

## *Supporting Information*

### **Radical Aminooxygenation of Alkenes with *N*-fluorobenzenesulfonimide (NFSI) and TEMPONa**

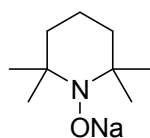
Yi Li, Marcel Hartmann, Constantin Gabriel Daniliuc and Armido Studer\*

*Institute of Organic Chemistry, Westfälische Wilhelms-University Münster, Corrensstrasse  
40, 48149 Münster, Germany  
[studer@uni-muenster.de](mailto:studer@uni-muenster.de)*

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**General:** All reactions involving air- or moisture-sensitive reagents or intermediates were carried out in pre-heated glassware under an argon atmosphere using standard *Schlenk* techniques. All solvents and reagents were purified according to standard procedures or were used as received from Aldrich, Fluka, Acros or ABCR. IR spectra were recorded on a *Digilab FTS 4000* with a *Specac MKII Golden Gate Single Reflexion ART System*.  $^1\text{H}$  NMR and  $^{13}\text{C}$  NMR spectra were recorded on a *DPX 300 or DD2 600* at 300 K. Spectra were calibrated relative to solvent's residual proton and carbon chemical shift:  $\text{CHCl}_3$  ( $\delta = 7.26$  for  $^1\text{H}$  NMR and  $\delta = 77.0$  for  $^{13}\text{C}$  NMR). TLC was performed using Merck silica gel 60 F-254 plates, detection of compounds with UV light or dipping into a solution of  $\text{KMnO}_4$  (1.5 g in 400 mL  $\text{H}_2\text{O}$ , 5 g  $\text{NaHCO}_3$ ), followed by heating. Flash column chromatography (FC) was performed using Merck or Fluka silica gel 60 (40-63  $\mu\text{m}$ ) applying a pressure of about 0.2 bar. Mass spectra were recorded on a *Finnigan MAT 4200S*, a *Bruker Daltonics Micro Tof*, a *Waters-Micromass Quatro LCZ* (ESI); peaks are given in  $m/z$  (% of basis peak).

#### **General procedure for the preparation of sodium 2,2,6,6-tetramethylpiperidine-1-olate (TEMPONa) solution**

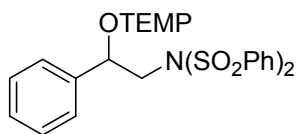


Freshly cleaned sodium (1.4 eq.) was placed in a flame-dried Schlenk-tube under argon. The sodium was melted with a heat-gun (200 °C) until a sodium mirror was formed at the bottom of the tube. The tube was allowed to cool to room temperature, TEMPO (1.0 eq.), THF (1.7M) and naphthalene (10 mol%) were added under argon. The reaction mixture was stirred at room temperature until a dark blue-black color persisted (1-2 h). For safety reasons the sodium was cut under hexan.

#### **General procedure for the arylaminoxylation of alkenes (GP1)**

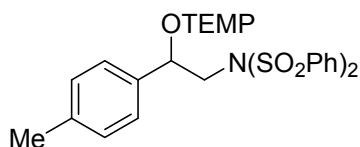
The TEMPONa-solution 1.7 M (3.0 eq.) in THF was added to a flame-dried Schlenk-tube containing *N*-fluorobenzenesulfonimide (0.75 mmol, 3.0 eq.) and alkene (0.25 mmol, 1.0 eq.) in  $\alpha,\alpha,\alpha$ -trifluorotoluene (1.0 mL) via syringe-pump over 6 h at room temperature. After completion, aqueous  $\text{NaHCO}_3$  was added and the aqueous layer was extracted with diethylether. The combined organic layers were dried over  $\text{MgSO}_4$ , filtered and concentrated *in vacuo*. Crude product was purified by flash column chromatography on silica gel.

***N*-(2-phenyl-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)-*N*-(phenylsulfonyl)benzenesulfonamide (2a)**



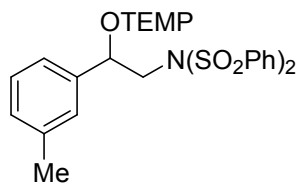
According to **GP1** with styrene (26 mg, 0.25 mmol, 1.0 eq.). Crude product was purified by flash chromatography on silica gel by using a 30:1 mixture of pentane/diethylether to provide analytically pure product as colorless oil (125 mg, 0.23 mmol, 90%). **<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>, 300 K): δ = 7.73 – 7.51 (*m*, 6H, CH<sub>arom</sub>), 7.50 – 7.30 (*m*, 9H, CH<sub>arom</sub>), 5.41 – 5.22 (*m*, 1H, OCH), 4.44 (*ddd*, *J* = 13.4, 11.0, 2.2 Hz, 1H, NH<sub>α</sub>H<sub>β</sub>), 4.21 – 4.00 (*m*, 1H, NH<sub>α</sub>H<sub>β</sub>), 1.64 – 0.77 (*m*, 18H, 3 × CH<sub>2</sub>, 4 × CH<sub>3</sub>). **<sup>13</sup>C NMR** (75 MHz, CDCl<sub>3</sub>, 300K): δ = 140.0 (C), 138.7 (C), 133.7 (2 × CH), 129.4 (2 × CH), 128.9 (4 × CH), 128.7 (4 × CH), 128.2 (2 × CH), 128.0 (CH), 84.2 (OCH), 60.1 (2 × C), 50.3 (NCH<sub>2</sub>), 40.6 (2 × CH<sub>2</sub>), 34.5 (2 × CH<sub>3</sub>), 20.3 (2 × CH<sub>3</sub>), 17.2 (CH<sub>2</sub>). **HRMS** (ESI) exact mass calculated for C<sub>29</sub>H<sub>36</sub>N<sub>2</sub>O<sub>5</sub>S<sub>2</sub>H ([M+H]<sup>+</sup>): 557.2138, found: 557.2147. **IR** (neat): 2932<sub>w</sub>, 1449<sub>m</sub>, 1375<sub>s</sub>, 1168<sub>s</sub>, 1084<sub>w</sub>, 1042<sub>w</sub>, 918<sub>w</sub>, 829<sub>m</sub>, 752<sub>m</sub>, 720<sub>m</sub>, 686<sub>m</sub>, 583<sub>s</sub>, 551<sub>s</sub>.

***N*-(phenylsulfonyl)-*N*-(2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)-2-(*p*-tolyl)ethyl)benzenesulfonamide (2b)**



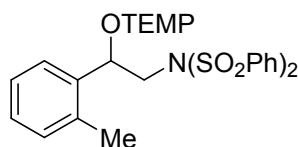
According to **GP1** with 1-methyl-4-vinylbenzene (30 mg, 0.25 mmol, 1.0 eq.). Crude product was purified by flash chromatography on silica gel by using a 10:1 mixture of pentane/diethylether to provide analytically pure product as colorless oil (92 mg, 0.17 mmol, 66%). **<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>, 300 K): δ = 7.52 (*t*, *J* = 7.4 Hz, 6H, CH<sub>arom</sub>), 7.33 (*t*, *J* = 7.6 Hz, 4H, CH<sub>arom</sub>), 7.15 (*d*, *J* = 7.6 Hz, 2H, CH<sub>arom</sub>), 7.02 (*d*, *J* = 7.6 Hz, 2H, CH<sub>arom</sub>), 5.12 (*dd*, *J* = 11.0, 4.6 Hz, 3H, OCH), 4.29 (*dd*, *J* = 14.9, 11.0 Hz, 1H, NH<sub>α</sub>H<sub>β</sub>), 3.95 (*dd*, *J* = 14.9, 4.6 Hz, 1H, NH<sub>α</sub>H<sub>β</sub>), 2.33 (*s*, 3H, CH<sub>3</sub>), 1.42 – 0.68 (*m*, 18H, 3 × CH<sub>2</sub>, 4 × CH<sub>3</sub>). **<sup>13</sup>C NMR** (75 MHz, CDCl<sub>3</sub>, 300K): δ = 138.7 (2 × C), 137.5 (C), 137.0 (C), 133.8 (2 × CH), 129.3 (2 × CH), 129.0 (4 × CH), 129.0 (2 × CH), 128.8 (2 × CH), 84.0 (OCH), 60.0 (2 × C), 50.3 (NCH<sub>2</sub>), 40.7 (2 × CH<sub>2</sub>), 34.6 (2 × CH<sub>3</sub>), 21.6 (CH<sub>3</sub>), 20.3 (2 × CH<sub>3</sub>), 17.3 (CH<sub>2</sub>). **HRMS** (ESI) exact mass calculated for C<sub>30</sub>H<sub>38</sub>N<sub>2</sub>O<sub>5</sub>S<sub>2</sub>H ([M+H]<sup>+</sup>): 571.2295, found: 571.2297. **IR** (neat): 2972<sub>w</sub>, 2930<sub>m</sub>, 1516<sub>w</sub>, 1448<sub>m</sub>, 1374<sub>s</sub>, 1361<sub>m</sub>, 1166<sub>s</sub>, 1133<sub>w</sub>, 1084<sub>w</sub>, 1041<sub>w</sub>, 1000<sub>w</sub>, 834<sub>m</sub>, 820<sub>m</sub>, 798<sub>m</sub>, 740<sub>m</sub>, 720<sub>s</sub>, 684<sub>s</sub>, 621<sub>w</sub>, 581<sub>s</sub>.

***N*-(phenylsulfonyl)-*N*-(2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)-2-(*m*-tolyl)ethyl)benzenesulfonamide (2c)**



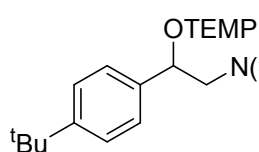
According to **GP1** with 1-methyl-3-vinylbenzene (30 mg, 0.25 mmol, 1.0 eq.). Crude product was purified by flash chromatography on silica gel by using a 10:1 mixture of pentane/diethylether to provide analytically pure product as colorless oil (99 mg, 0.18 mmol, 70%). **<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>, 300 K):  $\delta$  = 7.85 (*t*, *J* = 8.5 Hz, 6H, CH<sub>arom</sub>), 7.68 (*d*, *J* = 7.0 Hz, 4H, CH<sub>arom</sub>), 7.44 (*d*, *J* = 24.5 Hz, 4H, CH<sub>arom</sub>), 5.44 (*dd*, *J* = 10.8, 4.1 Hz, 1H, OCH), 4.63 (*dd*, *J* = 14.7, 10.7 Hz, 1H, NH <sub>$\alpha$</sub> H <sub>$\beta$</sub> ), 4.30 (*dd*, *J* = 14.9, 4.2 Hz, 1H, NH <sub>$\alpha$</sub> H <sub>$\beta$</sub> ), 2.54 (*s*, 3H, CH<sub>3</sub>), 1.86 – 1.00 (*m*, 18H, 3 × CH<sub>2</sub>, 4 × CH<sub>3</sub>). **<sup>13</sup>C NMR** (75 MHz, CDCl<sub>3</sub>, 300K):  $\delta$  = 135.5 (C), 134.3 (2 × C), 133.2 (C), 129.6 (2 × CH), 125.8 (CH), 124.6 (4 × CH), 124.5 (4 × CH), 124.4 (CH), 124.0 (CH), 121.9 (CH), 79.7 (OCH), 55.8 (2 × C), 46.0 (NCH<sub>2</sub>), 36.3 (2 × CH<sub>2</sub>), 30.4 (2 × CH<sub>3</sub>), 17.3 (CH<sub>3</sub>), 16.0 (2 × CH<sub>3</sub>), 13.0 (CH). **HRMS** (ESI) exact mass calculated for C<sub>30</sub>H<sub>38</sub>N<sub>2</sub>O<sub>5</sub>S<sub>2</sub>H ([M+H]<sup>+</sup>): 571.2295, found: 571.2293. **IR** (neat): 2972*w*, 2932*m*, 1585*w*, 1448*m*, 1374*s*, 1361*m*, 1167*s*, 1132*w*, 1084*m*, 1042*m*, 974*w*, 815*s*, 781*m*, 743*m*, 719*s*, 685*s*, 631*w*, 577*s*.

***N*-(phenylsulfonyl)-*N*-(2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)-2-(*o*-tolyl)ethyl)benzenesulfonamide (2d)**



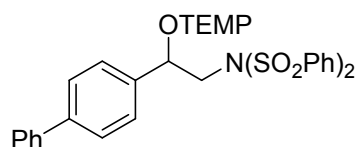
According to **GP1** with 1-methyl-3-vinylbenzene (30 mg, 0.25 mmol, 1.0 eq.). Crude product was purified by flash chromatography on silica gel by using a 10:1 mixture of pentane/diethylether to provide analytically pure product (98 mg, 0.18 mmol, 70%). **<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>, 300 K):  $\delta$  = 7.91 – 7.79 (*m*, 7H, CH<sub>arom</sub>), 7.67 (*t*, *J* = 7.8 Hz, 5H, CH<sub>arom</sub>), 7.55 – 7.42 (*m*, 2H, CH<sub>arom</sub>), 5.83 (*s*, 1H, OCH), 4.72 (*s*, 1H, NH <sub>$\alpha$</sub> H <sub>$\beta$</sub> ), 4.40 (*s*, 1H, NH <sub>$\alpha$</sub> H <sub>$\beta$</sub> ), 2.48 (*s*, 3H, CH<sub>3</sub>), 1.86 – 1.02 (*m*, 18H, 3 × CH<sub>2</sub>, 4 × CH<sub>3</sub>). **<sup>13</sup>C NMR** (75 MHz, CDCl<sub>3</sub>, 300K):  $\delta$  = 139.0 (C), 138.4 (2 × C), 137.8 (C), 133.7 (2 × CH), 130.4 (CH), 129.0 (5 × CH), 128.7 (4 × CH), 127.9 (CH), 126.1 (CH), 79.2 (OCH), 60.0 (2 × C), 50.7 (NCH<sub>2</sub>), 40.7 (2 × CH<sub>2</sub>), 34.7 (2 × CH<sub>3</sub>), 20.4 (CH<sub>3</sub>), 19.9 (2 × CH<sub>3</sub>), 17.3 (CH<sub>2</sub>). **HRMS** (ESI) exact mass calculated for C<sub>30</sub>H<sub>38</sub>N<sub>2</sub>O<sub>5</sub>S<sub>2</sub>H ([M+H]<sup>+</sup>): 571.2295, found: 571.2293. **IR** (neat): 2972*w*, 2932*m*, 1575*w*, 1448*m*, 1372*s*, 1365*m*, 1167*s*, 1132*w*, 1084*m*, 1042*m*, 974*w*, 781*m*, 743*m*, 719*s*, 679*s*, 642*w*, 581*s*.

***N*-(2-(4-(tert-butyl)phenyl)-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)-*N*-(phenylsulfonyl)benzenesulfonamide (2e)**



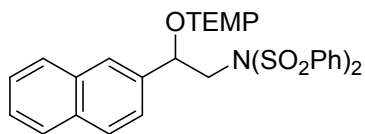
According to **GPI** with 1-(tert-butyl)-4-vinylbenzene (40 mg, 0.25 mmol, 1.0 eq.). Crude product was purified by flash chromatography on silica gel by using a 20:1 mixture of pentane/diethylether to provide analytically pure product (114 mg, 0.188 mmol, 75%). **<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>, 300 K): δ = 7.51 (*d*, *J* = 7.9 Hz, 6H, CH<sub>arom</sub>), 7.38 – 7.14 (*m*, 8H, CH<sub>arom</sub>), 5.14 (*dd*, *J* = 10.9, 4.3 Hz, 1H, OCH), 4.36 (*dd*, *J* = 14.9, 10.9 Hz, 1H, NH<sub>α</sub>H<sub>β</sub>), 3.94 (*dd*, *J* = 14.9, 4.4 Hz, 1H, NH<sub>α</sub>H<sub>β</sub>), 1.42 – 0.68 (*m*, 27H, 3 × CH<sub>2</sub>, 7 × CH<sub>3</sub>). **<sup>13</sup>C NMR** (75 MHz, CDCl<sub>3</sub>, 300K): δ = 150.1 (C), 137.6 (2 × CH), 135.5 (C), 132.7 (2 × CH), 128.3 (2 × CH), 128.0 (4 × CH), 127.7 (4 × CH), 123.9 (2 × CH), 82.5 (OCH), 59.1 (2 × C), 48.5 (NCH<sub>2</sub>), 39.7 (2 × CH<sub>2</sub>), 33.6 (C), 33.2 (2 × CH<sub>3</sub>), 30.6 (3 × CH<sub>3</sub>), 19.2 (2 × CH<sub>3</sub>), 16.3 (CH<sub>2</sub>). **HRMS** (ESI) exact mass calculated for C<sub>33</sub>H<sub>44</sub>N<sub>2</sub>O<sub>5</sub>S<sub>2</sub>H ([M+H]<sup>+</sup>): 613.2764, found: 613.2764. **IR** (neat): 2965*w*, 2932*w*, 1715*w*, 1448*m*, 1374*s*, 1361*m*, 1167*s*, 1085*m*, 825*s*, 744*s*, 720*s*, 685*s*, 621*w*, 580*s*, 548*s*.

***N*-(2-([1,1'-biphenyl]-4-yl)-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)-*N*-(phenylsulfonyl)benzenesulfonamide (2f)**



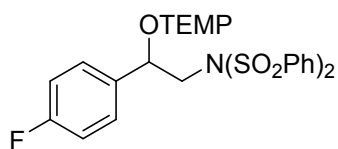
According to **GPI** with 1-(tert-butyl)-4-vinylbenzene (40 mg, 0.25 mmol, 1.0 eq.). Crude product was purified by flash chromatography on silica gel by using a 20:1 mixture of pentane/diethylether to provide analytically pure product (100 mg, 0.165 mmol, 66%). **<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>, 300 K): δ = 7.71 – 7.63 (*m*, 6H, CH<sub>arom</sub>), 7.60 – 7.56 (*m*, 2H, CH<sub>arom</sub>), 7.55 – 7.52 (*m*, 2H, CH<sub>arom</sub>), 7.49 (*d*, *J* = 7.9 Hz, 2H, CH<sub>arom</sub>), 7.45 (*d*, *J* = 8.2 Hz, 2H, CH<sub>arom</sub>), 7.44 – 7.33 (*m*, 5H, CH<sub>arom</sub>), 5.32 (*dd*, *J* = 11.1, 4.8 Hz, 1H, OCH), 4.46 (*dd*, *J* = 15.0, 11.1 Hz, 1H, NH<sub>α</sub>H<sub>β</sub>), 4.06 (*dd*, *J* = 15.1, 4.8 Hz, 1H, NH<sub>α</sub>H<sub>β</sub>), 1.55 – 0.76 (*m*, 18H, 3 × CH<sub>2</sub>, 4 × CH<sub>3</sub>). **<sup>13</sup>C NMR** (75 MHz, CDCl<sub>3</sub>, 300K): δ = 140.7 (C), 140.5 (C), 139.1 (C), 138.5 (2 × C), 133.7 (2 × CH), 129.8 (2 × CH), 128.9 (2 × CH), 128.9 (4 × CH), 128.7 (4 × CH), 127.4 (CH), 127.0 (2 × CH), 126.8 (2 × CH), 84.1 (OCH), 60.0 (2 × C), 50.0 (NCH<sub>2</sub>), 40.6 (2 × CH<sub>2</sub>), 34.8 (2 × CH<sub>3</sub>), 20.3 (2 × CH<sub>3</sub>), 17.2 (CH<sub>2</sub>). **HRMS** (ESI) exact mass calculated for C<sub>35</sub>H<sub>40</sub>N<sub>2</sub>O<sub>5</sub>S<sub>2</sub>H ([M+H]<sup>+</sup>): 633.2451, found: 633.2450. **IR** (neat): 2971*w*, 2932*w*, 1448*m*, 1374*s*, 1361*m*, 1167*s*, 828*s*, 764*m*, 719*s*, 697*m*, 684*s*, 581*s*, 547*s*, 520*m*.

***N*-(2-(naphthalen-2-yl)-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)-*N*-(phenylsulfonyl)benzenesulfonamide (2g)**



According to **GP1** with 2-vinylnaphthalene (39 mg, 0.25 mmol, 1.0 eq.). Crude product was purified by flash chromatography on silica gel by using a 20:1 mixture of pentane/diethylether to provide analytically pure product (65 mg, 0.11 mmol, 43%). **<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>, 300 K):  $\delta$  = 7.86 (*t*, *J* = 7.7 Hz, 2H, CH<sub>arom</sub>), 7.71 (*dd*, *J* = 7.6, 1.8 Hz, 1H, CH<sub>arom</sub>), 7.63 (*d*, *J* = 8.2 Hz, 2H, CH<sub>arom</sub>), 7.58 – 7.37 (*m*, 8H, CH<sub>arom</sub>), 7.11 (*t*, *J* = 7.8 Hz, 4H, CH<sub>arom</sub>), 5.41 (*dd*, *J* = 11.1, 4.7 Hz, 1H, OCH), 4.49 (*dd*, *J* = 15.1, 11.1 Hz, 1H, NH <sub>$\alpha$</sub> H <sub>$\beta$</sub> ), 4.11 (*dd*, *J* = 15.1, 4.7 Hz, 1H, NH <sub>$\alpha$</sub> H <sub>$\beta$</sub> ), 1.52 – 0.68 (*m*, 18H, 3 × CH<sub>2</sub>, 4 × CH<sub>3</sub>). **<sup>13</sup>C NMR** (75 MHz, CDCl<sub>3</sub>, 300K):  $\delta$  = 138.5 (2 × C), 137.4 (C), 133.5 (2 × CH), 133.3 (C), 133.2 (C), 128.8 (CH), 128.7 (4 × CH), 128.4 (5 × CH), 128.0 (CH), 127.7 (CH), 126.4 (CH), 126.0 (CH), 125.9 (CH), 84.5 (OCH), 60.1 (2 × C), 50.4 (NCH<sub>2</sub>), 40.6 (2 × CH<sub>2</sub>), 31.9 (2 × CH<sub>3</sub>), 20.2 (2 × CH<sub>3</sub>), 17.2 (CH<sub>2</sub>). **HRMS** (ESI) exact mass calculated for C<sub>33</sub>H<sub>38</sub>N<sub>2</sub>O<sub>5</sub>S<sub>2</sub>H ([M+H]<sup>+</sup>): 607.2295, found: 607.2299. **IR** (neat): 2929<sub>w</sub>, 1448<sub>m</sub>, 1375<sub>s</sub>, 1169<sub>s</sub>, 816<sub>m</sub>, 751<sub>m</sub>, 720<sub>m</sub>, 686<sub>m</sub>, 593<sub>s</sub>, 549<sub>s</sub>.

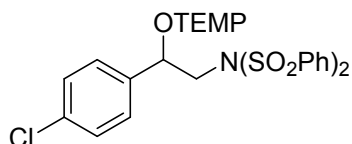
***N*-(2-(4-fluorophenyl)-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)-*N*-(phenylsulfonyl)benzenesulfonamide (2h)**



According to **GP1** with 1-fluoro-4-vinylbenzene (31 mg, 0.25 mmol, 1.0 eq.). Crude product was purified by flash chromatography on silica gel by using a 20:1 mixture of pentane/diethylether to provide analytically pure product as colorless oil (105 mg, 0.183 mmol, 73%). **<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>, 300 K):  $\delta$  = 7.72 (*d*, *J* = 7.8 Hz, 4H, CH<sub>arom</sub>), 7.65 (*t*, *J* = 7.4 Hz, 2H, CH<sub>arom</sub>), 7.49 (*t*, *J* = 7.7 Hz, 4H, CH<sub>arom</sub>), 7.32 (*dd*, *J* = 8.4, 5.4 Hz, 2H, CH<sub>arom</sub>), 6.99 (*t*, *J* = 8.6 Hz, 2H, CH<sub>arom</sub>), 5.26 (*dd*, *J* = 11.0, 4.8 Hz, 1H, OCH), 4.36 (*dd*, *J* = 14.9, 11.0 Hz, 1H, NH <sub>$\alpha$</sub> H <sub>$\beta$</sub> ), 4.11 (*dd*, *J* = 15.0, 4.8 Hz, 1H, NH <sub>$\alpha$</sub> H <sub>$\beta$</sub> ), 1.60 – 0.72 (*m*, 18H, 3 × CH<sub>2</sub>, 4 × CH<sub>3</sub>). **<sup>13</sup>C NMR** (75 MHz, CDCl<sub>3</sub>, 300K):  $\delta$  = 162.6 (*d*, *J* = 246.0 Hz, C), 138.6 (2 × C), 135.8 (*d*, *J* = 3.0 Hz, C), 133.8 (2 × CH), 130.8 (*d*, *J* = 7.9 Hz, 2 × CH), 128.8 (4 × CH), 128.8 (2 × CH), 115.2 (*d*, *J* = 20.8 Hz, 2 × CH), 83.6 (OCH), 60.0 (*d*, *J* = 7.9 Hz, 2 × C), 50.6 (NCH<sub>2</sub>), 40.6 (2 × CH<sub>2</sub>), 34.3 (2 × CH<sub>3</sub>), 20.5 (2 × CH<sub>3</sub>), 17.2 (CH<sub>2</sub>). **HRMS** (ESI) exact mass calculated for C<sub>29</sub>H<sub>35</sub>FN<sub>2</sub>O<sub>5</sub>S<sub>2</sub>H ([M+H]<sup>+</sup>): 575.2044, found:

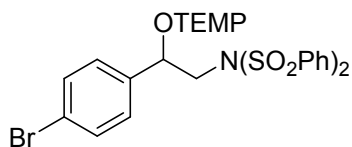
575.2041. **IR** (neat): 2932w, 1605w, 1511m, 1449m, 1376s, 1223m, 1168s, 1085m, 842m, 811m, 722m, 583s.

***N*-(2-(4-chlorophenyl)-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)-*N*-(phenylsulfonyl)benzenesulfonamide (2i)**



According to **GPI** with 1-chloro-4-vinylbenzene (35 mg, 0.25 mmol, 1.0 eq.). Crude product was purified by flash chromatography on silica gel by using a 10:1 mixture of pentane/diethylether to provide analytically pure product (98 mg, 0.17 mmol, 67%). **<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>, 300 K): δ = 7.73 (*dd*, *J* = 21.8, 7.4 Hz, 6H, CH<sub>arom</sub>), 7.56 (*t*, *J* = 7.5 Hz, 4H, CH<sub>arom</sub>), 7.33 (*q*, *J* = 8.2 Hz, 4H, CH<sub>arom</sub>), 5.32 (*dd*, *J* = 10.9, 4.7 Hz, 1H, OCH), 4.40 (*dd*, *J* = 15.0, 11.0 Hz, 1H, NH<sub>α</sub>H<sub>β</sub>), 4.14 (*dd*, *J* = 15.0, 4.8 Hz, 1H, NH<sub>α</sub>H<sub>β</sub>), 1.67 – 0.75 (*m*, 18H, 3 × CH<sub>2</sub>, 4 × CH<sub>3</sub>). **<sup>13</sup>C NMR** (75 MHz, CDCl<sub>3</sub>, 300K): δ = 138.8 (C), 138.6 (2 × C), 134.0 (2 × CH), 133.8 (C), 130.5 (2 × CH), 128.9 (4 × CH), 128.9 (4 × CH), 128.5 (2 × CH), 83.9 (OCH), 60.2 (2 × C), 50.5 (NCH<sub>2</sub>), 40.6 (2 × CH<sub>2</sub>), 34.8 (2 × CH<sub>3</sub>), 20.4 (2 × CH<sub>3</sub>), 17.3 (CH<sub>2</sub>). **HRMS** (ESI) exact mass calculated for C<sub>29</sub>H<sub>35</sub>ClN<sub>2</sub>O<sub>5</sub>S<sub>2</sub>H ([M+H]<sup>+</sup>): 591.1749, found: 591.1752. **IR** (neat): 2933w, 1449m, 1375s, 1361m, 1167s, 1087m, 824s, 790m, 739s, 720s, 684s, 592s, 579s.

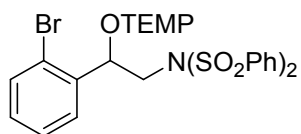
***N*-(2-(4-bromophenyl)-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)-*N*-(phenylsulfonyl)benzenesulfonamide (2j)**



According to **GPI** with 1-bromo-4-vinylbenzene (46 mg, 0.25 mmol, 1.0 eq.). Crude product was purified by flash chromatography on silica gel by using a 10:1 mixture of pentane/diethylether to provide analytically pure product (105 mg, 0.165 mmol, 66%). **<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>, 300 K): δ = 7.55 (*dd*, *J* = 17.1, 7.7 Hz, 6H, CH<sub>arom</sub>), 7.39 (*t*, *J* = 7.7 Hz, 4H, CH<sub>arom</sub>), 7.29 (*d*, *J* = 8.0 Hz, 2H, CH<sub>arom</sub>), 7.12 (*d*, *J* = 7.9 Hz, 2H, CH<sub>arom</sub>), 5.13 (*dd*, *J* = 11.0, 4.8 Hz, 1H, OCH), 4.22 (*dd*, *J* = 15.0, 11.0 Hz, 1H, NH<sub>α</sub>H<sub>β</sub>), 3.94 (*dd*, *J* = 15.0, 4.9 Hz, 1H, NH<sub>α</sub>H<sub>β</sub>), 1.45 – 0.59 (*m*, 18H, 3 × CH<sub>2</sub>, 4 × CH<sub>3</sub>). **<sup>13</sup>C NMR** (75 MHz, CDCl<sub>3</sub>, 300K): δ = 139.2 (C), 138.5 (2 × C), 133.9 (2 × CH), 131.4 (2 × CH), 130.8 (2 × CH), 128.9 (4 × CH), 128.8 (4 × CH), 122.0 (C), 83.9 (OCH), 60.1 (2 × C), 50.4 (NCH<sub>2</sub>), 40.6 (2 × CH<sub>2</sub>),

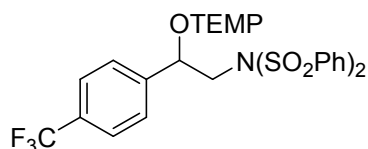
34.8 ( $2 \times \text{CH}_3$ ), 20.4 ( $2 \times \text{CH}_3$ ), 17.2 ( $\text{CH}_2$ ). **HRMS** (ESI) exact mass calculated for  $\text{C}_{29}\text{H}_{35}\text{BrN}_2\text{O}_5\text{S}_2\text{H}$  ( $[\text{M}+\text{H}]^+$ ): 635.1244, found: 635.1248. **IR** (neat): 2972 $w$ , 2932 $w$ , 1448 $m$ , 1375 $s$ , 1361 $m$ , 1168 $s$ , 1085 $m$ , 1012 $m$ , 822 $s$ , 790 $m$ , 720 $s$ , 686 $s$ , 611 $m$ , 592 $s$ , 550 $s$ .

***N*-(2-(2-bromophenyl)-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)-*N*-(phenylsulfonyl)benzenesulfonamide (2k)**



According to **GPI** with 1-bromo-2-vinylbenzene (46 mg, 0.25 mmol, 1.0 eq.). Crude product was purified by flash chromatography on silica gel by using a 20:1 mixture of pentane/diethylether to provide product with small amounts of impurities (95 mg, 0.16 mmol, 62%).  **$^1\text{H}$  NMR** (300 MHz,  $\text{CDCl}_3$ , 300 K):  $\delta = 7.78 - 7.72$  ( $m$ , 4H,  $\text{CH}_{\text{arom}}$ ), 7.60 ( $t$ ,  $J = 7.3$  Hz, 3H,  $\text{CH}_{\text{arom}}$ ), 7.44 ( $t$ ,  $J = 7.8$  Hz, 4H,  $\text{CH}_{\text{arom}}$ ), 7.39 – 7.30 ( $m$ , 2H,  $\text{CH}_{\text{arom}}$ ), 7.18 – 7.01 ( $m$ , 1H,  $\text{CH}_{\text{arom}}$ ), 5.83 – 5.74 ( $m$ , 1H, OCH), 4.38 – 4.29 ( $m$ , 2H,  $\text{NCH}_2$ ), 1.52 – 0.74 ( $m$ , 18H,  $3 \times \text{CH}_2$ ,  $4 \times \text{CH}_3$ ).  **$^{13}\text{C}$  NMR** (75 MHz,  $\text{CDCl}_3$ , 300K):  $\delta = 138.9$  ( $2 \times \text{C}$ ), 133.6 ( $2 \times \text{CH}$ ), 132.7 (C), 130.9 (CH), 129.5 (CH), 128.8 ( $6 \times \text{CH}$ ), 128.7 ( $4 \times \text{CH}$ ), 127.3 (C), 81.8 (OCH), 60.2 ( $2 \times \text{C}$ ), 51.4 ( $\text{NCH}_2$ ), 40.7 ( $2 \times \text{CH}_2$ ), 34.6 ( $2 \times \text{CH}_3$ ), 20.6 ( $2 \times \text{CH}_3$ ), 17.2 ( $\text{CH}_2$ ). **HRMS** (ESI) exact mass calculated for  $\text{C}_{29}\text{H}_{35}\text{BrN}_2\text{O}_5\text{S}_2\text{H}$  ( $[\text{M}+\text{H}]^+$ ): 635.1244, found: 635.1245. **IR** (neat): 2364 $w$ , 2337 $w$ , 1449 $m$ , 1375 $s$ , 1354 $m$ , 1169 $s$ , 1086 $m$ , 1038 $m$ , 832 $m$ , 758 $m$ , 720 $m$ , 686 $m$ , 580 $s$ , 553 $m$ .

