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Supplementary Information for

Efficient and Eco-friendly Catalyst-free Synthesis of *N*-Sulfonylimines from Sulfonamides and Aldehydes: Crucial role of Al_2O_3 as a Reusable Dehydrating Agent

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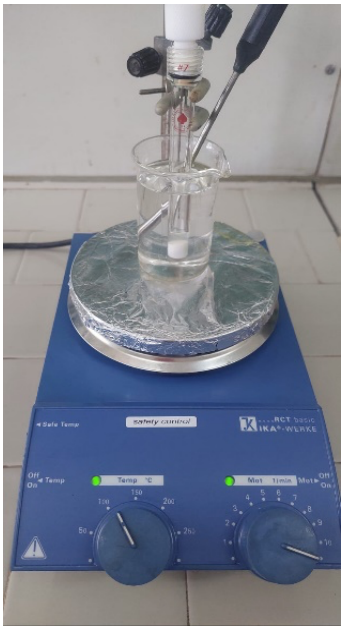
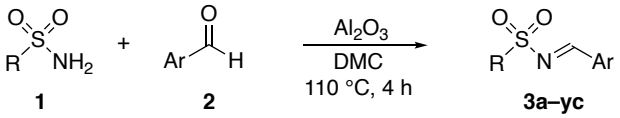
Table of contents

General methods

^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker AV 500 MHZ or AV 300 MHZ, using $CDCl_3$ as solvent. Chemical shifts (δ) are expressed in ppm relative to residual solvents ($CDCl_3$ δ_H 7.26, δ_C 77.16*).* In all ^{1}H NMR spectra, the number of protons for each signal and coupling constants values (Hertz) are indicated. Multiplicity is also reported and designated by the following abbreviations: s (singlet), d (doublet), t (triplet), q (quartet) m (multiplet). High-resolution mass spectroscopy (HRMS) was performed on a Q Extractive mass Spectrometer (Thermo Fisher). Melting points were measured with Kofler heating bench. Thin-layer chromatography (TLC) was performed on Merck 60F-254 precoated silica gel sheets (0.25 mm). Dimethyl carbonate (anhydrous ≥ 99%) and alumine (aluminum oxide, activated, neutral, 150 mesh, 58 Å, Fe_2O_3 max 0.03%) were purchased from Sigma-Aldrich. All chemicals obtained from commercial suppliers were used without further purification, except aldehydes were distilled or recrystallized (solid reagents) before use. Anhydrous solvents were prepared according to the standard methods. Alumine was activated at 200°C under reduced pressure (2 mbar) overnight before use.

General procedure for the synthesis of *N*-sulfonylimines



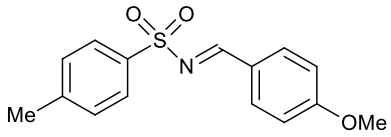


1 **2** **3a–yc**

A stirred mixture of sulfonamide **1** (1.2 mmol), aldehyde **2** (1 mmol), and 2 equiv. of activated Al_2O_3 in anhydrous DMC (1 mL) was heated in an 8-mL pressure tube at 110 °C for 4 h (reaction monitored by TLC: cyclohexane/AcOEt 8:3). After the reaction was complete, the mixture was filtered, and aluminum oxide was then washed thoroughly with 4 mL of hot DMC (60 °C). The collected filtrate was concentrated with the rotary evaporator under reduced pressure and crystallized from ethanol/water to yield pure *N*-sulfonylimines **3**. The DMC fractions, recovered after evaporation with the rotary evaporator, were subjected to fractional distillation at atmospheric pressure.

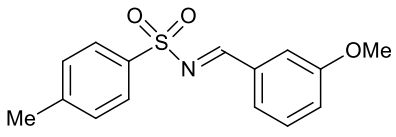
N-Sulfonylimines **3a**,[1] **3b**,[1] **3c**,[2] **3d**,[2] **3e**,[1] **3f**,[1] **3g**,[1] **3h**,[2] **3i** ,[3] **3j** ,[1] **3k**,[1] **3l** ,[1] **3m**,[1] **3n**,[2] **3o**,[2] **3p**,[1] **3q**,[2] **3r**,[4] **3s**,[5] **3t**,[5] **3v**,[5] **and 3x** [6] are known compounds and previously reported in the literature.

N-sulfonylimines **3u**, **3w**, **3ya**, **3yb**, and **3yz** are new compounds.



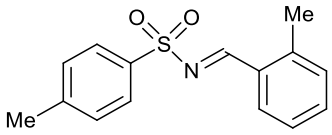
3a

***(E)-N*-(4-methoxybenzylidene)-4-methylbenzenesulfonamide 3a.**[1] According to the general procedure, the product was isolated as white solid; m = 269 mg; 93% (mp = 126–128 °C); ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 8.94 (3H, s), 7.95–7.82 (4H, m), 7.33 (2H, d, *J* = 8.1 Hz), 6.96, (2H, d, *J* = 9 Hz), 3.88 (3H, s), 2.43 (3H, s); ^{13}C NMR (75 MHz; $CDCl_3$) δ 169.3, 165.4, 144.4, 135.9, 133.9, 129.9, 128.0, 125.4, 114.8, 55.8, 21.8.



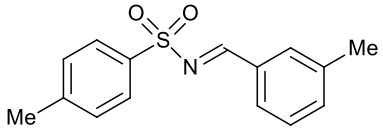
3b

***(E)-N*-(3-methoxybenzylidene)-4-methylbenzenesulfonamide 3b.**[1] According to the general procedure, the product was isolated as white solid; m = 270 mg; 93% (mp = 56–58 °C); ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 8.98 (1H, s), 7.89 (2H, d, *J* = 8.1 Hz), 7.51–7.31 (5H, m), 7.20–7.10 (1H, m), 3.83 (3H, s), 2.44 (3H, s); ^{13}C NMR (75 MHz; $CDCl_3$) δ 170.3, 160.2, 144.8, 135.2, 133.8, 130.2, 130.0, 128.3, 125.5, 122.3, 113.4, 55.7, 21.8.



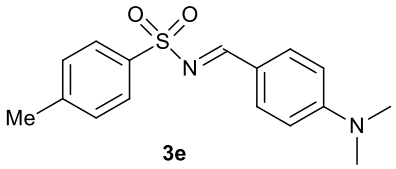
3c

***(E)*-4-methyl-*N*-(2-methylbenzylidene)benzenesulfonamide 3c.**[2] According to the general procedure, the product was isolated as white solid; m = 254 mg; 93% (mp = 91–92 °C); ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 9.34 (1H, s), 8.00 (1H, d, *J* = 7.8 Hz), 7.89 (2H, d, *J* = 8.1 Hz), 7.47 (1H, t, *J* = 7.5 Hz), 7.34 (2H, d, *J* = 7.8 Hz), 7.31–7.20 (2H, m), 2.60 (3H, s), 2.43 (3H, s); ^{13}C NMR (75 MHz; $CDCl_3$) δ 168.8, 144.6, 142.4, 135.5, 134.7, 131.7, 130.8, 130.5, 129.9, 128.1, 126.7, 21.8, 19.8.



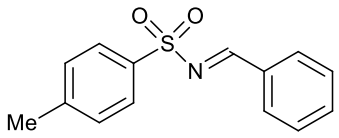
3d

***(E)*-4-methyl-*N*-(3-methylbenzylidene)benzenesulfonamide 3d.**[2] According to the general procedure, the product was isolated as white solid; m = 255 mg; 93% (mp = 88–90 °C); ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 8.99 (1H, s), 7.88 (2H, d, *J* = 8.4 Hz), 7.76 (1H, s), 7.70 (1H, d, *J* = 7.2 Hz), 7.46–7.31 (4H, m), 2.43 (3H, s), 2.38 (3H, s); ^{13}C NMR (75 MHz; $CDCl_3$) δ 170.5, 144.7, 139.2, 136.0, 135.3, 132.5, 131.5, 129.9, 129.1, 128.2, 21.8, 21.3.



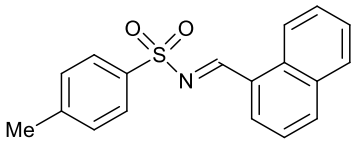
3e

***(E)*-*N*-(4-(dimethylamino)benzylidene)-4-methylbenzenesulfonamide 3e.**[1] According to the general procedure, the product was isolated as yellow solid; m = 287 mg; 95% (mp = 171–173 °C); ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 8.81 (1H, s), 7.85 (2H, d, *J* = 8.4 Hz), 7.76 (2H, d, *J* = 9.0 Hz), 7.29 (2H, d, *J* = 8.4 Hz), 6.65 (2H, d, *J* = 9.0 Hz), 3.09 (6 H, s), 2.41 (3H, s); ^{13}C NMR (75 MHz; $CDCl_3$) δ 169.1, 155.0, 143.7, 137.0, 134.1, 129.7, 127.7, 119.9, 111.4, 40.2, 21.7.



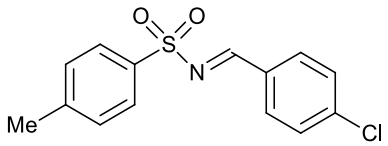
3f

***(E)-N*-benzylidene-4-methylbenzenesulfonamide 3f.**[1] According to the general procedure, the product was isolated as white solid; m = 249 mg; 96% (mp = 111–113 °C); ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 9.03 (1H, s), 7.92 (2H, d, *J* = 7.2 Hz), 7.89 (2H, d, *J* = 8.4 Hz) 7.67–7.55 (1H, m), 7.53–7.45 (2H, m), 7.34 (2H, d, *J* = 7.8 Hz), 2.43 (3H, s); ^{13}C NMR (75 MHz; $CDCl_3$) δ 170.3, 144.7, 135.4, 135.0, 132.6, 131.4, 129.9, 129.3, 128.2, 21.8.



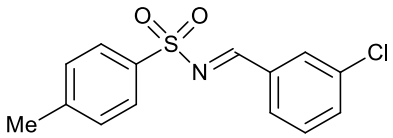
3g

***(E)*-4-methyl-*N*-(naphthalen-1-ylmethylene)benzenesulfonamide 3g.**[1] According to the general procedure, the product was isolated as white solid; m = 269 mg; 87% (mp = 133–135 °C); ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 9.61 (1H, s), 8.99 (1H, d, *J* = 8.7 Hz), 8.15 (1H, dd, *J* = 7.3 Hz, *J* = 1.2 Hz), 8.10 (1H, d, *J* = 8.2 Hz), 8.00–7.87 (3H, m), 7.72–7.63 (1H, m), 7.62–7.53 (2H, m), 7.36 (2H, d, *J* = 8.0 Hz), 2.43 (3H, s); ^{13}C NMR (75 MHz; $CDCl_3$) δ 169.9, 144.7, 136.3, 135.6, 135.3, 133.9, 132.0, 130.0, 129.2, 129.1, 128.2, 127.8, 127.1, 125.2, 124.4, 21.8.



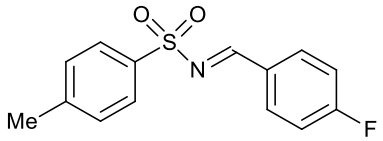
3h

***(E)-N*-(4-chlorobenzylidene)-4-methylbenzenesulfonamide 3h.**[2] According to the general procedure, the product was isolated as white solid; m = 264 mg; 90% (mp = 174–176 °C); ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 8.99 (1H, s), 7.88 (2H, d, *J* = 8.5 Hz), 7.86 (2H, d, *J* = 9.0 Hz) 7.46 (2H, d, *J* = 8.5 Hz), 7.35 (2H, d, *J* = 8.0 Hz), 2.44 (3H, s); ^{13}C NMR (75 MHz; $CDCl_3$) δ 168.8, 144.9, 141.6, 135.0, 132.5, 130.9, 130.0, 129.7, 128.3, 21.8.



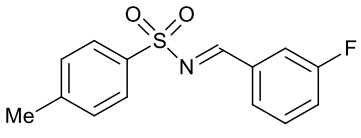
3i

***(E)-N*-(3-chlorobenzylidene)-4-methylbenzenesulfonamide 3i.**[3] According to the general procedure, the product was isolated as white solid ; m = 243 mg; 83% (mp = 80–82°C); ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 8.97 (1H, s), 7.92 (1H, t, *J* = 1.8 Hz), 7.88 (2H, d, *J* = 8.1), 7.77 (1H, dt, *J* = 7.7 Hz, *J* = 1.3 Hz), 7.56 (1H, ddd, *J* = 8.1 Hz, *J* = 2.1 Hz, *J* = 1.2), 7.43 (1H, t, *J* = 7.8 Hz), 7.35 (2H, d, J = 8.1 Hz), 2.44 (3H, s); ^{13}C NMR (75 MHz; $CDCl_3$) δ 168.7, 145.0, 135.5, 134.8, 134.1, 130.5, 130.3, 130.0, 129.9, 128.3, 126.6, 21.8.



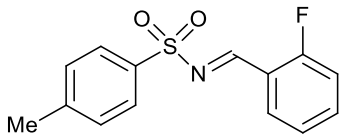
3j

***(E)-N*-(4-fluorobenzylidene)-4-methylbenzenesulfonamide 3j.**[1] According to the general procedure, the product was isolated as white solid ; m = 252 mg; 91% (mp = 109–111 °C); ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 8.99 (1H, s), 7.95 (2H, dd, *J* = 8.7 Hz, *J* = 5.4 Hz), 7.88 (2H, d, *J* = 8.4 Hz), 7.35 (2H, d, *J* = 8.1 Hz), 7.17 (2H, t, *J* = 8.6 Hz), 2.44 (3H, s); ^{13}C NMR (75 MHz; $CDCl_3$) δ 168.7, 167.0 (d, *J* = 257.0 Hz), 144.8, 135.2, 133.9 (d, *J* = 9.8 Hz), 130.0, 128.9 (d, *J* = 3.0 Hz), 128.2, 116.8 (2C, d, *J* = 22.5), 21.8.



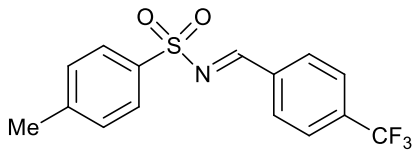
3k

***(E)-N*-(3-fluorobenzylidene)-4-methylbenzenesulfonamide 3k.**[1] According to the general procedure, the product was isolated as white solid ; m = 255 mg; 92% (mp = 82–84 °C); ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 9.00 (1H, d, *J* = 1.5 Hz), 7.88 (2H, d, *J* = 8.4 Hz), 7.71–7.61 (2H, m), 7.53–7.43 (1H, m), 7.35 (2H, d, *J* = 8.1 Hz), 7.32–7.27 (1H, m), 2.44 (3H, s); ^{13}C NMR (75 MHz; $CDCl_3$) δ 168.8 (d, *J* = 2.3 Hz), 163.0 (d, *J* = 247.7 Hz), 145.0, 134.9, 134.6 (d, *J* = 7.5 Hz), 130.9 (d, *J* = 8.3 Hz), 130.0, 128.3, 128.0 (d, *J* = 3.0 Hz), 122.1 (d, *J* = 21.8 Hz), 116.7 (d, *J* = 22.5 Hz), 21.8.



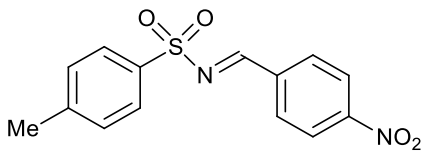
3l

***(E)*-*N*-(2-fluorobenzylidene)-4-methylbenzenesulfonamide 3l.**[1] According to the general procedure, the product was isolated as white solid ; m = 244 mg; 88% (mp = 135–137 °C); ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 9.35 (1H, s), 8.16–8.00 (1H, m), 7.89 (2H, d, *J* = 8.4 Hz), 7.68–7.53 (1H, m), 7.35 (2H, d, *J* = 8.1 Hz), 7.23 (1H, t, *J* = 7.7 Hz), 7.19–7.11 (1H, m), 2.44 (3H, s); ^{13}C NMR (75 MHz; $CDCl_3$) δ 164.4 (d, *J* = 258,8 Hz), 163.8 (d, *J* = 6.0 Hz), 144.9, 137.1 (d, *J* = 9.0 Hz), 134.9, 130.0, 129.5 (d, *J* = 0.8 Hz), 128.4, 125.0 (d, *J* = 3.8 Hz), 120.6 (d, *J* = 8.3 Hz), 116.5 (d, *J* = 21.0 Hz), 21.8.



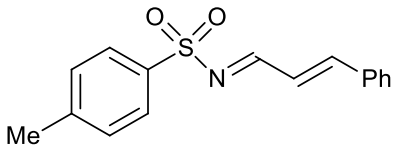
3m

***(E)*-4-methyl-*N*-(4-(trifluoromethyl)benzylidene)benzenesulfonamide 3m.**[1] According to the general procedure, the product was isolated as white solid ; m = 284 mg; 87% (mp = 154–156 °C); ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 9.07 (1H, s), 8.04 (2H, d, *J* =8.1 Hz), 7.90 (2H, d, *J* = 8.1 Hz), 7.74 (2H, d, *J* = 8.1 Hz), 7.36 (2H, d, *J* = 8.1 Hz), 2.44 (3H, s); ^{13}C NMR (75 MHz; $CDCl_3$) δ 168.5, 145.2, 135.9 (q, *J* = 33.0 Hz), 135.7, 134.9, 131.4, 130.1, 128.4, 126.2 (q, *J* = 3.8 Hz), 123.5 (q, *J* = 271.0 Hz), 21.7.



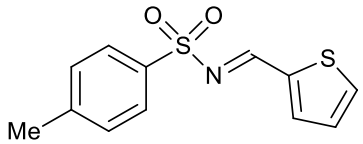
3n

***(E)*-4-methyl-*N*-(4-nitrobenzylidene)benzenesulfonamide 3n.**[2] According to the general procedure, the product was isolated as yellow solid; m = 188 mg; 62% (mp = 207–209 °C); ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 9.10 (1H, s), 8.33 (2H, d, *J* = 8.9 Hz), 8.11 (2H, d, *J* = 8.9 Hz), 7.90 (2H, d, *J* = 8.4 Hz), 7.38 (2H, d, *J* = 8.1 Hz), 2.46 (3H, s); ^{13}C NMR (75 MHz; $CDCl_3$) δ 167.4, 151.3, 145.5, 138.0, 134.3, 132.0, 130.2, 128.5, 124.3, 21.9.



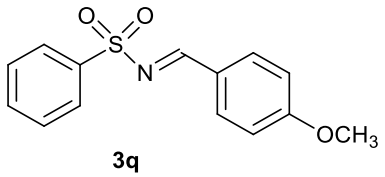
3o

***(E)*-4-methyl-*N*-(*(E)*-3-phenylallylidene)benzenesulfonamide 3o.**[2] According to the general procedure, the product was isolated as yellow solid ; m = 256 mg; 90% (mp = 110–112 °C); ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 8.77 (1H, d, *J* = 9.4 Hz), 7.86 (2H, d, *J* = 8.3 Hz), 7.60–7.53 (2H, m), 7.49 (1H, d, *J* = 15.9 Hz), 7.45–7.38 (3H, m), 7.34 (2H, d, *J* = 8.0 Hz), 6.98 (1H, dd, *J* = 15.8 Hz, *J* = 9.4 Hz), 2.43 (3H, s); ^{13}C NMR (75 MHz; $CDCl_3$) δ 171.0, 154.0, 144.6, 135.5, 134.3, 131.8, 129.9, 129.3, 128.8, 128.1, 124.9, 21.8.



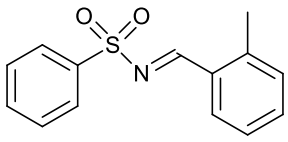
3p

***(E)*-4-methyl-*N*-(thiophen-2-ylmethylene)benzenesulfonamide 3p.**[1] According to the general procedure, the product was isolated as yellow solid; m = 244 mg; 92% (mp = 98–100 °C); ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 9.11 (1H, s), 7.86 (2H, d, *J* = 8.1 Hz), 7.77 (2H, d, *J* = 4.2 Hz), 7.33 (2H, d, *J* = 8.1 Hz), 7.20 (1H, t, *J* = 4.5 Hz), 2.43 (3H, s); ^{13}C NMR (75 MHz; $CDCl_3$) δ 162.4, 144.6, 139.2, 138.3, 136.8, 135.6, 129.9, 129.0, 128.1, 21.8.



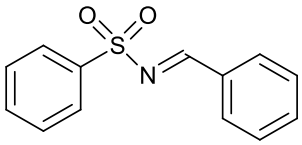
3q

***(E)*-*N*-(4-methoxybenzylidene)benzenesulfonamide 3q.**[2] According to the general procedure, the product was isolated as white solid; m = 256 mg; 93% (mp = 130–132 °C); ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 8.96 (1H, s), 8.10–7.94 (2H, m), 7.94–7.80 (2H, m), 7.72–7.57 (1H, m), 7.57–7.49 (2H, m), 7.07–6.85 (2H, m), 3.88 (3H, s); ^{13}C NMR (75 MHz; $CDCl_3$) δ 169.7, 165.4, 138.8, 133.9, 133.3, 129.1, 127.9, 125.2, 114.8, 55.7.



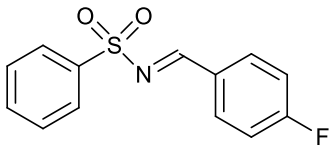
3r

***(E)-N*-(2-methylbenzylidene)benzenesulfonamide 3r.**[4] According to the general procedure, the product was isolated as white solid; m = 230 mg; 89% (mp = 77–79 °C); ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 9.36 (1H, s), 8.10–7.96 (3H, m), 7.67–7.44 (4H, m), 7.34–7.22 (2H, m), 2.61 (3H, s); ^{13}C NMR (75 MHz; $CDCl_3$) δ 169.3, 142.5, 138.5, 134.9, 133.6, 131.7, 130.8, 130.4, 129.3,128.0, 126.8, 19.8.



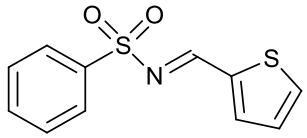
3s

***(E)-N*-benzylidenebenzenesulfonamide 3s.**[5] According to the general procedure, the product was isolated as white solid; m = 237 mg; 97% (mp = 77–79°C); ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 9.06 (1H, s), 8.12–7.97 (2H, m), 7.96–7.90 (2H, m), 7.66–7.46 (6H, m); ^{13}C NMR (75 MHz; $CDCl_3$) δ 170.8, 138.3, 135.2, 133.7, 132.4, 131.5, 129.3, 128.2.



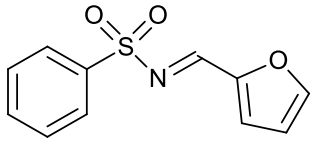
3t

***(E)-N*-(4-fluorobenzylidene)benzenesulfonamide 3t.**[5] According to the general procedure, the product was isolated as white solid; m = 216 mg; 82% (mp = 110–112 °C); ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 9.03 (1H, s), 8.10–7.93 (4H, m), 7.70–7.60 (1H, m), 7.59–7.51 (2H, m), 7.18 (2H, t, *J* = 8.5 Hz); ^{13}C NMR (75 MHz; $CDCl_3$) δ 169.2, 167.0 (d, *J* = 257.3 Hz), 138.2, 134.0 (d, *J* = 9.8 Hz), 132.8, 129.3, 128.8 (d, *J* = 2.8 Hz), 128.1, 116.6 (d, *J* = 22.1 Hz).



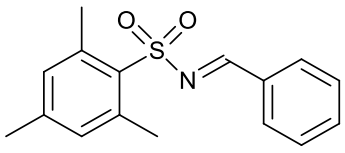
3u

***(E)-N*-(thiophen-2-ylmethylene)benzenesulfonamide 3u.** According to the general procedure, the product was isolated as white solid; m = 236 mg; 94% (mp = 130–132 °C); ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 9.14 (1H, br s), 8.03- 7.91 (2H, m), 7.79 (2H, d, *J* = 4.2 Hz), 7.66–7.58 (1H, m), 7.57–7.49 (2H, m), 7.21 (1H, t, *J* = 4.4 Hz); ^{13}C NMR (75 MHz; $CDCl_3$) δ 162.8, 139.4, 138.6, 138.2, 137.1, 133.6, 129.3, 129.1, 128.0; HRMS (TOF MS Cl^+) calculated for $C_{11}H_{10}NO_2S_2$ $[M+H]^+$: 252.0153, found: 252.0151.



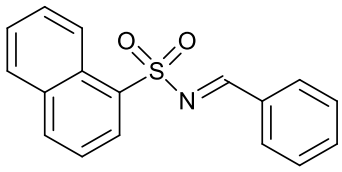
3v

***(E)-N*-(furan-2-ylmethylene)benzenesulfonamide 3v.**[5] According to the general procedure, the product was isolated as white solid; m = 212 mg; 90% (mp = 126–128 °C); ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 8.83 (1H, s), 8.06–7.95 (2H, m), 7.75 (1H, d, *J* = 1.5 Hz), 7.67–7.58 (1H, m), 7.57–7.48 (2H, m), 7.35 (1H, dd, *J* = 3,6 Hz, *J* = 0.6 Hz), 6.65 (1H, dd, *J* = 3,6 Hz, *J* = 1,5 Hz); ^{13}C NMR (75 MHz; $CDCl_3$) δ 156.2, 150.0, 149.3, 138.5, 133.6, 129.3, 128.1, 124.9, 113.9.



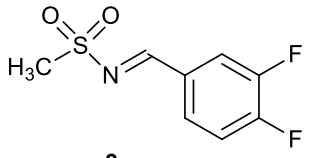
3w

***(E)-N*-benzylidene-2,4,6-trimethylbenzenesulfonamide 3w.** According to the general procedure, the product was isolated as white solid; m = 264 mg; 92% (mp = 87–89 °C); ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 9.03 (1H, s), 7.92 (2H, d, *J* = 7.2 Hz), 7.61 (1H, t, *J* = 7.4 Hz), 7.49 (2H, t, *J*= 7.5 Hz), 6.98 (2H, s), 2.71 (6H, s), 2.31 (3H, s); ^{13}C NMR (75 MHz; $CDCl_3$) δ 169.1, 143.4, 140.4, 134.8, 132.8, 132.3, 132.1, 131.3, 129.3, 23.2, 21.2; HRMS (TOF MS Cl^+) calculated for $C_{16}H_{18}NO_2S$ $[M+H]^+$: 288.1058, found: 288.1062.



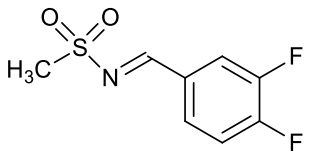
3x

***(E)-N*-benzylidenenaphthalene-1-sulfonamide 3x.**[6] According to the general procedure, the product was isolated as white solid; m = 215 mg; 73% (mp = 121–123 °C); ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 9.16(1H, s), 8.88 (1H, d, *J* = 8.4 Hz), 8.41 (1H, dd, *J* = 7.5 Hz, *J* = 1.1 Hz), 8.11 (1H, d, *J* = 8.1 Hz), 8.00–7.82 (3H, m), 7.77–7.67 (1H, m), 7.65–7.52 (3H, m), 7.50–7.38 (2H, m); ^{13}C NMR (75 MHz; $CDCl_3$) δ 170.8, 135.3, 135.2, 134.3, 133.8, 132.5, 131.5, 129.7, 129.3, 129.2, 129.0, 128.5, 127.1, 125.6, 124.4.



