

Supporting Information

K₂CO₃ Promoted Direct Sulfenylation of Indoles: A Facile Approach of 3-Sulfenylindoles

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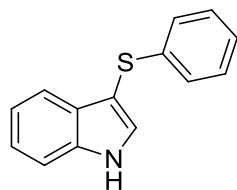
General experimental procedures

Unless otherwise stated, all reactions were carried out under air, all reagents and solvents were obtained from commercial sources and used without any further purification. ^1H NMR spectra were recorded at 400 MHz using TMS as internal standard. ^{13}C NMR spectra were recorded at 100 MHz using TMS as internal standard. The multiplicities are reported as follows: singlet (s), doublet (d), doublet of doublets (dd), triplet (t), quartet (q), multiplet (m). Coupling constants are reported in Hertz (Hz). Mass spectroscopy data were collected on HRMS-EI and HRMS-ESI instrument.

General procedure for synthesis of 3-sulfenylindoles (3)

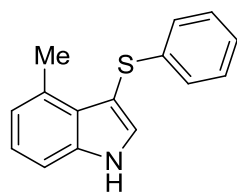
A 25 mL flask equipped with a magnetic stirring bar was charged with substituted indoles (**1**, 0.5 mmol), disulfides (**2**, 0.5 mmol) and K_2CO_3 (0.25 mmol) in DMSO (2 mL). The mixture was allowed to stir under open atmosphere at 100 °C for 9 h. Saturated aqueous NaCl (20 mL), and EtOAc (20 mL) were added to the cooled reaction mixture successively. The organic phase was separated, and the aqueous phase was further extracted with EtOAc (2×20 mL). The combined organic layers were dried over anhydrous Na_2SO_4 and concentrated. The residue was purified by column chromatography on silica gel using petroleum ether/ ethyl acetate (10:1) as eluent to provide the desired products.

Characterization data of compounds 3a-3v



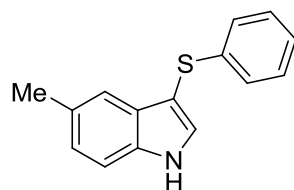
3a

3-(Phenylthio)-1H-indole (3a).¹ Eluent: petroleum ether / ethyl acetate (10:1). White solid. ^1H NMR (DMSO- d_6 , 400 MHz) δ 11.76 (s, 1H), 7.81 (d, 1H, $J = 1.2$ Hz), 7.55 (d, 1H, $J = 8.0$ Hz), 7.46 (d, 1H, $J = 8.0$ Hz), 7.16-7.22 (m, 3H), 7.03-7.10 (m, 4H). ^{13}C NMR (DMSO- d_6 , 100 MHz) δ 139.3, 136.8, 132.4, 128.8, 128.7, 125.3, 124.7, 122.1, 120.1, 118.3, 112.4, 99.3.



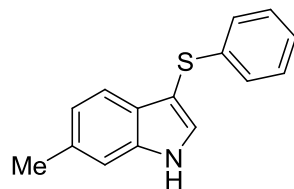
3b

4-Methyl-3-(phenylthio)-1H-indole (3b) [New compound]. Eluent: petroleum ether / ethyl acetate (20:1). Brown oil. ^1H NMR (CDCl_3 , 400 MHz) δ 8.23 (s, 1H), 7.26 (d, 1H, $J = 2.4$ Hz), 7.08-7.16 (m, 4H), 6.98-7.04 (m, 3H), 6.86 (d, 1H, $J = 7.2$ Hz), 2.62 (s, 3H). ^{13}C NMR (CDCl_3 , 100 MHz) δ 141.7, 137.1, 132.1, 129.0, 127.1, 125.3, 124.7, 123.2, 122.6, 109.8, 102.1, 18.8. HRMS (EI) Calcd for $\text{C}_{15}\text{H}_{13}\text{NS}$ (M^+) 239.0769; Found, 239.0769.



3c

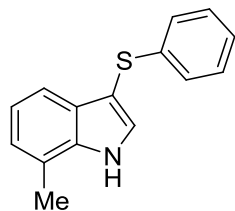
5-methyl-3-(phenylthio)-1H-indole (3c).⁴ Eluent: petroleum ether / ethyl acetate (10:1). White solid. ^1H NMR (DMSO-d_6 , 400 MHz) δ 11.64 (s, 1H), 7.74 (s, 1H), 7.43 (d, 1H, $J = 8.4$ Hz), 7.26 (s, 1H), 7.16-7.19 (m, 2H), 7.02-7.05 (m, 4H), 2.34 (s, 3H). ^{13}C NMR (DMSO-d_6 , 100 MHz) δ 139.5, 135.1, 132.5, 129.0, 128.8, 128.8, 125.1, 124.6, 123.8, 117.8, 112.1, 98.5, 21.2.



3d

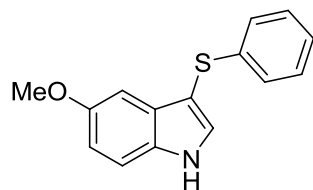
6-Methyl-3-(phenylthio)-1H-indole (3d).⁵ Eluent: petroleum ether / ethyl acetate (10:1). White solid. ^1H NMR (DMSO-d_6 , 400 MHz) δ 11.59 (s, 1H), 7.70 (s, 1H), 7.30-7.33 (m, 2H), 7.15-7.19 (m, 2H), 7.04 (d, 3H, $J = 7.2$ Hz), 6.91 (d, 1H, $J = 8.0$

Hz), 2.41 (s, 3H). ^{13}C NMR (DMSO- d_6 , 100 MHz) δ 139.4, 137.2, 131.7, 131.4, 128.7, 126.6, 125.2, 124.6, 121.9, 118.1, 112.1, 99.1, 21.3.



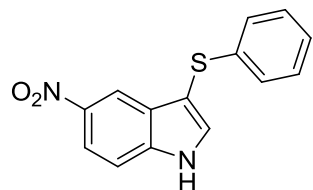
3e

7-Methyl-3-(phenylthio)-1H-indole (3e).⁶ Eluent: petroleum ether / ethyl acetate (10:1). Brown oil. ^1H NMR (CDCl_3 , 400 MHz) δ 8.12 (s, 1H), 7.44 (d, 1H, $J = 7.2$ Hz), 7.28 (s, 1H), 7.06-7.12 (m, 4H), 7.00-7.05 (m, 3H), 2.40 (s, 3H). ^{13}C NMR (CDCl_3 , 100 MHz) δ 139.5, 136.2, 130.7, 128.9, 128.8, 126.0, 124.9, 123.7, 121.2, 121.1, 117.4, 103.0, 16.6.



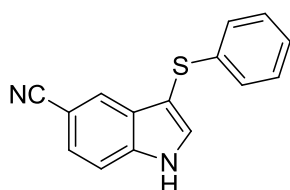
3f

5-Methoxy-3-(phenylthio)-1H-indole (3f).¹ Eluent: petroleum ether / ethyl acetate (5:1). Brown oil. ^1H NMR (CDCl_3 , 400 MHz) δ 8.37 (s, 1H), 7.30 (s, 1H), 7.20 (s, 1H, $J = 8.8$ Hz), 7.07-7.16 (m, 4H), 7.00-7.04 (m, 2H), 6.88 (d, 1H, $J = 8.8$ Hz), 3.72 (s, 3H). ^{13}C NMR (CDCl_3 , 100 MHz) δ 155.1, 139.5, 131.7, 131.5, 130.0, 128.9, 125.8, 124.9, 113.6, 112.7, 101.9, 100.9, 55.9.



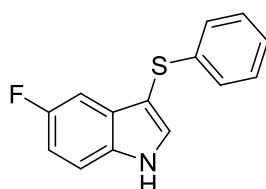
3g

5-Nitro-3-(phenylthio)-1H-indole (3g) [New compound]. Eluent: petroleum ether / ethyl acetate (5:1). Yellow solid. ^1H NMR (DMSO- d_6 , 400 MHz) δ 12.42 (s, 1H), 8.27 (s, 1H), 8.06 (d, 2H, $J = 9.2$ Hz), 7.68 (d, 1H, $J = 9.2$ Hz), 7.17-7.21 (m, 2H), 7.06 (d, 3H, $J = 7.2$ Hz). ^{13}C NMR (DMSO- d_6 , 100 MHz) δ 141.5, 140.0, 138.0, 136.4, 129.0, 128.2, 125.7, 125.2, 117.5, 114.8, 113.1, 102.8. HRMS (EI) Calcd for $\text{C}_{14}\text{H}_{10}\text{N}_2\text{O}_2\text{S}$ (M) $^+$ 270.0463; Found, 270.0466.



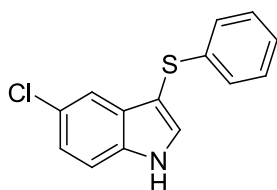
3h

3-(Phenylthio)-1H-indole-5-carbonitrile (3h).³ Eluent: petroleum ether / ethyl acetate (5:1). Yellow solid. ^1H NMR (DMSO- d_6 , 400 MHz) δ 12.29 (s, 1H), 8.02 (d, 1H, $J = 1.6$ Hz), 7.86 (s, 1H), 7.70 (d, 1H, $J = 8.4$ Hz), 7.54 (d, 1H, $J = 8.4$ Hz), 7.19 (t, 2H, $J = 7.6$ Hz), 7.07 (t, 3H, $J = 7.6$ Hz). ^{13}C NMR (DMSO- d_6 , 100 MHz) δ 138.6, 138.2, 135.2, 128.9, 128.6, 125.8, 125.2, 125.0, 123.6, 120.2, 113.8, 102.5, 101.2.



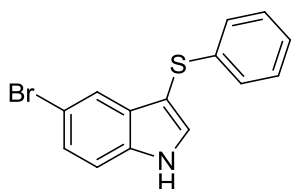
3i

5-Fluoro-3-(phenylthio)-1H-indole (3i) [New compound]. Eluent: petroleum ether / ethyl acetate (10:1). White solid. ^1H NMR (DMSO- d_6 , 400 MHz) δ 11.88 (s, 1H), 7.88 (s, 1H), 7.53-7.56 (dt, 1H, $J = 4.4$ Hz, 4.4 Hz), 7.17-7.20 (t, 2H, $J = 7.6$ Hz), 7.13 (d, 1H, $J = 9.2$ Hz), 7.04-7.07 (m, 4H). ^{13}C NMR (DMSO- d_6 , 100 MHz) δ 157.7 ($J_{CF} = 233.4$ Hz), 138.8, 134.4, 133.3, 129.4 ($J_{CF} = 9.8$ Hz), 128.8, 125.4, 124.9, 112.1 ($J_{CF} = 310.6$ Hz), 112.0 ($J_{CF} = 326.9$ Hz), 103.0 ($J_{CF} = 24.5$ Hz), 99.6 ($J_{CF} = 4.3$ Hz). HRMS (EI) Calcd for $\text{C}_{14}\text{H}_{10}\text{NOF}$ (M) $^+$ 243.0518; Found, 243.0516.



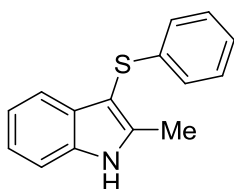
3j

5-Chloro-3-(phenylthio)-1H-indole (3j).⁴ Eluent: petroleum ether / ethyl acetate (10:1). White solid. ¹H NMR (DMSO-d₆, 400 MHz) δ 11.95 (s, 1H), 7.89 (s, 1H), 7.56 (d, 1H, *J* = 8.4 Hz), 7.40 (s, 1H), 7.17-7.21 (m, 3H), 7.03-7.08 (m, 3H). ¹³C NMR (DMSO-d₆, 100 MHz) δ 138.7, 135.2, 134.2, 130.0, 128.9, 125.4, 125.0, 124.9, 122.2, 117.4, 114.1, 99.3.



3k

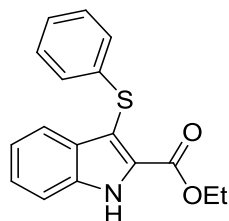
5-Bromo-3-(phenylthio)-1H-indole (3k).¹ Eluent: petroleum ether / ethyl acetate (10:1). White solid. ¹H NMR (DMSO-d₆, 400 MHz) δ 11.97 (s, 1H), 7.87 (s, 1H), 7.56 (s, 1H), 7.51 (d, 1H, *J* = 8.4 Hz), 7.31 (d, 1H, *J* = 8.4 Hz), 7.17-7.20 (m, 2H), 7.03-7.07 (m, 3H). ¹³C NMR (DMSO-d₆, 100 MHz) δ 138.7, 135.5, 134.1, 130.6, 128.9, 125.4, 124.9, 124.8, 120.4, 114.5, 113.0, 99.2.



3l

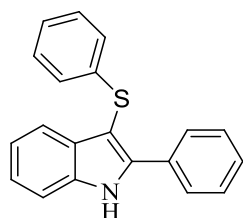
2-Methyl-3-(phenylthio)-1H-indole (3l).¹ Eluent: petroleum ether / ethyl acetate (10:1). Brown solid. ¹H NMR (DMSO-d₆, 400 MHz) δ 11.71 (s, 1H), 7.45 (d, 1H, *J* = 8.4 Hz), 7.40 (d, 1H, *J* = 7.6 Hz), 7.12-7.18 (m, 3H), 7.00-7.06 (m, 4H), 2.50 (s, 3H).

^{13}C NMR (DMSO- d_6 , 100 MHz) δ 142.1, 139.2, 135.7, 129.7, 128.8, 125.0, 124.5, 121.4, 119.9, 117.7, 111.3, 96.4, 11.7.



