CLJ-WL-M210
EXPNO    2
PROCNO   1
Date_    20190109
Time     9.12
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       150
DS       0
SWH      24038.461 MHz
FIDRES   0.366798 MHz
AQ       1.361988 sec
RG       194.26
DW       20.800 usec
DE       6.50 usec
TE       300.2 K
D1       2.00000000 sec
D11      0.23000000 sec
TDO      1
    
```

```

===== CHANNEL f1 =====
SF01   100.6228293 MHz
NUC1    13C
P1      8.54 usec
SI      32768
SF      100.6127825 MHz
WDW     EM
SSB     0
LB      1.00 MHz
GB      0
PC      1.40
    
```

Electronic Supplementary Information

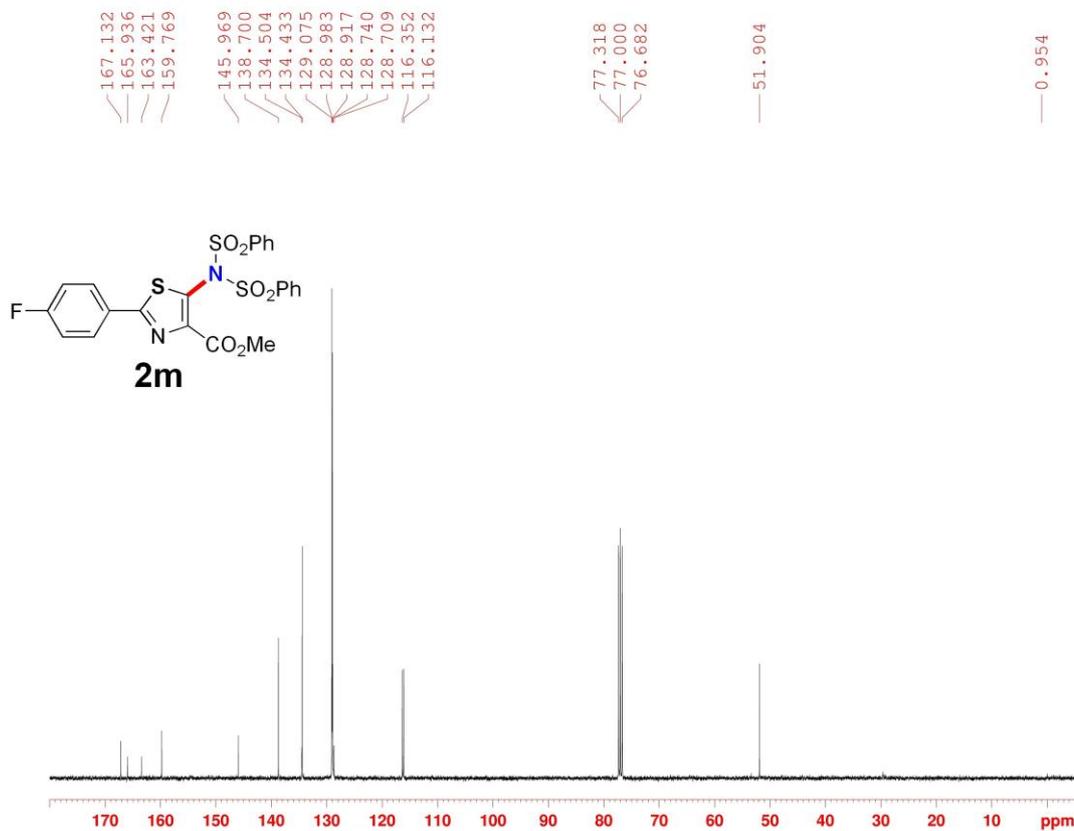


```

NAME      CLJ-WL-M209
EXPNO     1
PROCNO    1
Date_     20190109
Time      9.17
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         12
DS         0
SWH        8012.820 Hz
FIDRES     0.122266 Hz
AQ         4.0894966 sec
RG         47.53
DW         62.400 usec
DE         6.50 usec
TE         300.2 K
D1         1.00000000 sec
TDO        1
    
```

```

===== CHANNEL f1 =====
SFO1     400.1324710 MHz
NUC1      1H
P1        8.04 usec
SI        65536
SF        400.1300079 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
    
```



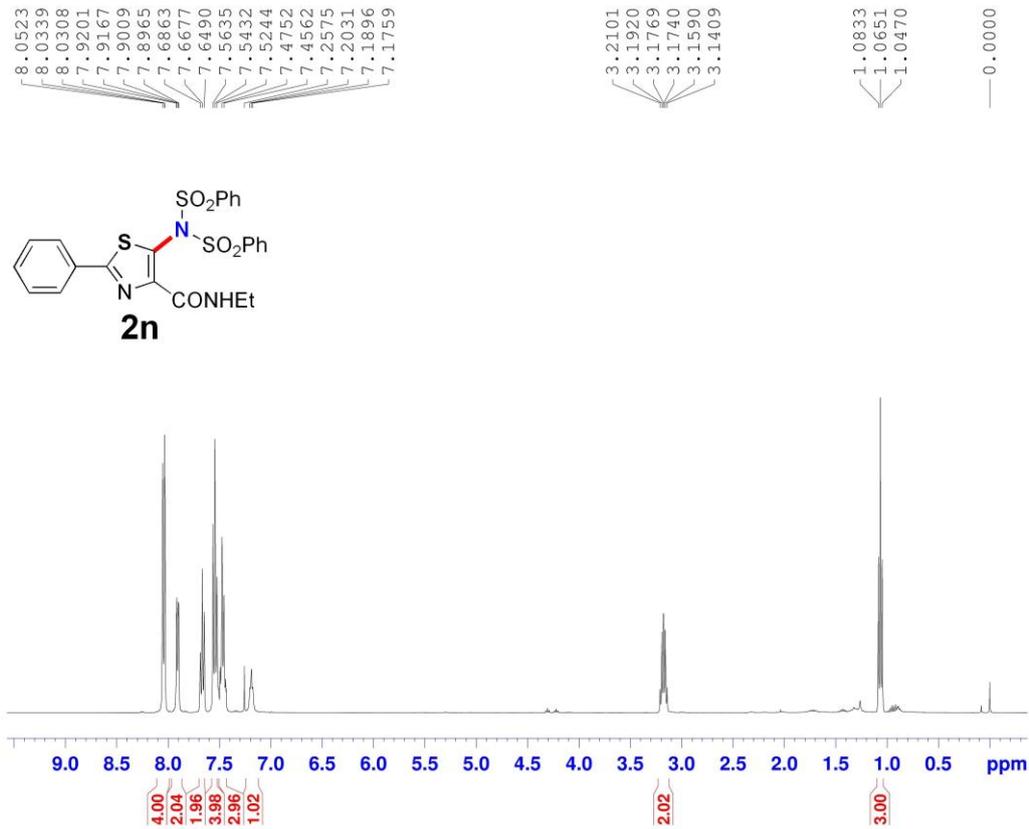
```

NAME      CLJ-WL-M209
EXPNO     2
PROCNO    1
Date_     20190109
Time      9.23
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         200
DS         0
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.361988 sec
RG         194.26
DW         20.800 usec
DE         6.50 usec
TE         300.2 K
D1         2.00000000 sec
D11        0.20000000 sec
TDO        1
    
```

```

===== CHANNEL f1 =====
SFO1     100.6228293 MHz
NUC1      13C
P1        8.54 usec
SI        32768
SF        100.6127761 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
    
```

Electronic Supplementary Information

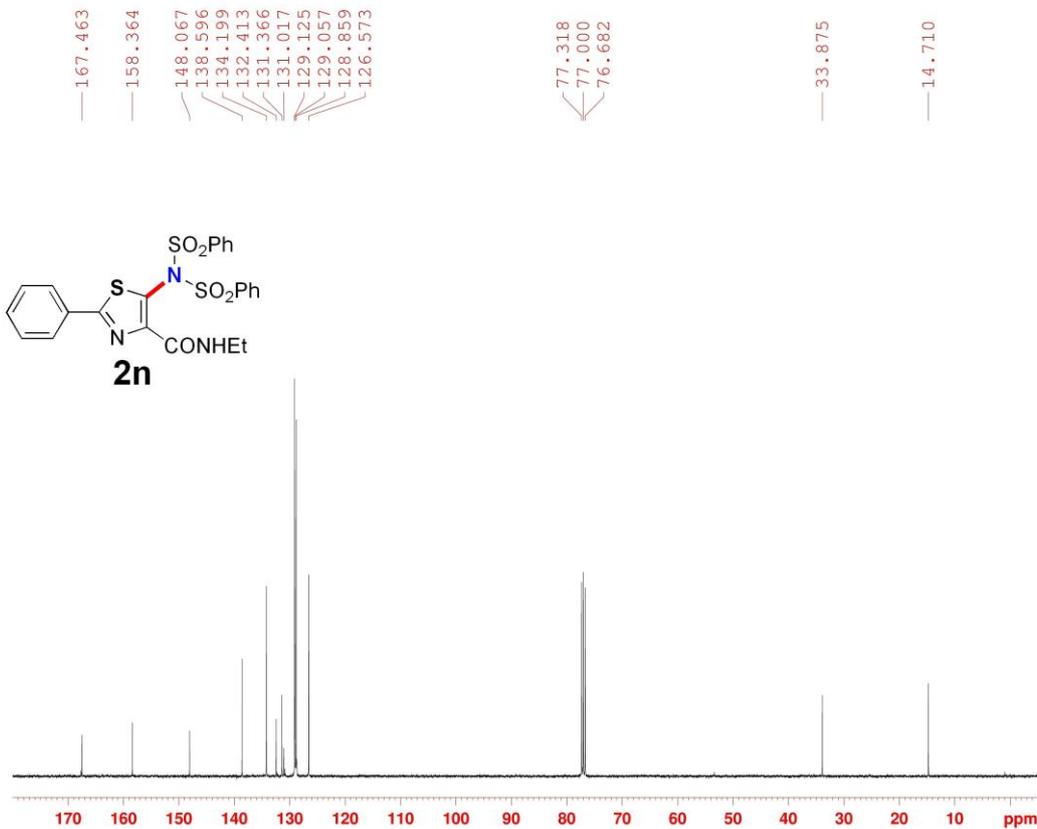


```

NAME      CLJ-WL-M176
EXPNO     1
PROCNO    1
Date_     20181209
Time      15.02
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD        65536
SOLVENT   CDCl3
NS        12
DS        0
SWH       8012.820 Hz
FIDRES    0.122266 Hz
AQ        4.0894966 sec
RG        23.9
DW        62.400 usec
DE        6.50 usec
TE        300.1 K
D1        1.00000000 sec
TDO       1
    
```

```

===== CHANNEL f1 =====
SFO1     400.1324710 MHz
NUC1     1H
P1       8.04 usec
SI       65536
SF       400.1300106 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
    
```



```

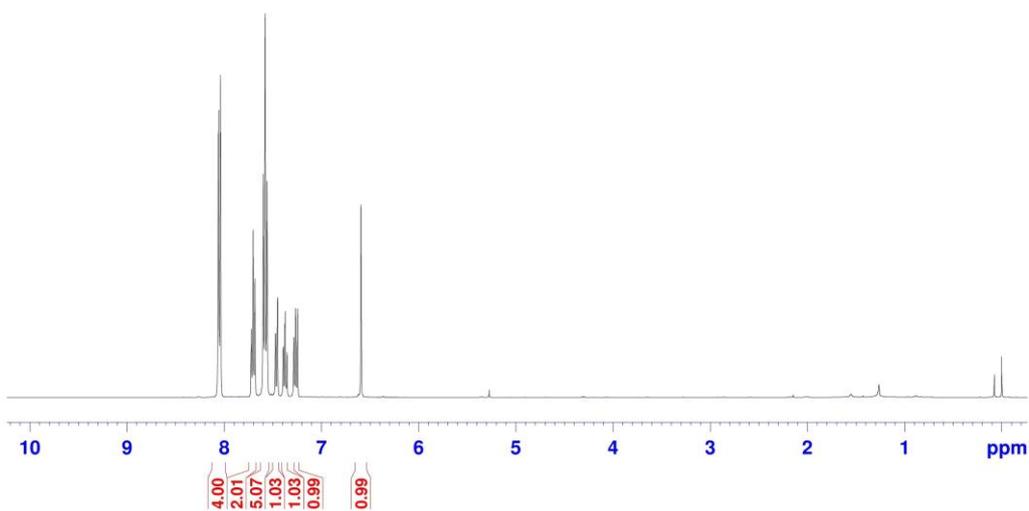
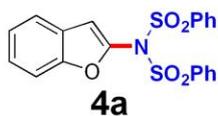
NAME      CLJ-WL-M176
EXPNO     2
PROCNO    1
Date_     20181209
Time      15.22
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS        339
DS        0
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ        1.361988 sec
RG        194.26
DW        20.800 usec
DE        6.50 usec
TE        300.1 K
D1        2.00000000 sec
D11       0.23000000 sec
TDO       1
    
```

```

===== CHANNEL f1 =====
SFO1     100.6228293 MHz
NUC1     13C
P1       8.54 usec
SI       32768
SF       100.6127776 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
    
```

Electronic Supplementary Information

8.0572
8.0389
8.0357
7.7214
7.7027
7.6869
7.6841
7.6814
7.5988
7.5784
7.5597
7.4738
7.4723
7.4531
7.4515
7.3948
7.3917
7.3766
7.3737
7.3559
7.3528
7.2853
7.2829
7.2656
7.2445
6.5928
6.5909



```

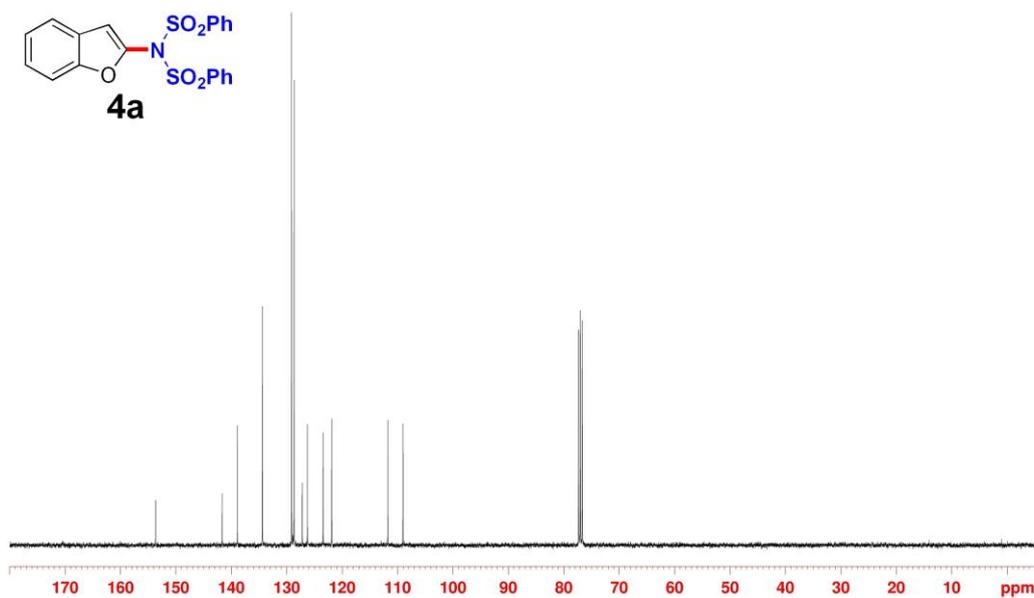
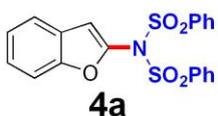
NAME      CLJ-WL-M196
EXPNO    1
PROCNO    1
Date_    20181225
Time     9.44
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD        65536
SOLVENT   CDCl3
NS        12
DS        0
SWH       8012.820 Hz
FIDRES    0.122266 Hz
AQ        4.0894966 sec
RG        37.75
DW        62.400 usec
DE        6.50 usec
TE        300.1 K
D1        1.00000000 sec
D11       0
TDO       1
    
```

```

----- CHANNEL f1 -----
SF01     400.1324710 MHz
NUC1      1H
P1        8.54 usec
SI        65536
SF        400.1300157 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
    
```

153.665
141.645
138.904
134.385
129.125
128.684
127.180
126.269
123.426
121.892
111.771
109.017

77.318
77.000
76.682



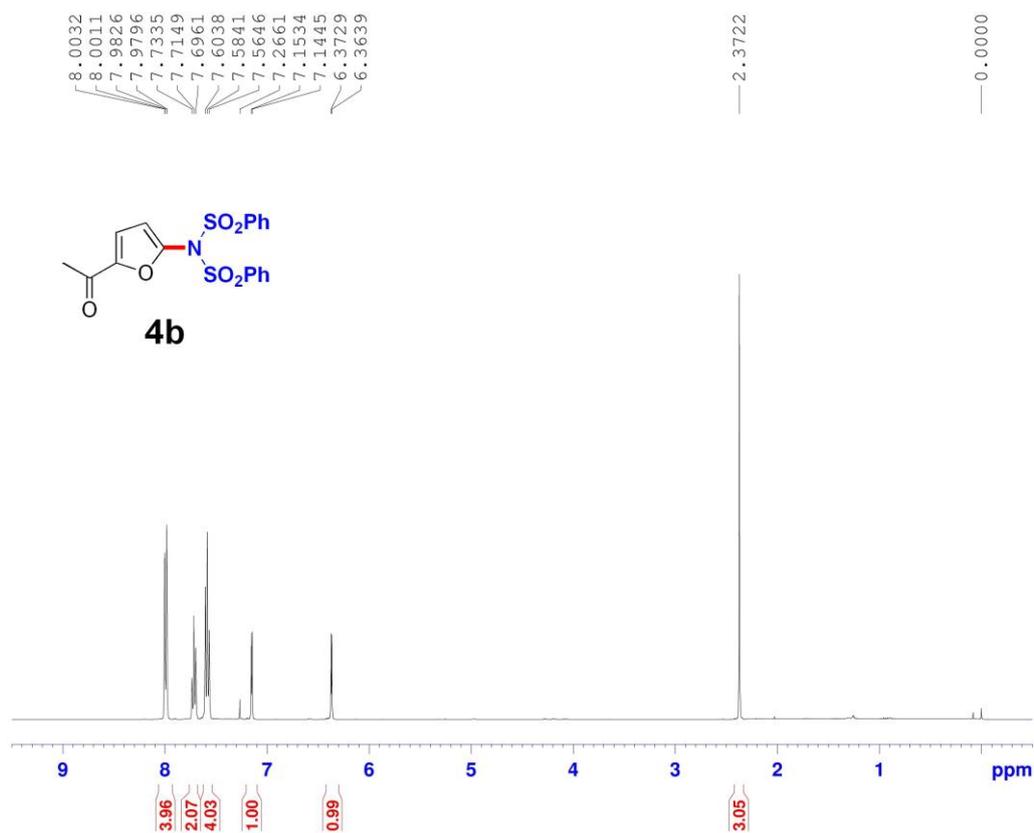
```

