Supporting Information

(Part I: Analytical Data)

Iron-catalyzed synthesis of polysubstituted pyrroles *via* [4C+1N] cyclization of 4-acetylenic ketones with primary amines

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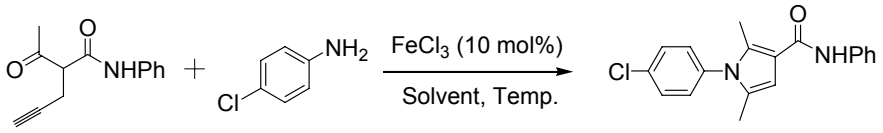
I. General

All reagents were purchased from commercial sources and used without treatment, unless otherwise indicated. The products were purified by column chromatography over silica gel. ^{1}H NMR and ^{13}C NMR spectra were recorded at 25 °C at 500 MHz and 125 MHz, respectively, with TMS as internal standard. IR spectra (KBr) were recorded on FTIR-spectrophotometer in the range of 400-4000 cm^{-1}. Elemental analysis (EA) was performed using a VarioEL analyzer. High-resolution mass spectra (HRMS) were obtained using a Bruker micrOTOF □ focus spectrometer (ESI).

II. Optimization of the reaction conditions



1a1 **2a1**

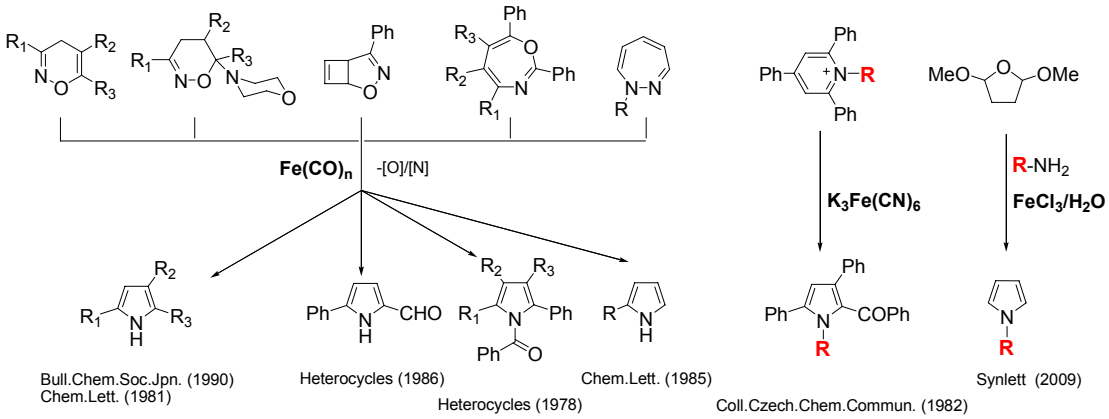
Entry	Solvent	Temperature (^{o}C)	Time (h)	Yield (%)[a]
1	toluene	rt	24	86
2	toluene	40	7	89
3	toluene	60	1.5	89
4	DCE	60	2	80
5	THF	60	3	45
6	DMF	60	9	30
7	CH_3CN	60	2	0

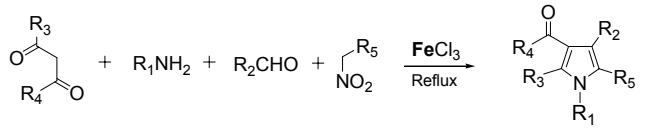
[a] Isolated yields.

Experimental Section

Typical procedure: To a solution of 2-acetyl-N-phenylpent-4-ynamide **1a1** (215 mg, 1 mmol) and 4-chloroaniline (152 mg, 1.2 mmol) in toluene (1.0 mL), $FeCl_3$ (17 mg, 0.1 mmol) was added. The reaction mixture was warmed to 60 ^{o}C and stirred until the staring material was consumed (monitored by TLC). Upon cooling to room temperature, the reaction mixture was quenched with 1M HCl (2 mL). The organic and aqueous layers were separated, and the aqueous layer was extracted with dichloromethane (3 × 10 mL). The combined organic layers were dried over $MgSO_4$ and filtered. The filtrate was concentrated in vacuo, and then the residue was purified by silica gel column chromatography (petroleum ether : diethyl ether = 10 : 3) to afford **2a1** (288 mg, 89% yield).

III. A summary on the iron-catalyzed reactions for the synthesis of pyrroles



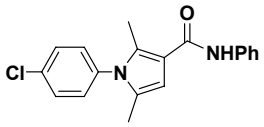


J. Org. Chem. (2010)

IV. Representative examples for the metal-catalyzed synthesis of pyrroles

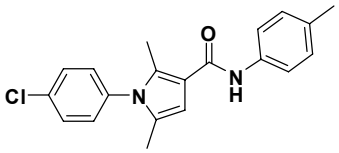
[**Au**] b) A. Saito, T. Konishi, Y. Hanzawa, *Org. Lett.* **2010**, *12*, 372–374; [**Ag**] c) S. Aggarwal, H.-J. Knölker, *Org. Biomol. Chem.* **2004**, *2*, 3060–3062; [**Pd**] d) R. Dhawan, B. A. Arndtsen, *J. Am. Chem. Soc.* **2004**, *126*, 468–469; [**Rh**] e) A. Mizuno, H. Kusama, N. Iwasawa, *Angew. Chem.* **2009**, *121*, 8468–8470; *Angew. Chem. Int. Ed.* **2009**, *48*, 8318; [**Ru**] f) V. Cadierno, J. Gimeno, N. Nebra, *Chem. Eur. J.* **2007**, *13*, 9973–9981; [**Cu**] g) R. Martin, M. R. Rivero, S. L. Buchwald, *Angew. Chem.* **2006**, *118*, 7237–7240; *Angew. Chem. Int. Ed.* **2006**, *45*, 7079–7082; [**In**] h) X. Liu, L. Huang, F. Zheng, Z. Zhan. *Adv. Synth. Catal.* **2008**, *350*, 2778-2788; [**Mg**] i) L. Lu, G. Chen, S. Ma, *Org. Lett.* **2006**, *8*, 835–838; [**Zr**] j) S. Zhang, X. Sun, W. -X. Zhang, Z. Xi, *Chem. Eur. J.* **2009**, *15*, 12608–12617; [**Co**] k) Y. C. Wong, K. Parthasarathy, C. Cheng, *J. Am. Chem. Soc.* **2009**, *131*, 18252–18253; [**Zn**] l) A. S. Demir, M. Emrullahoglu, G. Ardahan, *Tetrahedron* **2007**, *63*, 461–468; [**Bi**] m) S. Rivera, D. Bandyopadhyay, B. K. Banik, *Tetrahedron Lett.* **2009**, *50*, 5445–5448; [**Ni**] n) P. F. dos S. Filho, U. Schuchardt, *Angew. Chem.* **1977**, *16*, 559–572; [**Sc**] o) B. Zuo, J. Chen, M. Liu, J. Ding, H. Wu, W. Su, *J. Chem. Res.* **2009**, 14–16; [**Yb**] p) T. Sasada, T. Sawada, R. Ikeda, N. Sakai, T. Konakahara, *Eur. J. Org. Chem.* **2010**, 4237–4244.

V. Spectra analytical data of compounds **2**, **3** and **4**



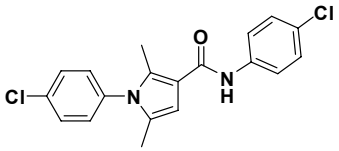
1-(4-Chlorophenyl)-2,5-dimethyl-*N*-phenyl-1*H*-pyrrole-3-carboxamide (**2a1**)

White solid: mp 174-176 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ = 2.02 (s, 3H), 2.35 (s, 3H), 6.20 (s, 1H), 7.08-7.11 (m, 1H), 7.15 (dd, *J* = 1.5, 6.5 Hz, 2H), 7.32-7.36 (m, 2H), 7.48 (dd, *J* = 1.5, 6.5 Hz, 2H), 7.48 (s, 1H), 7.60-7.61 (m, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ = 12.26, 12.72, 104.50, 114.54, 119.75, 123.57, 128.85, 128.92, 129.44, 129.68, 134.58, 134.98, 136.01, 138.51, 163.89; **IR** (KBr, cm^{-1}) 3309, 3056, 1643, 1595, 1534, 1492, 1433, 1405, 1306, 1242, 1089, 1025, 794, 756, 693.



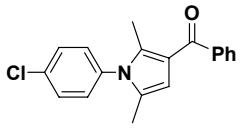
1-(4-Chlorophenyl)-2,5-dimethyl-*N*-*p*-tolyl-1*H*-pyrrole-3-carboxamide (**2a2**)

White solid: mp 184-185 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ = 2.00 (s, 3H), 2.32 (s, 3H), 2.34 (s, 3H), 6.19 (s, 1H), 7.13 (d, *J* = 8.0 Hz, 4H), 7.47 (d, *J* = 8.0 Hz, 4H), 7.50 (s, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ = 12.21, 12.67, 20.80, 104.54, 114.63, 119.83, 128.71, 129.35, 129.42, 129.63, 133.06, 134.50, 134.74, 135.94, 136.04, 163.83; **IR** (KBr, cm^{-1}) 3347, 3285, 1631, 1597, 1492, 1406, 1311, 1249, 1089, 1030, 805, 769, 528.



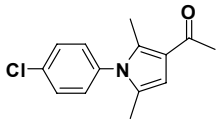
N,1-Bis(4-chlorophenyl)-2,5-dimethyl-1*H*-pyrrole-3-carboxamide (**2a3**)

White solid: mp 190-191 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ = 2.00 (s, 3H), 2.33 (s, 3H), 6.18 (s, 1H), 7.14 (dd, *J* = 2.5, 7.0 Hz, 2H), 7.27 (dd, *J* = 2.5, 7.0 Hz, 2H), 7.48 (dd, *J* = 2.0, 7.0 Hz, 2H), 7.50 (s, 1H), 7.56 (dd, *J* = 2.0, 7.0 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ = 12.25, 12.66, 104.51, 114.33, 120.99, 128.39, 128.85, 128.96, 129.42, 129.71, 134.68, 135.21, 135.98, 137.21, 163.84; **IR** (KBr, cm^{-1}) 3305, 1634, 1574, 1492, 1403, 1089, 825, 507.



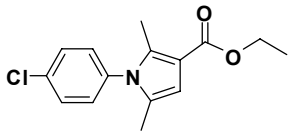
(1-(4-Chlorophenyl)-2,5-dimethyl-1*H*-pyrrol-3-yl)(phenyl)methanone (**2a4**)

White solid: mp 157-158 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ = 1.99 (s, 3H), 2.33 (s, 3H), 6.21 (d, *J* = 1.0 Hz, 1H), 7.18 (dd, *J* = 2.0, 6.5 Hz, 2H), 7.43-7.51 (m, 5H), 7.83 (dd, *J* = 1.0, 8.0 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ = 12.61, 12.95, 110.07, 119.72, 127.91, 128.22, 128.98, 129.32, 129.69, 131.01, 137.63, 135.89, 137.11, 140.66, 192.27; **IR** (KBr, cm^{-1}) 3050, 1631, 1717, 1493, 1409, 1251, 1099, 837, 731, 714, 697; **Elemental analysis** (%) Calcd for $C_{19}H_{16}ClNO$: C, 73.66; H, 5.21; N, 4.52; Found: C, 73.71; H, 5.24; N, 4.49; **HRMS** (ESI-TOF) Calcd for $C_{19}H_{17}ClNO$ $[M+H]^+$ 310.1005; Found 310.0993.



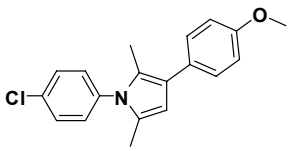
1-(1-(4-Chlorophenyl)-2,5-dimethyl-1*H*-pyrrol-3-yl)ethanone (**2a5**)

Yellow oil; **^{1}H NMR** (500 MHz, $CDCl_3$) δ = 1.99 (s, 3H), 2.31 (s, 3H), 2.42 (s, 3H), 6.33 (s, 1H), 7.13 (m, 2H), 7.49 (m, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ = 12.63, 12.85, 28.64, 108.22, 120.63, 128.47, 129.35, 129.69, 134.66, 135.58, 135.82, 195.02; **IR** (KBr, cm^{-1}) 3727, 3059, 1654, 1521, 1495, 1411, 1225, 1092, 1013, 344, 842, 651, 502.



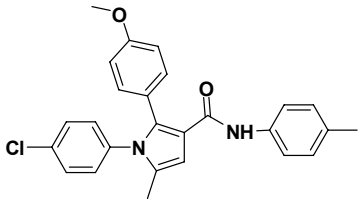
Ethyl 1-(4-chlorophenyl)-2,5-dimethyl-1*H*-pyrrole-3-carboxylate (**2a6**)

Yellow oil; **^{1}H NMR** (500 MHz, $CDCl_3$) δ = 1.35 (t, *J* = 7.0 Hz, 3H), 1.97 (s, 3H), 2.28 (s, 3H), 4.28 (q, *J* = 7.0 Hz, 2H), 6.37 (s, 1H), 7.13 (d, *J* = 8.5 Hz, 2H), 7.47 (d, *J* = 8.5 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ = 12.32, 12.64, 14.53, 59.32, 107.71, 111.71, 128.60, 129.46, 129.64, 134.51, 136.04, 136.17, 165.58; **IR** (KBr, cm^{-1}) 3726, 3626, 1696, 1495, 1383, 1281, 1084, 1049, 673.



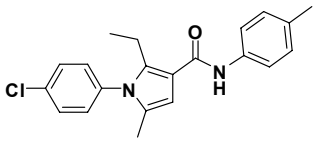
1-(4-Chlorophenyl)-3-(4-methoxyphenyl)-2,5-dimethyl-1*H*-pyrrole (**2a7**)

Yellow solid: mp 119-120 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ = 2.06 (s, 3H), 2.11 (s, 3H), 3.83 (s, 3H), 6.10 (s, 1H), 6.93 (d, *J* = 7.0 Hz, 2H), 7.21 (dd, *J* = 2.0, 7.0 Hz, 2H), 7.35 (dd, *J* = 2.0, 7.0 Hz, 2H), 7.45 (dd, *J* = 2.0, 7.0 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ = 12.09, 12.82, 55.24, 106.85, 113.76, 120.99, 124.41, 128.36, 128.86, 129.36, 129.61, 133.60, 137.36, 157.40; **IR** (KBr, cm^{-1}) 3727, 2167, 1494, 1243, 1090, 1042, 834, 797, 673.



1-(4-Chlorophenyl)-2-(4-methoxyphenyl)-5-methyl-*N*-*p*-tolyl-1*H*-pyrrole-3-carboxam -ide (**2b1**)

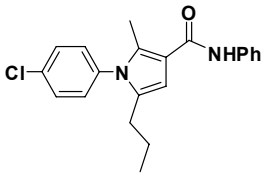
Yellow solid: mp 198-199 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ = 2.09 (s, 3H), 2.25 (s, 3H), 3.80 (s, 3H), 6.64 (s, 1H), 6.84 (d, *J* = 8.5 Hz, 2H), 6.99 (d, *J* = 8.5 Hz, 2H), 7.01 (d, *J* = 8.0 Hz, 2H), 7.09 (d, *J* = 8.0 Hz, 2H), 7.13 (s, 1H), 7.17 (d, *J* = 8.0 Hz, 2H), 7.27 (dd, *J* = 2.0, 8.0 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ = 12.92, 20.75, 55.24, 108.52, 114.19, 117.94, 119.12, 123.07, 129.14, 129.24, 129.69, 130.21, 132.65, 132.80, 133.32, 133.79, 135.88, 136.38, 159.74, 162.67; **IR** (KBr, cm^{-1}) 3389, 3282, 1656, 1602, 1517, 1492, 1252, 1177, 1028, 848, 812, 640; **Elemental analysis** (%) Calcd for $C_{26}H_{23}ClN_2O_2$: C, 72.47; H, 5.38; N, 6.50; Found: C, 72.53; H, 5.44; N, 6.51; **HRMS** (ESI-TOF) Calcd for $C_{26}H_{24}ClN_2O_2$ $[M+H]^+$ 431.1511; Found 431.1521; Calcd for $C_{26}H_{23}ClN_2NaO_2$ $[M+Na]^+$ 453.1342; Found 453.1340.



1-(4-Chlorophenyl)-2-ethyl-5-methyl-*N*-*p*-tolyl-1*H*-pyrrole-3-carboxamide (**2b2**)

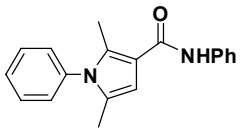
White solid: mp 178-179 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ = 1.02 (t, *J* = 7.0 Hz, 3H), 1.98 (s, 3H), 2.32 (s, 3H), 2.79 (q, *J* = 7.0 Hz, 2H), 6.17 (s, 1H), 7.13(d, *J* = 8.0 Hz, 2H), 7.17 (d, *J* = 8.0 Hz, 2H), 7.42 (s, 1H), 7.48 (d, *J* = 8.0 Hz, 2H), 7.49 (d, *J* = 8.0 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ = 12.57, 14.59, 18.98, 20.76, 104.45,

113.84, 119.80, 128.70, 129.34, 129.55, 129.64, 133.02, 134.64, 135.95, 136.07, 141.10, 163.42; **IR** (KBr, cm^{-1}) 3278, 1633, 1596, 1492, 1268, 1245, 1089, 854, 736.



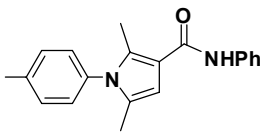
1-(4-Chlorophenyl)-2-methyl-*N*-phenyl-5-propyl-1*H*-pyrrole-3-carboxamide (**2c**)

White solid: mp 169-170 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ = 0.80 (t, *J* = 8.0 Hz, 3H), 1.42 (q, *J* = 8.0 Hz, 2H), 2.21 (t, *J* = 8.0 Hz, 2H), 2.26 (s, 3H), 6.12 (s, 1H), 7.01-7.04 (m, 1H), 7.08 (d, *J* = 8.0 Hz, 2H), 7.26-7.29 (m, 2H), 7.41 (d, *J* = 8.0 Hz, 2H), 7.42 (s, 1H), 7.54 (d, *J* = 8.0 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ = 12.13, 13.79, 21.90, 28.69, 103.57, 114.55, 119.78, 123.57, 128.92, 129.62, 129.66, 133.80, 134.62, 134.89, 136.04, 138.54, 163.96; **IR** (KBr, cm^{-1}) 3293, 1630, 1526, 1494, 1259, 1091, 1019, 799, 688; **Elemental analysis** (%) Calcd for $C_{21}H_{21}ClN_2O$: C, 71.48; H, 6.00; N, 7.94; Found: C, 71.40; H, 6.13; N, 7.91; **HRMS** (ESI-TOF) Calcd for $C_{21}H_{22}ClN_2O$ $[M+H]^+$ 353.1410; Found 353.1415; Calcd for $C_{21}H_{21}ClN_2NaO$ $[M+Na]^+$ 375.1231; Found 375.1235.



2,5-Dimethyl-*N*,1-diphenyl-1*H*-pyrrole-3-carboxamide (**2d1**)

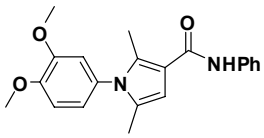
Yellow solid: mp 85-87 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ = 2.01 (s, 3H), 2.35 (s, 3H), 6.21 (s, 1H), 7.06-7.09 (m, 1H), 7.19 (d, *J* = 8.0 Hz, 2H), 7.31-7.34 (m, 2H), 7.44-7.51 (m, 3H), 7.55 (s, 1H), 7.61 (d, *J* = 8.0 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ = 12.26, 12.67, 104.25, 114.25, 119.81, 123.45, 128.10, 128.52, 128.85, 128.89, 129.35, 135.03, 137.53, 138.64, 164.11; **IR** (KBr, cm^{-1}) 3256, 1596, 1577, 1543, 1497, 1439, 1258, 1087, 820, 758, 690.



2,5-Dimethyl-*N*-phenyl-1-*p*-tolyl-1*H*-pyrrole-3-carboxamide (**2d2**)

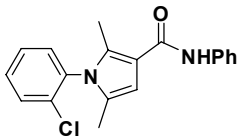
White solid: mp 132-133 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ = 2.00 (s, 3H), 2.34 (s,

3H), 2.43 (s, 3H), 6.19 (s, 1H), 7.05-7.08 (m, 3H), 7.27-7.31 (m, 2H), 7.33 (d, *J* = 8.0 Hz, 2H), 7.55 (s, 1H), 7.61 (d, *J* = 8.0 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ = 12.27, 12.70, 21.15, 104.07, 114.08, 119.75, 123.39, 127.80, 128.85, 128.97, 129.97, 134.85, 135.15, 138.49, 138.70, 164.12; **IR** (KBr, cm^{-1}) 3325, 1644, 1595, 1574, 1514, 1336, 1309, 1246, 1048, 908, 758, 732; **HRMS** (ESI-TOF) Calcd for $C_{20}H_{21}N_2O$ $[M+H]^+$ 305.1648; Found: 305.1641.



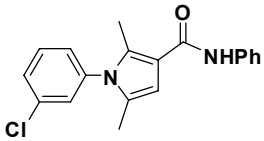
1-(3,4-Dimethoxyphenyl)-2,5-dimethyl-*N*-phenyl-1*H*-pyrrole-3-carboxamide (**2d3**)

Brown solid: mp 225-227 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ = 2.04 (s, 3H), 2.37 (s, 3H), 3.88 (s, 3H), 3.95 (s, 3H), 6.18 (s, 1H), 6.69 (d, *J* = 2.0 Hz, 1H), 6.78 (dd, *J* = 2.0, 8.0 Hz, 1H), 6.95 (d, *J* = 8.0 Hz, 1H), 7.07-7.10 (m, 1H), 7.32-7.36 (m, 2H), 7.47 (s, 1H), 7.61 (dd, *J* = 1.0, 8.0 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ = 12.26, 12.67, 56.03, 56.09, 103.89, 111.10, 114.05, 119.89, 120.32, 123.50, 124.79, 128.92, 129.02, 129.26, 130.30, 135.38, 138.62, 149.15, 164.04; **IR** (KBr, cm^{-1}) 3727, 3625, 1647, 1531, 1437, 1309, 1240, 673, 649.



1-(2-Chlorophenyl)-2,5-dimethyl-*N*-phenyl-1*H*-pyrrole-3-carboxamide (**2d4**)

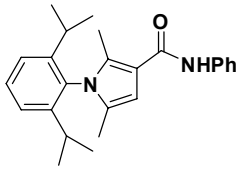
Yellow solid: mp 78-79 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ = 1.95 (s, 3H), 2.30 (s, 3H), 6.25 (s, 1H), 7.05-7.08 (m, 1H), 7.24-7.26 (m, 1H), 7.30-7.44 (m, 2H), 7.40-7.43 (m, 2H), 7.55-7.63 (m, 4H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ = 11.75, 12.17, 104.26, 114.42, 119.77, 123.39, 127.77, 128.69, 128.79, 130.22, 130.29, 130.35, 133.44, 135.07, 135.28, 138.58, 163.93; **IR** (KBr, cm^{-1}) 3423, 3347, 1643, 1596, 1534, 1487, 1436, 1310, 1245, 798, 753, 692.



1-(3-Chlorophenyl)-2,5-dimethyl-*N*-phenyl-1*H*-pyrrole-3-carboxamide (**2d5**)

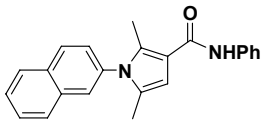
White solid: mp 163-165 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ = 2.00 (s, 3H), 2.34 (s,

3H), 6.22 (s, 1H), 7.06-7.09 (m, 2H), 7.19 (s, 1H), 7.30-7.33 (m, 2H), 7.40-7.43 (m, 2H), 7.61-7.63 (m, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ = 12.32, 12.72, 104.78, 113.14, 114.72, 114.84, 119.88, 123.57, 126.54, 128.45, 128.77, 128.90, 130.25, 130.43, 134.90, 138.70, 164.02; **IR** (KBr, cm^{-1}) 3327, 1648, 1596, 1498, 1485, 1436, 1311, 1252, 810, 759, 692.



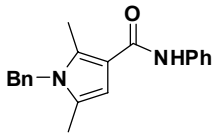
1-(2,6-Diisopropylphenyl)-2,5-dimethyl-*N*-phenyl-1*H*-pyrrole-3-carboxamide (**2d6**)

White solid: mp 100-101 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ = 1.12 (dd, *J* = 2.0, 9.0 Hz, 12H), 1.92 (s, 3H), 2.27 (s, 3H), 2.33 (q, *J* = 9.0 Hz, 2H), 6.27 (s, 1H), 7.08-7.10 (m, 1H), 7.27 (d, *J* = 8.0 Hz, 2H), 7.32-7.35 (m, 2H), 7.43-7.46 (m, 1H), 7.57 (s, 1H), 7.63 (d, *J* = 8.0 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ = 12.40, 12.45, 15.22, 23.87, 24.03, 27.90, 104.13, 113.99, 119.71, 123.36, 124.08, 128.74, 128.84, 129.61, 132.33, 135.36, 138.67, 146.92, 164.12; **IR** (KBr, cm^{-1}) 3296, 1639, 1537, 1457, 1323, 1253, 773, 752, 692.



2,5-Dimethyl-1-(naphthalen-2-yl)-*N*-phenyl-1*H*-pyrrole-3-carboxamide (**2d7**)

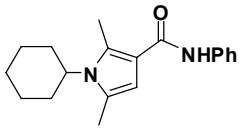
White solid: mp 222-223 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ = 2.04 (s, 3H), 2.39 (s, 3H), 6.24 (s, 1H), 7.07-7.10 (m, 1H), 7.24-7.28 (m, 1H), 7.32-7.35 (m, 1H), 7.54-7.69 (m, 2H), 7.54-7.69 (m, 6H), 7.88 (s, 1H), 7.89-7.97 (m, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ = 12.13, 12.55, 104.10, 114.14, 119.52, 123.21, 125.52, 126.65, 126.74, 127.59, 127.74, 128.64, 128.89, 129.17, 132.51, 133.00, 134.70, 135.02, 138.43, 163.81; **IR** (KBr, cm^{-1}) 3257, 3057, 1638, 1596, 1577, 1498, 1309, 1257, 795, 759, 690.



1-Benzyl-2,5-dimethyl-*N*-phenyl-1*H*-pyrrole-3-carboxamide (**2d8**)

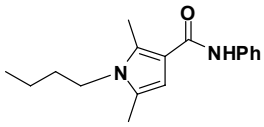
White solid: mp 170-171 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ = 2.15 (s, 3H), 2.52 (s,

3H), 5.05 (s, 2H), 6.16 (s, 1H), 6.90 (d, *J* = 8.0 Hz, 2H), 7.06-7.09 (m, 1H), 7.24-7.34 (m, 4H), 7.49 (s, 1H), 7.59 (d, *J* = 8.0 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ = 11.17, 12.21, 46.68, 61.49, 104.32, 113.96, 119.72, 123.39, 125.45, 127.36, 128.20, 128.84, 128.85, 134.54, 136.90, 138.63, 164.09; **IR** (KBr, cm^{-1}) 3249, 3027, 1638, 1536, 1495, 1435, 1250, 753, 691.