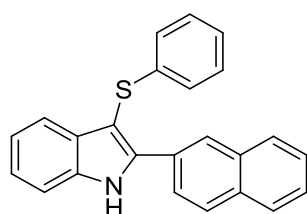
3m

Ethyl 3-(phenylthio)-1H-indole-2-carboxylate (3m).² Eluent: petroleum ether / ethyl acetate (10:1). White solid. ^1H NMR (DMSO- d_6 , 400 MHz) δ 9.73 (s, 1H), 7.62 (d, 1H, $J = 8.0$ Hz), 7.44 (d, 1H, $J = 8.4$ Hz), 7.33 (t, 1H, $J = 7.6$ Hz), 7.06-7.17 (m, 6H), 4.40 (q, 2H, $J = 7.2$ Hz), 1.29 (t, 3H, $J = 7.2$ Hz). ^{13}C NMR (DMSO- d_6 , 100 MHz) δ 161.7, 138.1, 136.0, 130.1, 128.9, 128.8, 127.2, 126.1, 125.3, 121.7, 121.5, 112.3, 110.4, 61.6, 14.2.



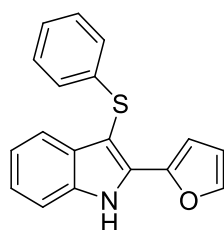
3n

2-Phenyl-3-(phenylthio)-1H-indole (3n).² Eluent: petroleum ether / ethyl acetate (10:1). yellow oil. ^1H NMR (CDCl_3 , 400 MHz) δ 8.74 (s, 1H), 7.60-7.65 (m, 3H), 7.28-7.32 (m, 4H), 7.18 (d, 1H, $J = 6.4$ Hz), 7.07-7.10 (m, 5H), 6.97 (s, 1H). ^{13}C NMR (CDCl_3 , 100 MHz) δ 142.3, 139.5, 136.1, 131.5, 131.3, 129.0, 128.8, 128.8, 128.4, 125.7, 124.8, 123.4, 121.2, 120.0, 111.5, 99.1.



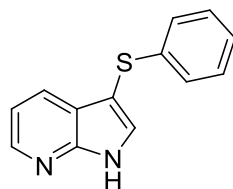
3o

2-(Naphthalen-2-yl)-3-(phenylthio)-1H-indole (3o) [New compound]. Eluent: petroleum ether / ethyl acetate (10:1). White solid. ^1H NMR (DMSO- d_6 , 400 MHz) δ 12.36 (s, 1H), 8.50 (s, 1H), 8.16 (d, 1H, $J = 8.4$ Hz), 7.99 (d, 1H, $J = 8.8$ Hz), 7.92 (d, 2H, $J = 4.0$ Hz), 7.68 (d, 1H, $J = 8.0$ Hz), 7.60 (d, 1H, $J = 8.0$ Hz), 7.52-7.54 (m, 2H), 7.30 (t, 1H, $J = 7.6$ Hz), 7.13-7.22 (m, 5H), 7.05 (t, 1H, $J = 6.8$ Hz). ^{13}C NMR (DMSO- d_6 , 100 MHz) δ 142.0, 139.1, 136.5, 132.7, 132.6, 130.8, 129.0, 128.9, 128.1, 128.0, 127.6, 127.4, 126.7, 126.7, 125.9, 125.2, 124.8, 122.9, 120.6, 118.8, 112.1, 97.3. HRMS (EI) Calcd for $\text{C}_{24}\text{H}_{17}\text{NS}$ (M) $^+$ 351.1082; Found, 351.1086.



3p

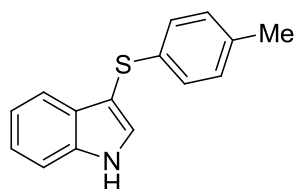
2-(Furan-2-yl)-3-(phenylthio)-1H-indole (3p) [New compound]. Eluent: petroleum ether / ethyl acetate (10:1). Brown solid. ^1H NMR (DMSO- d_6 , 400 MHz) δ 12.26 (s, 1H), 7.87 (s, 1H), 7.56 (d, 1H, $J = 8.0$ Hz), 7.51 (d, 1H, $J = 8.0$ Hz), 7.16-7.24 (m, 3H), 7.08-7.12 (m, 2H), 7.05 (d, 3H, $J = 7.6$ Hz), 6.63 (s, 1H). ^{13}C NMR (DMSO- d_6 , 100 MHz) δ 145.7, 143.1, 138.0, 136.1, 133.0, 130.4, 129.0, 125.3, 124.9, 122.9, 120.7, 118.4, 112.3, 112.2, 109.7, 95.8. HRMS (EI) Calcd for $\text{C}_{18}\text{H}_{13}\text{NOS}$ (M) $^+$ 291.0718; Found, 291.0714.



3q

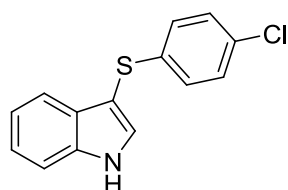
3-(Phenylthio)-1H-pyrrolo[2,3-*b*]pyridine (3q).⁷ Eluent: petroleum ether / ethyl acetate (2:1). White solid. ^1H NMR (DMSO- d_6 , 400 MHz) δ 12.35 (s, 1H), 8.32 (s,

1H), 7.95 (s, 1H), 7.79 (d, 1H, $J = 7.6$ Hz), 7.15-7.18 (m, 2H), 7.08-7.11 (m, 1H), 7.04 (d, 3H, $J = 8.0$ Hz). ^{13}C NMR (DMSO- d_6 , 100 MHz) δ 148.9, 143.6, 138.6, 133.2, 128.9, 126.7, 125.5, 125.0, 121.0, 116.6, 98.7.



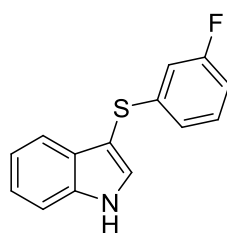
3r

3-(*p*-tolylthio)-1H-indole (3r).¹ Eluent: petroleum ether / ethyl acetate (10:1). White solid. ^1H NMR (DMSO- d_6 , 400 MHz) δ 11.71 (s, 1H), 7.77 (d, 1H, $J = 2.4$ Hz), 7.54 (d, 1H, $J = 8.4$ Hz), 7.45 (d, 1H, $J = 7.6$ Hz), 7.19 (t, 1H, $J = 8.0$ Hz), 7.07 (t, 1H, $J = 7.2$ Hz), 6.98 (s, 4H), 2.18 (s, 3H). ^{13}C NMR (DMSO- d_6 , 100 MHz) δ 136.7, 135.5, 134.1, 132.1, 129.4, 128.7, 125.8, 122.1, 120.0, 118.4, 112.3, 100.1, 20.4.



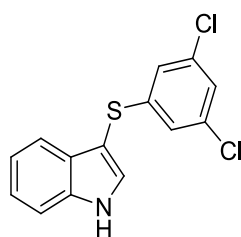
3s

3-(4-Chlorophenylthio)-1H-indole (3s).¹ Eluent: petroleum ether / ethyl acetate (10:1). White solid. ^1H NMR (DMSO- d_6 , 400 MHz) δ 11.80 (s, 1H), 7.81 (s, 1H), 7.55 (d, 1H, $J = 8.4$ Hz), 7.43 (d, 1H, $J = 7.6$ Hz), 7.18-7.23 (m, 3H), 7.08 (t, 1H, $J = 7.6$ Hz), 7.03 (d, 2H, $J = 8.0$ Hz). ^{13}C NMR (DMSO- d_6 , 100 MHz) δ 138.4, 136.8, 132.6, 129.3, 128.7, 128.4, 126.8, 122.2, 120.2, 118.2, 112.4, 98.8.



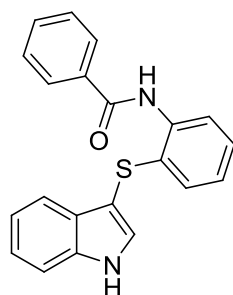
3t

3-(3-Fluorophenylthio)-1H-indole (3t).⁸ Eluent: petroleum ether / ethyl acetate (10:1). White solid. ¹H NMR (DMSO-d₆, 400 MHz) δ 11.85 (s, 1H), 7.85 (s, 1H), 7.51 (d, 2H, *J* = 45.6 Hz), 6.78-7.22 (m, 6H). ¹³C NMR (DMSO-d₆, 100 MHz) δ 162.5 (*J*_{CF} = 244.6 Hz), 142.4 (*J*_{CF} = 6.2 Hz), 136.8, 132.8, 130.5 (*J*_{CF} = 9.1 Hz), 128.5, 122.3, 121.1, 120.3, 118.2, 112.5, 111.6 (*J*_{CF} = 36.6 Hz), 111.5 (*J*_{CF} = 7.1 Hz), 98.3.



3u

3-(3,5-Dichlorophenylthio)-1H-indole (3u) [New compound]. Eluent: petroleum ether / ethyl acetate (10:1). White solid. ¹H NMR (DMSO-d₆, 400 MHz) δ 11.90 (s, 1H), 7.88 (d, 1H, *J* = 2.8 Hz), 7.56 (d, 1H, *J* = 8.0 Hz), 7.43 (d, 1H, *J* = 8.0 Hz), 7.28 (s, 1H), 7.24 (t, 1H, *J* = 7.6 Hz), 7.13 (t, 1H, *J* = 7.6 Hz), 6.99 (d, 2H, *J* = 1.6 Hz). ¹³C NMR (DMSO-d₆, 100 MHz) δ 144.3, 136.8, 134.5, 133.3, 128.1, 124.3, 123.0, 122.4, 120.5, 118.0, 112.6, 97.0. HRMS (EI) Calcd for C₁₄H₉NSCl₂ (M)⁺ 292.9833; Found, *m/z* (%) = 292.9834 ([M]⁺, 100), 294.9809 ([M+2]⁺, 70.44).



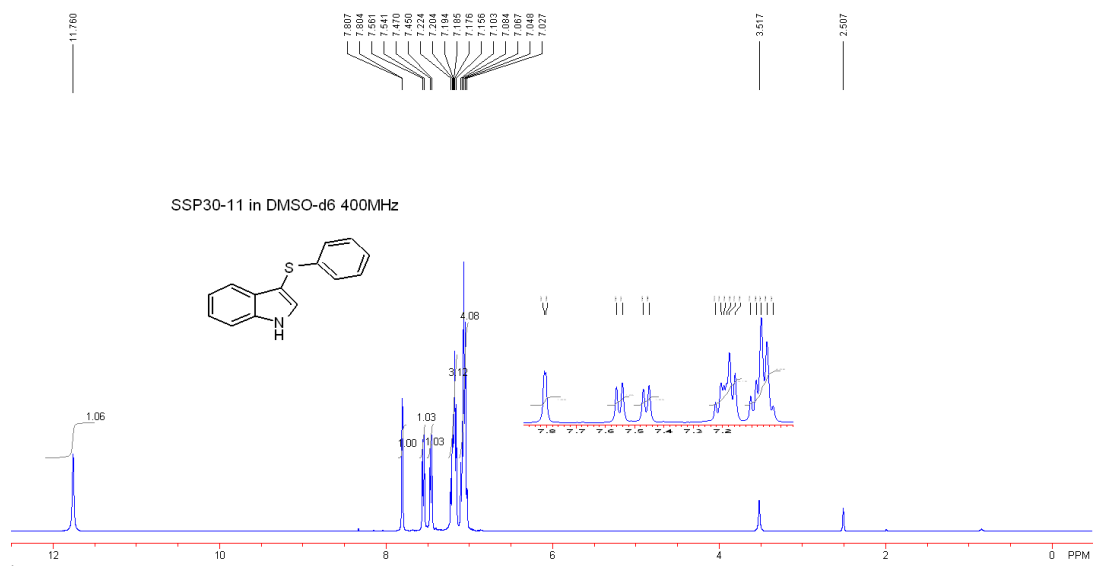
3v

N-(2-(1H-indol-3-ylthio)phenyl)benzamide (3v) [New compound]. Eluent: petroleum ether / ethyl acetate (5:1). White solid. ¹H NMR (DMSO-d₆, 400 MHz) δ 11.70 (s, 1H), 10.17 (s, 1H), 8.10 (d, 2H, *J* = 6.8 Hz), 7.75 (s, 1H), 7.57-7.62 (m, 3H), 7.51 (d, 1H, *J* = 8.0 Hz), 7.41 (t, 2H, *J* = 8.0 Hz), 7.19 (t, 1H, *J* = 8.0 Hz), 7.13 (t, 1H,

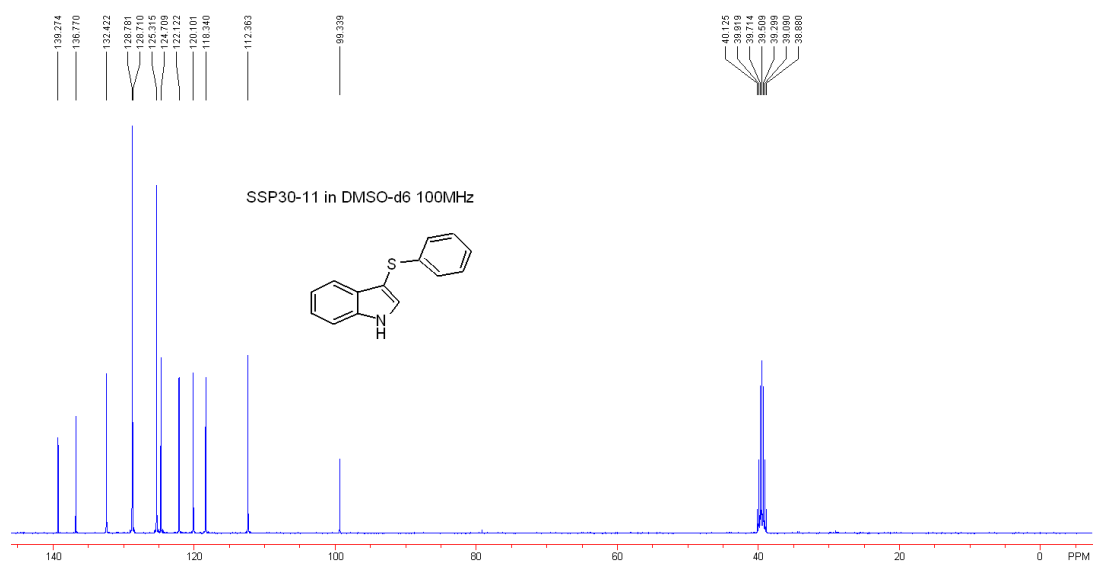
$J = 7.2$ Hz), 7.02-7.09 (m, 2H), 6.77 (d, 1H, $J = 7.6$ Hz). ^{13}C NMR (DMSO- d_6 , 100 MHz) δ 165.6, 136.8, 136.6, 134.3, 132.4, 131.7, 128.7, 128.5, 127.7, 127.6, 126.7, 126.5, 125.1, 122.1, 120.0, 118.4, 112.3, 99.2. HRMS (EI) Calcd for $\text{C}_{21}\text{H}_{16}\text{N}_2\text{OS}$ (M)⁺ 344.0983; Found, 344.0988.

