Supplementary Information

Efficient three-component one-pot synthesis of fully substituted pyridin-2(1*H*)-ones via tandem Knoevenagel condensation/ring-opening of cyclopropane/intramolecular cyclization

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I. General

All reagents were purchased from commercial sources and used without treatment, unless otherwise indicated. ^{1}H NMR and ^{13}C NMR spectra were recorded at 25 ºC on a 500 MHz and 125 MHz, respectively, and TMS as internal standard. Elemental analyses were measured on an E-2400 analyzer (Perkin-Elmer). Mass spectra were recorded on Agilient 1100 LCMsD mass spectrometer.

II. Synthesis and analytical data of 2a-j.

The substituted acetylacetamide substrates **1**, were prepared according to procedures reported by our group. For reference, see: Z. Zhang, Q. Zhang, S. Sun, T. Xiong, Q. Liu, *Angew. Chem. Int. Ed.* 2007, **46**, 1726–1729; and Pan, W.; Dong, D.; Wang, K.; Zhang, J.; Wu, R.; Xiang, D.; Liu, Q. *Org. Lett.* **2007**, *9*, 2421–2423.

General procedure for the preparation of **2** (**2a** as an example): 2 mmol 1-acetyl-*N*-phenylcyclopropanecarboxamide was dispensed in 5 ml DMF. 1.1 mmol malononitrile was added at room temperature, and then 2 mmol piperidine was added and stirring for 2.5 hours. After the starting material **1a** was consumed as indicated by TLC, the reaction mixture was extracted with CH_2Cl_2 (3 × 10 mL). The combined organic phase was washed with water (3 × 20 mL), dried over $MgSO_4$, filtered and concentrated in *vacuo.* The crude product was purified by recrystalization in ethanol.

2-amino-4-methyl-6-oxo-1-phenyl-5-(2-(piperidin-1-yl)ethyl)-1,6-dihydropyridine -3-carbonitrile (2a)

White solid. m.p. 148-150 ºC. ^{1}H NMR ($CDCl_3$, 500 MHz) $\delta = 1.42$ (s, 2H), 1.56-1.59 (m, 4H), 2.34 (s, 3H), 2.38-2.41 (m, 4H) , 2.47 (s, 4H), 2.69-2.72 (m, 2H), 4.83 (s, 2H), 7.25-7.27 (m, 2H), 7.53 (d, J = 7.5 Hz, 1H), 7.57-7.60 (m, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 17.7, 23.8, 24.0, 25.6, 54.0, 57.2, 73.0, 116.4, 117.3, 128.0, 129.7, 130.3, 134.2, 146.45, 152.9, 161.0. MS calcd *m/z* 336.2, Found 337.2 $[(M + 1)]^{+}$. Anal. Calcd for $C_{20}H_{24}N_4O$: C,71.40; H, 7.19; N, 16.65; Found: C, 71.84; H, 7.04; N, 16.28.

2-amino-1-(4-chlorophenyl)-4-methyl-6-oxo-5-(2-(piperidin-1-yl)ethyl)-1,6-dihydropyridine-3-carbonitrile (2b)

White solid. m.p. 164-166 ºC. ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 1.43 (s, 2H), 1.58 (t, *J* = 5.5 Hz, 4H), 2.34 (s, 3H), 2.36-2.40 (m, 2H), 2.47 (s, 4H), 2.70 (t, *J* = 8.0 Hz, 2H), 4.78 (s, 2H), 7.22 (d, *J* = 8.5 Hz, 2H), 7.57 (d, *J* = 8.0 Hz, 2H). ^{13}C NMR (DMSO-d_6, 125 MHz) δ = 17.7, 23.9, 24.1, 25.6, 53.9, 57.3, 71.4, 113.4, 117.9, 130.1, 130.9, 133.9, 134.2, 147.0, 154.3, 160.8. MS calcd *m/z* 370.2, Found 371.2 $[(M + 1)]^+$. Anal. Calcd for $C_{20}H_{23}ClN_4O$: C, 64.77; H, 6.25; N, 15.11; Found: C, 64.88; H, 6.55; N, 14.89.

2-amino-4-methyl-6-oxo-5-(2-(piperidin-1-yl)ethyl)-1-p-tolyl-1,6-dihydropyridine-3-carbonitrile (2c)

White solid. m.p. 176-178 ºC. ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 1.42 (s, 2H), 1.56-1.59 (m, 4H), 2.33 (s, 3H), 2.39-2.42 (m, 5H) , 2.48 (s, 4H), 2.69-2.72 (m, 2H), 4.80 (s, 2H), 7.13 (d, *J* = 8.0 Hz, 2H), 7.38 (d, *J* = 8.0 Hz, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 17.9, 21.2, 24.0, 24.3, 25.8, 54.2, 57.4, 73.2, 116.5, 117.6, 127.8, 131.2, 131.6, 140.2, 146.6, 153.3, 161.4.MS calcd *m/z* 350.2, Found 351.2 $[(M + 1)]^+$. Anal. Calcd for $C_{21}H_{26}N_4O$: C, 71.97; H, 7.48; N, 15.99; Found: C, 71.84; H, 7.04; N, 16.28.

2-amino-1-(2,4-dimethylphenyl)-4-methyl-6-oxo-5-(2-(piperidin-1-yl)ethyl)-1,6-dihydropyridine-3-carbonitrile (2d)

White solid. m.p. 118-120 ºC. ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 1.42 (s, 2H), 1.57 (t, *J* = 5.5 Hz, 4H), 2.05 (s, 3H), 2.34 (s, 3H), 2.38-2.41 (m, 5H), 2.47 (s, 4H), 2.69-2.72 (m, 2H), 4.83 (s, 2H), 7.02 (d, *J* = 8.0 Hz, 1H), 7.18 (d, *J* = 8.0 Hz, 1H), 7.27 (s, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 17.0, 17.9, 21.1, 24.0, 24.3, 25.8, 54.2, 57.4, 72.9, 116.5, 117.7, 127.7, 130.6, 132.7, 135.6, 140.3, 146.6, 153.0, 160.8. MS calcd *m/z* 364.2, Found 365.2 $[(M + 1)]^+$. Anal. Calcd for $C_{22}H_{28}N_4O$: C, 72.50; H, 7.74; N, 15.37; Found: C, 72.42; H, 7.58; N, 15.45.

2-amino-4-methyl-6-oxo-1-phenyl-5-(2-(piperidin-1-yl)propyl)-1,6-dihydropyridine-3-carbonitrile (2f)

White solid. m.p. 74-76 ºC. ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 0.98 (d, *J* = 6.5 Hz,

3H), 1.42 (d, *J* = 5.0 Hz, 2H), 1.50–1.56 (m, 4H), 2.34 (s, 3H), 2.38-2.46 (m, 1H), 2.56 (d, *J* = 5.5 Hz, 4H), 2.75-2.78 (m, 1H) , 2.80 (d, *J* = 6.5 Hz, 1H), 4.77 (s, 2H), 7.24-7.27 (m, 2H), 7.53 (t, *J* = 7.5 Hz, 1H), 7.59 (t, *J* = 7.5 Hz, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 15.2, 18.5, 24.8, 26.4, 29.1, 49.6, 59.3, 73.5, 117.7, 128.2, 128.3, 130.1, 130.7, 134.5, 146.7, 153.0, 161.6. MS calcd *m/z* 350.2, Found 351.2 $[(M + 1)]^+$. Anal. Calcd for $C_{21}H_{26}N_4O$: C, 71.97; H, 7.48; N, 15.99; Found: C, 71.59; H, 7.19; N, 16.11.

2-amino-4-methyl-5-(2-morpholinoethyl)-6-oxo-1-phenyl-1,6-dihydropyridine-3-carbonitrile (2g)

White solid. m.p. 222-224 ºC. ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 2.34 (s, 3H), 2.41-2.44 (m, 2H), 2.52 (s, 4H), 2.68-2.71 (m, 2H) , 3.70 (t, *J* = 5.0 Hz, 4H), 4.90 (s, 2H), 7.25 (d, *J* = 8.0 Hz, 2H), 7.54 (d, *J* = 7.5 Hz, 1H), 7.58-7.61 (m, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 18.0, 23.9, 53.4, 57.0, 66.8, 73.3, 116.1, 117.5, 128.2, 130.0, 130.6, 134.3, 146.9, 157.2, 161.3. MS calcd *m/z* 338.2, Found 339.2 $[(M + 1)]^+$. Anal. Calcd for $C_{19}H_{22}N_4O_2$: C, 67.44; H, 6.55; N, 16.56; Found: C, 67.82; H, 6.31; N, 16.28.

2-amino-1-(2,4-dimethylphenyl)-4-methyl-5-(2-morpholinoethyl)-6-oxo-1,6-dihydropyridine-3-carbonitrile (2h)

White solid. m.p. 200-202 ºC. ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 2.06 (s, 3H), 2.34 (s, 3H), 2.38 (s, 3H), 2.40-2.44 (m, 2H), 2.52 (d, *J* = 2.5 Hz, 4H), 2.71 (t, *J* = 7.5 Hz, 2H) , 3.70 (t, *J* = 4.5 Hz, 4H), 4.81 (s, 2H), 7.03 (d, *J* = 7.5 Hz, 1H), 7.19 (d, *J* = 8.0 Hz, 1H), 7.23 (s, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 17.0, 17.9, 21.1, 23.8, 53.4, 57.1, 66.8, 72.9, 116.0, 117.6, 127.7, 128.7, 130.5, 132.8, 135.6, 140.4, 146.7, 153.1, 160.8. MS calcd *m/z* 366.2, Found 369.2 $[(M + 1)]^+$. Anal. Calcd for $C_{19}H_{22}N_4O_2$: C, 68.83; H, 7.15; N, 15.29; Found: C, 68.49; H, 7.04; N, 15.34.

2-amino-1-(2-chlorophenyl)-4-methyl-5-(2-morpholinoethyl)-6-oxo-1,6-dihydropyridine-3-carbonitrile (2i)

White solid. m.p. 218-220 ºC. ^{1}H NMR (DMSO-d_6, 500 MHz) δ = 2.23 (s, 3H), 2.25 (s, 2H), 2.37 (s, 4H), 2.52 (t, *J* = 7.5 Hz, 2H), 3.54 (t, *J* = 4.5 Hz, 4H) , 6.82 (s, 2H), 7.38-7.40 (m, 1H), 7.49-7.53 (m, 2H), 7.66-7.67 (m, 1H). ^{13}C NMR ($CDCl_3$, 125

MHz) δ = 18.3, 24.2, 53.8, 57.6, 66.8, 71.8, 113.7, 118.3, 129.5, 131.1, 131.6, 131.8, 132.5, 133.3, 147.9, 150.5, 160.6. MS calcd *m/z* 372.1, Found 373.1 $[(M + 1)]^+$. Anal. Calcd for $C_{19}H_{21}ClN_4O_2$: C, 61.21; H, 5.68; N, 15.03; Found: C, 61.49; H, 5.49; N, 15.29.

2-amino-1-(2,4-dimethylphenyl)-4-methyl-6-oxo-5-(2-(pyrrolidin-1-yl)ethyl)-1,6-dihydropyridine-3-carbonitrile (2j)

White solid. m.p. 166-168 ºC. ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 1.78 (s, 4H), 2.05 (s, 3H), 2.35 (s, 3H), 2.38 (s, 3H), 2.52-2.55 (m, 2H), 2.60 (s, 4H), 2.73-2.76 (m, 2H), 4.83 (s, 2H), 7.03 (d, *J* = 8.0 Hz, 1H), 7.18 (d, *J* = 7.5 Hz, 1H), 7.22 (s, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 17.0, 17.9, 21.1, 23.3, 26.2, 53.8, 54.5, 73.0, 116.1, 117.6, 127.7, 128.7, 130.6, 132.7, 135.6, 140.4, 146.8, 153.1, 160.9. MS calcd *m/z* 350.2, Found 351.2 $[(M + 1)]^+$. Anal. Calcd for $C_{21}H_{26}N_4O$: C,71.97; H, 7.48; N, 15.99; Found: C, 72.22; H, 7.58; N, 15.45.

III. Synthesis and analytical data of 3a-f.

General procedure for the preparation of **3** (**3a** as an example): 2 mmol 1-acetyl-*N*-phenylcyclopropanecarboxamide was dispensed in 5 ml DMF. 2.1 mmol malononitrile was added at room temperature, and then 0.2 mmol piperidine was added and stirring for 2.5 hours. After the starting material **1a** was consumed as indicated by TLC, the reaction mixture was extracted with CH_2Cl_2 (3 × 10 mL). The combined organic phase was washed with water (3 × 20 mL), dried over $MgSO_4$, filtered and concentrated in *vacuo*. The crude product was purified by recrystalization in ethanol.

2-(2-(6-amino-5-cyano-4-methyl-2-oxo-1-phenyl-1,2-dihydropyridin-3-yl)ethyl)malononitrile (3a)

White solid. m.p. 219-221 ºC. ^{1}H NMR (DMSO-d_6, 500 MHz) δ = 2.04-2.09 (m, 2H), 2.25 (s, 3H), 2.59 (t, *J* = 7.5 Hz, 2H), 4.74 (t, *J* = 6.5 Hz, 1H) , 6.56 (s, 2H), 7.23 (d, *J* = 7.0 Hz, 2H), 7.50-7.57 (m, 3H). ^{13}C NMR (DMSO-d_6, 125 MHz) δ = 17.66, 21.91, 23.50, 28.63, 71.84, 112.39, 114.48, 117.76, 128.73, 129.46, 130.15, 135.03, 147.80,

154.59, 161.04. MS calcd *m/z* 317.1, Found 318.1 $[(M + 1)]^+$. Anal. Calcd for $C_{18}H_{15}N_5O$: C, 68.13; H, 4.76; N, 22.07; Found: C, 68.39; H, 4.59; N, 21.96.

2-(2-(6-amino-1-(4-chlorophenyl)-5-cyano-4-methyl-2-oxo-1,2-dihydropyridin-3-yl)ethyl)malononitrile (3b)

White solid.m.p.158-160 ºC. ^{1}H NMR (DMSO-d_6, 500 MHz) δ = 2.05 (d, *J* = 6.5 Hz, 2H), 2.23 (s, 3H), 2.57 (t, *J* = 7.0 Hz, 2H), 4.67 (s, 1H), 6.75 (s, 2H), 7.25 (d, *J* = 8.5 Hz, 2H), 7.59 (d, *J* = 8.5 Hz, 2H). ^{13}C NMR (DMSO-d_6, 125 MHz) δ = 17.9, 22.1, 23.6, 28.9, 71.2, 112.4, 114.7, 118.0, 130.5, 131.0, 134.2, 134.4, 148.5, 154.8, 161.3. MS calcd *m/z* 351.1, Found 352.1 $[(M + 1)]^+$. Anal. Calcd for $C_{18}H_{14}ClN_5O$: C, 61.46; H, 4.01; N, 19.91; Found: C, 61.22; H, 4.12; N, 19.99.

2-(2-(6-amino-5-cyano-4-methyl-2-oxo-1-p-tolyl-1,2-dihydropyridin-3-yl)ethyl)malononitrile (3c)

White solid. m.p. 202-204ºC. ^{1}H NMR (DMSO-d_6, 500 MHz) δ = 1.99-2.08 (m, 2H), 2.24 (s, 3H), 2.38 (s, 3H), 2.58 (t, *J* = 7.5 Hz, 2H), 4.75 (t, *J* = 6.5 Hz, 1H) , 6.54 (s, 2H), 7.10 (d, *J* = 8.0 Hz, 2H), 7.35 (d, *J* = 8.0 Hz, 2H). ^{13}C NMR (DMSO-d_6, 125 MHz) δ = 17.6, 20.8, 21.8, 23.5, 28.6, 71.7, 112.3, 114.4, 117.7, 128.4, 130.6, 132.3, 138.8, 147.6, 154.7, 161.0. MS calcd *m/z* 331.1, Found 332.1 $[(M + 1)]^+$. Anal. Calcd for $C_{19}H_{17}N_5O$: C, 68.87; H, 5.17; N, 21.13; Found: C, 68.50; H, 5.33; N, 21.01.

2-(2-(6-amino-5-cyano-1-(2,4-dimethylphenyl)-4-methyl-2-oxo-1,2-dihydropyridin-3-yl)ethyl)malononitrile (3d)

White solid. m.p. 176-178 ºC. ^{1}H NMR (DMSO-d_6, 500 MHz) δ = 1.89 (s, 3H), 2.02-2.07 (m, 2H), 2.58 (t, *J* = 7.5 Hz, 2H), 4.69 (d, *J* = 6.0 Hz, 1H) , 6.53 (s, 2H), 6.98 (d, *J* = 8.0 Hz, 1H), 7.15 (d, *J* = 8.0 Hz, 1H), 7.22 (s, 1H). ^{13}C NMR (DMSO-d_6, 125 MHz) δ = 17.4, 18.4, 21.5, 22.6, 24.3, 29.3, 72.5, 113.1, 115.1, 118.5, 129.0, 129.2, 132.0, 132.8, 136.1, 139.8, 148.8, 155.0, 161.3. MS calcd *m/z* 345.2, Found 346.2 $[(M + 1)]^+$. Anal. Calcd for $C_{20}H_{19}N_5O$: C, 69.55; H, 5.54; N, 20.28; Found: C, 69.26; H, 5.33; N, 20.55.

2-(1-(6-amino-5-cyano-4-methyl-2-oxo-1-phenyl-1,2-dihydropyridin-3-yl)propan-2-yl)malononitrile (3e)

White solid. m.p. 170-172 ºC. ^{1}H NMR (DMSO-d_6, 500 MHz) δ = 1.06 (d, *J* = 6.5

Hz, 3H), 2.23 (s, 3H), 2.40-2.42 (m, 1H), 2.45-2.50 (m, 1H), 2.55 (t, *J* = 7.0 Hz, 1H), 4.71 (d, *J* = 4.0 Hz, 1H) , 6.54 (s, 2H), 7.20-7.26 (m, 2H), 7.51 (d, *J* = 7.0 Hz, 1H), 7.55 (t, *J* = 7.5 Hz, 2H). ^{13}C NMR (DMSO-d_6, 125 MHz) δ = 17.5, 18.7, 28.8, 31.1, 35.2, 72.7, 112.4, 114.2, 114.7, 118.5, 129.3, 129.5, 130.2, 130.9, 135.7, 149.2, 155.3, 162.1. MS calcd *m/z* 331.1, Found 332.1 $[(M + 1)]^+$. Anal. Calcd for $C_{19}H_{17}N_5O$: C, 68.87; H, 5.17; N, 21.13; Found: C, 68.39; H, 4.99; N, 21.01.