***N*-(phenylsulfonyl)-*N*-(2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)-2-(4-(trifluoromethyl)phenyl)ethyl)benzenesulfonamide (2l)**

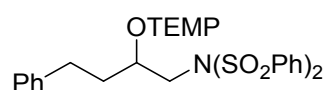


According to **GPI** with 1-(trifluoromethyl)-4-vinylbenzene (43 mg, 0.25 mmol, 1.0 eq.). Crude product was purified by flash chromatography on silica gel by using a 20:1 mixture of pentane/diethylether to provide analytically pure product (61 mg, 0.10 mmol, 39%).  **$^1\text{H}$  NMR** (300 MHz,  $\text{CDCl}_3$ , 300 K):  $\delta = 7.67 - 7.57$  ( $m$ , 5H,  $\text{CH}_{\text{arom}}$ ), 7.56 – 7.40 ( $m$ , 9H,  $\text{CH}_{\text{arom}}$ ), 5.36 – 5.26 ( $m$ , 1H, OCH), 4.36 ( $dd$ ,  $J = 15.0, 11.0$  Hz, 1H,  $\text{NH}_\alpha\text{H}_\beta$ ), 4.11 – 4.02 ( $m$ , 1H,  $\text{NH}_\alpha\text{H}_\beta$ ), 1.53 – 0.65 ( $m$ , 18H,  $3 \times \text{CH}_2$ ,  $4 \times \text{CH}_3$ ).  **$^{13}\text{C}$  NMR** (75 MHz,  $\text{CDCl}_3$ , 300K):  $\delta = 144.1$  (C), 138.4 ( $2 \times \text{C}$ ), 133.9 ( $2 \times \text{CH}$ ), 130.0 ( $q$ ,  $J = 32.0$  Hz, C), 129.4 ( $2 \times \text{CH}$ ), 128.7 ( $8 \times \text{CH}$ ), 125.1 ( $q$ ,  $J = 3.7$  Hz,  $2 \times \text{CH}$ ), 124.2 ( $q$ ,  $J = 272.2$  Hz,  $\text{CF}_3$ ), 83.9 (OCH), 60.1



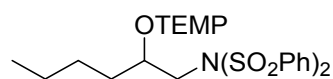
(2 × C), 50.3 (NCH<sub>2</sub>), 40.6 (2 × CH<sub>2</sub>), 34.5 (2 × CH<sub>3</sub>), 20.2 (2 × CH<sub>3</sub>), 17.1 (CH<sub>2</sub>). **HRMS** (ESI) exact mass calculated for C<sub>20</sub>H<sub>35</sub>F<sub>3</sub>N<sub>2</sub>O<sub>5</sub>S<sub>2</sub>H ([M+H]<sup>+</sup>): 625.2012, found: 625.2014. **IR** (neat): 2973<sub>w</sub>, 2933<sub>w</sub>, 1449<sub>m</sub>, 1376<sub>s</sub>, 1362<sub>m</sub>, 1234<sub>s</sub>, 1167<sub>s</sub>, 1123<sub>s</sub>, 1085<sub>m</sub>, 1066<sub>s</sub>, 1019<sub>m</sub>, 829<sub>s</sub>, 786<sub>m</sub>, 743<sub>m</sub>, 720<sub>m</sub>, 686<sub>m</sub>, 602<sub>m</sub>, 582<sub>s</sub>, 550<sub>s</sub>.

***N*-(4-phenyl-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)butyl)-*N*-(phenylsulfonyl)benzenesulfonamide (2m)**



According to **GP1** with but-3-en-1-ylbenzene (33 mg, 0.25 mmol, 1.0 eq.). Crude product was purified by flash chromatography on silica gel by using a 20:1 mixture of pentane/diethylether to provide analytically pure product (101 mg, 0.173 mmol, 69%). **<sup>1</sup>H NMR** (600 MHz, CDCl<sub>3</sub>, 300 K): δ = 8.00 (*dd*, *J* = 8.5, 1.3 Hz, 4H, CH<sub>arom</sub>), 7.70 – 7.59 (*m*, 2H, CH<sub>arom</sub>), 7.55 – 7.49 (*m*, 4H, CH<sub>arom</sub>), 7.29 – 7.25 (*m*, 2H, CH<sub>arom</sub>), 7.20 – 7.16 (*m*, 1H, CH<sub>arom</sub>), 7.14 – 7.11 (*m*, 2H, CH<sub>arom</sub>), 4.36 – 4.24 (*m*, 2H), 3.83 – 3.74 (*m*, 1H), 2.89 (*ddd*, *J* = 13.6, 11.3, 5.2 Hz, 1H), 2.46 (*ddd*, *J* = 13.6, 11.3, 5.6 Hz, 1H), 1.89 – 1.72 (*m*, 2H), 1.48 – 0.94 (*m*, 18H, 3 × CH<sub>2</sub>, 4 × CH<sub>3</sub>). **<sup>13</sup>C NMR** (151 MHz, CDCl<sub>3</sub>, 300K): δ = 142.3 (C), 139.6 (2 × C), 133.8 (2 × CH<sub>2</sub>), 129.0 (4 × CH), 128.4 (4 × CH), 128.4 (2 × CH), 128.3 (2 × CH), 125.7 (CH), 60.7 (C), 59.4 (C), 51.1 (NCH<sub>2</sub>), 40.6 (CH<sub>2</sub>), 40.3 (CH<sub>2</sub>), 34.4 (CH<sub>3</sub>), 34.2 (CH<sub>3</sub>), 33.6 (CH<sub>2</sub>), 31.8 (CH<sub>2</sub>), 20.7 (CH<sub>3</sub>), 20.4 (CH<sub>3</sub>), 17.3 (CH<sub>2</sub>). **HRMS** (ESI) exact mass calculated for C<sub>31</sub>H<sub>40</sub>N<sub>2</sub>O<sub>5</sub>S<sub>2</sub>H ([M+H]<sup>+</sup>): 585.2451, found: 585.2450. **IR** (neat): 2931<sub>w</sub>, 1449<sub>m</sub>, 1377<sub>s</sub>, 1170<sub>s</sub>, 1085<sub>w</sub>, 1043<sub>w</sub>, 795<sub>w</sub>, 719<sub>m</sub>, 868<sub>m</sub>, 581<sub>s</sub>, 551<sub>s</sub>.

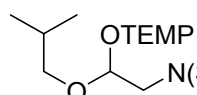
***N*-(phenylsulfonyl)-*N*-(2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)hexyl)benzenesulfonamide (2n)**



According to **GP1** with hex-1-ene (21 mg, 0.25 mmol, 1.0 eq.). Crude product was purified by flash chromatography on silica gel by using a 15:1 mixture of pentane/diethylether to provide analytically pure product (61 mg, 0.12 mmol, 46%). **<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>, 300 K): δ = 8.12 – 8.01 (*m*, 4H, CH<sub>arom</sub>), 7.77 – 7.63 (*m*, 2H, CH<sub>arom</sub>), 7.63 – 7.55 (*m*, 4H, CH<sub>arom</sub>), 4.32 – 4.14 (*m*, 2H), 3.74 (*ddd*, *J* = 13.3, 8.8, 2.7 Hz, 1H), 1.51 – 0.74 (*m*, 27H, 6 × CH<sub>2</sub>, 5 × CH<sub>3</sub>). **<sup>13</sup>C NMR** (125 MHz, CDCl<sub>3</sub>, 300K): δ = 139.4 (2 × C), 133.6 (2 × CH), 128.6 (4 × CH), 128.0 (4 × CH), 77.3 (OCH), 60.2

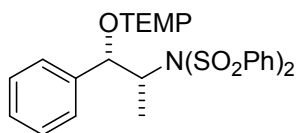
(C), 58.9 (C), 51.2 (NCH<sub>2</sub>), 40.2 (CH<sub>2</sub>), 39.9 (CH<sub>2</sub>), 34.0 (CH<sub>3</sub>), 33.8 (CH<sub>3</sub>), 30.9 (CH<sub>2</sub>), 27.6 (CH<sub>2</sub>), 22.5 (CH<sub>2</sub>), 20.2 (CH<sub>3</sub>), 20.0 (CH<sub>3</sub>), 16.9 (CH<sub>2</sub>), 13.7 (CH<sub>3</sub>). **HRMS** (ESI) exact mass calculated for C<sub>27</sub>H<sub>40</sub>N<sub>2</sub>O<sub>5</sub>S<sub>2</sub>H ([M+H]<sup>+</sup>): 537.2451, found: 537.2452. **IR** (neat): 2932w, 1449m, 1377s, 1361m, 1170s, 1086m, 805w, 753w, 743w, 720m, 687m, 582s, 551s.

***N*-(2-isobutoxy-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)-*N*-(phenylsulfonyl)benzenesulfonamide (2o)**



According to **GP1** with 2-methyl-1-(vinylloxy)propane (25 mg, 0.25 mmol, 1.0 eq.). Crude product was purified by flash chromatography on silica gel by using a 15:1 mixture of pentane/diethylether to provide analytically pure product (106 mg, 0.193 mmol, 77%). **<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>, 300 K): δ = 8.08 – 7.81 (*m*, 4H, CH<sub>arom</sub>), 7.59 – 7.52 (*m*, 2H, CH<sub>arom</sub>), 7.44 (*dd*, *J* = 8.4, 6.8 Hz, 4H, CH<sub>arom</sub>), 5.03 (*dd*, *J* = 7.4, 3.8 Hz, 1H, OCH), 3.95 – 3.70 (*m*, 2H, OCH<sub>2</sub>), 3.53 (*dd*, *J* = 9.1, 7.1 Hz, 1H, NH<sub>α</sub>H<sub>β</sub>), 2.77 (*dd*, *J* = 9.1, 6.8 Hz, 1H, NH<sub>α</sub>H<sub>β</sub>), 1.60 – 1.47 (*m*, 1H, CH), 1.45 – 0.93 (*m*, 18H, 3 × CH<sub>2</sub>, 4 × CH<sub>3</sub>), 0.68 (*dd*, *J* = 6.7, 2.3 Hz, 6H, 2 × CH<sub>3</sub>). **<sup>13</sup>C NMR** (75 MHz, CDCl<sub>3</sub>, 300K): δ = 139.9 (2 × C), 133.7 (2 × CH), 128.9 (4 × CH), 128.5 (4 × CH), 103.8 (OCH), 77.9 (OCH<sub>2</sub>), 60.5 (C), 59.8 (C), 50.3 (NCH<sub>2</sub>), 40.4 (2 × CH<sub>2</sub>), 33.9 (CH<sub>3</sub>), 33.4 (CH<sub>3</sub>), 28.4 (CH), 20.6 (CH<sub>3</sub>), 20.2 (CH<sub>3</sub>), 19.5 (CH<sub>3</sub>), 19.4 (CH<sub>3</sub>), 17.2 (CH<sub>2</sub>). **HRMS** (ESI) exact mass calculated for C<sub>27</sub>H<sub>40</sub>N<sub>2</sub>O<sub>6</sub>S<sub>2</sub>H ([M+H]<sup>+</sup>): 553.2401, found: 553.2388. **IR** (neat): 2931m, 1449m, 1378s, 1169s, 1086m, 889m, 792m, 742m, 720m, 686m, 584s, 551s.

***N*-(1-phenyl-1-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)propan-2-yl)-*N*-(phenylsulfonyl)benzenesulfonamide (2q)**

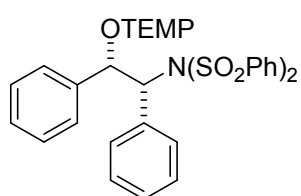


According to **GP1** (*E*)-prop-1-en-1-ylbenzene (30 mg, 0.25 mmol, 1.0 eq.). Crude product was purified by flash chromatography on silica gel by using a 25:1 mixture of pentane/diethylether to provide analytically pure product as a 15:1 mixture of diastereoisomers (107 mg, 0.183 mmol, 73%). Analytical data is given for the major diastereoisomer: **<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>, 300 K): δ = 7.93 (*d*, *J* = 7.7 Hz, 2H, CH<sub>arom</sub>), 7.60 (*t*, *J* = 7.3 Hz, 1H, CH<sub>arom</sub>), 7.53 – 7.39 (*m*, 4H, CH<sub>arom</sub>), 7.34 (*t*, *J* = 7.3 Hz, 1H, CH<sub>arom</sub>), 7.29 – 7.08 (*m*, 5H, CH<sub>arom</sub>), 6.99 (*d*, *J* = 7.9 Hz, 2H, CH<sub>arom</sub>), 5.48 (*d*, *J* = 9.6 Hz, 1H, OCH), 4.66 (*m*, *J* = 9.5, 6.8 Hz, 1H, NCH), 1.46 (*d*, *J* =

6.8 Hz, 3H, CH<sub>3</sub>), 1.37 – 0.08 (*m*, 18H, 3 × CH<sub>2</sub>, 4 × CH<sub>3</sub>). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>, 300K): δ = 141.9 (C), 138.9 (C), 137.8 (C), 134.1 (CH), 133.0 (CH), 132.5 (2 × CH), 129.4 (2 × CH), 129.1 (2 × CH), 128.3 (2 × CH), 128.1 (CH), 127.9 (2 × CH), 127.5 (2 × CH), 86.2 (OCH), 60.3 (C), 60.0 (NCH), 59.2 (C), 40.8 (2 × CH<sub>2</sub>), 34.8 (CH<sub>3</sub>), 33.1 (CH<sub>3</sub>), 20.7 (2 × CH<sub>3</sub>), 19.0 (CH<sub>3</sub>), 17.1 (CH<sub>2</sub>). HRMS (ESI) exact mass calculated for C<sub>30</sub>H<sub>38</sub>N<sub>2</sub>O<sub>6</sub>S<sub>2</sub>H ([M+H]<sup>+</sup>): 571.2295, found: 571.2295. IR (neat): 2932w, 1449m, 1376s, 1347m, 1166s, 1083m, 855m, 721m, 685m, 593s.

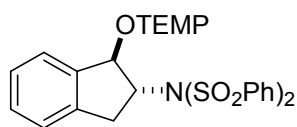
Using (*Z*)-prop-1-en-1-ylbenzene afforded the same product (105 mg, 0.180 mmol, 72%)

***N*-(1,2-diphenyl-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)-*N*-(phenylsulfonyl)benzenesulfonamide (2r)**



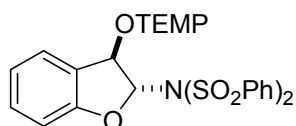
According to **GP1** with (*E*)-1,2-diphenylethene (40 mg, 0.25 mmol, 1.0 eq.). Crude product was purified by flash chromatography on silica gel by using a 25:1 mixture of pentane/diethylether to provide analytically pure product as a 20:1 mixture of diastereoisomers (91 mg, 0.15 mmol, 58%). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>, 300 K): δ = 8.22 (*d*, *J* = 16.8 Hz, 3H, CH<sub>arom</sub>), 8.07 (*s*, 2H, CH<sub>arom</sub>), 7.89 (*s*, 6H, CH<sub>arom</sub>), 7.61 (*d*, *J* = 15.4 Hz, 7H, CH<sub>arom</sub>), 7.37 (*s*, 2H, CH<sub>arom</sub>), 6.50 (*d*, *J* = 9.3, 1H, CH), 6.18 (*d*, *J* = 9.1 Hz, 1H, CH), 1.89 – 0.45 (*m*, 18H, 3 × CH<sub>2</sub>, 4 × CH<sub>3</sub>). <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>, 300K): δ = 142.6 (C), 138.9 (C), 138.4 (C), 138.1 (C), 134.3 (CH), 133.5 (CH), 130.8 (2 × CH), 129.6 (2 × CH), 129.1 (4 × CH), 128.8 (2 × CH), 128.5 (5 × CH), 128.3 (2 × CH), 128.0 (CH), 84.1 (OCH), 68.6 (NCH), 61.2 (C), 59.4 (C), 41.5 (CH<sub>2</sub>), 40.7 (CH<sub>2</sub>), 34.9 (CH<sub>3</sub>), 33.2 (CH<sub>3</sub>), 21.2 (CH<sub>3</sub>), 20.4 (CH<sub>3</sub>), 17.6 (CH<sub>2</sub>). HRMS (ESI) exact mass calculated for C<sub>35</sub>H<sub>40</sub>N<sub>2</sub>O<sub>5</sub>S<sub>2</sub>H ([M+H]<sup>+</sup>): 632.2451, found: 632.2447. IR (neat): 2972w, 2934w, 1448m, 1375m, 1357m, 1339m, 1165s, 1131m, 1081m, 973m, 833w, 843w, 751m, 721m, 689m, 684m, 608s, 573s, 559s, 539s.

***N*-(phenylsulfonyl)-*N*-(1-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)-2,3-dihydro-1H-inden-2-yl)benzenesulfonamide (2s)**



According to **GP1** with indene (29 mg, 0.25 mmol, 1.0 eq.). Crude product was purified by flash chromatography on silica gel by using a 20:1 mixture of pentane/diethylether to provide analytically pure product as colorless oil as a single diastereoisomer (98 mg, 0.17 mmol, 69%). **<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>, 300 K): δ = 8.15 – 8.02 (*m*, 4H, CH<sub>arom</sub>), 7.73 (*d*, *J* = 7.1 Hz, 1H, CH<sub>arom</sub>), 7.70 – 7.63 (*m*, 2H, CH<sub>arom</sub>), 7.56 (*t*, *J* = 7.6 Hz, 4H, CH<sub>arom</sub>), 7.29 – 7.17 (*m*, 2H, CH<sub>arom</sub>), 7.07 (*d*, *J* = 7.2 Hz, 1H, CH<sub>arom</sub>), 6.00 (*d*, *J* = 3.2 Hz, 1H, OCH), 5.29 – 5.23 (*m*, 1H, NCH), 3.31 – 2.98 (*m*, 2H, CH<sub>2</sub>), 1.55 – 0.48 (*m*, 18H, 3 × CH<sub>2</sub>, 4 × CH<sub>3</sub>). **<sup>13</sup>C NMR** (75 MHz, CDCl<sub>3</sub>, 300K): δ = 141.4 (C), 140.9 (C), 140.6 (2 × C), 133.7 (2 × CH), 129.0 (4 × CH), 128.4 (CH), 128.4 (5 × CH), 125.8 (CH), 123.9 (CH), 89.5 (OCH), 66.6 (NCH), 60.4 (C), 59.7 (C), 40.2 (CH<sub>2</sub>), 39.8 (CH<sub>2</sub>), 37.3 (CH<sub>2</sub>), 33.7 (CH<sub>3</sub>), 32.9 (CH<sub>3</sub>), 20.8 (CH<sub>3</sub>), 20.7 (CH<sub>3</sub>), 17.3 (CH<sub>2</sub>). **HRMS** (ESI) exact mass calculated for C<sub>30</sub>H<sub>26</sub>N<sub>2</sub>O<sub>5</sub>S<sub>2</sub>H ([M+H]<sup>+</sup>): 569.2138, found: 569.2136. **IR** (neat): 2931*w*, 1448*m*, 1370*m*, 1168*s*, 1132*w*, 1084*m*, 909*m*, 849*m*, 751*m*, 721*s*, 686*m*, 582*s*, 555*s*.

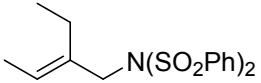
***N*-(phenylsulfonyl)-*N*-(3-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)-2,3-dihydrobenzofuran-2-yl)benzenesulfonamide (2t)**



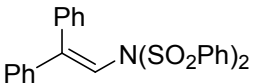
According to **GP1** with benzofuran (29 mg, 0.25 mmol, 1.0 eq.). Crude product was purified by flash chromatography on silica gel by using a 20:1 mixture of pentane/diethylether to provide analytically pure product as colorless oil as a single diastereoisomer (45 mg, 80 μmol, 32%). **<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>, 300 K): 8.11 – 8.04 (*m*, 4H, CH<sub>arom</sub>), 7.71 – 7.62 (*m*, 2H, CH<sub>arom</sub>), 7.55 (*dd*, *J* = 8.4, 6.8 Hz, 5H, CH<sub>arom</sub>), 7.30 – 7.22 (*m*, 1H, CH<sub>arom</sub>), 6.96 (*td*, *J* = 7.5, 1.0 Hz, 1H, CH<sub>arom</sub>), 6.76 (*d*, *J* = 8.1 Hz, 1H, CH, CH<sub>arom</sub>), 6.73 (*d*, *J* = 2.6 Hz, 1H, CH), 6.02 (*d*, *J* = 2.6 Hz, 1H, CH), 1.48 – 0.46 (*m*, 18H, 3 × CH<sub>2</sub>, 4 × CH<sub>3</sub>). **<sup>13</sup>C NMR** (75 MHz, CDCl<sub>3</sub>, 300K): 160.1 (C), 140.7 (2 × C), 134.0 (2 × CH), 130.3 (CH), 129.1 (4 × CH), 128.6 (5 × CH), 126.2 (C), 121.0 (CH), 109.6 (CH), 96.8 (OCH), 87.5 (NCH), 60.7 (C), 60.0 (C), 40.3 (CH<sub>2</sub>), 40.0 (CH<sub>2</sub>), 34.0 (CH<sub>3</sub>), 32.9 (CH<sub>3</sub>), 20.9 (2 × CH<sub>3</sub>), 17.3 (CH<sub>2</sub>). **HRMS** (ESI) exact mass calculated for C<sub>30</sub>H<sub>26</sub>N<sub>2</sub>O<sub>5</sub>S<sub>2</sub>H ([M+H]<sup>+</sup>): 571.1931, found: 571.1941. **IR** (neat):

2933w, 1600w, 1467w, 1376m, 1241w, 1174s, 1085m, 1003m, 968m, 907m, 817m, 732s, 684m, 578s, 546s.

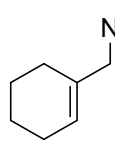
**(E)-N-(2-ethylbut-2-en-1-yl)-N-(phenylsulfonyl)benzenesulfonamide (4a)**

 According to **GPI** with (*E*)-1,2-diphenylethene (40 mg, 0.25 mmol, 1.0 eq.). Crude product was purified by flash chromatography on silica gel by using a 25:1 mixture of pentane/diethylether to provide analytically pure product (71 mg, 0.19 mmol, 75%). **<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>, 300 K): δ = 8.10 – 7.96 (*m*, 4H, CH<sub>arom</sub>), 7.66 – 7.58 (*m*, 2H, CH<sub>arom</sub>), 7.53 (*t*, *J* = 7.6 Hz, 4H, CH<sub>arom</sub>), 5.44 (*q*, *J* = 6.8 Hz, 1H, =CH), 4.40 (*s*, 2H, NCH<sub>2</sub>), 1.69 (*q*, *J* = 7.5 Hz, 2H, CH<sub>2</sub>), 1.55 – 1.43 (*m*, 3H, CH<sub>3</sub>), 0.89 (*t*, *J* = 7.5 Hz, 3H, CH<sub>3</sub>). **<sup>13</sup>C NMR** (75 MHz, CDCl<sub>3</sub>, 300K): δ = 140.5 (2 × C), 134.8 (C), 133.5 (2 × CH), 128.7 (4 × CH), 128.3 (4 × CH), 125.7 (CH), 54.6 (NCH<sub>2</sub>), 19.8 (CH<sub>2</sub>), 12.9 (CH<sub>3</sub>), 12.3 (CH<sub>3</sub>). **HRMS** (ESI) exact mass calculated for C<sub>18</sub>H<sub>21</sub>NO<sub>4</sub>S<sub>2</sub>Na ([M+Na]<sup>+</sup>): 402.0804, found: 402.0799. **IR** (neat): 2967w, 2934w, 1449m, 1374s, 1354m, 1168s, 1086m, 1030w, 813m, 720m, 686m, 597s, 551s.

**N-(2,2-diphenylvinyl)-N-(phenylsulfonyl)benzenesulfonamide (4b)**

 According to **GPI** with ethene-1,1-diylidibenzene (45 mg, 0.25 mmol, 1.0 eq.). Crude product was purified by flash chromatography on silica gel by using a 10:1 mixture of pentane/diethylether to provide analytically pure product (97 mg, 0.20 mmol, 81%). **<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>, 300 K): δ = 7.77 – 7.61 (*m*, 4H, CH<sub>arom</sub>), 7.61 – 7.53 (*m*, 2H, CH<sub>arom</sub>), 7.43 – 7.16 (*m*, 14H, CH<sub>arom</sub>), 6.13 (*s*, 1H, CH). **<sup>13</sup>C NMR** (75 MHz, CDCl<sub>3</sub>, 300K): δ = 152.4 (C), 134.0 (C), 138.8 (2 × C), 136.9 (C), 133.9 (2 × CH), 130.0 (2 × CH), 129.2 (CH), 128.9 (4 × CH), 128.8 (2 × CH), 128.8 (4 × CH), 128.4 (2 × CH), 128.4 (CH), 128.3 (2 × CH), 116.4 (CH). **HRMS** (ESI) exact mass calculated for C<sub>26</sub>H<sub>21</sub>NO<sub>4</sub>S<sub>2</sub>Na ([M+Na]<sup>+</sup>): 498.0804, found: 498.0807. **IR** (neat): 3062w, 1496w, 1448m, 1373s, 1354m, 1167s, 1083m, 1028w, 1000w, 897m, 809m, 753m, 720m, 683s, 673w, 574s, 546s.

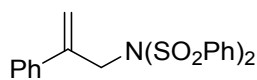
### *N*-(cyclohex-1-en-1-ylmethyl)-*N*-(phenylsulfonyl)benzenesulfonamide (**4c**)



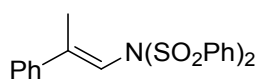
According to **GP1** with methylenecyclohexane (24 mg, 0.25 mmol, 1.0 eq.). Crude product was purified by flash chromatography on silica gel by using a 10:1 mixture of pentane/diethylether to provide analytically pure product (47 mg, 0.12 mmol, 47%). **<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>, 300 K): δ = 8.14 – 7.92 (*m*, 4H, CH<sub>arom</sub>), 7.66 – 7.60 (*m*, 2H, CH<sub>arom</sub>), 7.57 – 7.50 (*m*, 4H, CH<sub>arom</sub>), 5.70 (*s*, 1H, CH), 4.38 (*s*, 2H, NCH<sub>2</sub>), 1.95 – 1.85 (*m*, 2H), 1.73 (*s*, 2H), 1.40 – 1.22 (*m*, 4H). **<sup>13</sup>C NMR** (75 MHz, CDCl<sub>3</sub>, 300K): δ = 140.5 (2 × C), 133.8 (C), 133.6 (2 × CH), 130.9 (CH), 128.9 (2 × CH), 128.8 (3 × CH), 128.2 (3 × CH), 56.0 (NCH<sub>2</sub>), 25.4 (CH<sub>2</sub>), 25.1 (CH<sub>2</sub>), 21.9 (CH<sub>2</sub>), 21.7 (CH<sub>2</sub>). **HRMS** (ESI) exact mass calculated for C<sub>19</sub>H<sub>21</sub>NO<sub>4</sub>S<sub>2</sub>Na ([M+Na]<sup>+</sup>): 414.0804, found: 414.0800. **IR** (neat): 2929*w*, 1448*m*, 1372*s*, 1354*m*, 1167*s*, 1086*m*, 1021*m*, 996*m*, 915*m*, 839*m*, 801*m*, 719*s*, 685*s*, 570*s*, 548*s*.

### *N*-(2-phenylallyl)-*N*-(phenylsulfonyl)benzenesulfonamide (**4d**) and (*E*)-*N*-(2-phenylprop-1-en-1-yl)-*N*-(phenylsulfonyl)benzenesulfonamide (**4d'**)

According to **GP1** with prop-1-en-2-ylbenzene (30 mg, 0.25 mmol, 1.0 eq.). Crude product was purified by flash chromatography on silica gel by using a 30:1 mixture of pentane/diethylether to provide analytically pure product **4d** (37 mg, 90 μmol, 36%) along with isomer **4d'** (11 mg, 30 μmol, 11%).



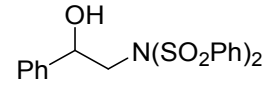
**4d**: **<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>, 300 K): δ = 8.10 – 8.00 (*m*, 4H, CH<sub>arom</sub>), 7.71 – 7.59 (*m*, 2H, CH<sub>arom</sub>), 7.52 (*t*, *J* = 7.7 Hz, 4H, CH<sub>arom</sub>), 7.31 (*s*, 5H, CH<sub>arom</sub>), 5.29 (*s*, 1H, =CH<sub>α</sub>H<sub>β</sub>), 5.11 (*s*, 1H, =CH<sub>α</sub>H<sub>β</sub>), 4.82 (*s*, 2H, NCH<sub>2</sub>). **<sup>13</sup>C NMR** (75 MHz, CDCl<sub>3</sub>, 300K): δ = 141.9 (C), 139.9 (2 × C), 138.7 (C), 133.9 (2 × CH), 128.9 (4 × CH), 128.5 (4 × CH), 128.4 (2 × CH), 128.1 (CH), 126.6 (2 × CH), 116.2 (CH<sub>2</sub>), 52.3 (CH<sub>2</sub>). **HRMS** (ESI) exact mass calculated for C<sub>21</sub>H<sub>19</sub>NO<sub>4</sub>S<sub>2</sub>Na ([M+Na]<sup>+</sup>): 436.0648, found: 436.0641. **IR** (neat): 3064*w*, 1449*m*, 1377*s*, 1358*m*, 1169*s*, 1085*m*, 913*w*, 815*m*, 754*m*, 722*m*, 686*m*, 629*m*, 578*s*, 549*s*.



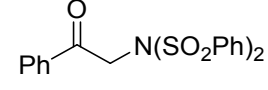
**4d'**: **<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>, 300 K): δ = 8.10 – 7.91 (*m*, 4H, CH<sub>arom</sub>), 7.74 – 7.62 (*m*, 2H, CH<sub>arom</sub>), 7.55 (*d*, *J* = 7.3 Hz, 4H, CH<sub>arom</sub>), 7.45 – 7.31 (*m*, 5H, CH<sub>arom</sub>), 6.09 (*d*, *J* = 1.4 Hz, 1H, NCH), 1.73 (*d*, *J* = 1.4, 3H, CH<sub>3</sub>).

$^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ , 300K):  $\delta$  = 150.0 (C), 139.7 (2  $\times$  C), 139.2 (C), 134.0 (2  $\times$  CH), 129.2 (4  $\times$  CH), 129.0 (CH), 128.7 (2  $\times$  CH), 128.4 (4  $\times$  CH), 126.5 (2  $\times$  CH), 117.2 (CH), 16.6 ( $\text{CH}_3$ ). HRMS (ESI) exact mass calculated for  $\text{C}_{21}\text{H}_{19}\text{NO}_4\text{S}_2\text{Na}$  ( $[\text{M}+\text{Na}]^+$ ): 436.0648, found: 436.0639. IR (neat): 3065w, 1449m, 1374s, 1355m, 1085m, 1064w, 909w, 811m, 720m, 685m, 587s, 551s.

### *N*-(2-hydroxy-2-phenylethyl)-*N*-(phenylsulfonyl)benzenesulfonamide (5)

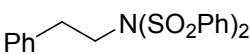
 **2a** (111 mg, 0.2 mmol, 1.0 eq.) was dissolved in a 1:3 mixture of  $\text{H}_2\text{O}/\text{AcOH}$  (0.6 mL/2.0 mL). To this solution Nano-Zn (66 mg, 1.0 mmol, 5.0 eq) was added and the solution was stirred at room temperature. After 3 h, additional Nano-Zn (66 mg, 1.0 mmol, 5.0 eq) was added and the solution was stirred over night at room temperature. NaOH (5.0 mL, 0.5 M) was added to quench the reaction. The mixture was extracted three times with DCM. The combined organic layers were washed with brine and dried over  $\text{MgSO}_4$ . The crude product was purified by flash column chromatography on silica gel by using a 5:1 mixture of pentane/diethylether as an eluent to provide analytical pure product as a colorless solid (83 mg, 0.20 mmol, 99%).  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ , 300 K):  $\delta$  = 8.16 – 8.02 (*m*, 4H,  $\text{CH}_{\text{arom}}$ ), 7.66 (*dd*,  $J$  = 8.3, 6.5 Hz, 2H,  $\text{CH}_{\text{arom}}$ ), 7.55 (*t*,  $J$  = 7.7 Hz, 4H,  $\text{CH}_{\text{arom}}$ ), 7.46 – 7.29 (*m*, 5H,  $\text{CH}_{\text{arom}}$ ), 5.15 (*t*,  $J$  = 6.2 Hz, 1H, OCH), 3.91 – 3.84 (*m*, 2H,  $\text{NCH}_2$ ), 2.91 (*s*, 1H, OH).  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ , 300K):  $\delta$  = 140.8 (C), 139.4 (2  $\times$  C), 134.1 (2  $\times$  CH), 129.1 (4  $\times$  CH), 128.7 (2  $\times$  CH), 128.5 (4  $\times$  CH), 128.2 (CH), 125.9 (2  $\times$  CH), 72.8 (OCH), 55.7 ( $\text{NCH}_2$ ). HRMS (ESI) exact mass calculated for  $\text{C}_{20}\text{H}_{19}\text{NO}_5\text{S}_2\text{Na}$  ( $[\text{M}+\text{Na}]^+$ ): 440.0597, found: 440.0597. IR (neat): 3066w, 1449m, 1371s, 1166s, 1084m, 1026m, 904m, 794m, 720m, 685m, 582s, 550s.

