Electronic Supplementary Information

Iodine-mediated 3-sulfenylation of indoles with sodium sulfinates

Praewpan Katrun,[a] Sakchai Hongthong,[a] Sornsiri Hlekhlai,[a] Manat Pohmakotr,[a] Vichai Reutrakul,[a] Darunee Soorukram,[a] Thaworn Jaipetch[b] and Chutima Kuhakarn*[a]

[a]Department of Chemistry and Center of Excellence for Innovation in Chemistry (PERCH-CIC), Faculty of Science, Mahidol University, Rama VI Road, Bangkok 10400, Thailand
[b]Mahidol University, Kanchanaburi Campus, Saiyok, Kanchanaburi 71150, Thailand

E-mail: chutima.kon@mahidol.ac.th

Contents

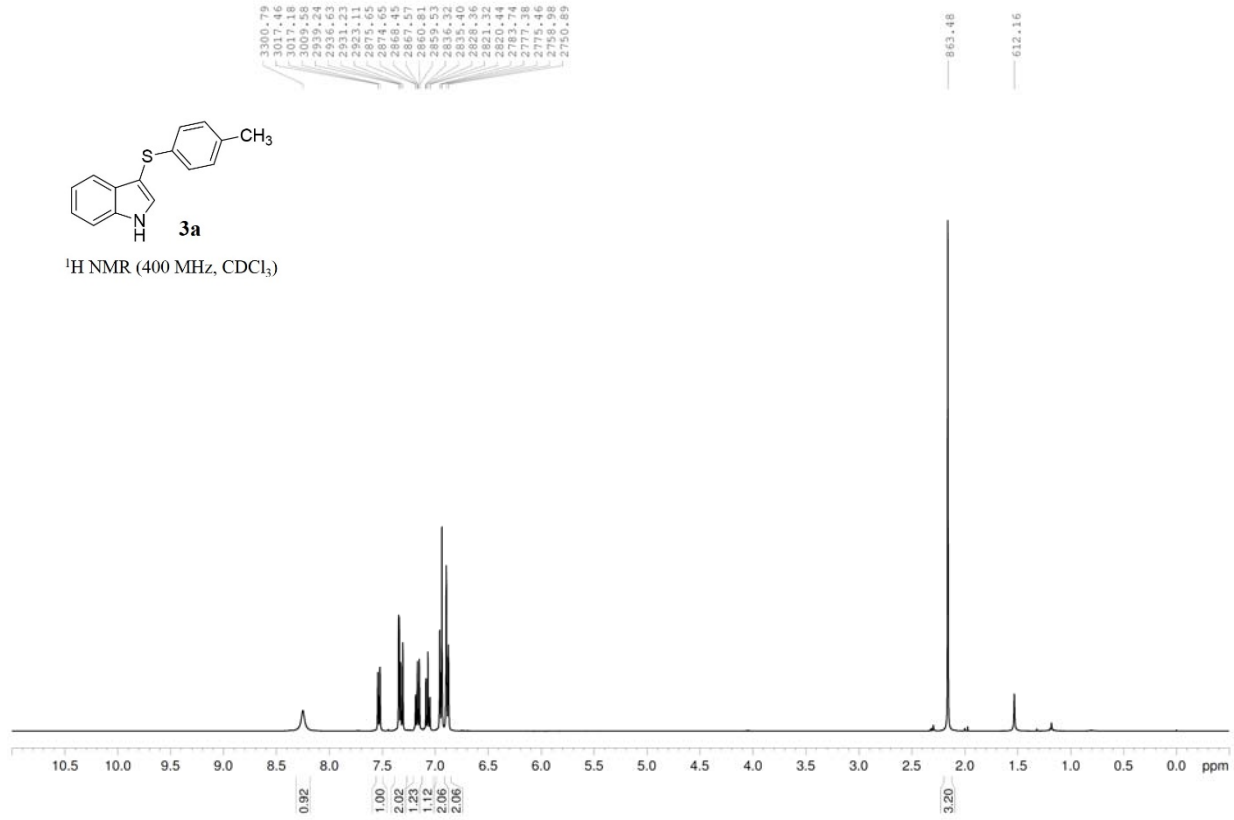
General information

^{1}H NMR spectra were recorded with a Bruker Ascend^{TM} 400 (400 MHz) and Bruker Avance-500 (500 MHz) spectrometer in $CDCl_3$ by using tetramethylsilane (δ = 0 ppm) as an internal standard. ^{13}C NMR spectra were recorded with a Bruker Ascend^{TM} 400 (400 MHz) and Bruker Avance-500 (500 MHz) spectrometer. Infrared spectra were recorded with a Perkin–Elmer 683 GX FTIR System spectrometer. High-resolution mass spectra (HRMS) were recorded with a Bruker micro TOF spectrometer in the ESI mode. Melting points were recorded with a digital Electrothermal Melting 9100 apparatus and were uncorrected. All reagents and solvents were obtained from commercial sources and used without further purification. Column chromatography was performed by using Merck silica gel 60 PF_{254} (Art 7734).

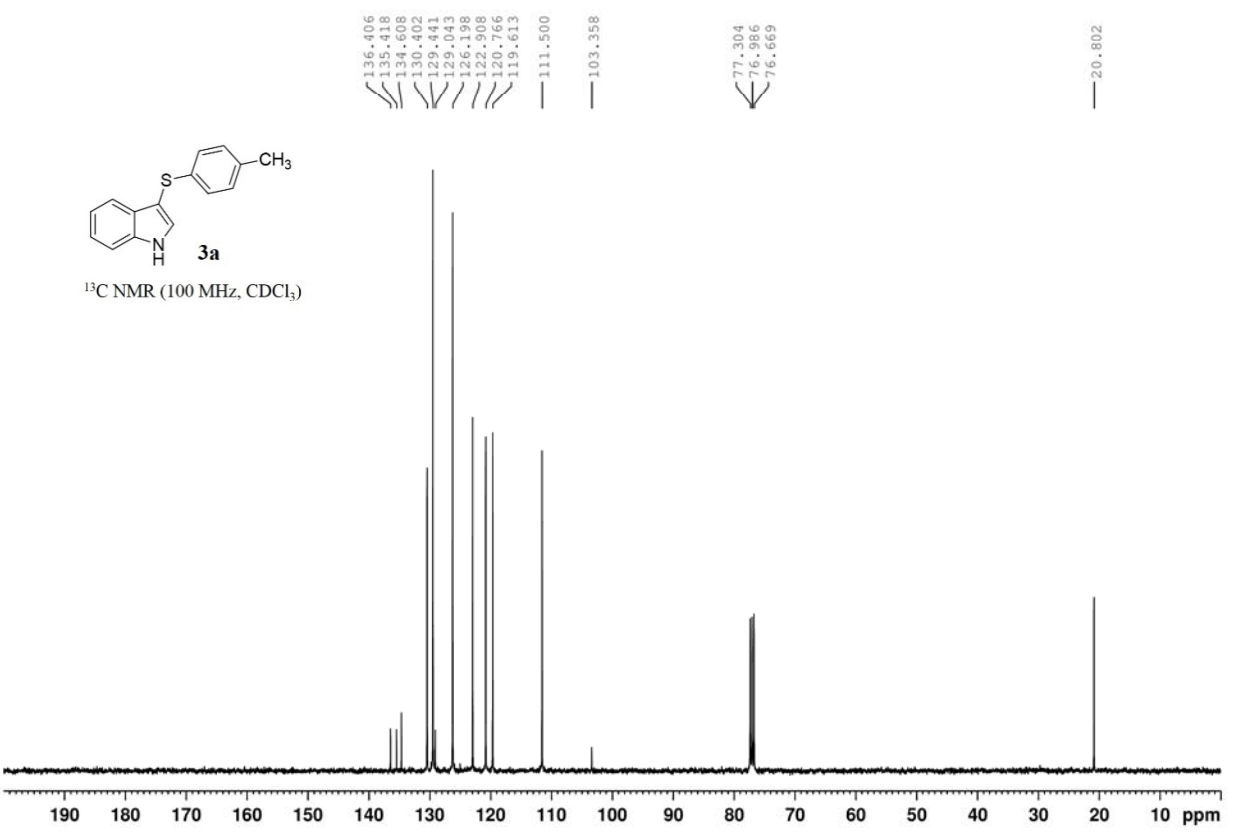
General procedure for the 3-sulfenylation of indoles

Iodine (127.0 mg, 0.50 mmol) was added to a solution of indole (0.5 mmol), sodium arenesulfinate (0.75 mmol) and triphenylphosphine (0.75 mmol) in EtOH (2 mL; 0.25 M), and the reaction mixture was stirred at refluxing temperature for 2-8 h. The reaction mixture was quenched by the addition of sat. aq $Na_2S_2O_3$ (5 mL). Further stirring was followed by extraction with EtOAc (2 × 20 mL). The combined organic extracts were washed with H_2O (20 mL) and brine (20 mL), dried ($MgSO_4$), filtered, and concentrated (aspirator). The residue was purified by column chromatography using acetone/hexanes as eluent to afford the corresponding product.

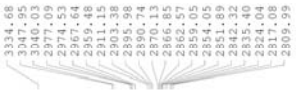
^{1}H and ^{13}C NMR spectra

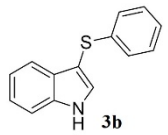


^{1}H NMR (400 MHz, $CDCl_3$)



^{13}C NMR (100 MHz, $CDCl_3$)





3b

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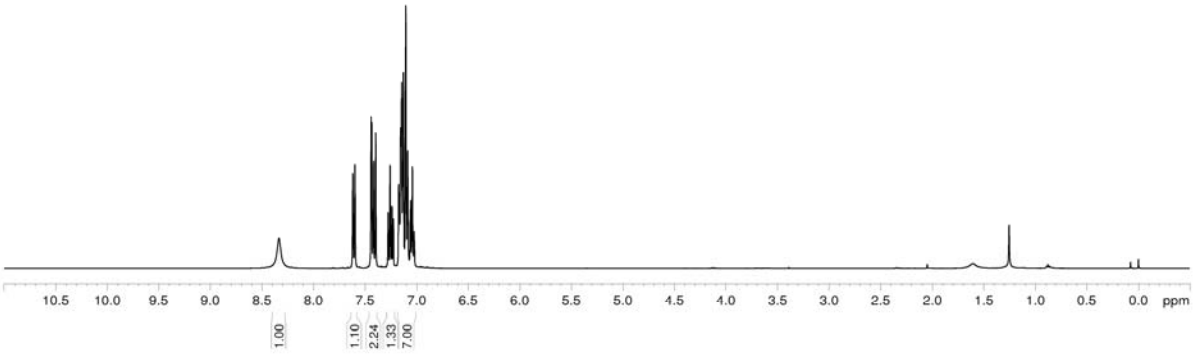

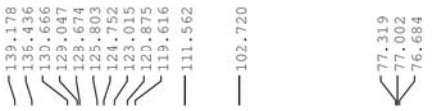

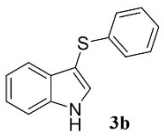

3b

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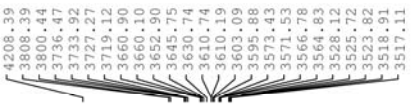

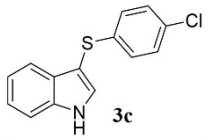

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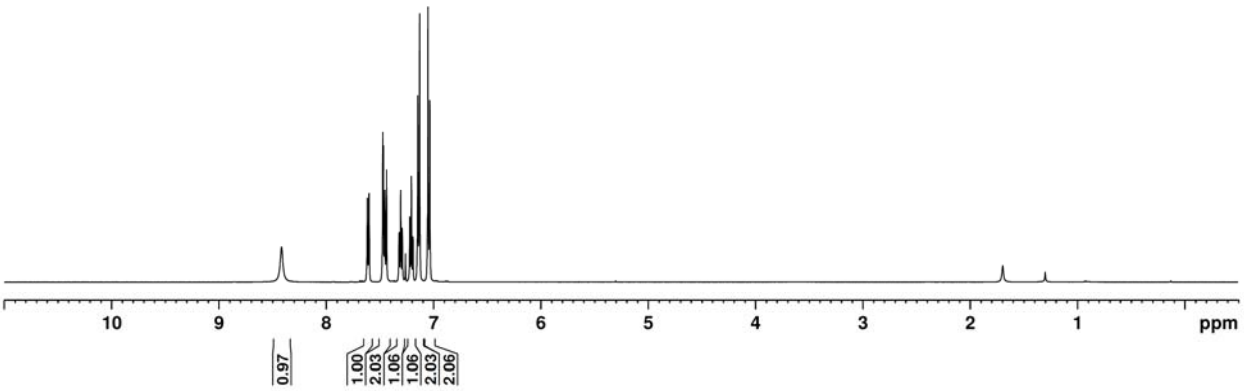
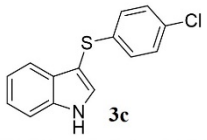
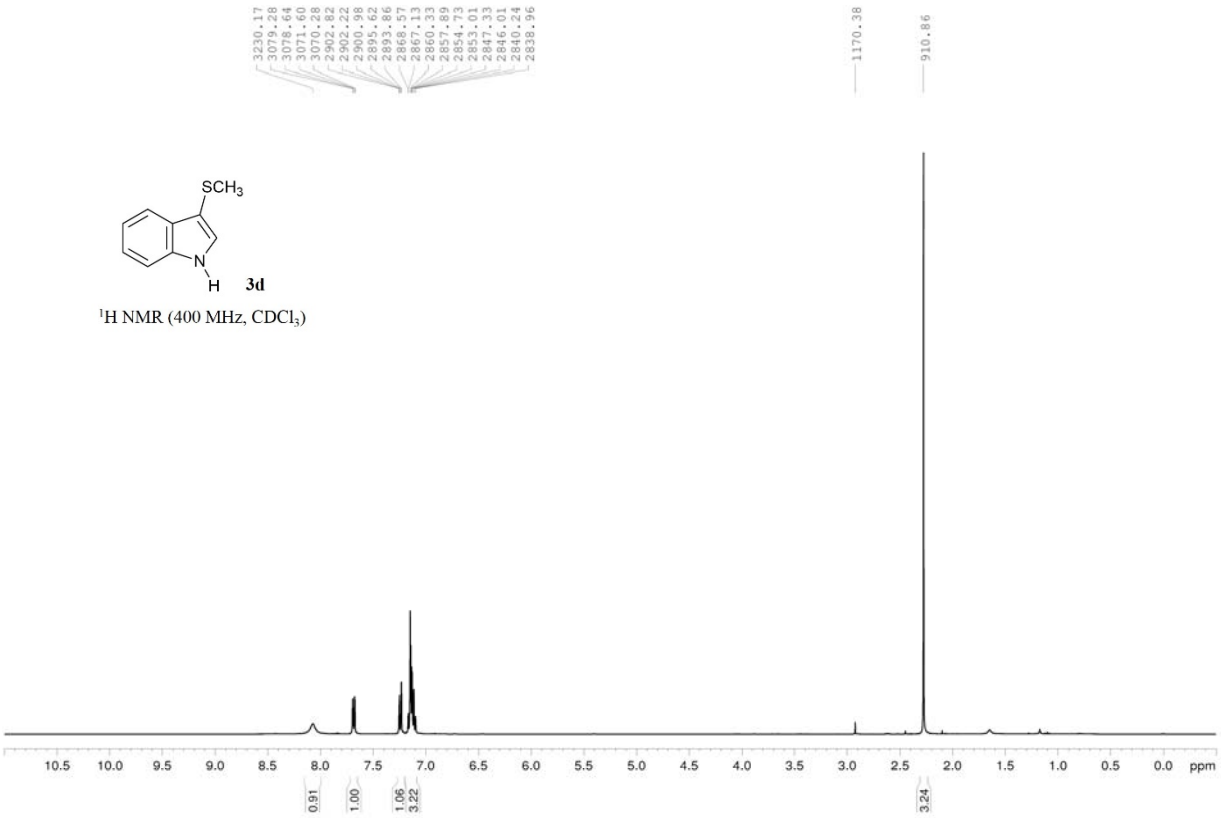




