Electronic Supporting Information

for

Access to N-Cyanosulfoximines by Transition Metal-Free Iminations of Sulfoxides

Carl Albrecht Dannenberg, Lars Fritze, Felix Krauskopf and Carsten Bolm*

Institute of Organic Chemistry, RWTH Aachen University, Landoltweg 1
52074 Aachen (Germany), Fax: (+49) 241-8092-391
E-mail: Carsten.Bolm@oc.rwth-aachen.de

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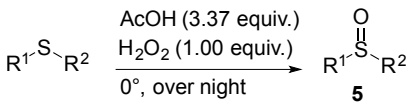
1. General Information

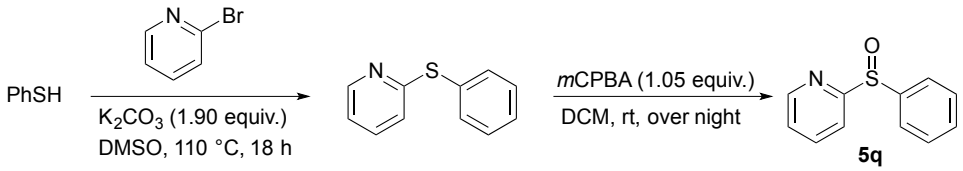
Unless otherwise stated, the starting materials were commercially available and used as received. ^{1}H, ^{13}C and ^{19}F NMR spectra were recorded either on Varian V-NMRS 600, Varian V-NMRS 400 or Varian Mercury 300 spectrometer, in $CDCl_3$. Chemical shifts (δ) are given in ppm relative to tetramethylsilane (TMS) and calibrated to residual chloroform peaks. Coupling constants (*J*) are reported in Hz and coupling patterns are described as br = broad, s = singlet, d = doublet, t = triplet, q = quartet, hept = heptet, td = triplet of doublets, m = multiplet. Infrared spectra were recorded on a Perkin Elmer FT-IR Spectrum 100 as KBr pellets or neat liquids; wave numbers are given in cm^{-1}. Mass spectra were recorded on a Finnigan SSQ 7000 spectrometer [electron ionization (EI), 70 eV] and peaks are listed according to their m/z values. High-resolution mass spectra (HRMS) were recorded on a Thermo Scientific LTQ Orbitrap XL spectrometer with positive ion mode. Elemental analyses were performed on an ElementarVario EL instrument. Melting points were measured with a Büchi Melting Point B-540 apparatus. Flash column chromatography was performed with Merck silica gel 60 (35–70 mesh). Reactions were monitored by thin layer chromatography (TLC) with aluminium sheets silica gel 60 F254 from Merck with detection by UV light and/or staining solutions ($KMnO_4$). Optical rotation was measured with a Perkin-Elmer model P241 (20 °C, λ = 589 nm). Specific rotations are reported as follows: $[\alpha]_D^{20}$ (c: g•100 mL^{-1}, in solvent). The unit is deg·cm^3·g^{-1}·dm^{-1}. The enantiomeric excess (*ee*) was determined by high-performance liquid chromatography (HPLC) on systems of an Agilent 1100 or 1200 series with chiral stationary phases (Chiralcel OB-H, Chiralpak AD-H) from Chiral Technologies Inc. The enantiomers were identified and confirmed by comparing their HPLC retention times with those of authentic enantiomerically pure compounds.

2. General procedure for the synthesis of sulfoxides 5



A 100 mL round-bottom flask, equipped with a magnetic stir bar, was charged with acetic acid (3.37 equiv.) and the corresponding sulfide (10.0 mmol, 1.00 equiv.) and cooled to 0 °C. Subsequently, hydrogen peroxide solution (30 wt%, 10.0 mmol, 1.00 equiv.) was added and the reaction mixture stirred over night at room temperature. After cooling to 0 °C, an aq. NaOH solution was added until the resulting mixture turned basic. The product was extracted with DCM, the organic phase was dried with $MgSO_4$, filtered and evaporated under reduced pressure. If necessary column chromatography was performed to purify the product.[1]

3. Synthesis of 2-(phenylsulfinyl)pyridine (5q)

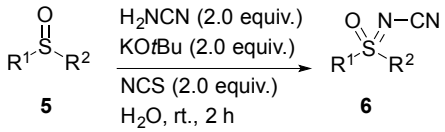


A 25 mL round-bottom flask, equipped with a magnetic stir bar, was charged with thiophenol (2.20 g, 20.0 mmol, 1.00 equiv.), 2-bromopyridine (1.92 mL, 20.0 mmol, 1.00 equiv.), K_2CO_3 (5.2 g, 38.0 mmol, 1.90 equiv.) and DMSO (16 mL). The solution was stirred for 18 h at 110 °C. Then, H_2O and DCM were added, the aqueous phase was extracted with DCM (3 x 50 mL), washed, dried with $MgSO_4$, filtered and purified by flash chromotography (80 g SiO_2, *n*-pentane:EtOAc 10:1) to yield 2.57 g (69%) of 2-(phenylthio)pyridine. A 250 mL round-bottom flask, equipped with a magnetic stir bar, was then charged with 2-(phenylthio)pyridine (2.57 g, 13.7 mmol, 1.00 equiv.) and DCM (140 mL). After cooling to 0 °C, 3-chloroperbenzoic acid (70 wt%, 2.49 g, 14.4 mmol, 1.05 equiv.) was slowly added and the mixture was stirred over night. A sat. aq. $NaHCO_3$ solution was then added (100 mL), and the product was extracted with DCM (3 x 50 mL). The organic phase was dried with $MgSO_4$, filtered and evaporated under reduced pressure. After flash chromotography (80 g SiO_2 *n*-pentane:EtOAc 5:1→1:1), 2.14 g (76%) 2-(phenylsulfinyl)pyridine (**5q**) was obtained as a colorless solid.[2]

Yield: 52% (over two steps), (eluent: *n*-pentane:EtOAc, 5:1) ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.27–7.31 (m, 1H), 7.40–7.48 (m, 3H), 7.75–7.81 (m, 2H), 7.84–7.89 (m, 1H), 8.00–8.01 (m, 1H), 8.53–8.56 (m, 1H) ppm; ^{13}C{^{1}H} NMR (151 MHz, $CDCl_3$) δ = 118.5, 124.8, 125.0, 129.3, 131.2, 138.2, 144.2, 149.9, 165.9 ppm.

The analytical data are in accordance with those reported in the literature.[2]

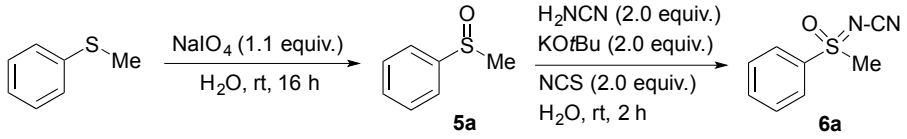
4. General procedure for the synthesis of *N*-cyanosulfoximines 6



A sealed tube, equipped with a magnetic stir bar, was charged with the respective sulfoxide **5** (0.20 mmol, 1.0 equiv.). Distilled water is added (1 mL). Then KO*t*Bu (45 mg, 0.40 mmol, 2.0 equiv.) followed by H_2NCN (17 mg, 0.40 mmol, 2.0 equiv.) were added and the mixture was stirred for 10 min. Subsequently, NCS (53 mg, 0.40 mmol, 2.0 equiv.) was added and the solution was stirred for 2 h at room temperature. The reaction mixture was transferred into a separating funnel containing DCM (15 mL) and distilled water (5 mL). Then, 1 M NaOH

(25 mL) was added into the separating funnel. The aqueous layer was extracted with DCM (3 x 10 mL), and the combined organic phase was dried over $MgSO_4$. After evaporation of the solvent the product was purified by column chromatography (8 g SiO_2, *n*-pentane:EtOAc, substrate dependent).

5. One-pot synthesis of 6a



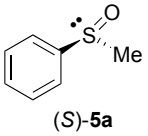
A sealed tube was charged with thioanisole (25 mg, 0.20 mmol, 1.0 equiv.), H_2O (1 mL) and $NaIO_4$ (45 mg, 0.21 mmol, 1.1 equiv.) and the mixture was stirred at room temperature for 16 h. Then, KO*t*Bu (45 mg, 0.40 mmol, 2.0 equiv.) followed by H_2NCN (17 mg, 0.40 mmol, 2.0 equiv.) was added and the mixture was stirred for 10 min. Subsequently, NCS (53 mg, 0.40 mmol, 2.0 equiv.) was added and the solution was stirred for 2 h at room temperature. The reaction mixture was transferred into a separating funnel containing DCM (15 mL) and distilled water (5 mL). Then, 1 M NaOH (25 mL) was added into the separating funnel. The aqueous layer was extracted with DCM (3 x 10 mL), and the combined organic phase was dried over $MgSO_4$. After evaporation of the solvent the product was purified by column chromatography (8 g SiO_2, *n*-pentane:EtOAc 2:1) to yield 31 mg (85%) of *N*-[methyl(oxo)(phenyl)-λ^6-sulfanylidene]cyanamide (**6a**) as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ = 3.34 (s, 3H), 7.68 (t, *J* = 7.8 Hz, 2H), 7.78 (t, *J* = 7.5 Hz, 1H), 7.99 (d, *J* = 7.3 Hz, 2H) ppm; ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ = 44.9, 111.9, 128.0, 130.4, 135.6, 136.1 ppm.

The analytical data are in accordance with those reported in the literature.[3]

6. Investigation of the stereochemistry

To investigate the stereochemistry of the described protocol, an enantiomerically enriched mixture of (*S*)-methylphenylsulfoxide **5a** was prepared after a procedure by Kagan and co-workers.[4]



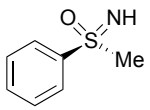
(*S*)-**5a**

$[\alpha]_D^{20}$ = –103.4 (c = 1.70 g•100 mL^{-1} in acetone), $[\alpha]_D^{22}$ = –140.2 [c = 2.05 g•100mL^{-1} in acetone, enantiopure (*S*)-enantiomer].[5] The enantiomeric excess (*ee*) was determined by HPLC (OB-H, heptane/*i*-PrOH = 70:30, 0.5 mL/min, λ = 210 nm, 20 °C). t_R (major) = 14.2 min, t_R (minor) = 24.0 min; *ee* = 72%. ^{1}H NMR (600 MHz,

$CDCl_3$) δ = 2.61 (s, 3H), 7.37–7.44 (m, 3H), 7.52–7.56 (m, 2H), ppm; $^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) δ = 43.8, 123.3, 129.2, 130.9, 145.5 ppm.

The analytical data are in accordance with those reported in the literature.[4]

The prepared sulfoxide (*S*)-**5a** was subjected to the procedure described under **4.** on a 2.0 mmol scale to prepare **6a**. Then, **6a** was converted to the corresponding *N*H-sulfoximine **9** after a procedure by Bolm and co-workers.[3] Measurements by chiral HPLC and polarimetric analysis revealed inversion of stereochemistry.



(*R*)-**9**

$[\alpha]_D^{20}$ = –25.6 (c = 1.11 g•100 mL^{-1} in acetone), $[\alpha]_D^{23}$ = –36.2 [c = 1.06 g•100mL^{-1} in acetone, enantiopure (*R*)-enantiomer].[6] The enantiomeric excess (*ee*) was determined by HPLC (AD-H, heptane/*i*-PrOH = 80:20, 0.6 mL/min, λ = 210 nm). t_R (major) = 19.1 min, t_R (minor) = 17.4 min; *ee* = 73%. ^1H NMR (600 MHz, $CDCl_3$) δ = 2.72 (brs, 1H), 3.05 (s, 3H), 7.47–7.52 (m, 2H), 7.54–7.59 (m, 1H), 7.96 (m, 2H), ppm; $^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) δ = 46.2, 127.7, 129.2, 133.0, 143.5 ppm.

The analytical data are in accordance with those reported in the literature.[3]

7. Characterisation data for sulfoxides 5

(Methylsulfinyl)benzene (5a)

^1H NMR (600 MHz, $CDCl_3$) δ = 2.70 (s, 3H), 7.45–7.54 (m, 3H), 7.61–7.65 (m, 2H) ppm. $^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) δ = 44.1, 123.6, 129.4, 131.1, 145.8 ppm.

The analytical data are in accordance with those reported in the literature.[7]

1-Methyl-4-(methylsulfinyl)benzene (5b)

Colorless solid, 1.052 g (99% yield), ^1H NMR (400 MHz, $CDCl_3$) δ = 2.41 (s, 3H), 2.69 (s, 3H), 7.30–7.34 (m, 2H), 7.51–7.55 (m, 2H) ppm; $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ = 21.5, 44.2, 123.7, 130.2, 141.6, 142.7 ppm.

The analytical data are in accordance with those reported in the literature.[7]

1-Methoxy-4-(methylsulfinyl)benzene (5c)

Colorless solid, 0.567 g (85% yield), ^{1}H NMR (400 MHz, $CDCl_3$) δ = 2.69 (s, 3H), 3.85 (s, 3H), 7.00–7.05 (m, 2H), 7.56–7.60 (m, 2H) ppm; $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) δ = 44.2, 55.7, 115.0, 125.6, 136.8, 162.1 ppm.

The analytical data are in accordance with those reported in the literature.[7]

1-Chloro-4-(methylsulfinyl)benzene (5d)

Colorless oil, 1.748 g (99% yield), ^{1}H NMR (400 MHz, $CDCl_3$) δ = 2.71 (s, 3H), 7.49–7.52 (m, 2H), 7.57–7.60 (m, 2H) ppm; $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) δ = 44.2, 125.1, 129.8, 137.4, 144.4 ppm.

The analytical data are in accordance with those reported in the literature.[7]

1-Bromo-4-(methylsulfinyl)benzene (5e)

Colorless solid, 1.304 g (99% yield), ^{1}H NMR (400 MHz, $CDCl_3$) δ = 2.71 (s, 3H), 7.50–7.54 (m, 2H), 7.65–7.69 (m, 2H) ppm; $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) δ = 44.2, 125.3, 125.6, 132.7, 145.0 ppm.

The analytical data are in accordance with those reported in the literature.[8]

1-Bromo-3-(methylsulfinyl)benzene (5f)

Colorless solid, 2.135 g (94% yield), ^{1}H NMR (400 MHz, $CDCl_3$) δ = 2.73 (s, 3H), 7.39 (t, *J* = 7.5 Hz, 1H), 7.52–7.55 (m, 1H), 7.60–7.64 (m, 1H), 7.79–7.81 (m, 1H) ppm; $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) δ = 44.2, 122.2, 123.7, 126.7, 130.9, 134.2, 148.0 ppm.

The analytical data are in accordance with those reported in the literature.[9]

1-Bromo-2-(methylsulfinyl)benzene (5g)

Colorless oil, 2.103 g (96% yield), ^{1}H NMR (400 MHz, $CDCl_3$) δ = 2.82 (s, 3H), 7.35–7.39 (m, 1H), 7.54–7.60 (m, 2H), 7.93–7.96 (m, 1H) ppm; $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) δ = 42.1, 118.6, 125.8, 128.9, 132.4, 133.0, 145.6 ppm.

The analytical data are in accordance with those reported in the literature.[9]

1-Fluoro-2-(methylsulfinyl)benzene (5h)

Colorless oil, 3.791 g (96% yield), ^{1}H NMR (400 MHz, $CDCl_3$) δ = 2.82 (s, 3H), 7.07–7.15 (m, 1H), 7.35–7.41 (m, 1H), 7.44–7.52 (m, 1H), 7.81–7.88 (m, 1H) ppm; $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) δ = 42.3, 115.8 (d, *J* = 20.3 Hz), 125.5 (m), 132.8 (d, *J* = 7.6 Hz), 133.0 (d, *J* = 16.9 Hz), 157.6 (d, *J* = 246.8 Hz) ppm. ^{19}F NMR (564 MHz, $CDCl_3$): δ = –114.8 (dt, *J* = 9.8, 6.2 Hz) ppm.

The analytical data are in accordance with those reported in the literature.[9]

1-(Methylsulfinyl)-4-nitrobenzene (5i)

Yellow solid, 393.6 mg (80% yield), ^{1}H NMR (400 MHz, $CDCl_3$) δ = 2.79 (s, 3H), 7.84 (d, *J* = 8.7 Hz, 2H), 8.40 (d, *J* = 8.7 Hz, 2H) ppm; $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) δ = 44.0, 124.7, 124.8, 144.1, 153.4 ppm.

The analytical data are in accordance with those reported in the literature.[10]

1-[4-(Methylsulfinyl)phenyl]ethan-1-one (5j)

Colorless solid, 188.3 mg (97% yield), ^{1}H NMR (400 MHz, $CDCl_3$) δ = 2.63 (s, 3H), 2.74 (s, 3H), 7.70–7.75 (m, 2H), 8.06–8.11 (m, 2H) ppm; $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) δ = 26.9, 43.9, 123.8, 129.2, 139.2, 151.0, 197.1 ppm.

The analytical data are in accordance with those reported in the literature.[11]

4-(Methylsulfinyl)benzaldehyde (5k)

Yellow solid, 104.0 mg (6% yield), ^{1}H NMR (600 MHz, $CDCl_3$) δ = 2.76 (s, 3H), 7.80 (d, *J* = 8.3 Hz, 2H), 8.02 (d, *J* = 8.3 Hz, 2H), 10.06 (s, 1H) ppm; $^{13}C\{^{1}H\}$ NMR (151 MHz, $CDCl_3$) δ = 43.9, 124.3, 130.5, 138.2, 152.5, 191.2 ppm.

The analytical data are in accordance with those reported in the literature.[12]

2-(Methylsulfinyl)pyridine (5l)

Colorless oil, 1.280 g (89% yield), ^{1}H NMR (400 MHz, $CDCl_3$) δ = 2.83 (s, 3H), 7.33–7.37 (m, 1H), 7.89–7.95 (m, 1H), 7.98–8.02 (m, 1H), 8.57–8.62 (m, 1H) ppm; $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) δ = 41.4, 119.3, 124.7, 138.2, 149.6, 166.1 ppm.

The analytical data are in accordance with those reported in the literature.[13]

(Cyclopropylsulfinyl)benzene (5m)

Colorless oil, 1.484 g (89% yield), ^{1}H NMR (400 MHz, $CDCl_3$) δ = 0.88–1.06 (m, 3H), 1.20–1.27 (m, 1H), 2.22–2.29 (m, 1H), 7.47–7.54 (m, 3H), 7.64–7.69 (m, 2H) ppm; $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) δ = 2.9, 3.5, 34.0, 124.1, 129.3, 131.0, 145.0 ppm.

The analytical data are in accordance with those reported in the literature.[8]

(Allylsulfinyl)benzene (5n)

Colorless oil, 188.3 mg (97% yield), ^{1}H NMR (400 MHz, $CDCl_3$) δ = 3.53 (qd, *J* = 12.6; 12.4; 7.5 Hz, 2H), 5.15–5.22 (m, 1H), 5.30–5.34 (m, 1H), 5.58–5.70 (m, 1H), 7.45–7.54 (m, 3H), 7.53–7.62 (m, 2H) ppm; $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) δ = 61.0, 124.0, 124.5, 125.4, 129.2, 131.2, 143.1 ppm.

The analytical data are in accordance with those reported in the literature.[8]

(Vinylsulfinyl)benzene (5o)

Yellow oil, 1.208 g (77% yield), ^{1}H NMR (600 MHz, $CDCl_3$) δ = 5.86–5.90 (m, 1H), 6.17–6.22 (m, 1H), 6.56–6.62 (m, 1H), 7.46–7.53 (m, 3H), 7.58–7.65 (m, 2H) ppm; $^{13}C\{^{1}H\}$ NMR (151 MHz, $CDCl_3$) δ = 120.8, 124.8, 129.6, 131.4, 143.2, 143.5 ppm.

The analytical data are in accordance with those reported in the literature.[14]

Sulfinyldibenzene (5p)

Colorless solid, 1.306 g (61% yield), ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.41–7.48 (m, 6H), 7.63–7.68 (m, 4H) ppm; $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) δ = 124.9, 129.5, 131.2, 145.8 ppm.

The analytical data are in accordance with those reported in the literature.[7]

(Methylsulfinyl)cyclohexane (5r)

Colorless liquid, 1.099 g (91% yield), ^{1}H NMR (600 MHz, $CDCl_3$) δ = 1.18–1.48 (m, 5H), 1.66–1.72 (m, 1H), 1.80–1.88 (m, 2H), 1.88–1.93 (m, 1H), 2.08–2.14 (m, 1H), 2.43–2.52 (m, 4H) ppm; $^{13}C\{^{1}H\}$ NMR (151 MHz, $CDCl_3$) δ = 24.9, 25.2, 25.5, 25.6, 26.1, 35.2, 60.9 ppm.

The analytical data are in accordance with those reported in the literature.[13]

Tetrahydrothiophene 1-oxide (5s)

Colorless liquid, 0.981 g (75% yield), ^{1}H NMR (400 MHz, $CDCl_3$) δ = 1.95–2.03 (m, 2H), 2.37–2.46 (m, 2H), 2.77–2.91 (m, 4H) ppm; $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) δ = 25.6, 54.6 ppm.

The analytical data are in accordance with those reported in the literature.[10]

8. Characterisation data for *N*-cyanosulfoximines 6

***N*-[Methyl(oxo)(phenyl)-λ^{6}-sulfanylidene]cyanamide (6a)**

Colorless solid, 33.5 mg (93% yield), (eluent: *n*-pentane:EtOAc 2:1), ^{1}H NMR (600 MHz, $CDCl_3$) δ = 3.34 (s, 3H), 7.65–7.70 (m, 2H), 7.75–7.80 (m, 1H), 7.80–7.96 (m, 2H) ppm; $^{13}C\{^{1}H\}$ NMR (151 MHz, $CDCl_3$) δ = 44.9, 111.9, 128.0, 130.4, 135.6, 136.1 ppm.

The analytical data are in accordance with those reported in the literature.[3]

***N*-[Methyl(oxo)(p-tolyl)-λ^{6}-sulfanylidene]cyanamide (6b)**

Colorless solid, 38.1 mg (98% yield), (eluent: *n*-pentane:EtOAc 2:1), ^{1}H NMR (600 MHz, $CDCl_3$) δ = 2.47 (s, 3H), 3.30 (s, 3H), 7.45 (d, *J* = 8.4 Hz, 2H), 7.84 (d, *J* = 8.4 Hz, 2H) ppm; $^{13}C\{^{1}H\}$ NMR (151 MHz, $CDCl_3$) δ = 21.8, 44.9, 112.1, 127.9, 130.9, 132.9, 147.1 ppm.