### *N*-(2-oxo-2-phenylethyl)-*N*-(phenylsulfonyl)benzenesulfonamide (6)

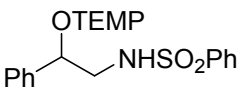
 **2a** (111 mg, 0.2 mmol, 1.0 eq.) and *m*CPBA (73 mg, 0.3 mmol, 1.5 eq., 70-75% in  $\text{H}_2\text{O}$ ) were dissolved in  $\text{CH}_2\text{Cl}_2$  (4 mL) and the reaction mixture was stirred over night at room temperature. Then water (10 ml) was added and the mixture was extracted three times with DCM. The combined organic layers were dried over  $\text{MgSO}_4$ . Crude product was purified by flash column chromatography on silica gel by using a 20:1 mixture of pentane/diethylether as an eluent to provide analytical pure product (74 mg,

0.18 mmol, 89%). **<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>, 300 K): δ = 8.11 – 8.05 (*m*, 3H, CH<sub>arom</sub>), 8.01 – 7.97 (*m*, 1H, CH<sub>arom</sub>), 7.89 – 7.85 (*m*, 2H, CH<sub>arom</sub>), 7.70 – 7.63 (*m*, 2H, CH<sub>arom</sub>), 7.61 – 7.53 (*m*, 4H, CH<sub>arom</sub>), 7.51 – 7.46 (*m*, 2H, CH<sub>arom</sub>), 7.45 – 7.41 (*m*, 1H, CH<sub>arom</sub>), 5.20 (*s*, 2H, CH<sub>2</sub>). **<sup>13</sup>C NMR** (75 MHz, CDCl<sub>3</sub>, 300K): δ = 191.0 (C), 170.5 (C), 139.4 (2 × C), 134.2 (2 × CH), 134.0 (CH), 133.8 (CH), 130.3 (CH), 129.9 (CH), 129.0 (3 × CH), 128.9 (3 × CH), 128.4 (CH), 128.1 (2 × CH), 53.7 (CH<sub>2</sub>). **HRMS** (ESI) exact mass calculated for C<sub>20</sub>H<sub>17</sub>NO<sub>5</sub>S<sub>2</sub>Na ([M+Na]<sup>+</sup>): 438.0440, found: 438.0432. **IR** (neat): 3067*w*, 1707*m*, 1449*m*, 1373*s*, 1168*s*, 1084*m*, 980*w*, 824*m*, 750*s*, 721*m*, 684*s*, 583*s*, 544*s*.

### *N*-phenethyl-*N*-(phenylsulfonyl)benzenesulfonamide (7)

 **2a** (111 mg, 0.20 mmol, 1.0 eq) and thiophenol (24 mg, 0.22 mmol, 1.1 eq.) were dissolved in *tert*-butyl benzene (3 mL). The reaction mixture was heated and stirred at 120 °C over night. After removal of the solvent, the crude product was purified directly by flash column chromatography on silica gel by using a 50:1 mixture of pentane/diethylether as an eluent to provide analytical pure product as colorless oil (77 mg, 0.19 mmol, 96%). **<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>, 300 K): δ = 8.04 – 7.82 (*m*, 4H, CH<sub>arom</sub>), 7.59 – 7.52 (*m*, 2H, CH<sub>arom</sub>), 7.46 (*dd*, *J* = 8.4, 6.8 Hz, 4H, CH<sub>arom</sub>), 7.24 – 7.08 (*m*, 5H, CH<sub>arom</sub>), 3.84 – 3.73 (*m*, 2H, CH<sub>2</sub>), 2.96 – 2.85 (*m*, 2H, CH<sub>2</sub>). **<sup>13</sup>C NMR** (75 MHz, CDCl<sub>3</sub>, 300K): δ = 140.0 (2 × C), 137.6 (C), 134.0 (2 × CH), 129.3 (4 × CH), 129.0 (2 × CH), 128.8 (2 × CH), 128.3 (4 × CH), 127.0 (CH), 50.6 (CH<sub>2</sub>), 36.7 (CH<sub>2</sub>). **HRMS** (ESI) exact mass calculated for C<sub>20</sub>H<sub>19</sub>NO<sub>4</sub>S<sub>2</sub>Na ([M+Na]<sup>+</sup>): 424.0648, found: 424.0643. **IR** (neat): 3065*w*, 1449*m*, 1375*s*, 1168*s*, 1087*m*, 1065*m*, 1024*w*, 856*m*, 740*m*, 720*m*, 579*s*, 550*s*.

### *N*-(2-phenyl-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)ethyl)benzenesulfonamide (8)

 Into a flask containing **2a** (111 mg, 0.20 mmol, 1.0 eq) in 3.2 ml DMF at room temperature was added 1 ml HOAc/NaOAc buffer solution (8 mol/L). Magnesium turnings (72 mg, 3.0 mmol, 15 eq.) were added portionwise. The reaction mixture was stirred 5 h at room temperature. Then water (5 ml) was added. The mixture was extracted with diethylether and the combined organic layers were dried over MgSO<sub>4</sub>. After removal of the solvent, the crude product was purified by flash column chromatography on silica gel by using a 20:1 mixture of pentane/diethylether as an eluent to



provide analytical pure product (83 mg, 0.20 mmol, 99%). **<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>, 300 K): δ = 7.80 – 7.73 (*m*, 2H, CH<sub>arom</sub>), 7.58 – 7.51 (*m*, 1H, CH<sub>arom</sub>), 7.46 (*dd*, *J* = 8.3, 6.5 Hz, 2H, CH<sub>arom</sub>), 7.32 – 7.28 (*m*, 3H, CH<sub>arom</sub>), 7.23 – 7.20 (*m*, 2H, CH<sub>arom</sub>), 5.78 (*t*, *J* = 5.9 Hz, 1H, NH), 4.94 (*dd*, *J* = 6.9, 5.3 Hz, 1H, OCH), 3.55 – 3.44 (*m*, 1H, NH<sub>α</sub>H<sub>β</sub>), 3.29 (*dd*, *J* = 12.3, 6.1 Hz, 1H, NH<sub>α</sub>H<sub>β</sub>), 1.48 – 0.88 (*m*, 18H, 3 × CH<sub>2</sub>, 4 × CH<sub>3</sub>). **<sup>13</sup>C NMR** (75 MHz, CDCl<sub>3</sub>, 300K): δ = 140.2 (C), 139.8 (2 × C), 132.6 (CH), 129.1 (2 × CH), 128.6 (2 × CH), 128.2 (CH), 127.2 (2 × CH), 127.1 (2 × CH), 83.4 (OCH), 61.0 (C), 60.2 (C), 48.9 (NCH<sub>2</sub>), 40.5 (2 × CH<sub>2</sub>), 34.2 (CH<sub>3</sub>), 33.4 (CH<sub>3</sub>), 20.8 (2 × CH<sub>3</sub>), 17.2 (CH<sub>2</sub>). **HRMS** (ESI) exact mass calculated for C<sub>23</sub>H<sub>32</sub>N<sub>2</sub>O<sub>3</sub>SH ([M+H]<sup>+</sup>): 417.2206, found: 417.2199. **IR** (neat): 2931*m*, 1447*m*, 1329*m*, 1163*s*, 1094*m*, 754*m*, 690*m*, 582*m*.

### **X-Ray crystallographic data:**

**X-Ray diffraction:** Data sets were collected with a D8 Venture Dual Source 100 CMOS diffractometer. Programs used: data collection: APEX2 V2014.5-0 (Bruker AXS Inc., 2014);<sup>[1]</sup> cell refinement: SAINT V8.34A (Bruker AXS Inc., 2013);<sup>[1]</sup> data reduction: SAINT V8.34A (Bruker AXS Inc., 2013);<sup>[1]</sup> absorption correction, SADABS V2014/2 (Bruker AXS Inc., 2014);<sup>[1]</sup> structure solution SHELXT-2014 (Sheldrick, 2014);<sup>[2]</sup> structure refinement SHELXL-2014 (Sheldrick, 2014)<sup>[2]</sup> and graphics, XP (Bruker AXS Inc., 2014).<sup>[2]</sup> *R*-values are given for observed reflections, and *wR*<sup>2</sup> values are given for all reflections.

**X-ray crystal structure analysis of 10 (stu7058):** A colorless plate-like specimen of C<sub>30</sub>H<sub>38</sub>N<sub>2</sub>O<sub>5</sub>S<sub>2</sub>, approximate dimensions 0.055 mm x 0.087 mm x 0.150 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured. A total of 1349 frames were collected. The total exposure time was 20.46 hours. The frames were integrated with the Bruker SAINT software package using a wide-frame algorithm. The integration of the data using a monoclinic unit cell yielded a total of 38943 reflections to a maximum θ angle of 66.59° (0.84 Å resolution), of which 9985 were independent (average redundancy 3.900, completeness = 100.0%, *R*<sub>int</sub> = 6.85%, *R*<sub>sig</sub> = 5.71%) and 8814 (88.27%) were greater than 2σ(*F*<sup>2</sup>). The final cell constants of *a* = 13.0848(4) Å, *b* = 13.5713(4) Å, *c* = 16.2698(5) Å, β = 94.205(2)°, volume = 2881.38(15) Å<sup>3</sup>, are based upon the refinement of the XYZ-centroids of 9946 reflections above 20 σ(*I*) with 6.773° < 2θ < 136.3°. Data were corrected for absorption effects using the multi-scan method (SADABS). The ratio of minimum to

maximum apparent transmission was 0.879. The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.7520 and 0.8970. The structure was solved and refined using the Bruker SHELXTL Software Package, using the space group  $P2_1$ , with  $Z = 4$  for the formula unit,  $C_{30}H_{38}N_2O_5S_2$ . The final anisotropic full-matrix least-squares refinement on  $F^2$  with 713 variables converged at  $R1 = 3.30\%$ , for the observed data and  $wR2 = 6.89\%$  for all data. The goodness-of-fit was 1.030. The largest peak in the final difference electron density synthesis was  $0.228 \text{ e}/\text{\AA}^3$  and the largest hole was  $-0.260 \text{ e}/\text{\AA}^3$  with an RMS deviation of  $0.041 \text{ e}/\text{\AA}^3$ . On the basis of the final model, the calculated density was  $1.316 \text{ g}/\text{cm}^3$  and  $F(000)$ , 1216  $e^-$ .

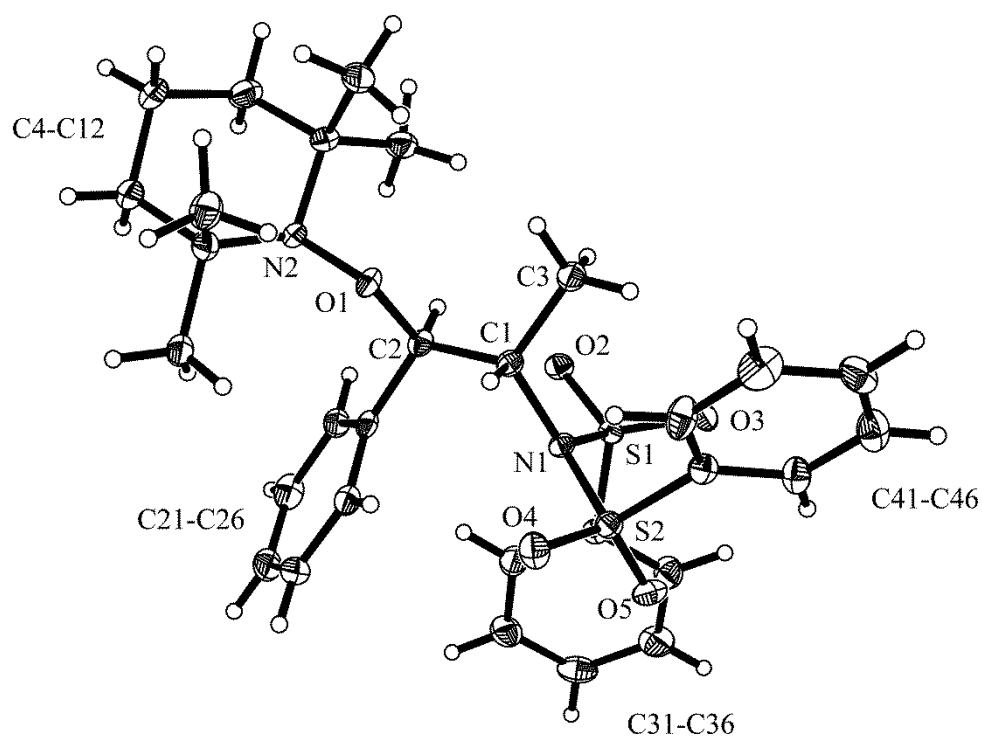
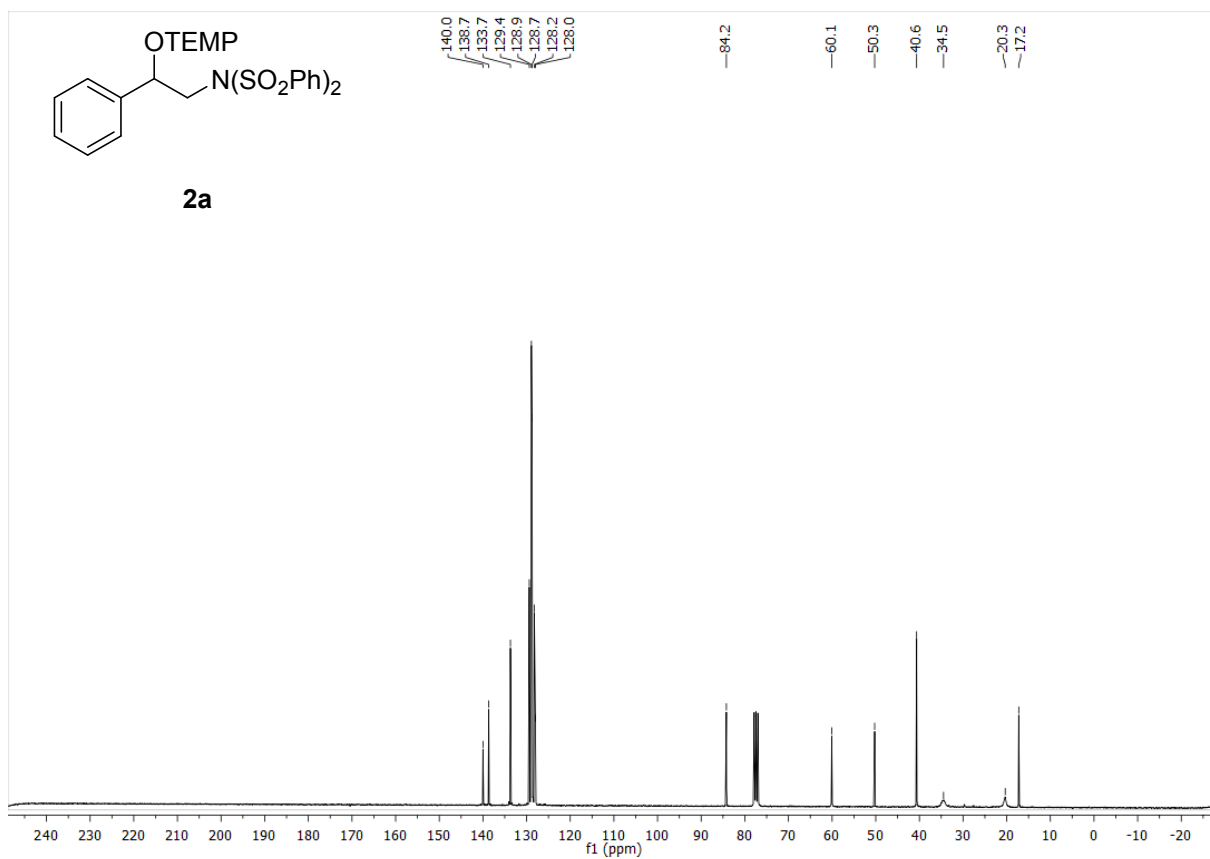
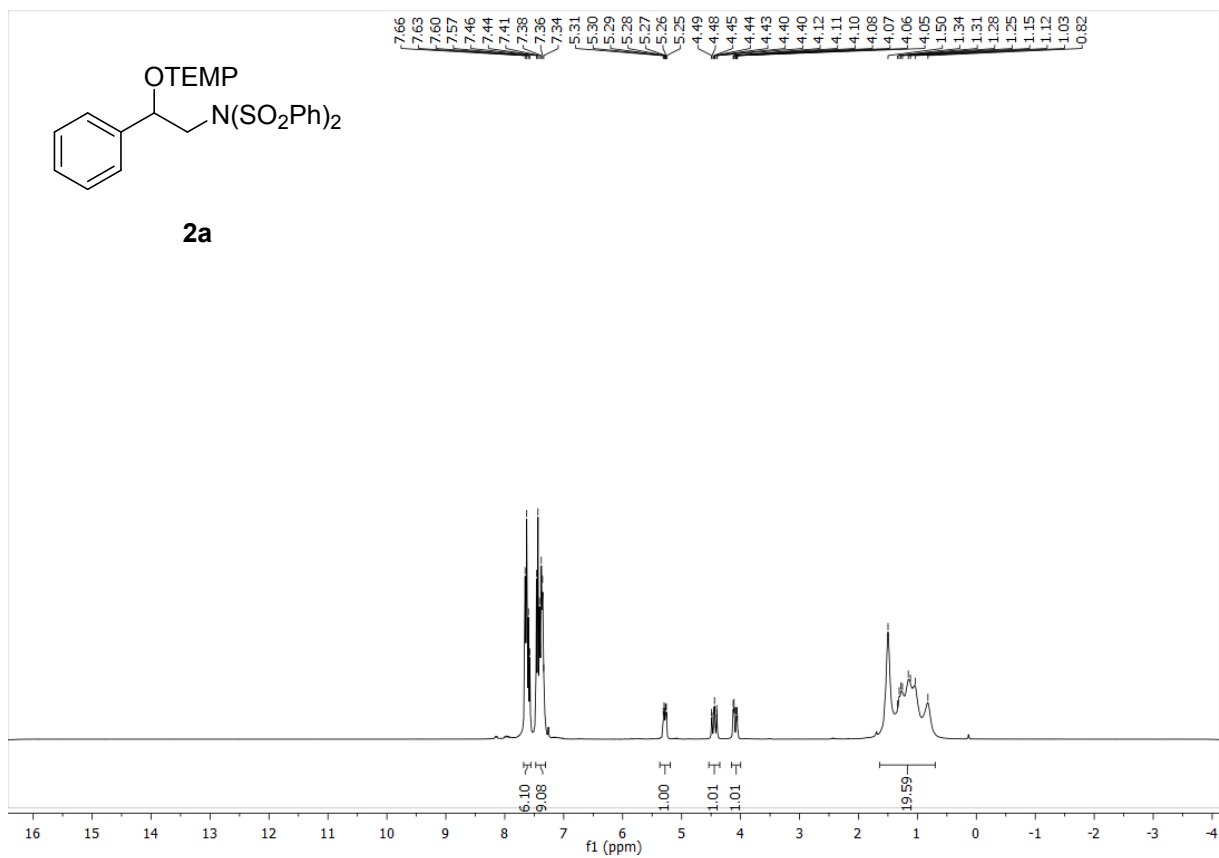
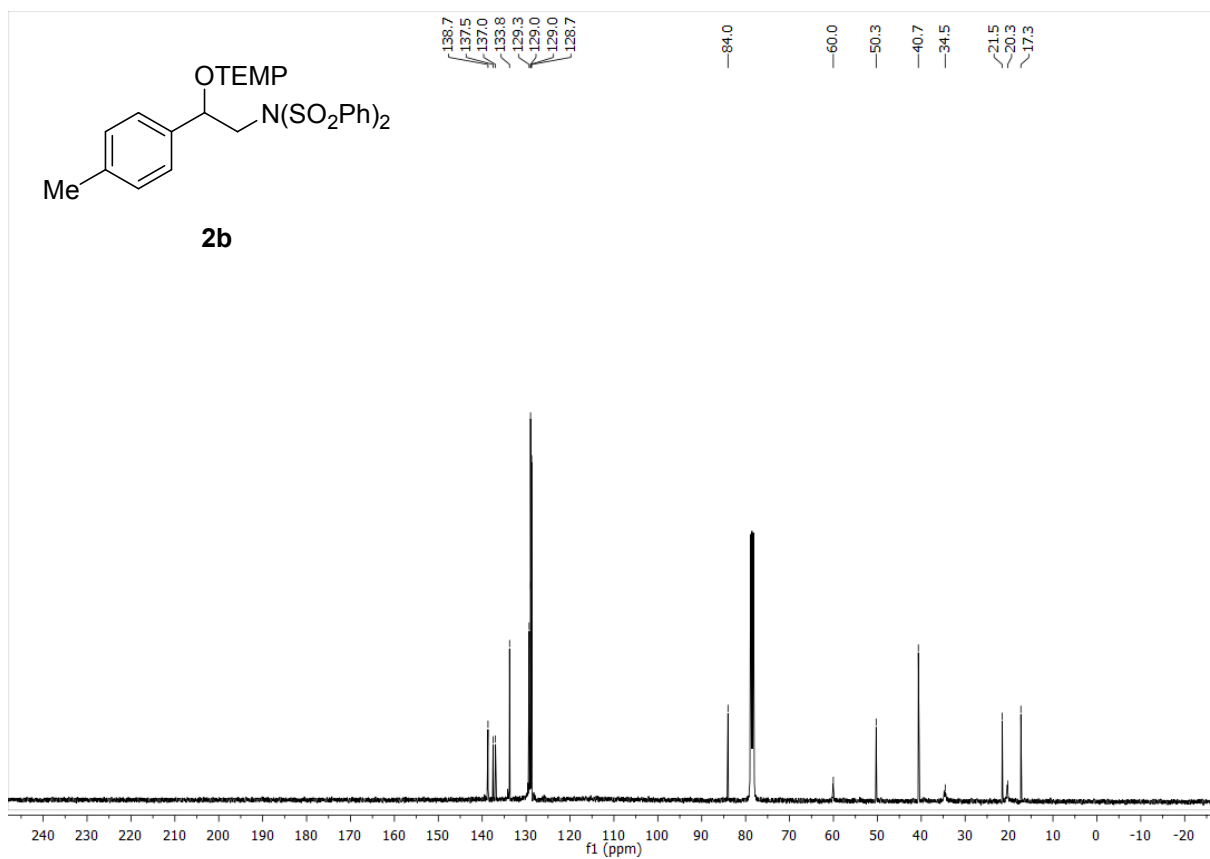
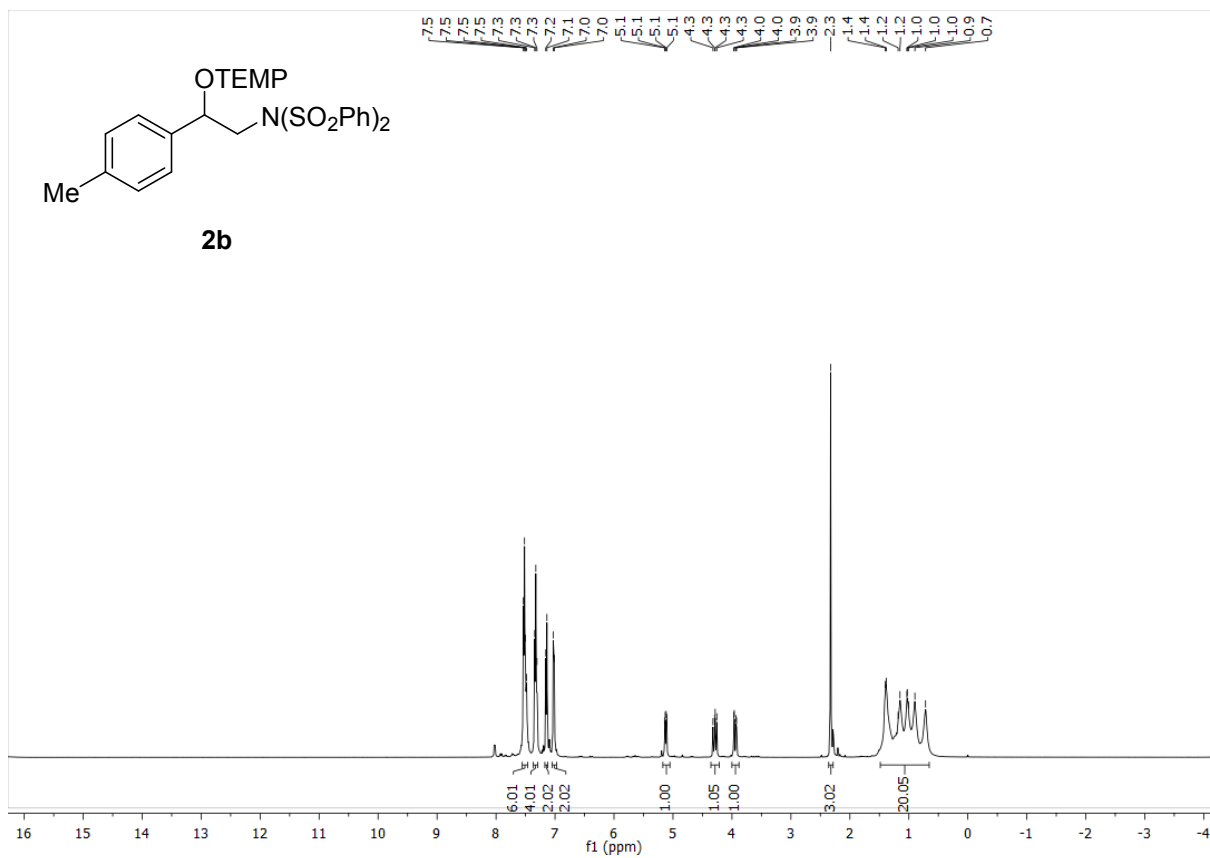
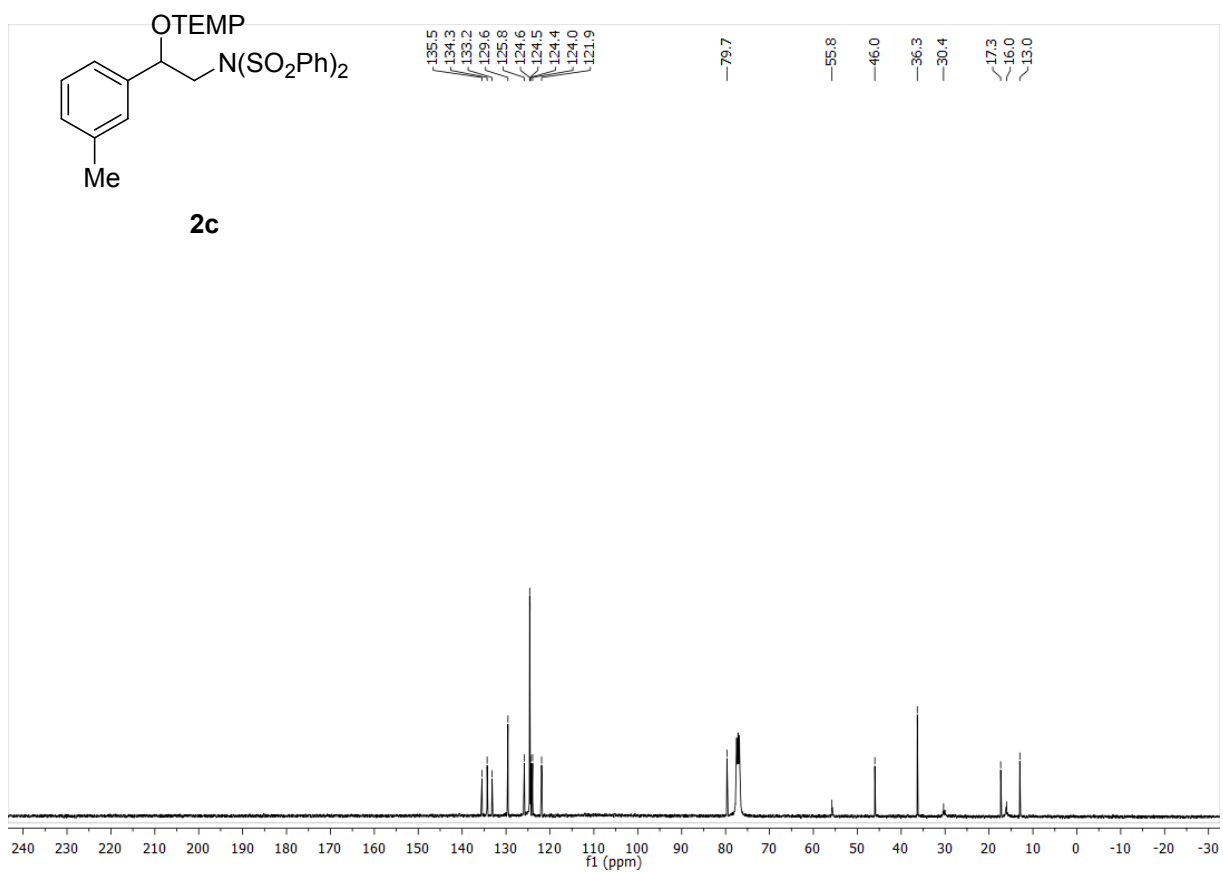
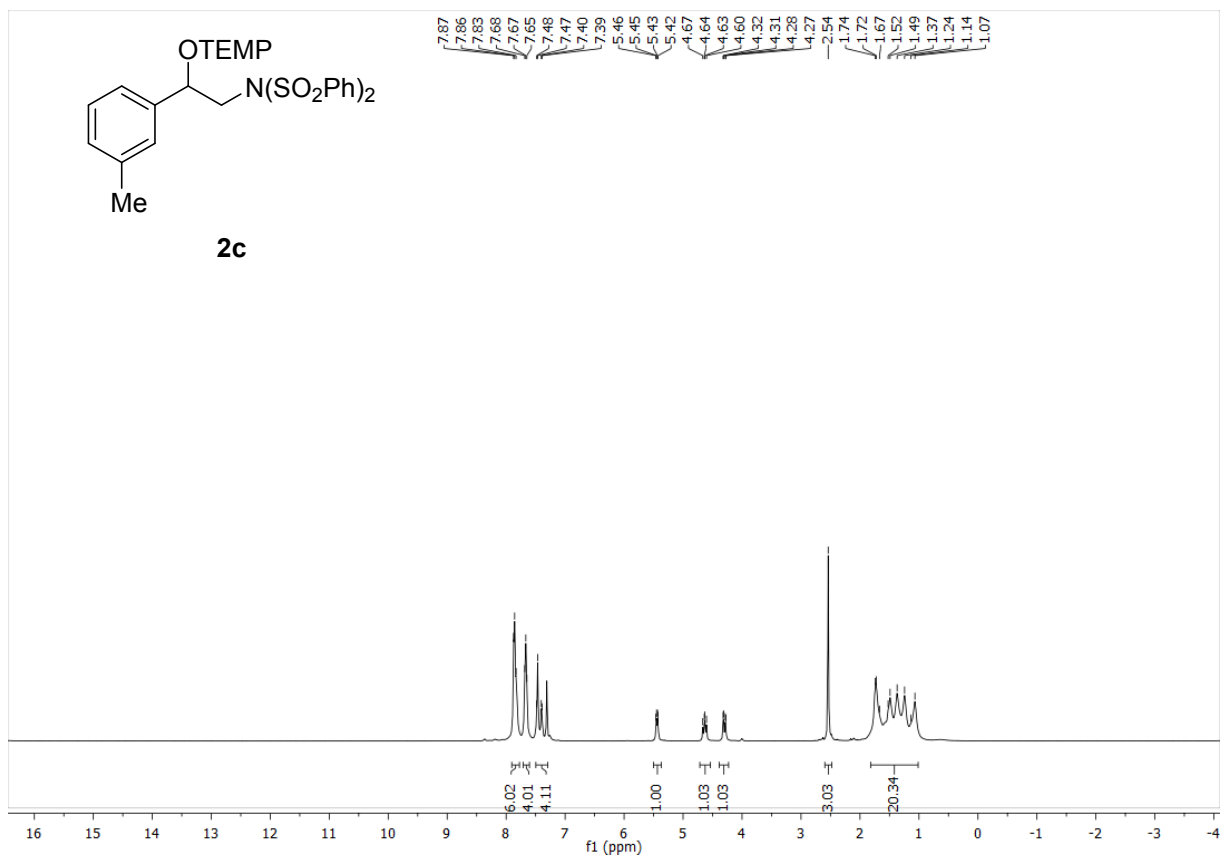
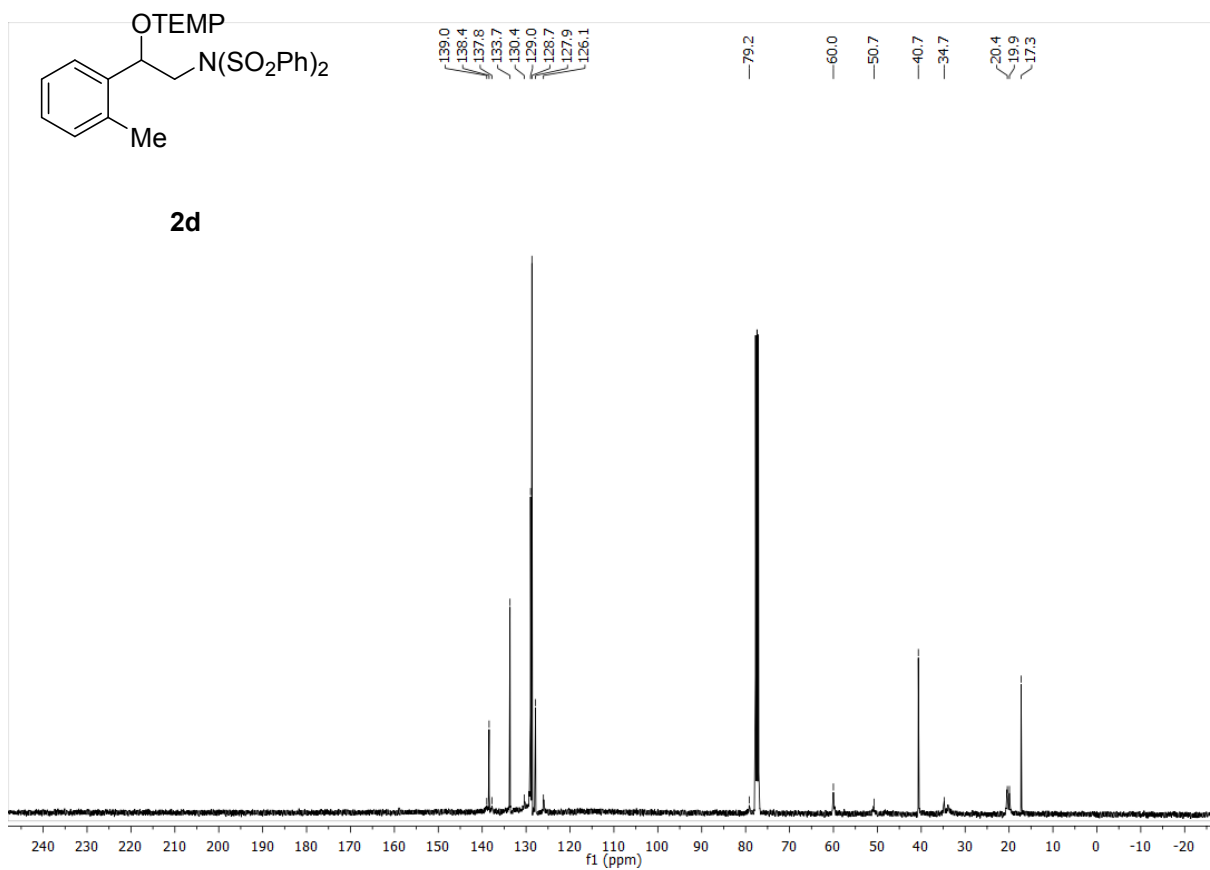
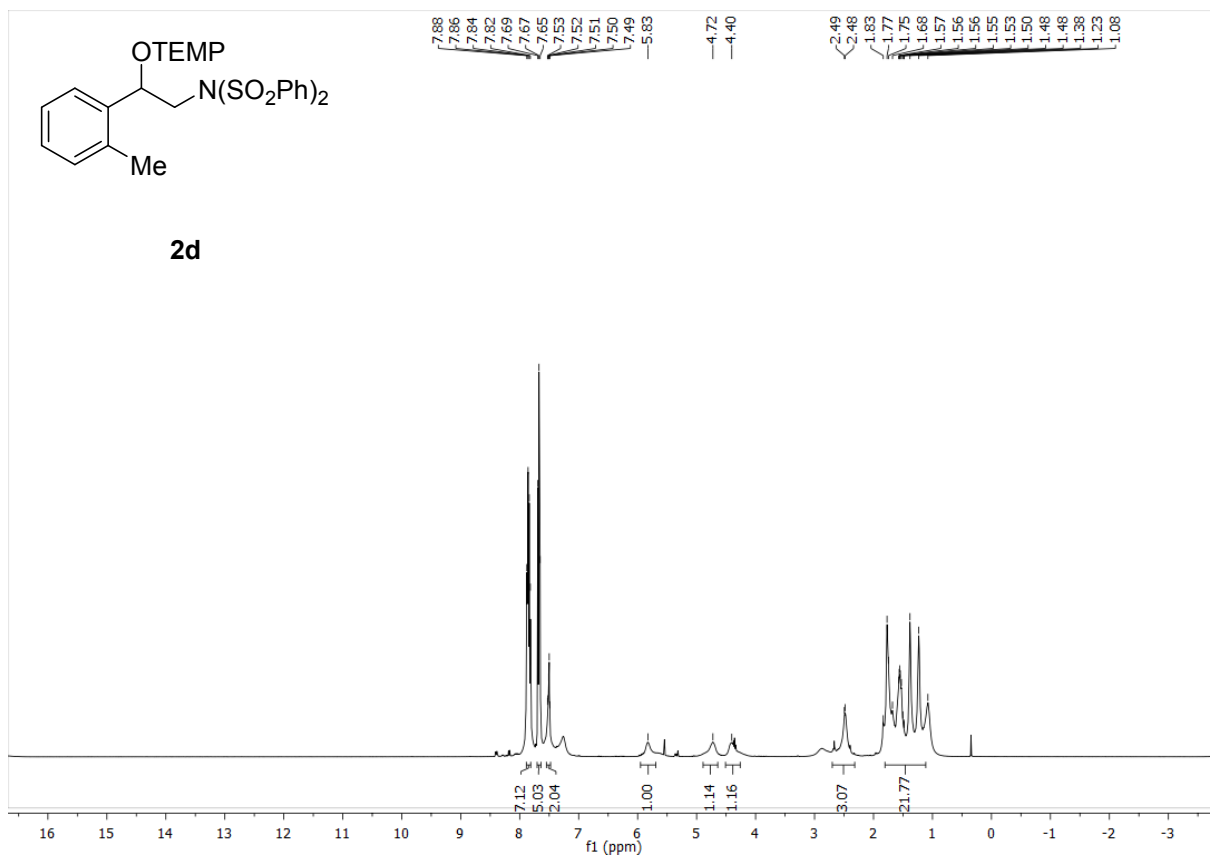


