

Supporting Information

A base-catalyzed cycloisomerization of 5-cyano-pentyne derivatives: efficient synthesis of 3-cyano-4,5-dihydro-1*H*-pyrroles

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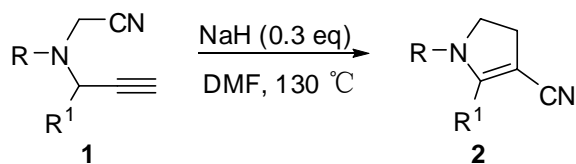
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I. General Information

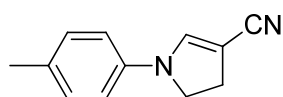
All reagents were commercial and were used without further purification. Chromatography was carried on flash silica gel (300-400 mesh). All reactions were monitored by TLC, which was performed on precoated aluminum sheets of silica gel 60 (F₂₅₄). Melting points were uncorrected. The ¹H NMR spectra were recorded at 500 MHz in CDCl₃ and the ¹³C NMR spectra were recorded at 125 MHz in CDCl₃ with TMS as internal standard. All coupling constants (*J* values) were reported in Hertz (Hz). High-resolution mass spectra (HRMS) were obtained using a Bruker microTOF II focus spectrometer (ESI). The compound **2a** was glued on a glass fiber. Data were collected at 293 K using graphite-monochromated Mo K α radiation (λ = 0.71073 Å) and IP technique in the range $2.19^\circ < \theta < 27.48^\circ$. Empirical absorption correction was applied. The structures were solved by the direct method and refined by the full-matrix least-squares method on F^2 using the SHELXS 97 crystallographic software package. Anisotropic thermal parameters were used to refine all non-hydrogen atoms. Hydrogen atoms were located from difference Fourier maps.

II. General Procedure for the Preparation of **2** from **1** (**1a** as Example):



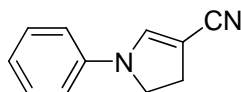
To a stirred solution of 2-(prop-2-ynyl(p-tolyl)amino)acetonitrile **1a** (0.5 mmol, 92 mg) in DMF (2.0 mL) was added NaH (NaH 60%, 0.25 mmol, 6 mg) in one portion. The reaction mixture was stirred for 7 h at 130 °C. After **1a** was consumed (monitored by TLC), the reaction mixture was poured into ice-water (20.0 mL) and extracted with CH₂Cl₂ (3×10 mL). The combined organic extracts were dried over anhydrous MgSO₄, filtered and concentrated under reduced pressure to yield the corresponding crude product, which was purified by chromatography (silica gel, petroleum ether/ethyl acetate = 10/1, V/V) to give **2a** (85 mg, 92%) as a white solid.

1-p-Tolyl-4,5-dihydro-1H-pyrrole-3-carbonitrile (**2a**):



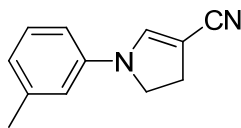
White solid; m.p. 110-112 °C. ¹H NMR (500 MHz, CDCl₃) δ: 2.29 (s, 3H), 2.97 (t, *J* = 10.0 Hz, 2H), 3.92 (t, *J* = 10.0 Hz, 2H), 6.74 (d, *J* = 8.5 Hz, 2H), 7.11 (d, *J* = 8.0 Hz, 2H), 7.40 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 20.4, 28.7, 48.9, 81.7, 113.9 (2C), 119.0, 130.0 (2C), 131.0, 138.5, 145.1; HRMS (ESI-TOF) Calcd for C₁₂H₁₃N₂⁺ ([M + H]⁺): 185.1073. Found 185.1080.

1-Phenyl-4,5-dihydro-1H-pyrrole-3-carbonitrile (**2b**):



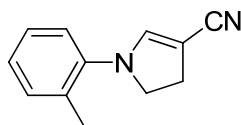
White solid; m.p. 95-97 °C. ¹H NMR (500 MHz, CDCl₃) δ: 2.98 (t, *J* = 10.0 Hz, 2H), 3.94 (t, *J* = 10.0 Hz, 2H), 6.83 (d, *J* = 8.0 Hz, 2H), 6.97 (t, *J* = 7.0 Hz, 1H), 7.31 (t, *J* = 7.5 Hz, 2H), 7.43 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 28.7, 48.7, 82.6, 113.9 (2C), 118.6, 121.4, 129.5 (2C), 140.7, 144.7; HRMS (ESI-TOF) Calcd for C₁₁H₁₁N₂⁺ ([M + H]⁺): 171.0917. Found 171.0923.

1-m-Tolyl-4,5-dihydro-1H-pyrrole-3-carbonitrile (2c):



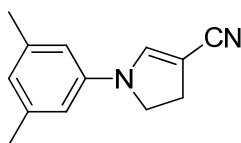
White solid; m.p. 138-140 °C. ^1H NMR (500 MHz, CDCl_3) δ : 2.34 (s, 3H), 2.98 (td, J = 10.0, 1.5 Hz, 2H), 3.93 (t, J = 10.0 Hz, 2H), 6.64 (d, J = 7.5 Hz, 2H), 6.79 (d, J = 8.0 Hz, 1H), 7.19 (t, J = 7.0 Hz, 1H), 7.44 (s, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ : 21.6, 28.7, 48.8, 82.4, 111.1, 114.7, 118.8, 122.4, 129.4, 139.6, 140.8, 144.9; HRMS (ESI-TOF) Calcd for $\text{C}_{12}\text{H}_{13}\text{N}_2^+$ ($[\text{M} + \text{H}]^+$): 185.1073. Found 185.1075.

1-o-Tolyl-4,5-dihydro-1H-pyrrole-3-carbonitrile (2d):



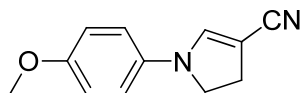
Light yellow liquid; ^1H NMR (500 MHz, CDCl_3) δ : 2.31 (s, 3H), 2.95 (td, J = 10.0, 1.5 Hz, 2H), 3.84 (t, J = 10.0 Hz, 2H), 6.97 (dd, J = 7.5, 1.0 Hz, 1H), 7.01 (s, 1H), 7.09-7.13 (m, 1H), 7.13-7.21 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 18.7, 29.3, 53.0, 80.9, 119.1, 122.5, 125.8, 127.0, 131.7, 131.9, 141.6, 151.3; HRMS (ESI-TOF) Calcd for $\text{C}_{12}\text{H}_{13}\text{N}_2^+$ ($[\text{M} + \text{H}]^+$): 185.1073. Found 185.1075.

1-(3,5-Dimethylphenyl)-4,5-dihydro-1H-pyrrole-3-carbonitrile (2e):



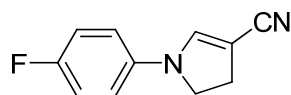
White solid; m.p. 170-172 °C. ^1H NMR (500 MHz, CDCl_3) δ : 2.29 (s, 6H), 2.95 (t, J = 10.0 Hz, 2H), 3.90 (t, J = 10.0 Hz, 2H), 6.45 (s, 2H), 6.62 (s, 1H), 7.41 (s, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ : 21.4 (2C), 28.7, 48.8, 82.0, 111.9 (2C), 118.9, 123.3, 139.3 (2C), 140.7, 145.0; HRMS (ESI-TOF) Calcd for $\text{C}_{13}\text{H}_{15}\text{N}_2^+$ ($[\text{M} + \text{H}]^+$): 199.1230. Found 199.1233.

1-(4-Methoxyphenyl)-4,5-dihydro-1H-pyrrole-3-carbonitrile (2f):



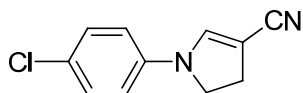
White solid; m.p. 104-106 °C. ^1H NMR (500 MHz, CDCl_3) δ : 2.96 (t, J = 10.0 Hz, 2H), 3.77 (s, 3H), 3.90 (t, J = 10.0 Hz, 2H), 6.78 (d, J = 9.0 Hz, 2H), 6.86 (d, J = 9.0 Hz, 2H), 7.33 (s, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ : 28.9, 49.5, 55.6, 81.1, 114.9 (2C), 115.5 (2C), 119.1, 134.9, 145.6, 154.8; HRMS (ESI-TOF) Calcd for $\text{C}_{12}\text{H}_{13}\text{N}_2\text{O}^+$ ($[\text{M} + \text{H}]^+$): 201.1022. Found 201.1018.

1-(4-Fluorophenyl)-4,5-dihydro-1H-pyrrole-3-carbonitrile (2g):



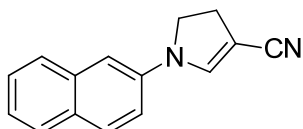
White solid; m.p. 95-97 °C. ^1H NMR (500 MHz, CDCl_3) δ : 2.99 (td, J = 9.5, 1.5 Hz, 2H), 3.92 (t, J = 10.0 Hz, 2H), 6.77-6.79 (m, 2H), 7.02 (t, J = 9.0 Hz, 2H), 7.35 (s, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ : 28.9, 49.2, 82.7, 115.2 (d, $^3J_{\text{C-F}}$ = 7.6 Hz, 2C), 116.2 (d, $^2J_{\text{C-F}}$ = 22.8 Hz, 2C), 118.6, 137.3, 145.0, 156.8 (d, $^1J_{\text{C-F}}$ = 239.8 Hz); HRMS (ESI-TOF) Calcd for $\text{C}_{11}\text{H}_{10}\text{FN}_2^+$ ($[\text{M} + \text{H}]^+$): 189.0823. Found 189.0829.

1-(4-Chlorophenyl)-4,5-dihydro-1H-pyrrole-3-carbonitrile (2h):



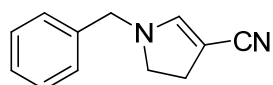
White solid; m.p. 99-101 °C. ^1H NMR (500 MHz, CDCl_3) δ : 2.99 (t, J = 10.0 Hz, 2H), 3.92 (t, J = 10.0 Hz, 2H), 6.75 (d, J = 9.0 Hz, 2H), 7.26 (d, J = 8.5 Hz, 2H), 7.37 (s, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ : 28.9, 48.9, 83.7, 115.0 (2C), 118.3, 126.4, 129.5 (2C), 139.5, 144.3; HRMS (ESI-TOF) Calcd for $\text{C}_{11}\text{H}_{10}\text{ClN}_2^+$ ($[\text{M} + \text{H}]^+$): 205.0527. Found 205.0538.

1-(Naphthalen-2-yl)-4,5-dihydro-1H-pyrrole-3-carbonitrile (2i):



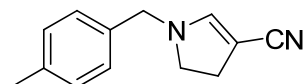
White solid; m.p. 184-186 °C. ^1H NMR (500 MHz, CDCl_3) δ : 3.01 (t, $J = 10.5$ Hz, 2H), 4.02 (t, $J = 10.5$ Hz, 2H), 7.01 (s, 1H), 7.12 (d, $J = 9.5$ Hz, 1H), 7.35 (d, $J = 7.0$ Hz, 1H), 7.43-7.47 (m, 1H), 7.54 (s, 1H), 7.69 (d, $J = 8.0$ Hz, 1H), 7.76 (t, $J = 9.0$ Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 28.7, 48.9, 83.0, 109.3, 115.0, 118.7, 124.1, 126.6, 127.0, 127.6, 128.9, 129.6, 134.1, 138.3, 144.7; HRMS (ESI-TOF) Calcd for $\text{C}_{15}\text{H}_{13}\text{N}_2^+$ ($[\text{M} + \text{H}]^+$): 221.1073. Found 221.1086.

1-Benzyl-4,5-dihydro-1H-pyrrole-3-carbonitrile (2j):



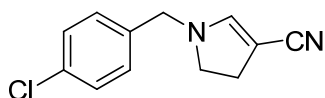
Light yellow liquid; ^1H NMR (500 MHz, CDCl_3) δ : 2.77 (t, $J = 10.0$ Hz, 2H), 3.32 (t, $J = 10.0$ Hz, 2H), 4.16 (s, 2H), 6.84 (s, 1H), 7.22 (d, $J = 7.5$ Hz, 2H), 7.31 (t, $J = 7.5$ Hz, 1H), 7.36 (t, $J = 7.5$ Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 29.3, 50.9, 54.4, 76.7, 119.7, 127.8 (2C), 127.9, 128.7 (2C), 135.8, 153.3; HRMS (ESI-TOF) Calcd for $\text{C}_{12}\text{H}_{13}\text{N}_2^+$ ($[\text{M} + \text{H}]^+$): 185.1073. Found 185.1073.