2-(2-(6-amino-1-(2-chlorophenyl)-5-cyano-4-methyl-2-oxo-1,2-dihydropyridin-3-yl)ethyl)malononitrile (3f)

White solid. m.p. 162-164 ºC. ^{1}H NMR (DMSO-d_6, 500 MHz) δ = 2.24-2.30 (m, 2H), 2.40 (s, 3H), 2.79-2.84 (m, 2H), 3.83 (t, *J* = 7.5 Hz, 1H), 4.90 (s, 2H), 7.36 (d, *J* = 7.0 Hz, 1H), 7.51-7.54 (m, 2H), 7.66-7.67 (m, 1H). ^{13}C NMR (DMSO-d_6, 125 MHz) δ = 18.2, 22.4, 24.0, 29.0, 72.1, 112.4, 114.9, 118.1, 129.2, 129.9, 131.1, 131.5, 133.1, 135.5, 149.0, 154.8, 160.6. MS calcd *m/z* 351.1, Found 352.1 $[(M + 1)]^+$. Anal. Calcd for $C_{18}H_{14}ClN_5O$: C, 61.46; H, 4.01; N, 19.91; Found: C, 61.67; H, 3.89; N, 19.69.

IV. Synthesis and analytical data of 4b-c, 5a-c.

Similar procedure as that of **2a-k** was applied for the preparation of **4a-c and 5a-c.**

9-(4-chlorophenyl)-8-imino-6-methyl-10-oxospiro[4.5]dec-6-ene-7-carbonitrile (4b)

White solid. m.p. 156-158 ºC. ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 1.86 (s, 2H), 1.97 (d, *J* = 5.0 Hz, 2H), 2.06 (s, 2H), 229 (s, 2H), 2.37 (s, 3H), 7.04 (s, 4H), 7.42 (s, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 19.2, 28.2, 39.2, 54.9, 107.3, 113.7, 121.97, 129.9, 130.2, 143.2, 144.1, 167.5, 174.4. MS calcd *m/z* 312.1, Found 313.1 $[(M + 1)]^+$. Anal. Calcd for $C_{18}H_{17}ClN_2O$: C, 69.12; H, 5.48; N, 8.96; Found: C, 69.39; H, 5.22; N, 8.77.

8-imino-9-(4-methoxyphenyl)-6-methyl-10-oxospiro[4.5]dec-6-ene-7-carbonitrile (4c)

White solid. m.p. 152-154 ºC. ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 1.86 (s, 2H), 1.97 (d, *J* = 5.0 Hz, 2H), 2.06 (s, 2H), 229 (s, 2H), 2.37 (s, 3H), 3.85 (s, 3H), 7.04 (s, 4H),

7.42 (s, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 19.4, 28.6, 39.5, 55.1, 55.8, 107.8, 115.3, 122.0, 129.9, 138.8, 143.3, 144.1, 157.2, 167.0, 174.6. MS calcd *m/z* 308.2, Found 309.2 $[(M + 1)]^+$. Anal. Calcd for $C_{19}H_{20}N_2O_2$: C, 74.00; H, 6.54; N, 9.08; Found: C, 73.88; H, 6.39; N, 8.89.

4,4-dicyano-2,2-diethyl-3-methyl-N-phenylbut-3-enamide (5a)

White solid. m.p. 160-162 ºC. ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 0.83 (t, *J* = 7.0 Hz, 6H), 1.77–1.85 (m, 2H), 2.18–2.23 (m, 2H), 2.33 (s, 3H), 7.11 (d, *J* = 7.0 Hz, 2H), 7.42 (s, 1H), 7.42-7.85 (m 3H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 8.2, 16.5, 31.0, 55.1, 110.0, 112.6, 127.6, 128.6, 129.2, 132.1, 150.1, 162.9, 170.3. MS calcd *m/z* 281.2, Found 282.2 $[(M + 1)]^+$. Anal. Calcd for $C_{17}H_{19}N_3O$: C, 72.57; H, 6.81; N, 14.94; Found: C, 72.39; H, 6.99; N, 14.66.

***N*-(4-chlorophenyl)-1-(1,1-dicyanoprop-1-en-2-yl)cyclopentanecarboxamide (5b)**

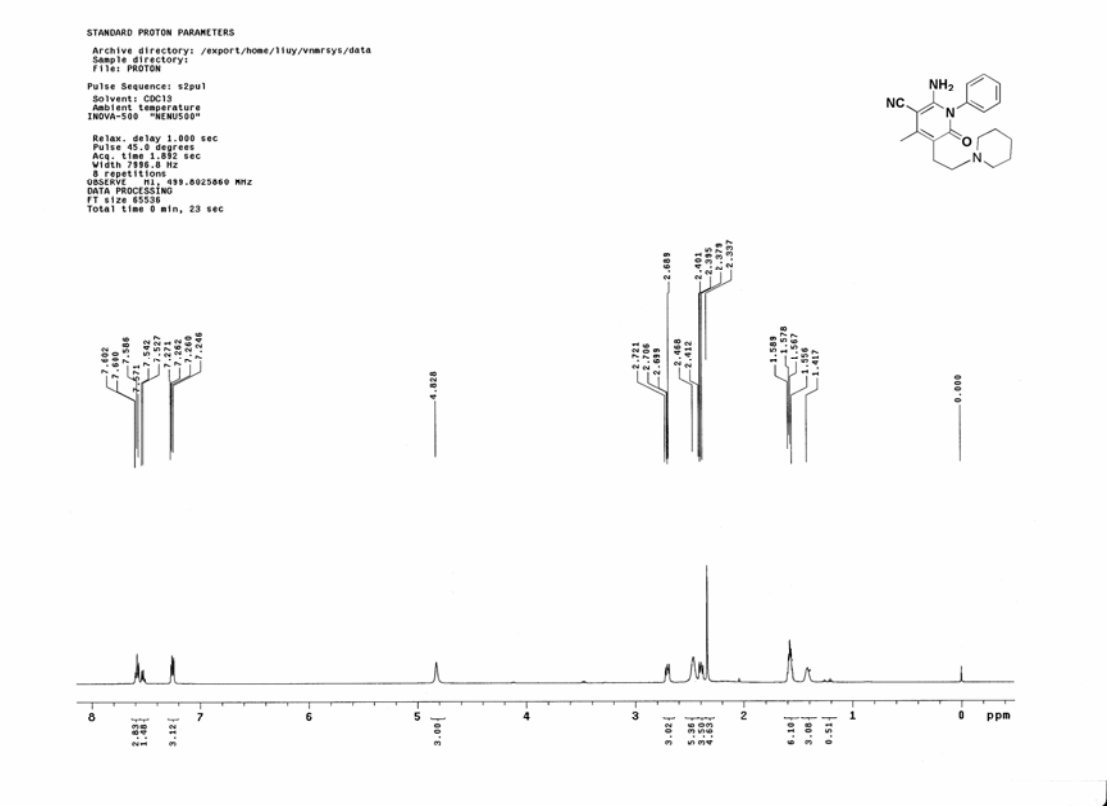
White solid. m.p. 180-182 ºC. ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 1.87 (d, *J* = 6.0 Hz, 2H), 1.91–1.95 (m, 2H), 2.04 (d, *J* = 6.5 Hz, 2H), 2.19-2.23 (m, 2H), 2.35 (s, 3H), 6.82 (d, *J* = 8.5 Hz, 2H), 7.34 (d, *J* = 8.5 Hz, 2H), 7.65 (s, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 19.2, 28.2, 39.2, 54.9, 107.3, 113.7, 122.0, 129.9, 130.2, 143.2, 144.1, 167.5, 174.4. MS calcd *m/z* 313.1, Found 314.1 $[(M + 1)]^+$. Anal. Calcd for $C_{17}H_{16}ClN_3O$: C, 65.07; H, 5.14; N, 13.39; Found: C, 64.95; H, 4.98; N, 13.41.

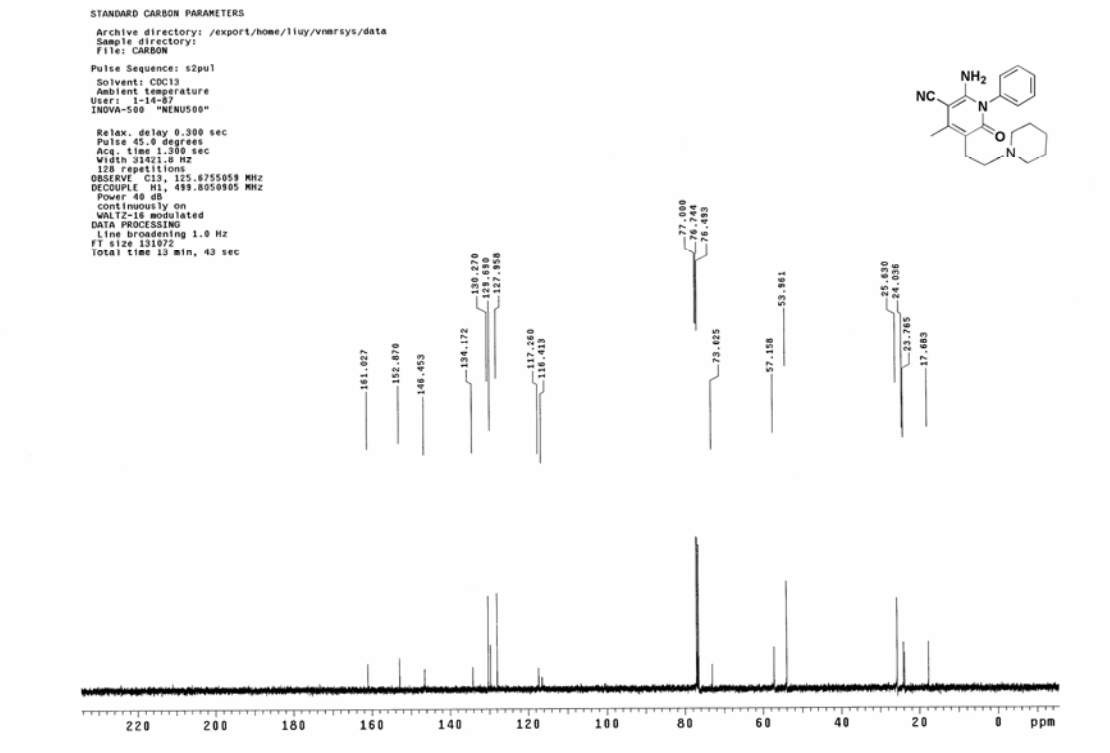
1-(1,1-dicyanoprop-1-en-2-yl)-*N*-(4-methoxyphenyl)cyclopentanecarboxamide (5c)

White solid. m.p. 170-172 ºC. ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 1.86 (d, *J* = 6.5 Hz, 2H), 1.89–1.96 (m, 2H), 2.04 (d, *J* = 5.5 Hz, 2H), 2.20-2.25 (m, 2H), 2.34 (s, 3H), 3.81 (s, 3H), 6.82 (d, *J* = 9.0 Hz, 2H), 6.91 (d, *J* = 9.0 Hz, 2H), 7.77 (s, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 19.4, 28.6, 39.5, 55.1, 55.8, 107.8, 115.3, 122.0, 130.0, 138.8, 143.3, 144.1, 157.2, 167.0, 174.6. MS calcd *m/z* 309.2, Found 310.2 $[(M + 1)]^+$. Anal. Calcd for $C_{18}H_{19}N_3O_2$: C, 69.88; H, 6.19; N, 13.58; Found: C, 69.59; H, 6.22; N, 13.41.

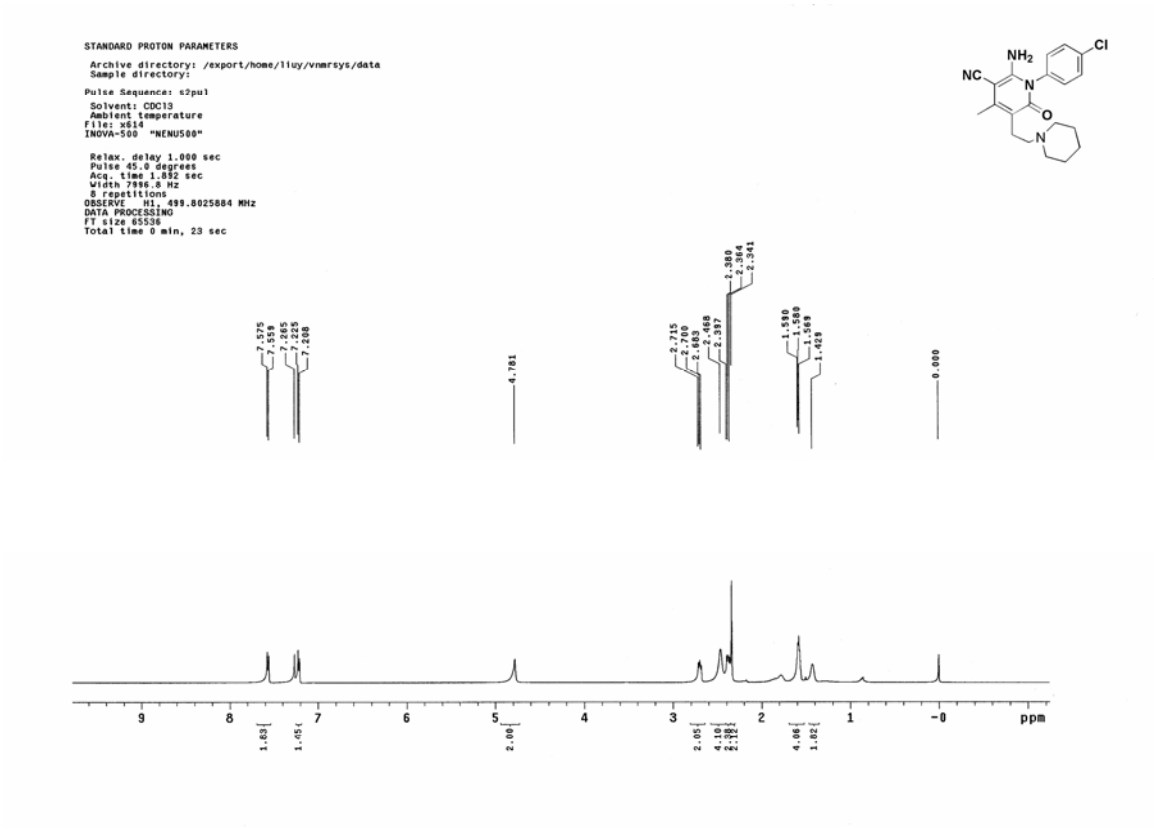
V. Copies of ^{1}H and ^{13}C NMR spectra for compounds 2-5