References

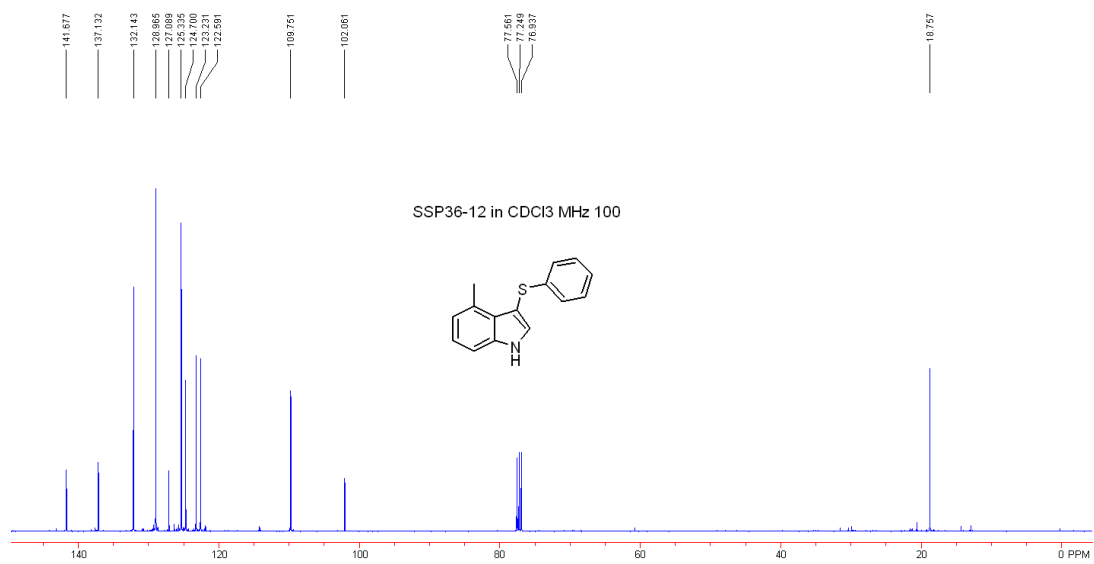
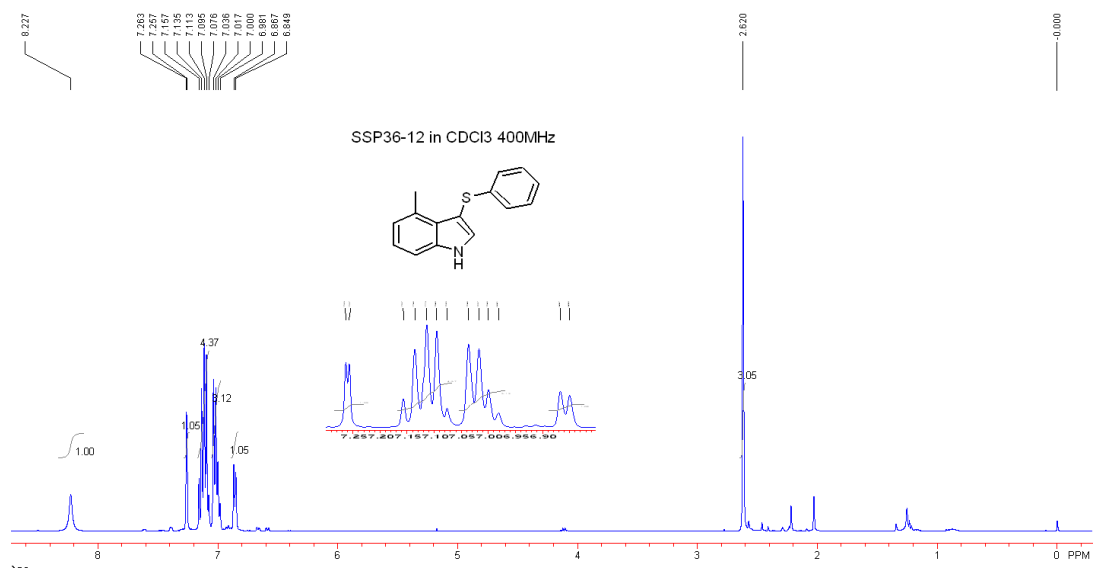
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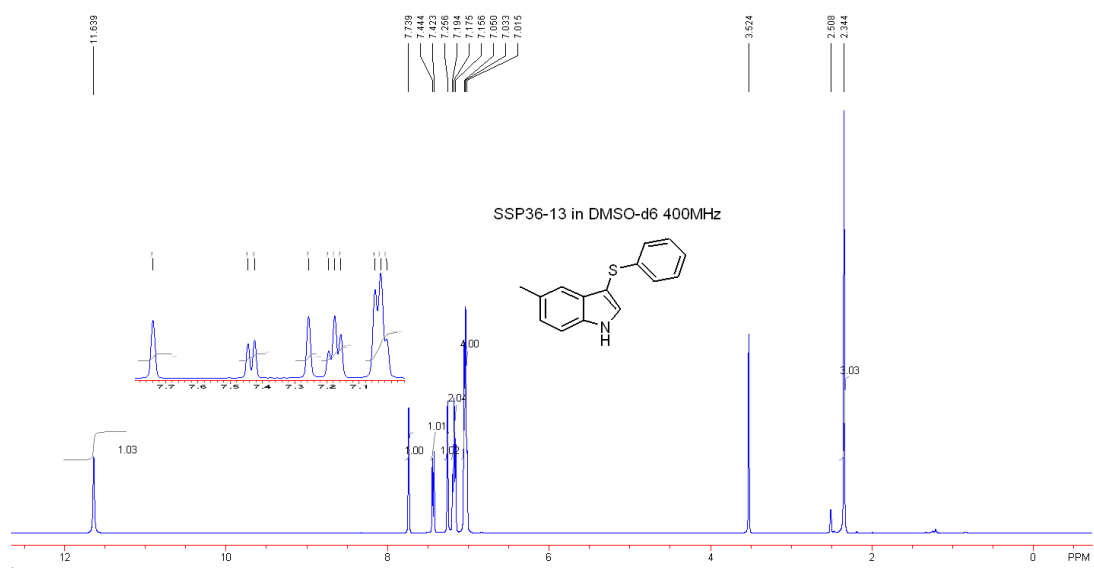


