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Platinum-Catalyzed Multi-step Reaction of Indoles with Alkynyl Alcohols

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Experimental procedure for the addition reaction of N-methyl indole (1a) with cyclic enol ether 6: A 25 ml round-bottomed flask containing PtCl_2 (0.050 mmol, 5.0 mol %) was evacuated and purged with nitrogen gas three times. Freshly distilled THF (3.0 ml), N-methyl indole (**1a**) (1.00 mmol) and 2,3-dihydro-5-methylfuran (**6**) (3.0 mmol) were sequentially added to the system and the reaction mixture was stirred at room temperature for 2 h. The mixture was filtered through a short Celite and silica gel pad and washed with dichloromethane several times. The filtrate was concentrated and the residue was purified on a silica gel column using hexanes-ethyl acetate as eluent to afford the cyclization product **3a**. Product **3s** was synthesized according to this procedure by using 2,3-dihydro-5-methylfuran (**6**) (1.2 mmol) or N-methyl indole (**1a**) (1.00 mmol) and **3a** (1.0 mmol) for 6 h or N-methyl indole (**1a**) (2.0 mmol) and pent-3-yn-1-ol (**2a**) (1.0 mmol) for 6 h. Product **3t** was also synthesized according to this procedure by using N-methyl indole (**1a**) (1.00 mmol) and **11** (1.2 mmol) for 3 h.

Experimental procedure for the addition reaction of N-methyl indole (1a) with 3-tetrahydro-2-(pent-3-ynyloxy)-2H-pyran (10b): A 25 ml round-bottomed flask containing PtCl_2 (0.050 mmol, 5.0 mol %) was evacuated and purged with nitrogen gas three times. Freshly distilled THF (3.0 ml), N-methyl indole (**1a**) (2.00 mmol) and 3-tetrahydro-2-(pent-3-ynyloxy)-2H-pyran (**10b**) were sequentially added to the system and the reaction mixture was stirred at room temperature for 2 h. The mixture was filtered through a short Celite and silica gel pad and washed with dichloromethane several times. The filtrate was concentrated and the residue was purified on a silica gel

column using hexanes-ethyl acetate as eluent to afford the cyclization products **3a** and **3t**.

Spectral data of compounds **3d-h**, **3k-m**, **3q**, and **3s-t** and copies of ^1H and ^{13}C NMR spectra of all compounds are listed bellow.

3-(Tetrahydro-2-methylfuran-2-yl)-7-methoxy-1-methyl-1H-

indole (3d): ^1H NMR (400 MHz, CDCl_3): δ = 7.27 (d, J = 8.0 Hz, 1 H; HC=), 6.97 (t, J = 8.0 Hz, 1 H; HC=), 6.84 (s, 1 H; HC=), 6.61 (d, J = 7.6 Hz, 1 H; HC=), 4.02-3.96 (m, 5 H; O-CH₂, O-CH₃), 3.92 (s, 3 H; N-CH₃), 2.41-2.39 (m, 1 H; CH₂), 2.04-2.00 (m, 3 H; CH₂), 1.67 (s, 3 H, CH₃); ^{13}C NMR (100 MHz, CDCl_3): δ = 148.11 (C), 127.85 (C), 127.63 (C), 126.62 (CH), 121.68 (C), 119.50 (CH), 113.43 (CH), 102.37 (CH), 82.00 (C), 67.45 (CH₂), 55.62 (O-CH₃), 38.64 (CH₂), 36.65 (N-CH₃), 28.90 (CH₃), 26.32 (CH₂); HRMS (m/z) calcd for C₁₅H₁₉O₂N 245.1416 found 245.1410.

5-Bromo-3-(tetrahydro-2-methylfuran-2-yl)-1-methyl-1H-

indole (3e): ^1H NMR (400 MHz, CDCl_3): δ = 7.84 (d, J = 1.6 Hz, 1 H; HC=), 7.28 (d, J = 8.8 Hz, 1 H; HC=), 7.13 (d, J = 8.4 Hz, 1 H; HC=), 6.95 (s, 1 H; HC=), 4.02-3.93 (m, 2 H; O-CH₂), 3.71 (s, 3 H; N-CH₃), 2.35-2.28 (m, 1 H; CH₂), 2.07-1.93 (m, 3 H; CH₂), 1.64 (s, 3 H, CH₃); ^{13}C NMR (100 MHz, CDCl_3): δ = 136.27 (C), 127.02 (C), 126.84 (CH), 124.12 (CH), 122.85 (CH), 121.22 (C), 112.22 (C), 110.69 (CH), 81.42 (C), 67.18 (CH₂), 38.48 (CH₂), 32.74 (N-CH₃), 28.59 (CH₃), 25.98 (CH₂); HRMS (m/z) calcd for C₁₄H₁₆ON 293.0415 found 293.0409.

3-(Tetrahydro-2-methylfuran-2-yl)-5-iodo-1-methyl-1H-indole

(3f): ^1H NMR (400 MHz, CDCl_3): δ = 8.01 (d, J = 1.2 Hz, 1 H; HC=), 7.42 (d, J = 8.4 Hz, 1 H; HC=), 7.02 (d, J = 8.4 Hz, 1 H; HC=), 6.89 (s, 1 H; HC=), 4.00-3.89 (m, 2 H; O-CH₂),

3.67 (s, 3 H; N-CH₃), 2.32-2.63 (m, 1 H; CH₂), 2.03-1.90 (m, 3 H; CH₂), 1.62 (s, 3 H, CH₃); ¹³C NMR (100 MHz, CDCl₃): δ = 136.69 (C), 129.61 (CH), 129.13 (CH), 127.93 (C), 125.95 (CH), 121.13 (CH), 111.29 (CH), 82.50 (C), 81.44 (C), 67.20 (CH₂), 38.52 (CH₂), 32.69 (N-CH₃), 28.66 (CH₃), 25.99 (CH₂); HRMS (m/z) calcd for C₁₄H₁₆ON 341.0277 found 341.0277.

3-(Tetrahydro-2-methylfuran-2-yl)-1-methyl-5-nitro-1H-

indole (3g): ¹H NMR (400 MHz, CDCl₃): δ = 8.65 (d, *J* = 2.0 Hz, 1 H; HC=), 8.06 (d, *J* = 9.2 Hz, 1 H; HC=), 7.26 (d, *J* = 9.2 Hz, 1 H; HC=), 7.08 (s, 1 H; HC=), 4.02-3.92 (m, 2 H; O-CH₂), 3.78 (s, 3 H; N-CH₃), 2.33-2.28 (m, 1 H; CH₂), 2.12-1.91 (m, 3 H; CH₂), 1.64 (s, 3 H, CH₃); ¹³C NMR (100 MHz, CDCl₃): δ = 141.03 (C), 140.36 (C), 128.19 (CH), 124.87 (C), 124.66 (C), 117.82 (CH), 117.13 (CH), 109.12 (C), 81.24 (C), 67.35 (CH₂), 38.79 (CH₂), 33.79 (N-CH₃), 28.74 (CH₃), 26.01 (CH₂); HRMS (m/z) calcd for C₁₄H₁₆O₃N₂ 260.1161 found 260.1167.

3-(Tetrahydro-2-methylfuran-2-yl)-1,2-dimethyl-1H-indole

(3h): ¹H NMR (400 MHz, CDCl₃): δ = 7.68 (d, *J* = 8.0 Hz, 1 H; HC=), 7.22 (d, *J* = 8.0 Hz, 1 H; HC=), 7.13 (t, *J* = 7.2 Hz, 1 H; HC=), 7.04 (t, *J* = 8.0 Hz, 1 H; HC=), 4.04-3.90 (m, 2 H; O-CH₂), 3.75 (s, 3 H; N-CH₃), 2.59 (s, 3 H; CH₃), 2.57-2.50 (m, 1 H; CH₂), 2.22-2.17 (m, 1 H; CH₂), 2.08-1.87 (m, 2 H; CH₂), 1.64 (s, 3 H, CH₃); ¹³C NMR (100 MHz, CDCl₃): δ = 136.99 (C), 132.03 (C), 126.07 (C), 120.50 (CH), 120.21 (CH), 118.90 (CH), 117.26 (C), 108.81 (CH), 83.69 (C), 67.03 (CH₂), 39.71 (CH₂), 31.84 (N-CH₃), 29.50 (CH₃), 29.32 (CH₃), 26.01 (CH₂); HRMS (m/z) calcd for C₁₅H₁₉ON 229.1467 found 229.1468.

3-(Tetrahydro-2-phenylfuran-2-yl)-1-methyl-1H-indole (3k):

¹H NMR (400 MHz, CDCl₃): δ = 7.57 (d, *J* = 8.0 Hz, 1 H; HC=),

7.52-7.50 (m, 2 H; HC=), 7.31-7.14 (m, 5 H; HC=), 7.00 (t, $J = 8.0$ Hz, 1 H; HC=), 6.84 (s, 1 H; HC=), 4.11-4.07 (m, 2 H; O-CH₂), 3.82 (s, 3 H; N-CH₃), 2.78-2.71 (m, 1 H; CH₂), 2.59-2.43 (m, 1 H; CH₂), 2.09-1.92 (m, 2 H; CH₂); ¹³C NMR (125 MHz, CDCl₃): $\delta = 146.46$ (C), 137.72 (C), 127.87 (CH), 126.72 (CH), 126.49 (CH), 126.05 (CH), 125.88 (C), 121.51 (CH), 121.08 (CH), 119.89 (C), 119.04 (CH), 109.11 (CH), 85.43 (C), 67.37 (CH₂), 38.65 (CH₂), 32.65 (N-CH₃), 25.80 (CH₂); HRMS (m/z) calcd for C₁₉H₁₉ON 277.1467 found 277.1470.

(E)-4-(1-methyl-1H-indol-3-yl)-4-phenylbut-3-en-1-ol and (Z)-4-(1-methyl-1H-indol-3-yl)-4-phenylbut-3-en-1-ol (3k'):

¹H NMR of *E* isomer (400 MHz, CDCl₃): $\delta = 7.37$ -7.32 (m, 3 H; HC=), 7.25-7.18 (m, 5 H; HC=), 7.08 (s, 1 H; HC=), 6.98 (t, $J = 8.0$ Hz, 1 H; HC=), 6.11 (t, $J = 7.2$ Hz, 1 H; HC=), 3.82 (s, 3 H; N-CH₃), 3.69 (t, $J = 7.2$ Hz, 2 H; O-CH₂), 2.53 (q, $J = 6.8, 14.6$ Hz, 2 H; CH₂); ¹H NMR of *Z* isomer (400 MHz, CDCl₃): $\delta = 7.61$ (d, $J = 8.0$ Hz, 1 H; HC=), 7.39-7.26 (m, 6 H; HC=), 7.21 (t, $J = 7.2$ Hz, 1 H; HC=), 7.01 (t, $J = 6.8$ Hz, 1 H; HC=), 6.65 (s, 1 H; HC=), 6.18 (t, $J = 7.2$ Hz, 1 H; HC=), 3.72 (t, $J = 7.2$ Hz, 2 H; O-CH₂), 3.68 (s, 3 H; N-CH₃), 2.53 (q, $J = 6.8, 15.0$ Hz, 2 H; CH₂); HRMS (m/z) calcd for C₁₉H₁₉ON 277.1467 found 277.1470.

3-(Tetrahydro-2-(thiophen-2-yl)furan-2-yl)-1-methyl-1H-

indole (31): ¹H NMR (400 MHz, CDCl₃): $\delta = 7.61$ (d, $J = 8.0$ Hz, 1 H; HC=), 7.32 (t, $J = 7.6$ Hz, 1 H; HC=), 7.22 (d, $J = 8.0$ Hz, 1 H; HC=), 7.05 (t, $J = 8.0$ Hz, 1 H; HC=), 6.98 (s, 1 H; HC=), 6.94-6.91 (m, 3 H; HC=), 4.17-4.10 (m, 2 H; O-CH₂), 3.72 (s, 3 H; N-CH₃), 2.77-2.72 (m, 1 H; CH₂), 2.66-2.62 (m, 1 H; CH₂), 2.15-2.09 (m, 2 H; CH₂); ¹³C NMR (125 MHz, CDCl₃): $\delta = 151.82$ (C), 137.65 (C), 127.08 (C), 126.60 (CH), 126.33 (CH), 125.71 (C), 124.76 (CH), 124.23 (CH),

121.52 (CH), 120.82 (CH), 119.63 (CH), 109.21 (CH), 83.52 (C), 67.06 (CH₂), 40.08 (CH₂), 32.66 (N-CH₃), 26.05 (CH₂); HRMS (m/z) calcd for C₁₇H₁₇ONS 283.1031 found 283.1036.

4-(1-Methyl-1*H*-indol-3-yl)-4-(thiophen-2-yl)but-3-en-1-ol

(**31'**): ¹H NMR of *E* isomer (400 MHz, CDCl₃): δ = 7.31-7.29 (m, 2 H; HC=), 7.25 (t, *J* = 8.0 Hz, 1 H; HC=), 7.11-7.03 (m, 3 H; HC=), 6.87 (m, 1 H; HC=), 6.11 (t, *J* = 7.2 Hz, 1 H; HC=), 6.79 (m, 1 H; =CH), 6.21 (t, *J* = 8.0 Hz, 1 H; HC=), 3.82 (s, 3 H, N-CH₃), 3.67 (t, *J* = 6.4 Hz, 2 H; O-CH₂), 2.41 (q, *J* = 6.8, 14.0 Hz, 2 H; CH₂); HRMS (m/z) calcd for C₁₇H₁₇ONS 283.1031 found 283.1036.

3-(2-Ethyl-tetrahydro-5-methylfuran-2-yl)-1-methyl-1*H*-indole (3m)

(1:1 diastereoisomer): ¹H NMR (500 MHz, CDCl₃): δ = 7.67 (d, *J* = 8.0 Hz, 1 H; HC=), 7.62 (d, *J* = 8.5 Hz, 1 H; HC=), 7.26 (d, *J* = 8.0 Hz, 2 H; HC=), 7.18 (t, *J* = 7.0 Hz, 2 H; HC=), 7.05 (t, *J* = 8.0 Hz, 2 H; HC=), 6.98 (s, 1 H; HC=), 6.93 (s, 1 H; HC=), 4.21-4.11 (m, 2 H; O-CH₂), 3.72 (s, 6 H; N-CH₃), 2.33-2.30 (m, 2 H; CH₂), 2.16-2.00 (m, 2 H; CH₂), 1.99-1.93 (m, 6 H; CH₂), 1.57-1.50 (m, 2 H; CH₂), 1.24 (t, *J* = 9.0 Hz, 4 H; CH₂), 0.80 (m, 6 H; CH₃); ¹³C NMR (125 MHz, CDCl₃): δ = 137.70 (C), 126.53 (CH), 125.95 (CH), 125.71 (C), 121.63 (C), 121.17 (CH), 121.06 (CH), 119.07 (CH), 120.73 (C), 120.62 (CH), 120.31 (CH), 118.56 (CH), 118.47 (CH), 109.19 (CH), 83.35 (C), 85.21 (C), 75.11 (CH), 74.13 (CH), 37.70 (CH₂), 36.52 (CH₂), 34.87 (CH₂), 34.06 (CH₂), 33.70 (CH₂), 33.48 (CH₂), 32.64 (N-CH₃), 21.73 (CH₃), 21.66 (CH₃), 9.14 (CH₃), 9.06 (CH₃); HRMS (m/z) calcd for C₁₆H₂₁ON 243.1623 found 243.1624.