The analytical data are in accordance with those reported in the literature.[15]

***N*-[(4-Methoxyphenyl)(methyl)(oxo)-λ^{6}-sulfanylidene]cyanamide (6c)**

Colorless solid, M.P. 104 °C, 42.0 mg (98% yield), (eluent: *n*-pentane:EtOAc 2:1), ^{1}H NMR (600 MHz, $CDCl_3$) δ = 3.29 (s, 3H), 3.89 (s, 3H), 7.09 (d, *J* = 8.9 Hz, 2H), 7.88 (d, *J* = 8.9 Hz, 2H) ppm; $^{13}C\{^{1}H\}$ NMR (151 MHz, $CDCl_3$) δ = 45.2, 56.1, 112.3, 115.6, 126.6, 130.3, 165.2

ppm; IR (ATR): v [cm^{-1}] = 3373 (vw), 3098 (vw), 2921 (s), 2858 (m), 2584 (vw), 2183 (vw), 1897 (vw), 1737 (vw), 1580 (vs), 1474 (s), 1316 (m), 1240 (vs), 1085 (vs), 976 (vs), 809 (vs); MS (EI, 70 eV): *m/z* (%) = 209 (22) $[M]^+$, 169 (13) $[M\text{-}NCN]^+$, 154 (100) $[M\text{-}NCN, -CH_3]^+$, 147 (10), 139 (7), 123 (15), 95 (13), 92 (17), 78 (8), 77 (13), 69 (11), 64 (15), 63 (42), 57 (9), 55 (8), 50 (9); HRMS (ESI): $C_9H_{10}O_2N_2NaS$, $[M+Na]^+$ Calcd. 233.0351, Found 233.0355.

_N_-[(4-Chlorophenyl)(methyl)(oxo)-λ^6-sulfanylidene]cyanamide (6d)

Colorless oil, 37.8 mg (88% yield), (eluent: *n*-pentane:EtOAc 3:2), ^{1}H NMR (600 MHz, $CDCl_3$) δ = 3.33 (s, 3H), 7.63–7.67 (m, 2H), 7.90–7.95 (m, 2H) ppm; $^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) δ = 44.8, 111.6, 129.5, 130.7, 134.5, 142.7 ppm.

The analytical data are in accordance with those reported in the literature.[15]

_N_-[(4-Bromophenyl)(methyl)(oxo)-λ^6-sulfanylidene]cyanamide (6e)

Colorless solid, 43.8 mg (85% yield), (eluent: *n*-pentane:EtOAc 3:2), ^{1}H NMR (600 MHz, $CDCl_3$) δ = 3.34 (s, 3H), 7.80–7.87 (m, 4H) ppm; $^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) δ = 44.9, 111.6, 129.5, 131.4, 133.8, 135.1 ppm.

The analytical data are in accordance with those reported in the literature.[15]

_N_-[(3-Bromophenyl)(methyl)(oxo)-λ^6-sulfanylidene]cyanamide (6f)

Colorless solid, M.P. 111 °C, 36.8 mg (71% yield), (eluent: *n*-pentane:EtOAc 2:1), ^{1}H NMR (600 MHz, $CDCl_3$) δ = 3.36 (s, 3H), 7.55–7.58 (m, 1H), 7.89–7.94 (m, 2H), 8.09–8.14 (m, 1H) ppm; $^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) δ = 44.8, 111.4, 124.3, 126.6, 130.8, 131.8, 138.0, 138.7 ppm; IR (ATR): v [cm^{-1}] = 3786 (vw), 3366 (vw), 3063 (m), 2997 (m), 2913 (m), 2306 (vw), 2187 (vs), 1896 (vw), 1733 (w), 1568 (w), 1408 (s), 1325 (w), 1183 (vs), 1092 (vs), 978 (vs), 803 (vs), 732 (s), 668 (w); MS (EI, 70 eV): *m/z* (%) = 260 (15), 259 (73) $[M]^+$, 258 (15), 257 (71), 219 (96), 217 (96) $[M\text{-}NCN]^+$, 209 (10), 205 (86), 202 (87) $[M\text{-}NCN, -CH_3]^+$, 182 (10), 180 (33), 176 (20), 174 (22), 173 (10), 171 (11), 156 (40), 154 (63) $[M–SO(CH_3)(NCN)]^+$, 142 (8), 141 (14), 140 (14), 139 (64), 124 (9), 121 (7), 119 (15), 111 (22), 108 (24), 96 (46), 95 (29), 93 (17), 92 (12), 90 (7), 79 (12), 75 (100), 77 (14), 76 (81), 75 (93), 74 (66), 69 (21), 67 (23), 64 (11), 63 (69), 52 (14), 51 (46), 50 (99), 47 (12); CHN ($C_8H_7BrN_2OS$) Calcd. C: 37.08% H: 2.72% N: 10.81%; Found: C: 37.03% H: 2.90% N: 10.63%.

***N*-[(2-Fluorophenyl)(methyl)(oxo)-λ^6-sulfanylidene]cyanamide (6h)**

Yellow oil, 21.0 mg [53% yield; isolated product contains small amounts (<5%) of impurities (such as solvents)], (eluent: *n*-pentane:EtOAc 4:1), ^{1}H NMR (600 MHz, $CDCl_3$) δ = 3.49 (s, 3H), 7.37 (t, *J* = 16.1 Hz, 1H), 7.47 (t, *J* = 7.7 Hz, 1H), 7.78–7.83 (m, 1H), 8.02–8.06 (m, 1H) ppm; $^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) δ = 43.8 (d, *J* = 3.6 Hz), 111.2, 118.1 (d, *J* = 20.9 Hz), 124.2 (d, *J* = 13.8 Hz), 125.8 (d, *J* = 3.6 Hz), 130.7, 138.3 (d, *J* = 8.9 Hz), 159.0 (d, *J* = 257.5 Hz) ppm; ^{19}F NMR (564 MHz, $CDCl_3$) δ = –107.3 (m) ppm; IR (ATR): ν [cm^{-1}] = 3490 (vw), 3020 (w), 2926 (w), 2197 (vs), 1828 (vw), 1718 (vw), 1592 (m), 1470 (m), 1253 (vs), 1195 (vs), 1074 (m), 978 (s), 827 (vs), 766 (vs); MS (EI, 70 eV): *m/z* (%) = 199 (28), 198 (100) $[M]^+$, 157 (79) $[M\text{-}NCN]^+$, 143 (100) $[M\text{-}NCN, \text{-}CH_3]^+$, 140 (26), 130 (6), 125 (25), 115 (33), 112 (14), 109 (10), 97 (17), 95 (18), 85 (13), 83 (37), 81 (5), 77 (11), 75 (31), 74 (11), 69 (12), 57 (11), 51 (15), 50 (14), 45 (9).; HRMS (ESI): $C_8H_7ON_2FNaS$, $[M+Na]^+$ Calcd. 221.0192, Found 221.0155.

***N*-[Methyl(4-nitrophenyl)(oxo)-λ^6-sulfanylidene]cyanamide (6i)**

Colorless solid, 13.9 mg [31% yield; isolated product contains small amounts (<5%) of impurities (such as solvents)], (eluent: *n*-pentane:EtOAc 3:1), ^{1}H NMR (600 MHz, $CDCl_3$) δ = 3.43 (s, 3H), 8.21–8.25 (m, 2H), 8.51–8.54 (m, 2H) ppm; $^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) δ = 44.7, 110.8, 125.5, 129.8, 142.0, 151.9 ppm.

The analytical data are in accordance with those reported in the literature.[3]

***N*-[(4-Acetylphenyl)(methyl)(oxo)-λ^6-sulfanylidene]cyanamide (6j)**

Colorless solid, M.P. 121 °C, 29.8 mg (67% yield), (eluent: *n*-pentane:EtOAc 2:1), ^{1}H NMR (600 MHz, $CDCl_3$) δ = 2.67 (s, 3H), 3.38 (s, 3H), 8.09 (d, *J* = 8.5 Hz, 2H), 8.20 (d, *J* = 8.5 Hz, 2H) ppm; $^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) δ = 27.1, 44.6, 111.5, 128.5, 129.9, 139.8, 142.3, 196.3 ppm; IR (ATR): ν [cm^{-1}] = 3382 (vw), 3098 (vw), 2982 (w), 2901 (w), 2642 (vw), 2361 (vw), 2190 (vs), 1990 (vw), 1896 (vw), 1686 (s), 1569 (vw), 1488 (vw), 1399 (m), 1363 (w), 1302 (vw), 1245 (vs), 1191 (vs), 1100 (w), 1067 (vw), 970 (vs), 823 (vs), 706 (m); MS (EI, 70 eV): *m/z* (%) = 223 (9), 222 (57) $[M]^+$, 182 (34) $[M\text{-}NCN]^+$, 169 (10), 167 (100) $[M\text{-}NCN, \text{-}CH_3]^+$, 152 (27), 145 (5), 139 (12), 124 (9), 104 (8), 76 (9); CHN ($C_{10}H_{10}N_2O_2S$) Calcd. C: 54.04% H: 4.544% N: 12.60%; Found: C: 53.82% H: 4.456% N: 12.52%.

***N*-[(4-Formylphenyl)(methyl)(oxo)-λ^6-sulfanylidene]cyanamide (6k)**

Colorless oil, 22.5 mg (54% yield), (eluent: *n*-pentane:EtOAc 3:1), ^{1}H NMR (600 MHz, $CDCl_3$) δ = 3.40 (s, 3H), 8.17–8.20 (m, 4H), 10.16 (s, 1H) ppm; $^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) δ =

44.6, 111.3, 128.9, 131.0, 140.8, 141.1, 190.3 ppm; IR (ATR): v [cm^{-1}] = 3640 (vw), 3386 (vw), 3091 (vw), 3014 (w), 2922 (w), 2853 (w), 2389 (vw), 2195 (vs), 2142 (vw), 1995 (vw), 1945 (vw), 1705 (vs), 1578 (w), 1383 (m), 1324 (vw), 1302 (w), 1247 (vs), 1194 (vs), 1087 (s), 976 (vs), 910 (vw), 817 (vs), 770 (vs), 735 (s), 688 (s); MS (EI, 70 eV): *m/z* (%) = 209 (17), 208 (92) $[M]^+$, 169 (9), 168 (97) $[M\text{-}NCN]^+$, 154 (8), 152 (100) $[M\text{-}NCN, -CH_3]^+$, 152 (8), 146 (5), 125 (45), 124 (8), 121 (58), 97 (29), 91 (9), 77 (44), 76 (20), 75 (11), 74 (11), 65 (15), 63 (14), 51 (38), 50 (22); HRMS (ESI): $C_9H_8O_2N_2NaS$, $[M+Na]^+$ Calcd. 231.0199, Found 231.0199.

N-[Methyl(oxo)(pyridin-2-yl)-λ^6-sulfanylidene]cyanamide (6l)

Yellow oil, 25.0 mg (69% yield), (eluent: *n*-pentane:EtOAc 2:1), ^1H NMR (600 MHz, $CDCl_3$) δ = 3.51 (s, 3H), 7.67–7.70 (m, 1H), 8.06–8.11 (m, 1H), 8.20 (d, *J* = 8.0 Hz, 1H), 8.79 (d, *J* = 4.7 Hz, 1H) ppm; $^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) δ = 39.8, 111.6, 122.5, 128.9, 139.1, 150.9, 154.7 ppm; IR (ATR): v [cm^{-1}] = 3485 (vw), 3014 (m), 2922 (m), 2661 (vw), 2339 (vw), 2193 (vs), 1578 (m), 1425 (m), 1244 (vs), 975 (vs), 770 (vs); MS (EI, 70 eV): *m/z* (%) = 265 (12), 263 (16), 181 (24) $[M]^+$, 166 (4) $[M\text{-}CH_3]^+$, 141 (10) $[M\text{-}NCN]^+$, 86 (10), 84 (64), 82 (100), 78 (16) 48 (8), 47 (20); HRMS (ESI): $C_7H_7ON_3NaS$, $[M+Na]^+$ Calcd. 233.0120, Found 233.0202.

N-[Cyclopropyl(oxo)(phenyl)-λ^6-sulfanylidene]cyanamide (6m)

Colorless oil, (31.5 mg) 78% yield, (eluent: *n*-pentane:EtOAc 2:1), ^1H NMR (600 MHz, $CDCl_3$) δ = 1.10–1.14 (m, 1H), 1.29–1.36 (m, 2H), 1.66–1.70 (m, 1H), 2.67–2.73 (m, 1H), 7.62–7.67 (m, 2H), 7.73–7.78 (m, 1H), 7.92–7.96 (m, 2H) ppm; $^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) δ = 5.9, 7.1, 33.6, 112.4, 127.8, 130.0, 135.2, 136.3 ppm; IR (ATR): v [cm^{-1}] = 3881 (vw), 3476 (vw), 3039 (w), 2662 (vw), 2330 (vw), 2193 (vs), 2109 (w), 1998 (vw), 1805 (w), 1445 (m), 1244 (vs), 1184 (vs), 1081 (s), 881 (s), 831 (vs), 732 (vs), 686 (s); MS (EI, 70 eV): *m/z* (%) = 207 (11), 205 (41) $[M]^+$, 165 (15) $[M\text{-}NCN]^+$, 124 (100) $[M\text{-}NCN, -C_3H_5]^+$, 97 (26), 78 (8), 77 (35), 65 (6), 51 (25); HRMS (ESI): $C_{10}H_{10}ON_2NaS$, $[M+Na]^+$ Calcd. 229.0404, Found 229.0406.

N-[Allyl(oxo)(phenyl)-λ^6-sulfanylidene]cyanamide (6n)

Colorless oil, 22.7 mg [55% yield; isolated product contains small amounts (<5%) of impurities (such as solvents)], (eluent: *n*-pentane:EtOAc 2:1), ^1H NMR (600 MHz, $CDCl_3$) δ = 4.08–4.12 (m, 2H), 5.27 (d, *J* = 16.1 Hz, 1H), 5.47 (d, *J* = 10.4 Hz, 1H), 5.69–5.77 (m, 1H), 7.64–7.67 (m, 2H), 7.75–7.80 (m, 1H), 7.89–7.93 (m, 2H) ppm; $^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) δ = 61.2, 112.0, 122.2, 127.8, 129.1, 130.0, 133.7, 135.6 ppm; IR (ATR): v [cm^{-1}] = 3840 (vw), 3456 (vw), 3066 (vw), 2917 (w), 2335 (vw), 2193 (vs), 2116 (w), 1802 (w), 1635

(w), 1444 (m), 1243 (vs), 1186 (vs), 1089 (s), 948 (m), 826 (s), 755 (m); MS (EI, 70 eV): *m/z* (%) = 206 (5) $[M]^+$, 126 (8), 125 (100) [M-NCN, $-C_3H_5]^+$, 117 (8), 109 (7), 97 (52), 78 (21), 77 (89) $[C_6H_5]^+$, 65 (9) 57 (7), 51 (50), 50 (13); HRMS (ESI): $C_{10}H_{10}ON_2NaS$, $[M+Na]^+$ Calcd. 229.0402, Found 229.0202.

_N_-(Oxodiphenyl-λ^6-sulfanylidene)cyanamide (6p)

Colorless solid, M.P. 101 °C, 7.3 mg [15% yield; isolated product contains small amounts (<5%) of impurities (such as solvents)], (eluent: *n*-pentane:EtOAc 4:1), ^{1}H NMR (600 MHz, $CDCl_3$) δ = 7.58–7.63 (m, 4H), 7.67–7.71 (m, 2H), 7.98–8.02 (m, 4H) ppm; $^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) δ = 112.0, 128.1, 130.2, 134.9, 137.5 ppm; IR (ATR): ν [cm^{-1}] = 3790 (vw), 3698 (vw), 3375 (vw), 3073 (w), 2924 (m), 2647 (vw), 2288 (vw), 2189 (vs), 2008 (w), 1913 (vw), 1780 (vw), 1581 (vw), 1449 (s), 1180 (vs), 1085 (vs), 1000 (s), 835 (s), 735 (vs), 682 (vs); MS (EI, 70 eV): *m/z* (%) = 242 (32) $[M]^+$, 202 (32) $[M\text{-}NCN]^+$, 185 (6), 174 (16), 173 (14), 155 (9), 154 (73), 152 (9), 141 (11), 125 (18) $[C_6H_5SO]^+$, 109 (91) $[C_6H_5S]^+$, 97 (57), 93 (19), 92 (18), 78 (12), 77 (40), 69 (12), 65 (52), 57 (10), 51 (100); HRMS (ESI): $C_{13}H_{10}ON_2NaS$, $[M+Na]^+$ Calcd. 265.0403, Found 265.0406.

_N_-[Oxo(phenyl)(pyridin-2-yl)-λ^6-sulfanylidene]cyanamide (6q)

Colorless solid, M.P. 108 °C, 8.4 mg (17% yield), (eluent: *n*-pentane:EtOAc 4:1), ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.55–7.59 (m, 1H), 7.60–7.66 (m, 2H), 7.71–7.77 (m, 1H), 8.00–8.06 (m, 1H), 8.11–8.16 (m, 2H), 8.31–8.35 (m, 1H), 8.70–8.74 (m, 1H) ppm; $^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) δ = 111.8, 123.3, 128.2, 129.6, 129.9, 134.4, 135.4, 138.9, 151.2, 155.7 ppm; IR (ATR): ν [cm^{-1}] = 3808 (vw, 2959 (m), 2656 (vw), 2194 (s), 1739 (vs), 1573 (w), 1430 (s), 1368 (s), 1215 (vs), 1085 (vs), 808 (vs); MS (EI, 70 eV): *m/z* (%) = 244 (27) $[M]^+$, 243 (16), 203 (25) $[M\text{-}NCN]^+$, 202 (8), 187 (9), 186 (63) [Ph–S–Py]$^+$, 170 (29), 156 (13), 155 (100), 154 (14), 125 (25), 109 (14), 97 (20), 78 (59), 77 (26), 52 (12), 51 (62), 50 (13); HRMS (ESI): $C_{12}H_9ON_3NaS$, $[M+Na]^+$ Calcd. 266.0356, Found 266.0359.

_N_-[Cyclohexyl(methyl)(oxo)-λ^6-sulfanylidene]cyanamide (6r)

Colorless oil, 17.9 mg [48% yield; isolated product contains small amounts (<5%) of impurities (such as solvents)], (eluent: *n*-pentane:EtOAc 1:1), ^{1}H NMR (600 MHz, $CDCl_3$) δ = 1.33–1.43 (m, 2H), 1.55–1.62 (m, 4H), 1.99–2.04 (m, 2H), 2.26–2.33 (m, 2H), 3.12 (s, 3H), 3.19–3.25 (m, 1H) ppm; $^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) δ = 24.6, 24.9, 25.5, 36.8, 64.3, 112.2 ppm; IR (ATR): ν [cm^{-1}] = 3881 (vw), 3478 (vw), 2931 (vs), 2864 (m), 2336 (vw), 2189

(vs), 1722 (w), 1638 (w), 1451 (m), 1235 (vs), 967 (s), 820 (vs); MS (EI, 70 eV): *m/z* (%) = 187 (16) $[M]^+$, 171 (7) $[M\text{-}CH_3]^+$, 105 (21), 104 (11), 89 (8), 83 (68) $[C_6H_{11}]^+$. 81 (18), 79 (9), 67 (16), 63 (24), 57 (11), 55 (100), 53 (13); HRMS (ESI): $C_8H_{14}ON_2NaS$, $[M+Na]^+$ Calcd. 209.0714, Found 209.0719.

N-(1-Oxidotetrahydro-1-λ^6-thiophen-1-ylidene)cyanamide (6s)

Colorless solid, 14.4 mg (50% yield), (eluent: *n*-pentane:EtOAc 1:1), ^{1}H NMR (600 MHz, $CDCl_3$) δ = 2.26–2.43 (m, 4H), 3.25–3.33 (m, 2H), 3.48–3.56 (m, 2H) ppm; $^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) δ = 23.6, 53.0, 112.5 ppm.
The analytical data are in accordance with those reported in the literature.[3]

N-[Dimethyl(oxo)-λ^6-sulfanylidene]cyanamide (6t)

Prepared on a 5.0 mmol scale, colorless solid, M.P. 85–86 °C, 133.8 mg (23% yield), (eluent: *n*-pentane:EtOAc 1:1), ^{1}H NMR (400 MHz, $CDCl_3$) δ = 3.34 (s, 6H). ppm; $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ = 111.9, 42.7 ppm; IR (ATR): v [cm^{-1}] = 3953 (vw), 3390 (vw), 3009 (s), 2919 (w), 2644 (vw), 2342 (vw), 2187 (vs), 1929 (vw), 1712 (vw), 1627 (vw), 1508 (vw), 1412 (w), 1333 (w), 1192 (vs), 1029 (s), 942 (s), 809 (vs), 693 (w); MS (EI, 70 eV): *m/z* (%) = 120 (6), 119 (47), 118 (100) $[M]^+$, 78 (22), 63 (35), 61 (7), 48 (6), 47 (5), 46 (10), 45 (19); CHN ($C_3H_6N_2OS$) Calcd. C: 30.50% H: 5.12% N: 23.71%; Found: C: 30.70% H: 5.14% N: 23.63%.