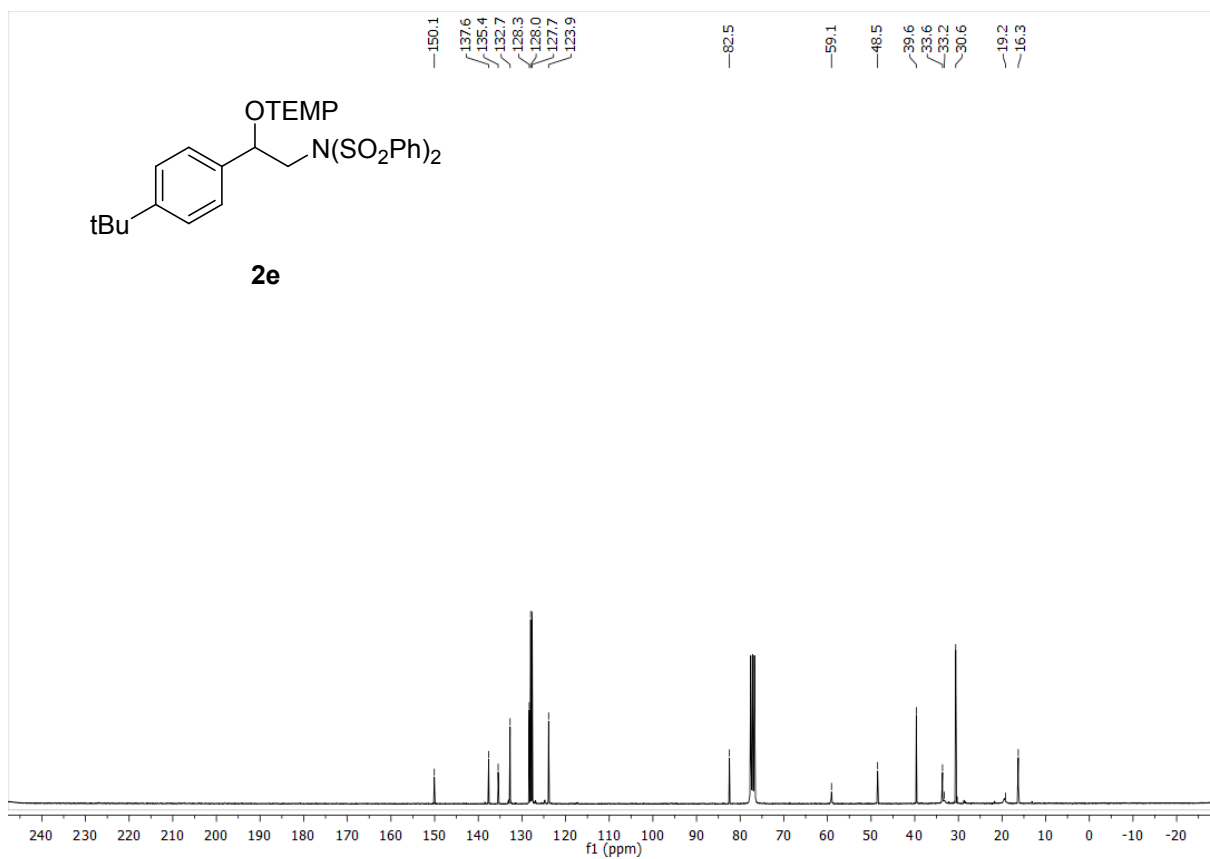
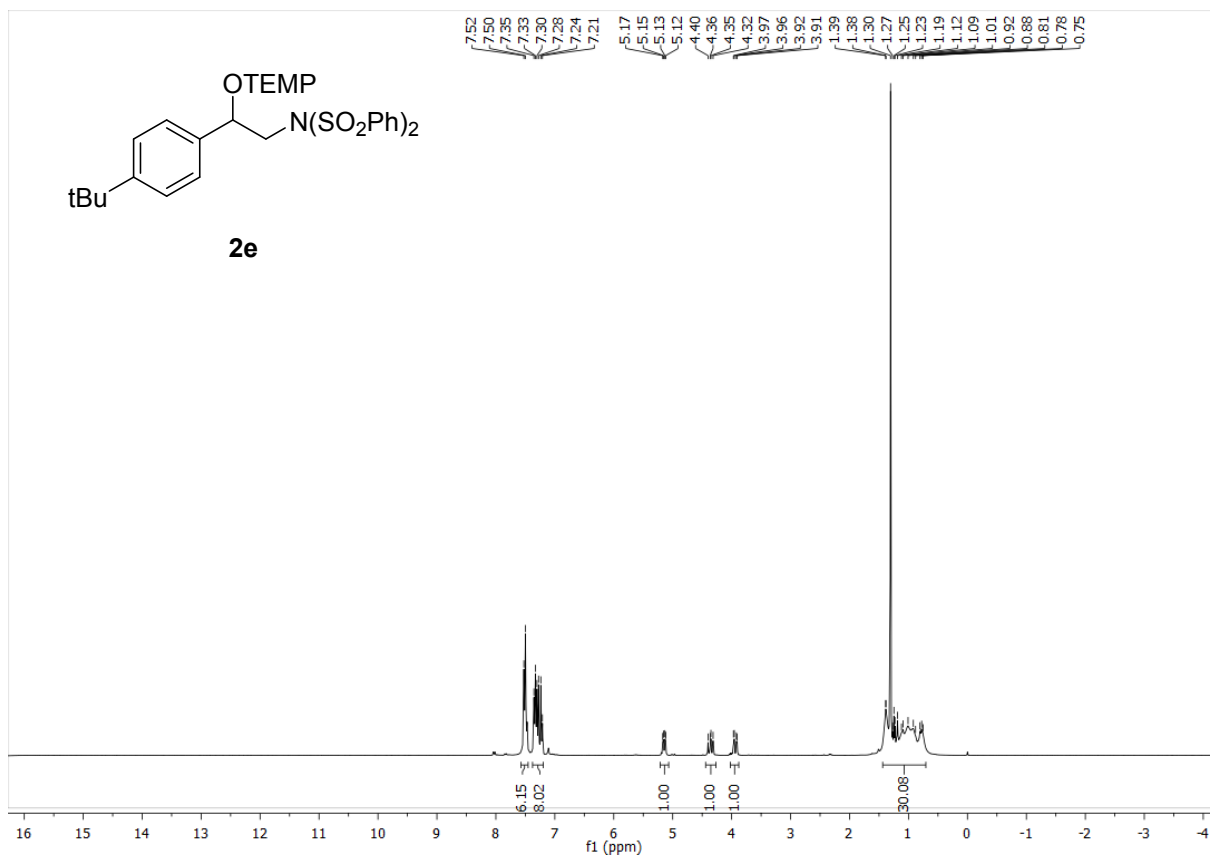
Figure 1. Crystal structure of compound **2q**.  
(Thermal ellipsoids are shown with 50% probability.)

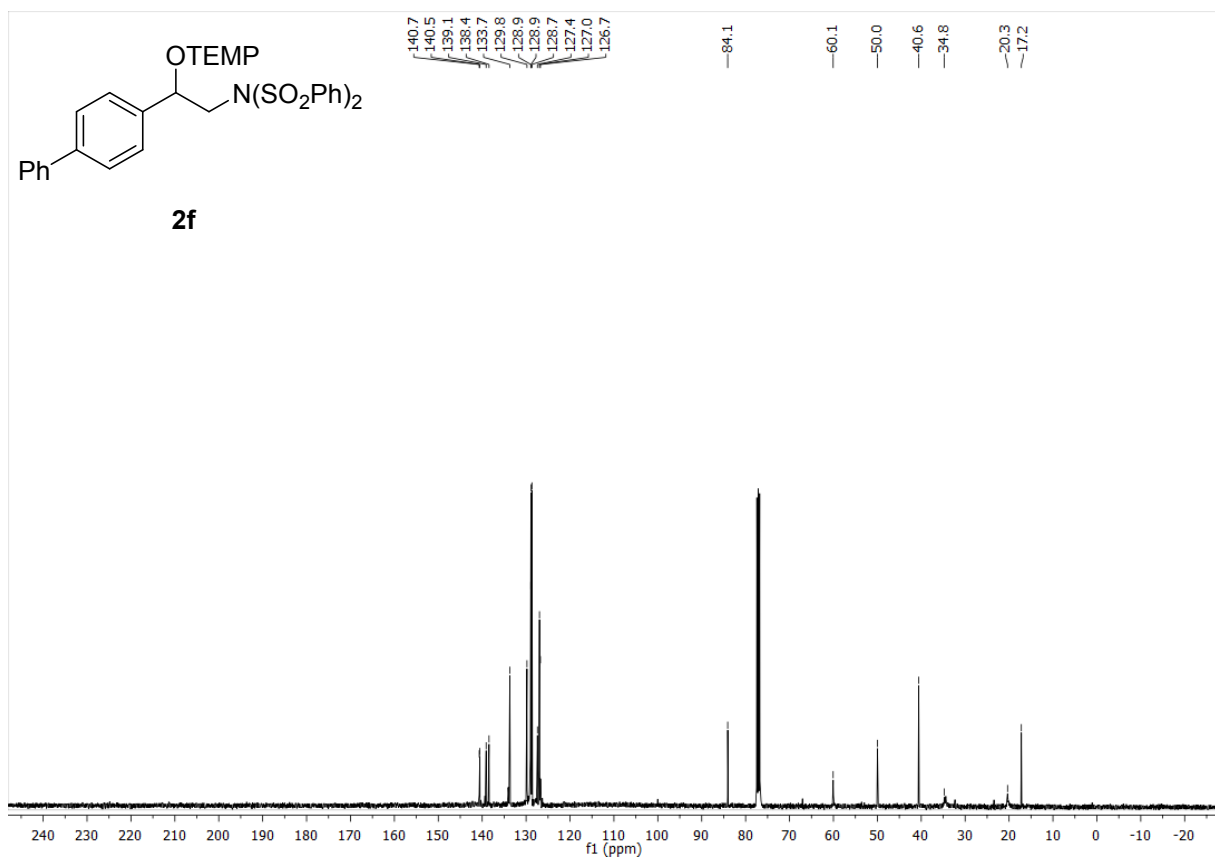
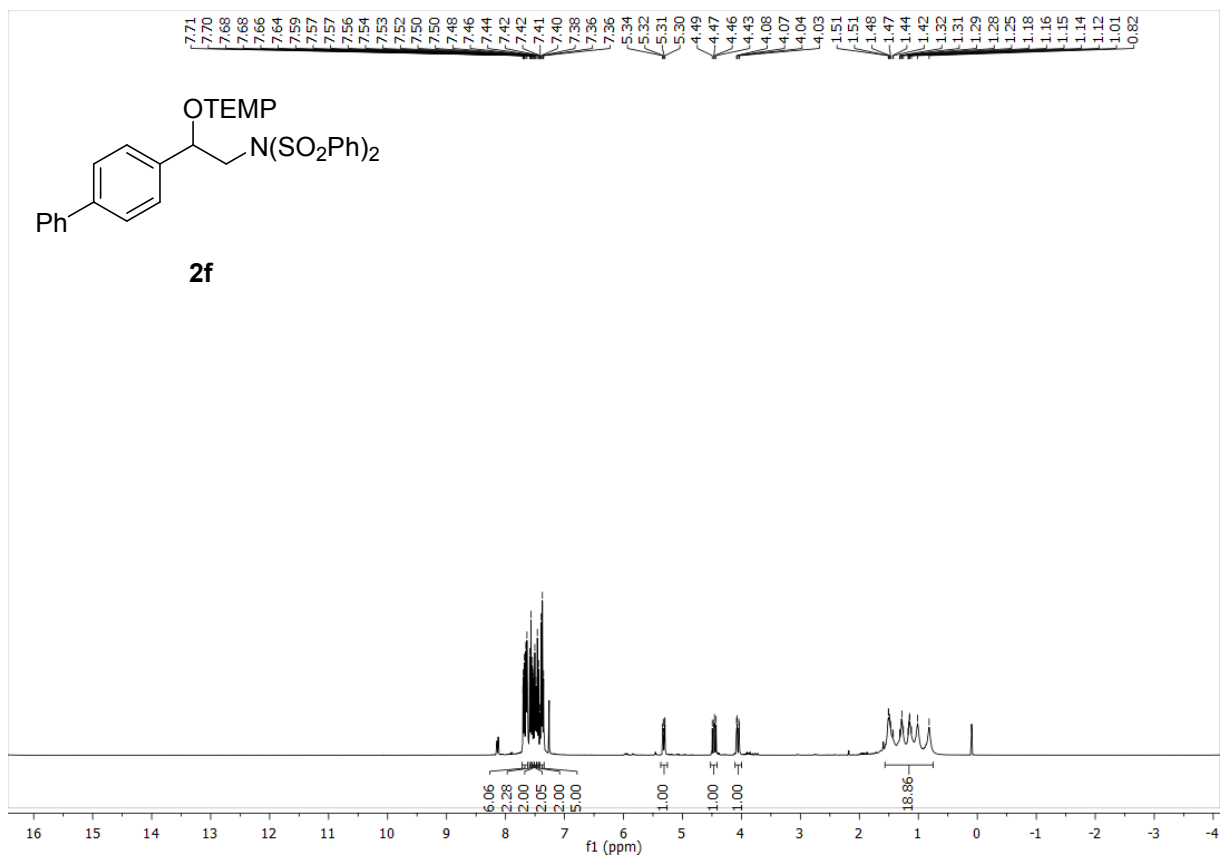




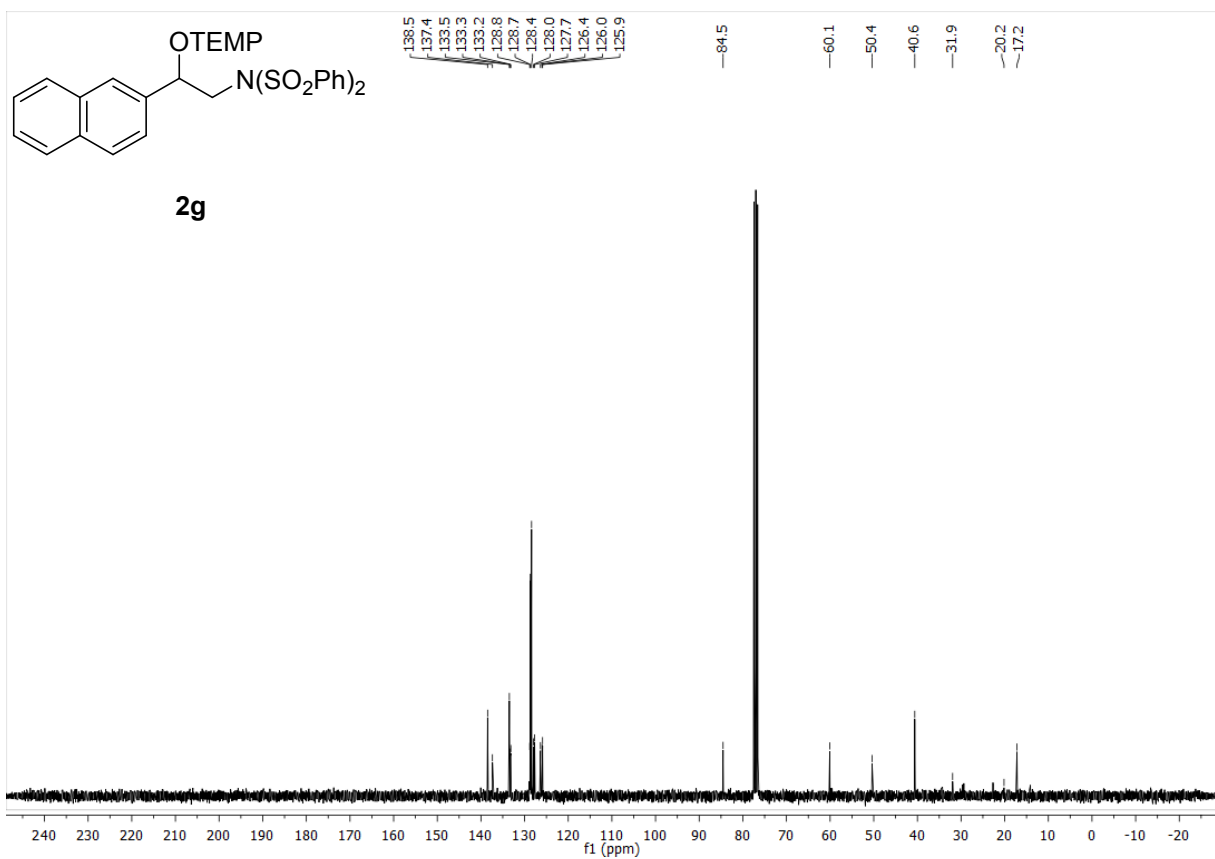
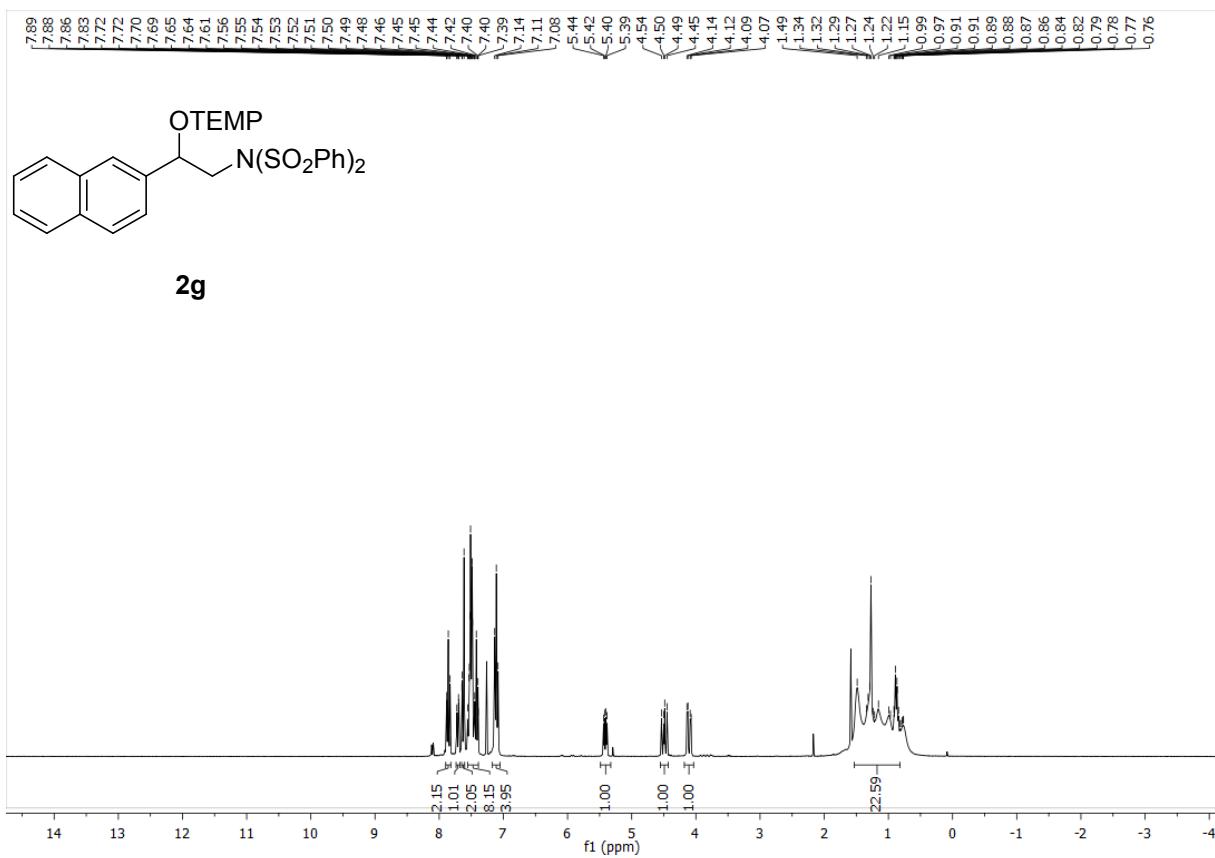


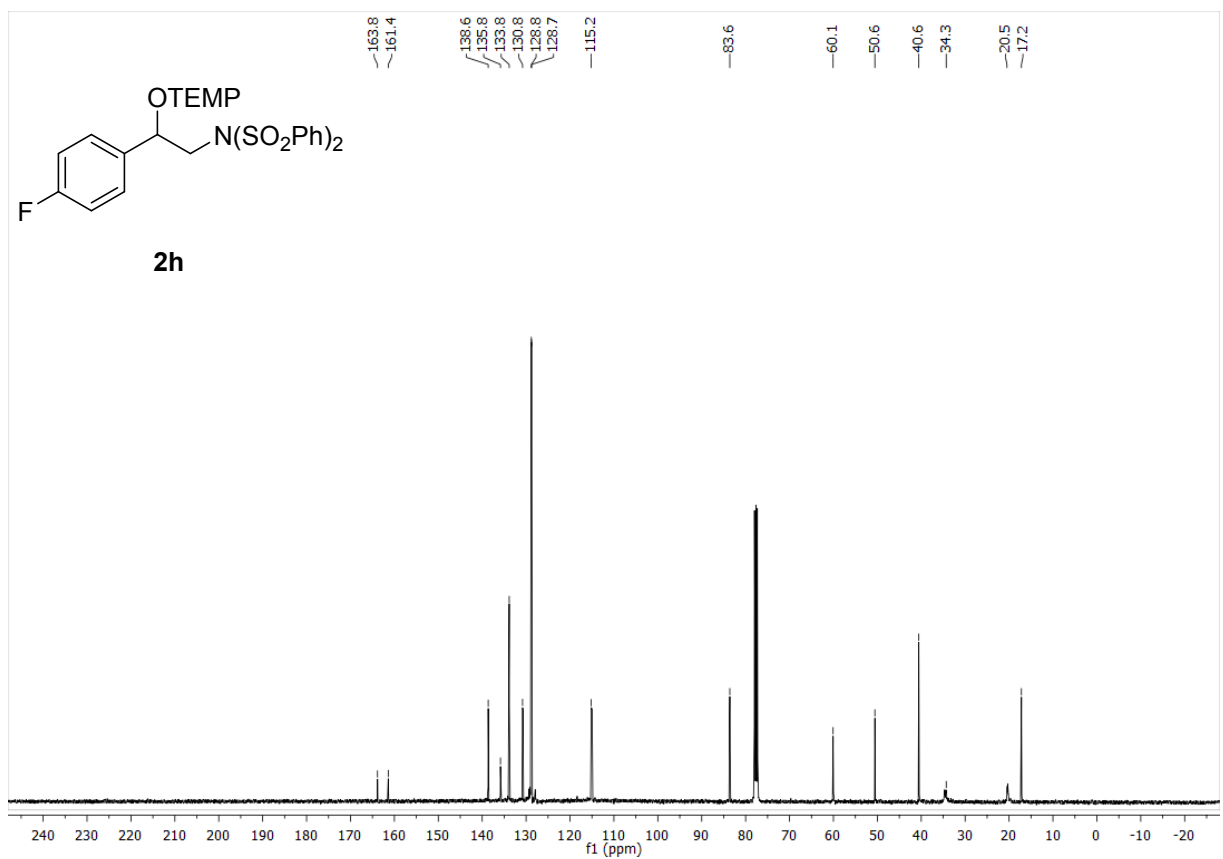
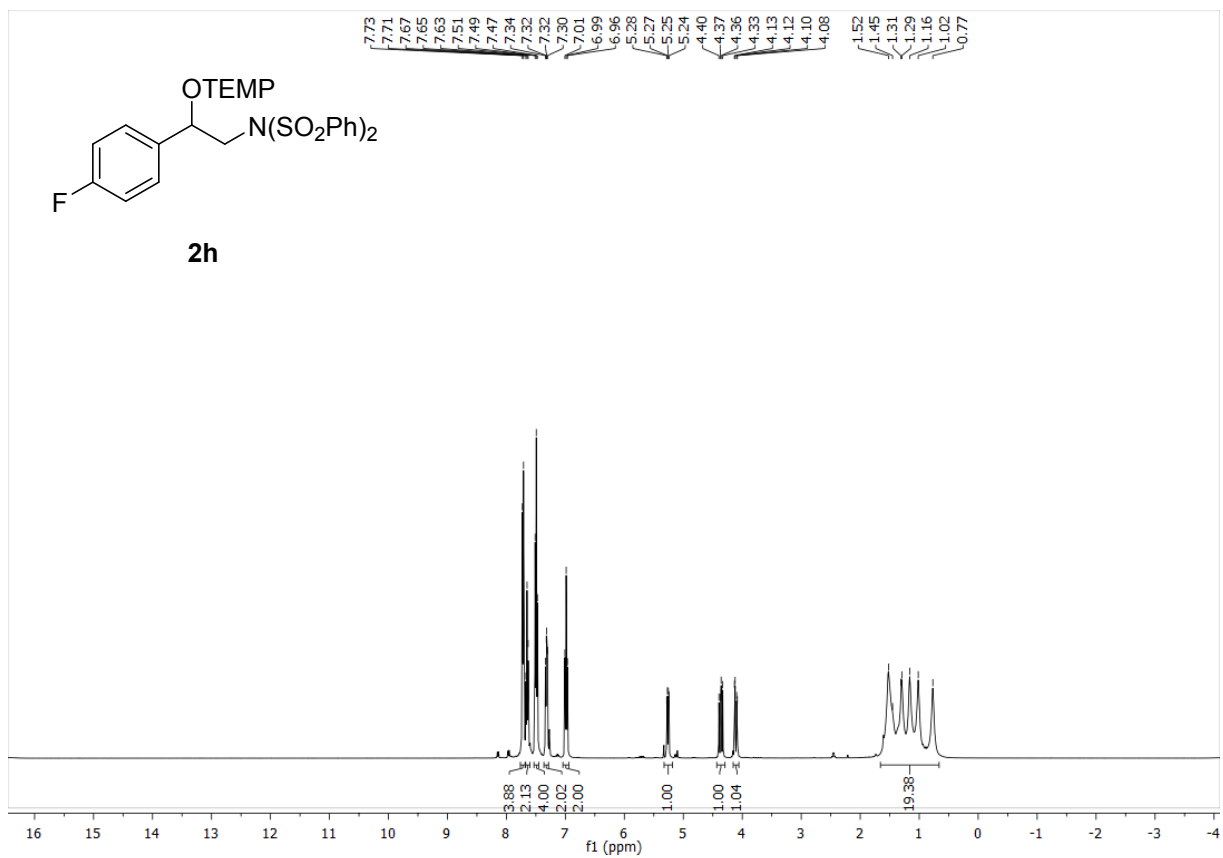