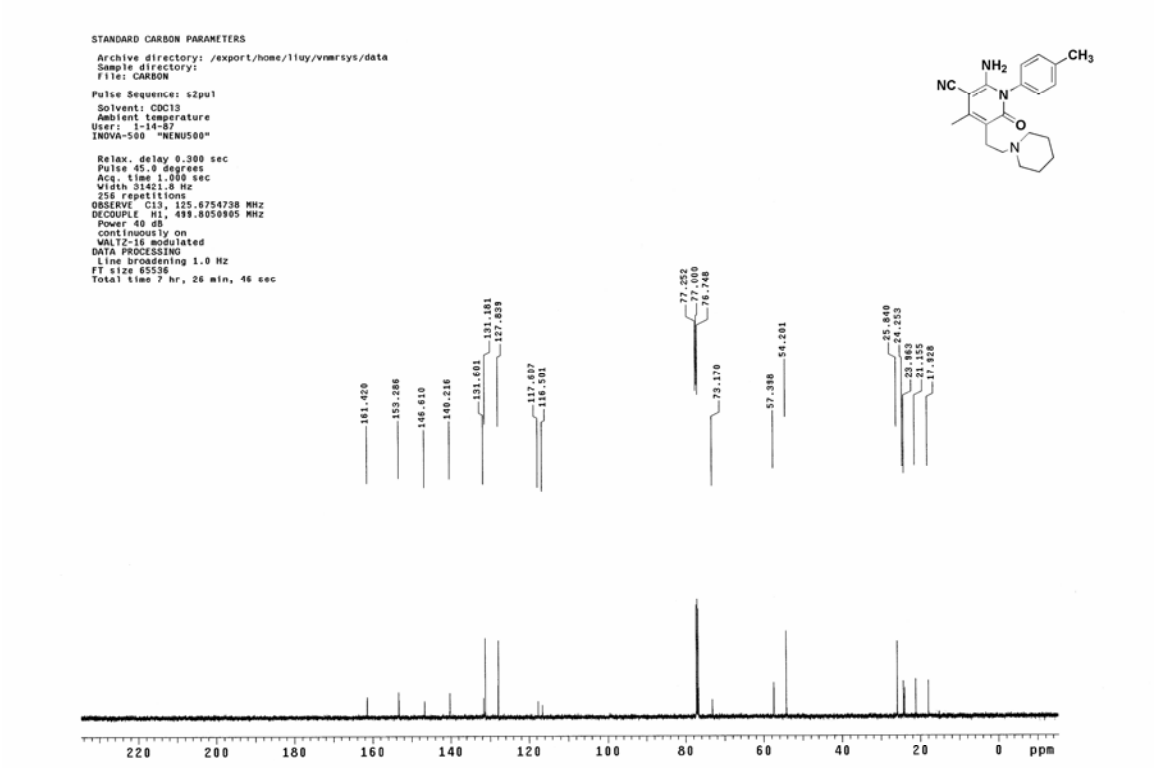
2a



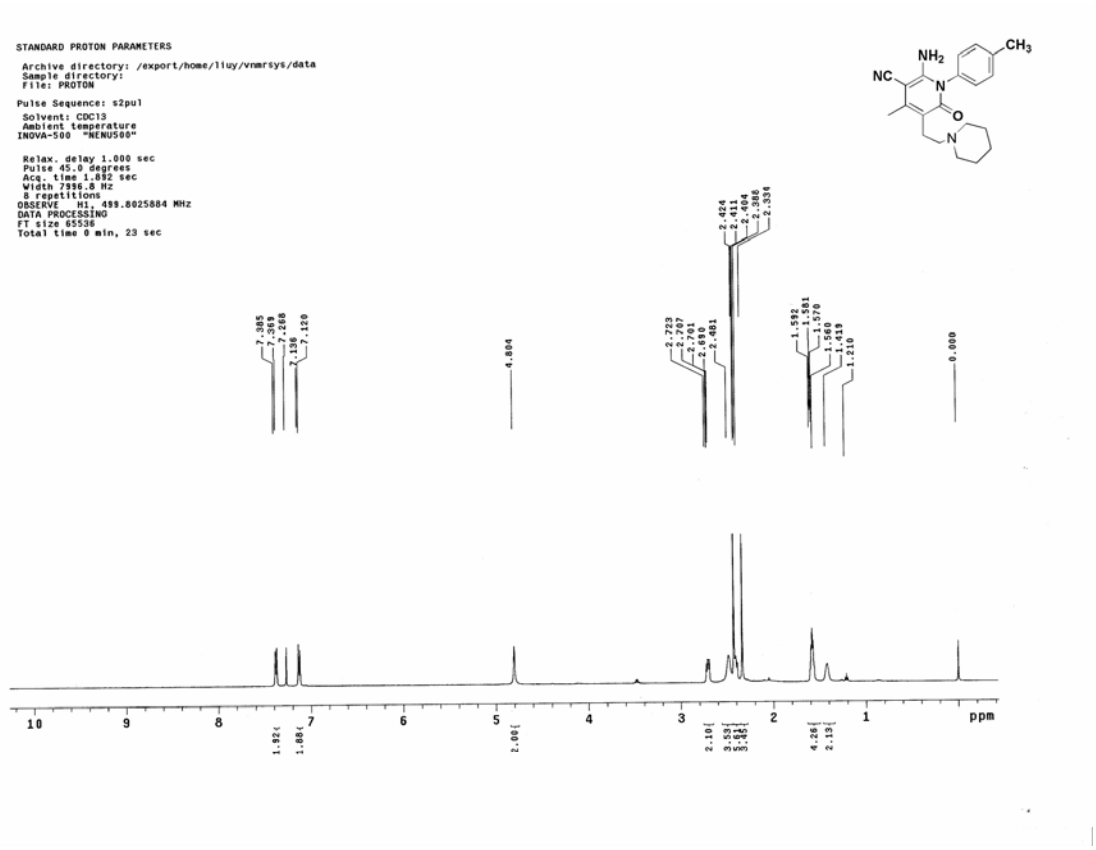


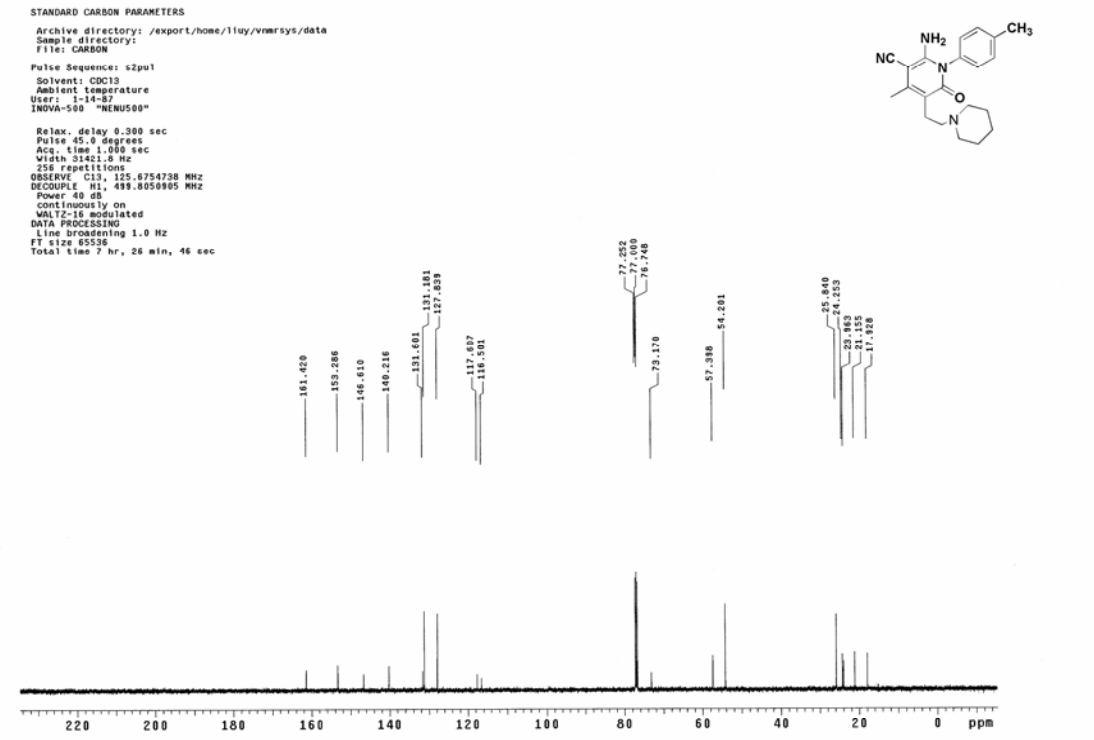
2b


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: x614
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7986.8 Hz
8 repetitions
OBSERVE H1, 499.8025884 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
NH2
Cl
NC
N
O
N
7.575
7.559
7.265
7.255
7.248
4.781
2.715
2.700
2.683
2.468
2.397
2.380
2.364
2.341
1.590
1.580
1.569
1.429
0.000
9
8
7
6
5
4
3
2
1
-0
ppm
1.63
1.45
2.00
2.05
4.10
2.10
4.06
1.02

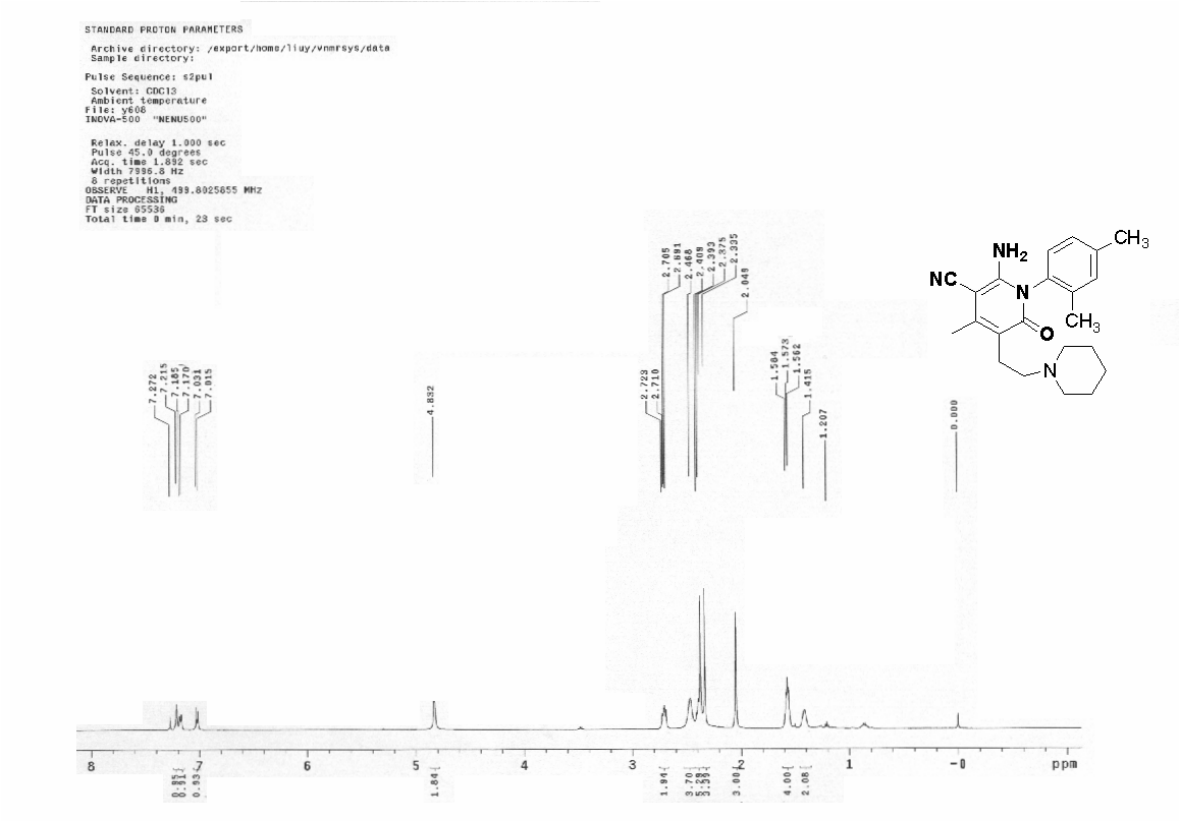

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.000 sec
Width 31421.8 Hz
256 repetitions
OBSERVE C13, 125.6754738 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 7 hr, 26 min, 46 sec
NH2
CH3
NC
N
O
N
161.420
153.286
146.610
140.216
131.601
131.181
127.839
117.607
116.501
77.252
77.000
76.748
73.170
57.388
54.201
25.840
24.253
23.463
21.155
17.928
220
200
180
160
140
120
100
80
60
40
20
0
ppm

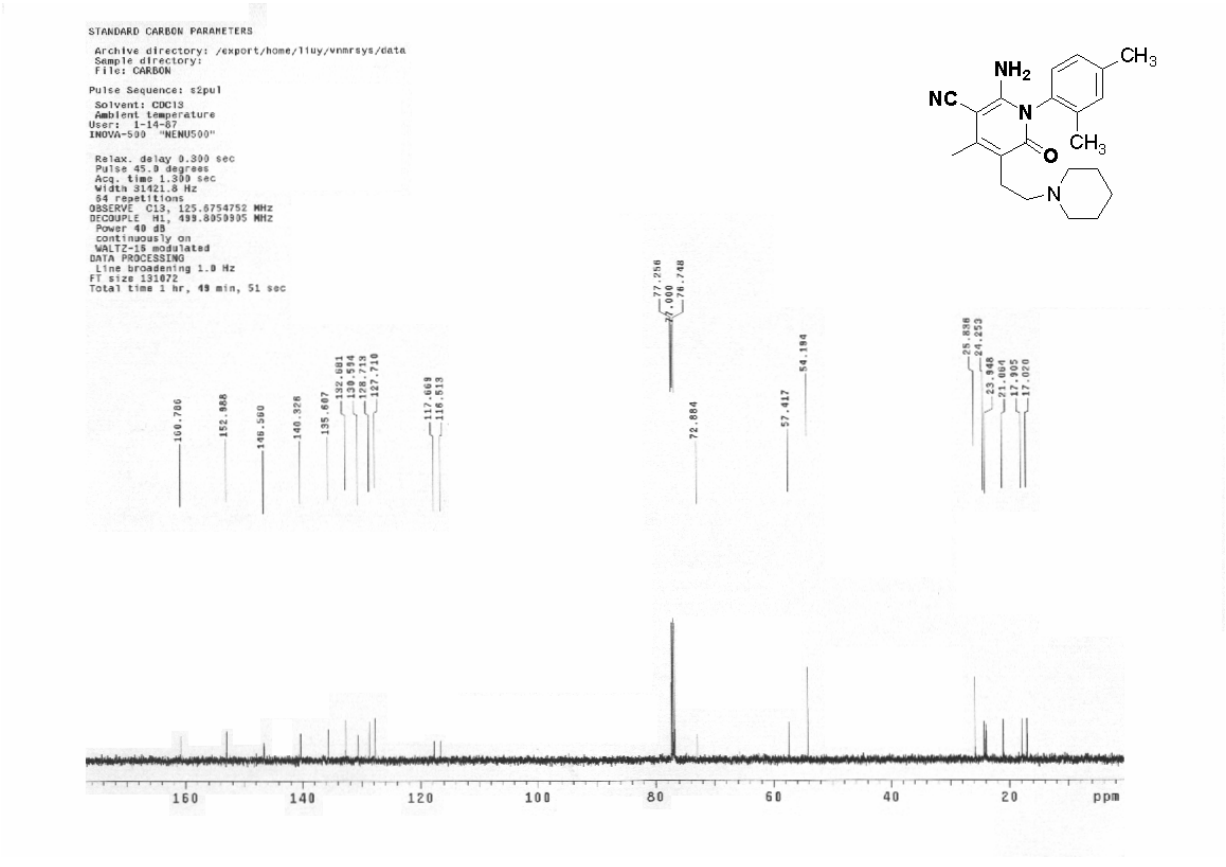
2c


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7996.8 Hz
8 repetitions
OBSERVE H1, 499.8025884 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
NH2
CH3
NC
N
O
N
7.385
7.369
7.288
7.136
7.120
4.804
2.424
2.411
2.404
2.388
2.334
2.723
2.707
2.701
2.690
2.481
1.591
1.581
1.570
1.560
1.419
1.210
0.000
ppm

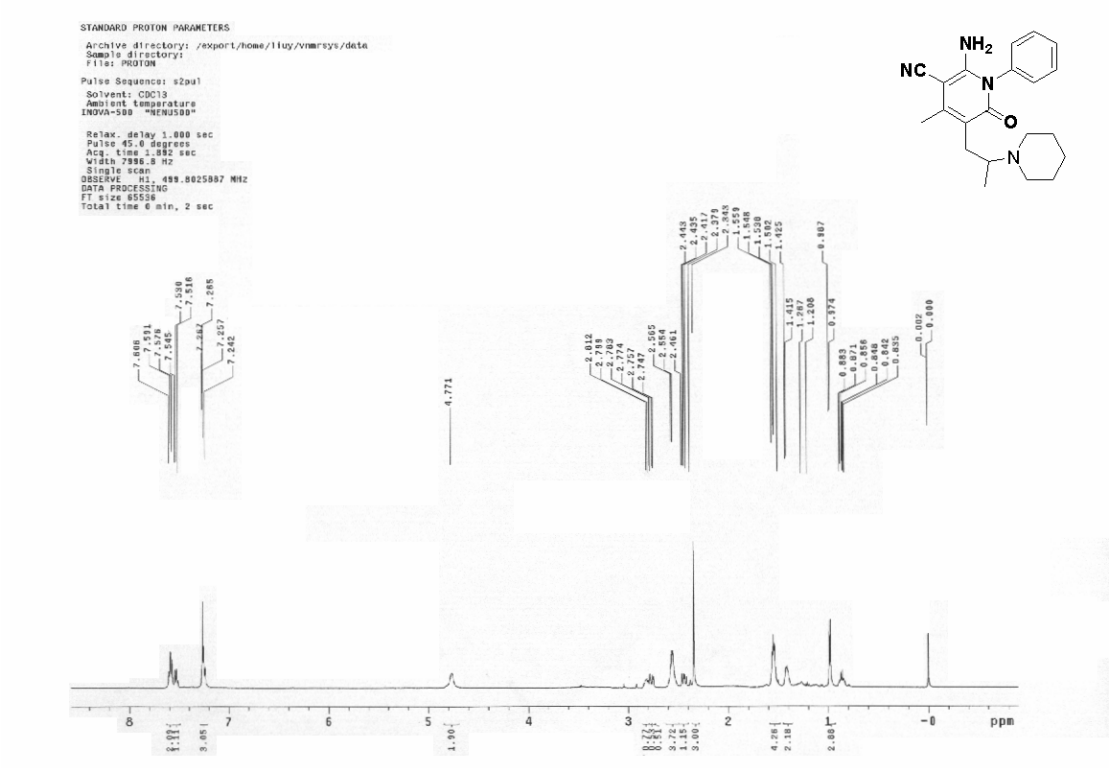

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.000 sec
Width 31421.8 Hz
256 repetitions
OBSERVE C13, 125.6754738 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 7 hr, 26 min, 46 sec
NH2
CH3
NC
N
O
N
161.420
159.286
148.610
140.216
131.601
131.181
127.839
117.607
116.591
77.252
77.000
76.748
73.170
57.388
54.201
25.840
24.253
23.963
21.155
17.928
ppm

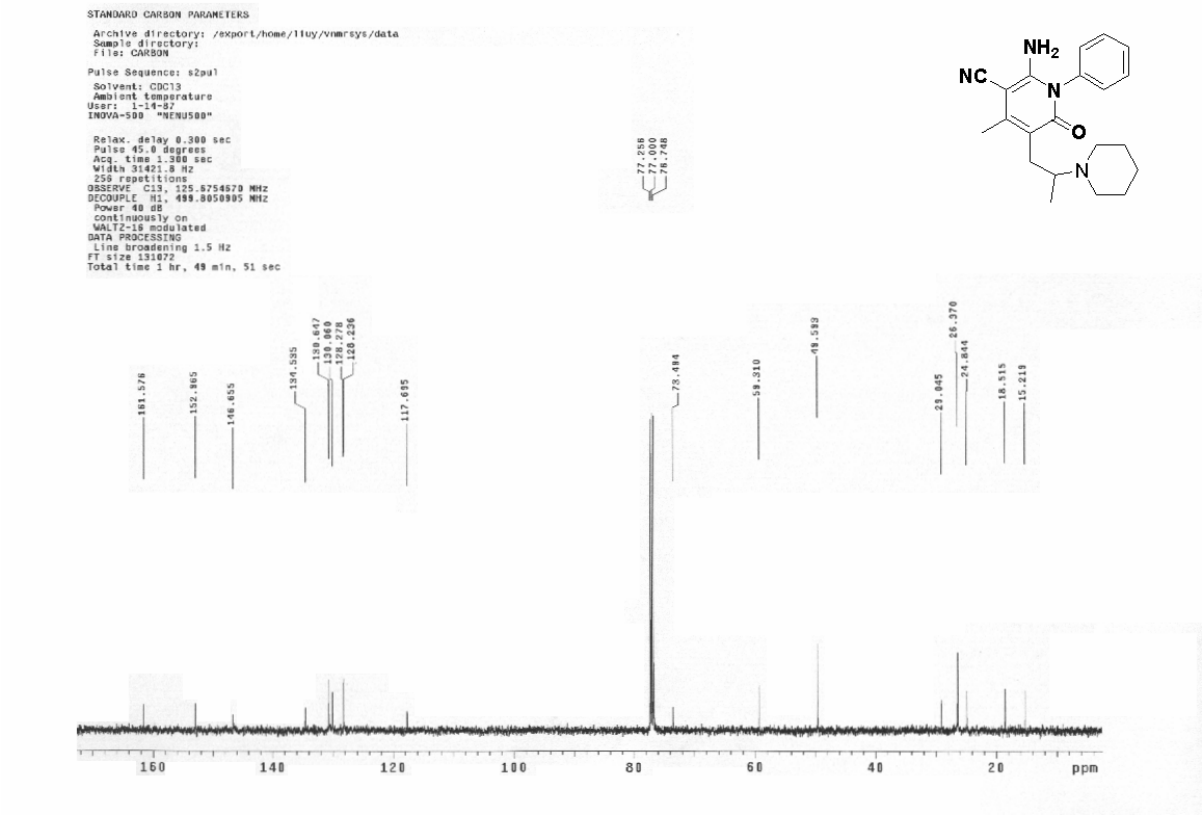
2d


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: y608
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7986.8 Hz
8 repetitions
OBSERVE H1, 499.8025655 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
NH2
CH3
NC
N
O
CH3
N
ppm

