Supporting Information

Tandem Michael addition/isocyanide insertion into C–C bond: A novel access to 2-acylpyrroles and medium-ring fused pyrroles

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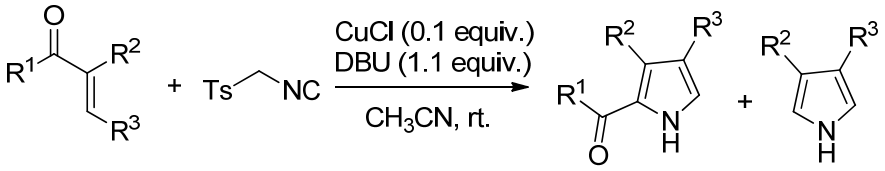
I. General information

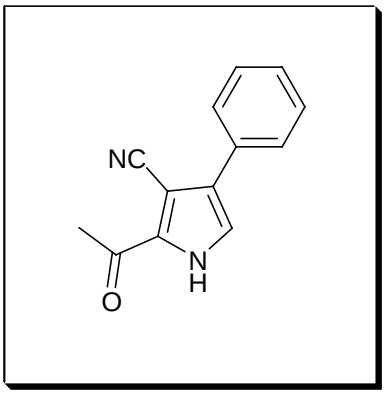
All reagents were commercial and used without further purification, unless otherwise indicated. Chromatography was carried on flash silica gel (300−400 mesh). All reactions were monitored by TLC, which was performed on precoated aluminum sheets of silica gel 60 (F254). Melting points were uncorrected. The ^{1}H NMR and ^{13}C NMR spectra were determined at 25ºC on a 500 MHz and 125 MHz, respectively, and TMS as internal standard. All shifts are given in ppm. High-resolution mass spectra (HRMS) were obtained using a Bruker microTOF II focus spectrometer (ESI). The substrates **1** and **5** were prepared by the similar method as reported references.[1,2]

II. Synthetic procedures/analytical data of compounds 3 and 6.



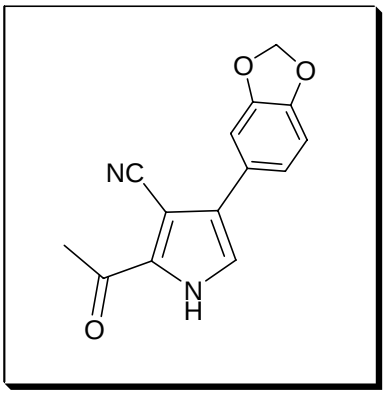
1 **2a** **3** **4**

General procedure for the synthesis of 3a-u (taking 3a as an example): To the mixture of **1a** (171.0 mg, 1.0 mmol), tosylmethyl isocyanide (215 mg, 1.1 mmol) and CuCl (10 mg, 0.1 mmol) in CH_3CN (5 mL) in ice-bath, a solution of DBU (0.165 mL, 1.1 mmol) in CH_3CN (2 mL) was added dropwise in 5 min under stirring. Then the temperature was allowed to rise to room temperature and the reaction was monitored by TLC. After the substrate **1a** was consumed, the resulting mixture was poured into brine (20 mL), and extracted with EtOAc (3×10 mL). The combined organic layers were dried over anhydrous $MgSO_4$, and concentrated under reduced pressure. Purification by flash column chromatography on silica gel (eluent: petroleum ether/EtOAc = 6:1) to give **3a** (153 mg, 73%) and **4a** (40 mg, 24%), respectively.

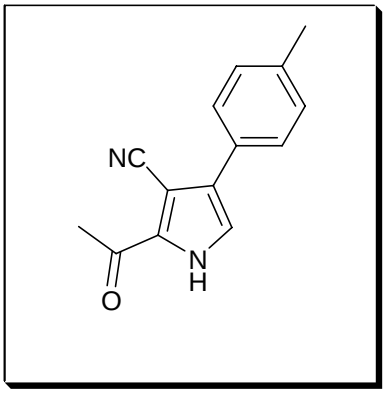


3a, 2-Acetyl-4-phenyl-1*H*-pyrrole-3-carbonitrile. White solid. m.p. 112–114 °C. ^{1}H NMR ($CDCl_3$, 500 Hz) δ 2.61 (s, 3H), 7.36 (t, *J* = 8.0 Hz, 1H), 7.43 (t, *J* = 8.0 Hz, 2H), 7.49 (d, *J* = 2.0 Hz, 1H), 7.64 (d, *J* = 7.5 Hz 2H), 7.84 (d, *J* = 2.0 Hz, 1H). ^{13}C NMR ($CDCl_3$, 125 Hz), δ 22.0, 97.6, 114.8, 116.5, 126.7, 127.7, 128.3, 129.0, 129.3, 131.0, 166.5. HRMS (ESI-TOF) Calcd for $C_{13}H_{11}N_2O^+$ ($[M+H]^+$) 211.0866. Found 211.0861.

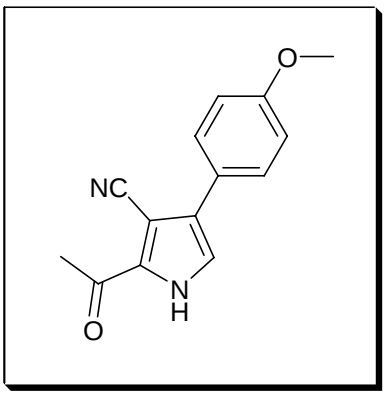
(1) Goswami, P.; Das, B. *Tetrahedron Lett.* **2009**, *50*, 897. (2) Pan, L.; Liu, Q. *Synlett* **2011**, 1073.



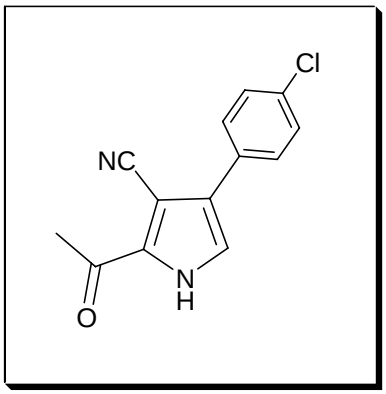
3b, 2-Acetyl-4-(benzo[*d*][1,3]dioxol-5-yl)-1*H*-pyrrole-3-carbonitrile. Light yellow solid. m.p. 165–167 °C. ^{1}H NMR ($CDCl_3$, 500 Hz) δ 2.62 (s, 3H), 6.01 (s, 2H), 6.88 (d, *J* = 8.0 Hz, 1H), 7.07 (d, *J* = 2.0 Hz, 1H), 7.07–7.14 (dd, *J* = 8.0 Hz, *J* = 2.0 Hz, 1H), 7.04 (s, 1H), 7.83 (s, 1H). ^{13}C NMR ($CDCl_3$, 125 Hz), δ22.0, 97.6, 101.3, 107.3, 108.8, 114.8, 116.0, 120.6, 124.9, 127.5, 129.1, 147.8, 148.2, 166.4. HRMS (ESI-TOF) Calcd for $C_{14}H_{11}N_2O_3^+$ ($[M+H]^+$) 255.0764 Found 255.0768.



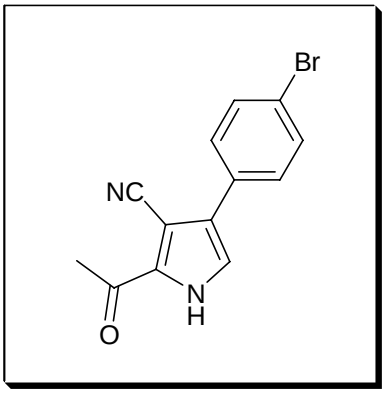
3c, 2-Acetyl-4-*p*-tolyl-1*H*-pyrrole-3-carbonitrile. White solid. m.p. 116–118 °C. ^{1}H NMR ($CDCl_3$, 500 Hz) δ 2.39 (s, 3H), 2.62 (s, 3H), 7.25 (d, *J* = 8.0 Hz, 2H), 7.46 (s, 1H), 7.53 (d, *J* = 8.0 Hz, 2H), 7.84 (s, 1H). ^{13}C NMR ($CDCl_3$, 125 Hz), δ 21.2, 22.0, 97.5, 114.9, 116.0, 126.6, 127.6, 128.0, 129.3, 129.6, 138.2, 166.4. HRMS (ESI-TOF) Calcd for $C_{14}H_{13}N_2O^+$ ($[M+H]^+$) 225.1022, Found 225.1019.



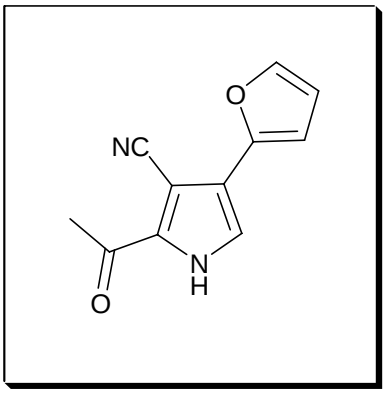
3d, 2-Acetyl-4-(4-methoxyphenyl)-1*H*-pyrrole-3-carbonitrile. White solid. m.p. 145–147 °C. ^{1}H NMR ($CDCl_3$, 500 Hz) δ 2.61 (s, 3H), 3.84 (s, 3H), 6.96 (d, *J* = 8.5 Hz, 2H), 7.42 (s, 1H), 7.57 (d, *J* = 8.5 Hz, 2H), 7.82 (s, 1H). ^{13}C NMR ($CDCl_3$, 125 Hz), δ 22.0 55.3, 87.6, 114.4, 115.0, 115.7, 123.5, 127.5, 128.0, 129.1, 159.7, 166.5. HRMS (ESI-TOF) Calcd for $C_{14}H_{13}N_2O_2^+$ ($[M+H]^+$) 241.0972, Found 241.0970.



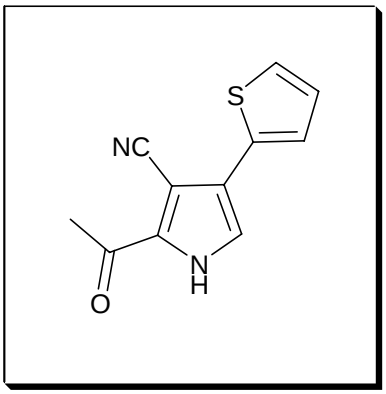
3e, 2-Acetyl-4-(4-chlorophenyl)-1*H*-pyrrole-3-carbonitrile. White solid. m.p. 182–184 °C. ^{1}H NMR ($CDCl_3$, 500 Hz) δ 2.63 (s, 3H), 7.41 (d, J = 8.5 Hz, 2H), 7.50 (s, 1H), 7.57 (d, J = 8.5 Hz, 2H), 7.85 (d, J = 2.0 Hz, 1H). ^{13}C NMR ($CDCl_3$, 125 Hz), δ 22.1, 94.5, 114.7, 116.6, 127.8, 128.0, 128.2, 129.3 129.5, 134.3, 166.4. HRMS (ESI-TOF) Calcd for $C_{13}H_9ClN_2NaO^+$ ($[M+Na]^+$) 267.0296, Found 267.0285.



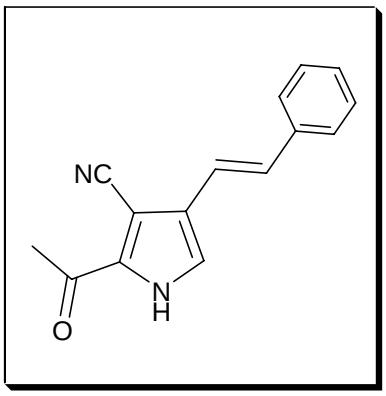
3f, 2-Acetyl-4-(4-bromophenyl)-1*H*-pyrrole-3-carbonitrile. White solid. m.p. 165–167°C. ^{1}H NMR ($CDCl_3$, 500 Hz) δ 2.64 (s, 3H), 7.51(s, 1H), 7.52 (d, J = 8.5 Hz, 2H), 7.57 (d, J = 8.5 Hz, 2H), 7.86 (s, 1H). ^{13}C NMR ($CDCl_3$, 125 Hz), δ 22.0, 97.4, 114.6, 116.6, 122.4, 127.8, 128.2, 128.3, 129.9, 132.2, 166.3. HRMS (ESI-TOF) Calcd for $C_{13}H_{10}BrN_2O^+$ ($[M+H]^+$) 288.9971, Found 288.9980.



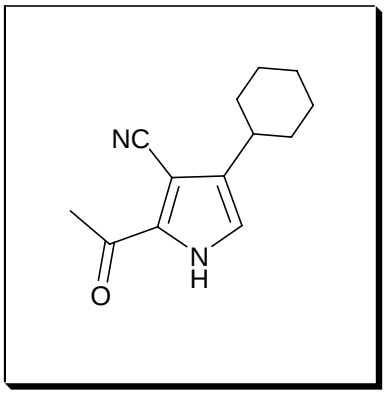
3g, 2-Acetyl-4-(furan-2-yl) -1*H*-pyrrole-3-carbonitrile. White solid. m.p. 154–156 °C. ^{1}H NMR ($CDCl_3$, 500 Hz) δ 2.62 (s, 3H), 6.49 (s, 1H), 6.88 (s, 1H), 7.44 (s, 1H), 7.54 (s, 1H), 7.82 (s, 1H). ^{13}C NMR ($CDCl_3$, 125 Hz), δ 22.0, 95.6, 107.3, 111.6, 114.3, 114.9, 120.1, 127.2, 142.0, 145.7, 166.3. HRMS (ESI-TOF) Calcd for $C_{11}H_8N_2NaO_2^+$ ($[M+Na]^+$) 223.0478, Found 223.0467.



3h, 2-Acetyl-4-(thiophen-2-yl)-1*H*-pyrrole-3-carbonitrile. Light pink solid. m.p. 162–164 °C. ^{1}H NMR ($CDCl_3$, 500 Hz) δ 2.62 (s, 3H), 7.10 (dd, J = 5.0 Hz, J = 3.5 Hz, 1H), 7.30 (dd, J = 5.0 Hz, J = 1.0 Hz, 1H), 7.47 (s, 1H), 7.52 (dd, J = 4.0 Hz, J = 1.0 Hz, 1H), 7.83 (s, 1H). ^{13}C NMR ($CDCl_3$, 125 Hz), δ 22.0, 97.2, 114.5, 115.9, 123.1, 125.1, 125.3, 127.5, 128.1, 132.7, 166.3. HRMS (ESI-TOF) Calcd for $C_{11}H_9N_2OS^+$ ($[M+H]^+$) 217.0430, Found 217.0424.

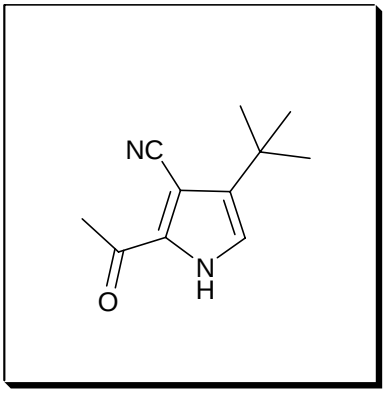


3i, 2-Acetyl-4-styryl-1*H*-pyrrole-3-carbonitrile. Light yellow solid. m.p. 167–169 °C. ^{1}H NMR ($CDCl_3$, 500 Hz) δ 2.60 (s, 3H), 6.93 (d, J = 16.5 Hz, 1H), 7.29 (t, J = 8.0 Hz, 1H), 7.30 (d, J = 16.5 Hz, 1H), 7.36 (t, J = 8.0 Hz, 2H), 7.41 (s, 1H), 7.49 (d, J = 8.0 Hz, 2H), 7.78 (s, 1H). ^{13}C NMR ($CDCl_3$, 125 Hz), δ 22.0, 97.1, 114.8, 117.2, 117.3, 126.5, 127.5, 128.2, 128.7, 131.0, 136.5, 166.3. HRMS (ESI-TOF) Calcd for $C_{15}H_{13}N_2O^+$ ($[M+H]^+$) 237.1022, Found 237.1022.

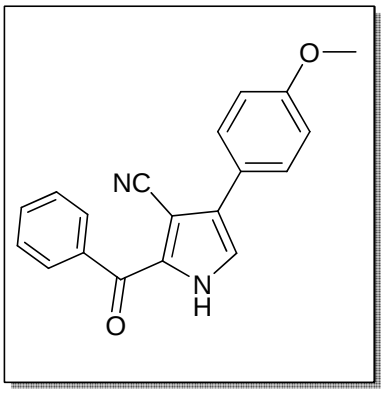


3j, 2-Acetyl-4-cyclohexyl-1H-pyrrole-3-carbonitrile. White solid. Light yellow oil. ^{1}H NMR ($CDCl_3$, 500 Hz) δ 1.21–1.27 (m, 1H), 1.30–1.44 (m, 4H), 1.74 (d, J = 14.0 Hz, 1H), 1.82 (d, J = 14.0 Hz, 2H), 1.99 (d, J = 14.0 Hz, 2H), 2.53 (s, 3H), 2.53–2.58 (m, 1H), 7.05 (s, 1H), 7.70 (s, 1H). ^{13}C NMR ($CDCl_3$, 125 Hz), δ 22.0, 25.8, 26.2, 33.1, 35.5, 98.5, 114.7, 115.3, 126.4, 135.8, 166.6. HRMS (ESI-TOF) Calcd for

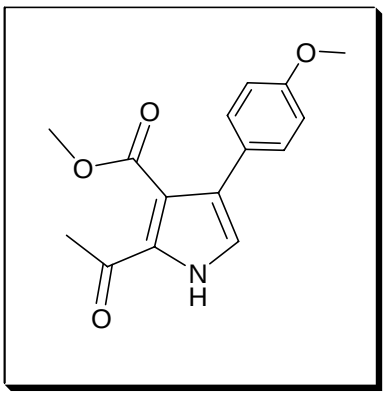
$C_{13}H_{17}N_2O^+$ ([M+H]$^+$) 217.1355, Found 217.1340.



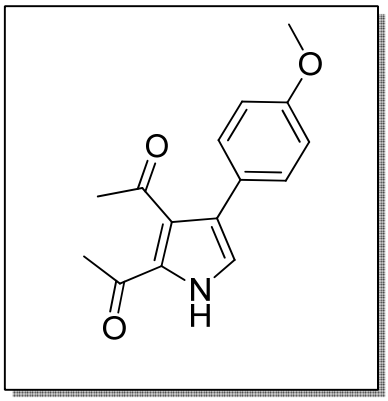
3k, 4-*tert*-Butyl-2-acetyl-1*H*-pyrrole-3-carbonitrile. Colorless oil. ^{1}H NMR ($CDCl_3$, 500 Hz) δ 1.36 (s, 9H), 2.55 (s, 3H), 7.06 (s, 1H), 7.74 (s, 1H). ^{13}C NMR ($CDCl_3$, 125 Hz), δ 22.0, 28.6, 31.3, 97.4, 114.9, 115.6, 128.0, 139.4, 166.6. HRMS (ESI-TOF) Calcd for $C_{11}H_{15}N_2O^+$ ([M+H]$^+$) 191.1179, Found 191.1169.



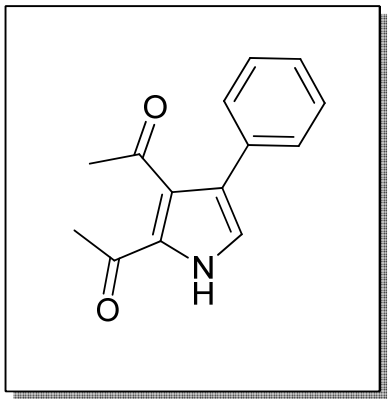
3l, 2-Benzoyl-4-(4-methoxyphenyl)-1*H*-pyrrole-3-carbonitrile. White solid. m.p. 126–128 °C. ^{1}H NMR ($CDCl_3$, 500 Hz) δ 3.84 (s, 3H), 6.96 (d, *J* = 9.0 Hz, 2H), 7.47 (d, *J* = 2.5 Hz, 1H), 7.56–7.60 (m, 4H), 7.70 (t, *J* = 7.5 Hz, 1H), 7.77 (s, 1H), 7.78 (d, *J* = 7.5 Hz, 2H). ^{13}C NMR ($CDCl_3$, 125 Hz), δ 55.3, 97.3, 114.4, 115.1, 117.5, 123.5, 128.0, 128.8, 128.9, 129.7, 129.8, 131.1, 133.5, 159.6, 166.4. HRMS (ESI-TOF) Calcd for $C_{19}H_{14}N_2NaO_2^+$ ([M+Na]$^+$) 325.0947, Found 325.0941.