6-Di(1*H*-indol-3-yl)heptan-1-ol (3q): IR (cm⁻¹): 3556.09 (OH), 3301.54 (NH); ¹H NMR (400 MHz, CDCl₃): δ = 7.93 (N-H), 7.34 (d, *J* = 8.0 Hz, 2 H; HC=), 7.34 (d, *J* = 8.0 Hz, 2 H;

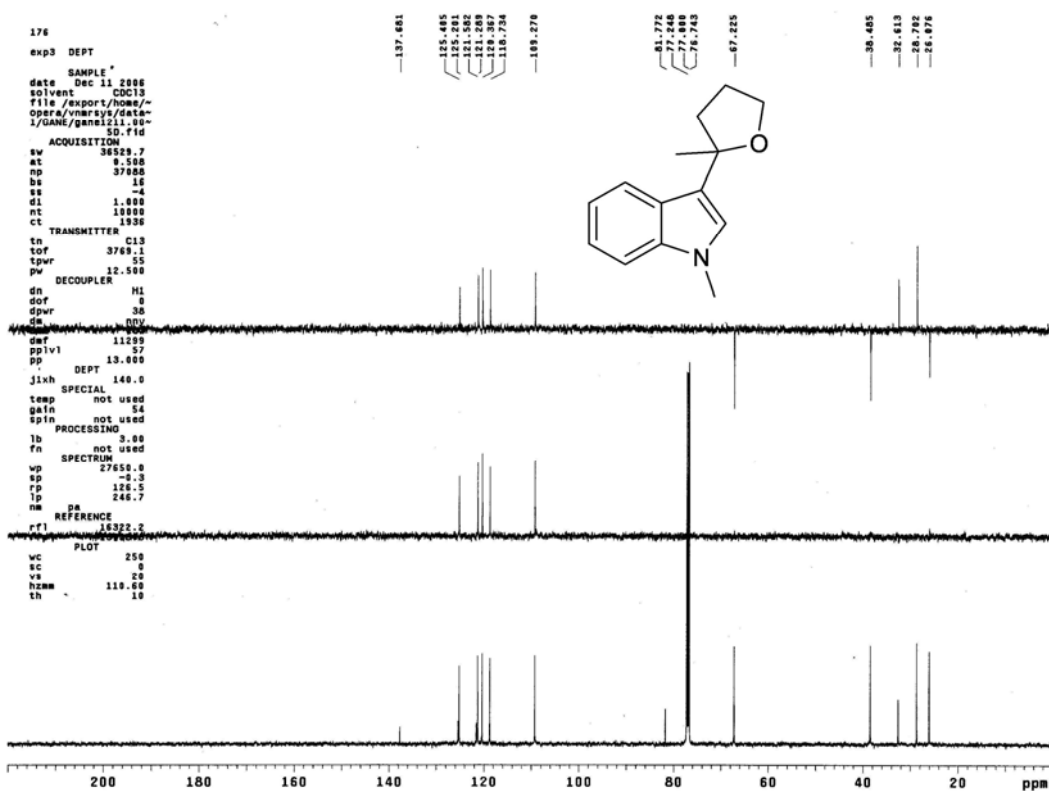
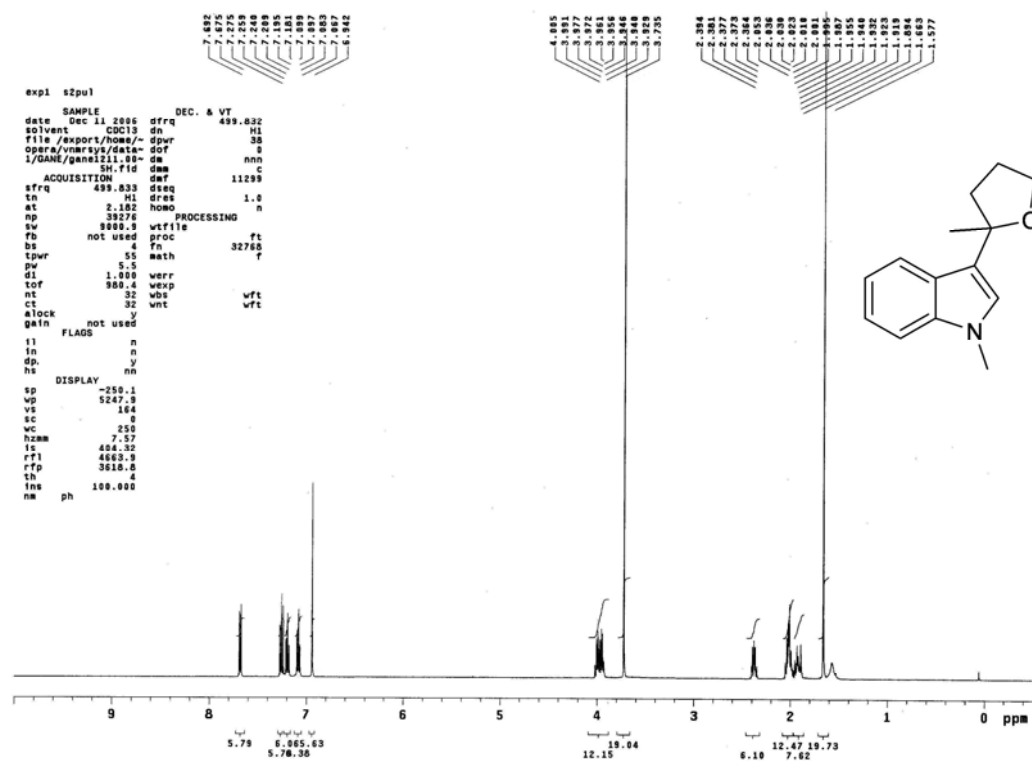
HC=), 7.27 (d, J = 8.0 Hz, 2 H; HC=), 7.06–7.03 (m, 4 H; HC=), 6.85–6.81 (m, 2 H; HC=), 3.48 (t, J = 6.4 Hz, 2 H; OCH₂), 2.34 (t, J = 7.6 Hz, 2 H; CH₂), 1.81 (s, 3 H, CH₃), 1.46–1.14 (m, 6 H; CH₂); ¹³C NMR (100 MHz, CDCl₃): δ = 136.98 (C), 126.36 (C), 124.31 (C), 121.28 (CH), 121.11 (CH), 121.10 (CH), 118.54 (CH), 110.94 (CH), 62.96 (CH₂), 40.29 (CH₂), 38.37 (C), 32.59 (CH₂), 26.92 (CH₃), 26.23 (CH₂), 24.24 (CH₂); HRMS (m/z) calcd for C₂₃H₂₆ON₂ 346.2045 found 346.2053.

4,4-Bis(1-methyl-1H-indol-3-yl)pentan-1-ol (3s): IR (cm⁻¹): 3470.25 (OH); ¹H NMR (400 MHz, CDCl₃): δ = 7.38 (d, J = 8.0 Hz, 2 H; HC=), 7.25 (d, J = 8.0 Hz, 2 H; HC=), 7.11 (t, J = 7.2 Hz, 2 H; HC=), 6.92 (s, 2 H; HC=), 6.86 (t, J = 7.2 Hz, 2 H; HC=), 3.73 (s, 3 H; N-CH₃), 3.75 (s, 3 H; N-CH₃), 3.54 (t, J = 6.8 Hz, 2 H; OCH₂), 2.43 – 2.39 (m, 2 H; CH₂), 1.84 (s, 3 H, CH₃), 1.46–1.42 (m, 2 H; CH₂); ¹³C NMR (100 MHz, CDCl₃): δ = 137.64 (C), 126.69 (C), 126.14 (CH), 122.50 (C), 121.30 (CH), 120.86 (CH), 118.03 (CH), 108.99 (CH), 63.62 (CH₂), 38.15 (CH₂), 36.64 (C), 32.66 (N-CH₃), 28.24 (CH₂), 27.34 (CH₃); HRMS (m/z) calcd for C₂₃H₂₆ON₂ 346.2045 found 346.2050.

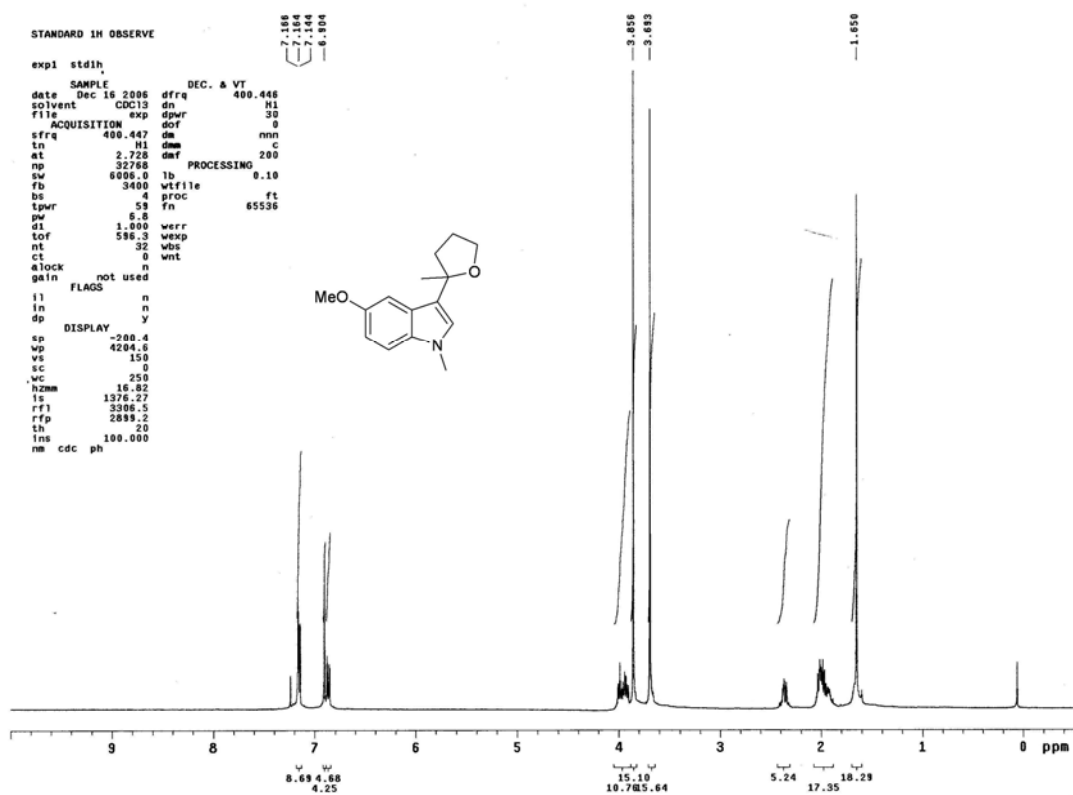
1-Methyl-3-(1-(1-methyl-1H-indol-3-yl)-5-(tetrahydro-2H-pyran-2-yloxy)pentyl)-1H-indole (3t): ¹H NMR (400 MHz, CDCl₃): δ = 7.61 (d, J = 8.0 Hz, 2 H; HC=), 7.26 (d, J = 8.0 Hz, 2 H; HC=), 7.19 (t, J = 8.0 Hz, 2 H; HC=), 7.04 (t, J = 8.0 Hz, 2 H; HC=), 6.85 (s, 2 H; HC=), 4.50–4.46 (m, 2 H; O-CH₂), 3.80 (m, 8 H; N-CH₃, O-CH₂), 3.46–3.33 (m, 2 H; O-CH₂), 2.26–2.20 (q, J = 7.6 Hz, 2 H; CH₂), 1.82–1.51 (m, 10 H; CH₂); ¹³C NMR (100 MHz, CDCl₃): δ = 137.17 (C), 127.45 (C), 126.15 (CH), 121.18 (CH), 119.67 (CH), 118.97 (C), 118.34 (CH), 109.02 (CH), 98.77 (CH), 67.49 (CH₂), 62.28

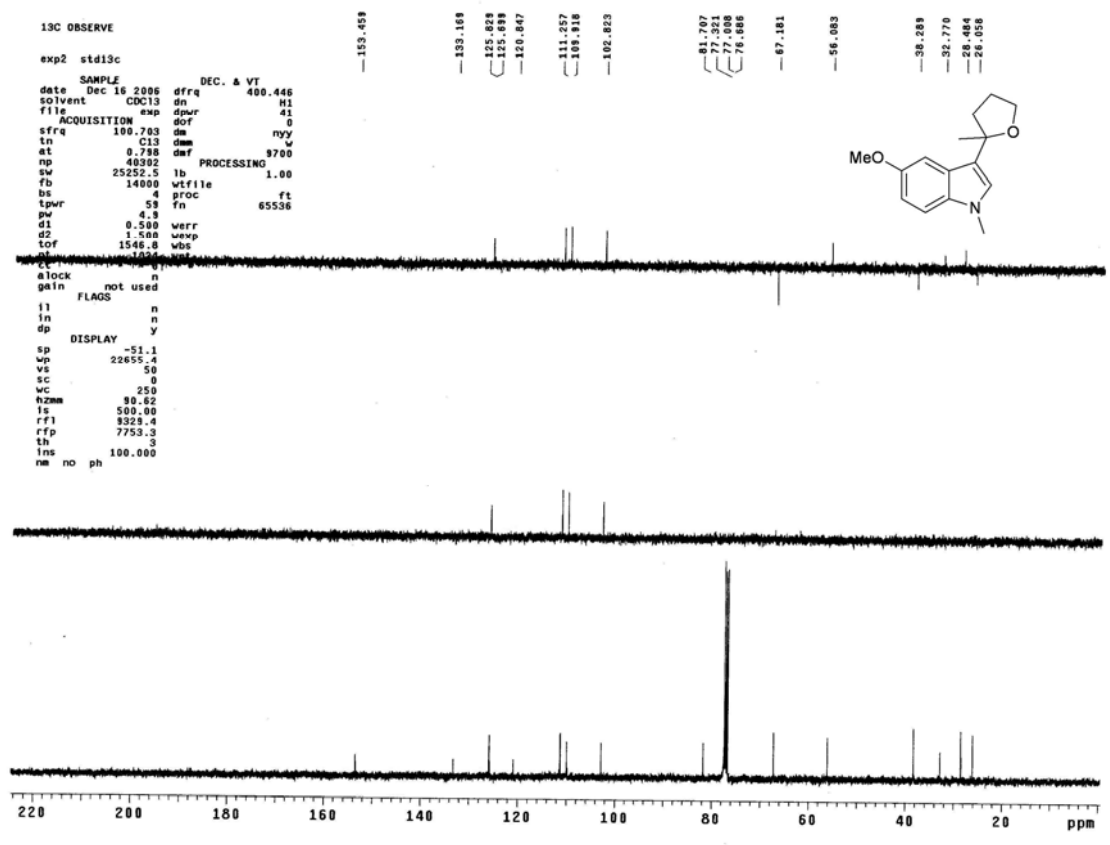
(CH₂), 36.12 (CH₂), 33.72 (C-H), 32.59 (N-CH₃), 30.70 (CH₂),
29.76 (CH₂), 25.44 (CH₂), 24.85 (CH₂), 19.63 (CH₂); HRMS
(m/z) calcd for C₂₈H₃₄O₂N₂ 430.2620 found 430.2629.

^1H and ^{13}C NMR spectra of compound **3a**.

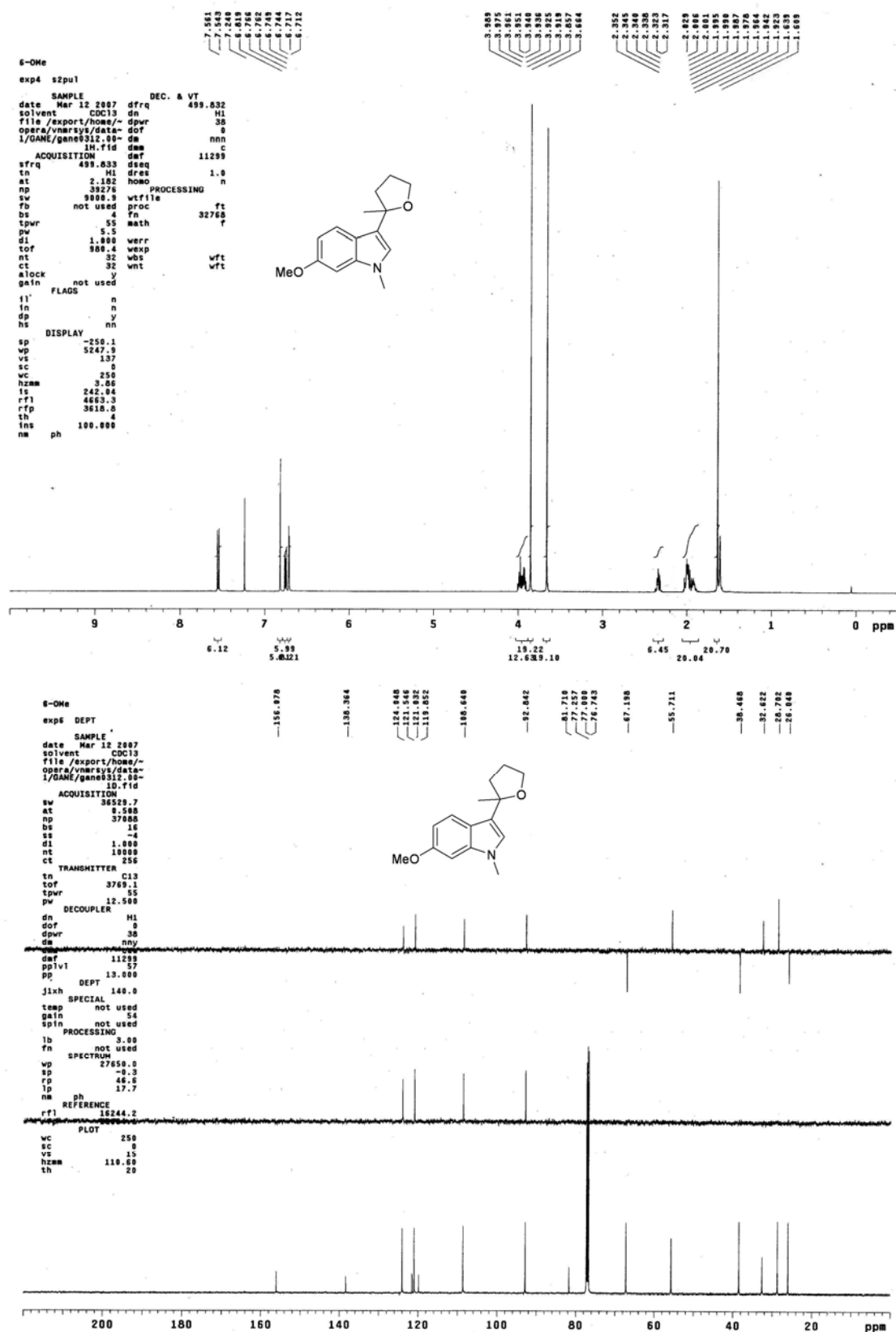


^1H and ^{13}C NMR spectra of compound **3b**.