1-(4-Methylbenzyl)-4,5-dihydro-1H-pyrrole-3-carbonitrile (2k):



White solid; m.p. 86-88 °C. ^1H NMR (500 MHz, CDCl_3) δ : 2.34 (s, 3H), 2.75 (t, $J = 10.0$ Hz, 2H), 3.31 (t, $J = 10.0$ Hz, 2H), 4.11 (s, 2H), 6.82 (s, 1H), 7.10 (d, $J = 8.0$ Hz, 2H), 7.16 (d, $J = 8.0$ Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 21.0, 29.3, 50.8, 54.1, 76.7, 119.8, 127.8 (2C), 129.3 (2C), 132.7, 137.6, 153.2; HRMS (ESI-TOF) Calcd for $\text{C}_{13}\text{H}_{15}\text{N}_2^+$ ($[\text{M} + \text{H}]^+$): 199.1230. Found 199.1229.

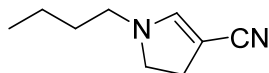
1-(4-Chlorobenzyl)-4,5-dihydro-1H-pyrrole-3-carbonitrile (2l):



White solid; m.p. 124-126 °C. ^1H NMR (500 MHz, CDCl_3) δ : 2.77 (t, $J = 10.0$ Hz, 2H), 3.30 (t, $J = 10.0$ Hz, 2H), 4.14 (s, 2H), 6.83 (s, 1H), 7.17 (d, $J = 8.0$ Hz, 2H), 7.33 (d, $J = 7.5$ Hz, 2H); ^{13}C NMR (125 MHz,

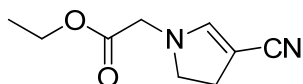
CDCl_3) δ : 29.3, 50.8, 53.7, 77.5, 119.4, 128.8 (2C), 129.1 (2C), 133.6, 134.4, 153.1; HRMS (ESI-TOF) Calcd for $\text{C}_{12}\text{H}_{12}\text{ClN}_2^+$ ($[\text{M} + \text{H}]^+$): 219.0684. Found 219.0680.

1-Butyl-4,5-dihydro-1H-pyrrole-3-carbonitrile (2m):



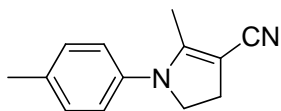
Light yellow liquid; ^1H NMR (500 MHz, CDCl_3) δ : 0.92 (t, $J = 7.0$ Hz, 3H), 1.29-1.34 (m, 2H), 1.46-1.52 (m, 2H), 2.78 (t, $J = 10.0$ Hz, 2H), 3.01 (t, $J = 7.0$ Hz, 2H), 3.40 (t, $J = 10.0$ Hz, 2H), 6.76 (s, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ : 13.6, 19.7, 29.2, 30.0, 49.9, 51.0, 75.1, 120.3, 153.6; HRMS (ESI-TOF) Calcd for $\text{C}_9\text{H}_{15}\text{N}_2^+$ ($[\text{M} + \text{H}]^+$): 151.1230. Found 151.1236.

Ethyl 2-(4-cyano-2,3-dihydro-1H-pyrrol-1-yl)acetate (2n):



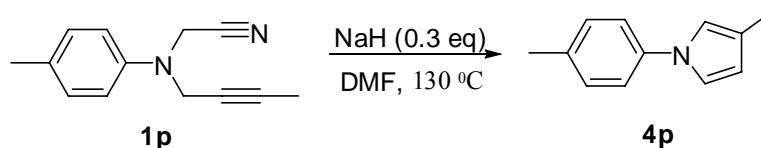
Light yellow liquid; ^1H NMR (500 MHz, CDCl_3) δ : 1.29 (t, $J = 7.0$ Hz, 3H), 2.83 (t, $J = 10.0$ Hz, 2H), 3.50 (t, $J = 10.0$ Hz, 2H), 3.78 (s, 2H), 4.21 (q, $J = 7.0$ Hz, 2H), 6.74 (s, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ : 14.1, 29.7, 51.0, 51.7, 61.4, 79.2, 119.1, 153.1, 168.9; HRMS (ESI-TOF) Calcd for $\text{C}_9\text{H}_{13}\text{N}_2\text{O}_2^+$ ($[\text{M} + \text{H}]^+$): 181.0972. Found 181.0971.

2-Methyl-1-p-tolyl-4,5-dihydro-1H-pyrrole-3-carbonitrile (2o):



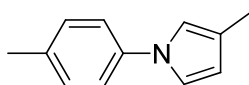
White solid; m.p. 98-100 °C. ^1H NMR (500 MHz, CDCl_3) δ : 2.00 (t, $J = 1.5$ Hz, 3H), 2.33 (s, 3H), 2.80 (td, $J = 9.5, 1.0$ Hz, 2H), 3.89 (t, $J = 9.5$ Hz, 2H), 6.93 (dd, $J = 6.5, 1.5$ Hz, 2H), 7.15 (d, $J = 8.0$ Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 13.7, 20.8, 27.3, 54.0, 79.1, 120.6, 129.1 (2C), 129.8 (2C), 134.8, 139.0, 160.6; HRMS (ESI-TOF) Calcd for $\text{C}_{13}\text{H}_{15}\text{N}_2^+$ ($[\text{M} + \text{H}]^+$): 199.1230. Found 199.1223.

III. General Procedure for the Preparation of 4p and 5p (4p as Example):



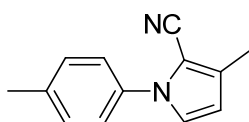
To a stirred solution of 2-(but-2-ynyl(p-tolyl)amino)acetonitrile **1p** (0.5 mmol, 99 mg) in DMF (2.0 mL) was added NaH (NaH 60%, 0.15 mmol, 6 mg) in one portion. The reaction mixture was stirred for 7 h at 130 °C. After **1p** was consumed (monitored by TLC), the reaction mixture was poured into ice-water (20.0 mL) and extracted with CH₂Cl₂ (3×10 mL). The combined organic extracts were dried over anhydrous MgSO₄, filtered and concentrated under reduced pressure to yield the corresponding crude product, which was purified by chromatography (silica gel, petroleum ether/ethyl acetate = 12/1, V/V) to give **4p** (74 mg, 86%) as a white solid.

3-Methyl-1-p-tolyl-1H-pyrrole (4p):



¹H NMR (500 MHz, CDCl₃) δ : 2.16 (s, 3H), 2.35 (s, 3H), 6.16 (t, J = 2.5 Hz, 1H), 6.83 (d, J = 1.0 Hz, 1H), 6.95 (t, J = 2.5 Hz, 1H), 7.18 (d, J = 8.5 Hz, 2H), 7.22-7.24 (m, 2H); ¹³C NMR (125 MHz, CDCl₃) δ : 11.9, 20.7, 111.5, 117.1, 118.9, 119.9 (2C), 120.7, 129.9 (2C), 134.8, 138.4; HRMS (ESI-TOF) Calcd for C₁₂H₁₄N⁺ ([M + H]⁺): 172.1121. Found 172.1131.

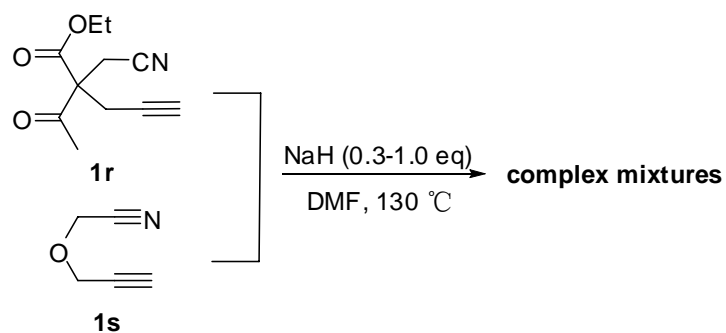
3-Methyl-1-p-tolyl-1H-pyrrole-2-carbonitrile (5p):



¹H NMR (500 MHz, CDCl₃) δ : 2.30 (s, 3H), 2.39 (s, 3H), 6.16 (d, J = 2.5 Hz, 1H), 6.93 (d, J = 2.5 Hz, 1H), 7.26 (d, J = 8.0 Hz, 2H), 7.30 (d, J = 8.5 Hz, 2H); ¹³C NMR (125 MHz, CDCl₃) δ : 11.9, 20.9, 103.0, 111.4, 113.9, 123.5 (2C), 126.3, 130.1 (2C), 133.9, 136.0, 137.9; HRMS (ESI-TOF) Calcd for C₁₃H₁₃N₂⁺ ([M + H]⁺): 197.1073. Found 197.1065.

IV. Results of Cyclization Reactions of **1r** or **1s**:

In addition, the cyclization reaction of ethyl 2-acetyl-2-(cyanomethyl)pent-4-ynoate **1r** or 2-(prop-2-ynyloxy)acetonitrile **1s** was attempted. However, when the substrate **1r** or **1s** was treated with NaH (0.3-1.0 equiv) in DMF at 130 °C for 5-10 h, a complex mixture was produced, in which no desired product could be isolated.



V. Crystal data and ORTEP drawing of compound 2a:

Crystal data for **2a**: C₁₂H₁₂N₂, white crystal, $M = 184.24$, Monoclinic, P2(1)/c, $a = 7.7787(10)$ Å, $b = 8.3955(11)$ Å, $c = 15.691(2)$ Å, $\alpha = 90.00^\circ$, $\beta = 93.859(2)^\circ$, $\gamma = 90.00^\circ$, $V = 1022.4(2)$ Å³, $Z = 4$, $T = 293(2)$, $F_{000} = 392$, $R_1 = 0.0523$, $wR_2 = 0.1252$. CCDC 994058.