NAME      CLJ-WL-M196
EXPNO    4
PROCNO    1
Date_    20181228
Time     8.97
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS        200
DS        0
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ        1.361988 sec
RG        194.26
DW        20.800 usec
DE        6.50 usec
TE        300.1 K
D1        2.00000000 sec
D11       0.23000000 sec
TDO       1
    
```

```

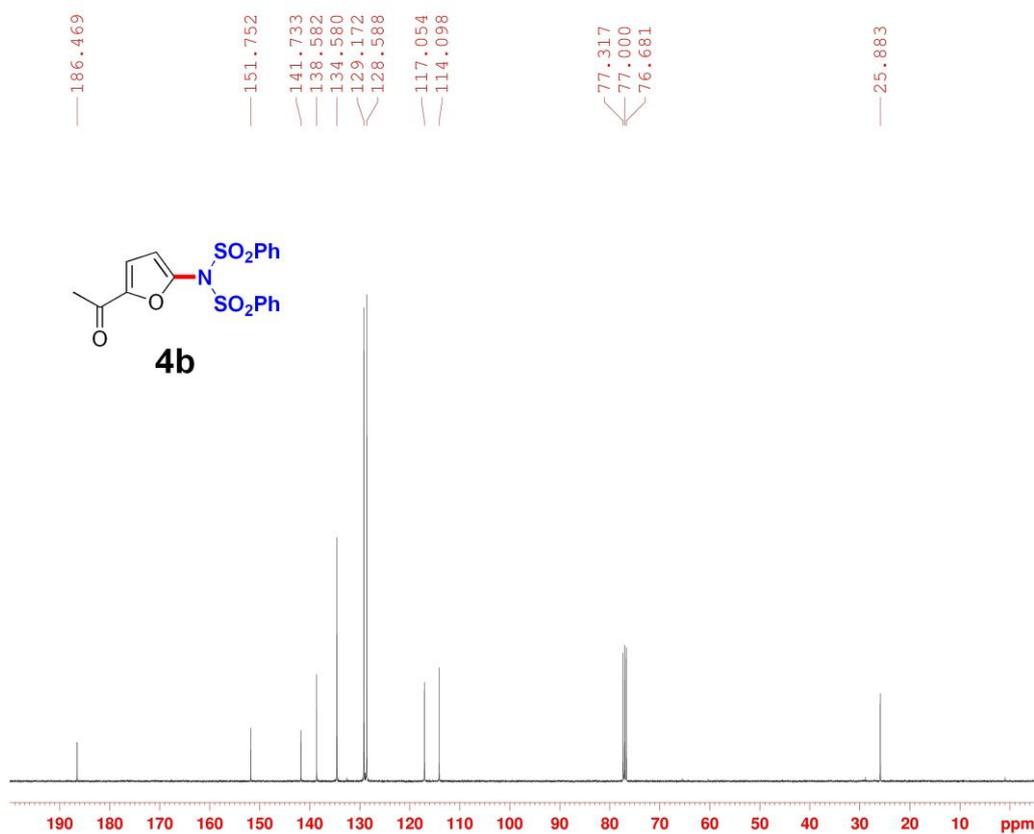
----- CHANNEL f1 -----
SF01     100.6228293 MHz
NUC1      13C
P1        8.54 usec
SI        32768
SF        100.6127746 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
    
```

Electronic Supplementary Information



```

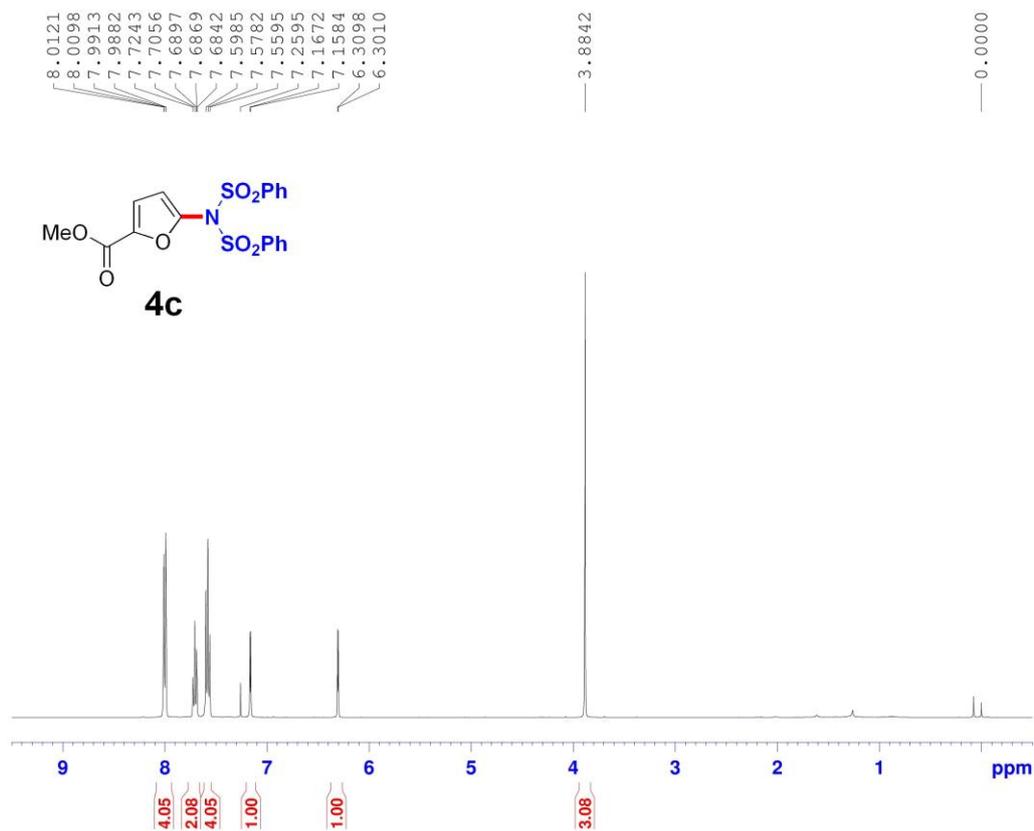
NAME      CLJ-WL-M193
EXPNO     1
PROCNO    1
Date_     20181225
Time      22.20
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         12
DS         0
SWH        8012.820 Hz
FIDRES     0.122266 Hz
AQ         4.0894966 sec
RG         23.9
DW         62.400 usec
DE         6.50 usec
TE         300.1 K
D1         1.00000000 sec
TDO        1
----- CHANNEL f1 -----
SF01      400.1324710 MHz
NUC1       1H
P1         8.04 usec
SI         65536
SF         400.1300068 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
    
```



```

NAME      CLJ-WL-M193
EXPNO     1
PROCNO    1
Date_     20181225
Time      22.30
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         512
DS         0
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.361988 sec
RG         194.26
DW         20.800 usec
DE         6.50 usec
TE         300.1 K
D1         2.00000000 sec
D11        0.23000000 sec
TDO        1
----- CHANNEL f1 -----
SF01      100.6228293 MHz
NUC1       13C
P1         8.54 usec
SI         32768
SF         100.6127774 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
    
```

Electronic Supplementary Information

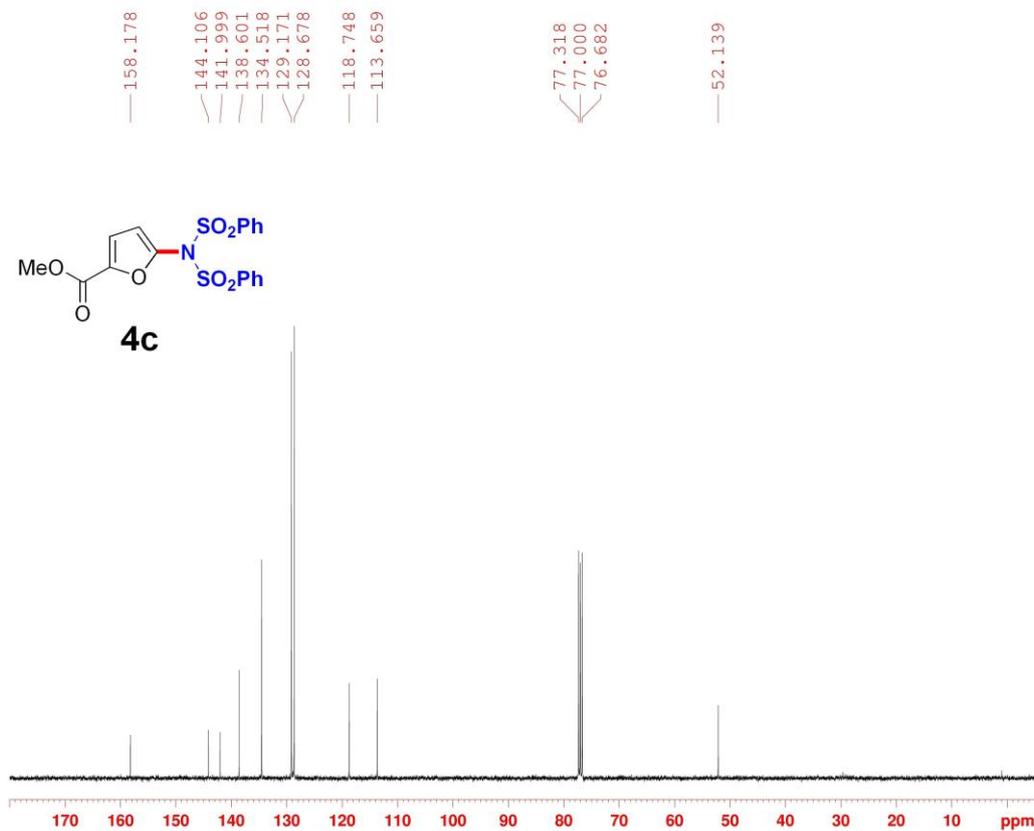


```

NAME      CLJ-WL-M194
EXPNO    1
PROCNO    1
Date_    20181226
Time     9.11
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD        65536
SOLVENT   CDCl3
NS         12
DS         0
SWH        8012.820 Hz
FIDRES     0.122266 Hz
AQ         4.0894966 sec
RG         51.19
DW         62.400 usec
DE         6.50 usec
TE         300.2 K
D1         1.00000000 sec
TDO        1
    
```

```

----- CHANNEL f1 -----
SFO1     400.1324710 MHz
NUC1      1H
P1        8.04 usec
SI        65536
SF        400.1300097 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
    
```



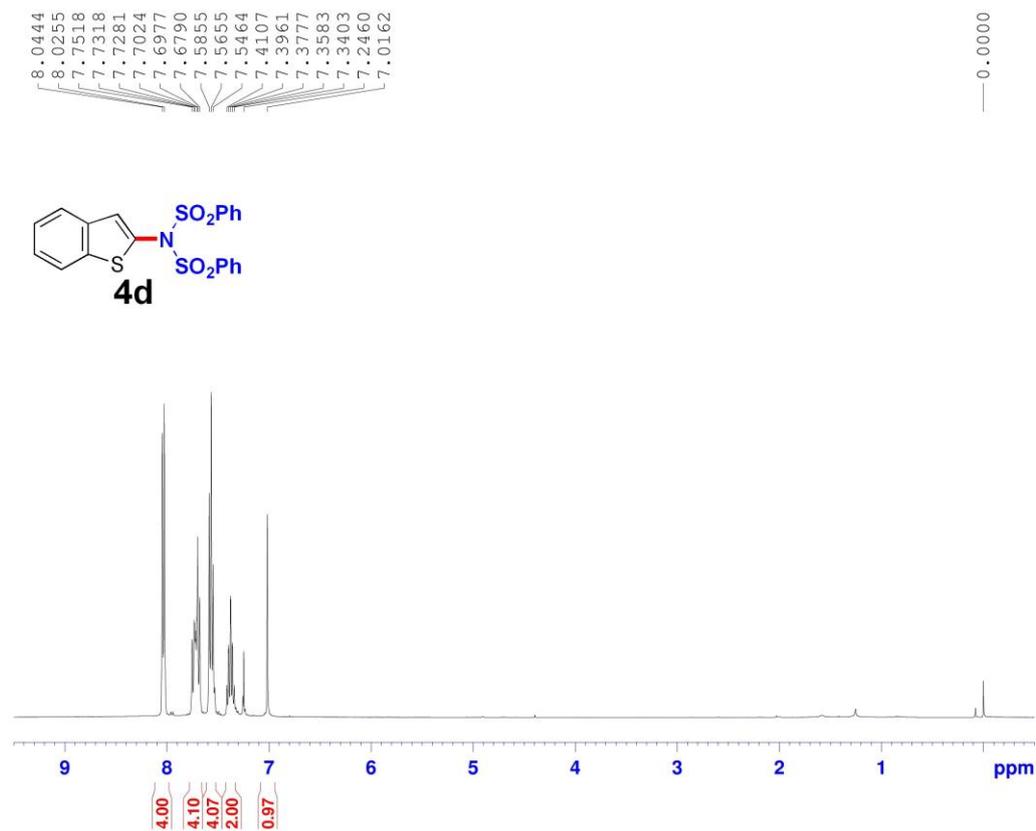
```

NAME      CLJ-WL-M194
EXPNO    2
PROCNO    1
Date_    20181226
Time     9.14
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS         154
DS         0
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.361988 sec
RG         194.26
DW         20.800 usec
DE         6.50 usec
TE         300.2 K
D1         2.00000000 sec
D11       0.20000000 sec
TDO        1
    
```

```

----- CHANNEL f1 -----
SFO1     100.6228293 MHz
NUC1      13C
P1        8.54 usec
SI        32768
SF        100.6127742 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
    
```

Electronic Supplementary Information

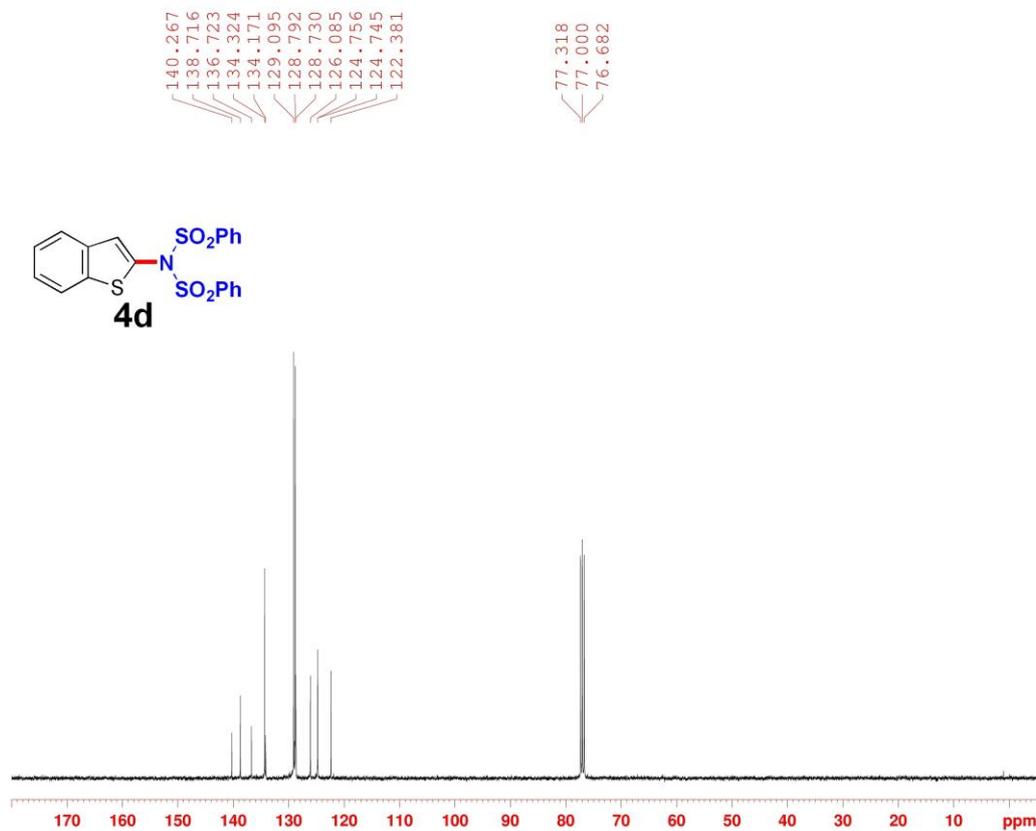


```

NAME      CLJ-WL-M255
EXPNO    1
PROCNO   1
Date_    20190310
Time     4.53
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zg30
TD       65536
SOLVENT  CDCl3
NS       8
DS       0
SWH      8012.820 MHz
FIDRES   0.122266 MHz
AQ       4.0894966 sec
RG       70.36
DW       62.400 usec
DE       6.50 usec
TE       300.7 K
D1       1.00000000 sec
TDO      1
    
```

```

===== CHANNEL f1 =====
SF01    400.1324710 MHz
NUC1     1H
P1       8.04 usec
SI       65536
SF       400.1300150 MHz
WDW      EM
SSB      0
LB       0.30 MHz
GB       0
PC       1.00
    