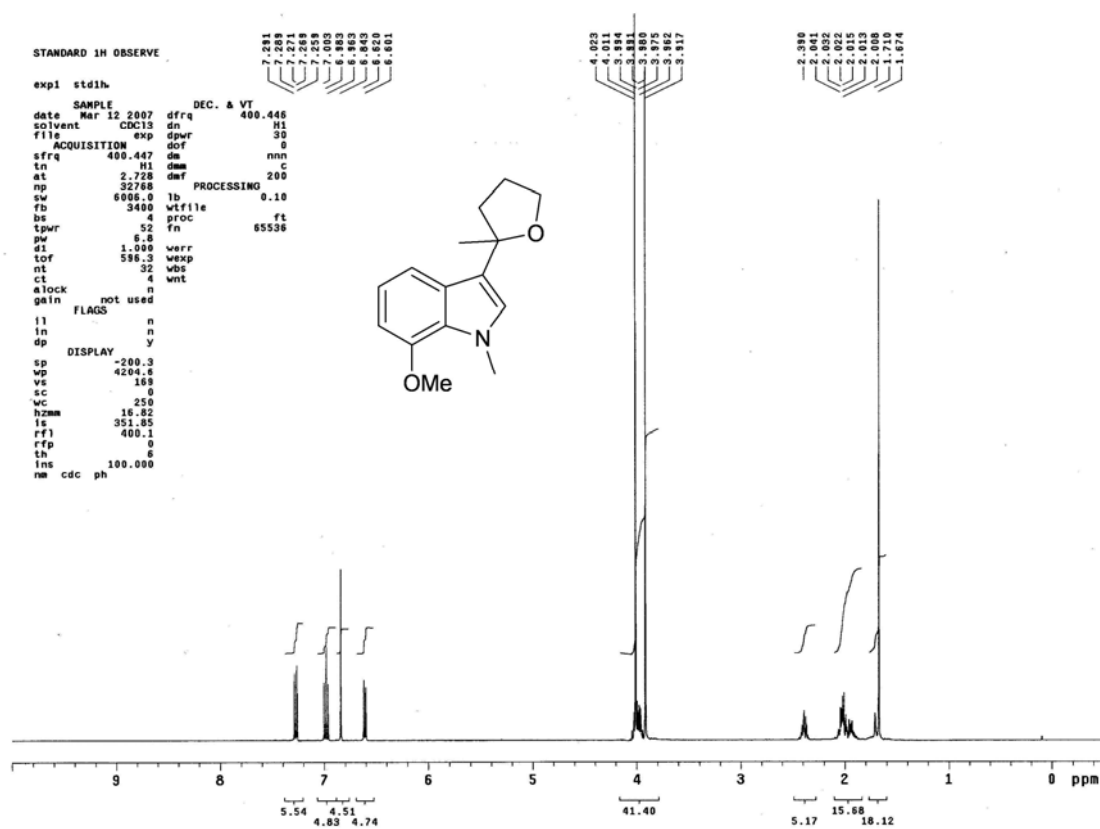




^1H and ^{13}C NMR spectra of compound **3c**.



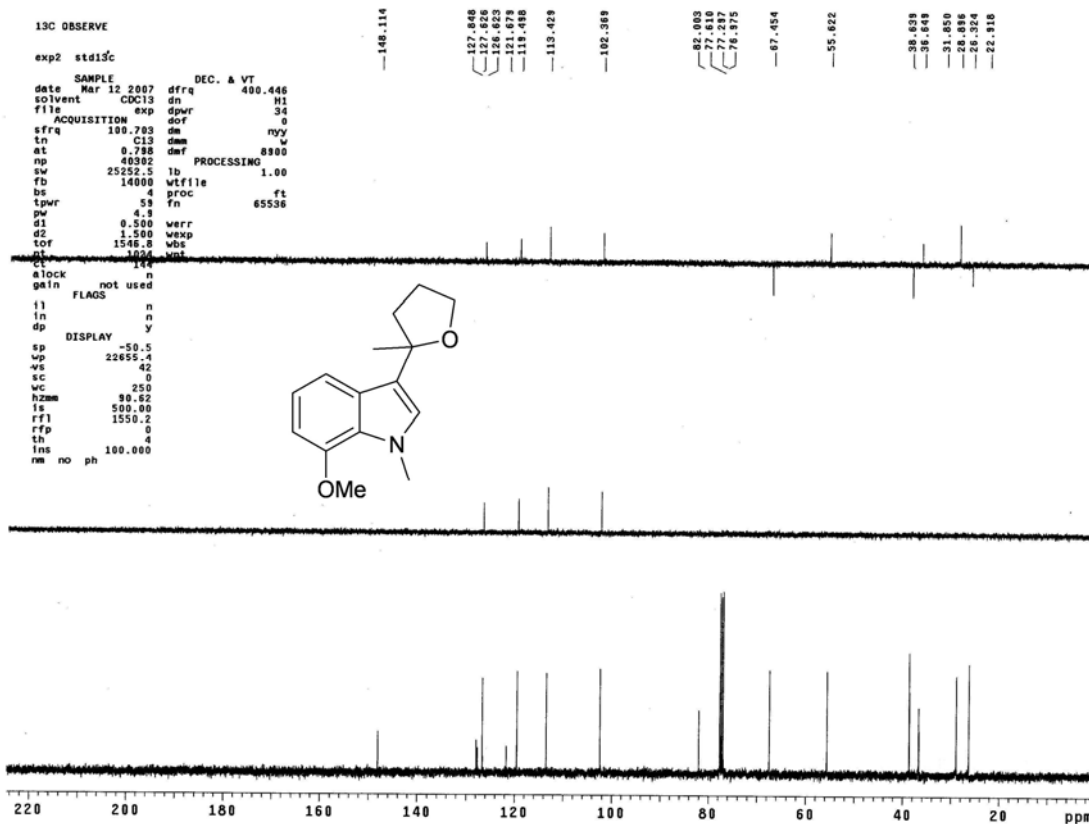
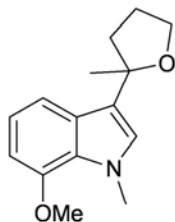
^1H and ^{13}C NMR spectra of compound **3d**.



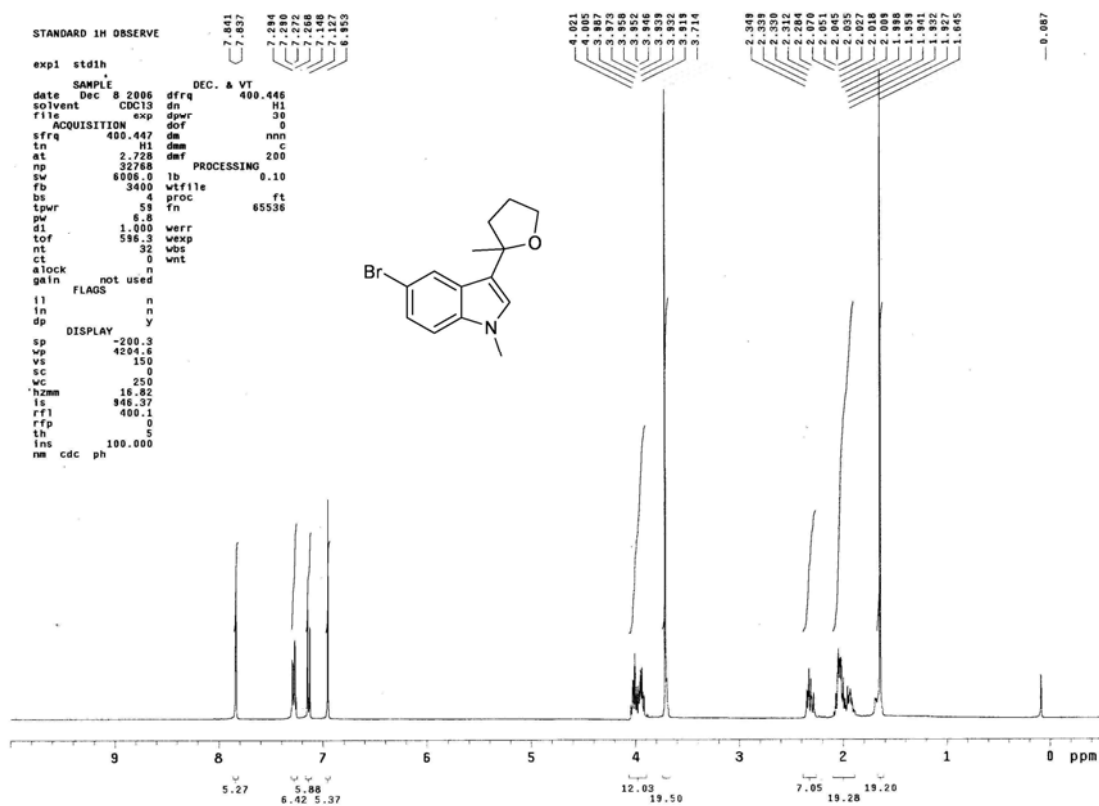
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^1H and ^{13}C NMR spectra of compound **3e**.



13C OBSERVE

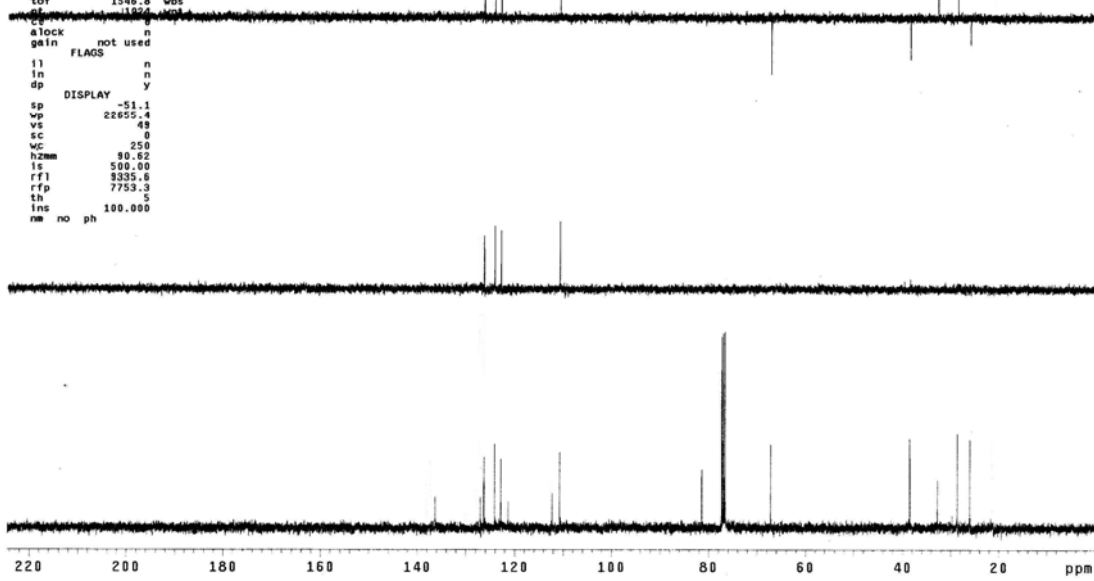
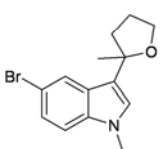
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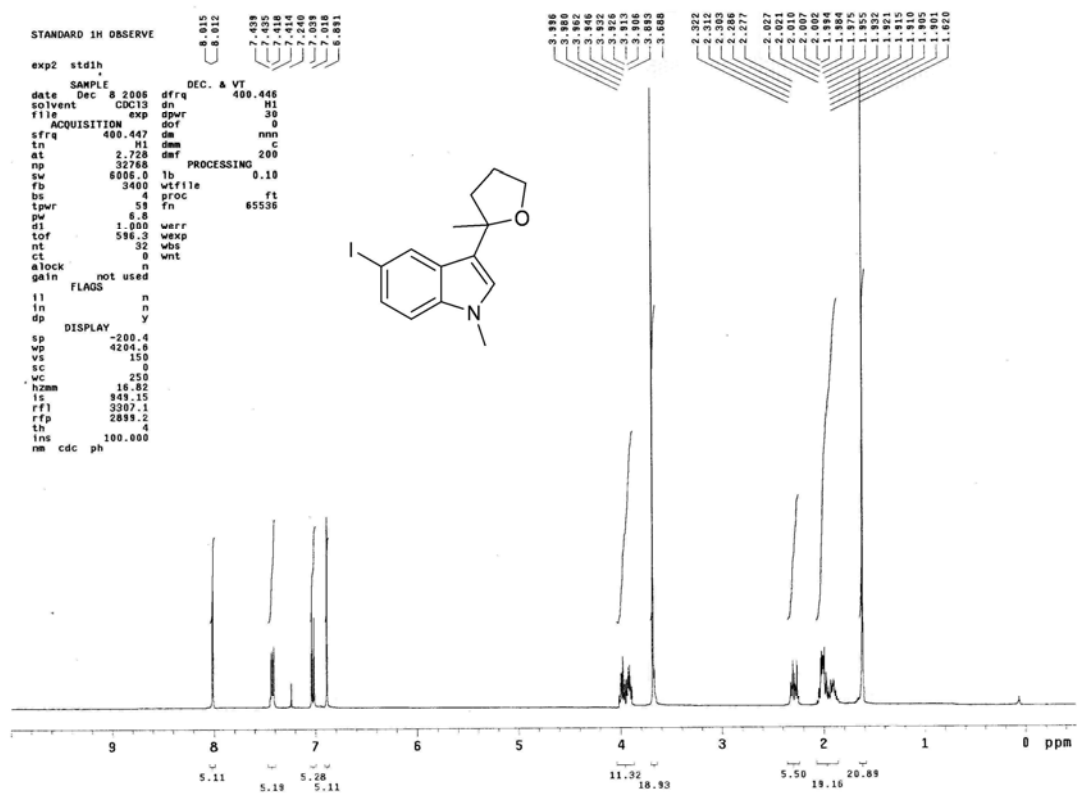
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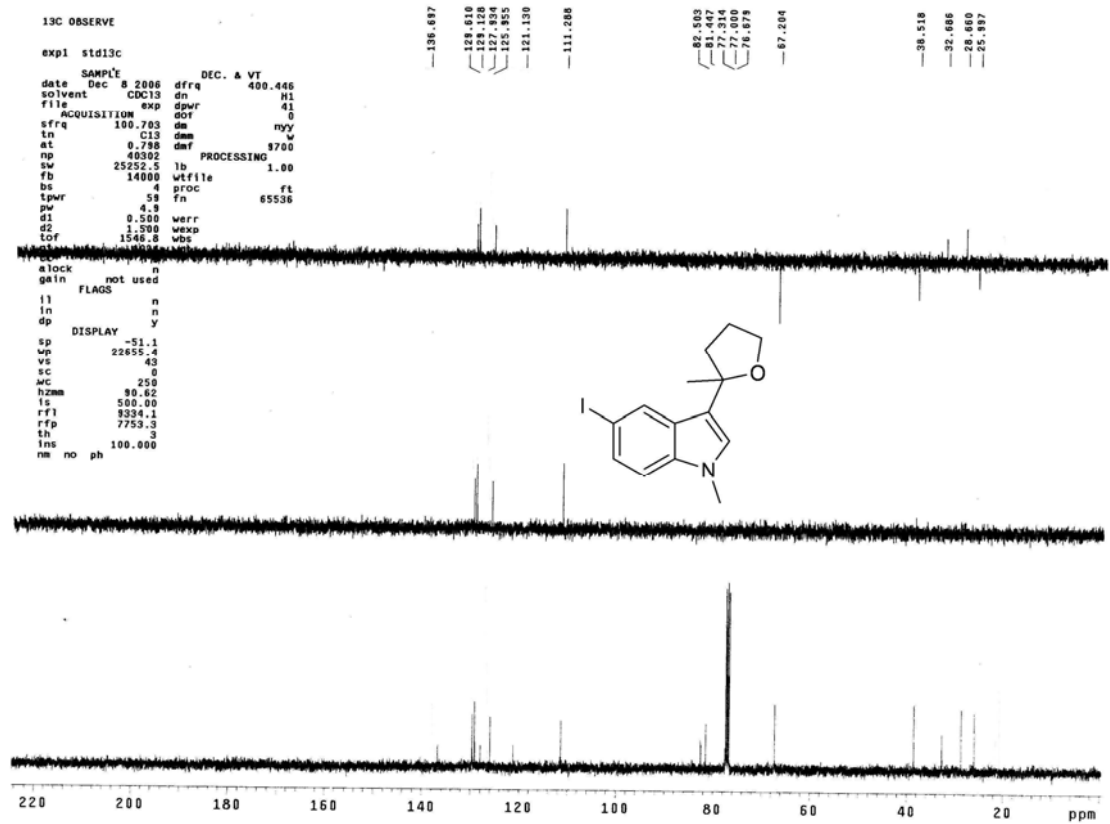
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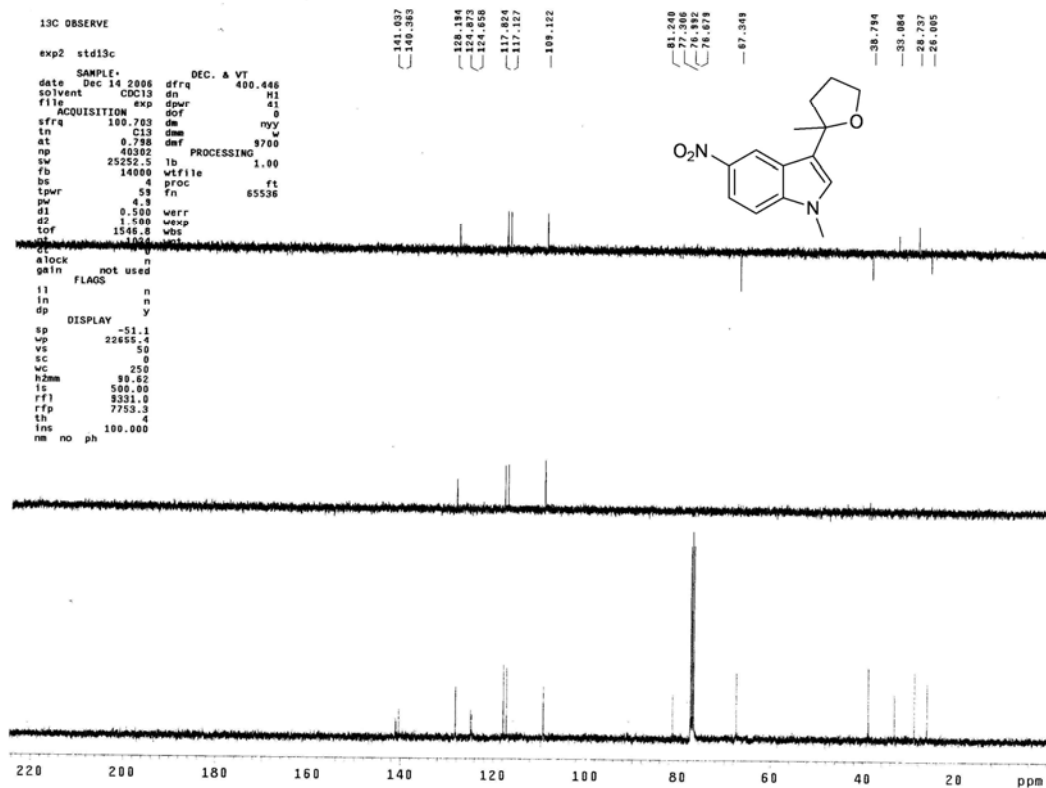
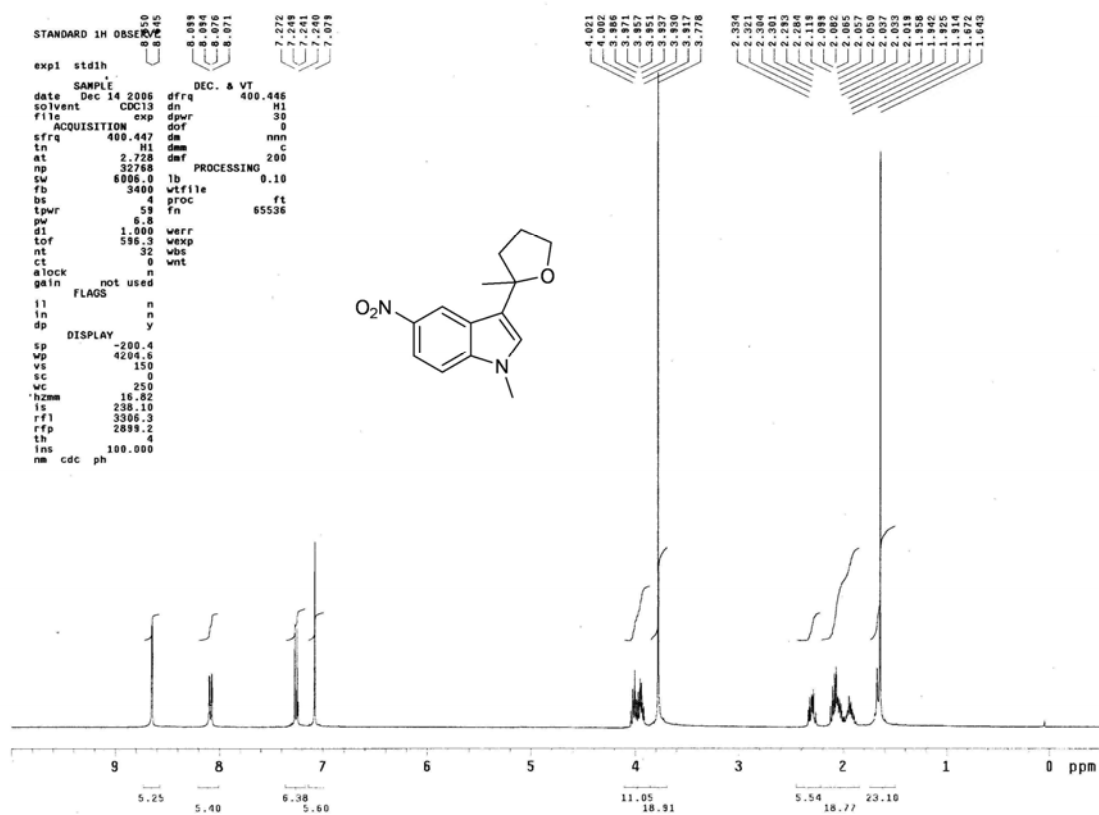