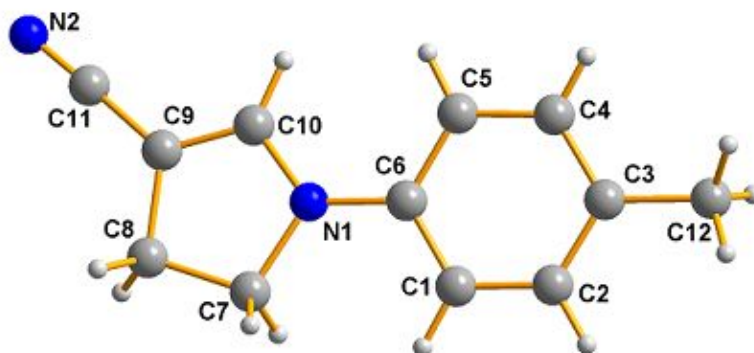


Figure 1 ORTEP drawing of **2a**

VI. ^1H and ^{13}C NMR spectra of substrates and products 2, 4 and 5:

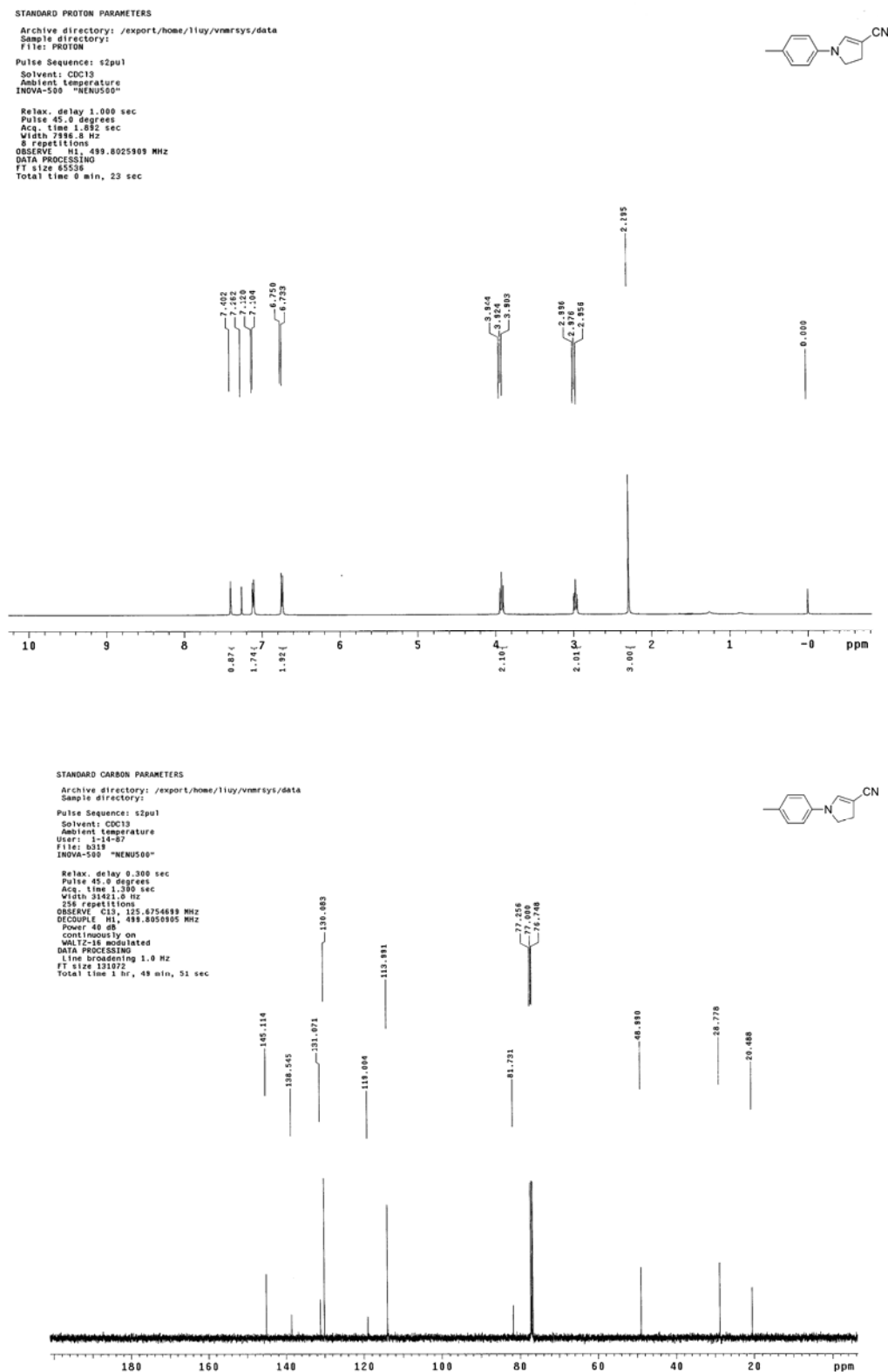
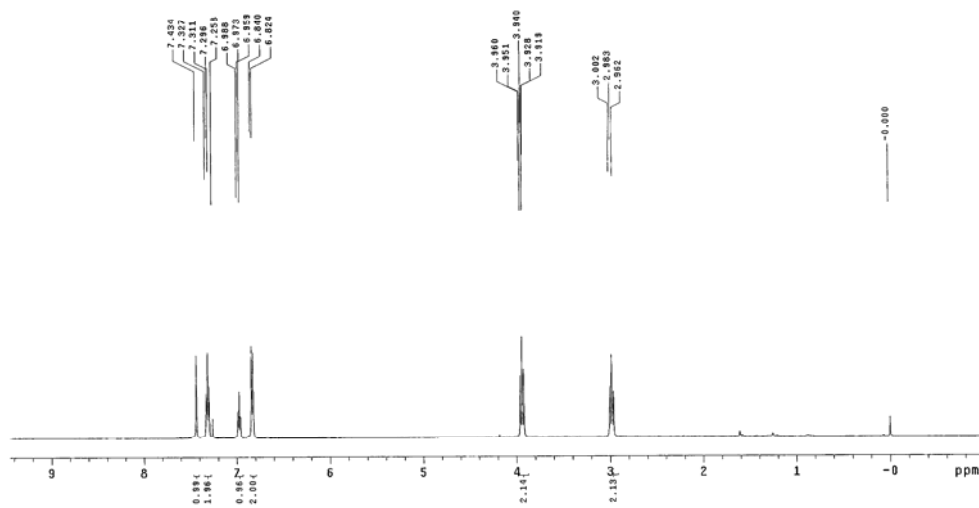
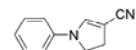


Figure 1. ^1H - (upper) and ^{13}C -NMR (lower) spectra of compound 2a.

STANDARD PROTON PARAMETERS
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 Sample directory:
 Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 File: h036
 INOVA-500 "NENU500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.892 sec
 Width 9102.3 Hz
 0 repetitions
 OBSERVE H1, 499.8025926 MHz
 DATA PROCESSING
 FT size 45536
 Total time 0 min, 23 sec



STANDARD CARBON PARAMETERS
 Archive directory: /export/home/ouyy/vnmr/sys/data
 Sample directory:
 Pulse Sequence: s2pul
 Solvent: cdcl3
 Ambient temperature
 User: j-14-87
 File: h037
 INOVA-500 "NENU500"
 Relax. delay 0.500 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 51401.8 Hz
 64 repetitions
 OBSERVE C13, 125.6754747 MHz
 DECOUPLE H1, 499.8050905 MHz
 Power 42 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.5 Hz
 FT size 131072
 Total time 3 hr, 56 sec

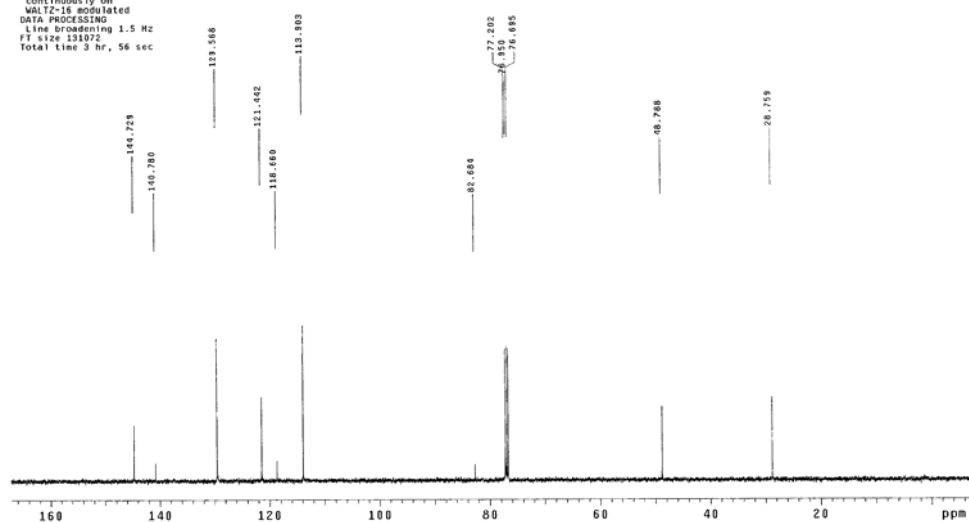
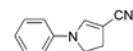
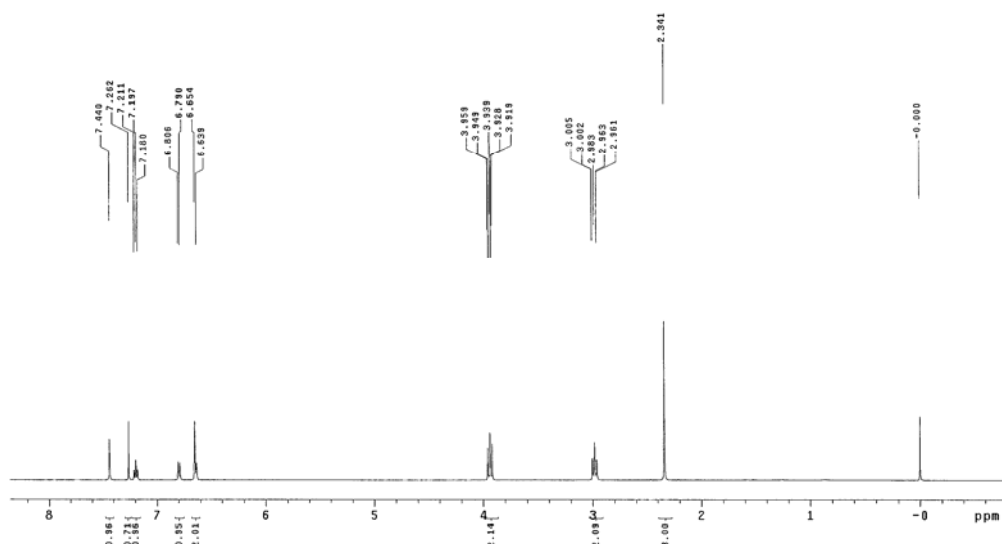
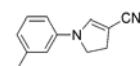


Figure 2. ^1H - (upper) and ^{13}C -NMR (lower) spectra of compound **2b**.

STANDARD PROTON PARAMETERS

Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: m662
INOVA-500 "NMR500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 3351.3 Hz
8 repetitions
OBSERVE H1, 499.8025918 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec



STANDARD CARBON PARAMETERS

Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: m671
INOVA-500 "NMR500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
360 repetitions
OBSERVE C13, 125.6754656 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 12 hr, 33 min, 54 sec

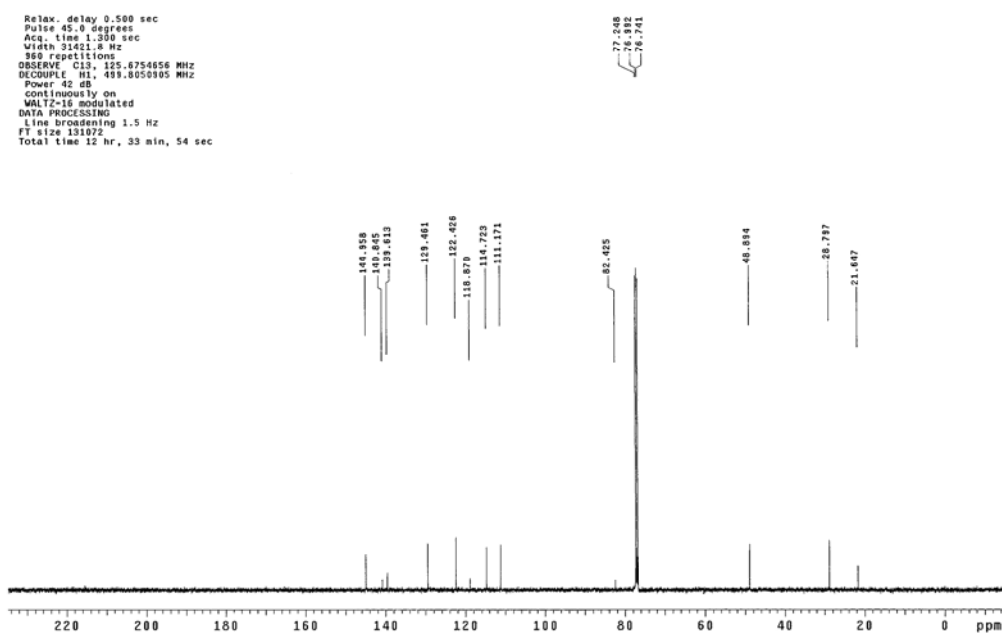
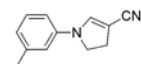
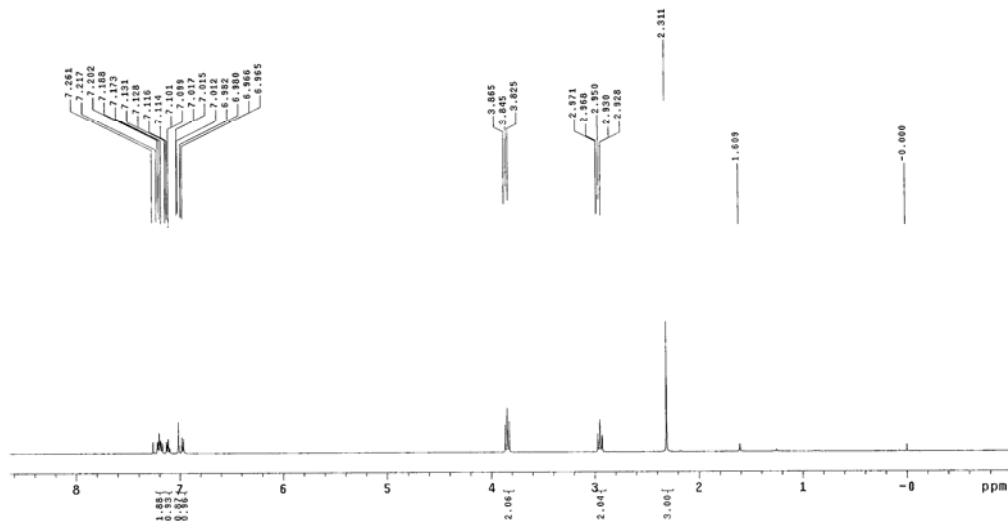
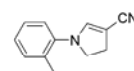


Figure 3. ¹H- (upper) and ¹³C-NMR (lower) spectra of compound 2c.