3c

^{13}C NMR (125 MHz, $CDCl_3$)



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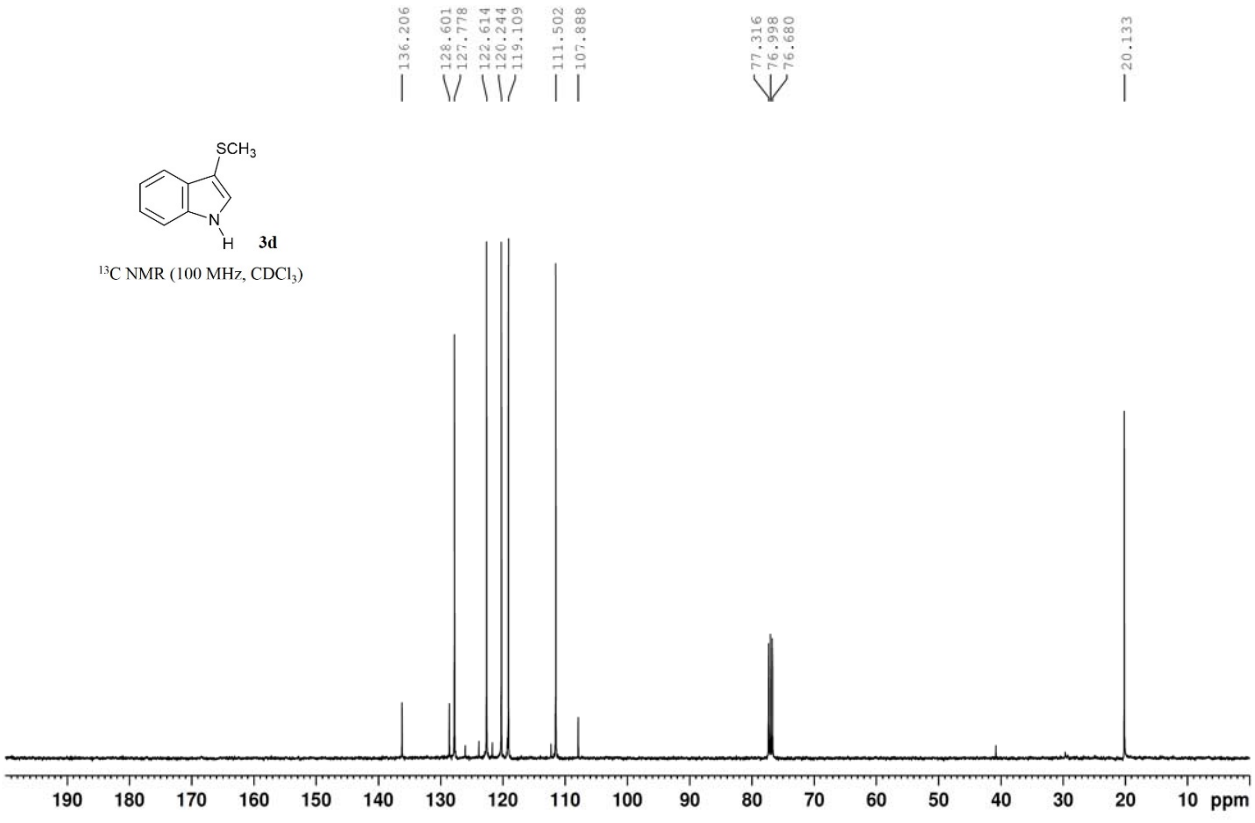

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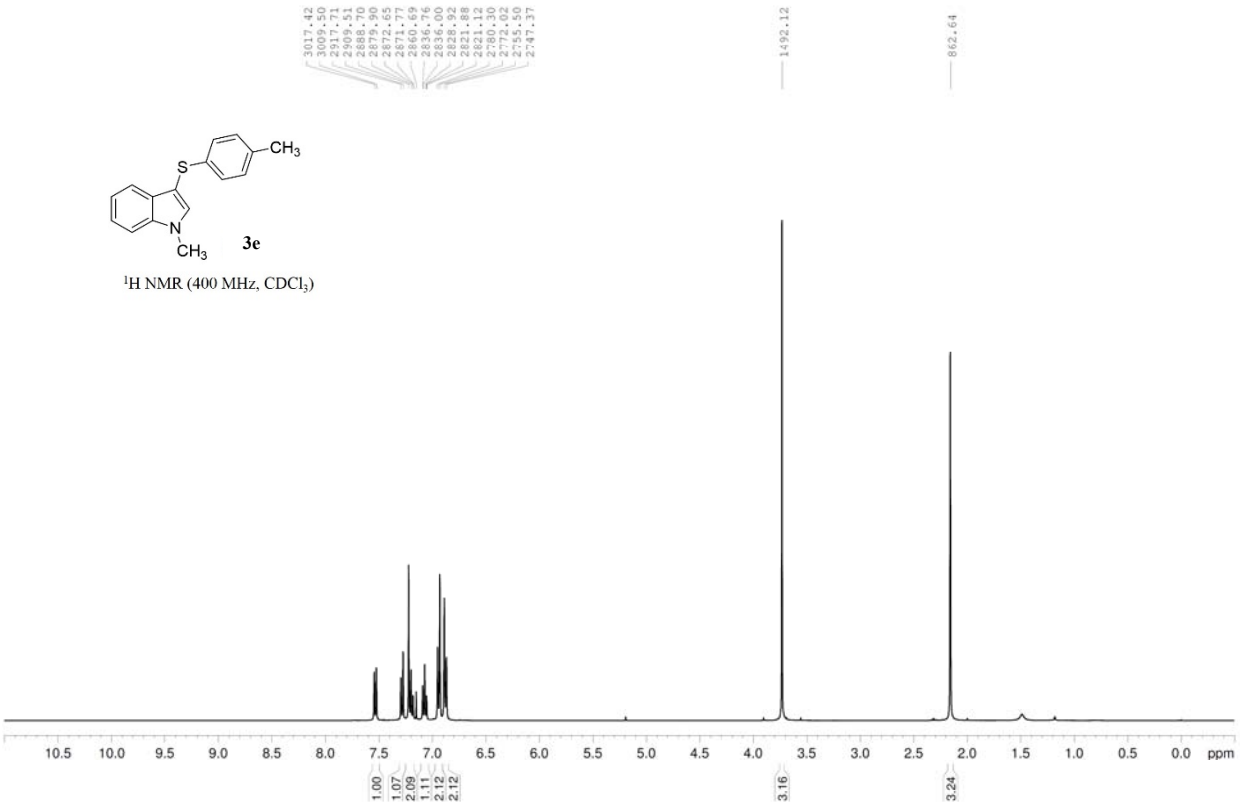

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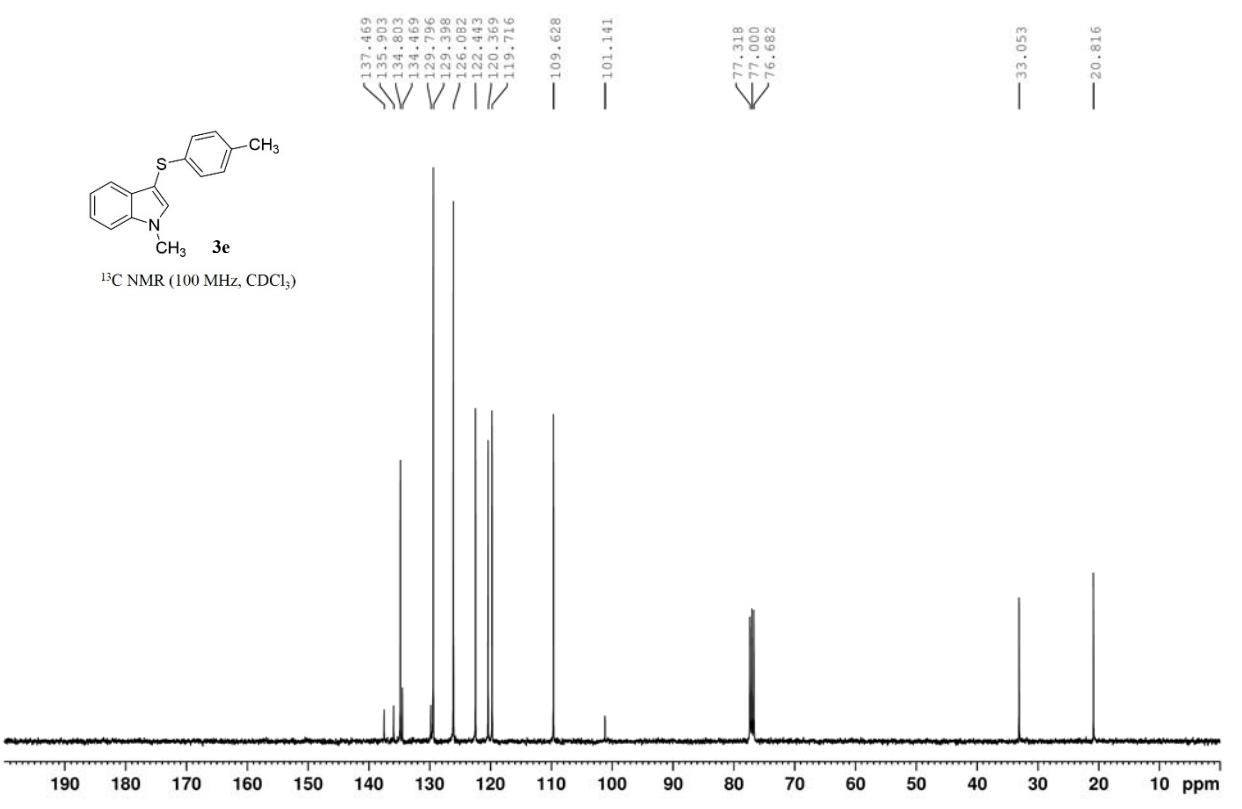

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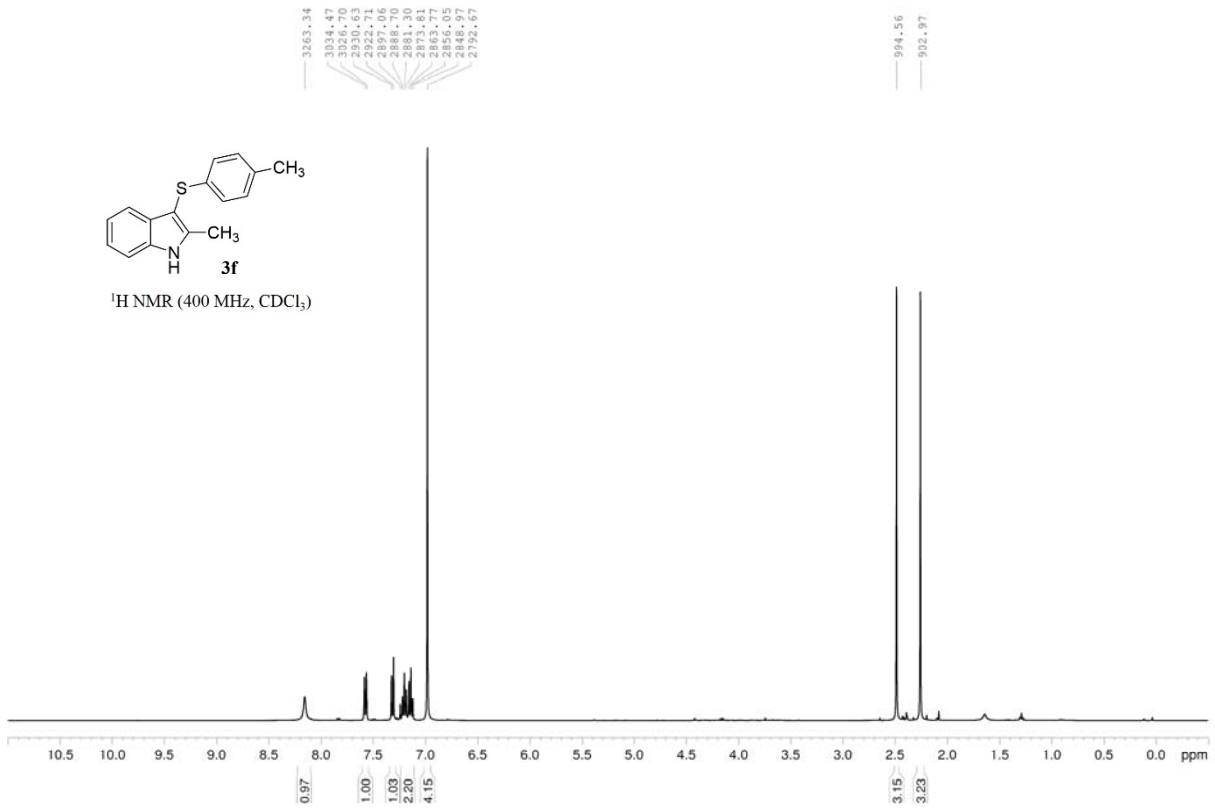

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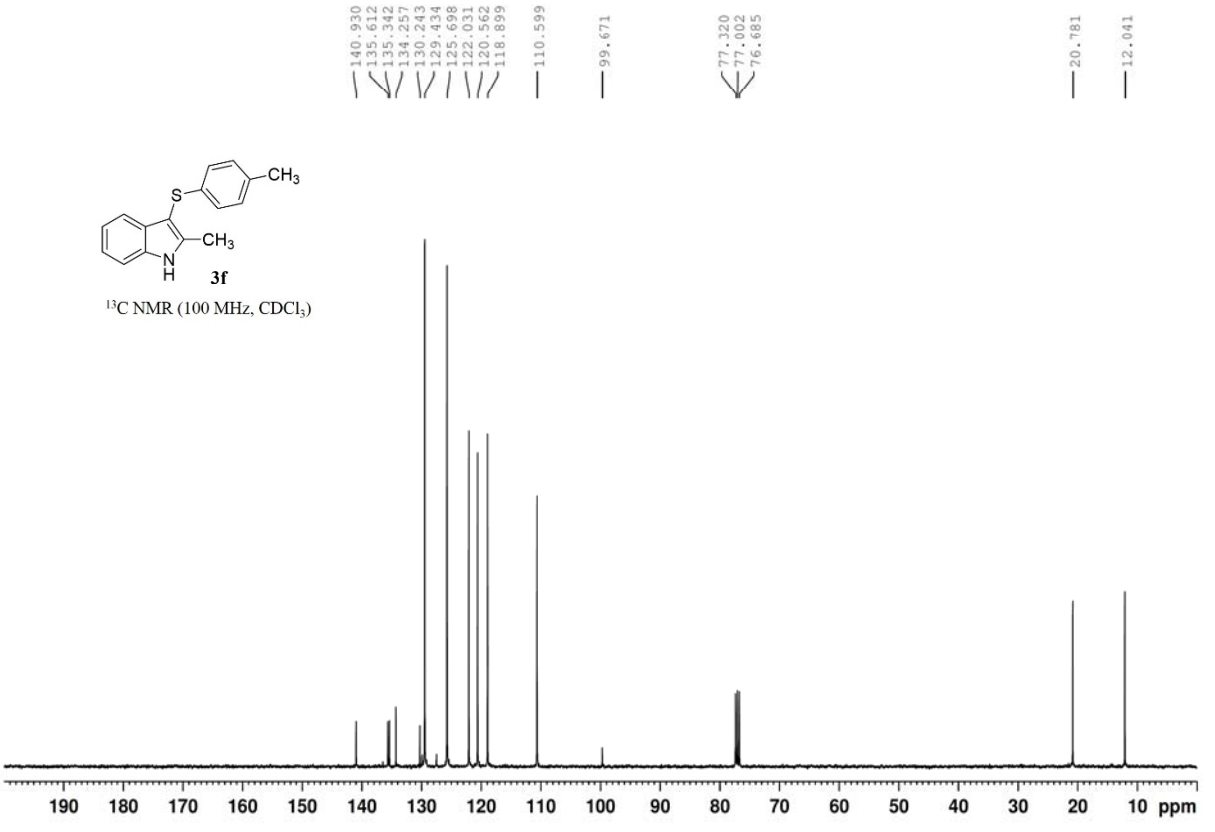