^1H and ^{13}C NMR spectra of compound **3f**.

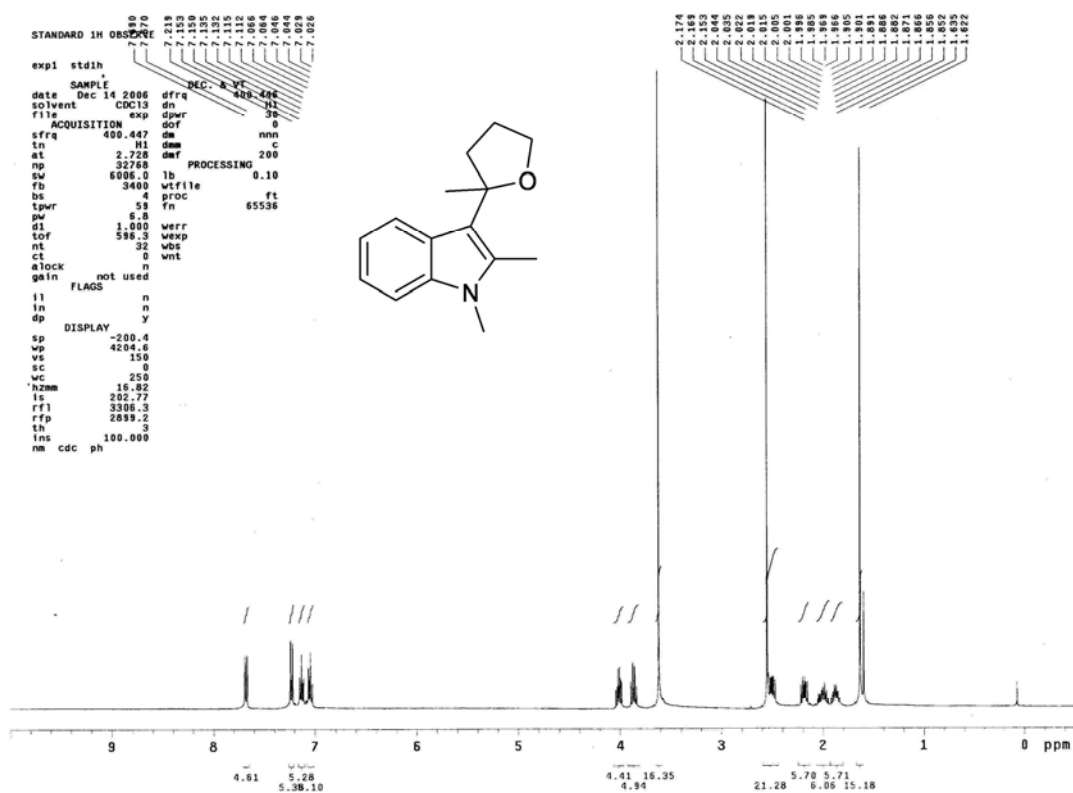


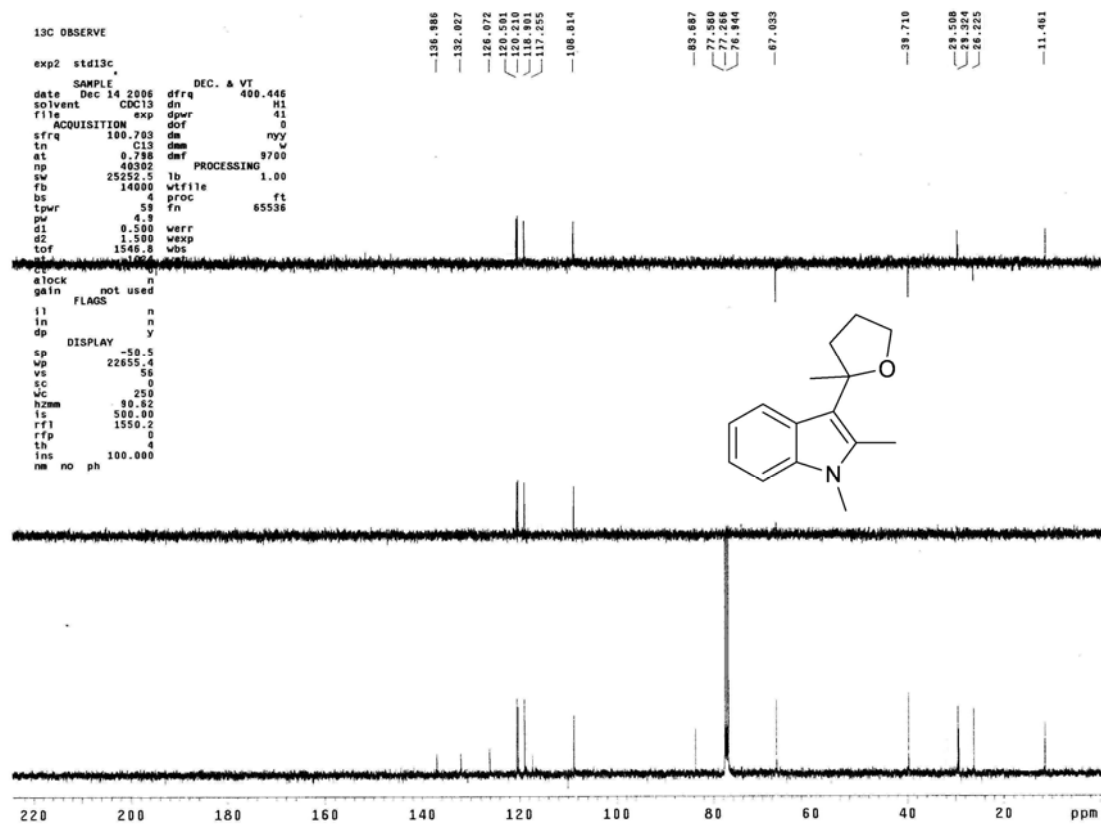


^1H and ^{13}C NMR spectra of compound **3g**.

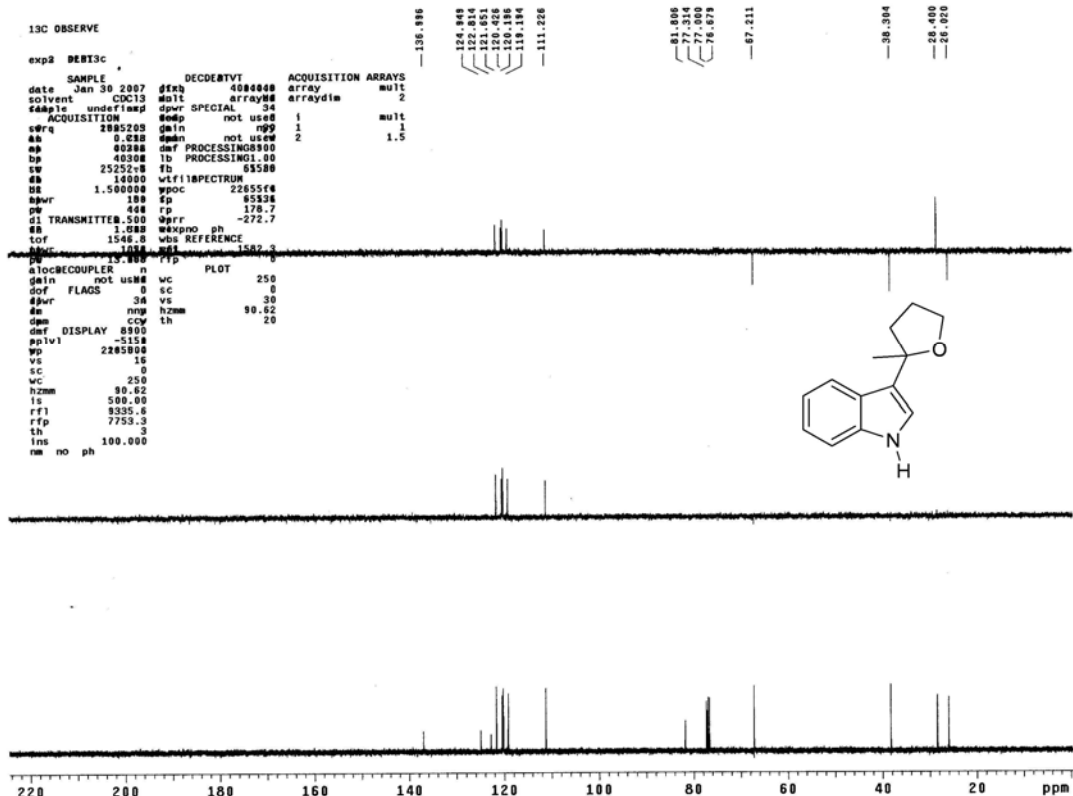
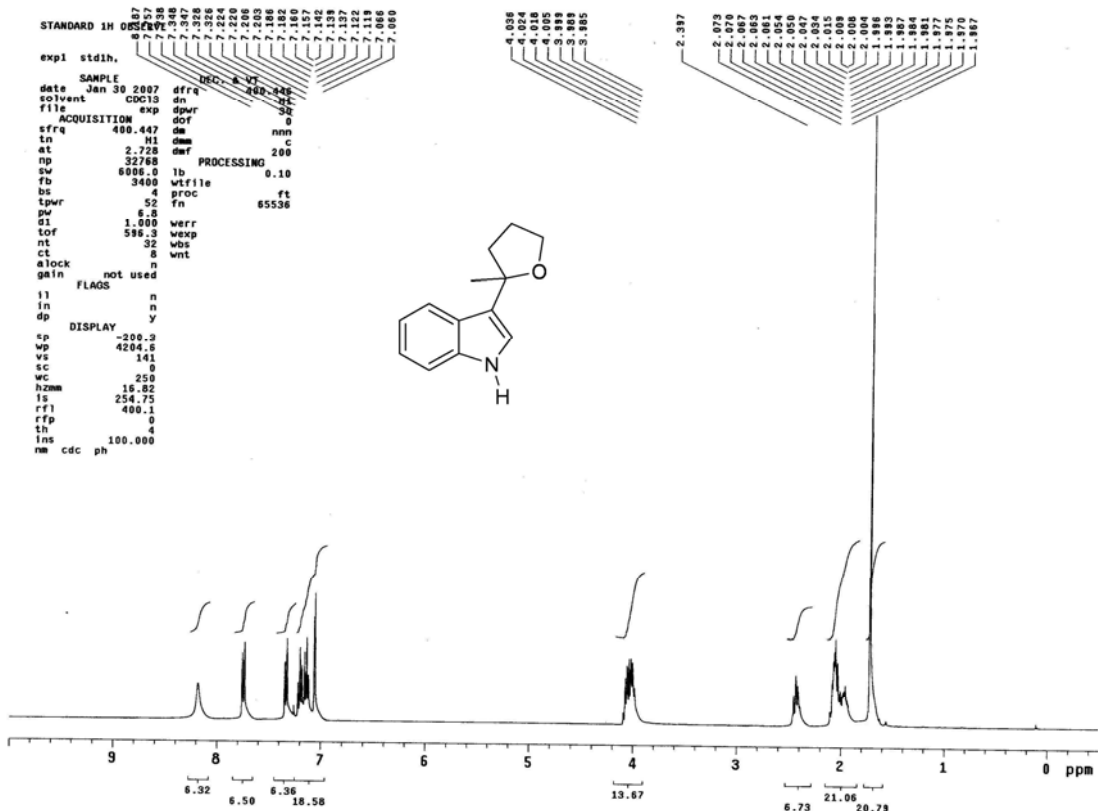


^1H and ^{13}C NMR spectra of compound **3h**.