^1H NMR of product **3a**

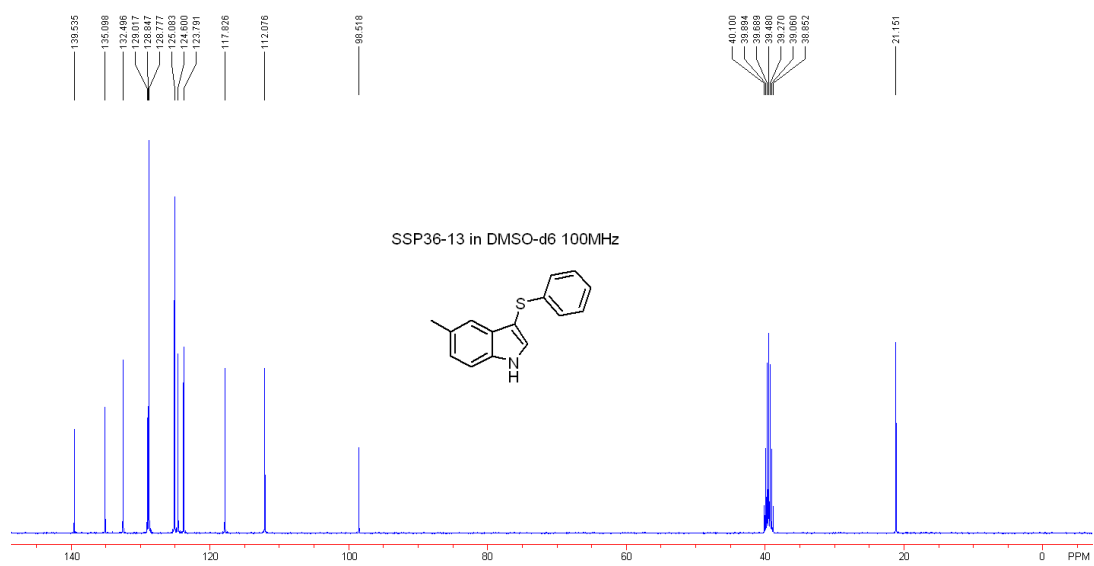


^{13}C NMR of product **3a**

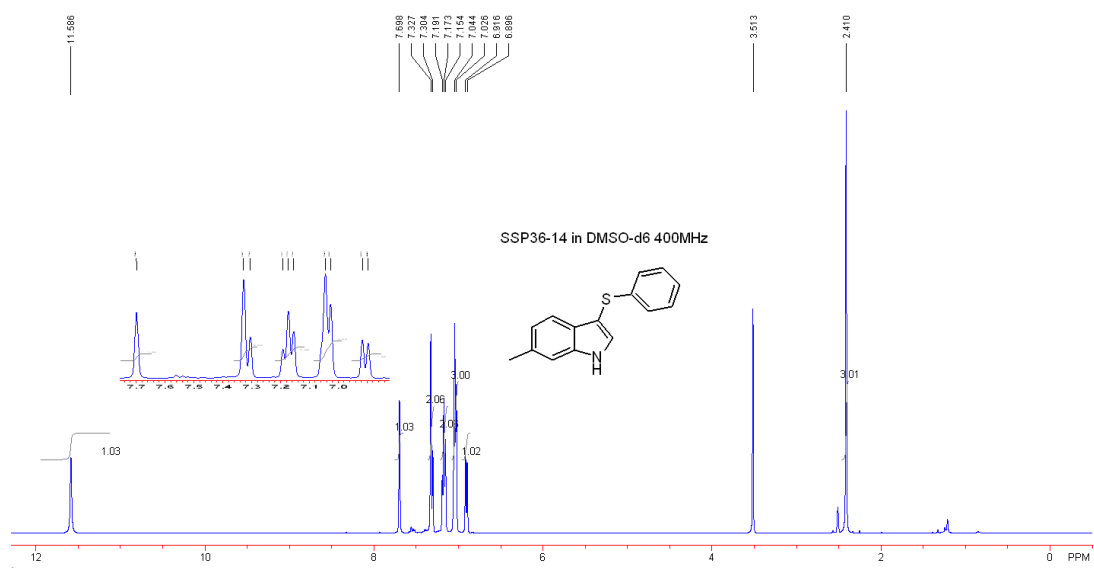




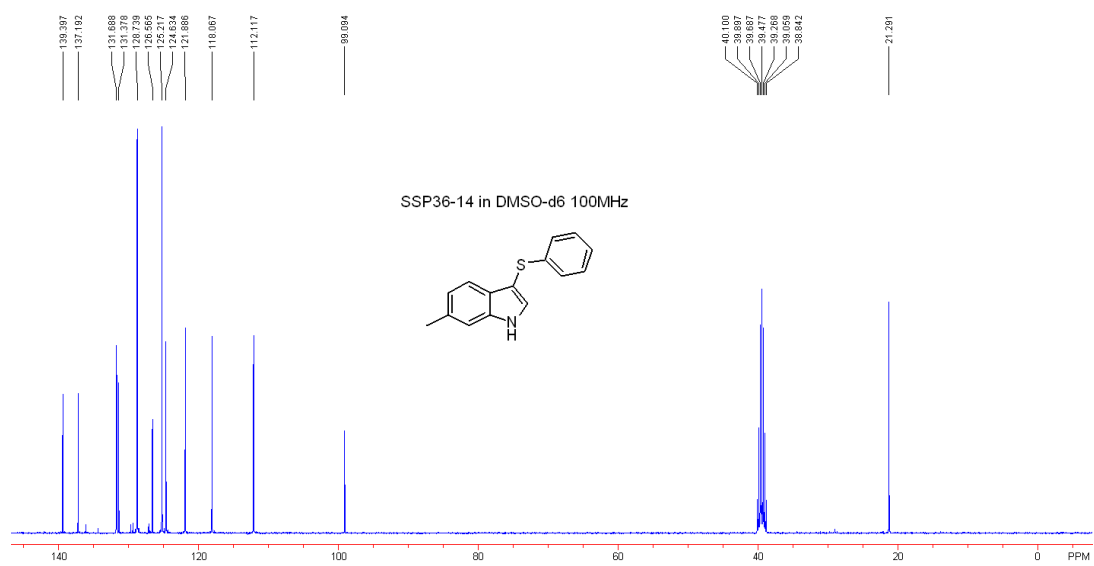
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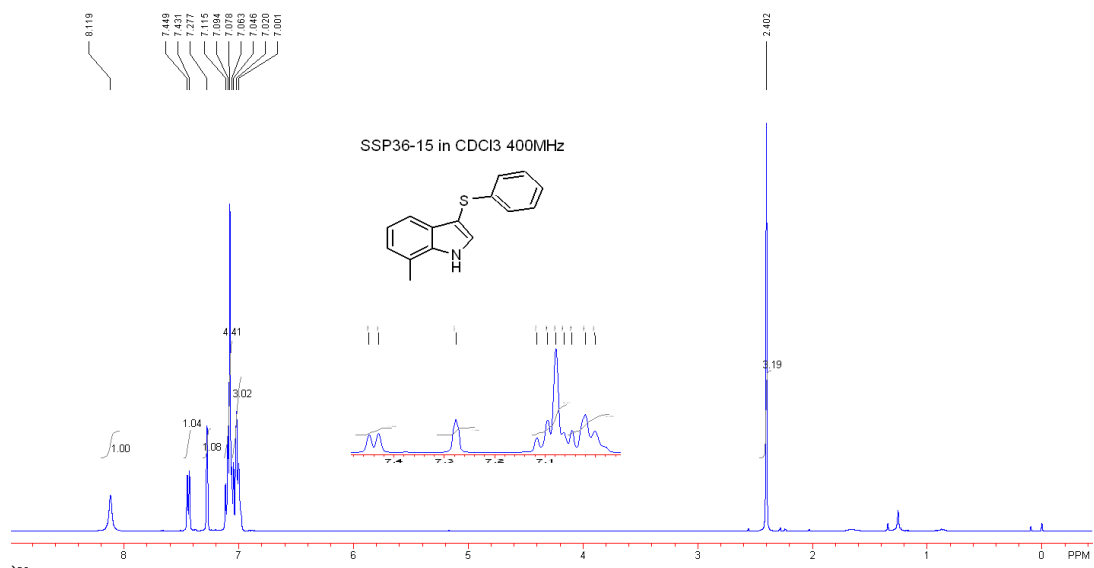
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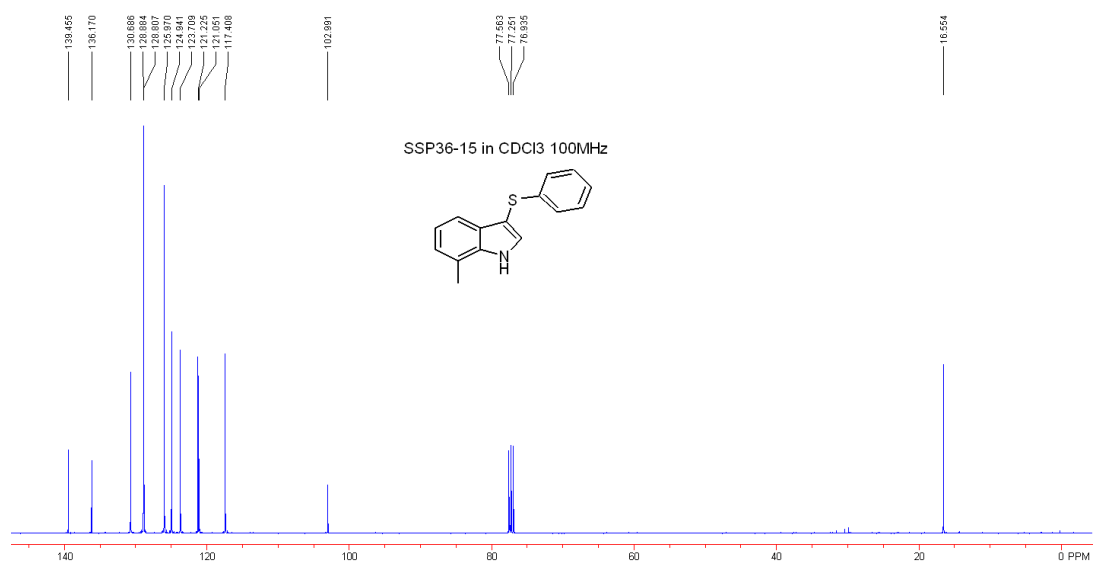
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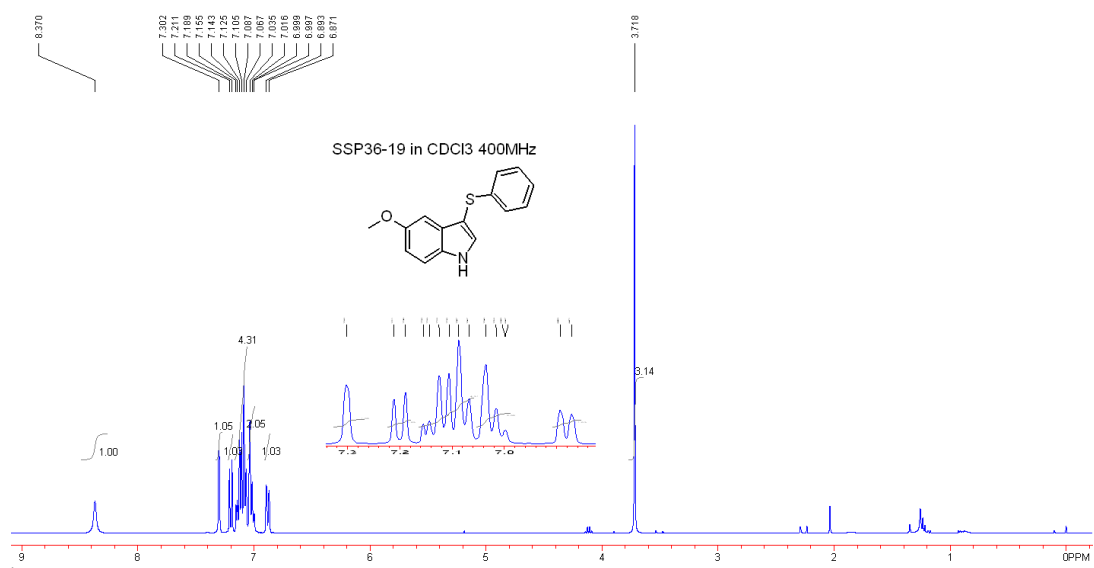
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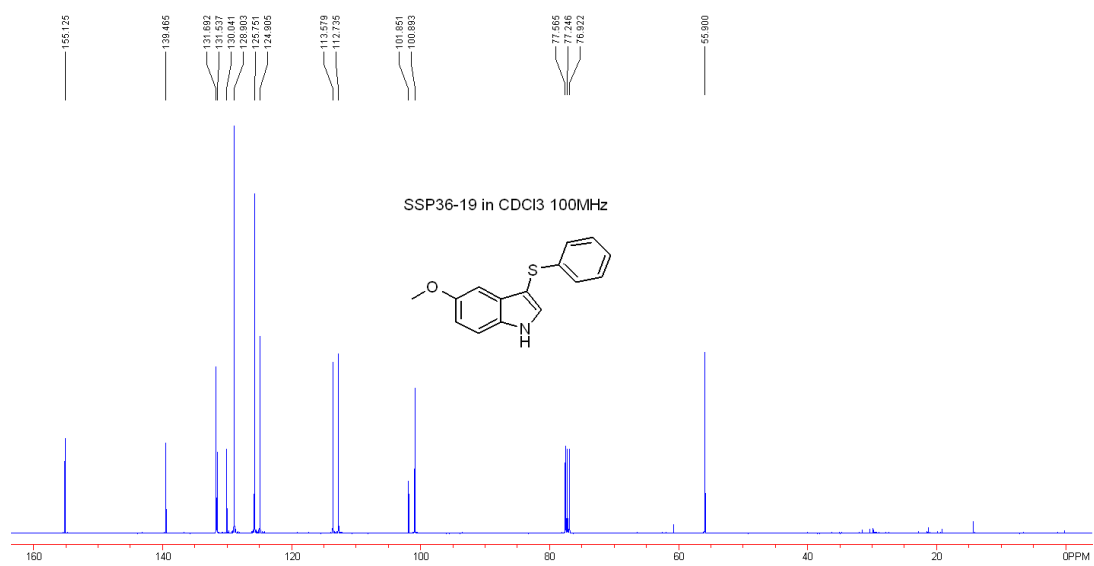
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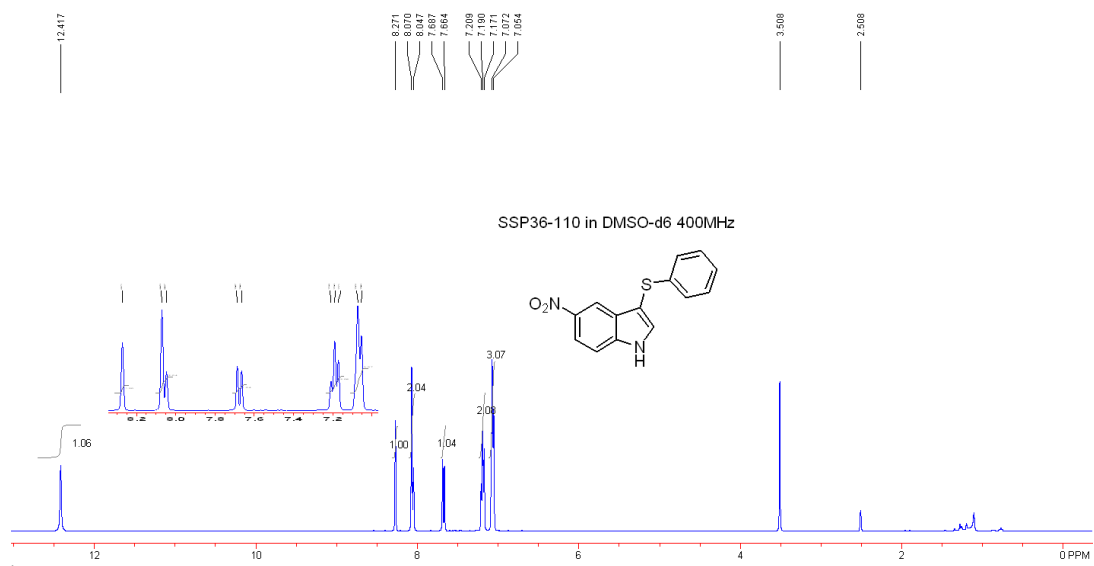