9. References

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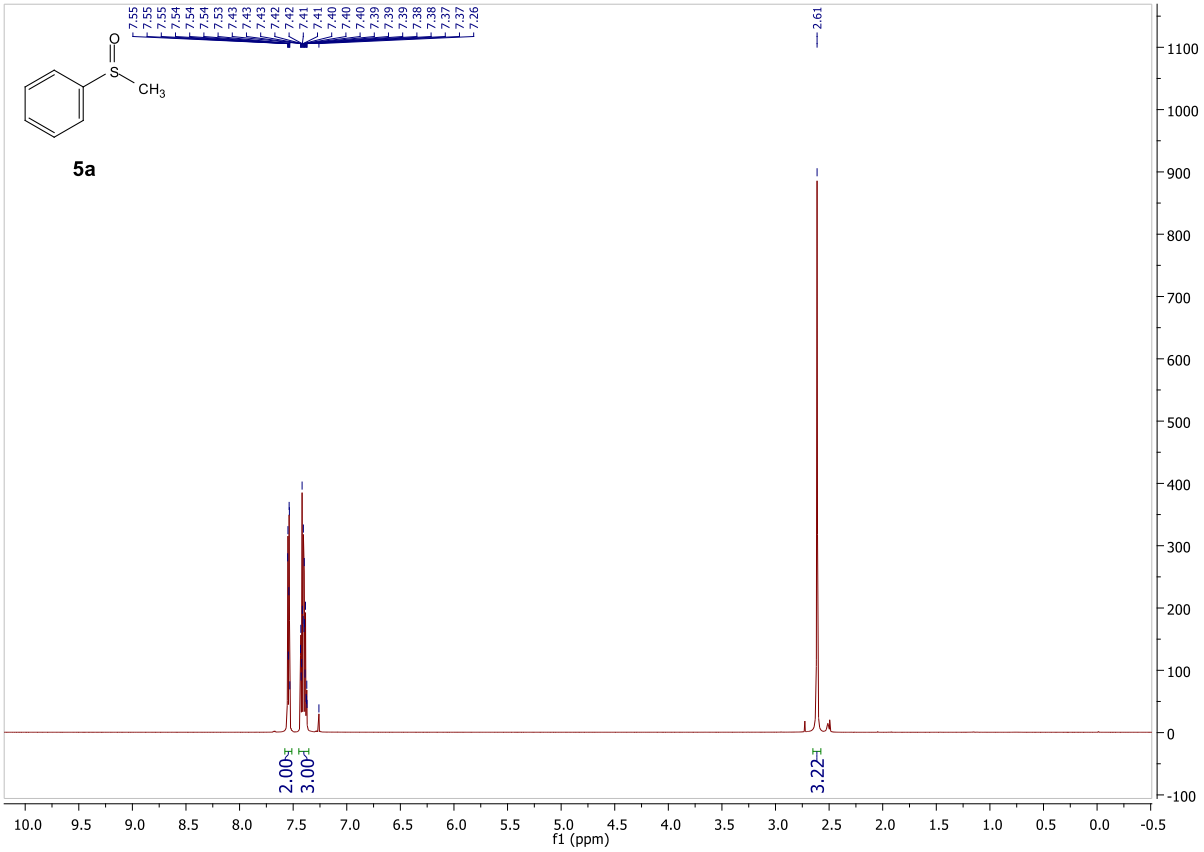
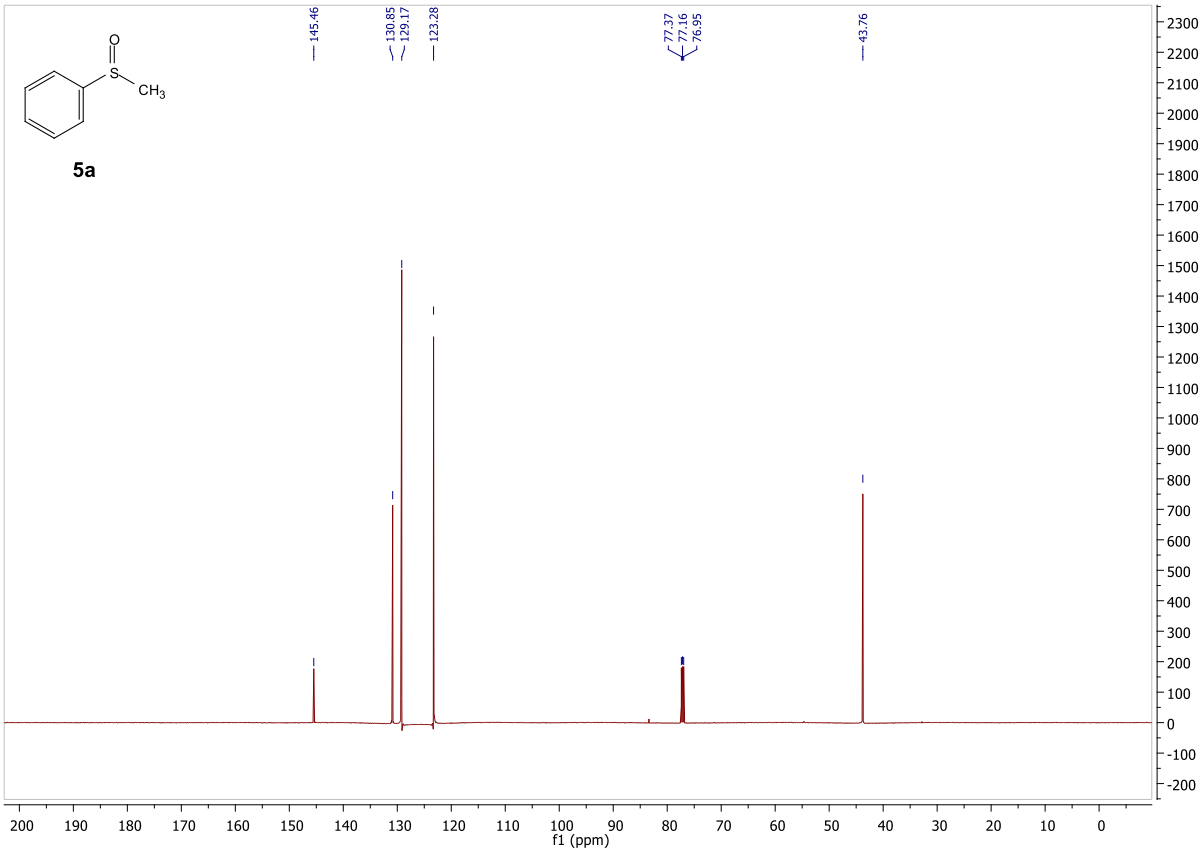
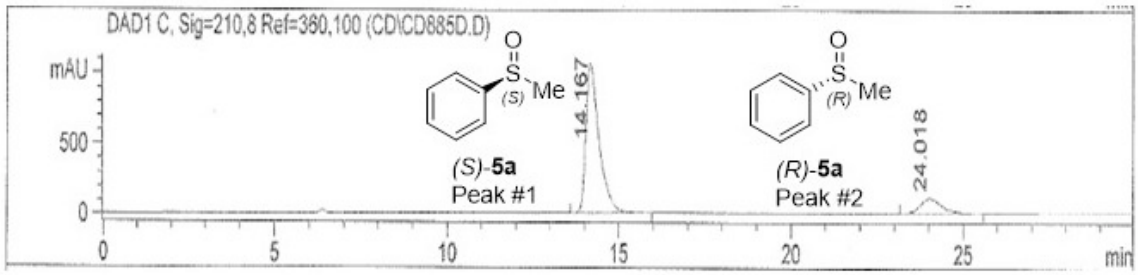
10. NMR Spectra and HPLC chromatograms







DAD1 C, Sig=210,8 Ref=360,100

Peak #	Ret. Time in min	Width in min	Height in mAU	Area in mAU*s	Area %
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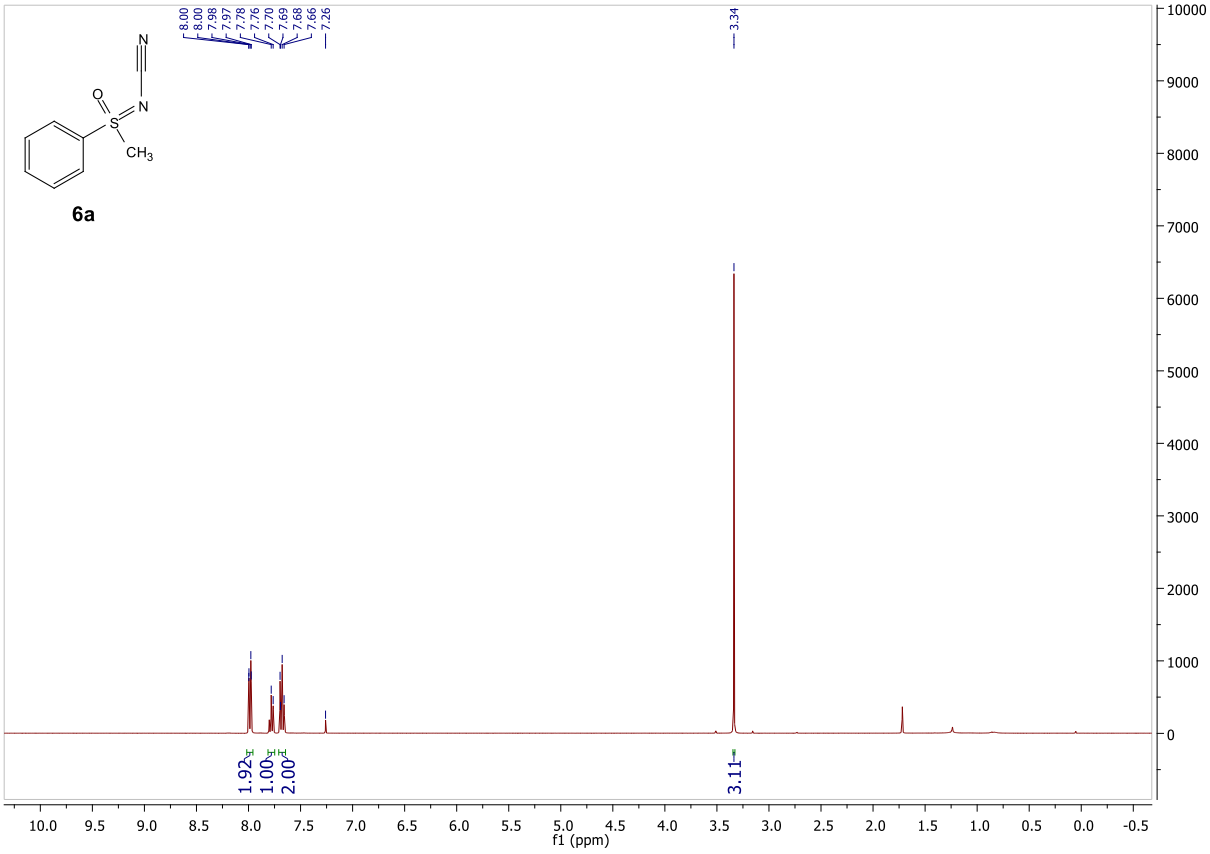
Total				33282.49707	100.0000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7.97
7.78
7.76
7.70
7.69
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7.66
7.26
3.34
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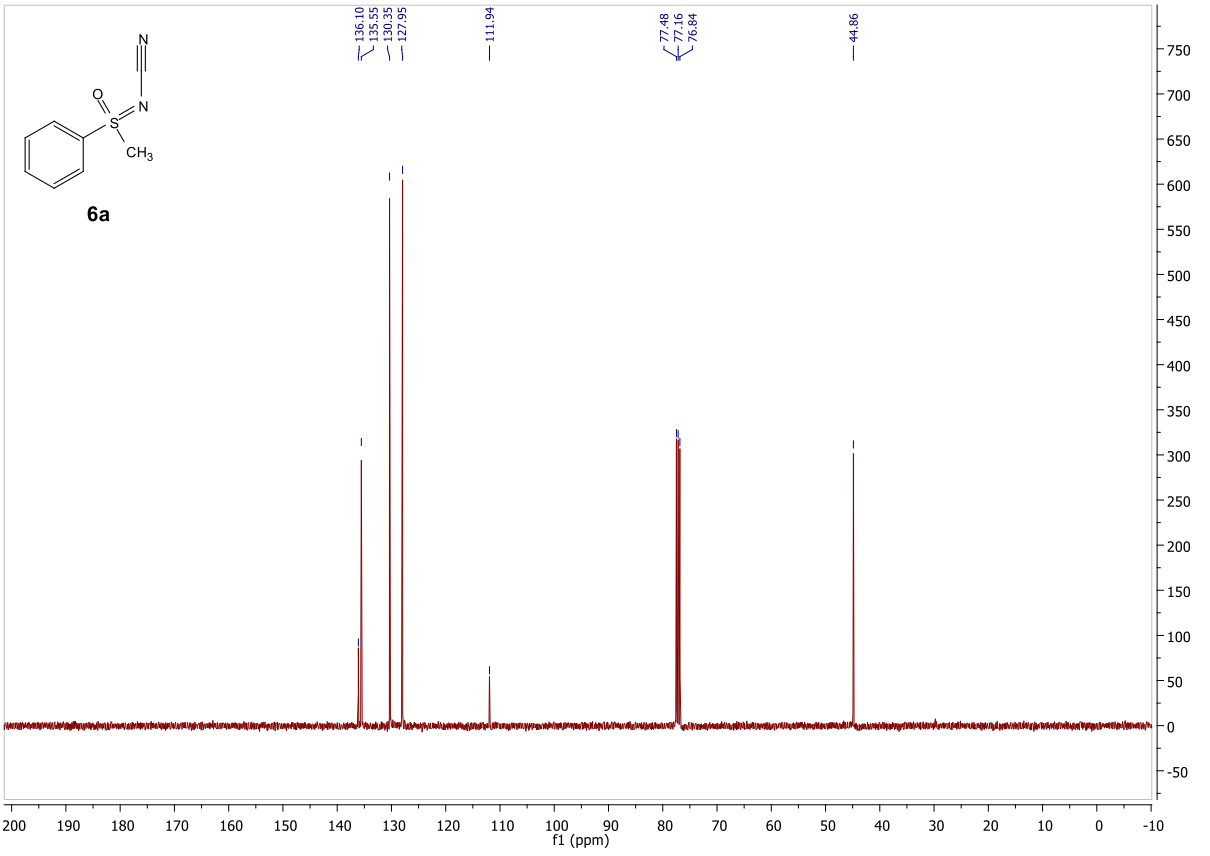
1.92
1.00
2.00
3.11
f1 (ppm)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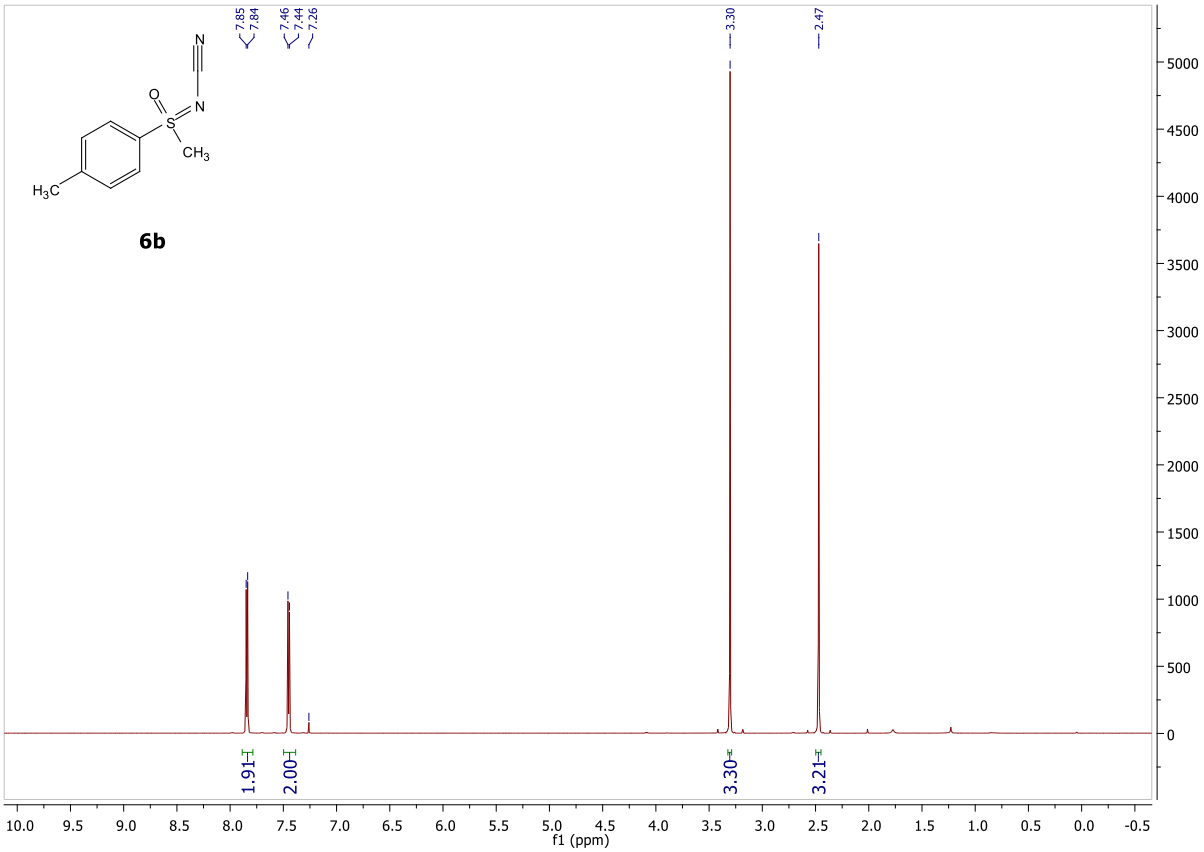
f1 (ppm)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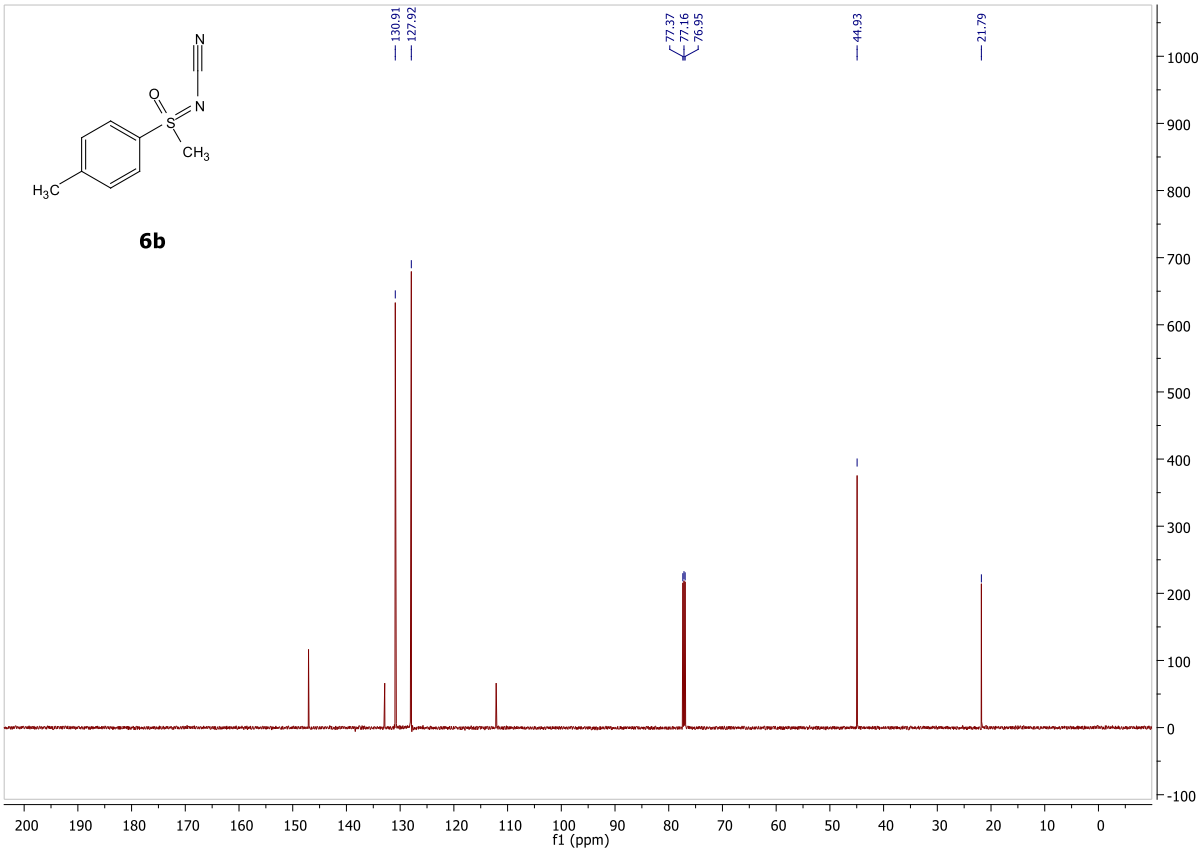
3.21
f1 (ppm)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6b
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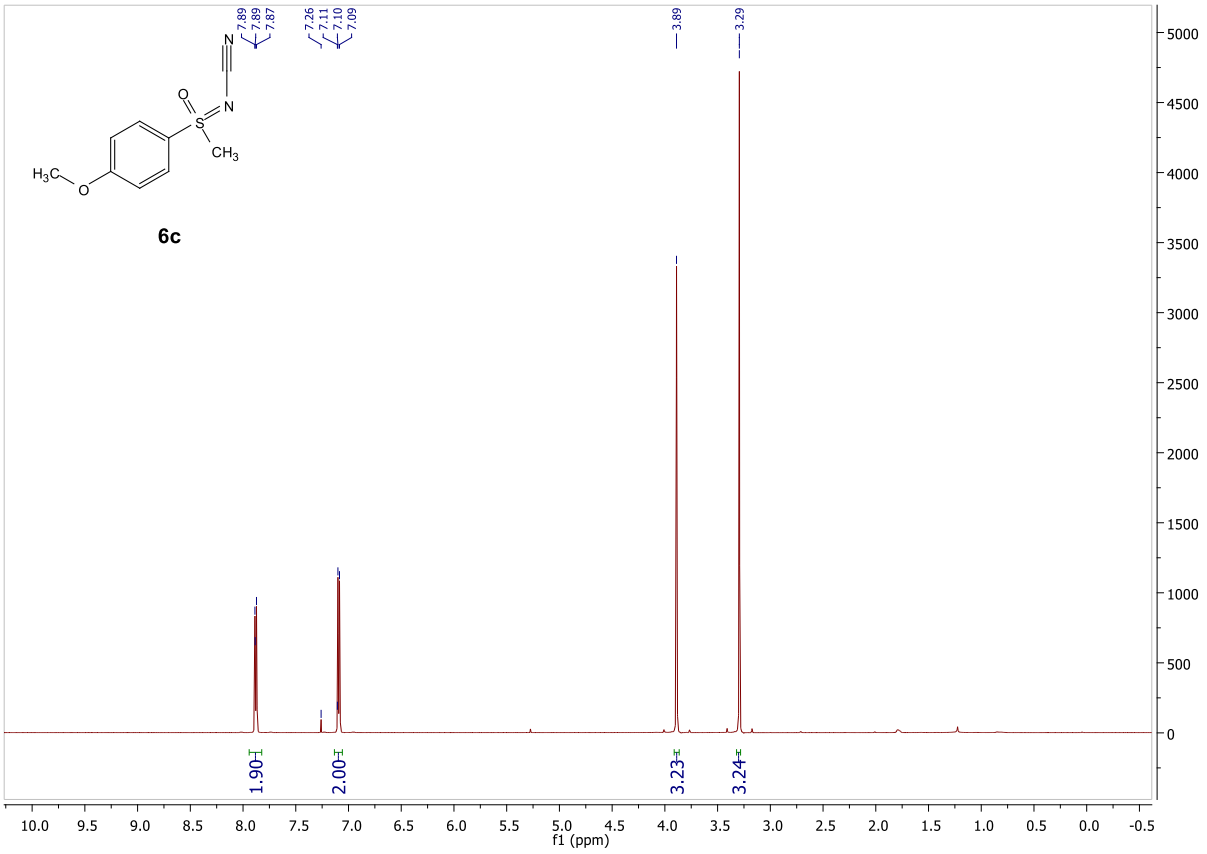
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7.89
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7.10
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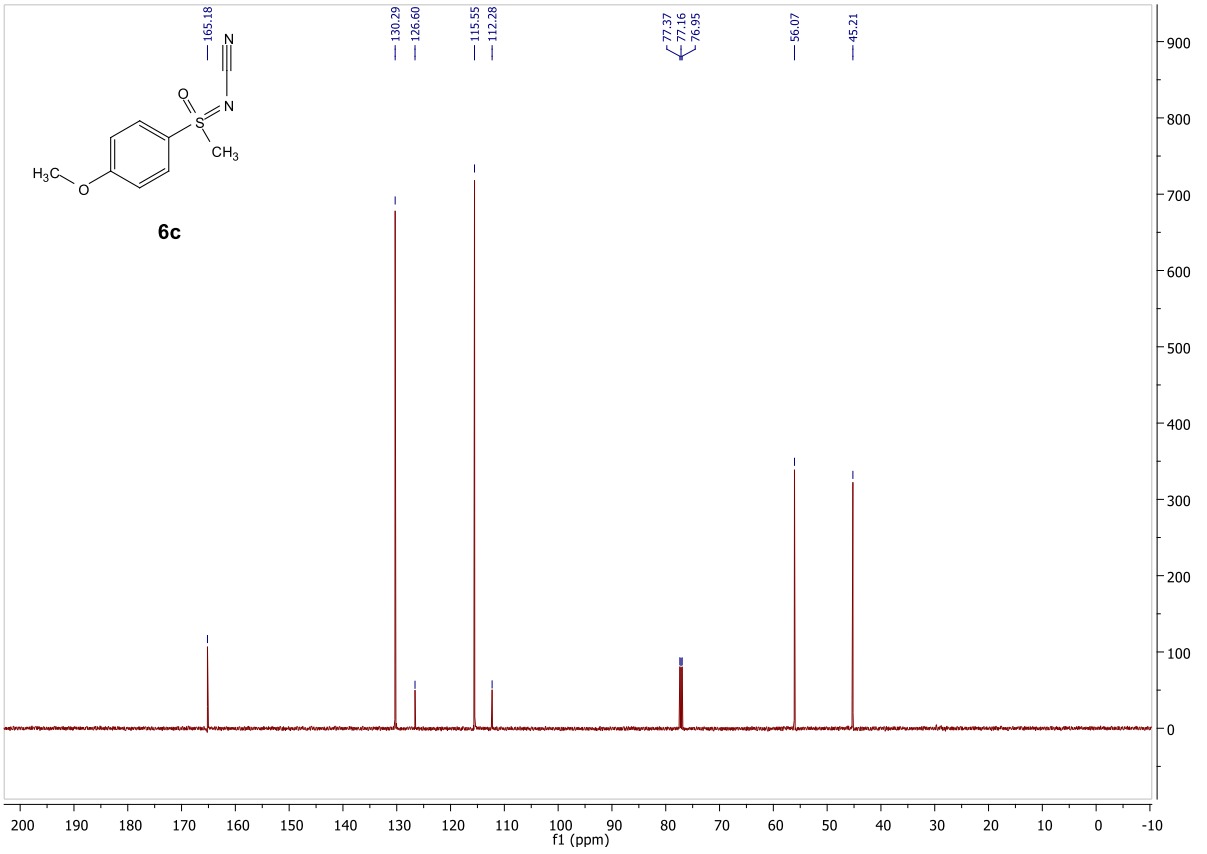
2.00
3.23
3.24
f1 (ppm)