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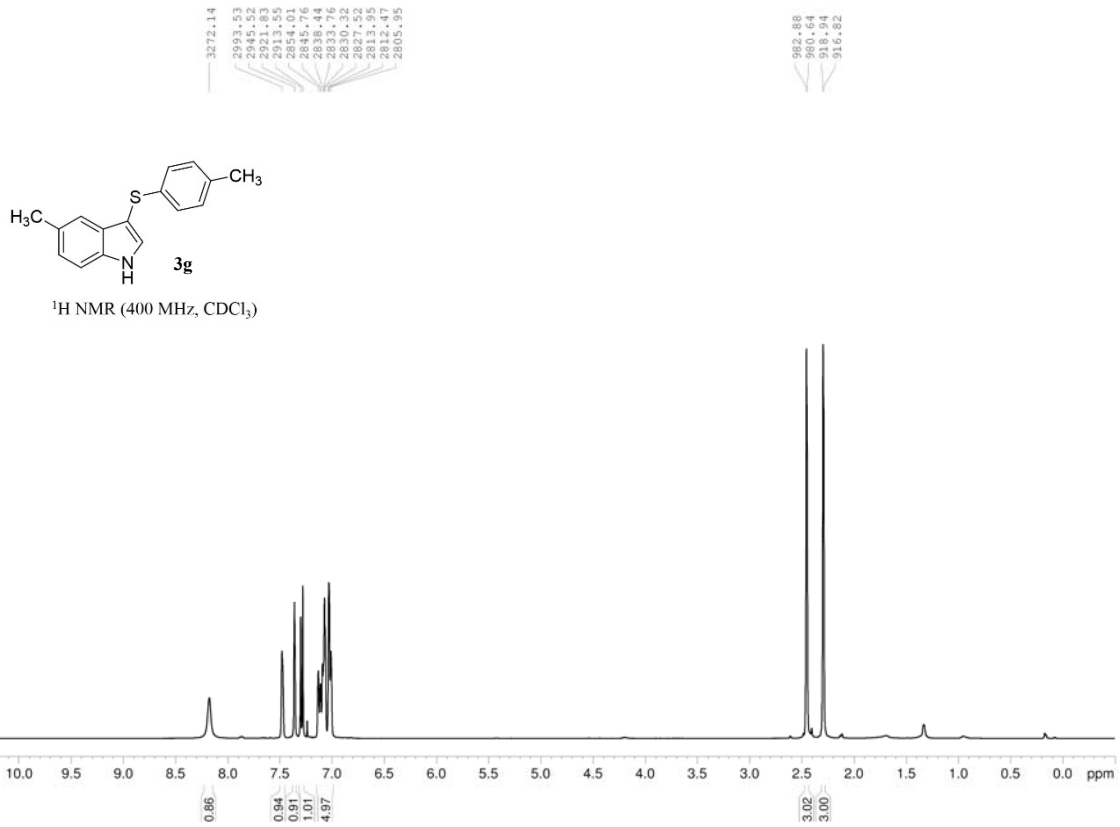

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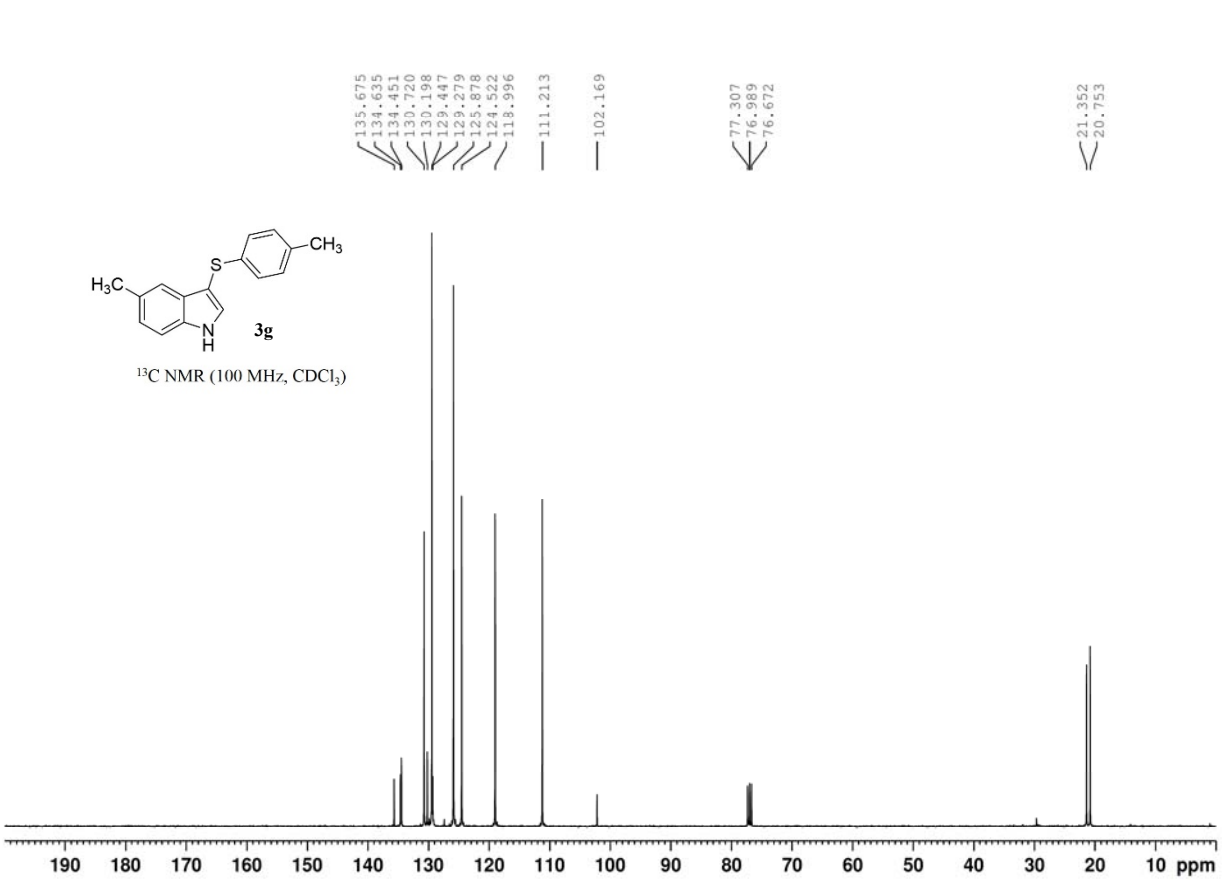

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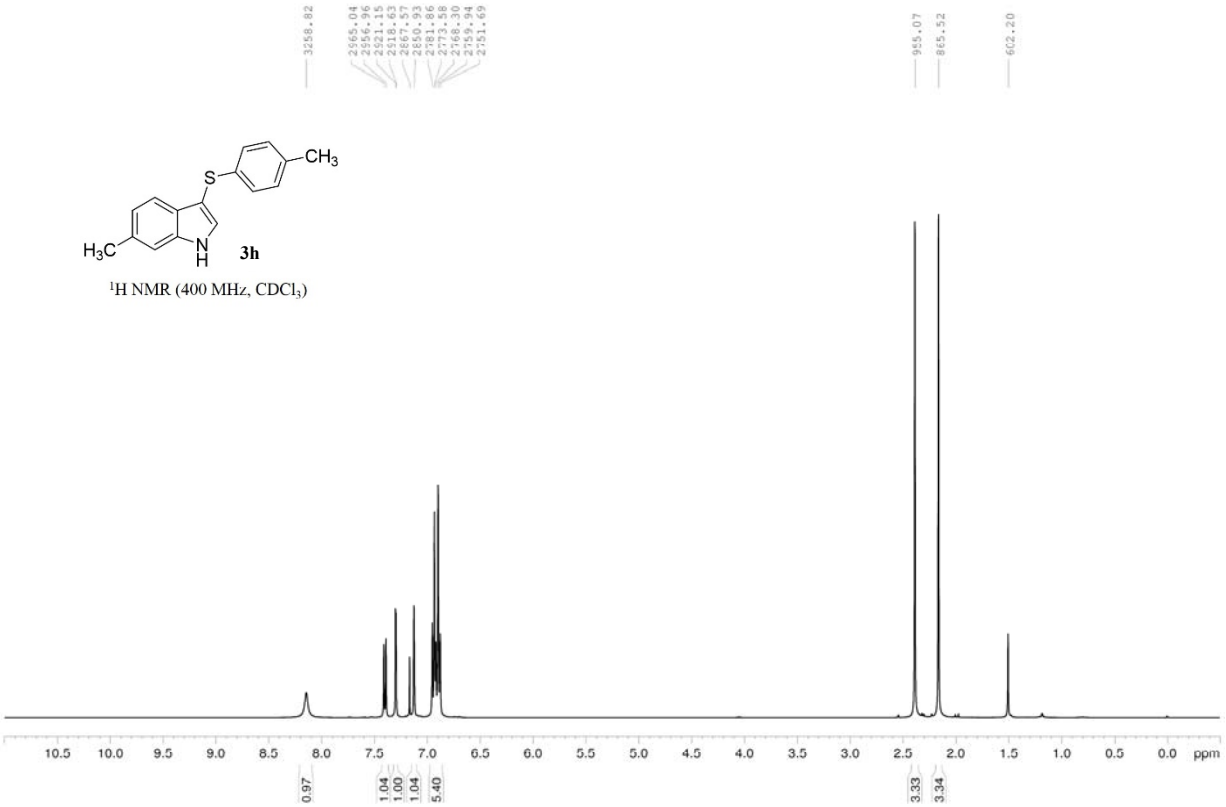

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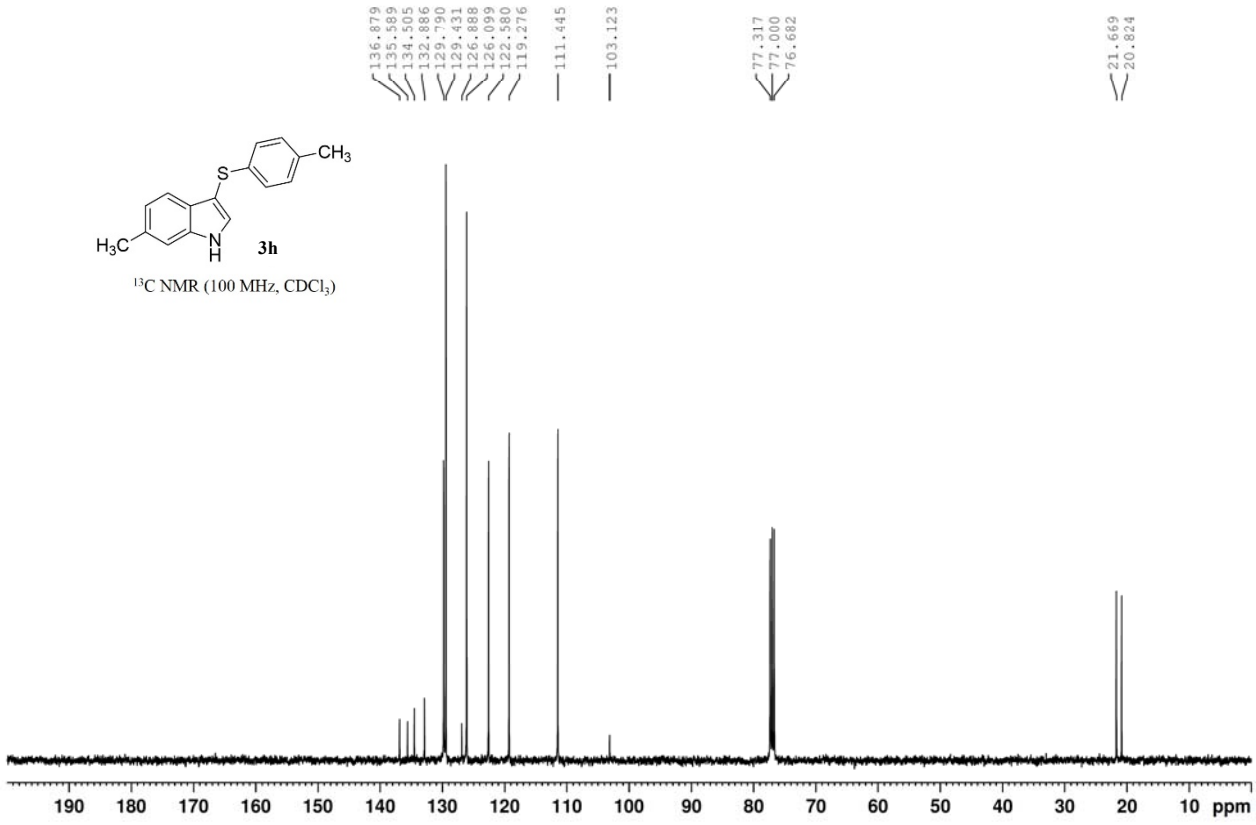