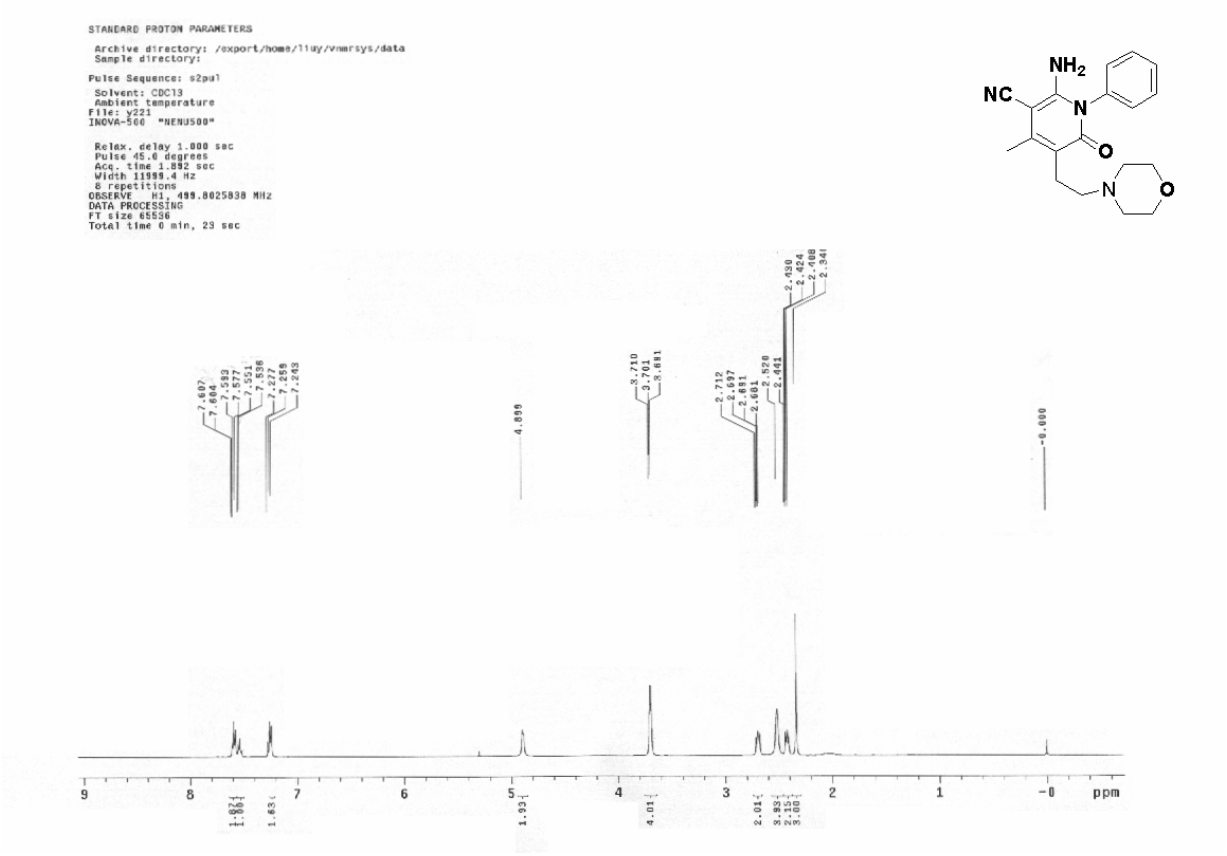

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
64 repetitions
OBSERVE C13, 125.6754752 MHz
DECOUPLE H1, 499.8059995 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 49 min, 51 sec
NH2
CH3
NC
N
O
CH3
N
ppm

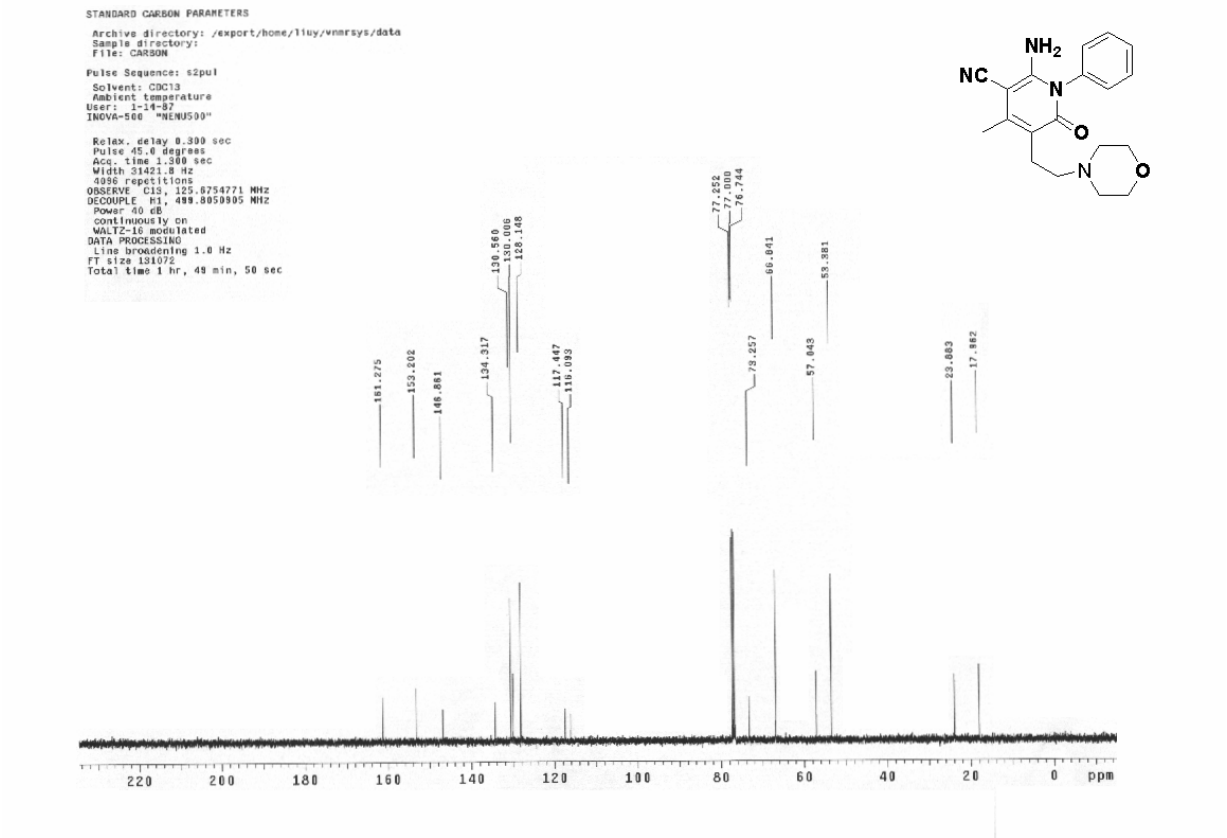
2f


STANDARD PROTON PARAMETERS
Archive directory: /export/home/lluy/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7998.8 Hz
Single scan
OBSERVE H1, 499.8025887 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 2 sec
NH2
NC
N
O
N
ppm

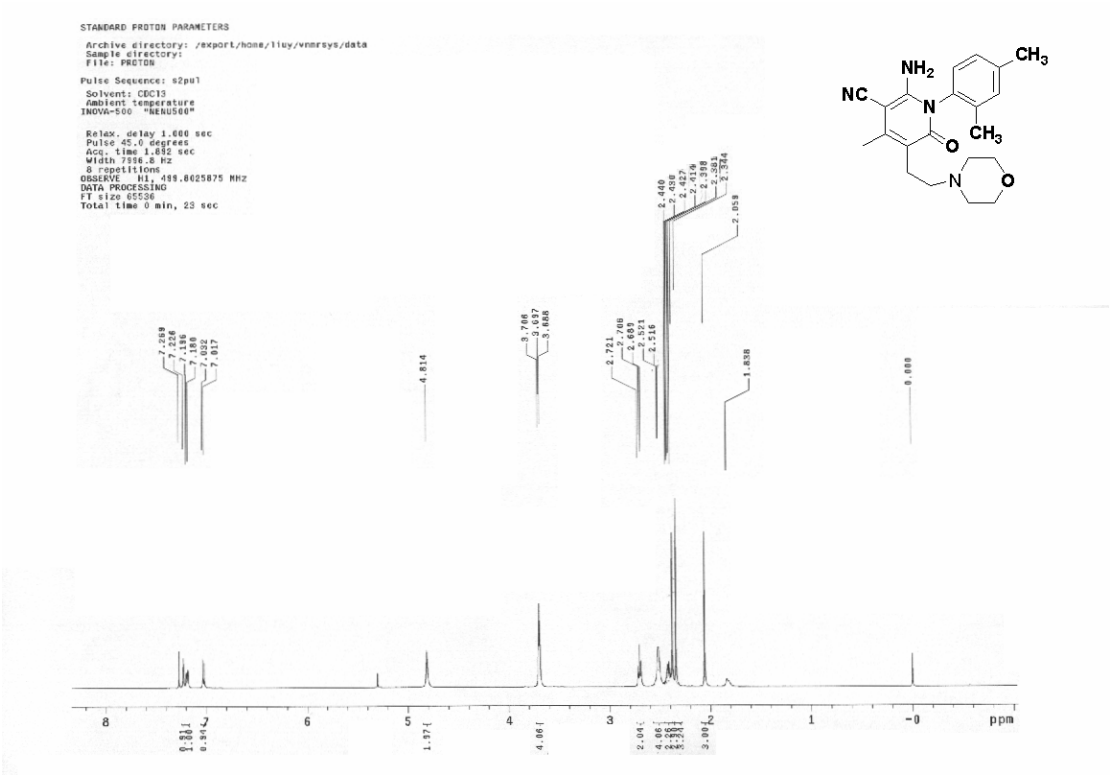

STANDARD CARBON PARAMETERS
Archive directory: /export/home/lluy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
250 repetitions
OBSERVE C13, 125.6754670 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 1 hr, 49 min, 51 sec
NH2
NC
N
O
N
ppm

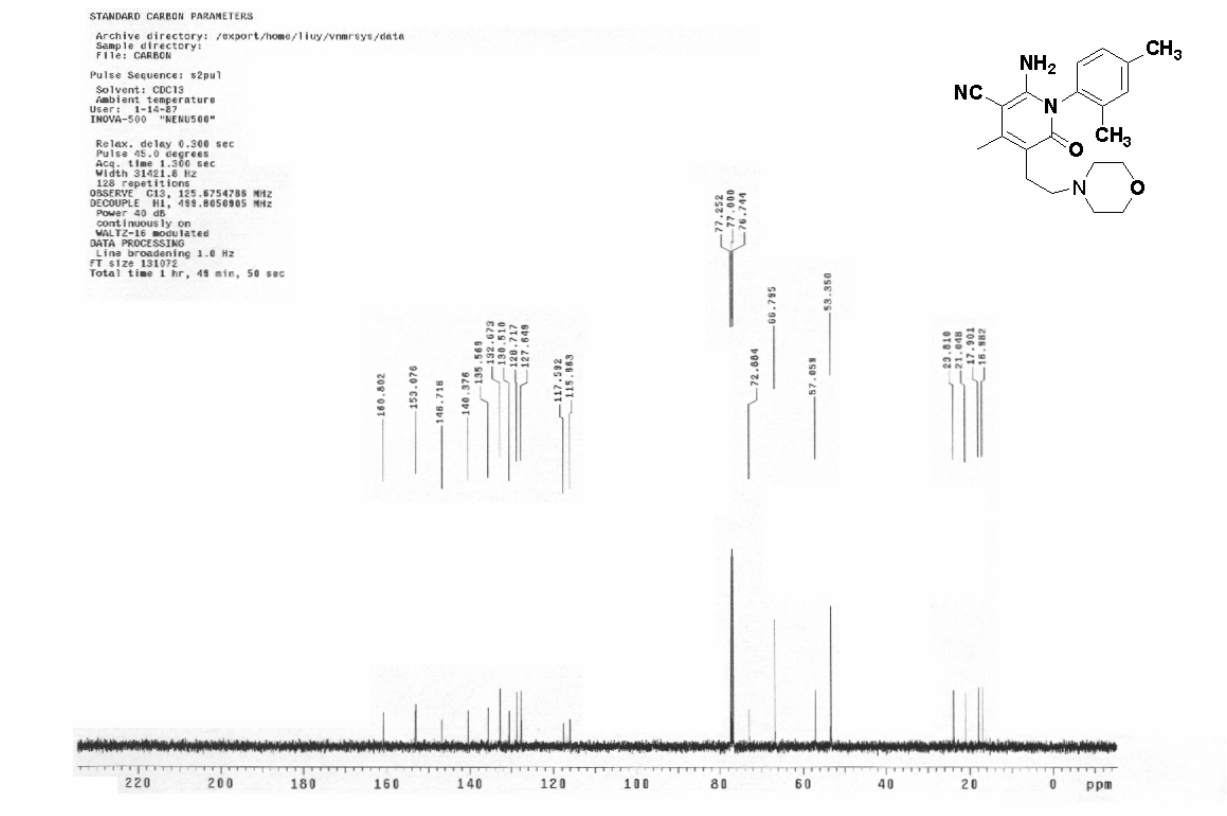
2g


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: y221
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 11989.4 Hz
8 repetitions
OBSERVE H1, 499.8025838 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
NH2
NC
N
O
N
O
7.607
7.604
7.573
7.572
7.551
7.536
7.277
7.259
7.243
4.899
3.710
3.701
3.681
2.712
2.697
2.681
2.661
2.520
2.442
2.430
2.424
2.408
2.341
-0.000
9
8
7
6
5
4
3
2
1
-0
ppm

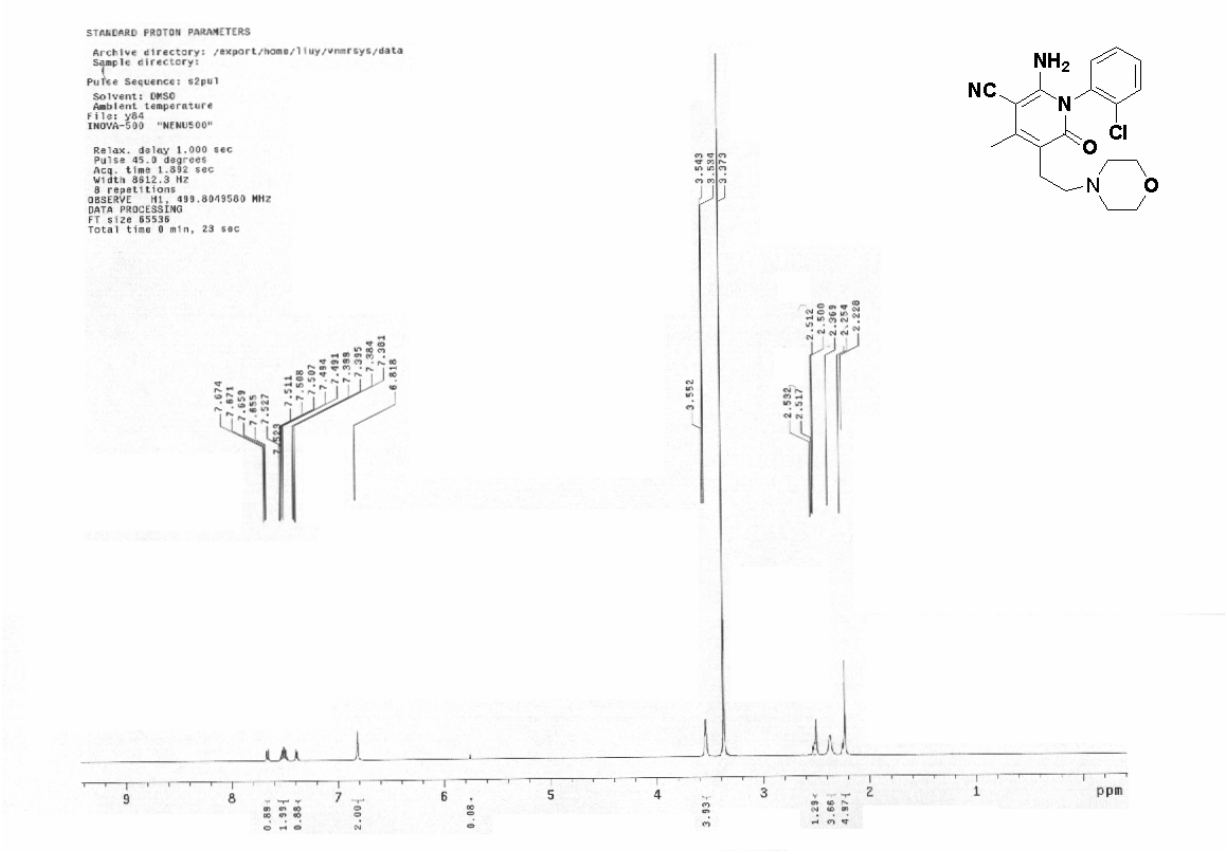

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
4096 repetitions
OBSERVE C13, 125.6754771 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 49 min, 50 sec
NH2
NC
N
O
N
O
161.275
153.202
146.861
134.317
130.560
130.006
128.148
117.447
110.093
77.458
77.000
76.744
73.257
66.841
57.043
53.381
23.083
17.862
220
200
180
160
140
120
100
80
60
40
20
0
ppm

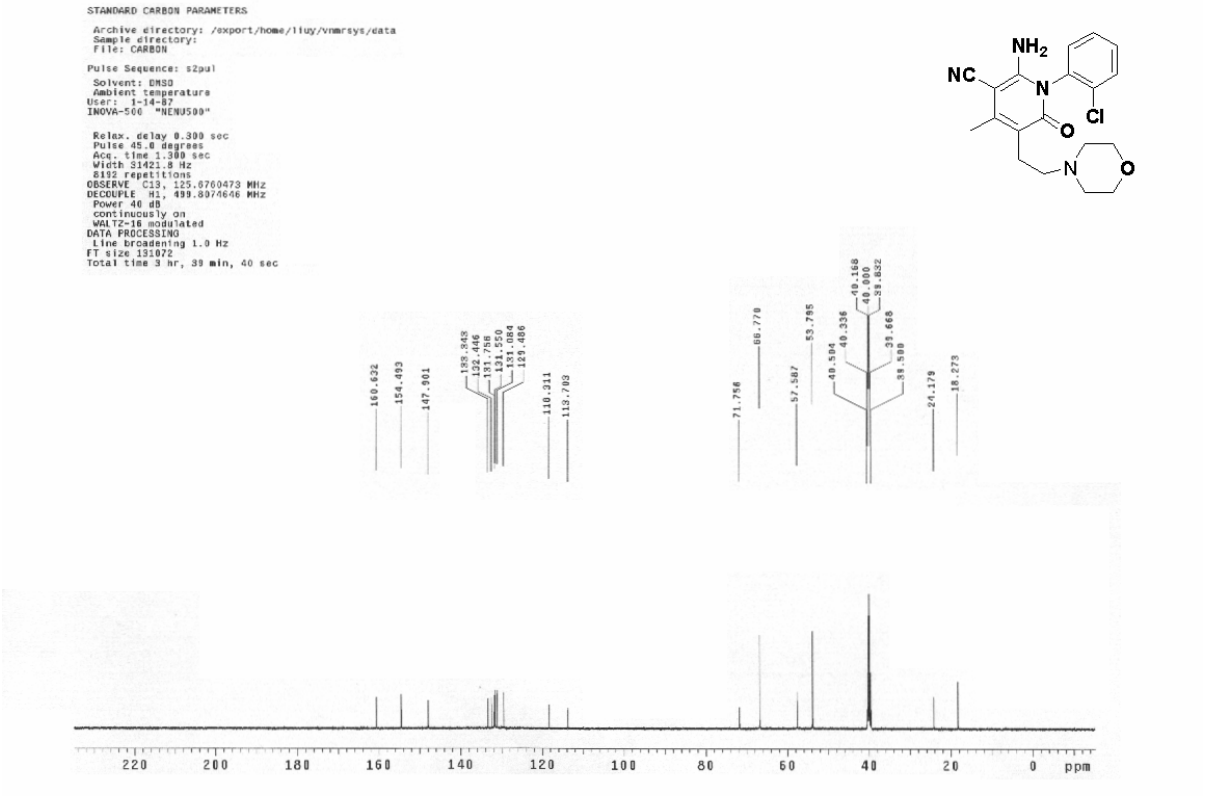
2h


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.882 sec
Width 7556.8 Hz
8 repetitions
OBSERVE H1, 499.8025875 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
NH2
CH3
NC
N
O
CH3
N
O
ppm