6c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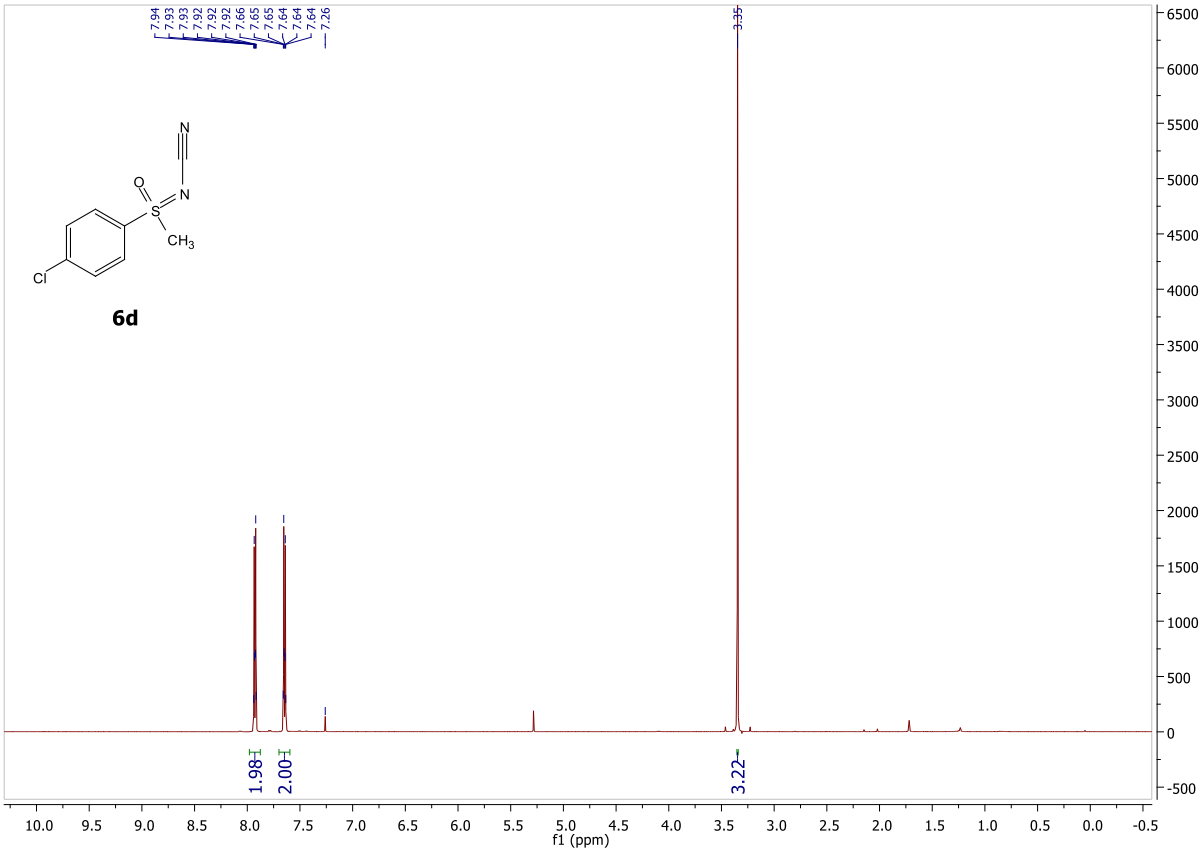
6c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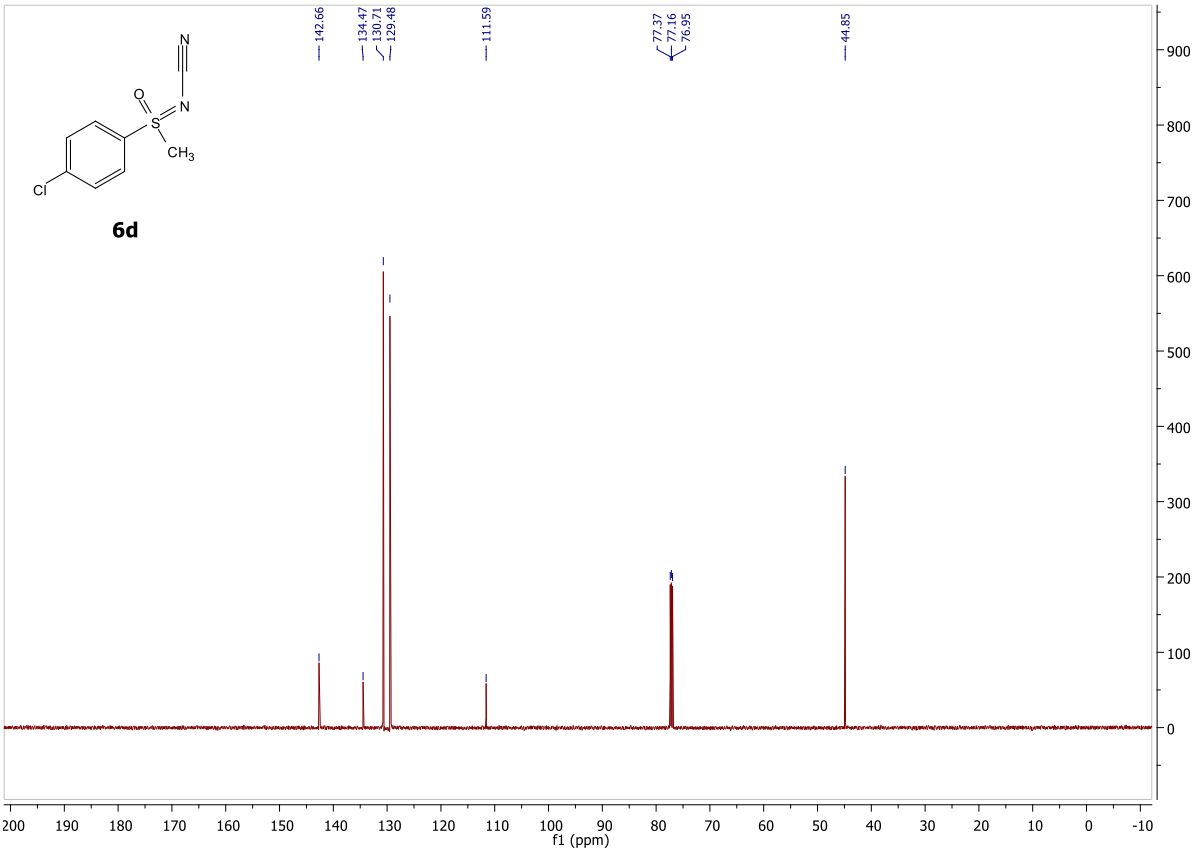
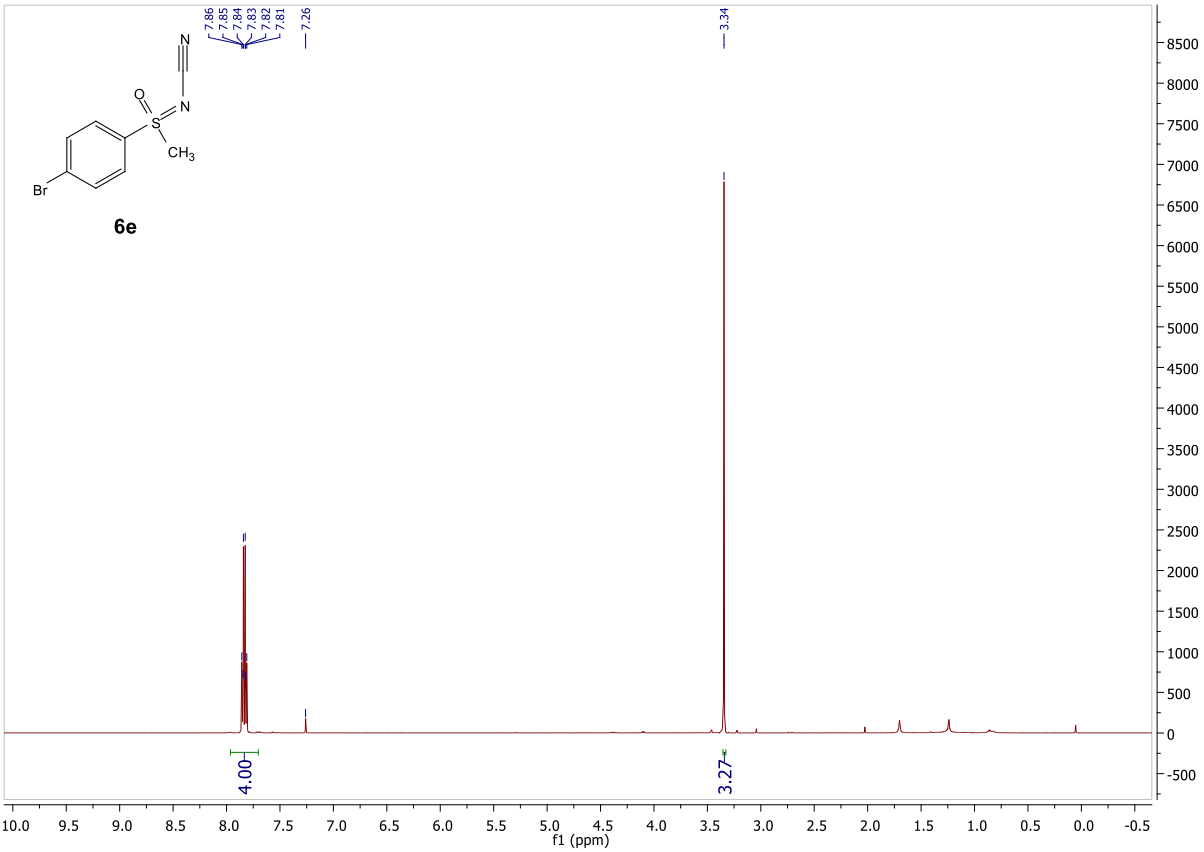
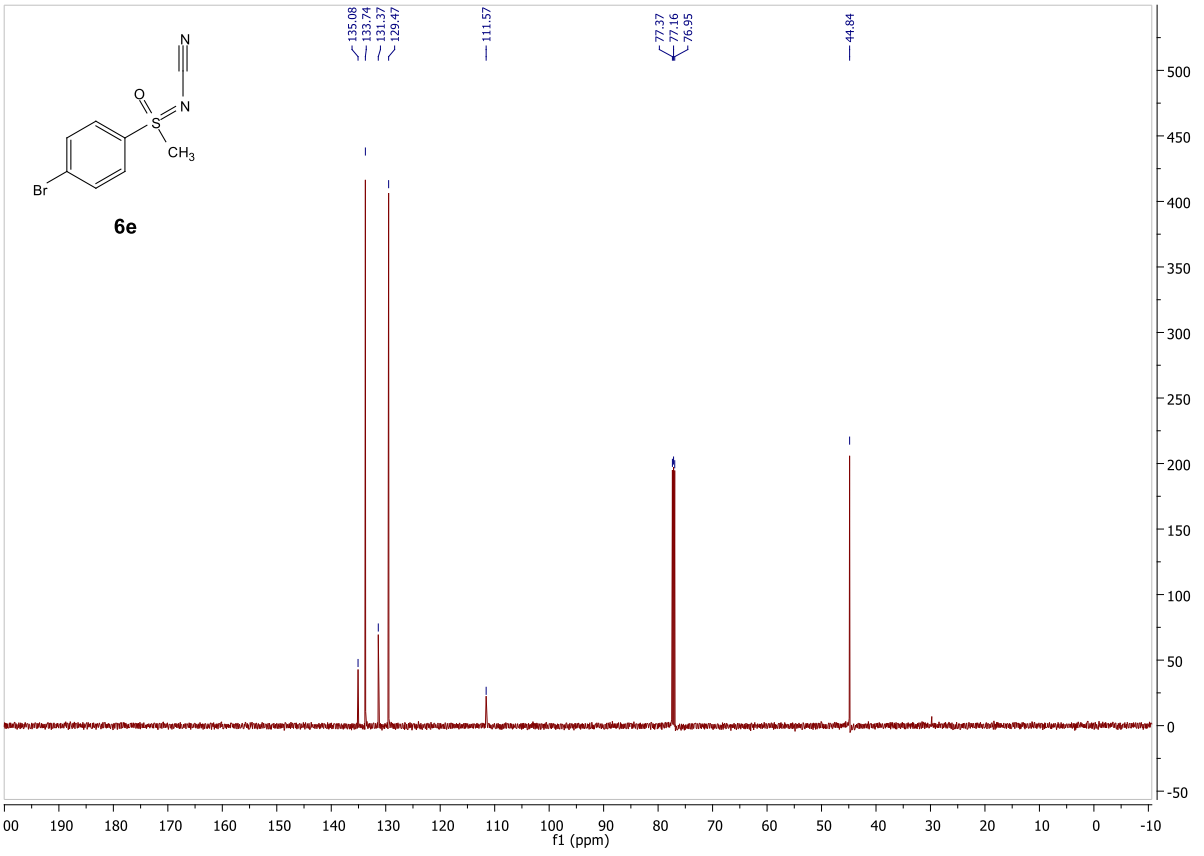
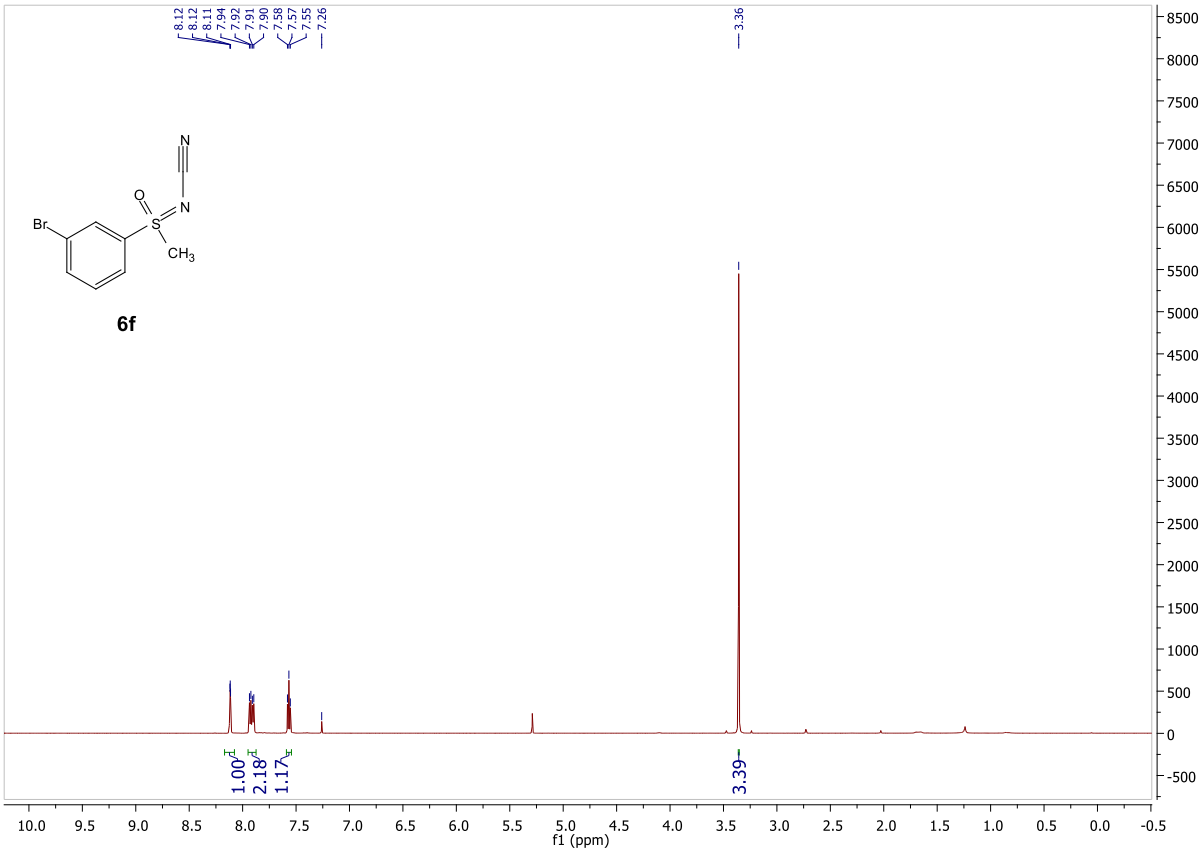
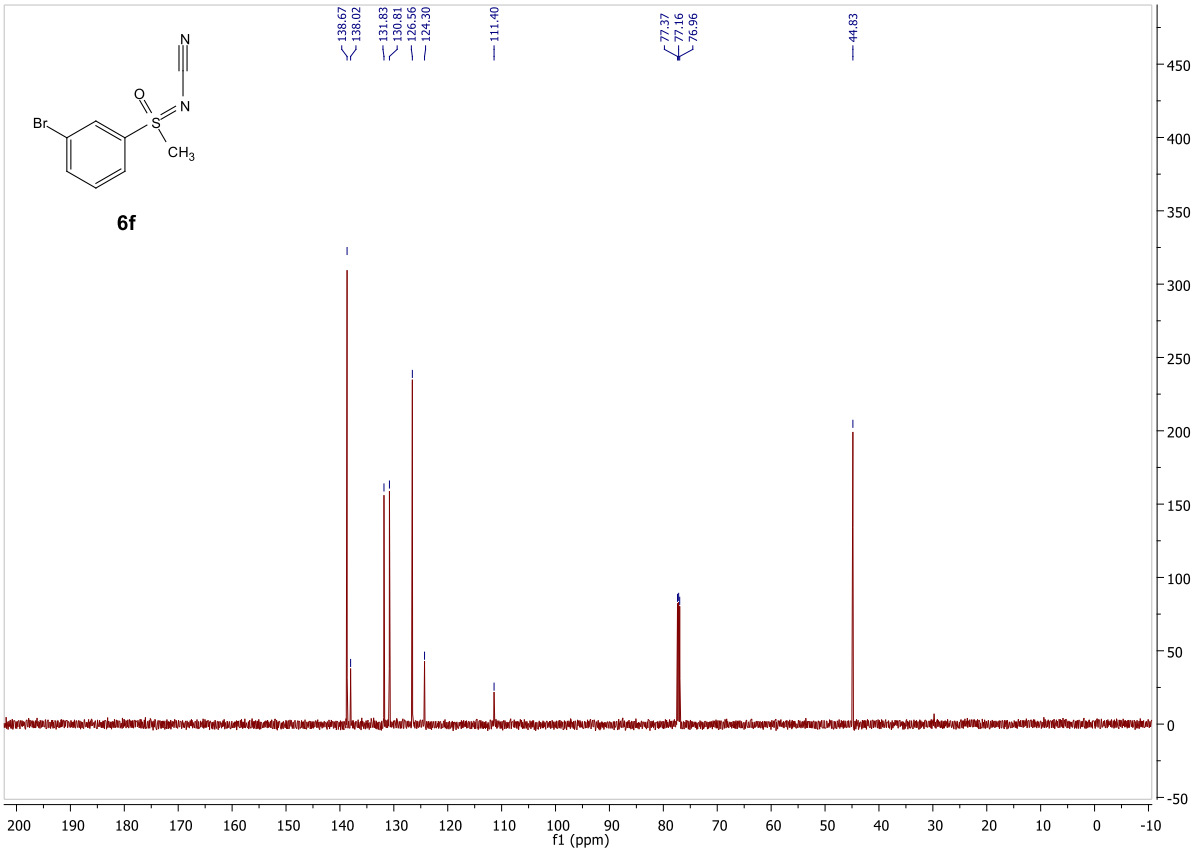
7.26
3.35
N
O
N
S
CH3
Cl
6d
1.98
2.00
3.22
f1 (ppm)

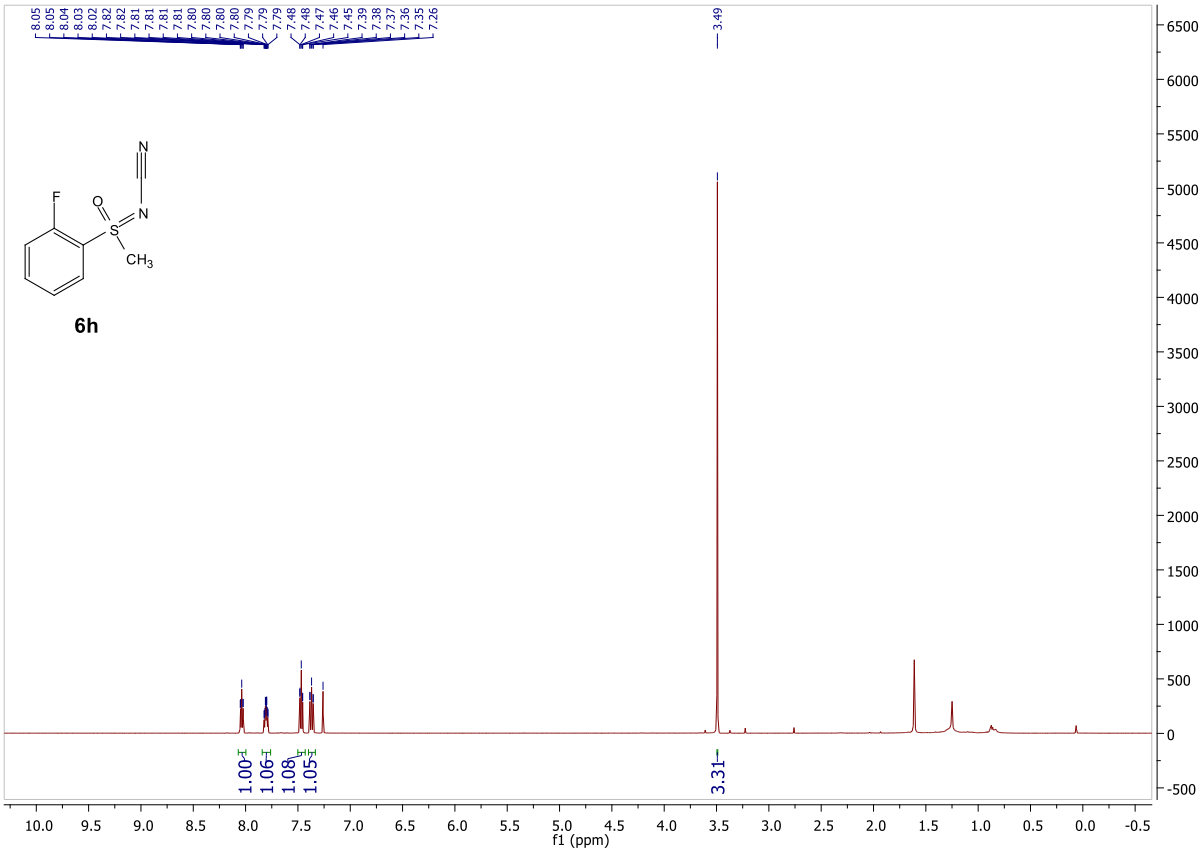

142.66
134.47
130.71
129.48
111.59
77.37
77.16
76.95
44.85
N
O
N
S
CH3
Cl
6d
f1 (ppm)