^1H and ^{13}C NMR spectra of compound **3i**.



STANDARD 1H OBSERVE

exp3 stdih
date Dec 8 2005 dfrq 400.446
solvent CDC13 dn H1
file ACQUISITION exp dpr 30
sfrq 400.447 dm nnn
in H1 dm C
at 2.728 daf 200
np 32768
sw 6006.9 lb wtfile 0.10
fb 3400 wfile
bs 4 proc ft
tipr 53 fn 65536
pw 6.8
d1 1.000 werr
tdr 536.5 wexp
nt 32 wbs
ct 0 wnt
elock n
gain not used
FLAGS
il n
in n
dp Y
DISPLAY
sp -200.3
wp 4204.6
vs 150
sc 0
wc 250
hzmm 16.82
ls 1214.78
rf1 400.1
rfp 0
th 6
ins 100.000
nm cdc ph

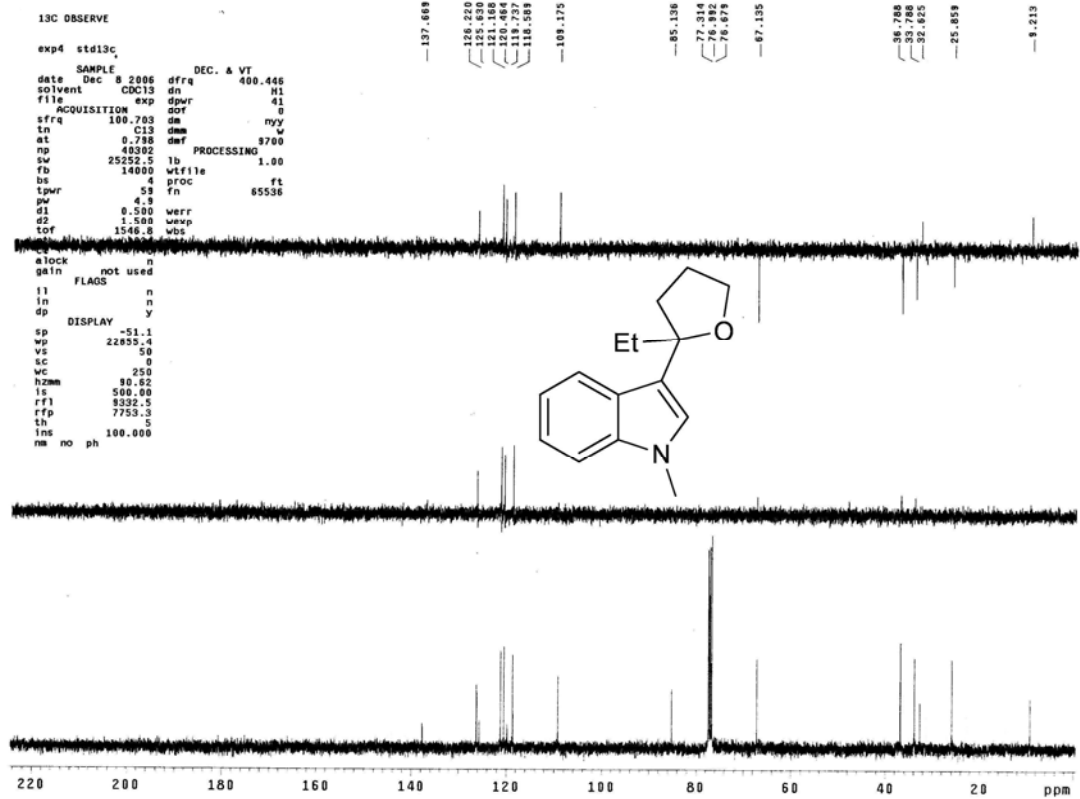
DEC. & VT

PROCESSING

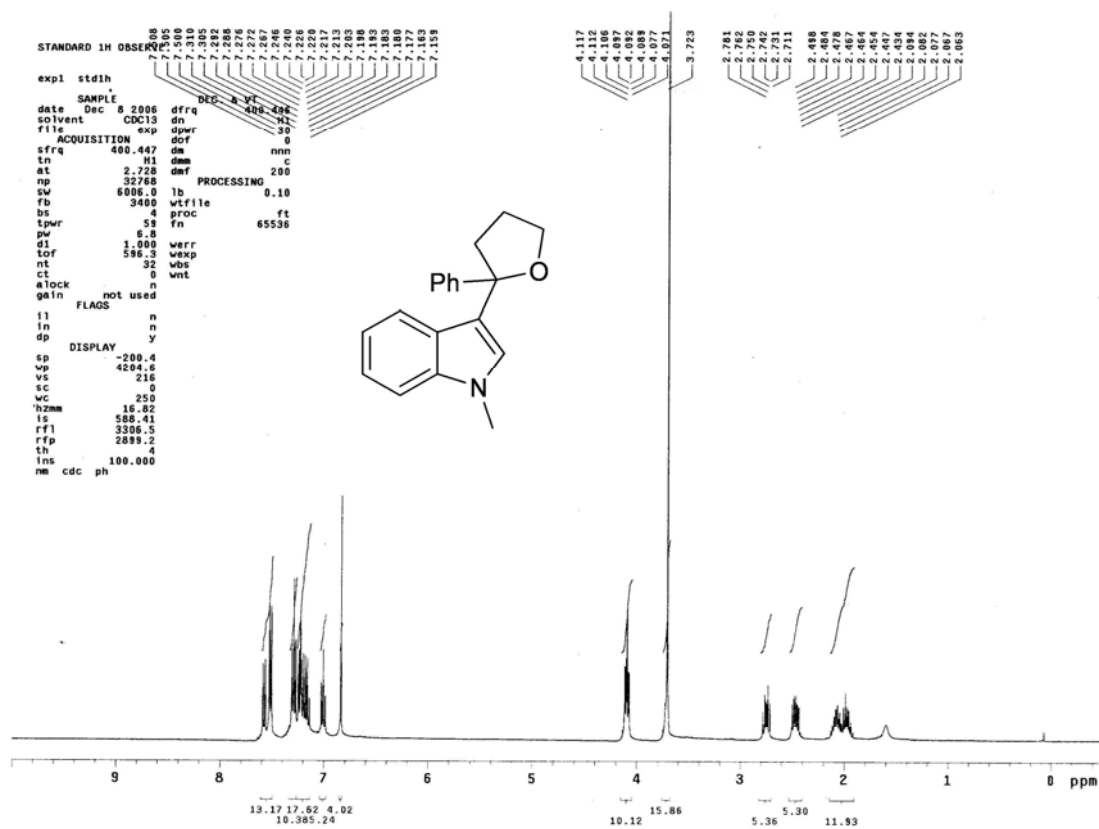
Chemical structure: CC1(C2=CC3=C(C=C2)C(=C1)N(C)C3)C4CCCCO4

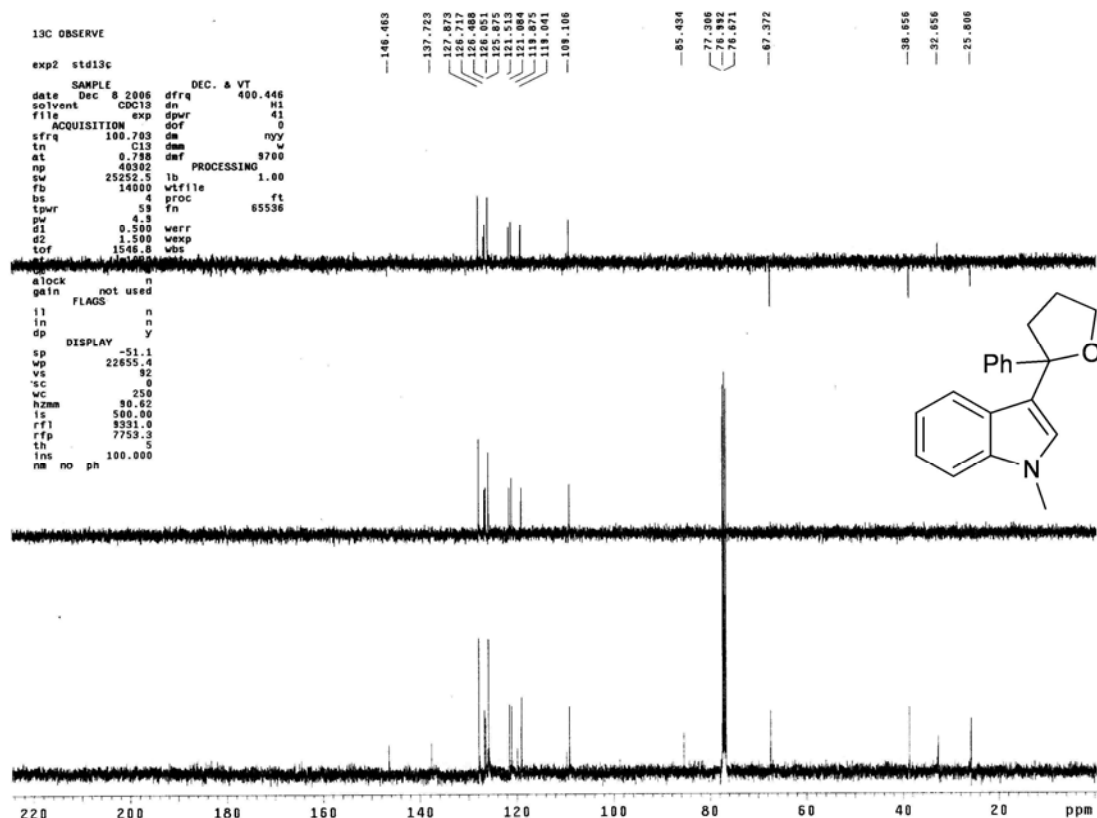
Peak list (ppm):

Peak	Chemical Shift (ppm)
1	12.51
2	8.57
3	7.18
4	7.10
5	7.01
6	6.99
7	6.91
8	6.83
9	6.75
10	6.67
11	6.59
12	6.51
13	6.43
14	6.35
15	6.27
16	6.19
17	6.11
18	6.03
19	5.95
20	5.87
21	5.79
22	5.71
23	5.63
24	5.55
25	5.47
26	5.39
27	5.31
28	5.23
29	5.15
30	5.07
31	5.00
32	4.92
33	4.84
34	4.76
35	4.68
36	4.60
37	4.52
38	4.44
39	4.36
40	4.28
41	4.20
42	4.12
43	4.04
44	3.96
45	3.88
46	3.80
47	3.72
48	3.64
49	3.56
50	3.48
51	3.40
52	3.32
53	3.24
54	3.16
55	3.08
56	3.00
57	2.92
58	2.84
59	2.76
60	2.68
61	2.60
62	2.52
63	2.44
64	2.36
65	2.28
66	2.20
67	2.12
68	2.04
69	1.96
70	1.88
71	1.80
72	1.72
73	1.64
74	1.56
75	1.48
76	1.40
77	1.32
78	1.24
79	1.16
80	1.08
81	1.00
82	0.92
83	0.84
84	0.76
85	0.68
86	0.60
87	0.52
88	0.44
89	0.36
90	0.28
91	0.20
92	0.12
93	0.04

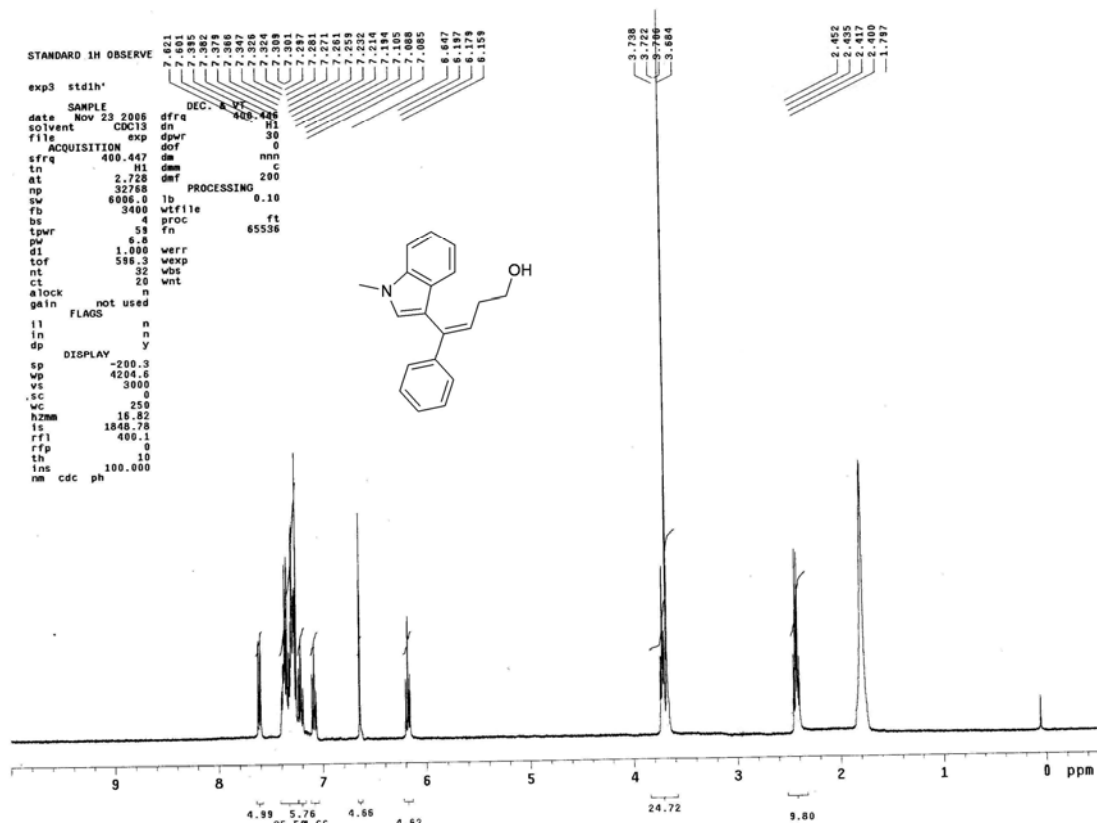
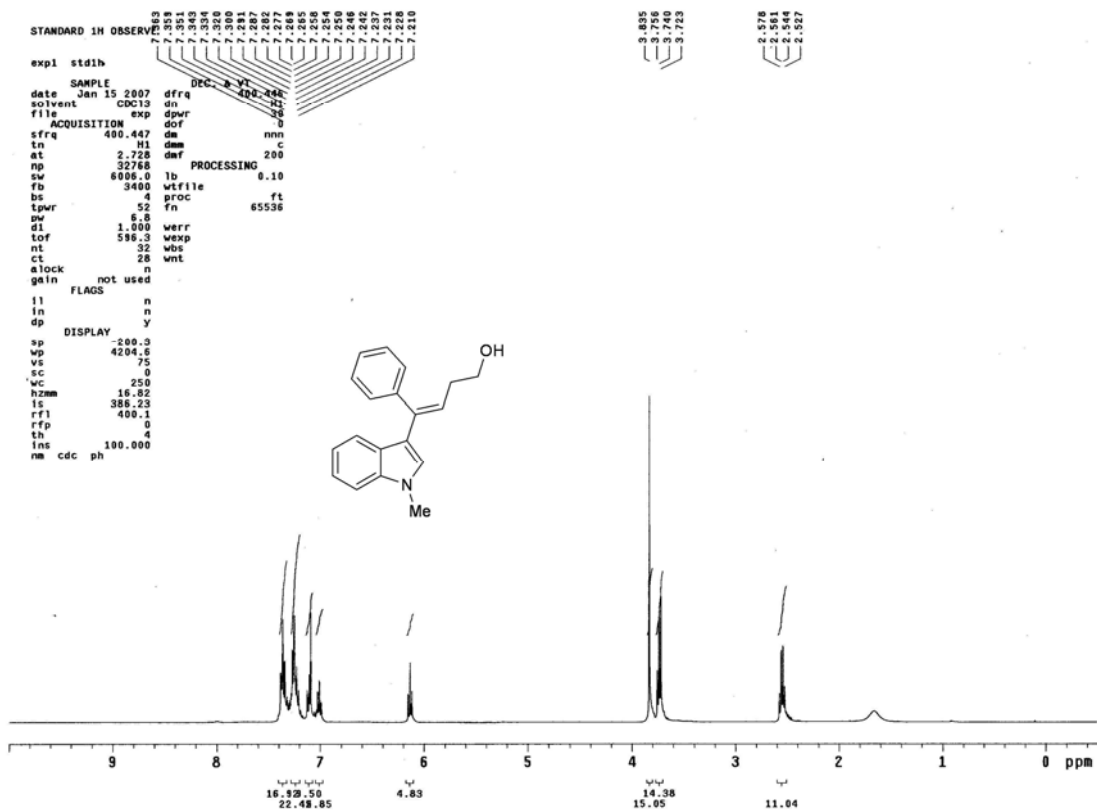


^1H and ^{13}C NMR spectra of compound **3k**.





¹H NMR spectra of E and Z form of compounds **3k'**.