```



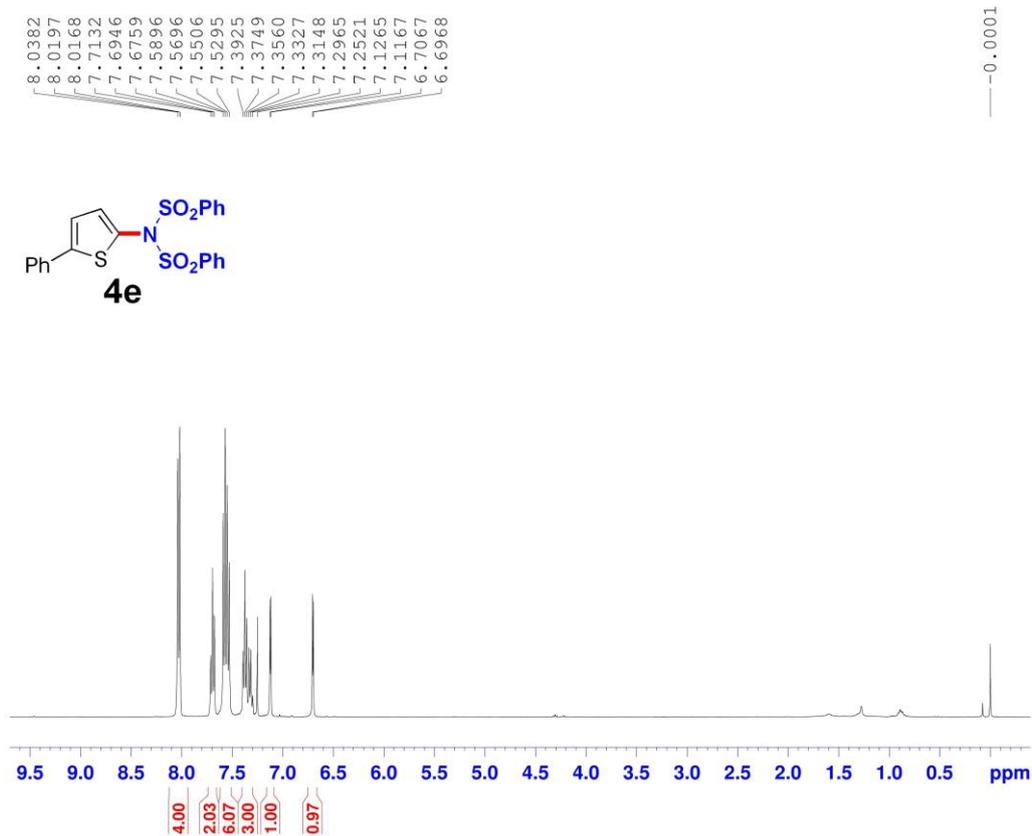
```

NAME      CLJ-WL-M255
EXPNO    2
PROCNO   1
Date_    20190310
Time     5.23
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       512
DS       0
SWH      24038.461 MHz
FIDRES   0.366798 MHz
AQ       1.361988 sec
RG       194.26
DW       20.800 usec
DE       6.50 usec
TE       300.6 K
D1       2.00000000 sec
D11      0.20000000 sec
TDO      1
    
```

```

===== CHANNEL f1 =====
SF01    100.6228293 MHz
NUC1     13C
P1       8.54 usec
SI       32768
SF       100.6127719 MHz
WDW      EM
SSB      0
LB       1.00 MHz
GB       0
PC       1.40
    
```

Electronic Supplementary Information

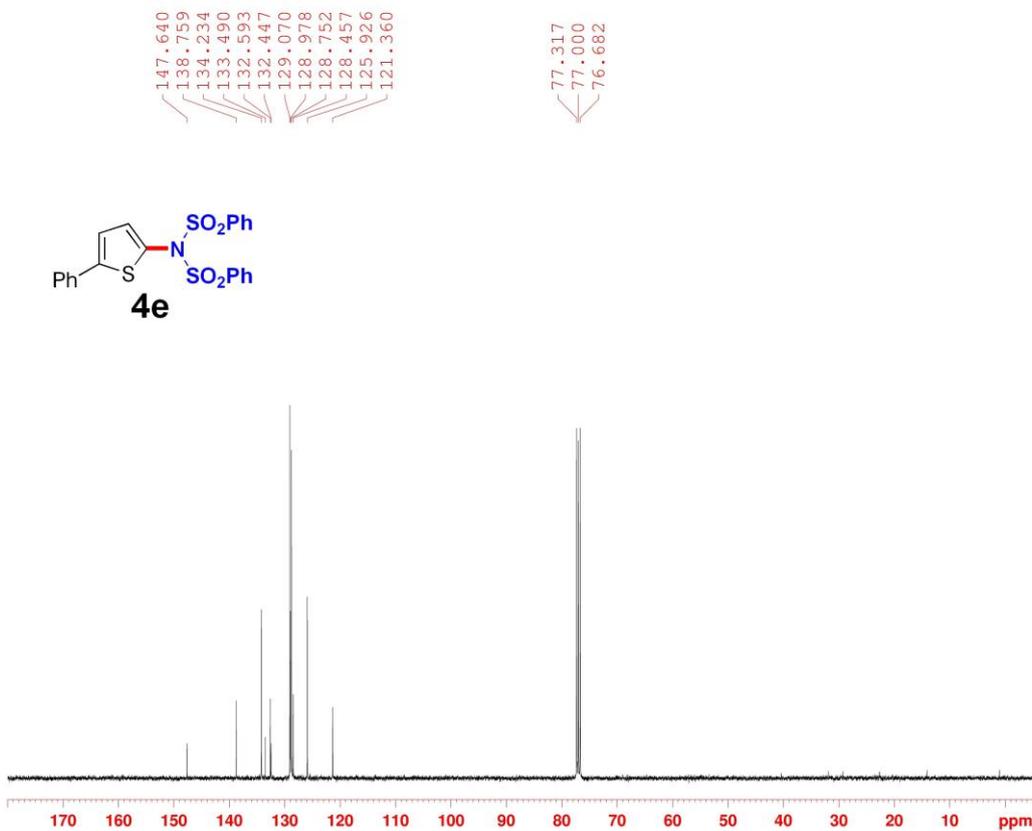


```

NAME      CLJ-WL-M175
EXPNO    1
PROCNO    1
Date_    20181209
Time     14.28
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD        65536
SOLVENT   CDCl3
NS        12
DS        0
SWH       8012.820 Hz
FIDRES    0.122266 Hz
AQ        4.0894966 sec
RG        63
DW        62.400 usec
DE        6.50 usec
TE        300.1 K
D1        1.00000000 sec
TDO       1
    
```

```

===== CHANNEL f1 =====
SF01     400.1324710 MHz
NUC1     1H
P1       8.04 usec
SI       65536
SF       400.1300128 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
    
```



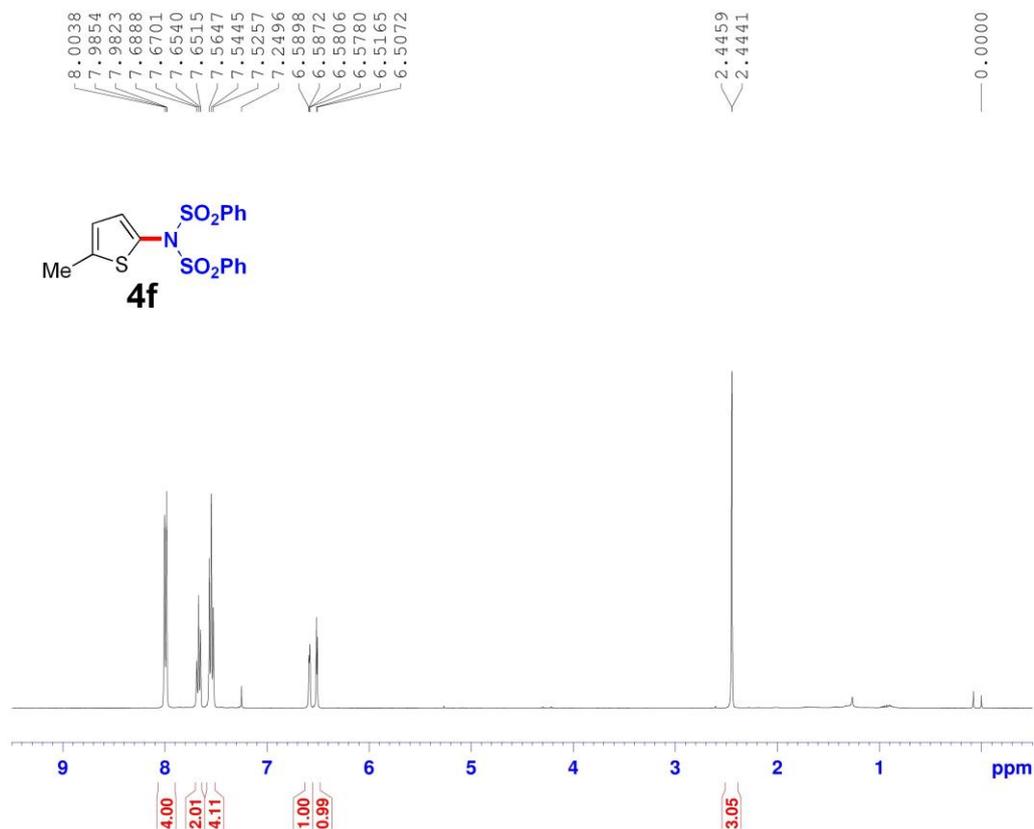
```

NAME      CLJ-WL-M175
EXPNO    2
PROCNO    1
Date_    20181209
Time     14.55
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS        466
DS        0
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ        1.361988 sec
RG        194.26
DW        20.800 usec
DE        6.50 usec
TE        300.1 K
D1        2.00000000 sec
D11      0.23000000 sec
TDO       1
    
```

```

===== CHANNEL f1 =====
SF01     100.6228293 MHz
NUC1     13C
P1       8.54 usec
SI       32768
SF       100.6127721 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
    
```

Electronic Supplementary Information

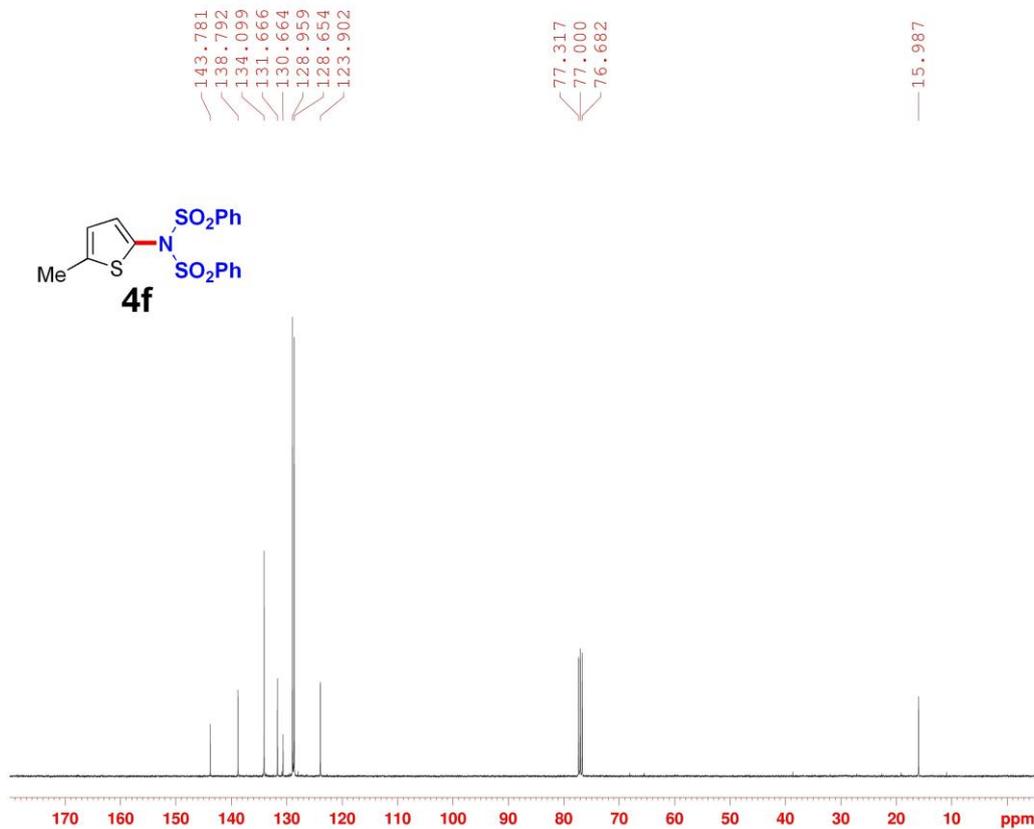


```

NAME      CLJ-WL-M173
EXPNO    1
PROCNO   1
Date_    20181209
Time     1.36
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zg30
TD       65536
SOLVENT  CDCl3
NS       12
DS       0
SWH      8012.820 Hz
FIDRES   0.122266 Hz
AQ       4.0894866 sec
RG       36.4
DW       62.400 usec
DE       6.50 usec
TE       300.2 K
D1       1.00000000 sec
TDO      1
    
```

```

===== CHANNEL f1 =====
SF01    400.1324710 MHz
NUC1     1H
P1       8.04 usec
SI       65536
SF       400.1300137 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
    
```



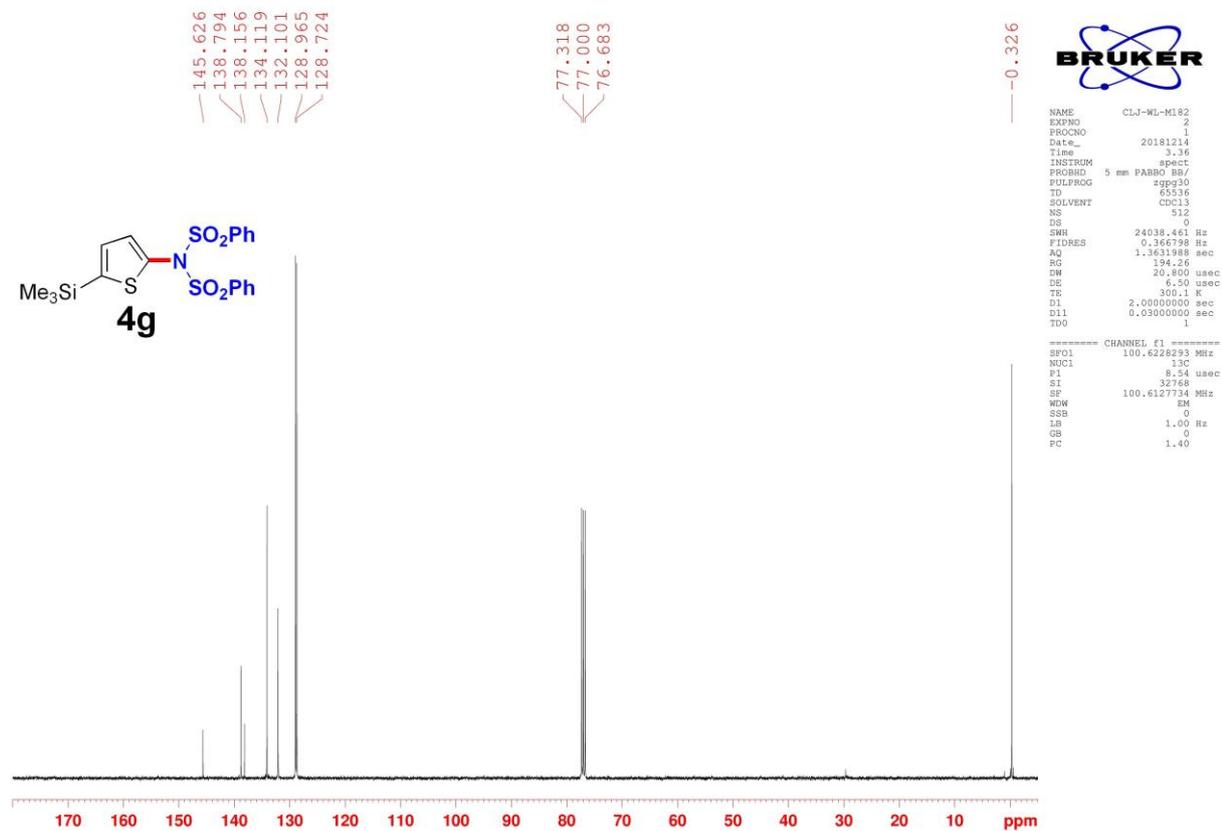
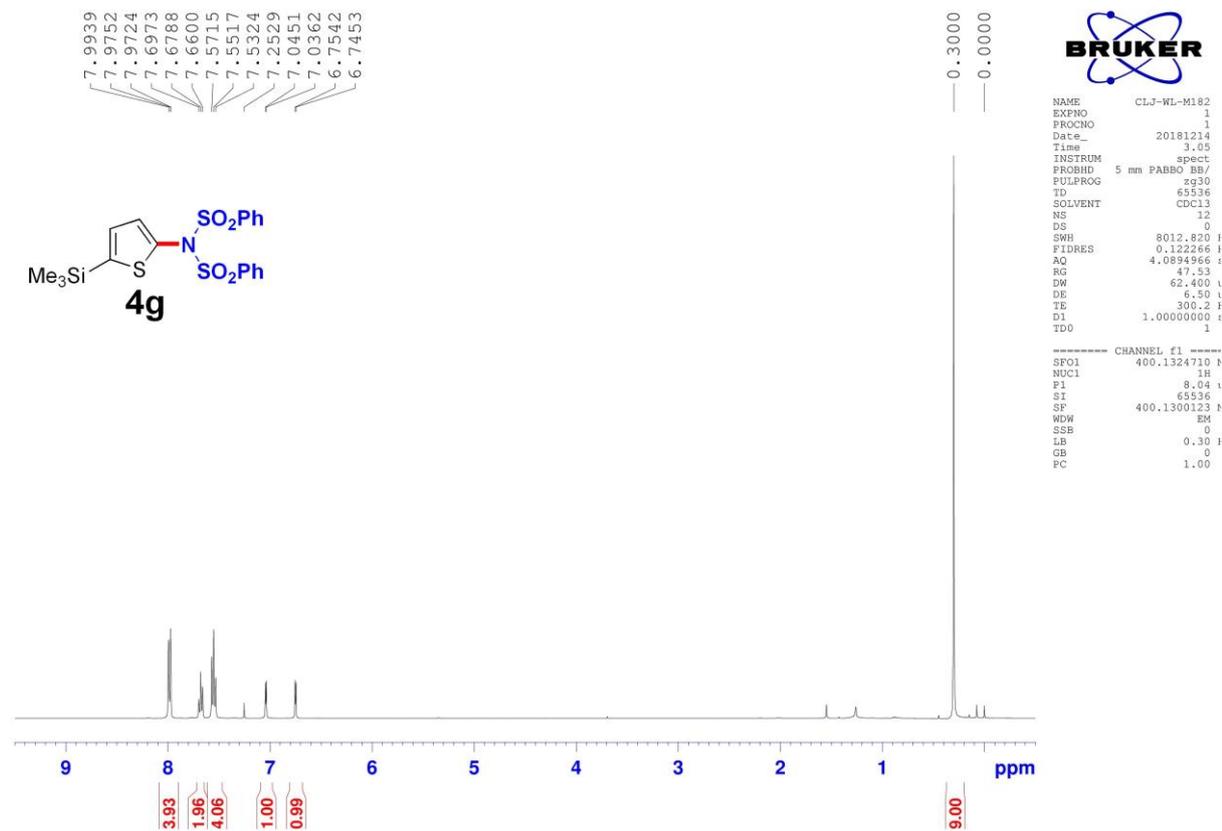
```

NAME      CLJ-WL-M173
EXPNO    2
PROCNO   1
Date_    20181209
Time     2.06
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       512
DS       0
SWH      24038.461 Hz
FIDRES   0.366798 Hz
AQ       1.361988 sec
RG       194.26
DW       20.800 usec
DE       6.50 usec
TE       300.1 K
D1       2.00000000 sec
D11      0.23000000 sec
TDO      1
    