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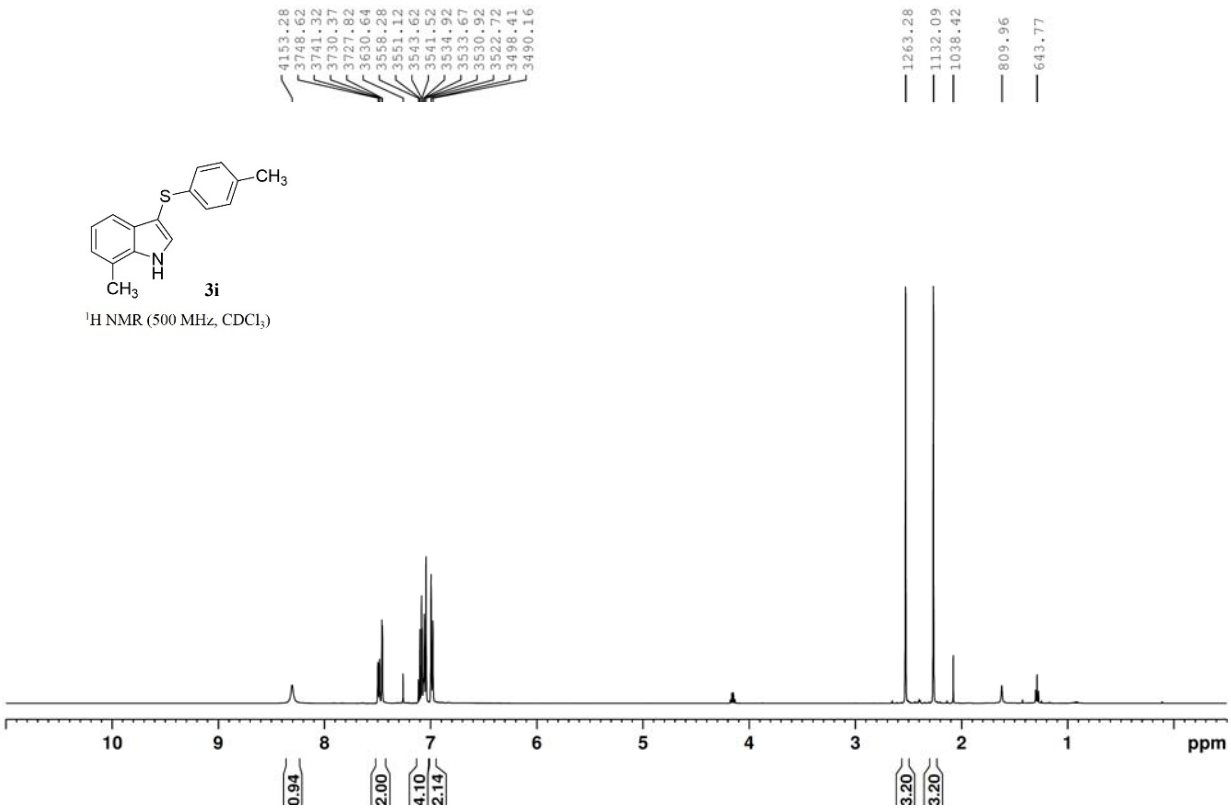
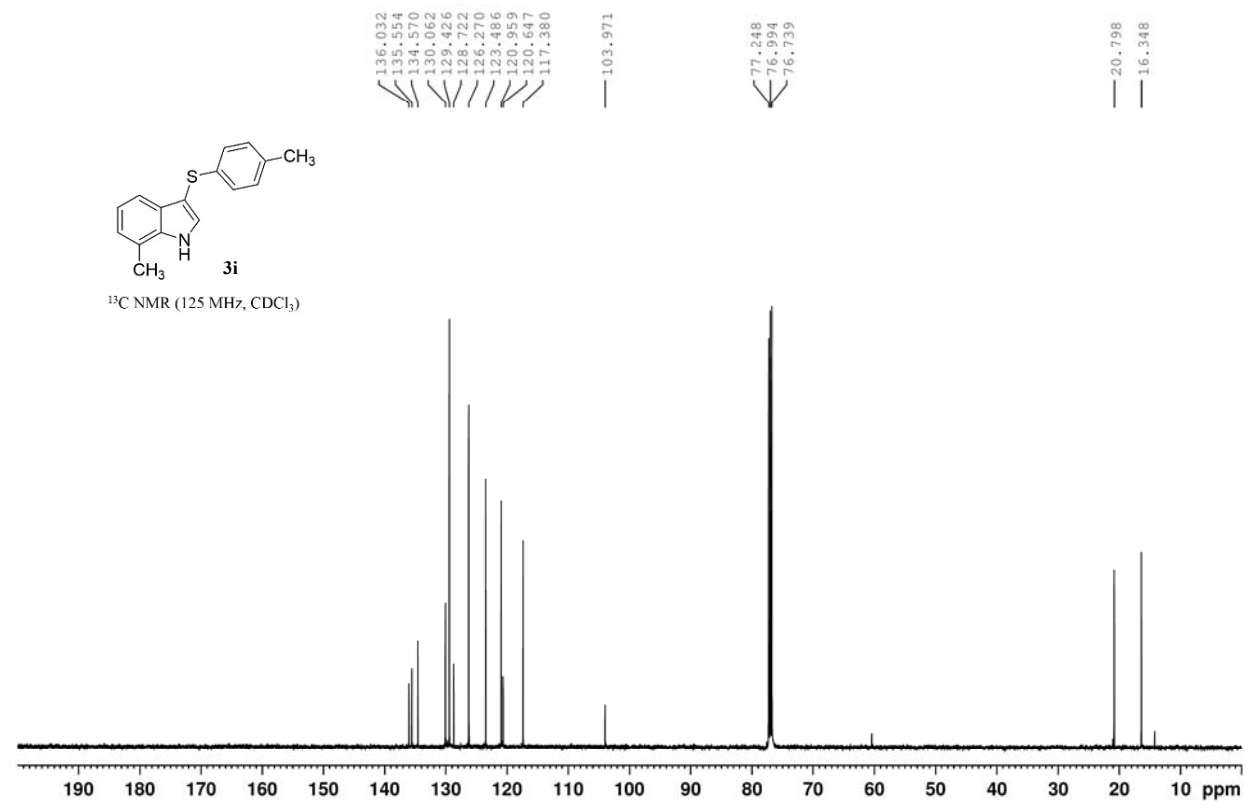


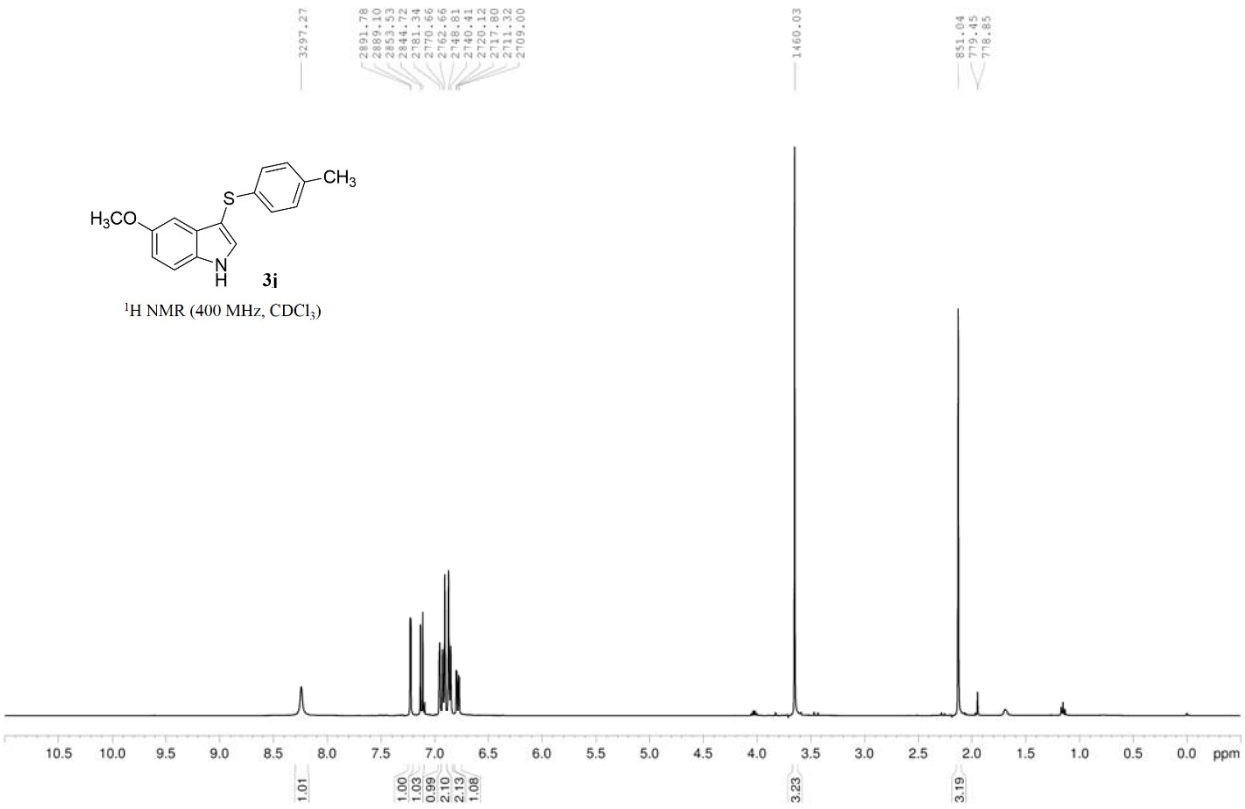
3i

^{1}H NMR (500 MHz, $CDCl_3$)



3i

^{13}C NMR (125 MHz, $CDCl_3$)



3i

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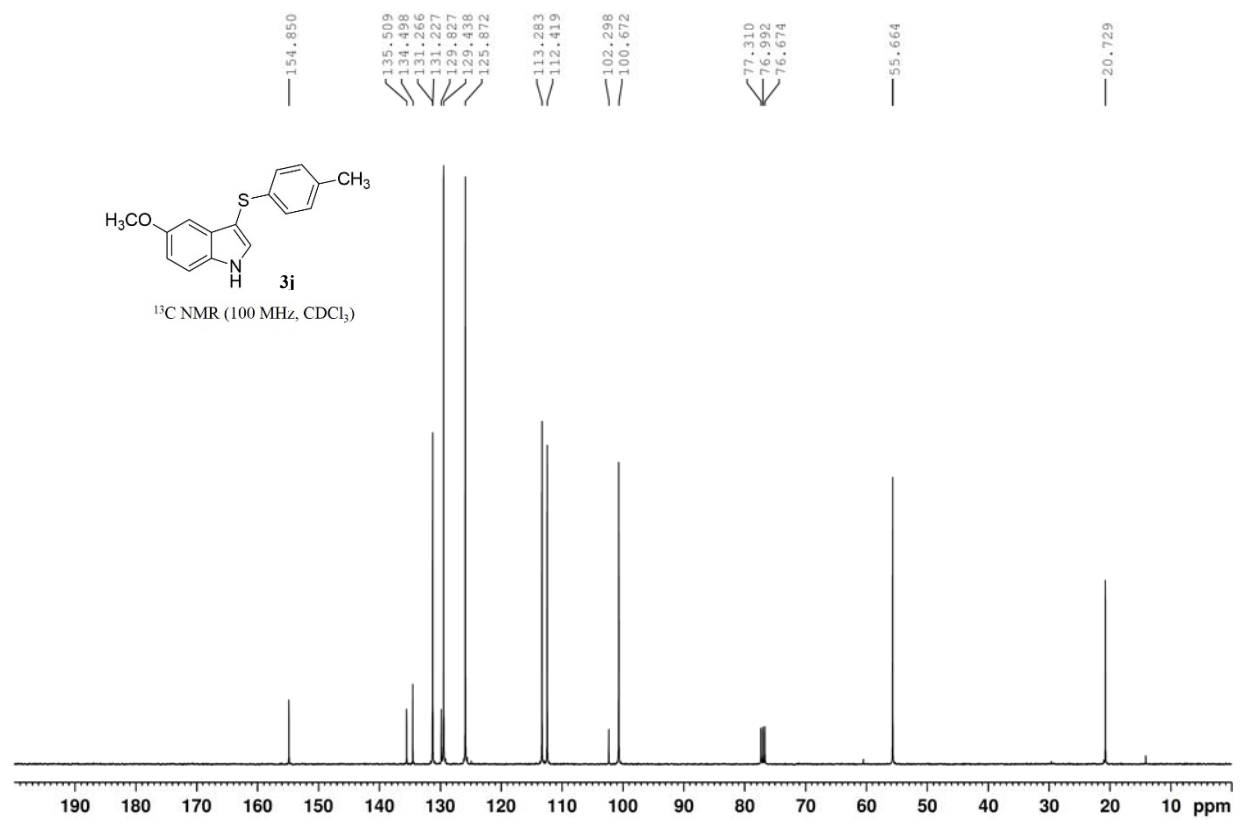

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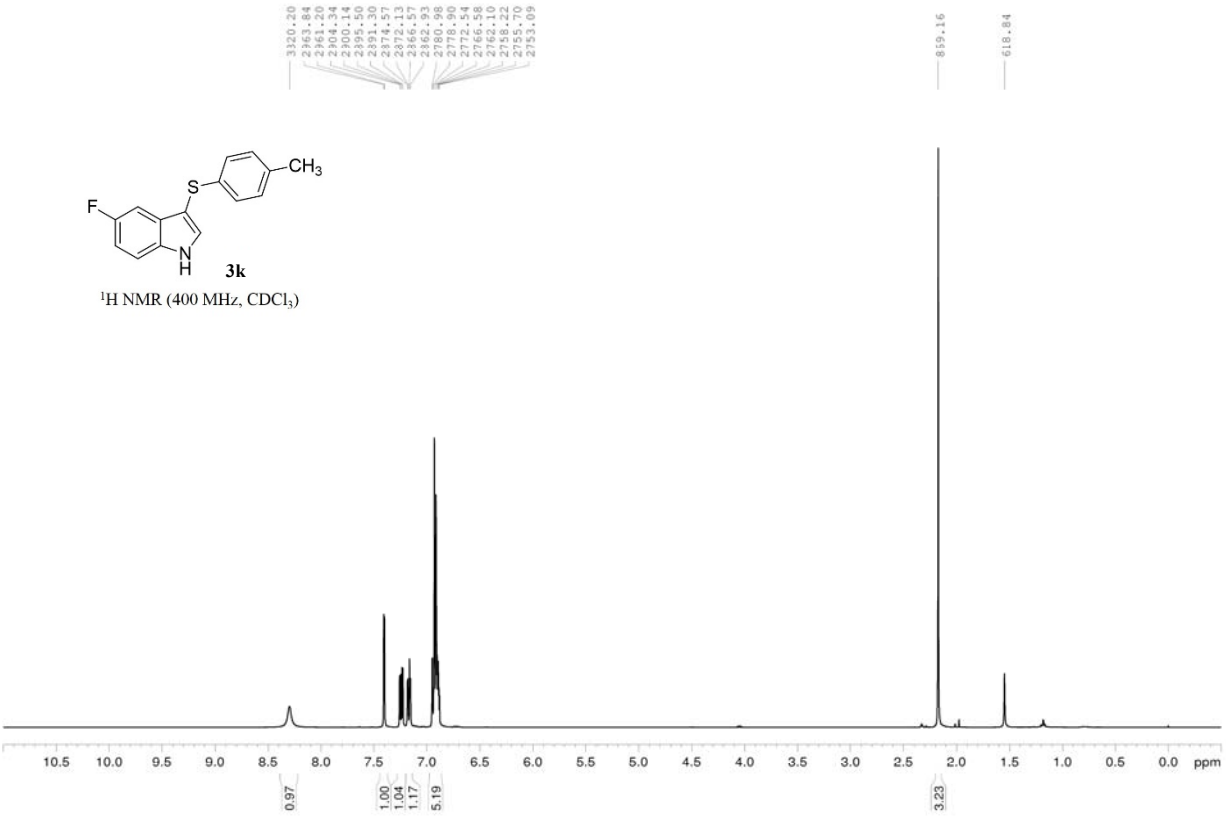