3ya

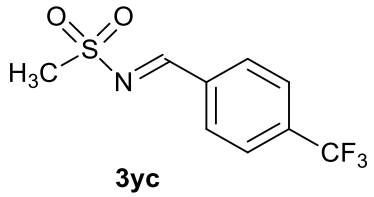
***(E)-N*-(4-fluorobenzylidene)methanesulfonamide 3ya.** According to the general procedure, the product was isolated as white solid; m = 177 mg; 88% (mp = 109–111 °C); ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 8.99 (1H, s), 7.99 (2H, dd, *J* = 8.8 Hz, *J* = 5.4 Hz), 7.22 (2H, t, *J* = 8.6 Hz), 3.13 (3H, s); ^{13}C NMR (75 MHz; $CDCl_3$) δ 170.2, 167.1 (d, *J* = 257.3 Hz), 140.0 (d, *J* = 9.8 Hz), 128.6 (d, *J* = 3.0 Hz), 116.9 (d, *J* = 22.5 Hz), 40.4; ^{19}F NMR (282 MHz; $CDCl_3$) δ –100.54; HRMS (TOF MS Cl^+) calculated for $C_8H_9FNO_2S$ $[M+H]^+$: 202.0338, found: 202.0336.



3yb

***(E)-N*-(3,4-difluorobenzylidene)methanesulfonamide 3yb.** According to the general procedure, the product was isolated as white solid; m = 164 mg; 75% (mp = 129–131 °C); ^{1}H NMR (300 MHz; $CDCl_3$) δ_H 8.96 (1H, s), 7.96–7.80 (1H, m), 7.79–7.58 (1H, m), 7.42–7.28 (1H, m), 3.14 (3H, s); ^{13}C NMR (75 MHz; $CDCl_3$) δ 169.4 (d, *J* = 0.8 Hz), 155.0 (dd, *J* = 259.2 Hz, *J* = 12.8 Hz), 151.1 (dd, *J* =251.1 Hz, *J* = 13.3 Hz), 129.4 (dd, *J* = 7.8 Hz, *J* = 3.3 Hz), 119.0 (dd, *J* = 18.2 Hz, *J* = 2.0 Hz), 118.7, 118.4, 40.4; ^{19}F NMR (282 MHz; $CDCl_3$) δ –125.03 (d, *J* = 20.7

Hz), –134.32 (d, *J* = 20.7 Hz); HRMS (TOF MS Cl^+) calculated for $C_8H_8F_2NO_2S$ $[M+H]^+$: 220.0244, found: 220.0240.

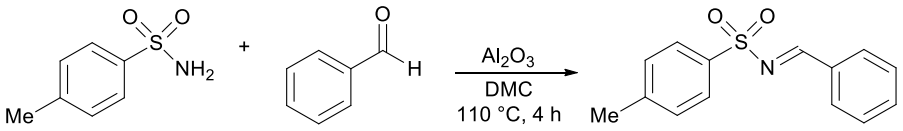


3yc

***(E)-N*-(4-(trifluoromethyl)benzylidene)methanesulfonamide yc.** According to the general procedure, the product was isolated as white solid; m = 208 mg; 83% (mp = 88–90 °C); ^1H NMR (300 MHz; $CDCl_3$) δ_H 9.09 (1H, s), 8.09 (2H, d, *J* = 8.1 Hz), 7.79 (2H, d, *J* = 8.2 Hz), 3.17 (3H, s); ^{13}C NMR (75 MHz; $CDCl_3$) δ 170.3, 136.2 (1C, q, *J* = 33.0 Hz), 135.2, 131.5, 126.3 (2C, q, *J* = 3.8 Hz), 123.4 (1C, q, *J* = 271.5 Hz), 40.3; ^{19}F NMR (282 MHz; $CDCl_3$) δ –63.32; HRMS (TOF MS Cl^+) calculated for $C_9H_9F_3NO_2S$ $[M+H]^+$: 252.0306, found: 252.0308.

Recycling and reuse of alumina

We have found that the use of Al_2O_3 for 2nd, 3rd, 4th, and 5th runs have shown the same efficiency as when it was used for the first time. Recovered Al_2O_3 after the filtration of the reaction crude was activated at 200 °C under reduced pressure (2 mbar) overnight and reused in another reaction. We found that, in all cases, the conversion to imine was complete (100%), and the isolated yield was always between 92 and 96% (product purified by recrystallization from ethanol–water).



3f

	Conversion (%)	Isolated yield (%)
Run 1	100	96
Run 2	100	92
Run 3	100	95
Run 4	100	96
Run 5	100	96

References

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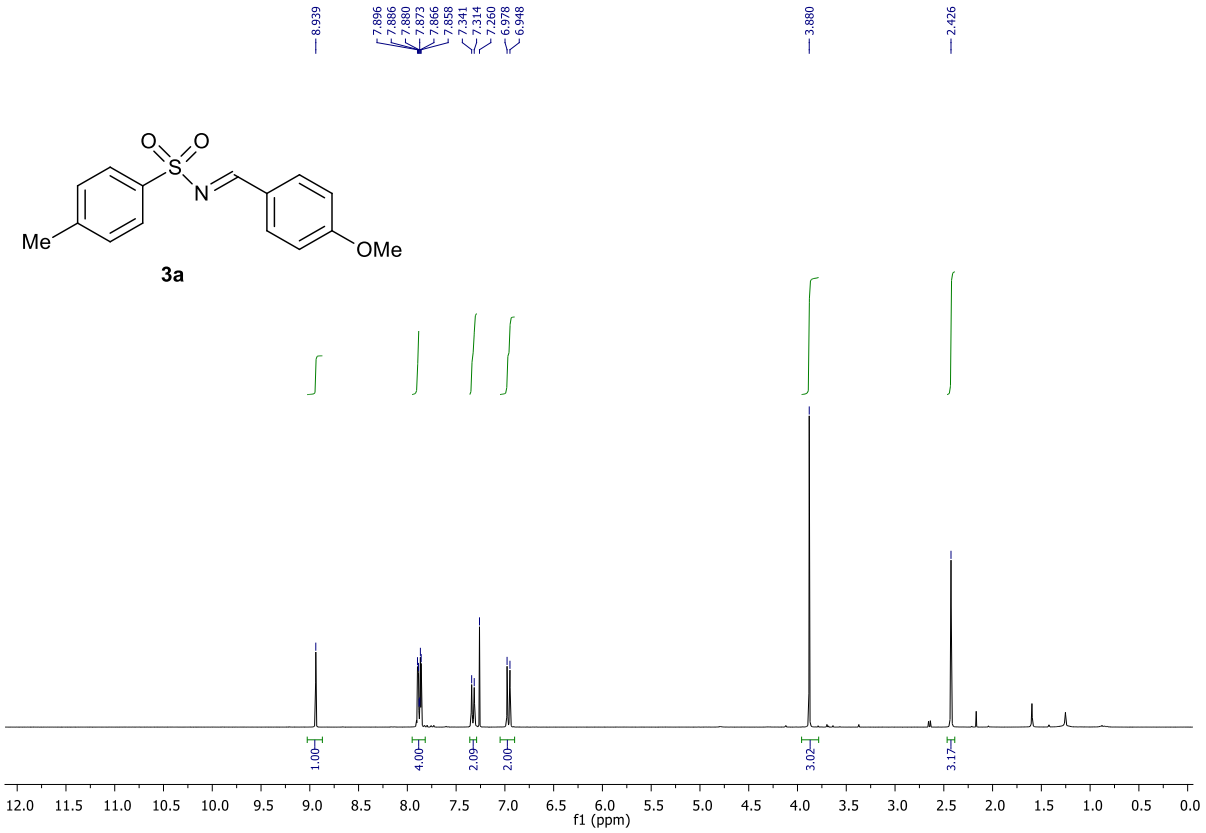
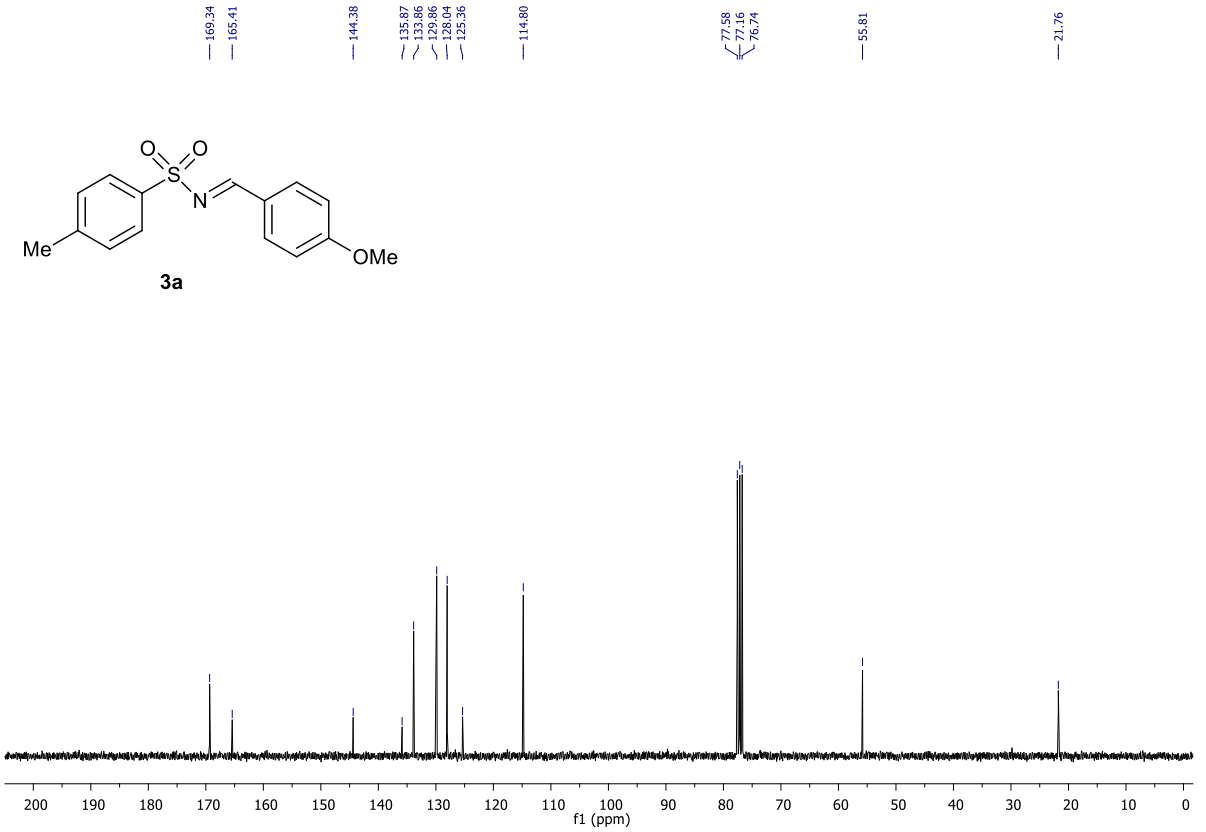
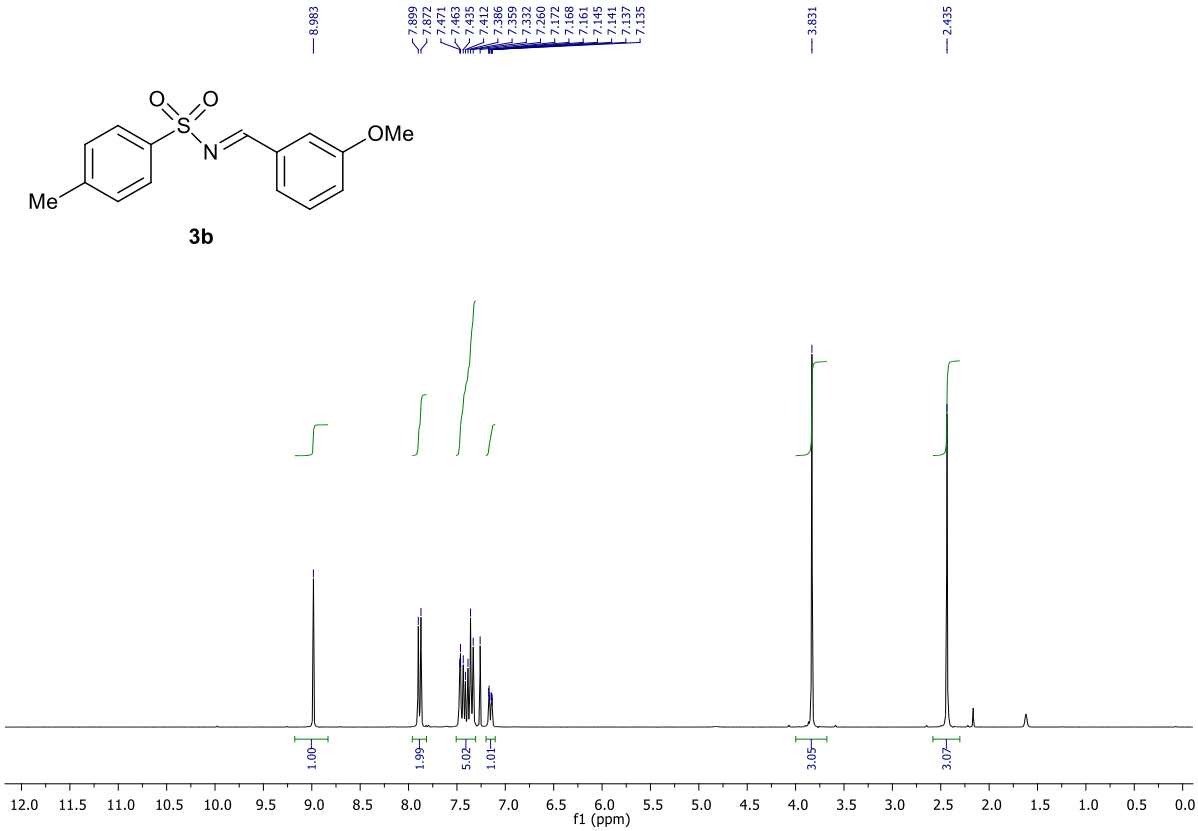
Copies of NMR spectra



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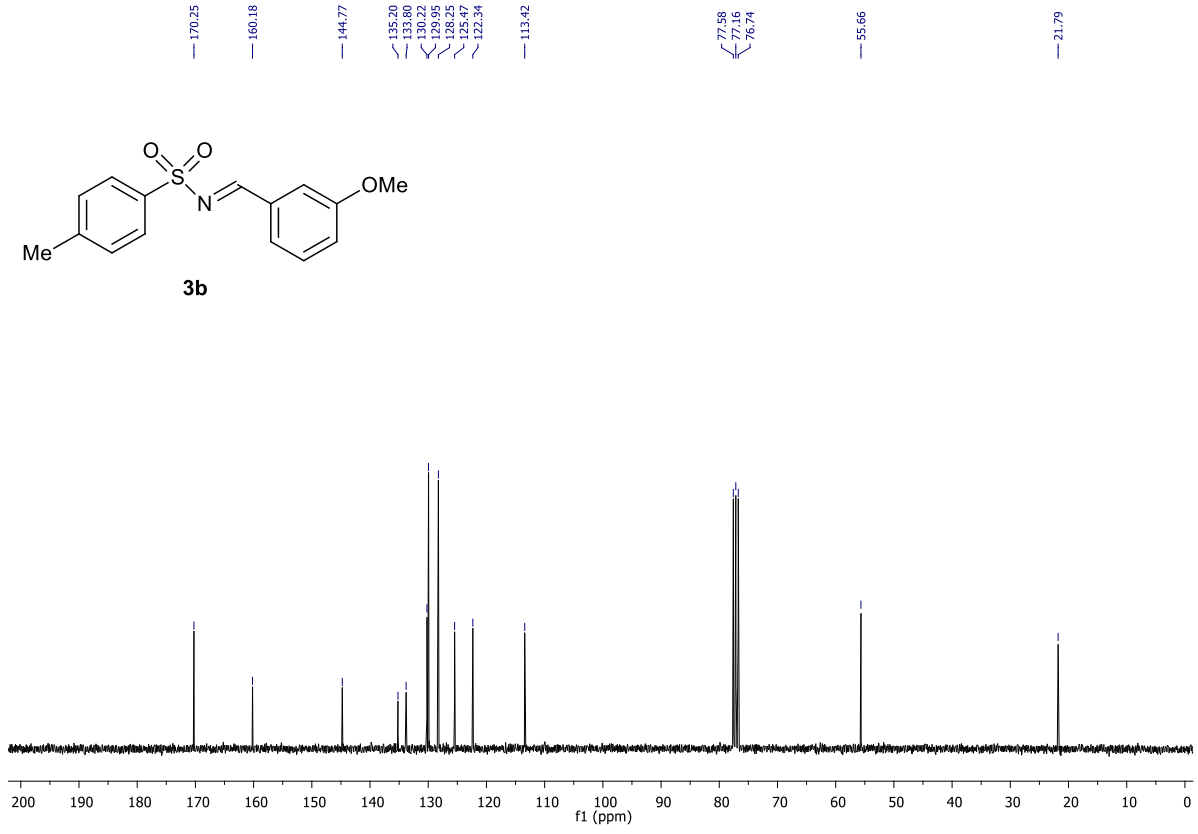
7.172
7.168
7.161
7.145
7.141
7.137
7.135
3.831
2.435
O
O
S
N
OMe
Me
1.00
1.99
5.02
1.01
3.05
3.07
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

3b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76.74
55.66
21.79
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O
S
N
OMe
Me
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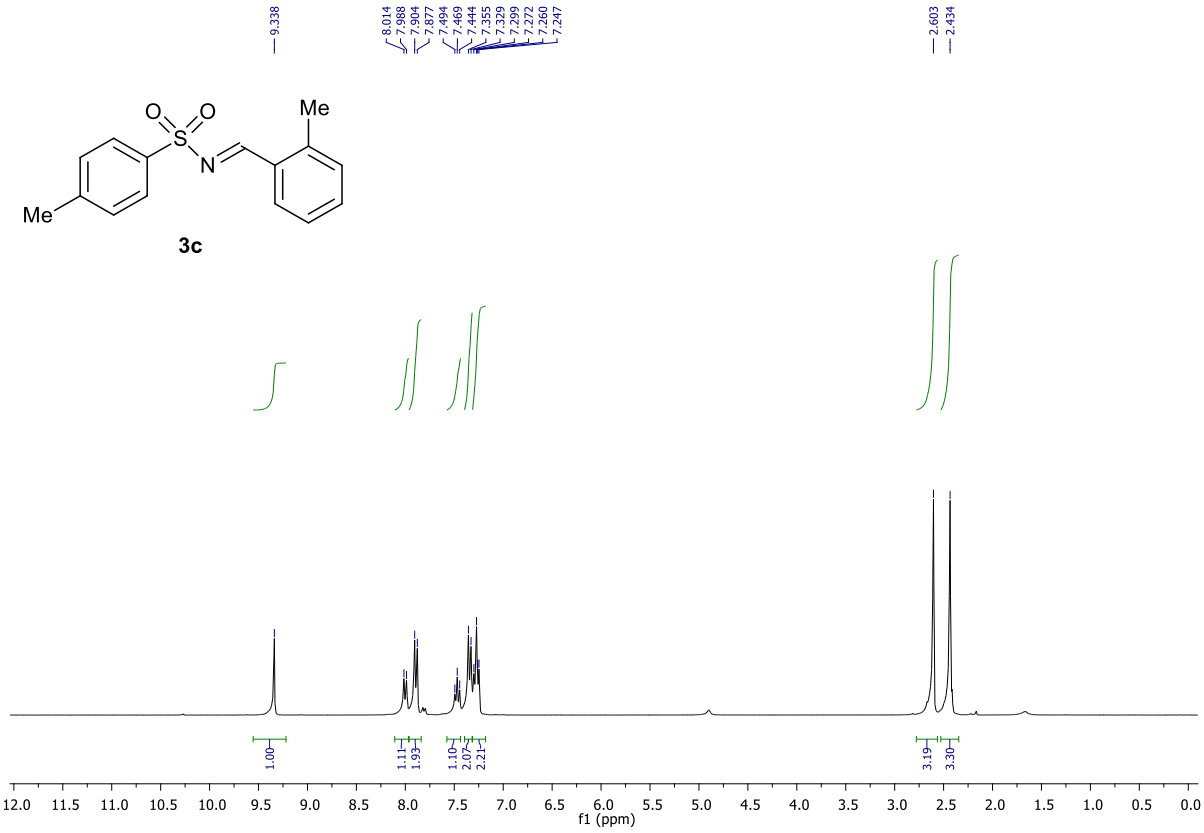
f1 (ppm)

3b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7.260
7.247
2.603
2.434
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1.10
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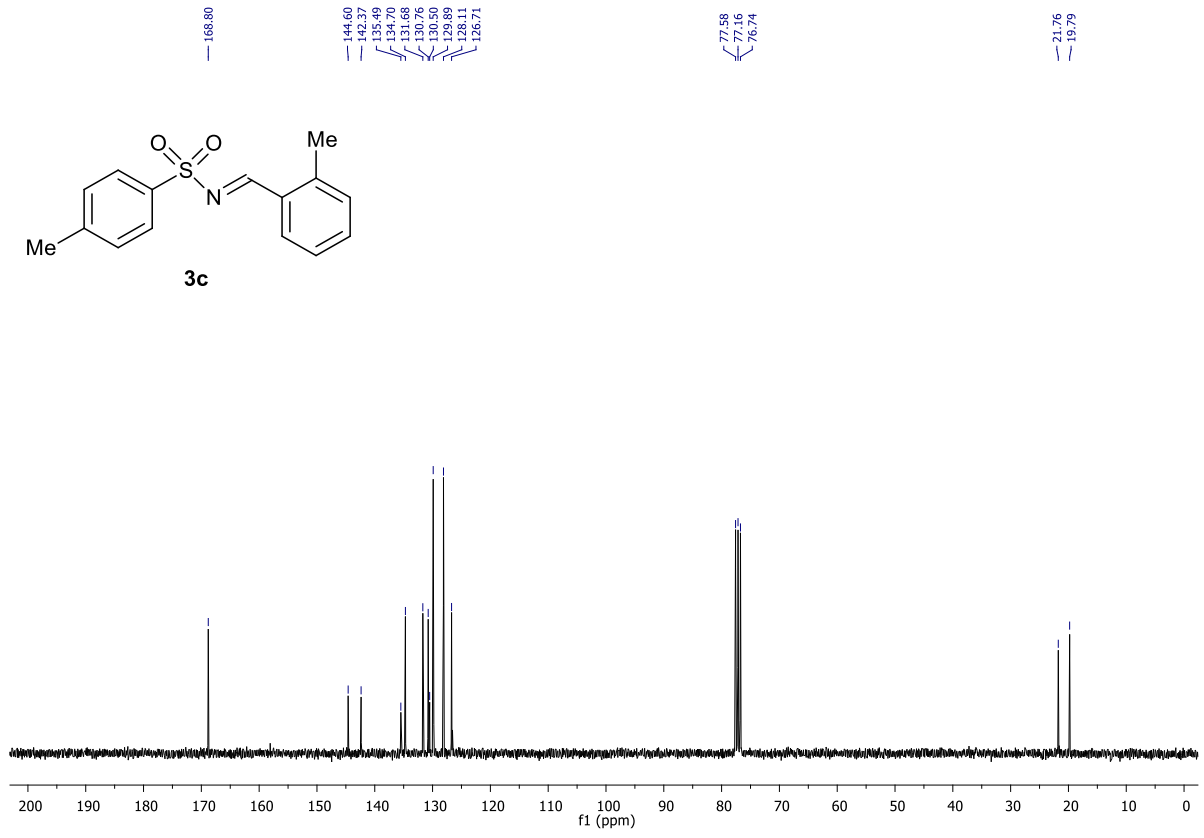
3.19
3.30
f1 (ppm)

3c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128.11
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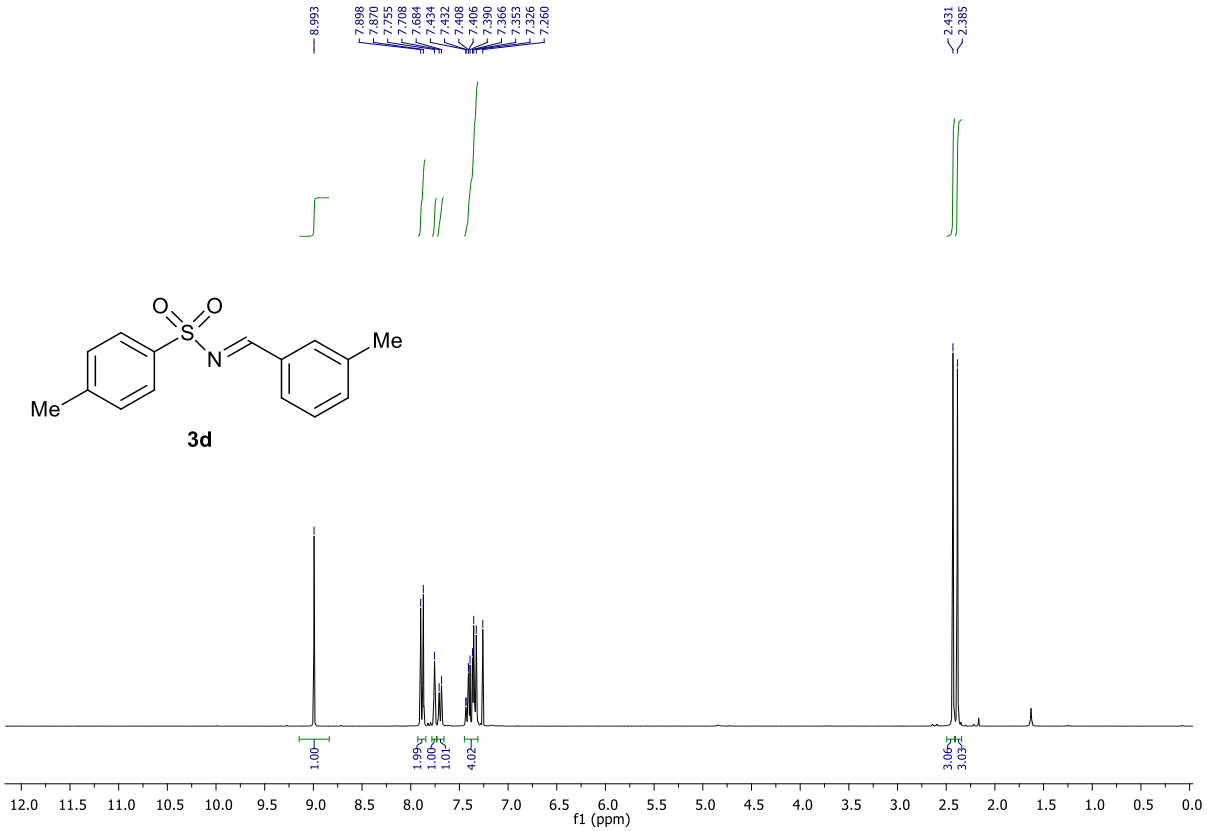
O
S
N
Me
Me
f1 (ppm)