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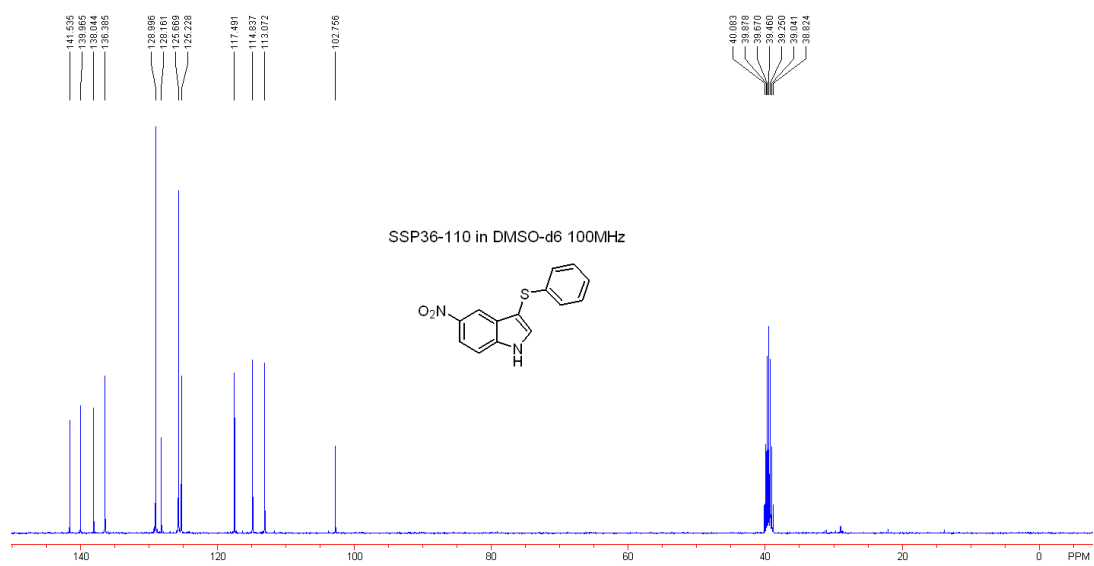
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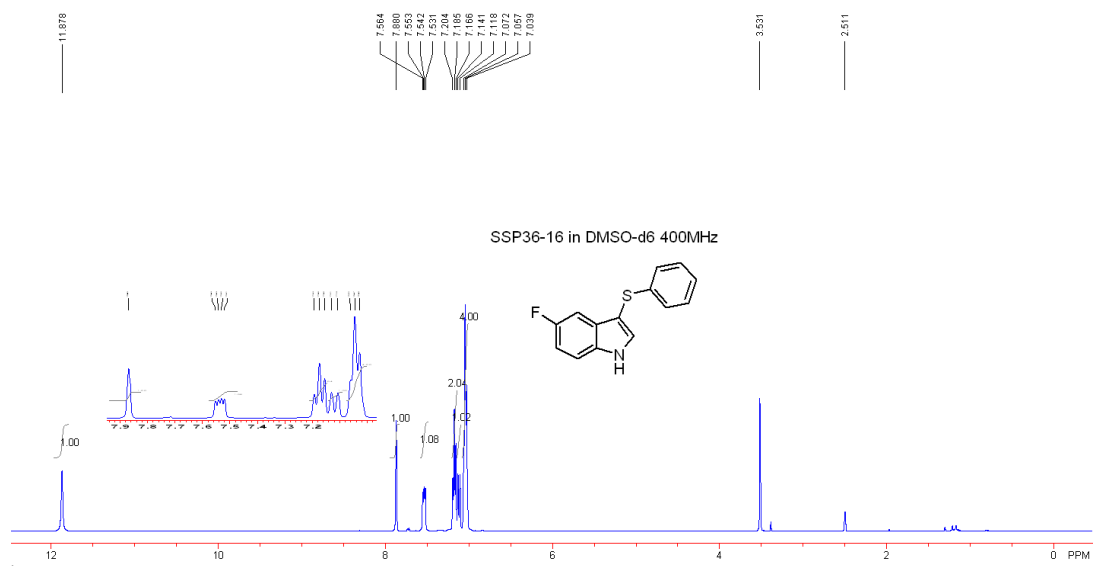
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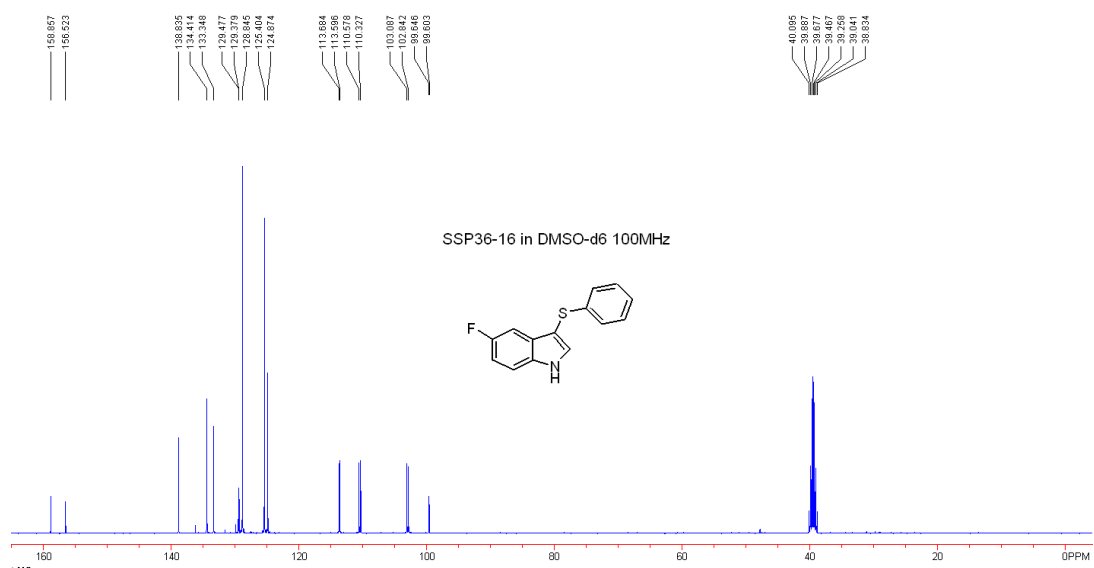
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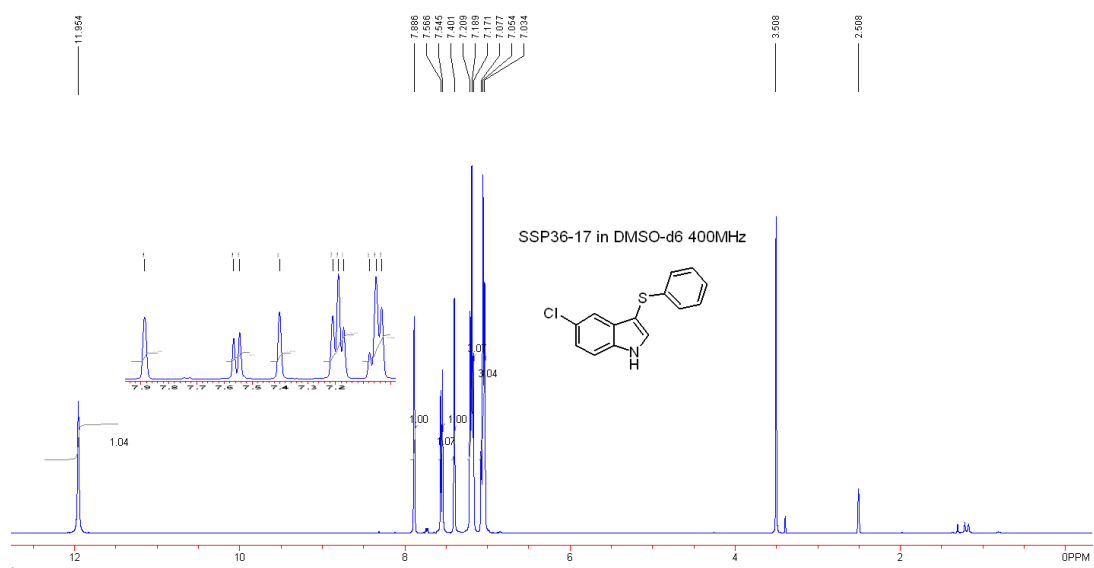
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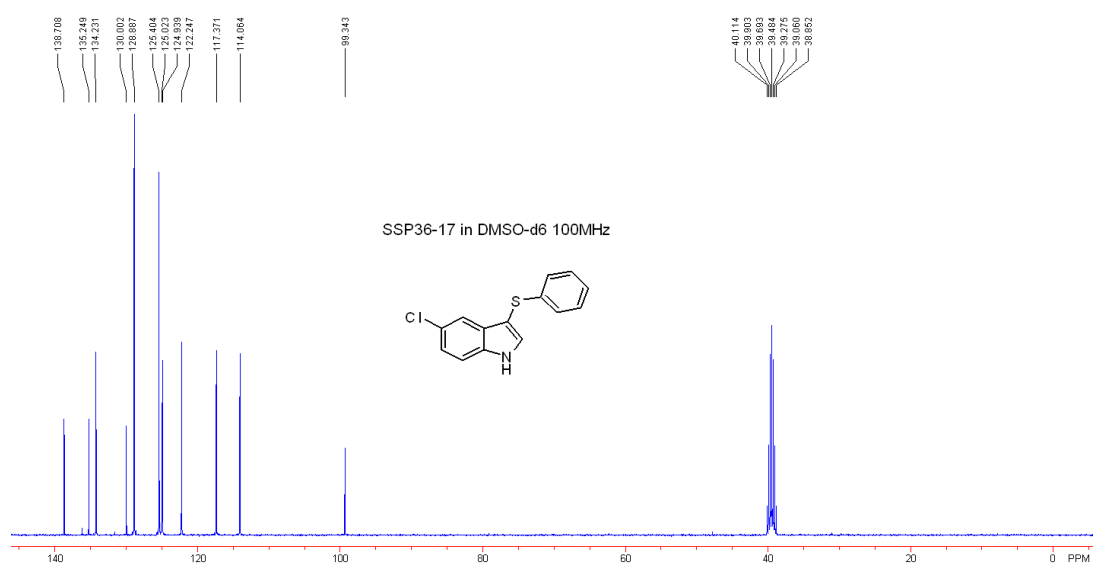
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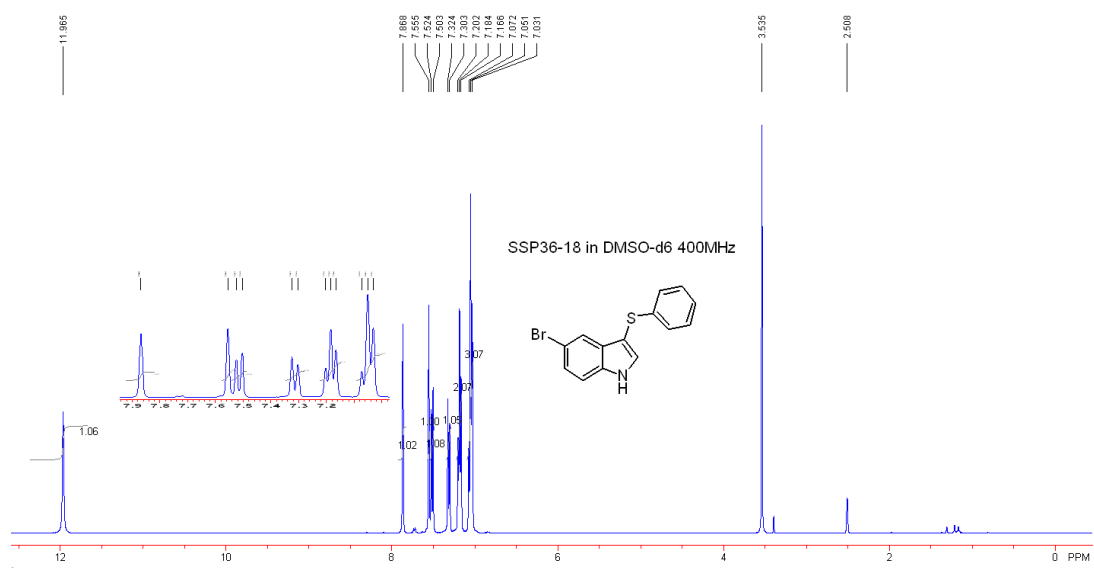
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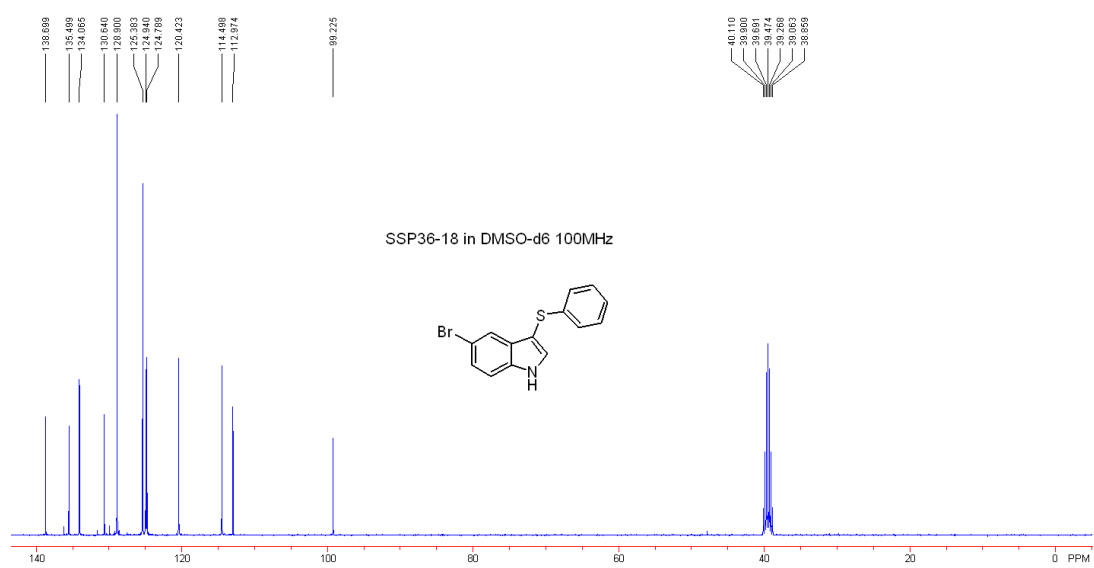