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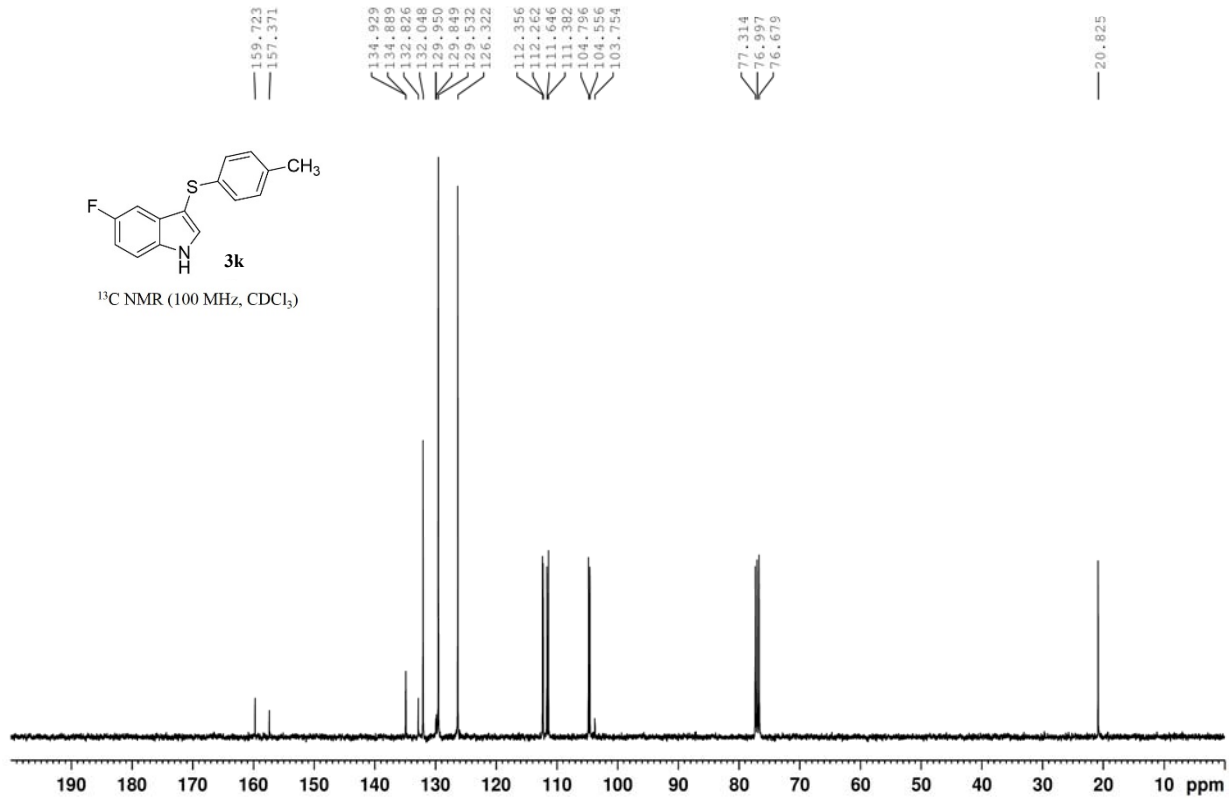

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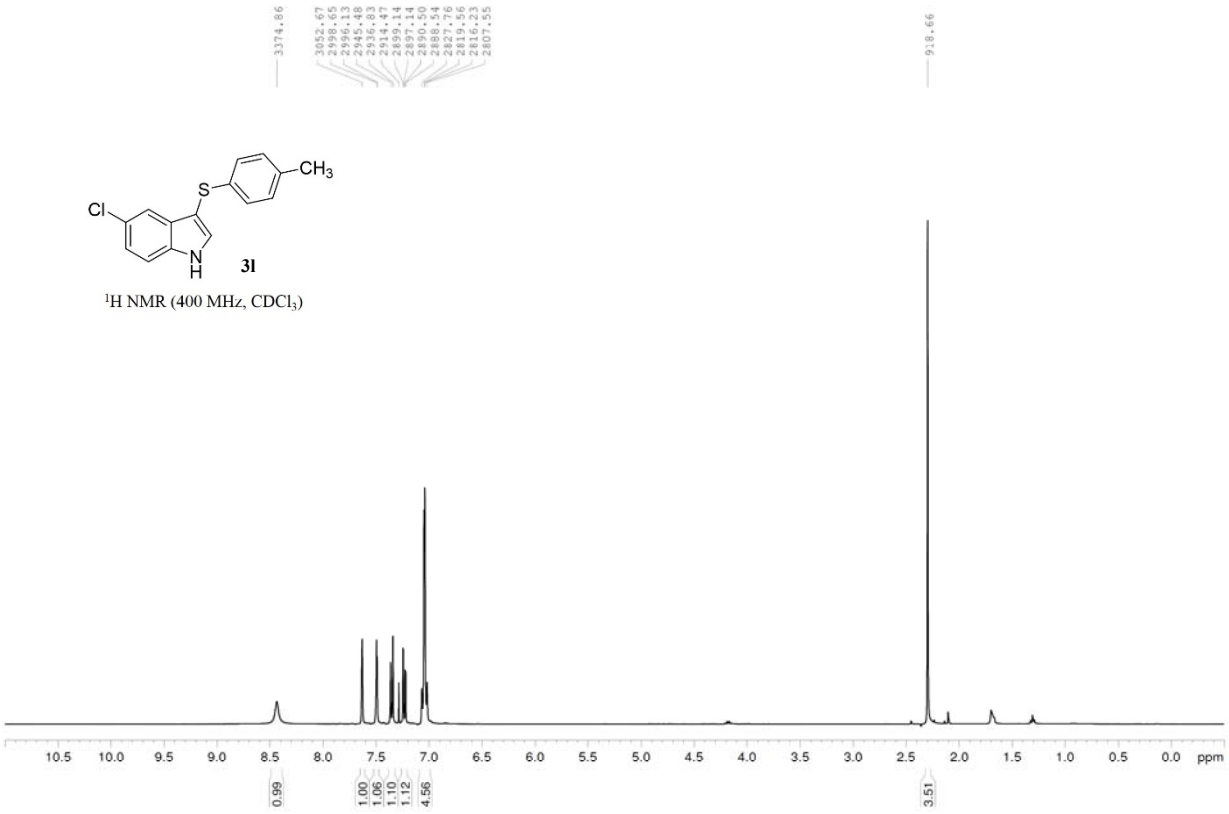

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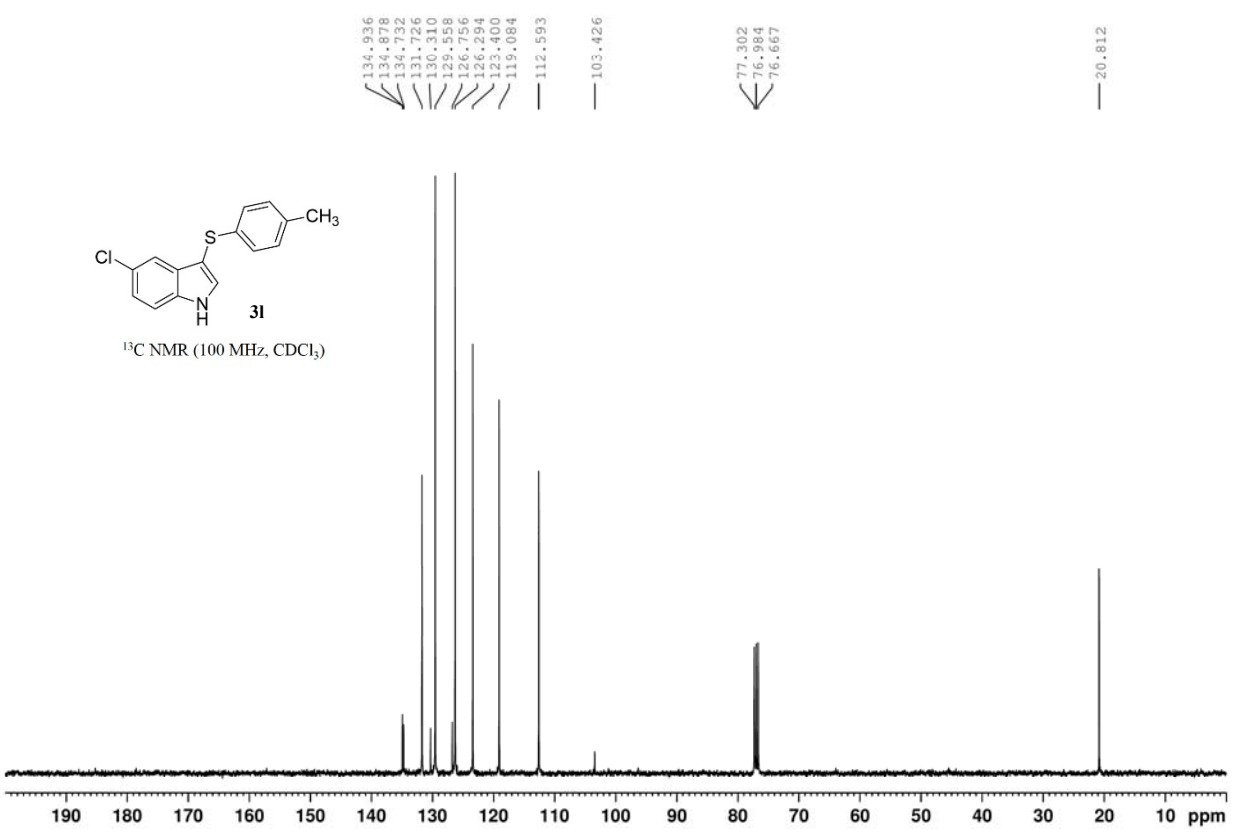

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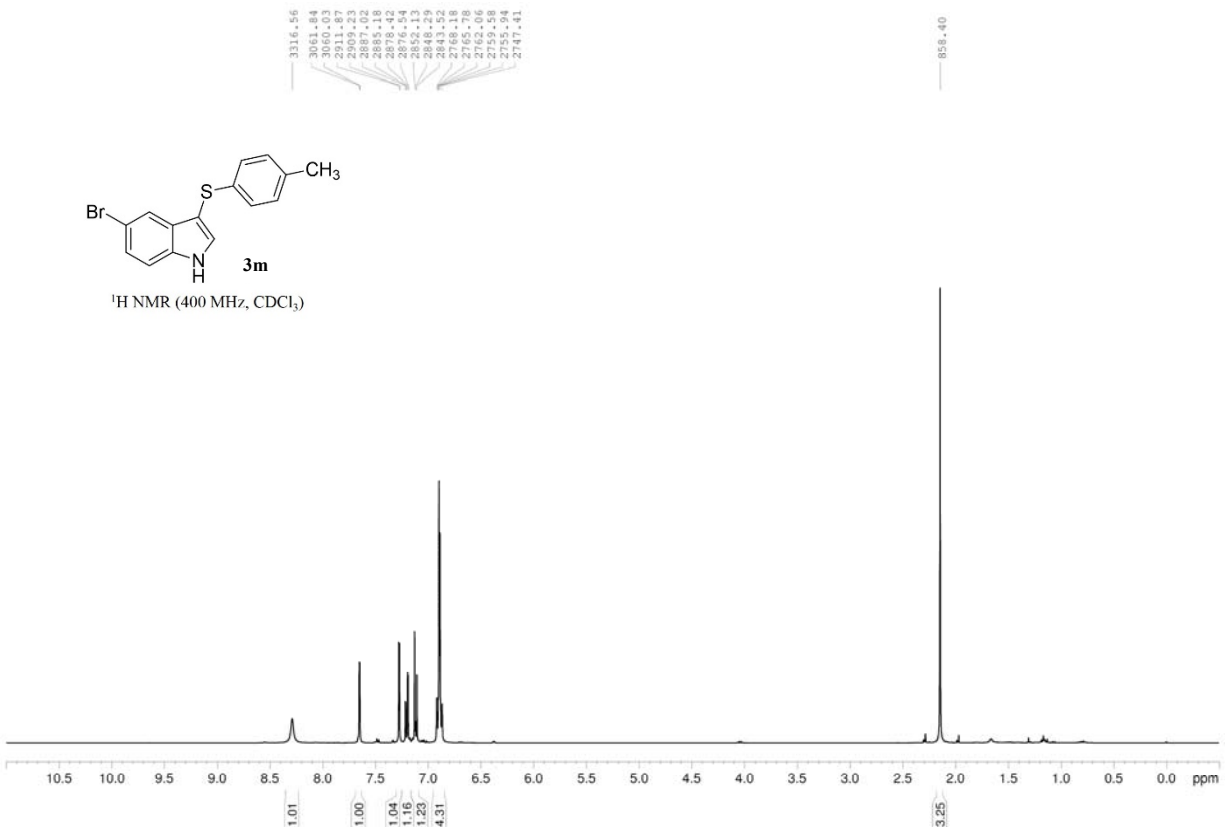

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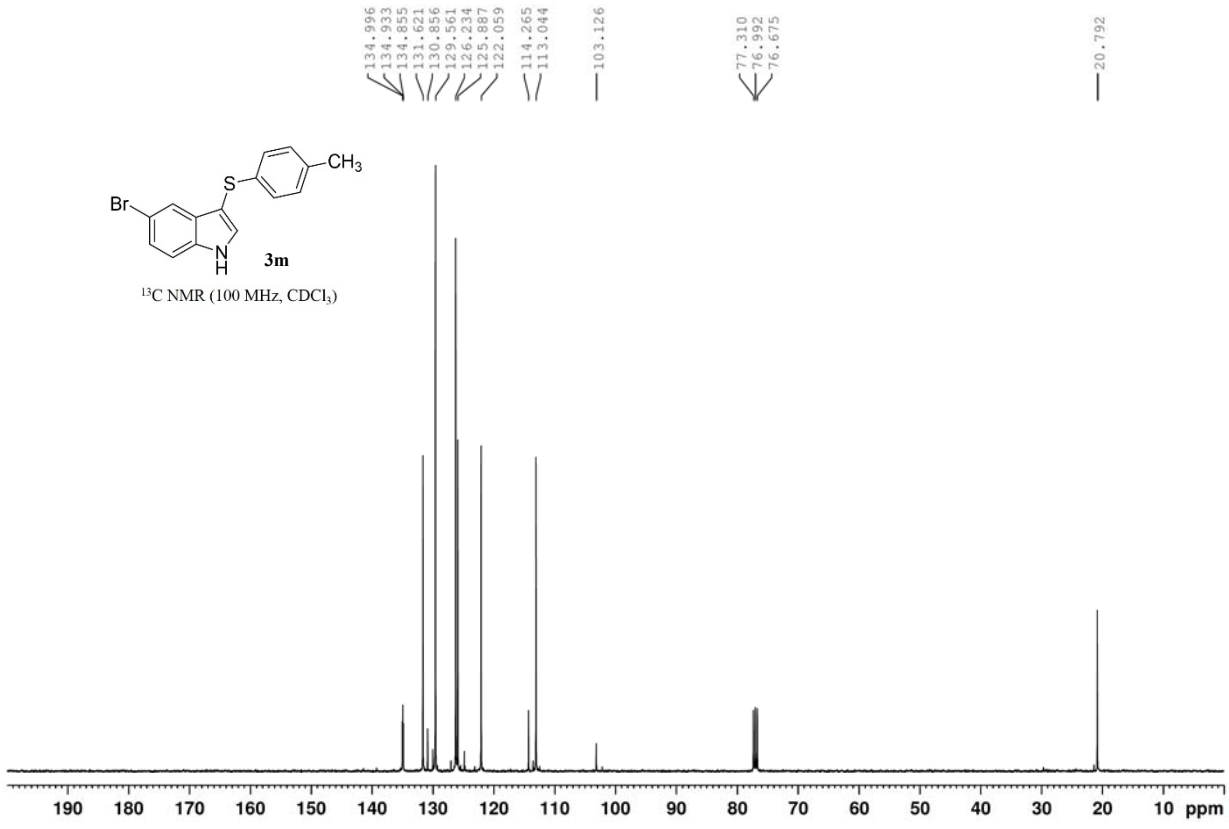