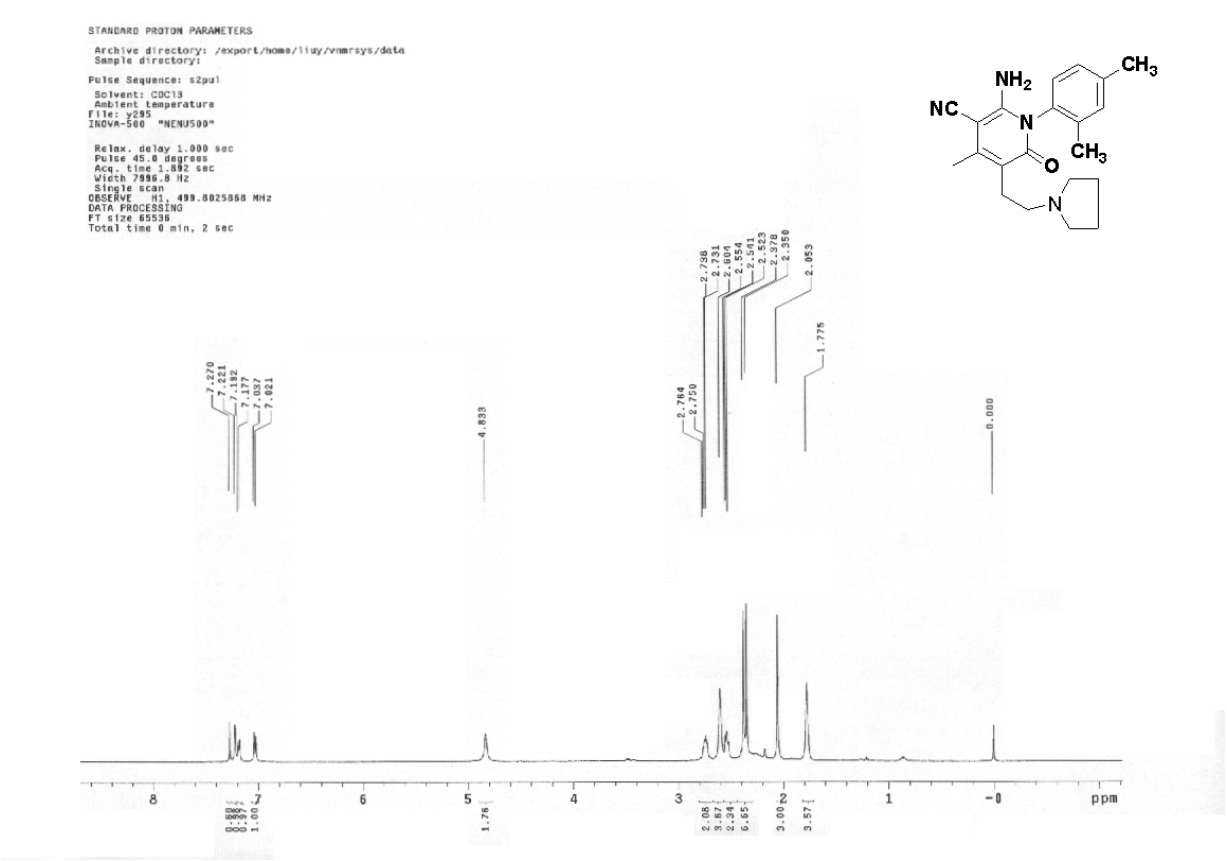

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.366 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754785 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 49 min, 50 sec
NH2
CH3
NC
N
O
CH3
N
O
ppm

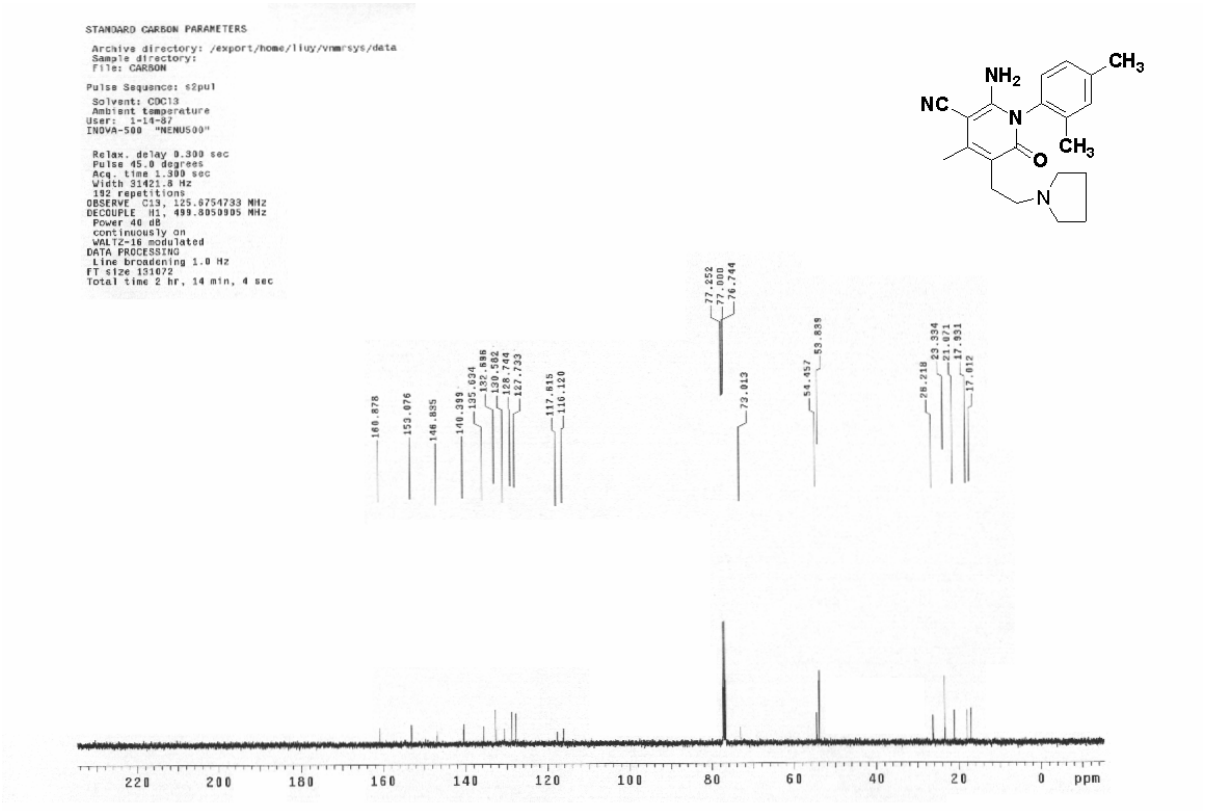
2i


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
File: y84
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8612.3 Hz
8 repetitions
OBSERVE H1, 499.8049580 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
NH2
NC
N
Cl
O
N
O
ppm

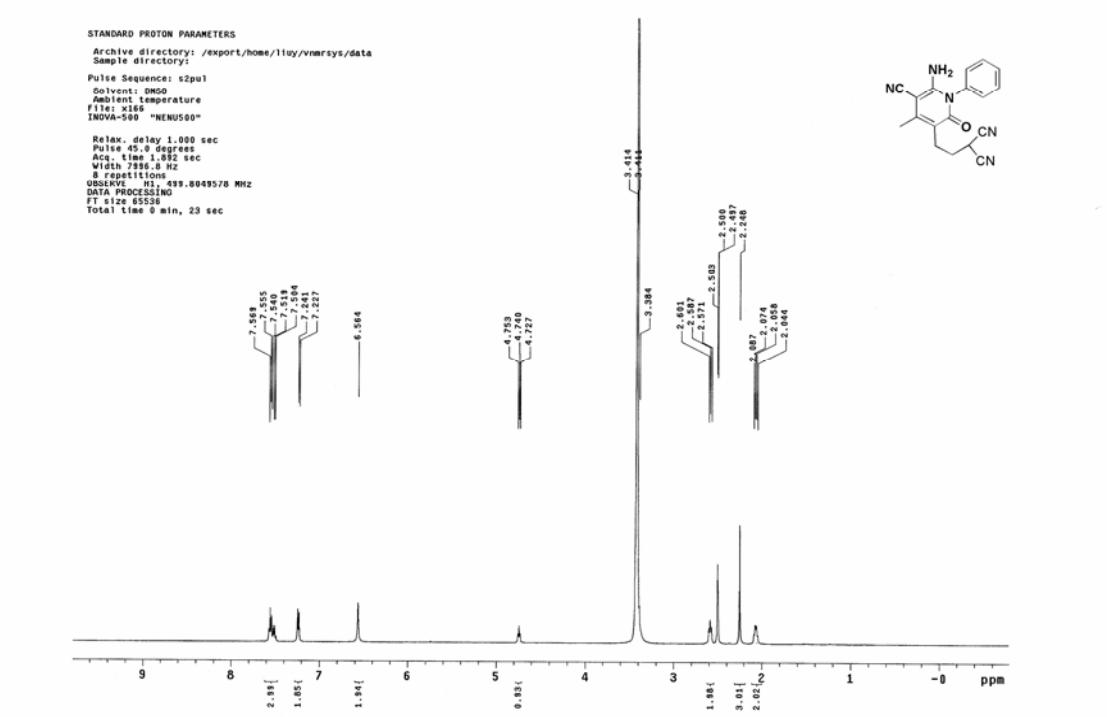

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
User: 1-14-07
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
8192 repetitions
OBSERVE C13, 125.6760473 MHz
DECOUPLE H1, 499.8074646 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 3 hr, 39 min, 40 sec
NH2
NC
N
Cl
O
N
O
ppm

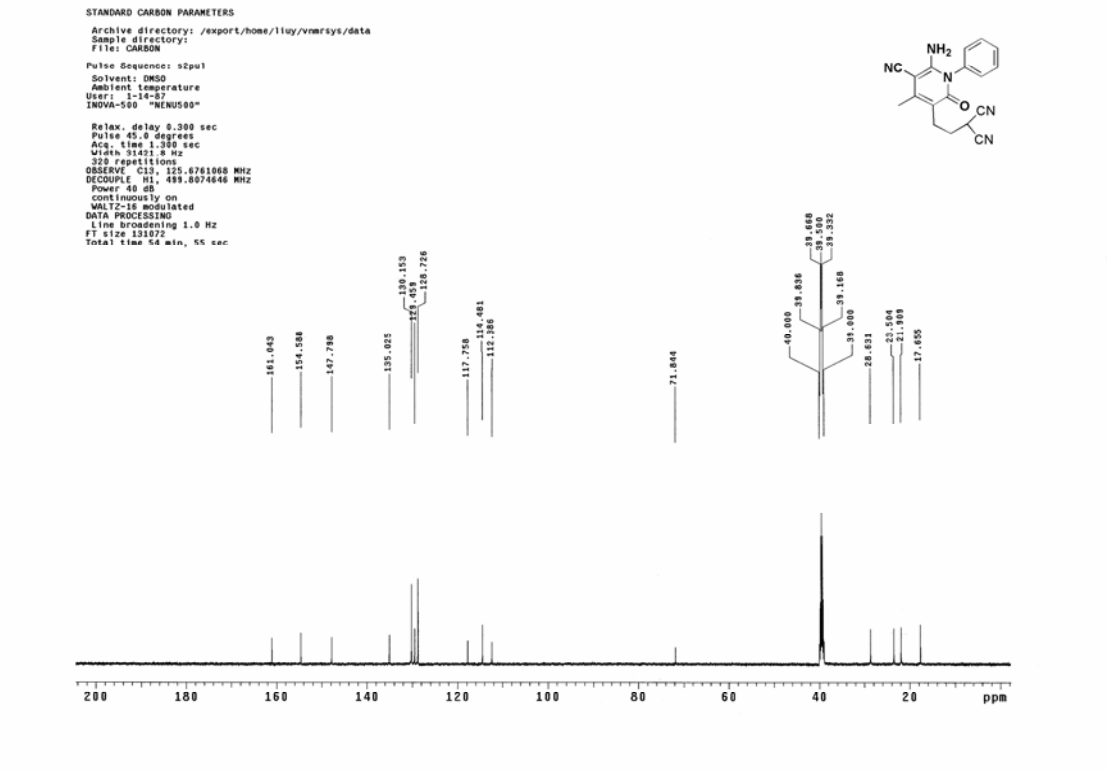
2j


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: y295
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7896.8 Hz
Single scan
OBSERVE H1, 499.8025868 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 2 sec
NH2
CH3
NC
N
O
CH3
N
ppm

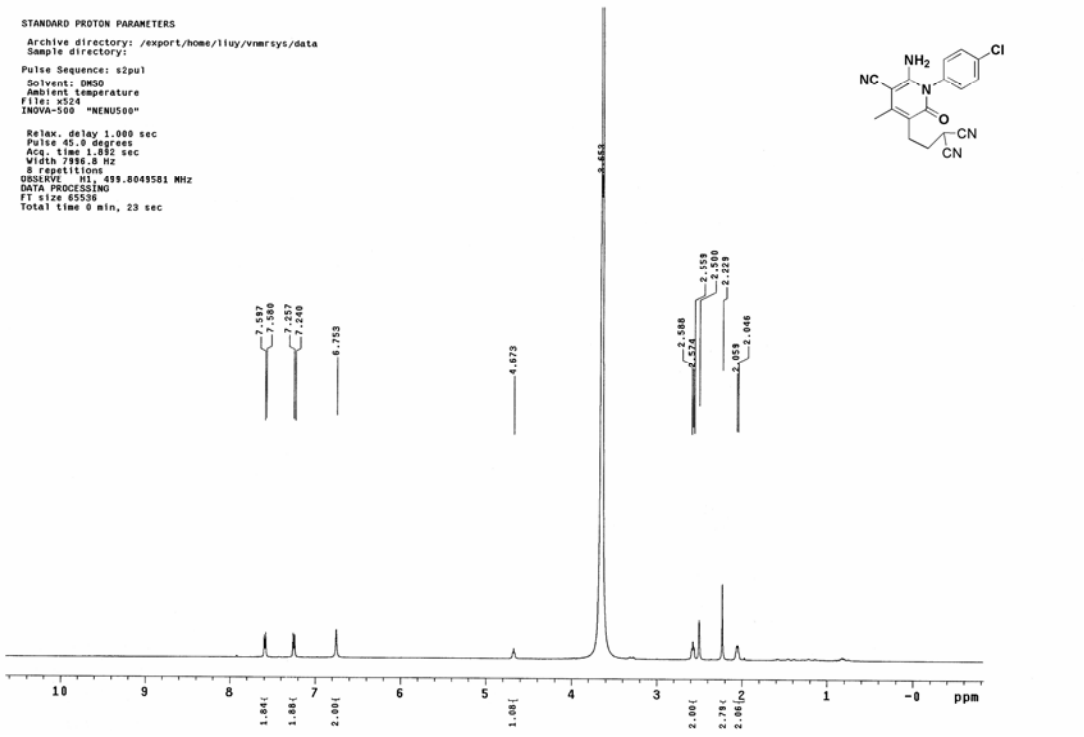

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-07
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
192 repetitions
OBSERVE C13, 125.6754733 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 2 hr, 14 min, 4 sec
NH2
CH3
NC
N
O
CH3
N
ppm

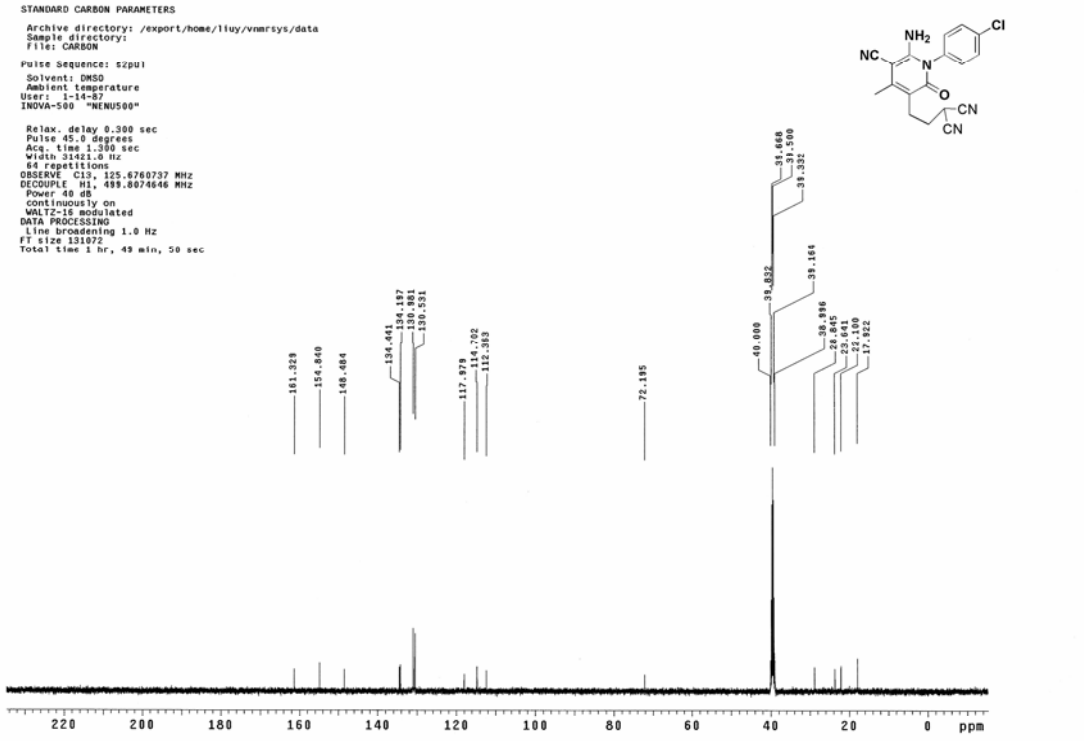
3a


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
File: x166
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7896.8 Hz
8 repetitions
OBSERVE H1, 499.8049578 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
NH2
NC
N
O
CN
CN
ppm