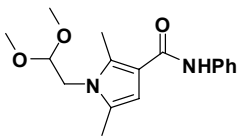
1-Cyclohexyl-2,5-dimethyl-*N*-phenyl-1*H*-pyrrole-3-carboxamide (**2d9**)

White solid: mp 68-69 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ = 1.17-1.24 (m, 1H), 1.35-1.42 (m, 2H), 1.74-1.76 (m, 1H), 1.85-2.04 (m, 6H), 2.31 (s, 3H), 2.66 (s, 3H), 3.96-3.41 (m, 1H), 6.04 (s, 1H), 7.04-7.07 (m, 1H), 7.29-7.32 (m, 2H), 7.43 (s, 1H), 7.56 (d, *J* = 8.0 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ = 25.38, 26.45, 31.89, 56.62, 61.49, 119.68, 123.27, 127.80, 128.81, 134.18, 138.70, 164.27; **IR** (KBr, cm^{-1}) 3338, 1643, 1529, 1498, 1435, 1304, 1243, 755, 693.



1-Butyl-2,5-dimethyl-*N*-phenyl-1*H*-pyrrole-3-carboxamide (**2d10**)

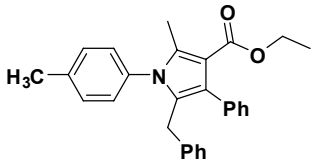
White solid: mp 86-88 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ = 0.96 (t, *J* = 7.5 Hz, 3H), 1.37 (q, *J* = 7.5 Hz, 2H), 1.57-1.63 (m, 2H), 2.23 (s, 3H), 2.58 (s, 3H), 3.76 (t, *J* = 7.5 Hz, 3H), 6.01 (s, 1H), 7.04-7.07 (m, 1H), 7.29-7.33 (m, 2H), 7.42 (s, 1H), 7.57 (d, *J* = 7.5 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ = 11.22, 12.27, 13.75, 20.05, 32.64, 43.39, 103.94, 113.47, 119.69, 123.29, 127.50, 128.83, 133.90, 138.71, 164.16; **IR** (KBr, cm^{-1}) 3306, 3057, 1645, 1529, 1437, 1307, 1242, 755, 693.



1-(2,2-Dimethoxyethyl)-2,5-dimethyl-*N*-phenyl-1*H*-pyrrole-3-carboxamide (**2d11**)

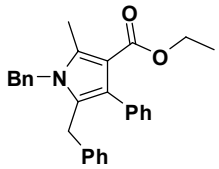
White solid: mp 141-142 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ = 2.25 (s, 3H), 2.60 (s, 3H), 3.34 (s, 6H), 3.90 (d, *J* = 5.5 Hz, 2H), 4.38 (t, *J* = 5.5 Hz, 1H), 6.07 (s, 1H), 7.04-7.07 (m, 1H), 7.26-7.32 (m, 2H), 7.44 (s, 1H), 7.56-7.58 (m, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ = 11.40, 12.49, 29.63, 46.54, 55.46, 104.28, 113.93, 119.73,

123.36, 128.64, 128.82, 134.60, 138.69, 164.08; **IR** (KBr, cm^{-1}) 3220, 1635, 1534, 1304, 1251, 1052, 755, 694; **Elemental analysis** (%) Calcd for $C_{17}H_{22}N_2O_3$: C, 67.53; H, 7.33; N, 9.26; Found: C, 67.57; H, 7.32; N, 9.24; **HRMS** (ESI-TOF) Calcd for $C_{17}H_{23}N_2O_3$ $[M+H]^+$ 303.1703; Found 303.1691.



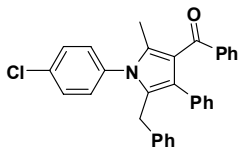
Ethyl 5-benzyl-2-methyl-4-phenyl-1-*p*-tolyl-1*H*-pyrrole-3-carboxylate (**3a**)

Yellow solid: mp 81-82 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ = 1.02 (t, *J* = 7.0 Hz, 3H), 2.27 (s, 3H), 2.36 (s, 3H), 3.68 (s, 2H), 4.01 (q, *J* = 7.0 Hz, 2H), 6.64-6.66 (m, 2H), 6.79 (d, *J* = 8.0 Hz, 2H), 7.03-7.04 (m, 3H), 7.07 (d, *J* = 8.0 Hz, 2H), 7.23-7.26 (m, 1H), 7.30-7.36 (m, 4H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ = 12.52, 13.92, 21.15, 30.61, 59.16, 110.94, 123.93, 125.60, 126.09, 127.48, 127.82, 128.04, 128.20, 129.42, 129.50, 130.39, 134.68, 136.37, 136.64, 138.32, 139.78, 165.99; **IR** (KBr, cm^{-1}) 3379, 1697, 1515, 1187, 1079, 783, 716, 524.



Ethyl 1,5-dibenzyl-2-methyl-4-phenyl-1*H*-pyrrole-3-carboxylate (**3b**)

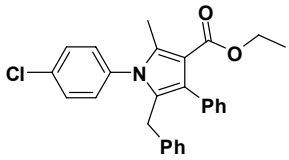
Yellow solid: mp 108-109 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ = 1.00 (t, *J* = 7.0 Hz, 3H), 2.47 (s, 3H), 3.76 (s, 2H), 4.08 (q, *J* = 7.0 Hz, 2H), 4.86 (s, 2H), 6.85 (d, *J* = 7.0 Hz, 2H), 7.01 (d, *J* = 7.0 Hz, 2H), 7.15-7.33 (m, 11H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ = 11.50, 13.86, 30.32, 47.14, 59.16, 111.08, 124.82, 125.51, 126.09, 127.33, 127.79, 128.20, 128.55, 128.82, 129.56, 130.36, 131.62, 135.98, 136.44, 137.04, 139.45, 165.99; **IR** (KBr, cm^{-1}) 3735, 3438, 1698, 1600, 1517, 1412, 1329, 1151, 1067, 1026, 735, 697, 510.



(5-Benzyl-1-(4-chlorophenyl)-2-methyl-4-phenyl-1*H*-pyrrol-3-yl)(phenyl)methanone (**3c**)

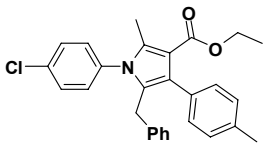
Brown solid: mp 70-71 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ = 2.13 (s, 3H), 3.83 (s,

2H), 6.69-6.71 (m, 2H), 6.91 (d, *J* = 7.0 Hz, 2H), 6.99-7.00 (m, 1H), 7.05-7.17 (m, 9H), 7.26-7.29 (m, 3H), 7.68 (d, *J* = 7.0 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ = 12.53, 30.78, 115.37, 120.80, 124.49, 125.88, 125.96, 127.53, 127.84, 127.98, 128.77, 129.50, 129.71, 129.73, 129.97, 131.50, 134.46, 135.15, 135.68, 135.89, 139.40, 139.45, 194.19; **IR** (KBr, cm^{-1}) 3443, 3060, 1631, 1598, 1493, 1451, 1091, 833, 695 **Elemental analysis** (%) Calcd for $C_{31}H_{24}ClNO$: C, 80.59; H, 5.24; N, 3.03; Found: C, 80.48; H, 5.28; N, 3.09; **HRMS** (ESI-TOF) Calcd for $C_{31}H_{25}ClNO$ $[M+H]^+$ 462.1626; Found 462.1619.



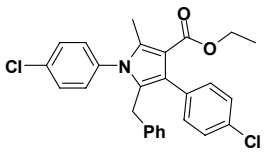
Ethyl 5-benzyl-1-(4-chlorophenyl)-2-methyl-4-phenyl-1*H*-pyrrole-3-carboxylate (**3d**)

Yellow solid: mp 96-97 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ = 1.02 (t, *J* = 7.0 Hz, 3H), 2.26 (s, 3H), 3.68 (s, 2H), 4.09 (q, *J* = 7.0 Hz, 2H), 6.65-6.67 (m, 2H), 6.83 (d, *J* = 7.0 Hz, 2H), 7.05-7.06 (m, 2H), 7.24-7.34 (m, 8H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ = 12.45, 13.89, 30.65, 59.27, 111.42, 124.37, 125.83, 126.27, 127.55, 127.98, 129.12, 129.31, 129.79, 130.33, 134.41, 135.88, 136.02, 136.41, 139.38, 165.78; **IR** (KBr, cm^{-1}) 3054, 1698, 1532, 1494, 1286, 1189, 1080, 833, 701, 664, 514.



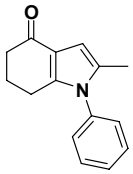
Ethyl 5-benzyl-1-(4-chlorophenyl)-2-methyl-4-*p*-tolyl-1*H*-pyrrole-3-carboxylate (**3e**)

Yellow oil; **^{1}H NMR** (500 MHz, $CDCl_3$) δ = 1.07 (t, *J* = 7.5 Hz, 3H), 2.25 (s, 3H), 2.35 (s, 3H), 3.67 (s, 2H), 4.11 (q, *J* = 7.5 Hz, 2H), 6.65-6.66 (m, 2H), 6.82 (d, *J* = 8.5 Hz, 2H), 7.04-7.05 (m, 3H), 7.14 (d, *J* = 8.0 Hz, 2H), 7.24 (d, *J* = 8.5 Hz, 2H), 7.26 (d, *J* = 8.0 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ = 12.48, 13.99, 21.16, 30.66, 59.23, 111.51, 124.39, 125.77, 127.95, 128.13, 128.52, 128.74, 129.17, 129.80, 130.17, 131.59, 135.89, 134.36, 135.98, 136.16, 139.47, 165.77.



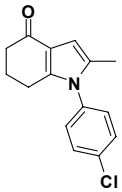
Ethyl 5-benzyl-1,4-bis(4-chlorophenyl)-2-methyl-1*H*-pyrrole-3-carboxylate (**3f**)

White solid: mp 90-91 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ = 1.07 (t, *J* = 7.0 Hz, 3H), 2.26 (s, 3H), 3.65 (s, 2H), 4.11 (q, *J* = 7.0 Hz, 2H), 6.63-6.65 (m, 2H), 6.83 (dd, *J* = 2.0, 6.5 Hz, 2H), 7.05-7.07 (m, 3H), 7.24-7.31 (m, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ = 12.48, 13.99, 30.57, 59.33, 111.32, 123.20, 125.95, 127.71, 127.87, 128.07, 129.18, 129.44, 129.74, 131.67, 132.21, 134.56, 134.60, 135.73, 136.65, 139.12, 165.51.



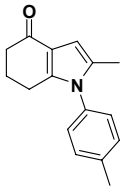
2-Methyl-1-phenyl-6,7-dihydro-1*H*-indol-4(5H)-one (**4a**)

Yellow solid: mp 140-141 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ = 2.05 (s, 3H), 2.07-2.17 (m, 2H), 2.47-2.54 (m, 4H), 6.38 (s, 1H), 7.22-7.24 (m, 2H), 7.45-7.52 (m, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ = 12.79, 22.98, 24.12, 38.17, 103.76, 120.43, 127.75, 128.82, 129.69, 131.57, 137.36, 144.59, 194.46; **IR** (KBr, cm^{-1}) 3623, 1649, 1593, 1494, 1396, 1308, 1230, 1008, 702, 579; **HRMS** (ESI-TOF) Calcd for $C_{15}H_{16}NO$ $[M+H]^+$ 226.1233; Found 226.1226.



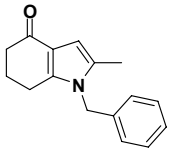
1-(4-Chlorophenyl)-2-methyl-5,6-dihydro-1*H*-indol-7(4H)-one) (**4b**)

Brown solid: mp 145-146 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ = 2.05 (s, 3H), 2.06-2.11 (m, 2H), 2.47-2.53 (m, 4H), 6.38 (s, 1H), 7.18 (d, *J* = 8.0Hz, 2H), 7.49 (d, *J* = 8.0Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ = 12.56, 22.67, 23.81, 37.84, 103.80, 120.36, 128.79, 129.73, 131.22, 134.60, 135.53, 144.26, 194.29; **IR** (KBr, cm^{-1}) 3279, 1653, 1494, 1468, 1091, 795, 505.



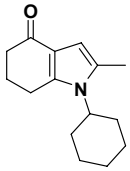
2-Methyl-1-*p*-tolyl-6,7-dihydro-1*H*-indol-4(5H)-one (**4c**)

Yellow solid: mp 133-134 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ = 2.04 (s, 3H), 2.04-2.10 (m, 2H), 2.44 (s, 3H), 2.46-2.53 (m, 4H), 6.36 (s, 1H), 7.10 (d , *J* = 8.0 Hz, 2H), 7.29 (d , *J* = 8.0 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ = 12.51, 21.11, 22.72, 23.87, 37.92, 103.32, 120.05, 127.22, 130.02, 131.42, 134.47, 138.59, 144.47, 194.22; **IR** (KBr, cm^{-1}) 3283, 1652, 1517, 1408, 858, 844, 784, 575; **Elemental analysis** (%) Calcd for $C_{16}H_{17}NO$: C, 80.30; H, 7.16; N, 5.85; Found: C, 80.38; H, 7.21; N, 5.88; **HRMS** (ESI-TOF) Calcd for $C_{16}H_{17}NNaO$ $[M+Na]^+$ 262.1207; Found 262.1202; Calcd for $C_{32}H_{34}N_2NaO_2$ $[2M+Na]^+$ 501.2526; Found 501.2512.



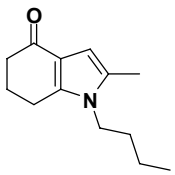
1-Benzyl-2-methyl-6,7-dihydro-1*H*-indol-4(5H)-one (**4d**)

Yellow solid: mp 137-138 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ = 2.08-2.13 (m, 2H), 2.14 (s, 3H), 2.45 (t, *J* = 6.0 Hz, 2H), 2.64 (t, *J* = 6.0 Hz, 2H), 5.03 (s, 2H), 6.34 (s, 1H), 6.92 (d , *J* = 8.0 Hz, 2H), 7.27-7.28 (m, 1H), 7.31-7.34 (m, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ = 11.79, 21.82, 23.52, 37.53, 46.91, 103.46, 119.78, 125.39, 127.36, 128.73, 130.46, 136.46, 143.59, 193.75; **IR** (KBr, cm^{-1}) 3471, 1650, 1606, 1526, 1494, 1478, 1199, 799, 731, 704, 569.



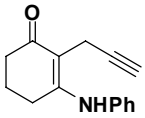
1-Cyclohexyl-2-methyl-6,7-dihydro-1*H*-indol-4(5H)-one (**4e**)

Yellow oil; **^{1}H NMR** (500 MHz, $CDCl_3$) δ = 1.18-1.27 (m, 2H), 1.37-1.41 (m, 2H), 1.81-1.94 (m, 6H), 2.11-2.15 (m, 2H), 2.27 (s, 3H), 2.42 (t, *J* = 6.0 Hz, 2H), 2.86 (t, *J* = 6.0 Hz, 2H), 3.89-3.94 (m, 1H), 6.25 (s, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ = 13.87, 24.13, 25.39, 26.36, 32.20, 37.56, 56.87, 104.28, 120.16, 130.39, 142.87, 193.95; **IR** (KBr, cm^{-1}) 3447, 1653, 1519, 1474, 1444, 1175, 1008, 795, 575.



1-Butyl-2-methyl-6,7-dihydro-1*H*-indol-4(5H)-one (**4f**)

Yellow oil; **^{1}H NMR** (500 MHz, $CDCl_3$) δ = 0.96 (t, *J* = 7.5 Hz, 3H), 1.35-1.41 (m, 2H), 1.60-1.66 (m, 2H), 2.11-2.16 (m, 2H), 2.22 (s, 3H), 2.44 (t, *J* = 6.5 Hz, 2H), 2.72 (t, *J* = 6.5 Hz, 2H), 3.75 (t , *J* = 6.5 Hz, 2H), 6.25 (s, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ = 12.05, 13.70, 20.03, 22.16, 23.81, 32.75, 37.75, 43.81, 103.37, 119.59, 130.08, 143.17, 193.79; **IR** (KBr, cm^{-1}) 3461, 1652, 1562, 1524, 1444, 1416, 1366, 1187, 999, 795, 570.



3-(Phenylamino)-2-(prop-2-ynyl)cyclohex-2-enone (**4a-i**)

^{1}H NMR (500 MHz, $CDCl_3$) δ = 1.90-1.95 (m, 2H), 2.11 (s, 1H), 2.40 (t, *J* = 6.5 Hz, 2H), 2.50 (t, *J* = 6.5 Hz, 2H), 3.45 (d , *J* = 1.0 Hz, 2H), 7.12 (d , *J* = 8.0 Hz, 2H), 7.15 (s, 1H), 7.22-7.27 (m, 1H), 7.36-7.39 (m, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ = 11.88, 21.72, 27.16, 36.34, 69.42, 81.85, 106.53, 125.02, 125.83, 129.29, 138.50, 169.08, 194.01; **IR** (KBr, cm^{-1}) 3288, 1570, 1496, 1395, 1308, 1184, 720.

Supporting Information

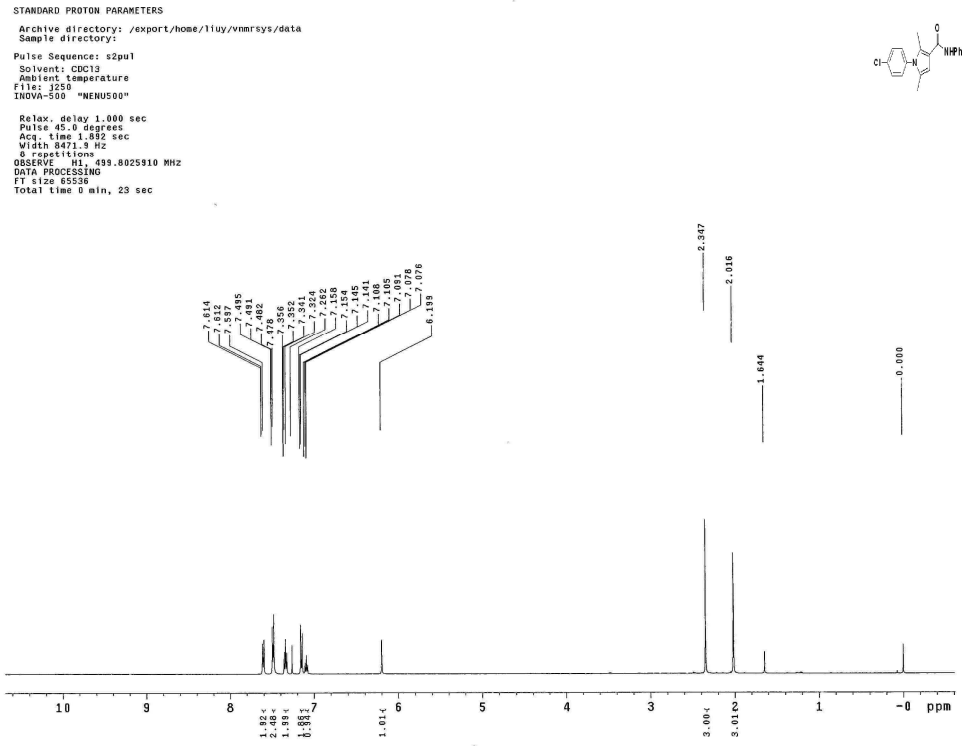
(Part II: Copies of Spectra)

Iron-catalyzed synthesis of polysubstituted pyrroles *via* [4C+1N] cyclization of 4-acetylenic ketones with primary amines

Yeming Wang, Xihe Bi,* Dehua Li, Peiqiu Liao, Yidong Wang, Jin Yang, Qian Zhang,* Qun Liu

Department of Chemistry, Northeast Normal University, 5268 Renmin Street, 130024 Changchun, CHINA

2a1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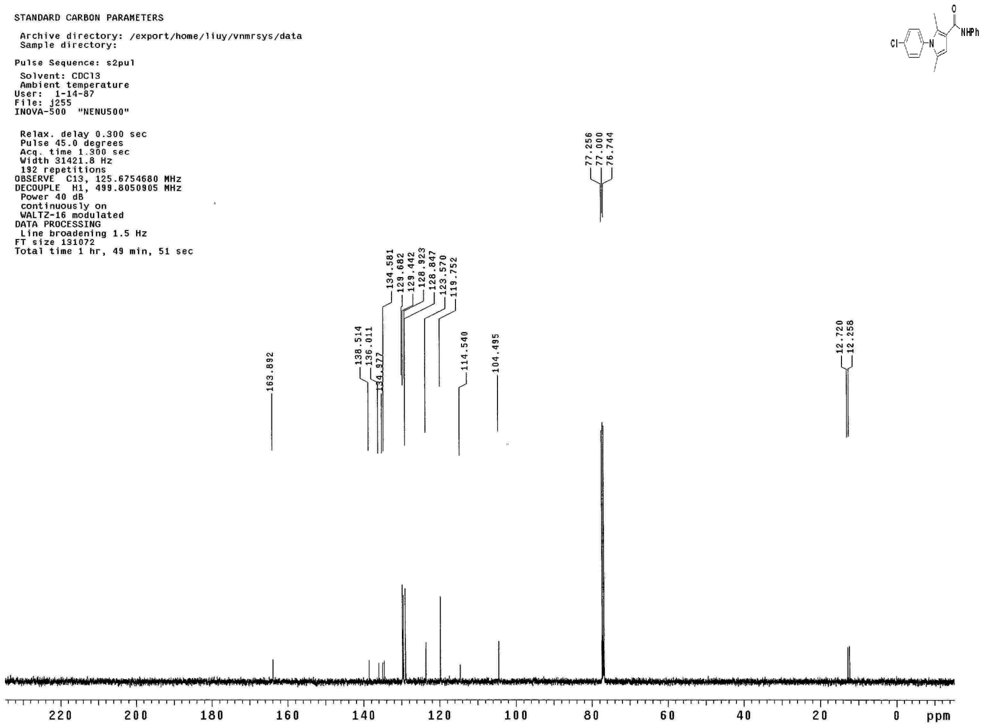
Ambient temperature
File: j250
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.882 sec
Width 8471.8 Hz
8 repetitions
OBSERVE H1, 499.8025910 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
NHPh
Cl
7.614
7.612
7.597
7.495
7.491
7.482
7.478
7.356
7.352
7.341
7.324
7.282
7.158
7.154
7.145
7.141
7.108
7.105
7.091
7.078
7.076
6.199
2.347
2.016
1.644
0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm
1.82
2.48
1.99
1.88
0.94
1.01
3.00
3.01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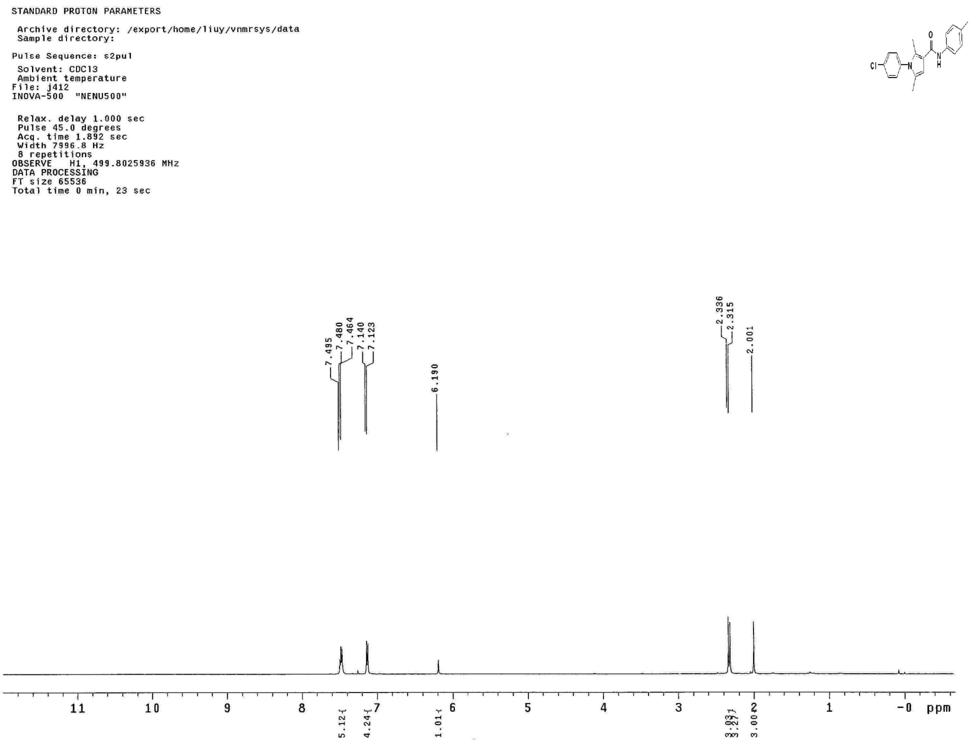
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: j255
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
192 repetitions
OBSERVE C13, 125.6754680 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 1 hr, 49 min, 51 sec
NHPh
Cl
N
O
163.892
138.514
136.011
134.927
134.581
129.682
129.442
128.923
128.847
123.570
119.752
114.540
104.495
77.256
77.000
76.744
12.720
12.258
220
200
180
160
140
120
100
80
60
40
20
0
ppm