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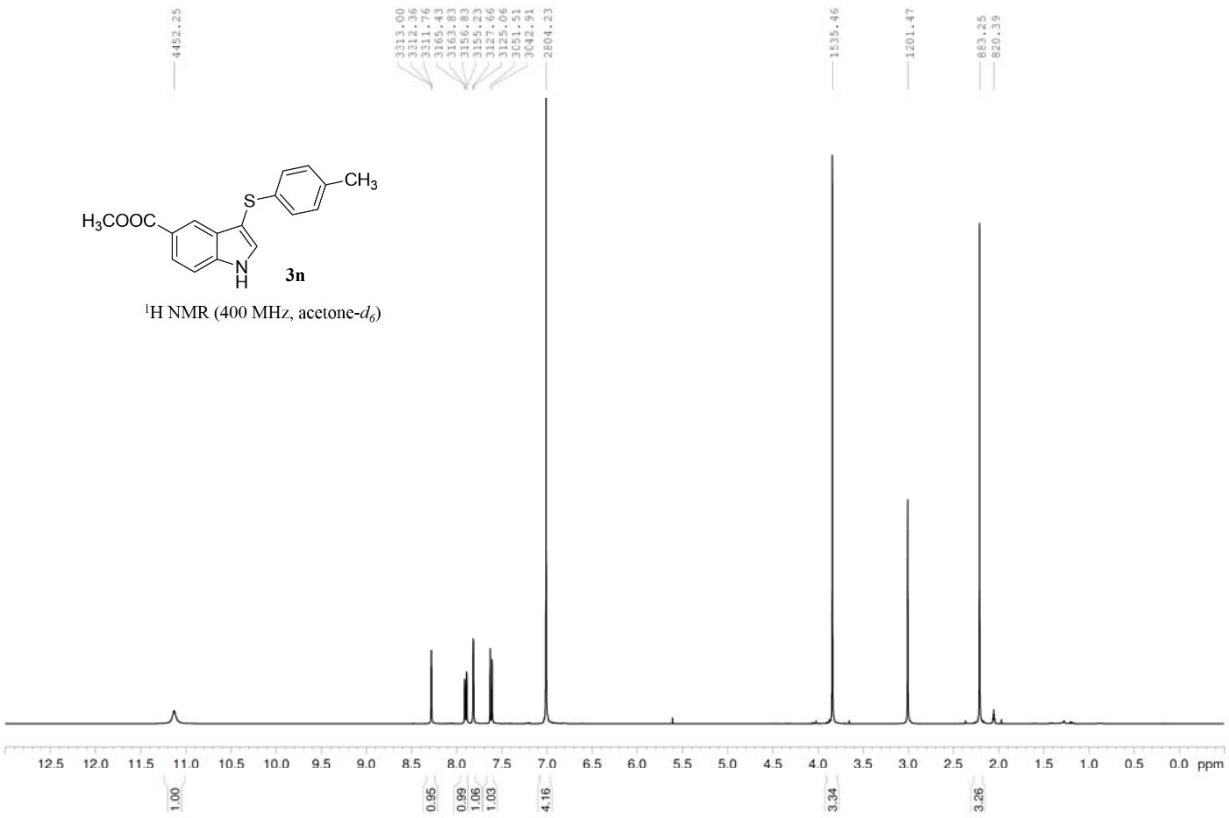
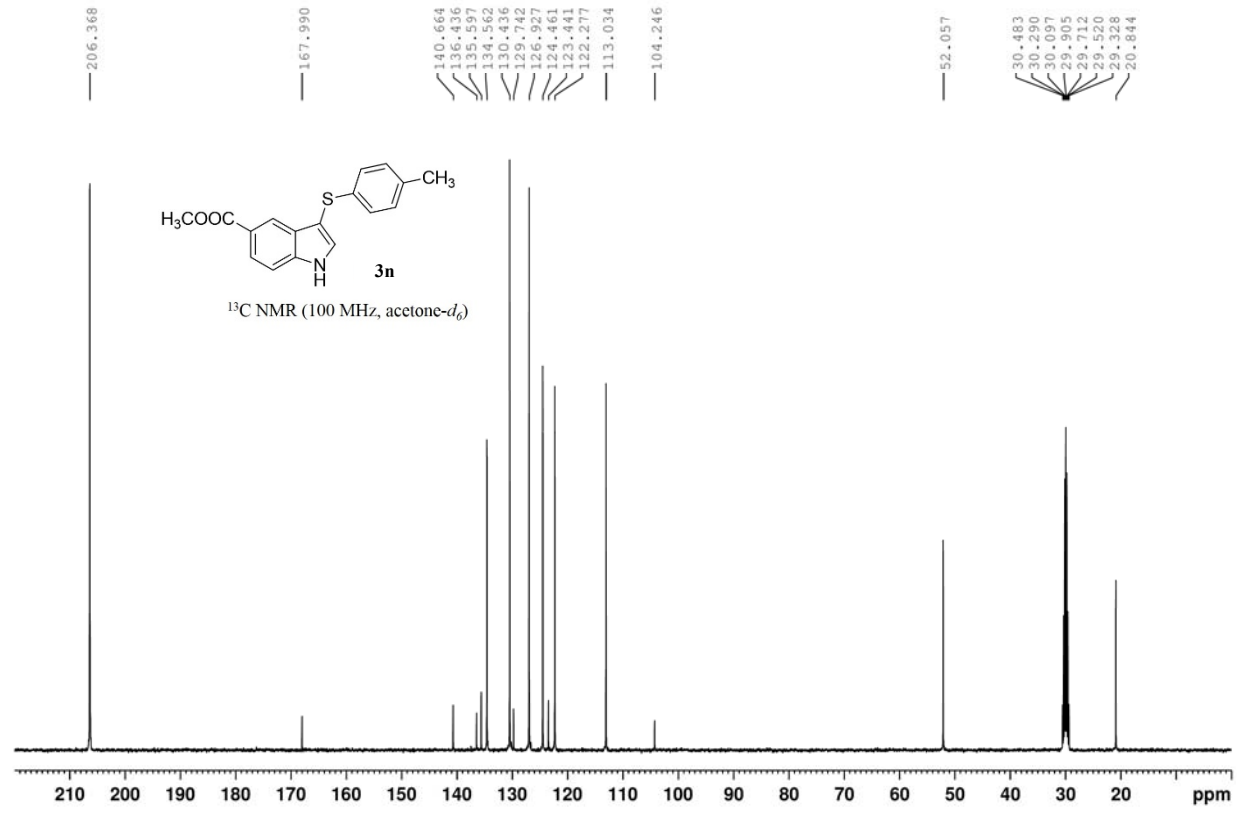


^{1}H NMR (400 MHz, acetone-d_6)



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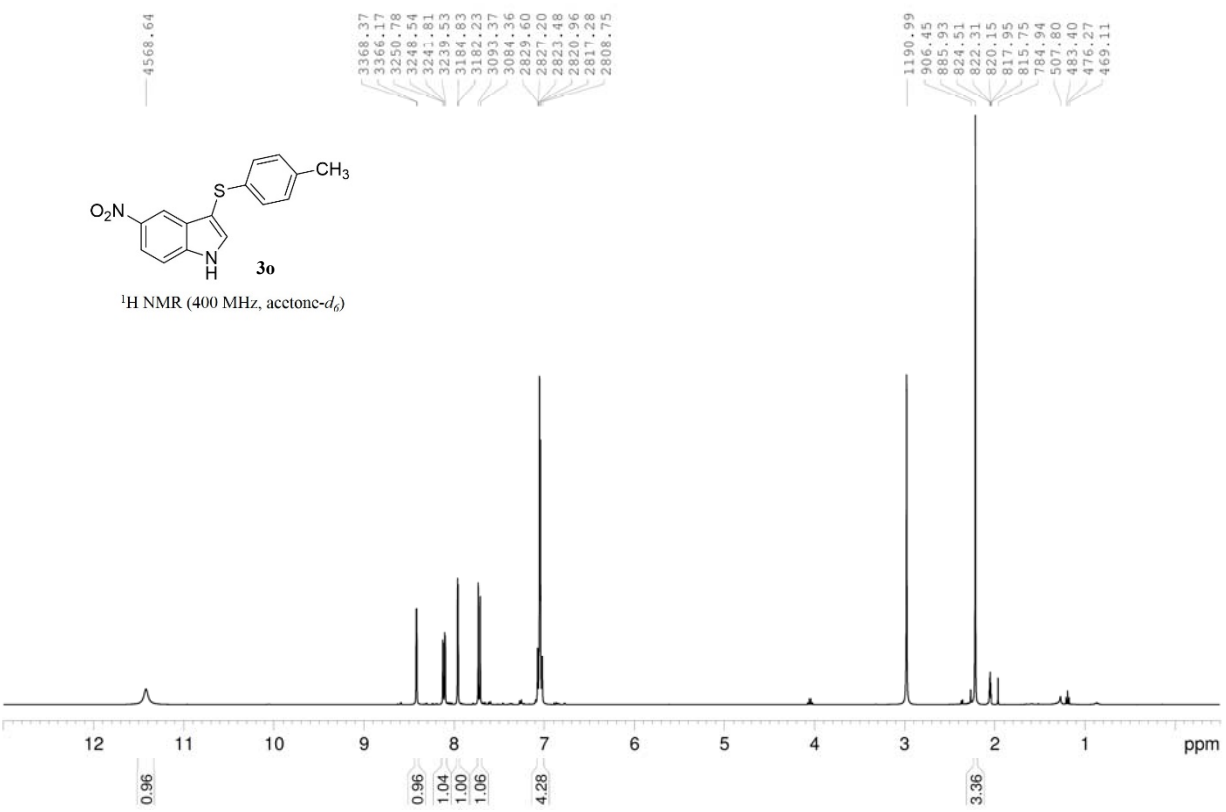
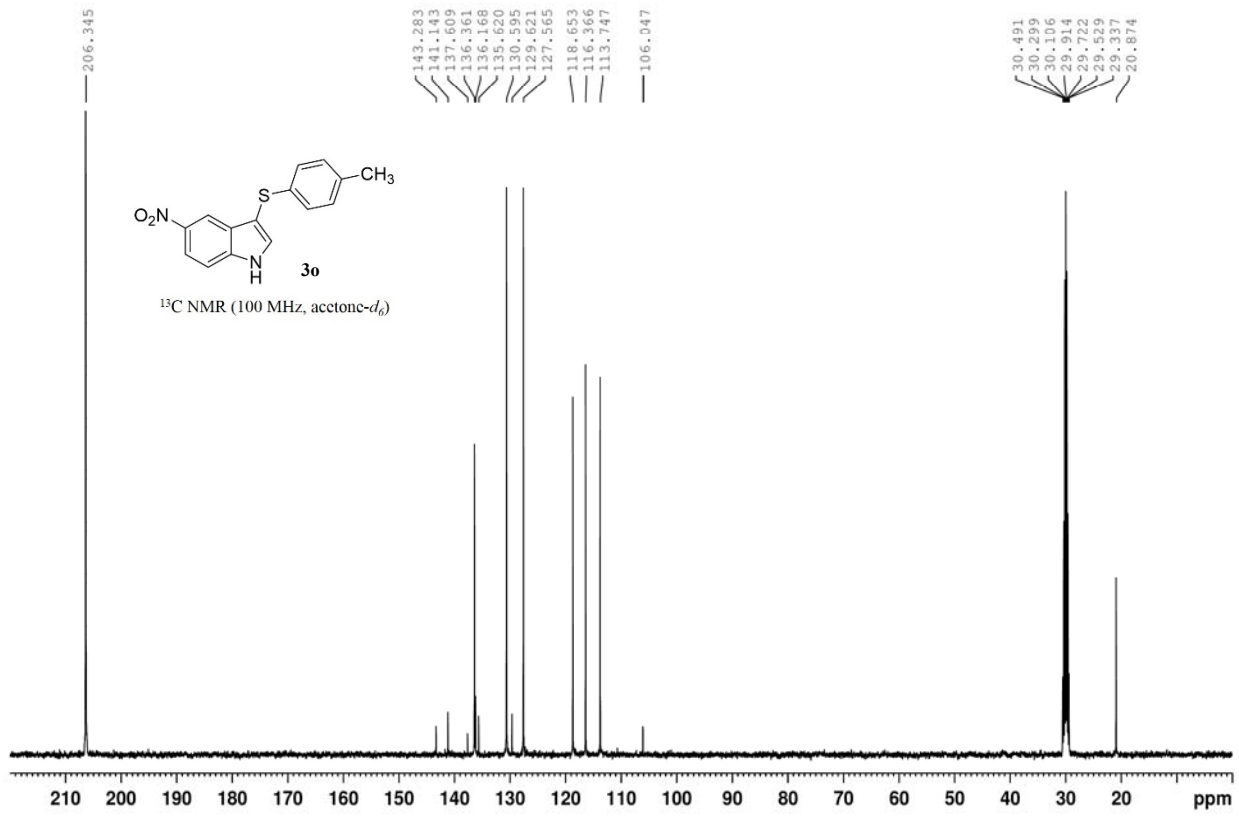


^{1}H NMR (400 MHz, acetone-d_6)



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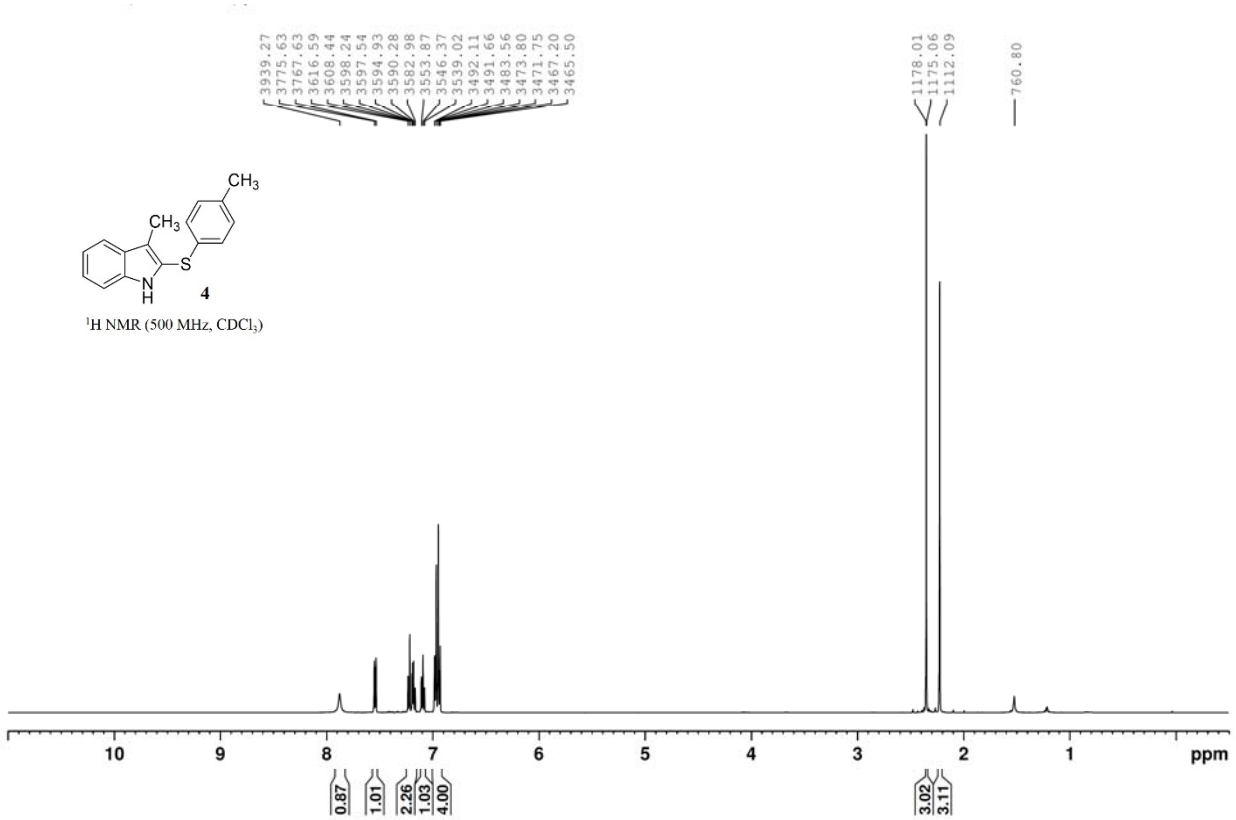