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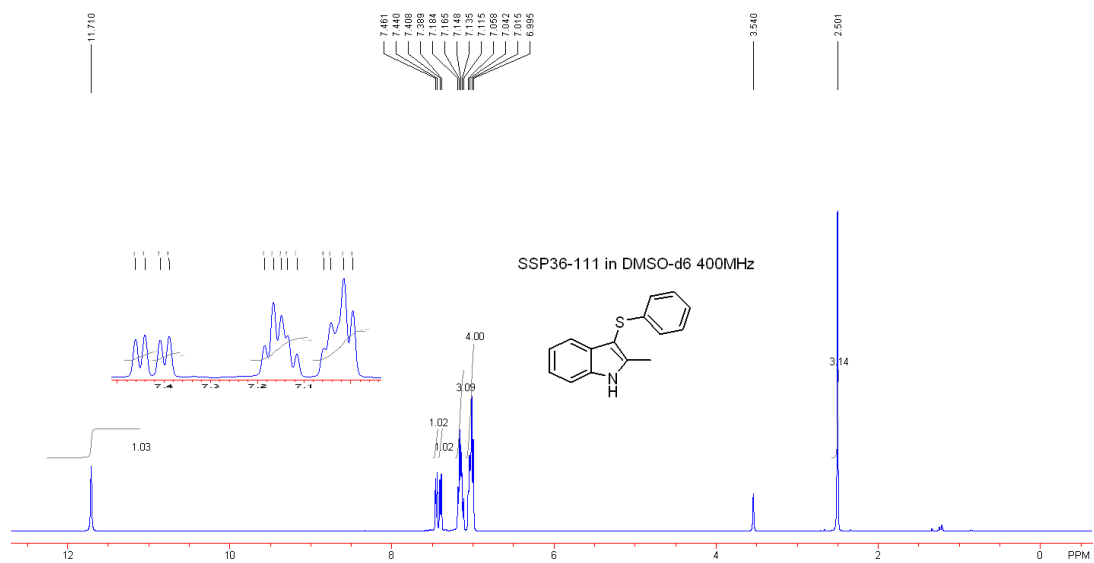
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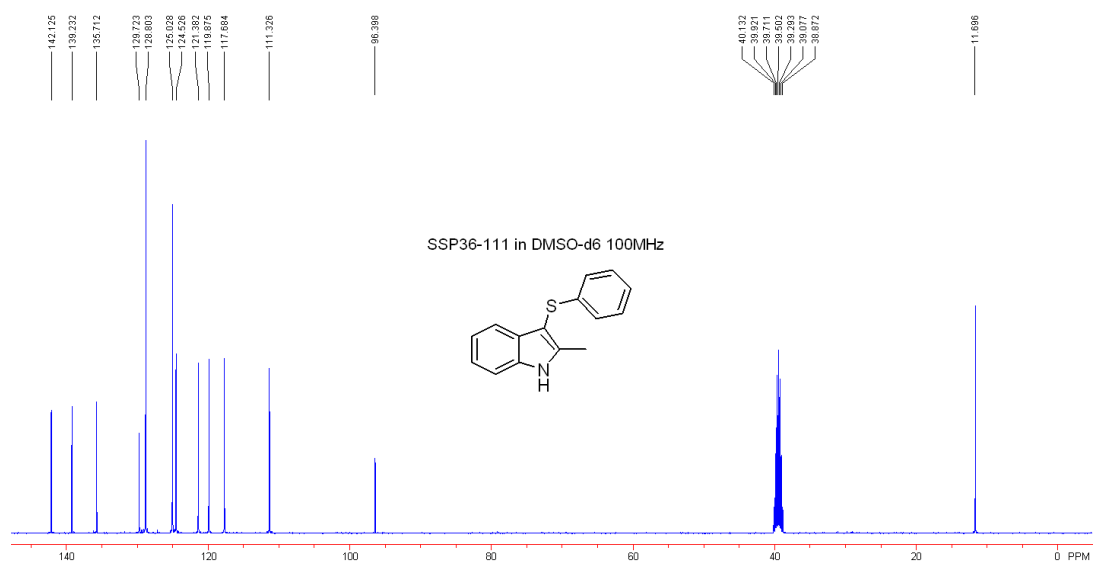
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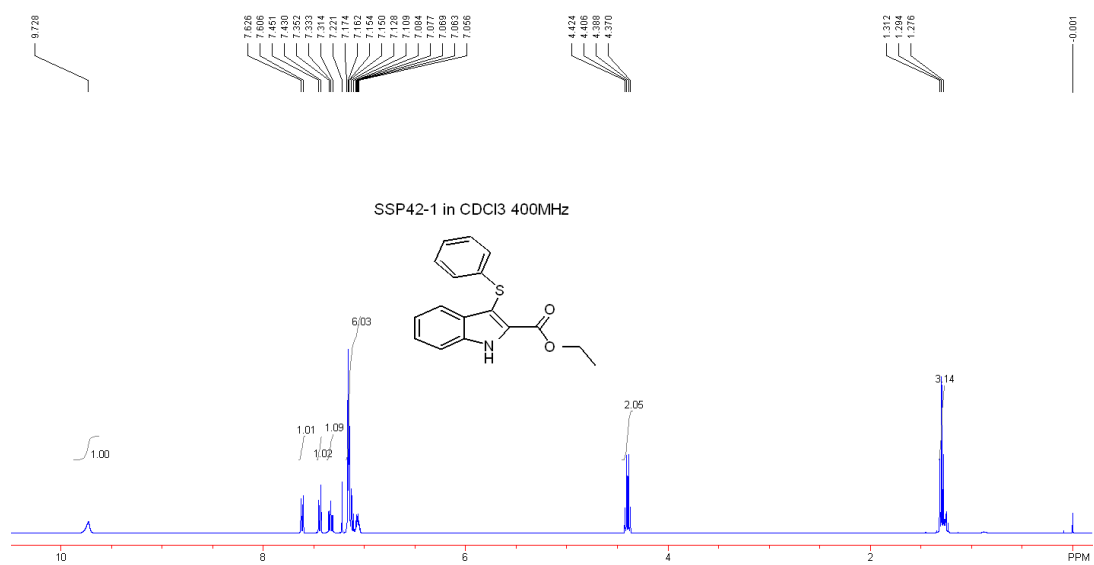
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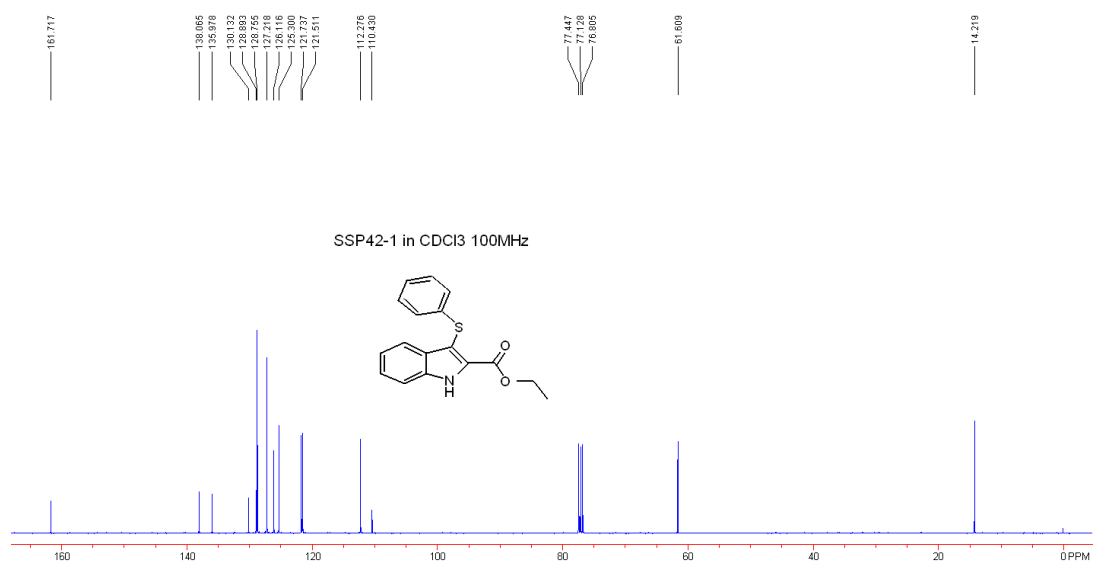
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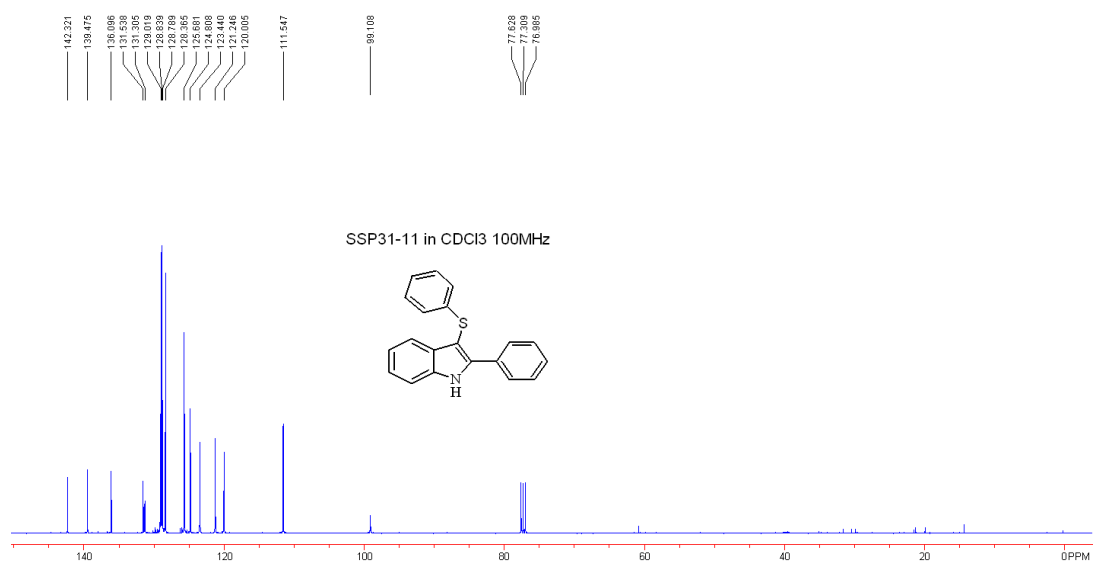
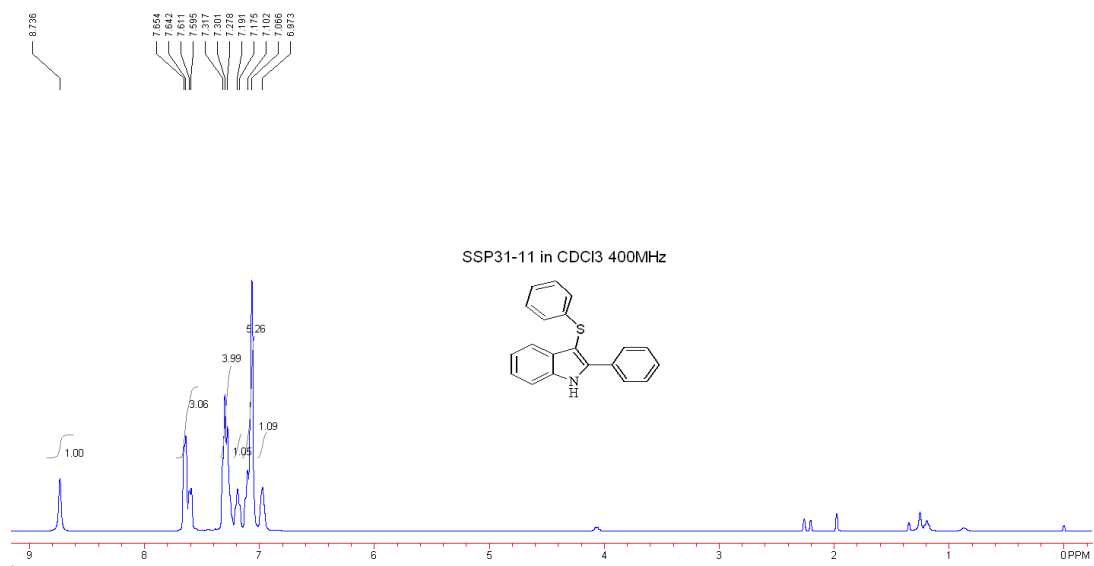
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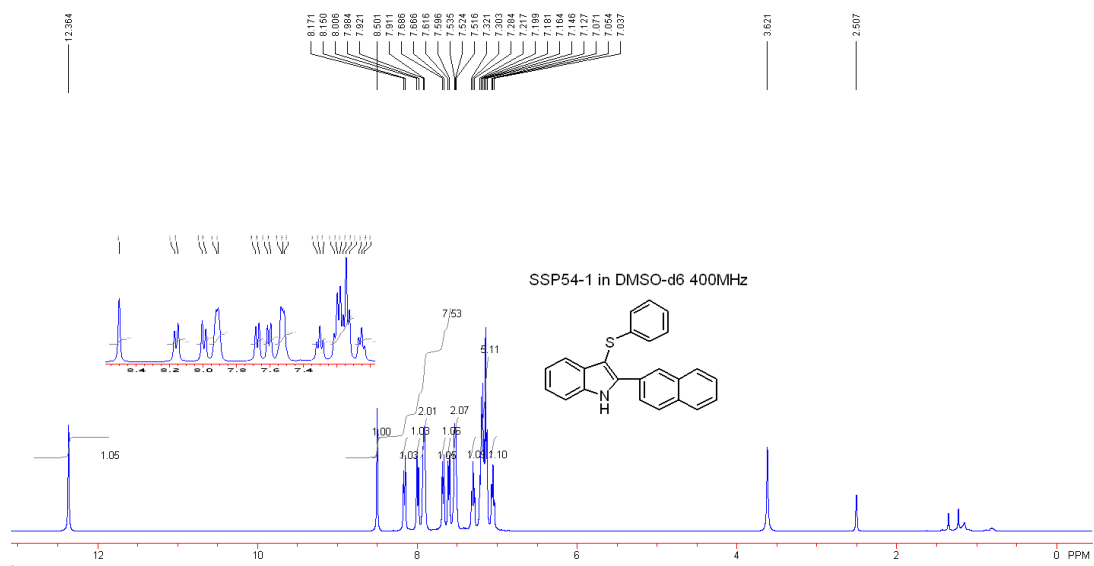