STANDARD PROTON PARAMETERS

Archive directory: /export/home/ouyy/vnmr/sys/data
 Sample directory:
 Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 File: n499
 INOVA-500 "NMR500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.852 sec
 Width 8331.6 Hz
 8 repetitions
 OBSERVE H1, 499.8025924 MHz
 DATA PROCESSING
 FT size 65536
 Total time 0 min, 23 sec



STANDARD CARBON PARAMETERS

Archive directory: /export/home/ouyy/vnmr/sys/data
 Sample directory:
 Pulse Sequence: s2pul
 Solvent: cdcl3
 Ambient temperature
 User: 1-14-87
 File: n491
 INOVA-500 "NMR500"
 Relax. delay 0.500 sec
 Pulse 45.0 degrees
 Acq. time 1.350 sec
 Width 31421.8 Hz
 64 repetitions
 OBSERVE C13, 125.6754699 MHz
 DECOUPLE H1, 499.8050905 MHz
 Power 42 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.5 Hz
 FT size 131072
 Total time 3 hr, 56 sec

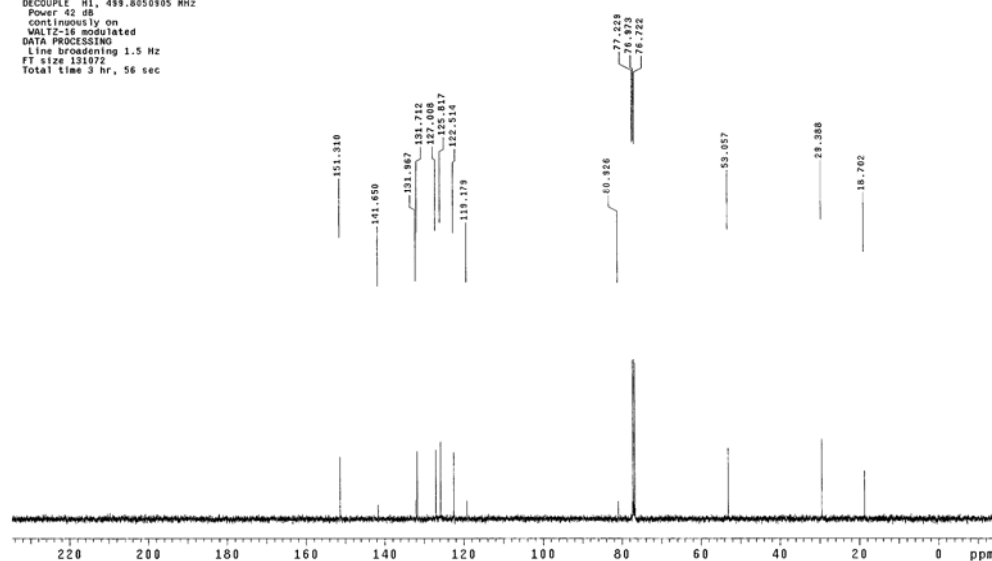
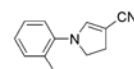


Figure 4. ¹H- (upper) and ¹³C-NMR (lower) spectra of compound **2d**.

STANDARD PROTON PARAMETERS

Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: 0719

INNOVA-500 "HENU500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.432 sec

Width 8508.7 Hz

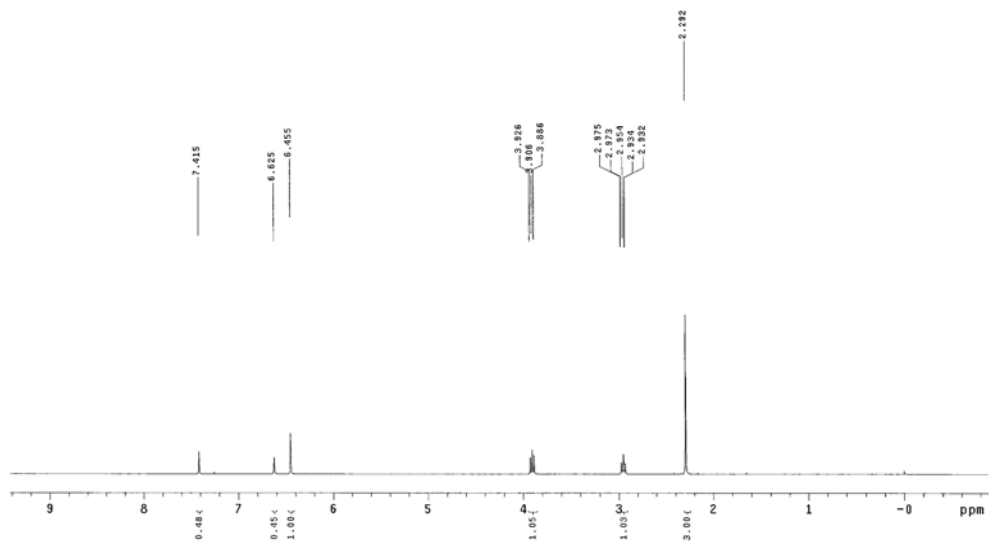
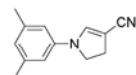
8 repetitions

OBSERVE H1, 499.8025938 MHz

DATA PROCESSING

FT size 65536

Total time 0 min, 23 sec



STANDARD CARBON PARAMETERS

Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul

Solvents: cdcl3

Ambient temperature

User: 1-14-87

File: 0719

INNOVA-500 "HENU500"

Relax. delay 0.500 sec

Pulse 45.0 degrees

Acq. time 1.390 sec

Width 31421.8 Hz

128 repetitions

OBSERVE C13, 125.6754728 MHz

DECOUPLE H1, 499.8050905 MHz

Power 42 dB

continuously on

WALTZ-16 modulated

Line broadening 1.5 Hz

FT size 131072

Total time 3 hr, 56 sec

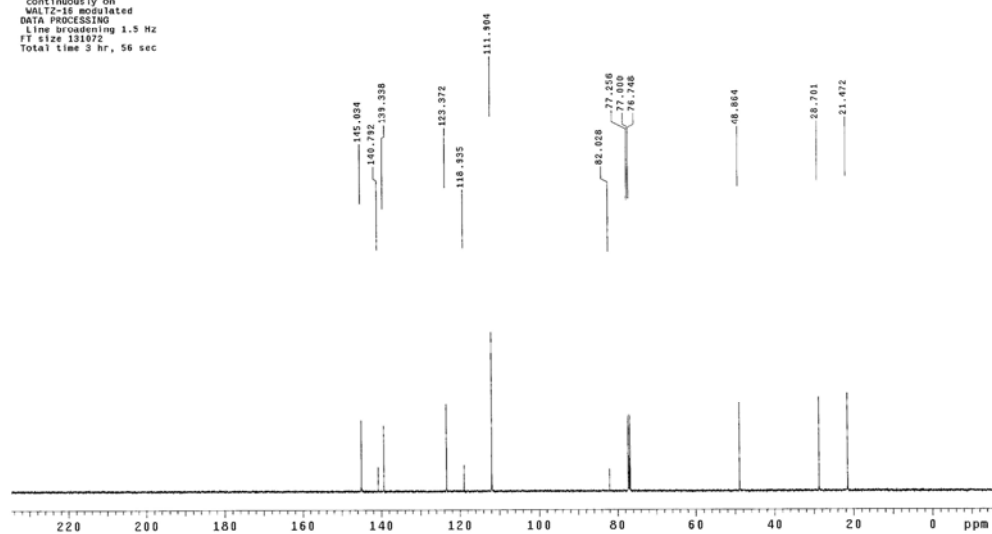
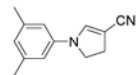
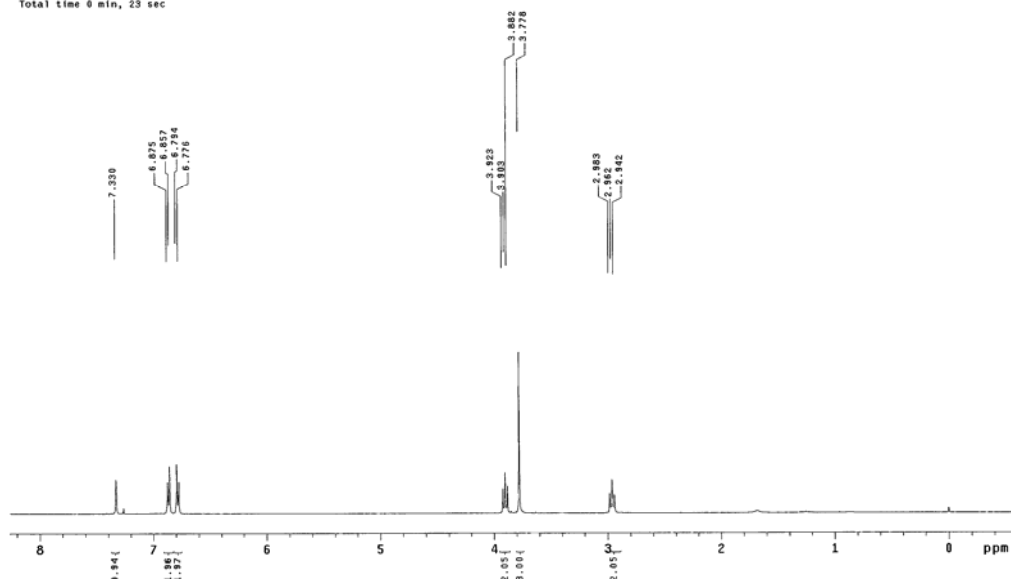
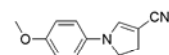


Figure 5. ¹H- (upper) and ¹³C-NMR (lower) spectra of compound **2e**.

STANDARD PROTON PARAMETERS

Archive directory: /export/home/ouyy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pul
 Solvent: CDCl₃
 Ambient temperature
 File: 1237
 INOVA-500 "NENU500"

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.882 sec
 Width 3177.5 Hz
 8 repetitions
 OBSERVE H1 499.8025892 MHz
 DATA PROCESSING
 FT size 85536
 Total time 8 min, 23 sec



STANDARD CARBON PARAMETERS

Archive directory: /export/home/ouyy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pul
 Solvent: cdcl3
 Ambient temperature
 User: 1-14-97
 File: 1238
 INOVA-500 "NENU500"

Relax. delay 0.500 sec
 Pulse 45.0 degrees
 Acq. time 1.380 sec
 Width 31421.8 Hz
 64 repetitions
 OBSERVE C13 125.6754603 MHz
 DECOUPLE H1 499.8050905 MHz
 Power 42 dB
 Continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.5 Hz
 FT size 131072
 Total time 2 hr, 3 min, 31 sec