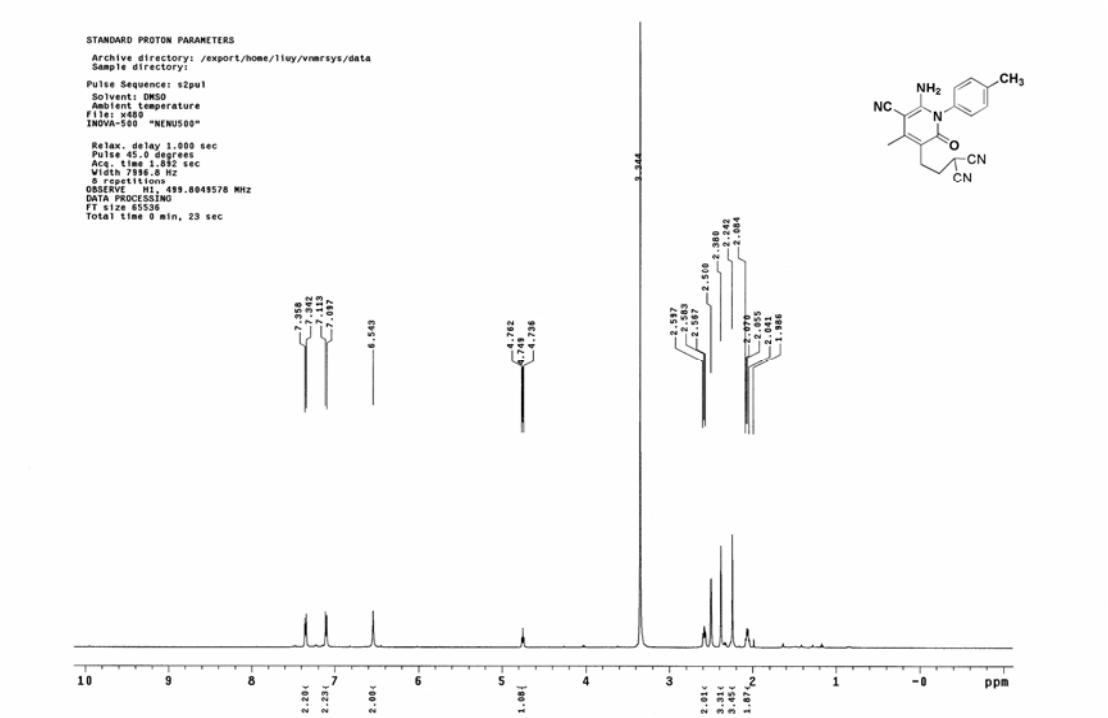

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
User: 1-14-07
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
320 repetitions
OBSERVE C13, 125.6761068 MHz
DECOUPLE H1, 499.8074646 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
NH2
NC
N
O
CN
CN
ppm

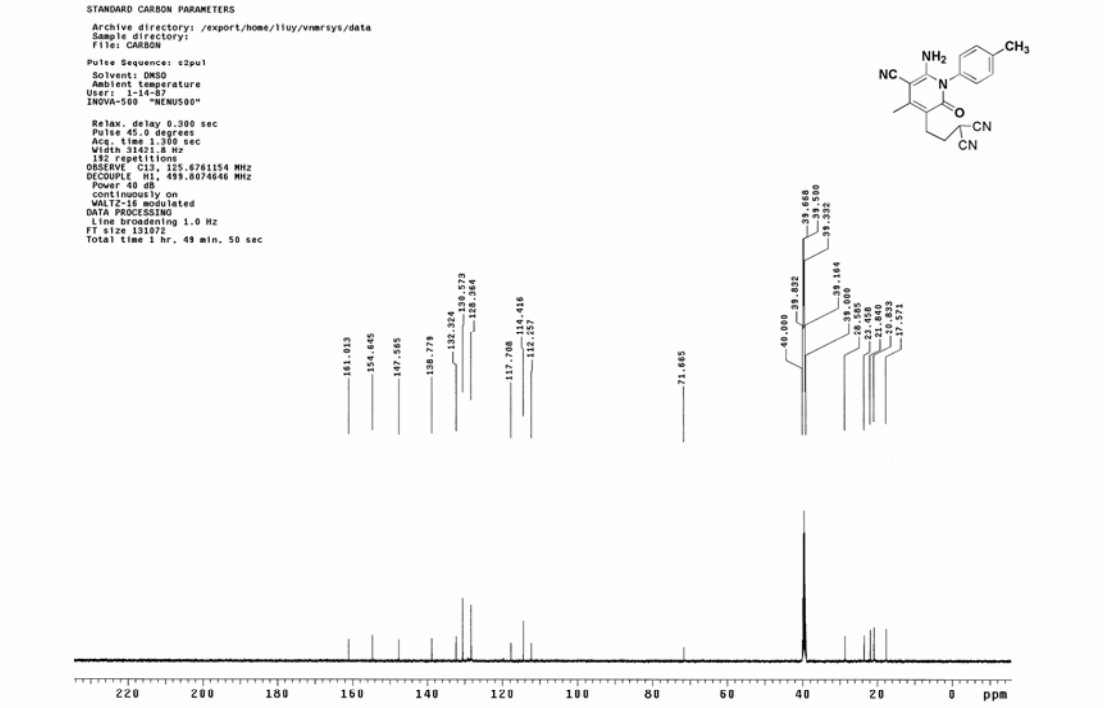
3b


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
File: x524
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.882 sec
Width 7996.8 Hz
8 repetitions
OBSERVE H1, 499.8049581 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
NH2
Cl
NC
N
O
CN
CN
7.597
7.580
7.257
7.240
6.753
4.673
3.453
2.559
2.500
2.328
2.588
2.574
2.059
2.046
1.04
1.08
2.00
1.08
2.00
2.79
2.06
10
9
8
7
6
5
4
3
1
-0
ppm

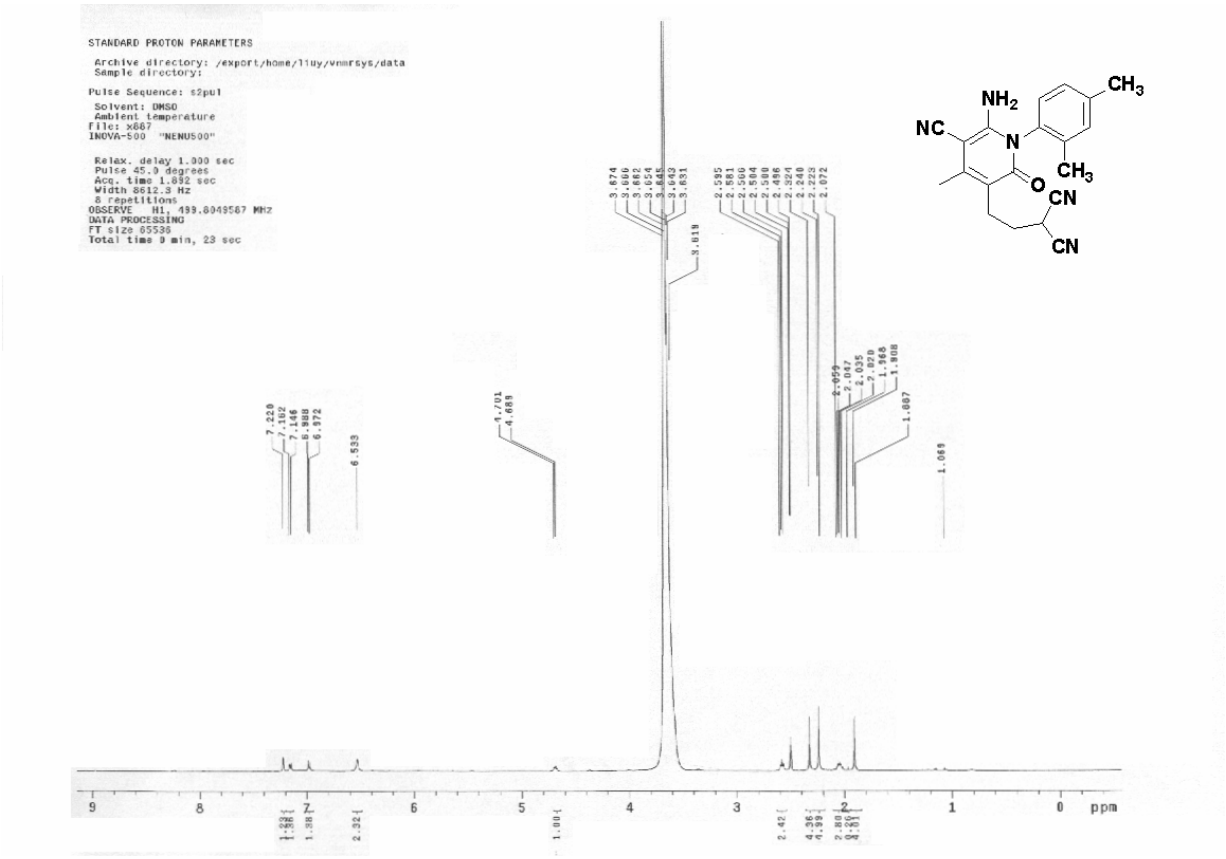

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.0 Hz
64 repetitions
OBSERVE C13, 125.6760737 MHz
DECOUPLE H1, 499.8074646 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 49 min, 50 sec
NH2
Cl
NC
N
O
CN
CN
161.329
154.840
148.484
134.441
134.187
130.981
130.591
117.979
114.702
112.383
72.195
40.000
39.832
39.666
39.500
39.332
39.164
38.998
28.845
23.641
22.100
17.922
220
200
180
160
140
120
100
80
60
40
20
0
ppm

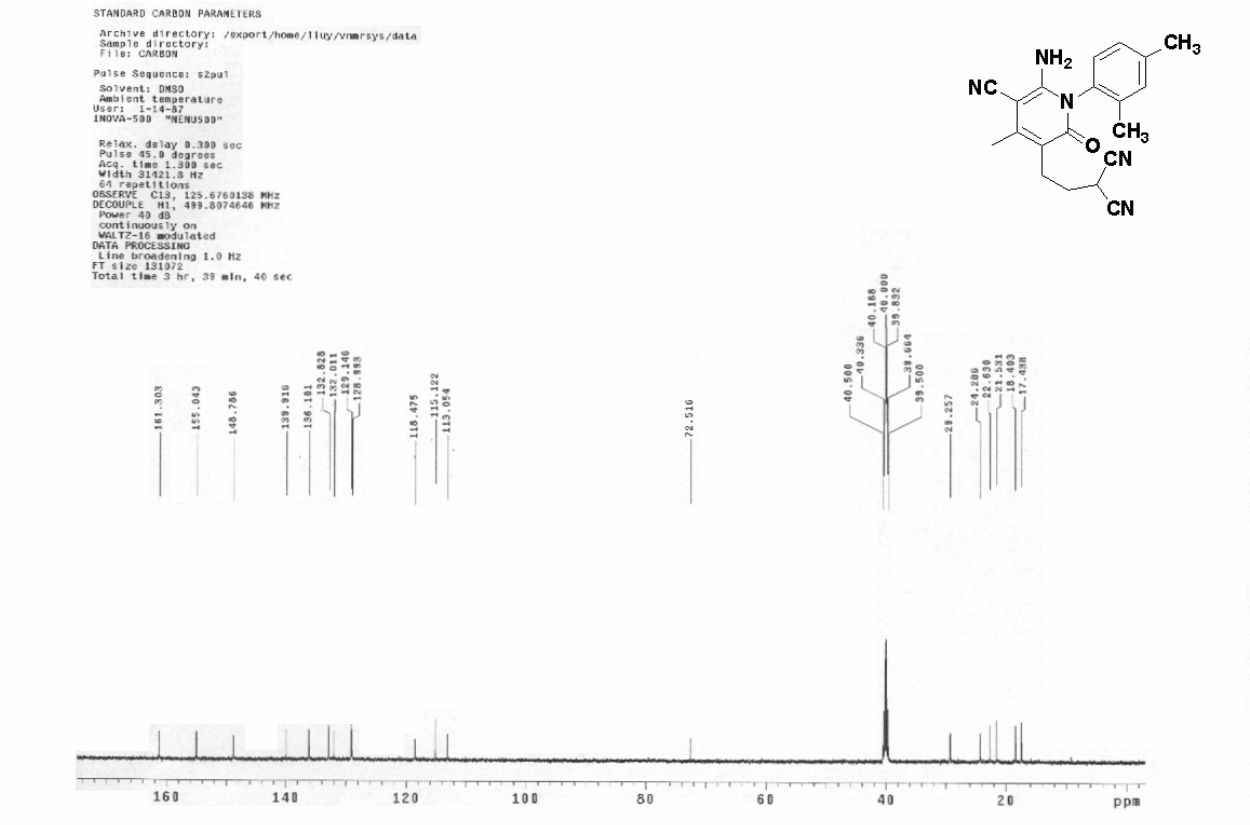
3c


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
File: x480
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7986.8 Hz
8 repetitions
OBSERVE H1, 499.8049578 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
NH2
CH3
NC
N
O
CN
CN
ppm

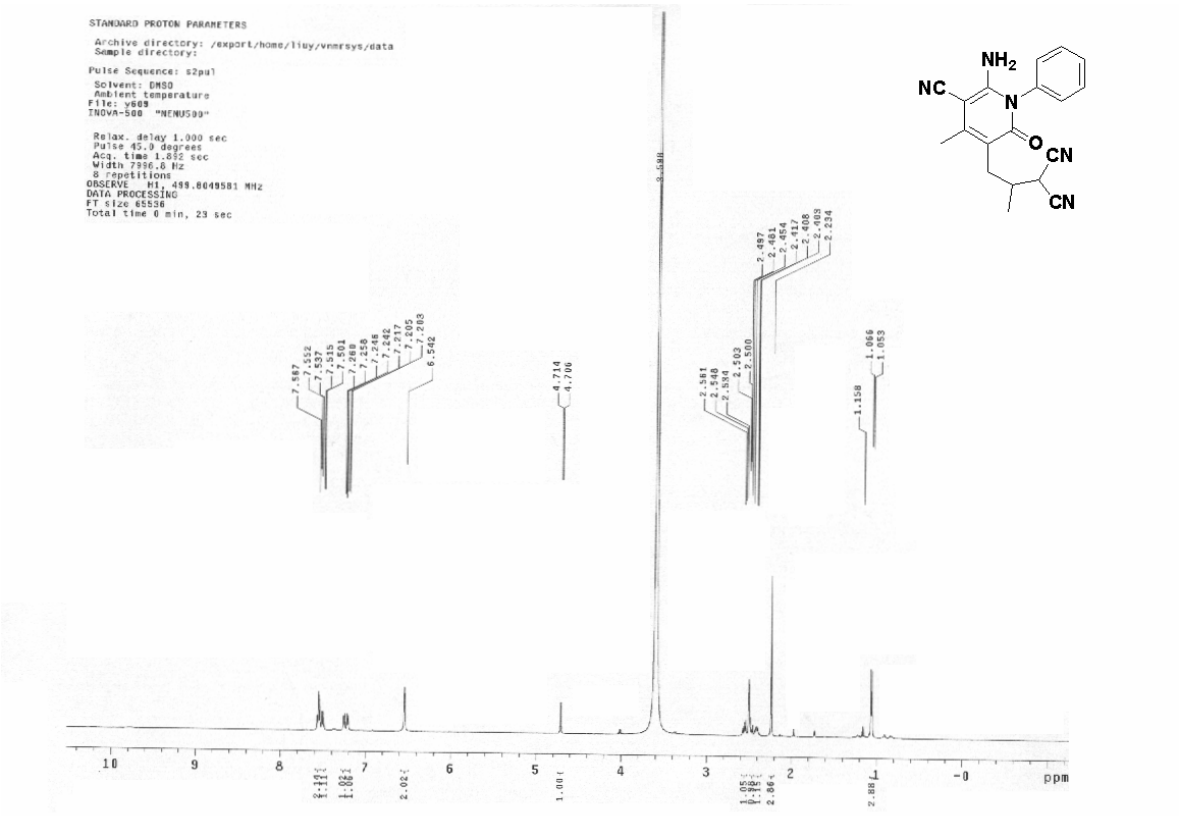

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
192 repetitions
OBSERVE C13, 125.6761154 MHz
DECOUPLE H1, 499.8074646 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 49 min, 50 sec
NH2
CH3
NC
N
O
CN
CN
ppm

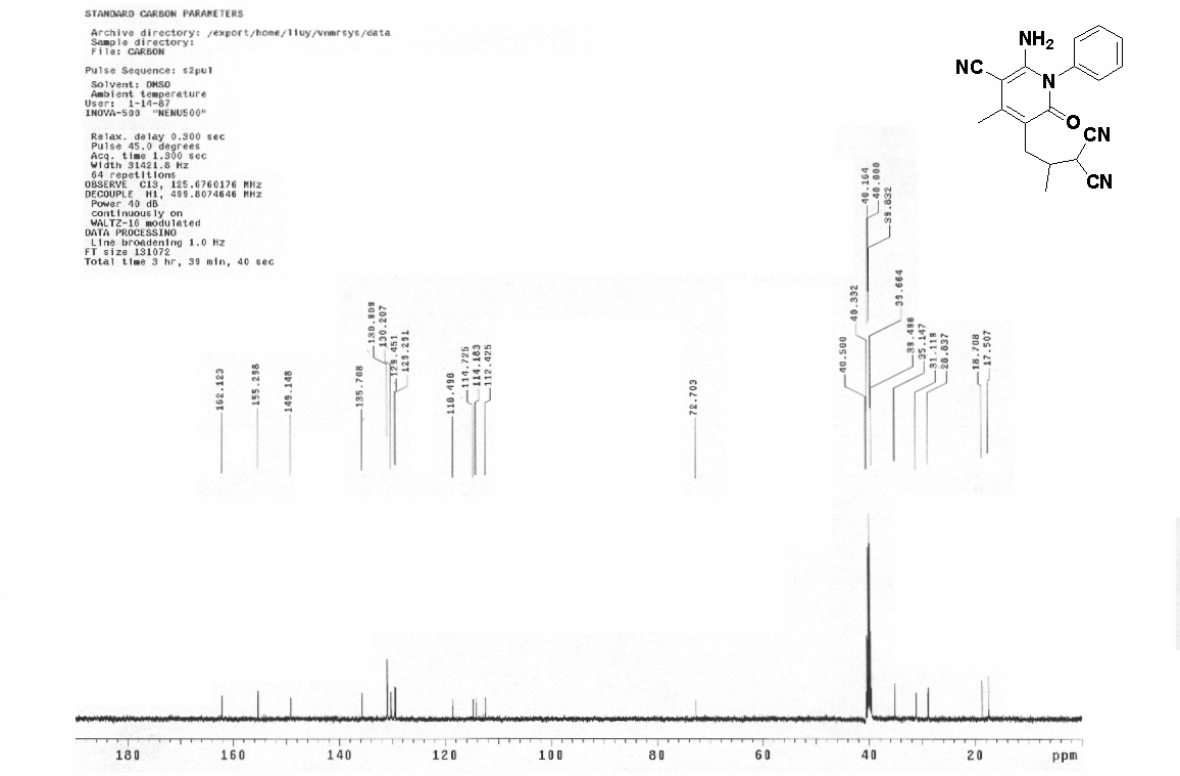
3d


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
File: x667
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8612.3 Hz
8 repetitions
OBSERVE H1, 499.8049587 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
NH2
CH3
NC
N
O
CN
CN
CH3
ppm