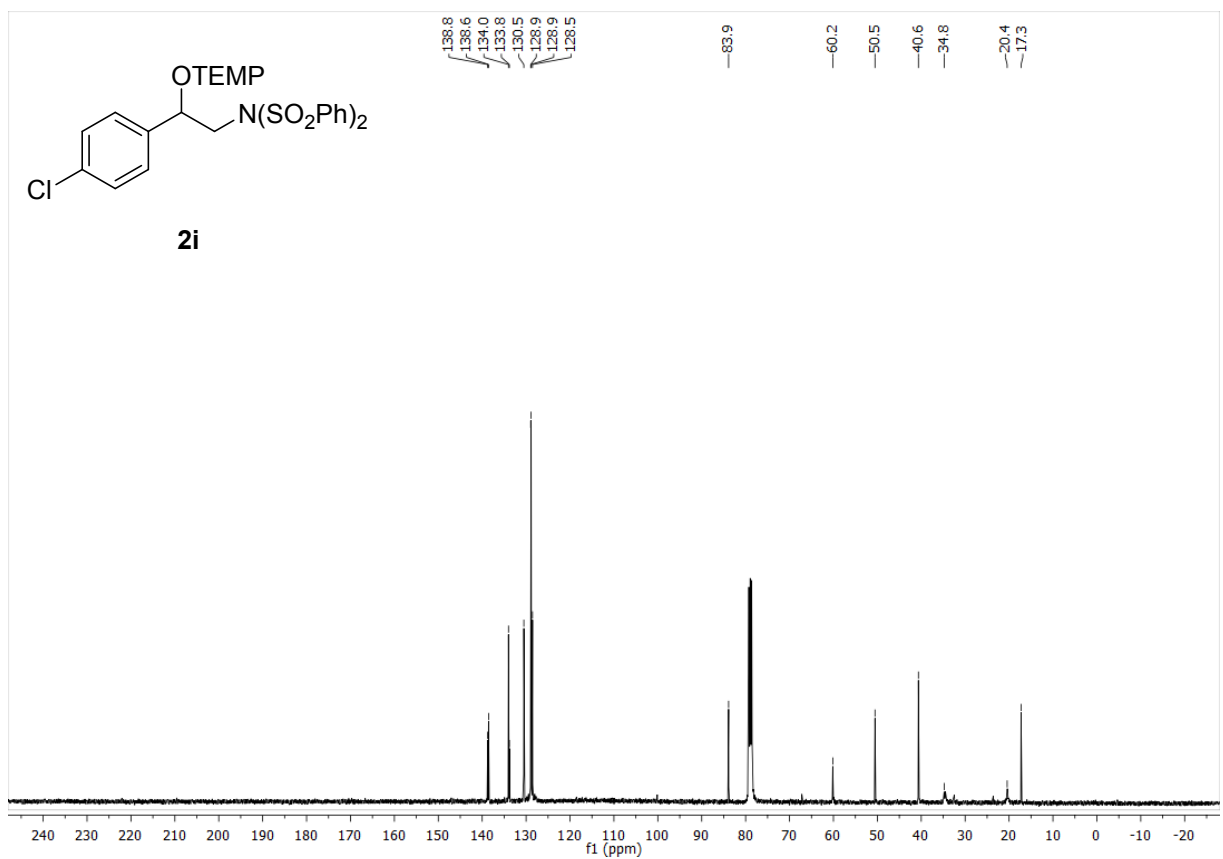
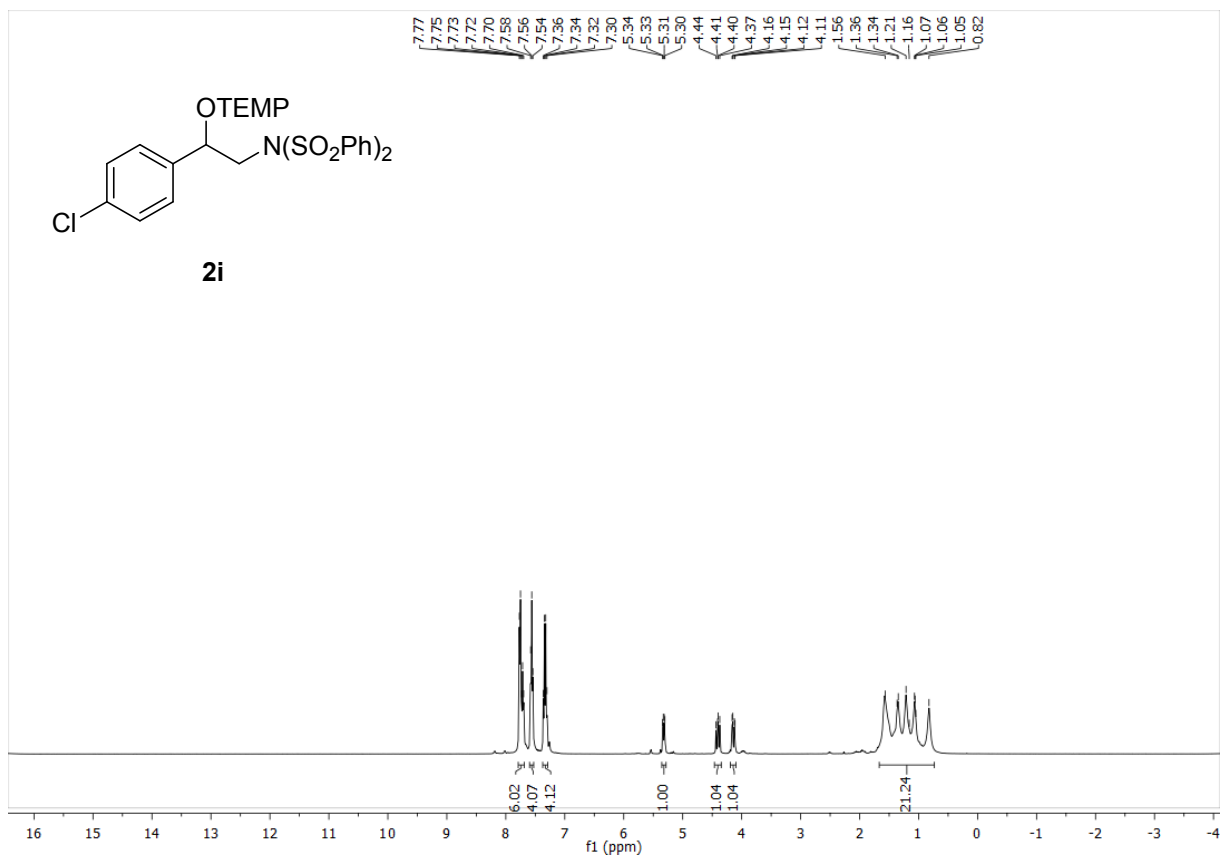


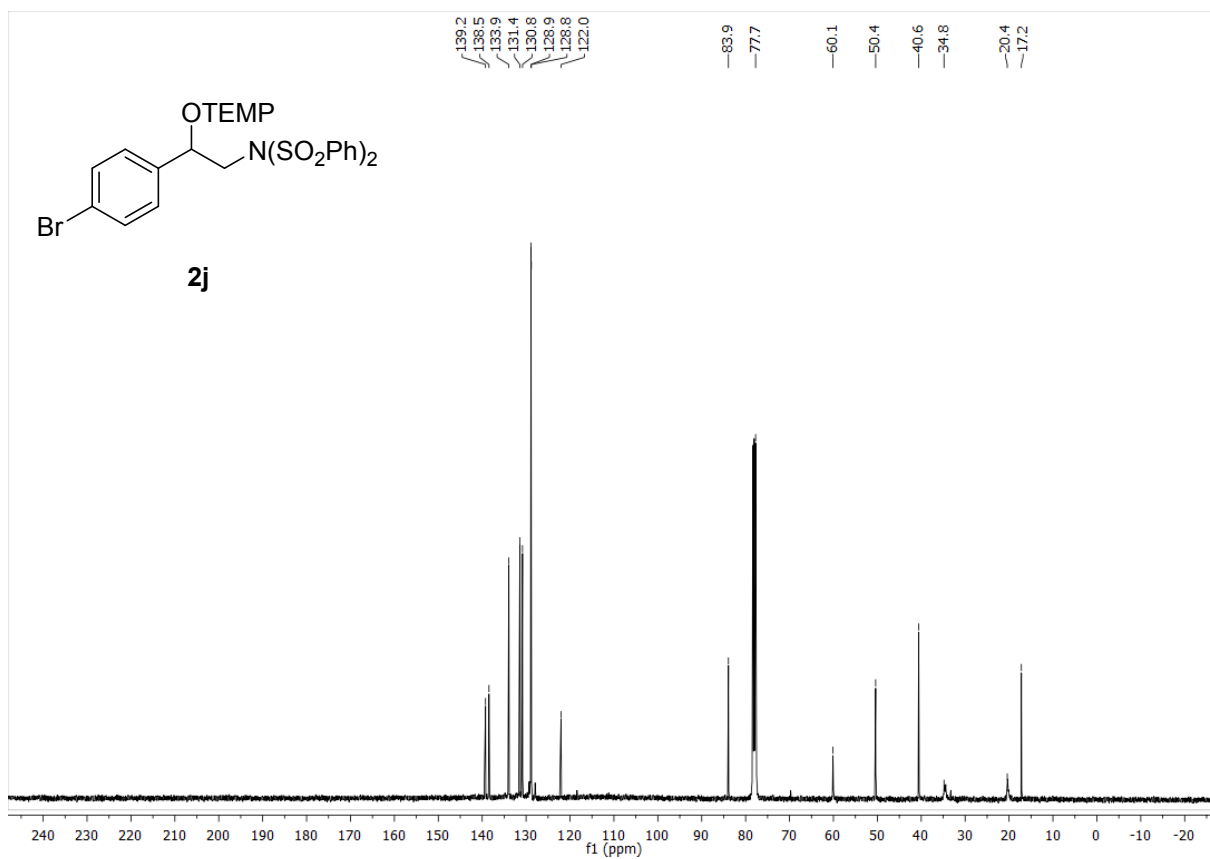
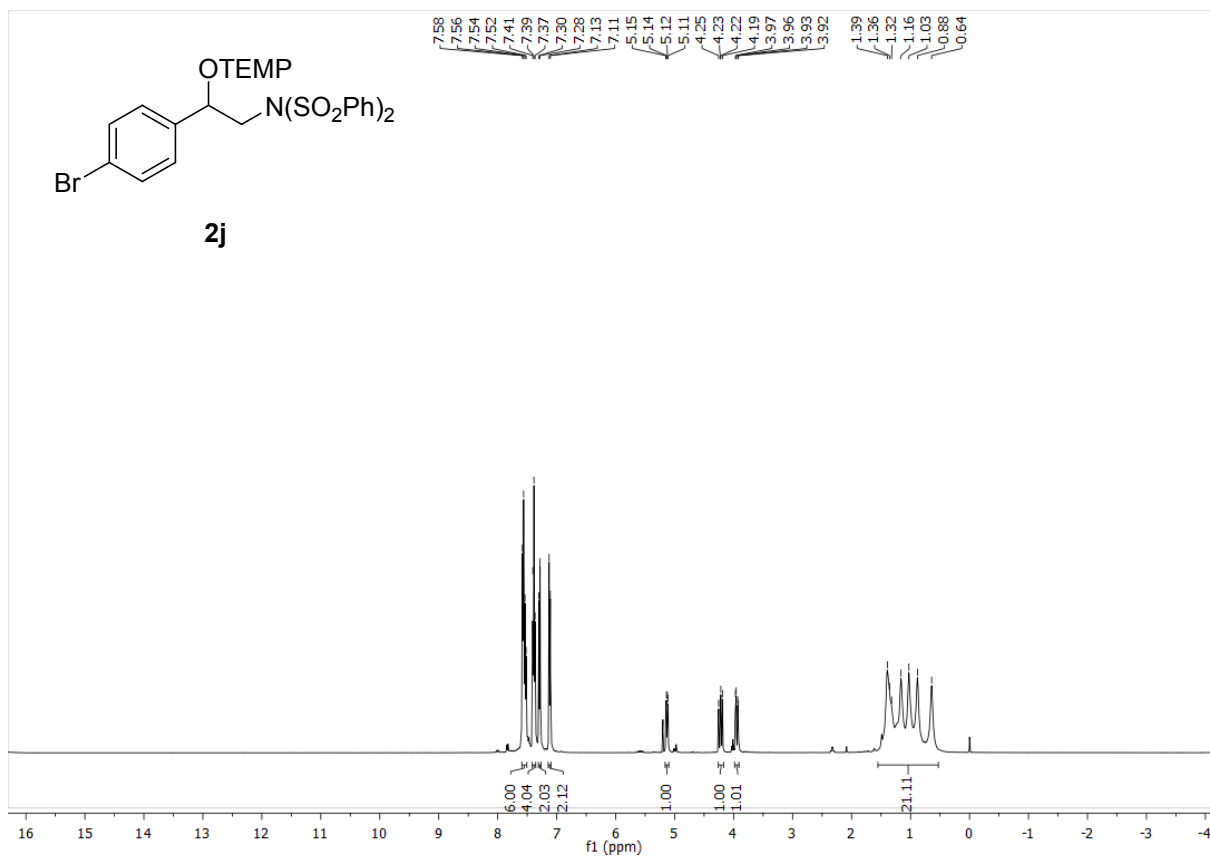


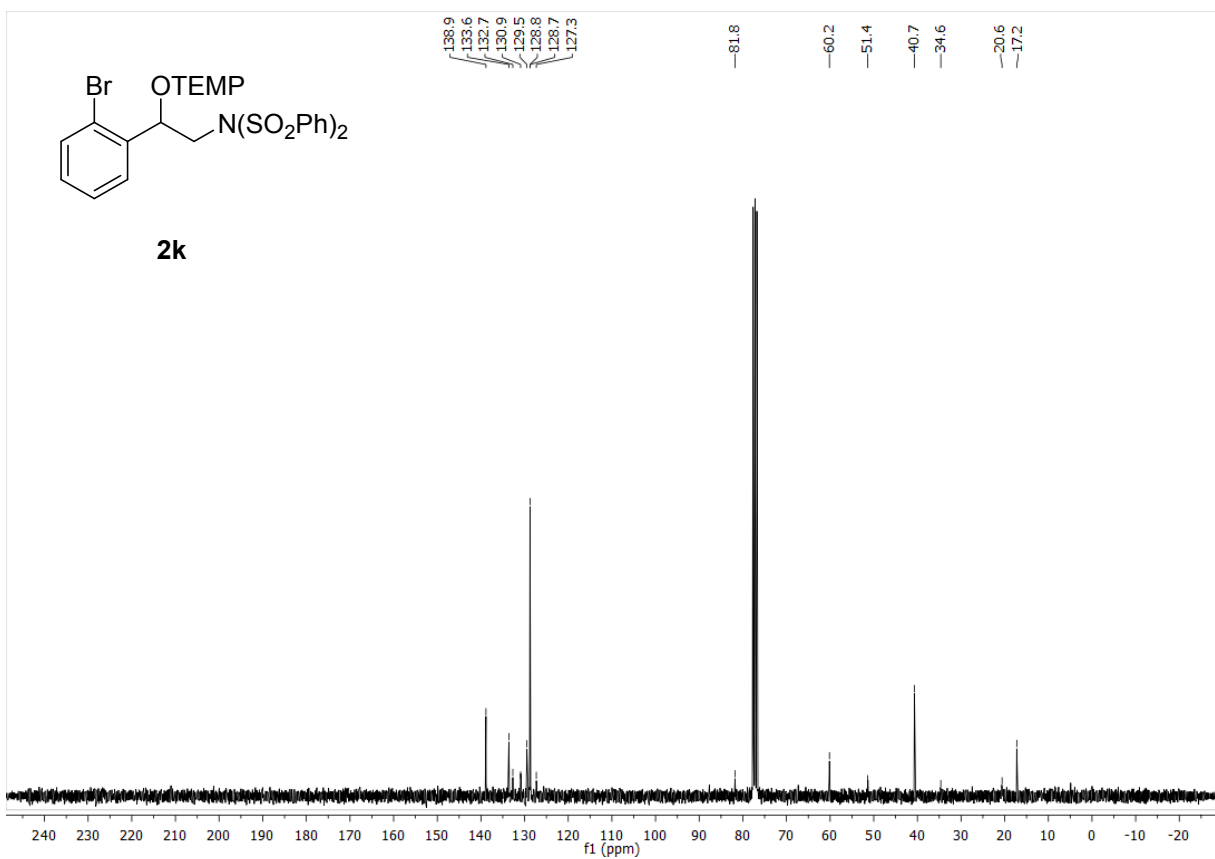
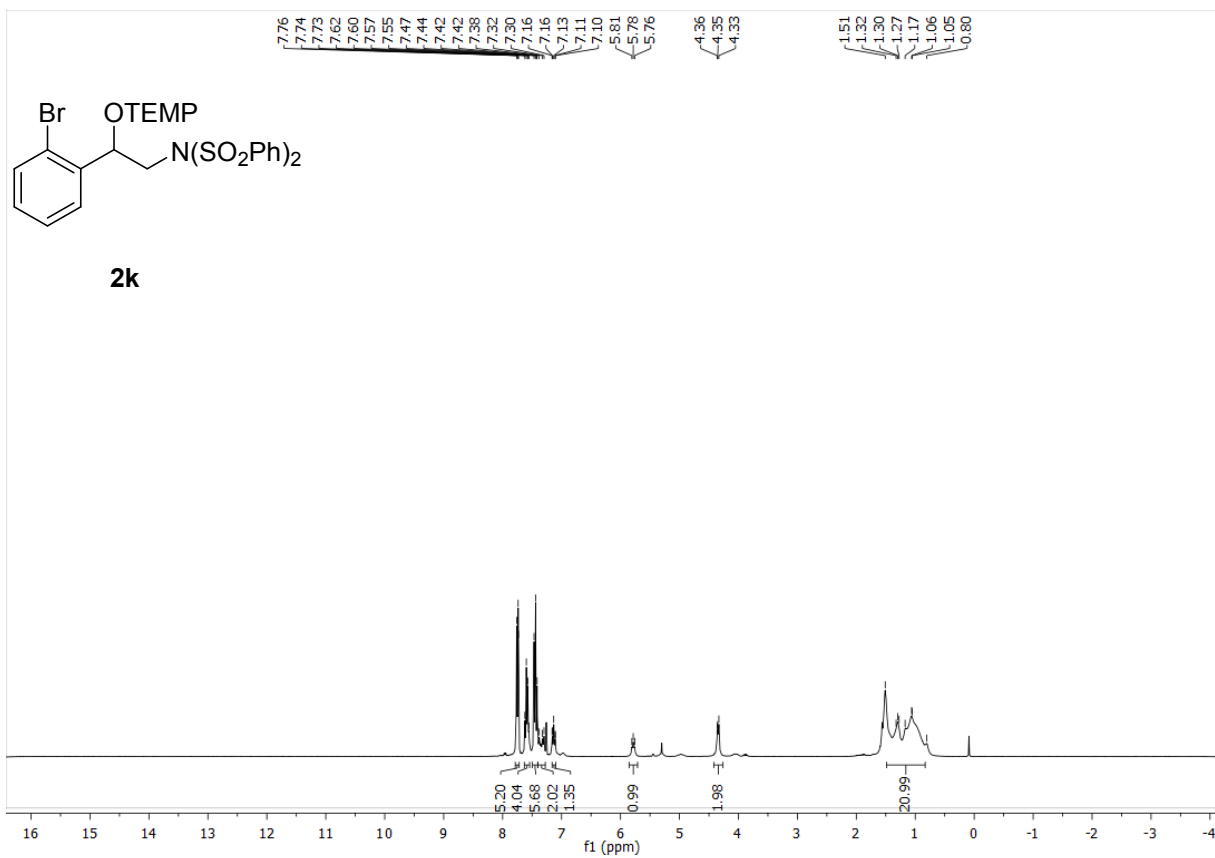


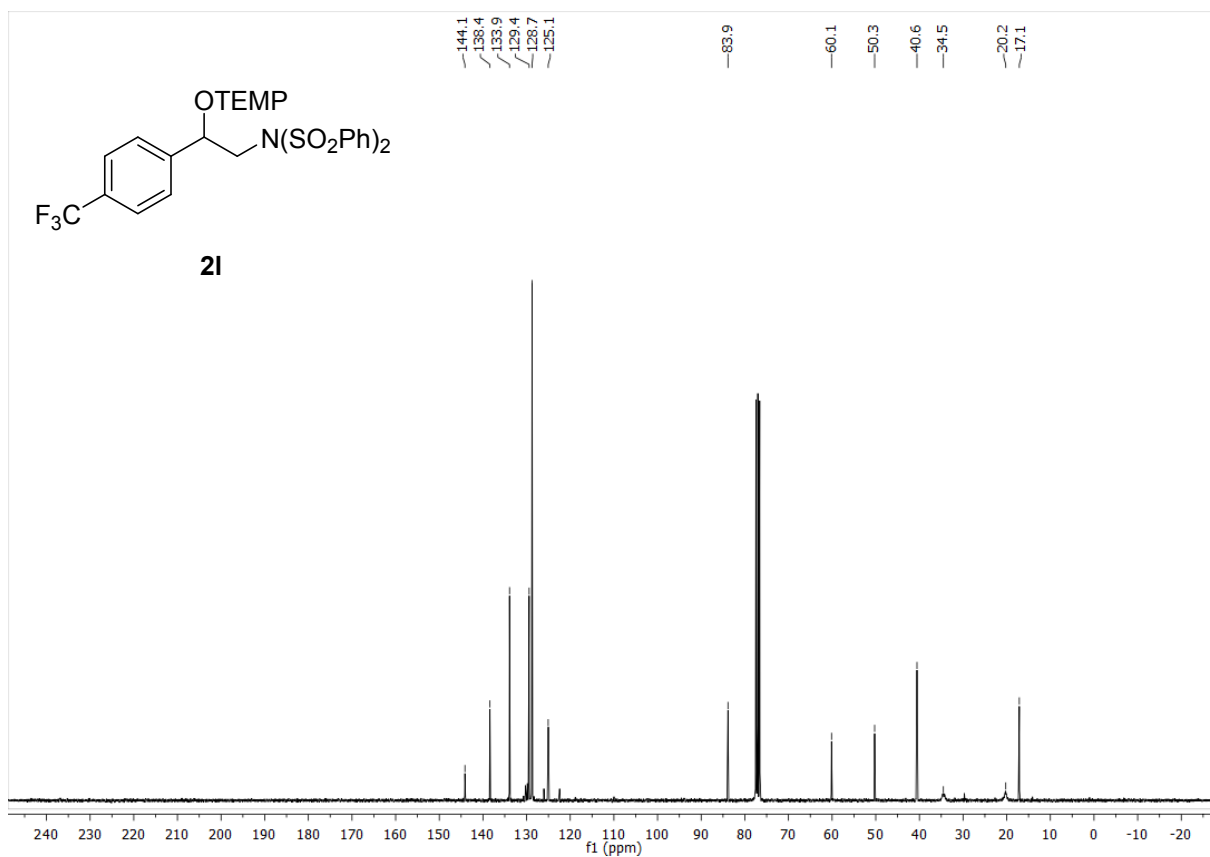
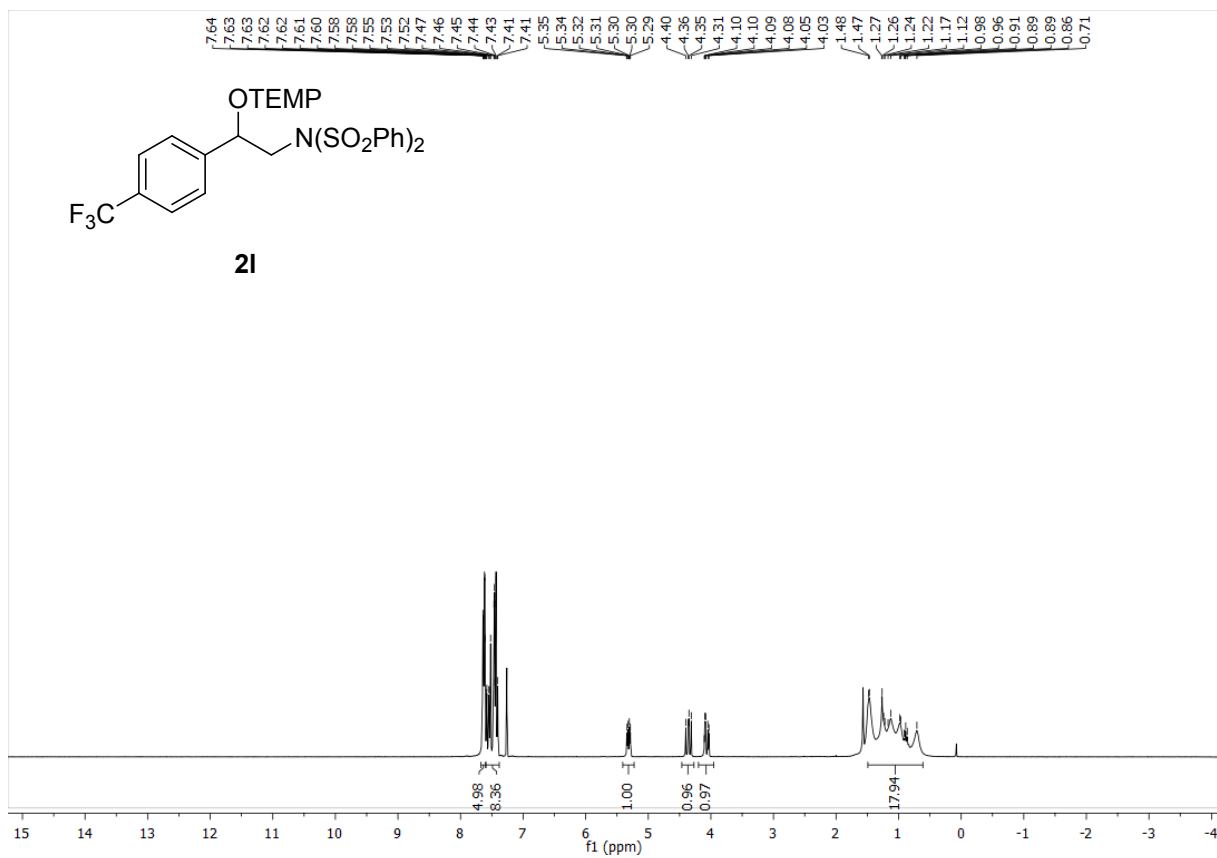


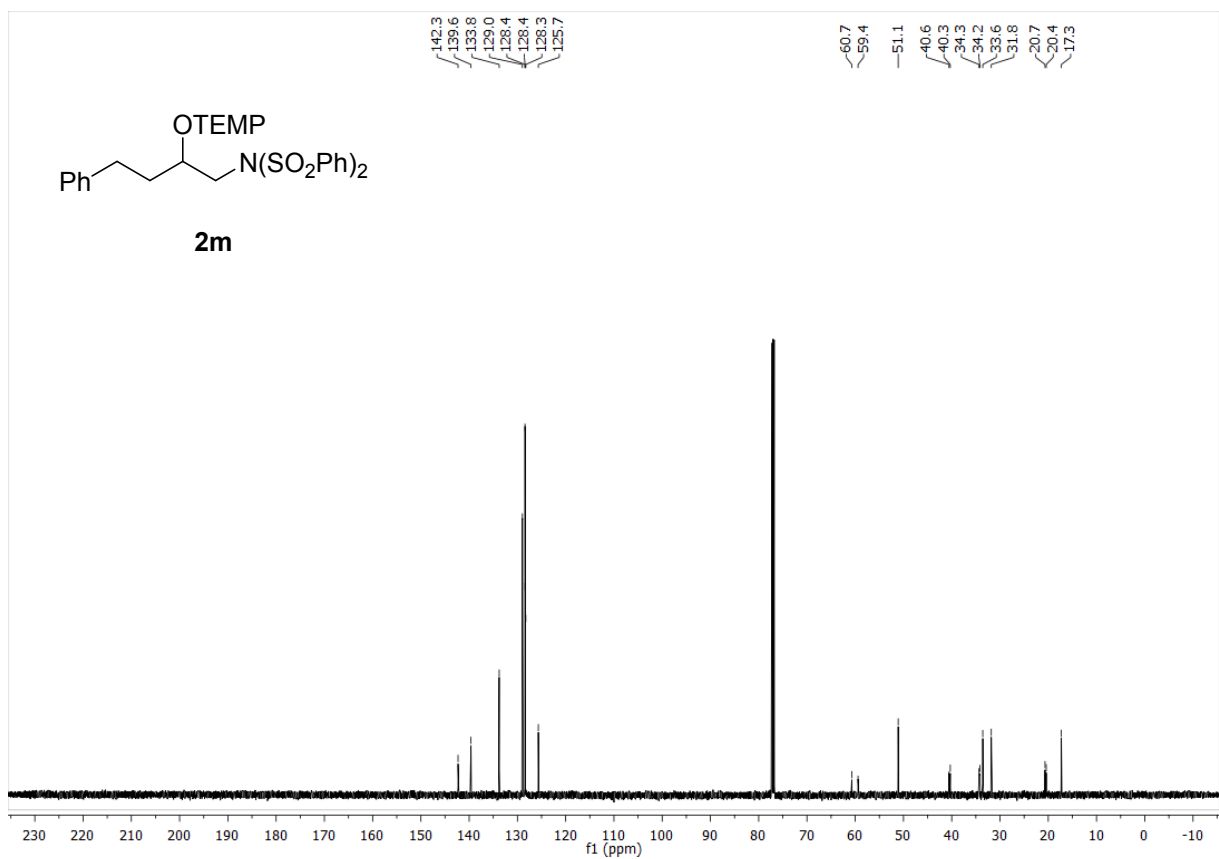
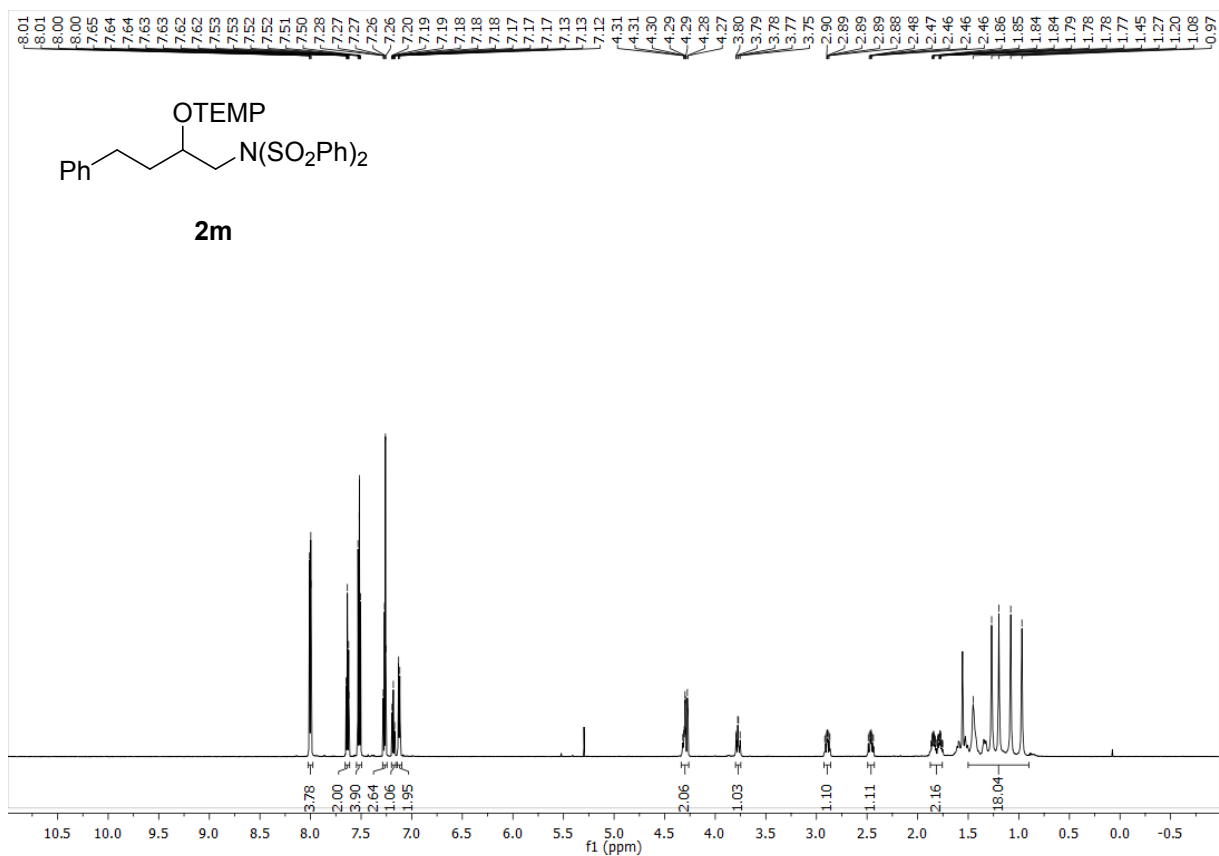


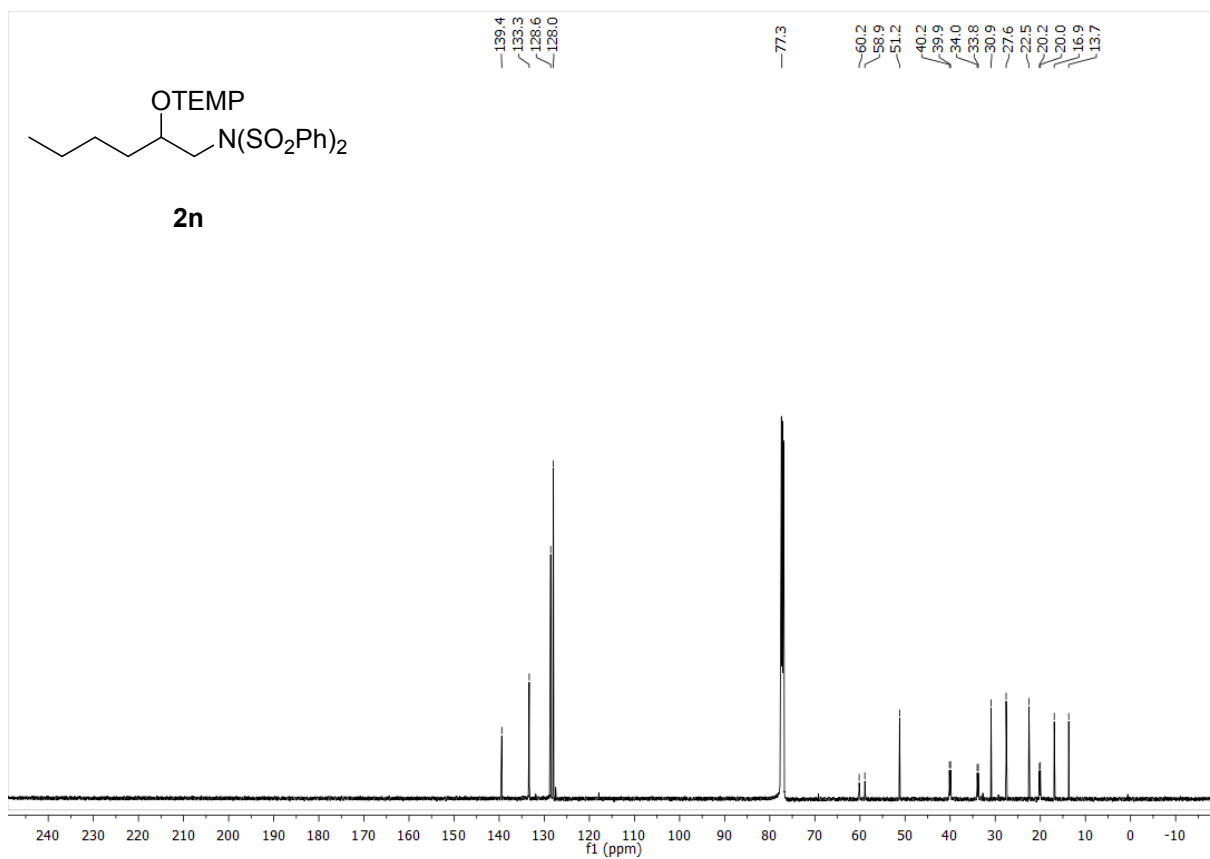
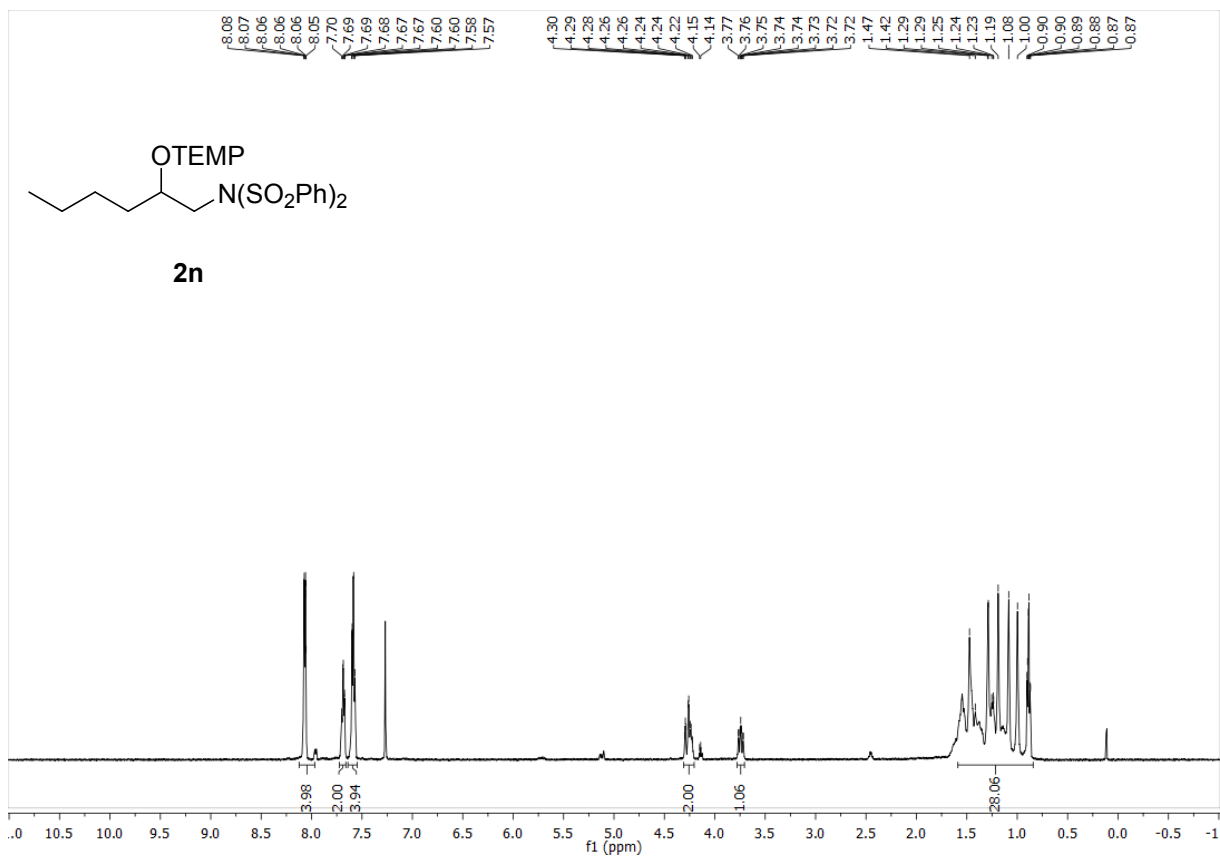