3c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O
O
S
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Me
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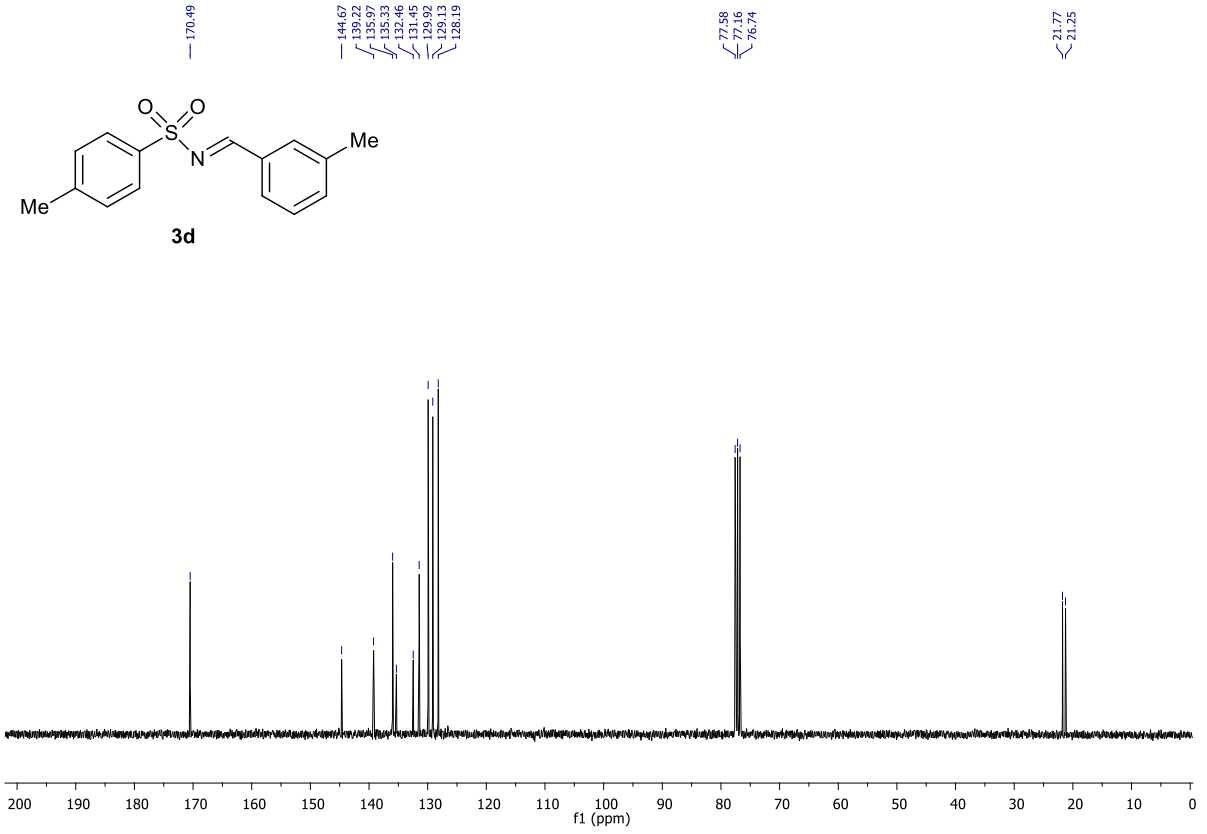
1.99
1.00
1.01
4.02
3.06
3.03
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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Me
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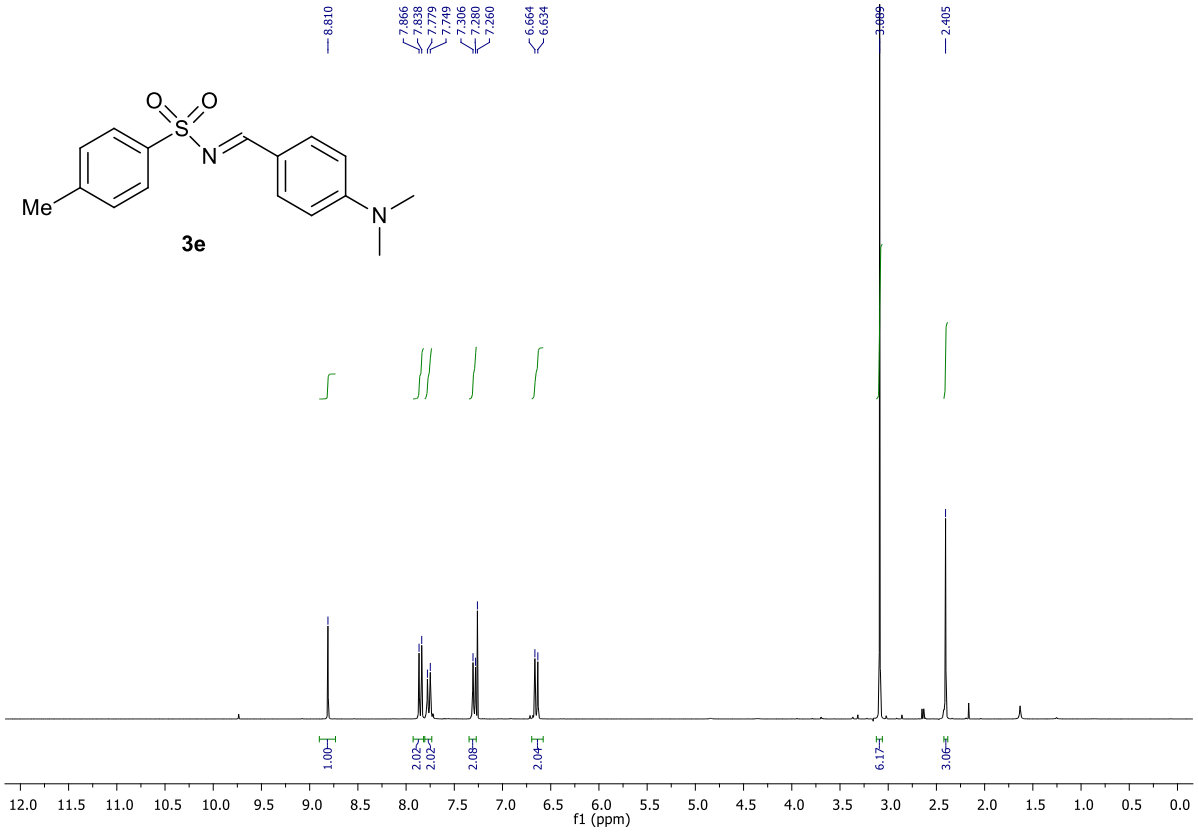
f1 (ppm)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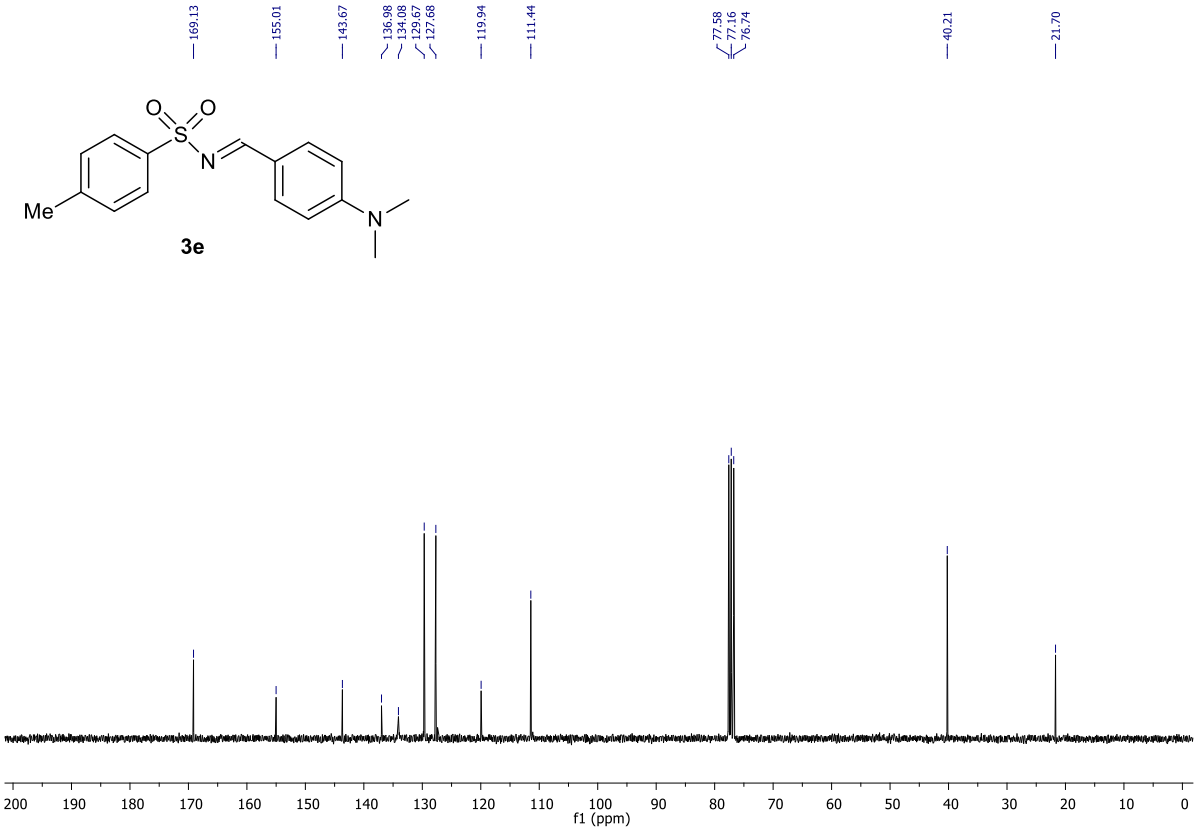
2.04
6.17
3.06
f1 (ppm)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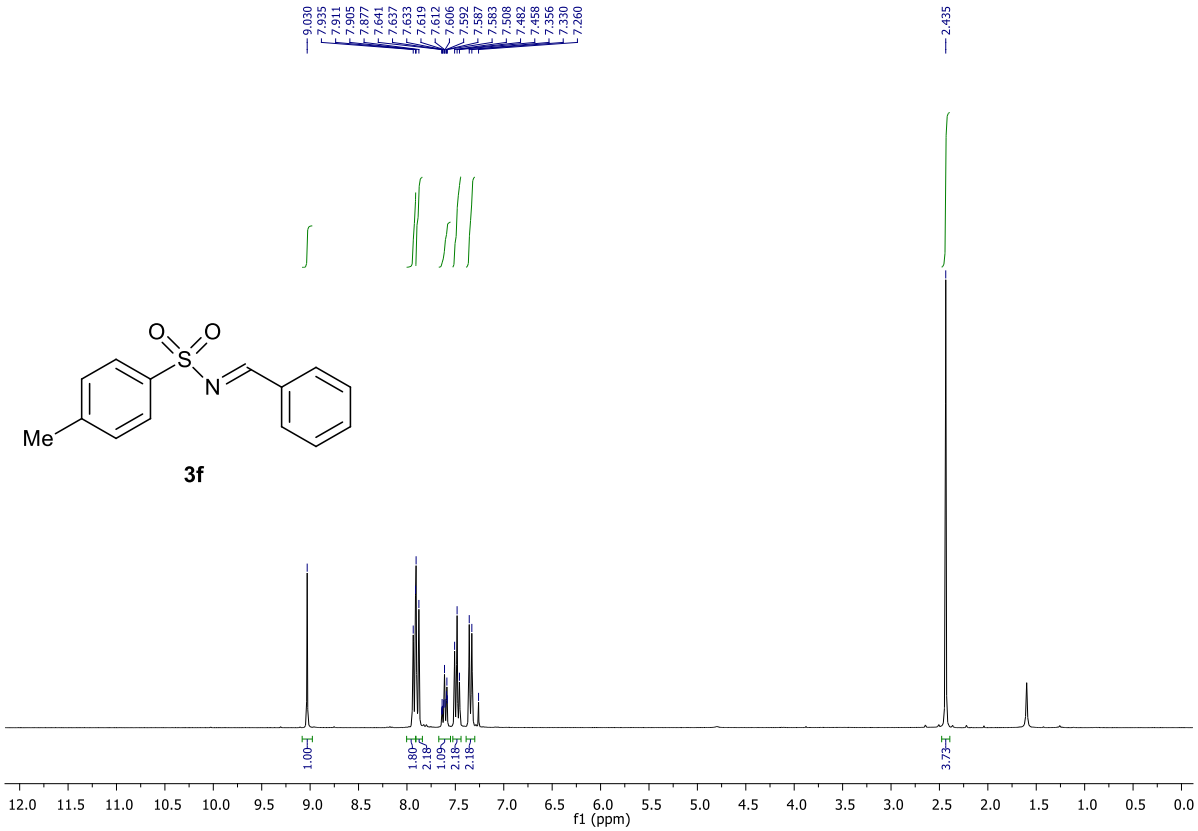
f1 (ppm)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7.592
7.587
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7.458
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7.330
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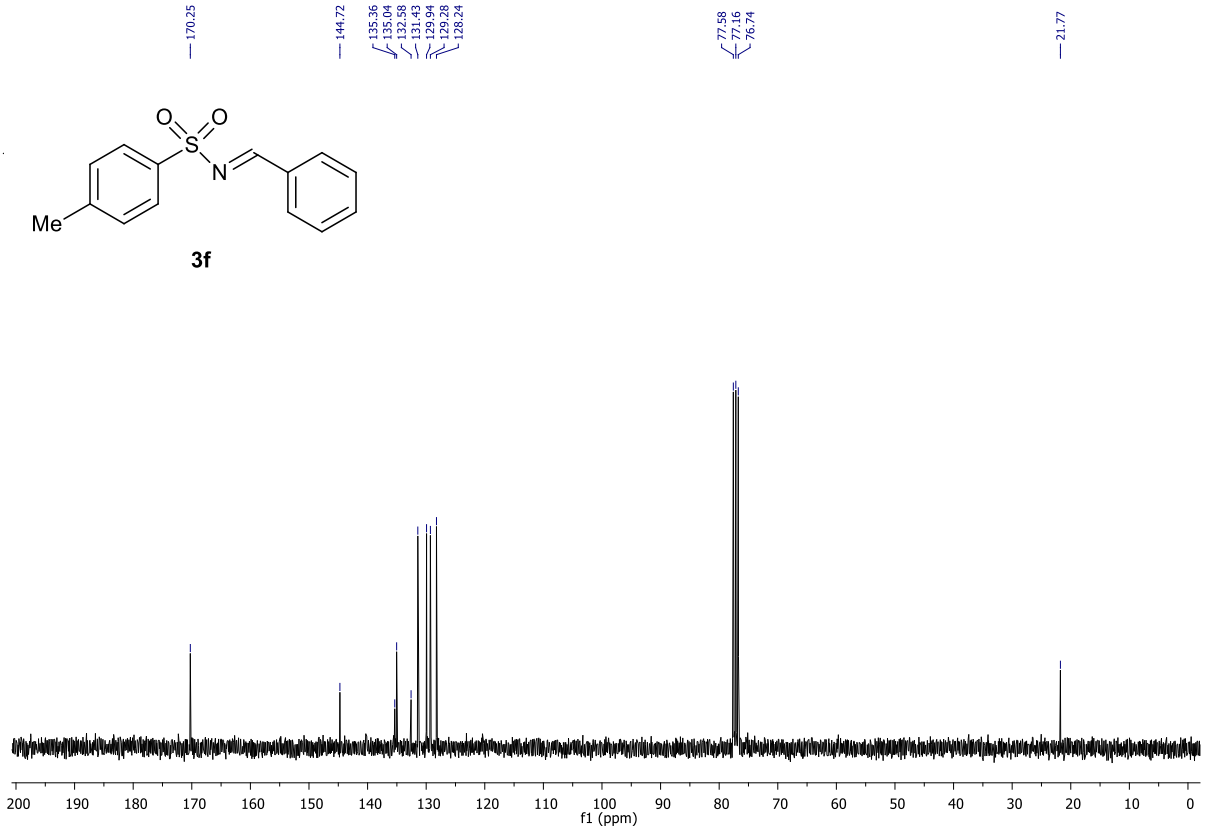
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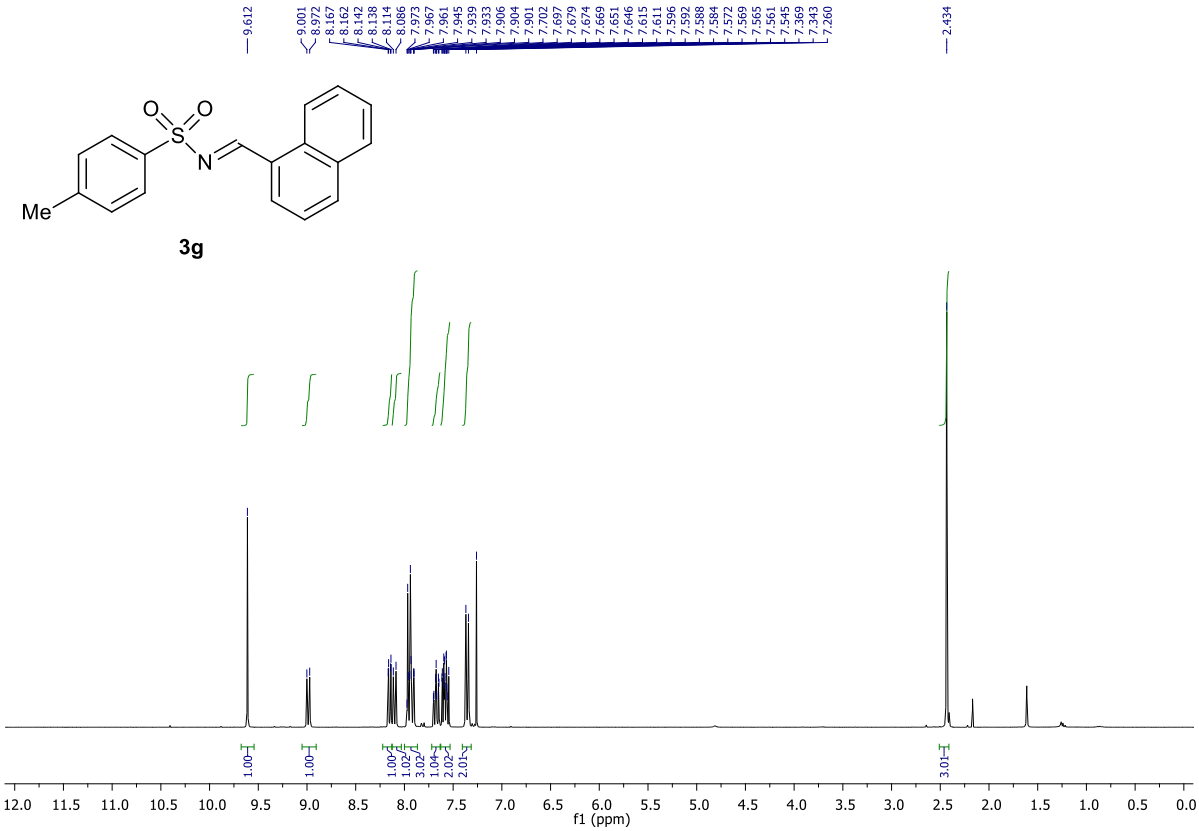
f1 (ppm)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2.01
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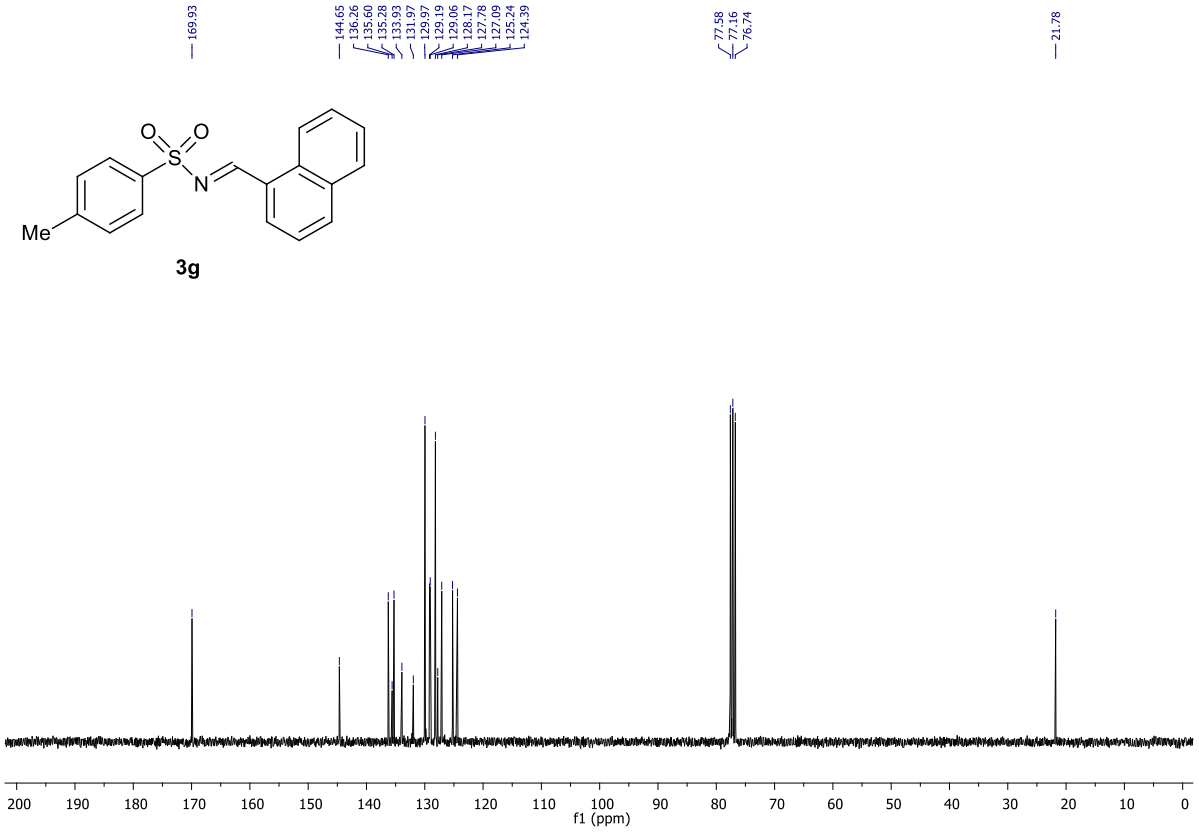
f1 (ppm)

3g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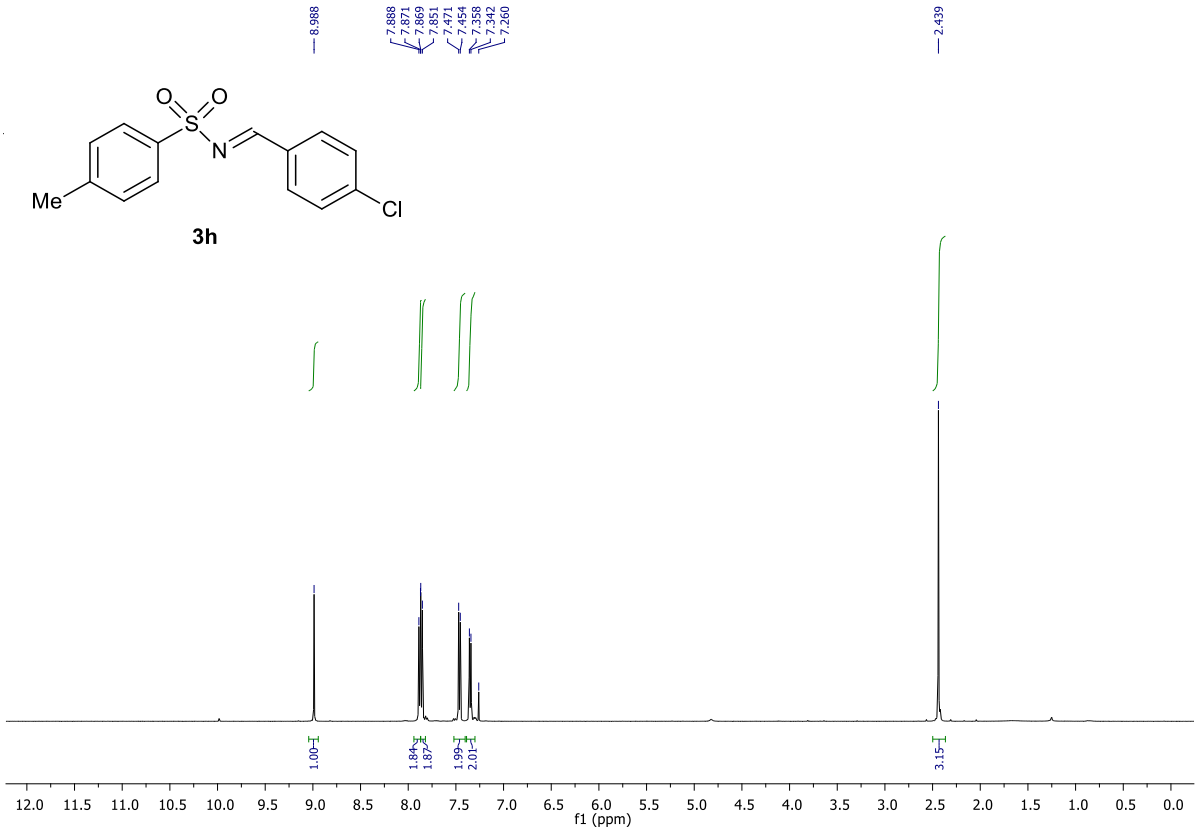
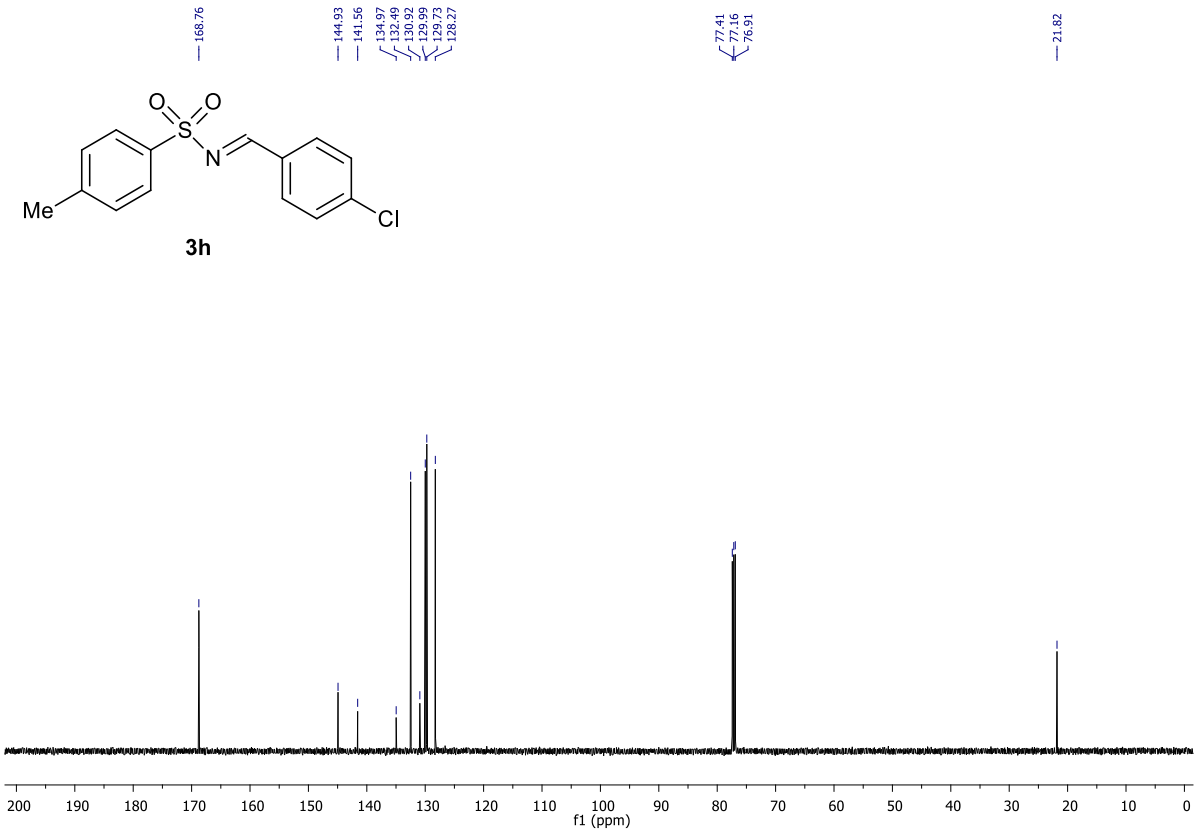
77.16
76.74
21.78
O
O
S
N
Me
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