STANDARD 1H OBSERVE

exp5 stdsh
SAMPLE
date dec 9 2006
solvent CDC13
file ACQUISITION
sfrq 400.447
tn 11
at 2.728
np 32768
sw 6006.0
fb 3400
tprw 58
pw 6.8
d1 1.000
tof 596.3
nt 32
ct 0
alock n
gain not used
FLAGS
l1 n
l2 n
dp y
DISPLAY
sp 200.3
wp 4204.6
ve 205
ec 0
wc 250
hzmm 16.82
ls 266.55
rfl 400.1
rfp 0
th 10
ins 100.000
nm cdc ph

DEC. & VS
dfrq 400.447
dn 30
dpr 0
dof 0
dm c
dms 200
lb 0.10
vfile ft
proc fn
warr
wexp
vbt
wnt

PROCESSING
0.10
65536

4.177
4.162
4.158
4.143
4.128
4.108

5.724

2.773
2.762
2.743
2.740
2.659
2.643
2.640
2.634
2.614
2.184
2.150
2.133
2.118
2.098
2.096

5.71 11.41
16.2615.85

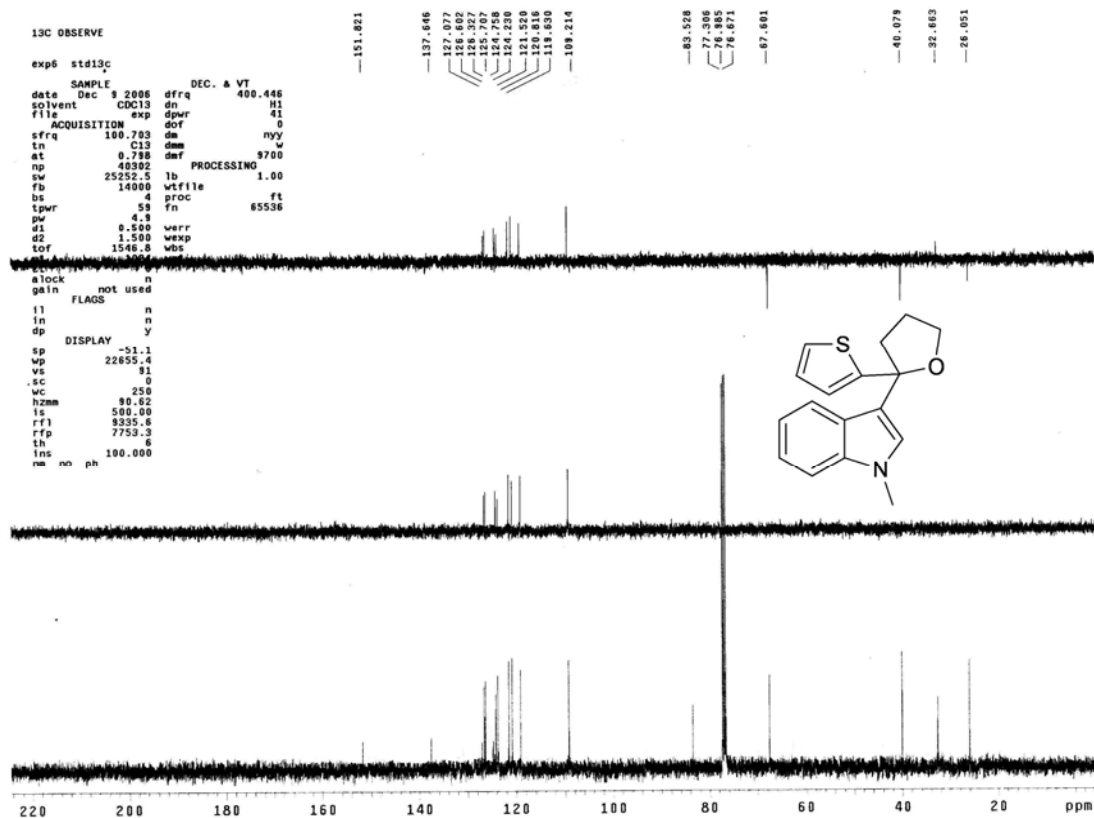
10.08
17.14

5.54
5.88

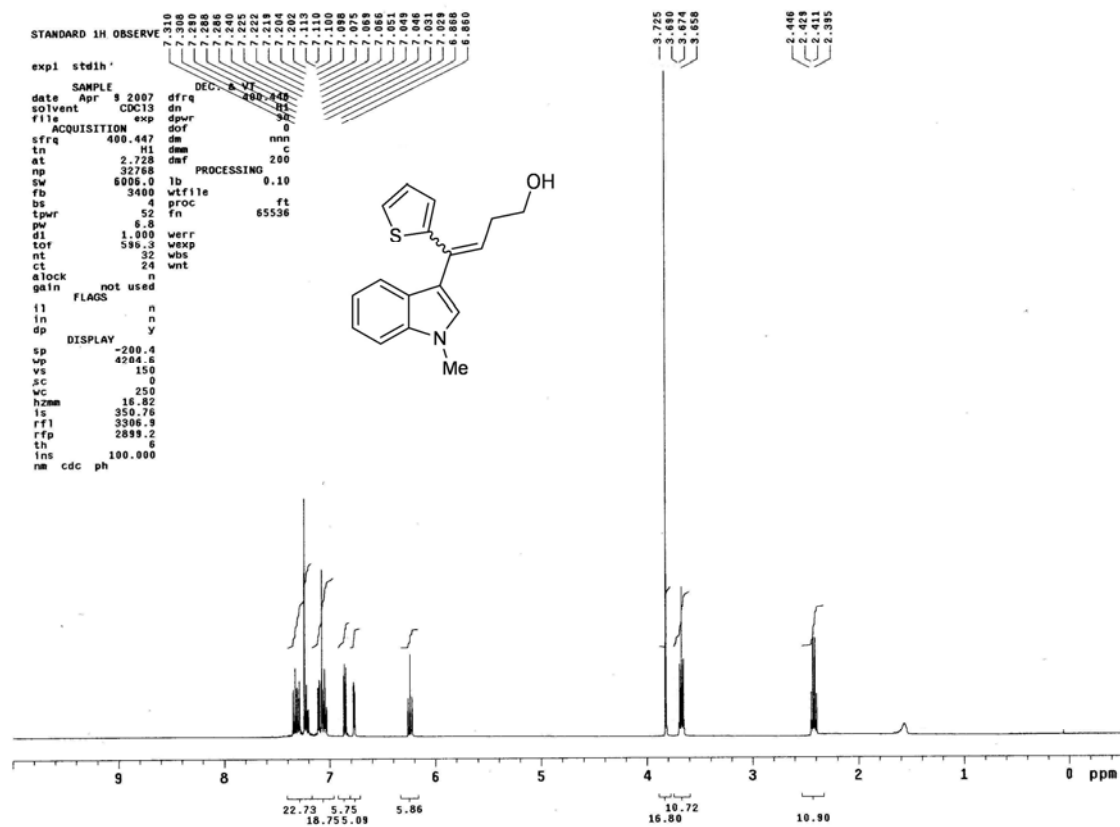
11.14

9 8 7 6 5 4 3 2 1 0 ppm

CN1C=C2C(=C1)c3ccccc3C2(C4CCO4)c5ccsc5



^1H NMR spectra of compound **31'**.



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expl szpu1

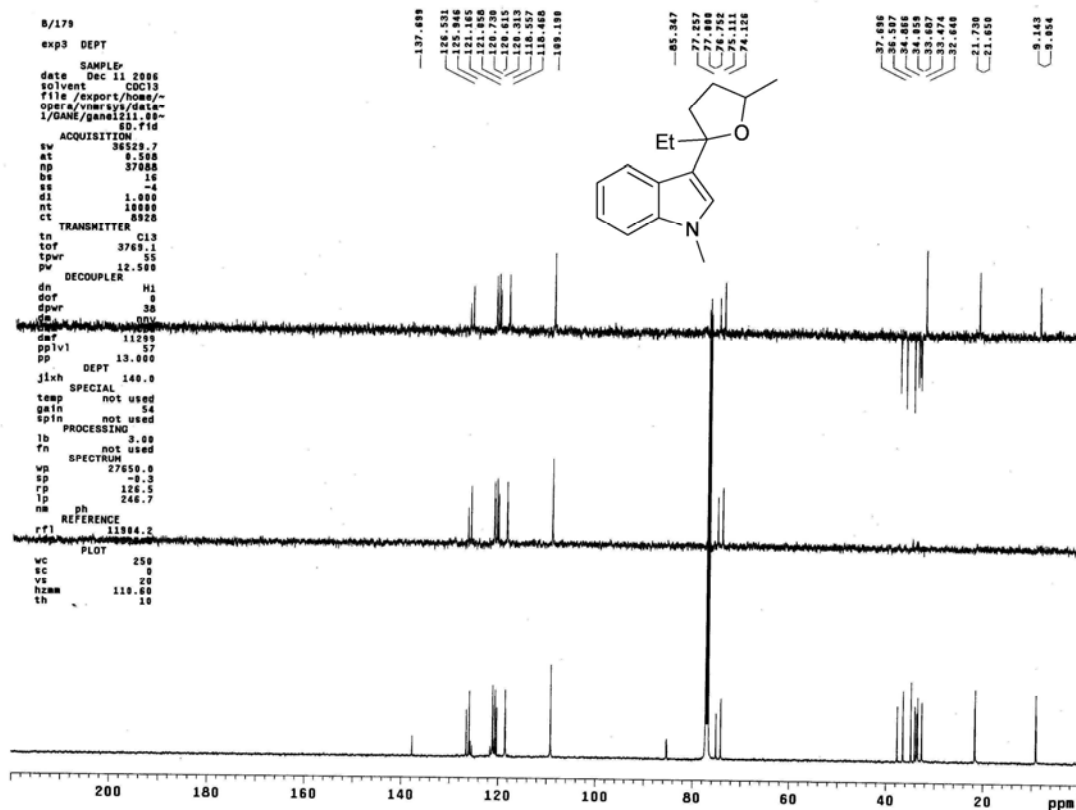
SAMPLE DEC. & VT
date Dec 11 2006 dfrq 499.832
solvent CDC13 dn
file /export/home/~ dpwr 38
opera/vmarsys/date/ dot 8
1/GANE/gane1211.00- dnm
6H.f1d dam
ACQUISITION day 11299
sfrq 499.833 dseq
tn 1n
at 2.102 homo 1.0
np 39274
sw 8889.9 vtfille
fb not used proc 32768
ls 1 fn
tpwr 55 math
pw 5.5
d1 1.000 verr
tof 980.4 wexp
nt 32 wft
ct 30 wnt
alock y
gain not used

FLAGS
f1 n
f2 n
dp y
hs nn

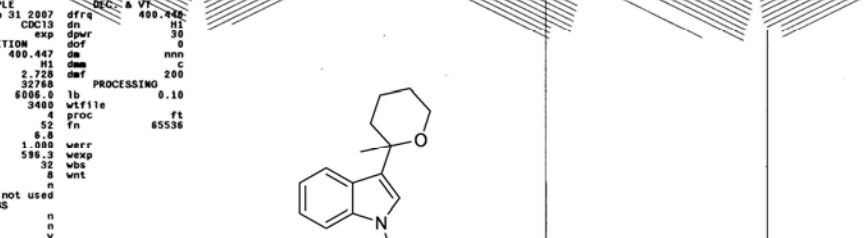
DISPLAY
sp -250.1
wp 5247.9
vc 166
sc 0
wc 250
hcam 2.50
ts 484.32
rf1 1045.1
rfp 1
th 3
ins
nm ph 100.000

Chemical structure: CC1(C)OCC1c2cnc3ccccc23

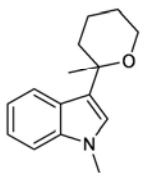
1H NMR spectrum (CDCl3) showing peaks at 7.684, 7.668, 7.652, 7.637, 7.266, 7.250, 7.234, 7.218, 7.181, 7.164, 7.089, 7.073, 7.041, 6.923, 6.902, 6.902, 4.212, 4.200, 4.184, 4.168, 4.152, 4.136, 4.120, 4.104, 4.114, 3.727, 2.331, 2.315, 2.301, 2.278, 2.262, 2.246, 2.230, 2.214, 2.197, 2.181, 2.165, 2.149, 2.133, 2.117, 2.101, 2.085, 2.069, 2.053, 2.037, 2.021, 2.005, 1.989, 1.973, 1.957, 1.941, 1.925, 1.909, 1.893, 1.877, 1.861, 1.845, 1.829, 1.813, 1.797, 1.781, 1.765, 1.749, 1.733, 1.717, 1.701, 1.685, 1.669, 1.653, 1.637, 1.621, 1.605, 1.589, 1.573, 1.557, 1.541, 1.525, 1.509, 1.493, 1.477, 1.461, 1.445, 1.429, 1.413, 1.397, 1.381, 1.365, 1.349, 1.333, 1.317, 1.301, 1.285, 1.269, 1.253, 1.237, 1.221, 1.205, 1.189, 1.173, 1.157, 1.141, 1.125, 1.109, 1.093, 1.077, 1.061, 1.045, 1.029, 1.013, 0.997, 0.981, 0.965, 0.949, 0.933, 0.917, 0.901, 0.885, 0.869, 0.853, 0.837, 0.821, 0.805, 0.789, 0.773, 0.757, 0.741, 0.725, 0.709, 0.693, 0.677, 0.661, 0.645, 0.629, 0.613, 0.597, 0.581, 0.565, 0.549, 0.533, 0.517, 0.501, 0.485, 0.469, 0.453, 0.437, 0.421, 0.405, 0.389, 0.373, 0.357, 0.341, 0.325, 0.309, 0.293, 0.277, 0.261, 0.245, 0.229, 0.213, 0.197, 0.181, 0.165, 0.149, 0.133, 0.117, 0.101, 0.085, 0.069, 0.053, 0.037, 0.021, 0.005.



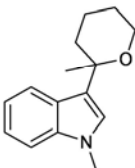
STANDARD 1H NMR

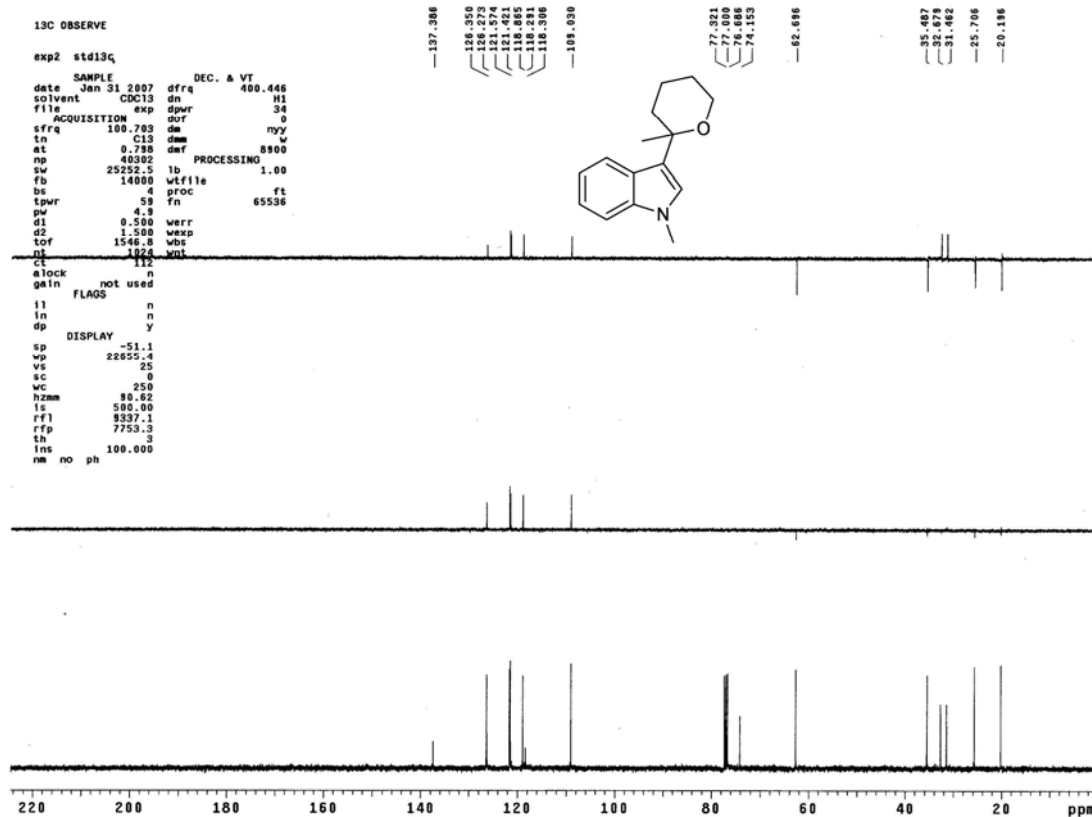
expl std1h,  2.241 1.829 1.804 1.789 1.777 1.772 1.751 1.740 1.732 1.728 1.670 1.652 1.641 1.627 1.615 1.585 1.460 1.453 3.750 3.746 3.740 3.536 3.531 3.509 3.483 3.475