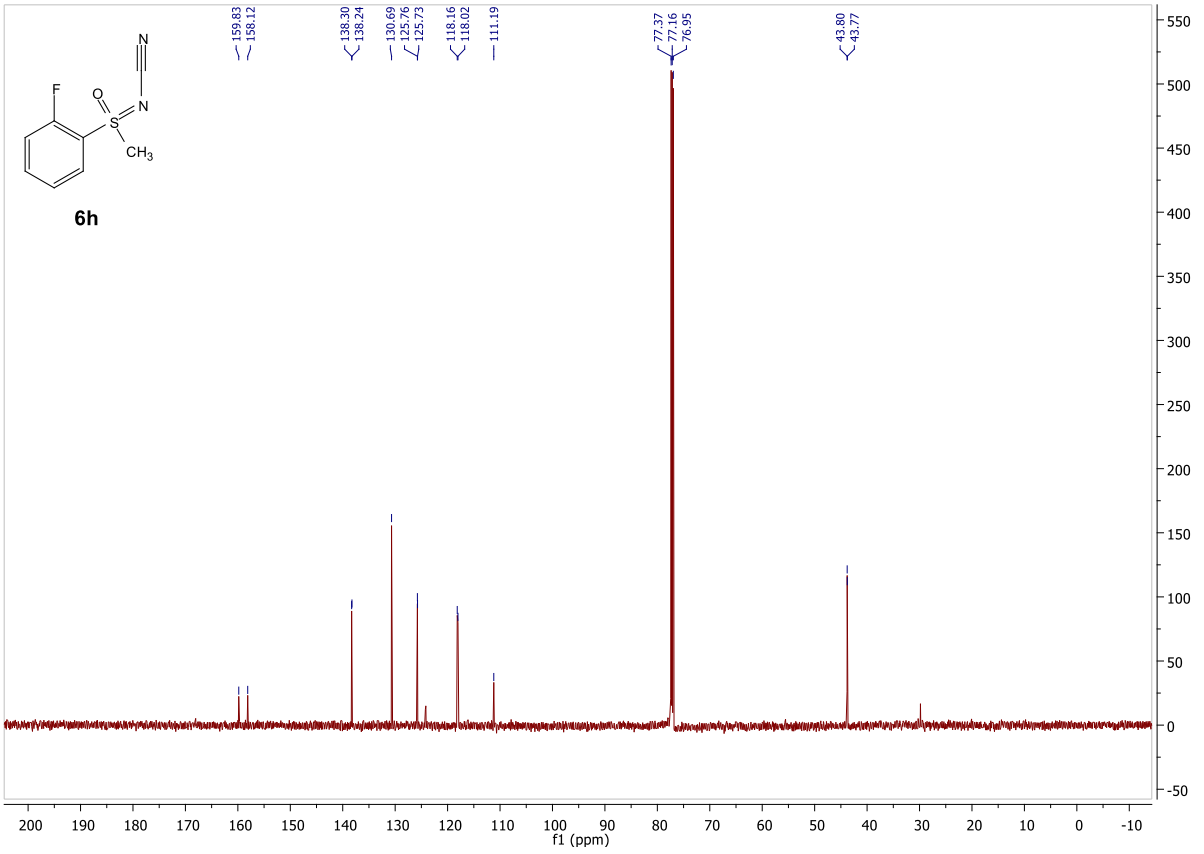

7.86
7.85
7.84
7.83
7.82
7.81
7.26
3.34
N
O
N
S
CH3
Br
6e
4.00
3.27
f1 (ppm)

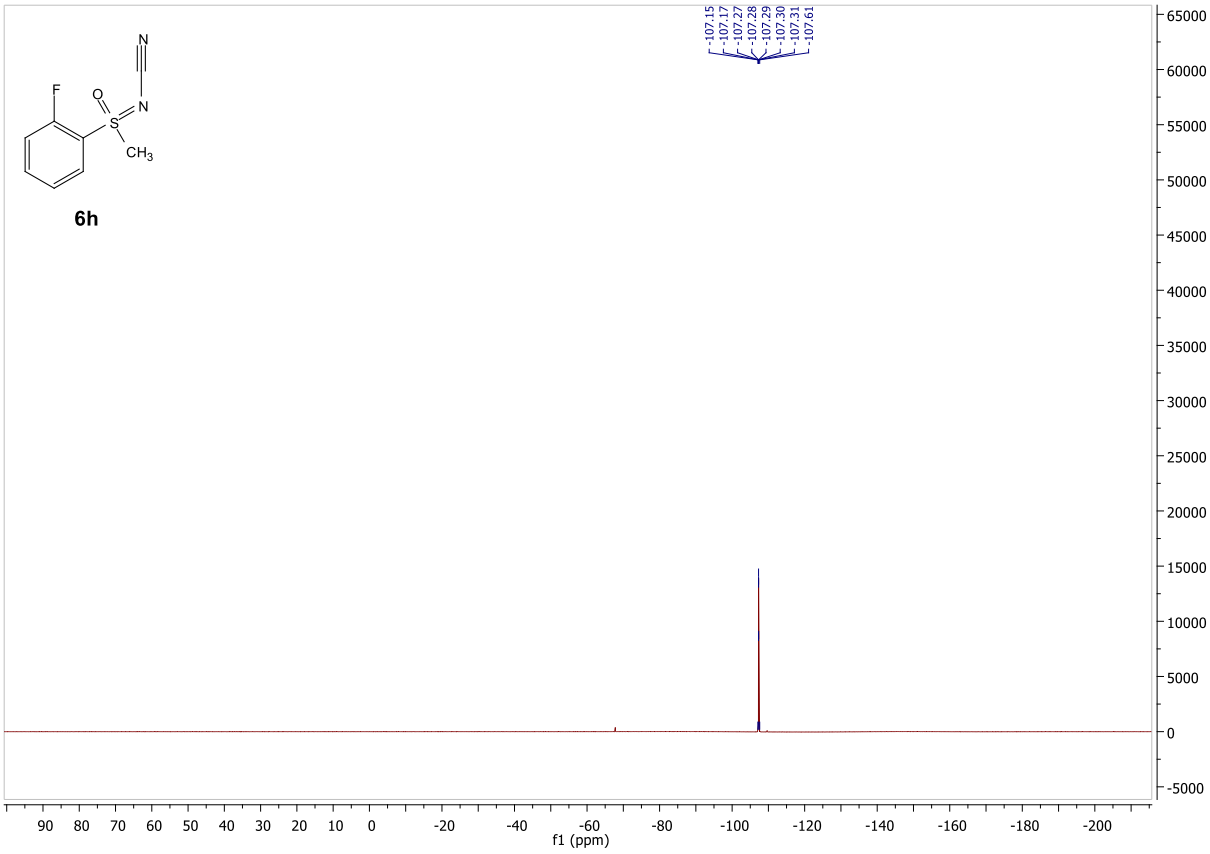

135.08
133.74
131.37
129.47
111.57
77.37
77.16
76.95
44.84
N
O
N
S
CH3
Br
6e
f1 (ppm)

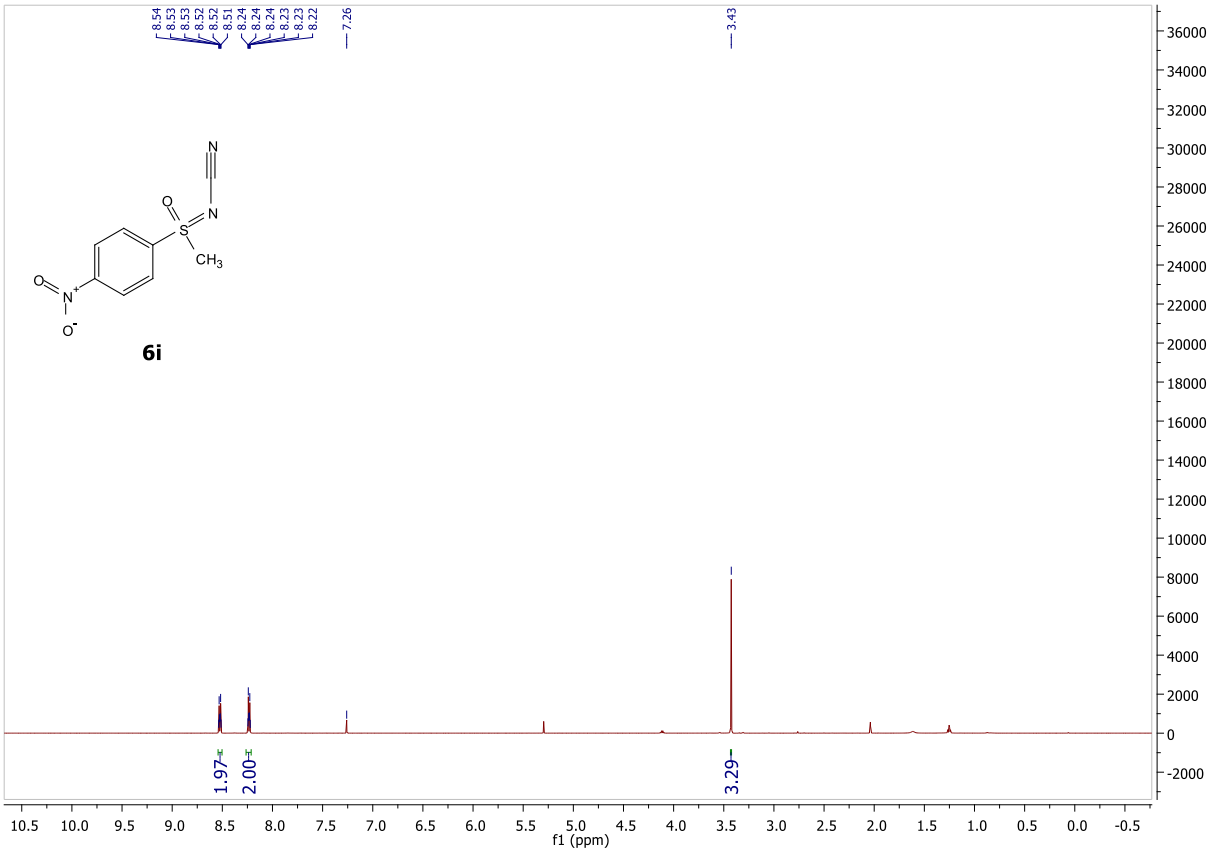

8.12
8.12
8.11
7.94
7.92
7.91
7.90
7.58
7.57
7.55
7.26
3.36
N
O
N
S
Br
CH3
6f
1.00
2.18
1.17
3.39
f1 (ppm)

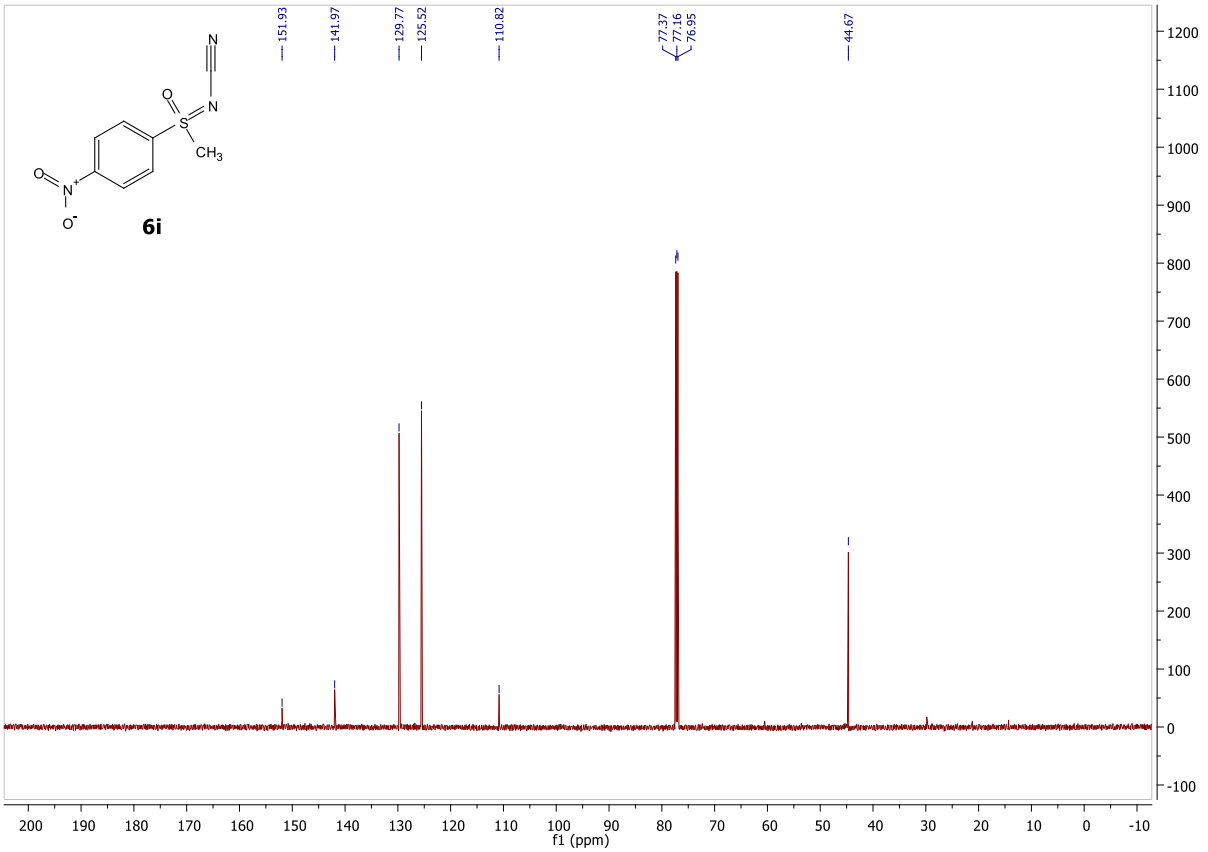

138.67
138.02
131.83
130.81
126.56
124.30
111.40
77.37
77.16
76.96
44.83
N
O
N
S
Br
CH3
6f
f1 (ppm)

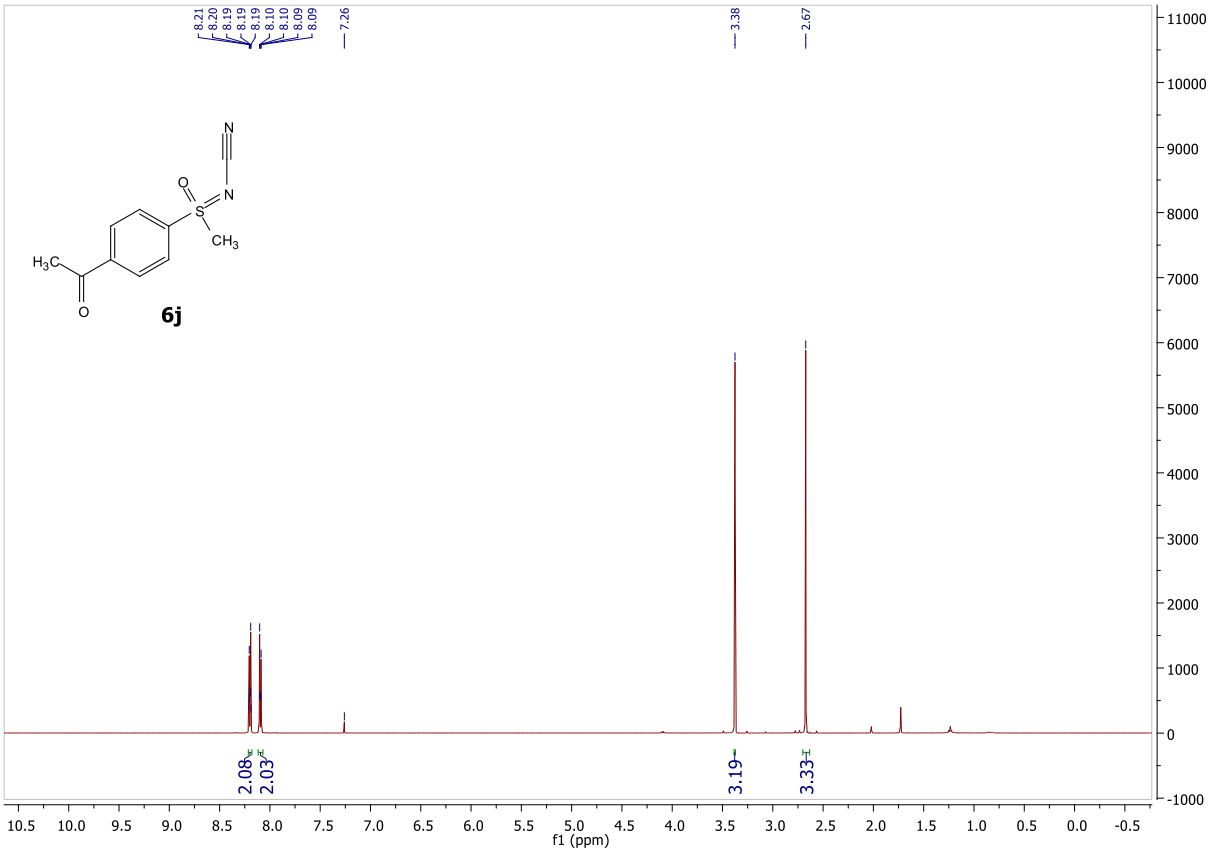

8.05
8.05
8.04
8.03
8.02
7.82
7.82
7.81
7.81
7.81
7.80
7.80
7.80
7.80
7.79
7.79
7.79
7.48
7.47
7.46
7.45
7.39
7.38
7.37
7.36
7.35
7.26
3.49
F
O
S
N
N
CH3
6h
1.00
1.06
1.08
1.05
3.31
f1 (ppm)

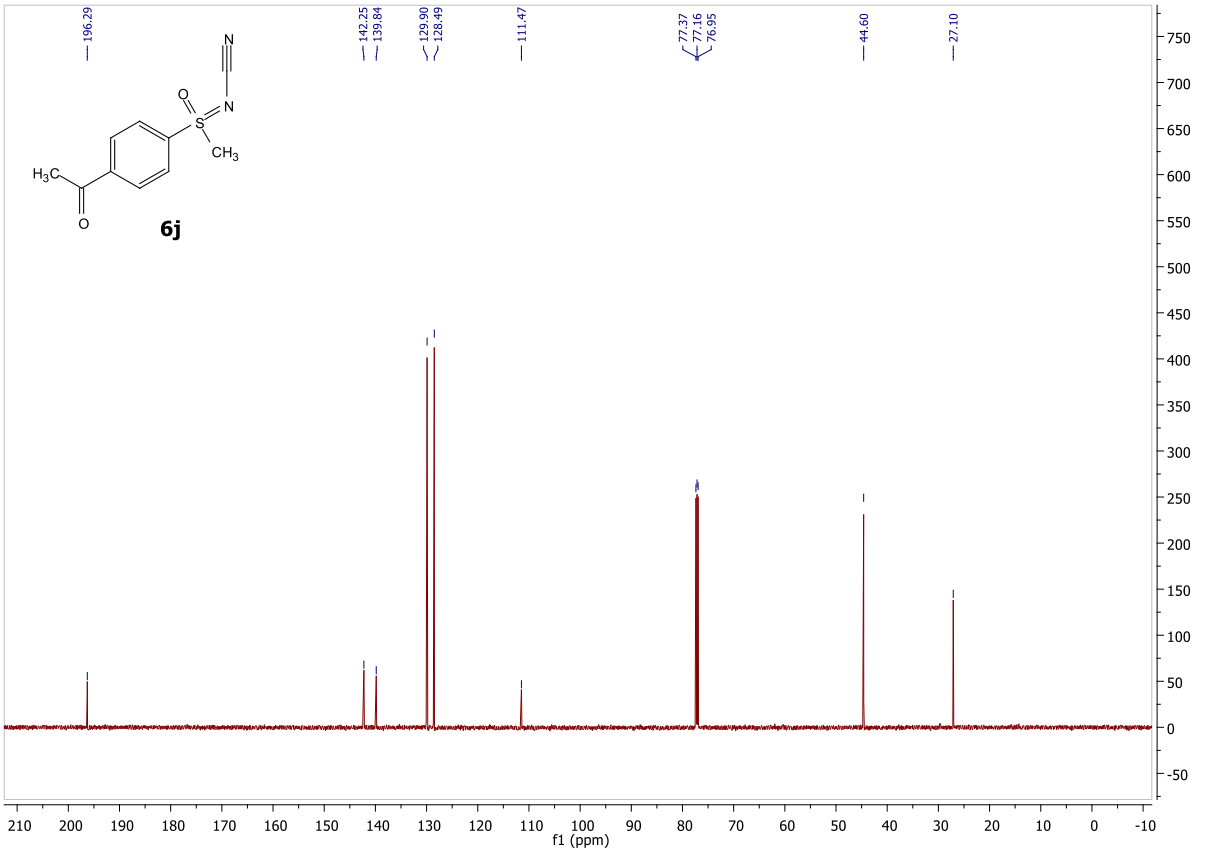

159.83
158.12
138.30
138.24
130.69
125.76
125.73
118.16
118.02
111.19
77.37
77.16
76.95
43.80
43.77
F
O
S
N
N
CH3
6h
f1 (ppm)