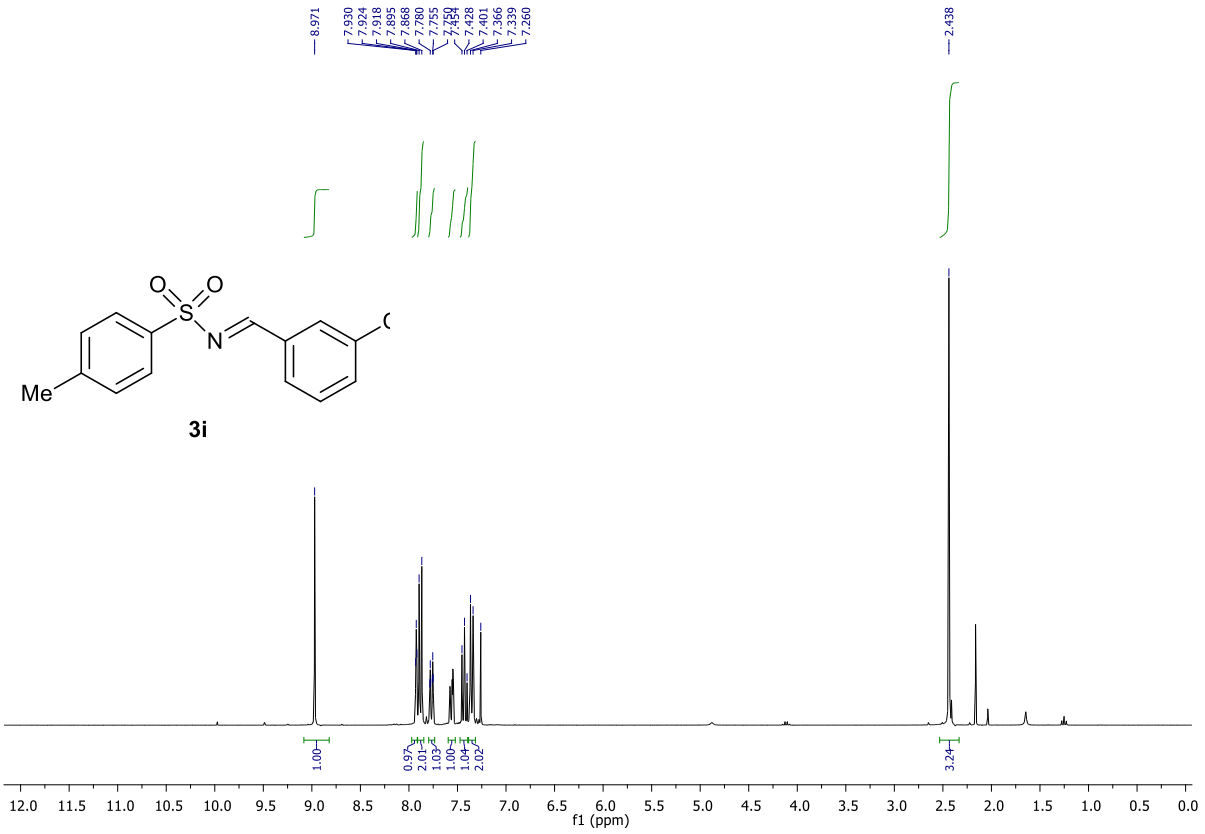
3g


8.988
7.888
7.871
7.869
7.851
7.471
7.454
7.358
7.342
7.260
2.439
O
O
S
N
Me
Cl
1.00
1.84
1.87
1.99
2.01
3.15
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

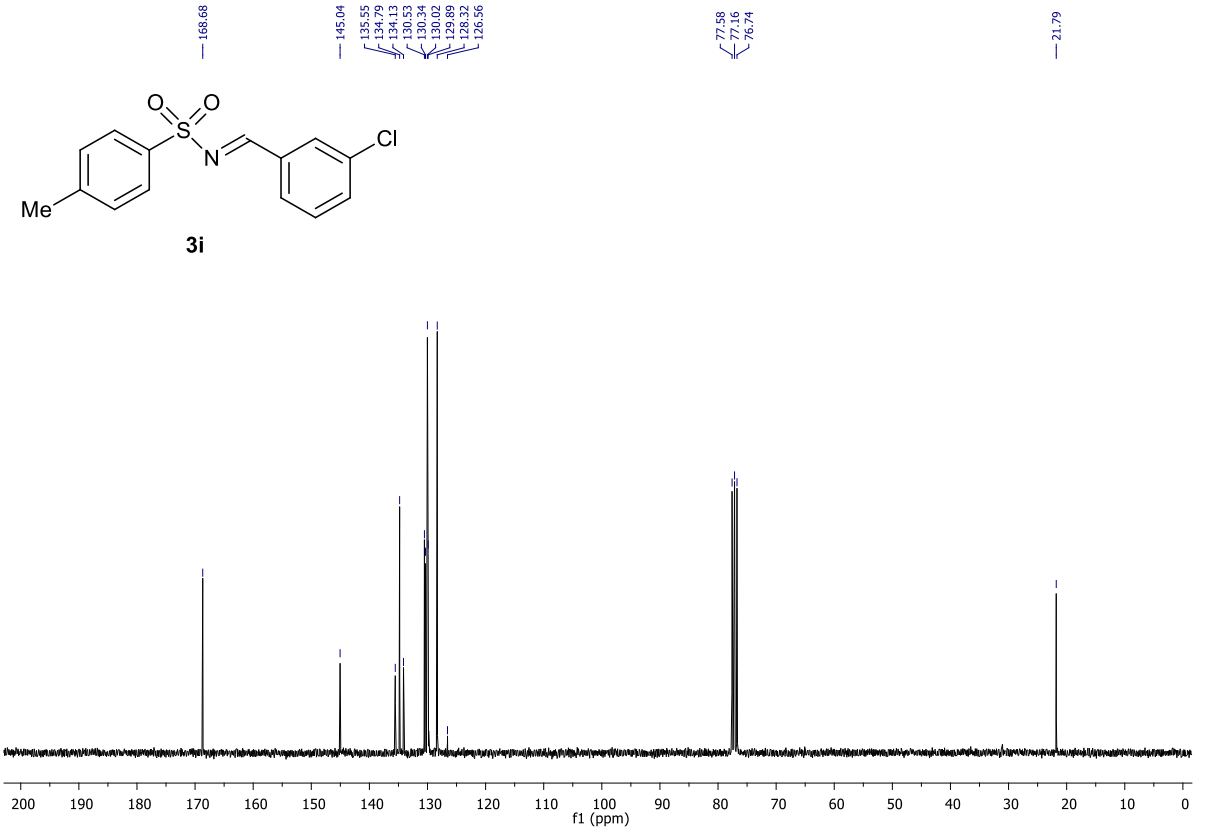
3h


168.76
144.93
141.56
134.97
132.49
130.92
129.99
129.73
128.27
77.41
77.16
76.91
21.82
O
O
S
N
Me
Cl
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

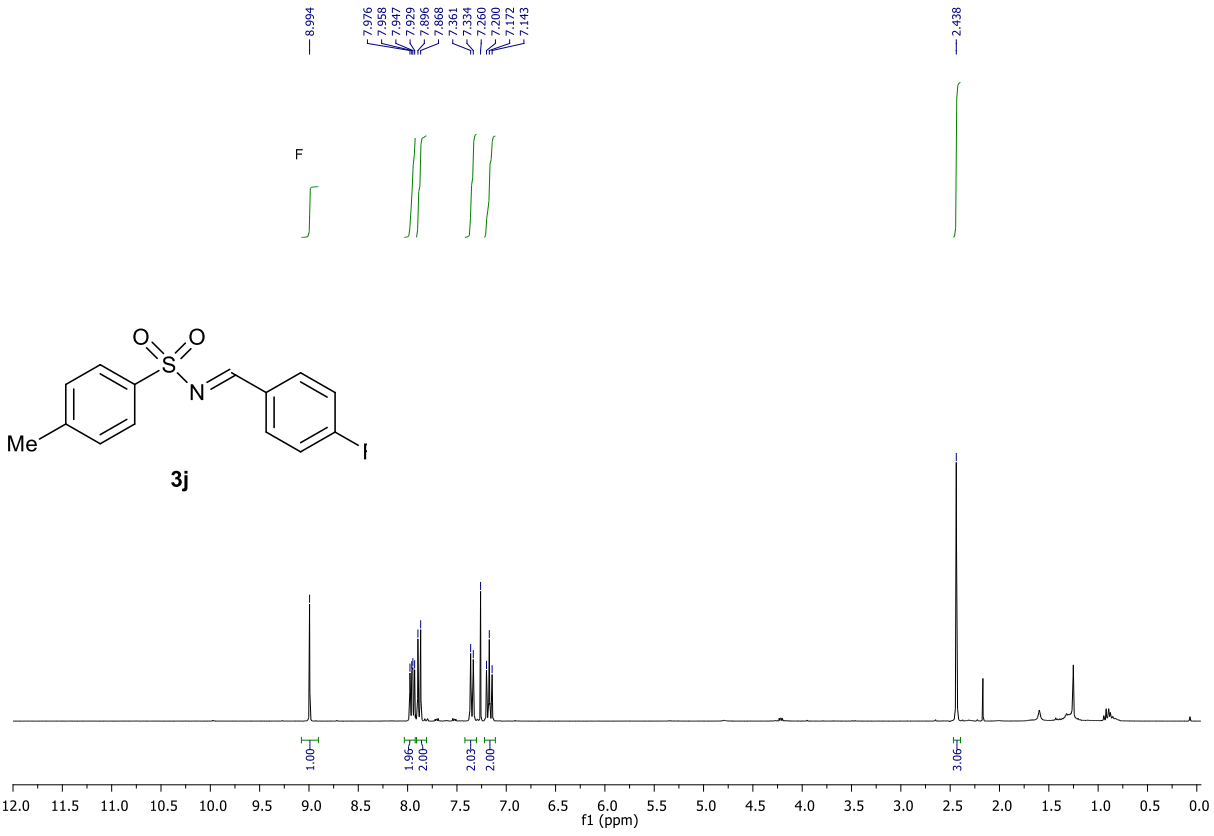
3h

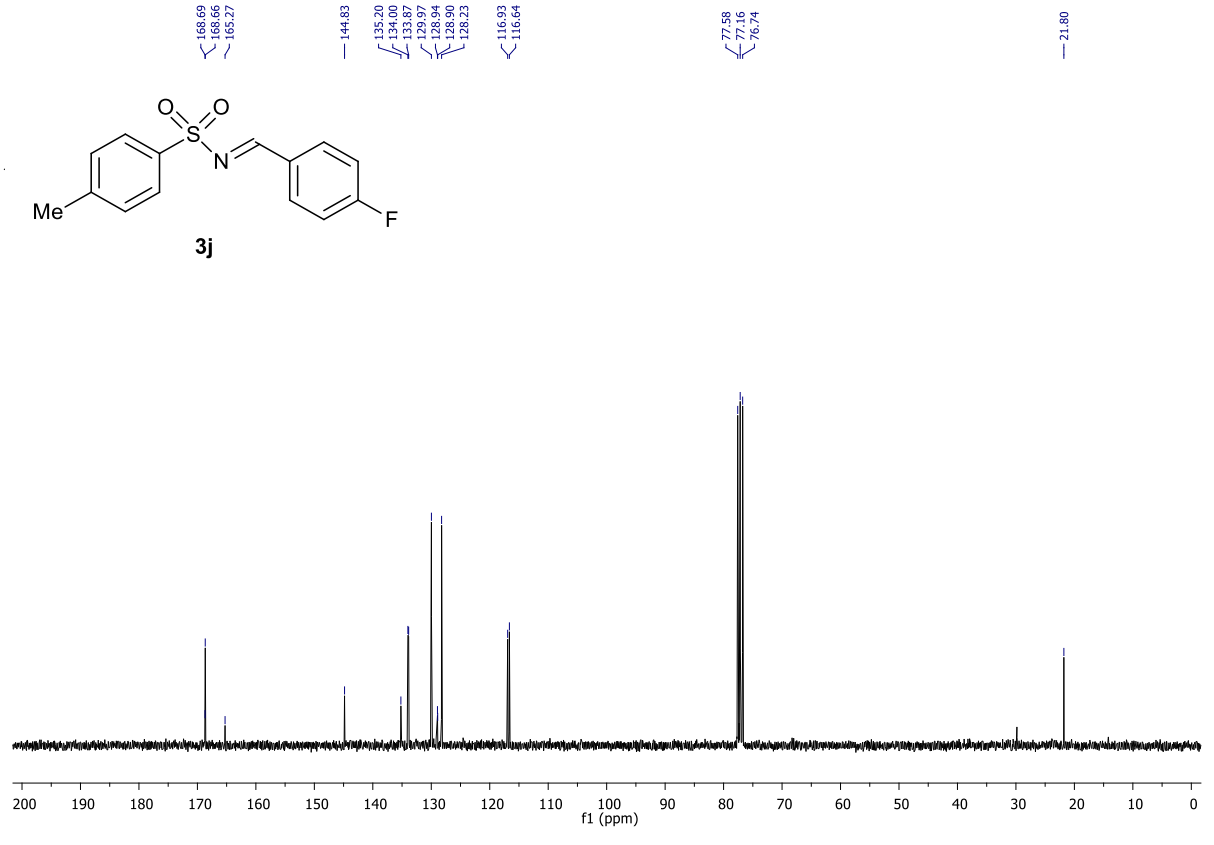

8.971
7.930
7.924
7.918
7.895
7.868
7.780
7.755
7.750
7.454
7.428
7.401
7.366
7.339
7.260
2.438
O
O
S
N
Me
1.00
0.97
2.01
1.03
1.00
1.04
2.02
3.24
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

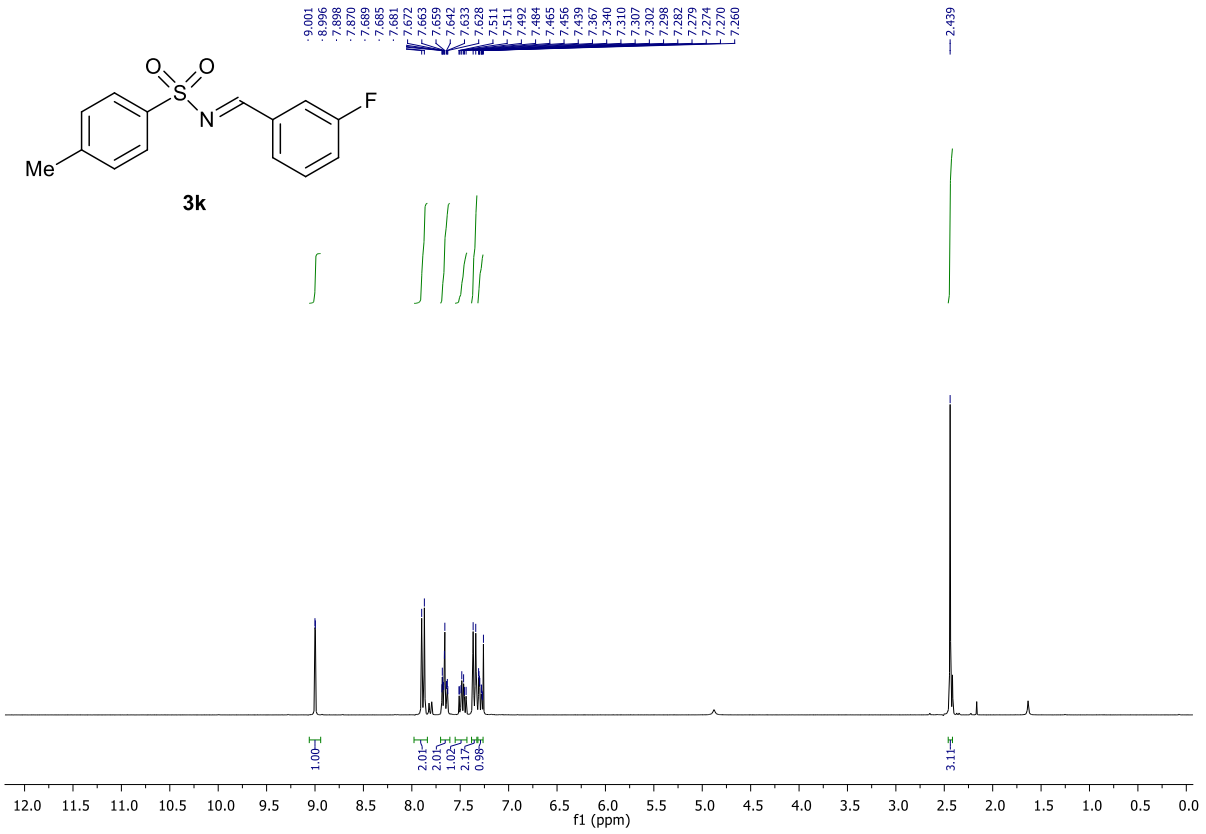
3i

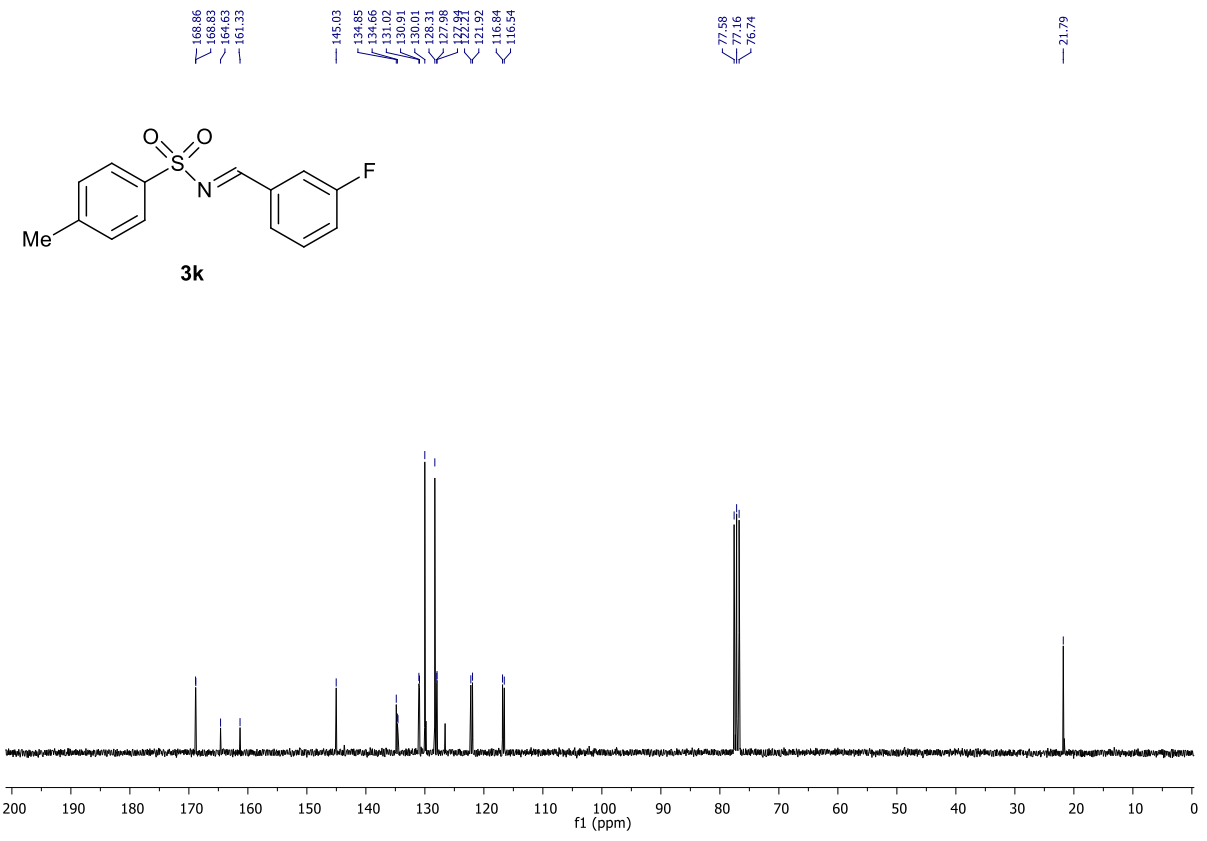

168.68
145.04
135.55
134.79
134.13
130.53
130.34
130.02
129.89
128.32
126.56
77.58
77.16
76.74
21.79
O
O
S
N
Cl
Me
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

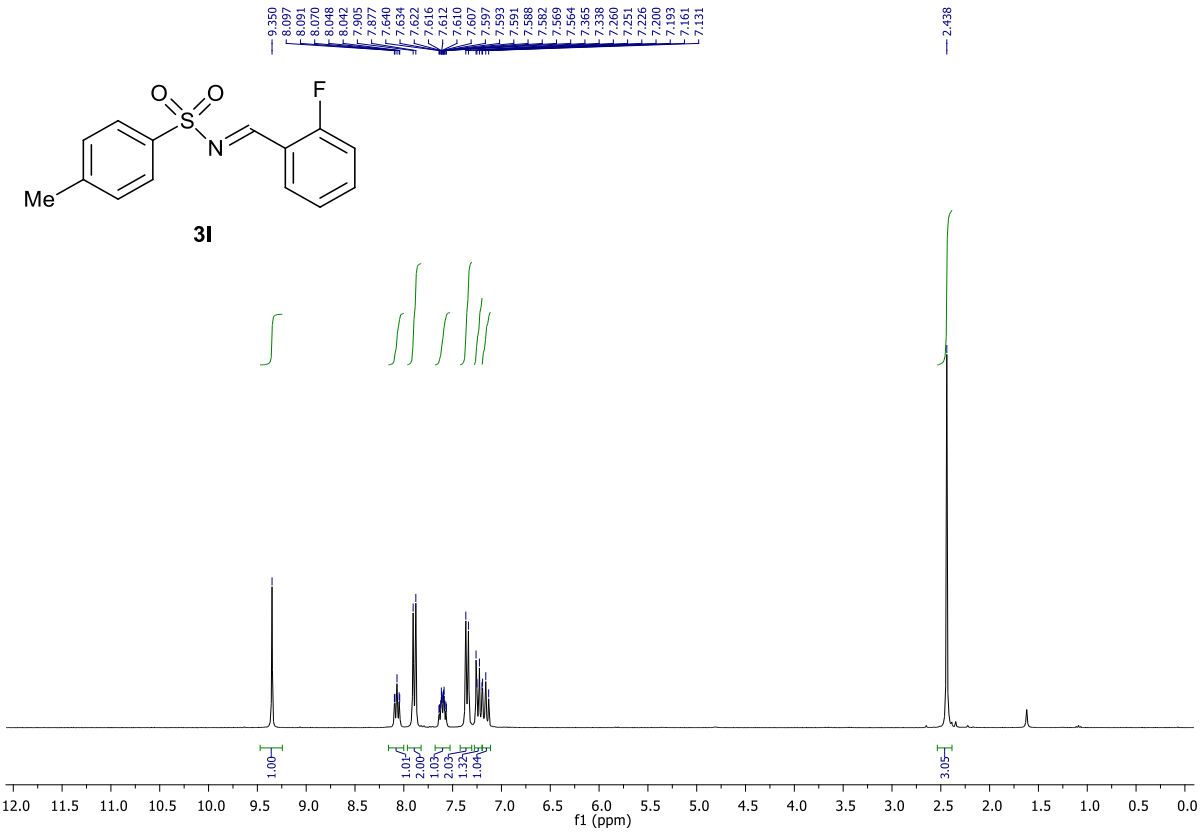
3i


8.994
7.976
7.958
7.947
7.929
7.896
7.868
7.361
7.334
7.260
7.200
7.172
7.143
2.438
F
Me
3j
1.00
1.96
2.00
2.03
2.00
3.06
f1 (ppm)

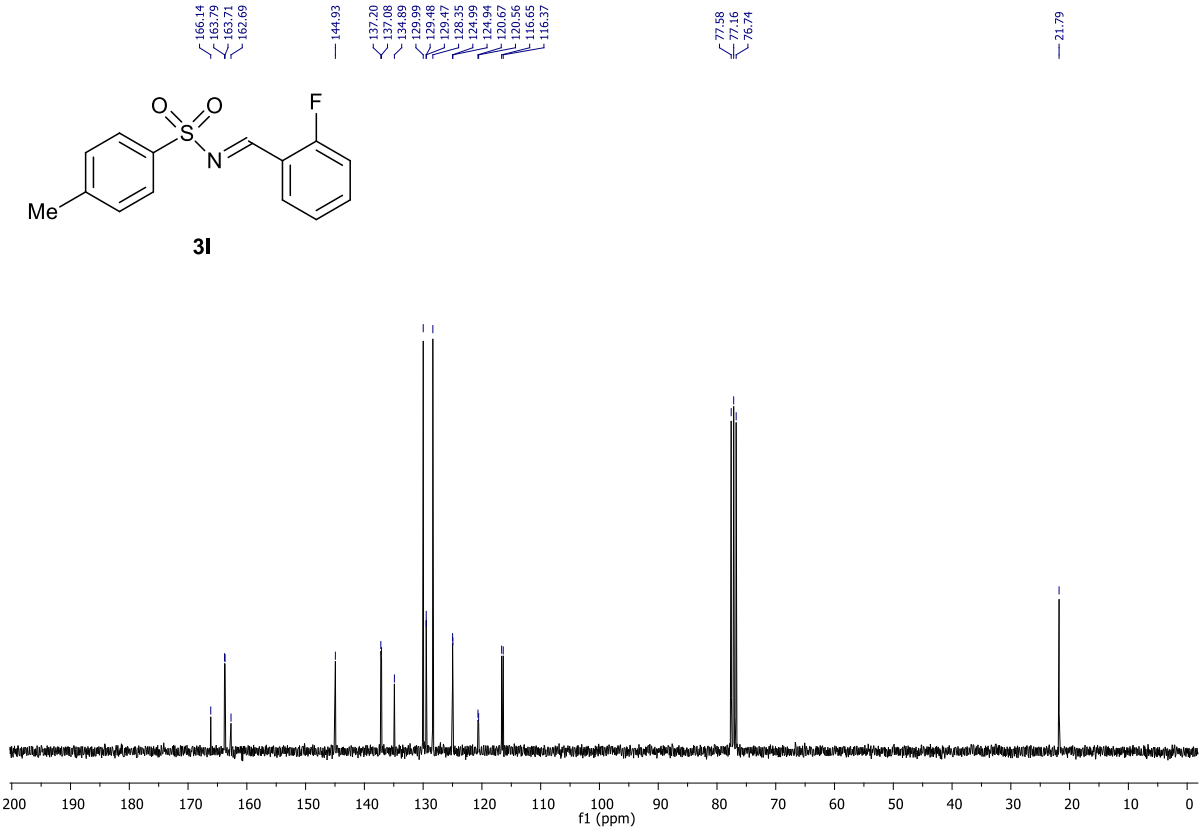

168.69
168.66
165.27
144.83
135.20
134.00
133.87
129.97
128.94
128.90
128.23
116.93
116.64
77.58
77.16
76.74
21.80
Me
F
3j
f1 (ppm)