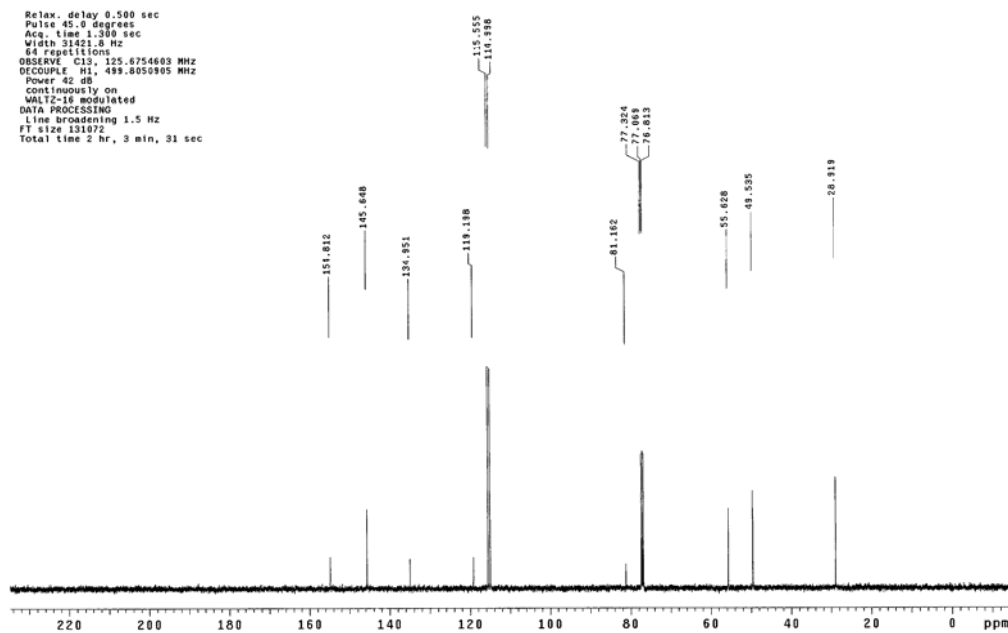
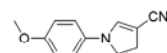
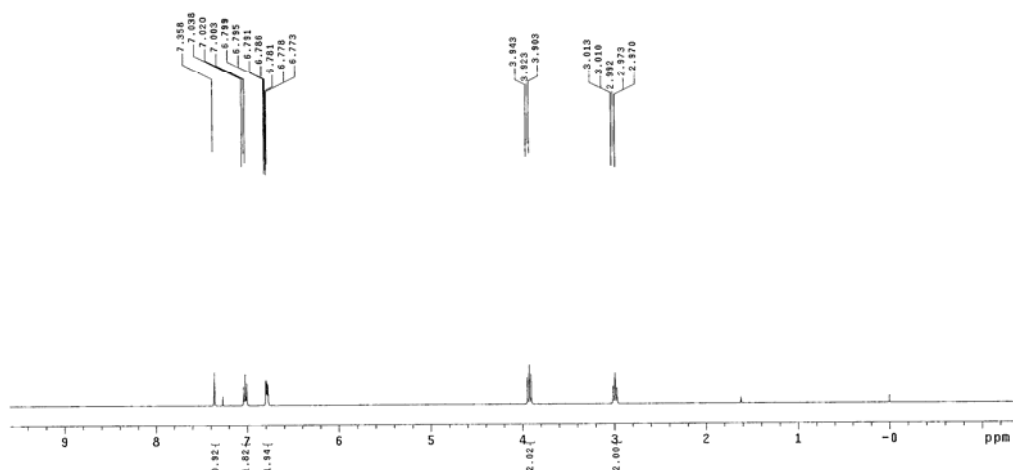
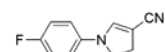


Figure 6. ¹H- (upper) and ¹³C-NMR (lower) spectra of compound **2f**.

STANDARD PROTON PARAMETERS

Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
File: n492
INOVA-500 "MENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8931.6 Hz
0 repetitions
OBSERVE H1, 499.8025897 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec



STANDARD CARBON PARAMETERS

Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pu1
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: n493
INOVA-500 "MENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.308 sec
Width 31421.8 Hz
64 repetitions
OBSERVE C13, 125.6754699 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec

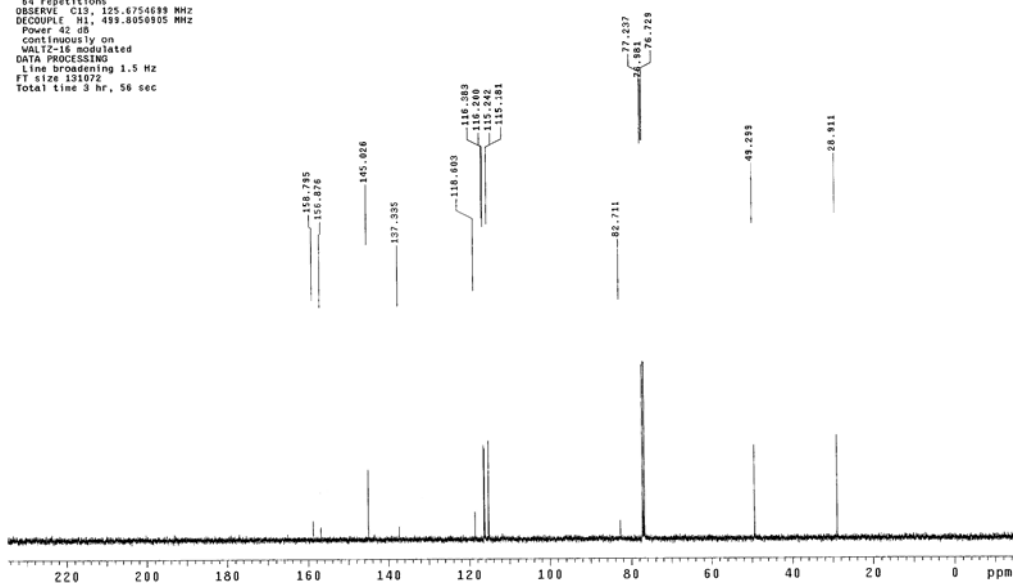
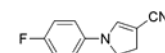


Figure 7. ¹H- (upper) and ¹³C-NMR (lower) spectra of compound **2g**.

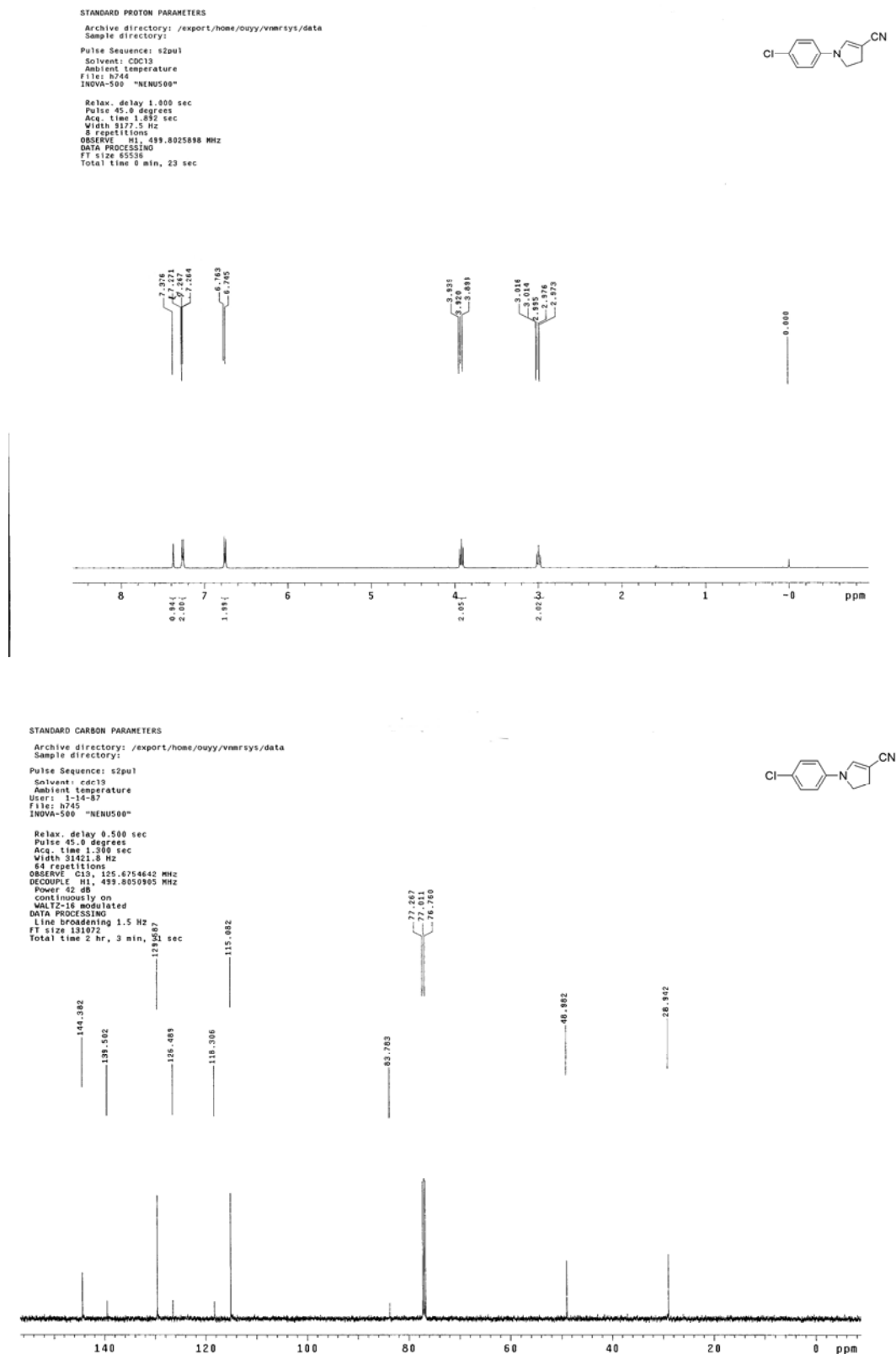
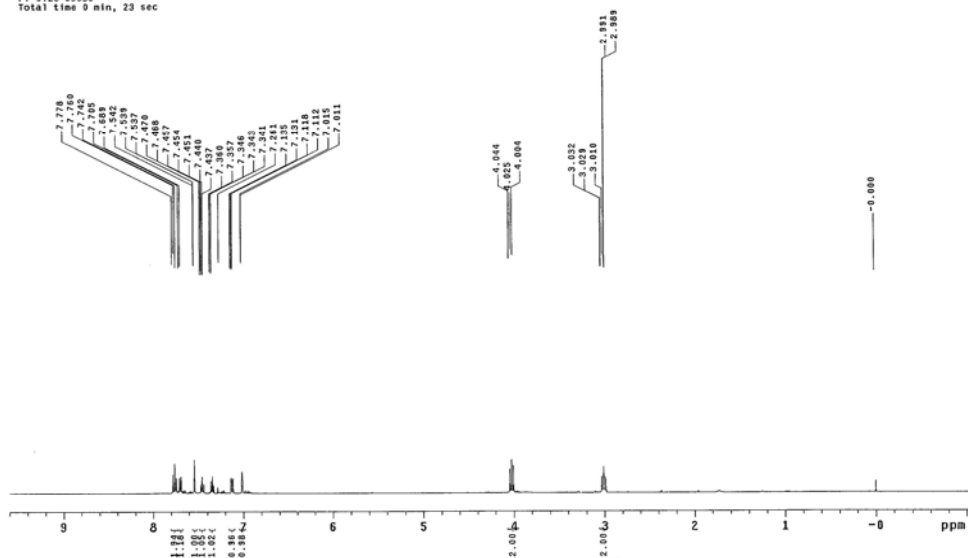


Figure 8. ¹H- (upper) and ¹³C-NMR (lower) spectra of compound **2h**.

STANDARD PROTON PARAMETERS

Archive directory: /export/home/ouyy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pul
 Solvent: CDCl₃
 Ambient temperature
 File: q728
 INOVA-500 "NMR500"

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.532 sec
 Width 8500.7 Hz
 8 repetitions
 OBSERVE H1, 499.8025914 MHz
 DATA PROCESSING
 FT size 65536
 Total time 0 min, 23 sec



STANDARD CARBON PARAMETERS

Archive directory: /export/home/ouyy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pul
 Solvent: cdcl3
 Ambient temperature
 User: j-10-87
 File: q728
 INOVA-500 "NMR500"

Relax. delay 0.500 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 31421.8 Hz
 135 repetitions
 OBSERVE C13, 125.6754704 MHz
 DECOUPLE H1, 499.8050905 MHz
 Power 42 dB
 Continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.5 Hz
 FT size 131072
 Total time 2 hr, 3 min, 31 sec