2a2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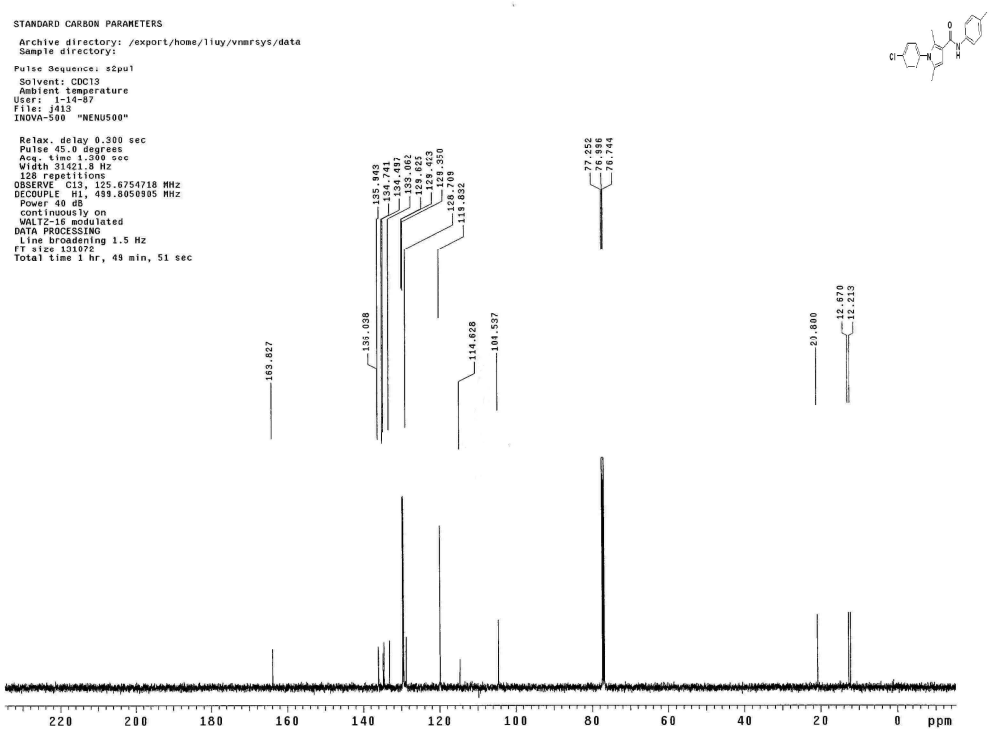
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: j412
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7996.8 Hz
8 repetitions
OBSERVE H1, 499.8025936 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
Cl
N
O
H
7.495
7.480
7.464
7.140
7.123
6.190
2.336
2.315
2.001
11
10
9
8
7
6
5
4
3
2
1
-0
ppm
5.12
4.24
1.01
3.03
3.27
3.00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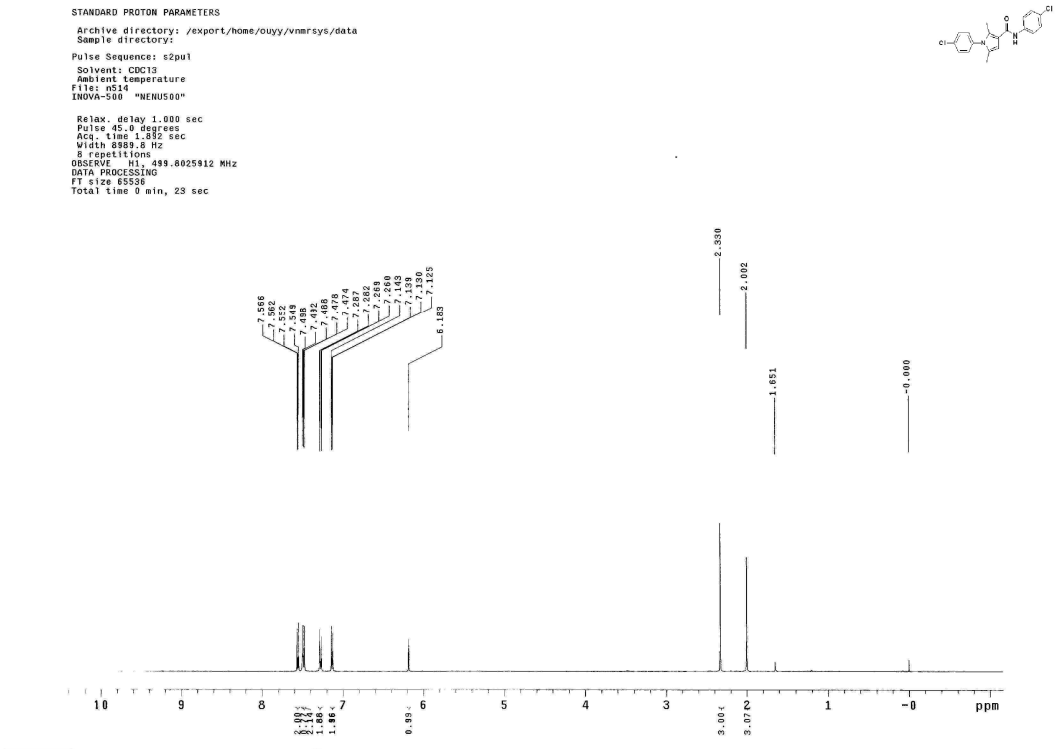
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: ja13
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754718 MHz
DECOUPLE H1, 499.8050805 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 1 hr, 49 min, 51 sec
163.827
139.038
135.943
134.741
134.497
133.062
129.805
129.423
128.350
126.709
119.092
114.628
104.537
77.252
76.998
76.744
21.800
12.670
12.213
220
200
180
160
140
120
100
80
60
40
20
0
ppm

2a3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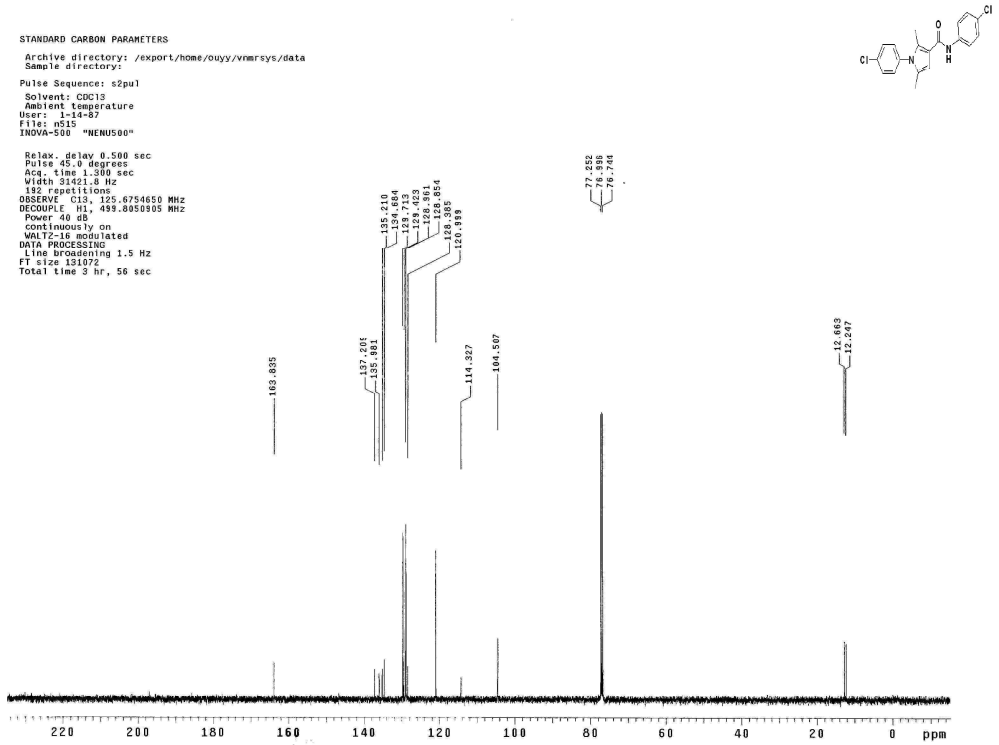
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: n514
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8889.8 Hz
8 repetitions
OBSERVE H1, 499.8025912 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
7.566
7.562
7.532
7.549
7.498
7.494
7.488
7.478
7.474
7.279
7.282
7.269
7.260
7.149
7.145
7.130
7.125
6.183
2.330
2.002
1.651
-0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm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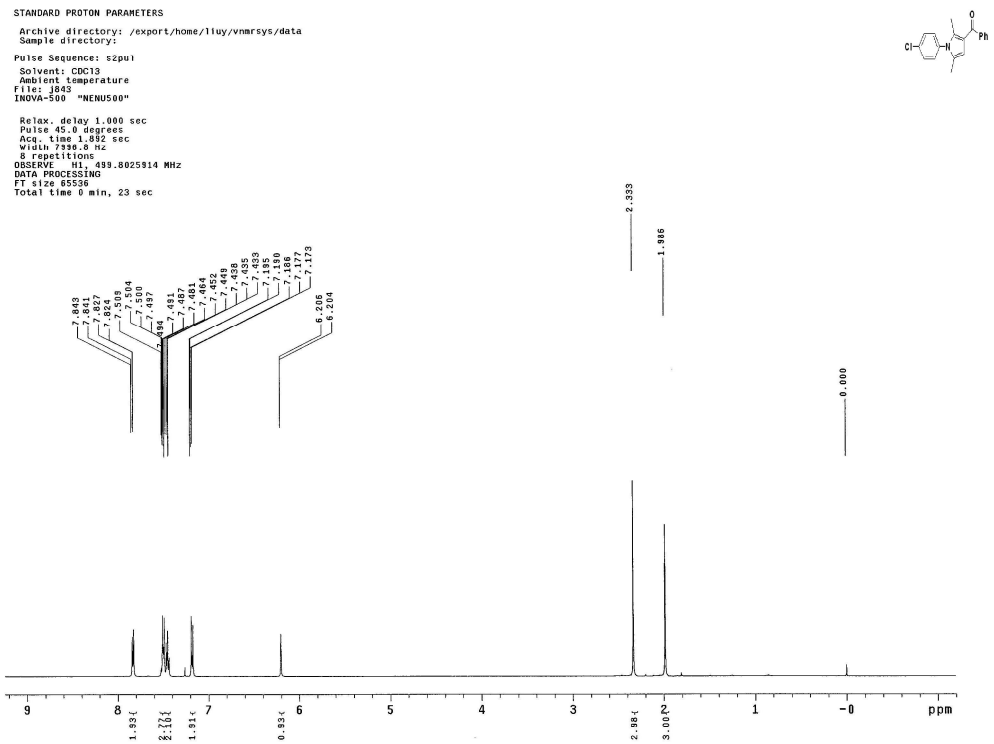
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: n515
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
192 repetitions
OBSERVE C13, 125.6754650 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
163.835
137.201
135.981
135.210
134.684
129.713
129.403
128.961
128.854
128.385
120.999
114.327
104.507
77.262
76.936
76.744
12.663
12.497
220
200
180
160
140
120
100
80
60
40
20
0
ppm

2a4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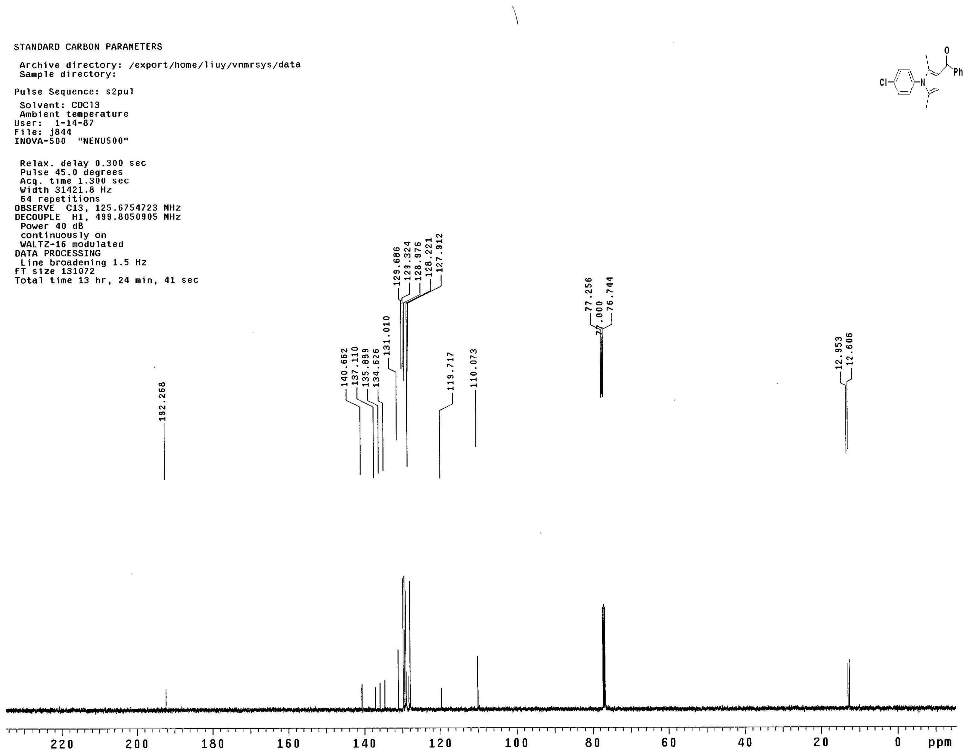
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: jb43
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7398.8 Hz
8 repetitions
OBSERVE H1, 499.8025914 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
7.843
7.841
7.827
7.824
7.509
7.504
7.500
7.497
7.494
7.481
7.468
7.464
7.461
7.452
7.448
7.438
7.435
7.433
7.195
7.190
7.186
7.177
7.173
6.206
6.204
2.333
1.986
0.000
9
8
7
6
5
4
3
2
1
0
ppm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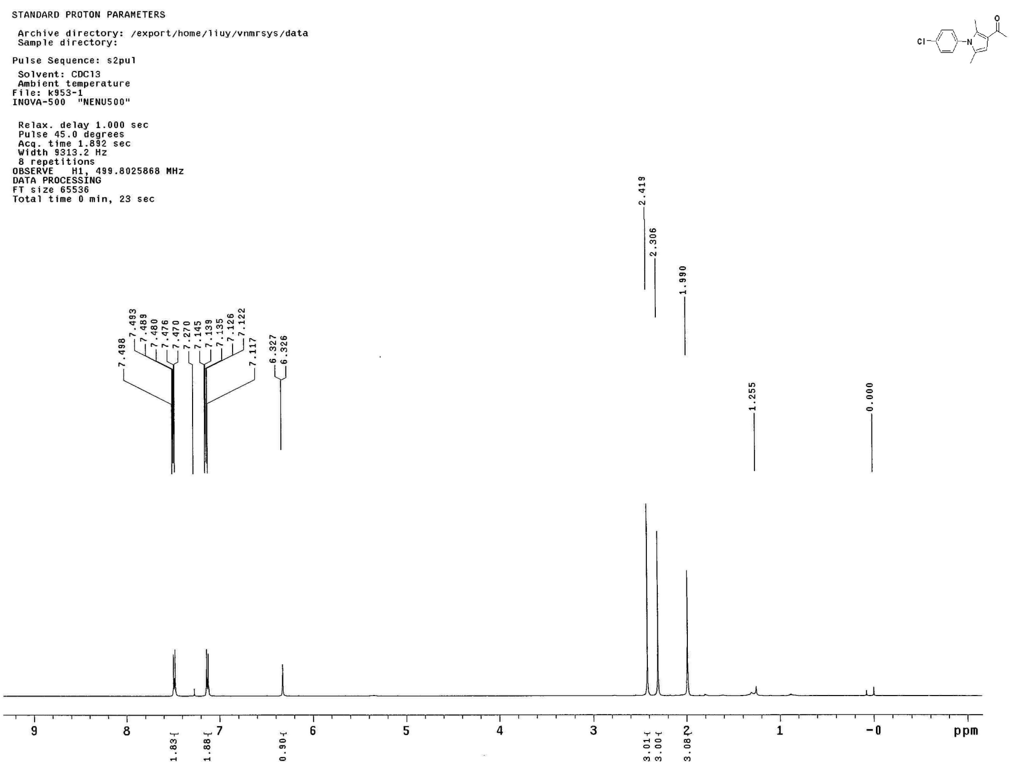
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: j844
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
64 repetitions
OBSERVE C13, 125.6754723 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 13 hr, 24 min, 41 sec
O
Ph
Cl
N
192.268
140.662
137.110
135.888
134.626
131.010
129.686
129.324
128.976
128.221
127.912
119.717
110.073
77.256
77.000
76.744
12.953
12.606
220
200
180
160
140
120
100
80
60
40
20
0
ppm

2a5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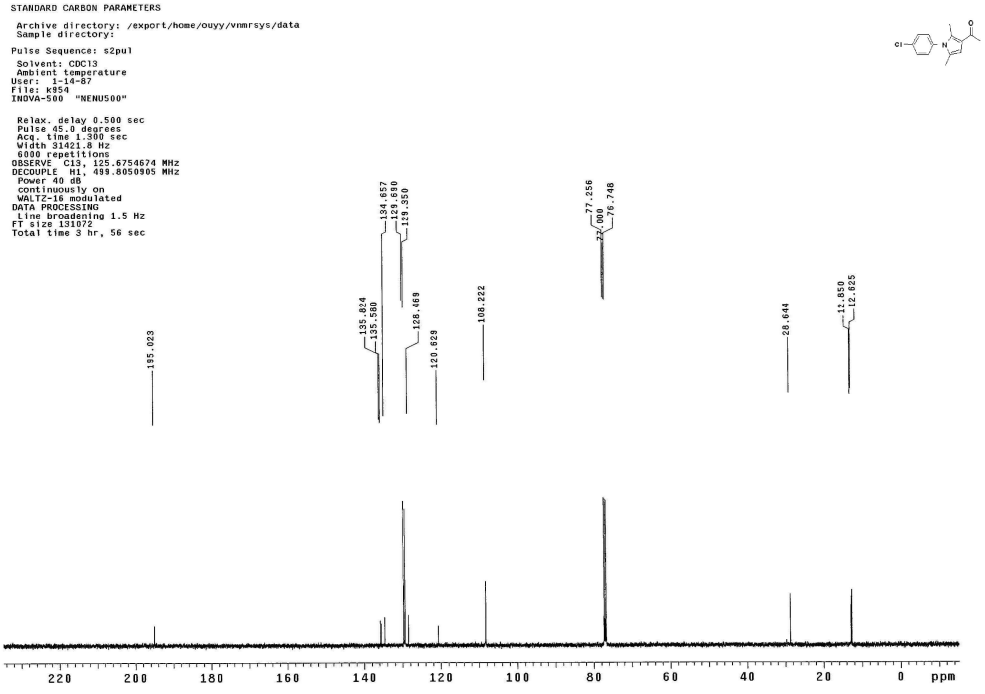
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: k953-1
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9313.2 Hz
8 repetitions
OBSERVE H1, 499.8025868 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
O
Cl
N
7.498
7.493
7.489
7.480
7.476
7.470
7.270
7.145
7.139
7.135
7.126
7.122
7.117
6.327
6.326
2.419
2.306
1.990
1.255
0.000
9
8
7
6
5
4
3
2
1
-0
ppm
1.83
1.88
0.90
3.01
3.00
3.08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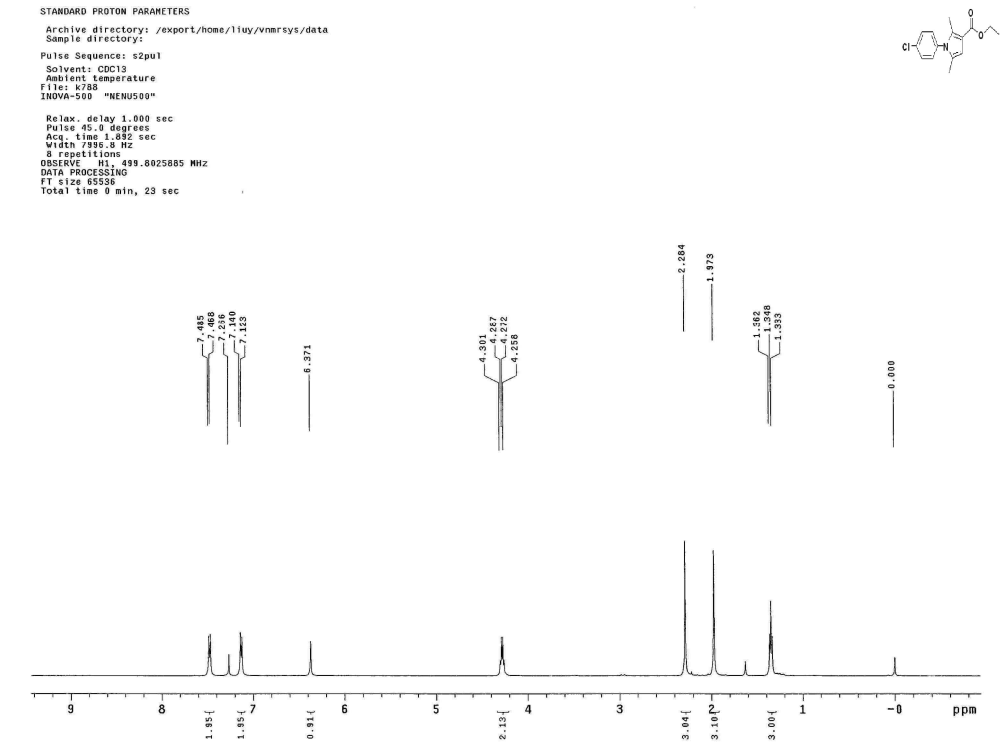
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: k854
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
6000 repetitions
OBSERVE C13, 125.6754674 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
195.023
135.824
135.580
134.657
129.680
129.350
128.469
120.629
108.222
77.256
77.000
76.748
28.644
12.850
12.625
220
200
180
160
140
120
100
80
60
40
20
0
ppm

2a6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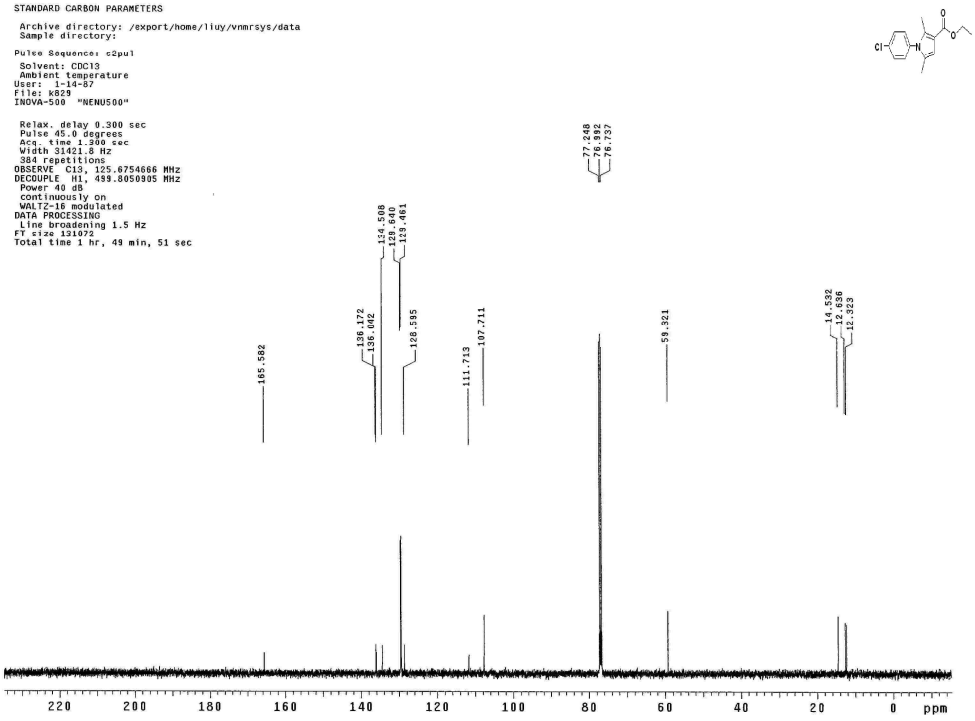
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: k788
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7986.8 Hz
8 repetitions
OBSERVE H1, 499.8025885 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
7.485
7.468
7.140
7.123
6.371
4.301
4.267
4.232
4.200
2.284
1.973
1.562
1.548
1.533
0.000
9
8
7
6
5
4
3
2
1
-0
ppm
1.95
1.95
0.91
2.19
3.04
3.10
3.00


STANDARD CARBON PARAMETERS
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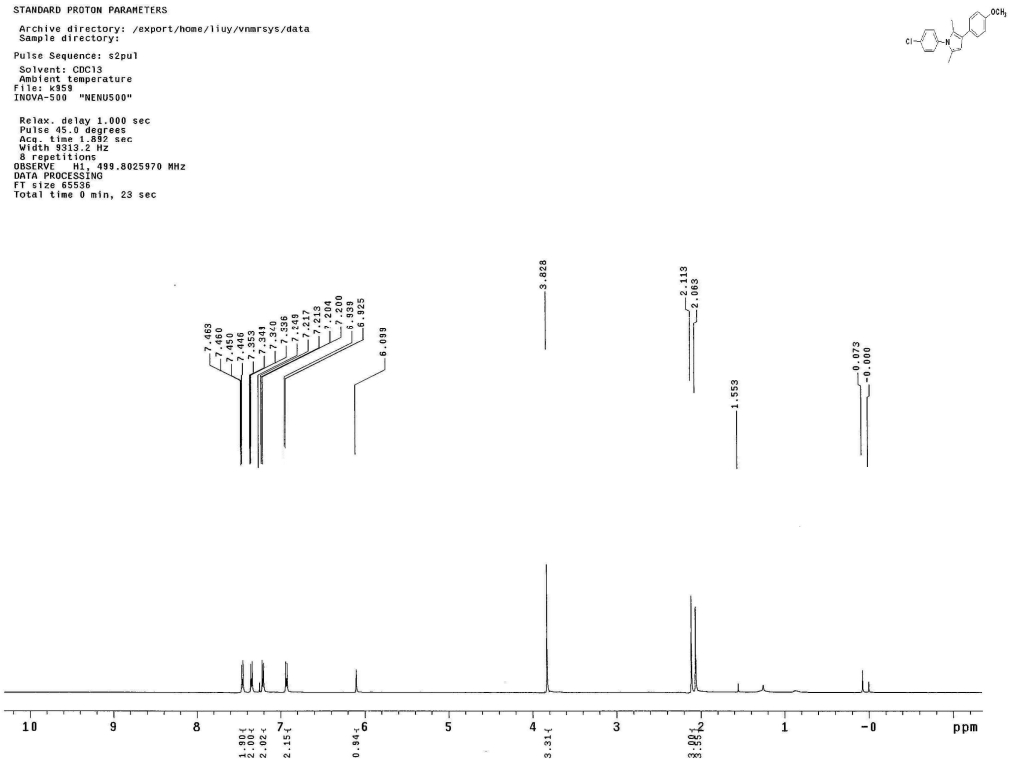
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: k829
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
384 repetitions
OBSERVE C13, 125.8754666 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 1 hr, 49 min, 51 sec
165.582
136.172
136.042
124.506
124.340
123.461
128.595
111.713
107.711
77.248
76.992
76.737
59.321
14.532
13.856
12.323
220
200
180
160
140
120
100
80
60
40
20
0
ppm