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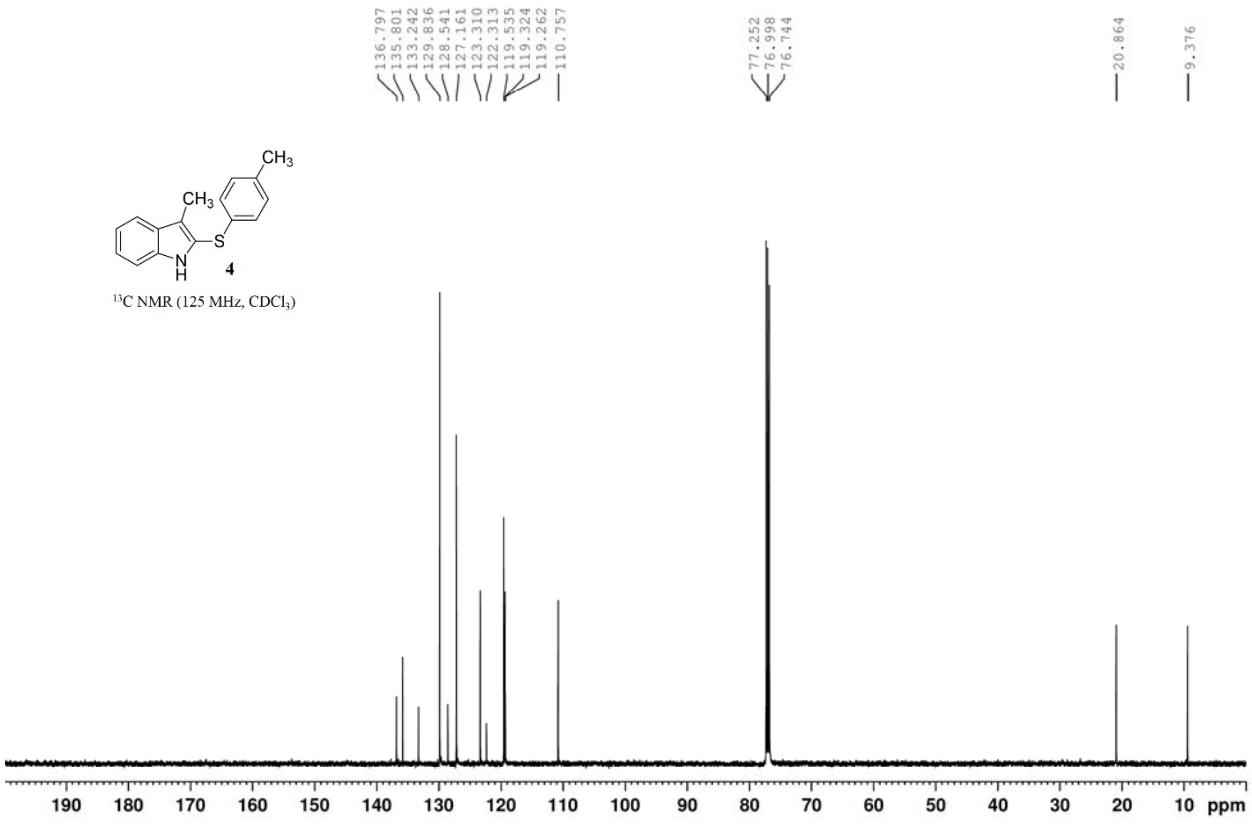
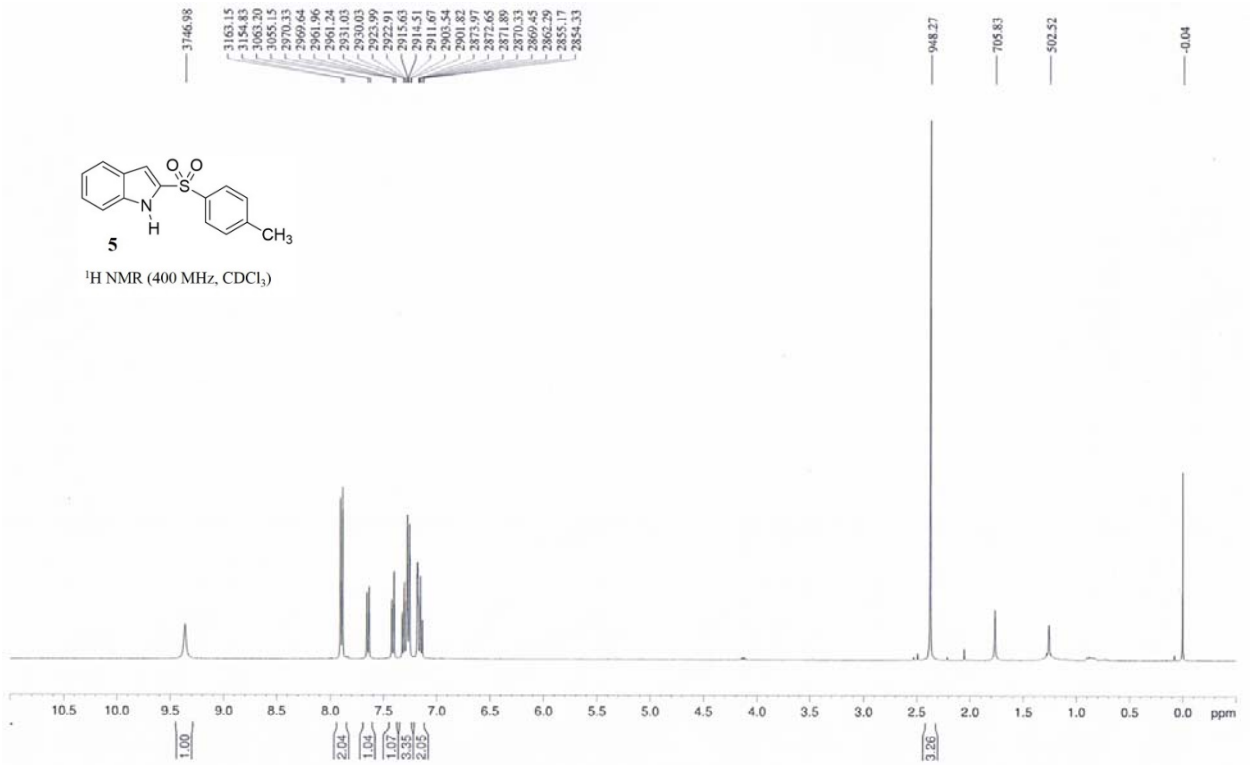


^{13}C NMR (125 MHz, $CDCl_3$)



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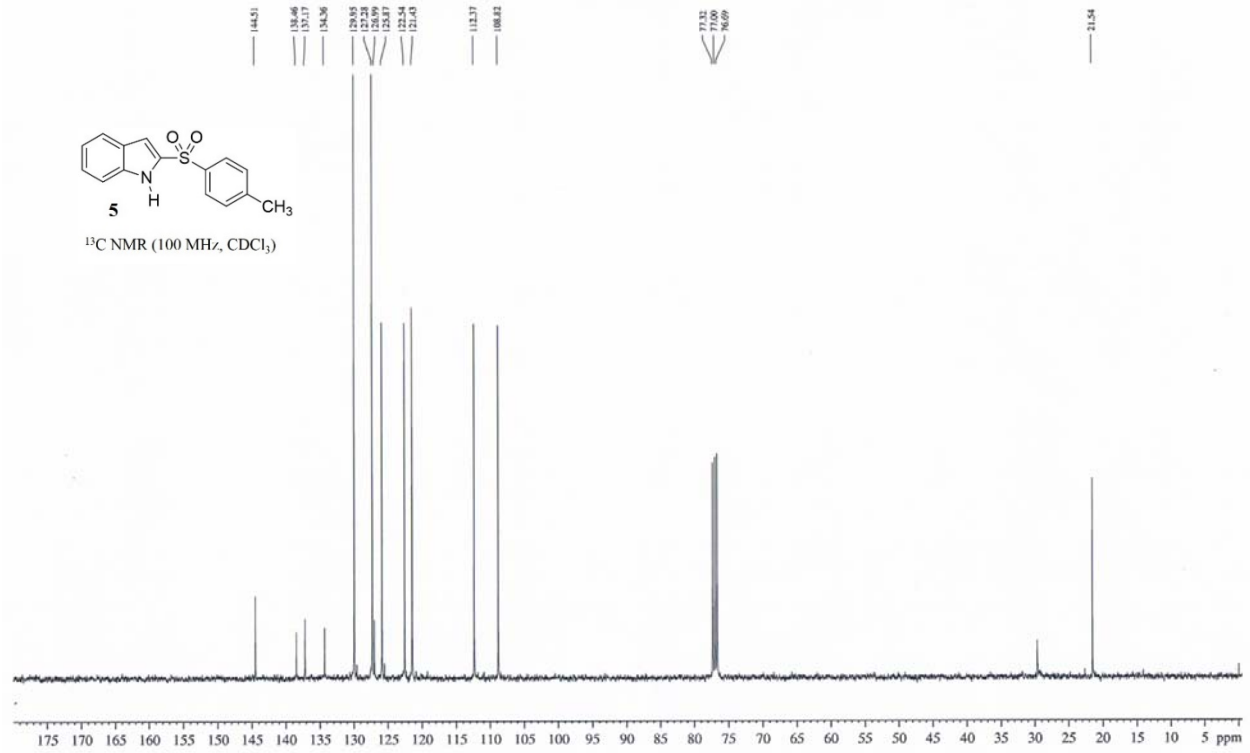

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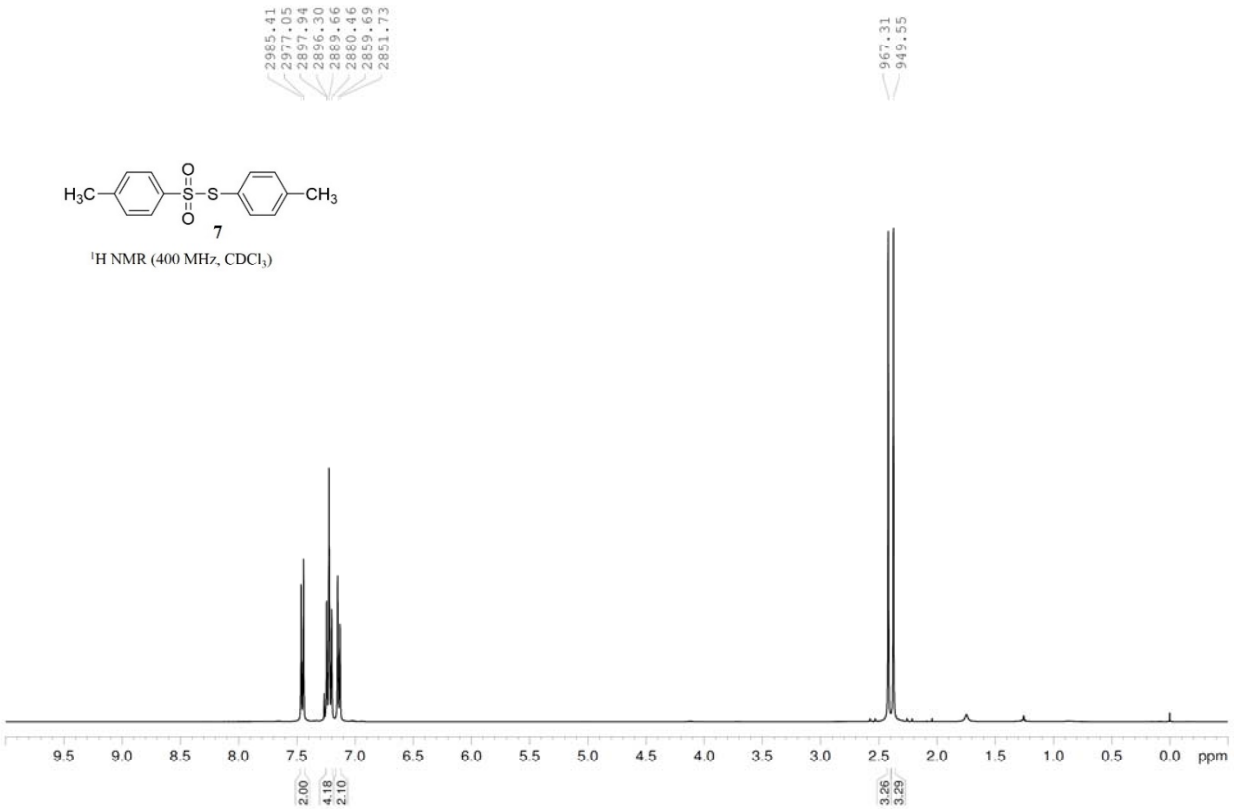
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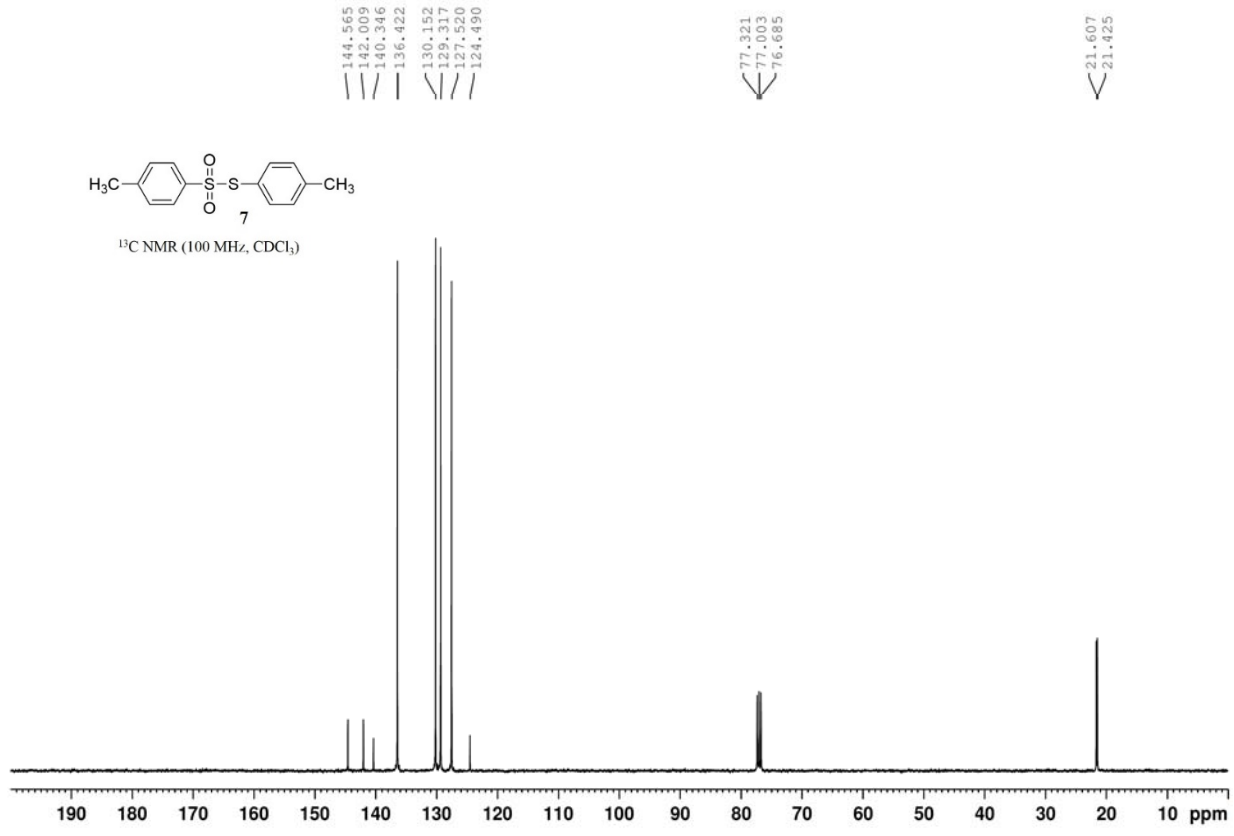
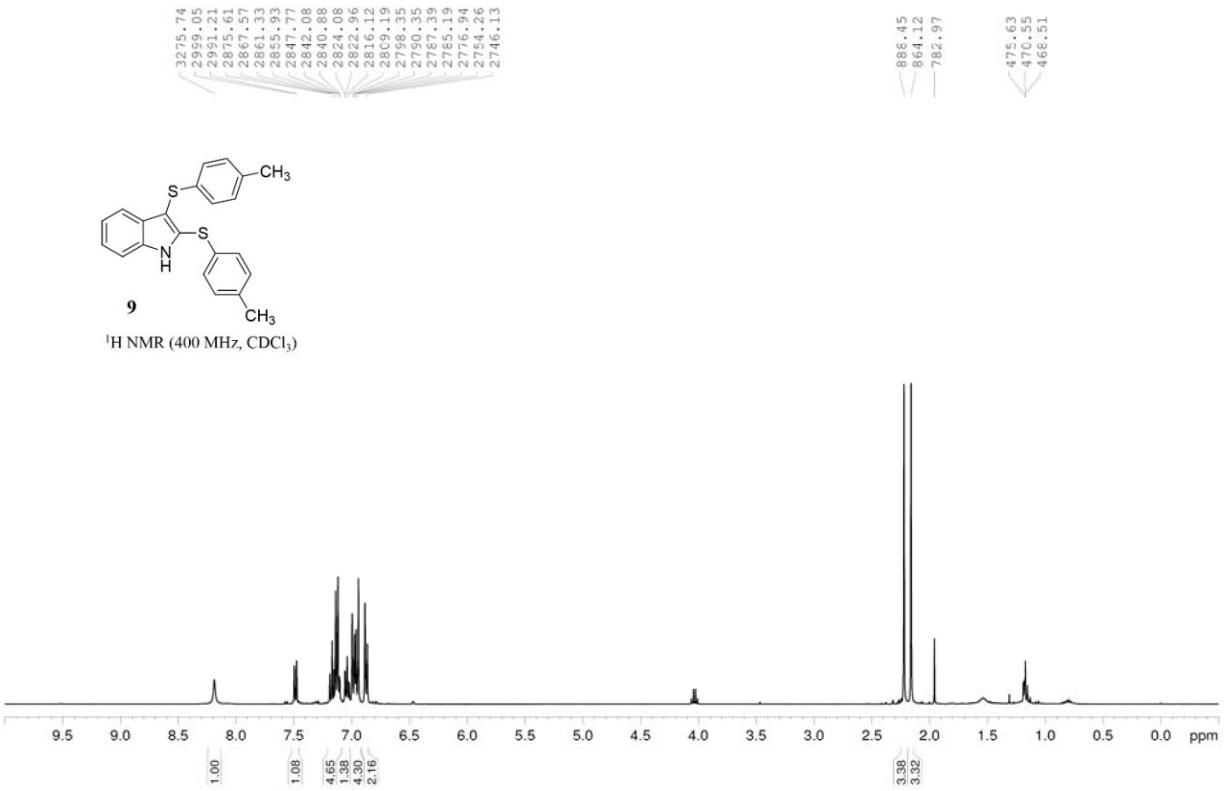
2977.05
2897.94
2896.30
2889.66
2880.46
2859.69
2851.73
967.31
949.55
H3C
CH3
7
1H NMR (400 MHz, CDCl3)
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.00
4.18
2.10
3.26
3.29