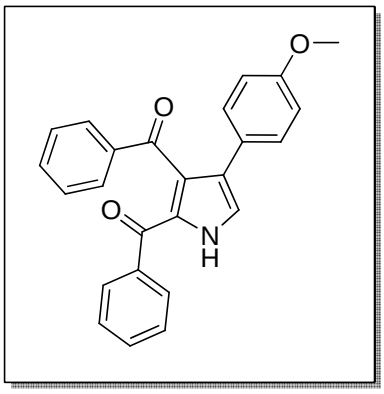
3m, Methyl 2-acetyl 4-(4-methoxyphenyl)-1H-pyrrole-3-carboxylate. White solid. m.p. 134–136 °C. ^{1}H NMR ($CDCl_3$, 500 Hz) δ 2.59 (s, 3H), 3.77 (s, 3H), 3.83 (s, 3H), 6.91 (d, *J* = 8.5 Hz, *2*H), 7.27 (s, 1H), 7.40 (d, *J* = 8.5 Hz, *2*H), 7.93 (s, 1H). ^{13}C NMR ($CDCl_3$, 125 Hz), δ 22.0, 51.3, 55.3, 113.4, 118.0, 125.4, 125.5, 129.2, 130.2, 159.1, 164.2, 167.3. HRMS (ESI-TOF) Calcd for $C_{15}H_{15}NNaO_4^+$ ([M+Na]$^+$) 296.0893, Found 296.0897.



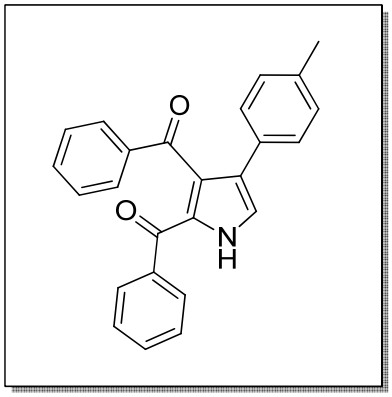
3n, 2,3-Diacetyl-4-(4-methoxyphenyl)pyrrole. Yellowish solid, m.p. 138–140 °C. ^{1}H NMR ($CDCl_3$ 500 Hz) δ 2.36 (s, 3H), 2.59 (s, 3H), 3.83 (s, 3H), 6.91 (d, J = 7.5 Hz, 2H), 7.21 (s, 1H), 7.33 (d, J = 7.5 Hz, 2H), 7.91 (s, 1H). ^{13}C NMR ($CDCl_3$, 125 Hz), δ 22.1, 28.7, 55.2, 113.5, 118.6, 124.8, 125.6, 127.0, 128.7, 130.2, 159.1, 167.4, 194.1. HRMS (ESI-TOF) Calcd for $C_{15}H_{15}NNaO_3^+$ ($[M+Na]^+$) 280.0944, Found 280.0951.



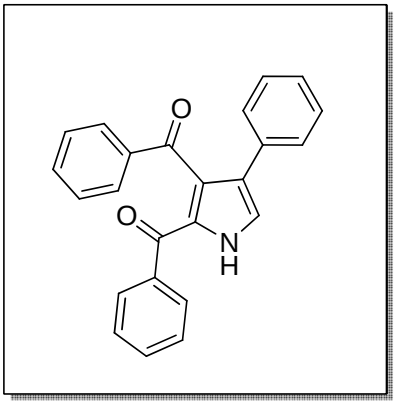
3o, 2,3-Diacetyl-4-phenylpyrrole. White solid, m.p. 93–95 °C. ^{1}H NMR ($CDCl_3$, 500 Hz) δ 2.36 (s, 3H), 2.61 (s, 3H), 7.26 (s, 1H), 7.33–7.41 (m, 5H), 7.93 (s, 1H). ^{13}C NMR ($CDCl_3$, 125 Hz), δ 22.1, 28.7, 119.1, 124.9, 127.5, 128.0, 129.0, 129.1, 133.3, 167.5, 194.1. HRMS (ESI-TOF) Calcd for $C_{14}H_{13}NNaO_2^+$ ($[M+Na]^+$) 250.0838, Found 250.0841.



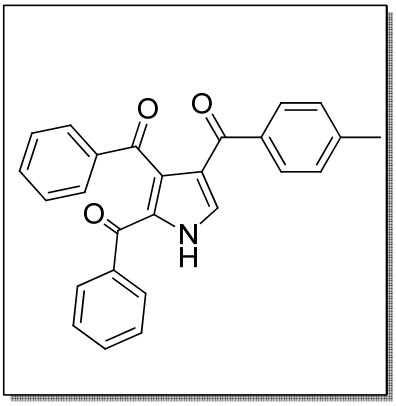
3p, (4-(Methoxyphenyl)-1*H*-pyrrole-2,3-diyl)bis(phenylmethanone). White solid. m.p. 186–188 °C. ^{1}H NMR ($CDCl_3$, 500 Hz) δ 3.76 (s, 3H), 6.80 (d, J = 8.5 Hz, 2H), 7.14 (d, J = 8.5 Hz, 2H), 7.15 (d, J = 8.5 Hz, 2H), 7.23 (d, J = 2.5 Hz, 1H), 7.27–7.29 (m, 4H), 7.32–7.35 (m, 1H), 7.37–7.41 (m, 3H), 9.59 (s, 1H). ^{13}C NMR ($CDCl_3$, 125 Hz), δ 55.2, 113.9, 121.9, 125.7, 127.6, 127.9, 128.0, 128.5, 129.0, 129.5, 131.0, 131.8, 132.6, 138.7, 139.2, 158.7, 186.7, 194.2. HRMS (ESI-TOF) Calcd for $C_{25}H_{19}NNaO_3^+$ ($[M+Na]^+$) 404.1257, Found 404.1257.



3q, (4-(*p*-Tolyl)-1*H*-pyrrole-2,3-diyl)bis(phenylmethanone). White solid. m.p. 179–181 °C. ^{1}H NMR ($CDCl_3$, 500 Hz) δ 2.28 (s, 3H), 7.06 (d, *J* = 8.0 Hz, 2H), 7.14–7.16 (m, 4H), 7.22–7.25 (m, 3H), 7.33 (t, *J* = 8.0 Hz, 2H), 7.38–7.42 (m, 4H), 9.89 (s, 1H). ^{13}C NMR ($CDCl_3$, 125 Hz), δ 21.0, 122.1, 127.7, 127.9, 128.0, 128.1, 128.3, 128.5, 129.0, 129.1, 130.2, 130.8, 131.8, 132.5, 136.6, 138.6, 139.1, 186.7, 194.1. HRMS (ESI-TOF) Calcd for $C_{25}H_{19}NNaO_2^+$ ($[M+Na]^+$) 388.1308, Found 388.1310.

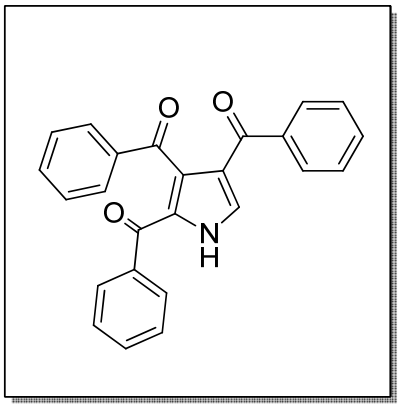


3r, (4-Phenyl-1*H*-pyrrole-2,3-diyl)bis(phenylmethanone). White solid. m.p. 150–152 °C. ^{1}H NMR ($CDCl_3$, 500 Hz) δ 7.15 (t, *J* = 7.5 Hz, 4H), 7.21 (d, *J* = 7.5 Hz, 1H), 7.24–7.29 (m, 3H), 7.33 (dd, *J* = 7.0, 3.5 Hz, 4H), 7.39–7.43 (m, 4H), 9.70 (s, 1H). ^{13}C NMR ($CDCl_3$, 125 Hz), δ 122.1, 126.9, 127.9, 128.1, 128.2, 128.3, 128.4, 128.5, 129.7, 129.1, 131.0, 131.9, 132.6, 133.2, 138.6, 139.1, 186.7, 194.0. HRMS (ESI-TOF) Calcd for $C_{24}H_{17}NNaO_2^+$ ($[M+Na]^+$)374.1151, Found 374.1157.

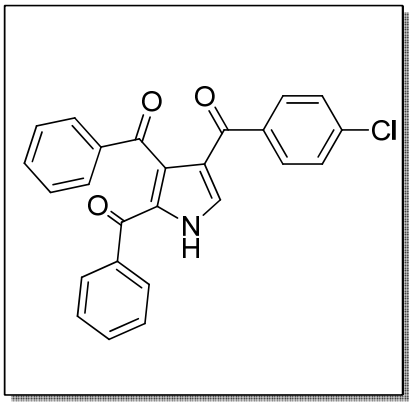


3s, (4-(Methlbenzoyl)-1*H*-pyrrole-2,3-diyl)bis(phenylmethanone). White solid. m.p. 162–164 °C. ^{1}H NMR ($CDCl_3$, 500 Hz) δ 2.37 (s, 3H), 7.10 (t, *J* = 7.5 Hz, 2H), 7.17 (t, *J* = 7.5 Hz, 4H), 7.29–7.35 (m, 2H), 7.39 (d, *J* = 3.0 Hz, 1H), 7.42 (d, *J* = 8.0 Hz, 2H), 7.51 (d, *J* = 7.5 Hz, 2H), 7.65 (d, *J* = 8.0 Hz, 2H), 11.03 (s,

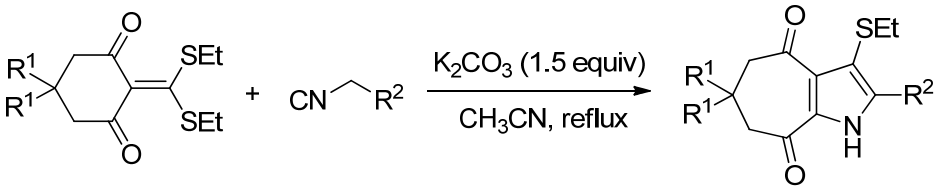
1H). ^{13}C NMR ($CDCl_3$, 125 Hz), δ 21.5, 126.2, 128.0, 128.0, 128.5, 128.7, 128.9, 129.0, 129.3, 130.7, 131.2, 132.2, 132.6, 135.4, 137.5, 138.1, 143.1, 186.8, 189.0, 193.1. HRMS (ESI-TOF) Calcd for $C_{26}H_{20}NO_3^+$ ($[M+H]^+$) 394.1438, Found 394.1430.



3t, (1*H*-pyrrole-2,3,4-triyl)tris(phenylmethanone). White solid. m.p. 126–128 °C. ^{1}H NMR ($CDCl_3$, 500 Hz) δ 7.11 (t, *J* = 8.0 Hz, 2H), 7.19 (t, *J* = 8.0 Hz, 2H), 7.31–7.36 (m, 4H), 7.38–7.45 (m, 3H), 7.51 (d, *J* = 7.0 Hz, 3H), 7.74 (d, *J* = 7.0 Hz, 2H), 10.86 (s, 1H). ^{13}C NMR (DMSO-d_6, 125 Hz), δ 126.2, 128.1, 128.4, 128.5, 128.8, 128.9, 129.1, 130.7, 131.2, 132.3, 132.4, 132.8 137.5, 138.0, 138.1, 187.0, 189.3, 192.9. HRMS (ESI-TOF) Calcd for $C_{25}H_{18}NO_3^+$ ($[M+H]^+$) 380.1281, Found 380.1298.



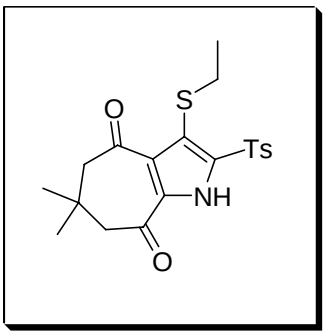
3u, (4-(Chlorobenzoyl)-1*H*-pyrrole-2,3-diyl)bis(phenylmethanone). White solid. m.p. 161–163 °C. ^{1}H NMR ($CDCl_3$, 500 Hz) δ 7.12 (t, *J* = 8.0 Hz, 2H), 7.19 (t, *J* = 8.0 Hz, 2H), 7.33–7.38 (m, 4H), 7.41–7.47 (m, 3H), 7.48 (d, *J* = 7.0 Hz, 2H), 7.69 (d, *J* = 8.5 Hz, 2H), 10.9, (s, 1H). ^{13}C NMR (DMSO-d_6, 125 Hz), δ 125.9, 128.1, 128.5, 128.7, 128.8, 130.5, 130.9, 131.0, 132.5, 132.9, 136.4, 137.5, 138.0, 138.8, 187.0, 188.1, 192.8. HRMS (ESI-TOF) Calcd for $C_{25}H_{17}ClNO_3^+$ ($[M+H]^+$) 414.0891, Found 414.0888.



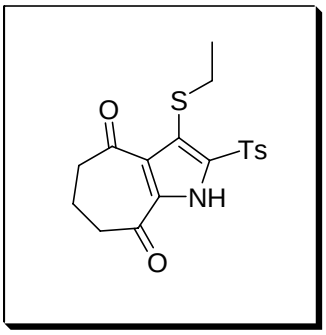
11 **2** **12**

General procedure for the synthesis of 6 (taking 6a as an example): To the mixture of **5a** (272.0 mg, 1.0 mmol) and tosylmethyl isocyanide **2a** (215 mg, 1.1 mmol) in CH_3CN (10 mL) was added K_2CO_3 (207 mg, 1.5 mmol) in one portion at room temperature. The reaction mixture was then heated to reflux under stirring, and the reaction mixture was monitored by TLC. After the substrate **5a** was consumed, the

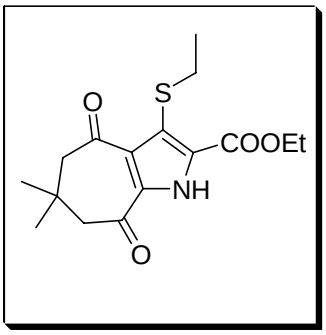
resulting mixture was poured into brine ice (30 mL) under stirring and neutralized with diluted hydrochloric acid, extracted with EtOAc (3 x 10 mL). The combined organic layers were dried over anhydrous $MgSO_4$, and concentrated under reduced pressure. Purification by flash column chromatography on silica gel (petroleum ether: EtOAc = 6:1) gave **6a** (332 mg, 82%).



6a, 3-(Ethylthio)-6,7-dihydro-6,6-dimethyl-2-tosylcyclohepta[*b*]pyrrole-4,8(1*H*,5*H*)-dione. yellowish solid, m.p. 139–141 °C. ^{1}H NMR ($CDCl_3$, 500 Hz) δ 1.01 (t, *J* = 7.5 Hz, 3H), 1.13 (s, 6H), 2.43 (s, 3H), 2.77 (q, *J* = 7.5 Hz, 2H), 2.86 (s, 2H), 2.88 (s, 2H), 7.33 (d, *J* = 8.0 Hz, 2H), 7.98 (d, *J* = 8.5 Hz, 2H), 10.2 (s, 1H). ^{13}C NMR ($CDCl_3$, 125 Hz), δ 13.8, 21.6, 29.0, 30.4, 31.5, 56.3, 58.4, 120.3, 128.4, 128.6, 129.6, 132.2, 136.6, 137.0, 145.3, 189.9, 193.6. HRMS (ESI-TOF) Calcd for $C_{20}H_{22}NO_4S_2^-$ ([M-H]$^-$) 404.0996. Found 404.0994.

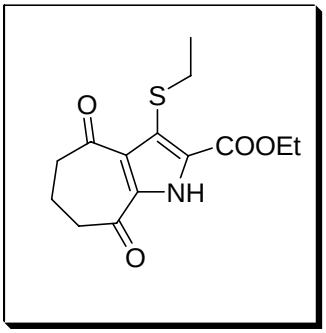


6b, 3-(Ethylthio)-6,7-dihydro-2-tosylcyclohepta[*b*]pyrrole-4,8(1*H*,5*H*)-dione. Yellowish solid, m.p. 140–142 °C. ^{1}H NMR ($CDCl_3$, 500 Hz) δ 1.02 (t, *J* = 7.5 Hz, 3H), 2.10 (m, 2H), 2.43 (s, 3H), 2.77 (q, *J* = 7.5 Hz, 2H), 2.88 (m, 4H), 7.33 (d, *J* = 8.0 Hz, 2H), 7.98 (d, *J* = 8.0 Hz, 2H), 10.3 (s, 1H). ^{13}C NMR ($CDCl_3$, 125 Hz), δ 13.8, 18.4, 21.7, 30.5, 42.0, 44.5, 120.8, 127.8, 128.8, 129.7, 131.6, 136.6, 137.6, 145.5, 191.1, 194.9. HRMS (ESI-TOF) Calcd for $C_{18}H_{18}NO_4S_2^-$ ([M-H]$^-$) 376.0683. Found 376.0677.

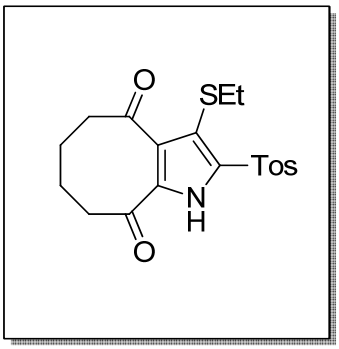


6c, Ethyl 3-(ethylthio)-1,4,5,6,7,8-hexahydri-6,6-dimethyl-4,8-dioxocycloheptal[*b*]pyrrole-2-carb -oxyate. Yellowish solid, m.p. 98–100 °C. ^{1}H NMR ($CDCl_3$, 500 Hz) δ 1.15 (s, 6H), 1.20 (t, *J* = 7.5 Hz, 3H), 1.41 (t, *J* = 7.5 Hz, 3H), 2.88 (s, 2H), 2.92 (s, 2H), 2.97 (q, *J* = 7.0Hz, 2H), 4.42 (q, *J* = 7.0 Hz, 2H), 10.0 (s,

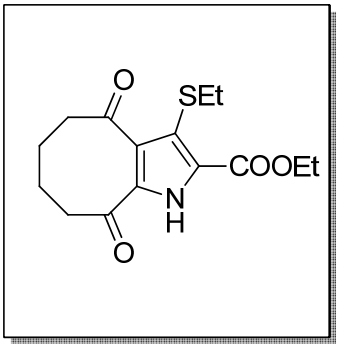
1H). ^{13}C NMR ($CDCl_3$, 125 Hz), δ 14.3, 14.4, 29.1, 30.1, 31.7, 56.5, 58.5, 61.7, 124.6, 127.4, 128.5, 128.5, 131.9, 159.1, 190.1, 193.9. HRMS (ESI-TOF) Calcd for $C_{16}H_{20}NO_4S^-$ ([M-H]$^-$) 322.1119. Found 322.1090.



6d, Ethyl 3-(ethylthio)-1,4,5,6,7,8-hexahydri-4,8-dioxocycloheptal[*b*]pyrrole-2-carboxyate. Yellow oil. ^{1}H NMR ($CDCl_3$, 500 Hz) δ 1.18 (t, J = 7.5 Hz, 3H), 1.42 (t, J = 7.5 Hz, 3H), 2.13 (m, 2H), 2.88 (m, 2H), 2.93 (m, 2H), 2.97 (q, J = 7.5 Hz, 2H), 4.43 (q ,J = 7.5 Hz, 2H), 10.2 (s, 1H). ^{13}C NMR ($CDCl_3$, 125 Hz), δ 14.2, 14.2, 18.6, 30.1, 41.8, 44.3, 61.6, 124.7, 127.8, 127.8, 131.4, 159.0, 191.4, 195.3. HRMS (ESI-TOF) Calcd for $C_{14}H_{16}NO_4S^-$ ([M-H]$^-$) 294.0806. Found 294.0801.