2a7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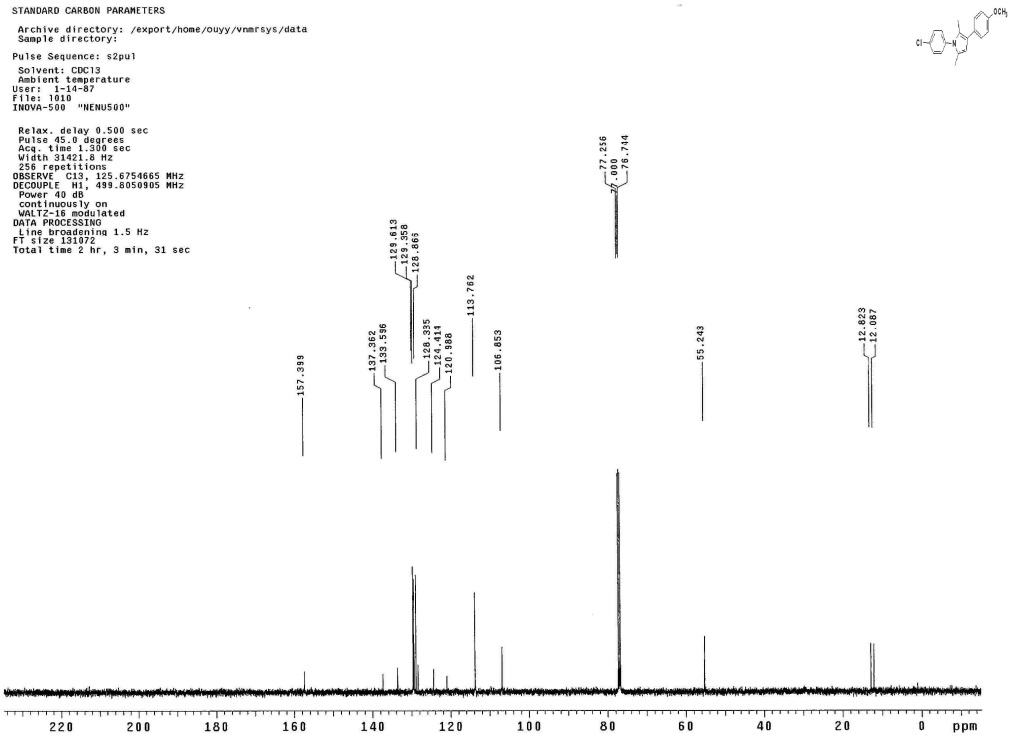
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: k859
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9313.2 Hz
8 repetitions
OBSERVE H1, 499.8025970 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
7.468
7.460
7.450
7.446
7.333
7.330
7.340
7.336
7.249
7.217
7.213
7.204
7.200
6.939
6.925
6.699
3.828
2.113
2.063
1.553
0.073
-0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm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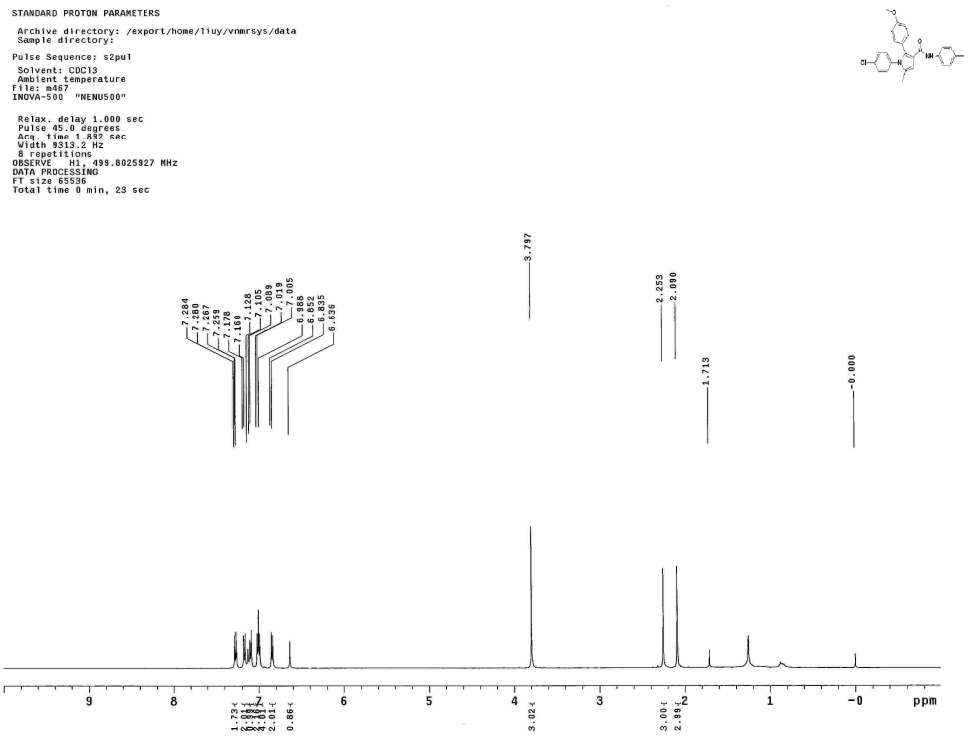
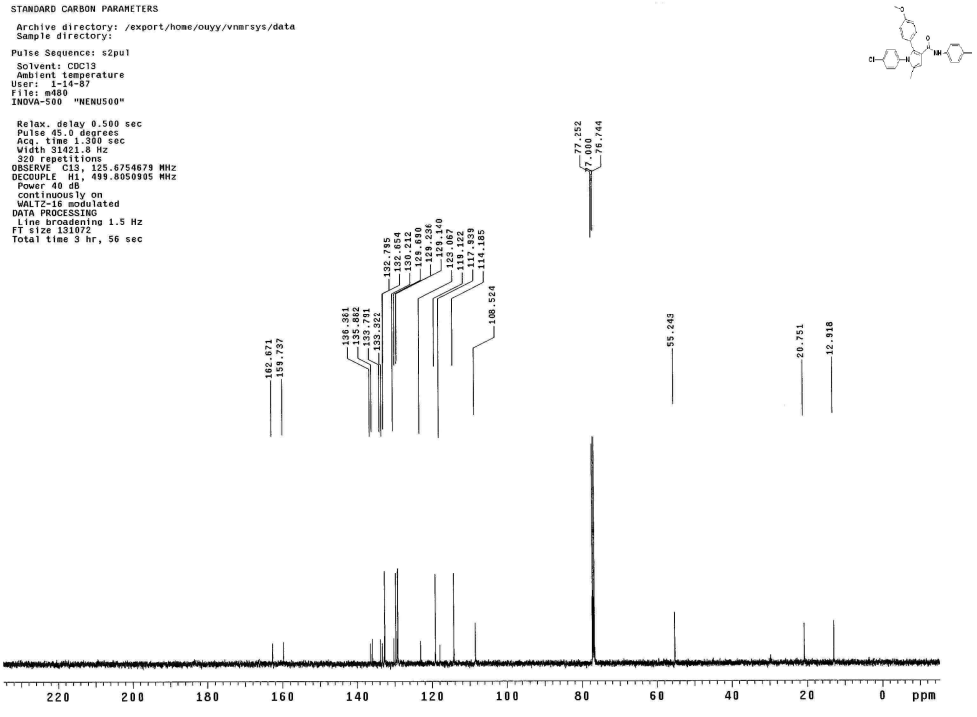
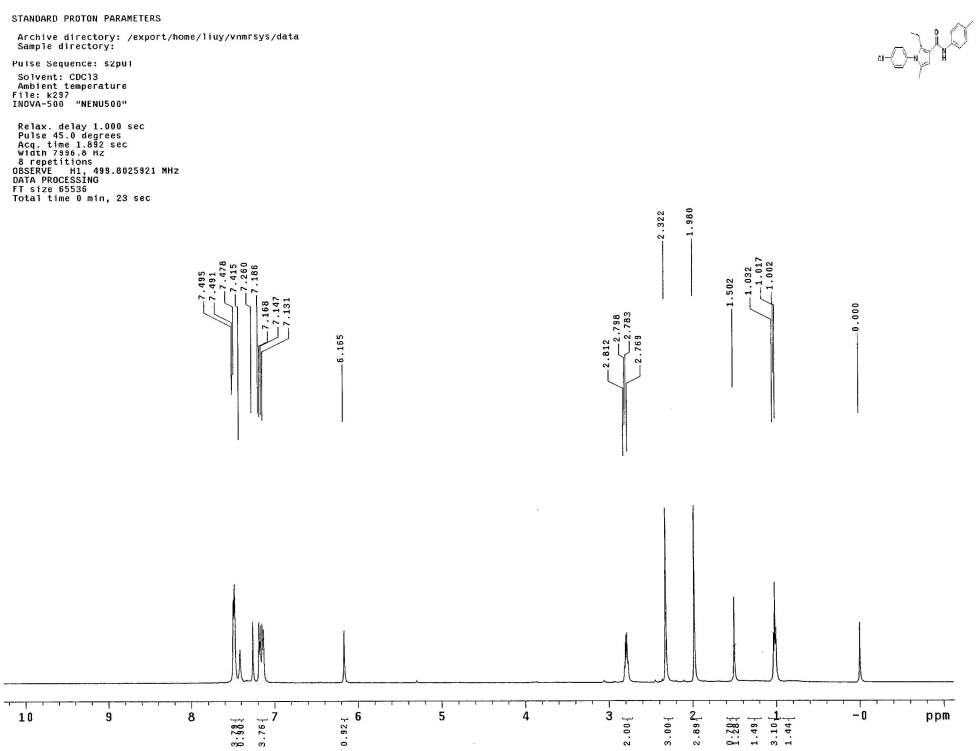
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: 1010
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
256 repetitions
OBSERVE C13, 125.6754665 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
157.399
137.362
133.596
129.613
129.358
128.880
128.335
124.414
120.388
113.762
106.853
77.256
77.000
76.744
55.243
12.823
12.087
220
200
180
160
140
120
100
80
60
40
20
0
ppm

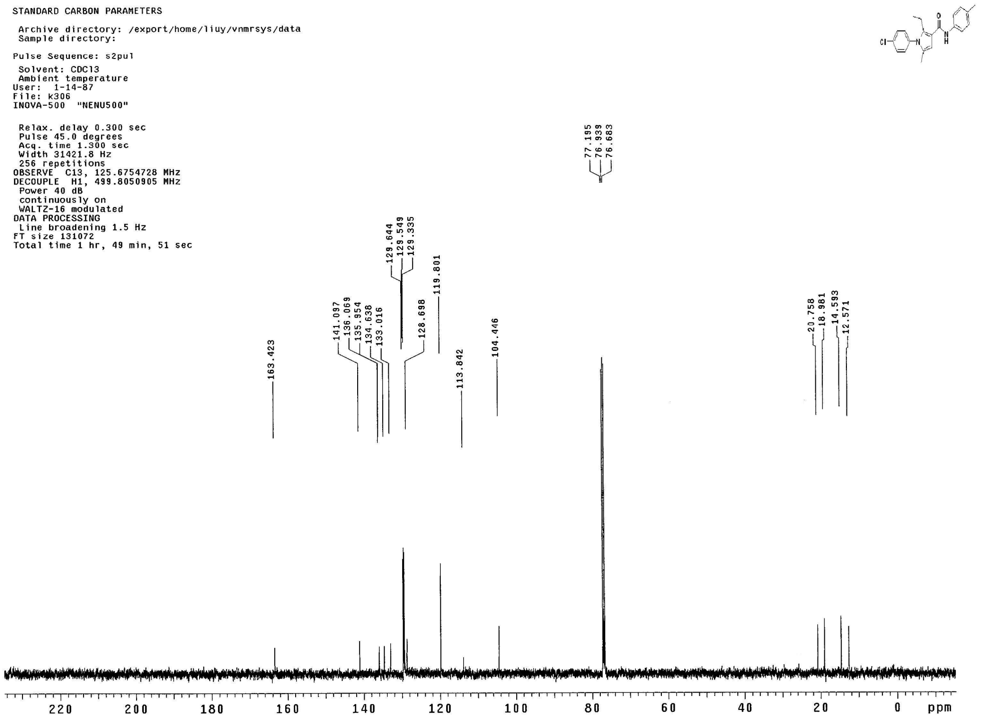
2b1


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: m467
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.882 sec
Width 9313.2 Hz
8 repetitions
OBSERVE H1, 499.8025927 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
7.284
7.280
7.167
7.163
7.158
7.160
7.128
7.105
7.089
7.019
7.005
6.856
6.852
6.835
6.836
3.797
2.253
2.090
1.713
0.000
9
8
7
6
5
4
3
2
1
0
ppm
1.73
1.00
2.01
0.86
3.02
3.00
2.99

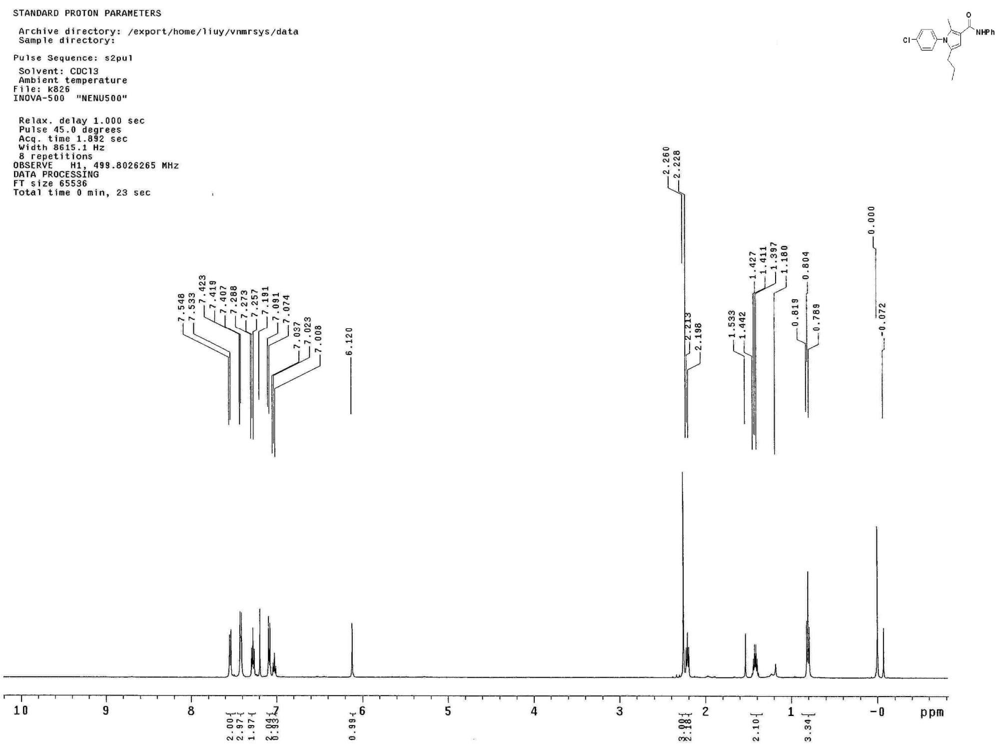

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: m480
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
320 repetitions
OBSERVE C13, 125.6754679 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 3 hr, 56 sec
77.252
77.000
76.744
162.671
159.737
136.861
135.872
133.791
133.322
132.785
132.654
130.212
129.630
129.236
129.140
123.067
119.122
117.938
114.185
108.524
55.243
20.751
12.918
220
200
180
160
140
120
100
80
60
40
20
0
ppm

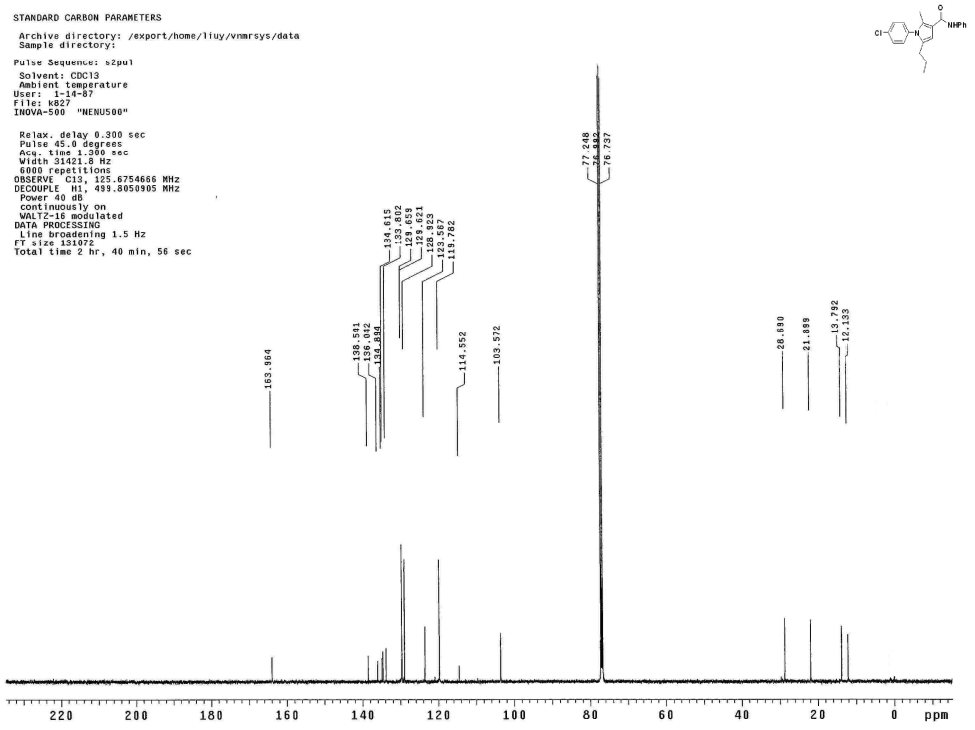
2b2


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: k237
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.882 sec
Width 7936.6 Hz
8 repetitions
OBSERVE H1, 499.8025921 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
7.495
7.491
7.418
7.415
7.260
7.186
7.165
7.147
7.131
6.165
2.812
2.798
2.783
2.769
2.322
1.980
1.502
1.032
1.017
1.002
0.000
10
9
8
7
6
5
4
3
2
1
0
ppm

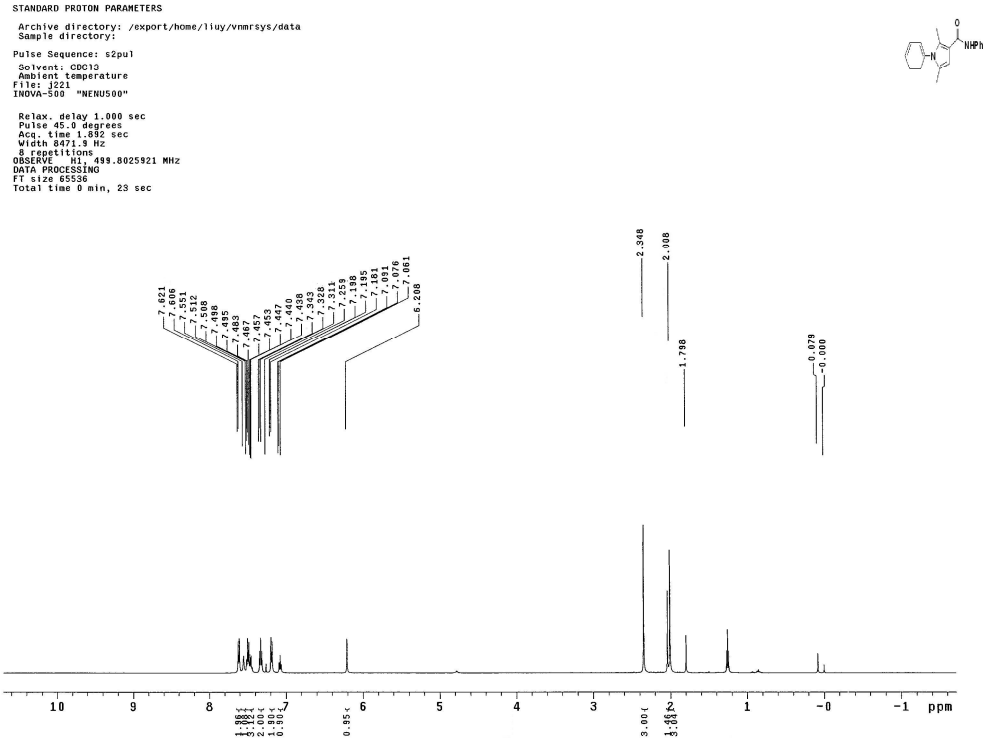

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: k306
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
256 repetitions
OBSERVE C13, 125.6754728 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
ppm

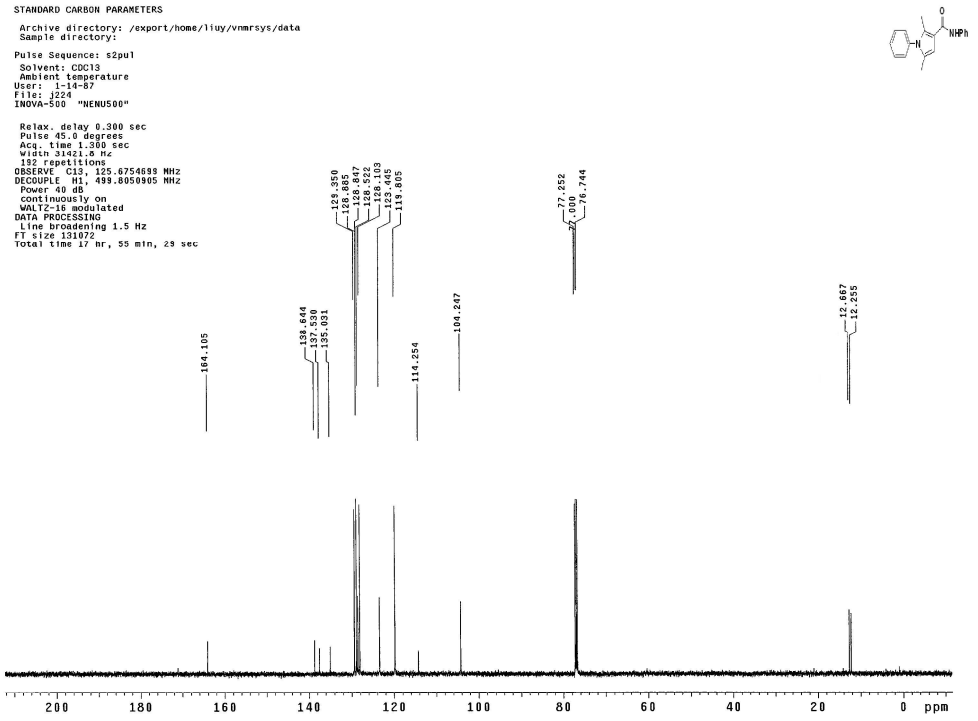
2c


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: k826
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8615.1 Hz
8 repetitions
OBSERVE H1, 499.8026265 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
NHPh
ppm

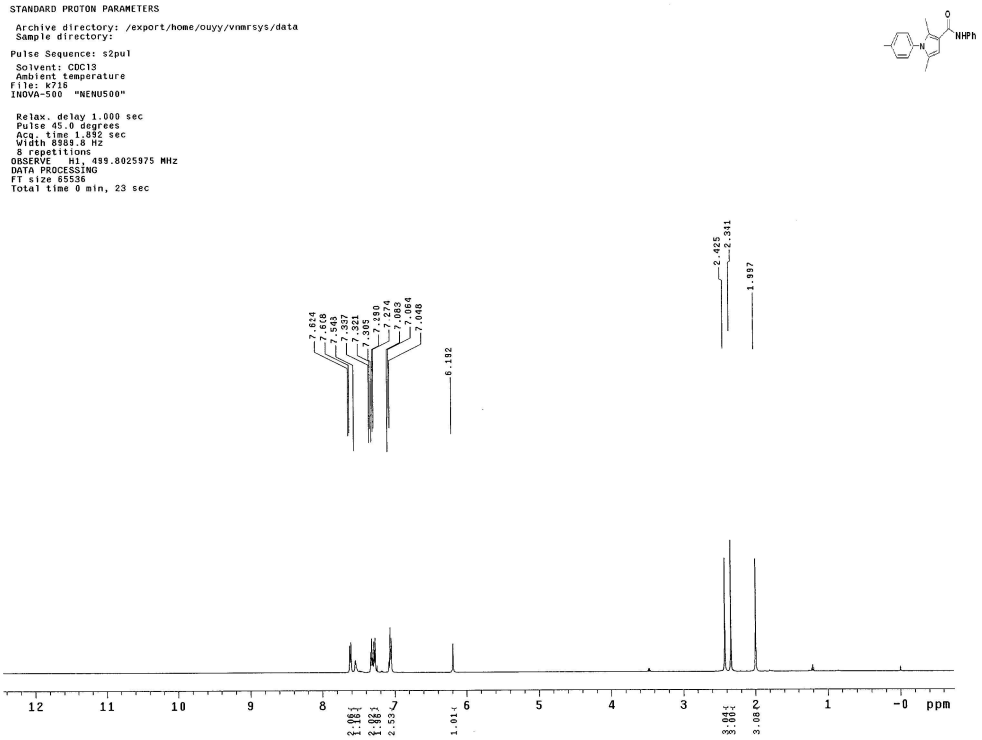

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: k827
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
6000 repetitions
OBSERVE C13, 125.6754666 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, 40 min, 56 sec
77.248
76.737
134.615
133.802
129.659
129.071
128.923
123.567
119.782
138.591
136.012
114.552
103.572
28.690
21.889
13.792
12.133
163.964
NHPh
Cl
220
200
180
160
140
120
100
80
60
40
20
0
ppm

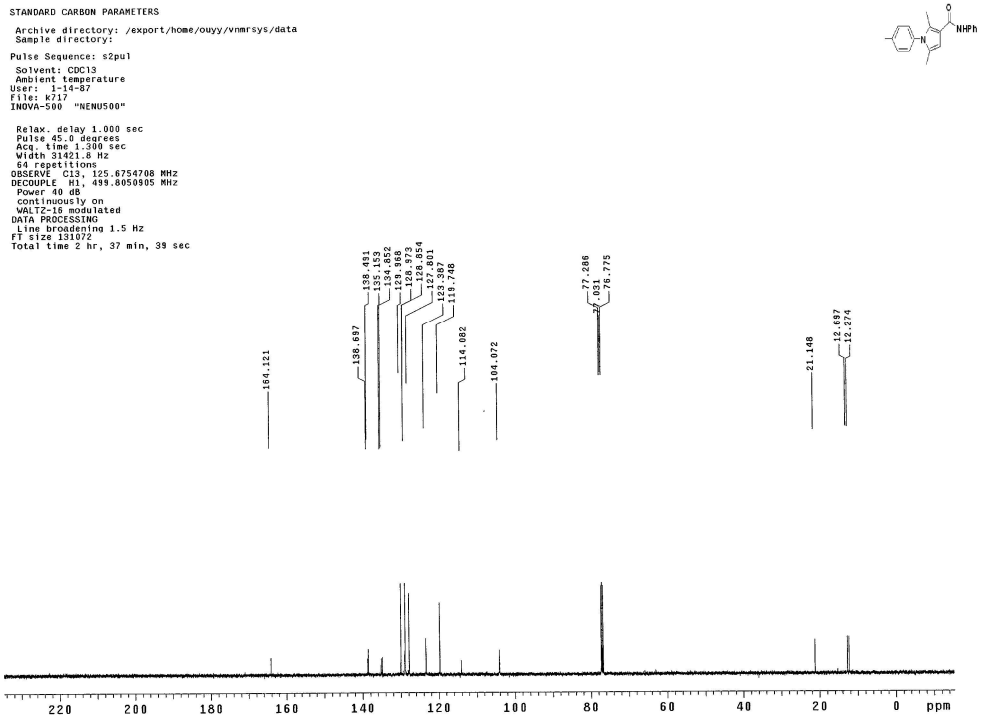
2d1


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: j221
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8471.9 Hz
8 repetitions
OBSERVE H1, 499.8025921 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
7.621
7.606
7.551
7.532
7.508
7.498
7.495
7.488
7.467
7.457
7.453
7.447
7.440
7.393
7.388
7.311
7.259
7.198
7.185
7.181
7.091
7.076
7.061
6.208
2.348
2.008
1.798
0.079
0.000
NHPh
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm

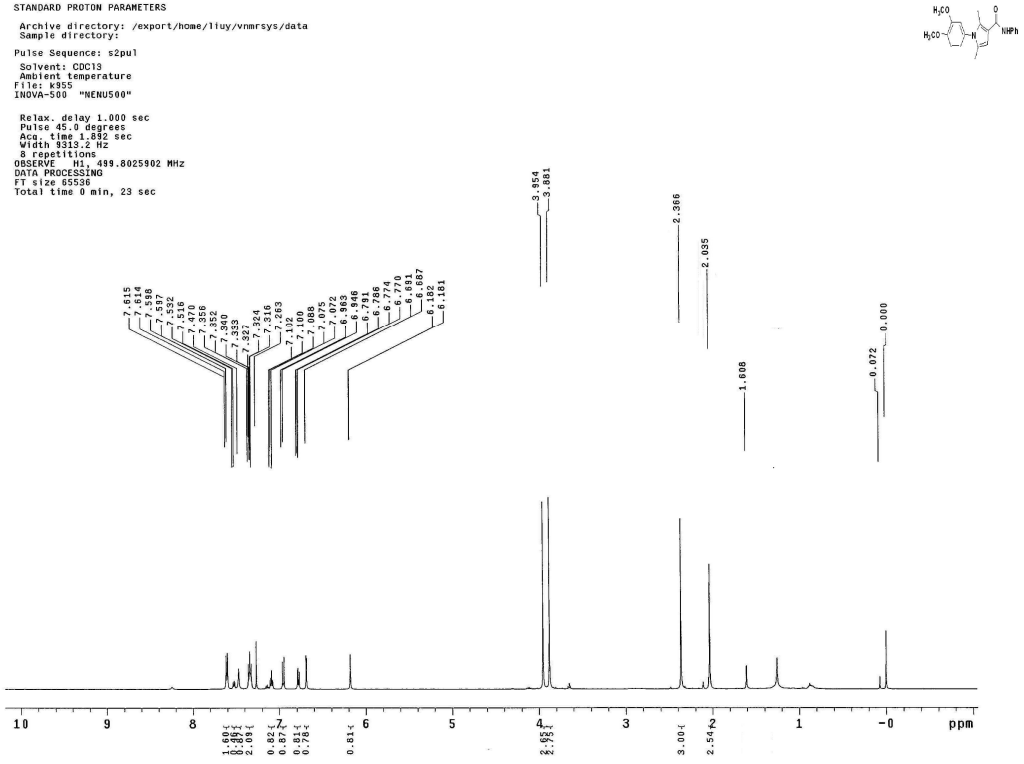

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: j224
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
192 repetitions
OBSERVE C13, 125.6754698 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 17 hr, 55 min, 29 sec
NHPh
164.105
138.644
137.530
135.031
129.350
128.885
128.847
128.522
128.103
123.465
119.805
114.254
104.247
77.252
77.000
76.744
12.667
12.255
200
180
160
140
120
100
80
60
40
20
0
ppm