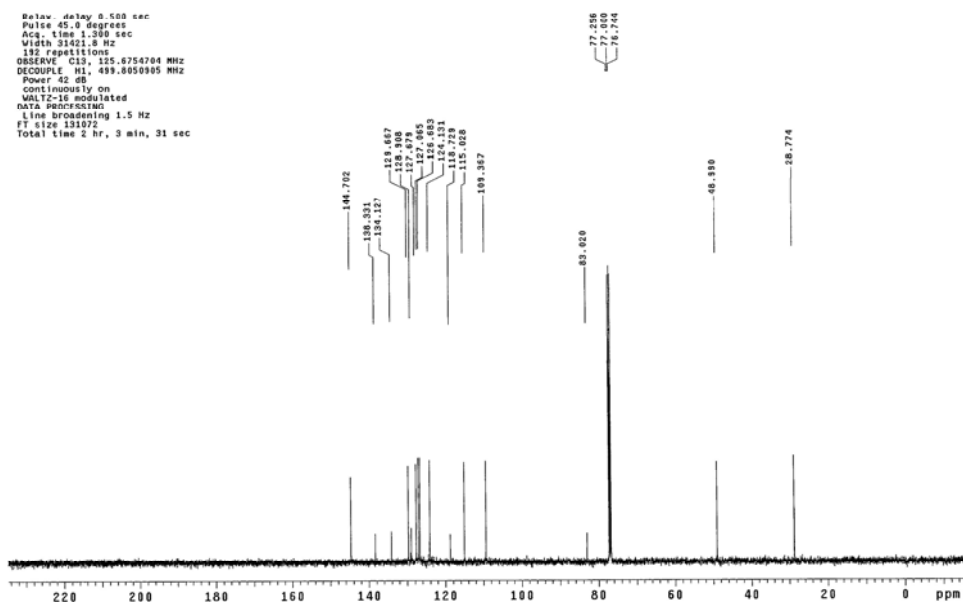
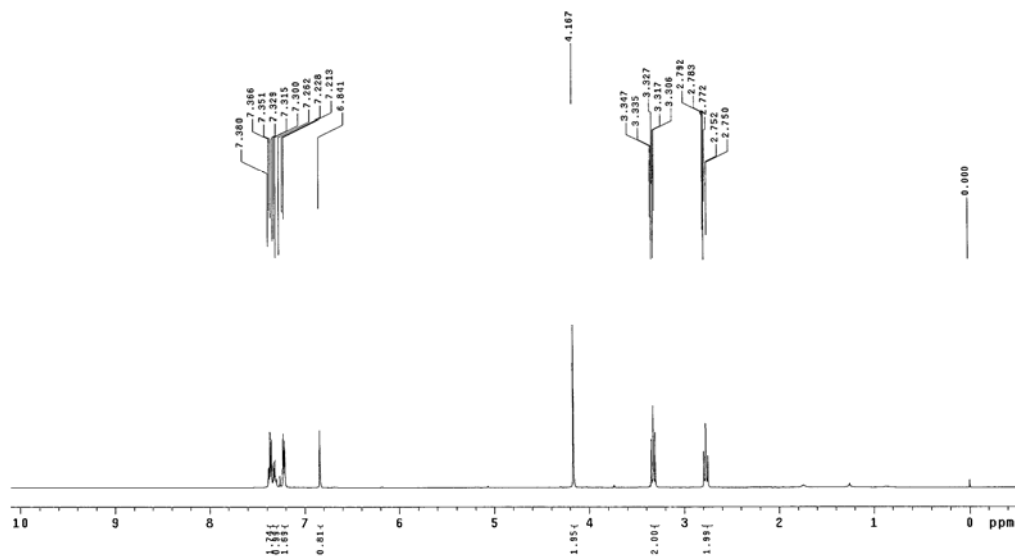
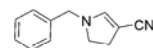


Figure 9. ¹H- (upper) and ¹³C-NMR (lower) spectra of compound **2i**.

STANDARD PROTON PARAMETERS

Archive directory: /export/home/liuy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pu1
 Solvent: CDCl₃
 Ambient temperature
 File: y177
 INOVA-500 "MENU500"

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.892 sec
 Width 7986.8 Hz
 8 repetitions
 OBSERVE H1: 499.8025907 MHz
 DATA PROCESSING
 FT size 65536
 Total time 0 min, 23 sec



STANDARD CARBON PARAMETERS

Archive directory: /export/home/liuy/vnmrsys/data
 Sample directory:
 File: CARBON
 Pulse Sequence: s2pu1
 Solvent: CDCl₃
 Ambient temperature
 User: l-14-57
 INOVA-500 "MENU500"

Relax. delay 0.300 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 31421.8 Hz
 256 repetitions
 OBSERVE C13: 125.6754742 MHz
 DECOUPLE H1: 499.8050905 MHz
 Power 60 dB
 Continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 131072
 Total time 1 hr, 49 min, 50 sec

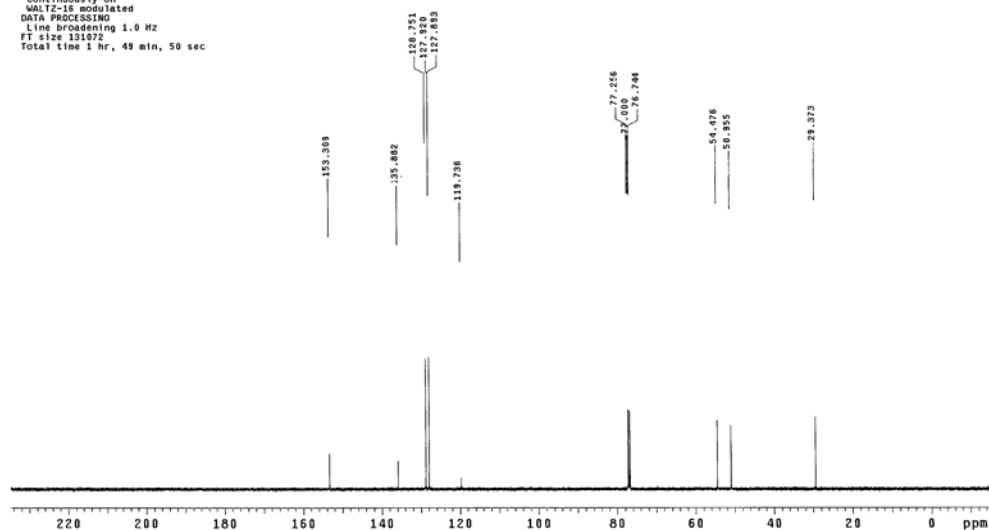
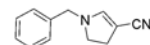
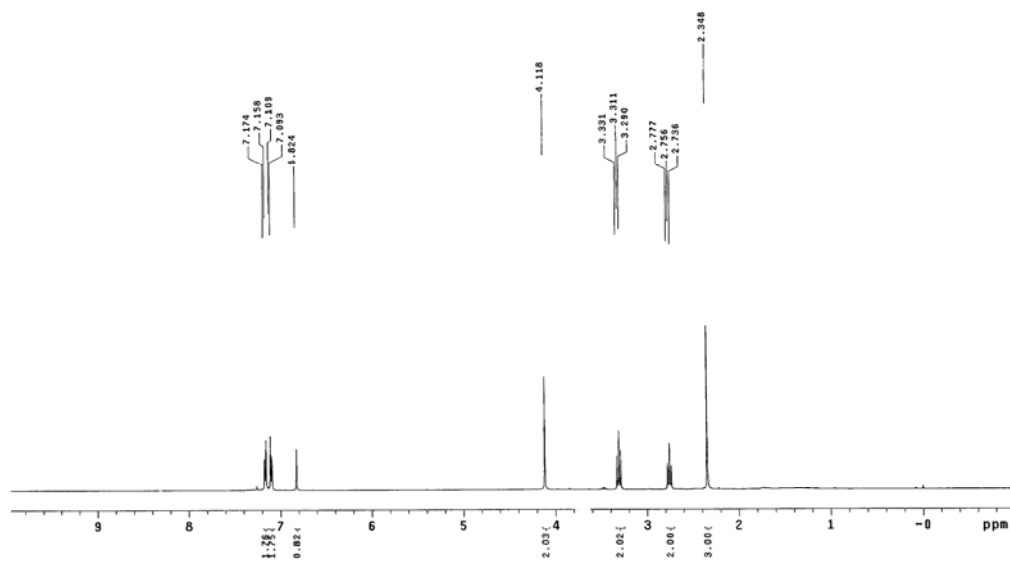
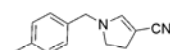


Figure 10. ¹H- (upper) and ¹³C-NMR (lower) spectra of compound **2j**.

STANDARD PROTON PARAMETERS

Archive directory: /export/home/luuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
File: m567
INOVA-500 "MENU500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.852 sec
Width 8319.2 Hz
8 repetitions
OBSERVE F1: 499.0025927 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec



STANDARD CARBON PARAMETERS

Archive directory: /export/home/luuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
User: j-14-87
File: m568
INOVA-500 "MENU500"

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13: 125.6754970 MHz
DECOUPLE H1: 499.0050905 MHz
Power 40 dB
Continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec

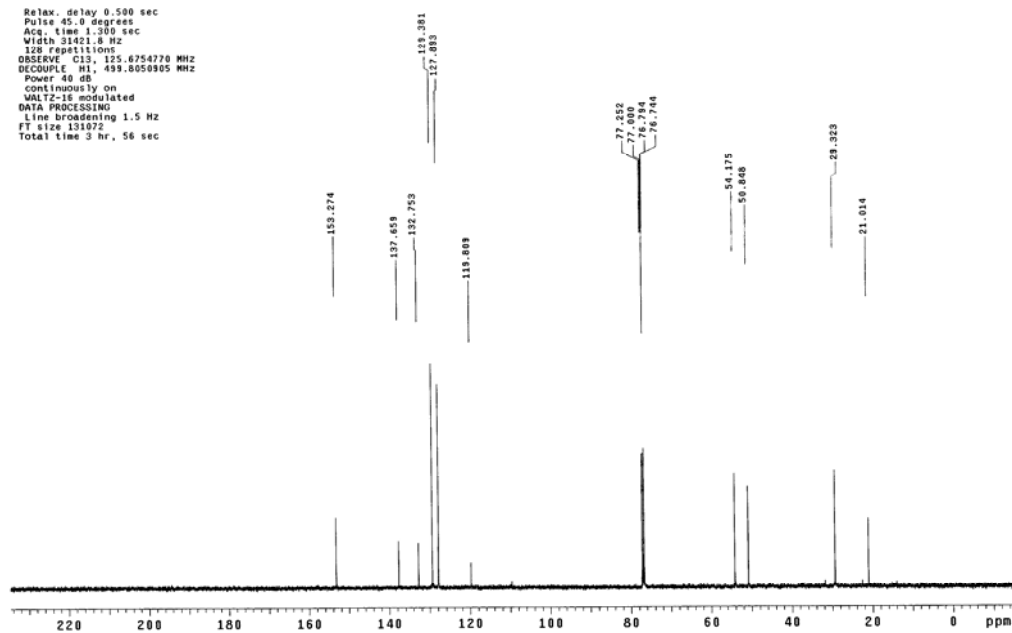
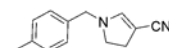
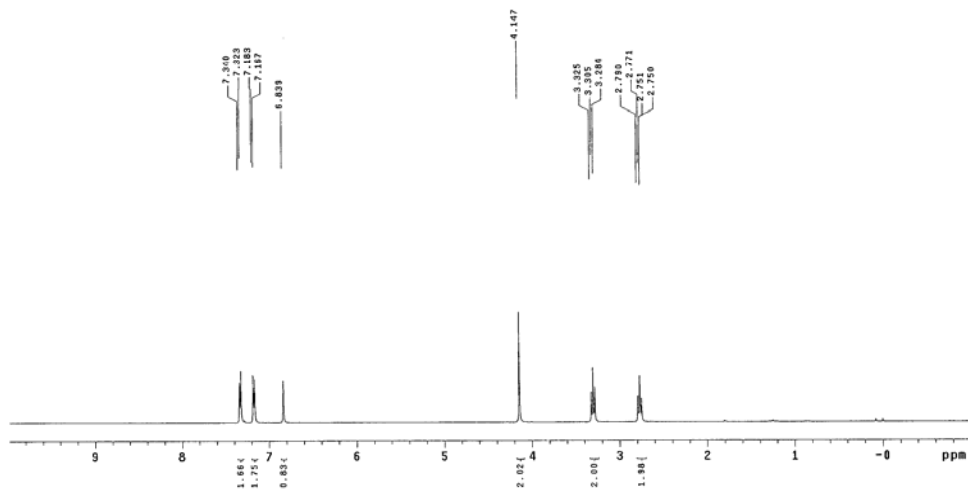
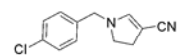


Figure 11. ¹H- (upper) and ¹³C-NMR (lower) spectra of compound **2k**.