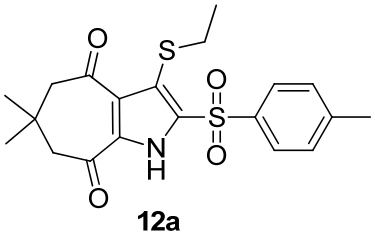
6e, 3-(Ethylthio)-2-tosyl-5,6,7,8-tetrahydro-1*H*-cycloocta[*b*]pyrrole-4,9-dione. White solid. m.p. 181–183 ^{o}C. ^{1}H NMR ($CDCl_3$, 500 Hz) δ 1.11 (t, J = 7.5 Hz, 3H), 1.88 (br, 4H), 2.43 (s, 3H), 2.66 (br, 4H), 2.79 (q, J = 7.5 Hz, 2H), 7.33 (d, J = 8.0 Hz, 2H), 7.98 (d, J = 8.0 Hz, 2H), 10.2 (s, 1H). ^{13}C NMR ($CDCl_3$, 125 Hz), δ 14.2, 21.5, 21.7, 25.2, 32.2, 37.7, 43.4, 117.7, 128.5, 129.7, 131.1, 133.2, 136.7, 137.2, 145.4, 190.7, 202.6. HRMS (ESI-TOF) Calcd for $C_{19}H_{22}NO_4S_2^+$ ([M+H]$^+$) 392.0985. Found 392.0993.



6f, Ethyl 3-(ethylthio)-4,9-dioxo-4,5,6,7,8,9-hexahydro-1*H*-cycloocta[*b*]pyrrole-2-carboxylate. Colorless oil. ^{1}H NMR ($CDCl_3$, 500 Hz) δ 1.18 (t, J = 7.5 Hz, 3H), 1.42 (t, J = 7.5 Hz, 3H), 1.89 (br, 4H), 2.68 (br, 4H), 2.88 (q, J = 7.5 Hz, 2H), 4.42 (q, J = 7.5 Hz, 2H), 10.04 (s, 1H). ^{13}C NMR ($CDCl_3$, 125 Hz), δ 14.2, 14.6, 21.6, 25.4, 30.8, 37.6, 43.6, 61.6, 120.2, 128.2, 131.0, 133.1, 159.1, 190.8, 203.3. HRMS (ESI-TOF) Calcd for $C_{15}H_{20}NO_4S^+$ ([M+H]$^+$) 310.1108. Found 310.1099.

III. Crystal data and ORTEP drawing of compound **6a**

6a: $C_{20}H_{23}N_1O_4S_2$, M = 405.51, monoclinic, space group P21/n, a = 15.619(2), b = 5.9270(8), c = 22.055(3) Å, V = 2003.3(5) Å^3, α = 90.0 β = 101.129(2), γ = 90.00, Z = 4, T = 273(2) K, F000 = 856, 9573 reflections collected, 2366 unique, R_1 = 0.0805, wR_2 = 0.1023 ($I > 2\sigma(I)$).



12a

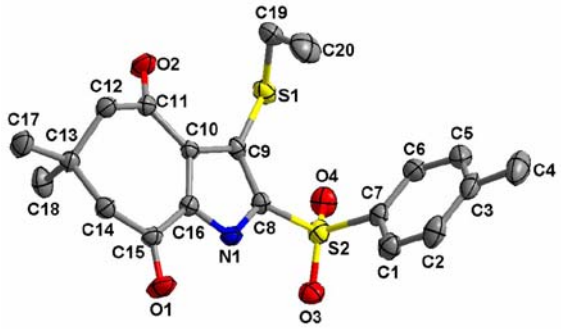
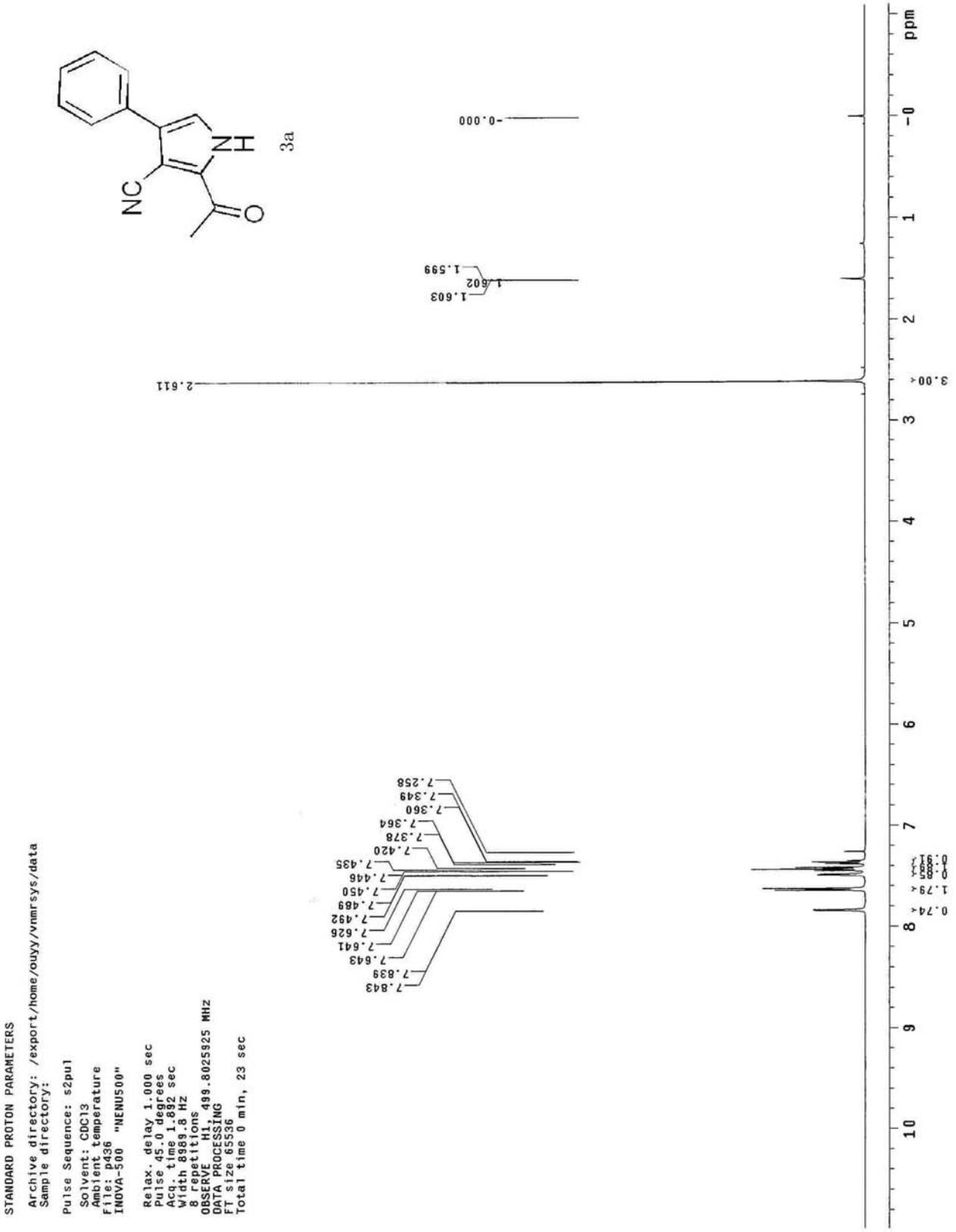
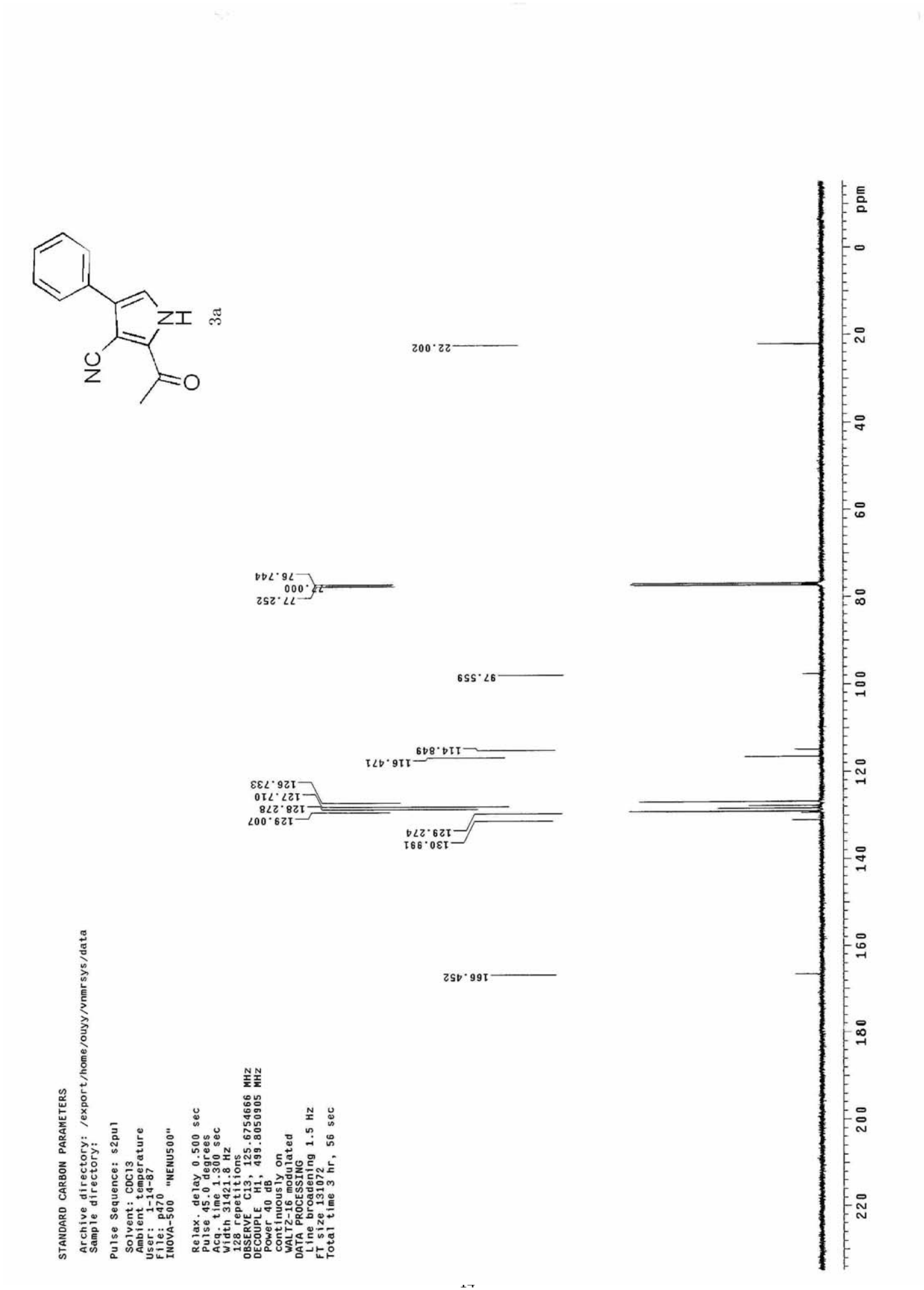
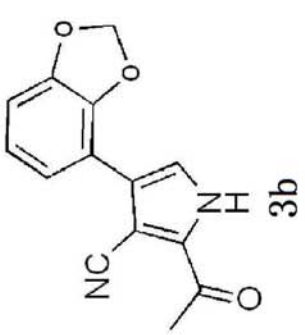


Fig 1. ORTEP diagram of **6a** (30% probability displacement ellipsoids and all hydrogen atoms are omitted).

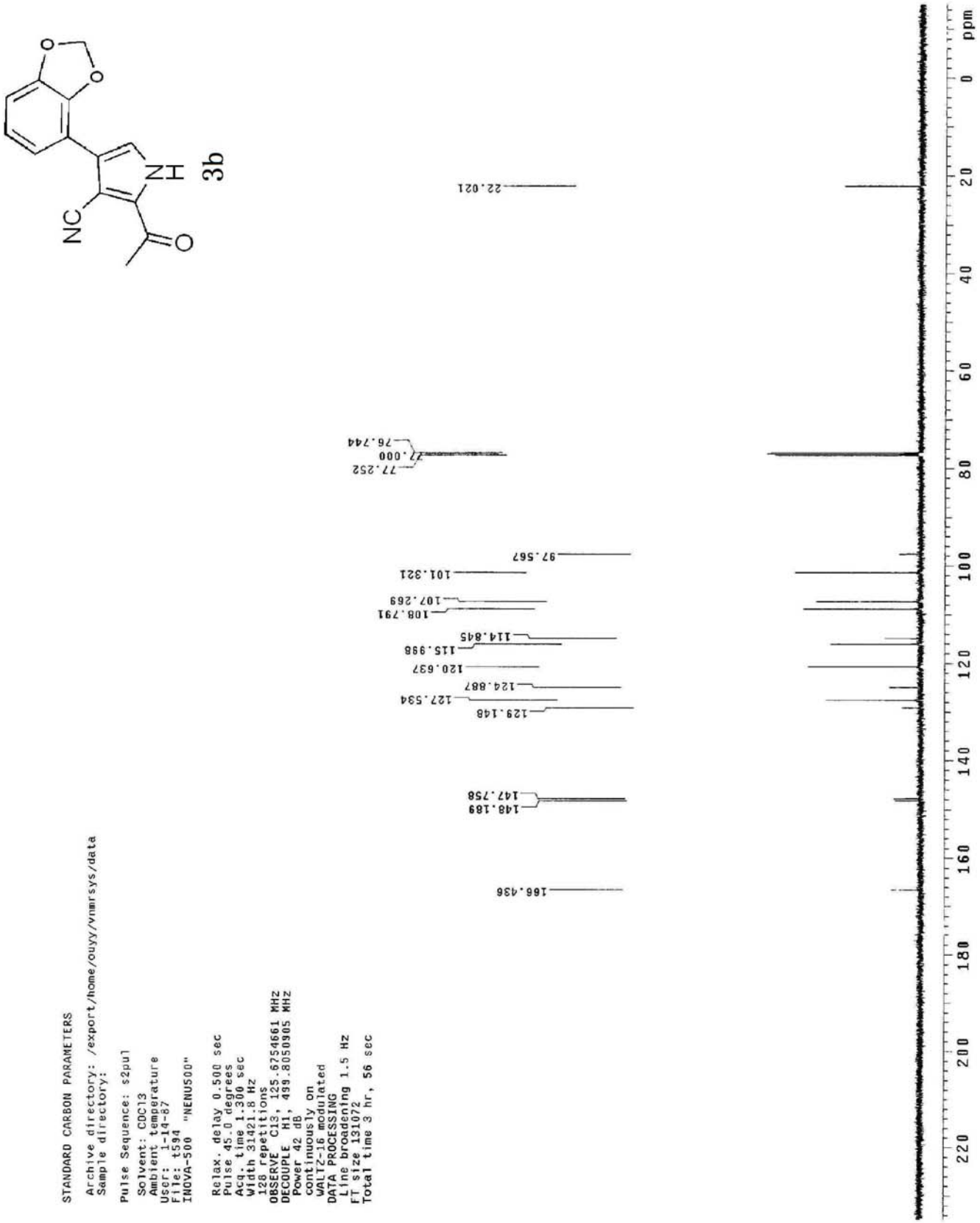
VI. Copies of ^{1}H NMR and ^{13}C NMR spectra of compounds **3** and **6**