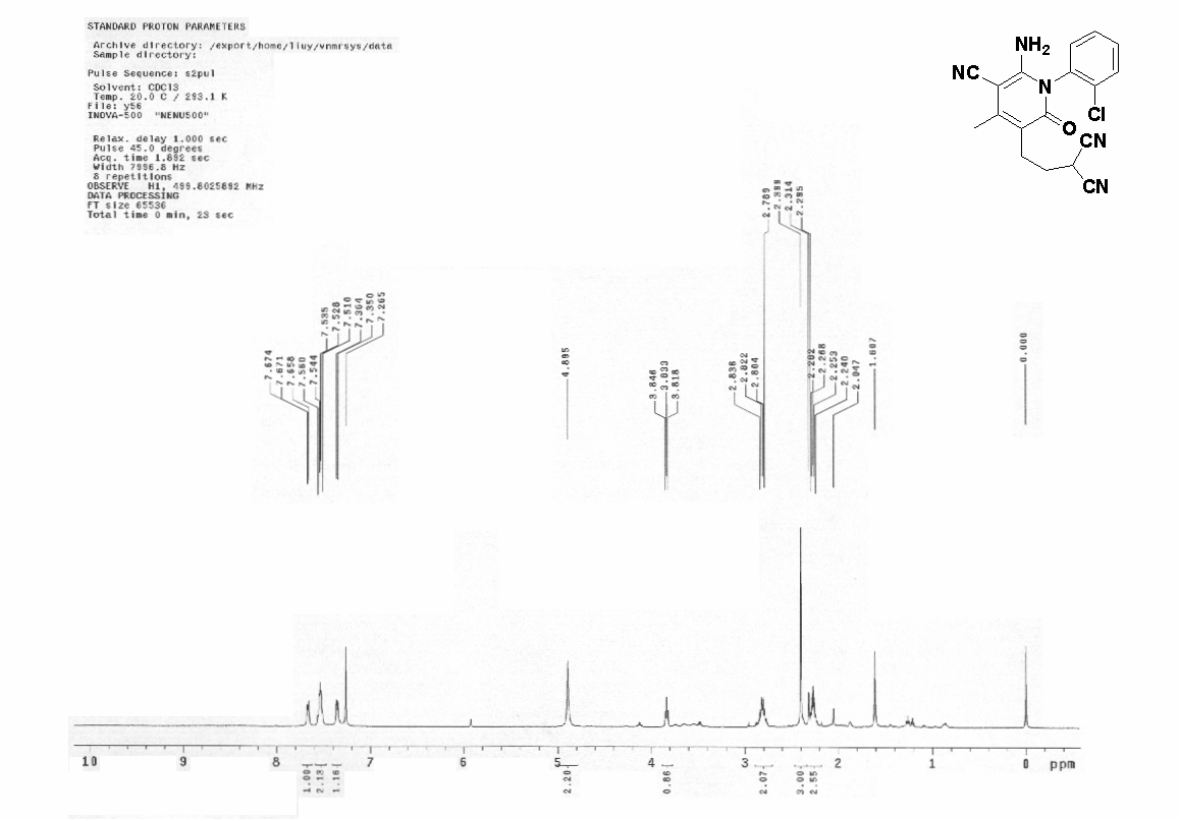

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
User: 1-14-57
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
64 repetitions
OBSERVE C13, 125.6760138 MHz
DECOUPLE H1, 499.8074646 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 3 hr, 39 min, 40 sec
NH2
CH3
NC
N
O
CN
CN
CH3
ppm

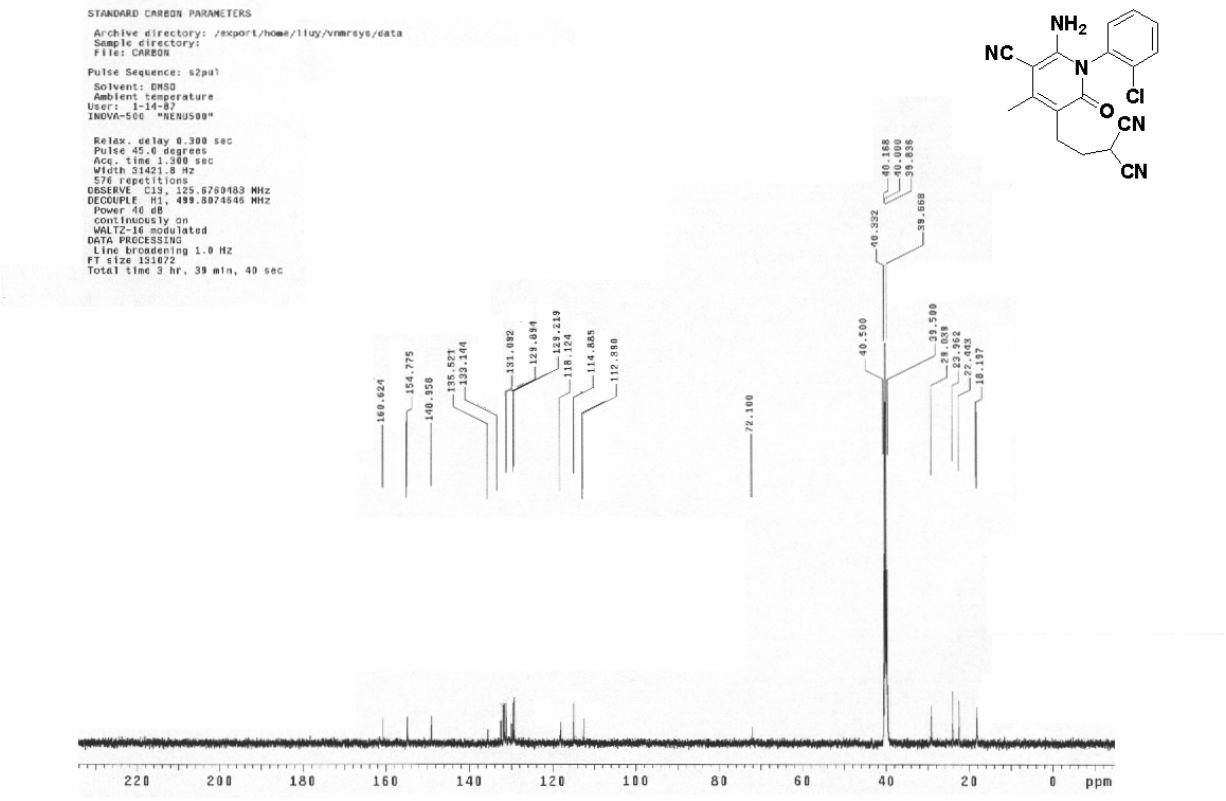
3e


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
File: y609
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7996.8 Hz
8 repetitions
OBSERVE H1, 499.8049581 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
NH2
NC
N
O
CN
CN
ppm

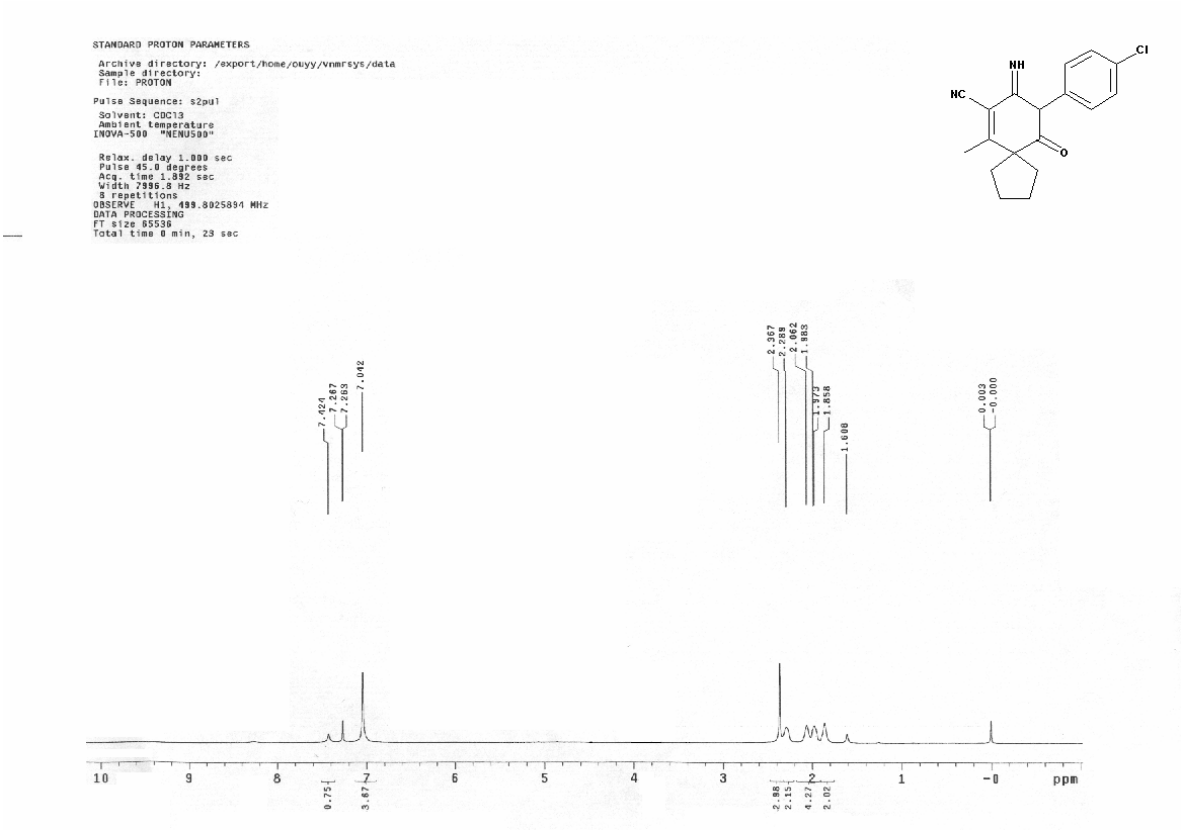

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
64 repetitions
OBSERVE C13, 125.6760176 MHz
DECOUPLE H1, 499.8074848 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 3 hr, 39 min, 40 sec
NH2
NC
N
O
CN
CN
ppm

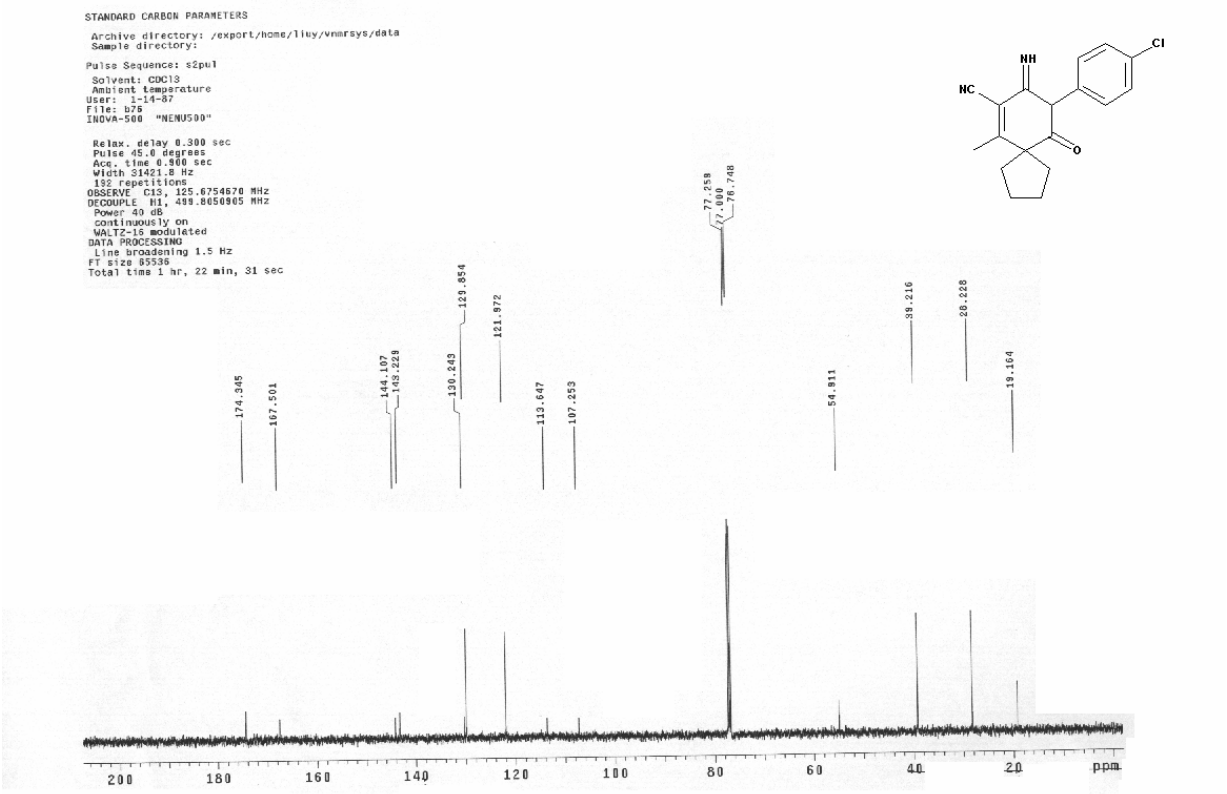
3f


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 20.0 C / 293.1 K
File: y56
INOVA-500 "NENUS00"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.882 sec
Width 7956.8 Hz
8 repetitions
OBSERVE H1, 499.8025692 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
NH2
NC
N
Cl
O
CN
CN
ppm

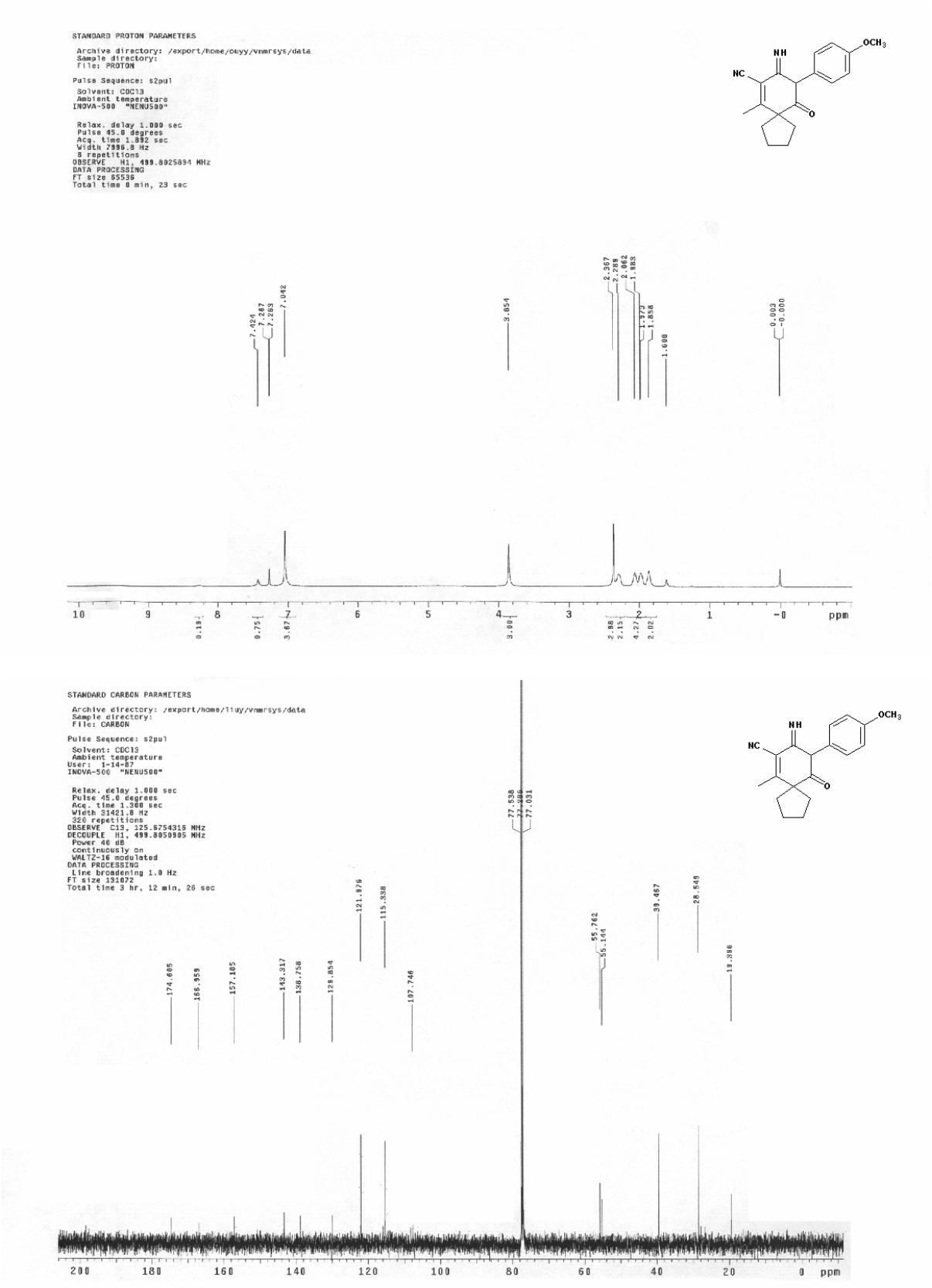

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
User: 1-14-87
INOVA-500 "NENUS00"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
576 repetitions
OBSERVE C13, 125.6760483 MHz
DECOUPLE H1, 499.8074646 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 3 hr, 39 min, 40 sec
NH2
NC
N
Cl
O
CN
CN
ppm

4b

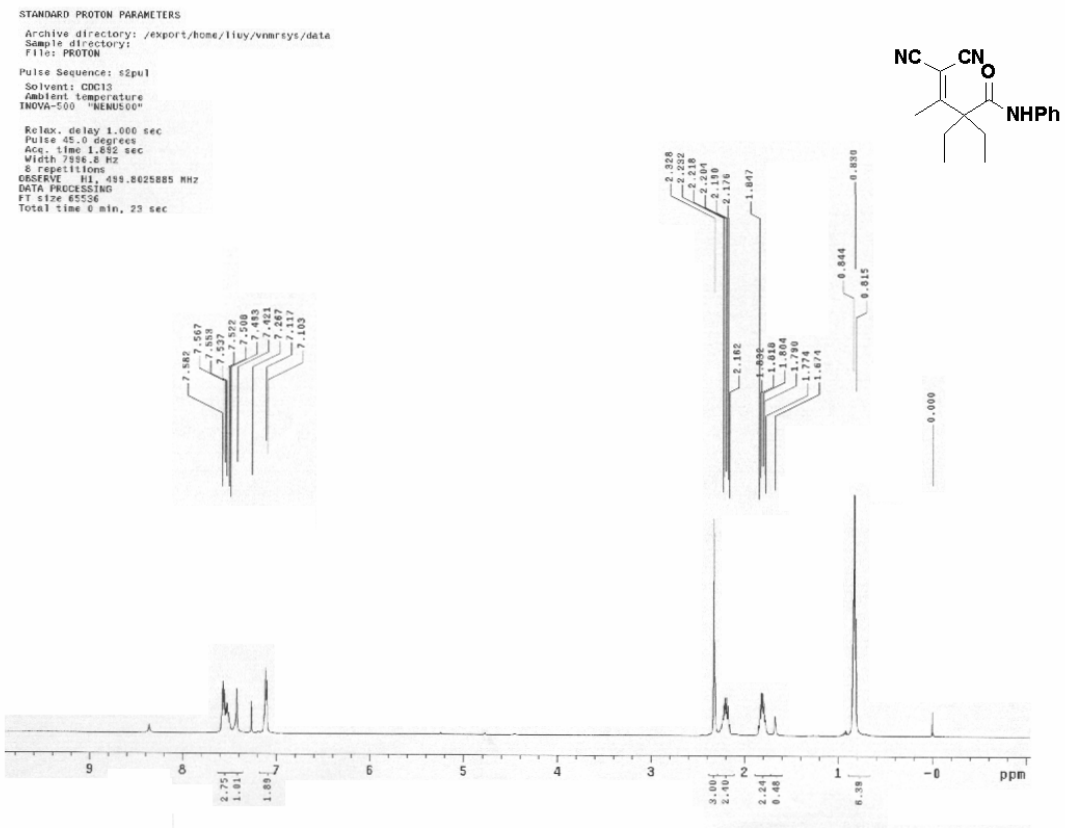

STANDARD PROTON PARAMETERS
Archive directory: /export/home/chyy/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7998.8 Hz
8 repetitions
OBSERVE H1, 499.8025894 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
NH
Cl
NC
O
7.424
7.287
7.263
7.042
2.367
2.288
2.062
1.883
1.973
1.858
1.608
0.000
0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm
0.75
3.87
2.88
2.15
4.27
2.02