SAMPLE DEC. & VT
date Jan 31 2007 dfrq 400.440
solvent CDCl3 dn H1
f114 exp dpr 30
ACQUISITION exp dof nm
sfrq 400.447 da nm
tn H1 dm C
at 2.728 daf 200
np 32768
sw 6006.0 lb 0.10
fb 3400 wfile
bs 4 proc ft
tpwr 52 fn 85536
gw 6.8
d1 1.080 warr
lof 596.3 wexp
nt 32 wbs
ct 8 wnt
elock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -200.3
wp 4504.6
vs 150
sc 9
wc 250
hzmm 16.82
ls 289.40
rf1 400.1
rf2 0
th 3
ins 100.000
nm cdc ph

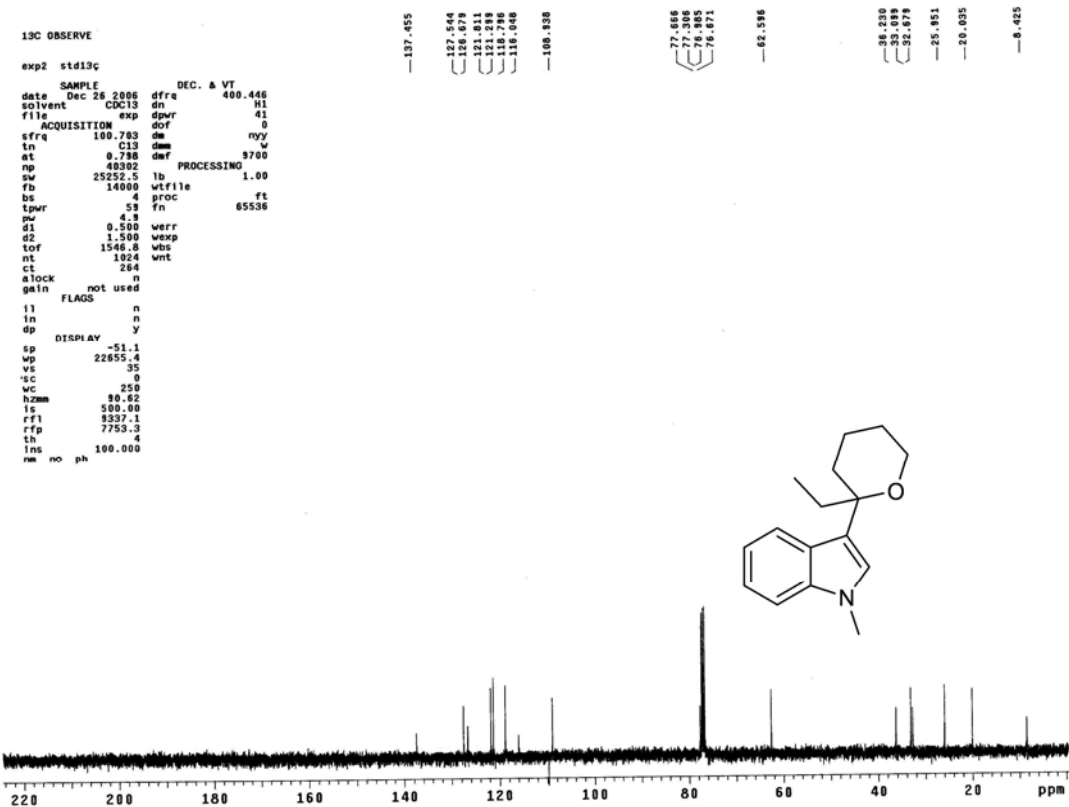
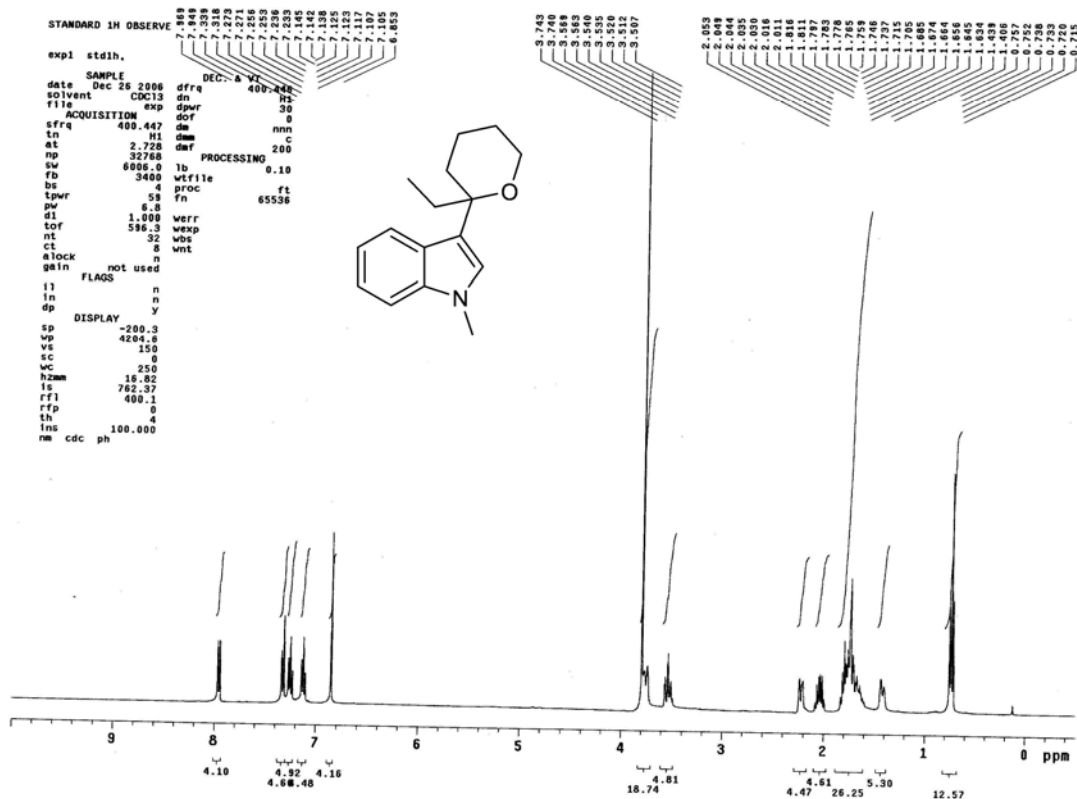


4.98 5.19 4.82 21.73 5.29 5.27 16.06 21.12 5.29





^1H and ^{13}C NMR spectra of compound 3o.



STANDARD IN OBSERVE

expl stdih

SAMPLE DEC. & VT
date Mar 15 2007 dfrq 400.448
solvent CDC13 dn H1
file exp dpr 30
ACQUISITION
tfrq 400.447 dm nmh
in H1 dam c
at 2.728 daf
np 32768 PROCESSING 200
sw 6006.0 lb 0.10
fb 3400 wfile
ss 4 proc ft
tpr 52 fn 65536
pw 6.8
d1 1.000 werr
tof 596.3 wexp
nt 32 vdx
ct 8 wnt
4lock not used
gain FLAGS
l1 n
ln n
dp y
DISPLAY
tp -200.3
vp 4204.5
vs 150
vc 0
wc 250
hzmm 16.82
ls 327.31
rf1 400.1
rfp 0
th 4
lms 100.000
rm cdc ph

7.485
7.484
7.385
7.384
7.264
7.263
7.254
7.253
7.111
7.110
7.108
7.107
6.884
6.883
6.844
6.843
3.755
3.525
3.522
3.505
3.489
2.380
2.389
2.388
1.827
1.827
1.597
1.579
1.578
1.565
1.565
1.488
1.487
1.452
1.455
1.455
1.317
1.317
1.278
1.278
1.202
1.202
1.185
1.185
1.163

OH

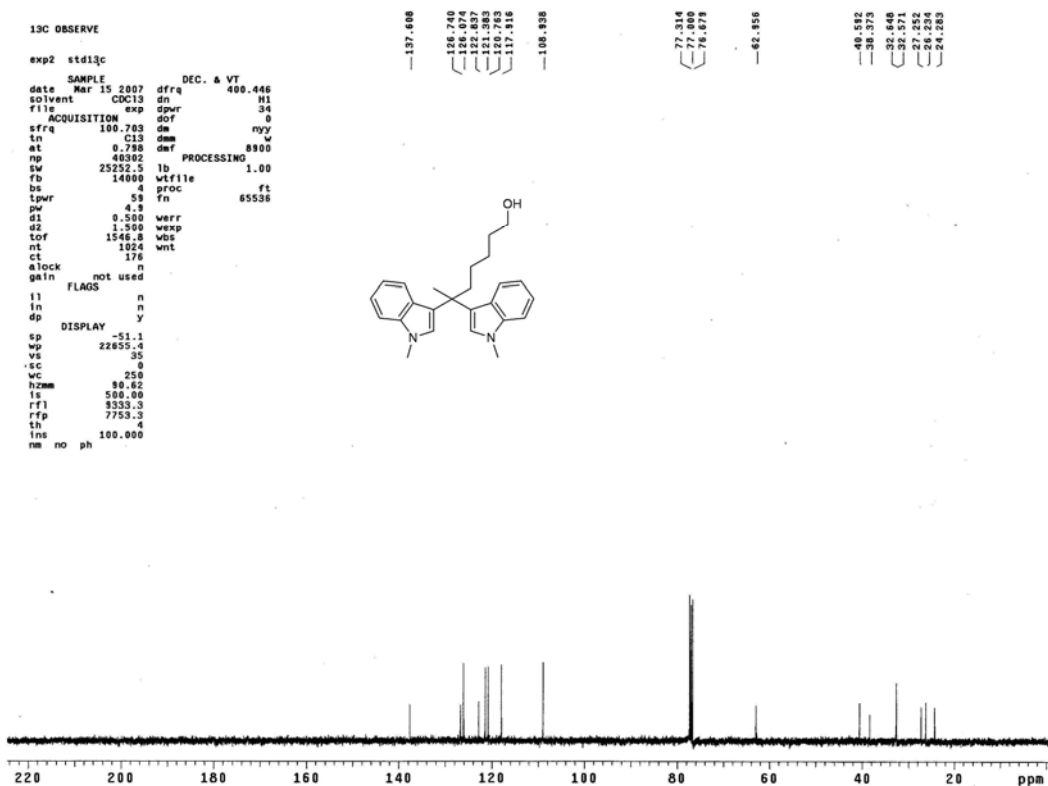
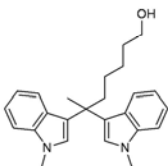
11 6.39 6.16
10 7.16 11.71
9 18.83
8 6.23
7 9.00
6 10.20
5 13.14
4
3
2
1
0 ppm

OCC1=CC=C2C(=C1)C(=C3C=CC(=C2)N3C4=CC=CC=C4)C5=CC=CC=C5

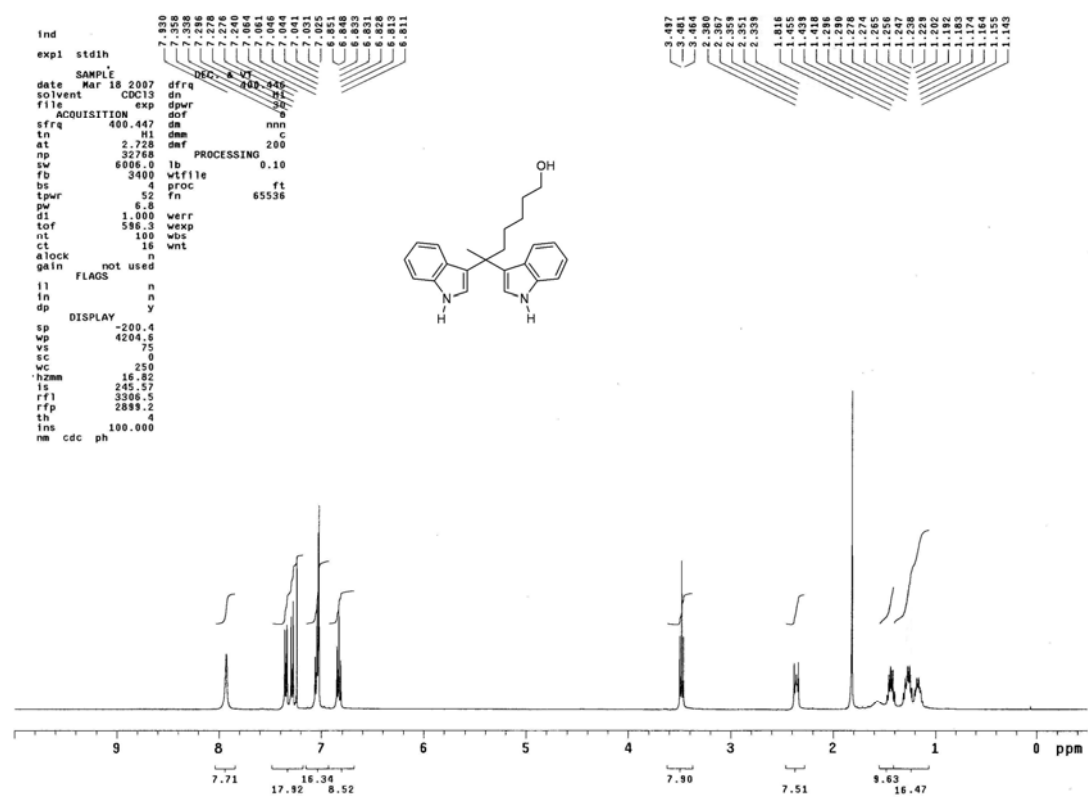
13C OBSERVE

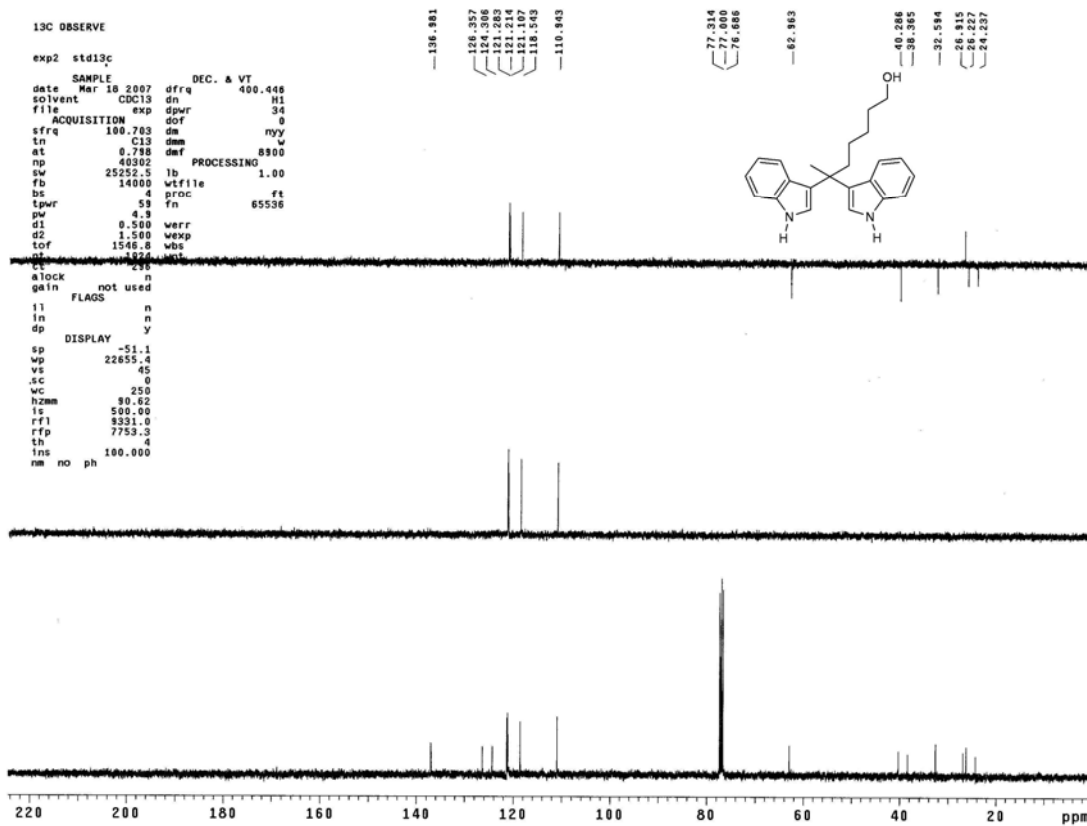
exp2 std13c

SAMPLE DEC. & VT
date Mar 15 2007 dfrq 400.446
solvent CDCl3 dn H1
file exp dpvr 34
ACQUISITION dof 0
sfrq 100.703 de myy
tn C13 dm w
at 0.788 der 8900
np 40302 PROCESSING
sw 25252.5 lb 1.00
fb 14000 wtfle
bs 4 proc ft
lpvr 59 fn 65536
pw 4.9
d1 0.500 verr
d2 1.500 wexp
tof 1546.8 vbs
nt 1024 wnt
ct 176
clock n
gain not used
FLAGS
l1 n
l2 n
dp y
DISPLAY
sp -51.1
wp 22855.4
vs 35
sc 0
wc 250
hzmm 80.62
ls 500.00
rf1 8333.3
rfp 7753.3
th 4
lms 100.000
nm no ph



^1H and ^{13}C NMR spectra of compound **3q**.