9.001
8.996
7.898
7.870
7.689
7.685
7.681
7.672
7.663
7.659
7.642
7.633
7.628
7.511
7.511
7.492
7.484
7.465
7.456
7.439
7.367
7.340
7.310
7.307
7.302
7.298
7.282
7.279
7.274
7.270
7.260
2.439
O
O
S
N
F
Me
3k
1.00
2.01
2.01
1.02
2.17
0.98
3.11
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

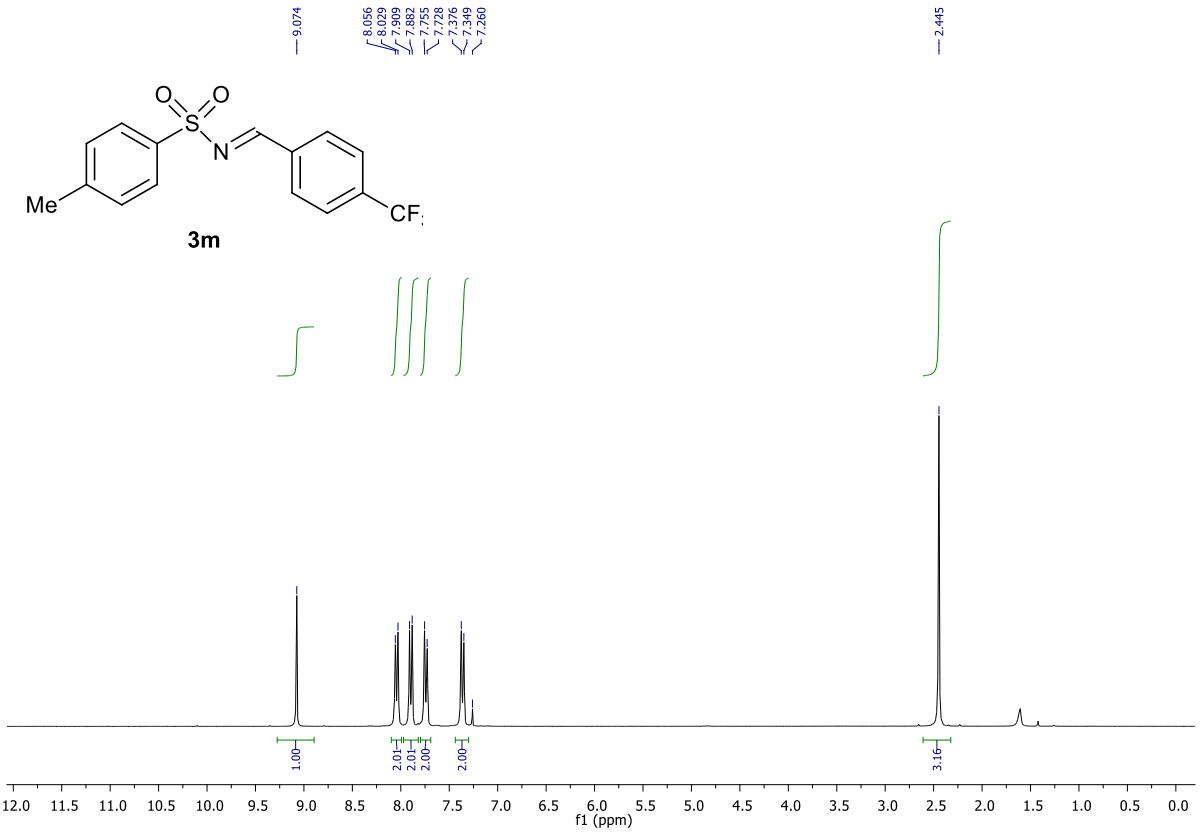

168.86
168.83
164.63
161.33
145.03
134.85
134.66
131.02
130.91
130.01
128.31
127.98
122.94
122.21
121.92
116.84
116.54
77.58
77.16
76.74
21.79
O
O
S
N
F
Me
3k
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

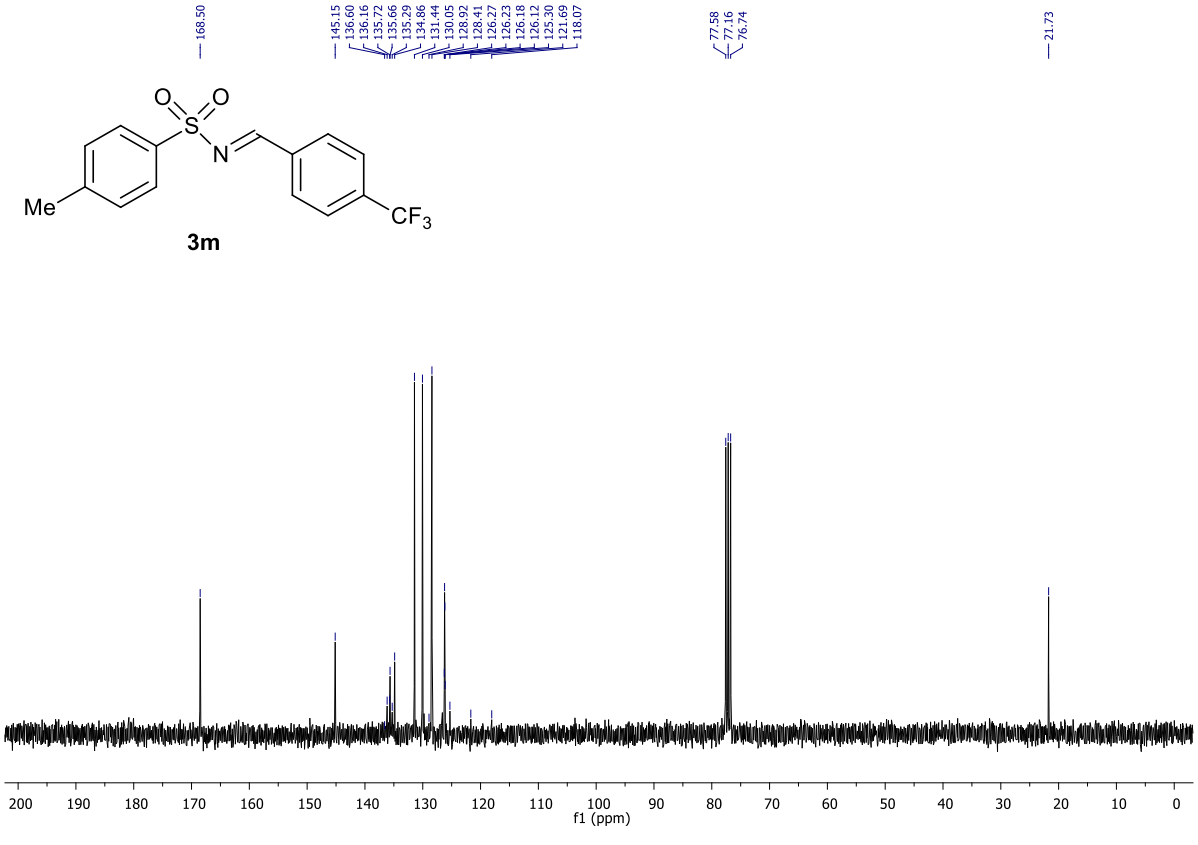

9.350
8.097
8.091
8.070
8.048
8.042
7.905
7.877
7.640
7.634
7.622
7.616
7.612
7.610
7.607
7.597
7.593
7.591
7.588
7.582
7.569
7.564
7.365
7.338
7.260
7.251
7.226
7.200
7.193
7.161
7.131
2.438
O
O
S
N
F
Me
1.00
1.01
2.00
1.03
2.03
1.32
1.04
3.05
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

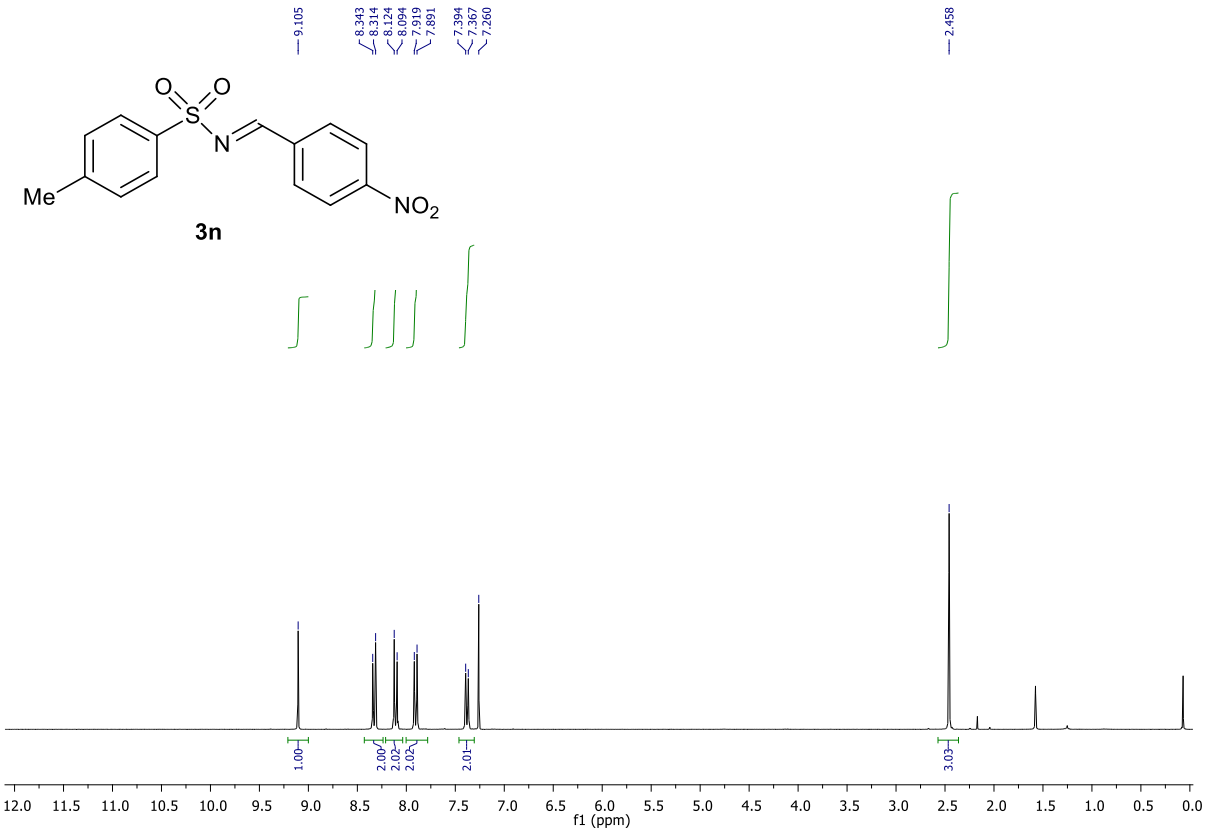
3l


166.14
163.79
163.71
162.69
144.93
137.20
137.08
134.89
129.99
129.48
129.47
128.35
124.99
124.94
120.67
120.56
116.65
116.37
77.58
77.16
76.74
21.79
O
O
S
N
F
Me
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

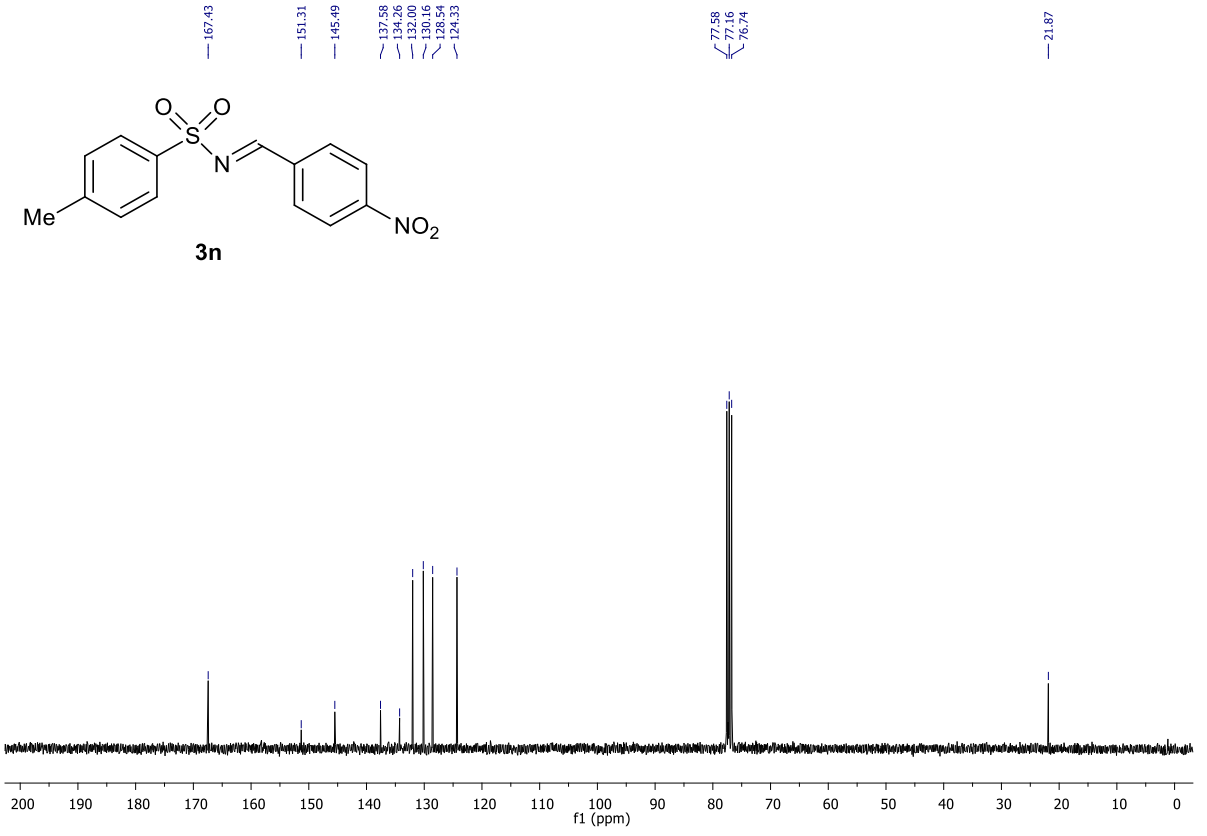
3l


9.074
8.056
8.029
7.909
7.882
7.755
7.728
7.376
7.349
7.260
2.445
O
O
S
N
Me
CF3
3m
1.00
2.01
2.01
2.00
2.00
3.16
f1 (ppm)

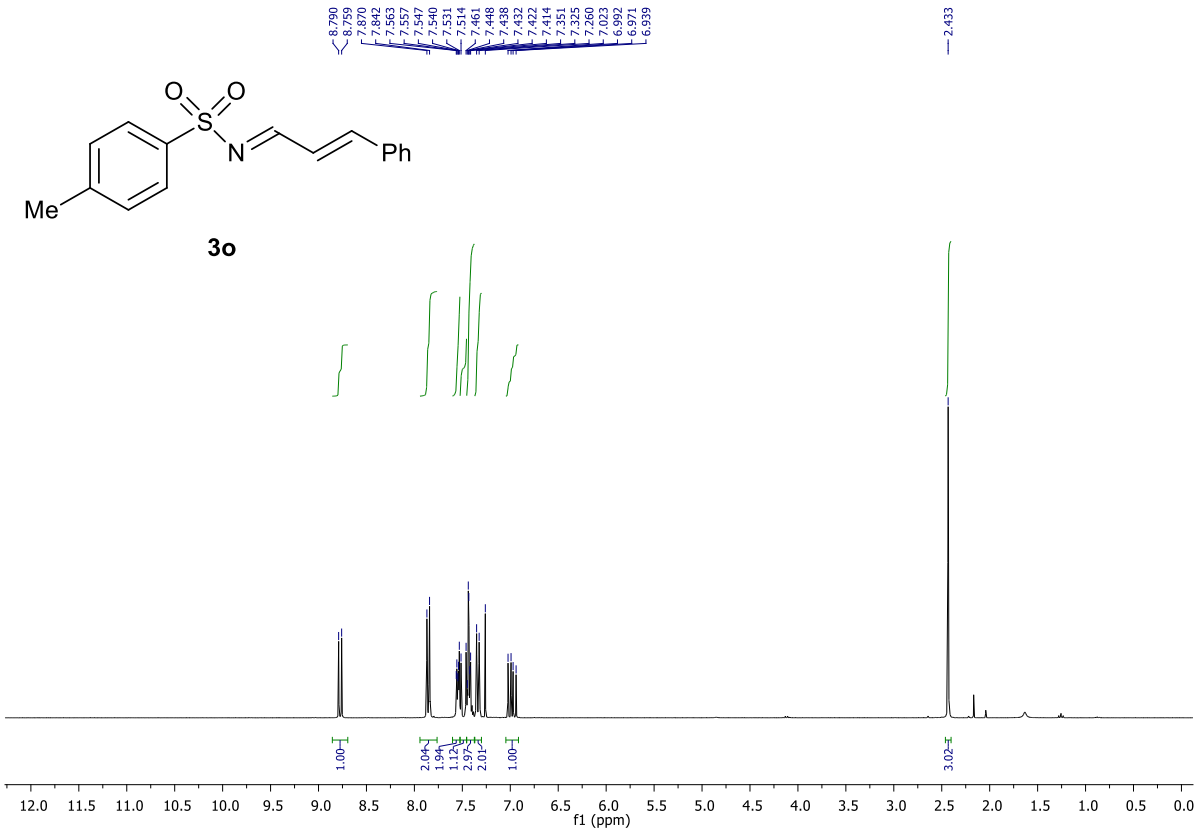

168.50
145.15
136.60
136.16
135.72
135.66
135.29
134.86
131.44
130.05
128.92
128.41
126.27
126.23
126.18
126.12
125.30
121.69
118.07
77.58
77.16
76.74
21.73
O
O
S
N
Me
CF3
3m
f1 (ppm)


9.105
8.343
8.314
8.124
8.094
7.919
7.891
7.394
7.367
7.260
2.458
O
O
S
N
Me
NO2
1.00
2.00
2.02
2.02
2.01
3.03
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

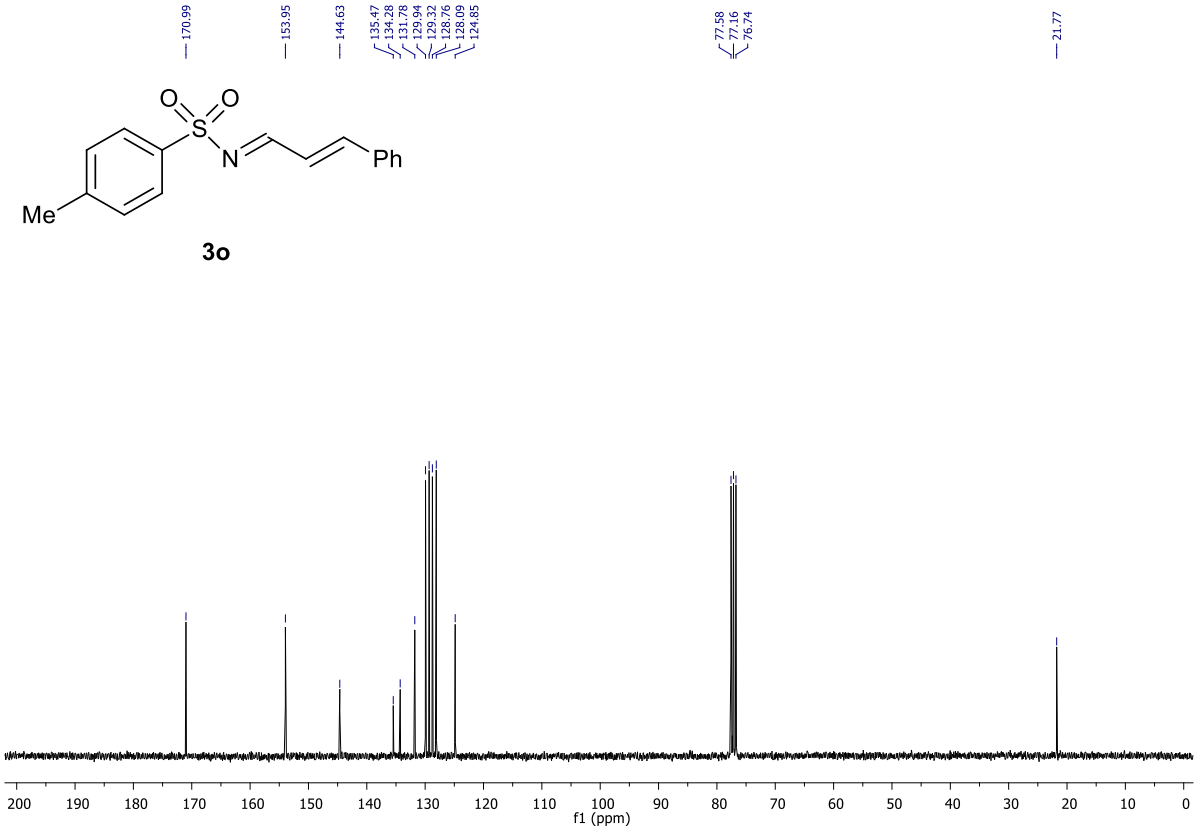
3n


167.43
151.31
145.49
137.58
134.26
132.00
130.16
128.54
124.33
77.58
77.16
76.74
21.87
O
O
S
N
Me
NO2
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

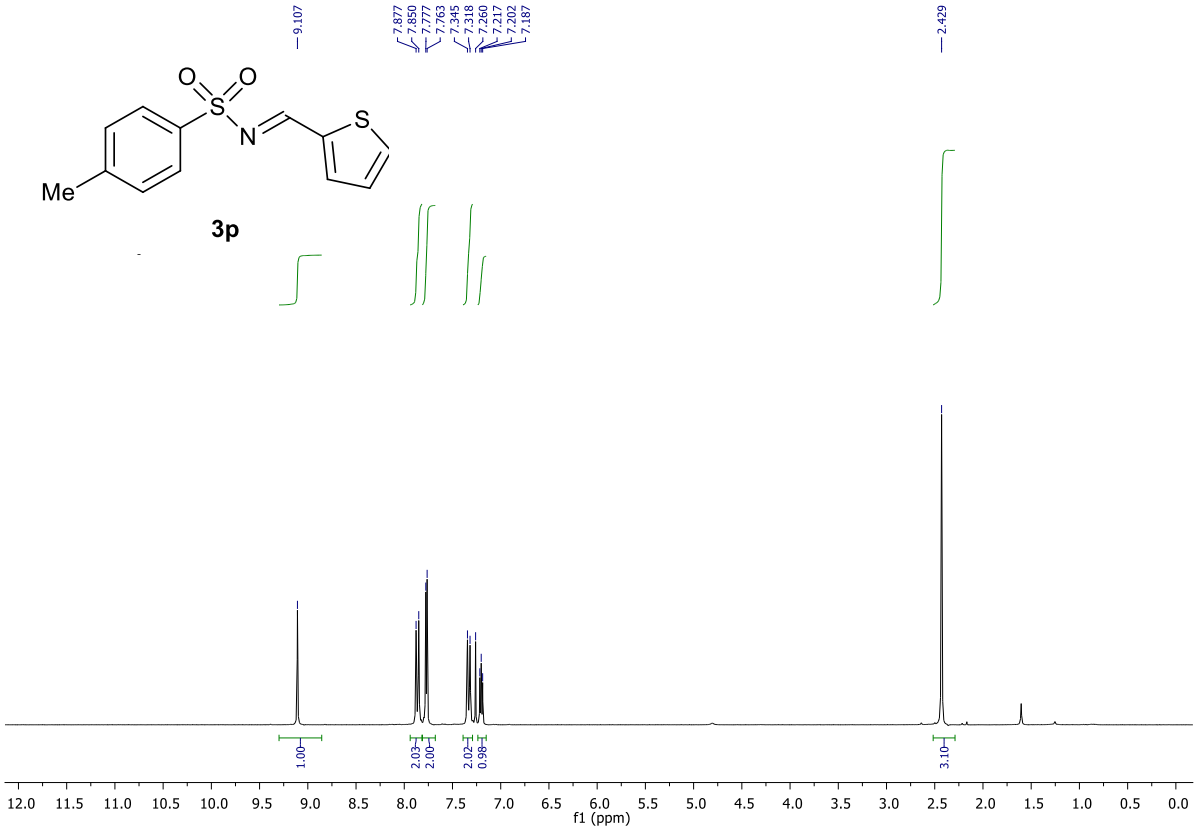
3n

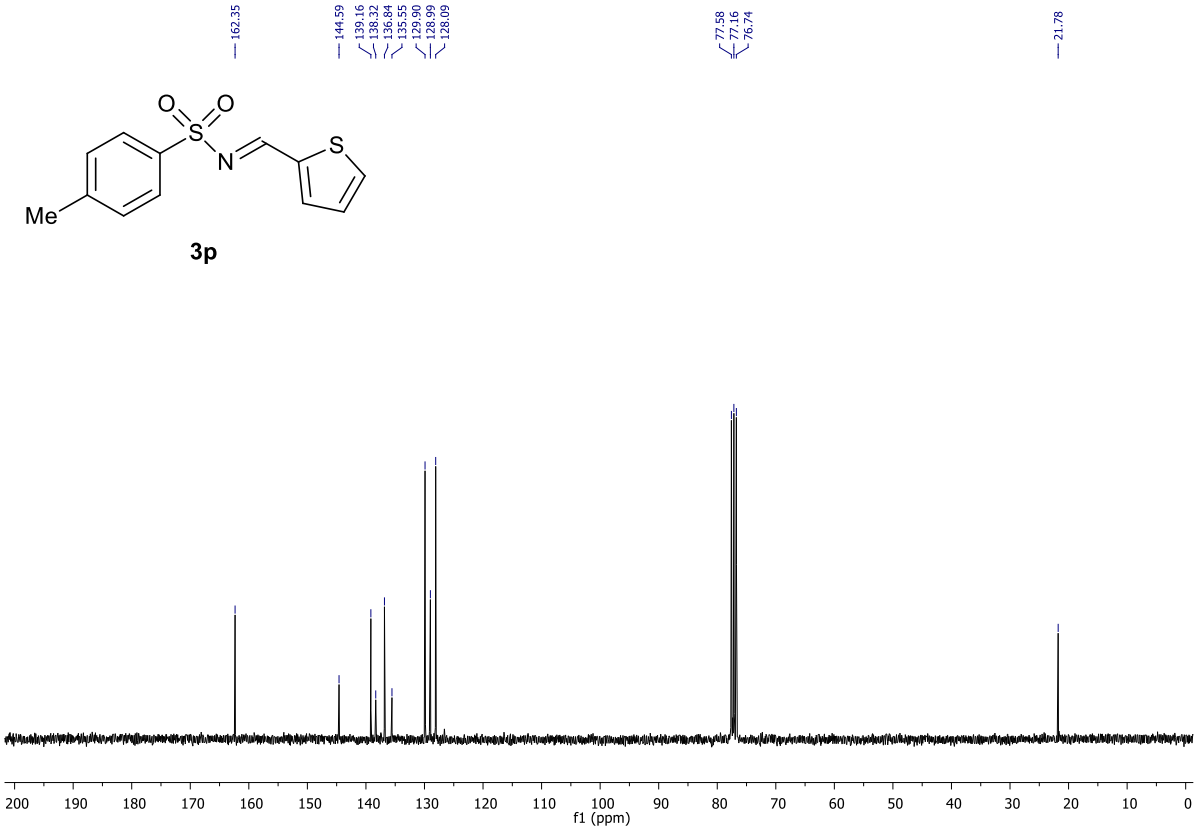

8.790
8.759
7.870
7.842
7.563
7.557
7.547
7.540
7.531
7.514
7.461
7.448
7.438
7.432
7.422
7.414
7.351
7.325
7.260
7.023
6.992
6.971
6.939
2.433
O
O
S
N
Ph
Me
1.00
2.04
1.94
1.12
2.97
2.01
1.00
3.02
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