```

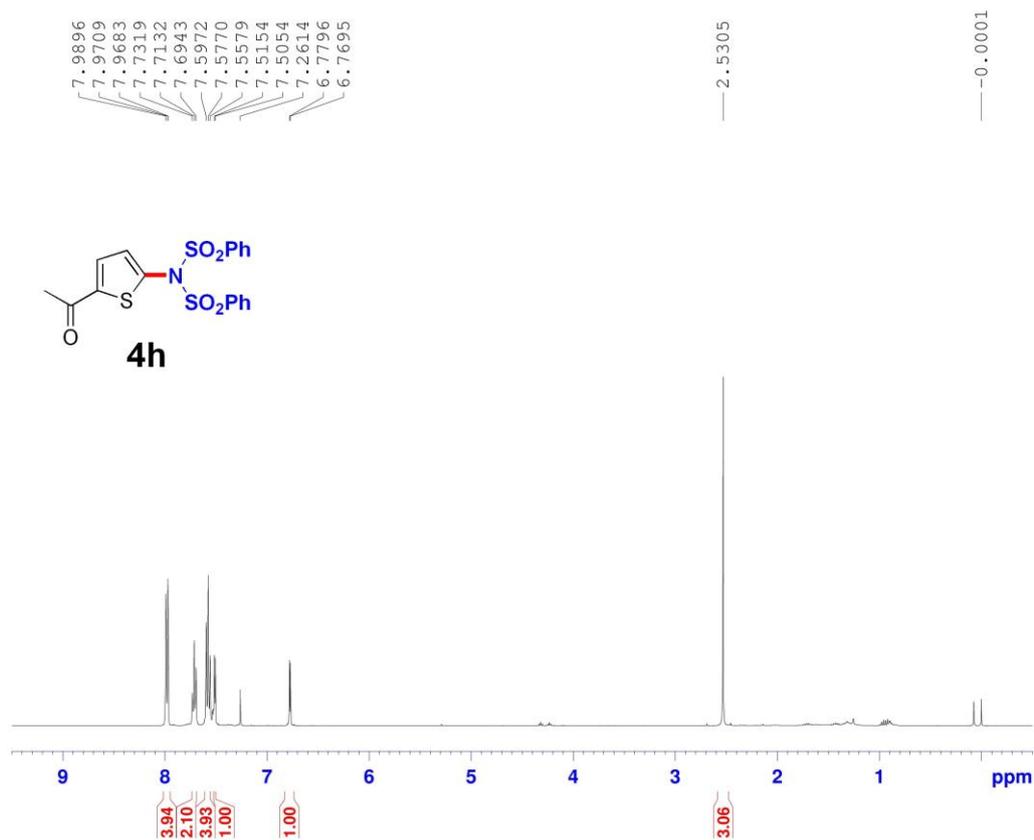
```

===== CHANNEL f1 =====
SF01    100.6228293 MHz
NUC1     13C
P1       8.54 usec
SI       32768
SF       100.6127767 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
    
```

Electronic Supplementary Information



Electronic Supplementary Information

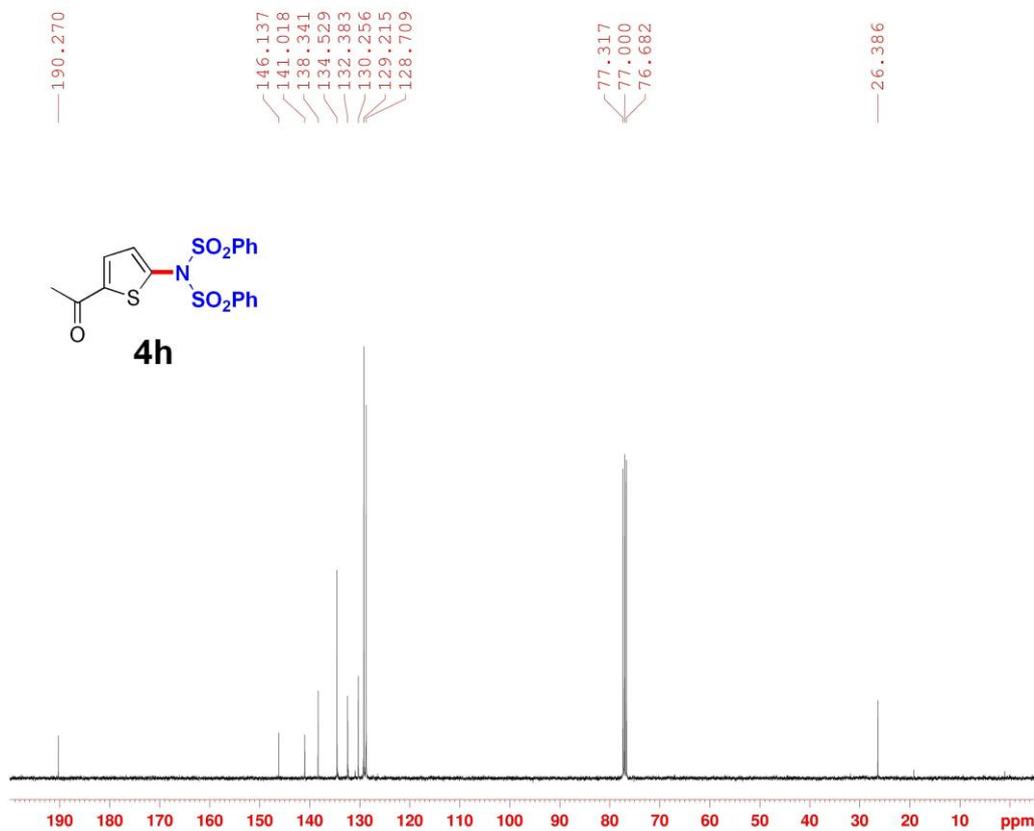


```

NAME      CLJ-WL-M172
EXPNO    1
PROCNO    1
Date_    20181205
Time     8.25
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD        65536
SOLVENT   CDCl3
NS        12
DS        0
SWH       8012.820 MHz
FIDRES    0.122266 MHz
AQ        4.0894966 sec
RG        37.75
DW        62.400 usec
DE        6.50 usec
TE        300.1 K
D1        1.00000000 sec
D11       0.23000000 sec
TDO       1
    
```

```

----- CHANNEL f1 -----
SF01     400.1324710 MHz
NUC1     1H
P1       8.04 usec
SI       65536
SF       400.1300091 MHz
WDW      EM
SSB      0
LB       0.30 MHz
GB       0
PC       1.00
    
```



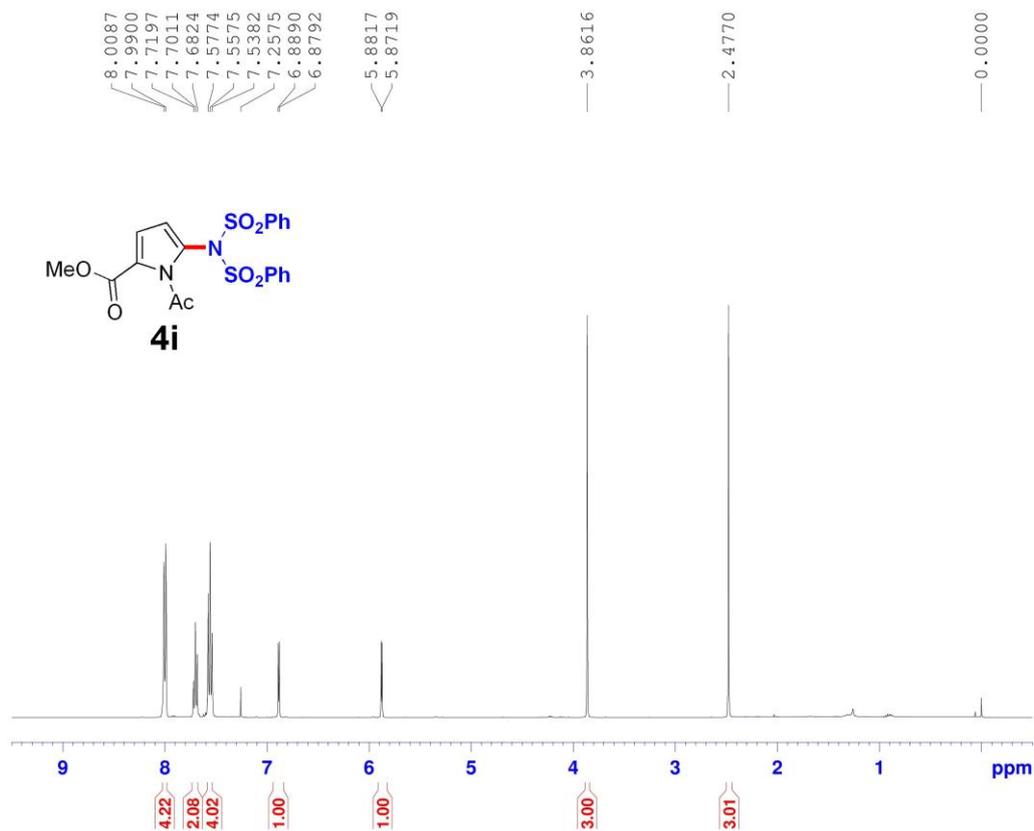
```

NAME      CLJ-WL-M172
EXPNO    2
PROCNO    1
Date_    20181205
Time     8.56
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS        512
DS        0
SWH       24038.461 MHz
FIDRES    0.366798 MHz
AQ        1.3611988 sec
RG        194.26
DW        20.800 usec
DE        6.50 usec
TE        300.1 K
D1        2.00000000 sec
D11       0.23000000 sec
TDO       1
    
```

```

----- CHANNEL f1 -----
SF01     100.6228293 MHz
NUC1     13C
P1       8.54 usec
SI       32768
SF       100.6127730 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
    
```

Electronic Supplementary Information

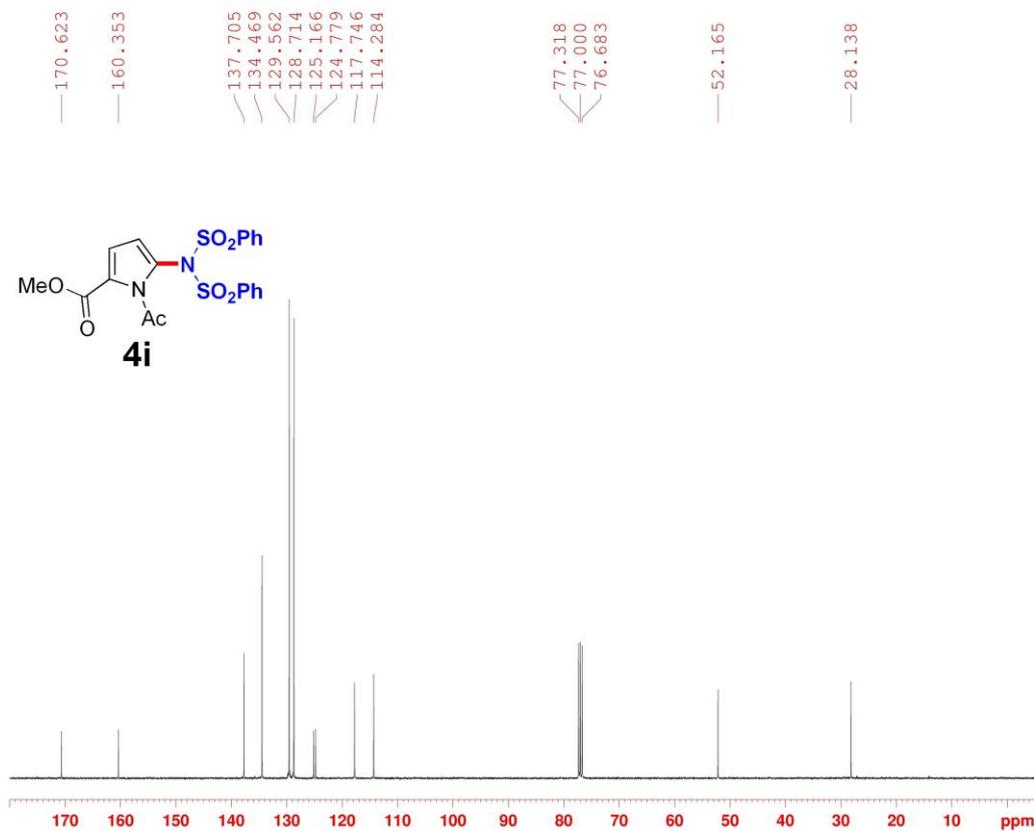


```

NAME      CLJ-WL-M244
EXPNO    1
PROCNO    1
Date_    20190302
Time     17.41
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD        65536
SOLVENT   CDC13
NS         8
DS         0
SWH       8012.820 Hz
FIDRES    0.122266 Hz
AQ        4.0894966 sec
RG        47.33
DW        62.400 usec
DE        6.50 usec
TE        300.2 K
D1        1.00000000 sec
D11       0.23000000 sec
TDO       1
    
```

```

===== CHANNEL f1 =====
SFO1     400.1324710 MHz
NUC1     1H
P1       8.04 usec
SI       65536
SF       400.1300104 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
    
```



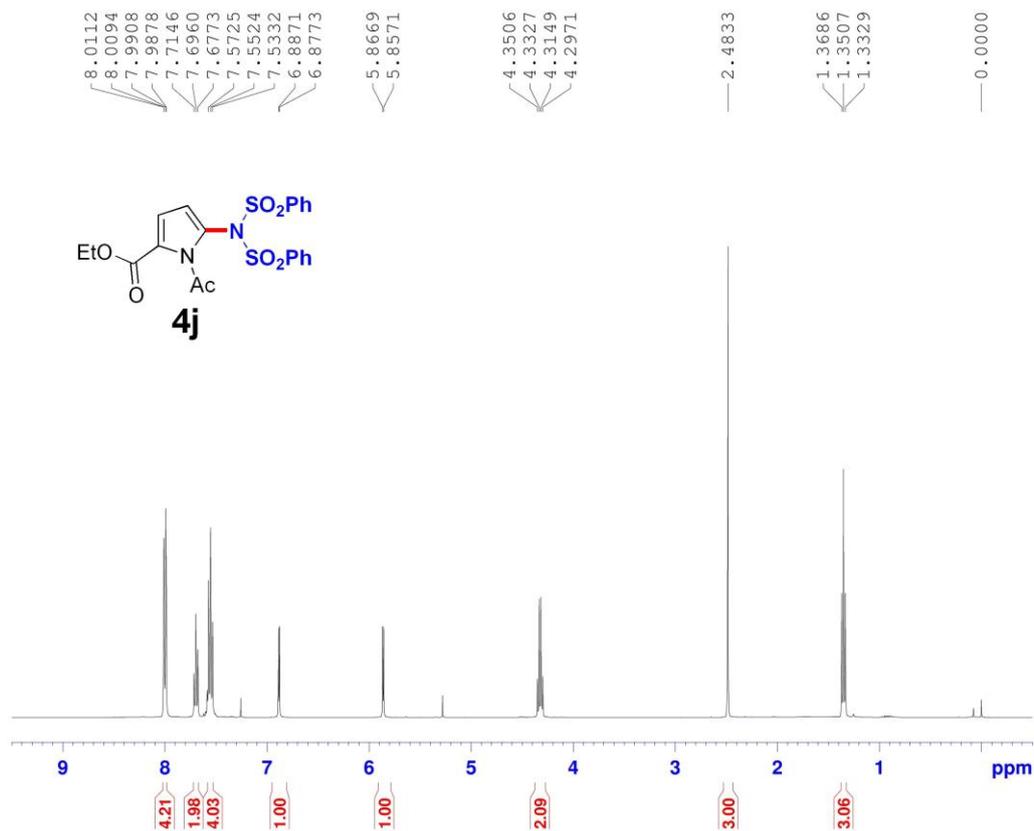
```

NAME      CLJ-WL-M230
EXPNO    2
PROCNO    1
Date_    20190124
Time     5.49
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD        65536
SOLVENT   CDC13
NS         0
DS         0
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ        1.361988 sec
RG        194.26
DW        20.800 usec
DE        6.50 usec
TE        300.2 K
D1        2.00000000 sec
D11       0.23000000 sec
TDO       1
    
```

```

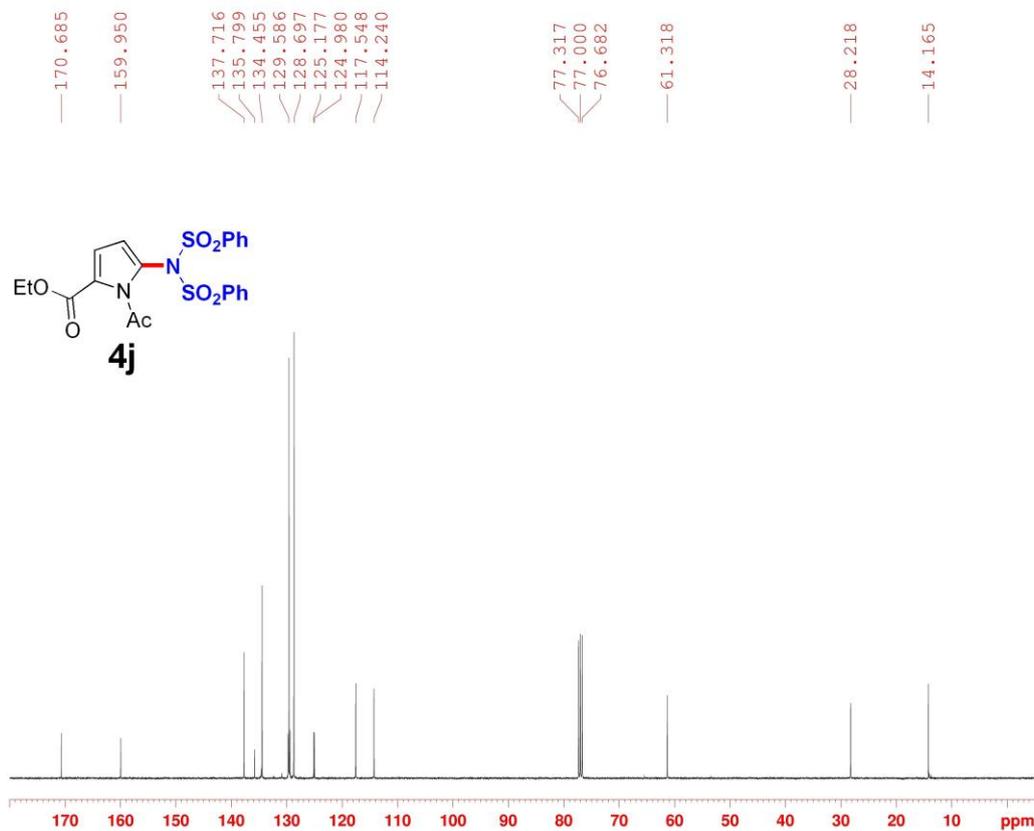
===== CHANNEL f1 =====
SFO1     100.6228293 MHz
NUC1     13C
P1       8.54 usec
SI       32768
SF       100.6127770 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
    