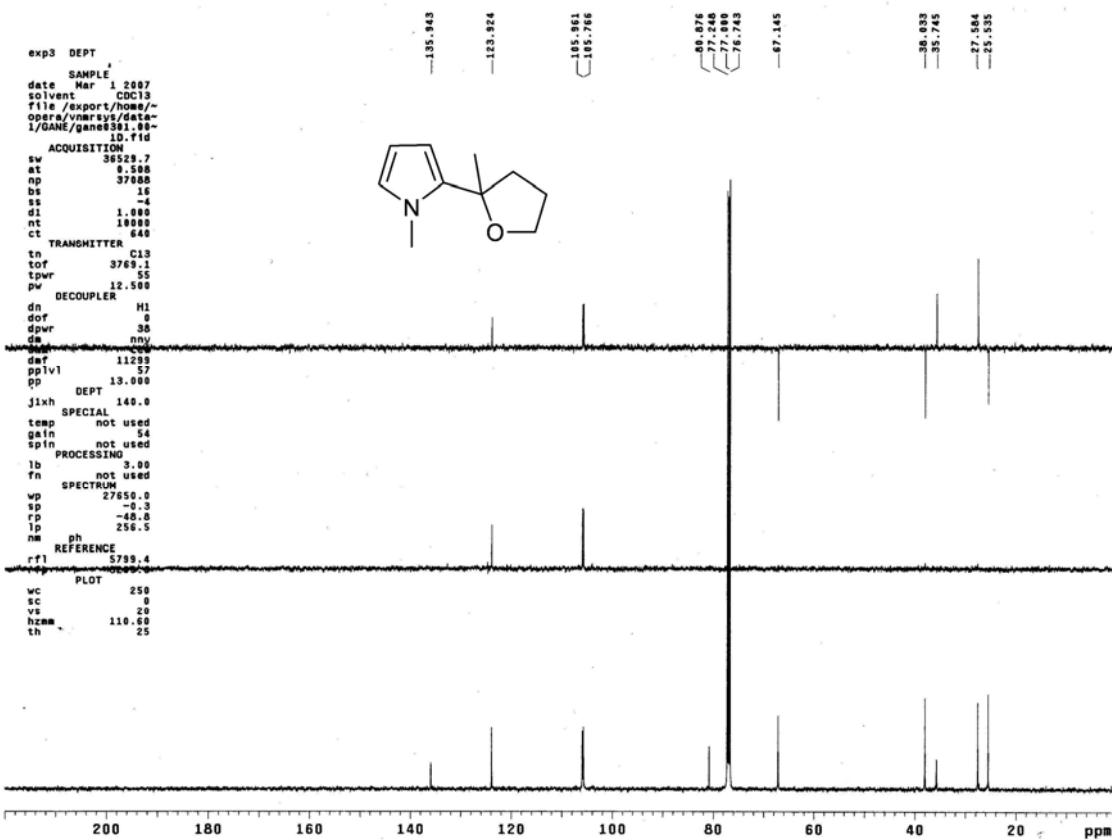
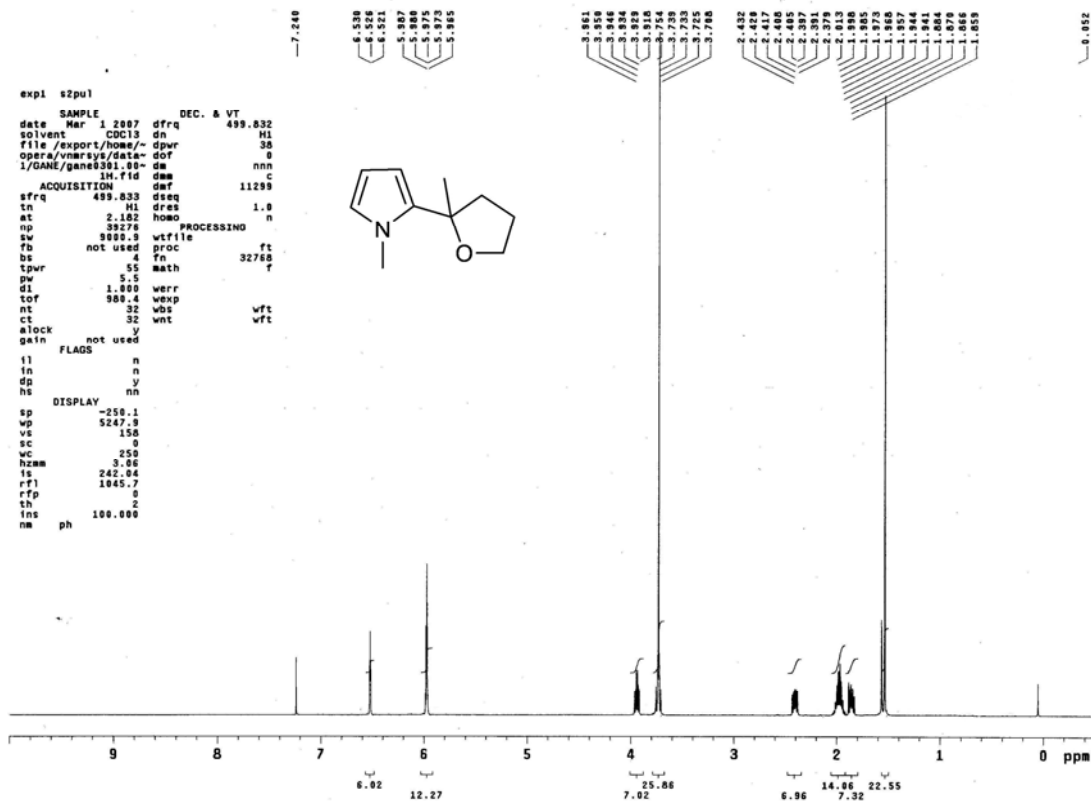
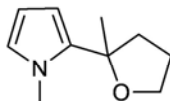


^1H and ^{13}C NMR spectra of compound **3r**.

```

exp1 szpul
SAMPLE
date Mar 1 2007 dfrq DEC. & VT 499.832
solvent CDCl3 dn H1
file /export/home/~ dpwr 38
opera/vnarsys/data- dof 0
1/GAME/gamess31.80- dm nnn
1H.fid dnm C
ACQUISITION dmf 11299
sfrq 499.833 dseq
tn H1 dres 1.0
at 2.102 homo n
np 39278 PROCESSING
sw 9800.9 wtf file ft
fb not used proc 32768 f
bs 4 fn
tpwr 5.5 math
pw 5.5
dl 1.000 verr
tof 980.4 wexp
nt 32 vbs vft
ct 32 wnt vft
aLock
gain not used
t1 FLAGS n
f1 n
dp y
hs nn
DISPLAY
sp -250.1
vp 5247.9
vs 150
sc 0
wc 250
hzmm 3.00
ts 242.04
rfl 1045.7
rfp 0
th 2
fns 100.000
nm ph

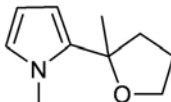
```



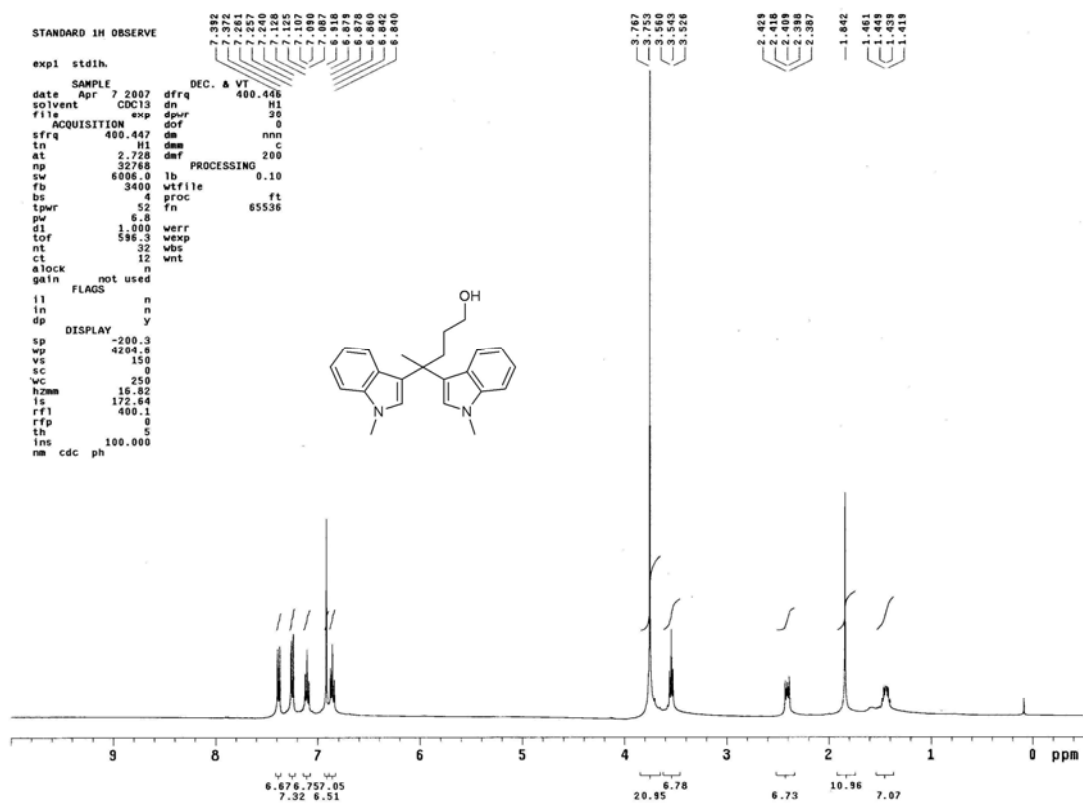
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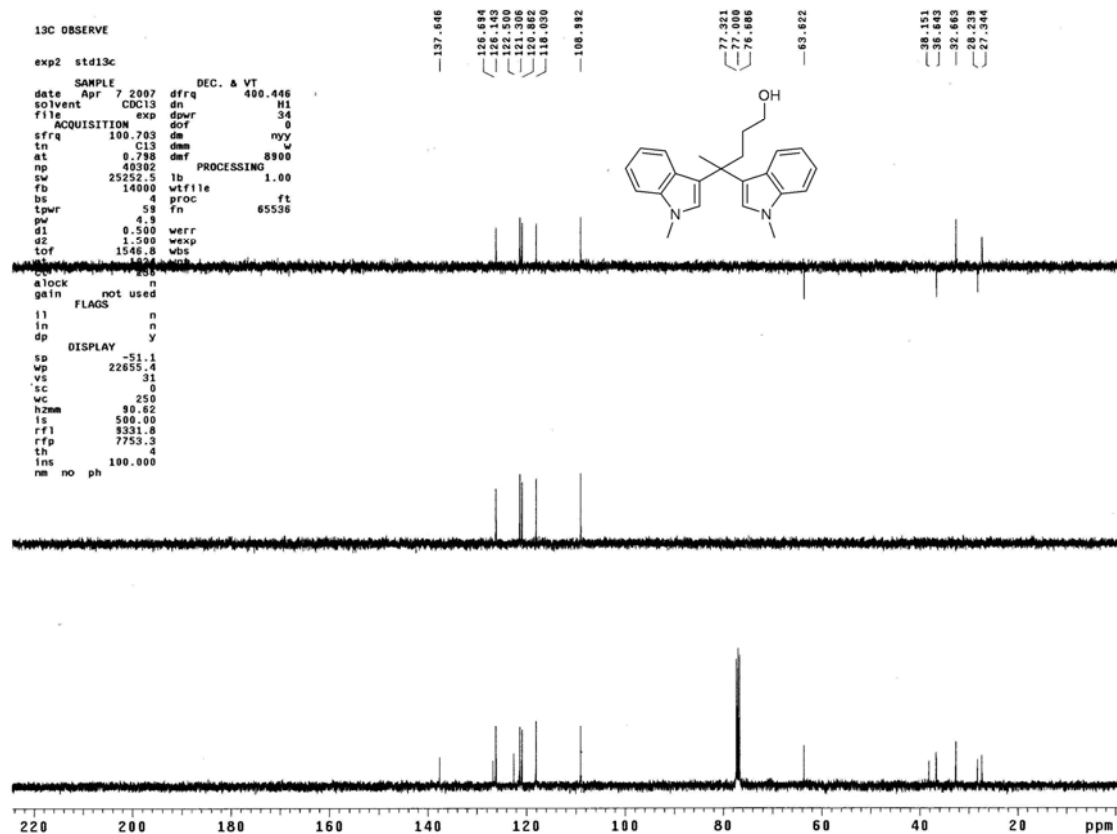
exp3 DEPT
SAMPLE
date Mar 1 2007
solvent CDCl3
file /export/home/~
opera/vnarsys/data-
1/GAME/gamess31.80-
10.fid
ACQUISITION
sw 36528.7
at 8.500
np 37088
bs 16
ss -4
dl 1.000
nt 18000
ct 640
TRANSMITTER C13
tn 3769.1
tof 55
tpwr 12.500
DECOUPLER
dn H1
dof 0
dpwr 38
dm nny
dmt 11299
pplvl 57
qp 13.000
DEPT 140.0
SPECIAL
temp not used
gain 54
spin not used
PROCESSING
lb 3.00
fn not used
SPECTRUM
wp 27650.0
sp -0.3
rp -48.8
lp 256.5
nm ph
REFERENCE
rfl 5799.4
sp 50499.5
PLOT
wc 250
sc 0
vs 20
hzmm 110.60
th 25

```

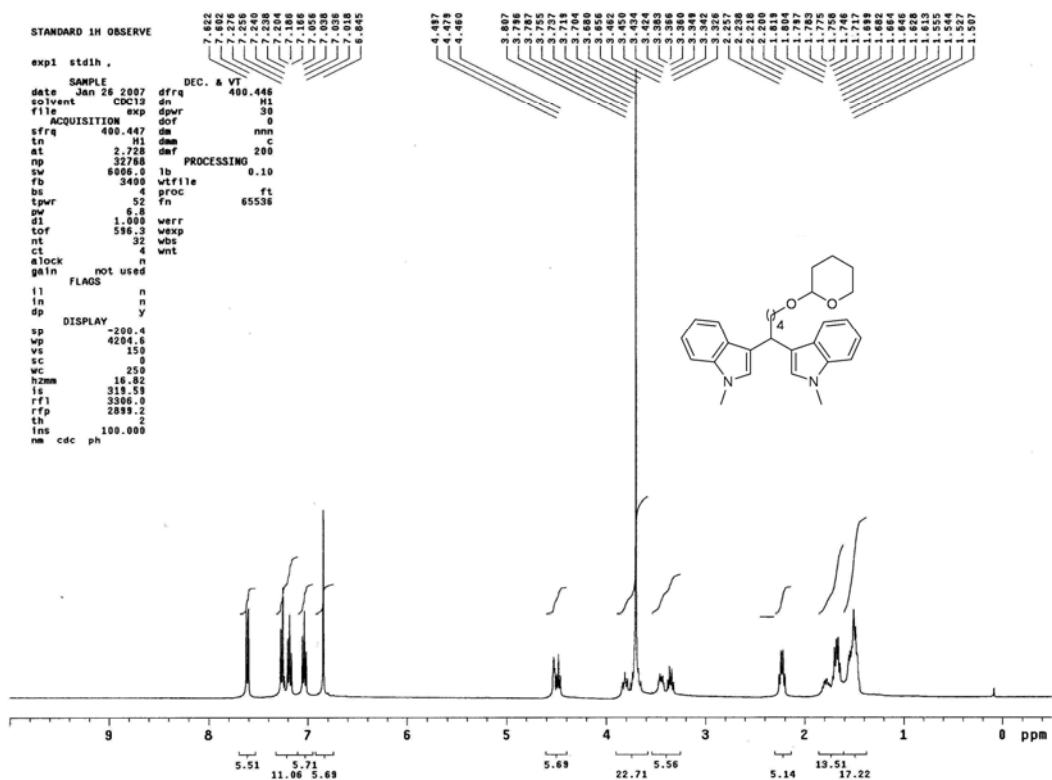


^1H and ^{13}C NMR spectra of compound **3s**.





^1H and ^{13}C NMR spectra of compound 3t.



exp5 std13c .

```

SAMPLE                                DEC. & VT
date  Jan 26 2007                    dffrq  400.446
solvent  CDCl3                       dn      H1
acq      f1a                          dof      34
sfrq     100.783                     dm      0
tn        1.38                       dme      8500
at        40392                      dmf      W
fb        25252.5                     lb      1.00
sw        14000                       wflrte   1
b         1000                       proc     85536
tprw      59                          fn
p         4
d1        0.500                      werr
d2        1.500                      wexp
d3        15.00                       wbs
d4        10.00                       wst
d5        1.00                        18
atlock    n
gain       not used
        FLAGS
ln         n
ln         n
dp         n
        DISPLAY
sp         -51.1
vc         22855.4
sc         25
wc         250
dms        90.52
rl         500.00
rf1         9334.1
rf2         7753.3
lns         3
th          100.000
no ph

```