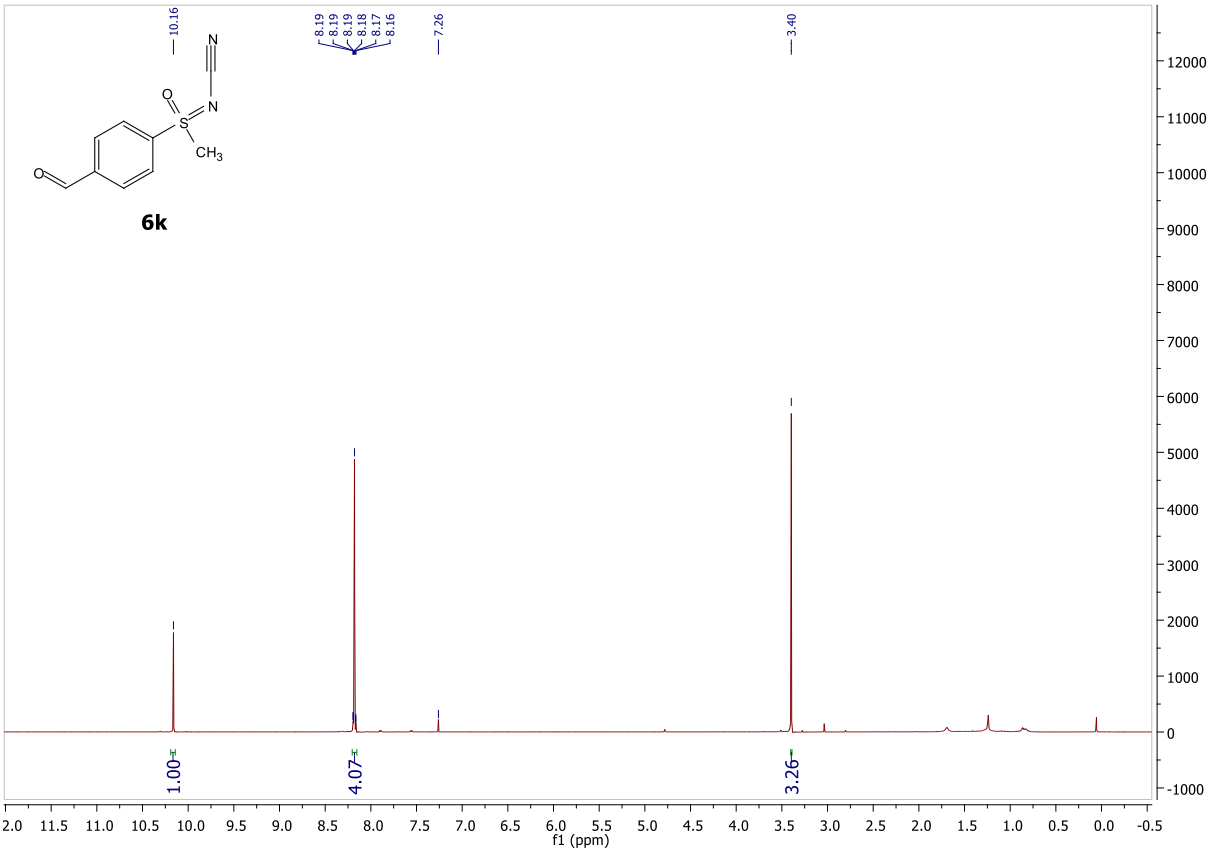

-107.15
-107.17
-107.27
-107.28
-107.29
-107.30
-107.31
-107.61
F
O
S
N
N
CH3
6h
f1 (ppm)

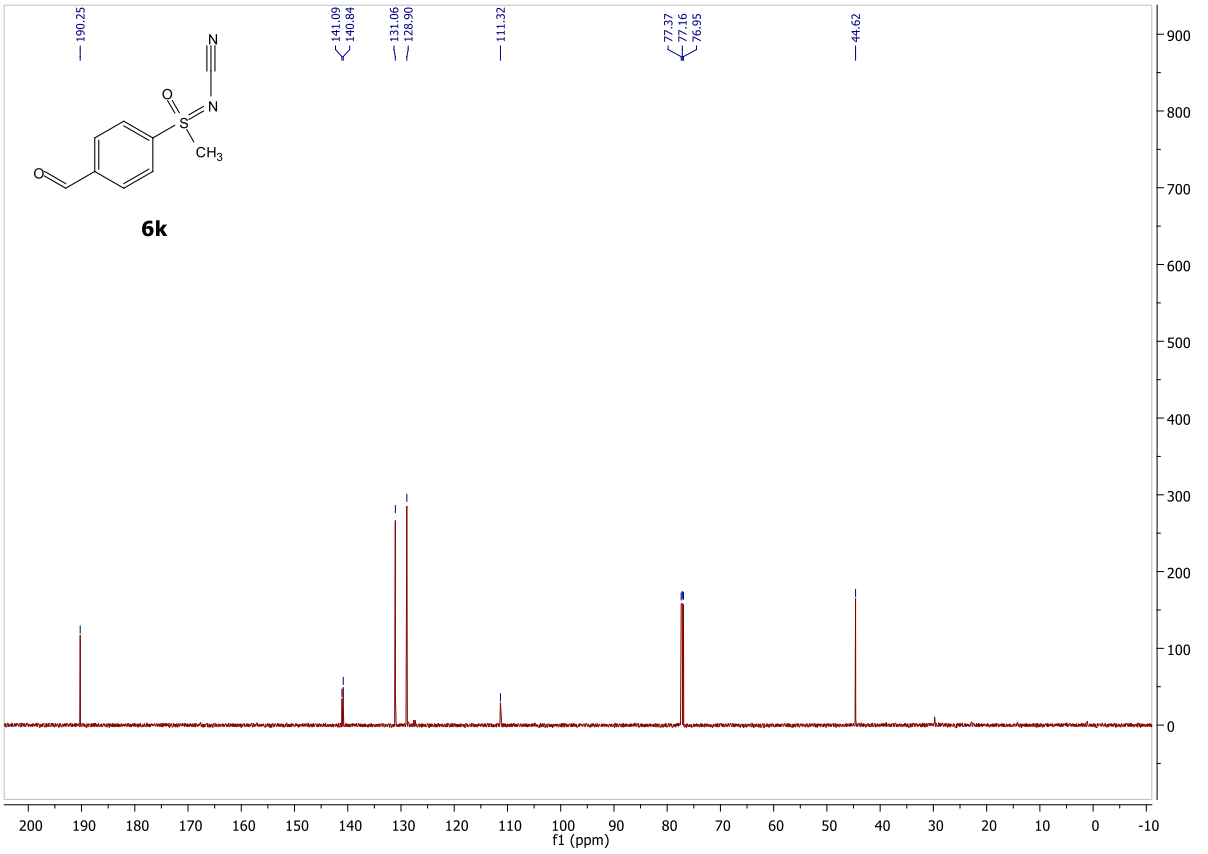

8.54
8.53
8.53
8.52
8.52
8.51
8.24
8.24
8.24
8.23
8.23
8.22
7.26
3.43
O
N
S
N
CH3
O
N+
O-
6i
1.97
2.00
3.29
f1 (ppm)


151.93
141.97
129.77
125.52
110.82
77.37
77.16
76.95
44.67
6i
f1 (ppm)

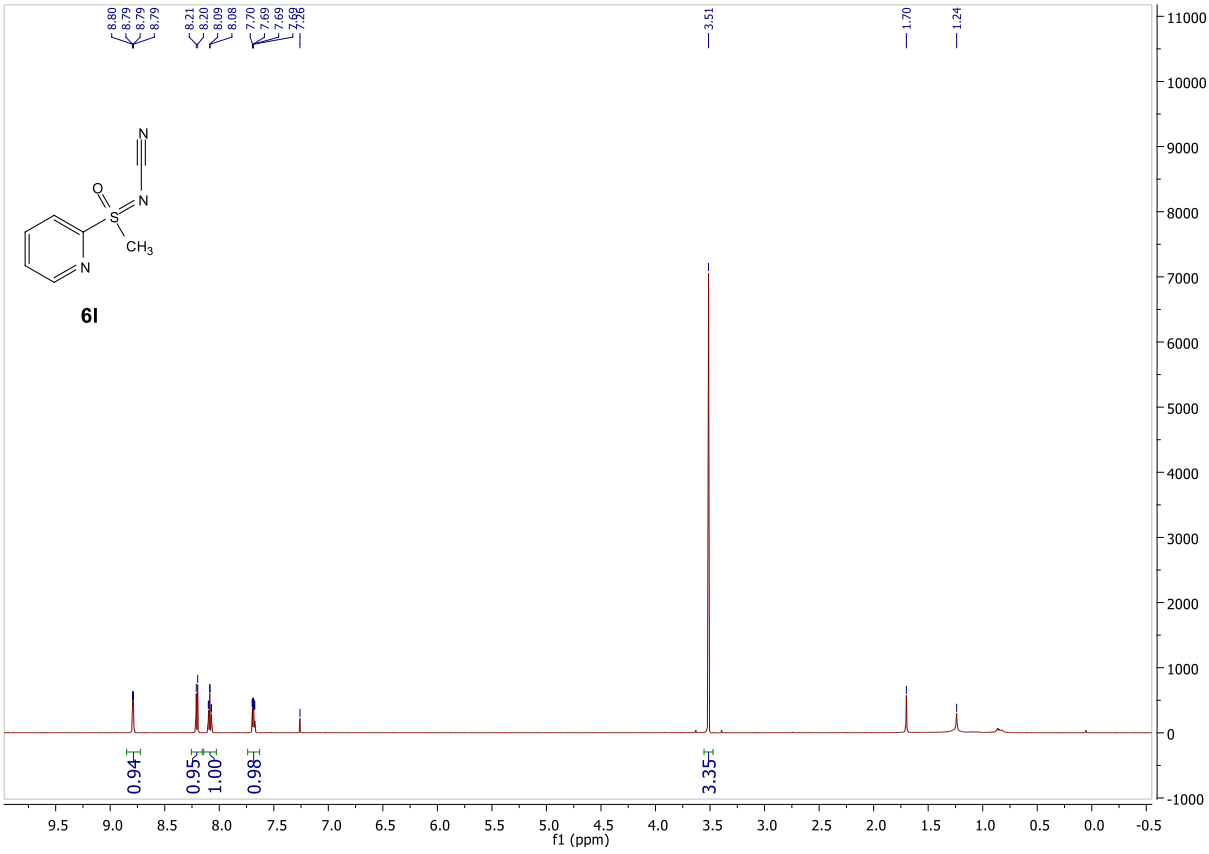

8.21
8.20
8.19
8.19
8.19
8.10
8.10
8.09
8.09
7.26
3.38
2.67
2.08
2.03
3.19
3.33
6j
f1 (ppm)


196.29
142.25
139.84
129.90
128.49
111.47
77.37
77.16
76.95
44.60
27.10
6j
f1 (ppm)

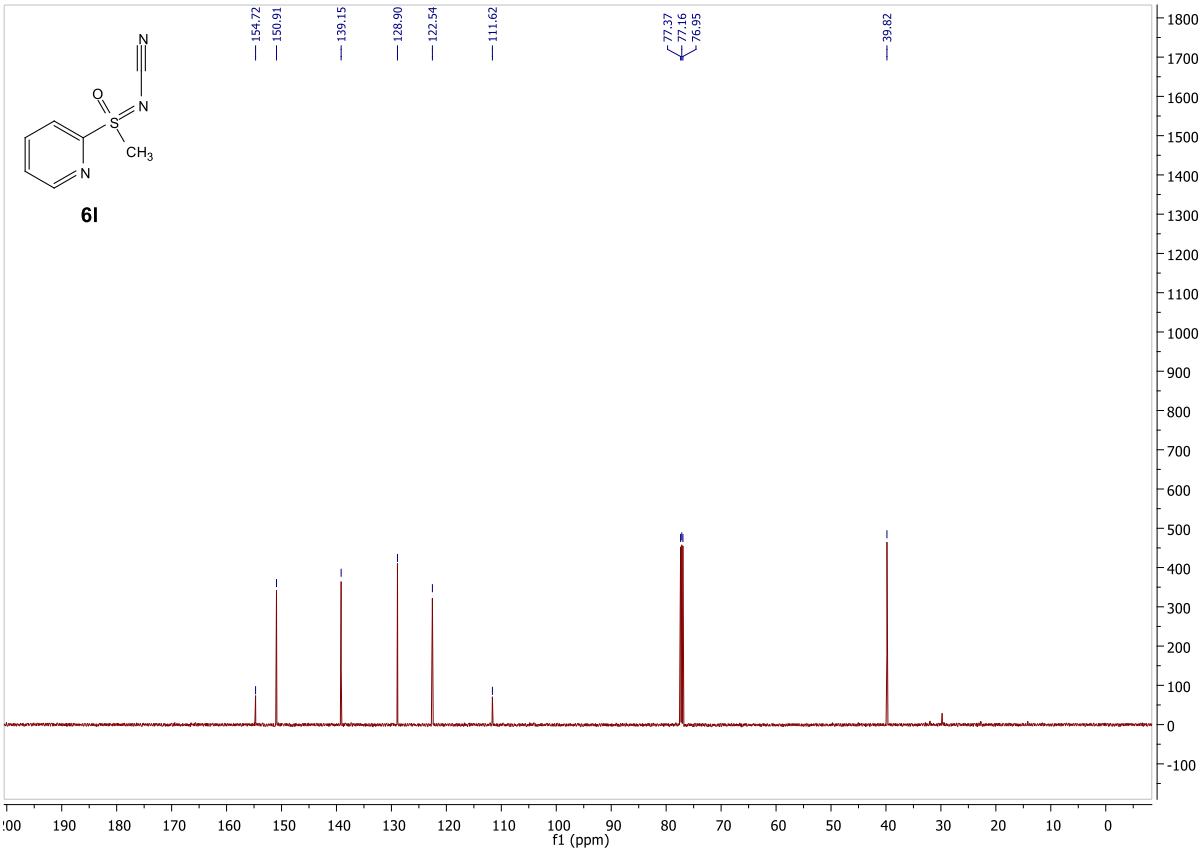

10.16
8.19
8.19
8.19
8.18
8.17
8.16
7.26
3.40
6k
1.00
4.07
3.26
f1 (ppm)

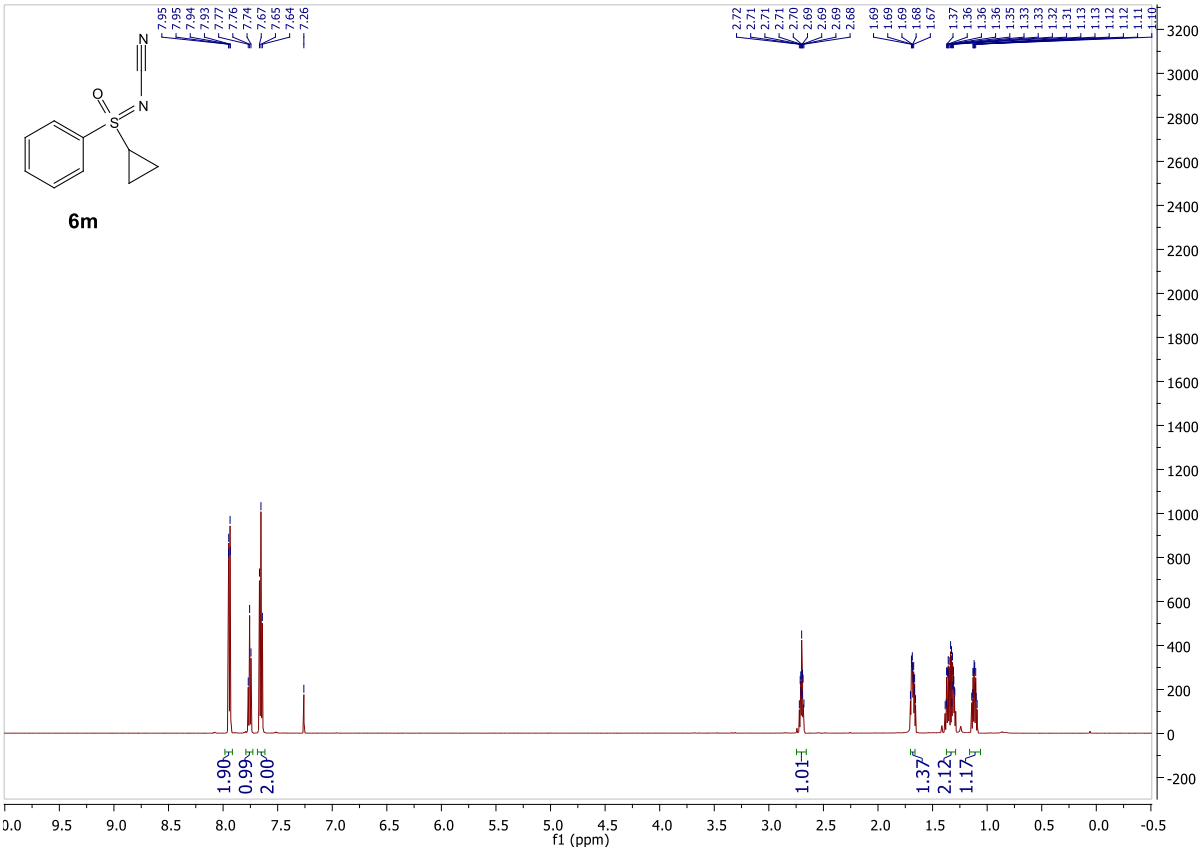

190.25
141.09
140.84
131.06
128.90
111.32
77.37
77.16
76.95
44.62
f1 (ppm)

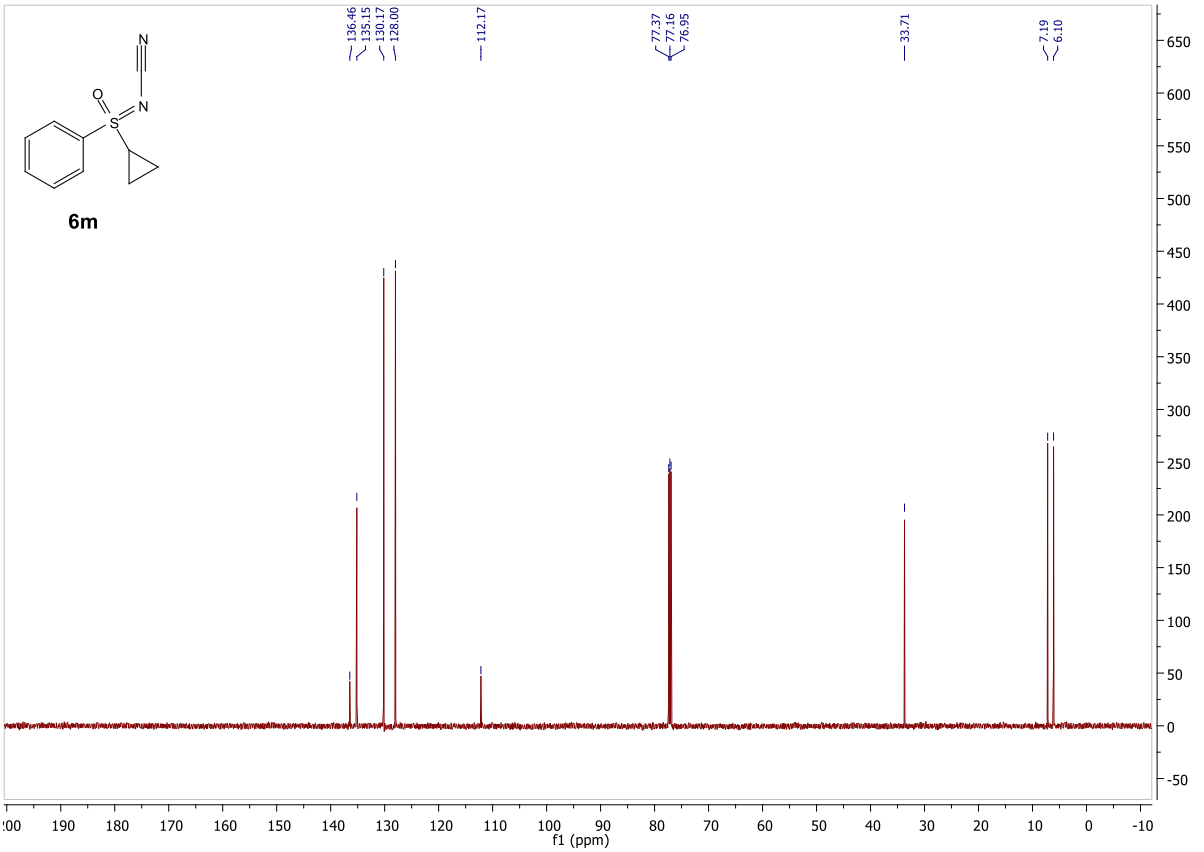
6k


8.80
8.79
8.79
8.79
8.21
8.20
8.09
8.08
7.70
7.69
7.69
3.51
1.70
1.24
0.94
0.95
1.00
0.98
3.35
f1 (ppm)

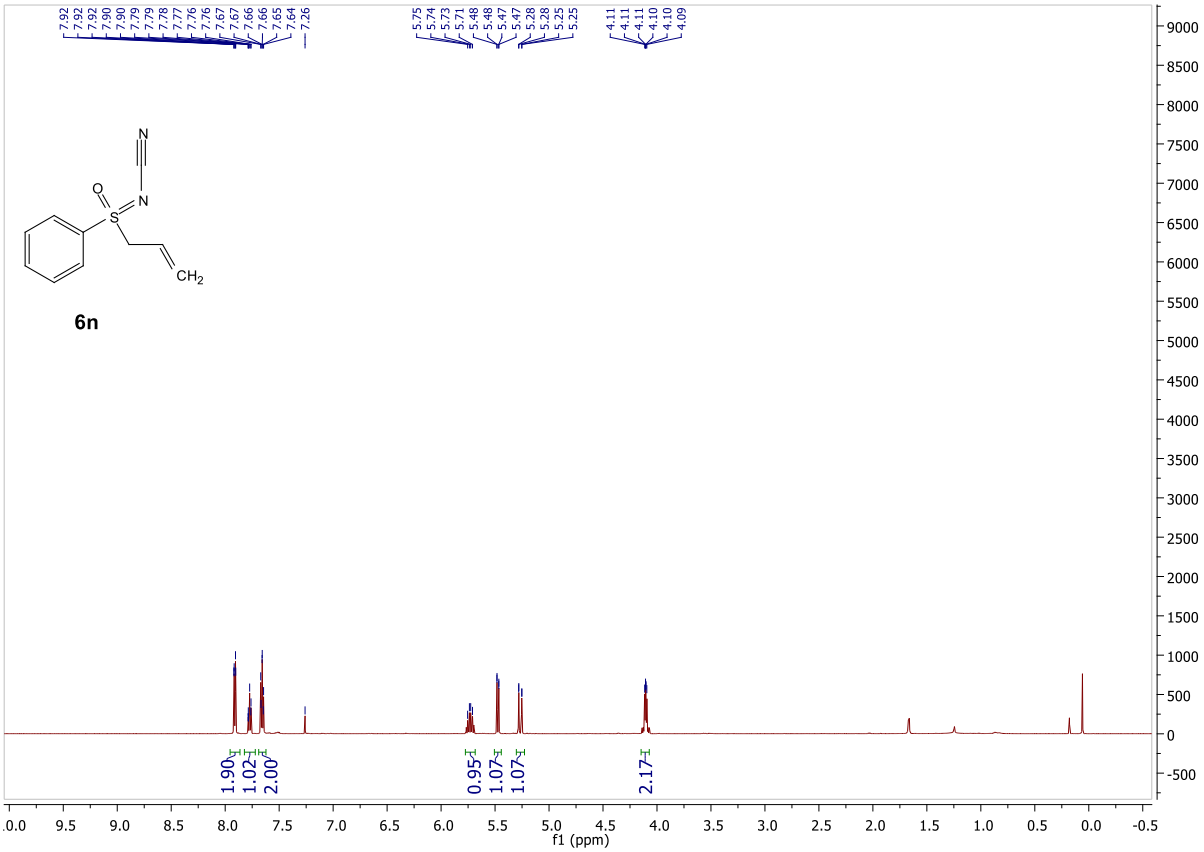
6l


6l
154.72
150.91
139.15
128.90
122.54
111.62
77.37
77.16
76.95
39.82
f1 (ppm)