STANDARD PROTON PARAMETERS
 Archive directory: /export/home/liuy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 File: 1652
 INOVA-500 "NENUS00"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.852 sec
 Width 8513.2 Hz
 8 repetitions
 OBSERVE H1, 499.8025805 MHz
 DATA PROCESSING
 FT size 65536
 Total time 9 min, 23 sec



STANDARD CARBON PARAMETERS
 Archive directory: /export/home/liuy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 User: 1-14-87
 File: 1703
 INOVA-500 "NENUS00"
 Relax. delay 0.500 sec
 Pulse 45.0 degrees
 Acq. time 1.200 sec
 Width 51421.6 Hz
 64 repetitions
 OBSERVE C13, 125.8754828 MHz
 DECOUPLE H1, 499.8050905 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.5 Hz
 FT size 131072
 Total time 2 hr, 3 min, 31 sec

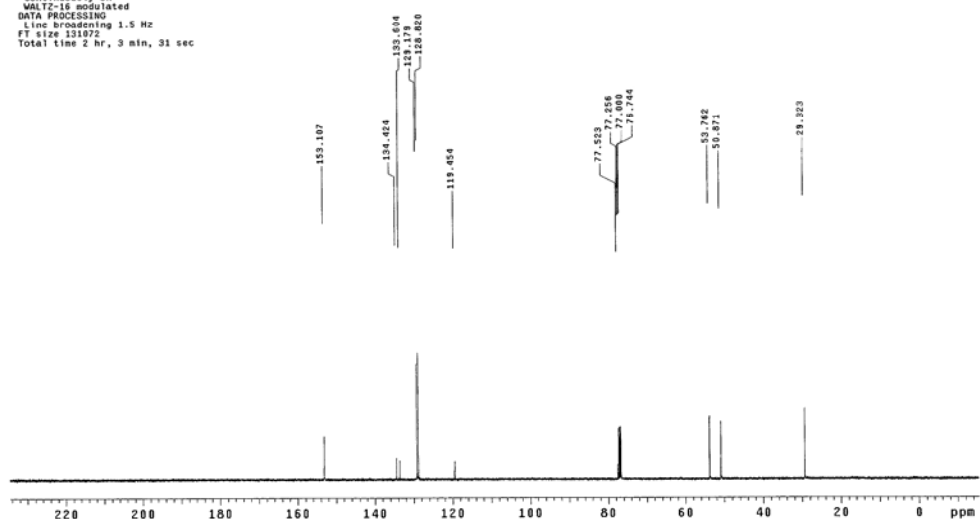
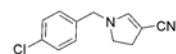
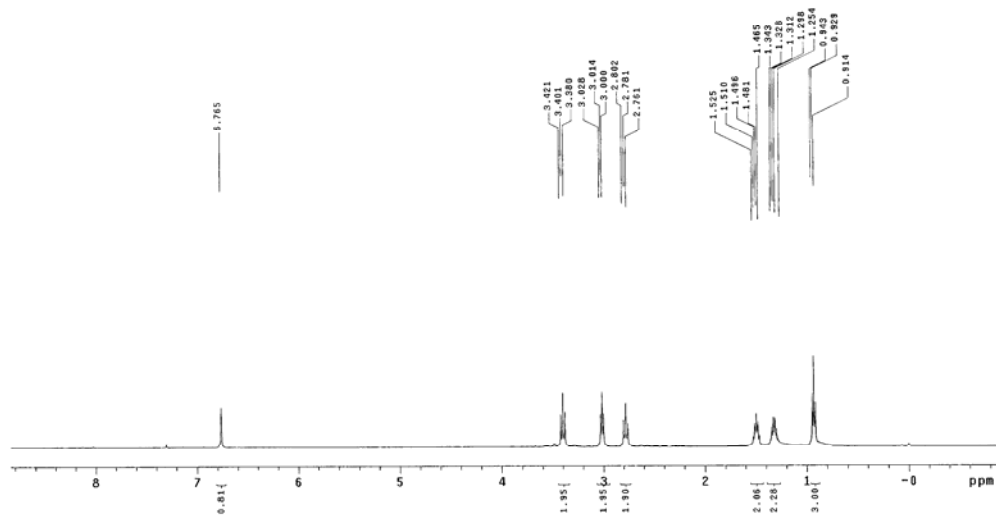
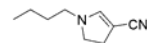


Figure 12. ^1H - (upper) and ^{13}C -NMR (lower) spectra of compound 21.

STANDARD PROTON PARAMETERS
 Archive directory: /export/home/liuy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 File: m025
 INOVA-500 "NENUS00"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.852 sec
 Width 8513.2 Hz
 8 repetitions
 OBSERVE H1, 499.8025700 MHz
 DATA PROCESSING
 FT size 65536
 Total time 9 min, 23 sec



STANDARD CARBON PARAMETERS
 Archive directory: /export/home/ouyy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 User: 1-14-87
 File: m027
 INOVA-500 "NENUS00"
 Relax. delay 0.500 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 31421.8 Hz
 84 repetitions
 OBSERVE C13, 125.6754641 MHz
 DECOUPLE H1, 499.8030805 MHz
 Power 40 dB
 Continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.5 Hz
 FT size 131072
 Total time 2 hr, 3 min, 31 sec

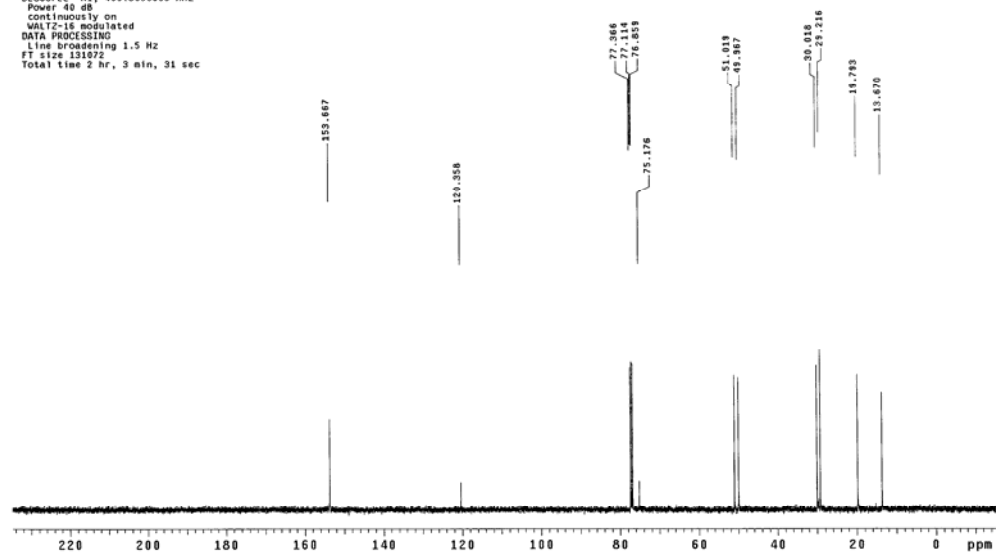
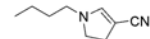


Figure 13. ^1H - (upper) and ^{13}C -NMR (lower) spectra of compound **2m**.

STANDARD PROTON PARAMETERS

Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: 0537

INOVA-500 "NMR500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.852 sec

Width 8560.7 Hz

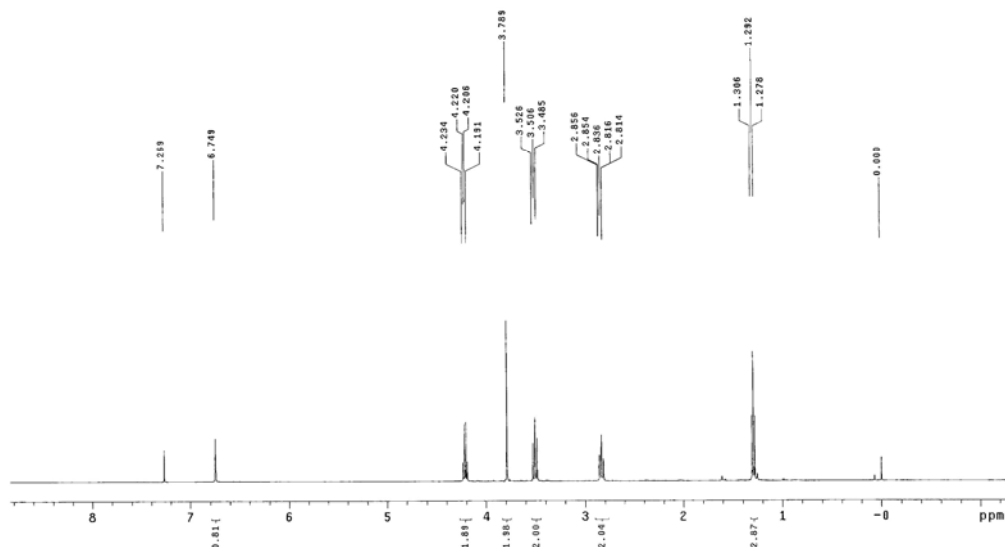
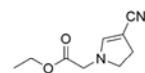
8 repetitions

OBSERVE H1, 499.8025883 MHz

DATA PROCESSING

FT size 65536

Total time 0 min, 23 sec



STANDARD CARBON PARAMETERS

Archive directory: /export/home/ouyy/vnmrsys/data

Sample directory:

Pulse Sequence: s2pul

Solvent: cdcl3

Ambient temperature

User: 1-14-87

File: 0538

INNOVA-500 "NMR500"

Relax. delay 0.500 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 31401.8 Hz

182 repetitions

OBSERVE C13, 125.6754685 MHz

DECOUPLE H1, 499.8050905 MHz

Power 42 dB

continuously on

WALTZ-16 modulated

Line broadening 1.5 Hz

FT size 131072

Total time 3 hr, 56 sec

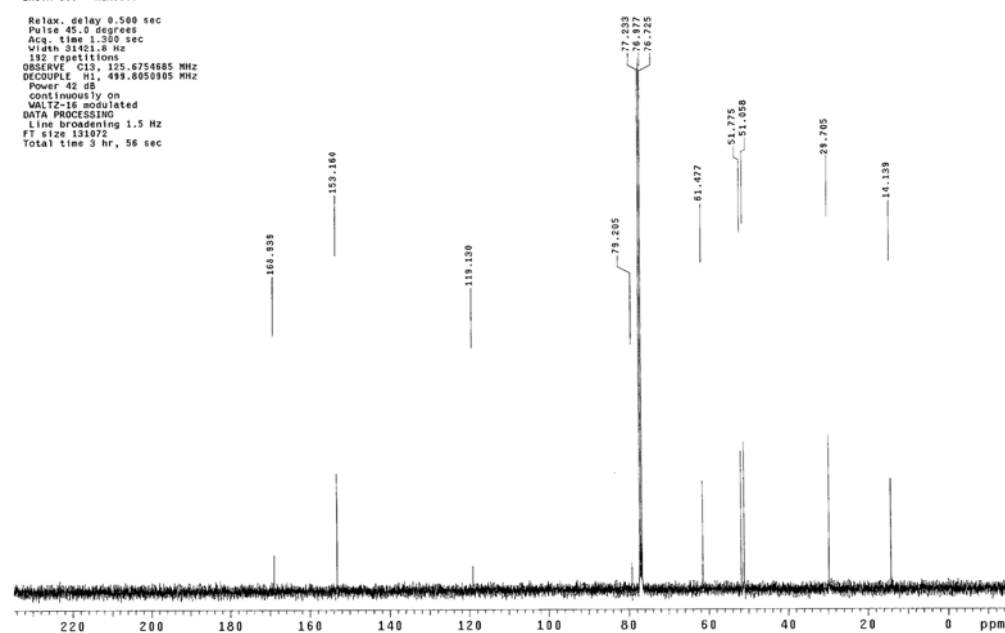
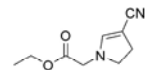
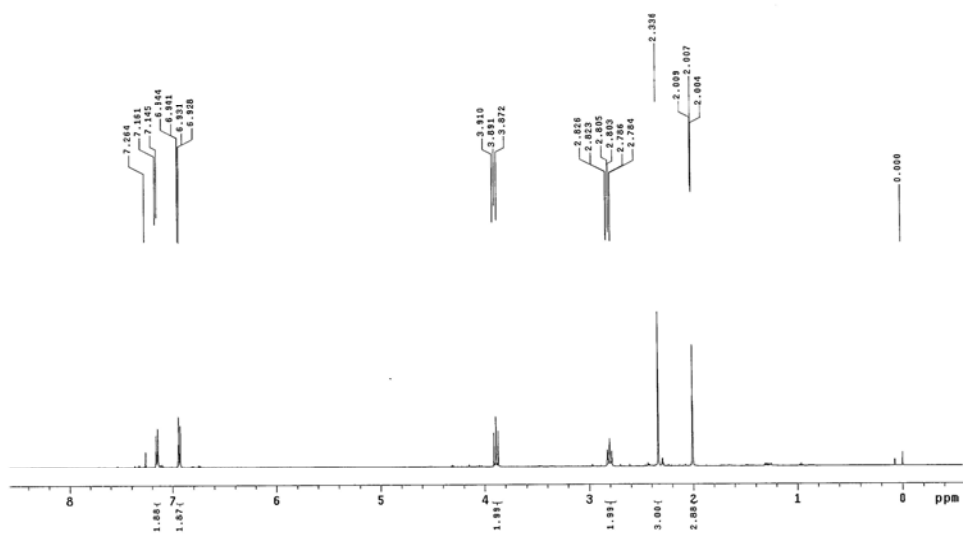
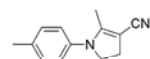


Figure 14. ¹H- (upper) and ¹³C-NMR (lower) spectra of compound 2n.