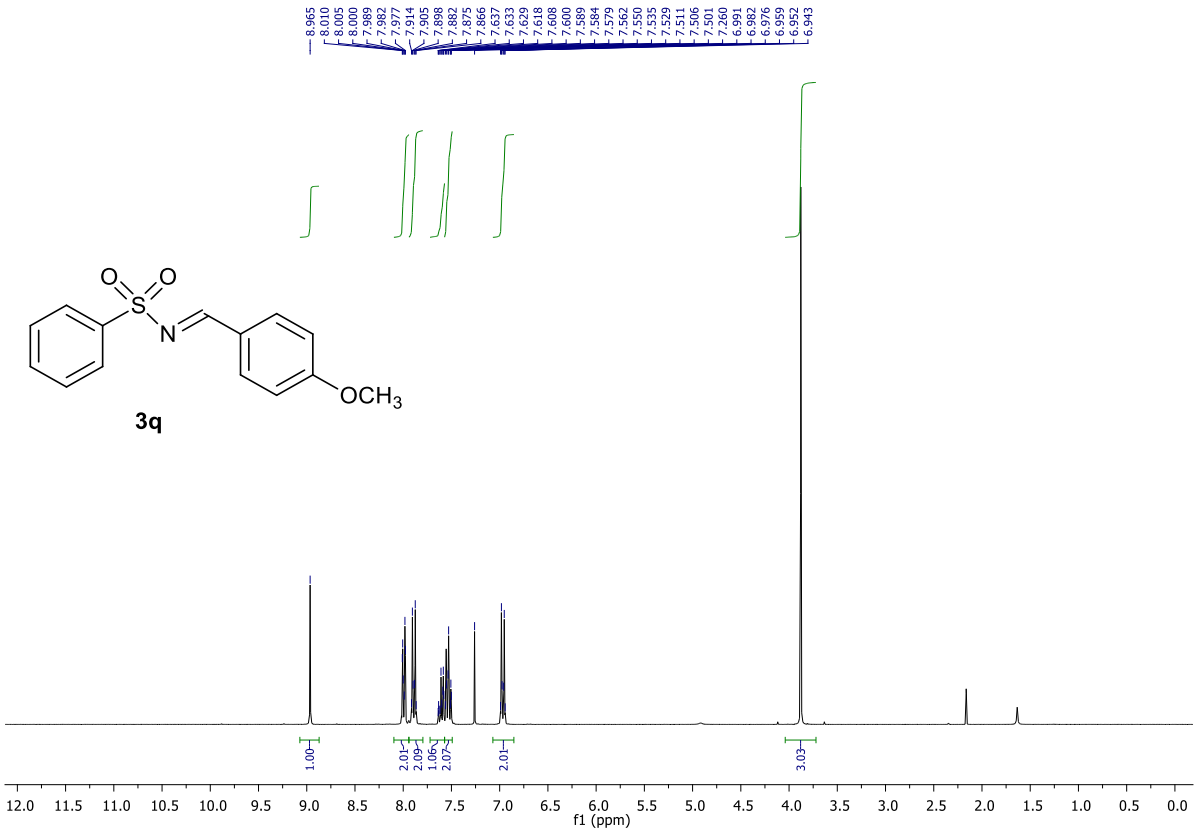
3o

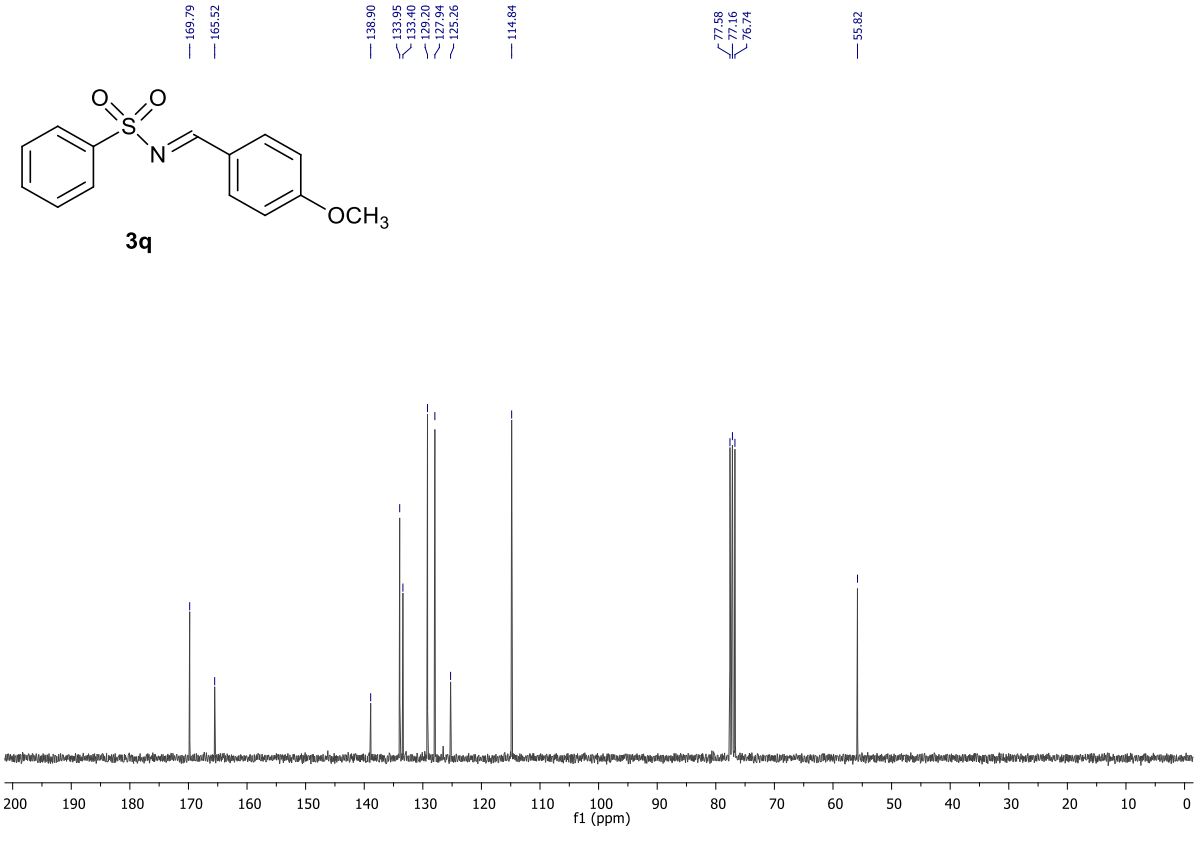

170.99
153.95
144.63
135.47
134.28
131.78
129.94
129.32
128.76
128.09
124.85
77.58
77.16
76.74
21.77
O
O
S
N
Ph
Me
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

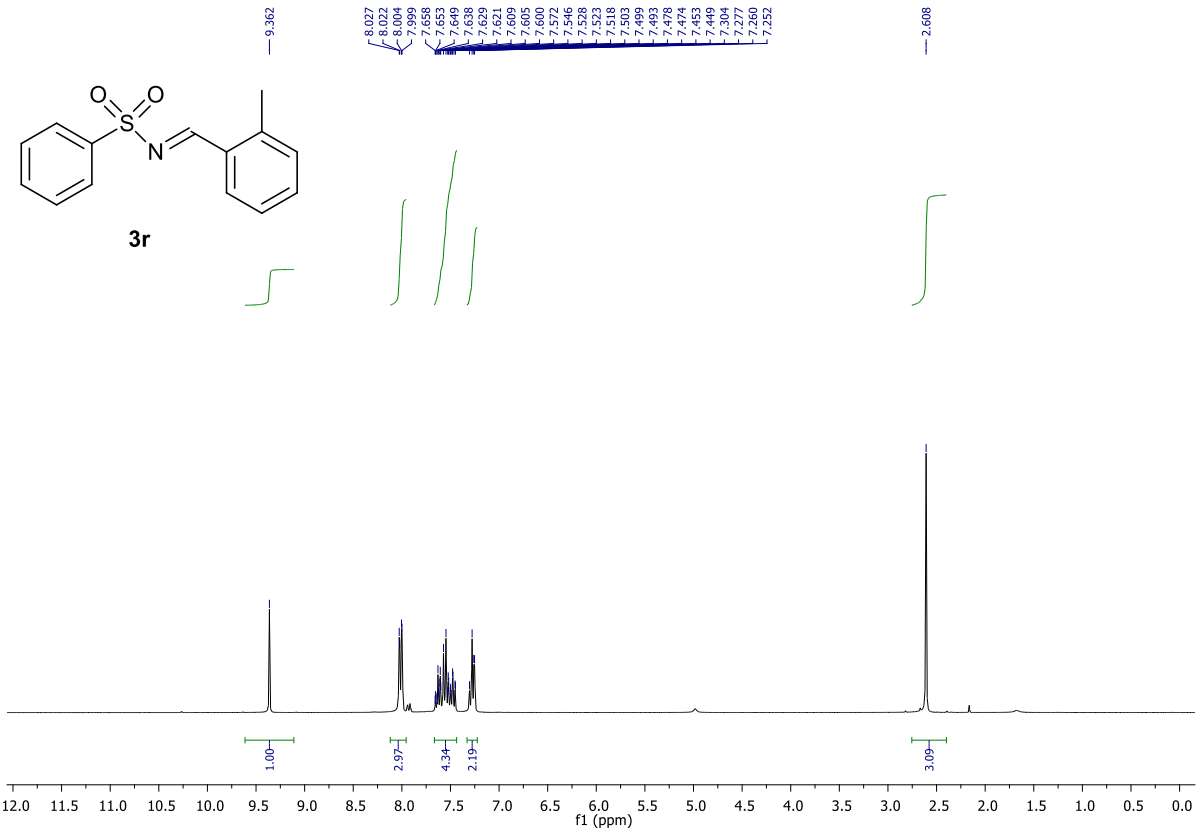
3o


9.107
7.877
7.850
7.777
7.763
7.345
7.318
7.260
7.217
7.202
7.187
2.429
O
O
S
N
S
Me
3p
1.00
2.03
2.00
2.02
0.98
3.10
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

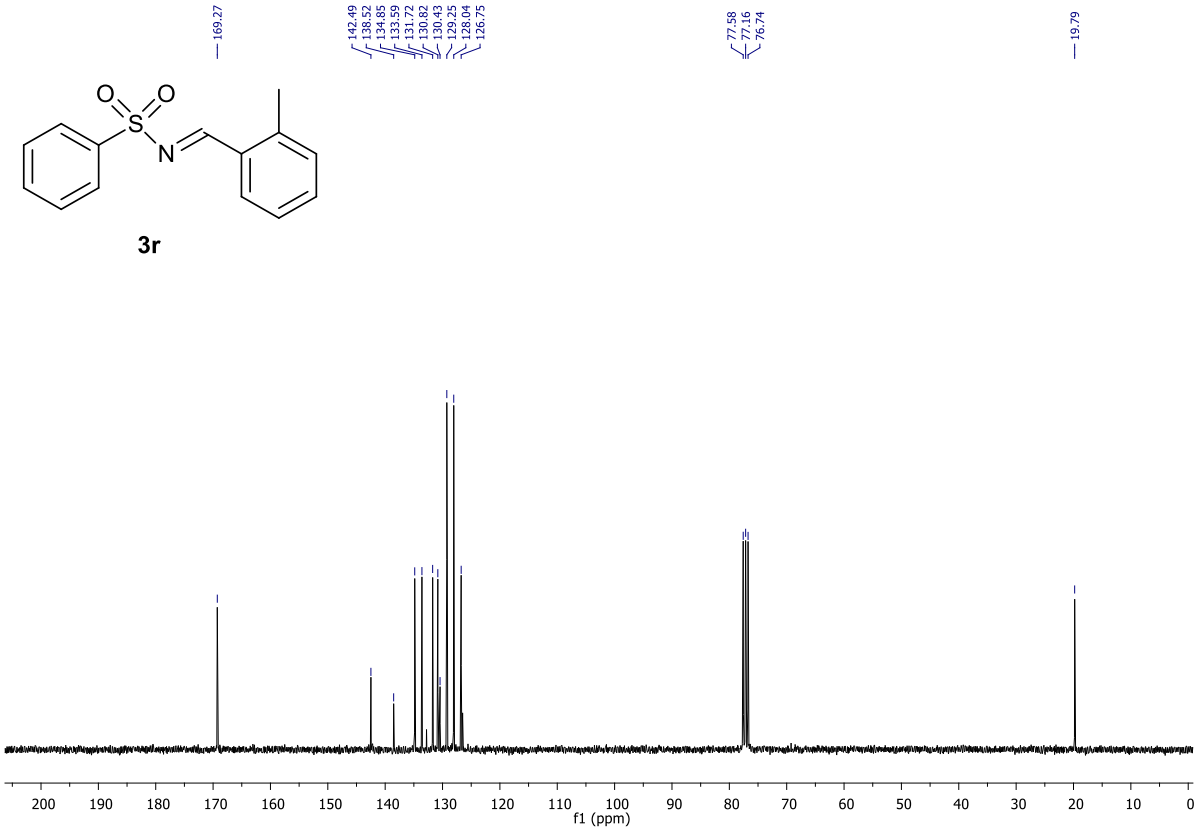

162.35
144.59
139.16
138.32
136.84
135.55
129.90
128.99
128.09
77.58
77.16
76.74
21.78
O
O
S
N
S
Me
3p
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


8.965
8.010
8.005
8.000
7.989
7.982
7.977
7.914
7.905
7.898
7.882
7.875
7.866
7.637
7.633
7.629
7.618
7.608
7.600
7.589
7.584
7.579
7.562
7.550
7.535
7.529
7.511
7.506
7.501
7.260
6.991
6.982
6.976
6.959
6.952
6.943
O
O
S
N
OCH3
3q
1.00
2.01
2.09
1.06
2.07
2.01
3.03
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

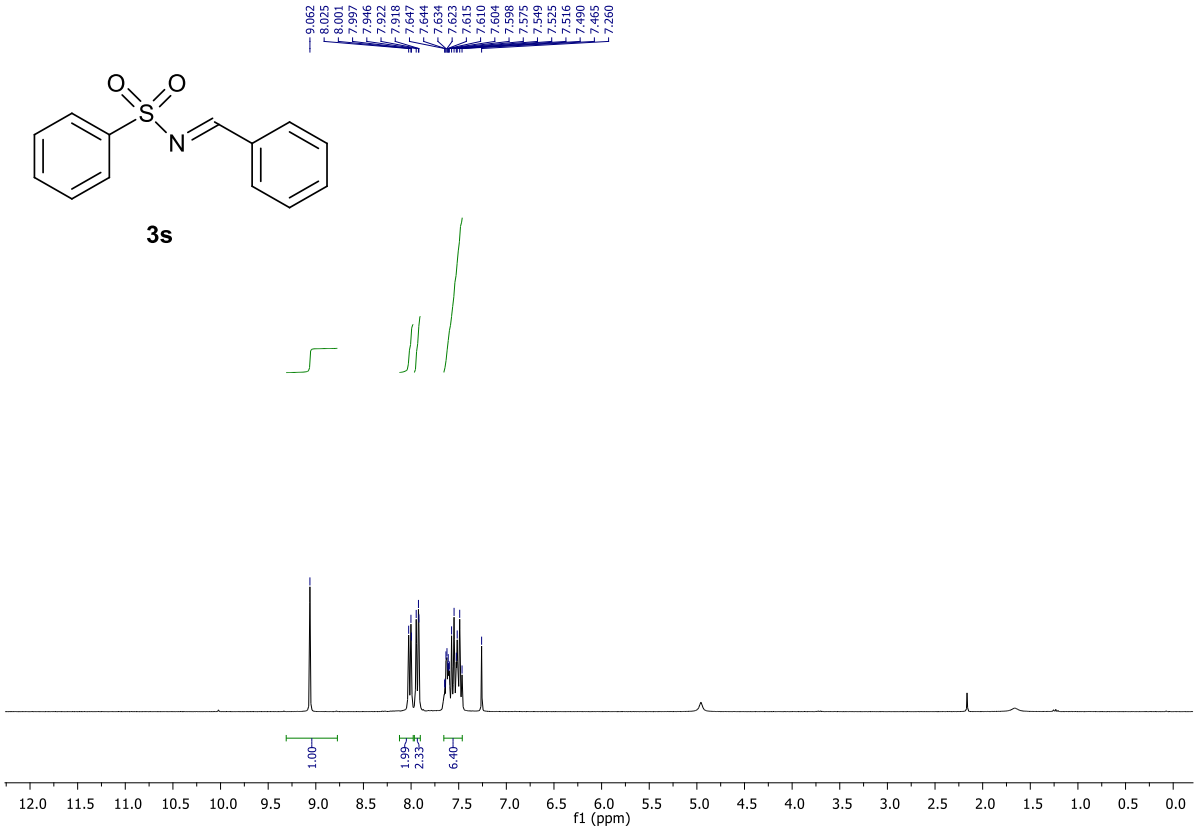

169.79
165.52
138.90
133.95
133.40
129.20
127.94
125.26
114.84
77.58
77.16
76.74
55.82
O
O
S
N
OCH3
3q
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


9.362
8.027
8.022
8.004
7.999
7.658
7.653
7.649
7.638
7.629
7.621
7.609
7.605
7.600
7.572
7.546
7.528
7.523
7.518
7.503
7.499
7.493
7.478
7.474
7.453
7.449
7.304
7.277
7.260
7.252
2.608
O
O
S
N
1.00
2.97
4.34
2.19
3.09
f1 (ppm)

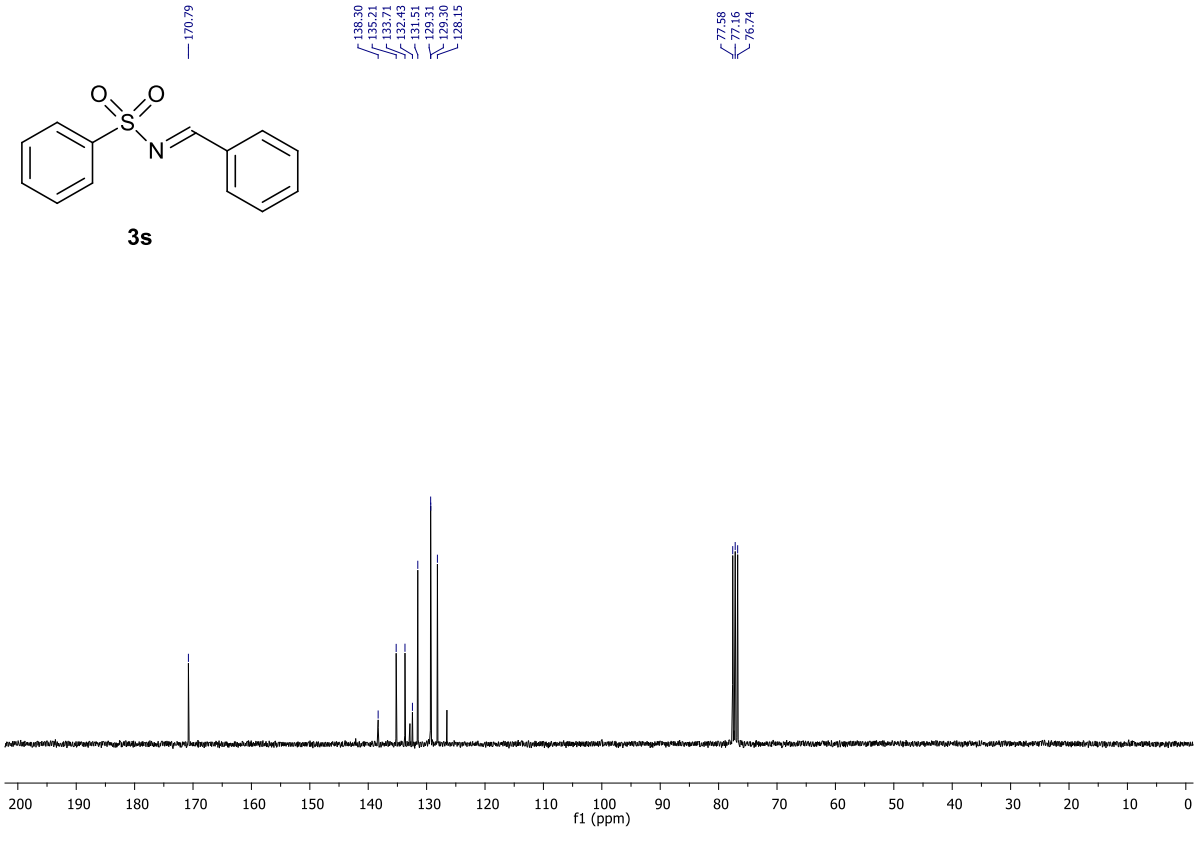
3r


169.27
142.49
138.52
134.85
133.59
131.72
130.82
130.43
129.25
128.04
126.75
77.58
77.16
76.74
19.79
O
O
S
N
f1 (ppm)

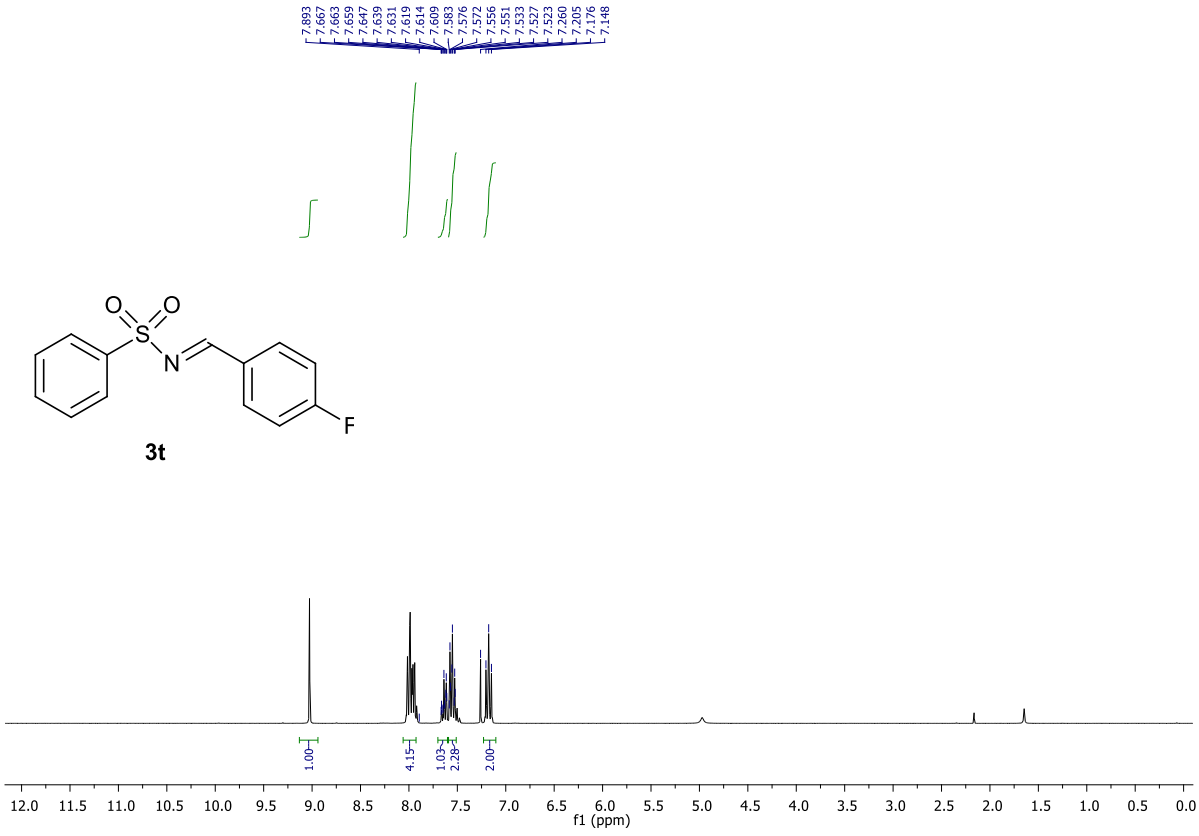
3r


9.062
8.025
8.001
7.997
7.946
7.922
7.918
7.647
7.644
7.634
7.623
7.615
7.610
7.604
7.598
7.575
7.549
7.525
7.516
7.490
7.465
7.260
O
O
S
N
1.00
1.99
2.33
6.40
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

3s


170.79
138.30
135.21
133.71
132.43
131.51
129.31
129.30
128.15
77.58
77.16
76.74
O
O
S
N
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