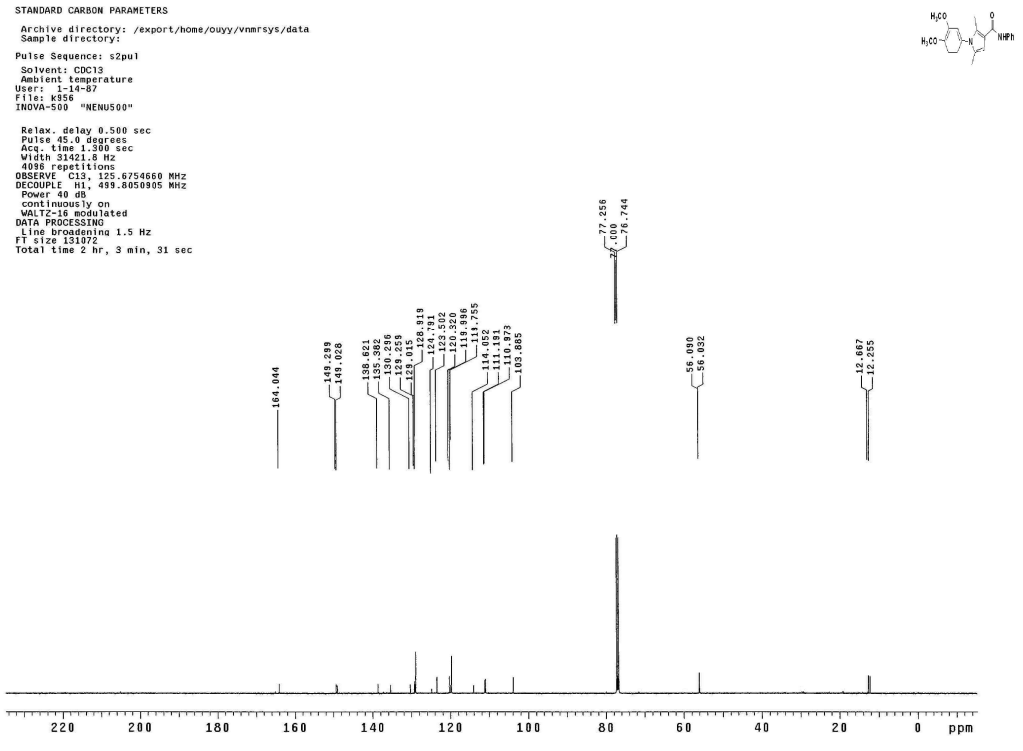
2d2


STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: k716
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8988.8 Hz
8 repetitions
OBSERVE H1, 499.8025975 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
NHPh
7.624
7.618
7.565
7.337
7.321
7.305
7.280
7.274
7.083
7.064
7.048
6.192
2.425
2.311
1.997
12
11
10
9
8
7
6
5
4
3
1
0
ppm

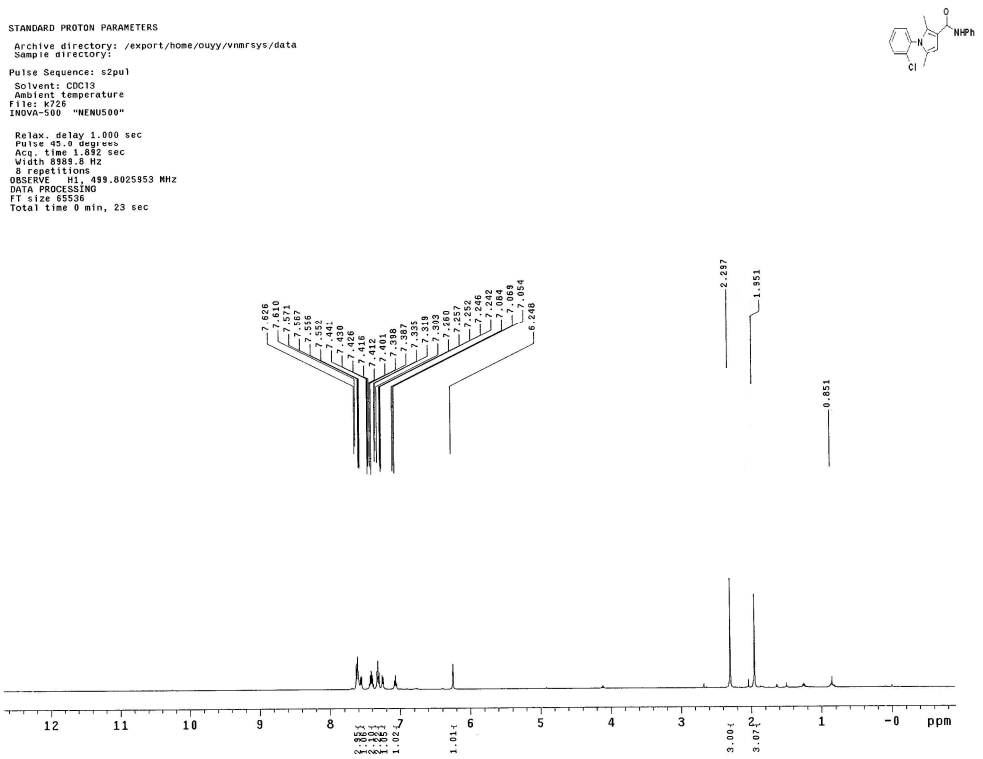

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: k717
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
64 repetitions
OBSERVE C13, 125.6754708 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, 37 min, 39 sec
NHPh
164.121
138.697
138.481
135.158
134.852
129.968
128.973
128.854
127.601
123.387
118.748
114.082
104.072
77.286
77.031
76.775
21.148
12.697
12.274
220
200
180
160
140
120
100
80
60
40
20
0
ppm

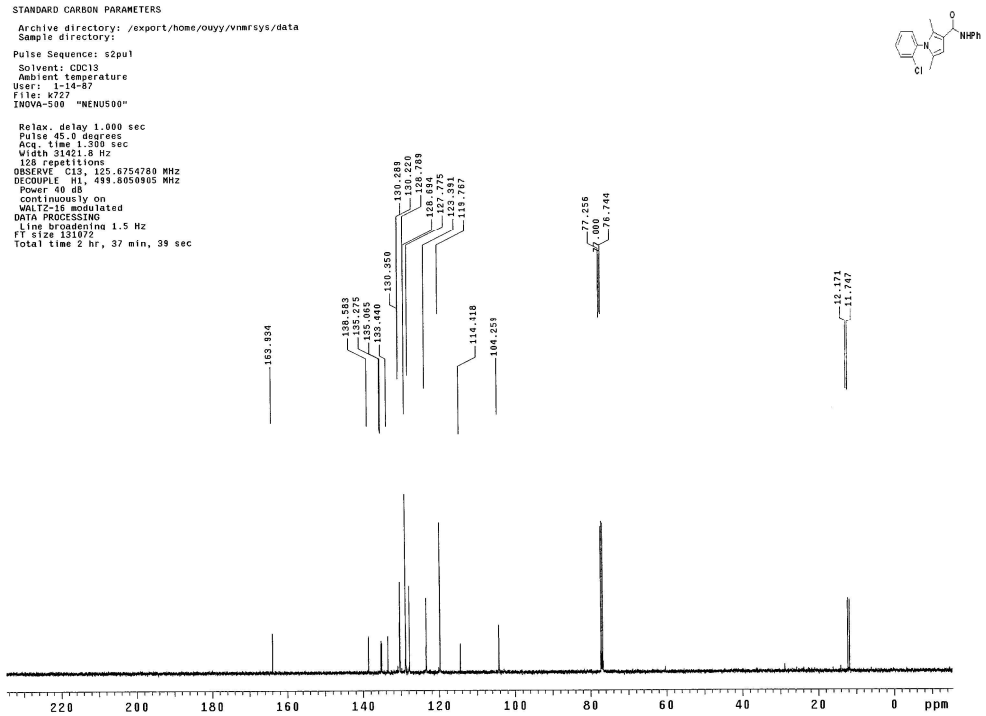
2d3


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: k955
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8318.2 Hz
8 repetitions
OBSERVE H1, 499.8025902 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
H3CO
H3CO
NHPh
7.615
7.614
7.598
7.597
7.532
7.516
7.470
7.356
7.352
7.340
7.333
7.327
7.324
7.285
7.263
7.112
7.100
7.088
7.075
7.072
6.963
6.946
6.791
6.786
6.774
6.770
6.681
6.667
6.182
6.181
3.854
3.681
2.366
2.035
1.608
0.072
0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm

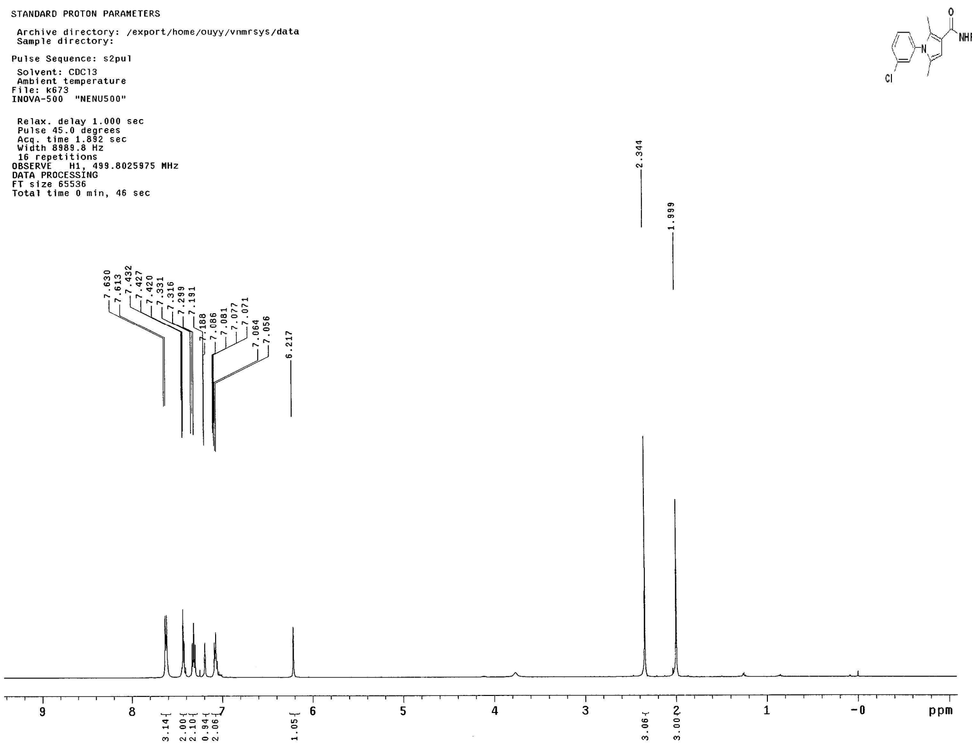

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: K956
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
4096 repetitions
OBSERVE C13, 125.6754660 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
H3CO
H3CO
O
NHPh
77.256
77.000
76.744
164.044
149.299
149.028
138.621
135.982
130.256
129.259
129.015
118.819
124.781
123.502
120.320
119.936
114.795
114.052
111.191
110.973
109.865
56.090
56.032
12.667
12.255
220
200
180
160
140
120
100
80
60
40
20
0
ppm

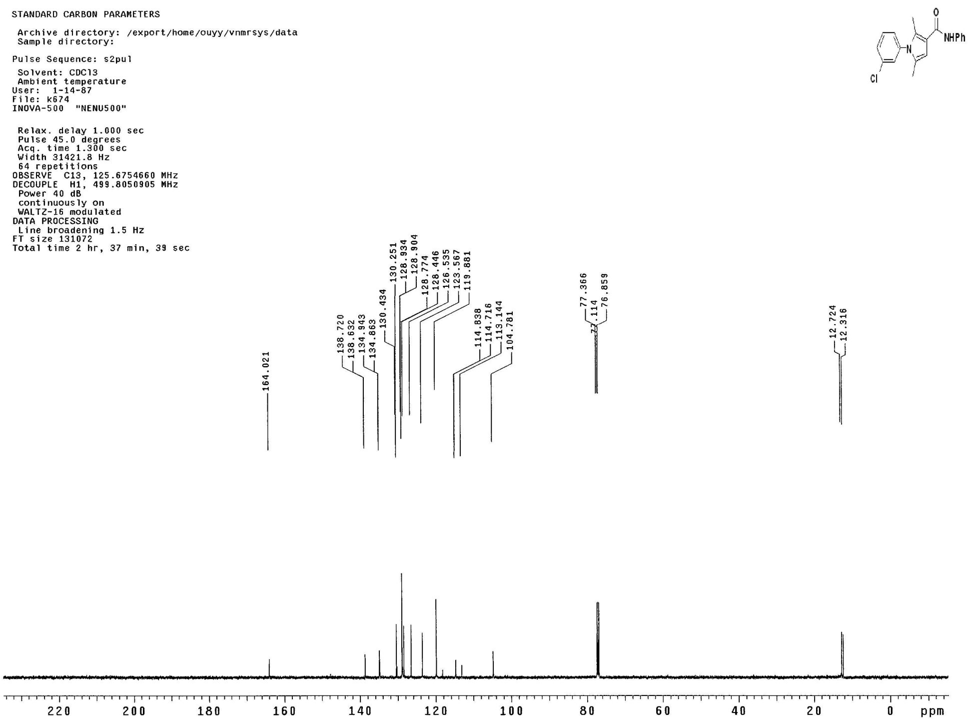
2d4


STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: k726
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8988.8 Hz
8 repetitions
OBSERVE H1, 499.8025953 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
NHPh
Cl
7.626
7.610
7.571
7.567
7.556
7.553
7.441
7.430
7.426
7.416
7.412
7.401
7.398
7.387
7.335
7.319
7.309
7.300
7.257
7.252
7.246
7.242
7.084
7.069
7.054
6.248
2.297
1.951
0.851
12
11
10
9
8
7
6
5
4
3
2
1
-0
ppm

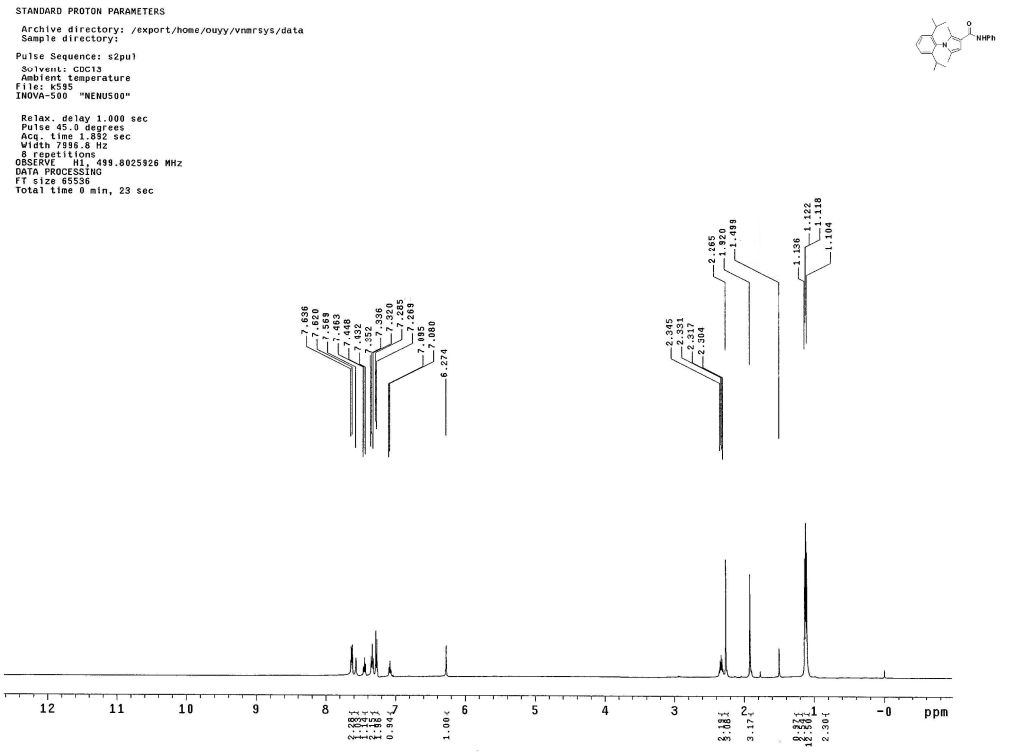

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: k727
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754780 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, 37 min, 39 sec
NHPh
Cl
163.934
138.583
135.275
135.005
133.440
130.350
130.289
129.020
128.789
128.694
127.775
123.391
119.767
114.418
104.259
77.256
77.000
76.744
12.171
11.747
220
200
180
160
140
120
100
80
60
40
20
0
ppm

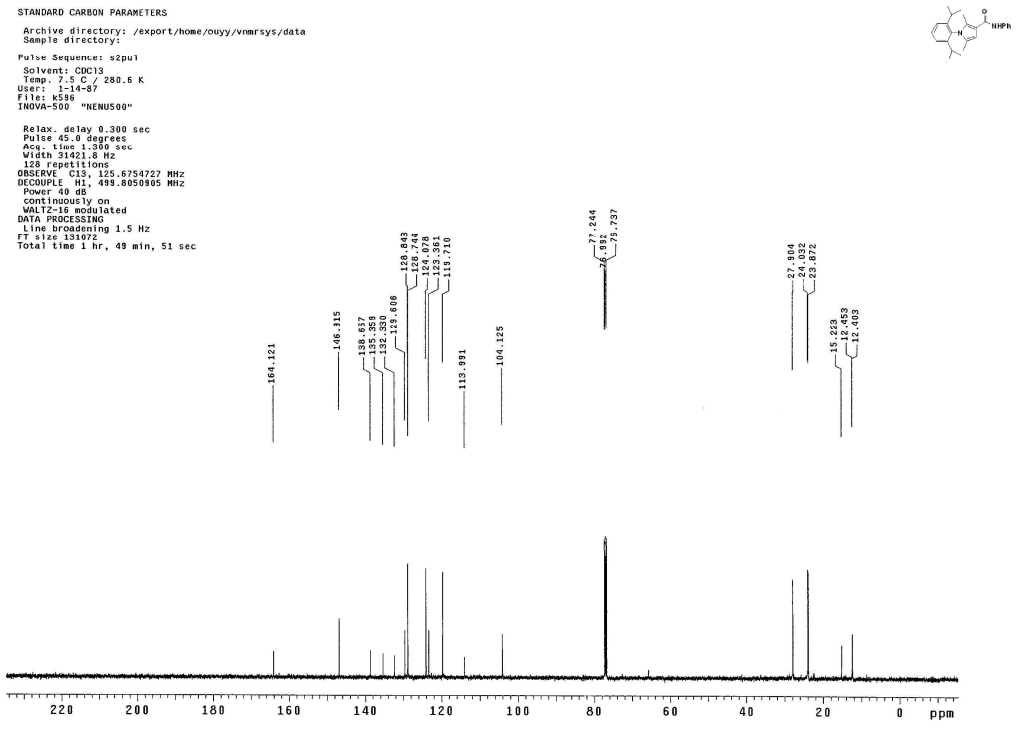
2d5


STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: k673
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8989.8 Hz
16 repetitions
OBSERVE H1, 499.8025975 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 46 sec
NHPh
Cl
7.630 7.613 7.432 7.427 7.420 7.331 7.316 7.299 7.181 7.188 7.086 7.081 7.077 7.071 7.064 7.056
6.217
2.344
1.999
3.14 2.00 2.10 0.94 2.06
1.05
3.06
3.00
ppm

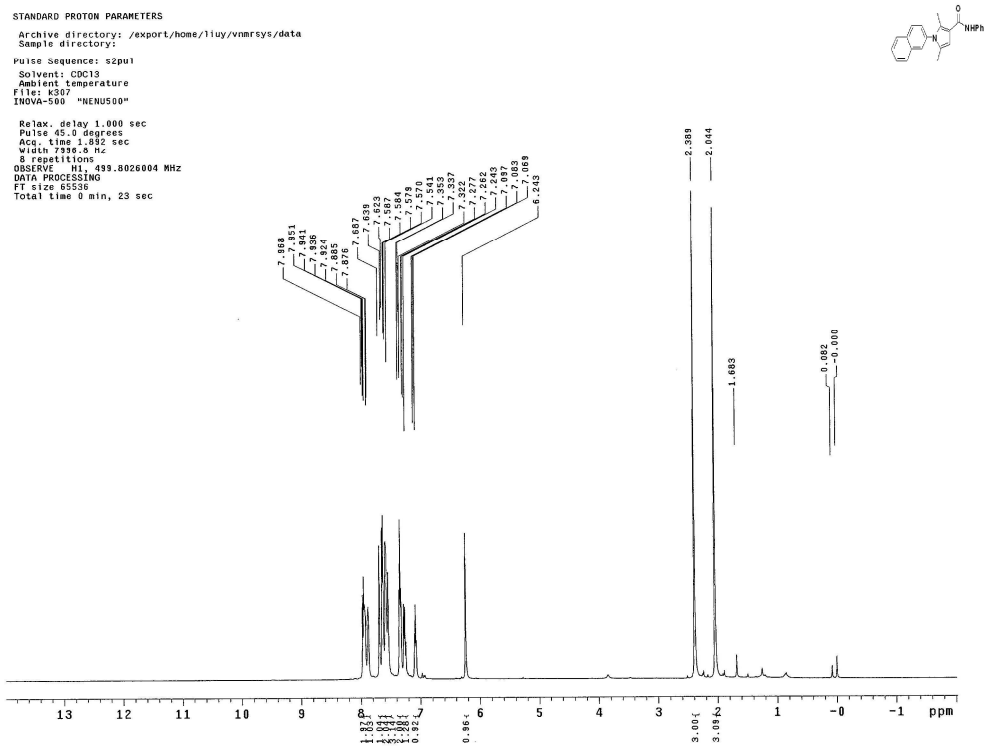

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: k674
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
64 repetitions
OBSERVE C13, 125.6754660 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, 37 min, 39 sec
NHPh
Cl
164.021
138.720 138.632 134.943 134.663 130.434
130.251 128.934 128.904 128.774 128.446 126.535 123.567 119.881
114.838 114.716 113.144 104.781
77.366 77.114 76.859
12.724 12.316
ppm

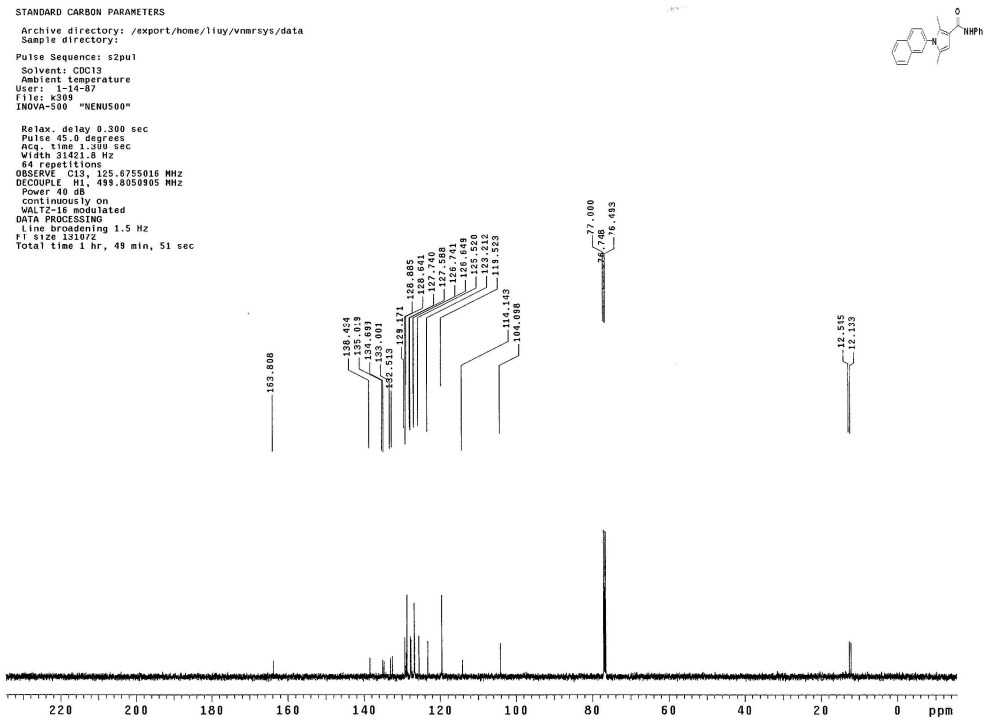
2d6


STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: k595
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.832 sec
Width 7996.8 Hz
8 repetitions
OBSERVE H1, 499.8025926 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
NHPh
7.636
7.620
7.589
7.469
7.448
7.432
7.352
7.336
7.320
7.285
7.269
7.165
7.080
6.274
2.345
2.331
2.317
2.304
2.265
1.920
1.499
1.136
1.122
1.118
1.104
ppm

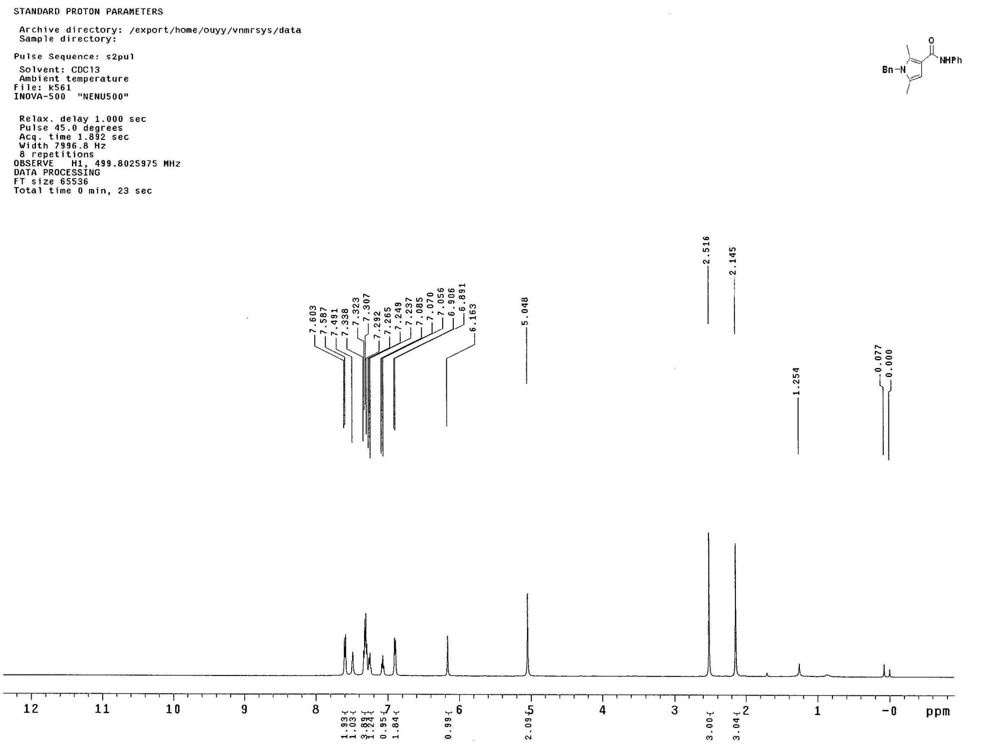

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 7.5 C / 280.6 K
User: 1-14-87
File: k596
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754727 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
NHPh
164.121
146.315
138.657
135.359
132.350
129.606
128.843
128.744
124.078
123.361
119.710
113.991
104.125
77.244
76.831
75.737
27.904
24.032
23.072
15.223
12.453
12.403
ppm