STANDARD PROTON PARAMETERS
 Archive directory: /export/home/ouyy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 File: s757
 INOVA-500 "HENU500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.892 sec
 Width 8500.7 Hz
 8 repetitions
 OBSERVE H1, 499.8025901 MHz
 DATA PROCESSING
 FT size 65536
 Total time 0 min, 23 sec



STANDARD CARBON PARAMETERS
 Archive directory: /export/home/ouyy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pul
 Solvent: cdcl3
 Ambient temperature
 User: 1-14-87
 File: s708
 INOVA-500 "HENU500"
 Relax. delay 0.500 sec
 Pulse 45.0 degrees
 Acq. time 1.890 sec
 Width 31421.8 Hz
 132 repetitions
 OBSERVE C13, 125.6754894 MHz
 DECOUPLE H1, 499.8058905 MHz
 Power 42 dB
 Continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.5 Hz
 FT size 131072
 Total time 2 hr, 3 min, 31 sec

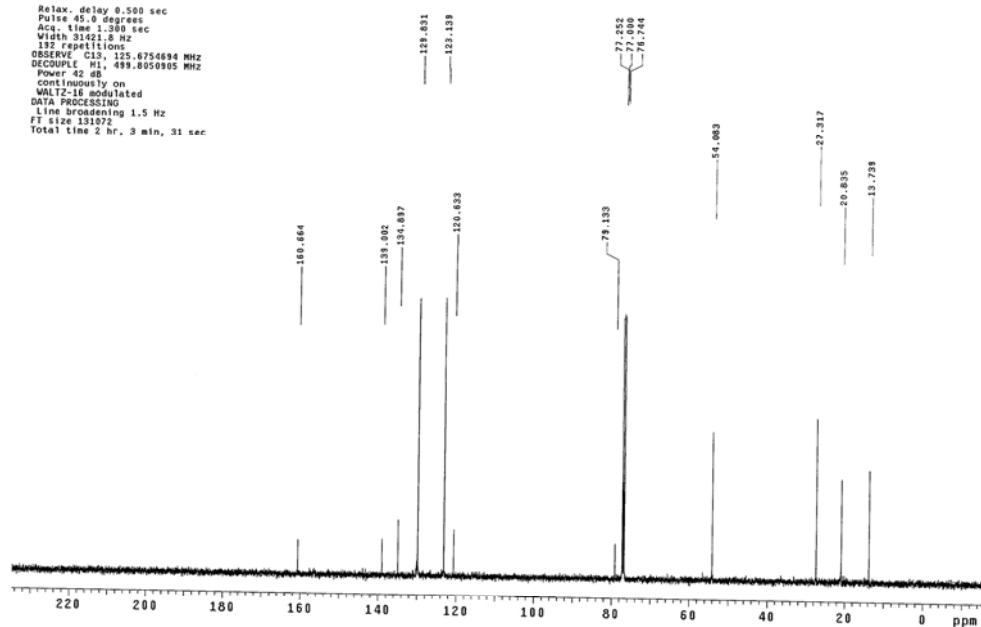
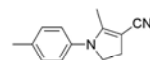
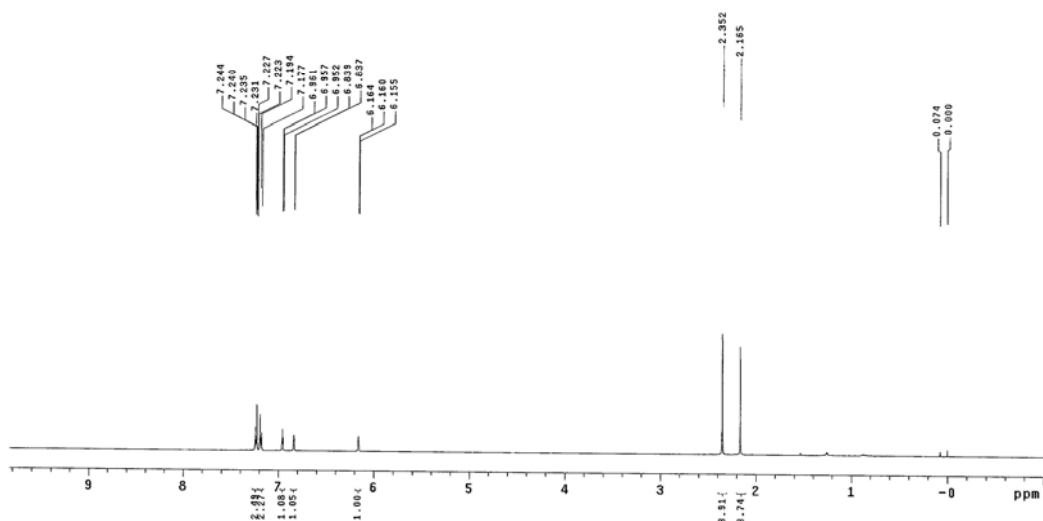


Figure 15. ^1H - (upper) and ^{13}C -NMR (lower) spectra of compound **2o**.

Cc1ccn(c1)-c2ccc(C)cc2

Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
File: q644
INOVA-500 "NENU500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8500.7 Hz
8 repetitions
OBSERVE H1, 499.8026041 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec

Cc1ccccc1N2C=CC=C2C

Pulse Sequence: zgpg30
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: q645
INNOVA-500 "NMR500"

```
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
192 repetitions
OBSERVE C13, 125.6754675 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
```

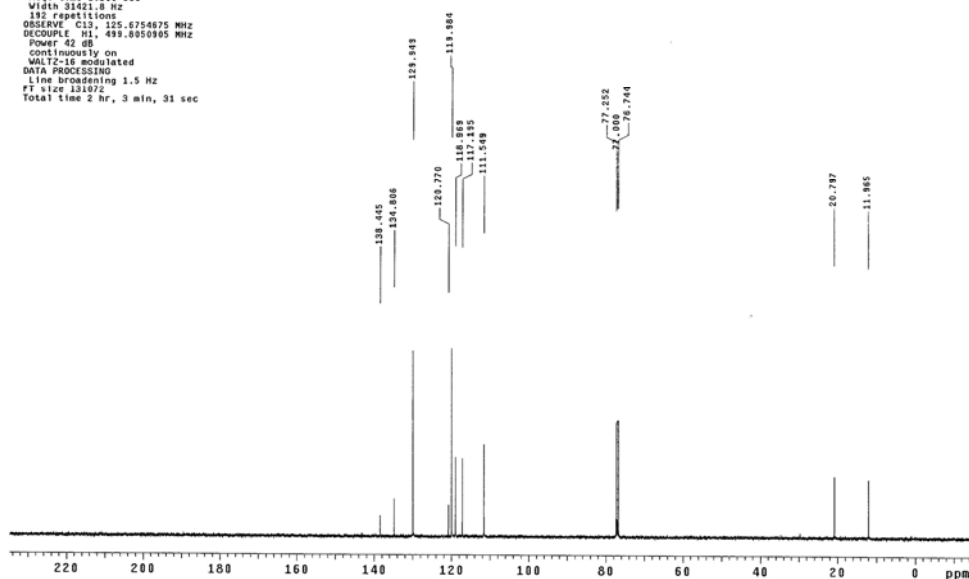
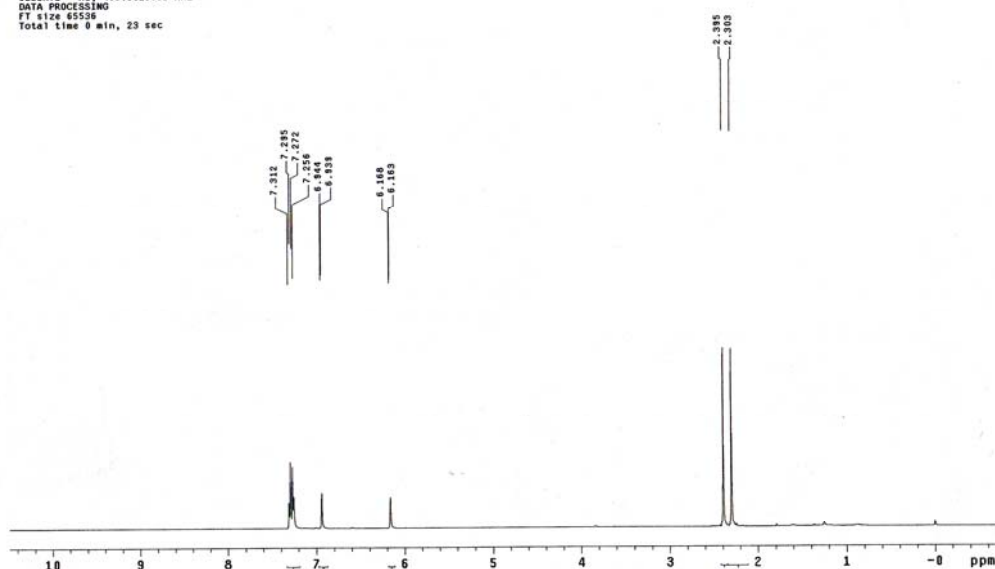
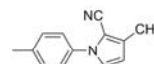


Figure 16. ^1H - (upper) and ^{13}C -NMR (lower) spectra of compound **4p**.

STANDARD PROTON PARAMETERS

Archive directory: /export/home/liuy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 File: c395
 INOVA-500 "NENUS00"

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.892 sec
 Width 7596.8 Hz
 8 repetitions
 OBSERVE H1, 499.9025963 MHz
 DATA PROCESSING
 FT size 65536
 Total time 9 min, 23 sec



STANDARD CARBON PARAMETERS

Archive directory: /export/home/liuy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 User: 1-14-97
 File: d47
 INOVA-500 "NENUS00"

Relax. delay 0.300 sec
 Pulse 45.0 degrees
 Acq. time 1.390 sec
 Width 31421.8 Hz
 256 repetitions
 OBSERVE C13, 125.6754685 MHz
 DECOUPLE H1, 499.8050905 MHz
 Power 40 dB
 Continuously on
 MALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.5 Hz
 FT size 131072
 Total time 1 hr, 49 min, 51 sec

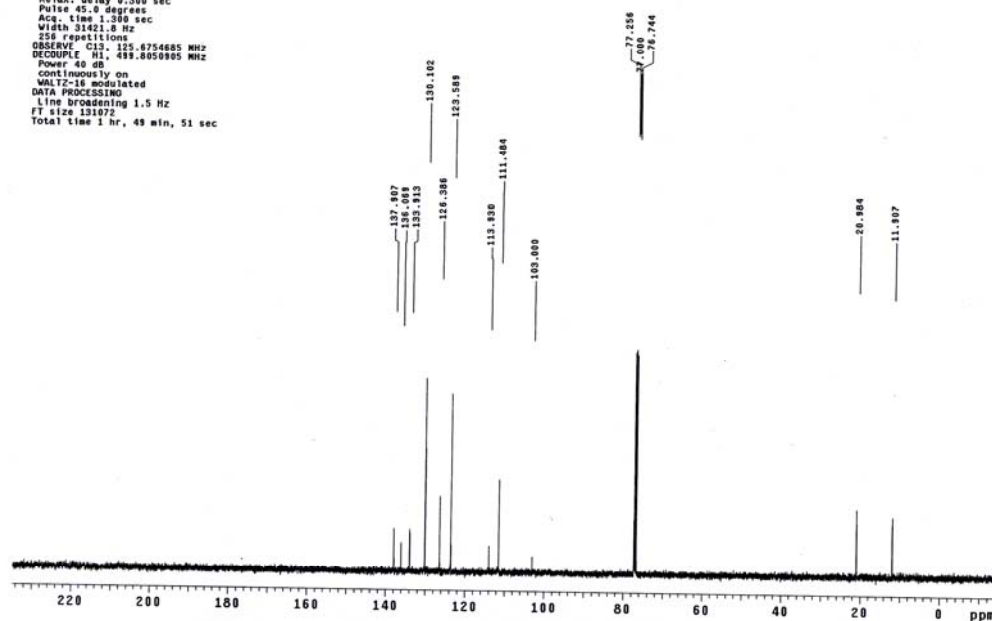
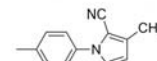


Figure 17. ^1H - (upper) and ^{13}C -NMR (lower) spectra of compound **5p**.