```

Electronic Supplementary Information



```

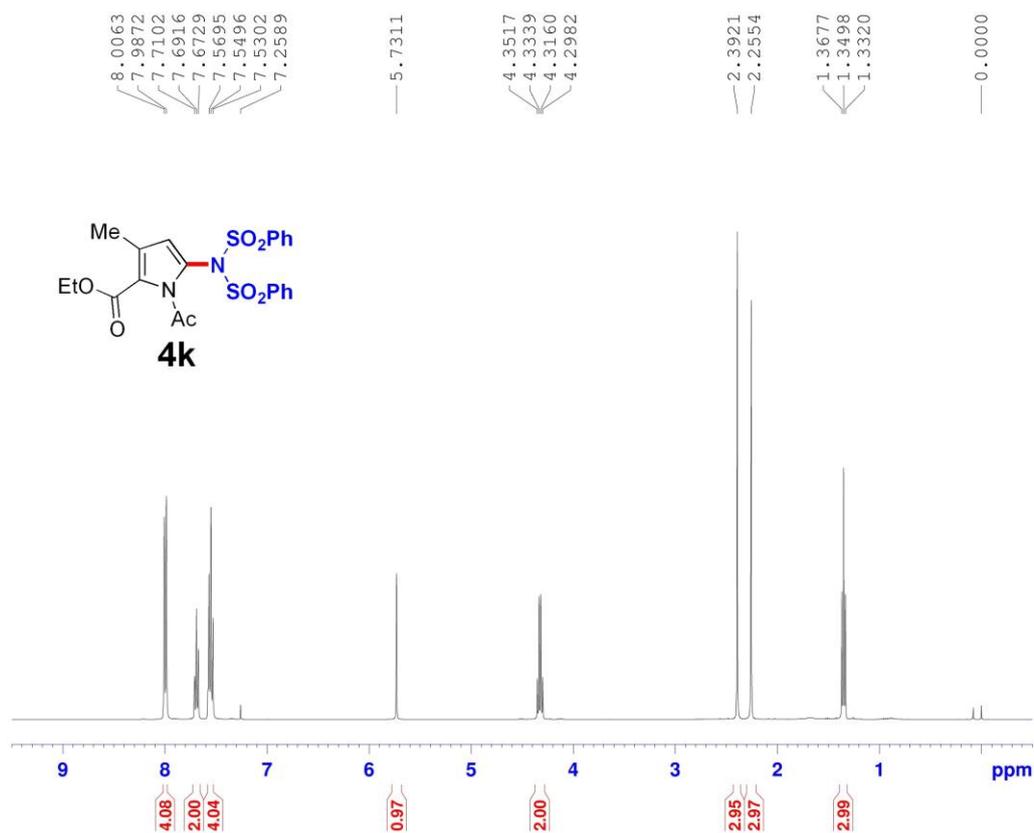
NAME      CLJ-WL-M272
EXPNO    1
PROCNO   1
Date_    20190320
Time     2.36
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zg30
TD       65536
SOLVENT  CDCl3
NS       8
DS       0
SWH      8012.820 Hz
FIDRES   0.122266 Hz
AQ       4.0894966 sec
RG       32.77
DW       62.400 usec
DE       6.50 usec
TE       300.5 K
D1       1.00000000 sec
TDO      1
===== CHANNEL f1 =====
SFO1    400.1324710 MHz
NUC1    1H
P1      8.04 usec
SI      65536
SF      400.1300107 MHz
WDW     EM
SSB     0
LB      0.30 Hz
GB      0
PC      1.00
    
```



```

NAME      CLJ-WL-M272
EXPNO    2
PROCNO   1
Date_    20190320
Time     3.06
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       512
DS       0
SWH      24038.461 Hz
FIDRES   0.366798 Hz
AQ       1.361988 sec
RG       194.26
DW       20.800 usec
DE       6.50 usec
TE       300.7 K
D1       2.00000000 sec
D11     0.23000000 sec
TDO      1
===== CHANNEL f1 =====
SFO1    100.6228293 MHz
NUC1    13C
P1      8.54 usec
SI      32768
SF      100.6127766 MHz
WDW     EM
SSB     0
LB      1.00 Hz
GB      0
PC      1.40
    
```

Electronic Supplementary Information

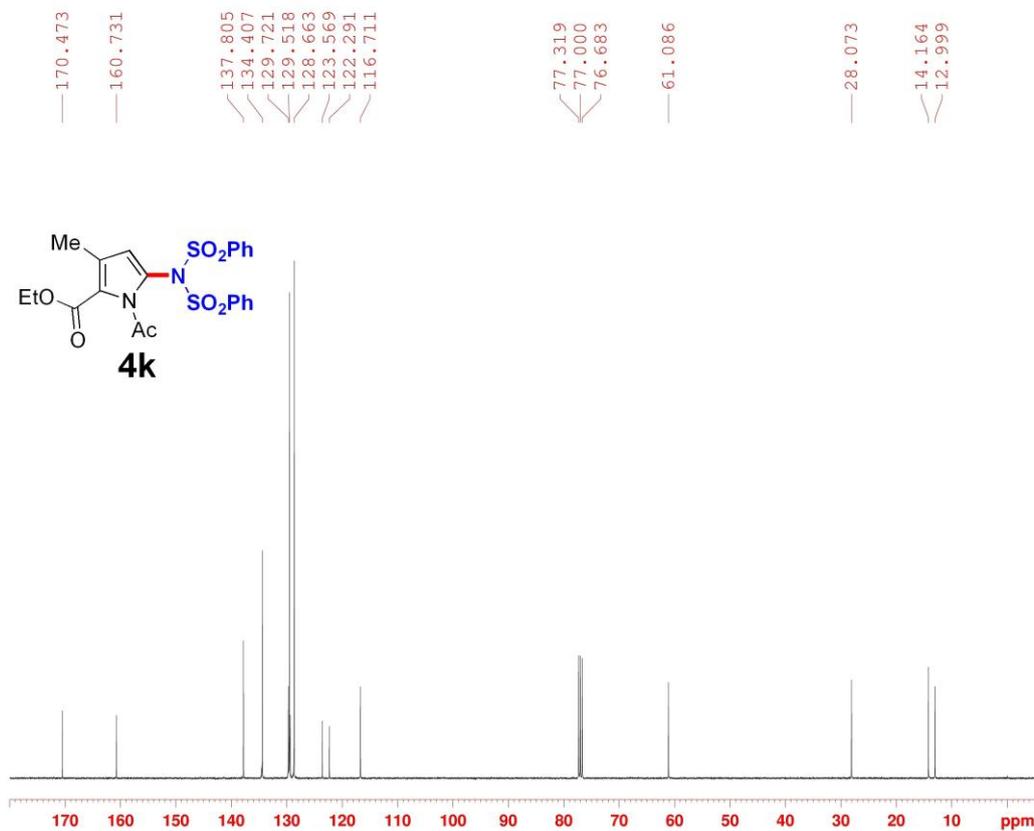


```

NAME      CLJ-WL-M274
EXPNO     1
PROCNO    1
Date_     20190322
Time      4.31
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD        65536
SOLVENT   CDCl3
NS        8
DS        0
SWH       8012.820 Hz
FIDRES    0.122266 Hz
AQ        4.0894966 sec
RG        21.11
DW        62.400 usec
DE        6.50 usec
TE        300.5 K
D1        1.00000000 sec
D11       0.23000000 sec
TDO       1
    
```

```

===== CHANNEL f1 =====
SFO1     400.1324710 MHz
NUC1     1H
P1       8.54 usec
SI       65536
SF       400.1300098 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
    
```



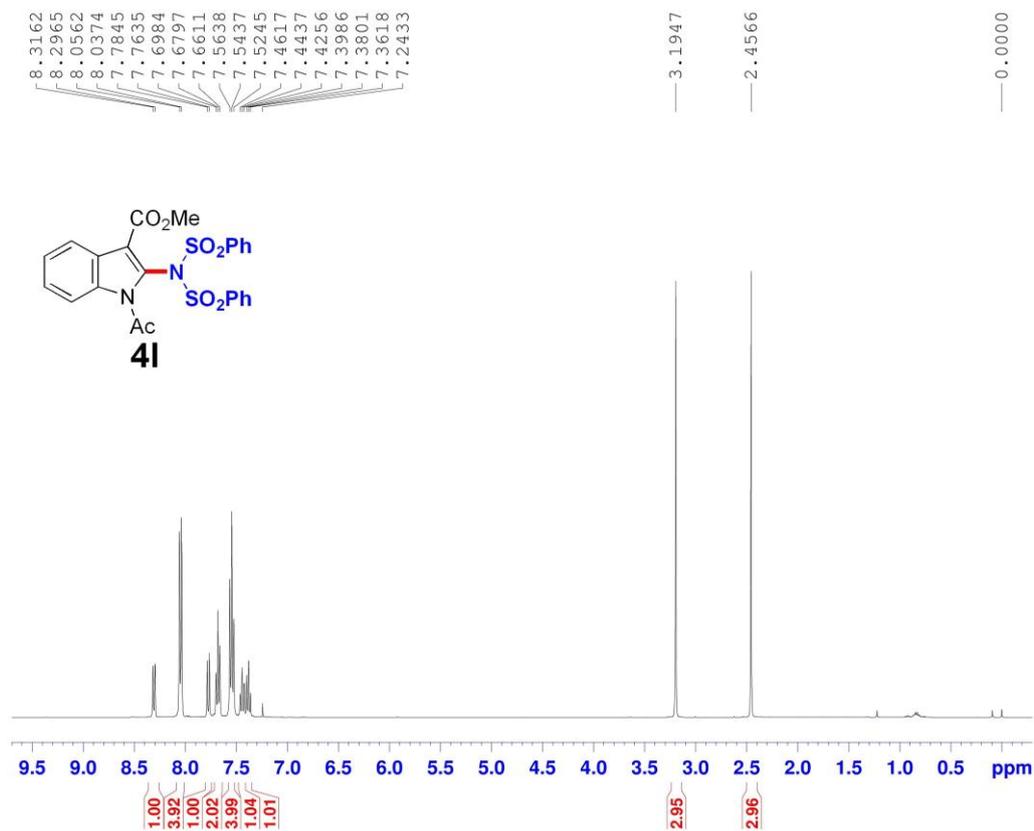
```

NAME      CLJ-WL-M274
EXPNO     2
PROCNO    1
Date_     20190322
Time      5.01
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS        512
DS        0
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ        1.361988 sec
RG        194.26
DW        20.800 usec
DE        6.50 usec
TE        300.6 K
D1        2.00000000 sec
D11       0.23000000 sec
TDO       1
    
```

```

===== CHANNEL f1 =====
SFO1     100.6228293 MHz
NUC1     13C
P1       13.00 usec
SI       32768
SF       100.6127792 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
    
```

Electronic Supplementary Information

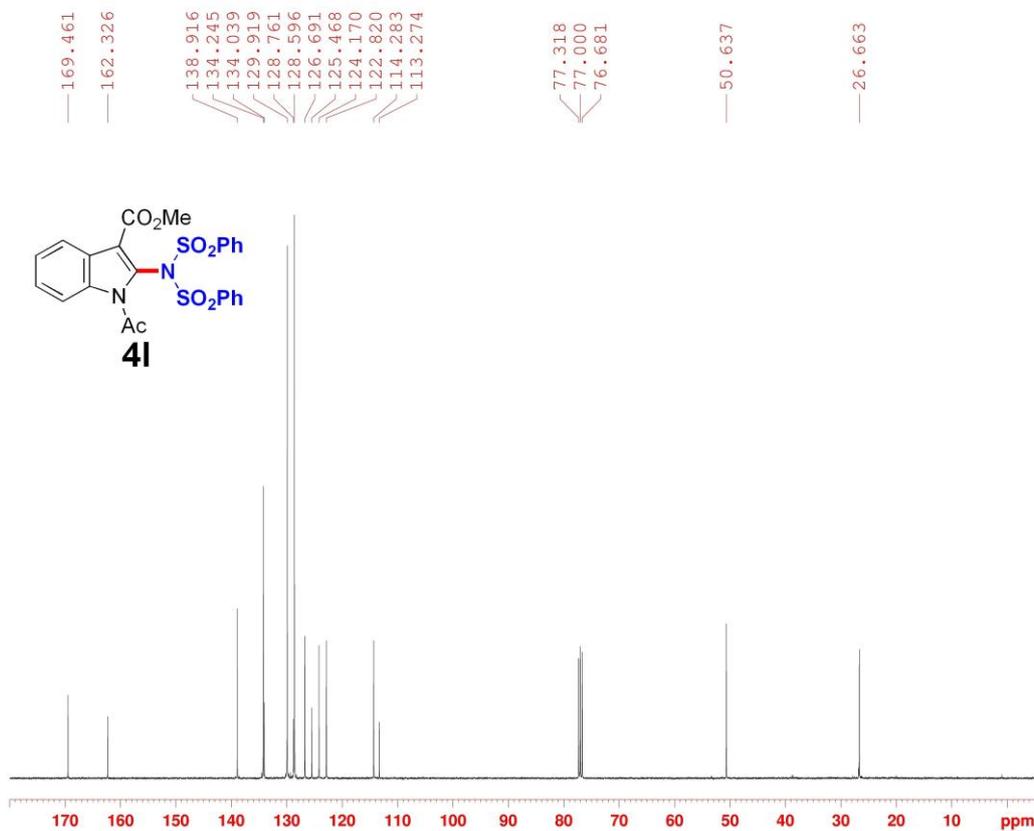


```

NAME      CLJ-WL-M250
EXPNO    1
PROCNO    1
Date_    20190307
Time     0.05
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD        65536
SOLVENT   CDCl3
NS        8
DS        0
SWH       8012.820 Hz
FIDRES    0.122266 Hz
AQ        4.0894966 sec
RG        16.03
DW        62.400 usec
DE        6.50 usec
TE        300.2 K
D1        1.00000000 sec
TDO       1
    
```

```

===== CHANNEL f1 =====
SF01     400.1324710 MHz
NUC1      1H
P1        8.04 usec
SI        65536
SF        400.1300160 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
    
```



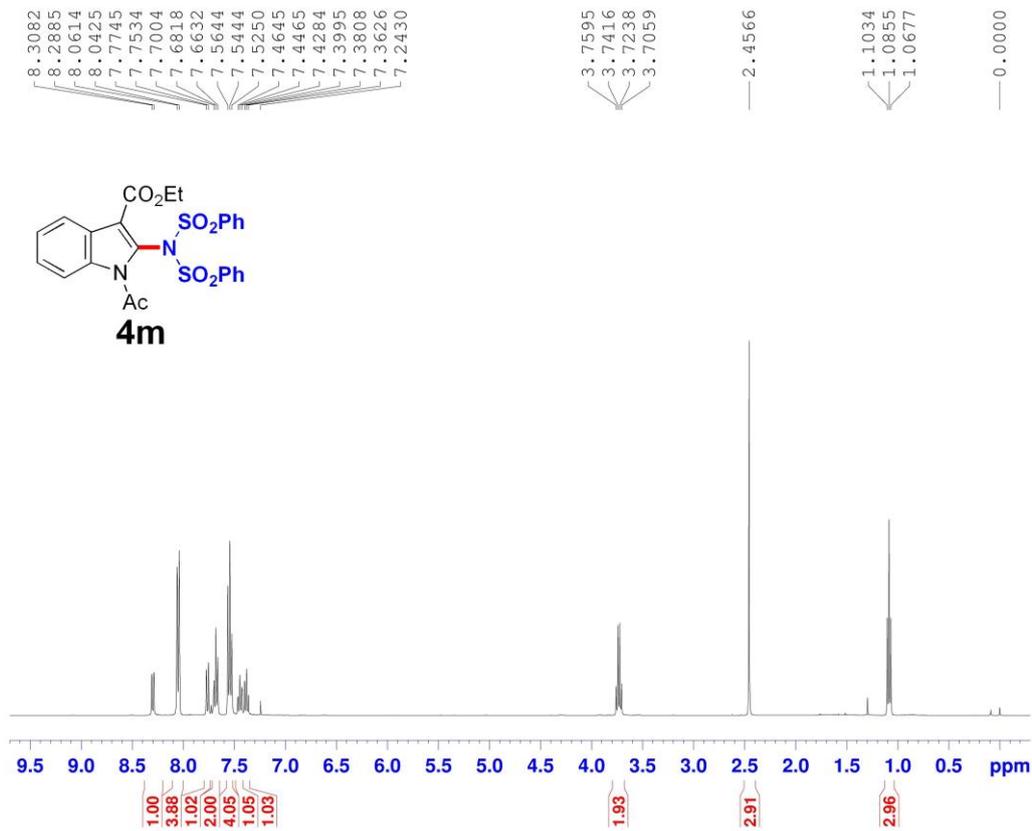
```

NAME      CLJ-WL-M250
EXPNO    2
PROCNO    1
Date_    20190307
Time     0.36
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS        512
DS        0
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ        1.361988 sec
RG        194.26
DW        20.800 usec
DE        6.50 usec
TE        300.7 K
D1        2.00000000 sec
D11       0.23000000 sec
TDO       1
    
```

```

===== CHANNEL f1 =====
SF01     100.6228293 MHz
NUC1      13C
P1        8.54 usec
SI        32768
SF        100.6127833 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
    