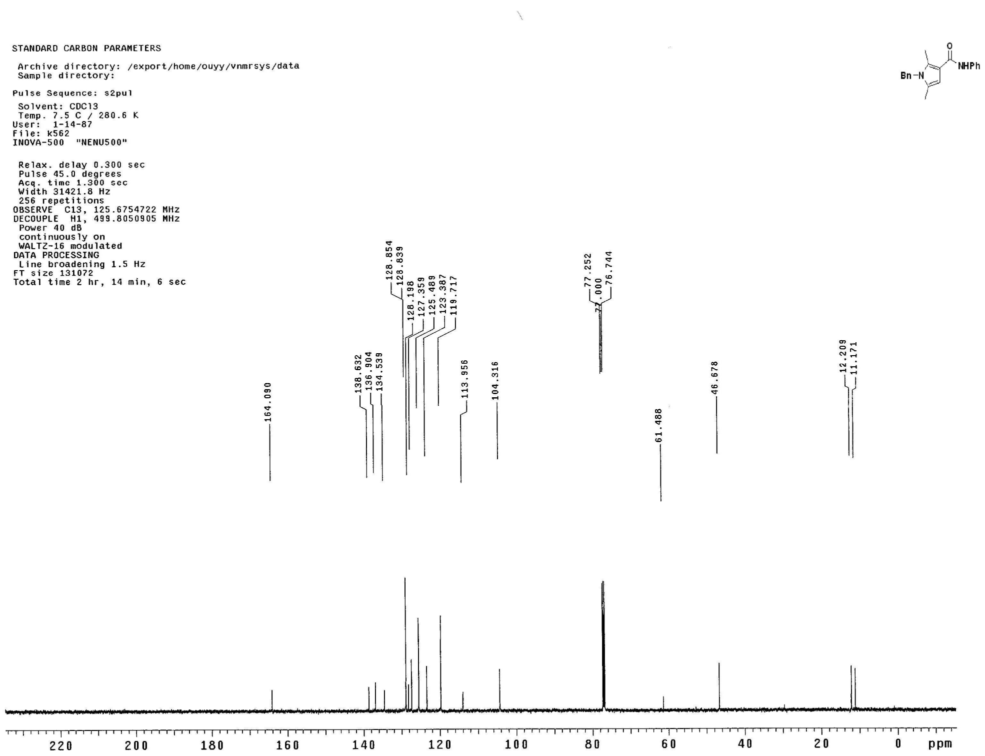
2d7


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: k307
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7936.5 Hz
8 repetitions
OBSERVE H1, 499.8026004 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
NHPh
ppm

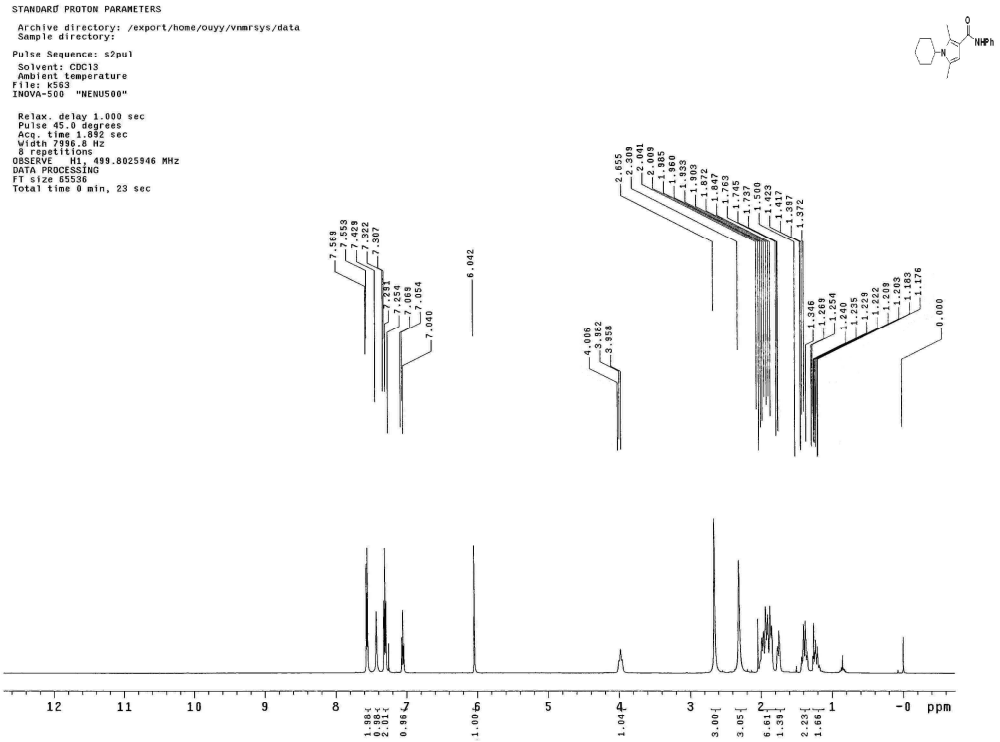

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: k309
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
64 repetitions
OBSERVE C13, 125.6755016 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
NHPh
ppm

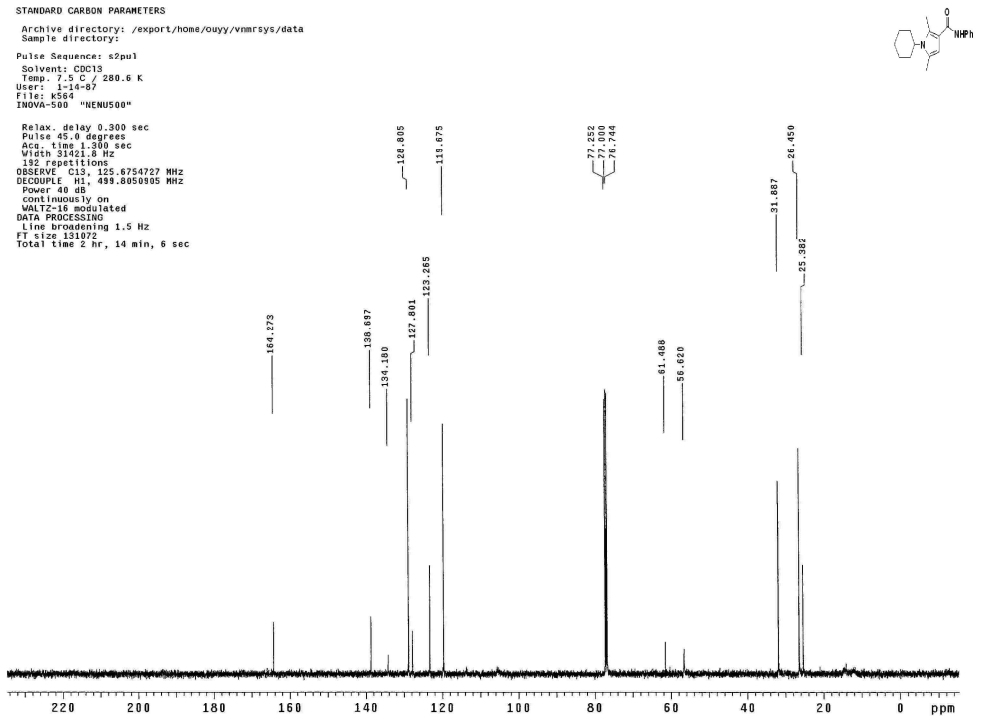
2d8


STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: k561
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, 499.8025975 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
Bn-N
NHPh
ppm

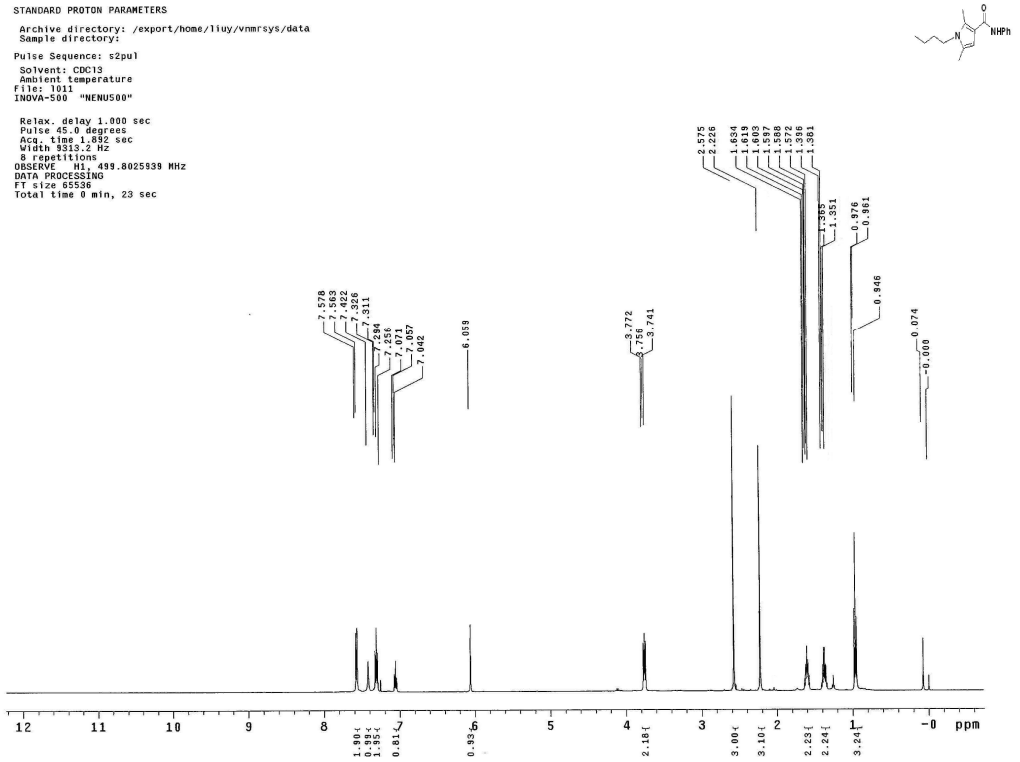

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 7.5 C / 280.6 K
User: 1-14-87
File: k562
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
256 repetitions
OBSERVE C13, 125.6754722 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, 14 min, 6 sec
Bn-N
NHPh
ppm

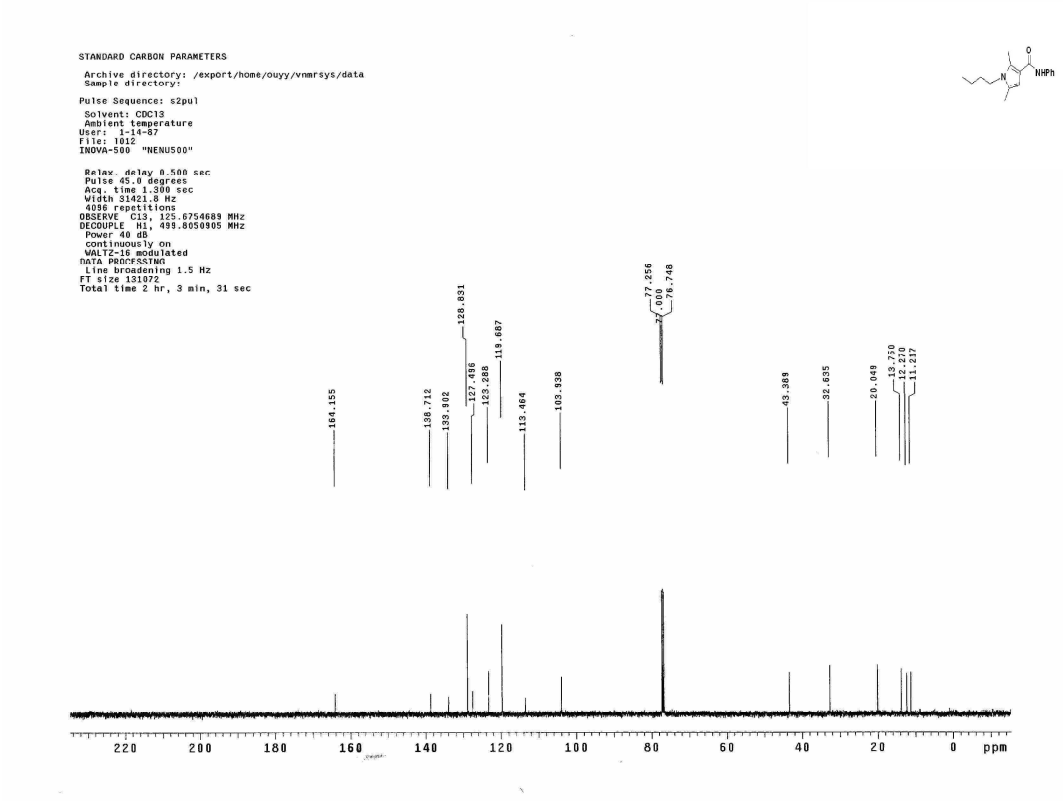
2d9


STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: k563
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, 499.8025946 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
NHPh
ppm

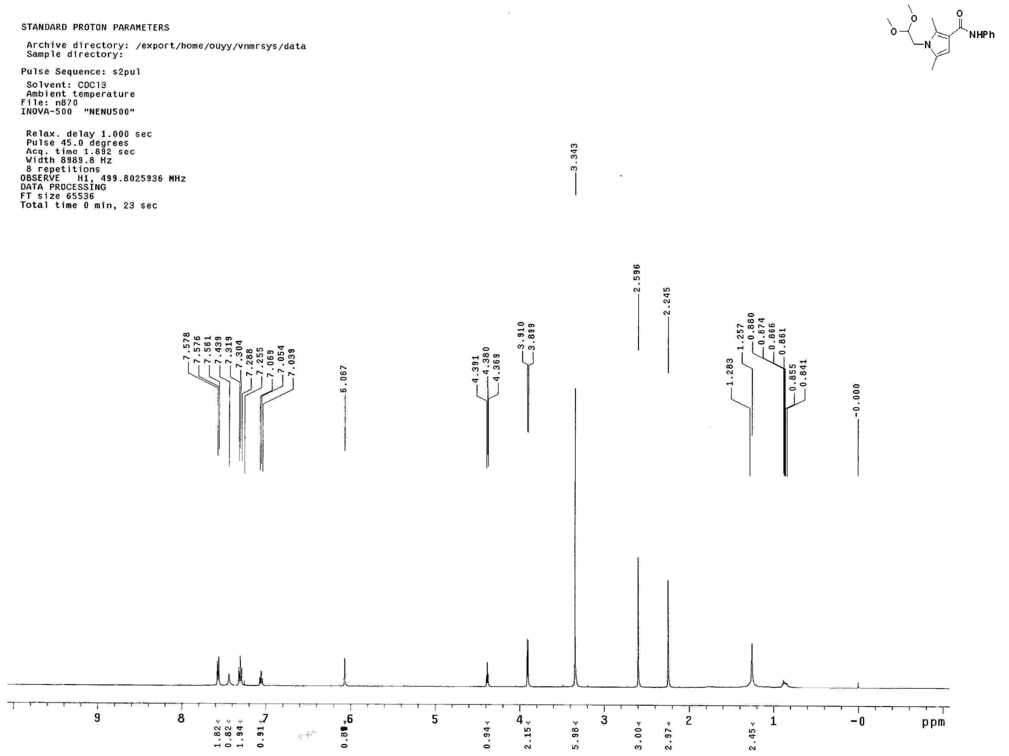

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 7.5 C / 280.6 K
User: 1-14-87
File: k564
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
192 repetitions
OBSERVE C13, 125.6754727 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, 14 min, 6 sec
NHPh
ppm

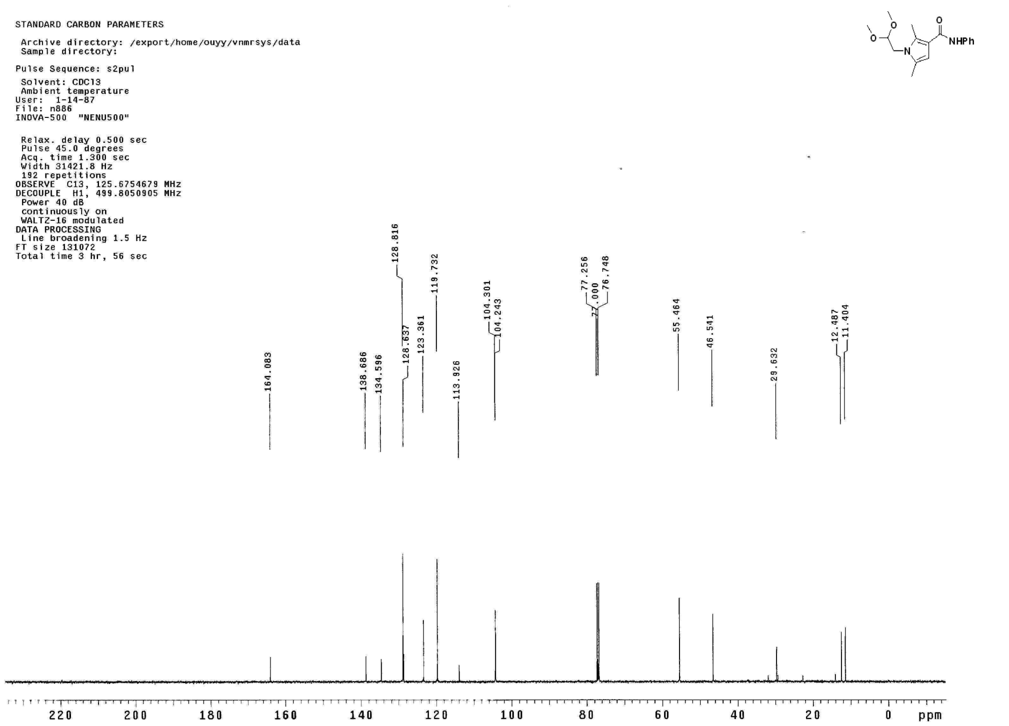
2d10


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 1011
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9313.2 Hz
8 repetitions
OBSERVE H1, 499.8025938 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
NHPh
ppm

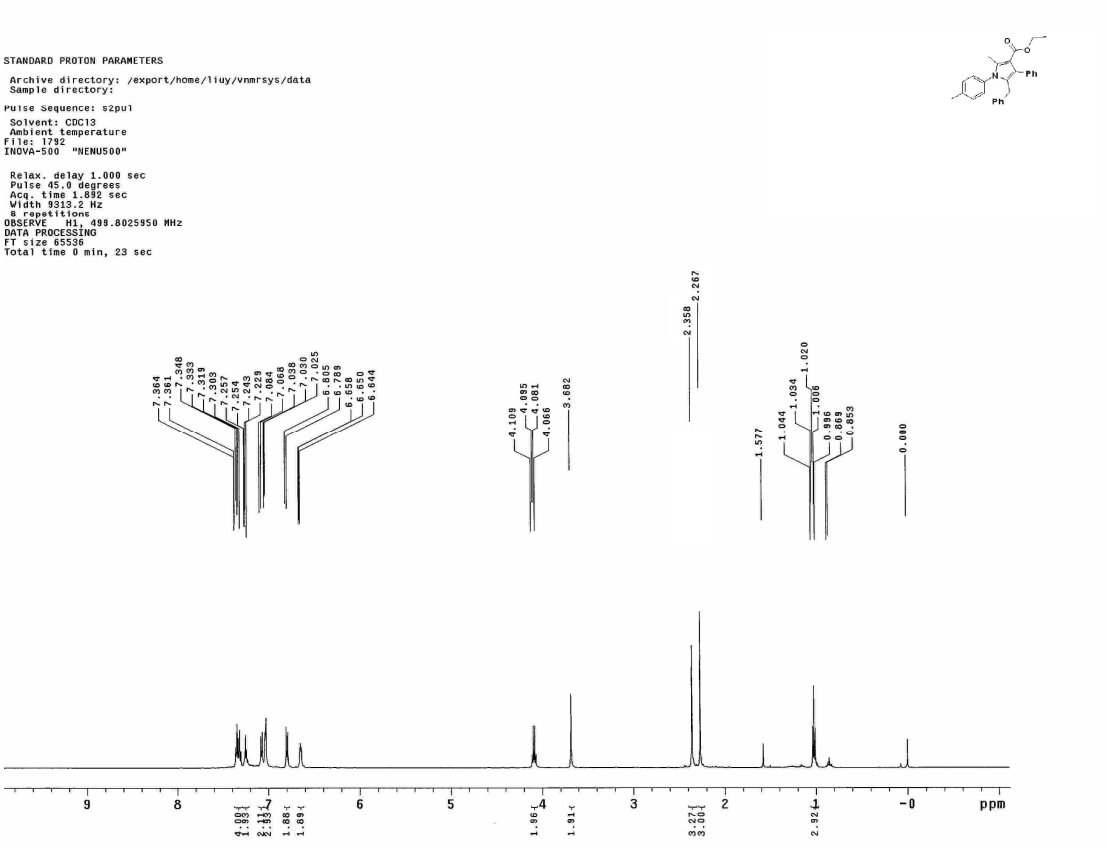

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: 1012
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
4096 repetitions
OBSERVE C13, 125.6754689 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
NHPh
ppm

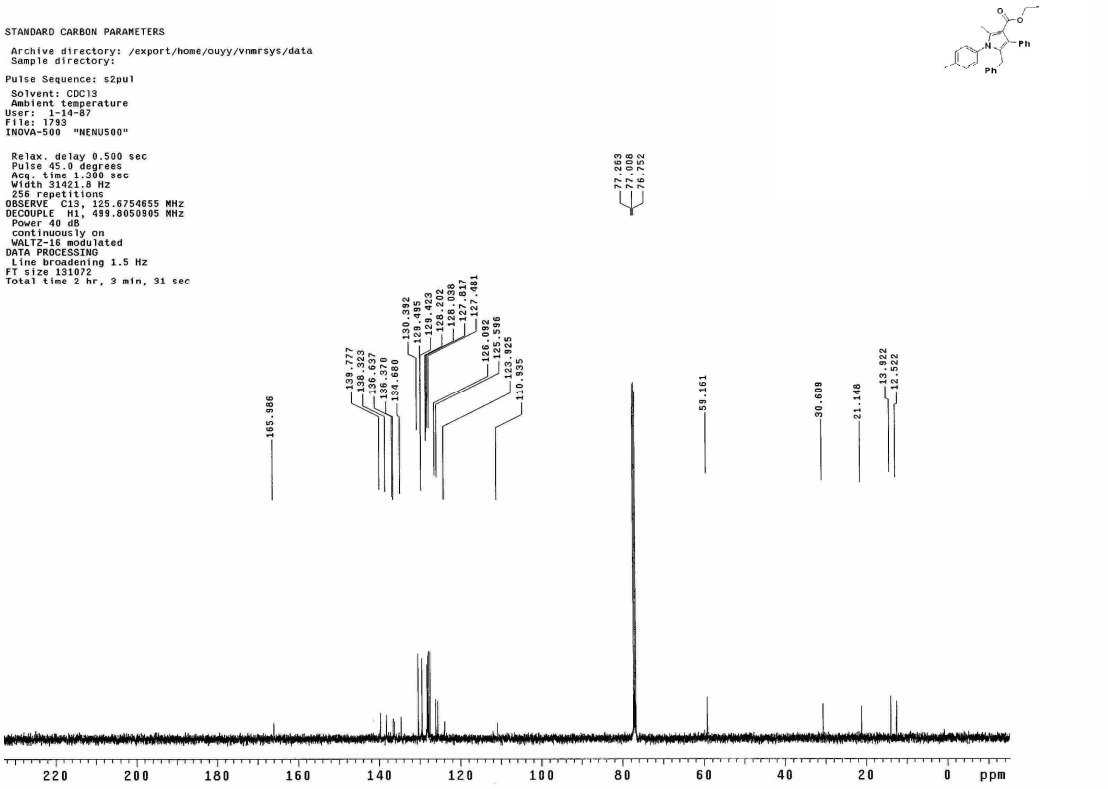
2d11


STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: n870
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8989.8 Hz
8 repetitions
OBSERVE H1, 499.8025936 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
NHPh
ppm

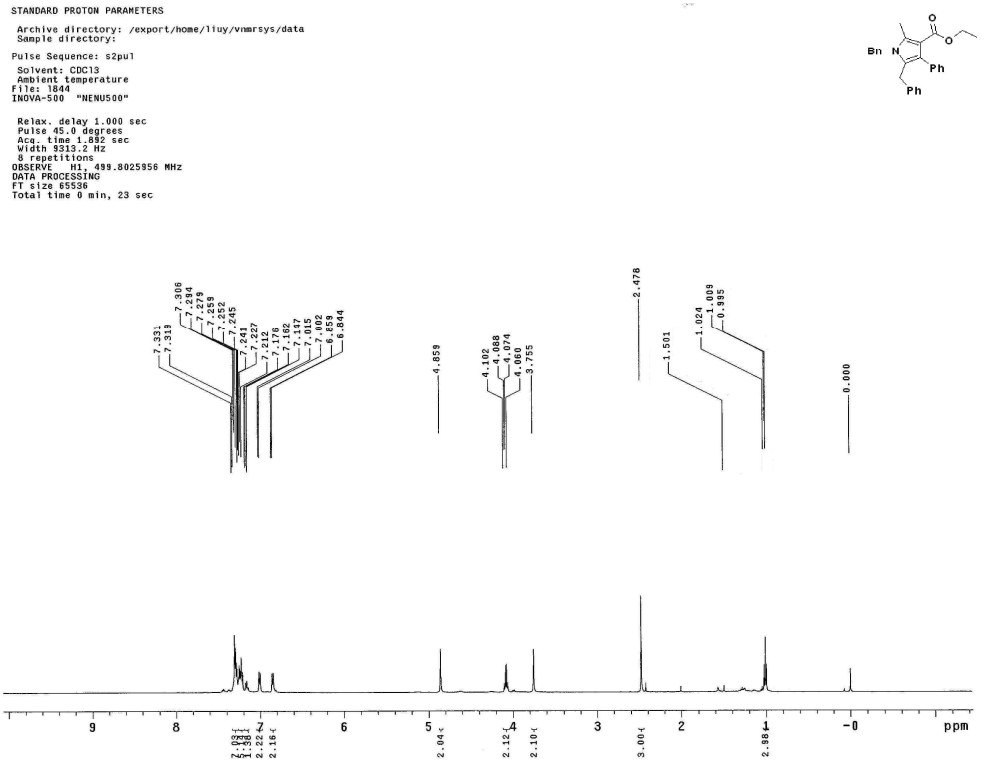

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: n886
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
192 repetitions
OBSERVE C13, 125.6754679 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 3 hr, 56 sec
NHPh
ppm