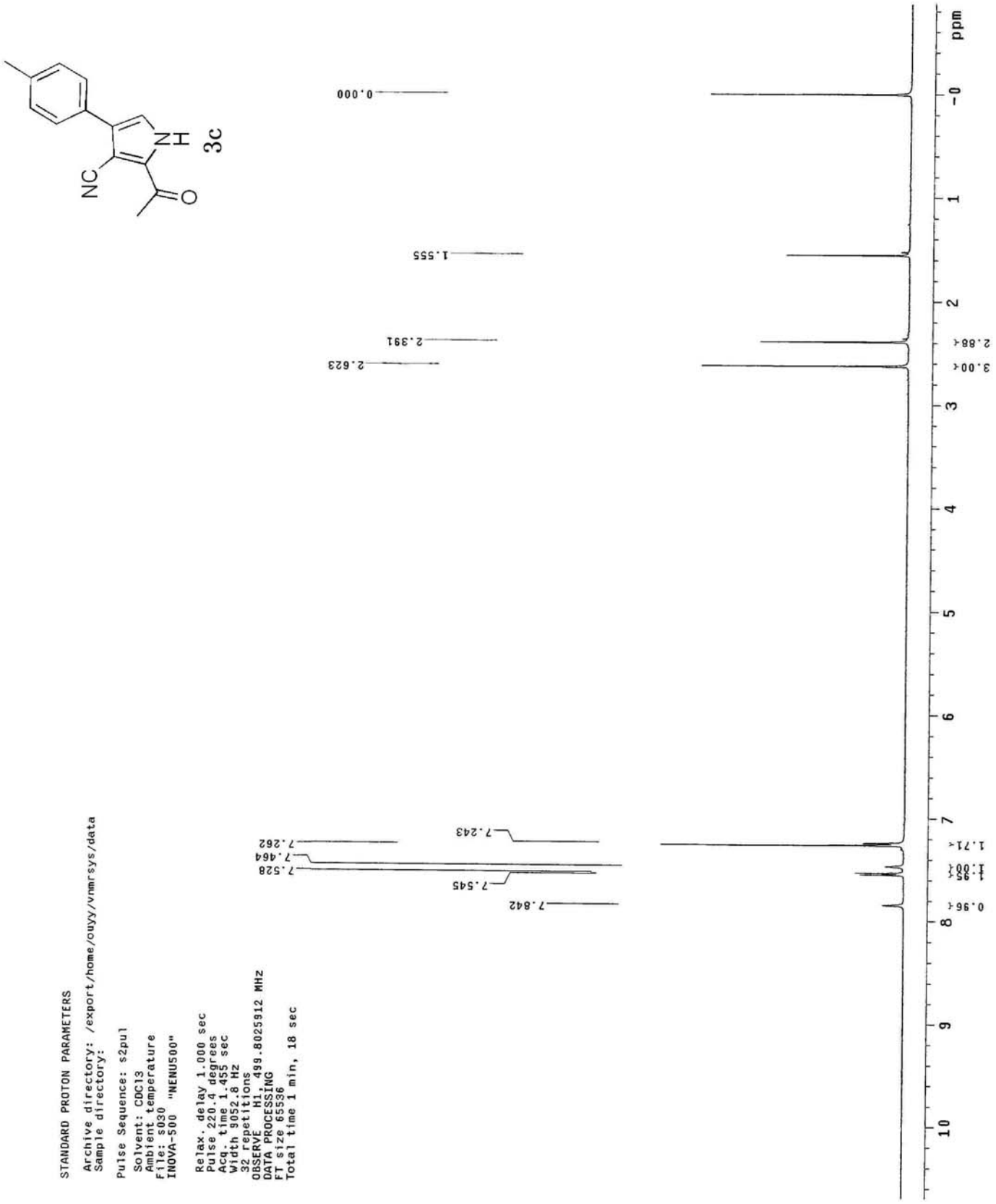


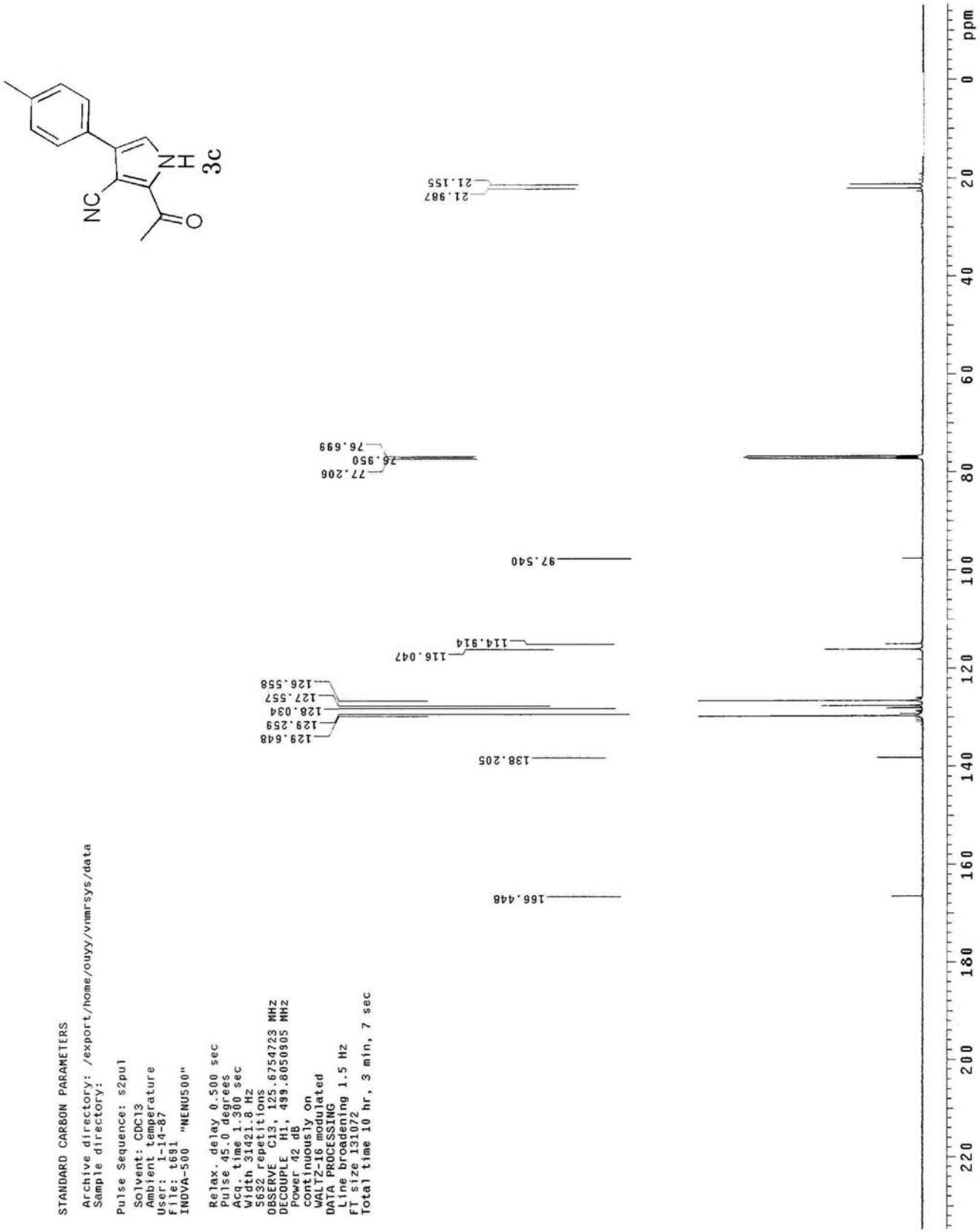


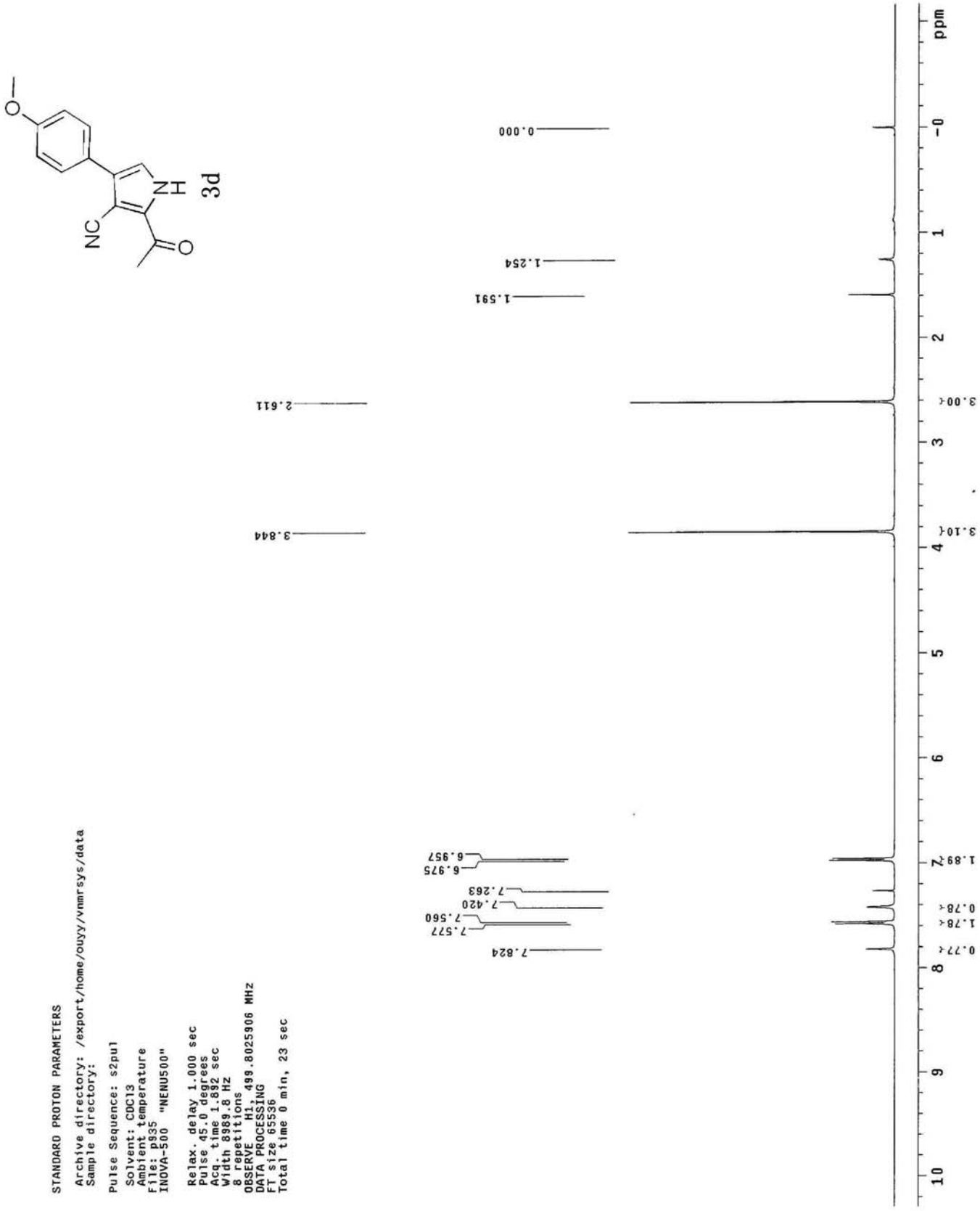


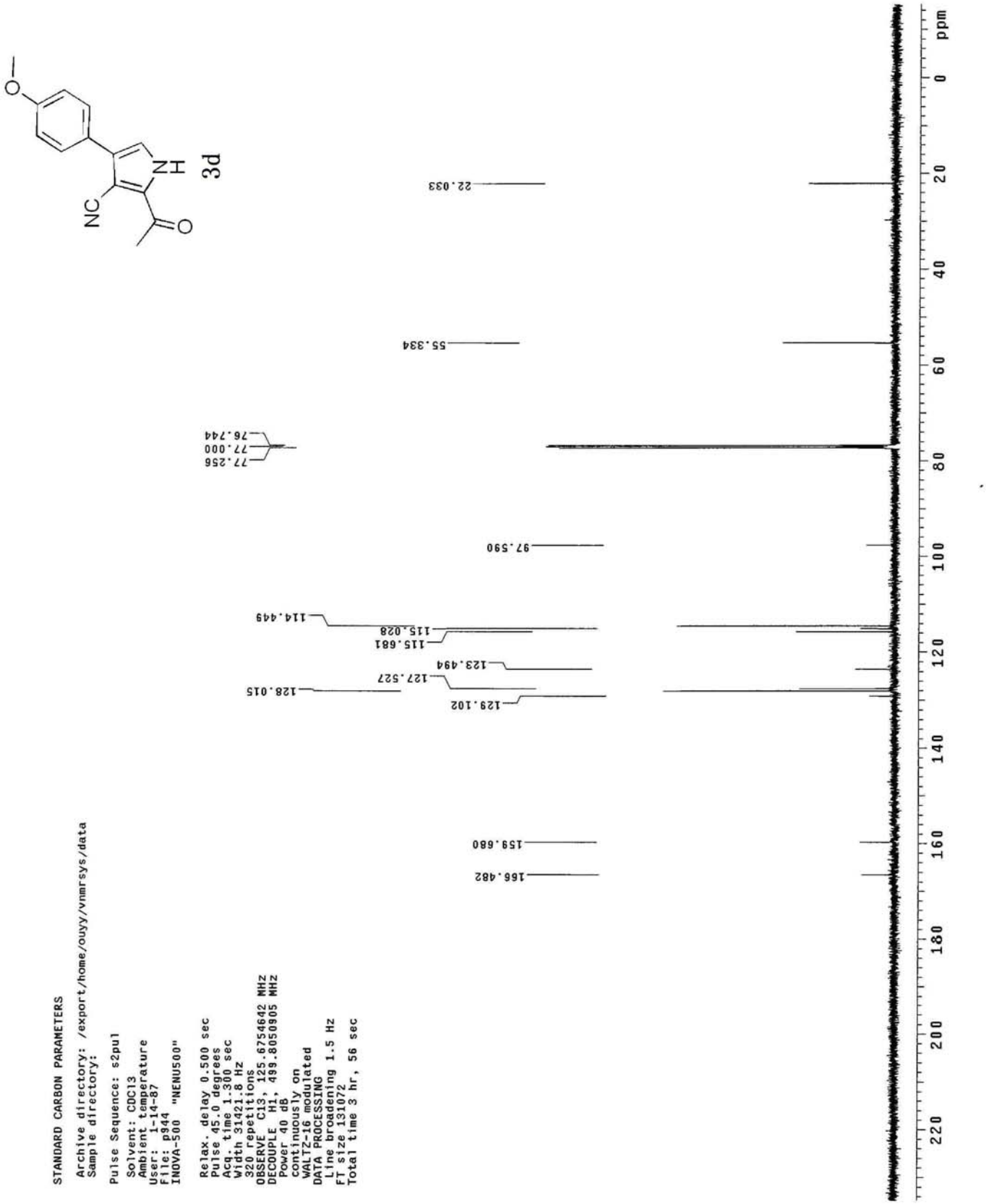
3b

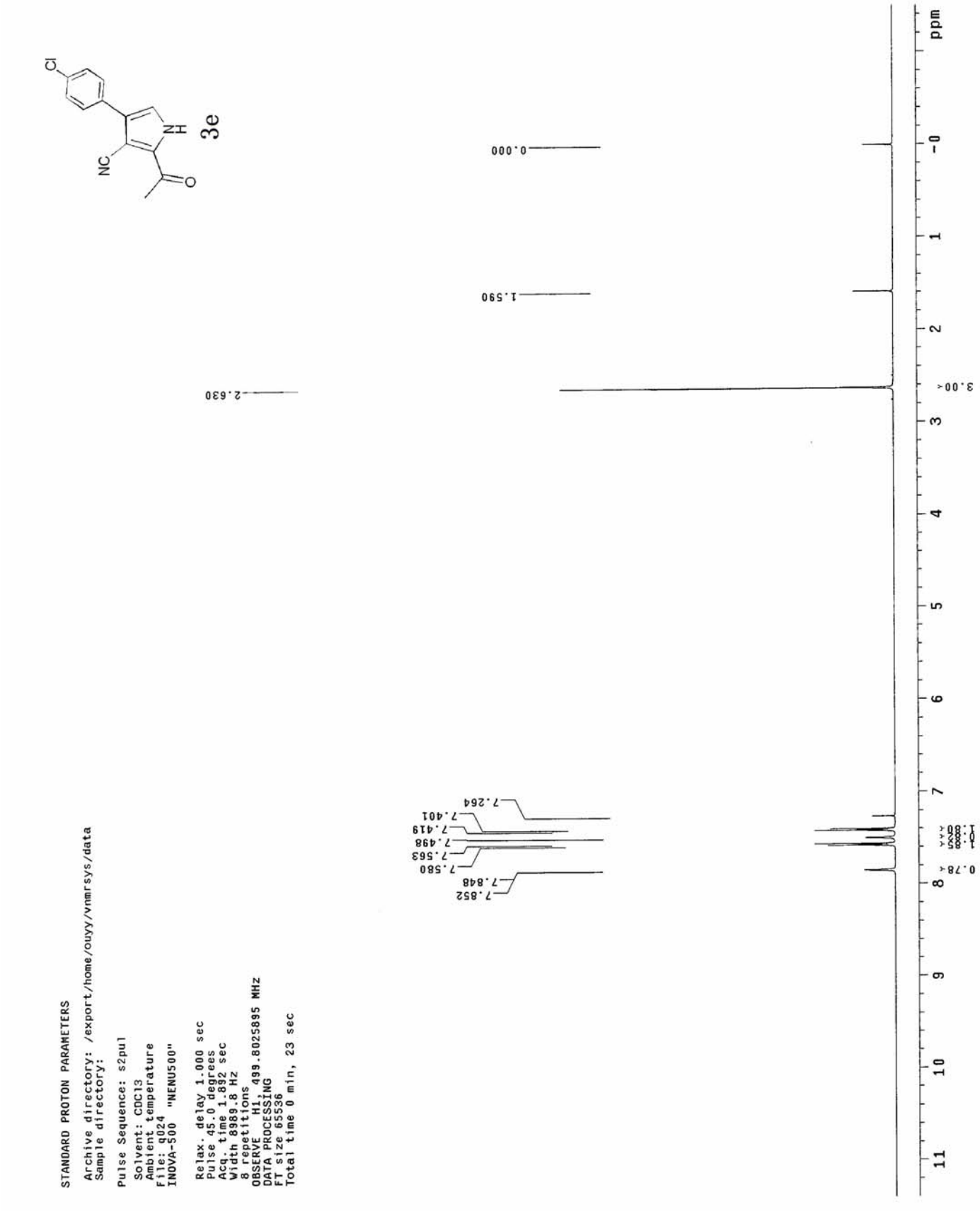












STANDARD PROTON PARAMETERS

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Sample directory:

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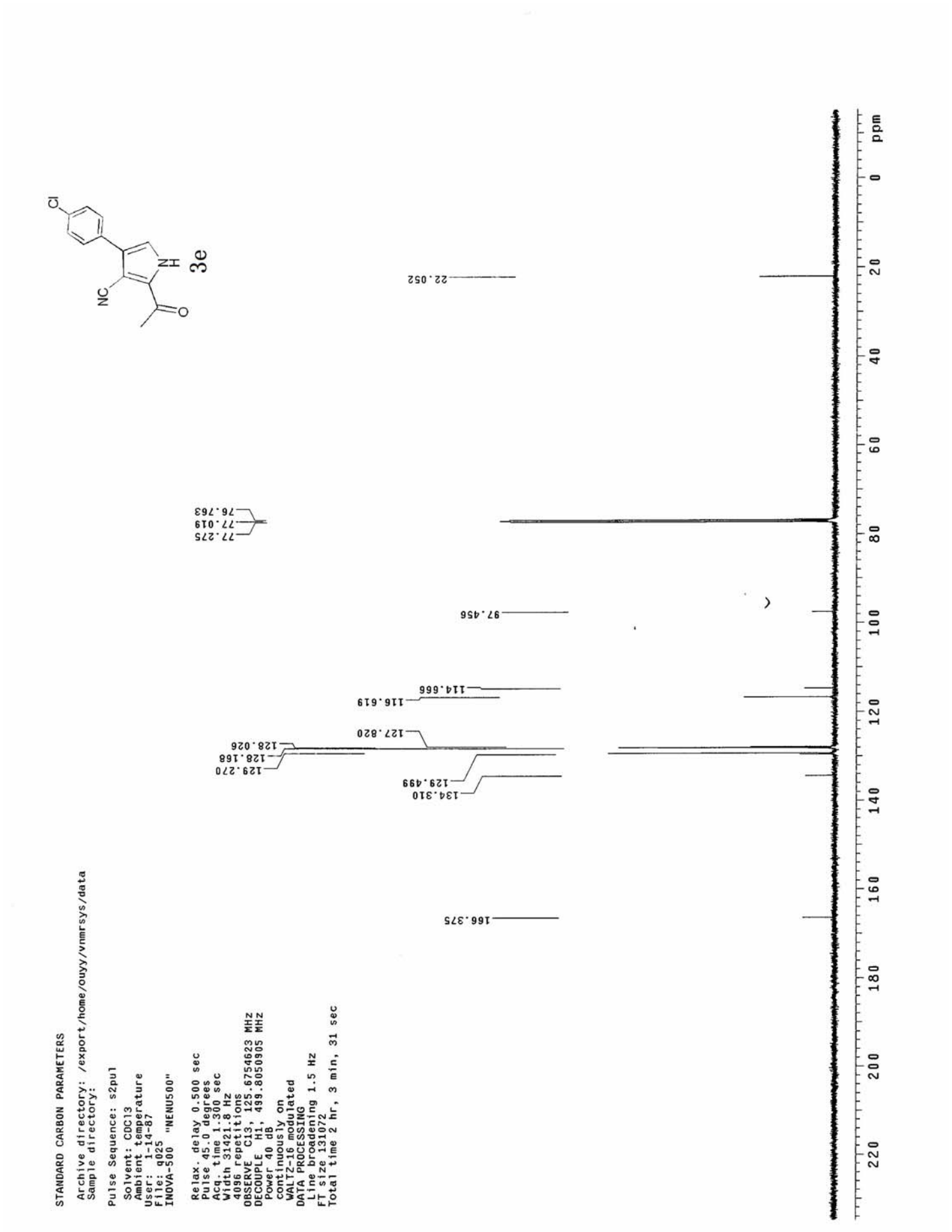
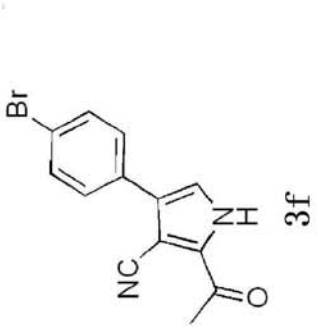
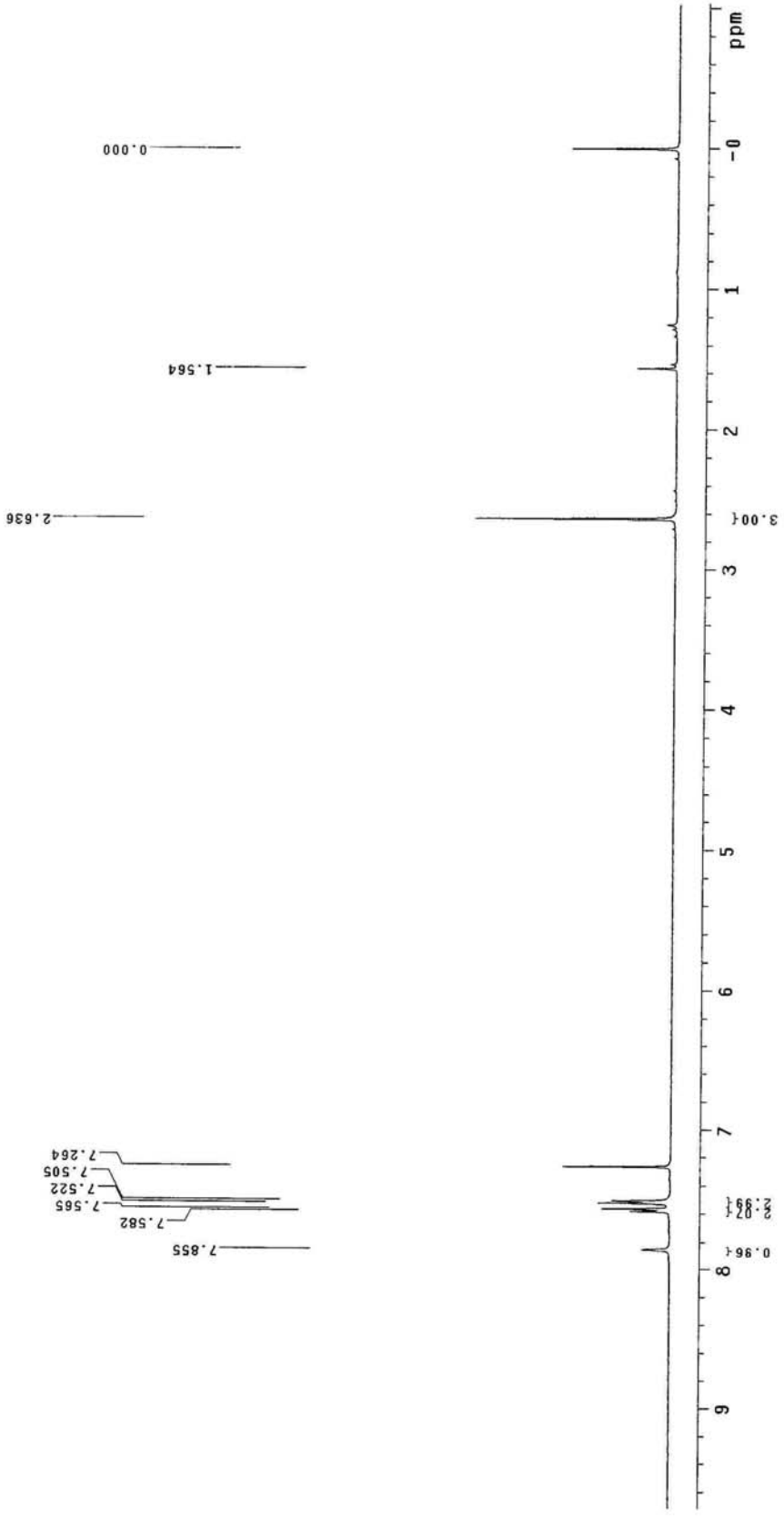
Solvent: CDCl3
Ambient temperature
File: q024
INOVA-500 "NENU500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8889.8 Hz
8 repetitions
OBSERVE H1, 499.8025895 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec





3f



STANDARD PROTON PARAMETERS

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Sample directory:

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Ambient temperature
File: s019
INOVA-500 "WENU500"

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Pulse 220.4 degrees
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16 repetitions
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DATA PROCESSING
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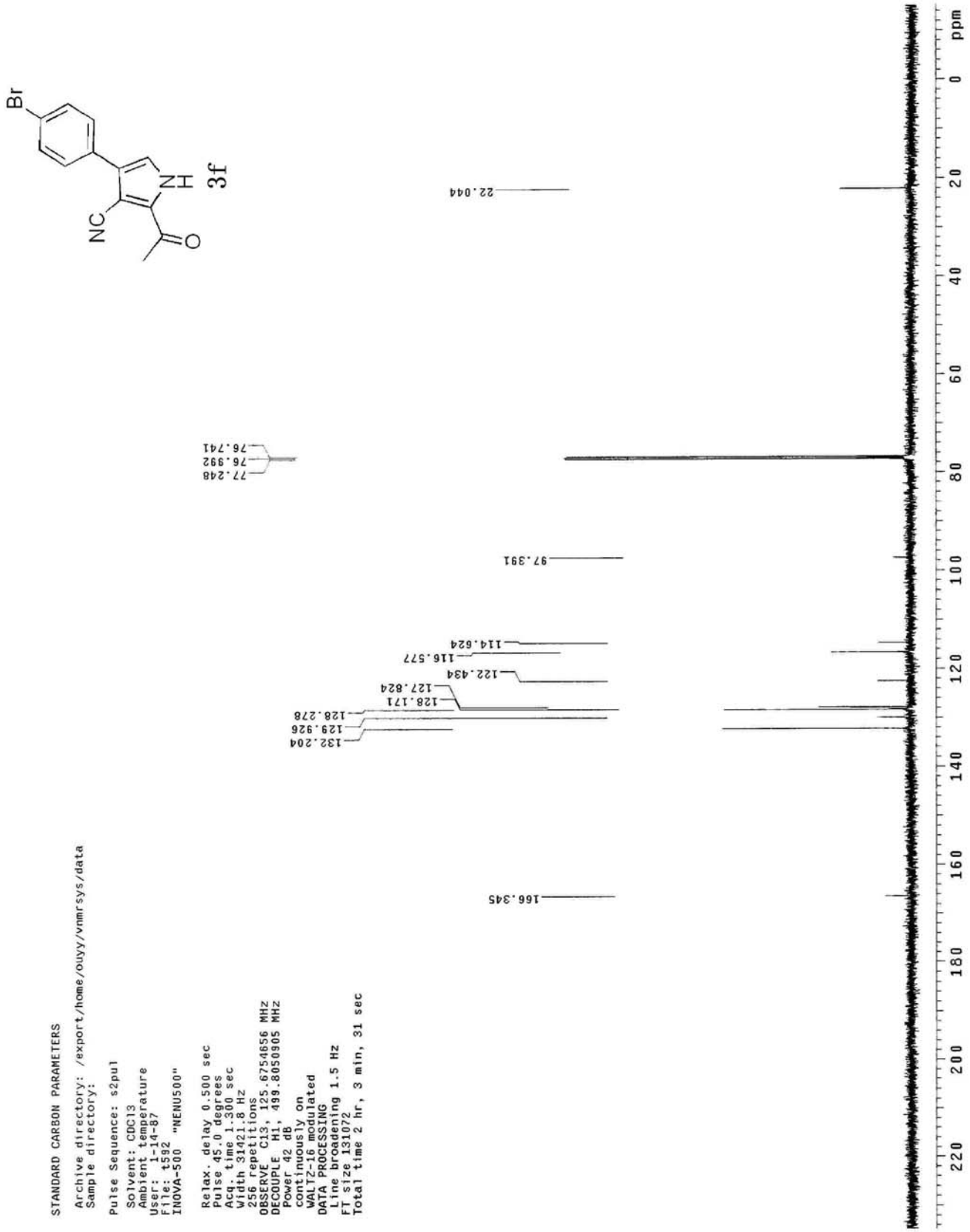
Total time 0 min, 39 sec



STANDARD CARBON PARAMETERS

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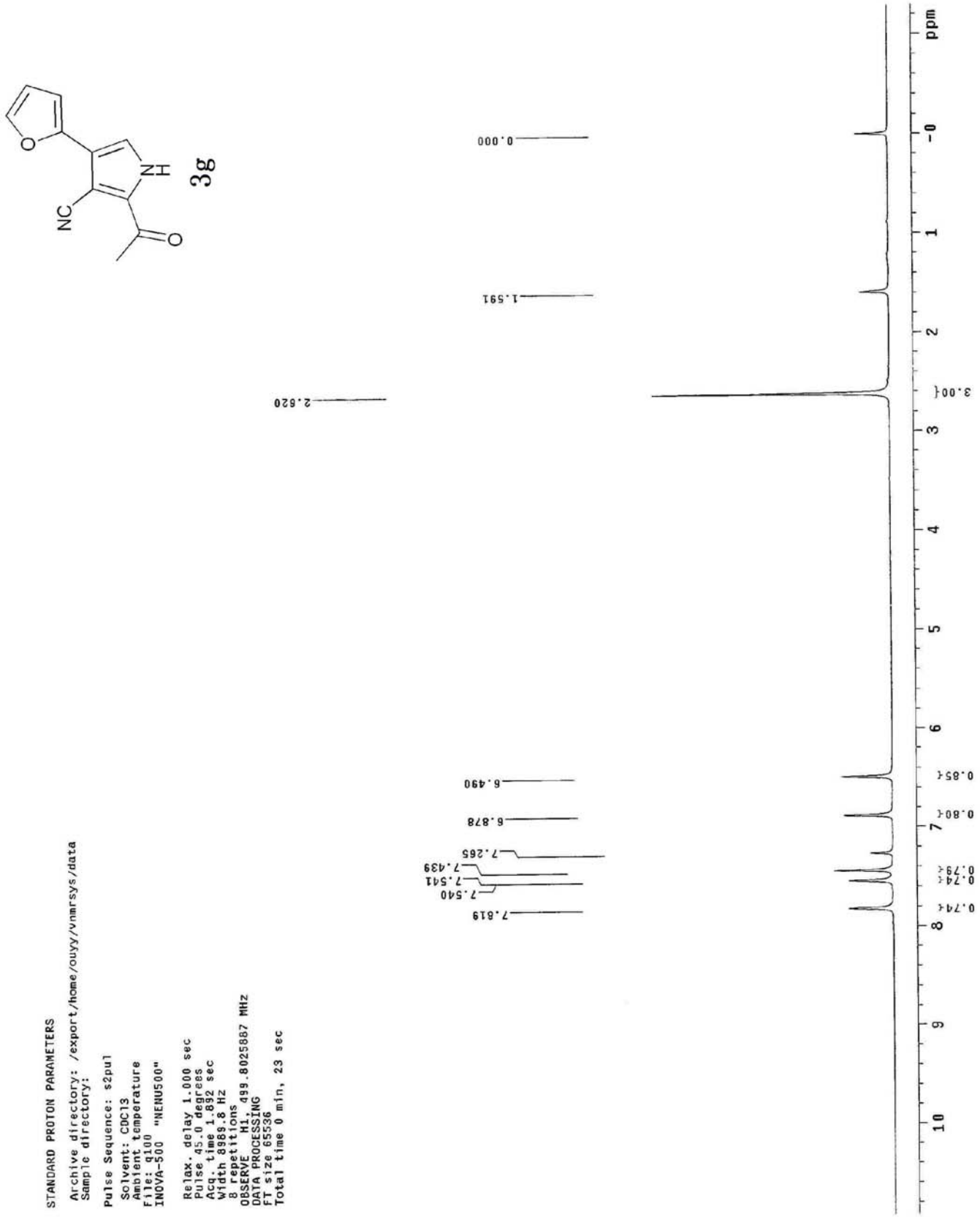
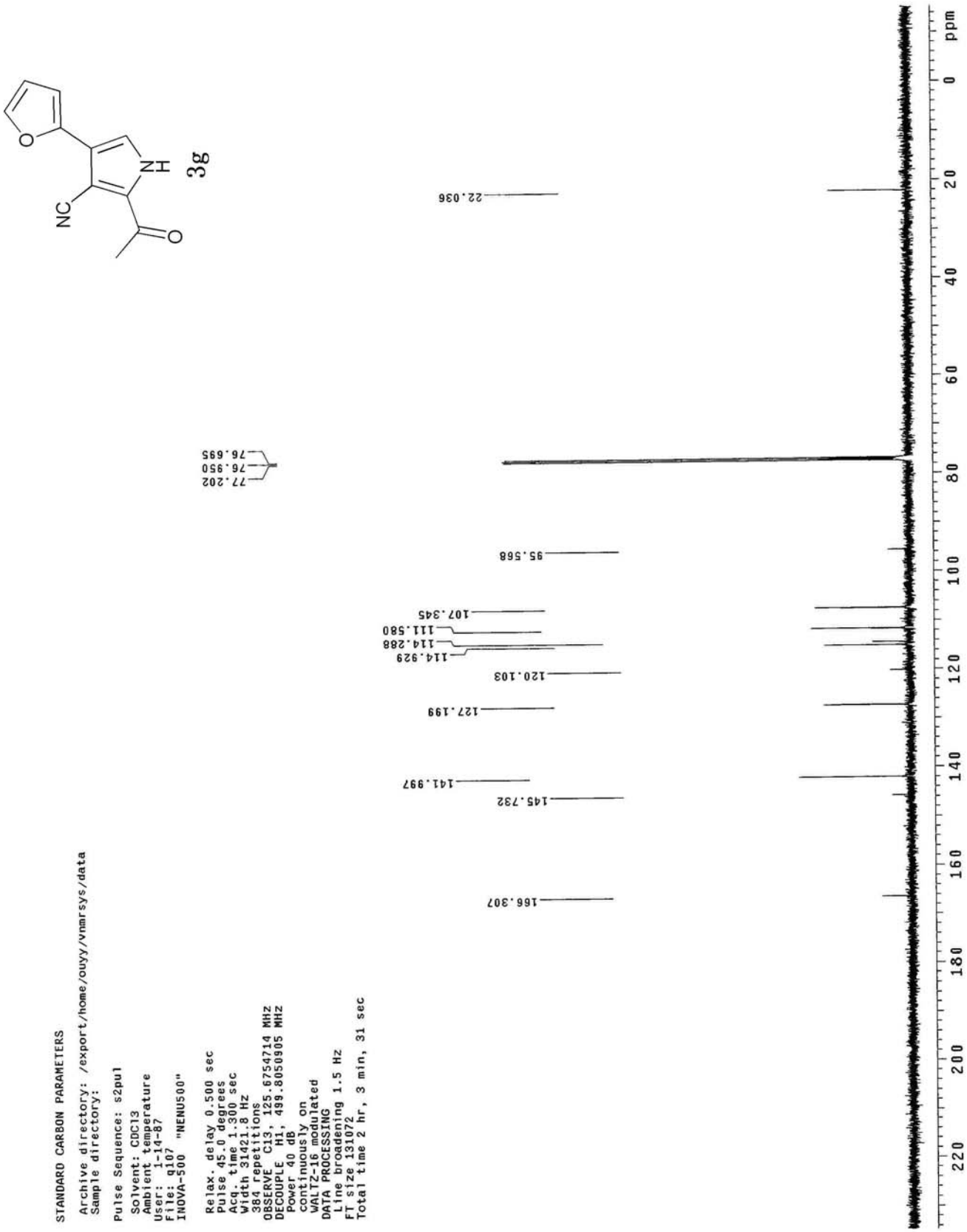
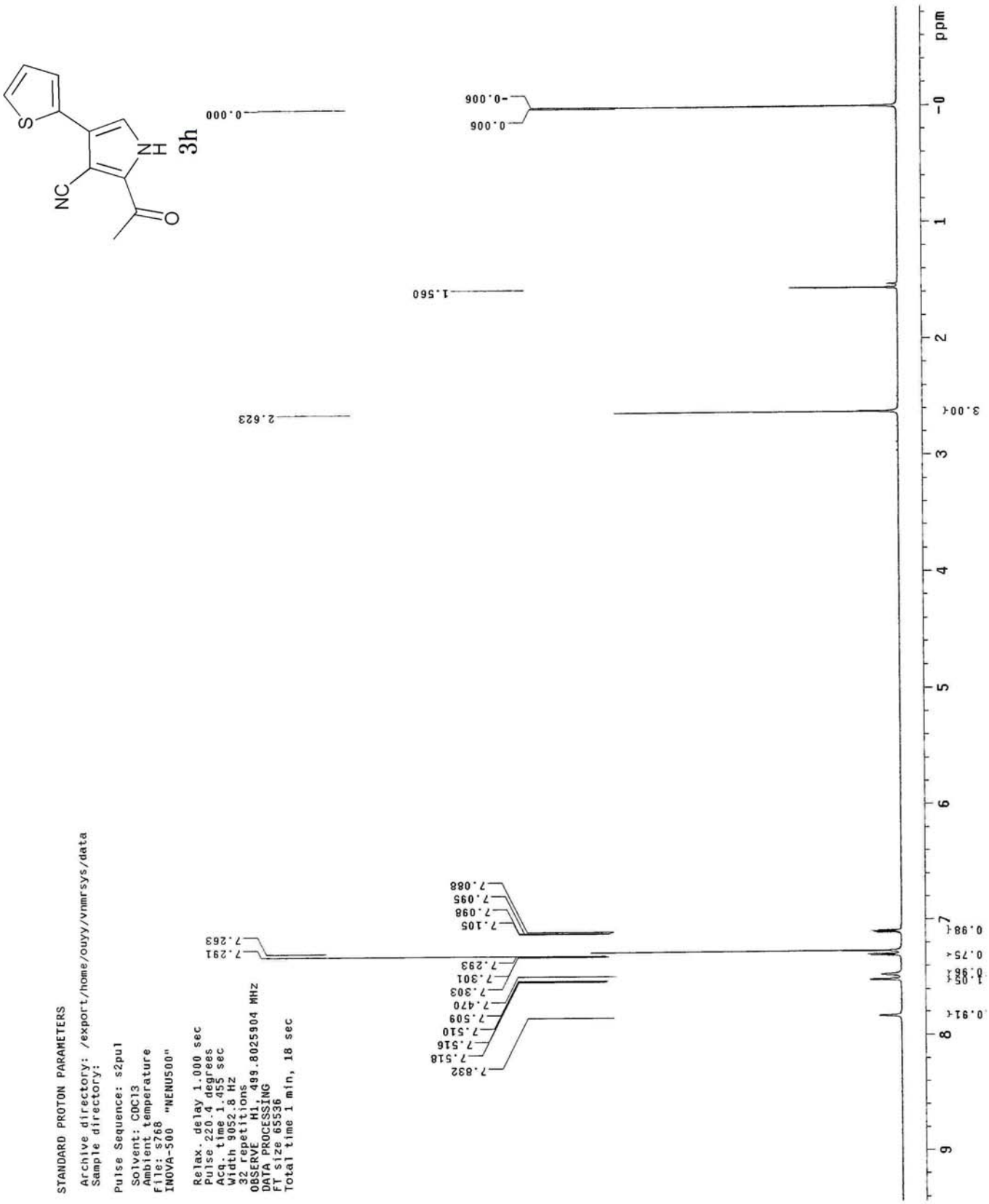
Sample directory:

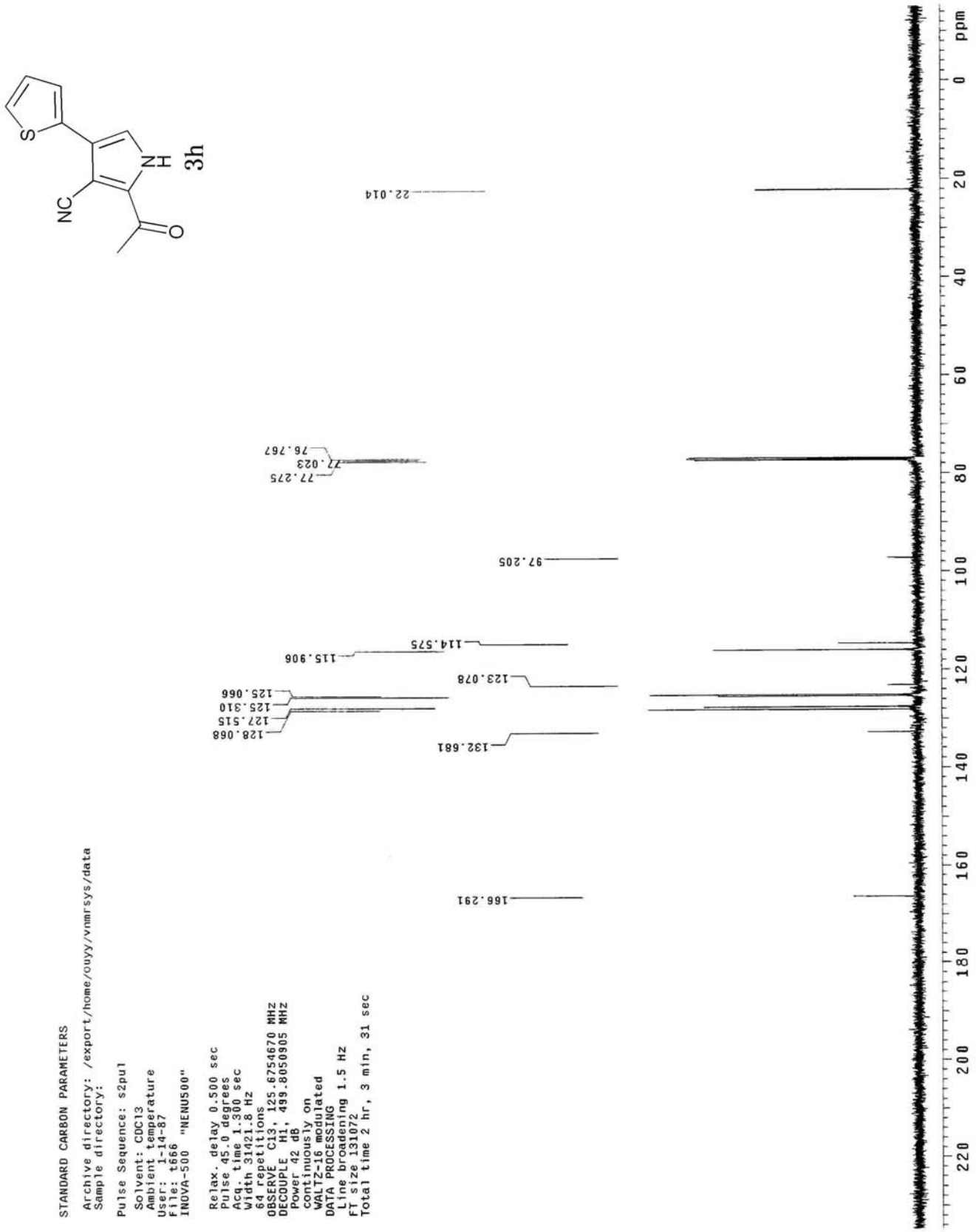
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: t592
INOVA-500 "NENU500"