¹H NMR of product 3m

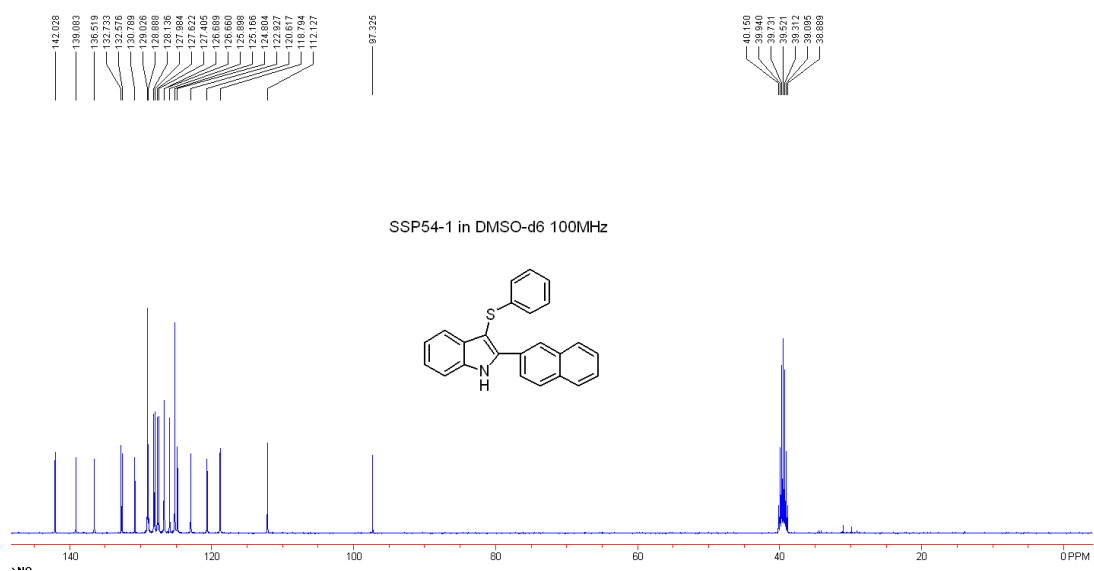


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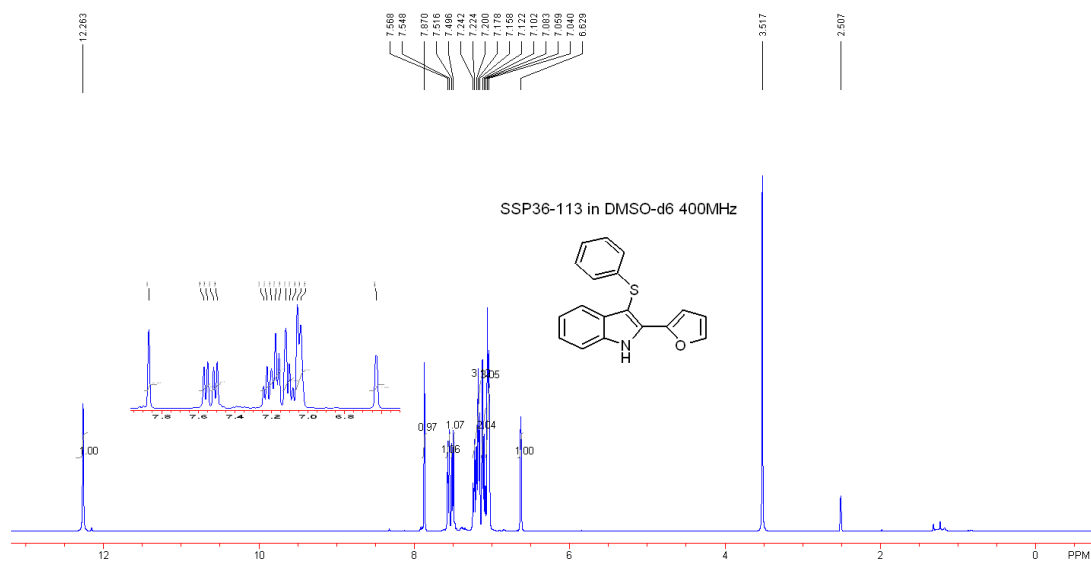