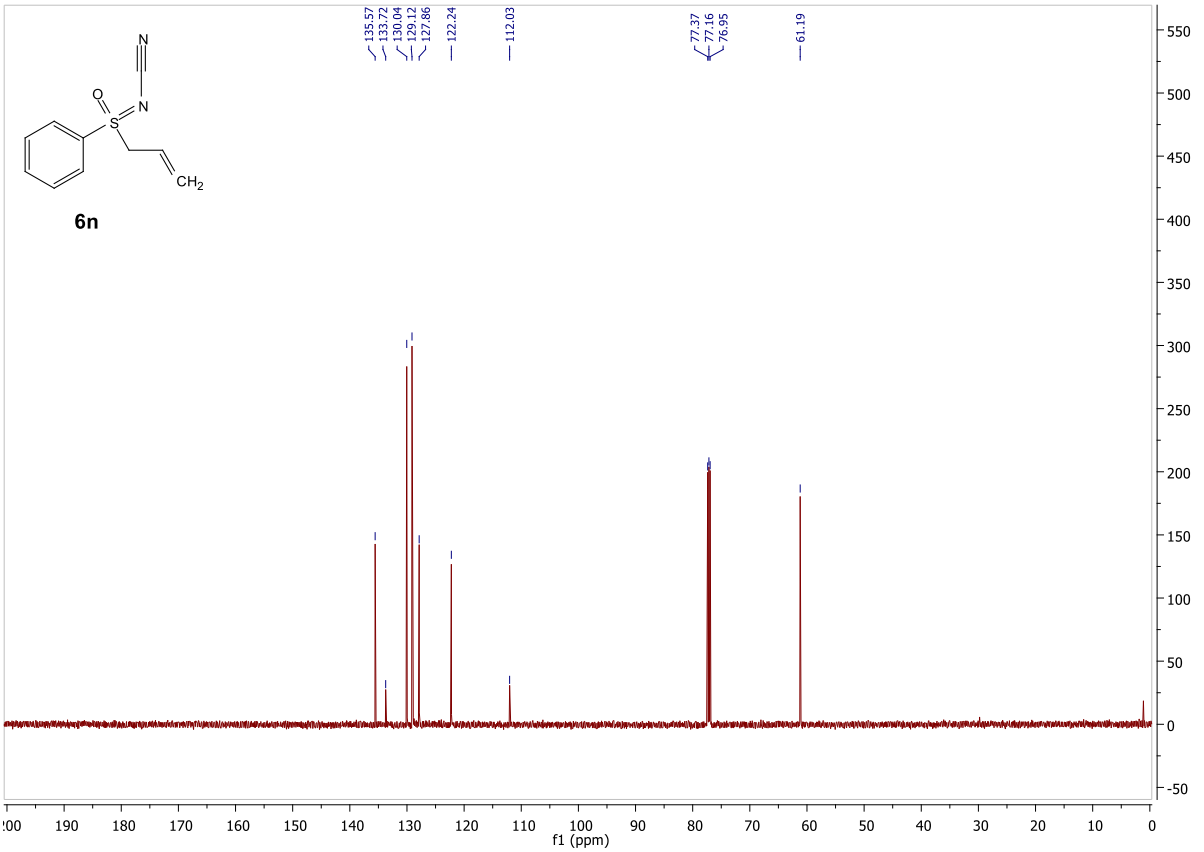

6m
7.95
7.95
7.94
7.93
7.77
7.76
7.74
7.67
7.65
7.64
7.26
2.72
2.71
2.71
2.71
2.70
2.69
2.69
2.69
2.68
1.69
1.69
1.69
1.68
1.67
1.37
1.36
1.36
1.35
1.33
1.33
1.32
1.31
1.13
1.13
1.12
1.12
1.11
1.10
1.90
0.99
2.00
1.01
1.37
2.12
1.17
f1 (ppm)


136.46
135.15
130.17
128.00
112.17
77.37
77.16
76.95
33.71
7.19
6.10
f1 (ppm)

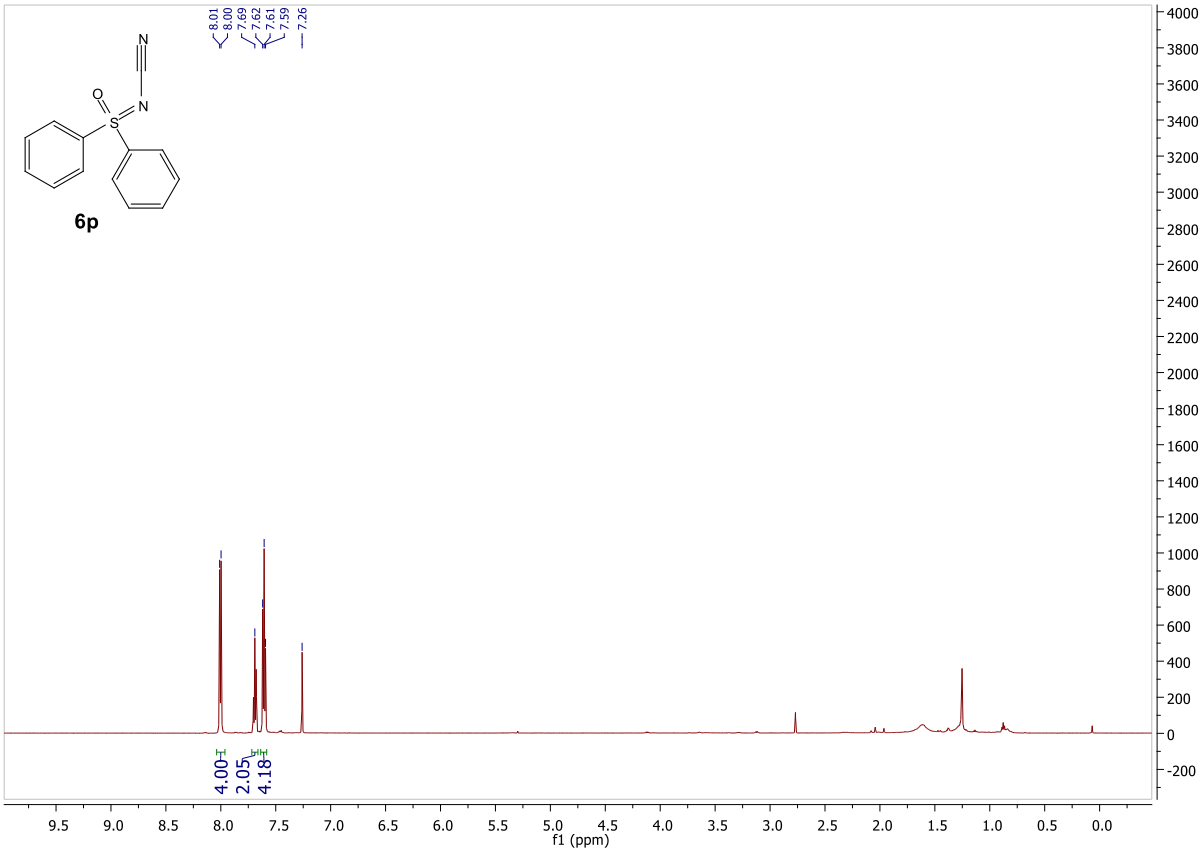
6m


7.92
7.92
7.92
7.90
7.90
7.79
7.79
7.78
7.77
7.76
7.76
7.67
7.67
7.66
7.66
7.65
7.64
7.26
5.75
5.74
5.73
5.71
5.48
5.48
5.47
5.47
5.28
5.25
5.25
4.11
4.11
4.11
4.10
4.10
4.09
1.90
1.02
2.00
0.95
1.07
1.07
2.17
f1 (ppm)

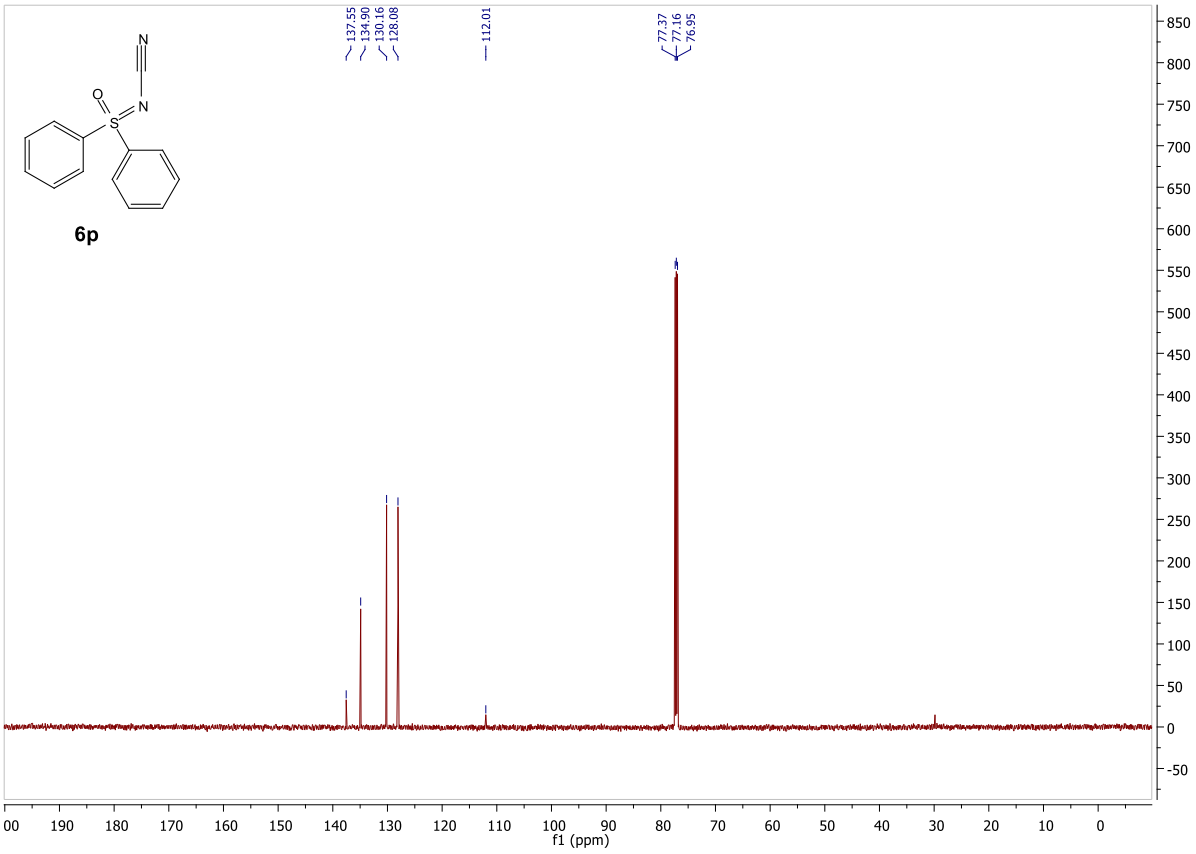
6n


135.57
133.72
130.04
129.12
127.86
122.24
112.03
77.37
77.16
76.95
61.19
f1 (ppm)

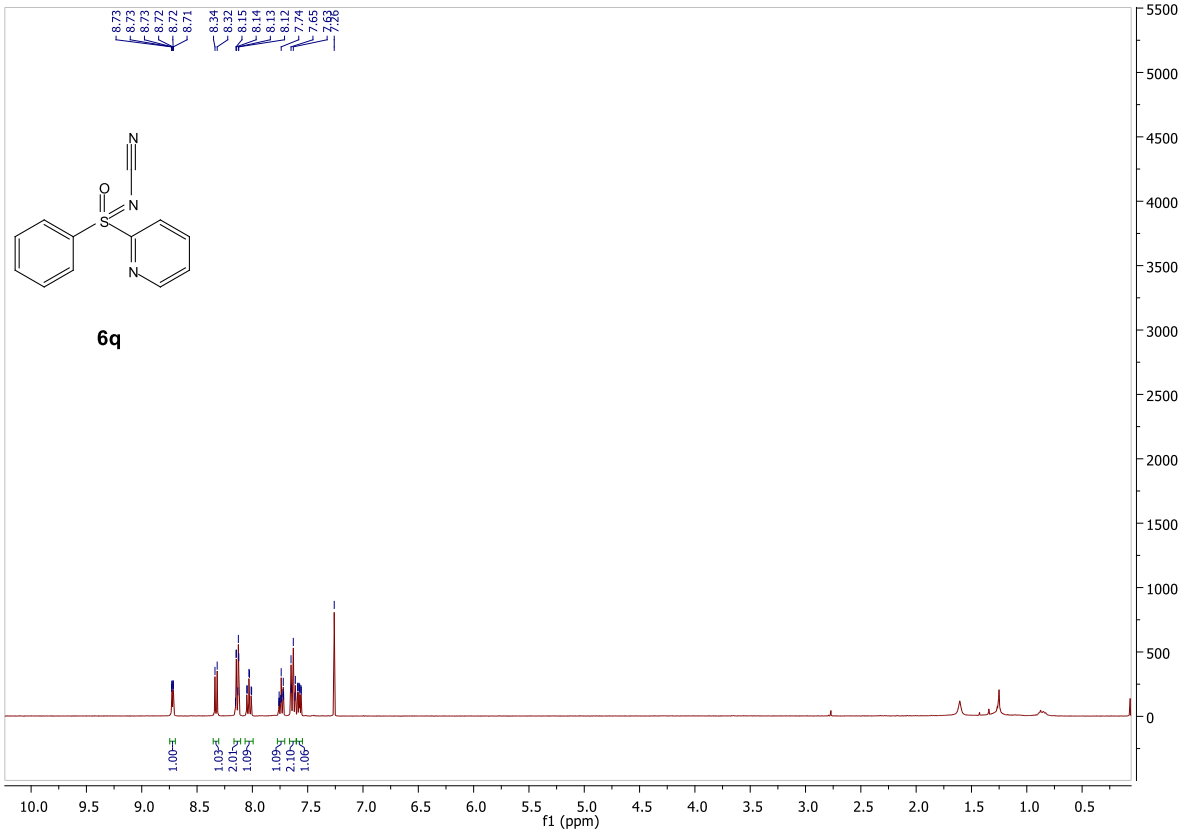
6n


8.01
8.00
7.69
7.62
7.61
7.59
7.26
4.00
2.05
4.18
f1 (ppm)

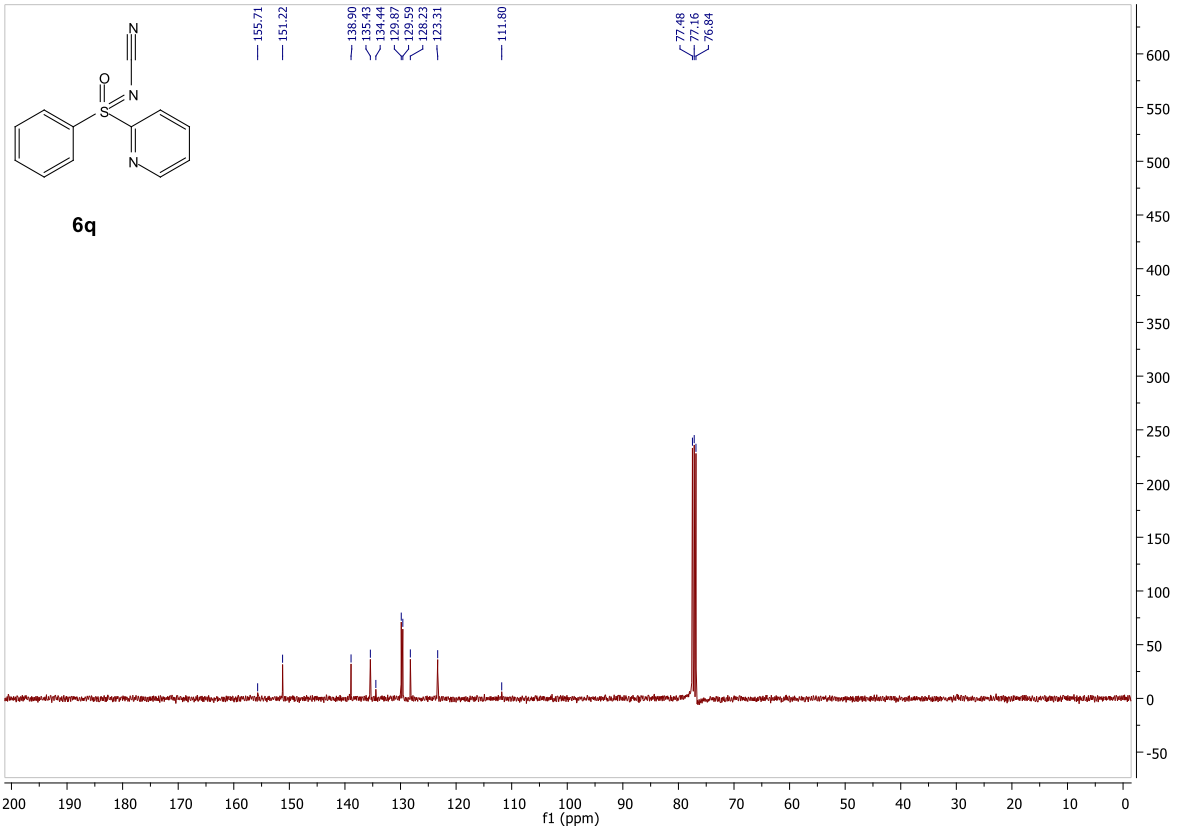
6p


137.55
134.90
130.16
128.08
112.01
77.37
77.16
76.95
f1 (ppm)

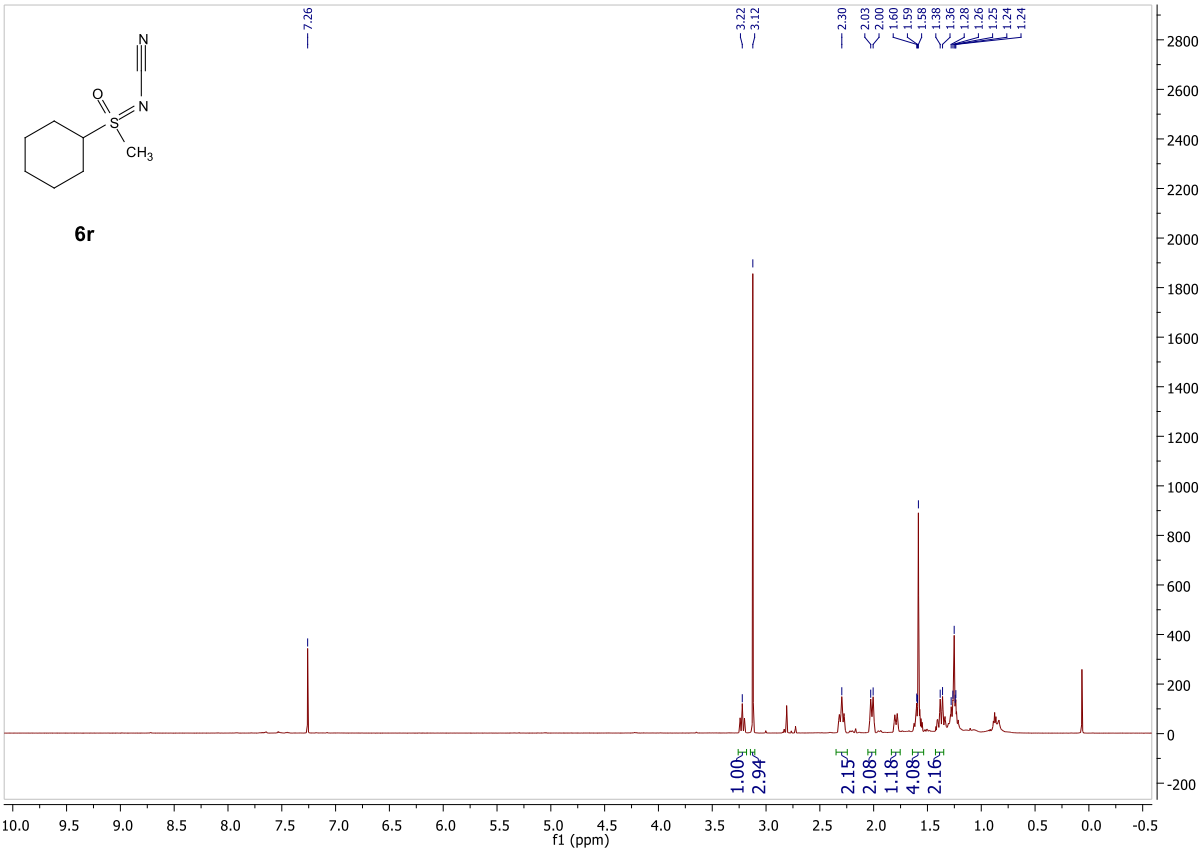
6p


8.73
8.73
8.73
8.72
8.72
8.71
8.34
8.32
8.15
8.14
8.13
8.12
7.74
7.65
7.63
7.28
1.00
1.03
2.01
1.09
1.09
2.10
1.06
f1 (ppm)