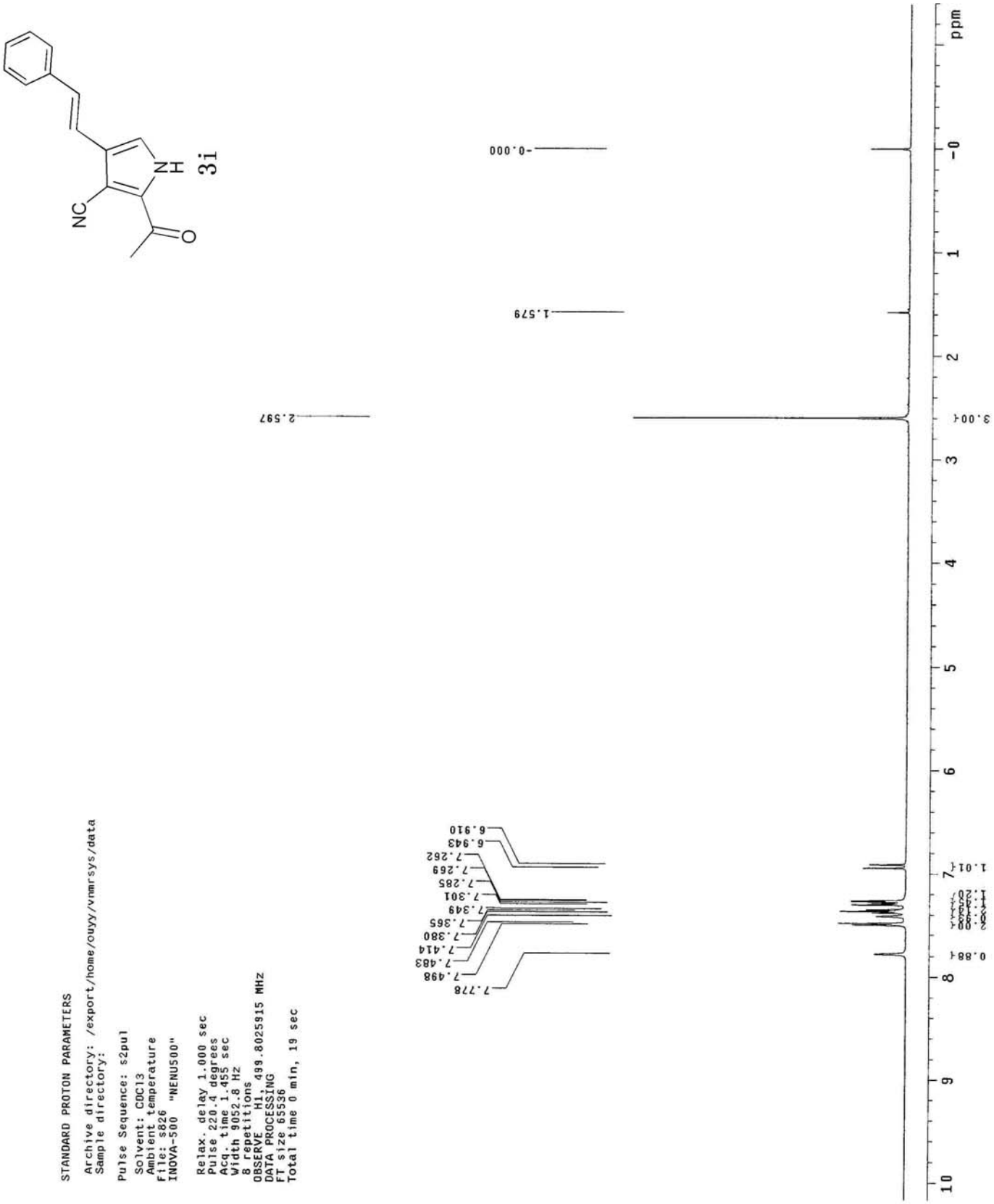
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
256 repetitions
OBSERVE C13, 125.6754656 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec










3i
NC
O
N
H
STANDARD PROTON PARAMETERS
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Solvent: CDCl3
Ambient temperature
File: s826
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Relax. delay 1.000 sec
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Acq. time 1.455 sec
Width 9052.8 Hz
8 repetitions
OBSERVE H1, 499.8025915 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 19 sec
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1.579
0.000
7.778
7.498
7.483
7.414
7.380
7.365
7.349
7.301
7.285
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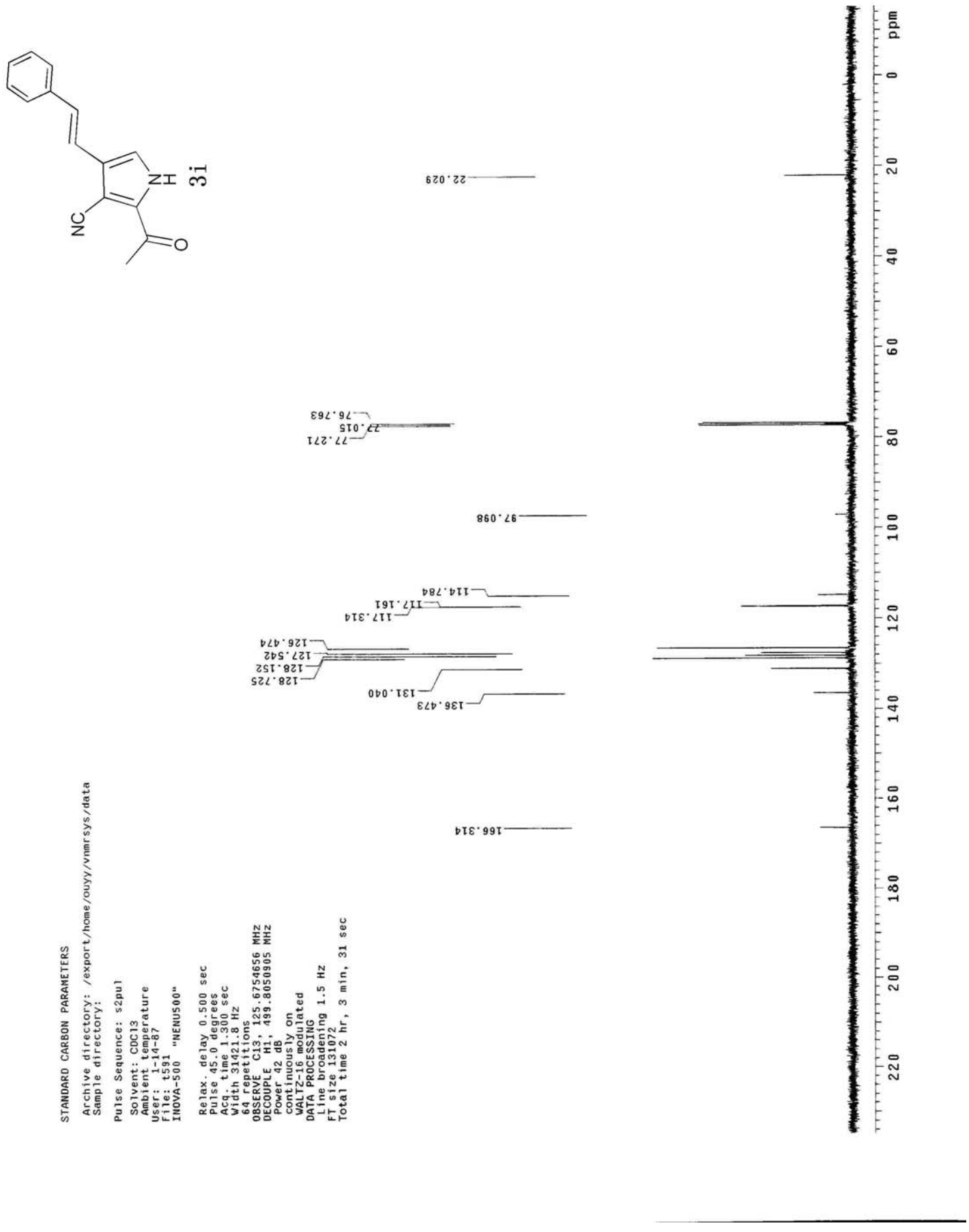
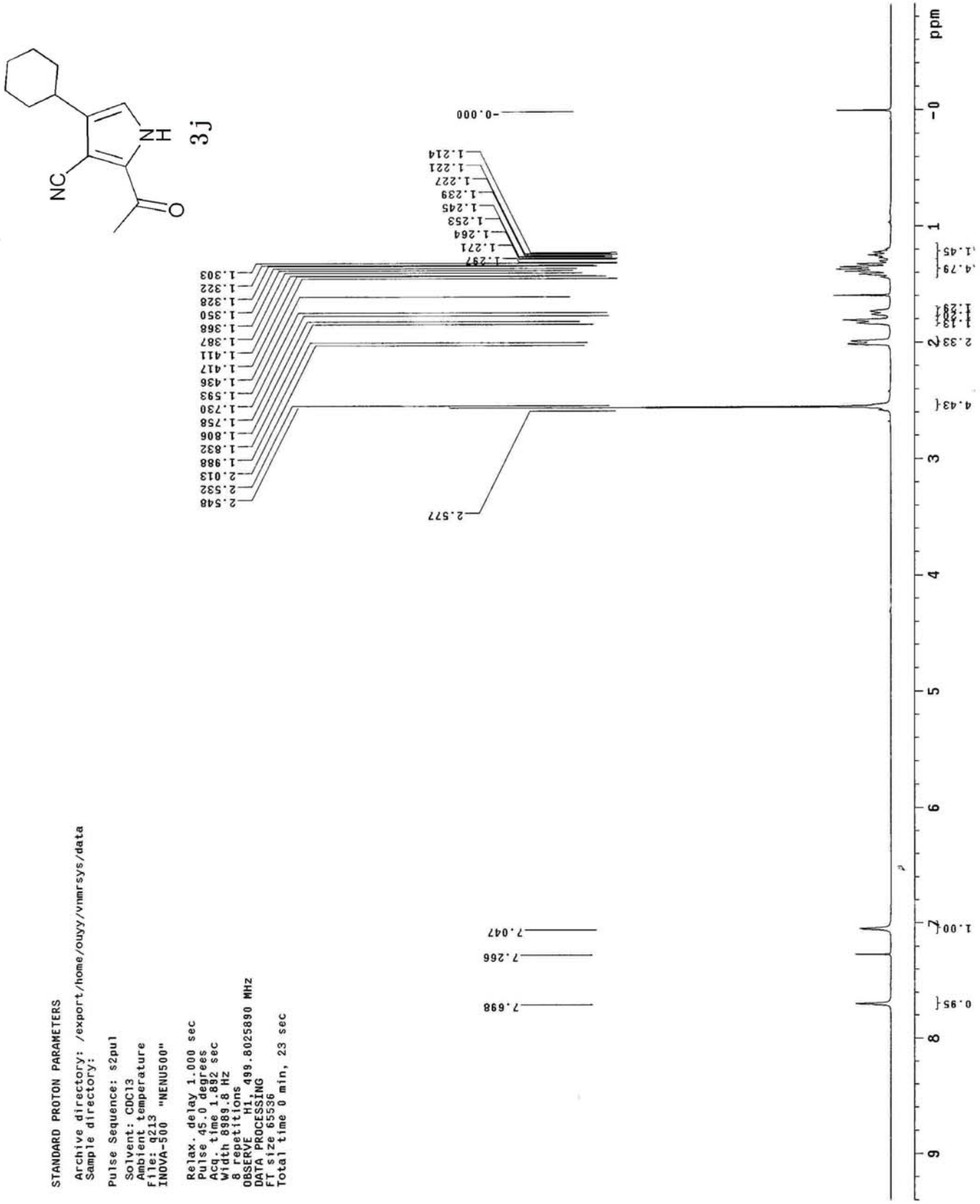
6.910
3.004
1.014
0.884
ppm




3j
NC
O
N
H
STANDARD PROTON PARAMETERS
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Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: q213
INOVA-500 "NENU500"
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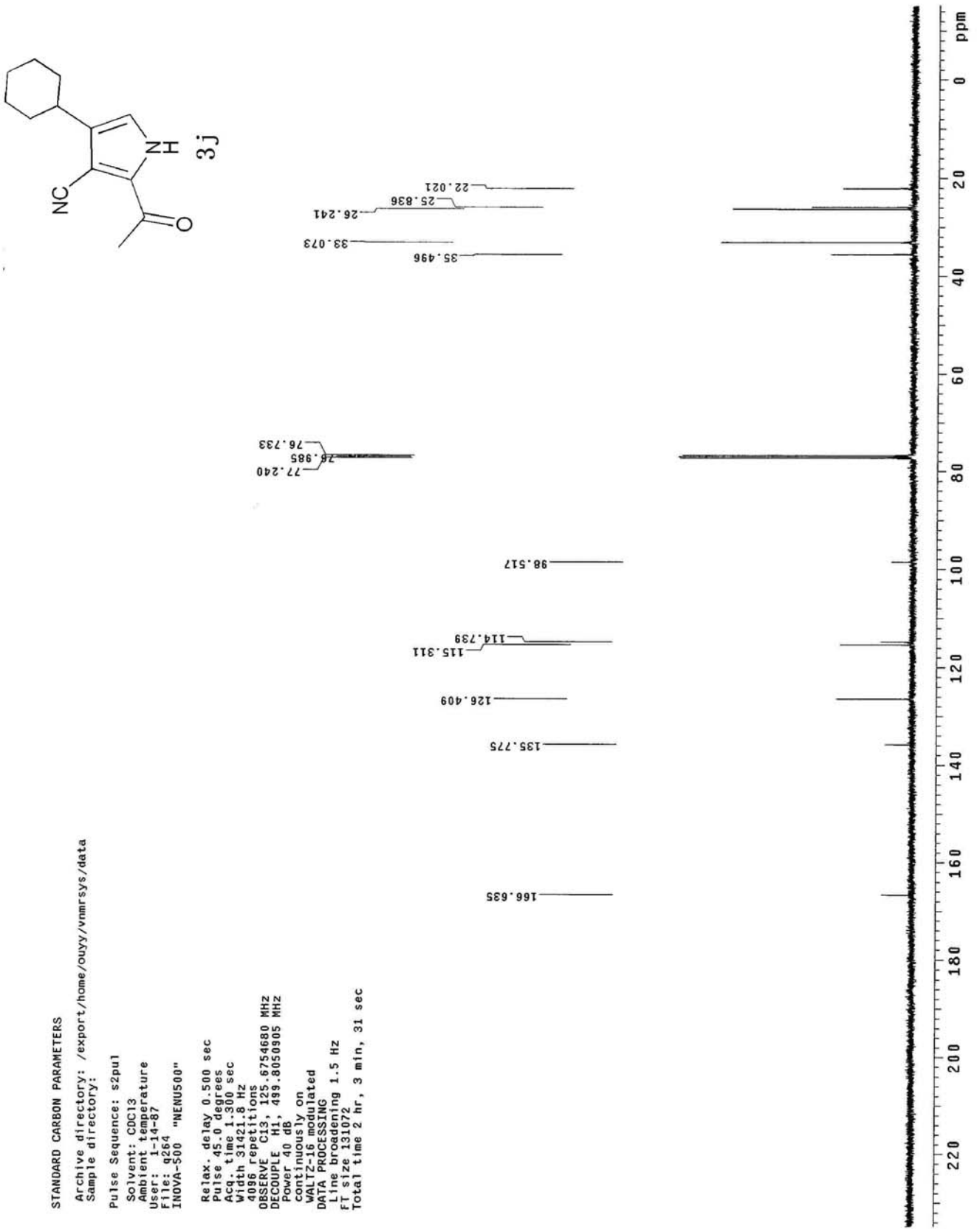
Pulse 45.0 degrees
Acq. time 1.882 sec
Width 8989.8 Hz
8 repetitions
OBSERVE H1, 499.8025890 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
ppm