3s


7.893
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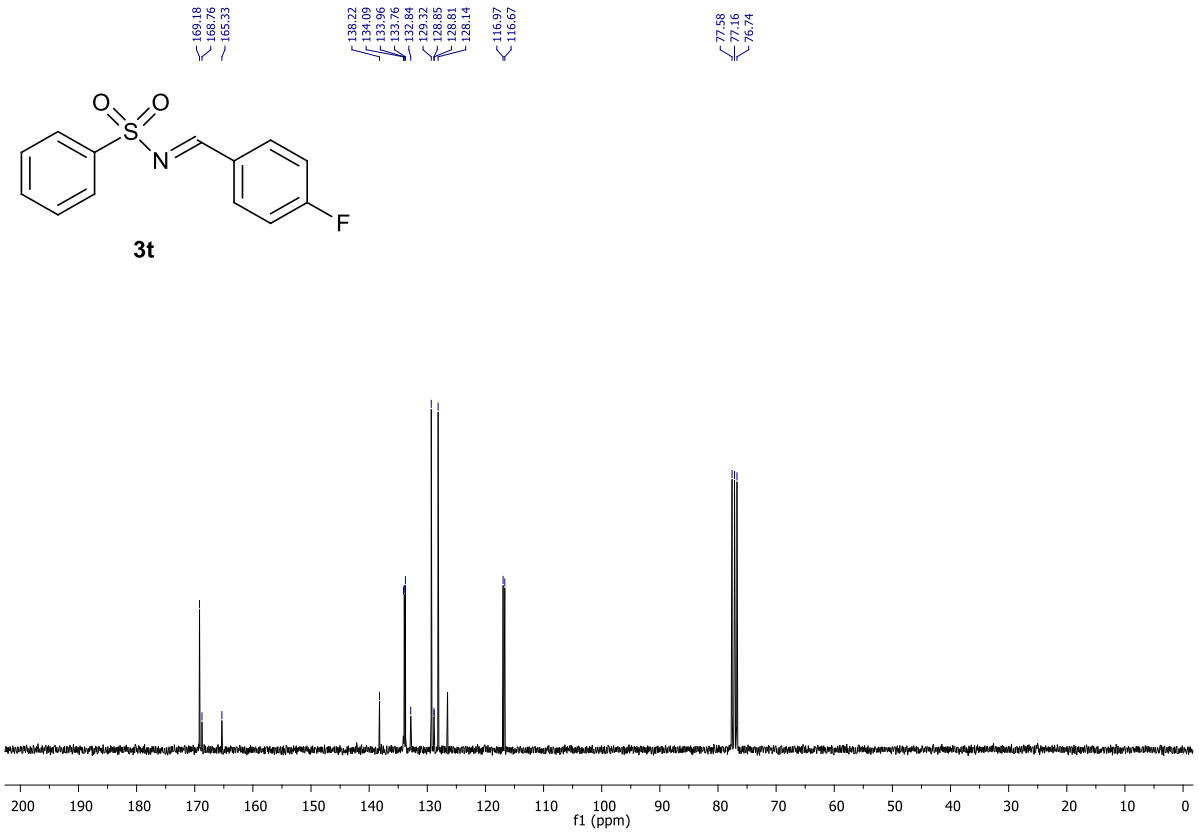
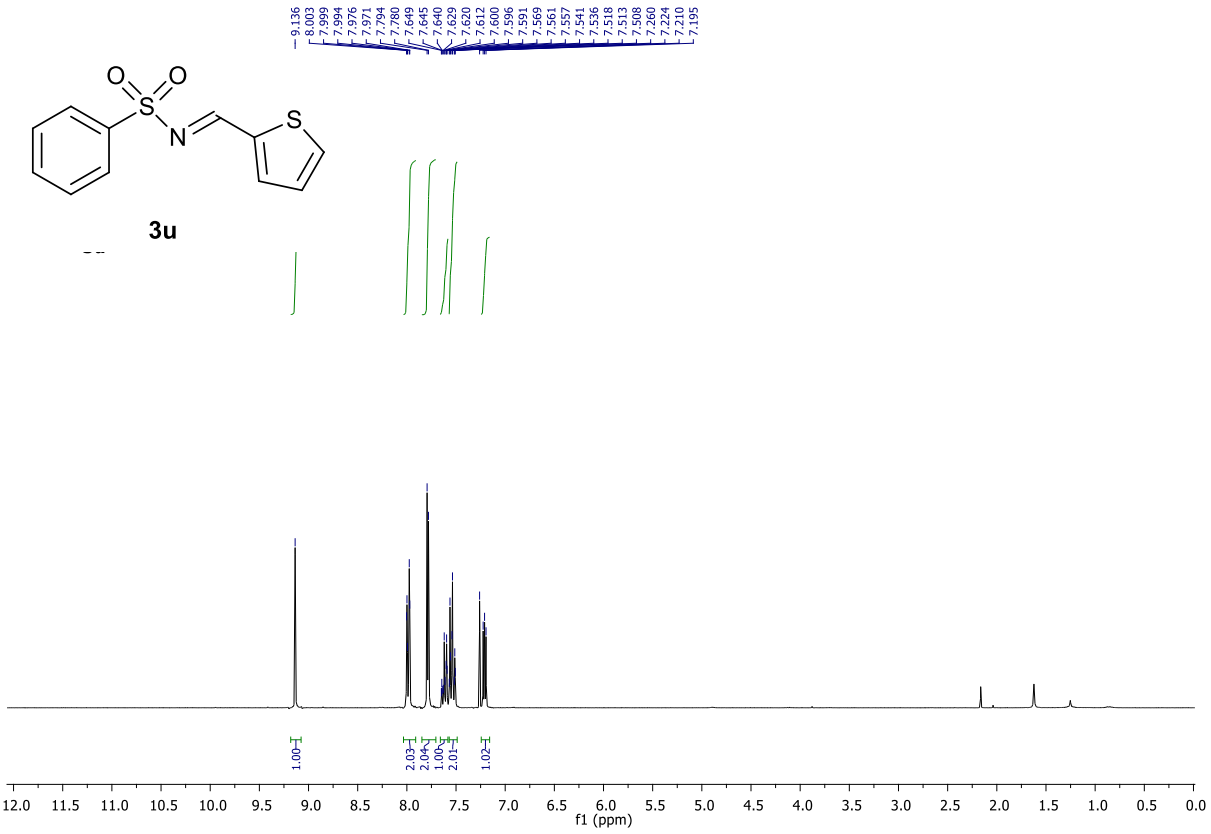
7.663
7.659
7.647
7.639
7.631
7.619
7.614
7.609
7.583
7.576
7.572
7.556
7.551
7.533
7.527
7.523
7.260
7.205
7.176
7.148
O
O
S
N
F
3t
1.00
4.15
1.03
2.28
2.00
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


169.18
168.76
165.33
138.22
134.09
133.96
133.76
132.84
129.32
128.85
128.81
128.14
116.97
116.67
77.58
77.16
76.74
O
O
S
N
F
3t
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


9.136
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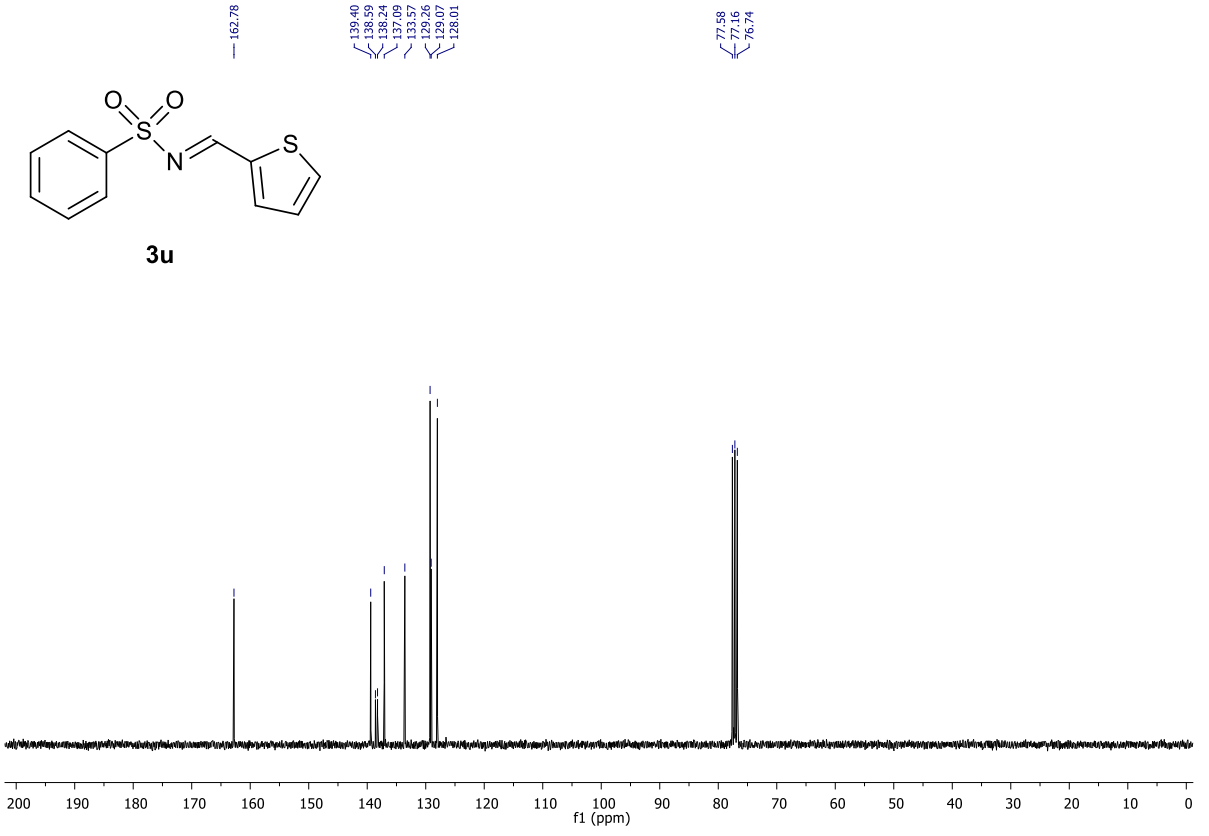
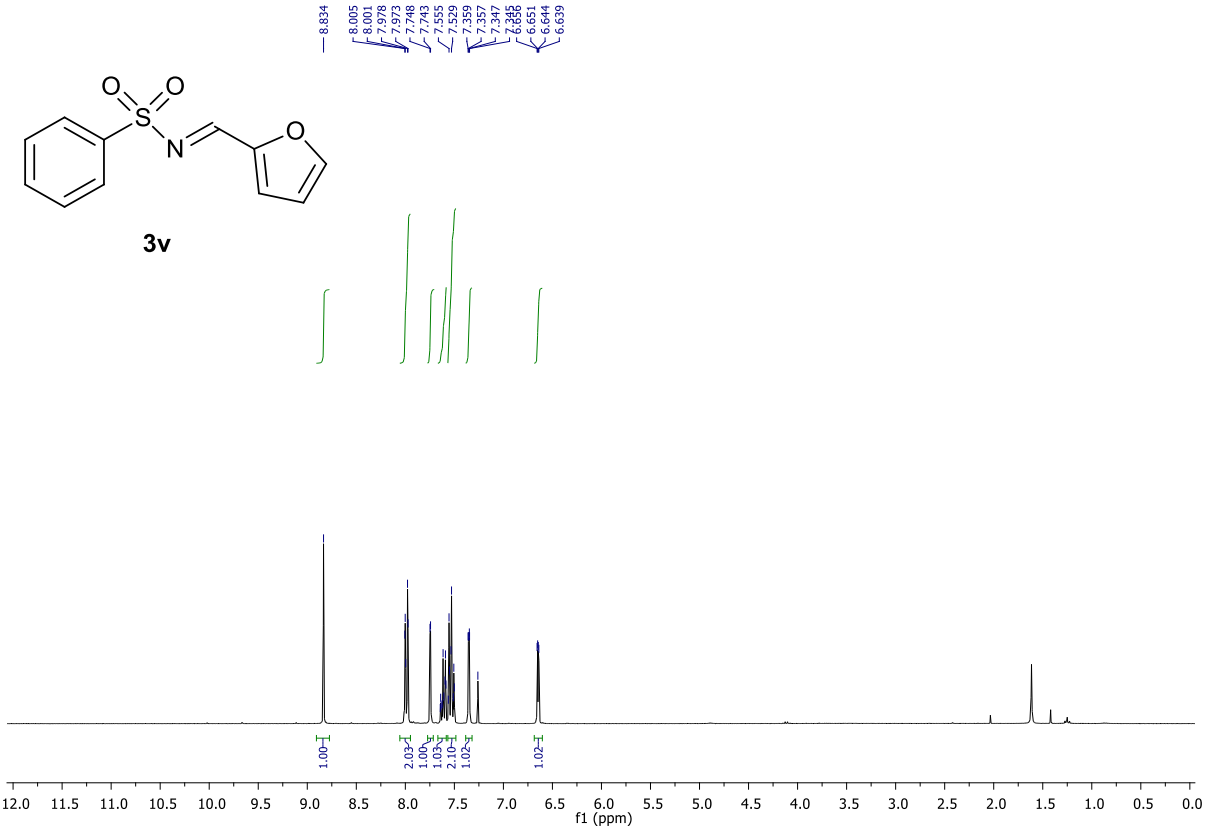
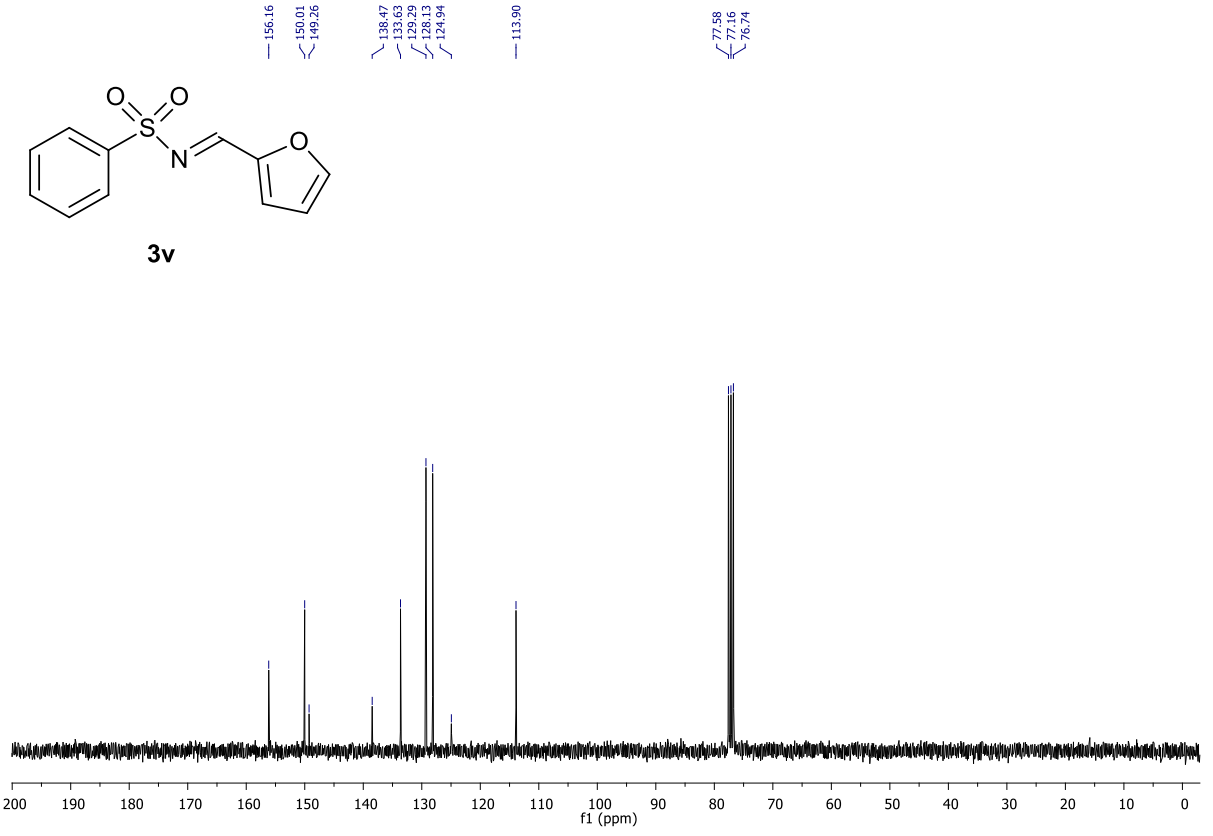
7.999
7.994
7.976
7.971
7.794
7.780
7.649
7.645
7.640
7.629
7.620
7.612
7.600
7.596
7.591
7.569
7.561
7.557
7.541
7.536
7.518
7.513
7.508
7.260
7.224
7.210
7.195
O
O
S
N
S
3u
1.00
2.03
2.04
1.00
2.01
1.02
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

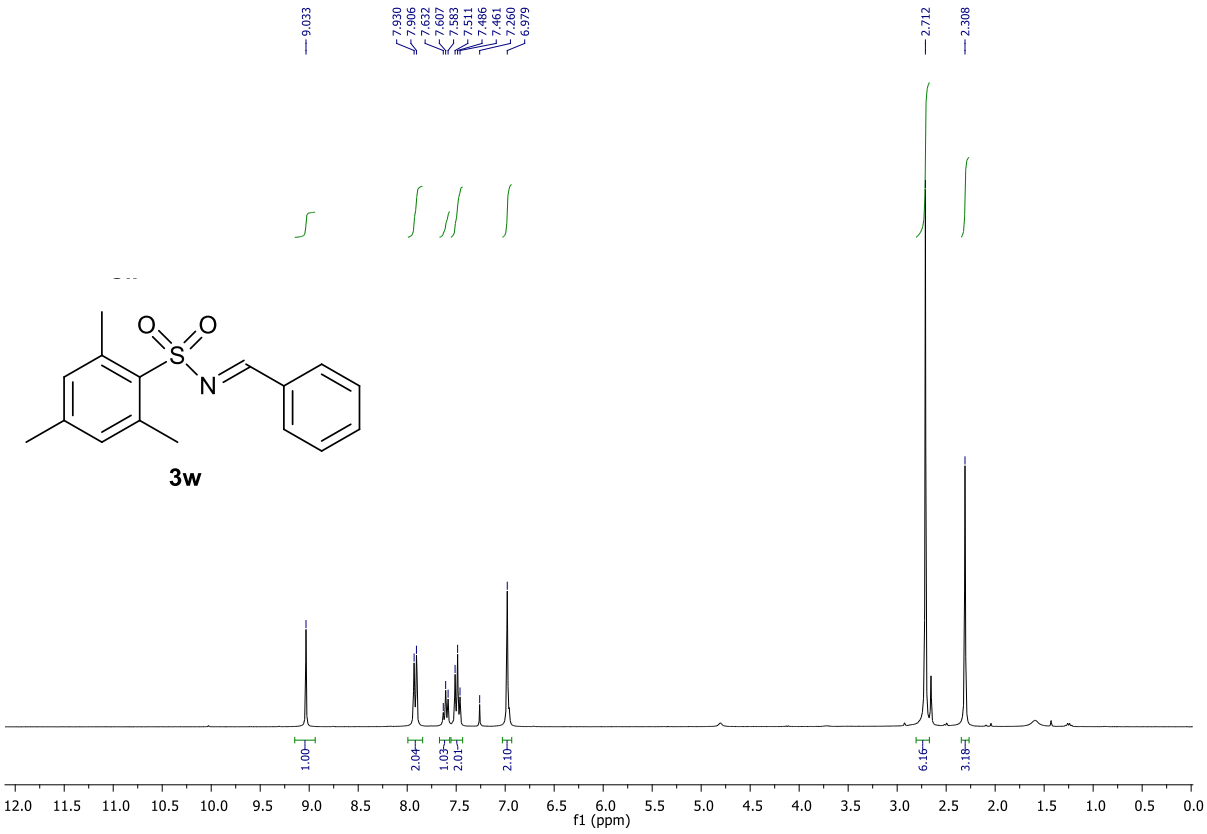

162.78
139.40
138.59
138.24
137.09
133.57
129.26
129.07
128.01
77.58
77.16
76.74
O
O
S
N
S
3u
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


8.834
8.005
8.001
7.978
7.973
7.748
7.743
7.555
7.529
7.359
7.357
7.347
6.651
6.644
6.639
1.00
2.03
1.06
1.05
2.10
1.02
1.02
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

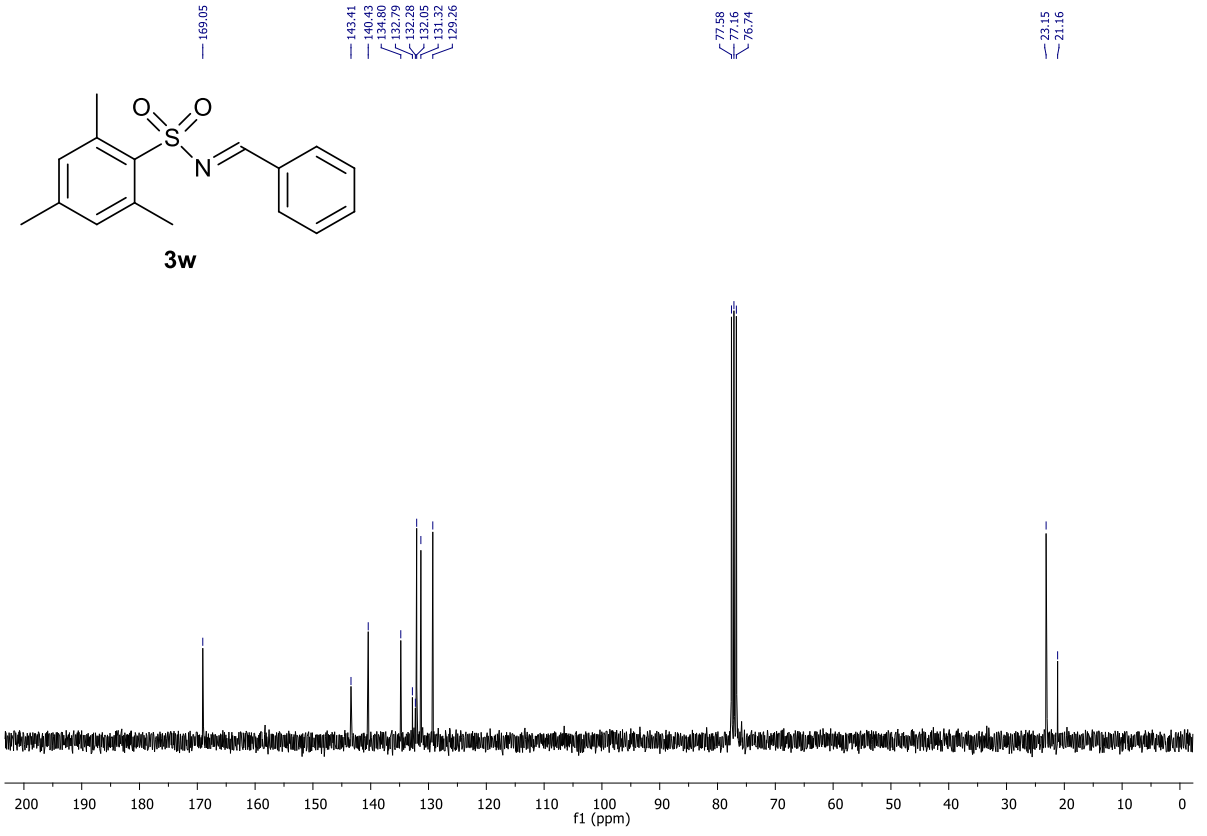
3v


156.16
150.01
149.26
138.47
133.63
129.29
128.13
124.94
113.90
77.58
77.16
76.74
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

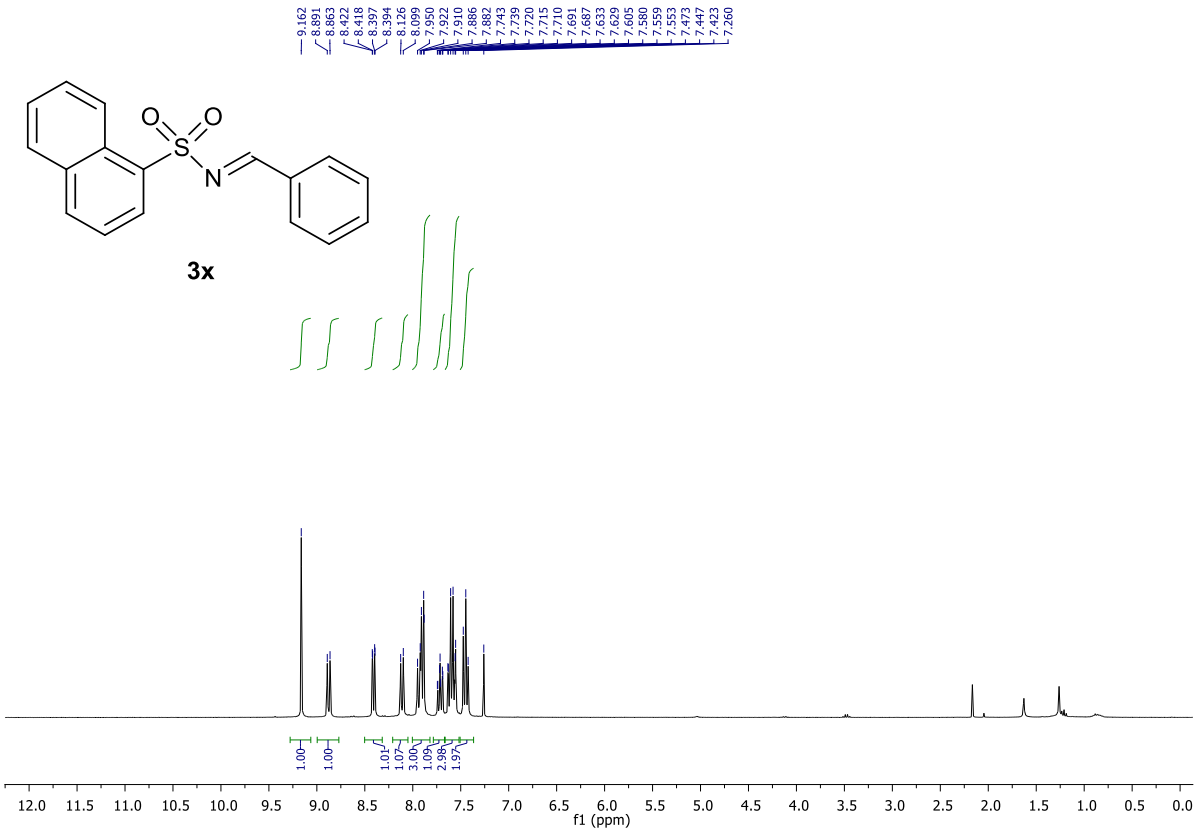
3v

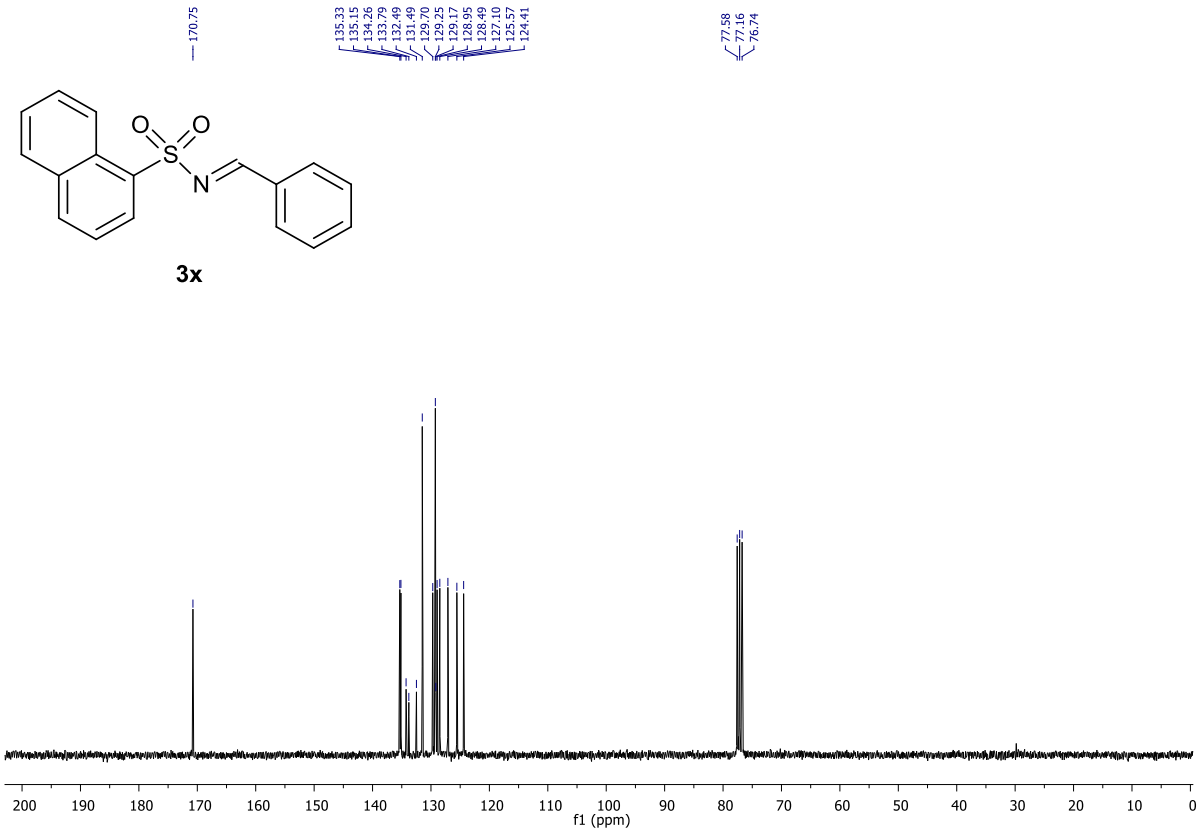

9.033
7.930
7.906
7.632
7.607
7.583
7.511
7.486
7.461
7.260
6.979
2.712
2.308
1.00
2.04
1.03
2.01
2.10
6.16
3.18
f1 (ppm)