```

Electronic Supplementary Information

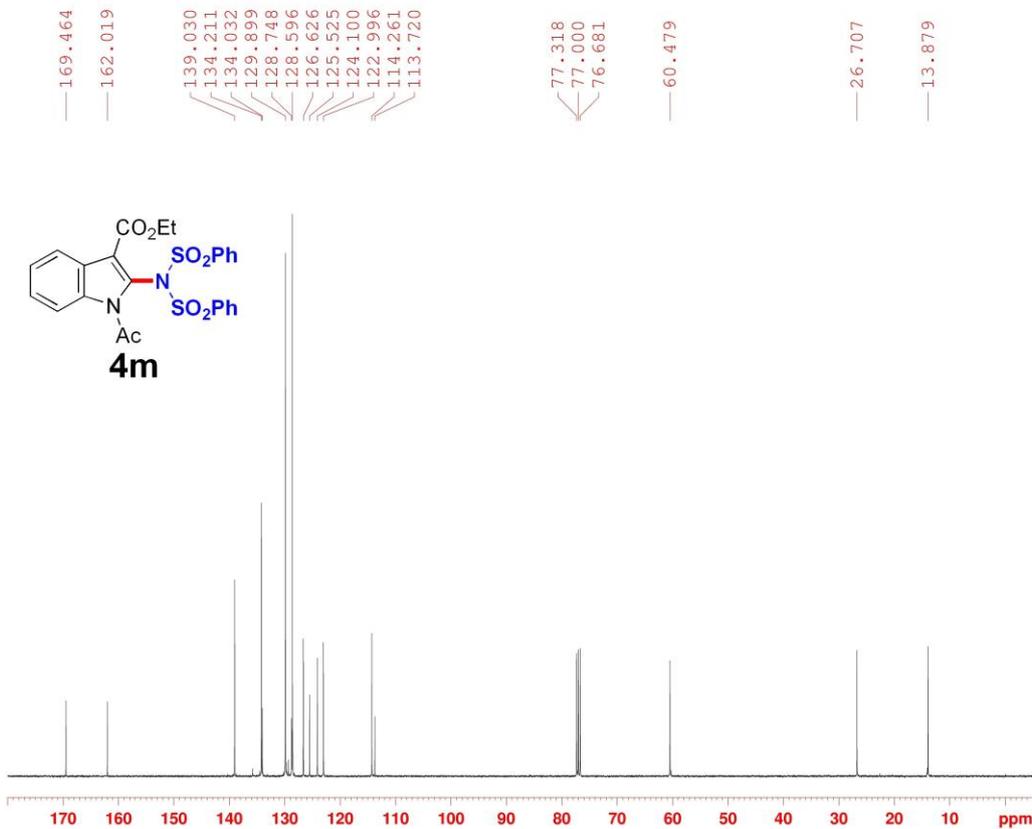


```

NAME      CLJ-WL-M267
EXPNO    2
PROCNO    1
Date_     20190316
Time      15.41
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         8
DS         0
SWH        8012.820 Hz
FIDRES     0.122266 Hz
AQ         4.0894866 sec
RG         18.6
DW         62.400 usec
DE         6.50 usec
TE         300.5 K
D1         1.00000000 sec
TDO        1
    
```

```

===== CHANNEL f1 =====
SF01     400.1324710 MHz
NUC1      1H
P1        8.04 usec
SI        65536
SF        400.1300161 MHz
WDW        EM
SSB        0
LB        0.30 Hz
GB        0
PC        1.00
    
```



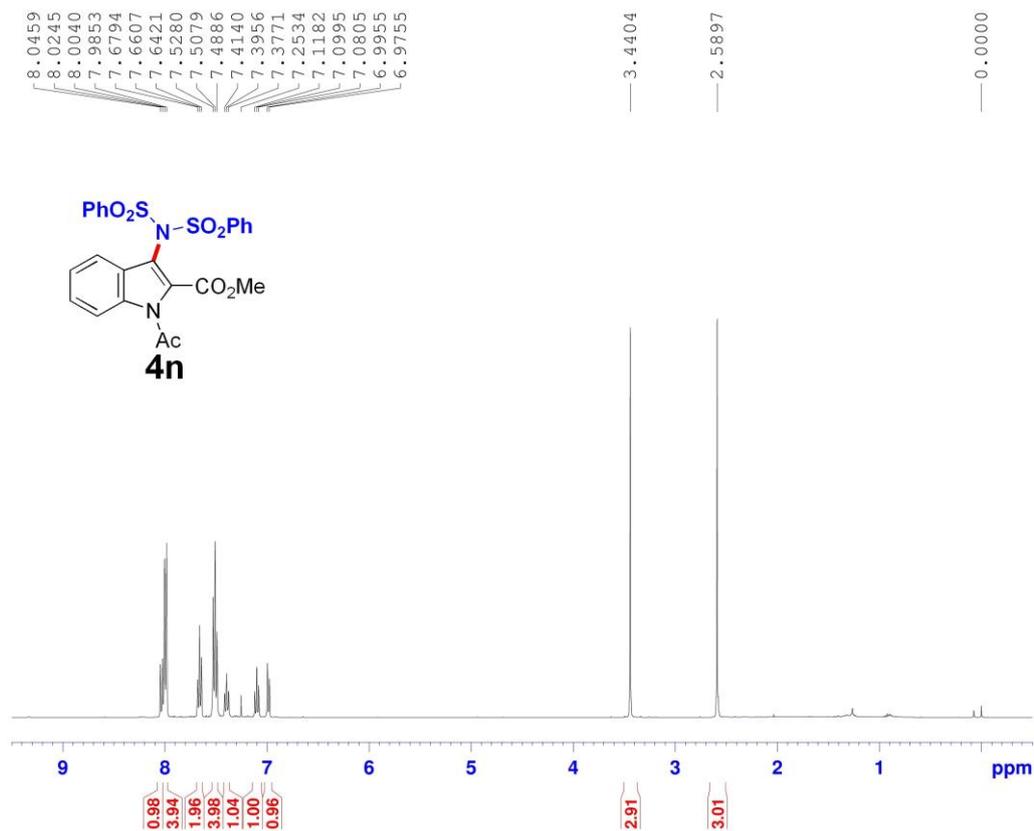
```

NAME      CLJ-WL-M267
EXPNO    4
PROCNO    1
Date_     20190323
Time      13.01
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         512
DS         0
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.361988 sec
RG         194.26
DW         20.800 usec
DE         6.50 usec
TE         300.6 K
D1         2.00000000 sec
D11        0.23000000 sec
TDO        1
    
```

```

===== CHANNEL f1 =====
SF01     100.6228293 MHz
NUC1      13C
P1        8.54 usec
SI        32768
SF        100.6127819 MHz
WDW        EM
SSB        0
LB        1.00 Hz
GB        0
PC        1.40
    
```

Electronic Supplementary Information

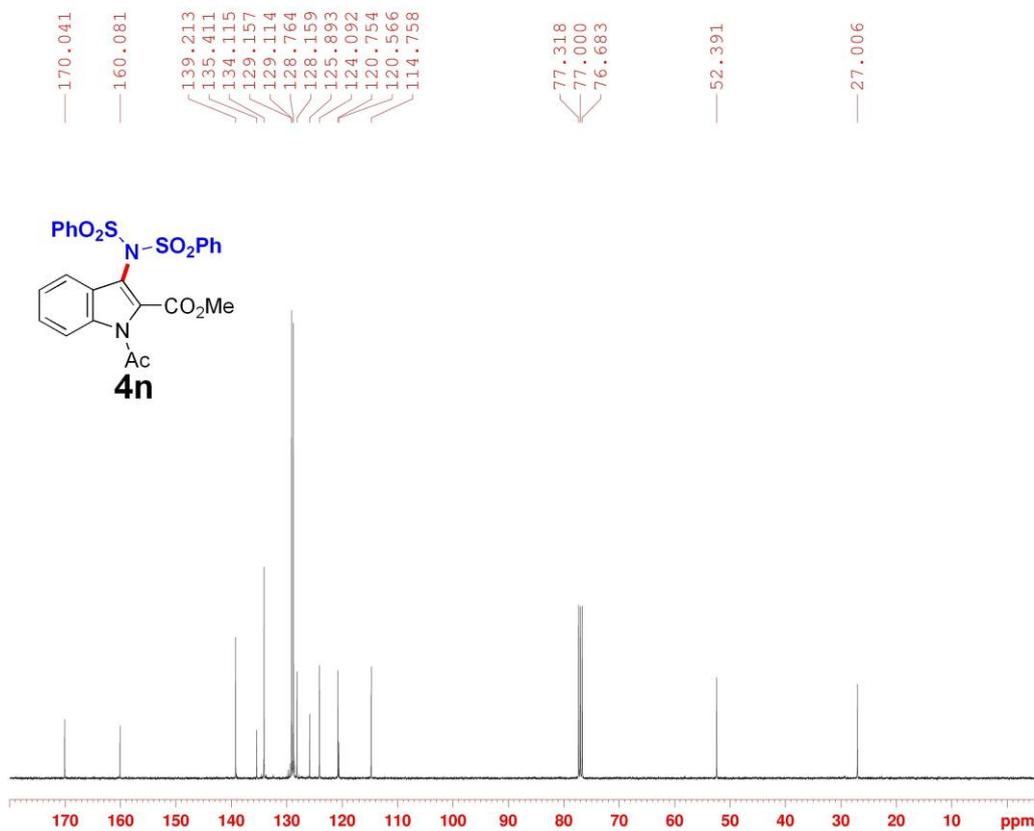


```

NAME      CLJ-WL-M264
EXPNO    1
PROCNO    1
Date_    20190314
Time     3.30
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD        65536
SOLVENT   CDCl3
NS        8
DS        0
SWH       8012.820 Hz
FIDRES    0.122266 Hz
AQ        4.0894966 sec
RG        231.9
DW        62.400 usec
DE        6.50 usec
TE        300.5 K
D1        1.00000000 sec
TDO       1
    
```

```

===== CHANNEL f1 =====
SFO1     400.1324710 MHz
NUC1     1H
P1       8.04 usec
SI       65536
SF       400.1300122 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
    
```



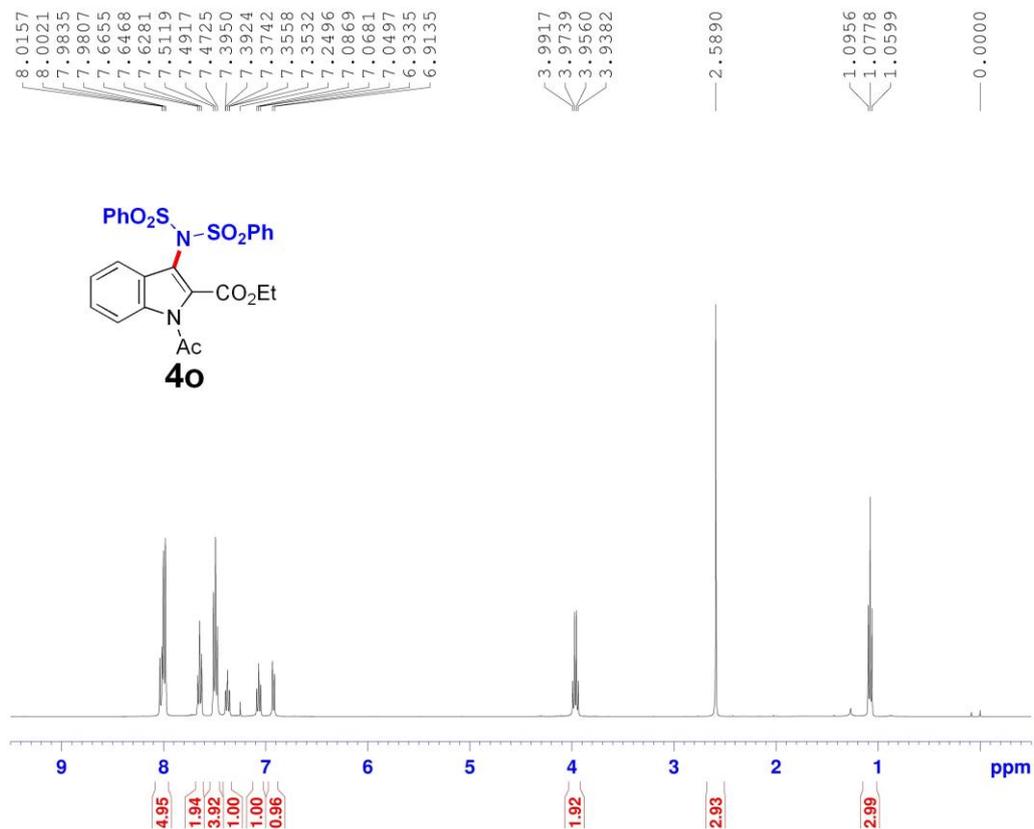
```

NAME      CLJ-WL-M264
EXPNO    2
PROCNO    1
Date_    20190314
Time     4.00
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS        512
DS        0
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ        1.361988 sec
RG        194.26
DW        20.800 usec
DE        6.50 usec
TE        300.7 K
D1        2.00000000 sec
D11       0.20000000 sec
TDO       1
    
```

```

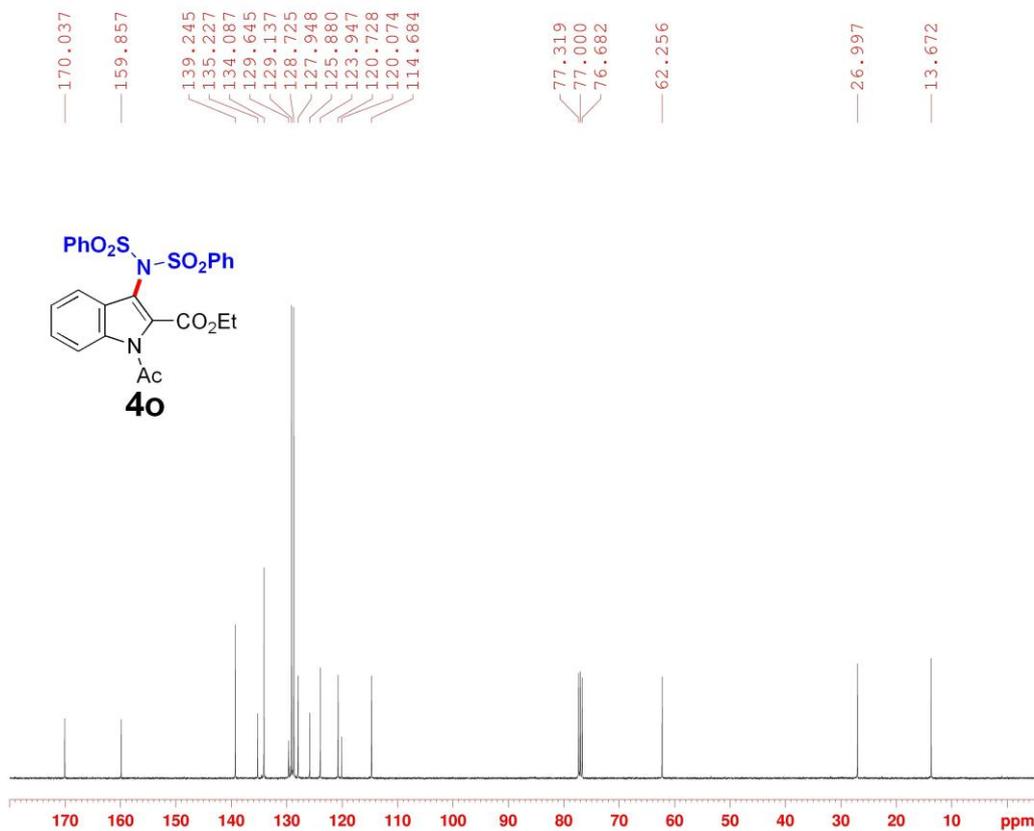
===== CHANNEL f1 =====
SFO1     100.6228293 MHz
NUC1     13C
P1       8.54 usec
SI       32768
SF       100.6127771 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
    
```

Electronic Supplementary Information



```

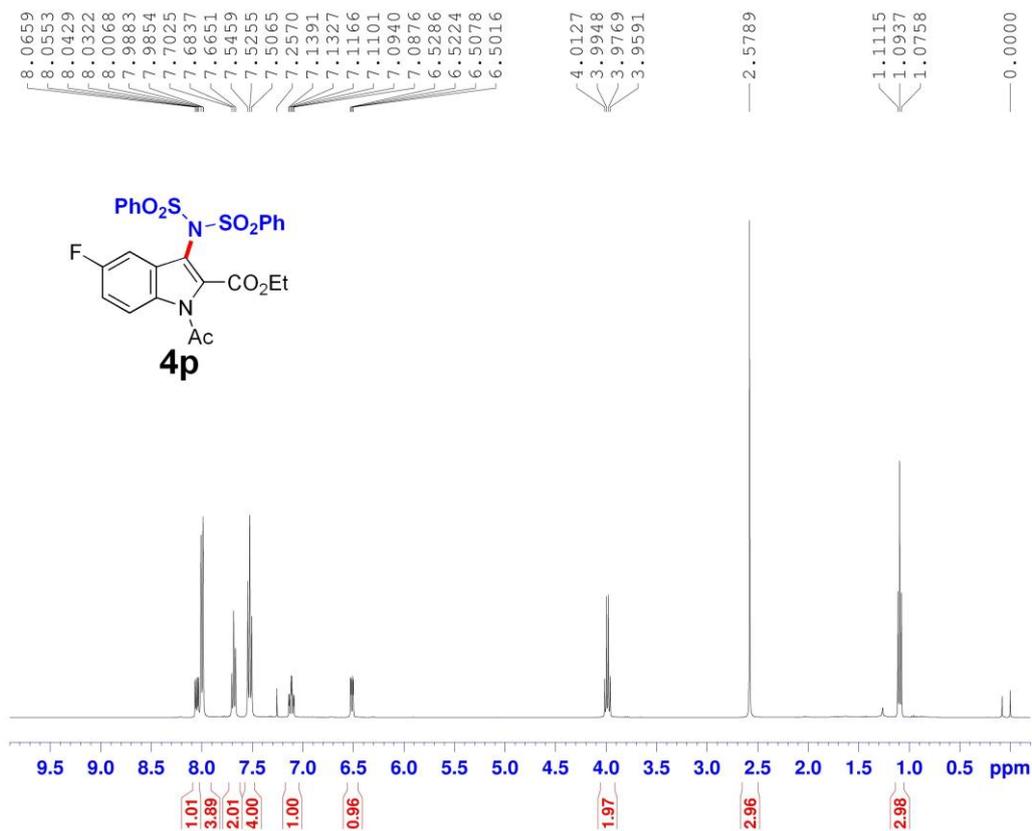
NAME      CLJ-WL-M252
EXPNO    1
PROCNO    1
Date_    20190307
Time     0.40
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD        65536
SOLVENT   CDCl3
NS        8
DS        0
SWH       8012.820 Hz
FIDRES    0.122266 Hz
AQ        4.0894866 sec
RG        18.6
DW        62.400 usec
DE        6.50 usec
TE        300.5 K
D1        1.00000000 sec
TDO       1
----- CHANNEL f1 -----
SF01     400.1324710 MHz
NUC1      1H
P1        8.54 usec
SI        65536
SF        400.1300136 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
    
```



```

NAME      CLJ-WL-M252
EXPNO    2
PROCNO    1
Date_    20190307
Time     1.10
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS        512
DS        0
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ        1.361988 sec
RG        194.26
DW        20.800 usec
DE        6.50 usec
TE        300.6 K
D1        2.00000000 sec
D11       0.23000000 sec
TDO       1
----- CHANNEL f1 -----
SF01     100.6228293 MHz
NUC1      13C
P1        8.54 usec
SI        32768
SF        100.6127806 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
    