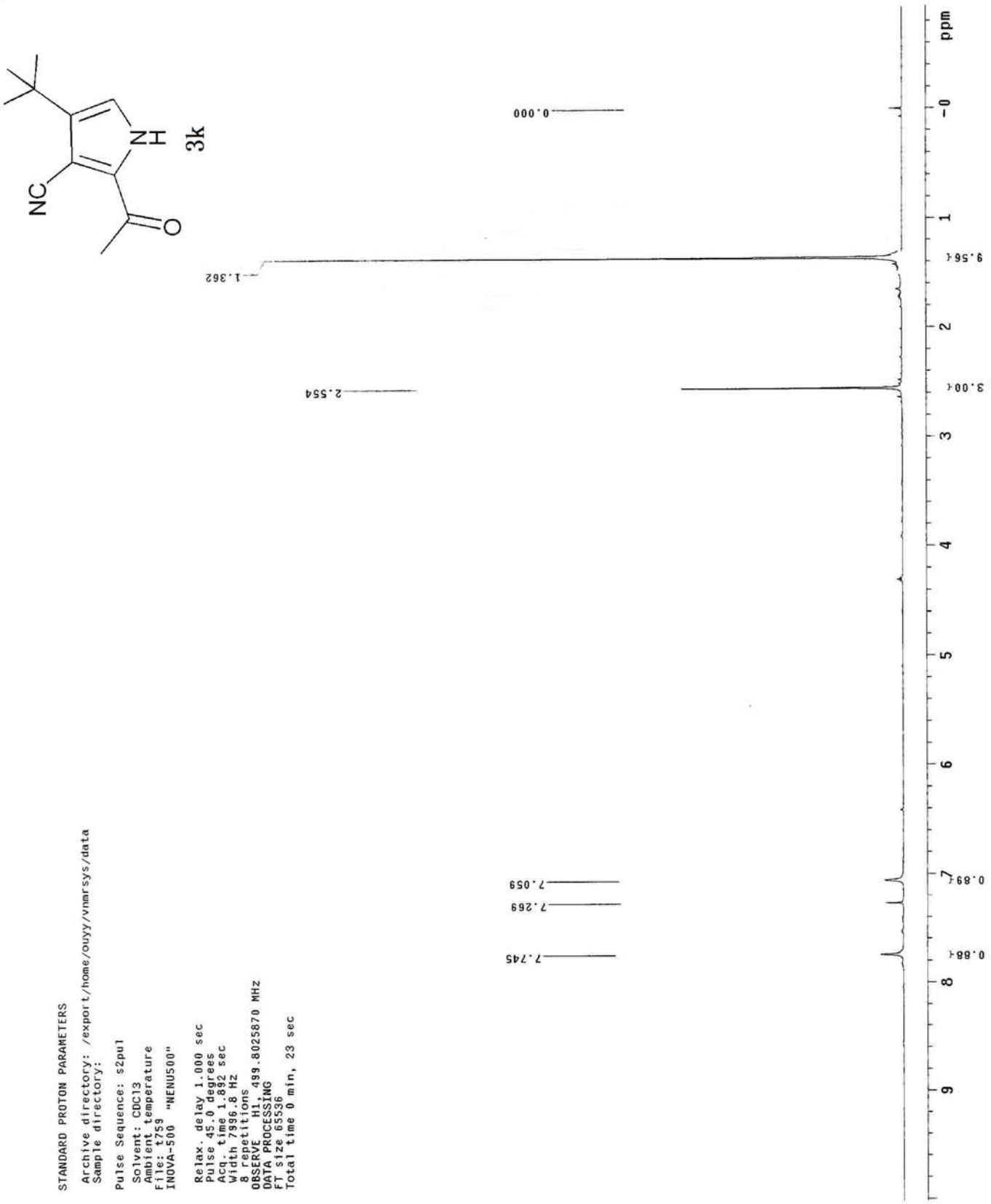


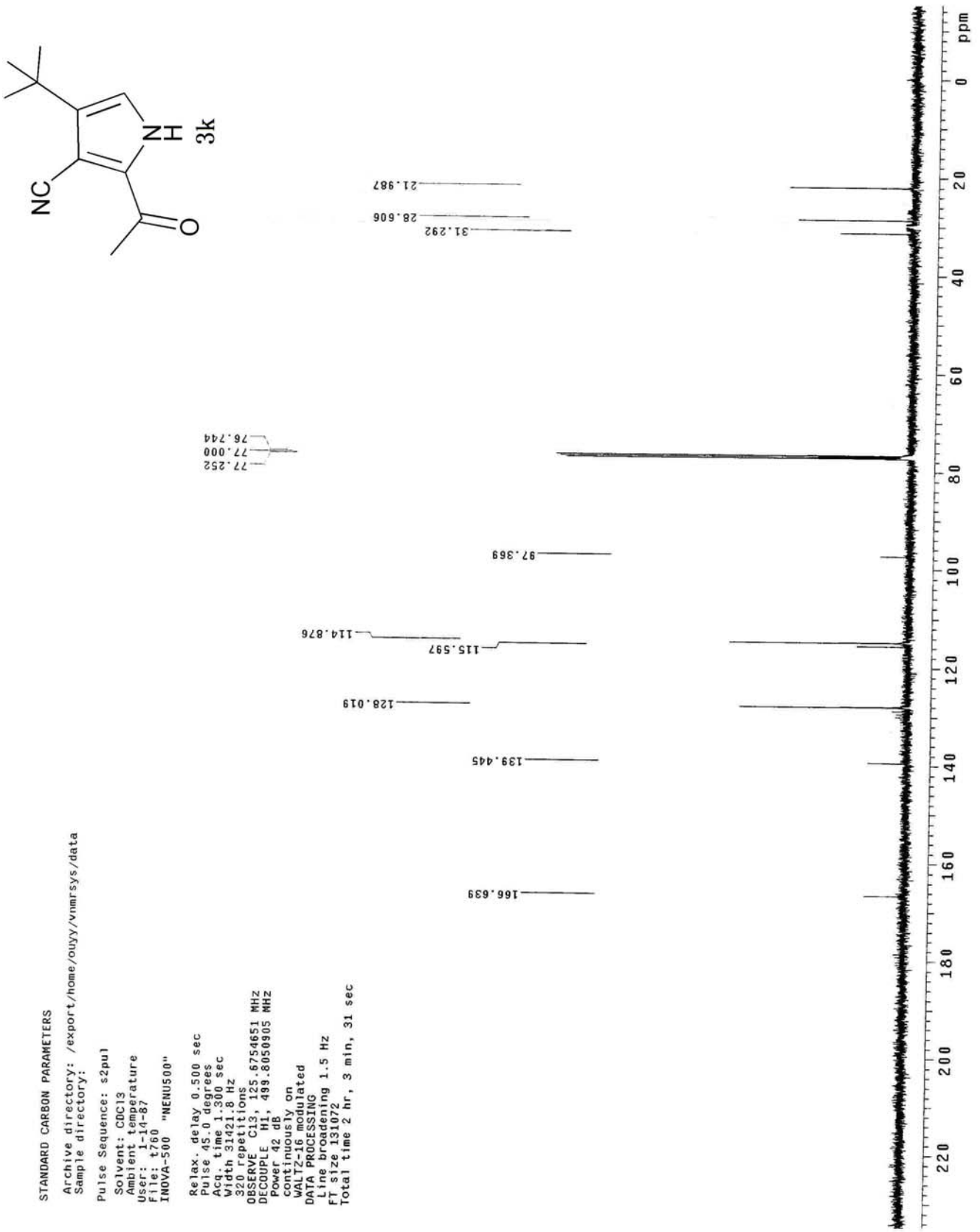
STANDARD CARBON PARAMETERS

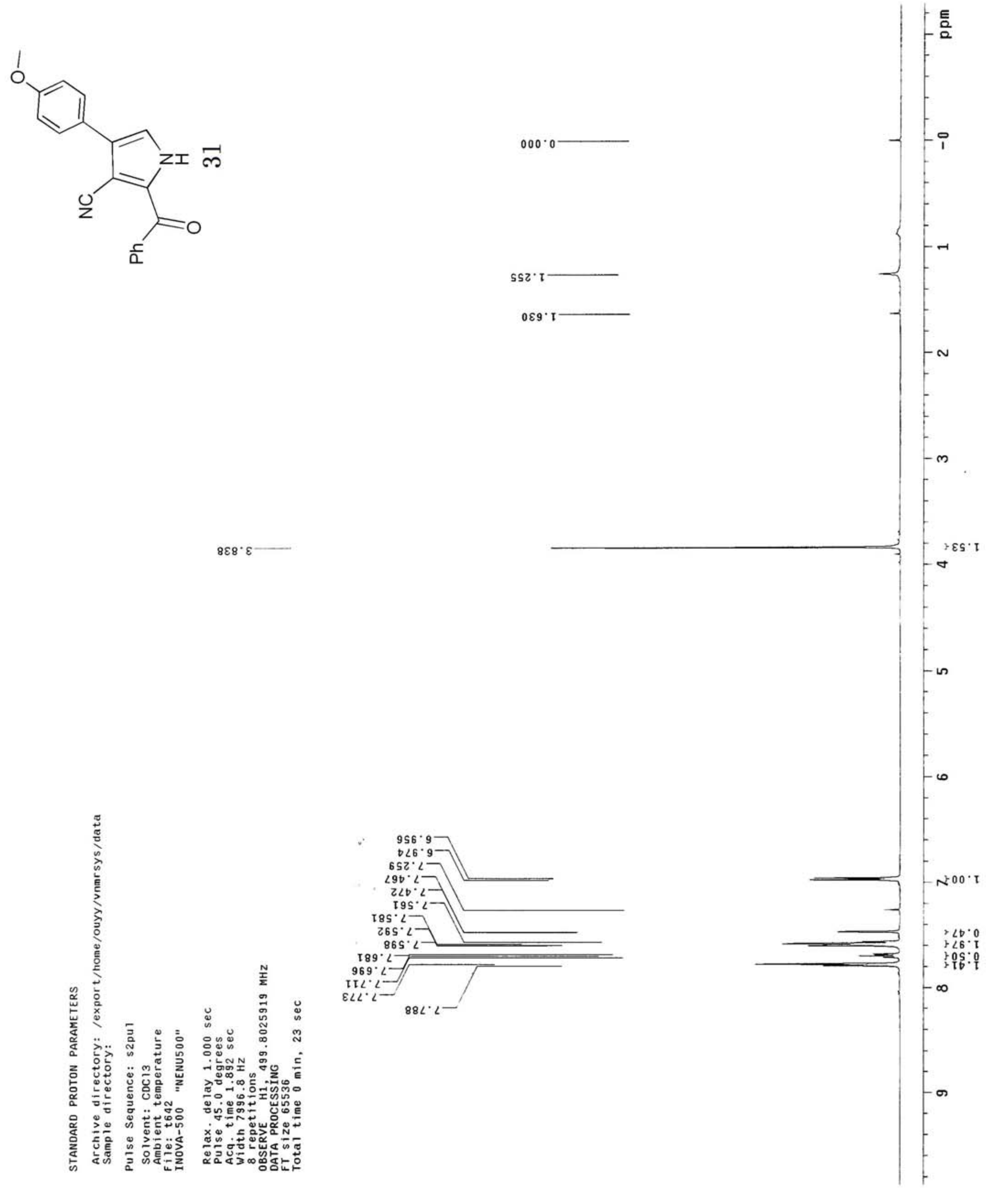
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:

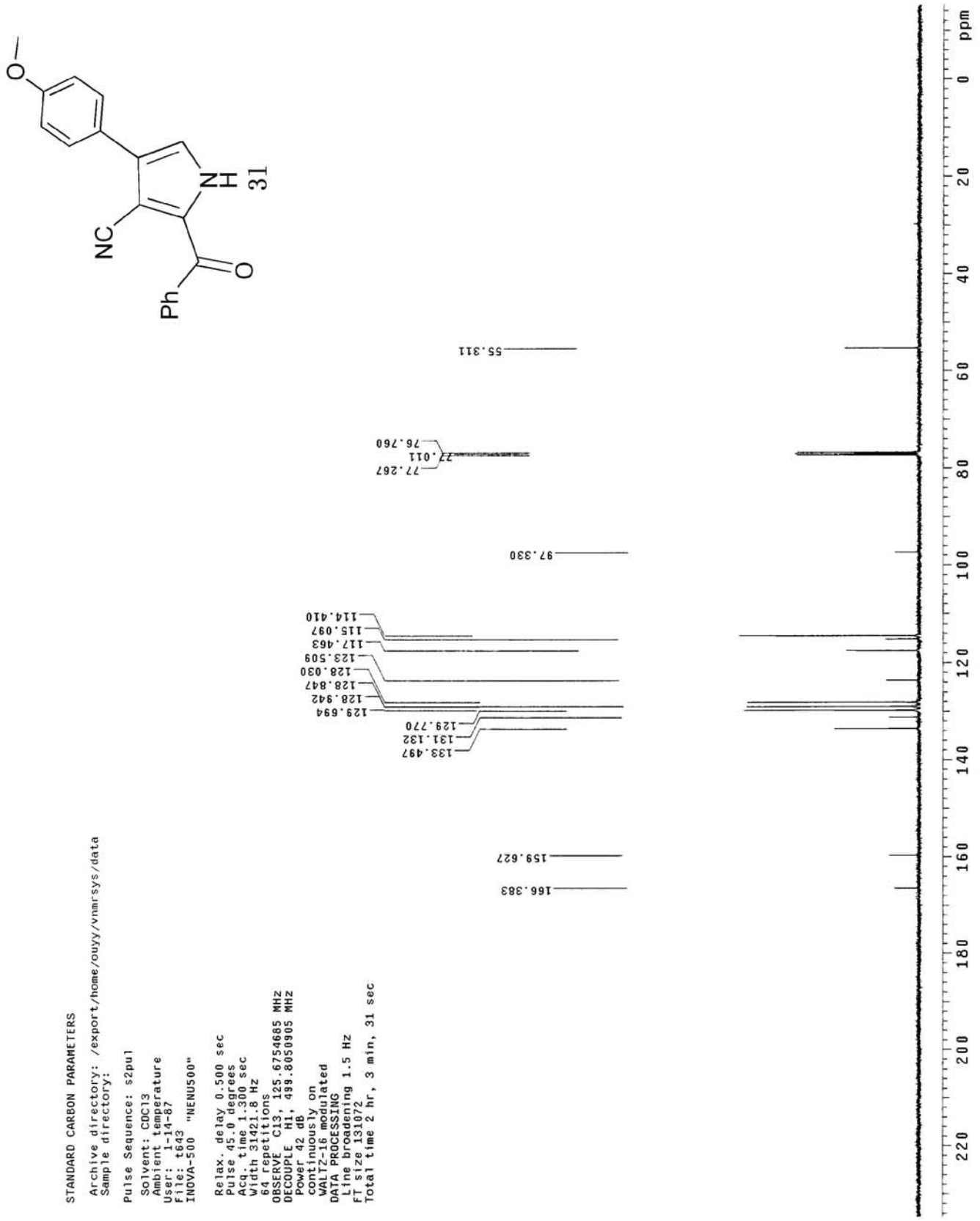
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: q264 "NENUS00"
INOVA-500

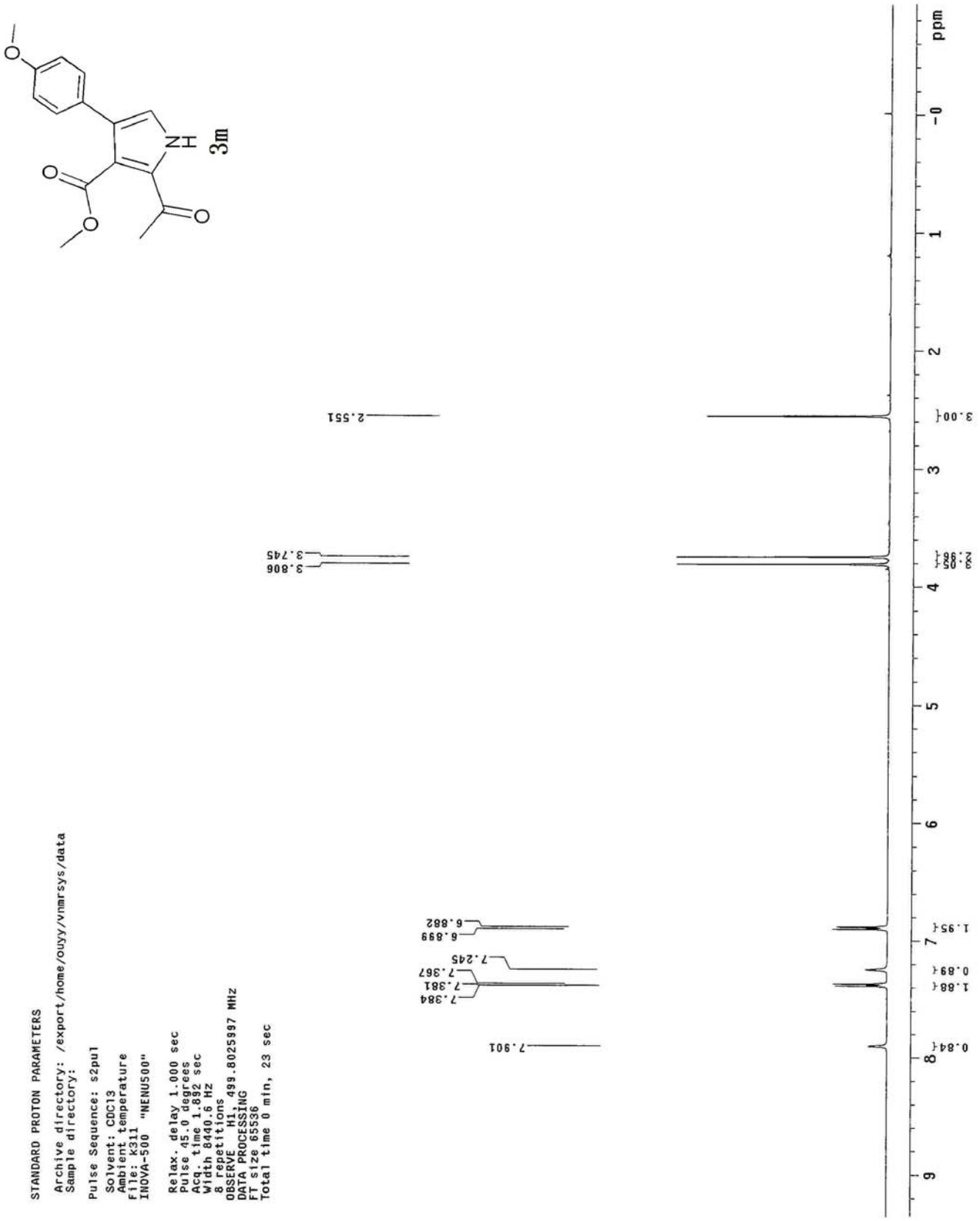
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
4096 repetitions
OBSERVE C13, 125.6754680 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec