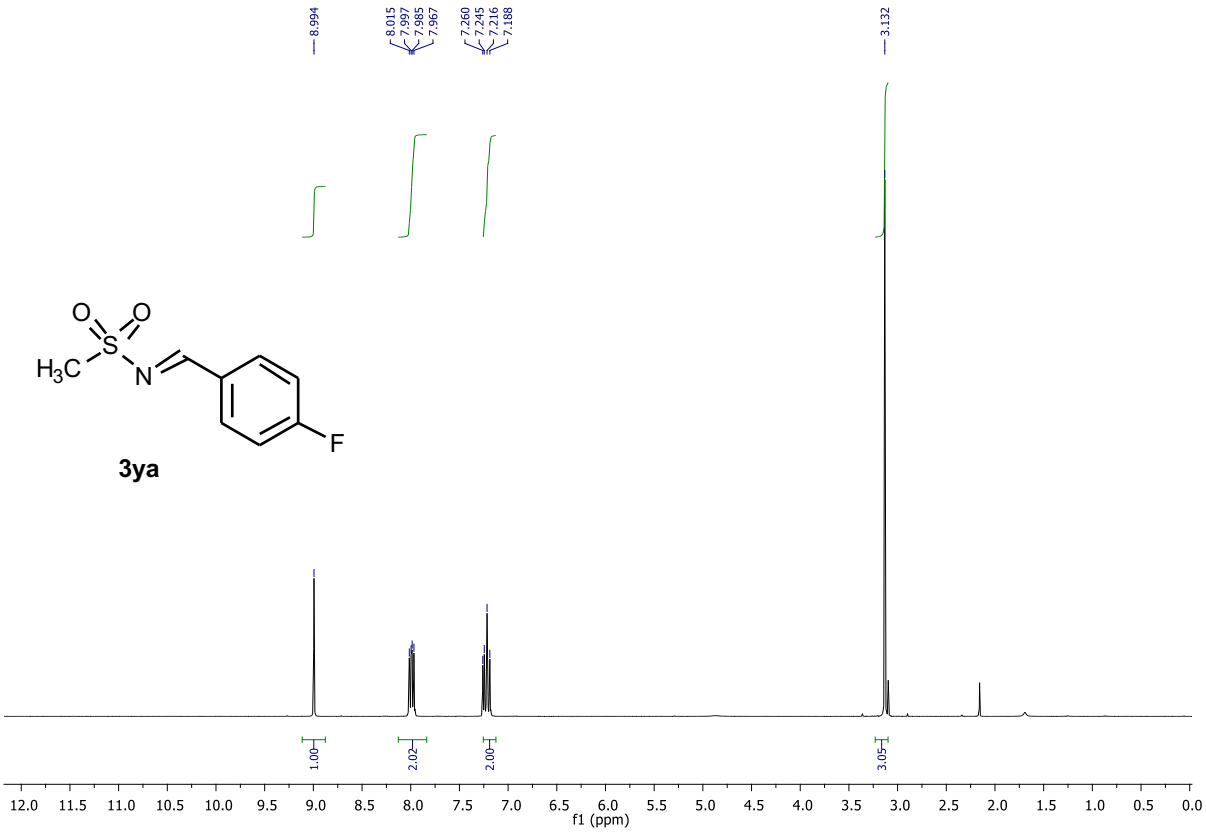
3w

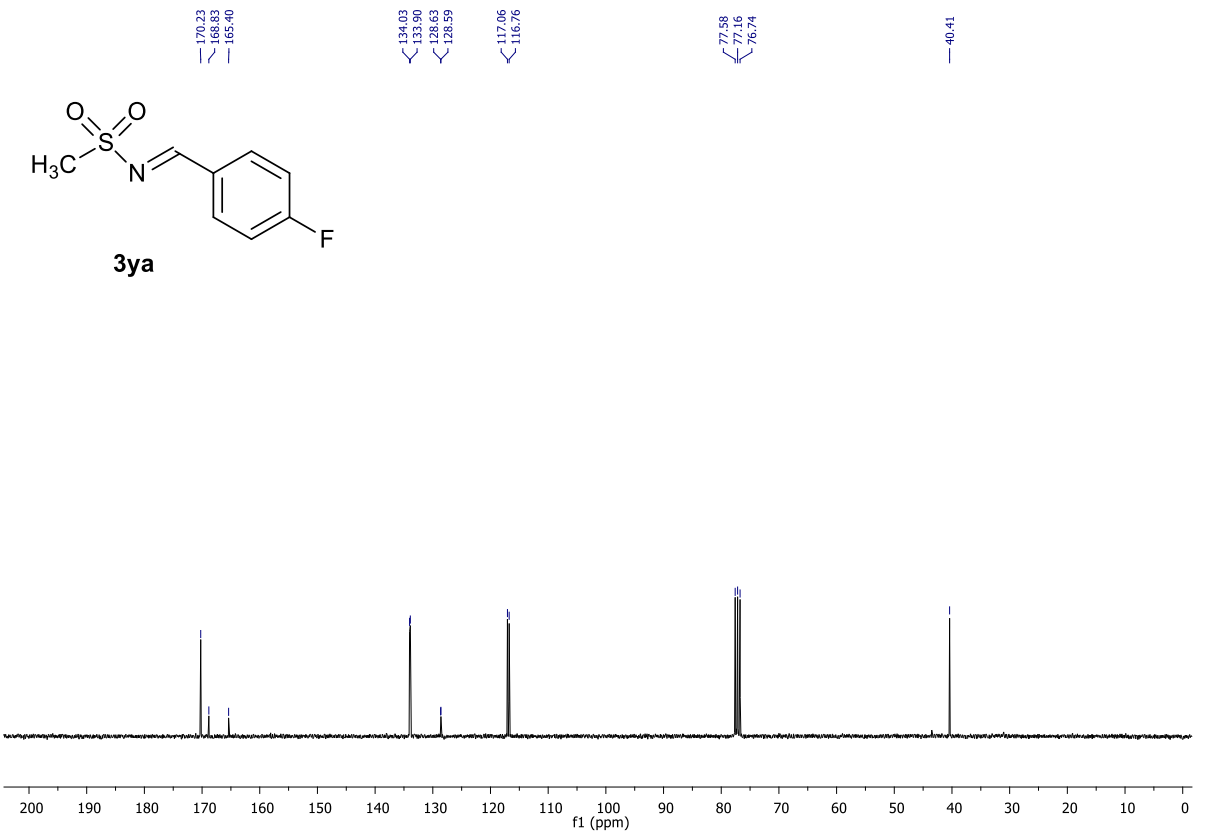

169.05
143.41
140.43
134.80
132.79
132.28
132.05
131.32
129.26
77.58
77.16
76.74
23.15
21.16
f1 (ppm)

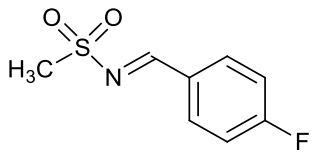
3w


9.162
8.891
8.863
8.422
8.418
8.397
8.394
8.126
8.099
7.950
7.922
7.910
7.886
7.882
7.743
7.739
7.720
7.715
7.710
7.691
7.687
7.633
7.629
7.605
7.580
7.559
7.553
7.473
7.447
7.423
7.260
O
O
S
N
3x
1.00
1.00
1.01
1.07
3.00
1.09
2.98
1.97
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


170.75
135.33
135.15
134.26
133.79
132.49
131.49
129.70
129.25
129.17
128.95
128.49
127.10
125.57
124.41
77.58
77.16
76.74
O
O
S
N
3x
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

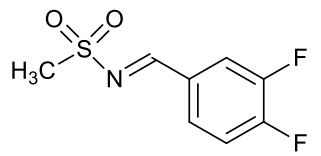

8.994
8.015
7.997
7.985
7.967
7.260
7.245
7.216
7.188
3.132
O
O
S
H3C
N
F
3ya
1.00
2.02
2.00
3.05
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


170.23
168.83
165.40
134.03
133.90
128.63
128.59
117.06
116.76
77.58
77.16
76.74
40.41
O
O
S
H3C
N
F
3ya
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



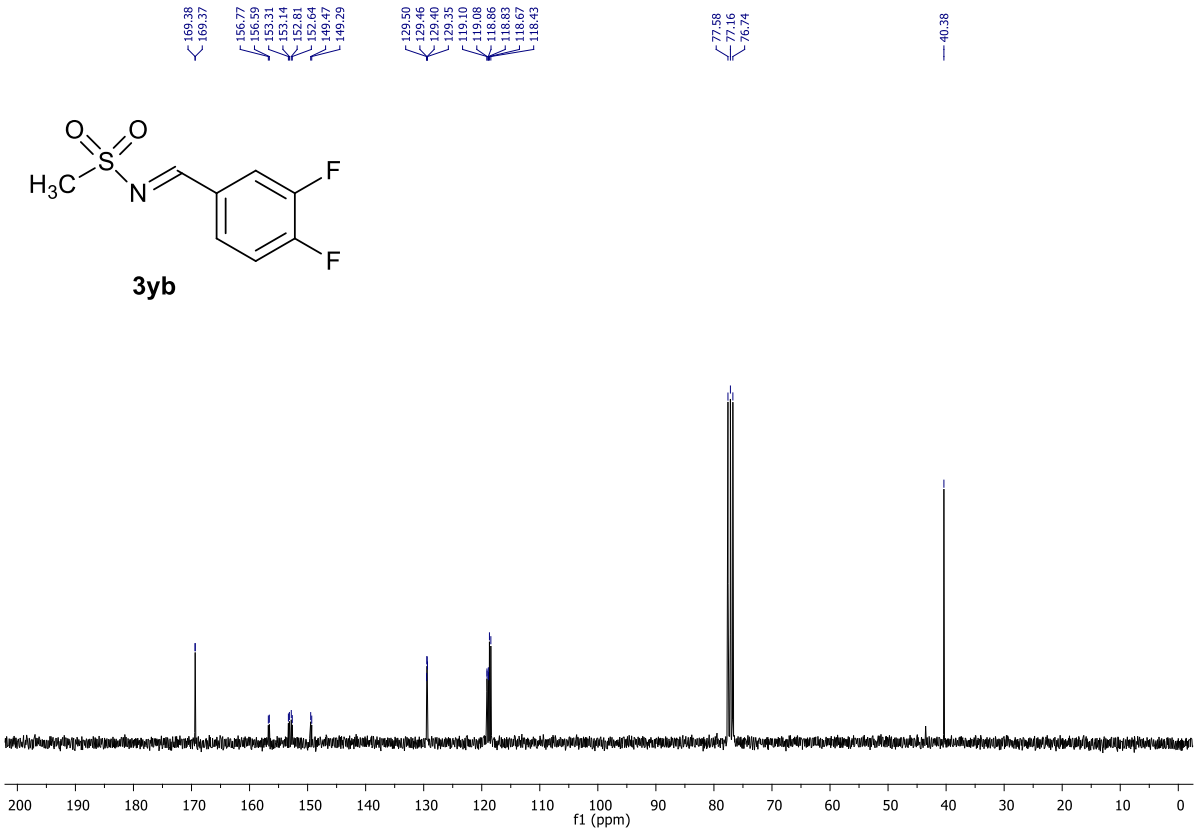
3ya

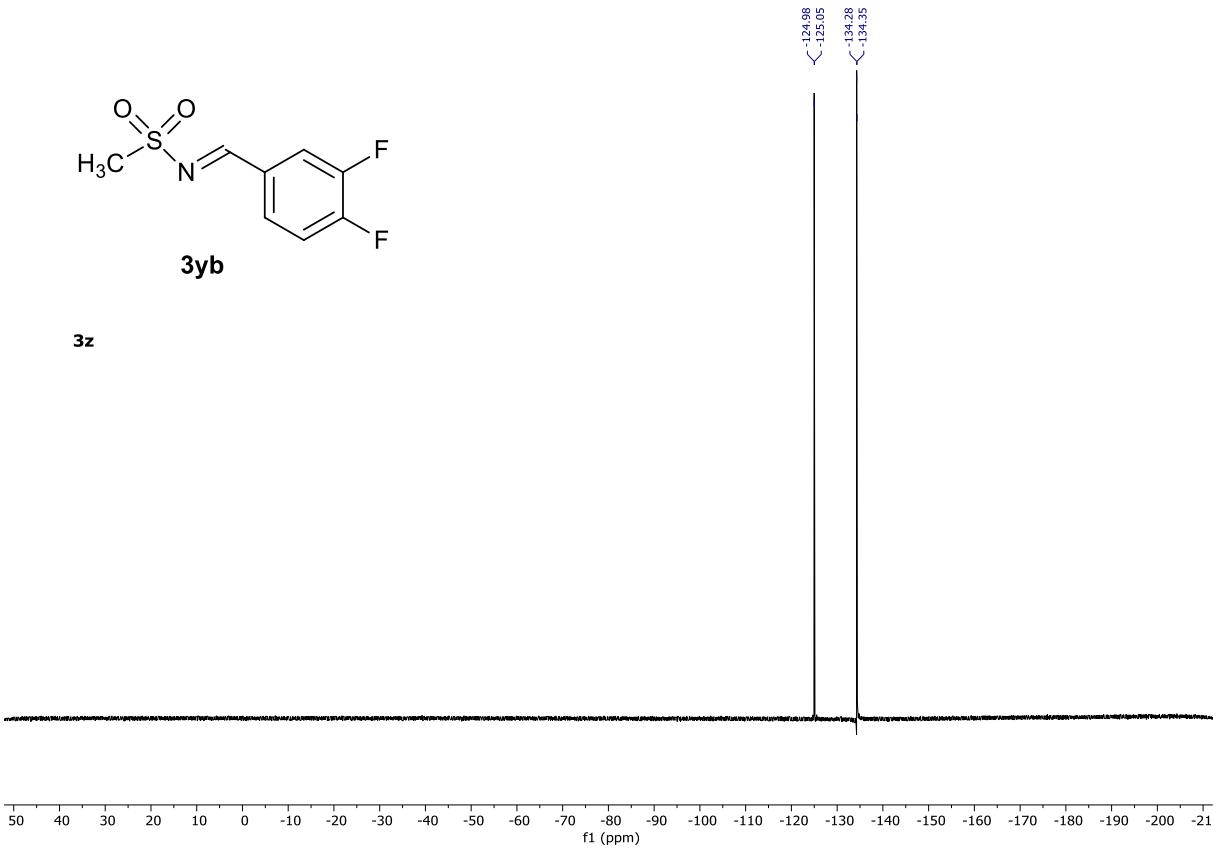
-100.54
50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -21
f1 (ppm)

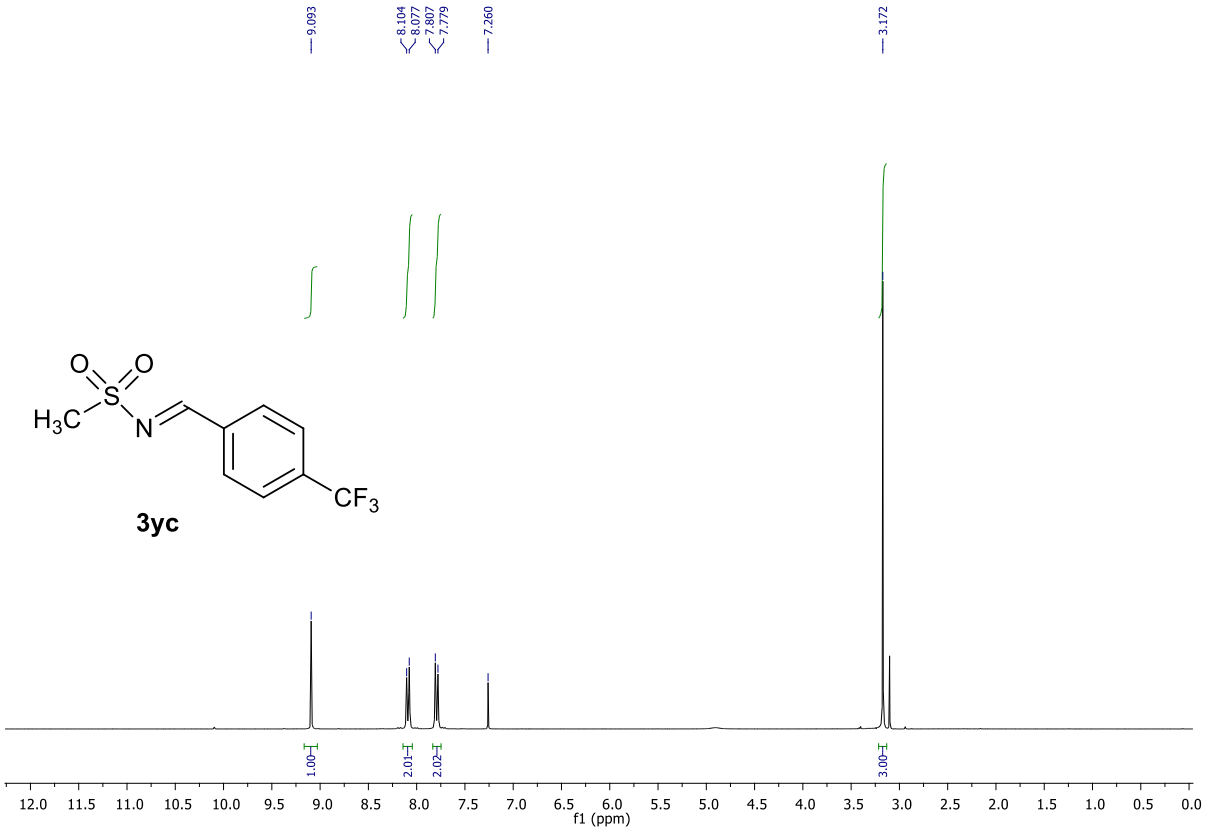


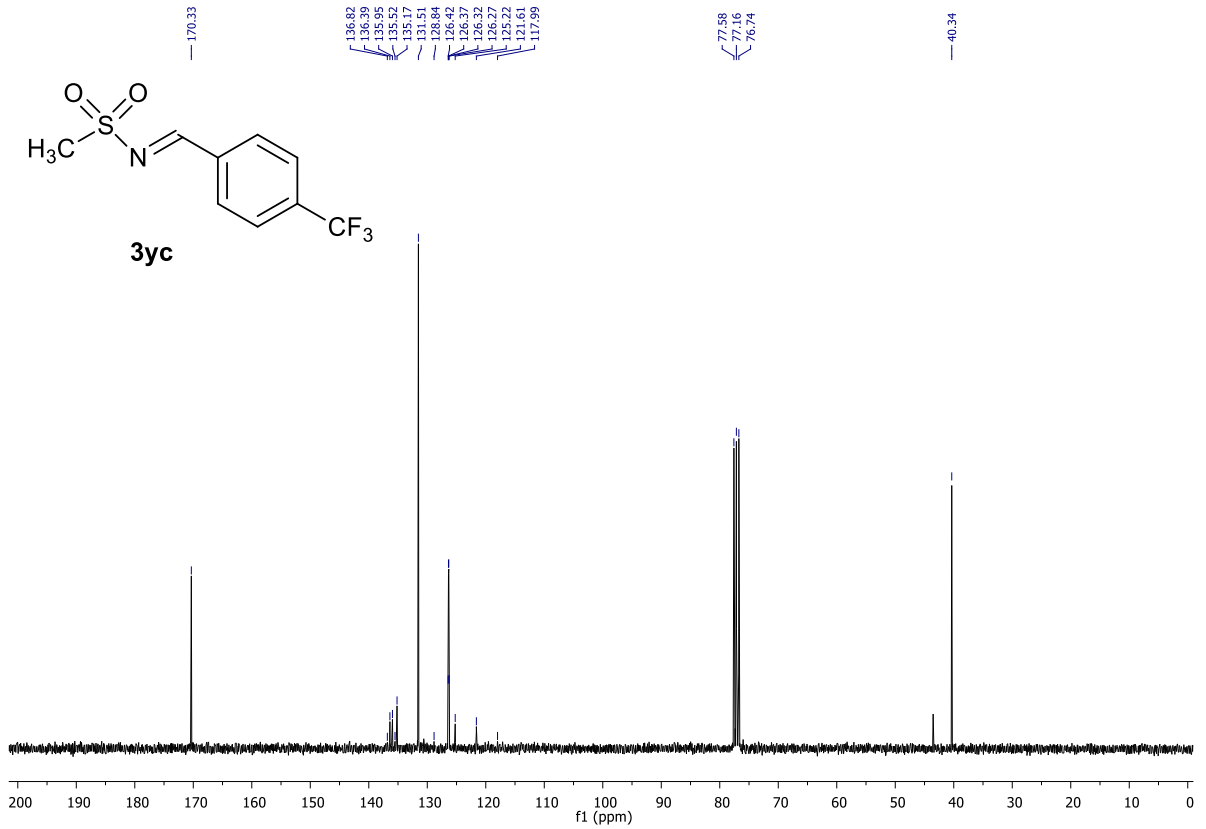
3yb

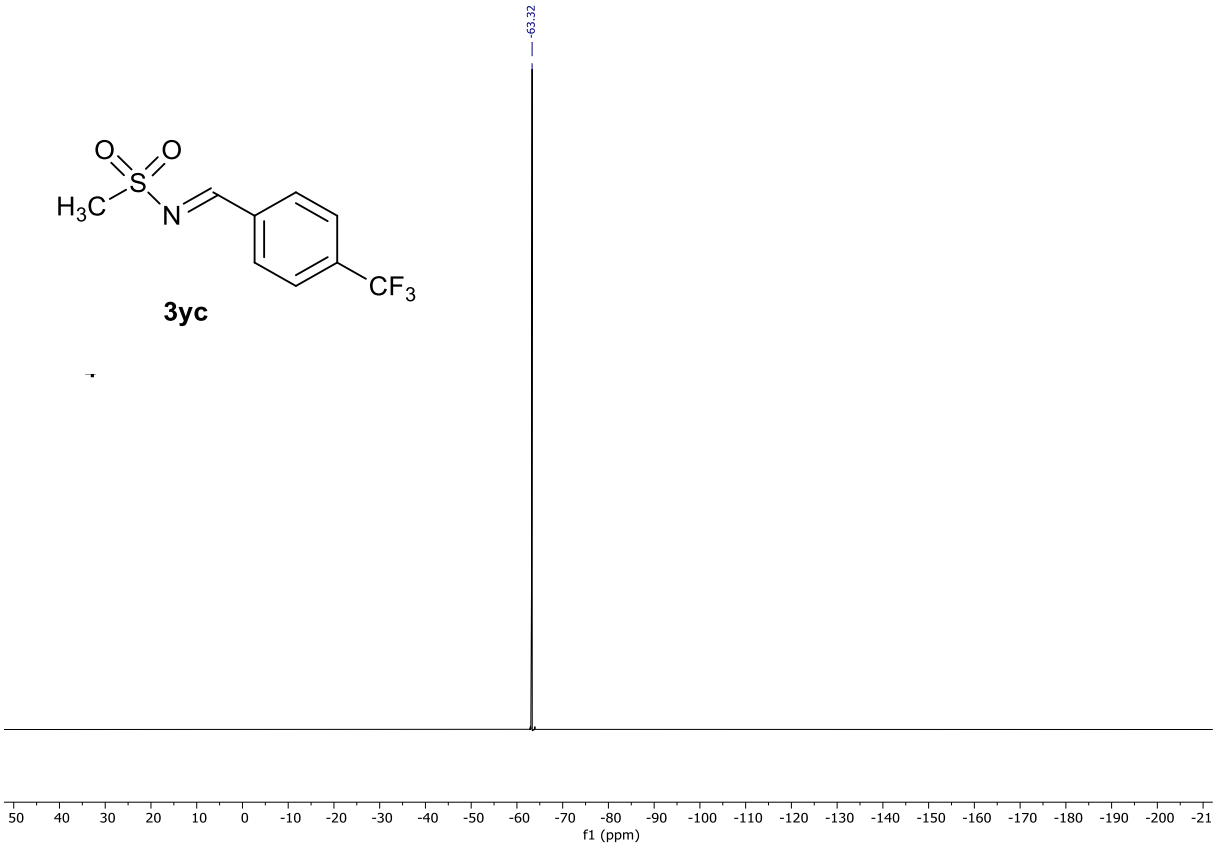
8.964
7.867
7.860
7.852
7.725
7.719
7.711
7.705
7.698
7.355
7.326
7.297
7.260
3.144
1.00
1.00
1.02
1.01
3.16
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


169.38
169.37
156.77
156.59
153.31
153.14
152.81
152.64
149.47
149.29
129.50
129.46
129.40
129.35
119.10
119.08
118.86
118.83
118.67
118.43
77.58
77.16
76.74
40.38
O
O
S
H3C
N
F
F
3yb
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


-124.98
-125.05
-134.28
-134.35
O
O
S
H3C
N
F
F
3yb
3z
50
40
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-21
f1 (ppm)


9.093
8.104
8.077
7.807
7.779
7.260
3.172
O
O
S
H3C
N
CF3
3yc
1.00
2.01
2.02
3.00
f1 (ppm)


170.33
136.82
136.39
135.95
135.52
135.17
131.51
128.84
126.42
126.37
126.32
126.27
125.22
121.61
117.99
77.58
77.16
76.74
40.34
O
O
S
H3C
N
CF3
3yc
f1 (ppm)


-63.32
O
O
S
H3C
N
CF3
3yc
50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -21
f1 (ppm)