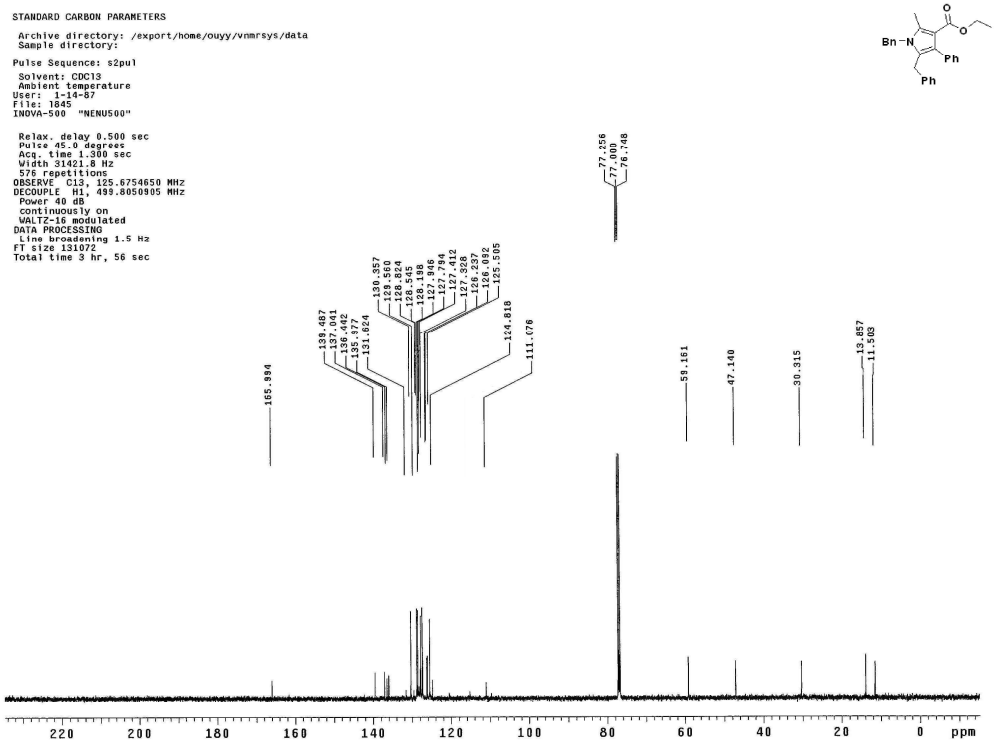
3a


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 1792
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8313.2 Hz
8 repetitions
OBSERVE H1, 499.8025950 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
Ph
Ph
7.364
7.361
7.348
7.333
7.319
7.303
7.297
7.254
7.243
7.229
7.084
7.068
7.038
7.030
7.025
6.805
6.789
6.658
6.650
6.644
4.109
4.095
4.081
4.066
3.682
2.358
2.267
1.577
1.044
1.034
1.020
1.006
0.986
0.869
0.853
0.000
ppm

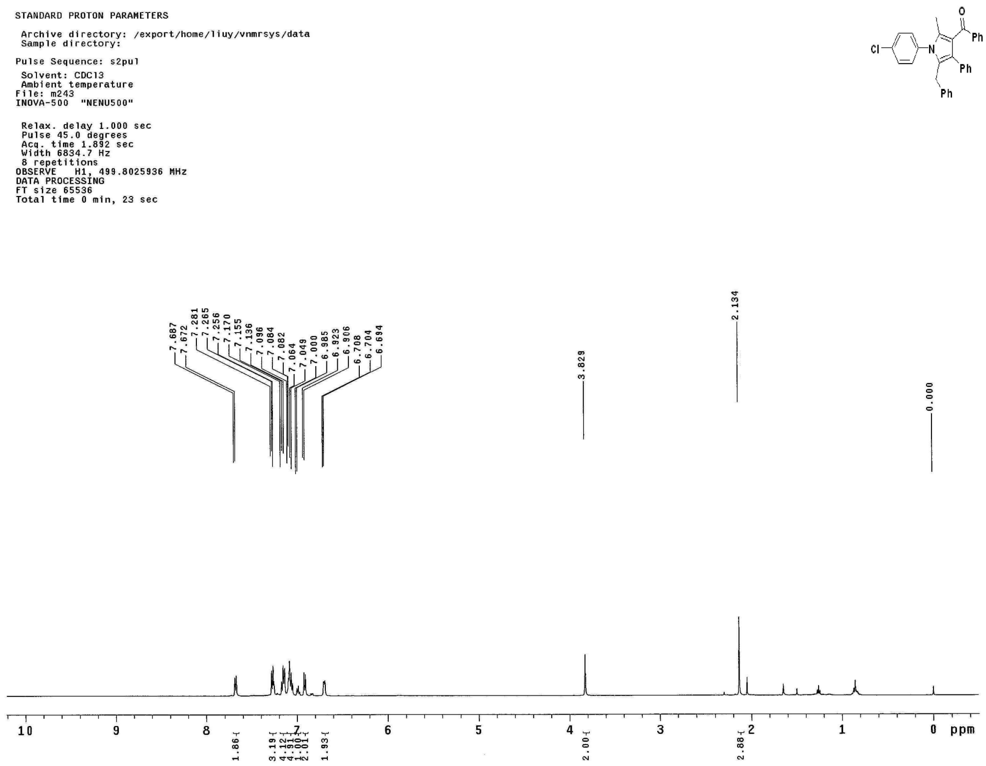

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: 1793
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
256 repetitions
OBSERVE C13, 125.6754655 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
Ph
Ph
77.263
77.008
76.752
165.986
139.777
138.323
136.637
136.370
134.680
130.392
129.495
129.423
128.202
128.088
127.487
127.481
126.092
125.586
123.925
110.935
59.161
30.609
21.148
13.922
12.522
ppm

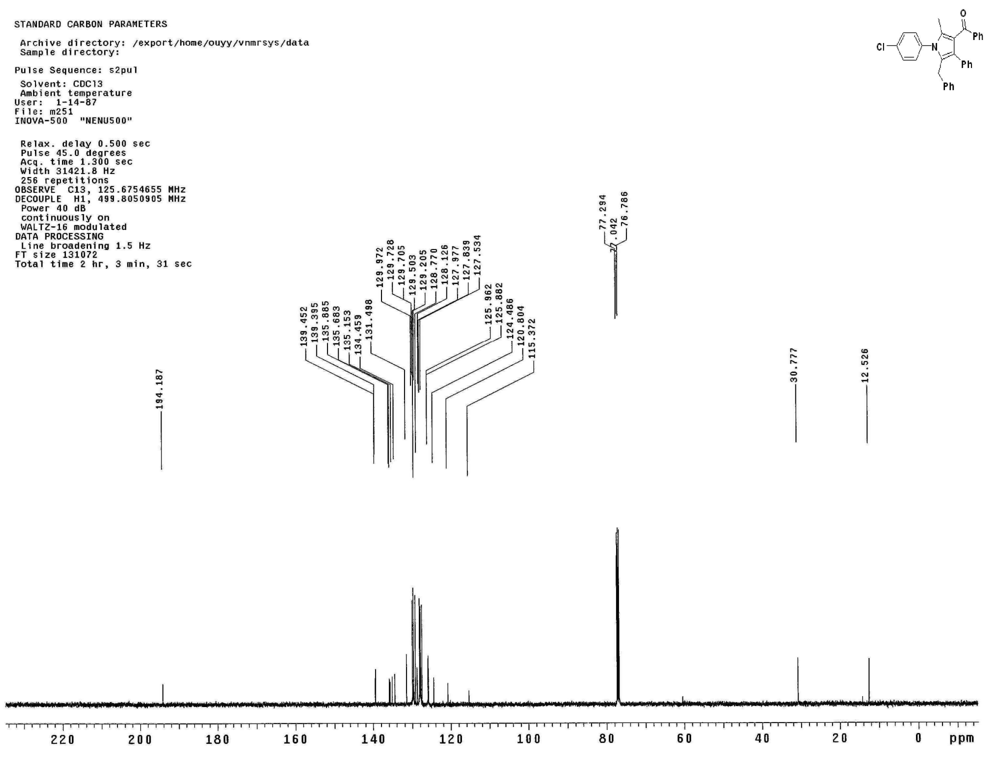
3b


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 1844
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9313.2 Hz
8 repetitions
OBSERVE H1, 499.8025956 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
Bn
N
O
O
Ph
Ph
ppm

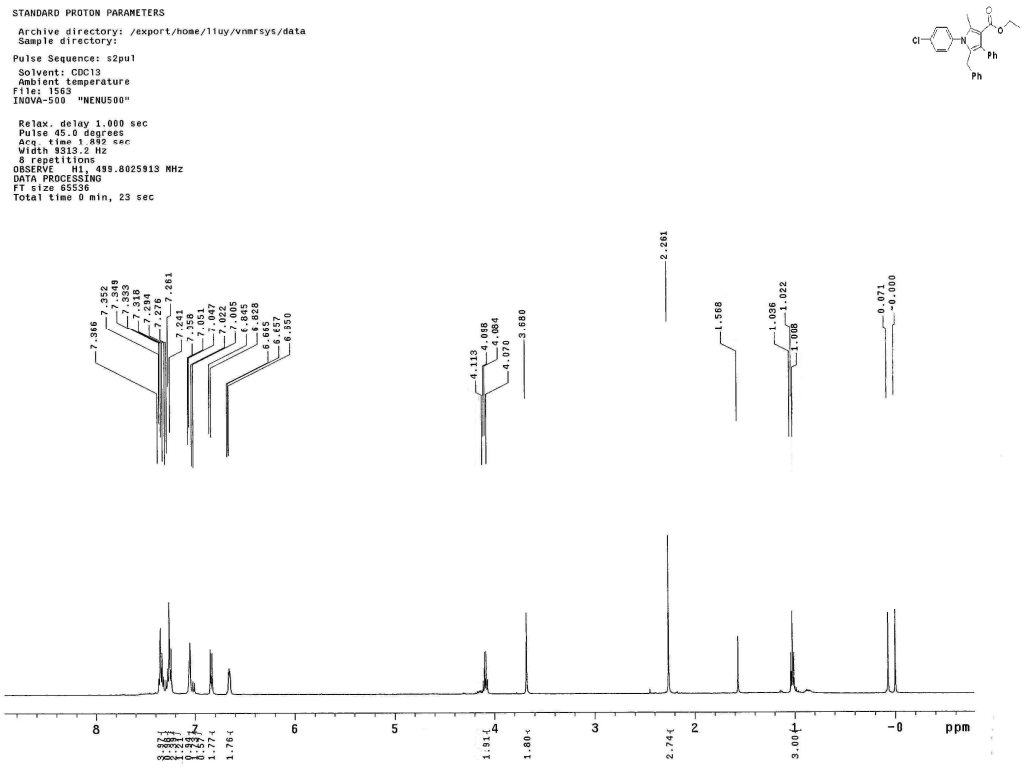

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: 1845
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
576 repetitions
OBSERVE C13, 125.6754650 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 3 hr, 56 sec
Bn
N
O
O
Ph
Ph
ppm

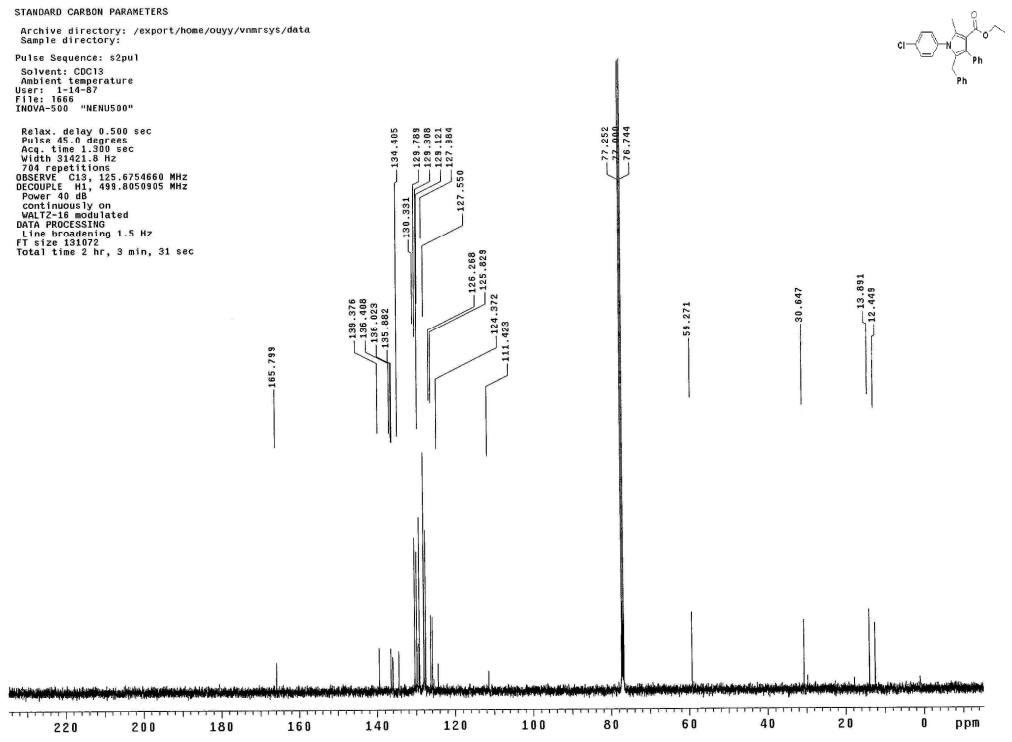
3c


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: m243
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 6834.7 Hz
8 repetitions
OBSERVE H1, 499.8025936 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
Cl
N
O
Ph
Ph
Ph
ppm

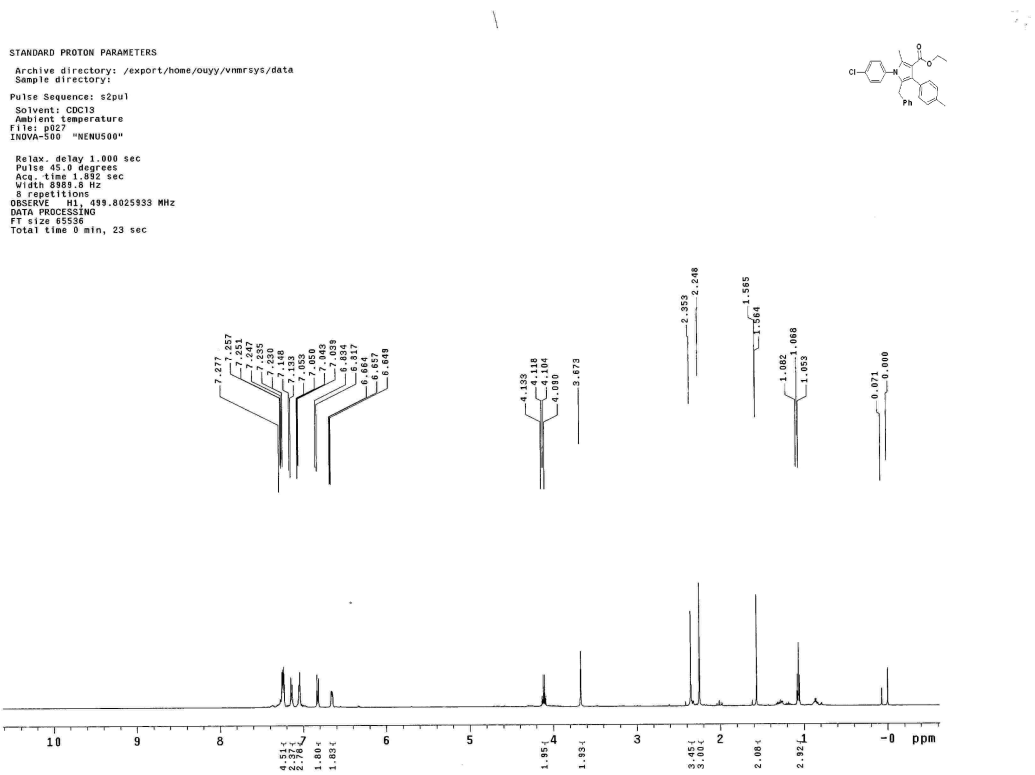

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: m251
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
256 repetitions
OBSERVE C13, 125.6754655 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
Cl
N
O
Ph
Ph
Ph
ppm

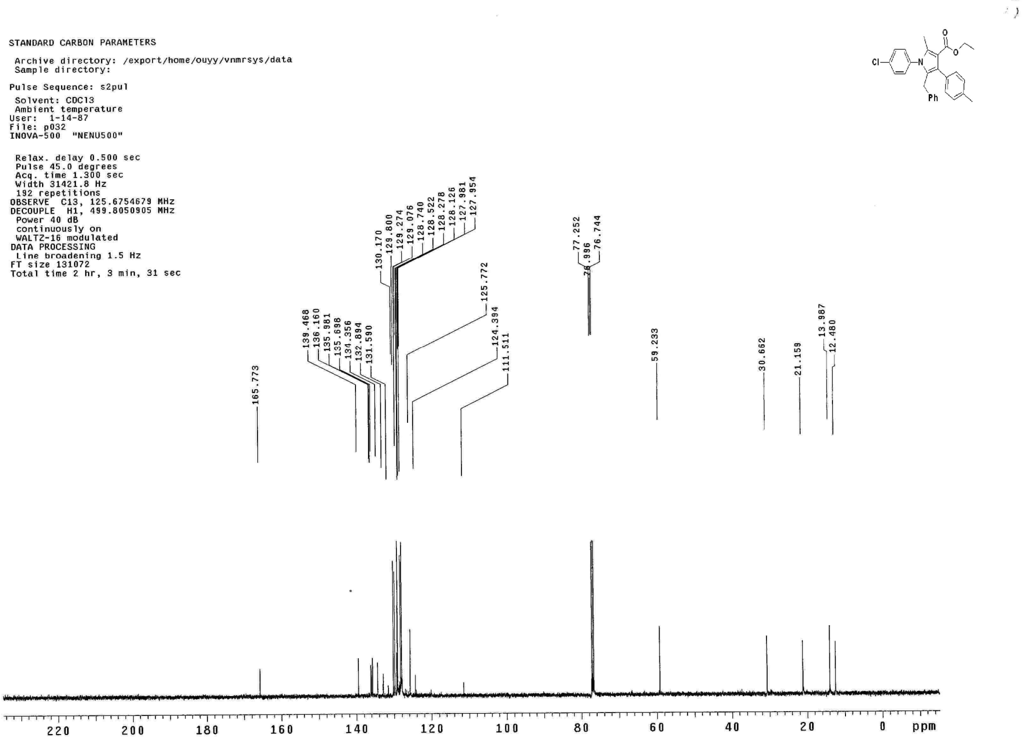
3d


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 1563
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9313.2 Hz
8 repetitions
OBSERVE H1, 499.8025913 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
ppm

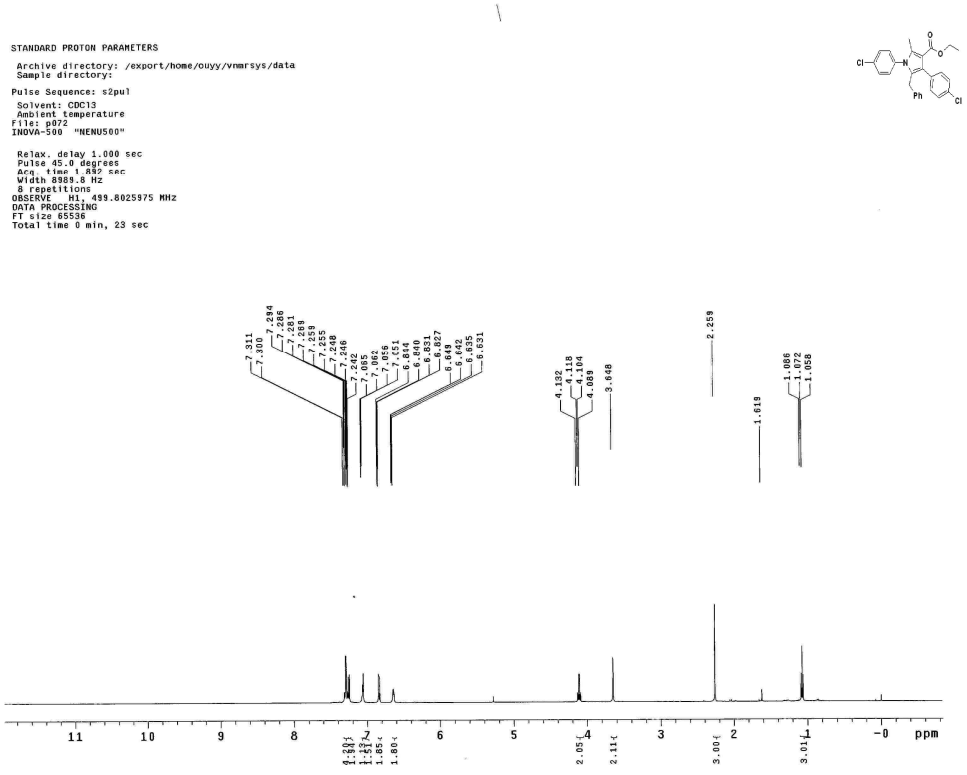

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: 1666
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
704 repetitions
OBSERVE C13, 125.6754660 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
ppm

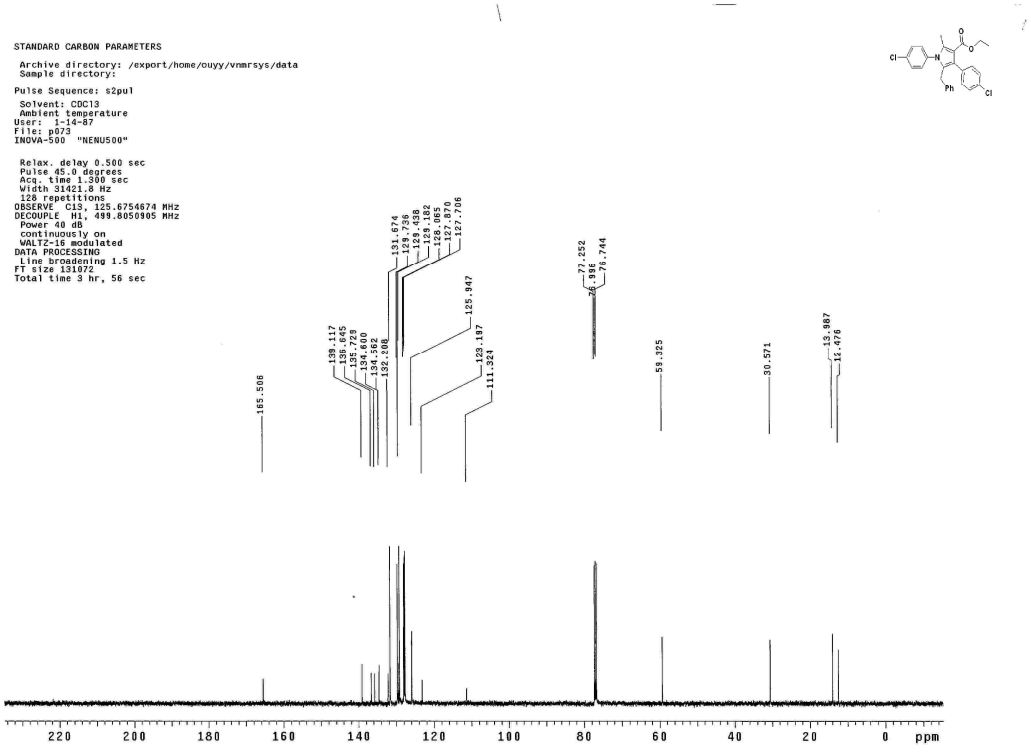
3e


STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: p027
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8989.8 Hz
8 repetitions
OBSERVE H1, 499.8025933 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
ppm

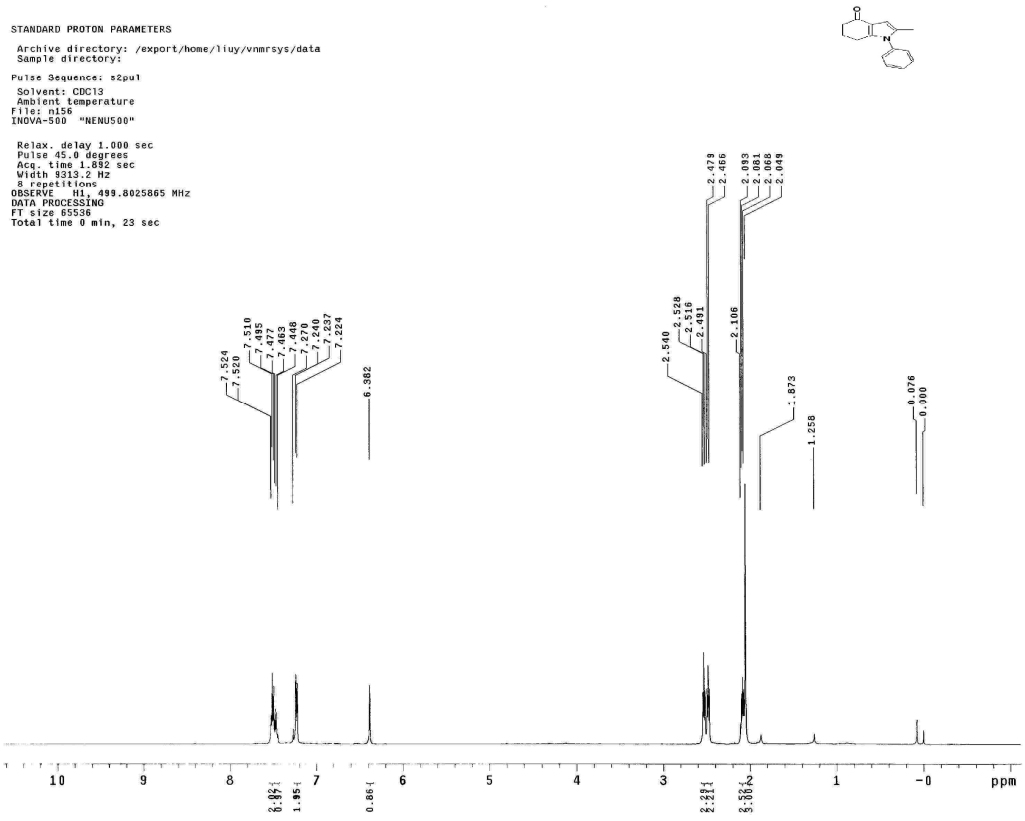

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: p032
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
192 repetitions
OBSERVE C13, 125.6754679 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
ppm

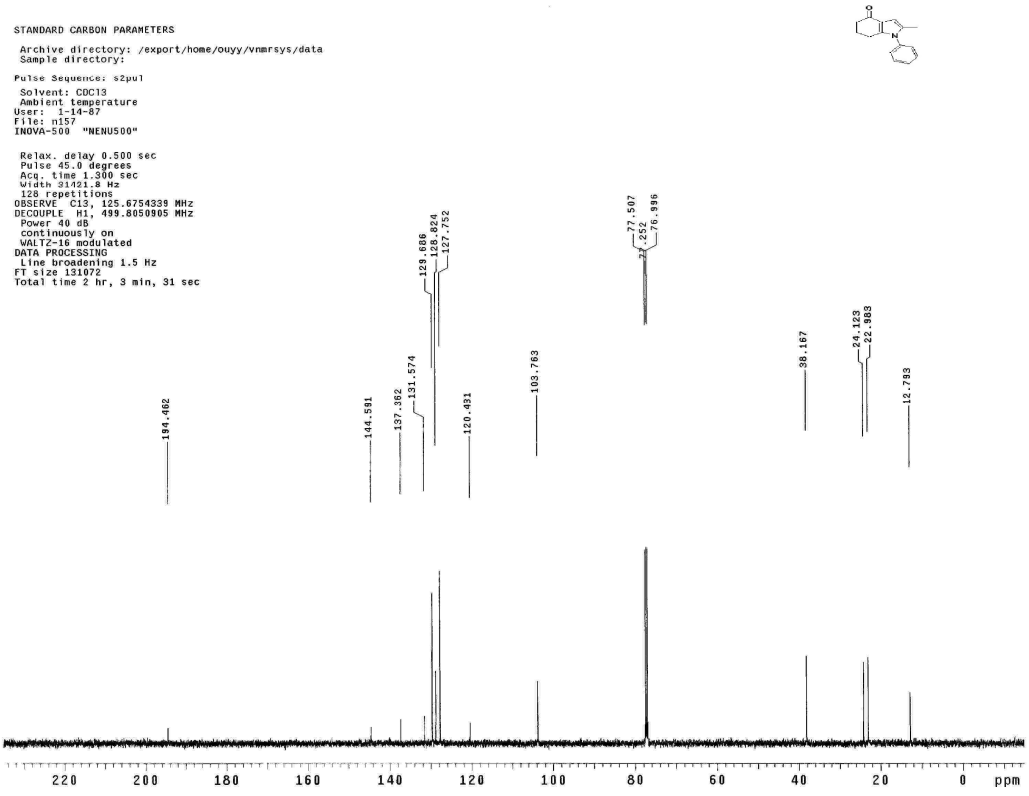
3f


STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: p072
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.882 sec
Width 8989.8 Hz
8 repetitions
OBSERVE H1, 499.8025975 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
7.311
7.300
7.294
7.286
7.281
7.269
7.259
7.255
7.248
7.240
7.242
7.085
7.065
7.056
7.051
6.844
6.840
6.831
6.827
6.648
6.640
6.635
6.631
4.132
4.118
4.104
4.089
3.648
2.259
1.619
1.086
1.072
1.058
ppm