144.565
142.009
140.346
136.422
130.152
129.317
127.520
124.490
77.321
77.003
76.685
21.607
21.425
H3C
CH3
7
13C NMR (100 MHz, CDCl3)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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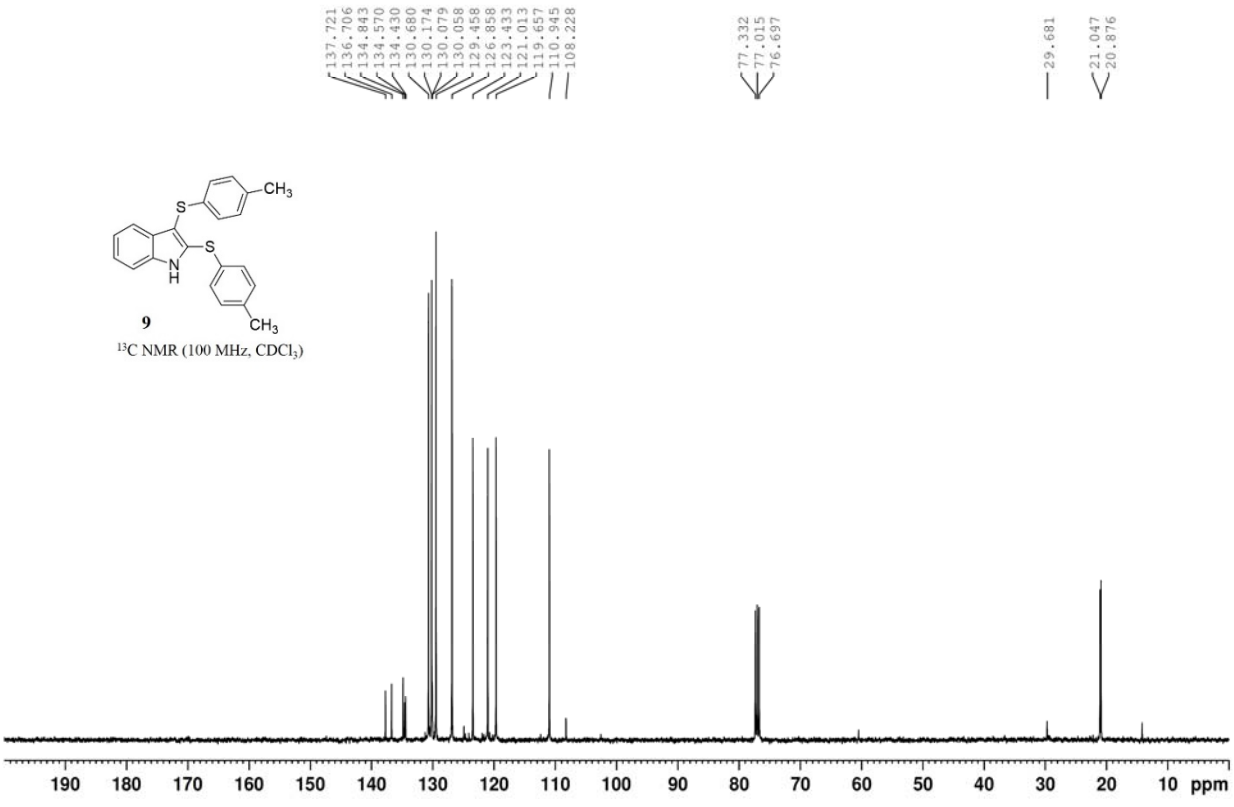
2999.05
2991.21
2875.61
2867.57
2861.33
2855.93
2847.77
2842.08
2840.88
2824.08
2822.96
2816.12
2809.19
2798.35
2790.35
2787.39
2785.19
2776.94
2754.26
2746.13
888.45
864.12
782.97
475.63
470.55
468.51
CH3
S
S
N
H
CH3
9
1H NMR (400 MHz, CDCl3)
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
1.08
4.65
1.38
4.30
2.16
3.38
3.32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134.430
130.680
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130.079
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129.458
126.858
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121.013
119.657
110.945
108.228
77.332
77.015
76.697
29.681
21.047
20.876
CH3
S
S
N
H
CH3
9
13C NMR (100 MHz, CDCl3)
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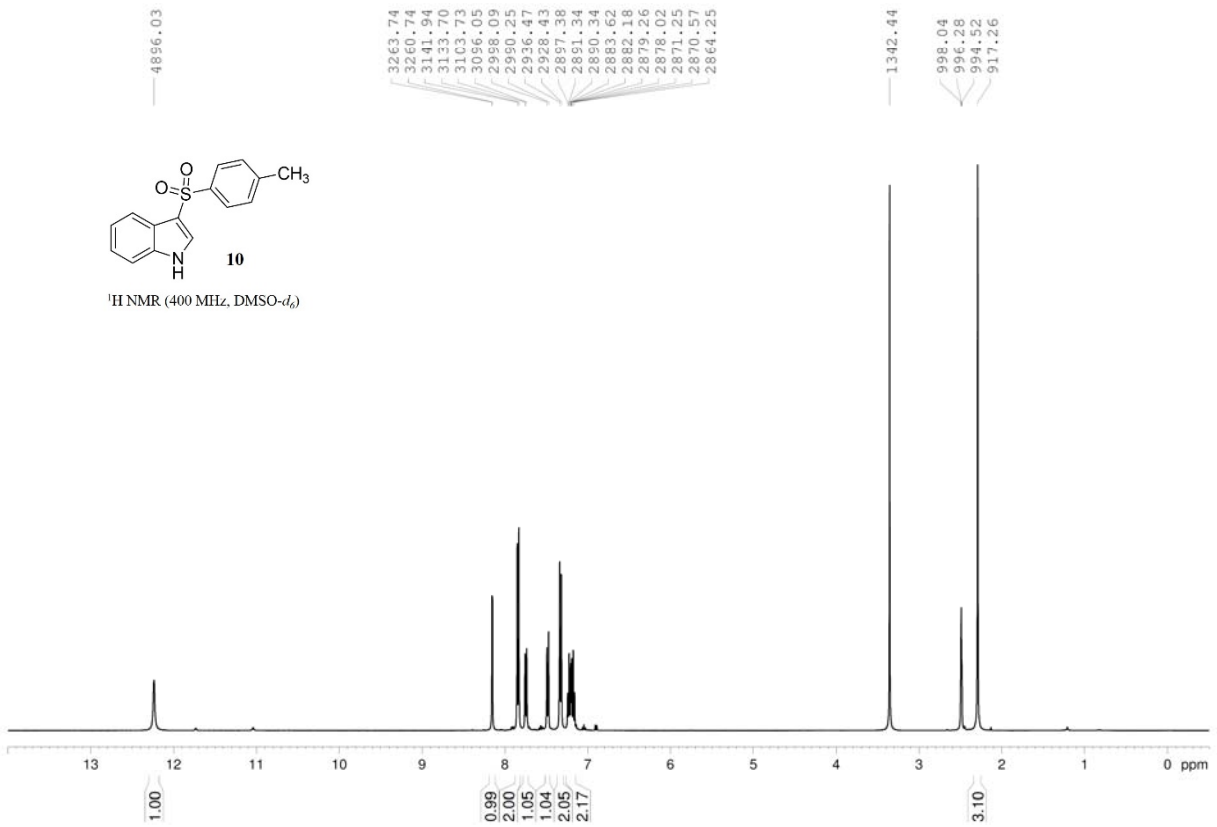
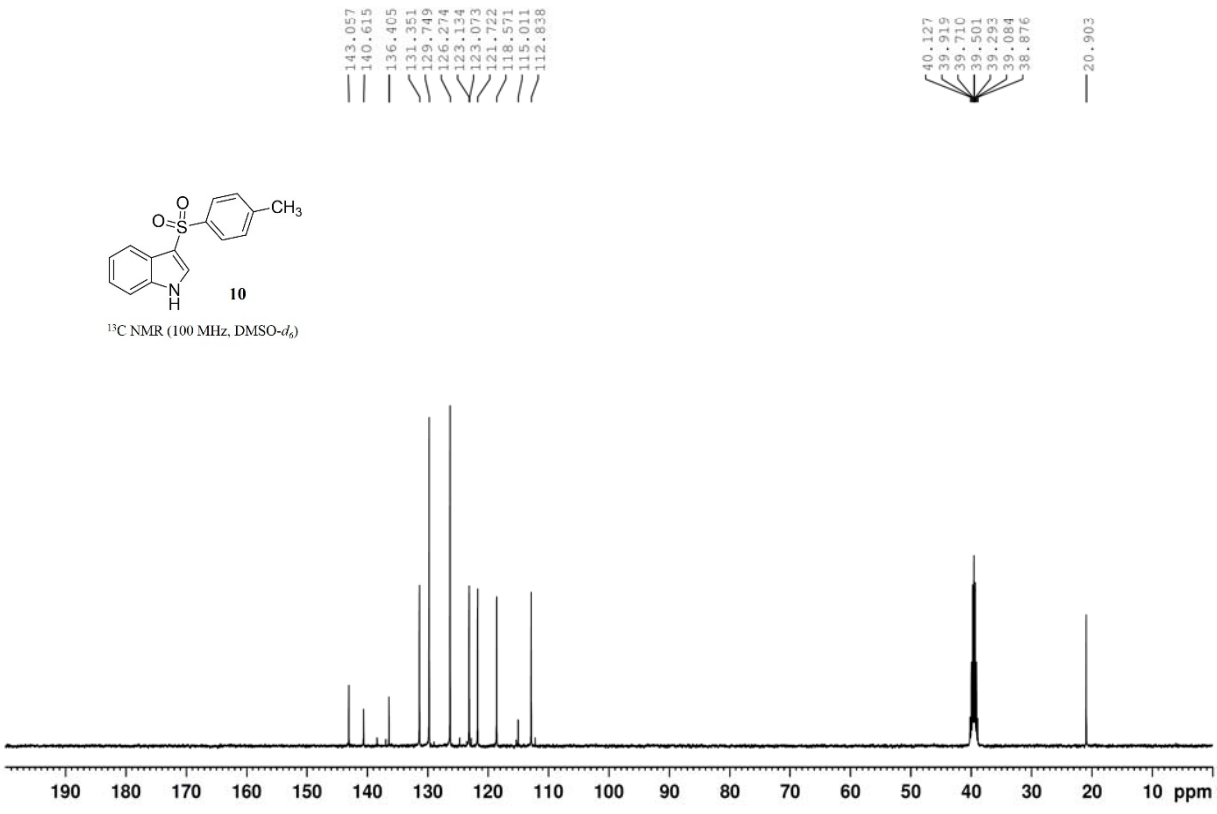


^{1}H NMR (400 MHz, DMSO-d_6)



^{13}C NMR (100 MHz, DMSO-d_6)