```

Electronic Supplementary Information

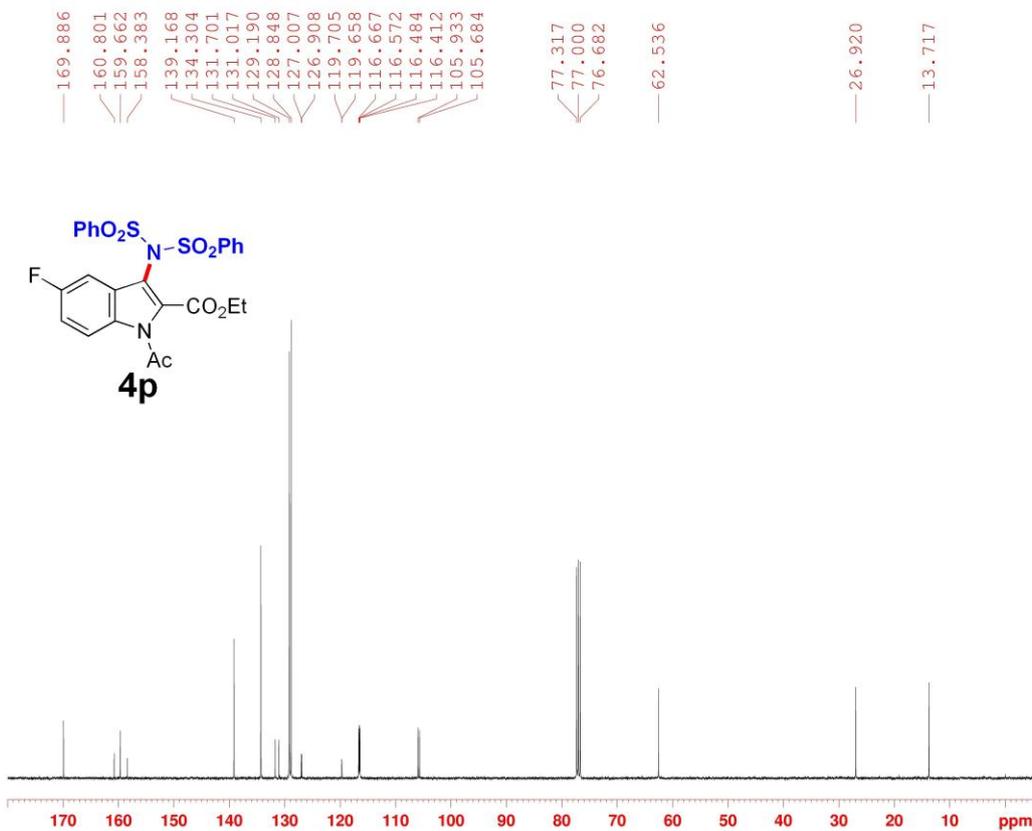


```

NAME      CLJ-WL-M270
EXPNO     2
PROCNO    1
Date_     20190316
Time      6.30
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         8
DS         0
SWH        8012.820 Hz
FIDRES     0.122266 Hz
AQ         4.0894966 sec
RG         47.33
DW         62.400 usec
DE         6.50 usec
TE         300.5 K
D1         1.00000000 sec
TDO        1
    
```

```

===== CHANNEL f1 =====
SFO1     400.1324710 MHz
NUC1      1H
P1        8.04 usec
SI        65536
SF        400.1300107 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
    
```



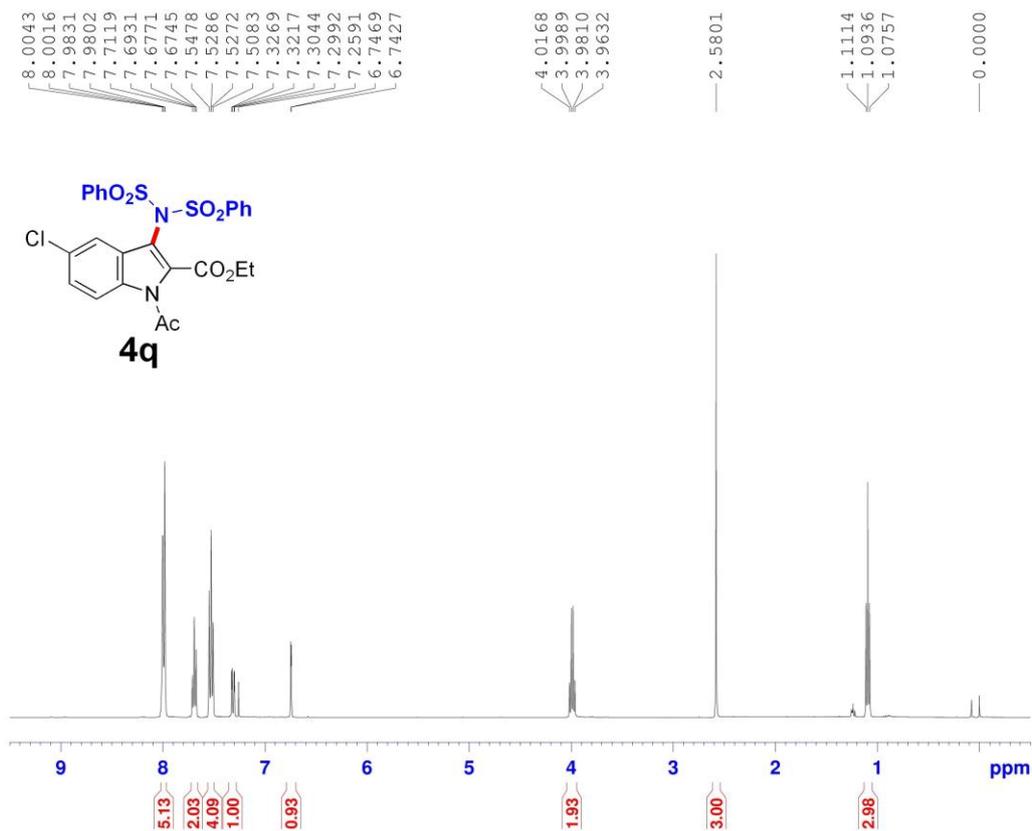
```

NAME      CLJ-WL-M270
EXPNO     1
PROCNO    1
Date_     20190316
Time      6.29
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         512
DS         0
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.361988 sec
RG         194.26
DW         20.800 usec
DE         6.50 usec
TE         300.7 K
D1         2.00000000 sec
D11       0.20000000 sec
TDO        1
    
```

```

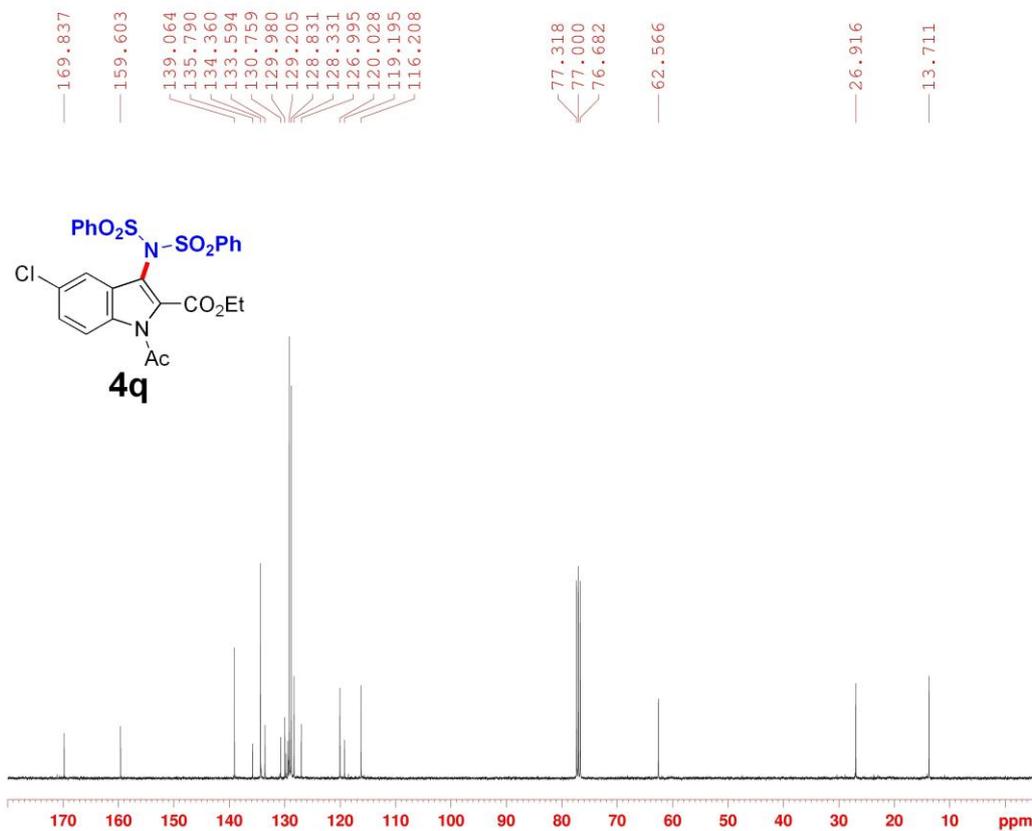
===== CHANNEL f1 =====
SFO1     100.6228293 MHz
NUC1      13C
P1        8.54 usec
SI        32768
SF        100.6127737 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
    
```

Electronic Supplementary Information



```

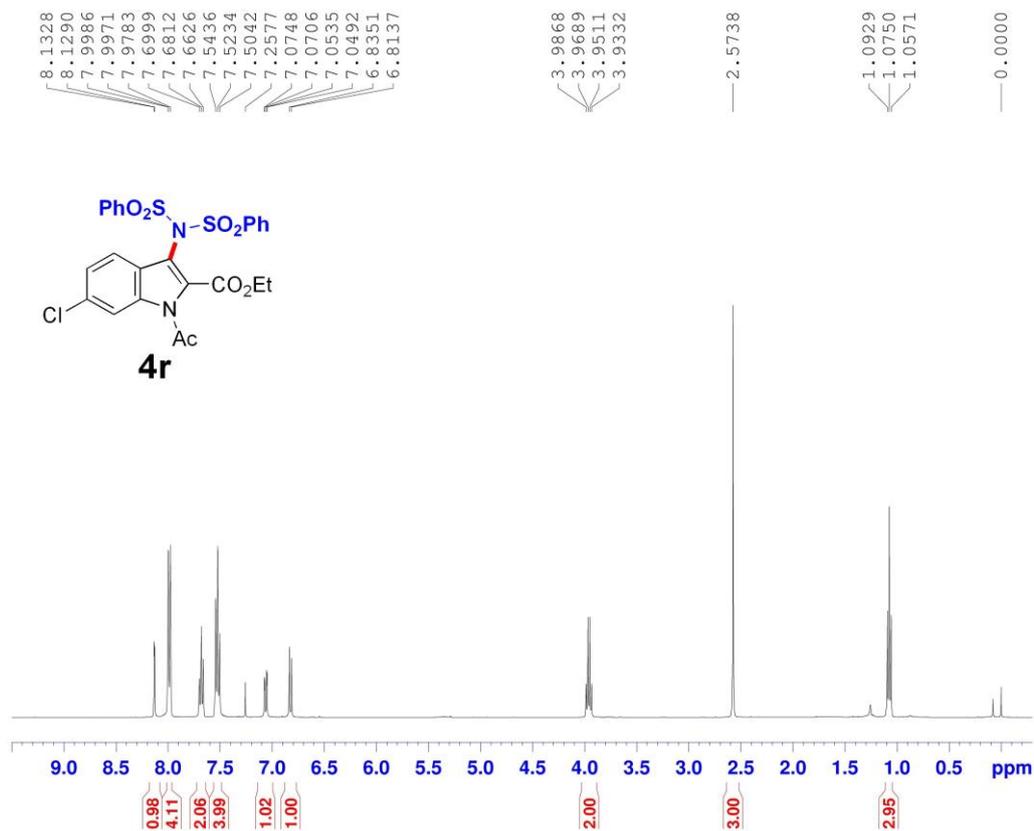
NAME      CLJ-WL-M266
EXPNO    2
PROCNO   1
Date_    20190316
Time     14.56
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zg30
TD       65536
SOLVENT  CDCl3
NS       8
DS       0
SWH      8012.820 Hz
FIDRES   0.122266 Hz
AQ       4.0894966 sec
RG       41.07
DW       62.400 usec
DE       6.50 usec
TE       300.5 K
D1       1.00000000 sec
TDO      1
----- CHANNEL f1 -----
SFO1    400.1324710 MHz
NUC1    1H
P1      8.04 usec
SI      65536
SF      400.1300099 MHz
WDW     EM
SSB     0
LB      0.30 Hz
GB      0
PC      1.00
    
```



```

NAME      CLJ-WL-M266
EXPNO    1
PROCNO   1
Date_    20190316
Time     14.55
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       512
DS       0
SWH      24038.461 Hz
FIDRES   0.366798 Hz
AQ       1.361988 sec
RG       194.26
DW       20.800 usec
DE       6.50 usec
TE       300.6 K
D1       2.00000000 sec
D11      0.20000000 sec
TDO      1
----- CHANNEL f1 -----
SFO1    100.6228293 MHz
NUC1    13C
P1      8.54 usec
SI      32768
SF      100.6127746 MHz
WDW     EM
SSB     0
LB      1.00 Hz
GB      0
PC      1.40
    
```

Electronic Supplementary Information

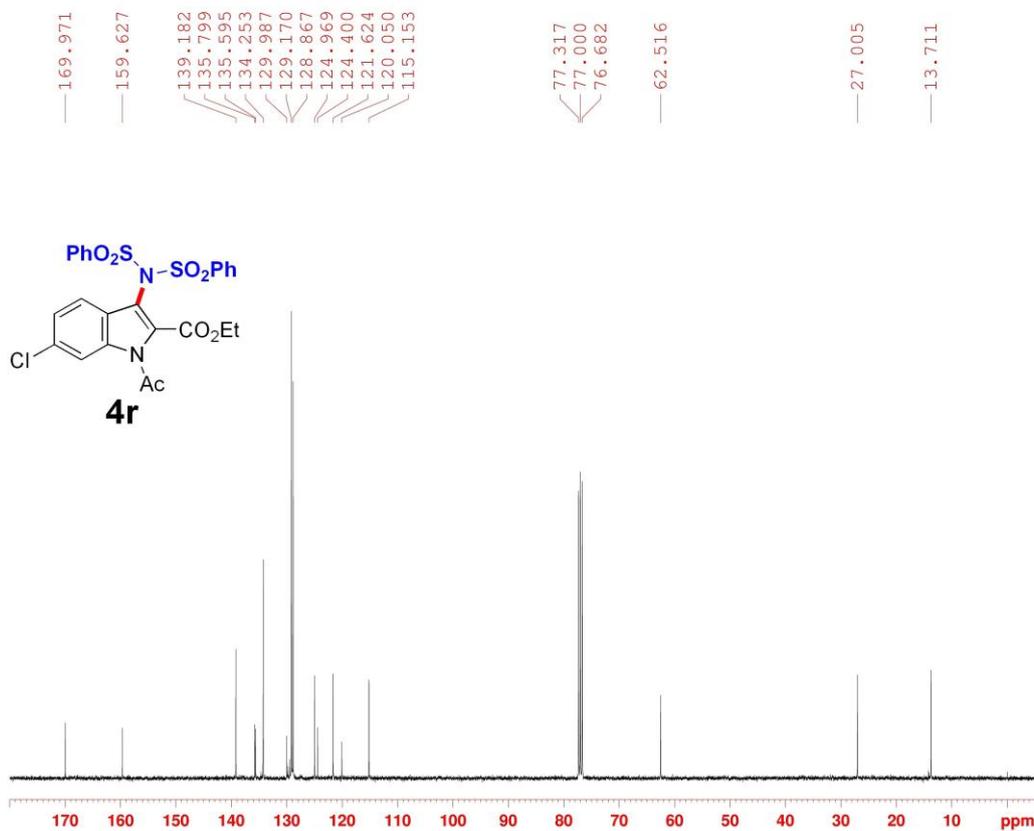


```

NAME      CLJ-WL-M271
EXPNO    1
PROCNO   1
Date_    20190320
Time     2.02
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zg30
TD       65536
SOLVENT  CDCl3
NS       0
DS       0
SWH      8012.820 Hz
FIDRES   0.122266 Hz
AQ       4.0894966 sec
RG       51.19
DW       62.400 usec
DE       6.50 usec
TE       300.5 K
D1       1.00000000 sec
TDO      1
    
```

```

===== CHANNEL f1 =====
SFO1    400.1324710 MHz
NUC1     1H
P1       8.04 usec
SI      65536
SF      400.1300105 MHz
WDW      EM
SSB      0
LB      0.30 Hz
GB      0
PC      1.00
    
```



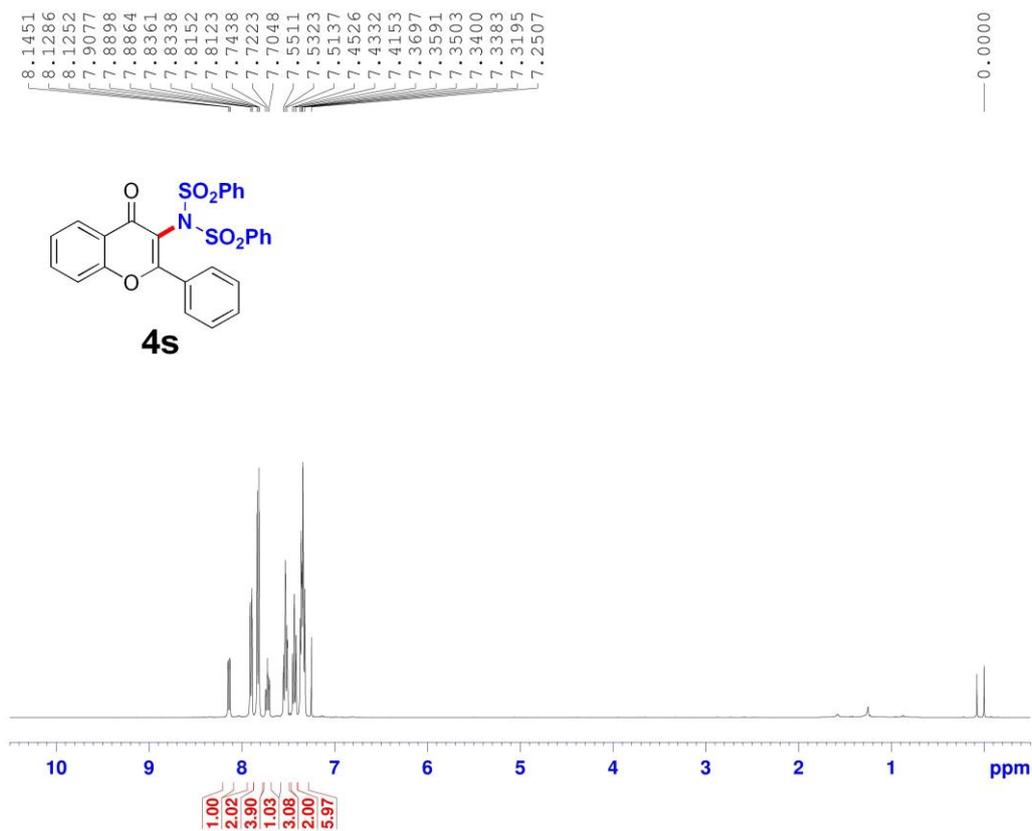
```

NAME      CLJ-WL-M271
EXPNO    2
PROCNO   1
Date_    20190320
Time     2.32
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       512
DS       0
SWH      24038.461 Hz
FIDRES   0.366798 Hz
AQ       1.361988 sec
RG       194.26
DW       20.800 usec
DE       6.50 usec
TE       300.6 K
D1       2.00000000 sec
D11      0.23000000 sec
TDO      1
    