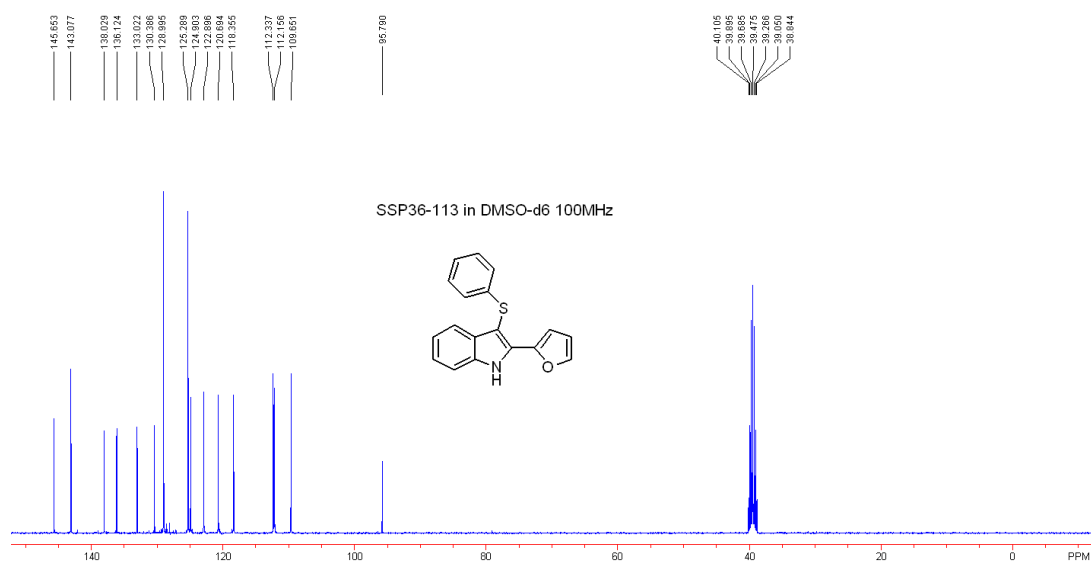
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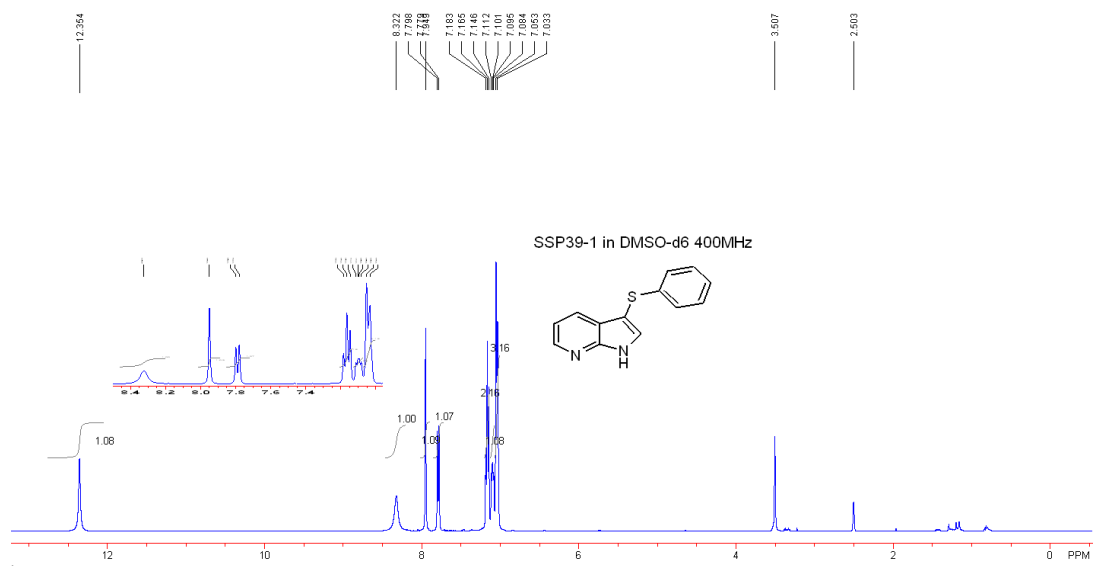
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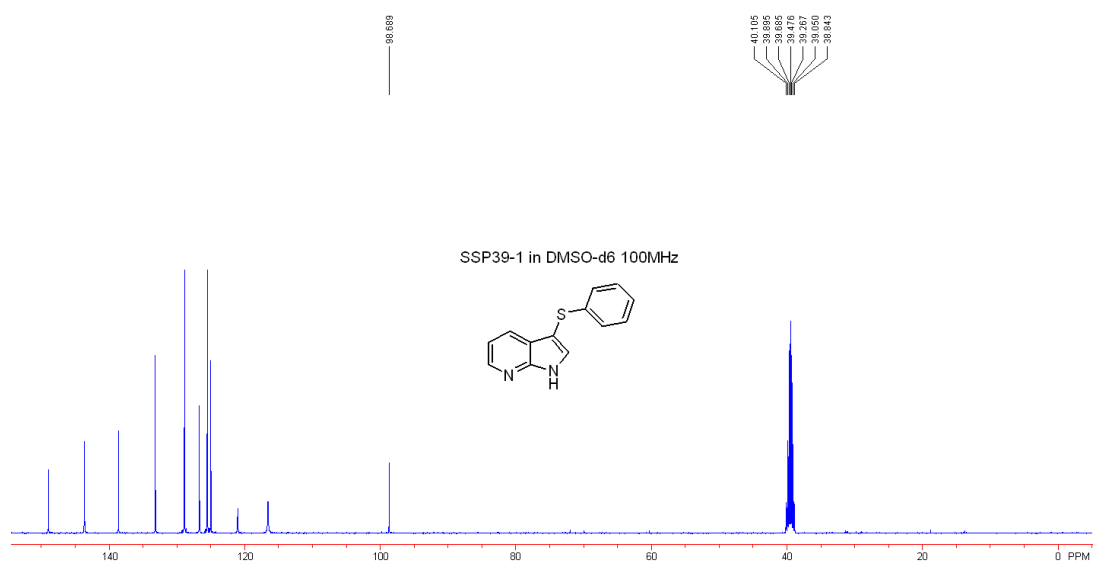
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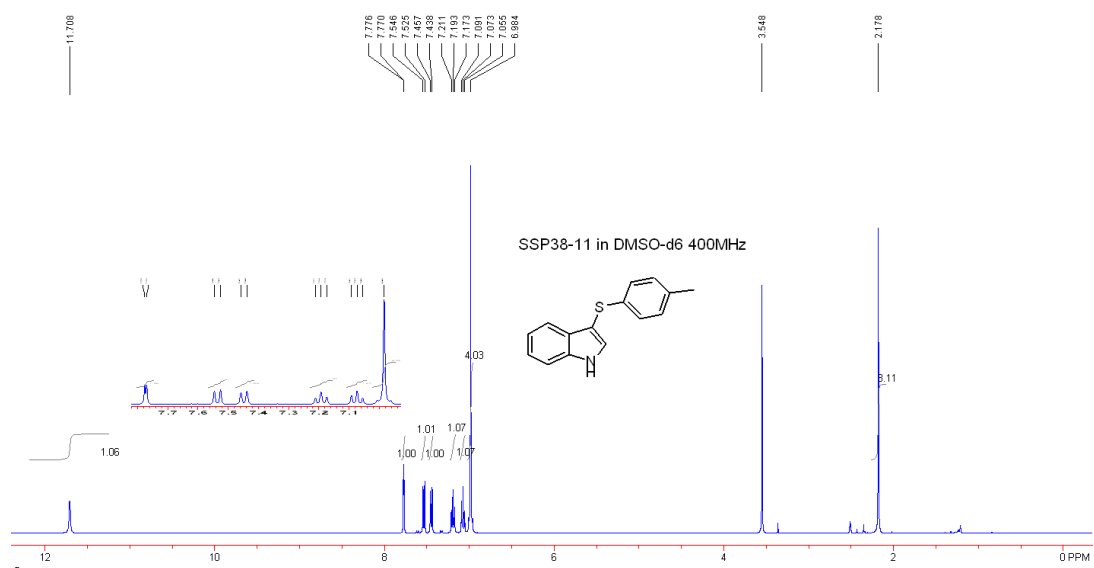
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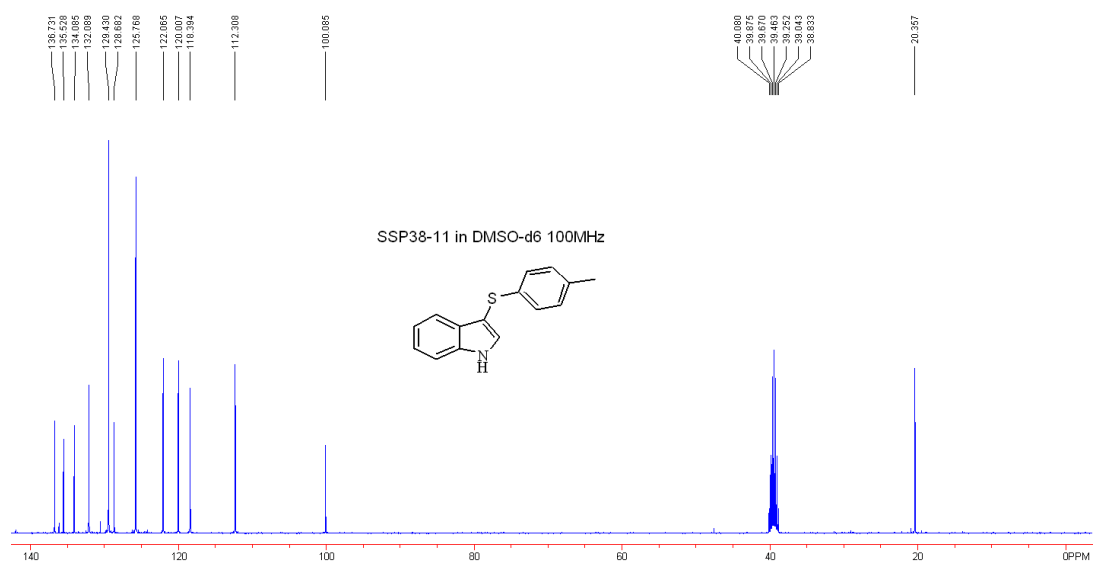
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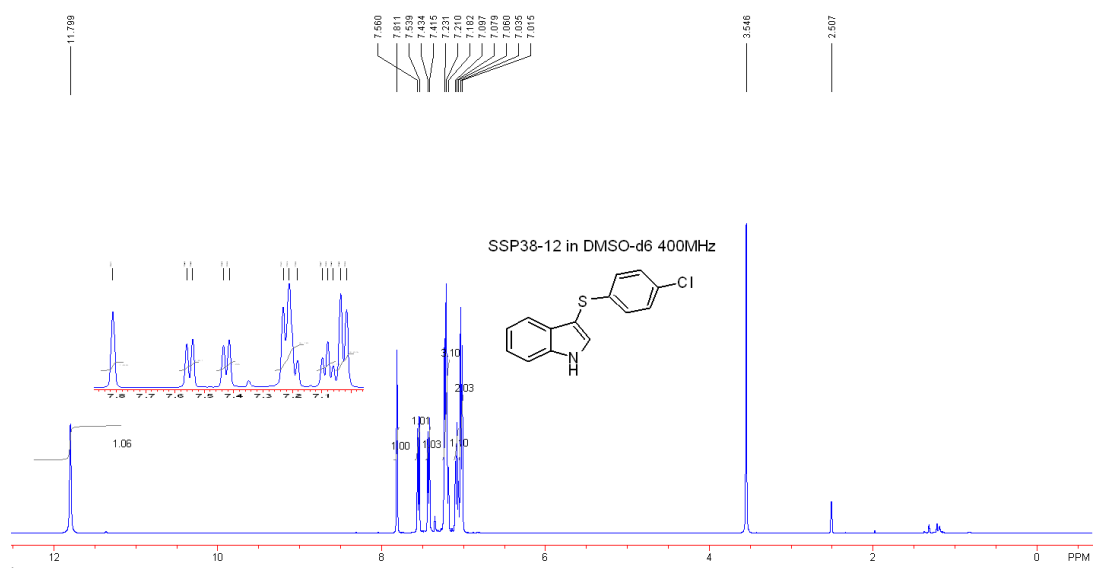
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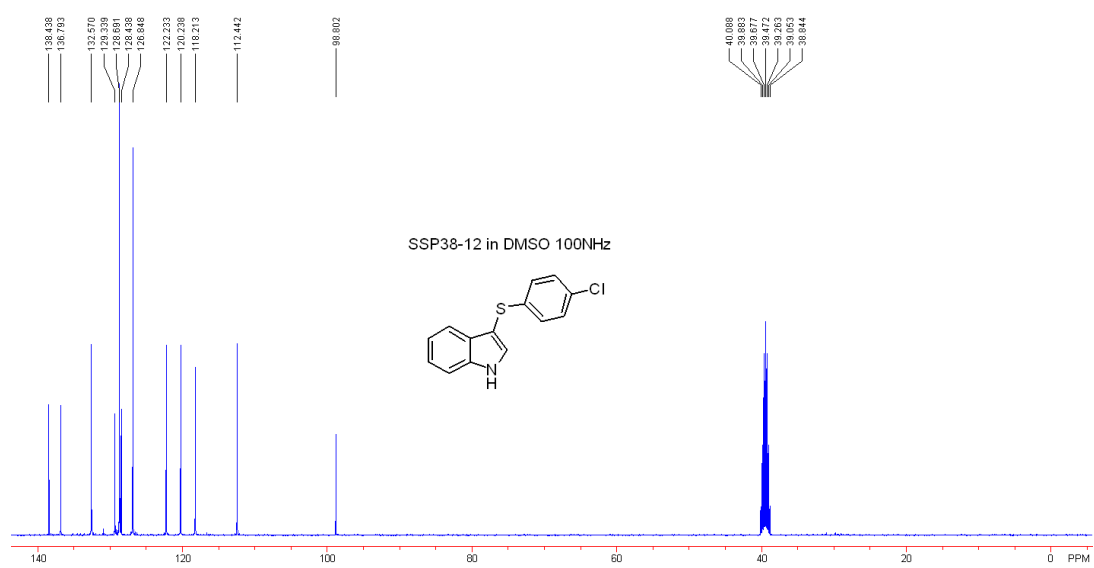
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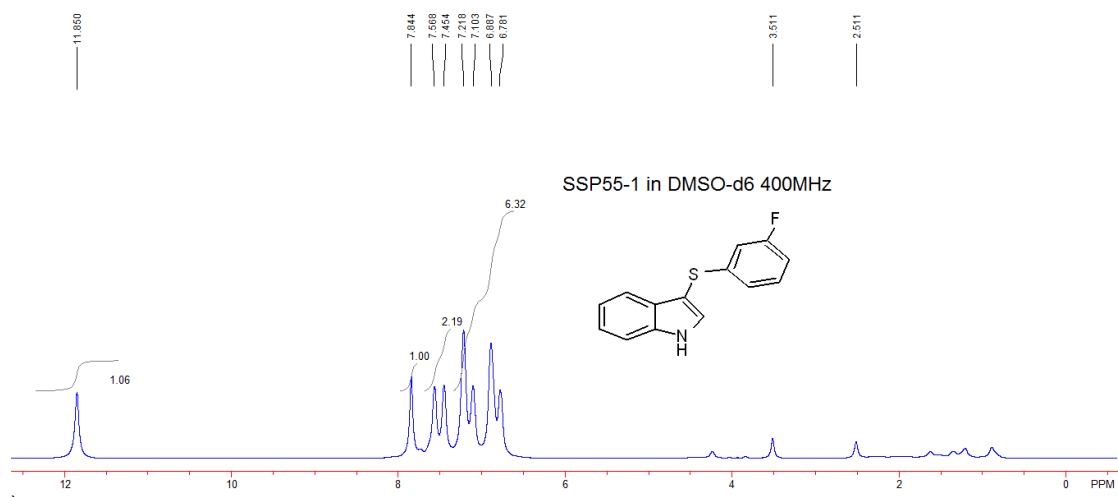
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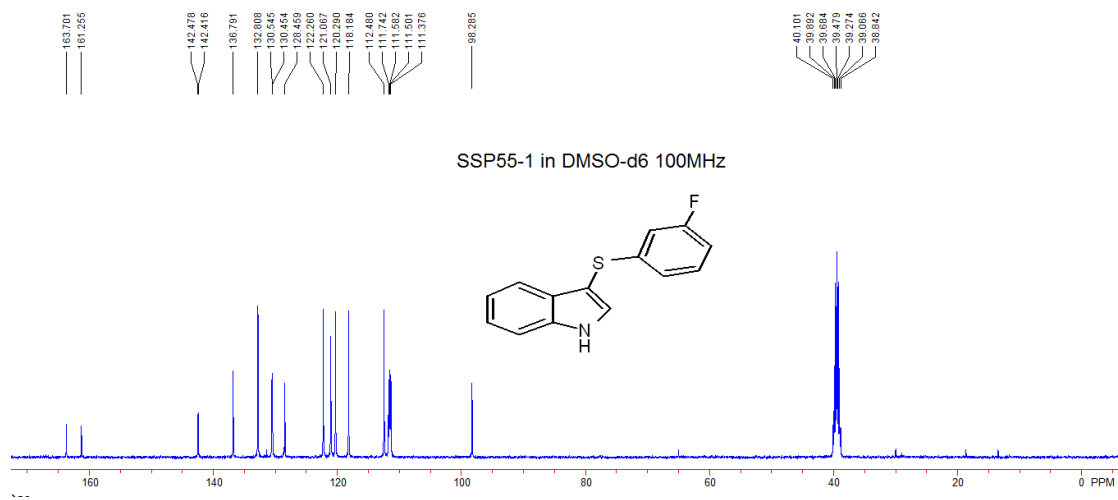
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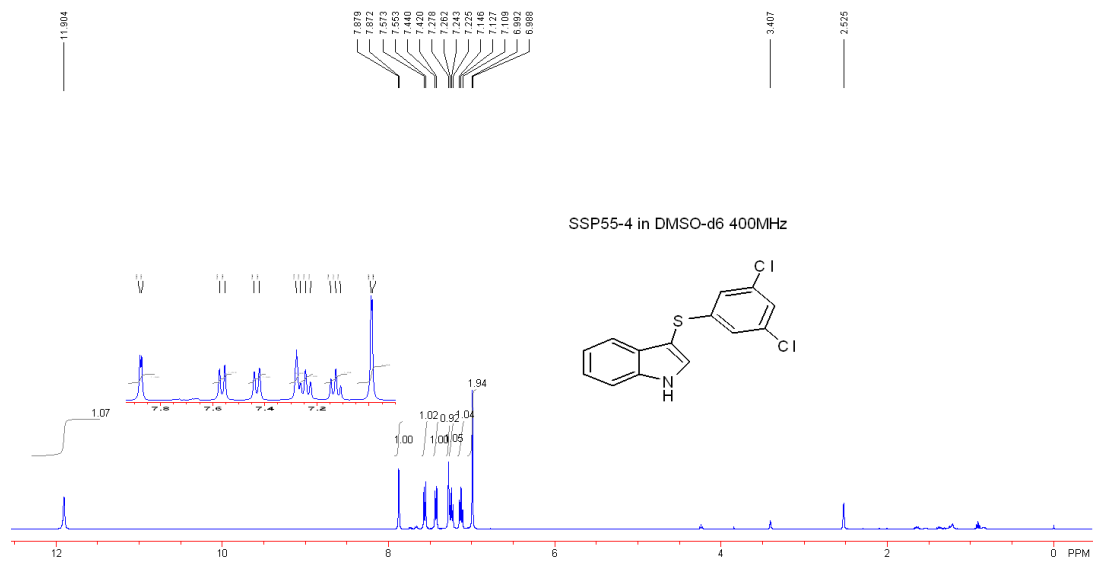
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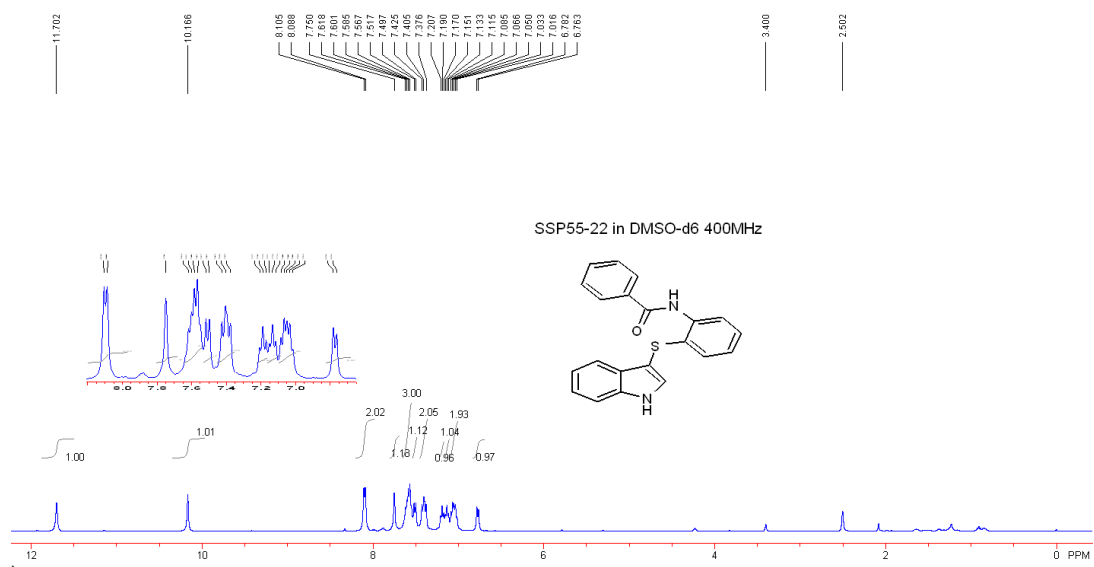
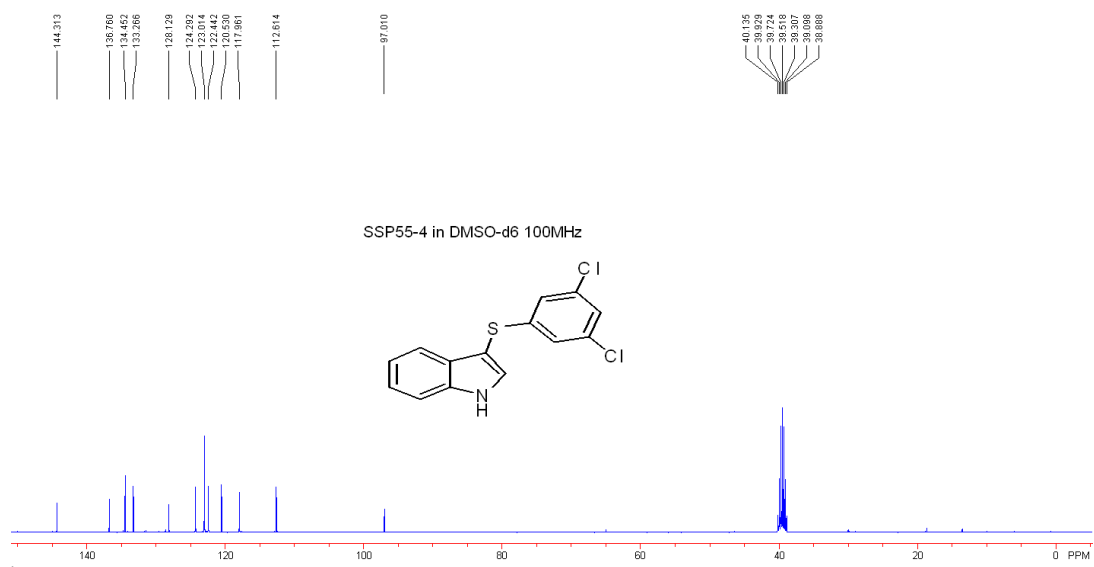
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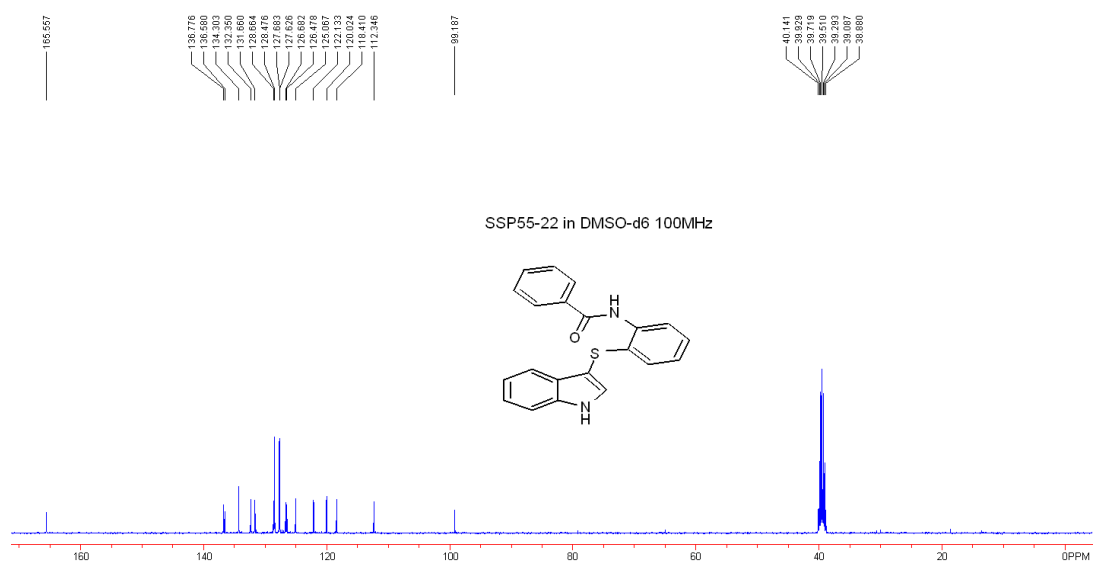


¹³C NMR of product 3t



¹H NMR of product 3u





¹³C NMR of product 3v