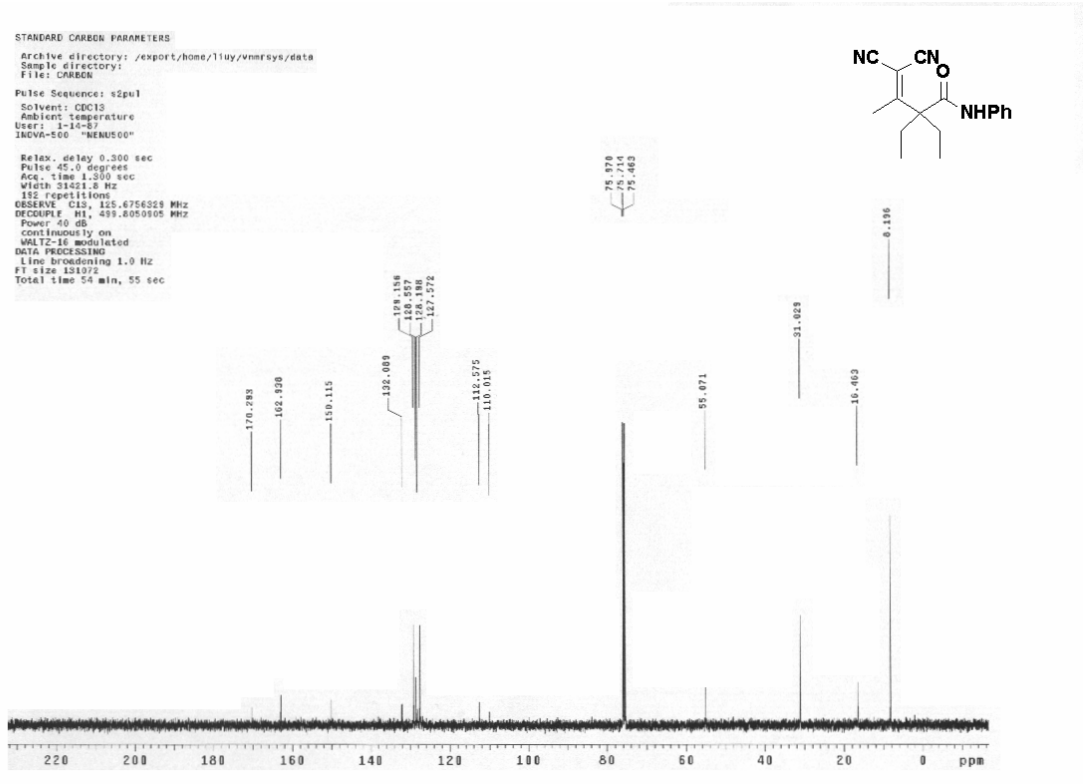

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-07
File: b75
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 0.900 sec
Width 31421.8 Hz
192 repetitions
OBSERVE C13, 125.6754670 MHz
DECOUPLE H1, 499.8650905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 65536
Total time 1 hr, 22 min, 31 sec
NH
Cl
NC
O
77.258
77.000
76.748
174.045
167.501
144.107
143.229
130.243
129.854
121.972
113.647
107.253
54.911
39.216
28.228
19.164
200
180
160
140
120
100
80
60
40
20
ppm

4c

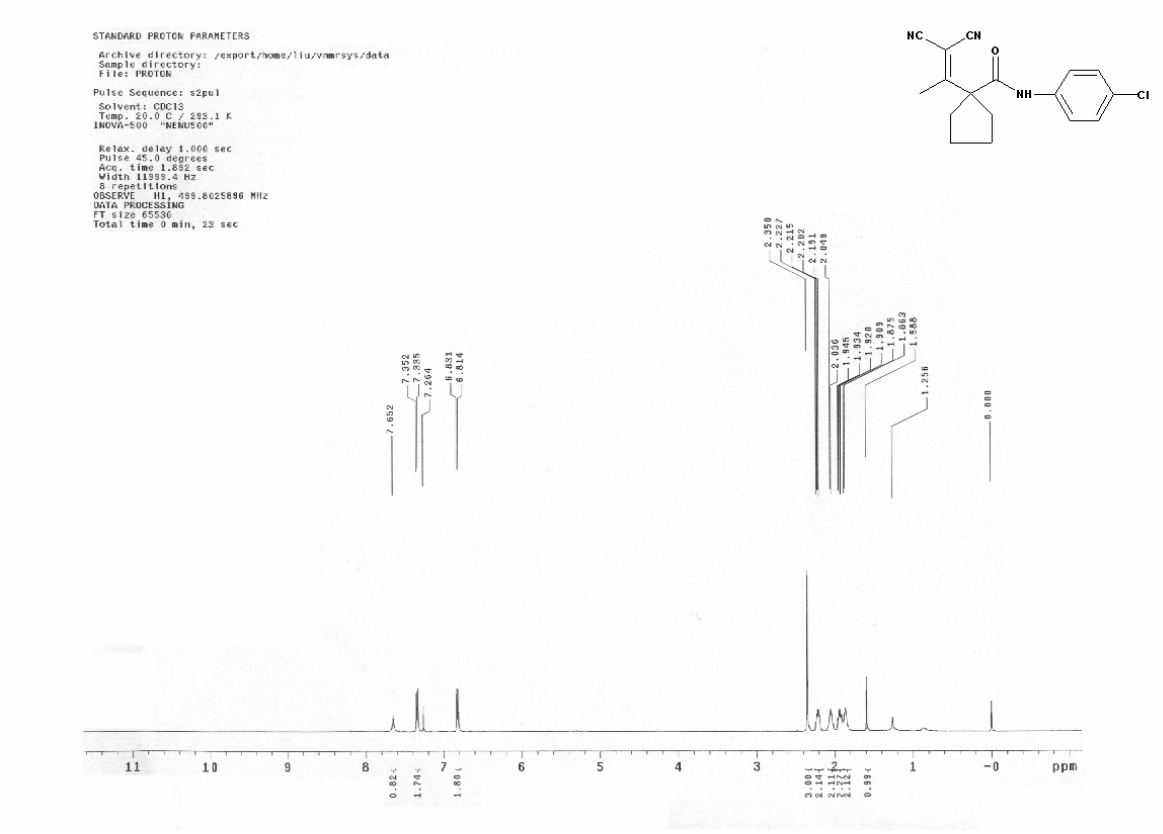

STANDARD PROTON PARAMETERS
Archive directory: /export/home/cuyy/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7986.8 Hz
8 repetitions
OBSERVE H1, 499.8025884 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
NH
NC
OCH3
O
7.424
7.287
7.263
7.042
3.854
2.067
2.028
2.062
1.983
1.970
1.858
1.688
0.003
0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm
0.19
0.75
3.67
3.00
2.08
2.15
4.27
2.02
STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.500 sec
Width 31421.8 Hz
320 repetitions
OBSERVE C13, 125.6754316 MHz
DECOUPLE H1, 499.8050905 MHz
Power 46 dB
continuously On
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 3 hr, 12 min, 26 sec
174.605
166.959
157.185
143.317
136.758
128.854
121.876
115.338
107.746
77.538
77.288
77.031
55.762
55.141
39.467
28.549
19.386
200
180
160
140
120
100
80
60
40
20
0
ppm

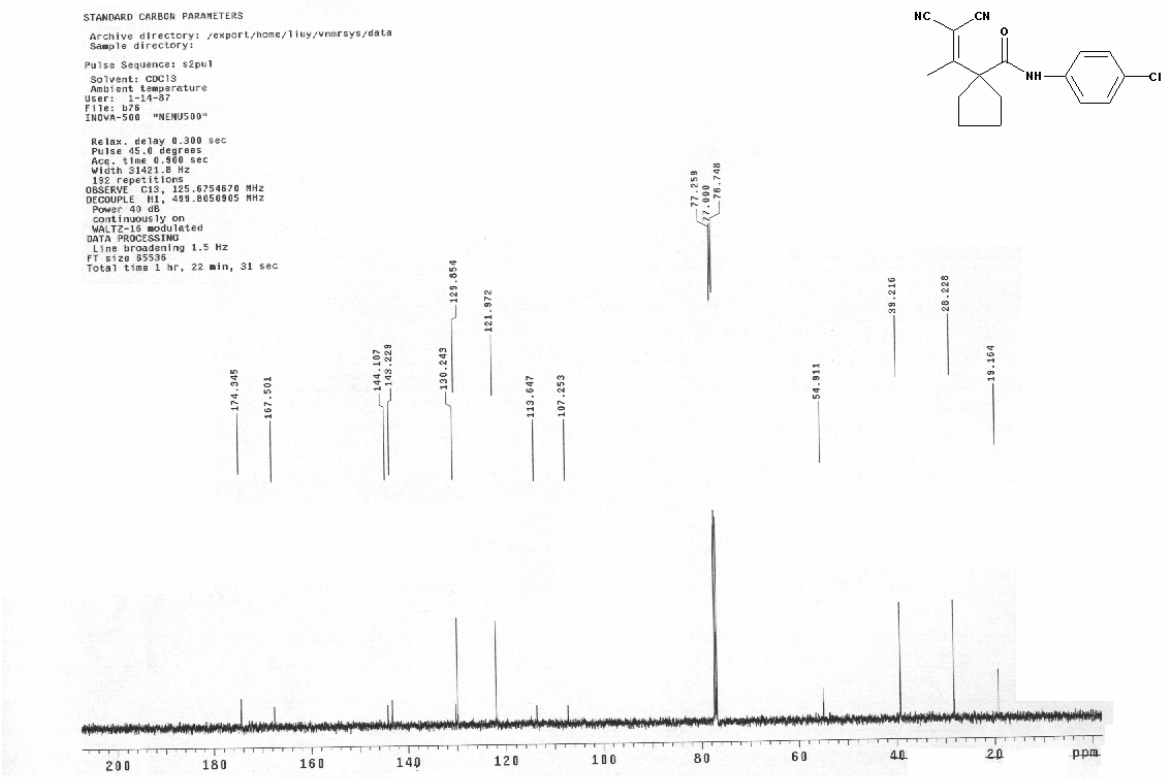
5a


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.852 sec
Width 7996.8 Hz
8 repetitions
OBSERVE H1, 499.8625885 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
NC
CN
O
NHPh
ppm

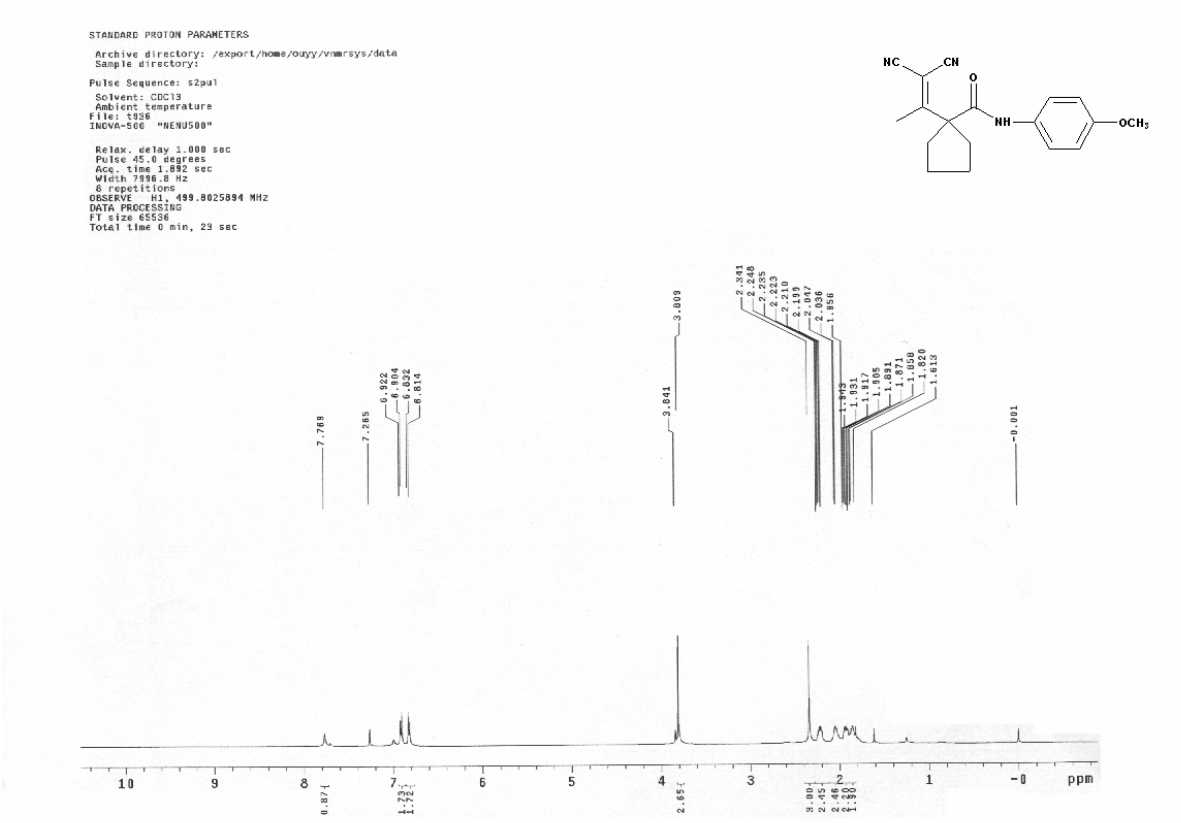

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31423.8 Hz
192 repetitions
OBSERVE C13, 125.6756328 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 54 min, 55 sec
NC
CN
O
NHPh
ppm

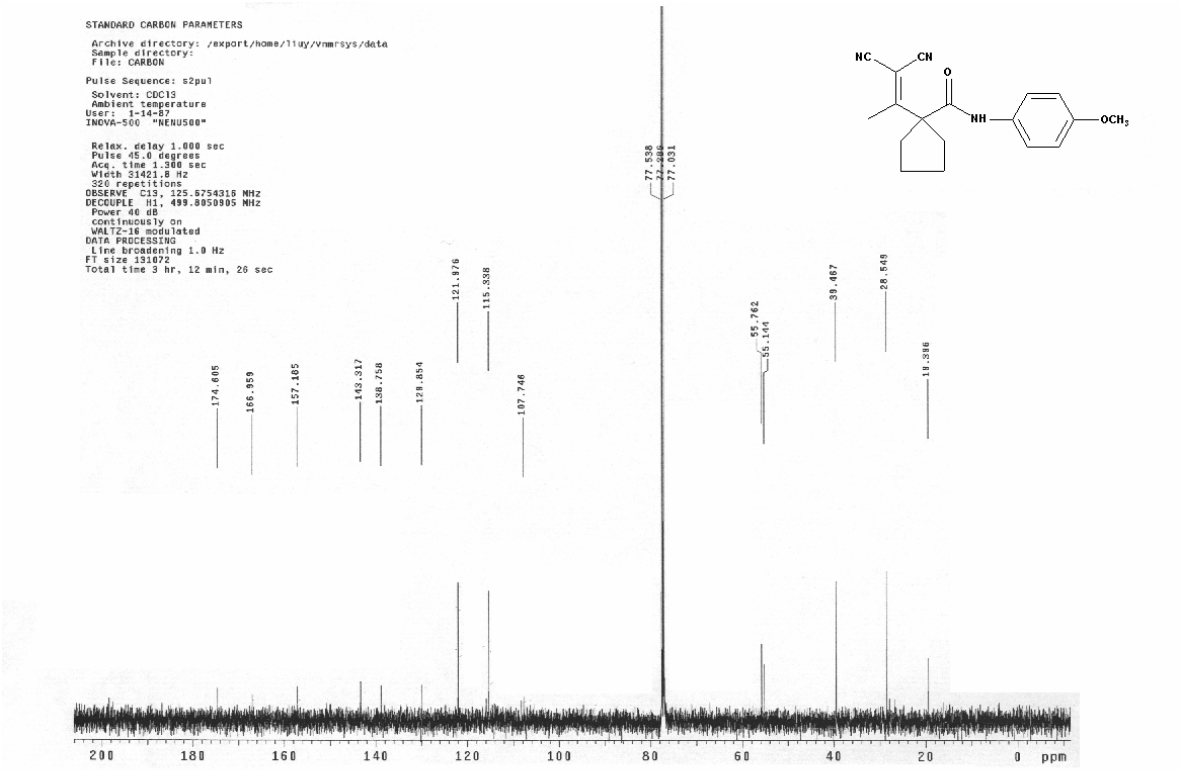
5b


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liu/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 20.0 C / 293.1 K
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 11993.4 Hz
8 repetitions
OBSERVE H1, 499.8025896 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
NC
CN
O
NH
Cl
ppm


STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: b76
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 0.900 sec
Width 31421.8 Hz
192 repetitions
OBSERVE C13, 125.6754670 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 65536
Total time 1 hr, 22 min, 31 sec
NC
CN
O
NH
Cl
ppm

5c


STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: t996
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.882 sec
Width 7996.8 Hz
8 repetitions
OBSERVE H1, 499.8025894 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 29 sec
NC
CN
O
NH
OCH3
7.768
7.265
6.922
6.904
6.832
6.814
3.841
3.809
2.341
2.248
2.235
2.223
2.210
2.195
2.047
2.036
1.956
1.903
1.881
1.817
1.805
1.691
1.671
1.660
1.620
1.613
-0.001
10
9
8
7
6
5
4
3
2
1
-0
ppm


STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
320 repetitions
OBSERVE C13, 125.6754318 MHz
DECOUPLE H1, 499.8050905 MHz
Power 46 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 3 hr, 12 min, 26 sec
NC
CN
O
NH
OCH3
174.606
166.859
157.485
143.317
138.758
128.854
121.976
115.338
107.746
77.538
77.286
77.031
55.762
55.144
39.467
28.549
18.386
200
180
160
140
120
100
80
60
40
20
0
ppm