```

```

===== CHANNEL f1 =====
SFO1    100.6228293 MHz
NUC1     13C
P1       8.54 usec
SI      32768
SF      100.6127731 MHz
WDW      EM
SSB      0
LB      1.00 Hz
GB      0
PC      1.40
    
```

Electronic Supplementary Information

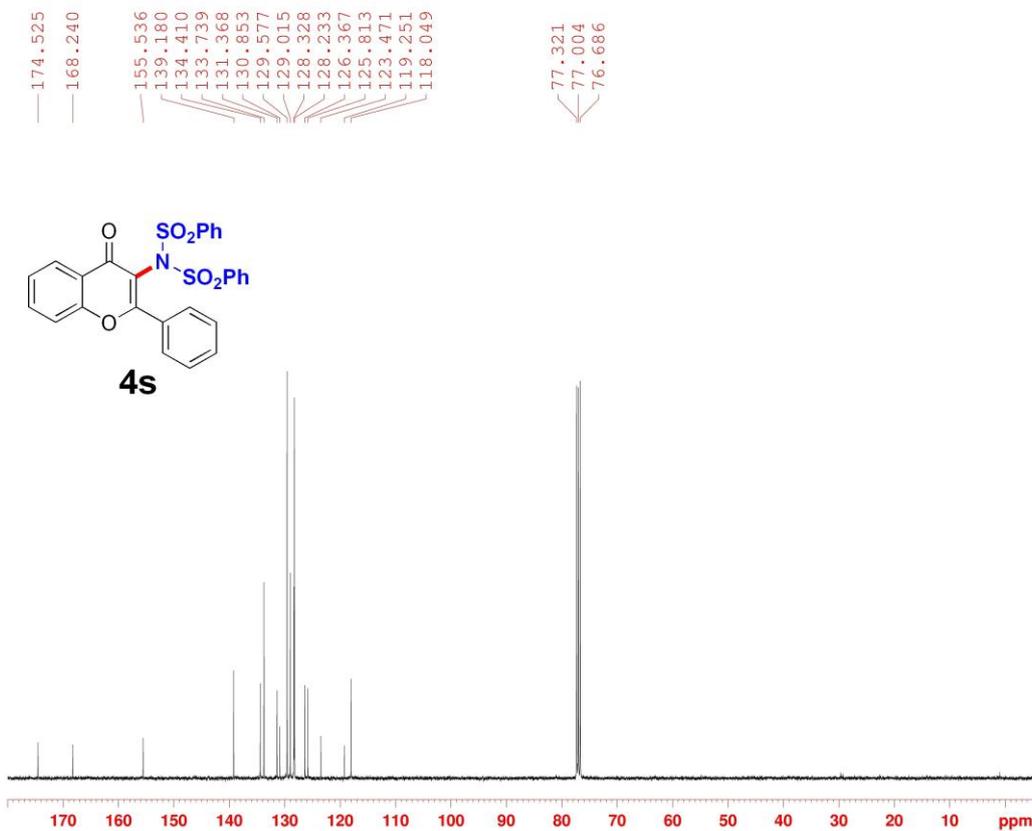


```

NAME      CLJ-WL-M231
EXPNO    1
PROCNO   1
Date_    20190124
Time     5.53
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zg30
TD       65536
SOLVENT  CDCl3
NS       8
DS       0
SWH      8012.820 Hz
FIDRES   0.122266 Hz
AQ       4.0894966 sec
RG       70.36
DW       62.400 usec
DE       6.50 usec
TE       300.1 K
D1       1.00000000 sec
D11      1
TDO      1
    
```

```

===== CHANNEL f1 =====
SF01    400.1324710 MHz
NUC1     1H
P1       8.04 usec
SI       65536
SF       400.1300132 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
    
```



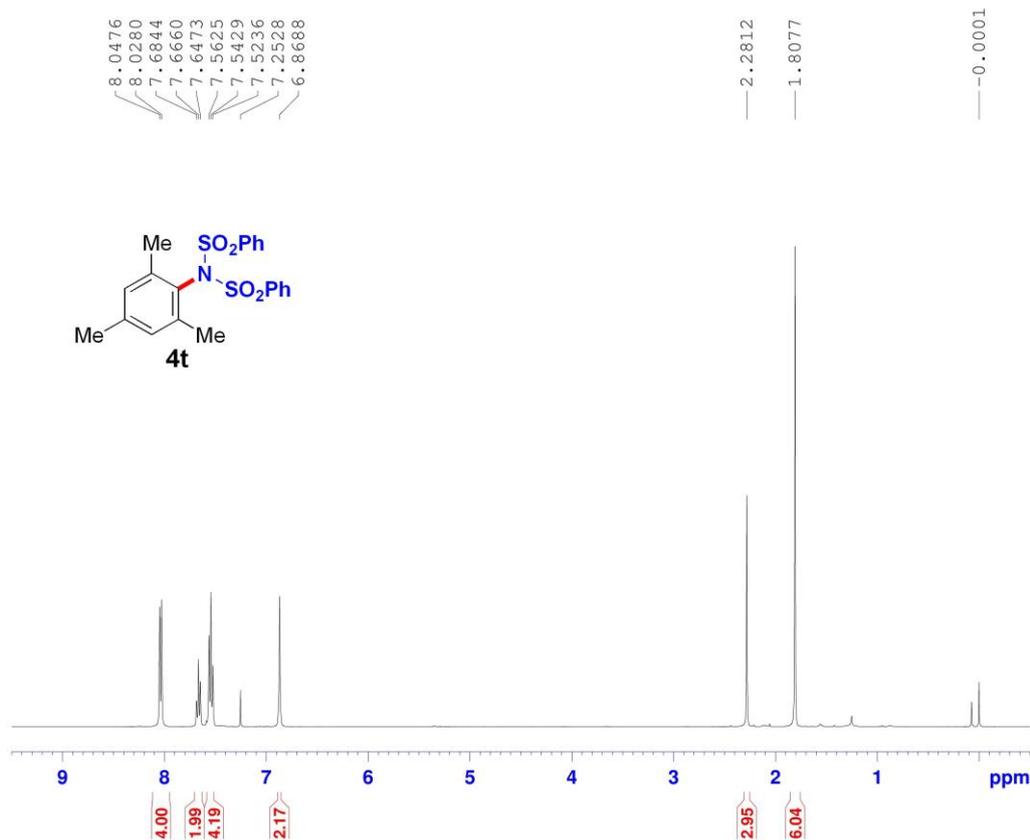
```

NAME      CLJ-WL-M231
EXPNO    2
PROCNO   1
Date_    20190124
Time     6.23
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       512
DS       0
SWH      24038.461 Hz
FIDRES   0.366798 Hz
AQ       1.361988 sec
RG       194.26
DW       20.800 usec
DE       6.50 usec
TE       300.1 K
D1       2.00000000 sec
D11      0.23000000 sec
TDO      1
    
```

```

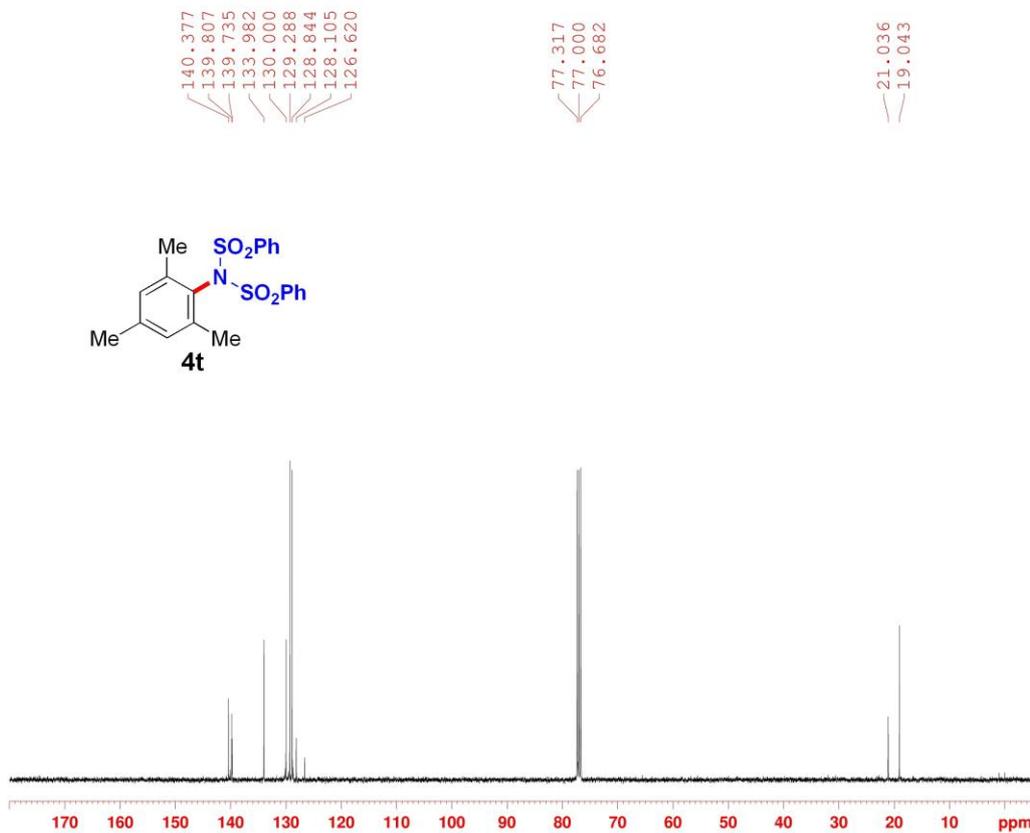
===== CHANNEL f1 =====
SF01    100.6228293 MHz
NUC1     13C
P1       8.54 usec
SI       32768
SF       100.6127755 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
    
```

Electronic Supplementary Information



```

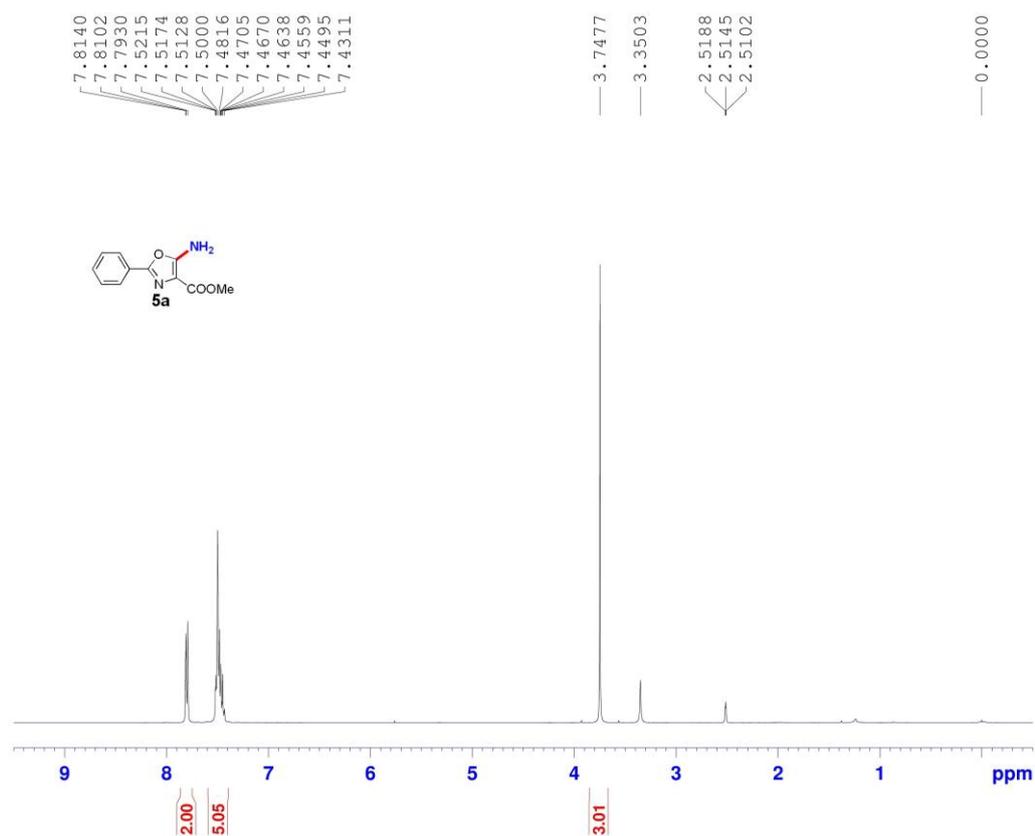
NAME      2019-05-13 miuqi_1
EXPNO     1
PROCNO    1
Date_     20190513
Time      19.53 h
INSTRUM   spect
PROBHD    z116098_0673 (
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        8012.820 Hz
FIDRES     0.244532 Hz
AQ          4.0894966 sec
RG          65.43
DW          62.400 usec
DE          6.50 usec
TE          298.4 K
D1          1.00000000 sec
D11         1
TDO         1
SFO1       400.1324708 MHz
NUC1       1H
P1          10.00 usec
SI          65536
SF          400.1300125 MHz
WDW         EM
SSB         0
LB          0.30 Hz
GB          0
PC          1.00
    
```



```

NAME      2019-05-13 miuqi_m307
EXPNO     2
PROCNO    1
Date_     20190513
Time      19.09 h
INSTRUM   spect
PROBHD    z116098_0673 (
PULPROG   zgpg30
TD         45536
SOLVENT   CDCl3
NS         256
DS         4
SWH        24038.461 Hz
FIDRES     0.133396 Hz
AQ          1.3631988 sec
RG          203.48
DW          20.800 usec
DE          6.50 usec
TE          297.6 K
D1          2.00000000 sec
D11         0.03000000 sec
TDO         1
SFO1       100.6228298 MHz
NUC1       13C
P1          10.00 usec
SI          32768
SF          100.6127734 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40
    
```

Electronic Supplementary Information

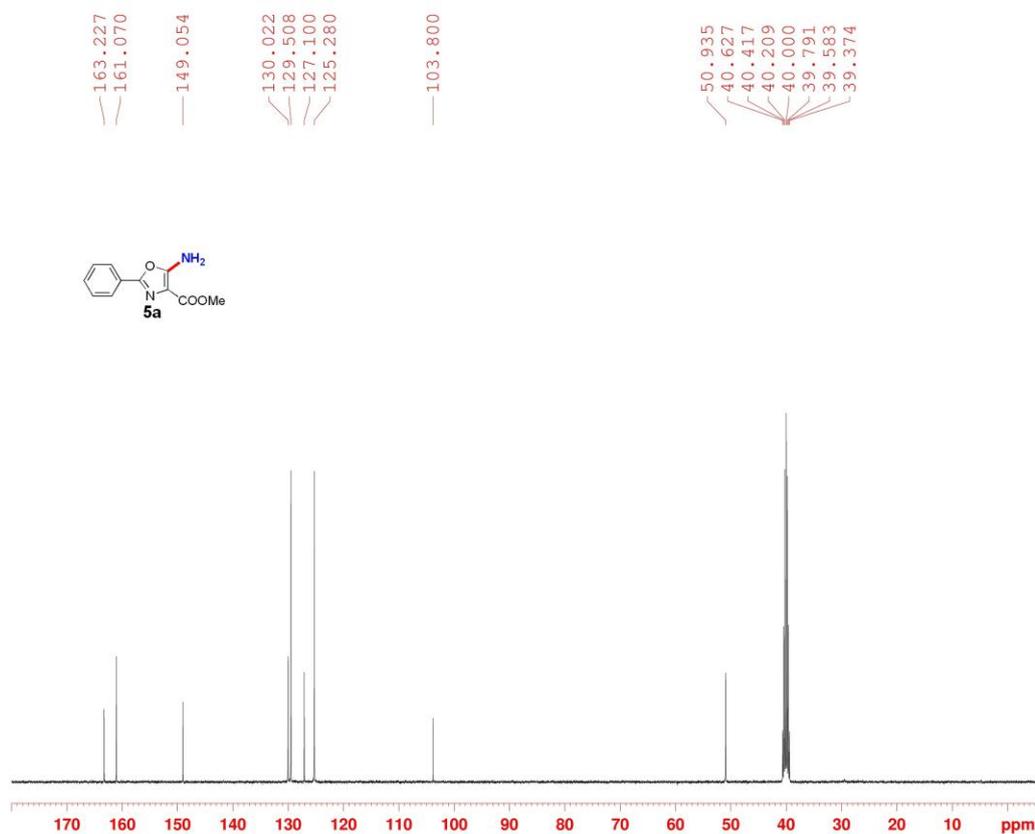


```

NAME      CLJ-WL-M288
EXPNO     1
PROCNO    1
Date_     20190413
Time      7.42
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg30
TD         65536
SOLVENT   DMSO
NS         8
DS         0
SWH       8012.820 Hz
FIDRES    0.122266 Hz
AQ         4.0894966 sec
RG         70.36
DM        62.400 usec
DE         6.50 usec
TE         300.2 K
D1         1.00000000 sec
TD0        1
    
```

```

===== CHANNEL f1 =====
SFO1     400.1324710 MHz
NUC1     1H
P1       8.04 usec
SI       65536
SF       400.1299972 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
    
```



```

NAME      CLJ-WL-M289
EXPNO     2
PROCNO    1
Date_     20190413
Time      8.46
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD         65536
SOLVENT   DMSO
NS         512
DS         0
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ         1.3631988 sec
RG         194.26
DM        20.800 usec
DE         6.50 usec
TE         300.7 K
D1         2.00000000 sec
D11       0.03000000 sec
TD0        1
    
```

```

===== CHANNEL f1 =====
SFO1     100.6228293 MHz
NUC1     13C
P1       8.54 usec
SI       32768
SF       100.6127685 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
    
```