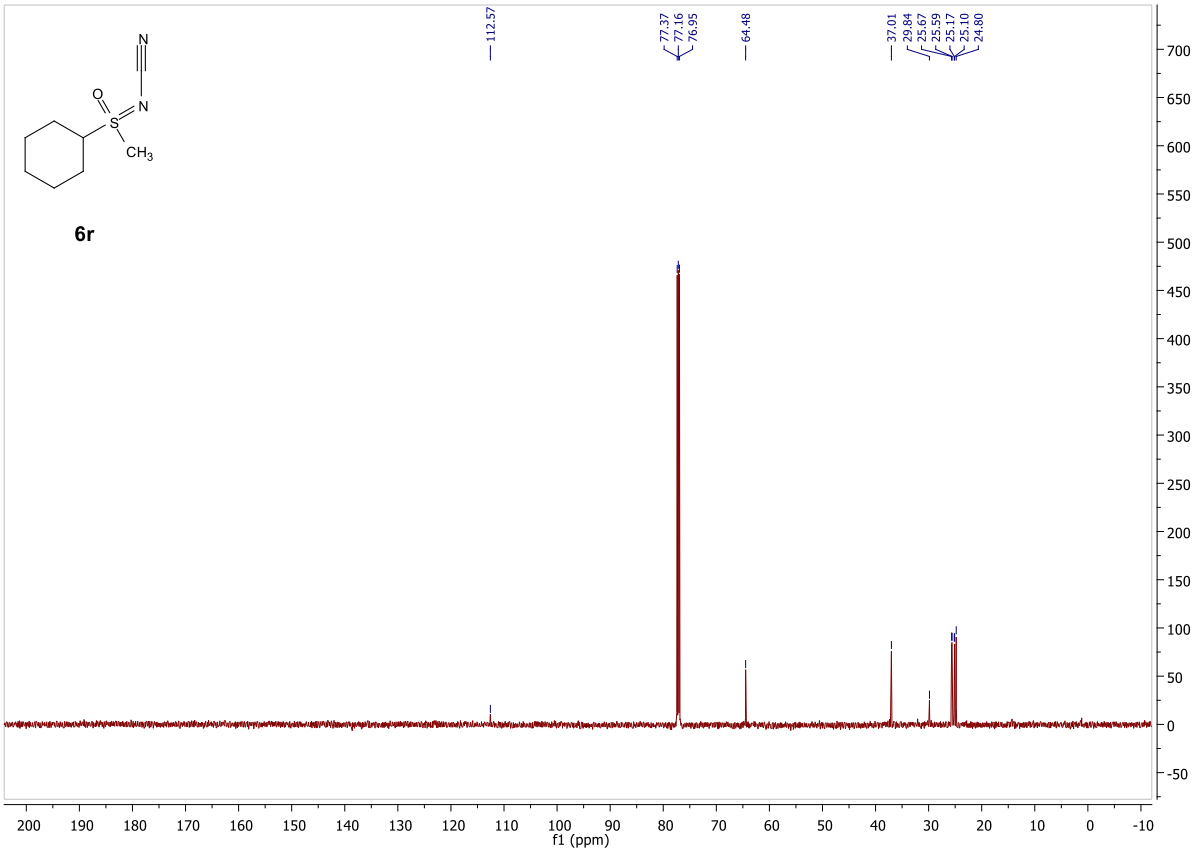
6q

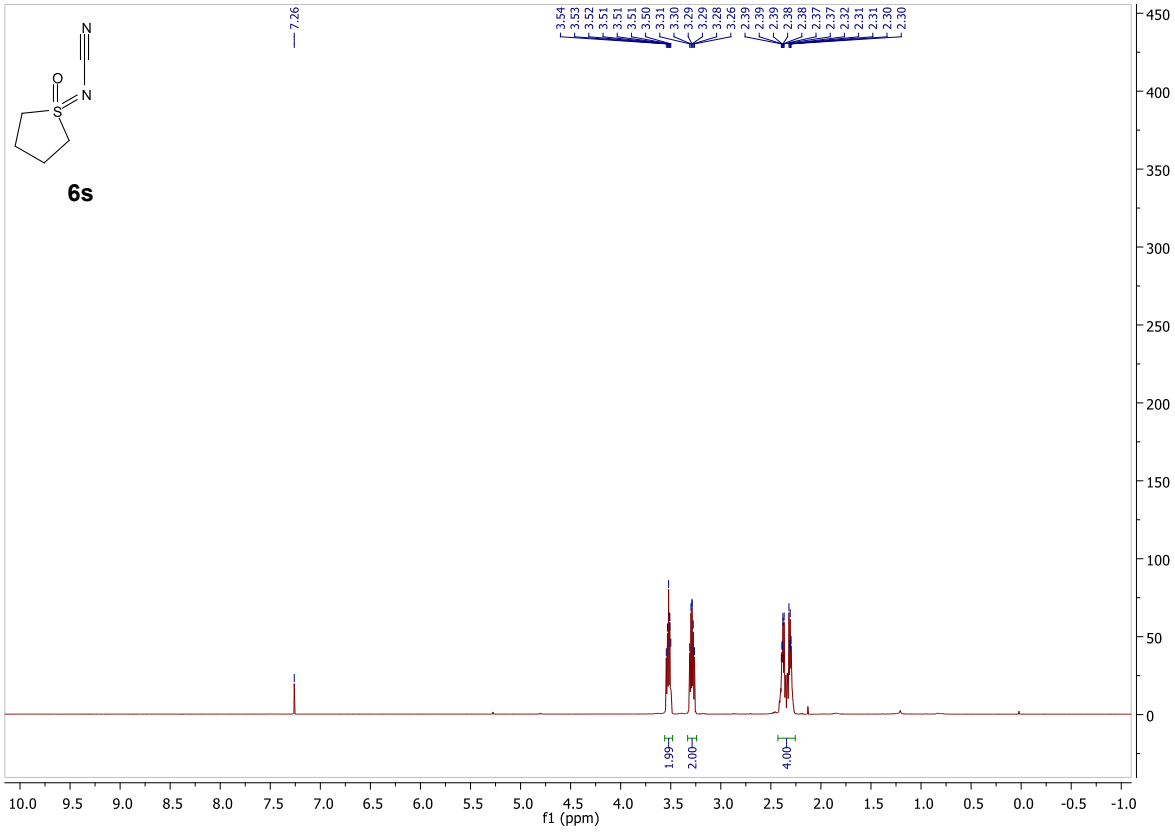

155.71
151.22
138.90
135.43
134.44
129.87
129.59
128.23
123.31
111.80
77.48
77.16
76.84
f1 (ppm)

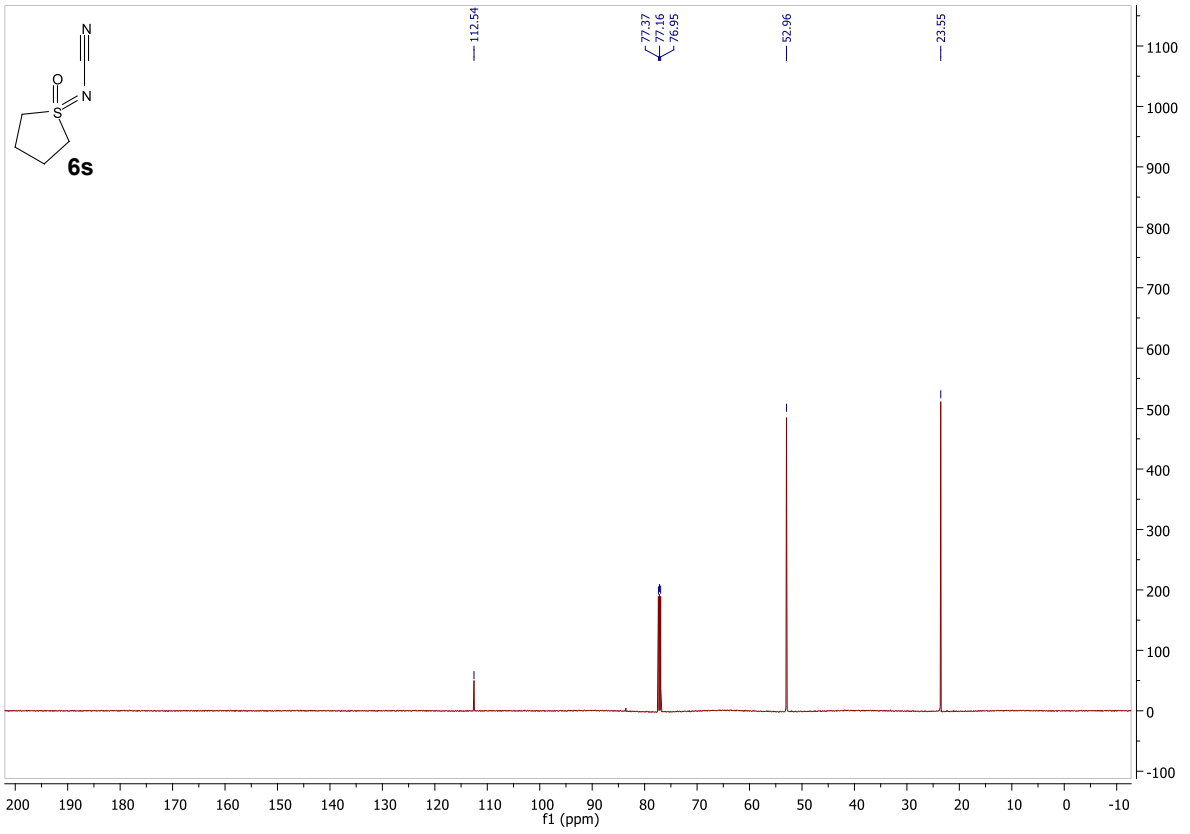
6q

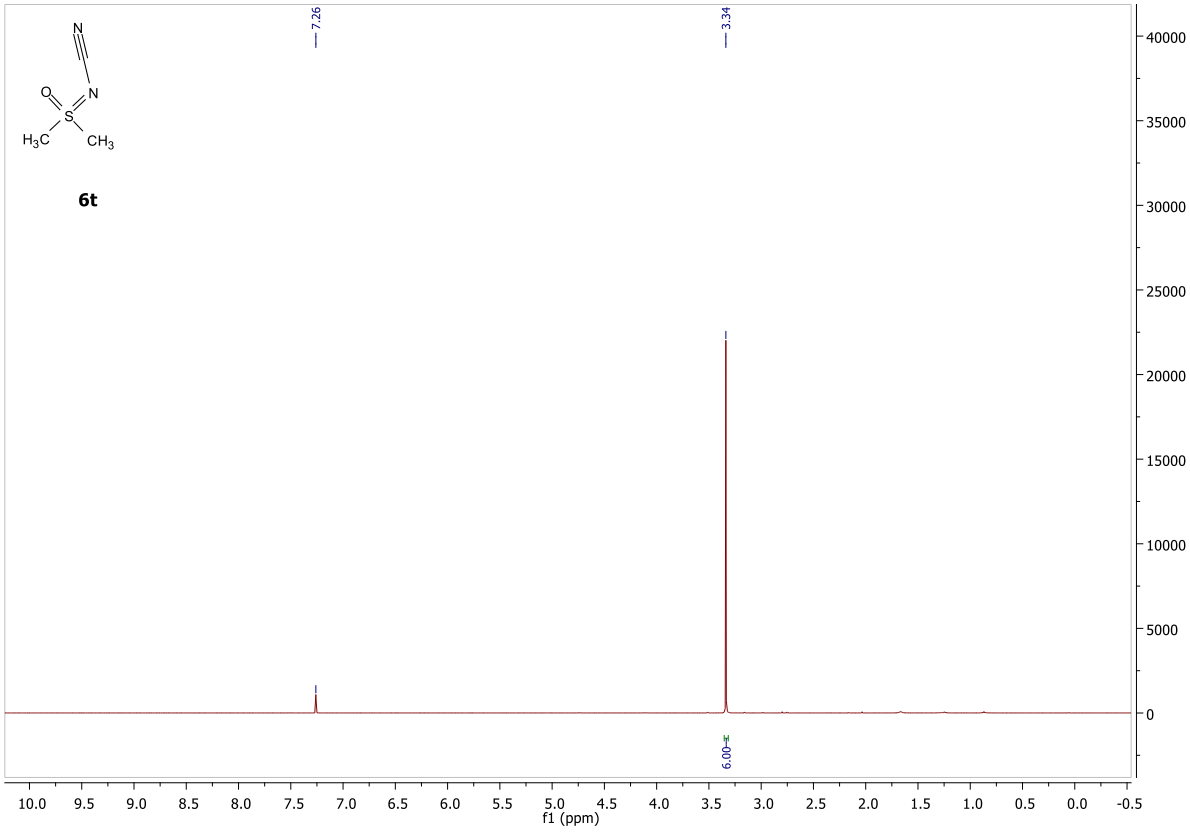

7.26
3.22
3.12
2.30
2.03
2.00
1.60
1.59
1.58
1.38
1.36
1.28
1.26
1.25
1.24
1.24
1.00
2.94
2.15
2.08
1.18
4.08
2.16
f1 (ppm)

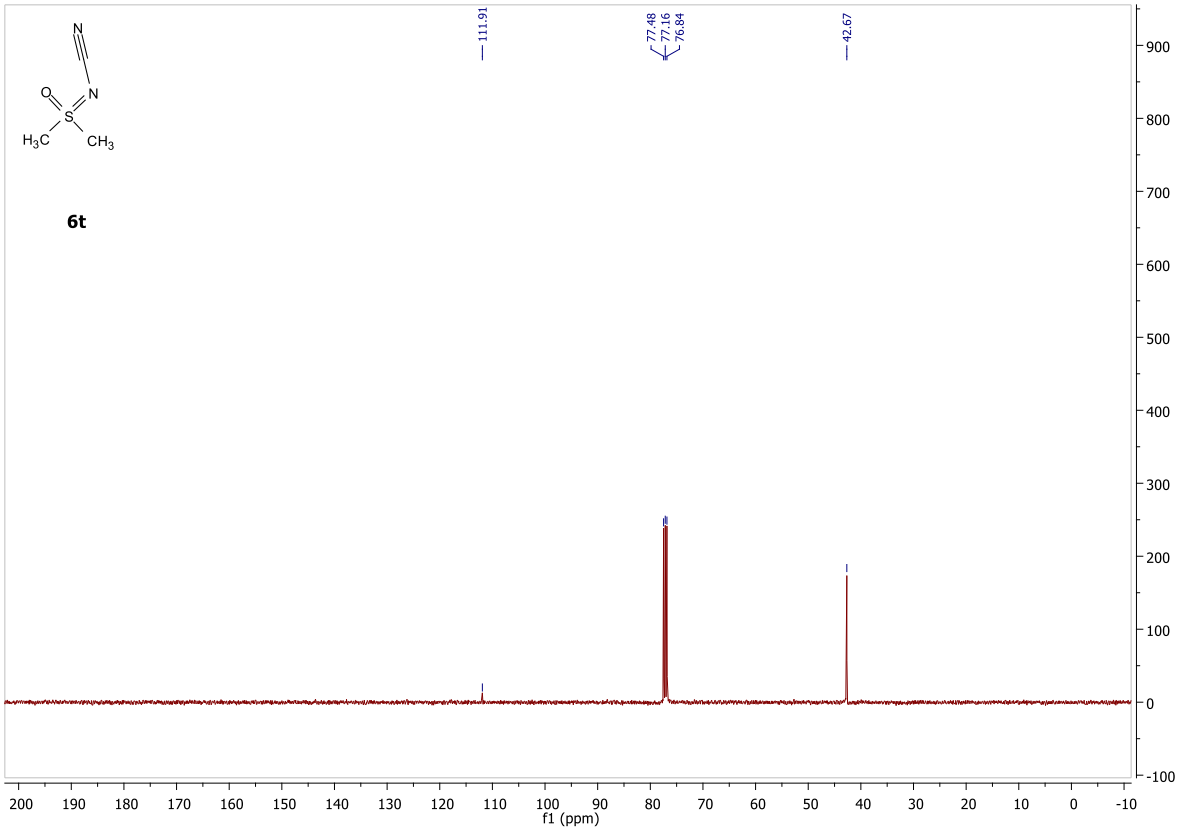
6r


6r
112.57
77.37
77.16
76.95
64.48
37.01
29.84
25.67
25.59
25.17
25.10
24.80
f1 (ppm)

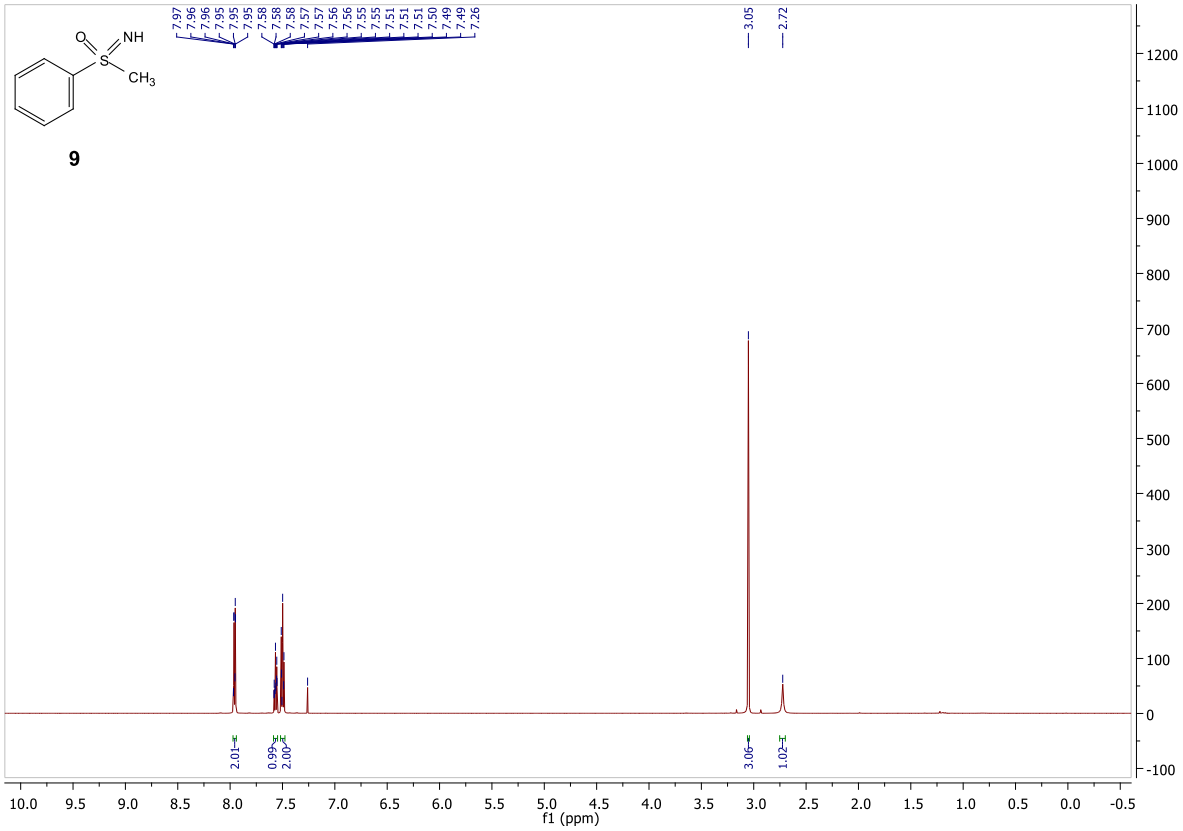

6s
7.26
3.54
3.53
3.52
3.51
3.51
3.51
3.50
3.31
3.30
3.29
3.29
3.28
3.26
2.39
2.39
2.39
2.38
2.38
2.37
2.37
2.32
2.31
2.31
2.30
2.30
1.99
2.00
4.00
f1 (ppm)


6s
112.54
77.37
77.16
76.95
52.96
23.55
f1 (ppm)

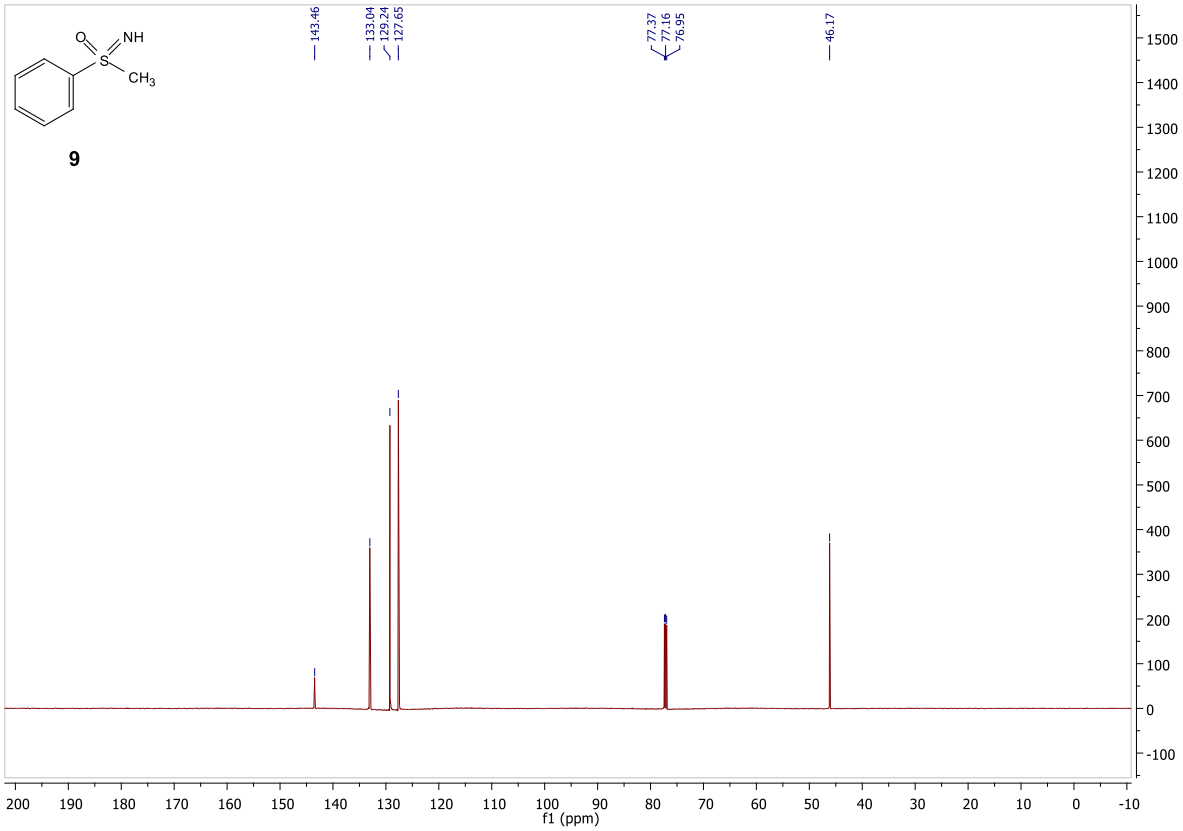

6t
7.26
3.34
6.00
f1 (ppm)

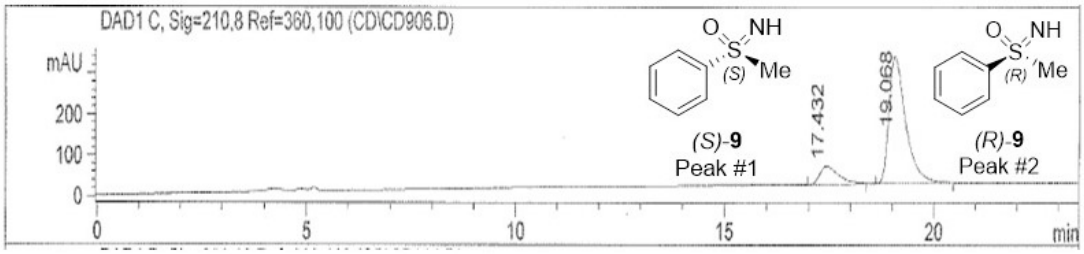

111.91
77.48
77.16
76.84
42.67
f1 (ppm)

6t


7.97
7.96
7.96
7.95
7.95
7.95
7.58
7.58
7.58
7.57
7.57
7.56
7.56
7.55
7.55
7.51
7.51
7.51
7.50
7.49
7.49
7.26
3.05
2.72
2.01
0.99
2.00
3.06
1.02
f1 (ppm)

9





DAD1 C, Sig=210,8 Ref=360,100

Peak #	Ret. Time in min	Width in min	Height in mAU	Area in mAU*s	Area %
1	17.432	0.4611	44.39227	1381.66479	13.7397
2	19.068	0.4191	309.35773	8674.34766	86.2603
Total				10056.01245	100.0000