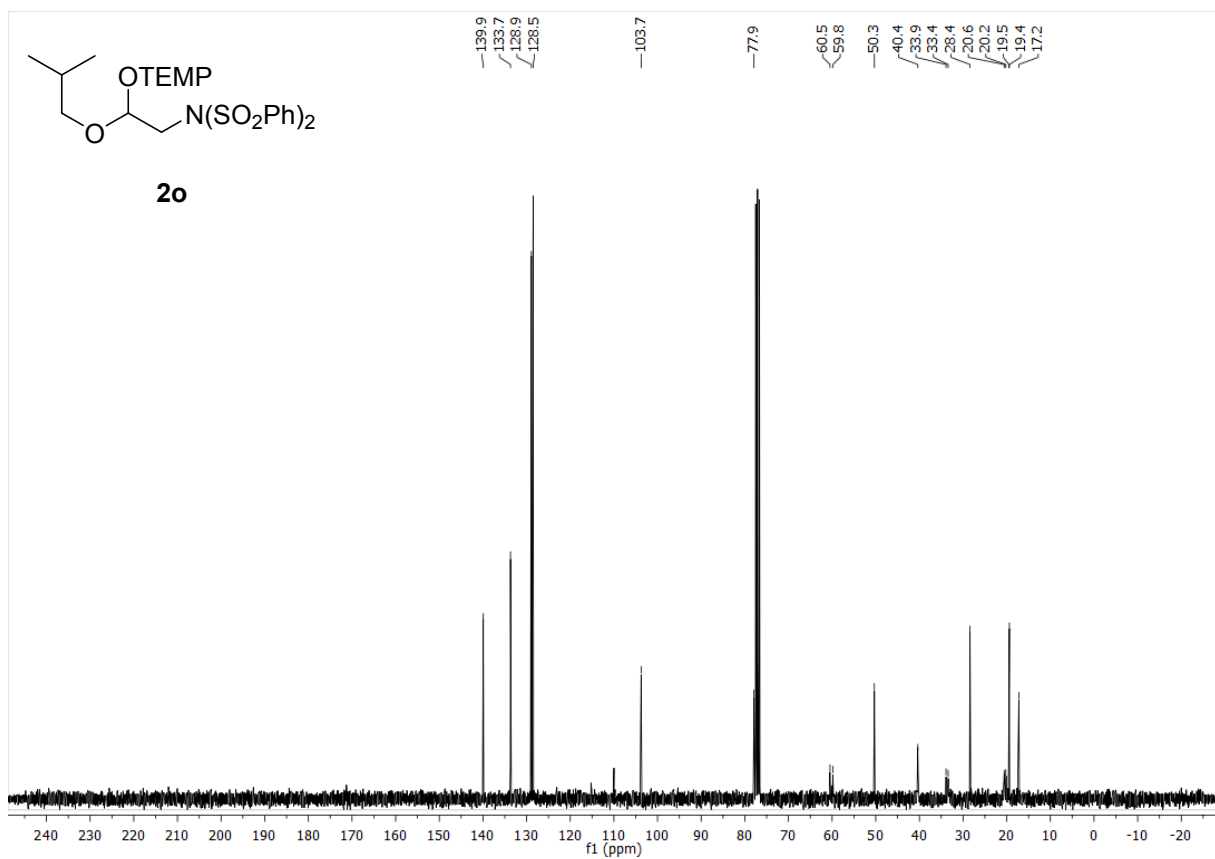
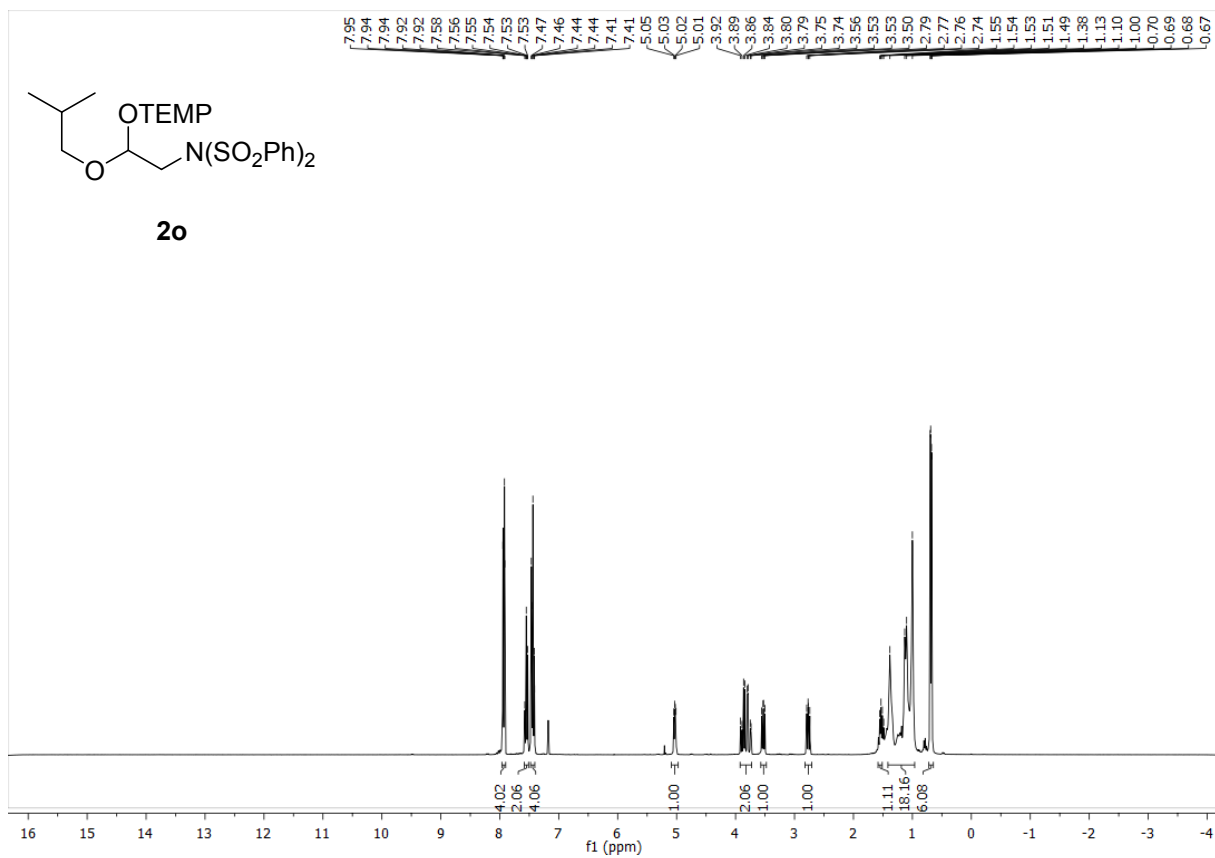


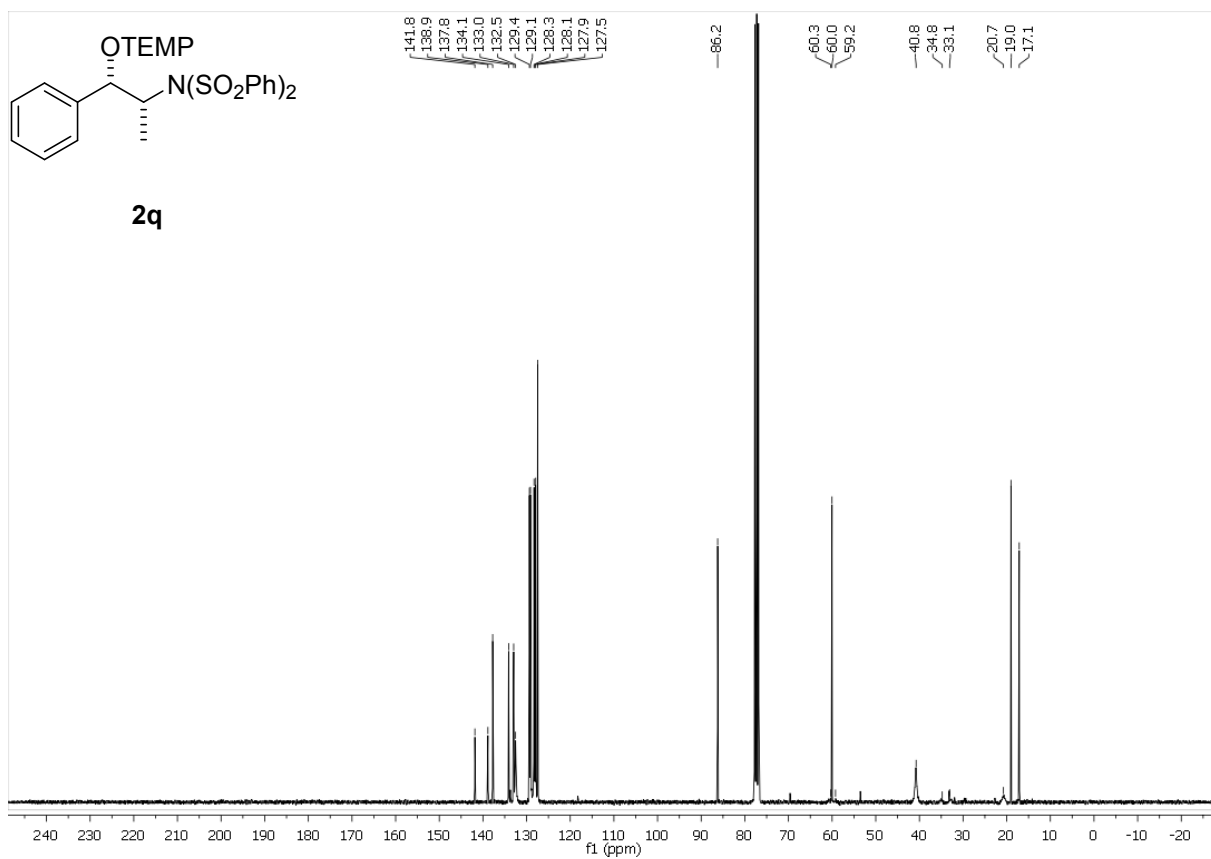
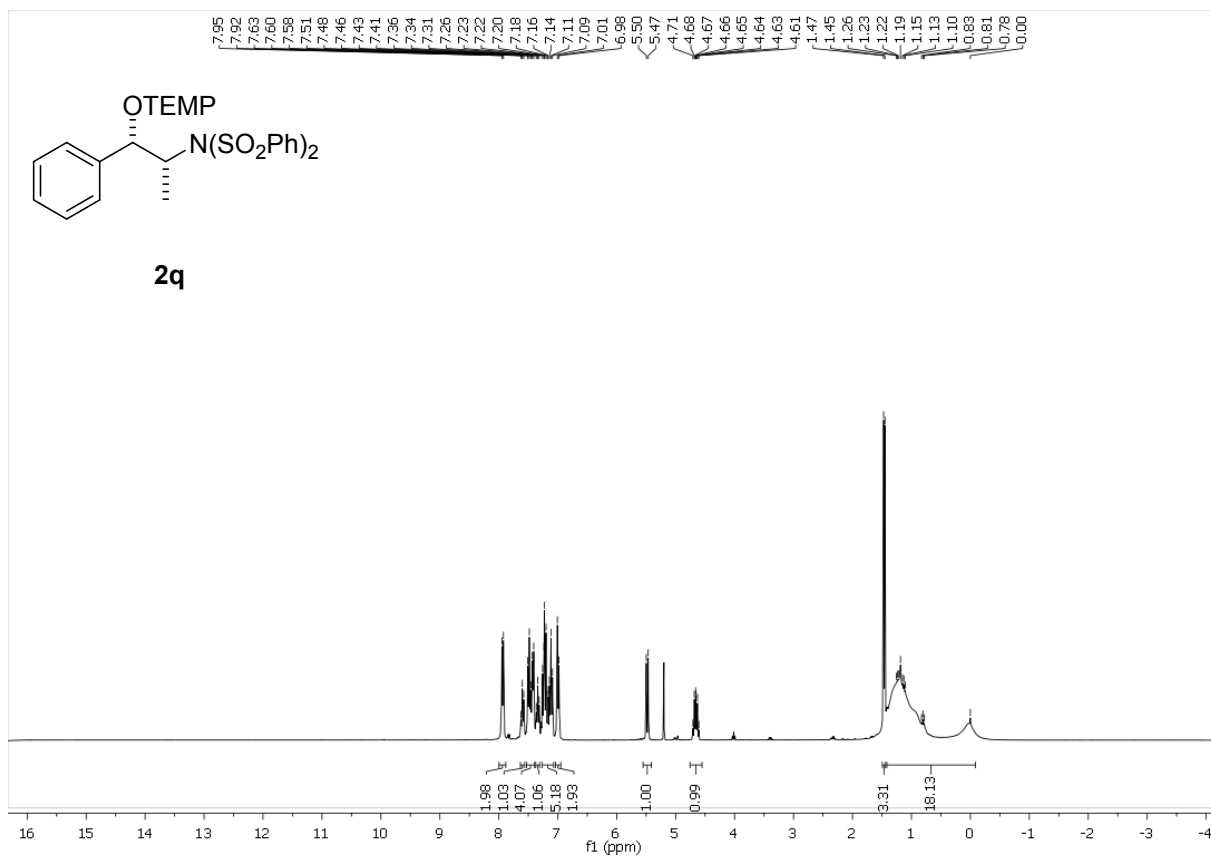


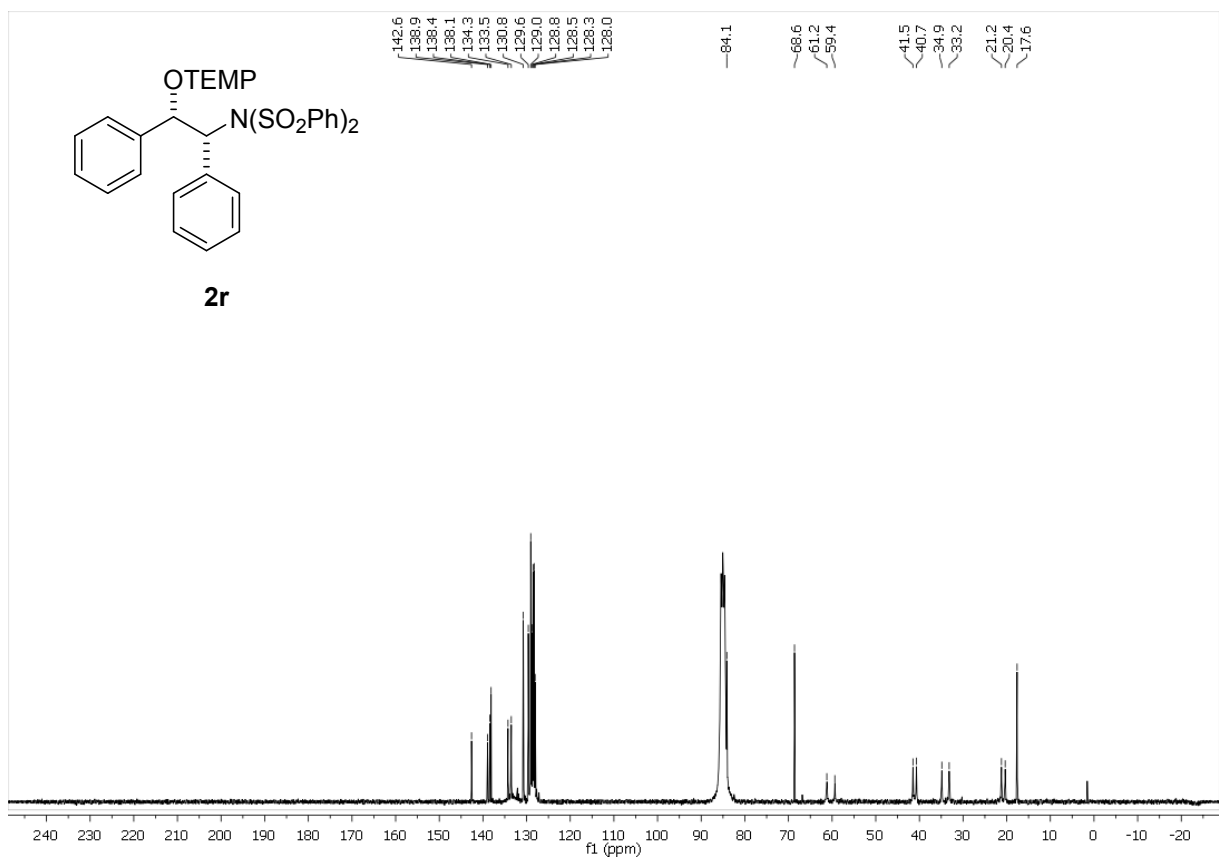
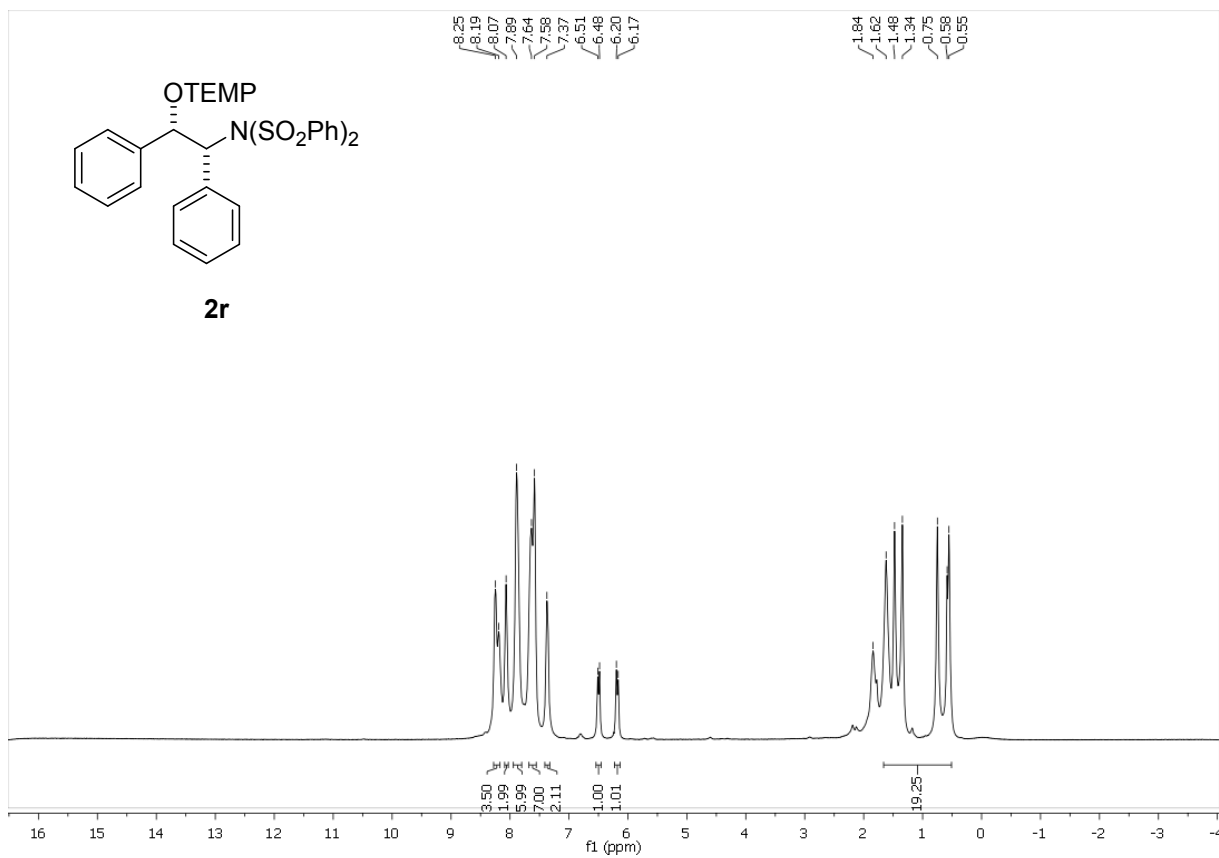


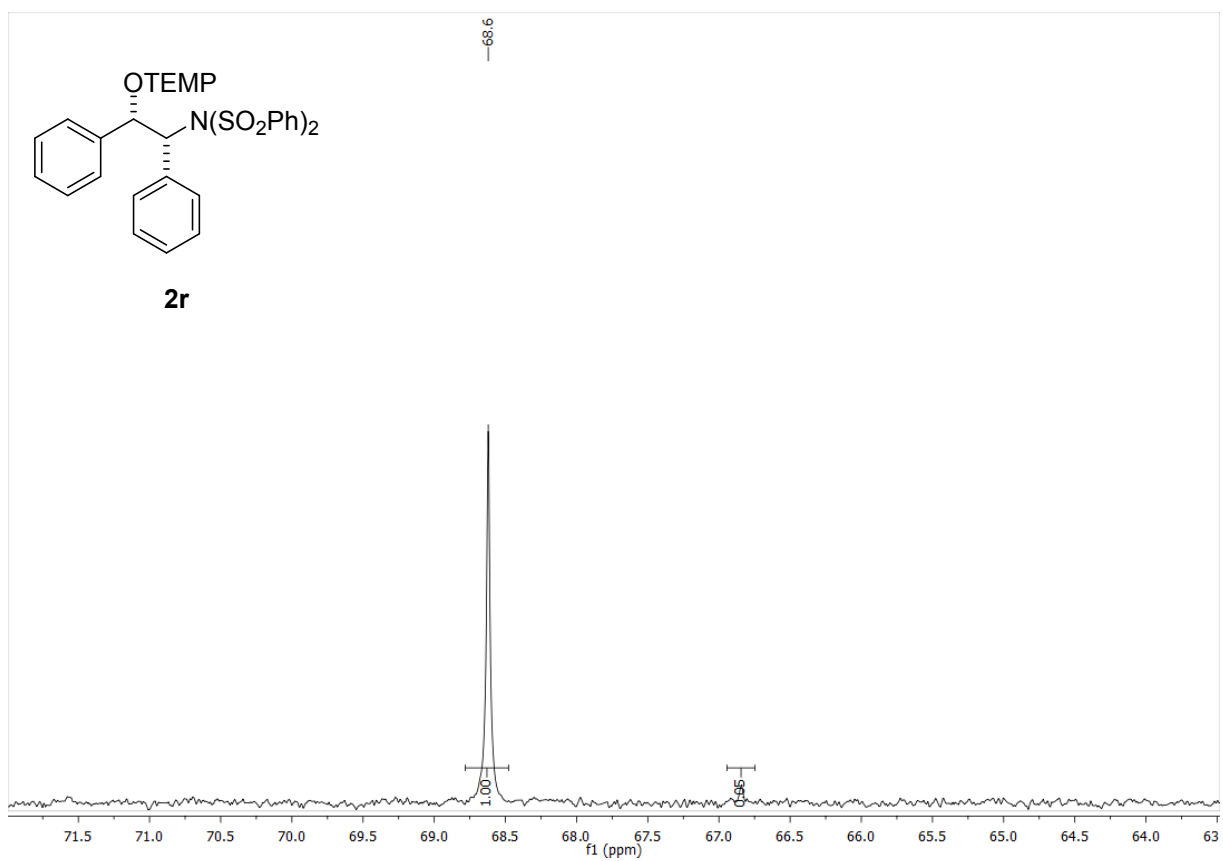
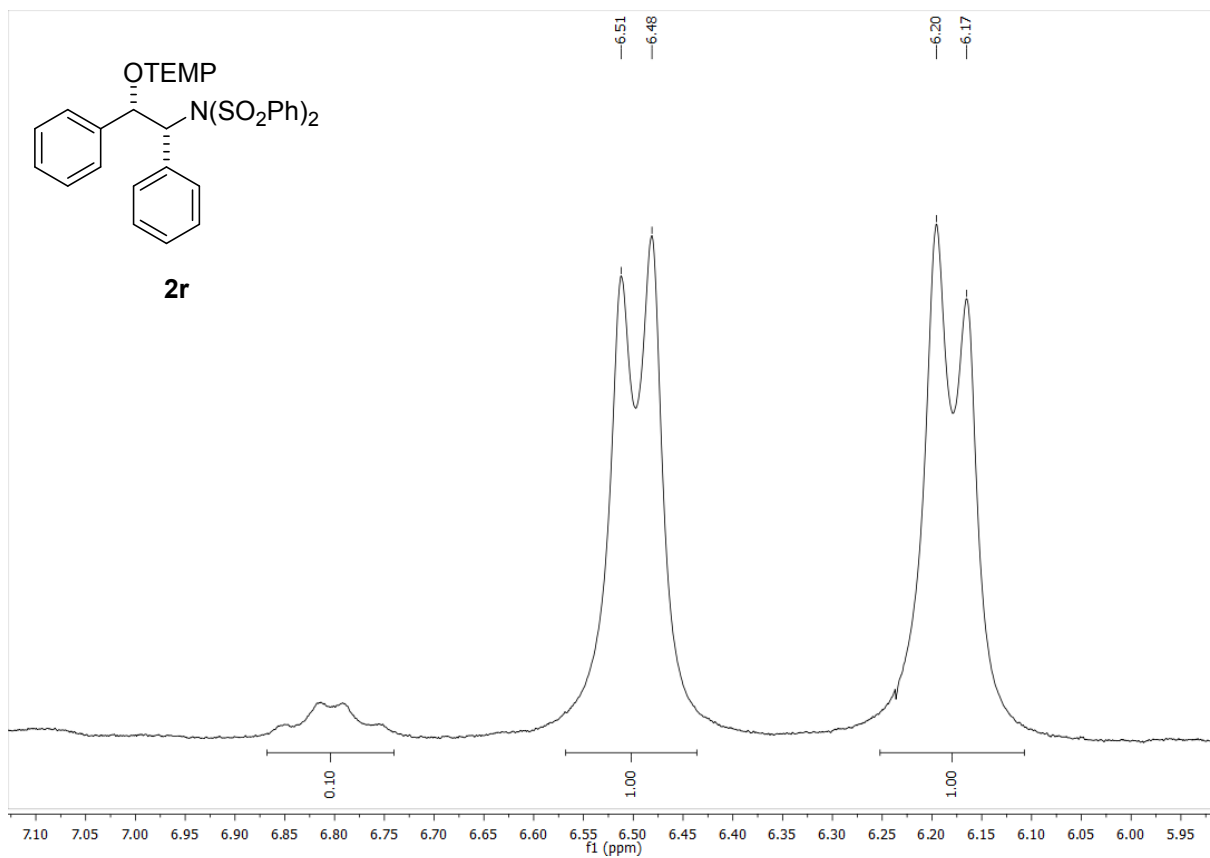


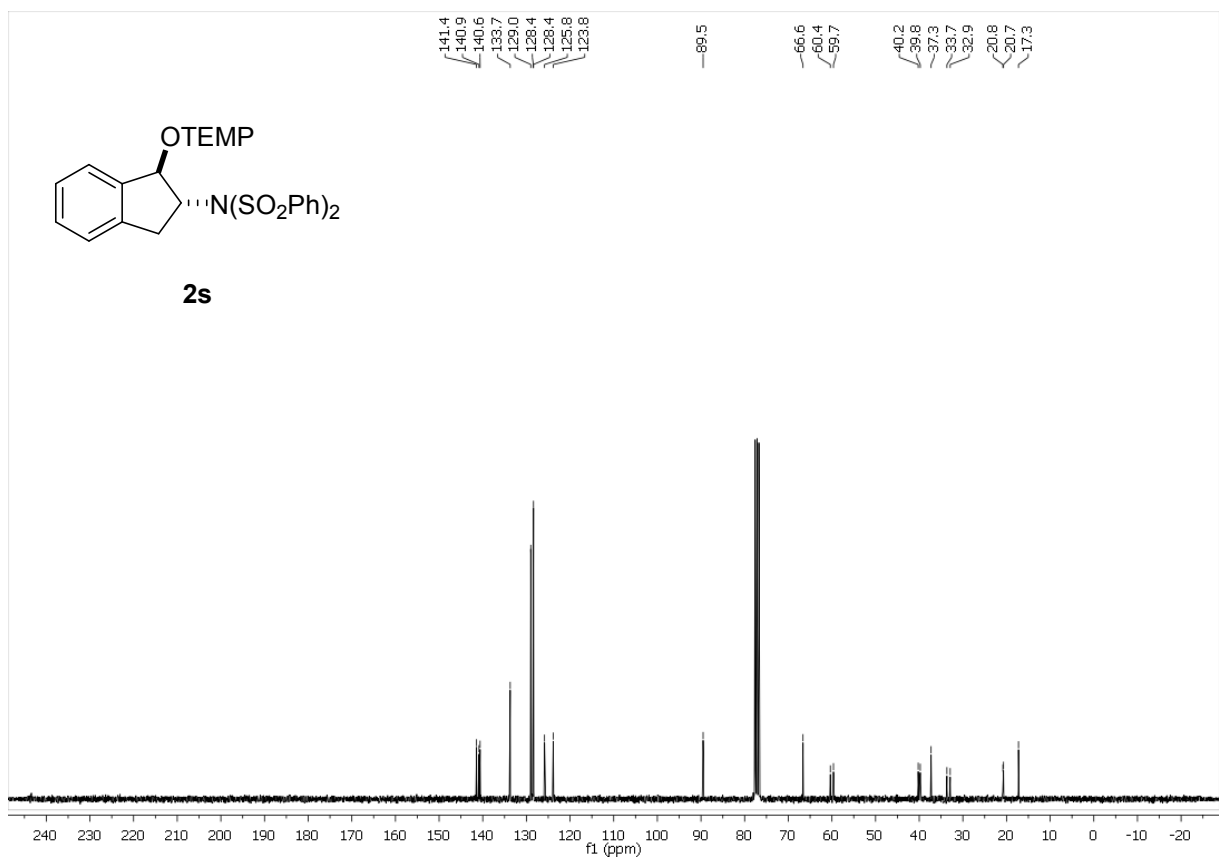
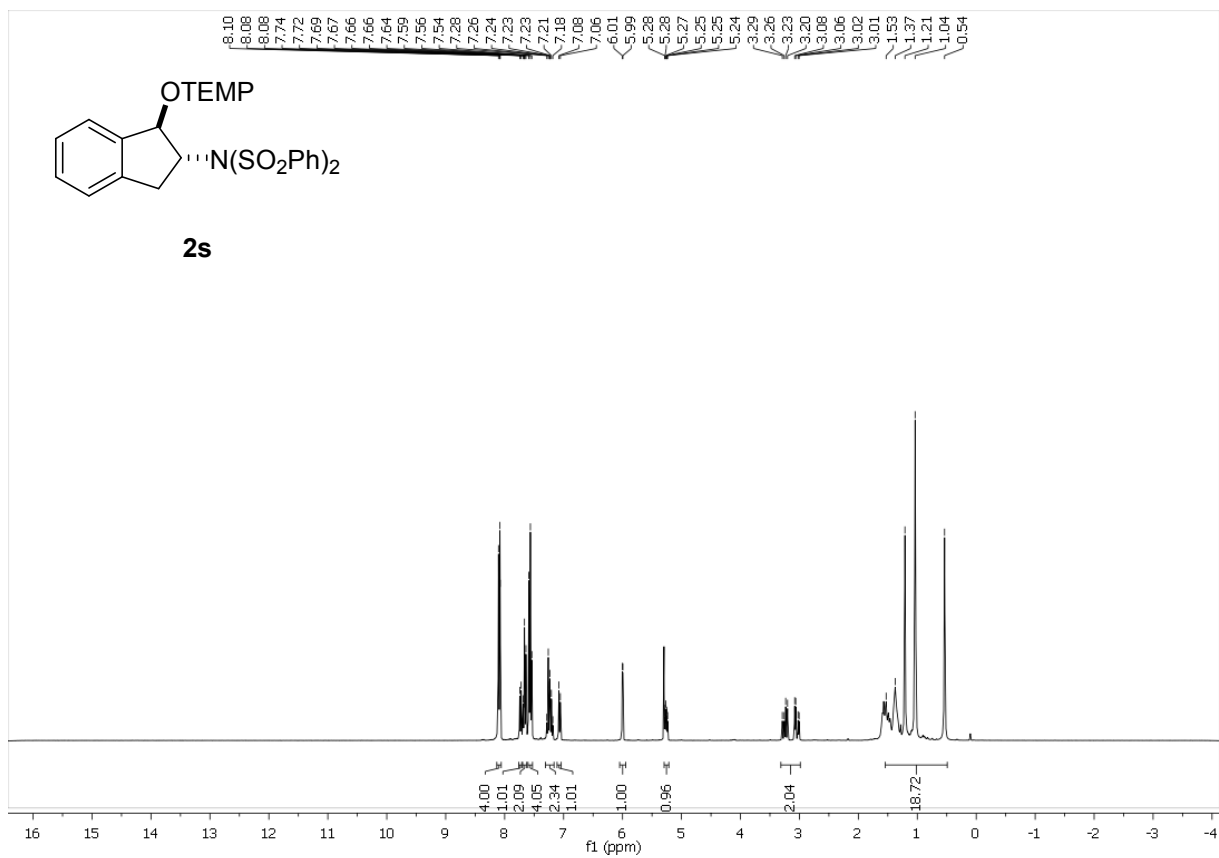