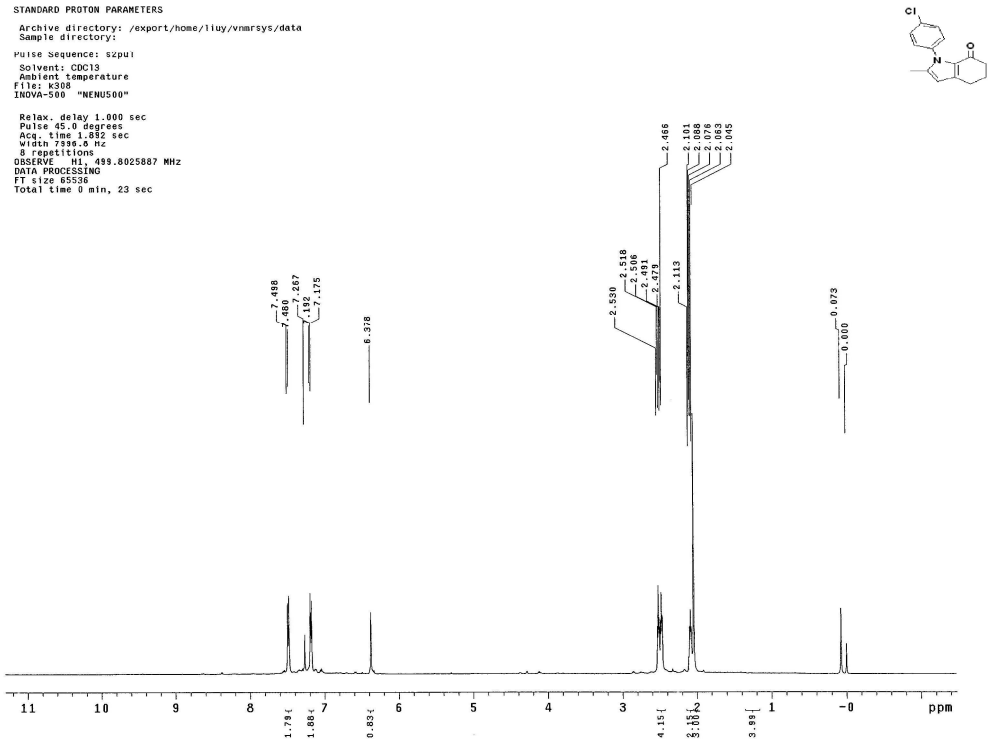

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: p073
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754674 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 3 hr, 56 sec
165.506
138.117
136.645
135.729
134.600
134.552
132.208
131.674
129.738
129.468
128.182
128.065
127.870
127.706
125.947
123.197
111.324
77.252
76.744
59.325
30.571
13.987
14.476
ppm

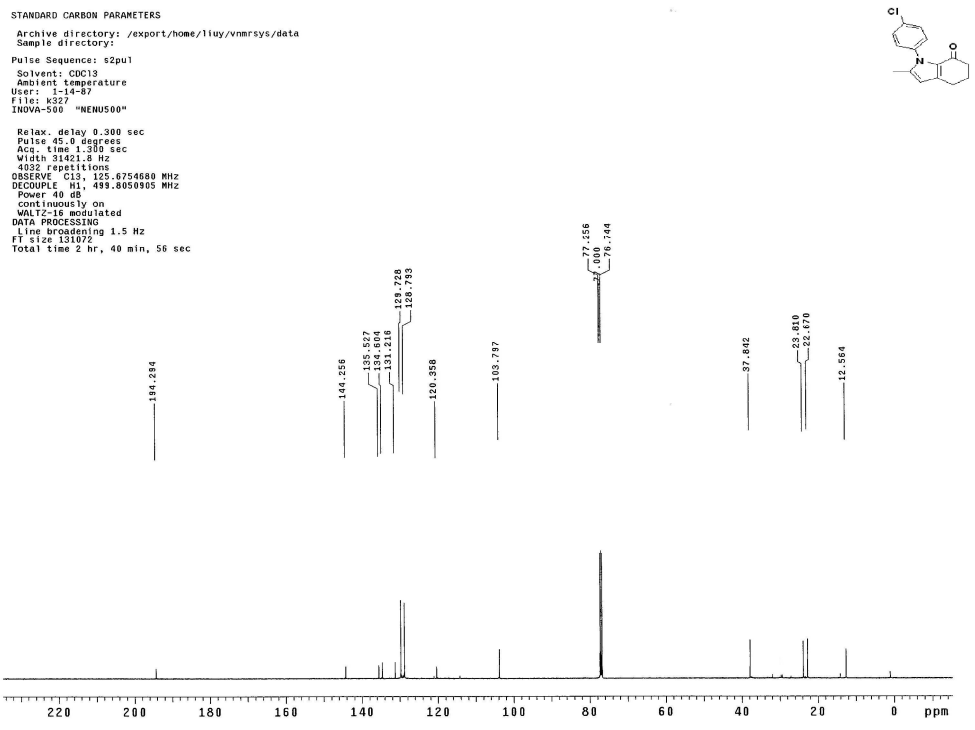
4a


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: n156
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8313.2 Hz
8 repetitions
OBSERVE H1, 499.8025865 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
7.524
7.520
7.510
7.495
7.477
7.463
7.448
7.270
7.260
7.237
7.224
6.382
2.540
2.528
2.516
2.491
2.479
2.466
2.106
2.093
2.081
2.068
2.049
1.873
1.258
0.076
0.000
ppm

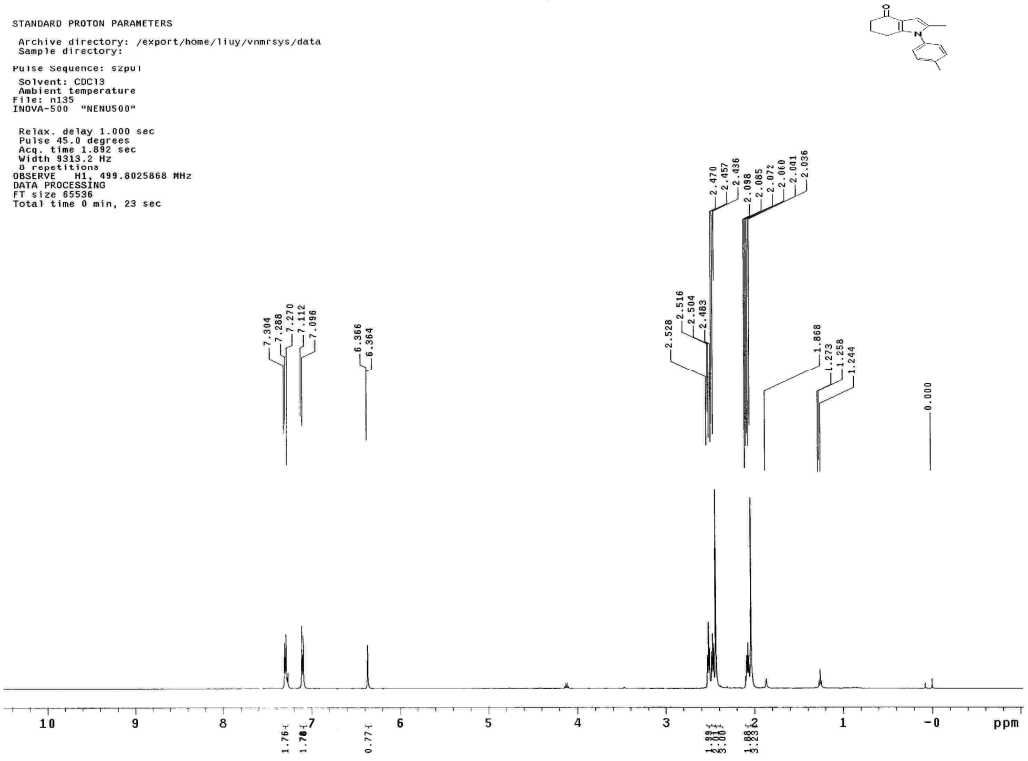

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: n157
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 21421.8 Hz
128 repetitions
OBSERVE C13, 125.6754339 MHz
DECOUPLE H1, 499.8050805 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
194.462
144.591
137.382
131.574
129.686
128.824
127.752
120.431
103.763
77.507
77.252
76.996
38.167
24.123
22.983
12.783
ppm

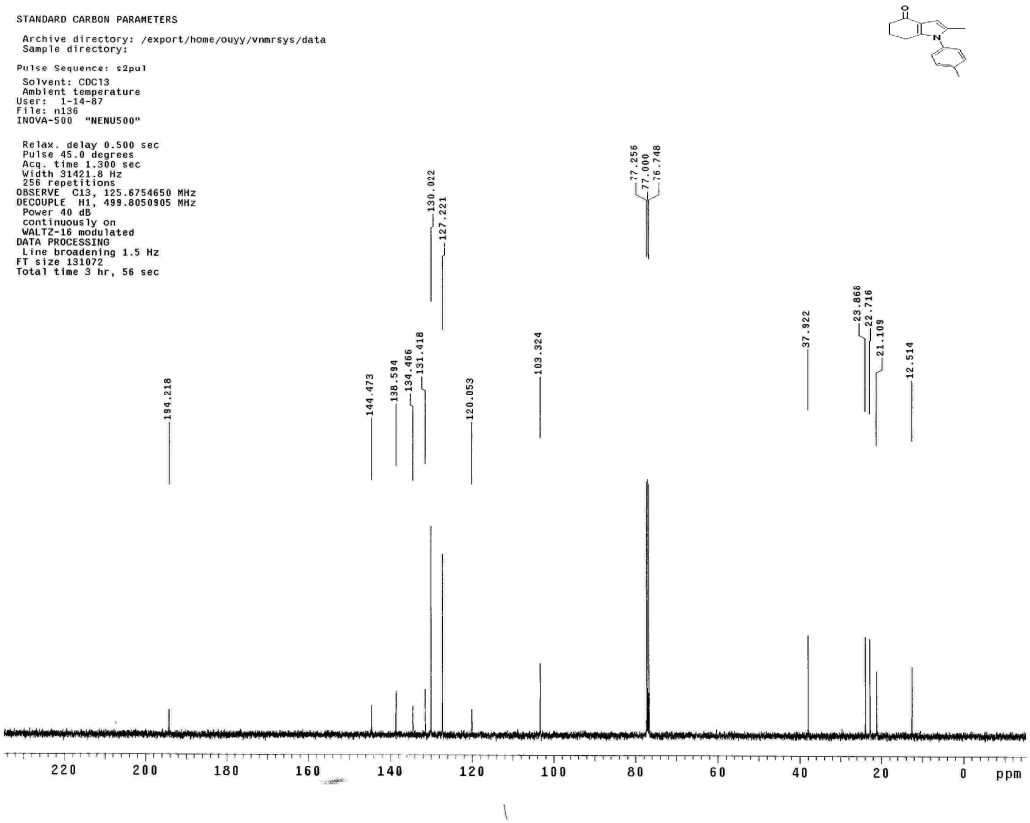
4b


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: k308
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, 499.8025887 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
Cl
O
N
7.498
7.480
7.267
7.192
7.175
6.378
2.466
2.101
2.088
2.076
2.063
2.045
2.530
2.518
2.506
2.491
2.479
2.113
0.073
0.000
11
10
9
8
7
6
5
4
3
2
1
-0
ppm
1.79
1.88
0.83
4.15
3.10
3.89

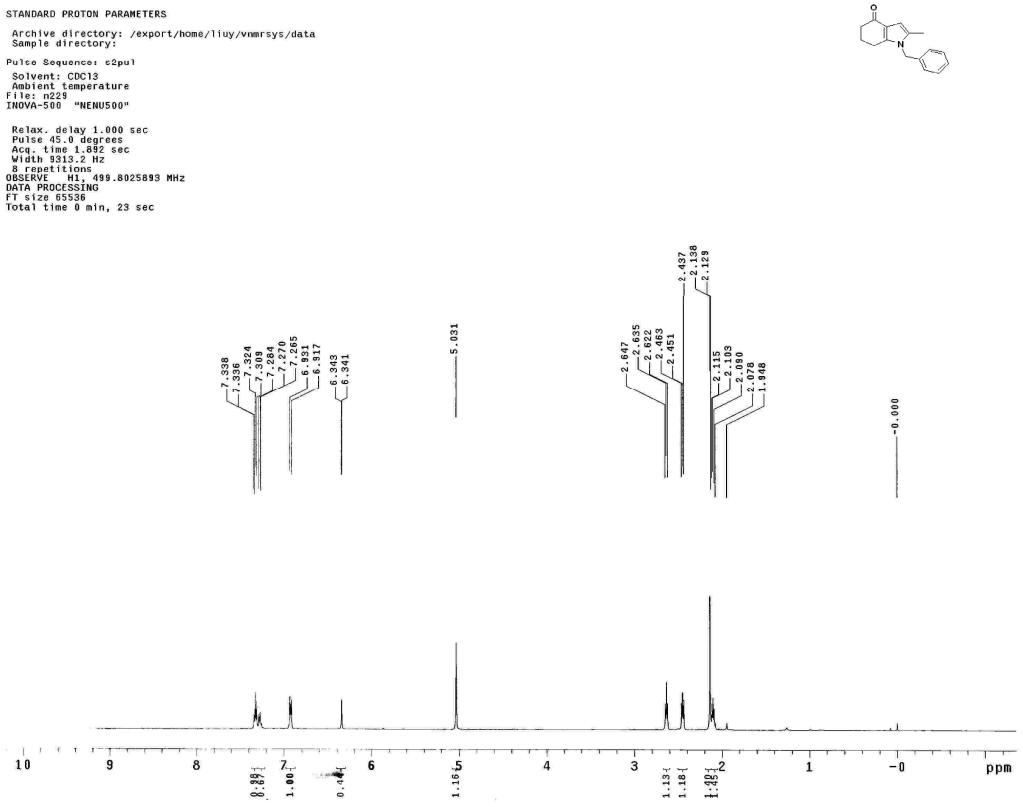

STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: k327
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
4032 repetitions
OBSERVE C13, 125.6754680 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, 40 min, 56 sec
Cl
O
N
194.284
144.256
135.527
134.608
134.218
129.728
128.793
120.358
103.797
77.156
77.100
76.744
37.842
23.810
22.670
12.564
220
200
180
160
140
120
100
80
60
40
20
0
ppm

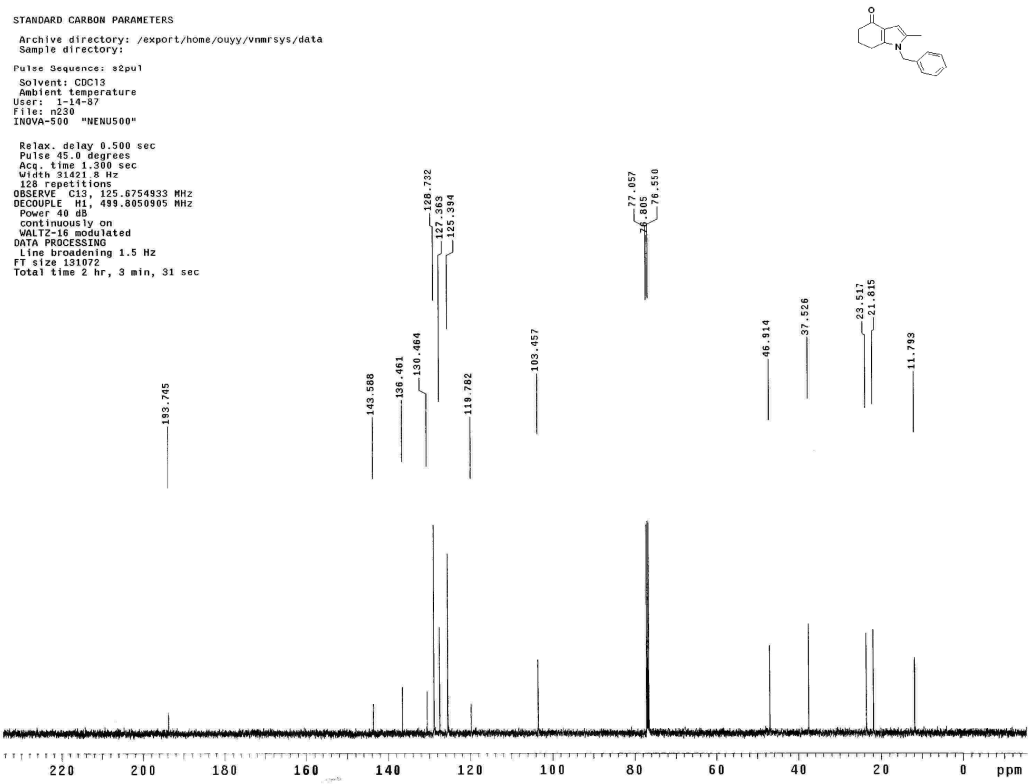
4c


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: n135
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9313.2 Hz
8 repetitions
OBSERVE H1, 499.8025868 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
7.304
7.288
7.270
7.112
7.096
6.366
6.364
2.528
2.516
2.504
2.483
2.470
2.457
2.436
2.098
2.085
2.072
2.060
2.041
2.036
1.868
1.273
1.258
1.244
0.000
ppm

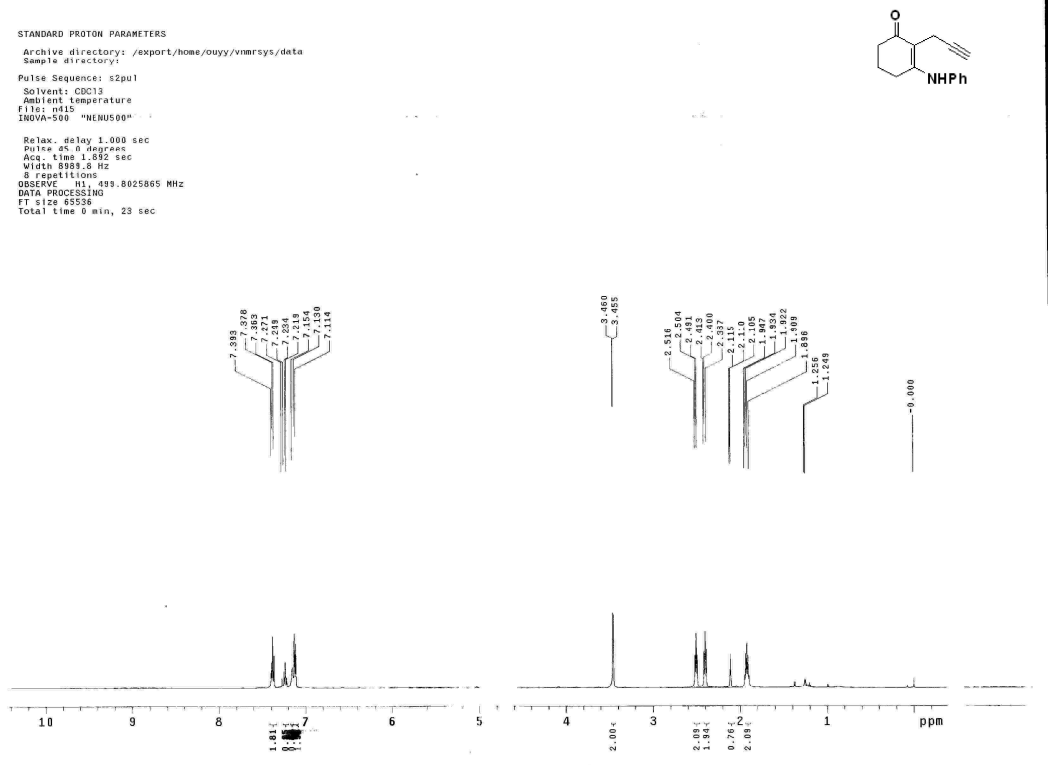

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: n136
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
256 repetitions
OBSERVE C13, 125.6754650 MHz
DECOUPLE H1, 499.8059805 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
194.218
144.473
138.594
134.466
131.418
130.022
127.221
120.053
103.324
77.256
77.000
76.748
37.922
23.866
22.716
21.109
12.514
ppm

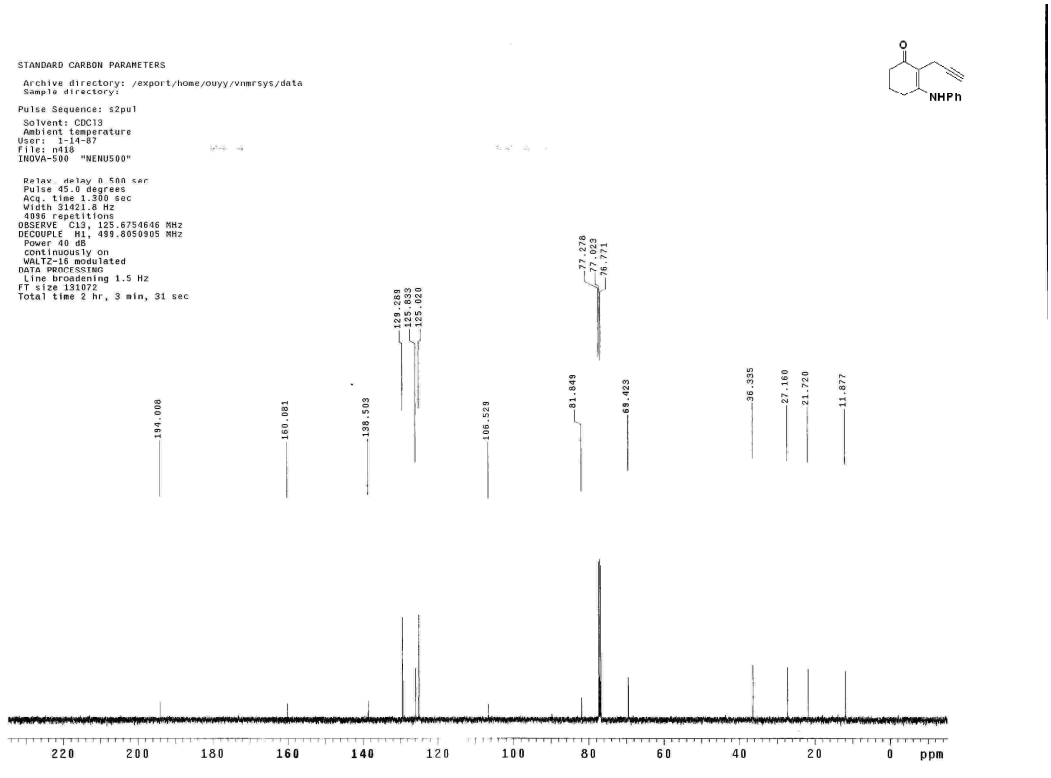
4d


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: n229
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.882 sec
Width 9313.2 Hz
8 repetitions
OBSERVE H1, 499.8025893 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
7.338
7.336
7.324
7.309
7.284
7.270
7.265
6.931
6.317
6.343
6.341
5.031
2.647
2.635
2.622
2.465
2.451
2.437
2.138
2.129
2.115
2.103
2.080
2.078
1.948
0.000
ppm

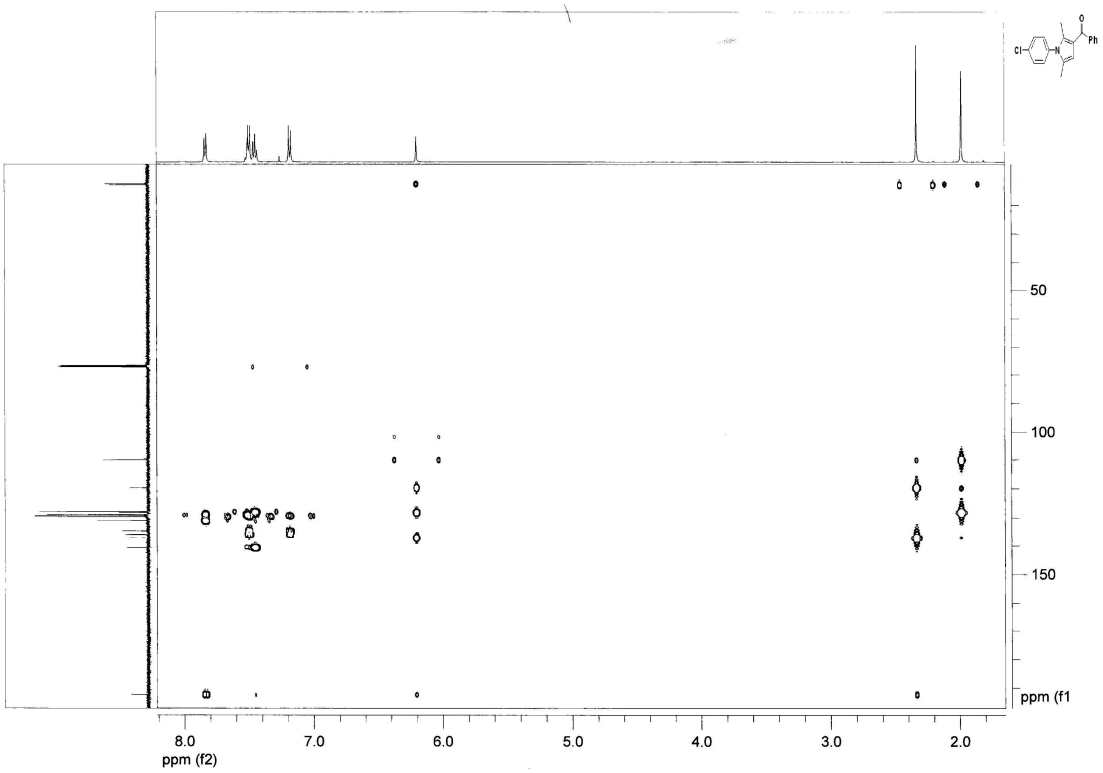

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: n230
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754933 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
193.745
143.588
136.461
130.464
128.732
127.363
125.394
119.782
103.457
77.057
76.805
76.550
46.914
37.526
23.517
21.815
11.793
ppm

4a-i





NOE Spectrum of 2a4


Cl
N
O
Ph
50
100
150
ppm (f1
8.0
7.0
6.0
5.0
4.0
3.0
2.0
ppm (f2)