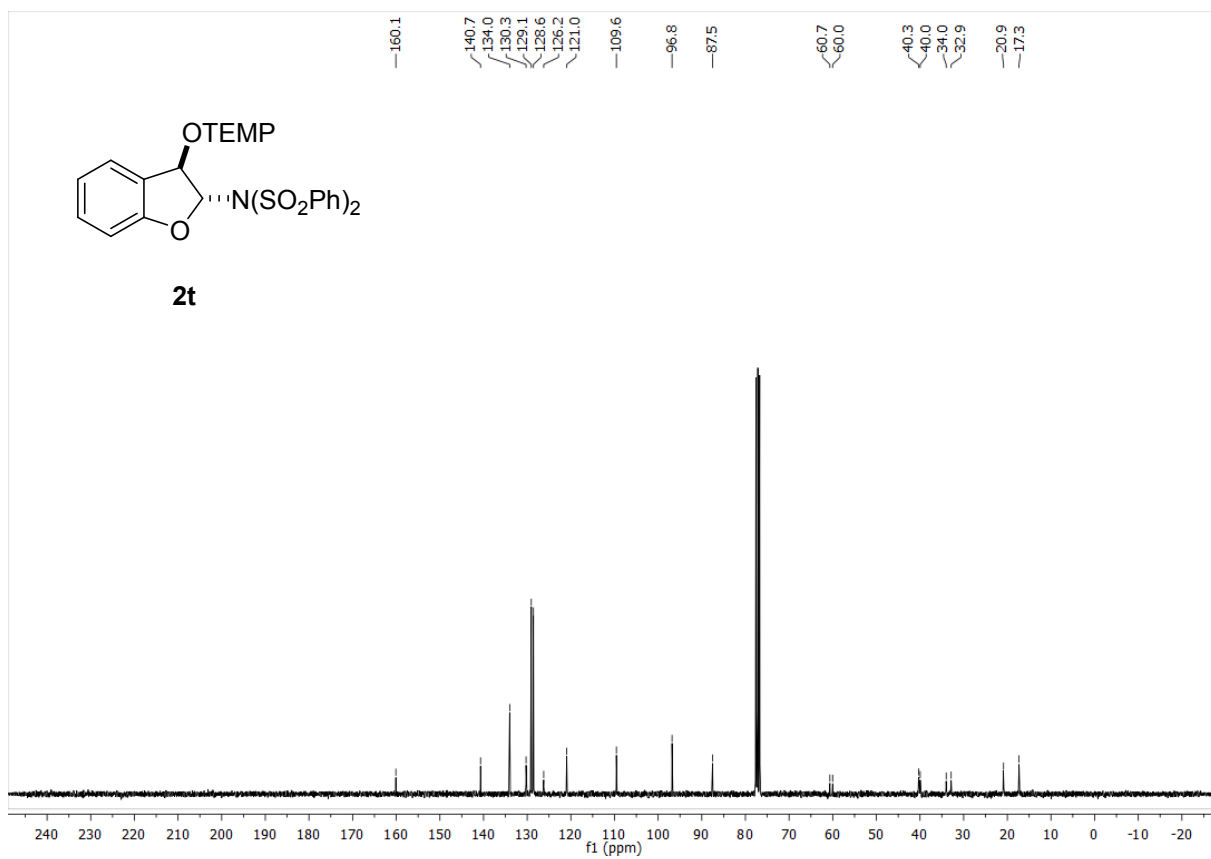
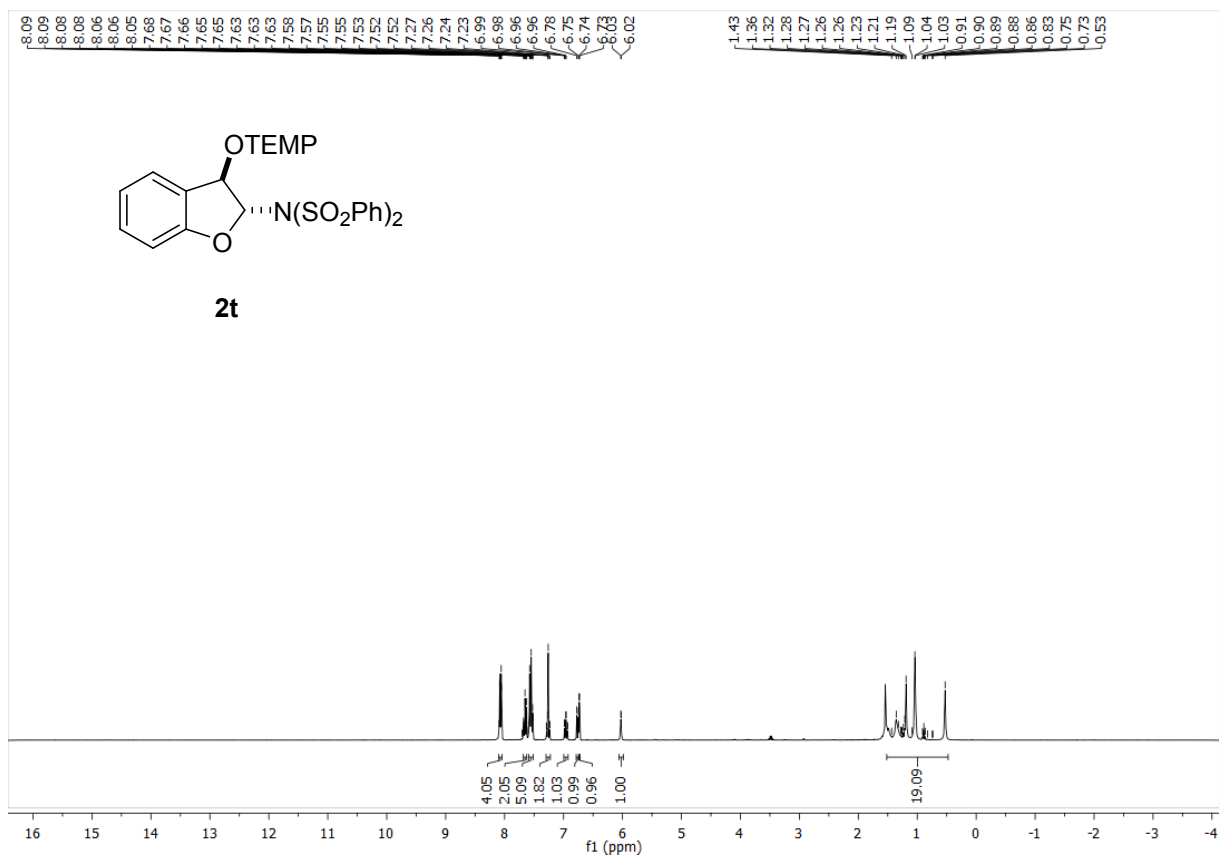


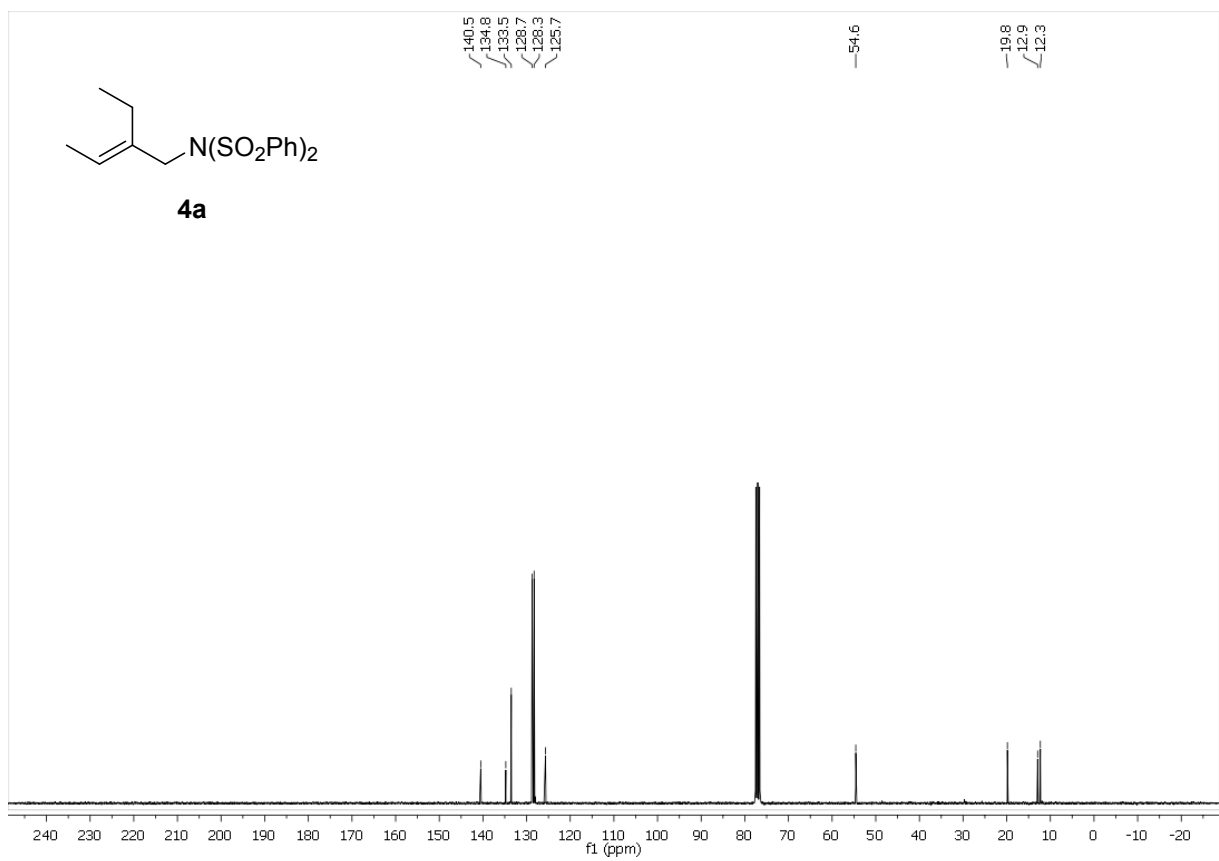
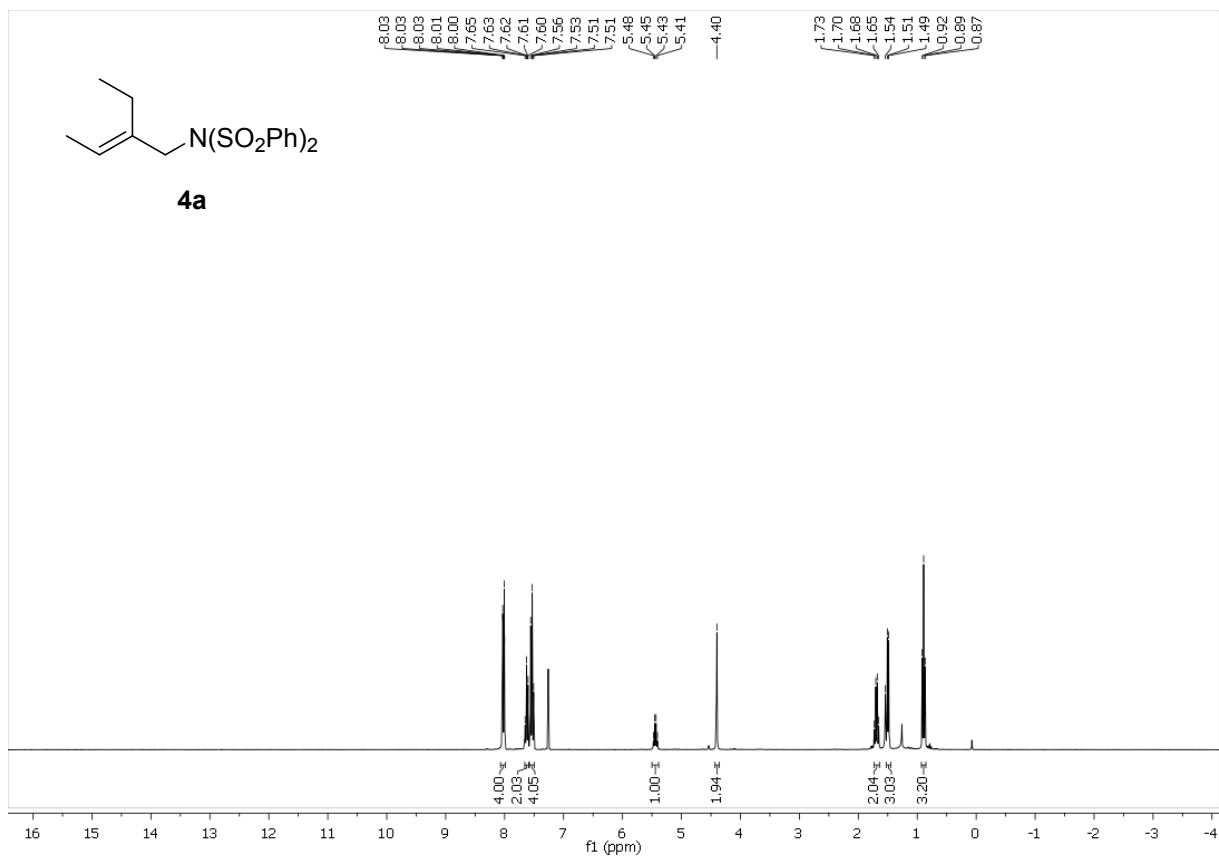


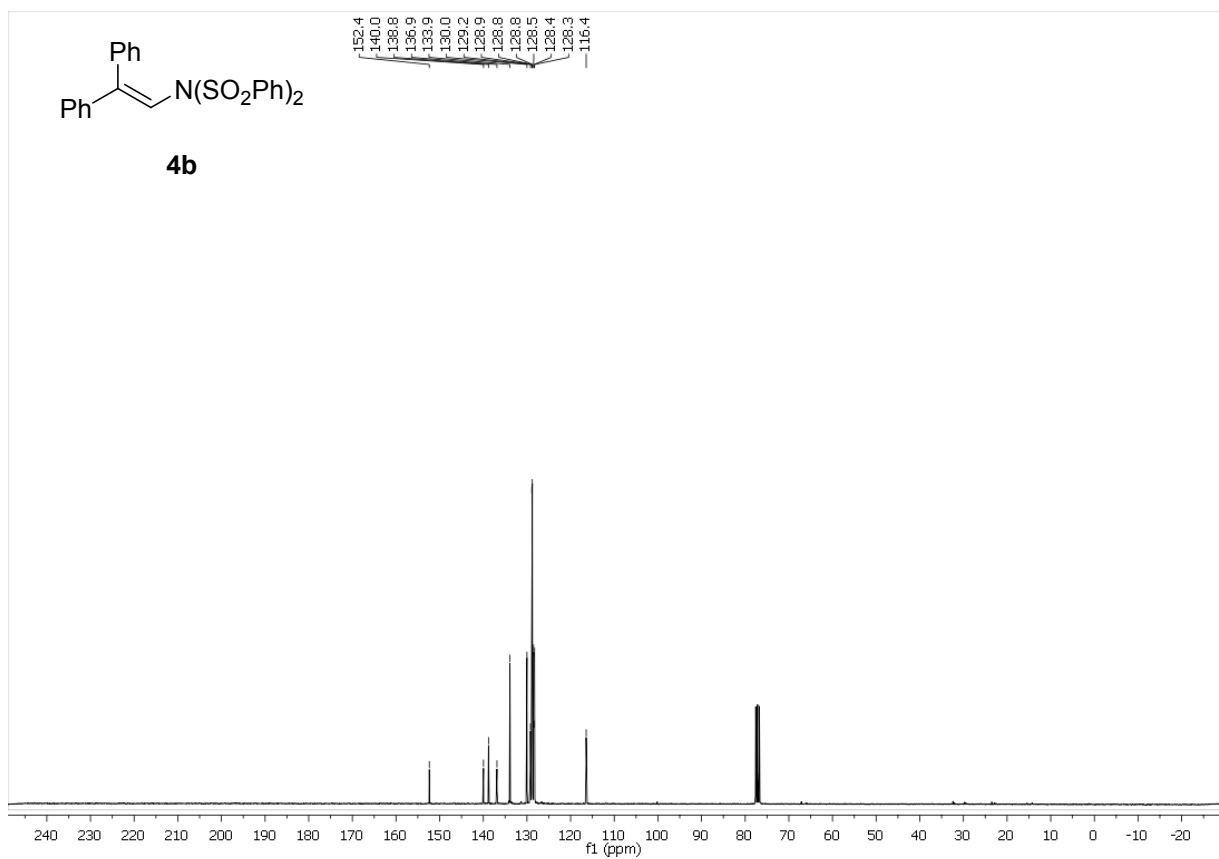
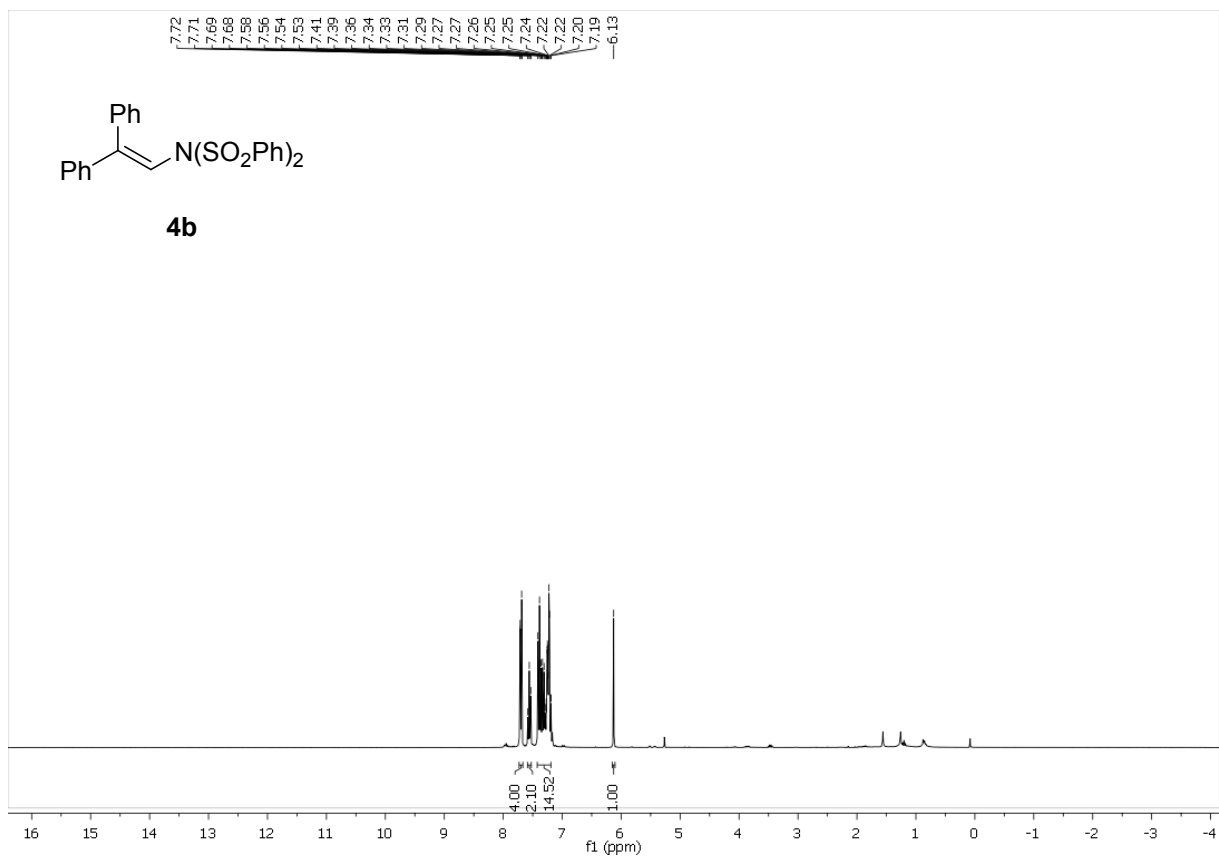




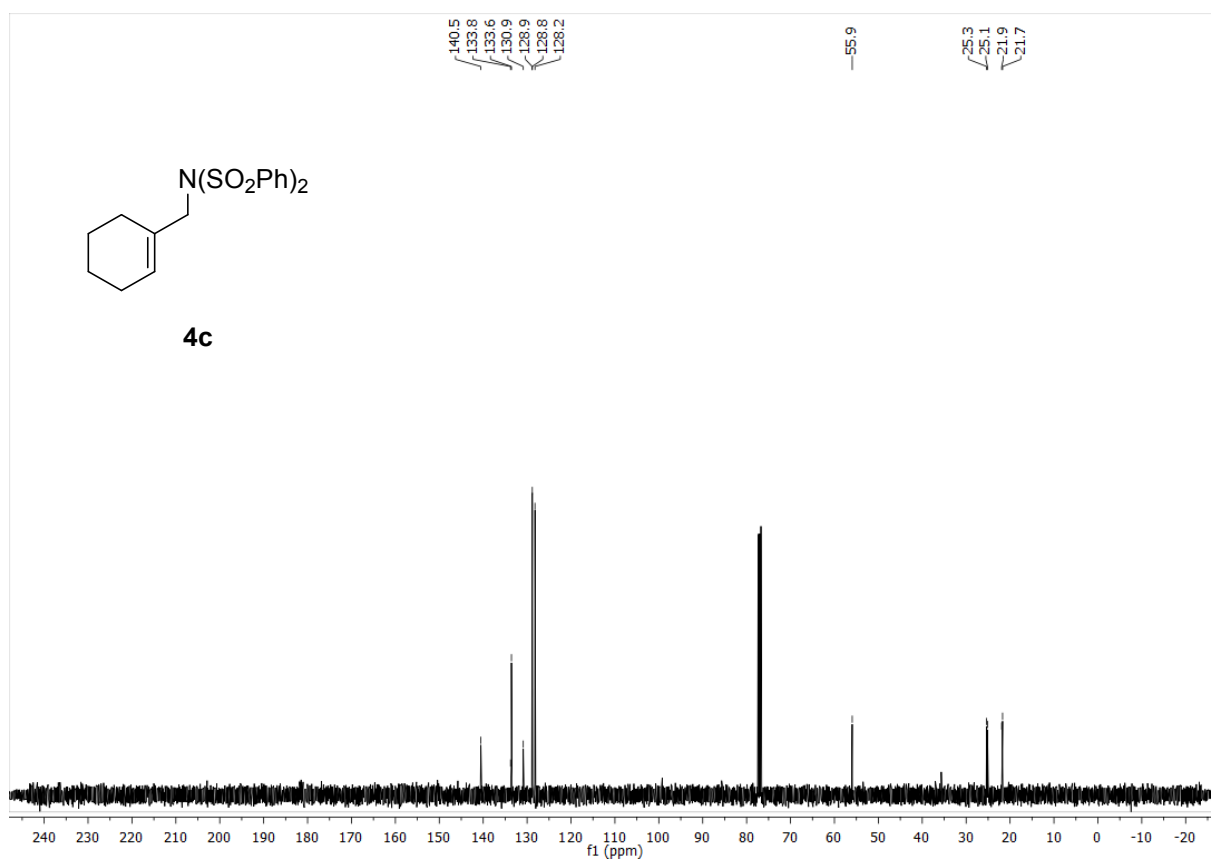
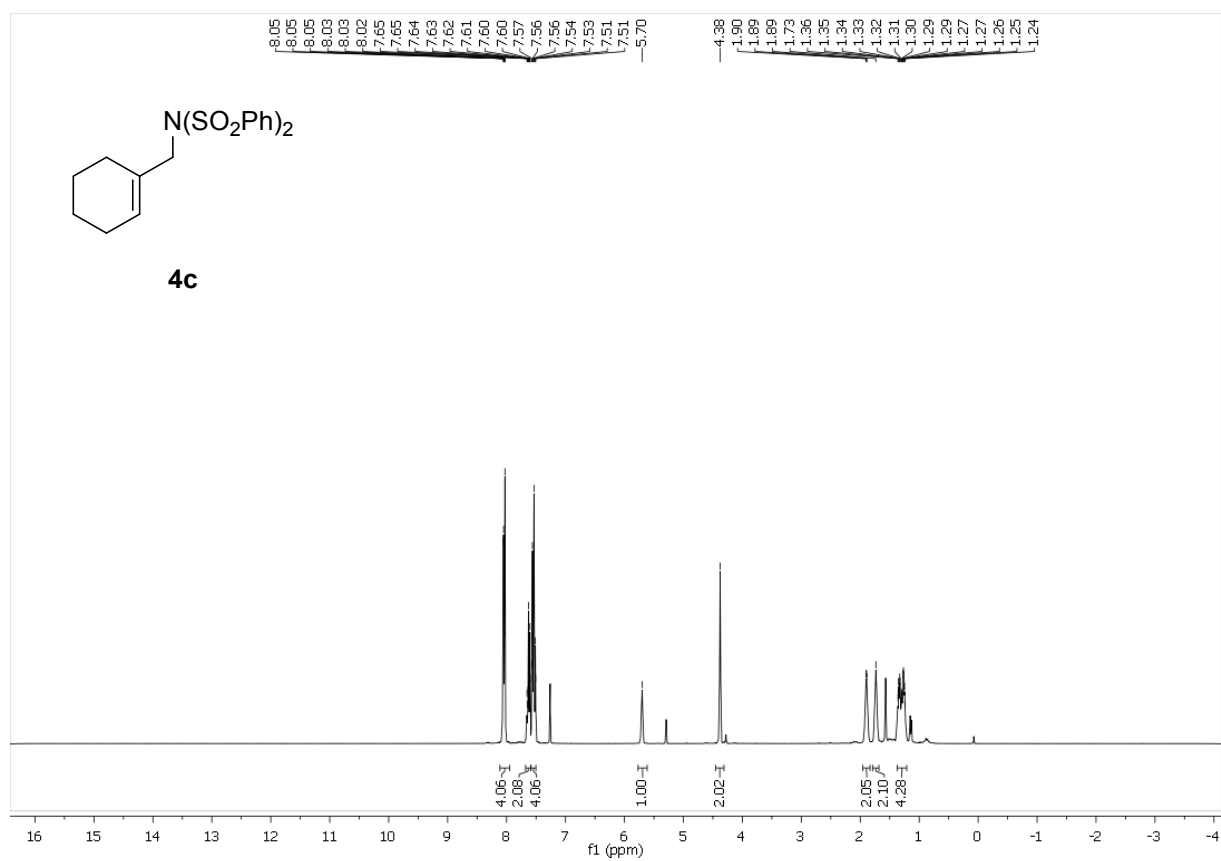


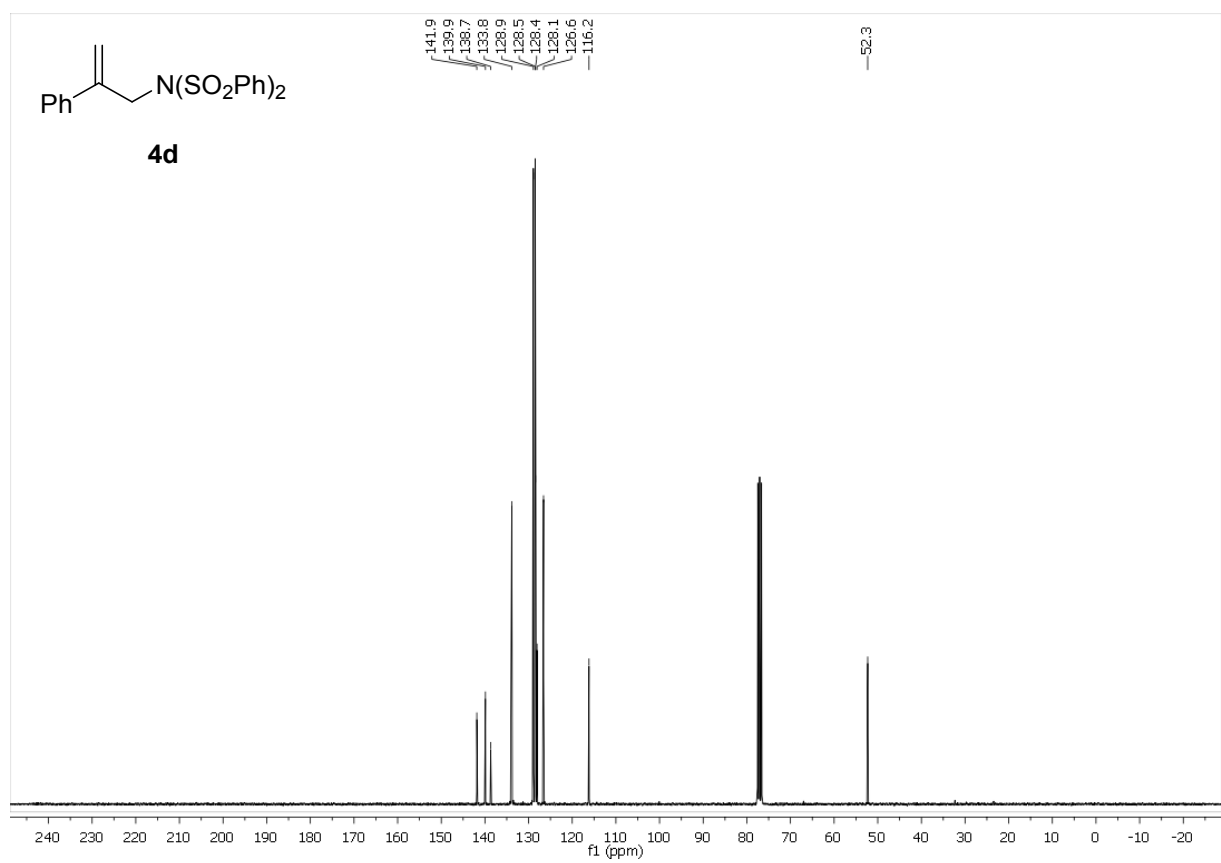
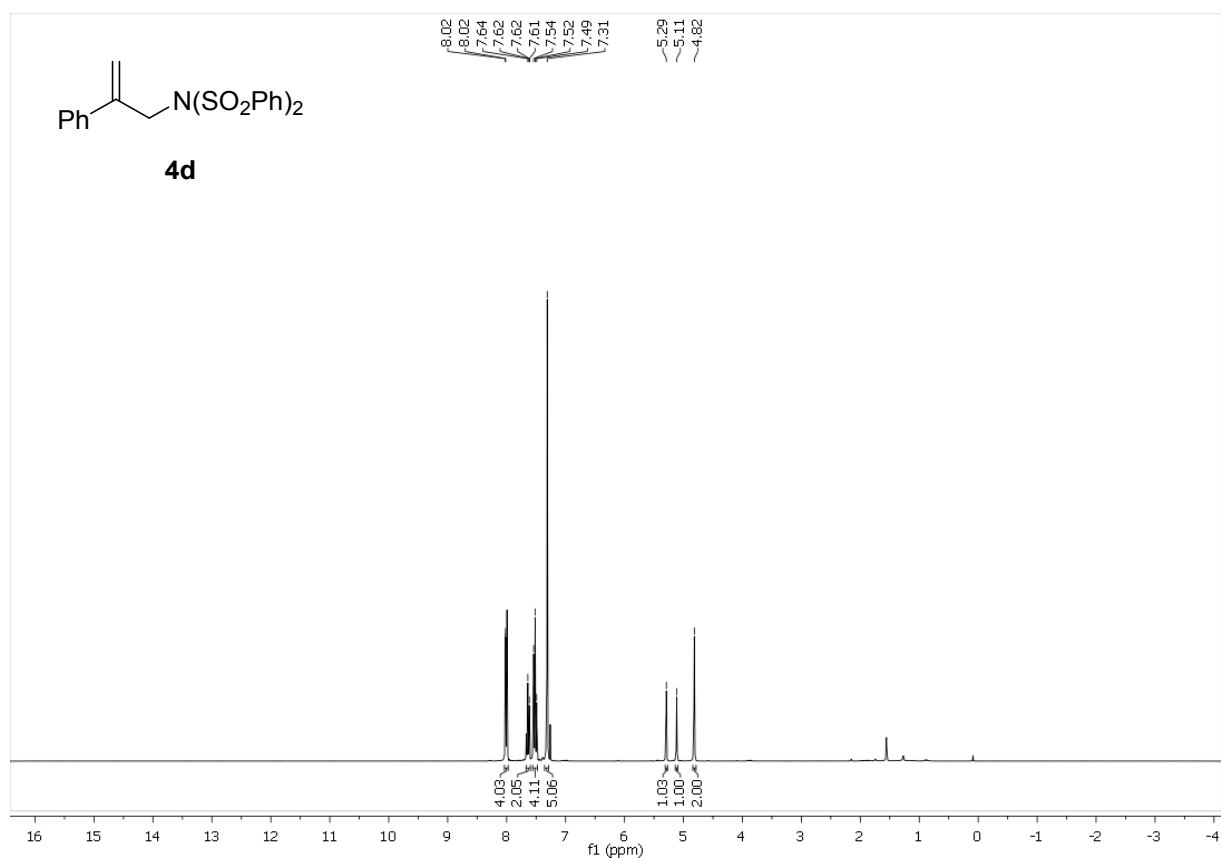


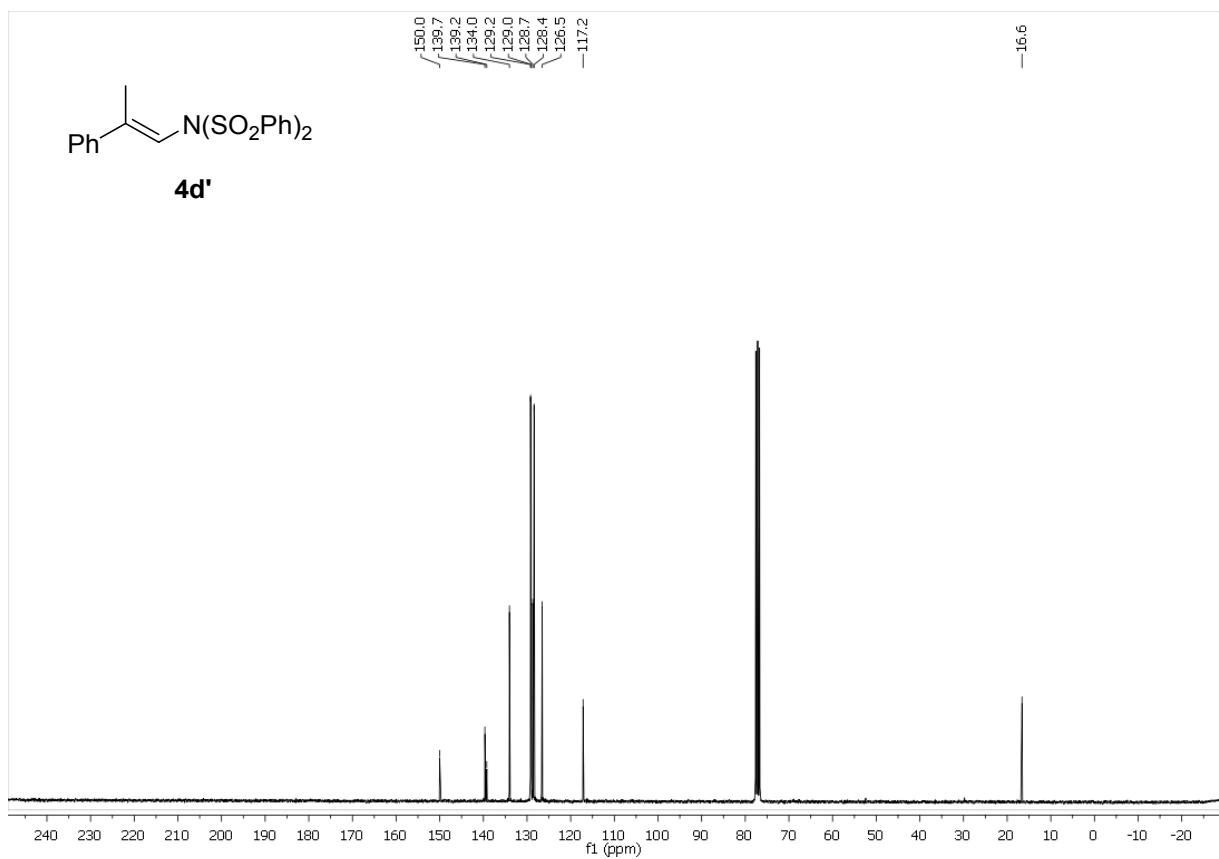
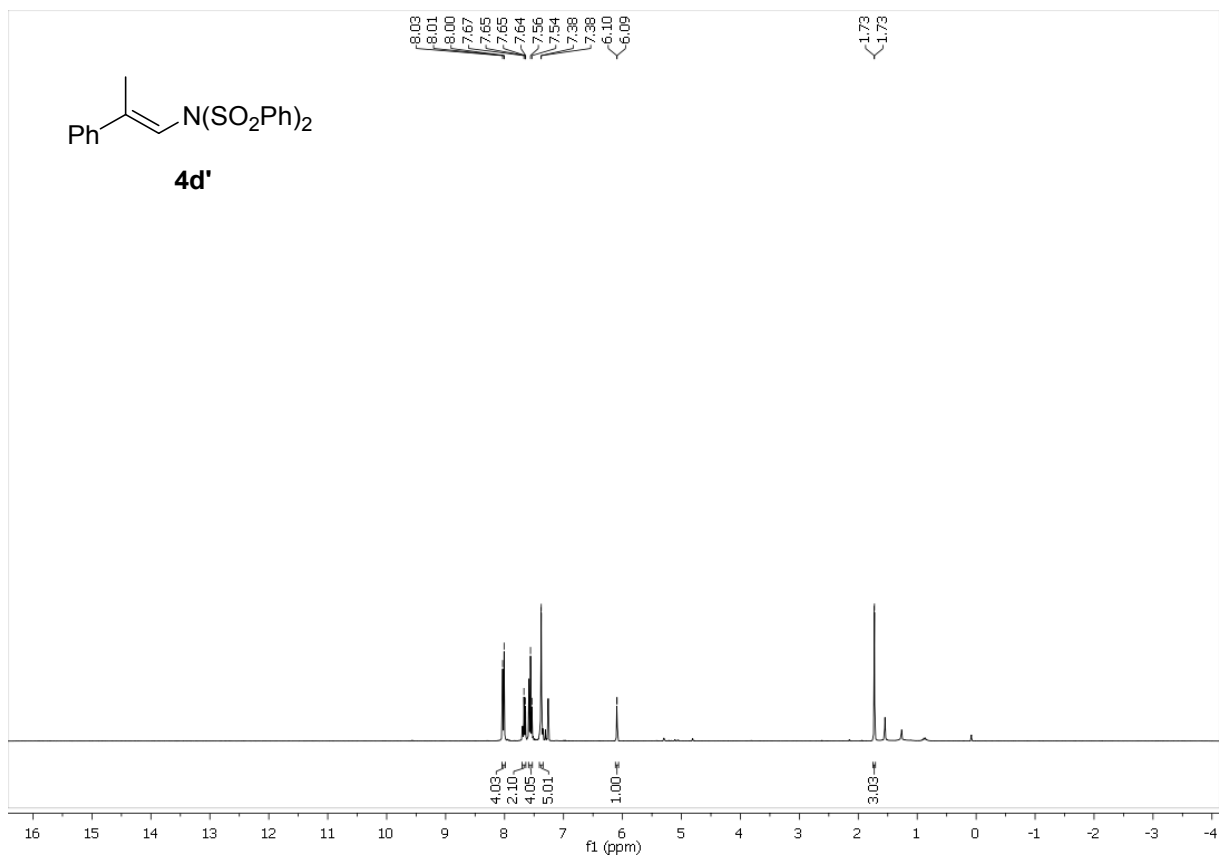


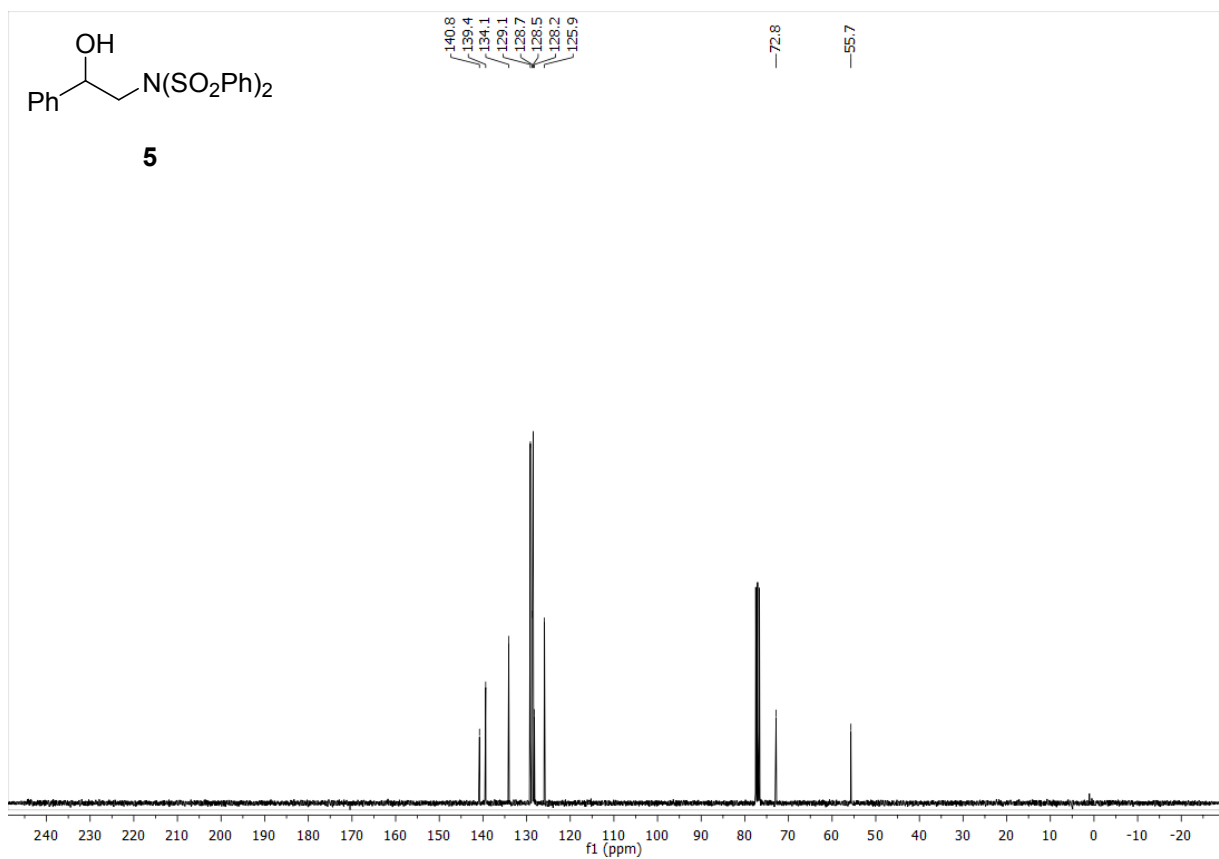
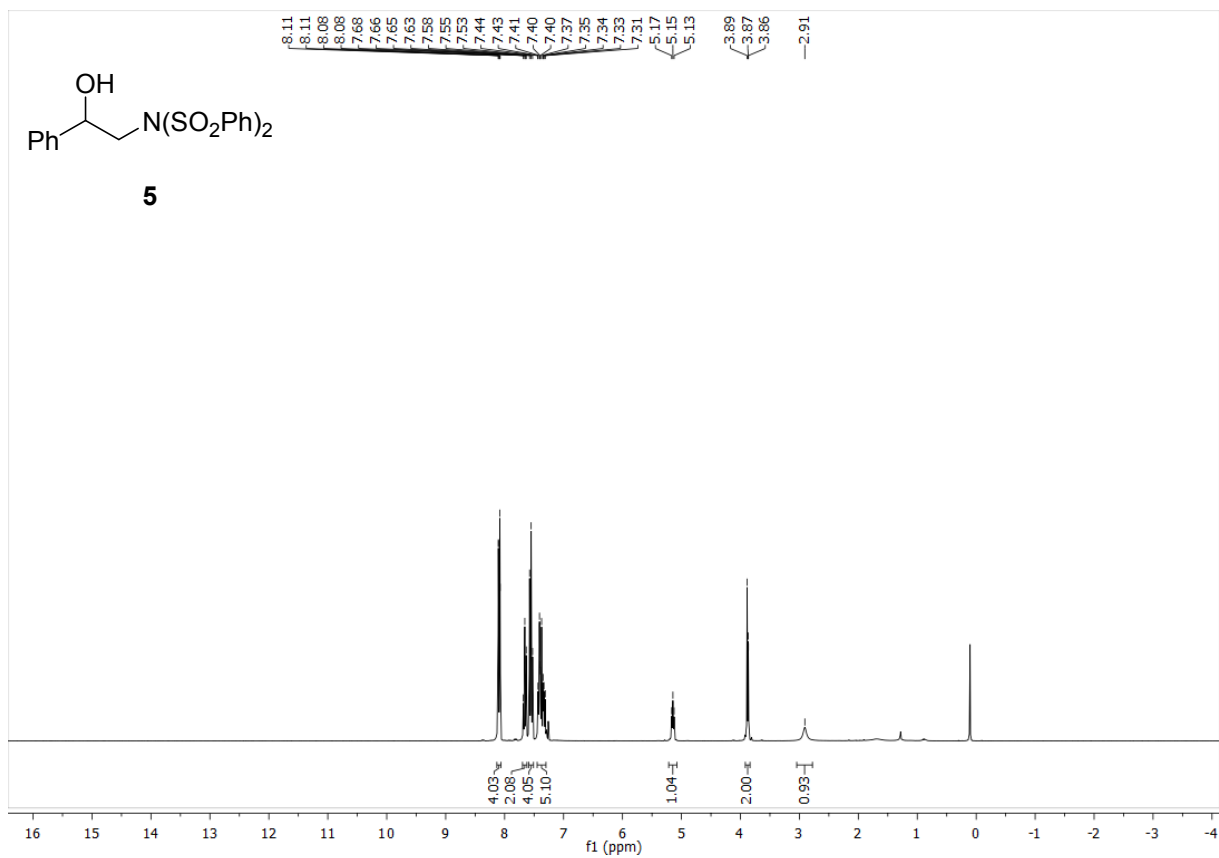


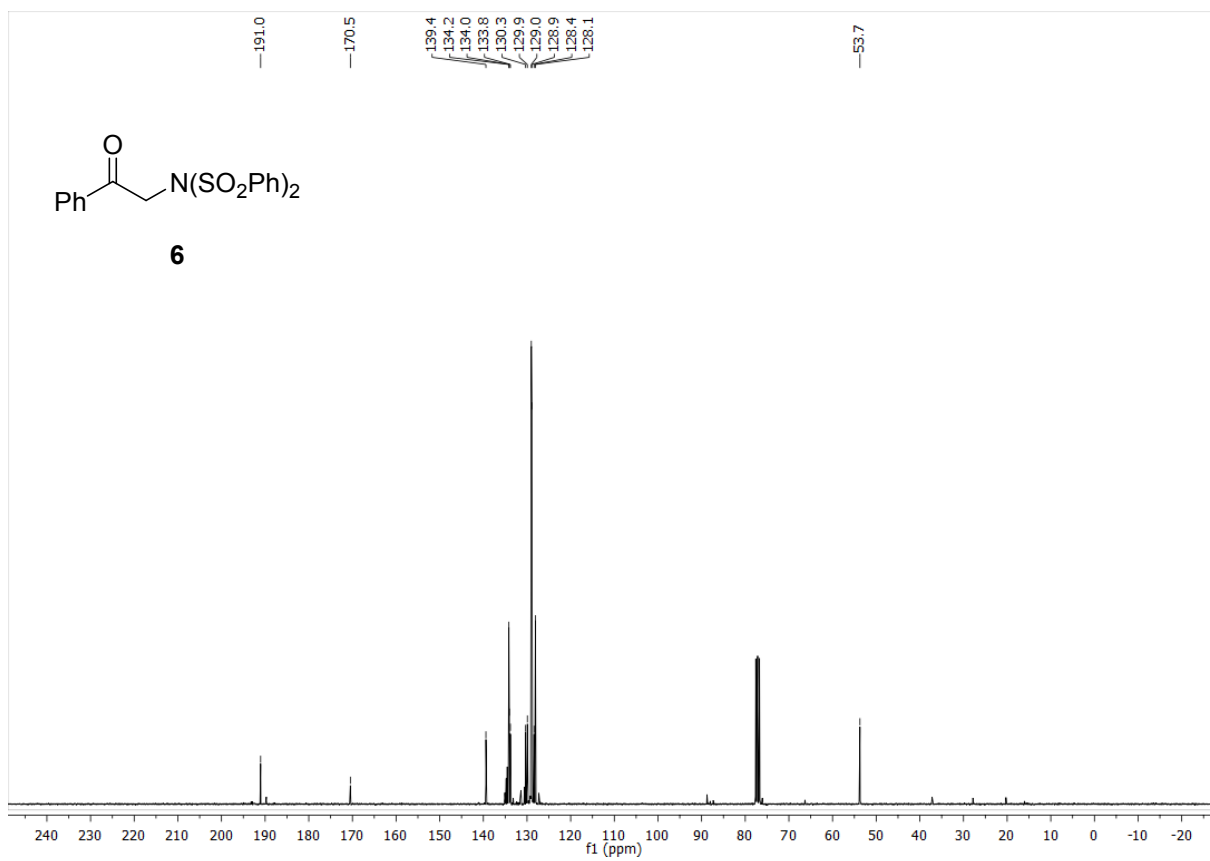
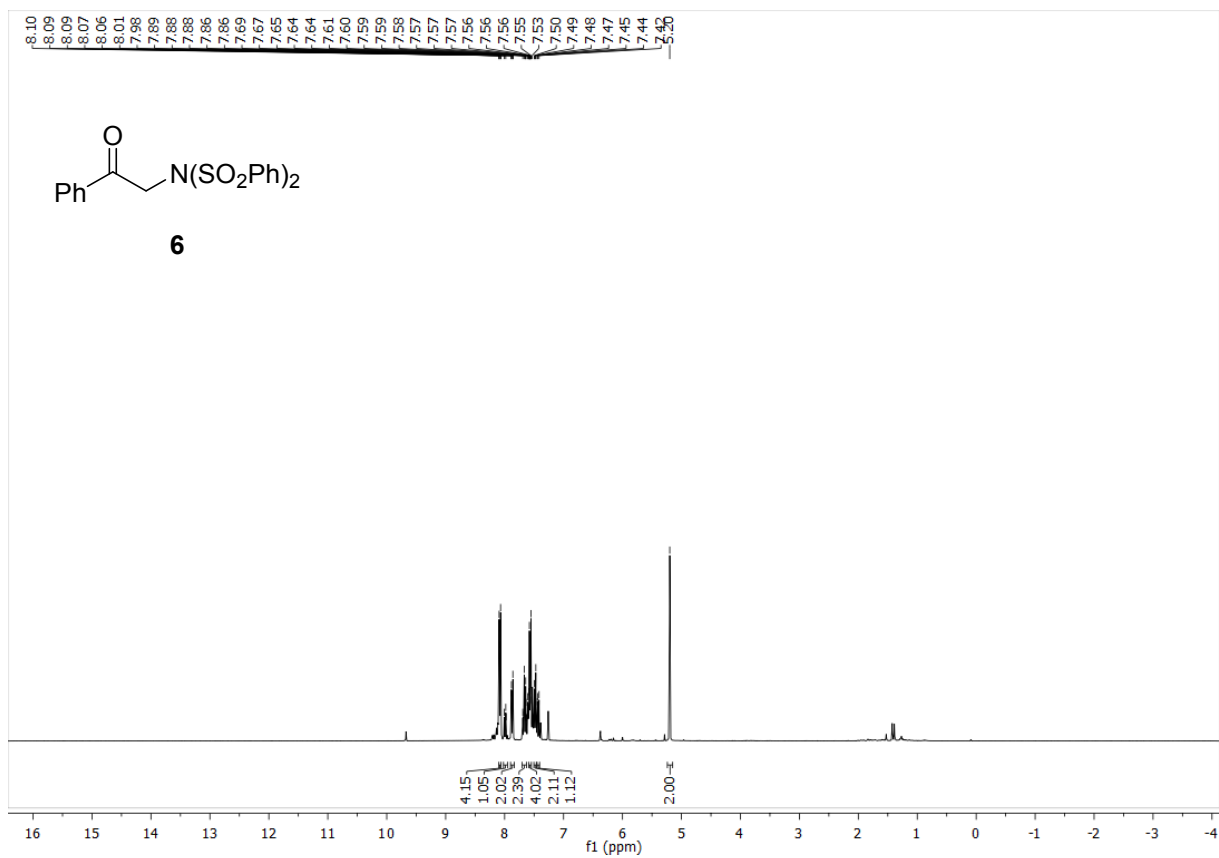


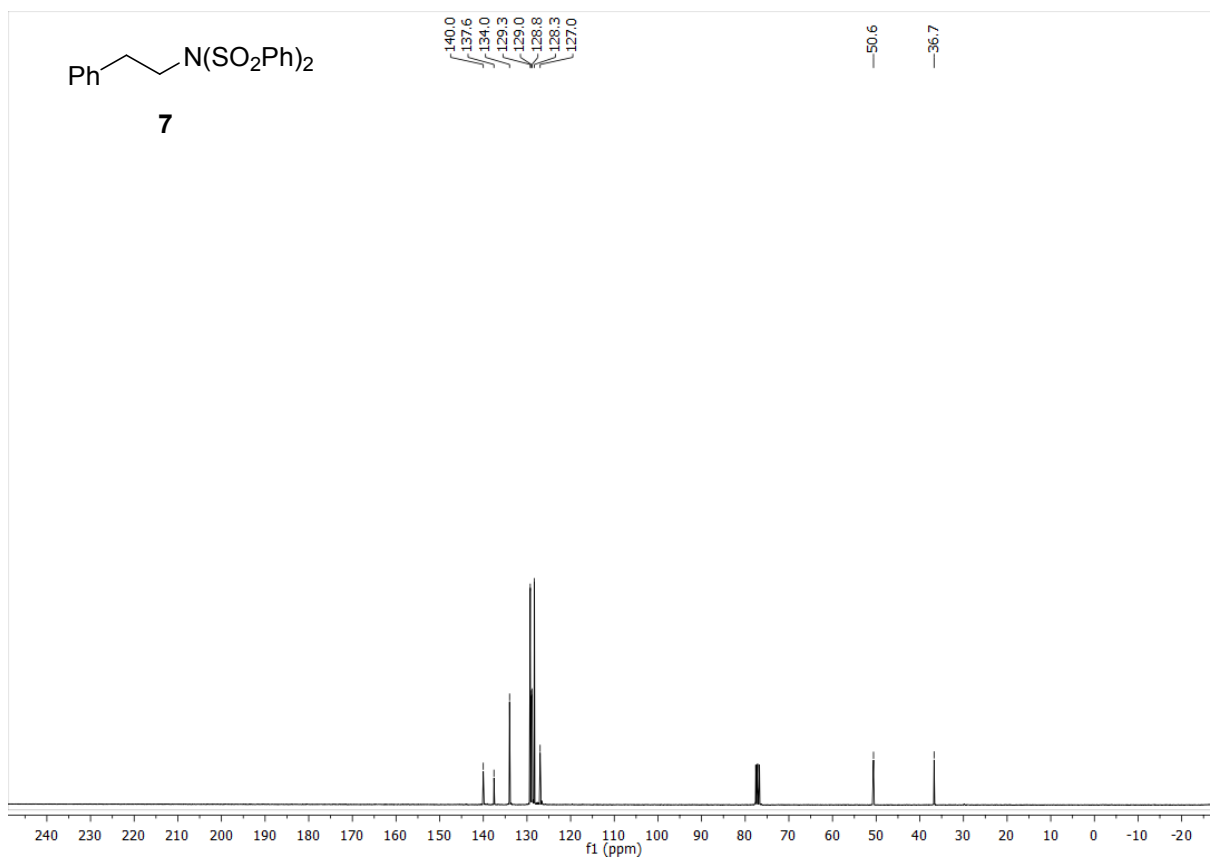
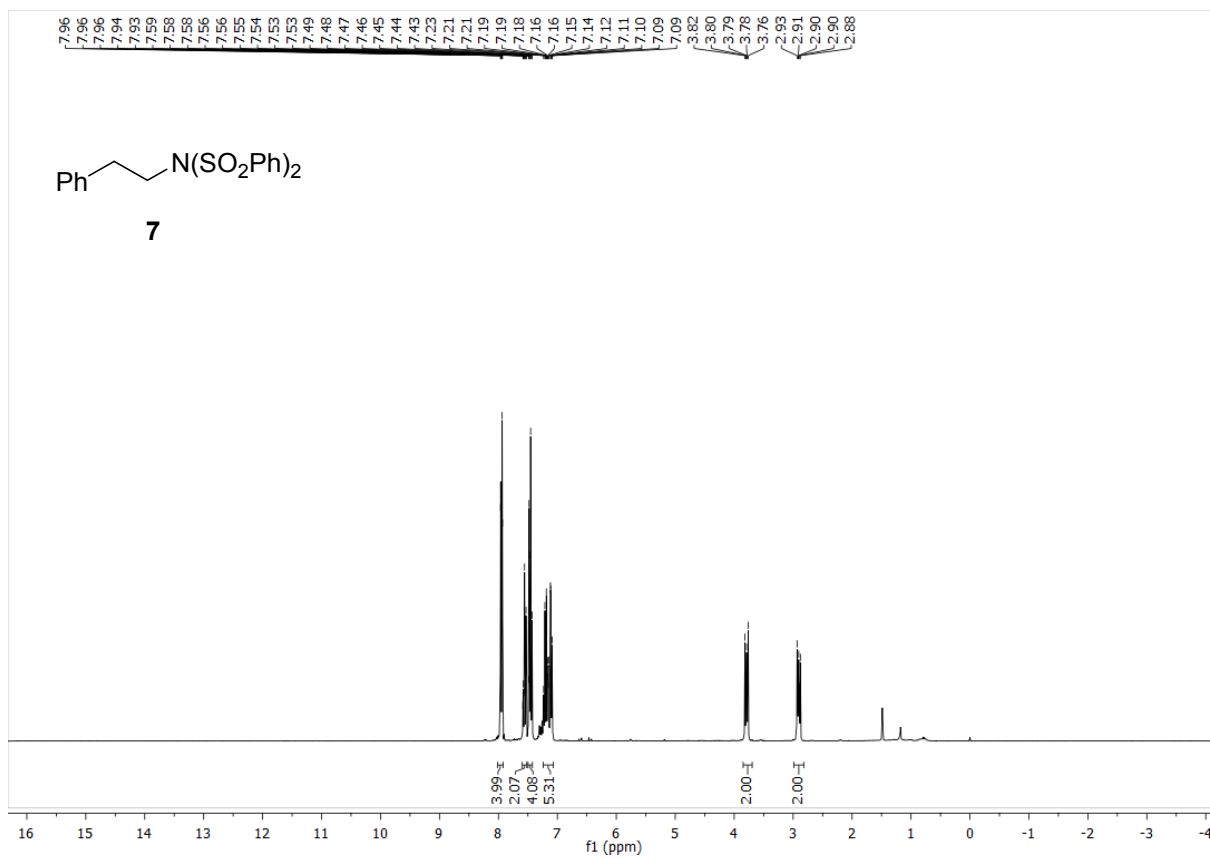


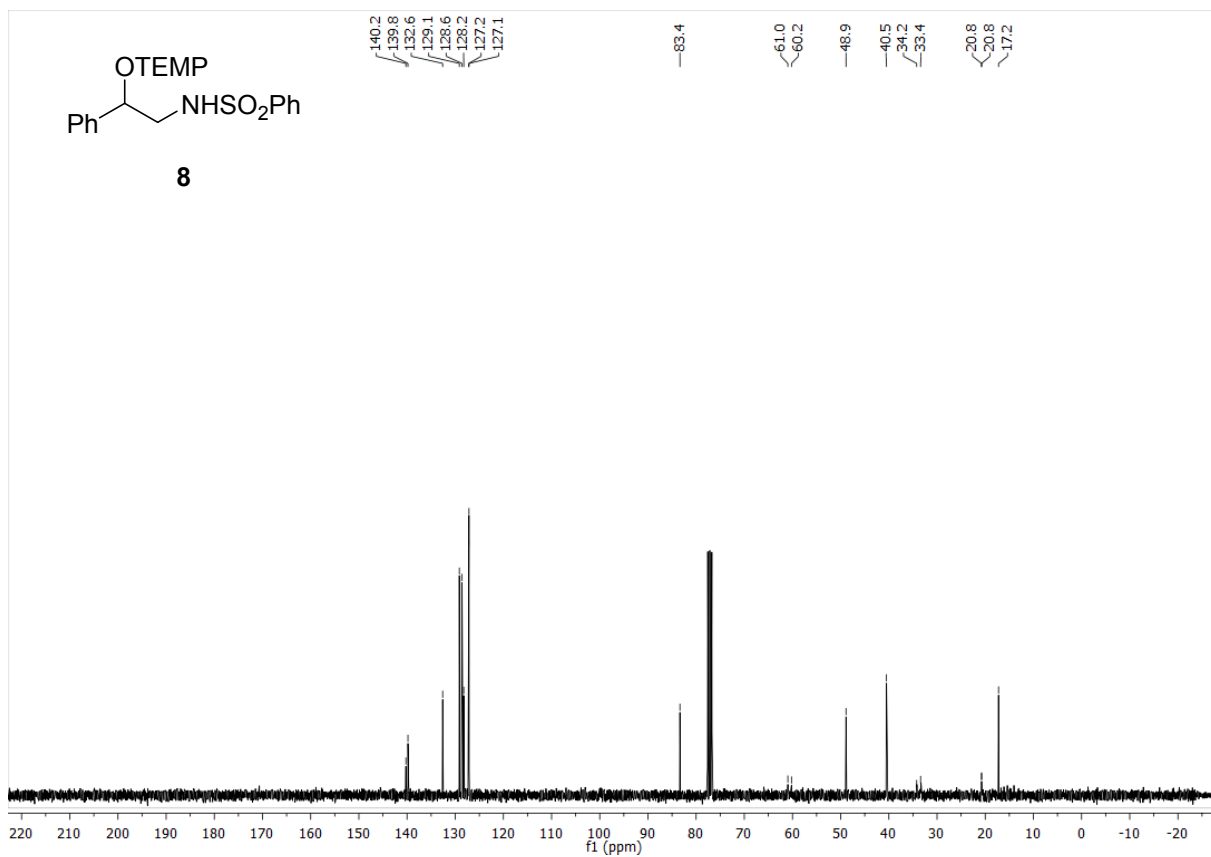
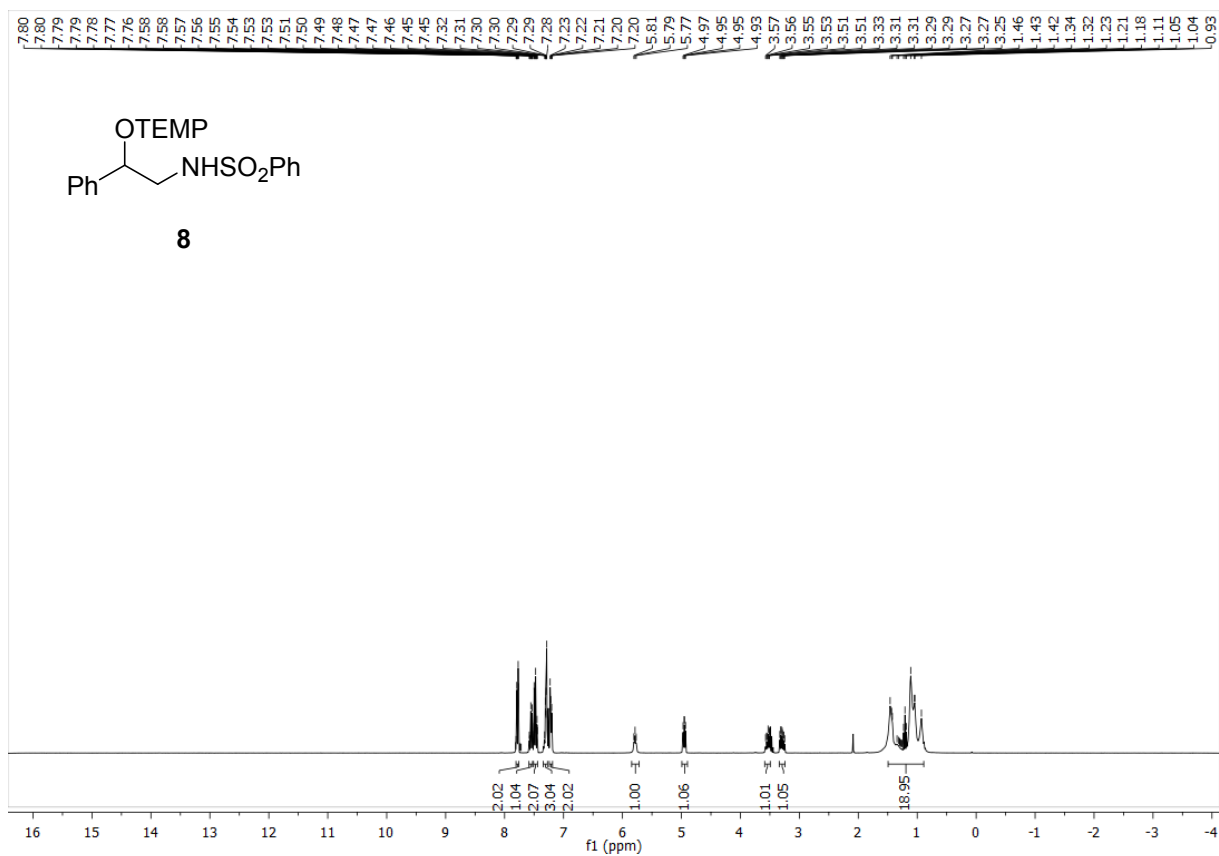












**Literature:**

- [1] *APEX2*, *SAINTE* and *SADABS* Bruker AXS Inc., Madison, Wisconsin, USA (2013).
- [2] *SHELXT* und *SHELXL* Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.