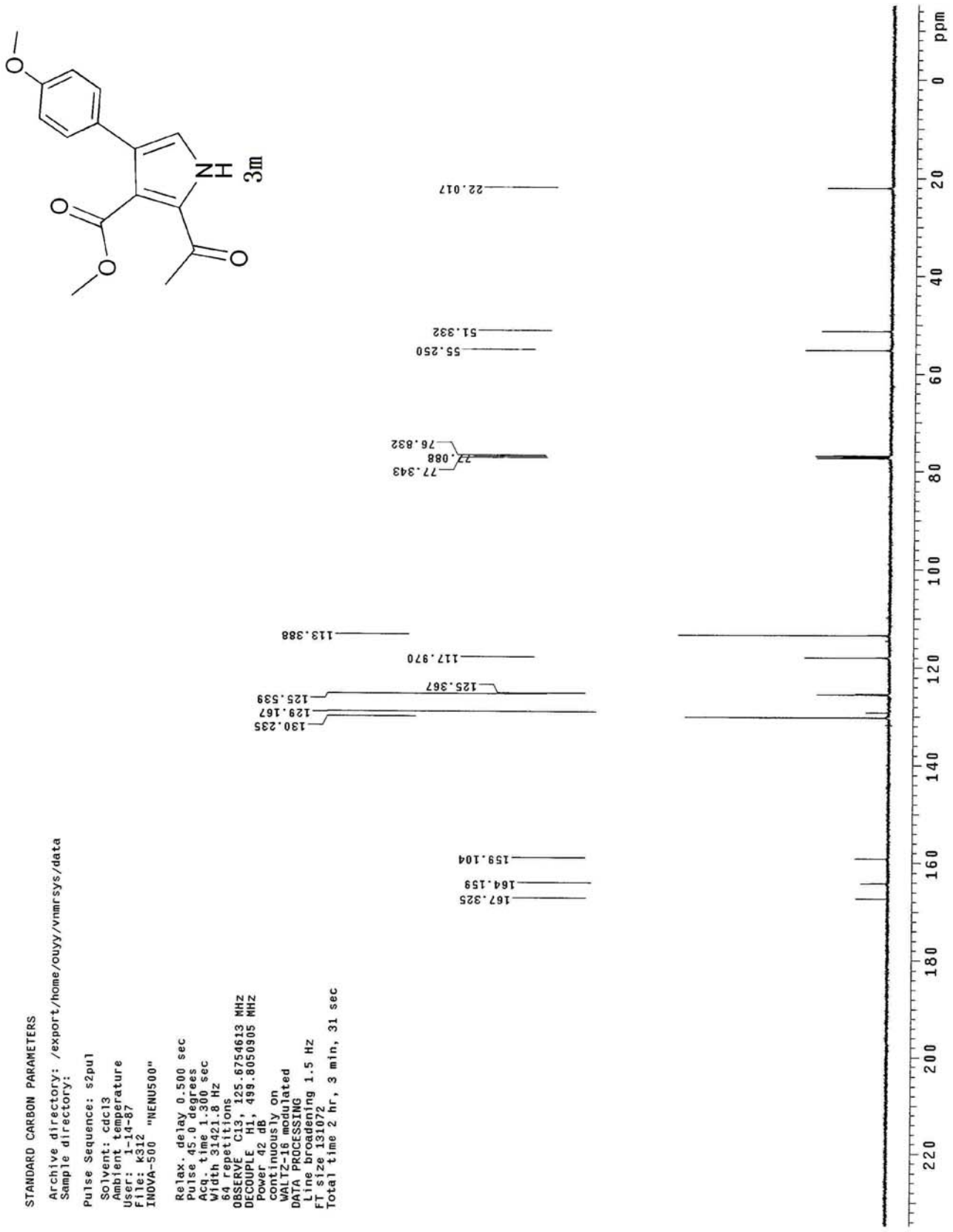


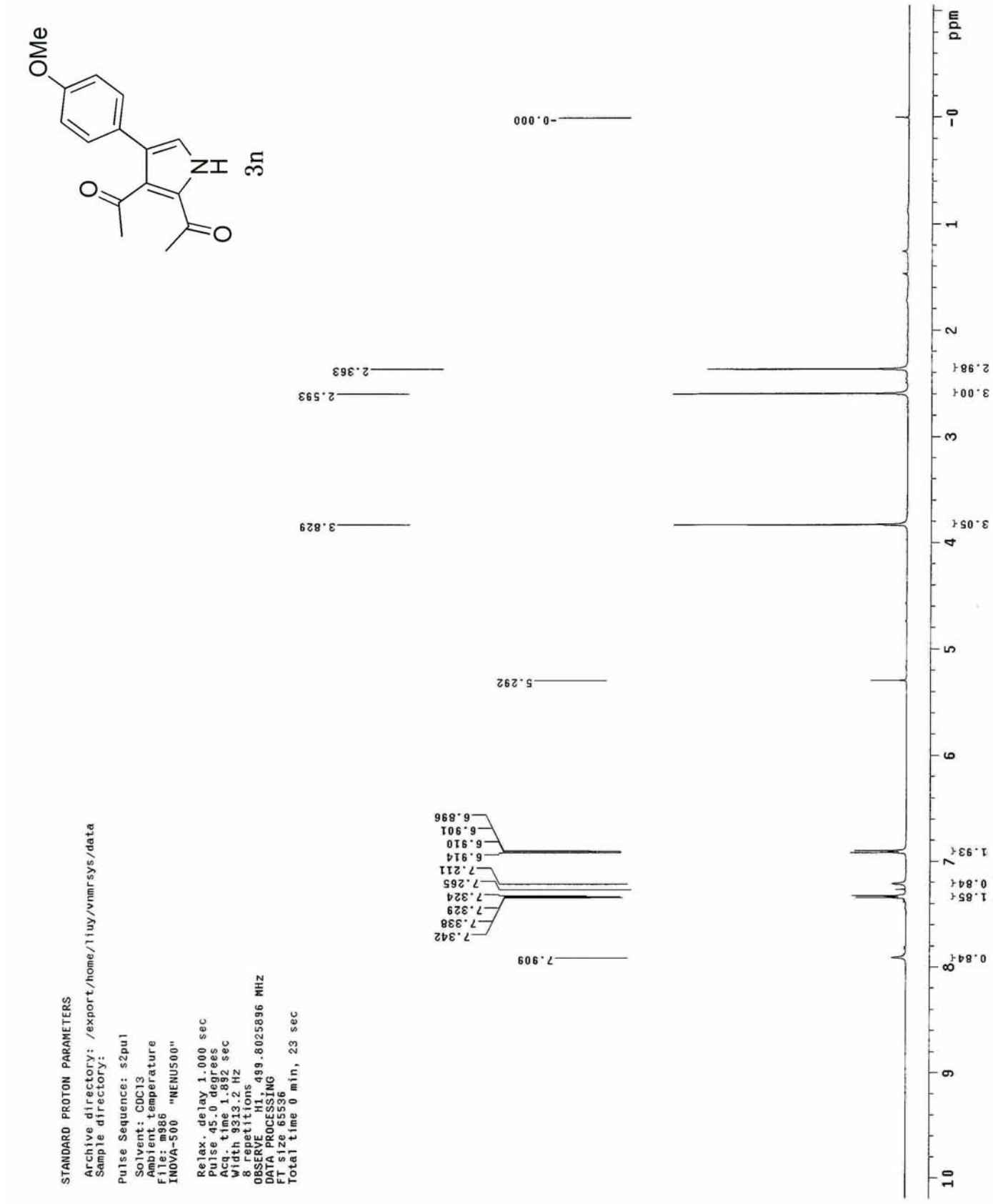


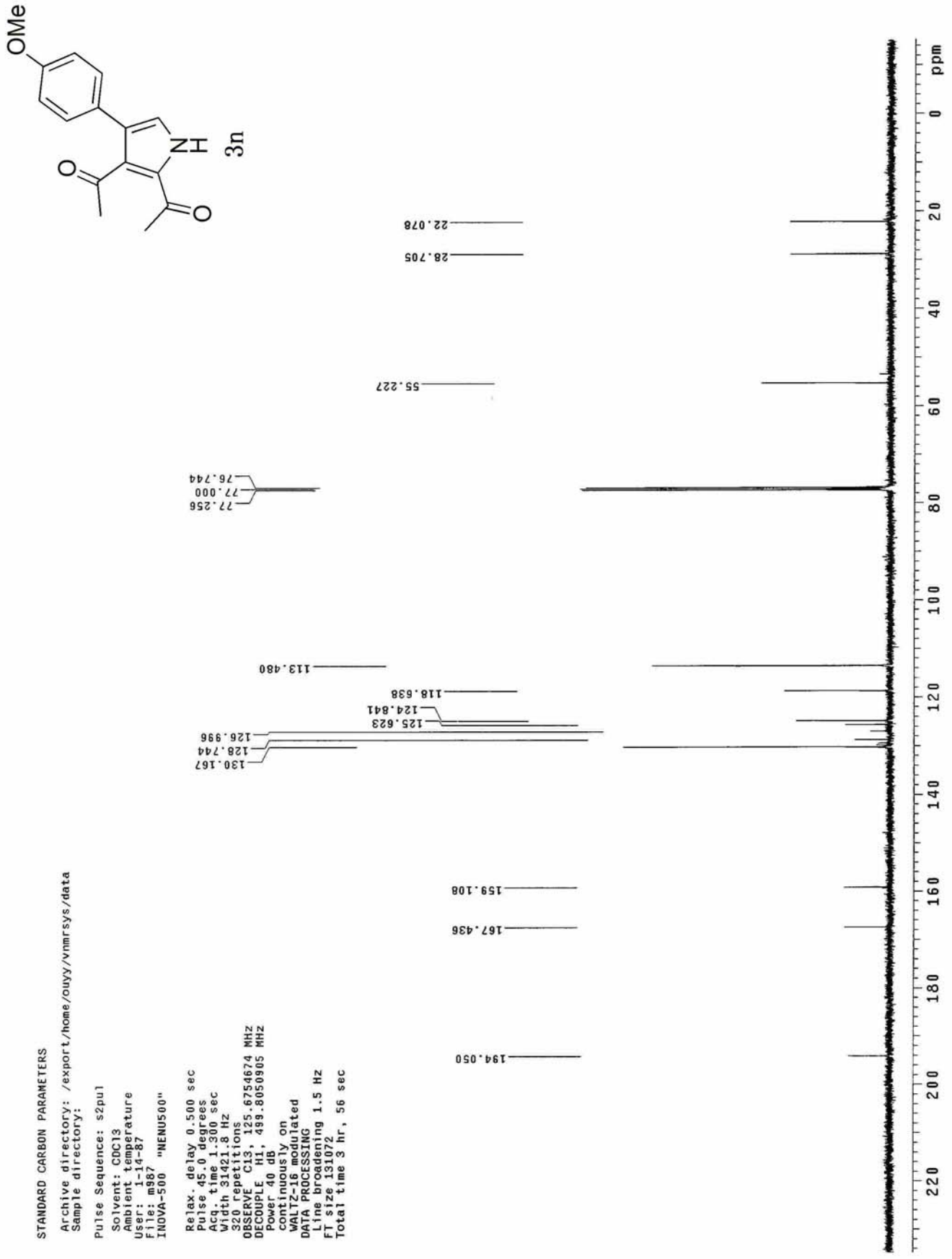

31
STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: t642
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7996.8 Hz
8 repetitions
OBSERVE H1, 499.8025919 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
0.000
1.255
1.630
3.838
6.956
6.974
7.259
7.467
7.472
7.561
7.581
7.592
7.598
7.681
7.696
7.711
7.773
7.788
1.53
1.00
0.47
1.97
0.50
1.41
ppm

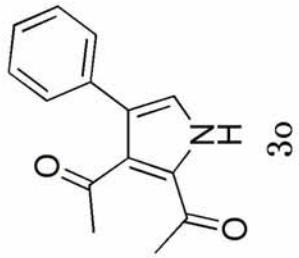

O
NC
N
H
O
Ph
31
55.311
76.760
77.011
77.267
97.330
114.410
115.097
117.463
123.509
128.030
128.847
128.942
129.694
129.770
131.132
133.497
159.627
166.383
220
200
180
160
140
120
100
80
60
40
20
0
ppm
STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: t643
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
64 repetitions
OBSERVE C13, 125.6754685 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec




3m
STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: k312 "NENU500"
INOVA-500
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
64 repetitions
OBSERVE C13, 125.6754613 MHz
DECOUPLE H1, 499.8050305 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
22.017
51.332
55.250
76.832
77.088
77.343
113.388
117.970
125.367
125.539
129.167
130.235
159.104
164.159
167.325
220
200
180
160
140
120
100
80
60
40
20
0
ppm



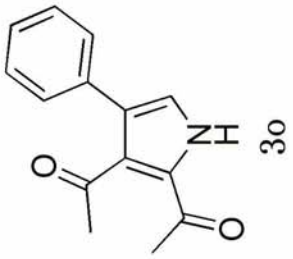




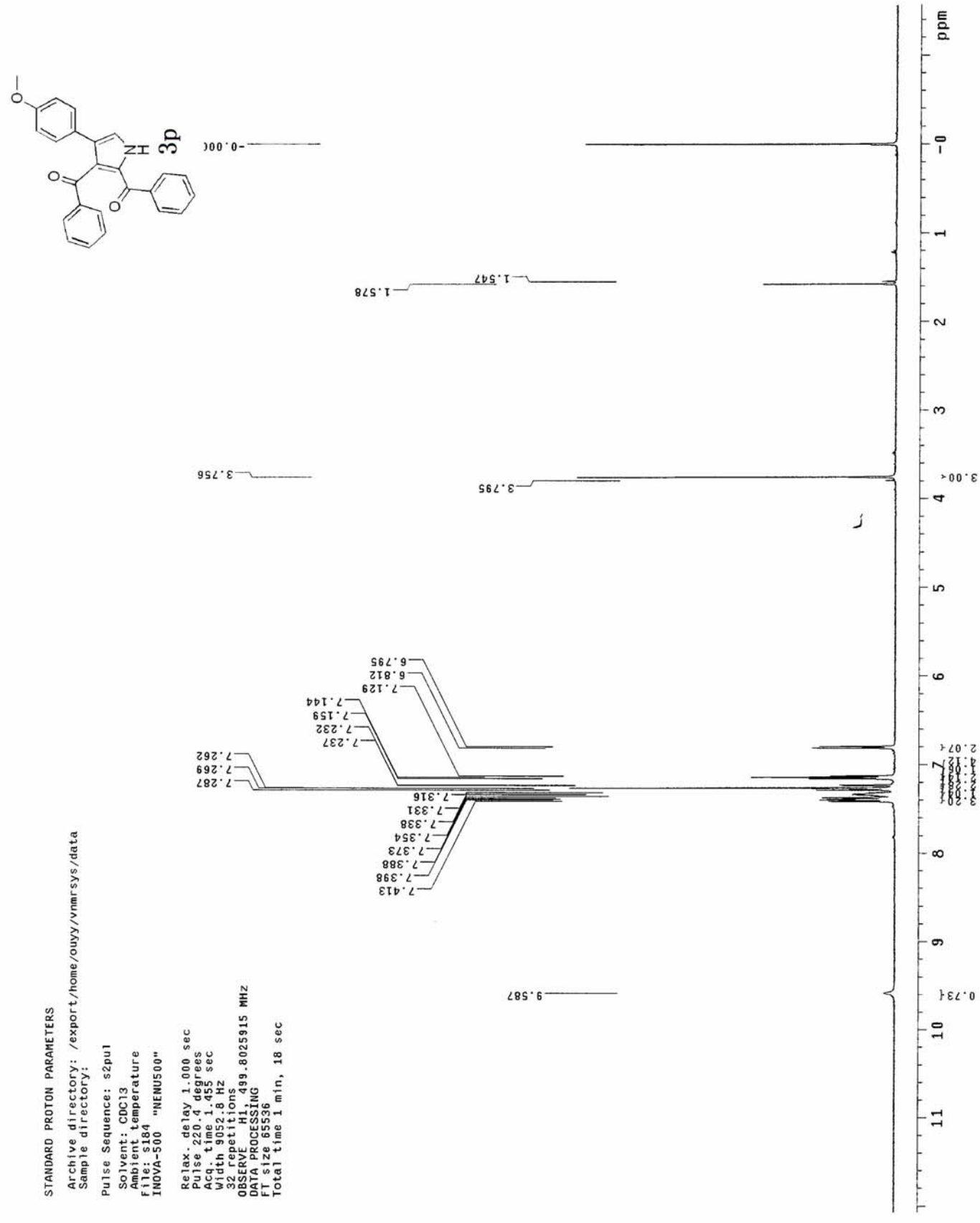
3o

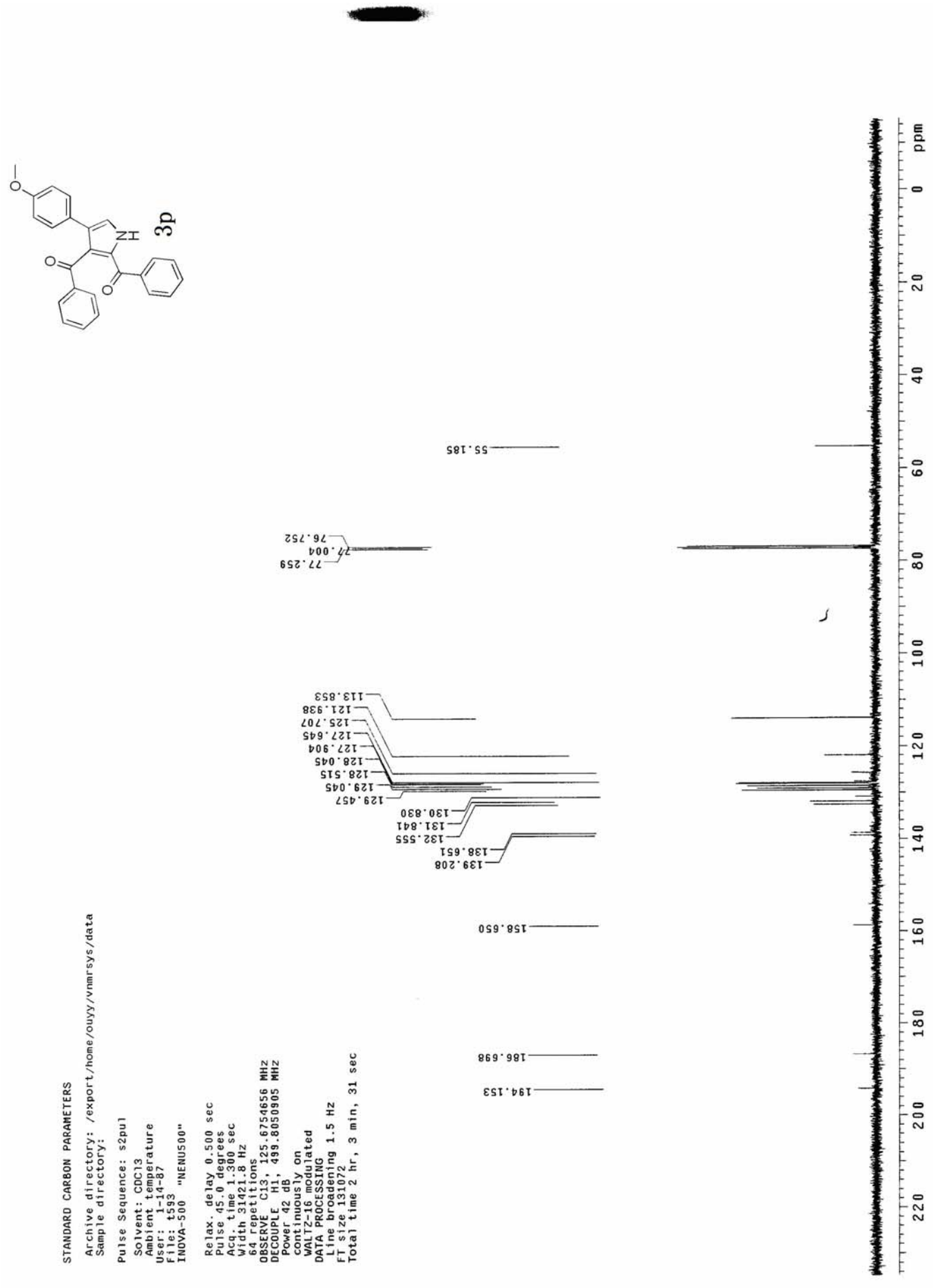

STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 1897 "NENUS00"
INOVA-500
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9313.2 Hz
8 repetitions
OBSERVE H1 499.8025904 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec

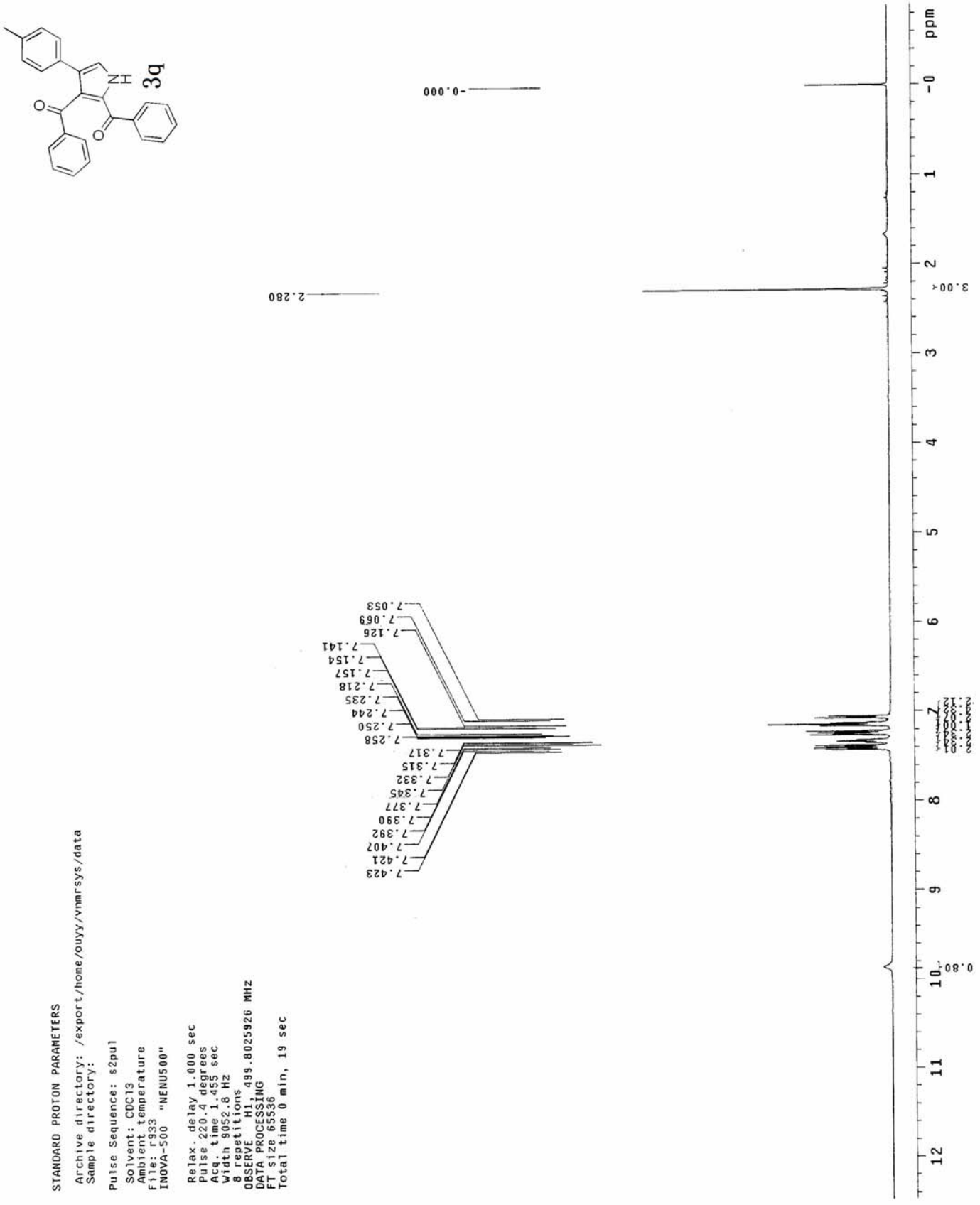

0.000
2.372
2.609
7.262
7.319
7.333
7.346
7.359
7.374
7.390
7.396
7.410
7.932
ppm
3.004
3.084
0.874
1.000
1.065
1.085

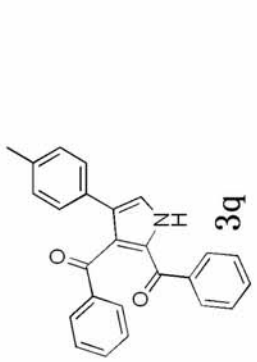

O
NH
O
3o

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: 1927
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, 125.6754684 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
22.120
28.724
76.733
76.989
77.240
119.050
124.868
127.515
128.026
128.999
129.079
133.306
167.470
194.050
ppm




3p
STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: t593 "NENUS00"
INOVA-500
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
64 repetitions
OBSERVE C13, 125.6754656 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
194.153
186.698
158.650
139.208
138.651
132.555
131.841
130.830
129.457
129.045
128.515
128.045
127.904
127.645
125.707
121.938
113.853
77.259
77.004
76.752
55.185
ppm



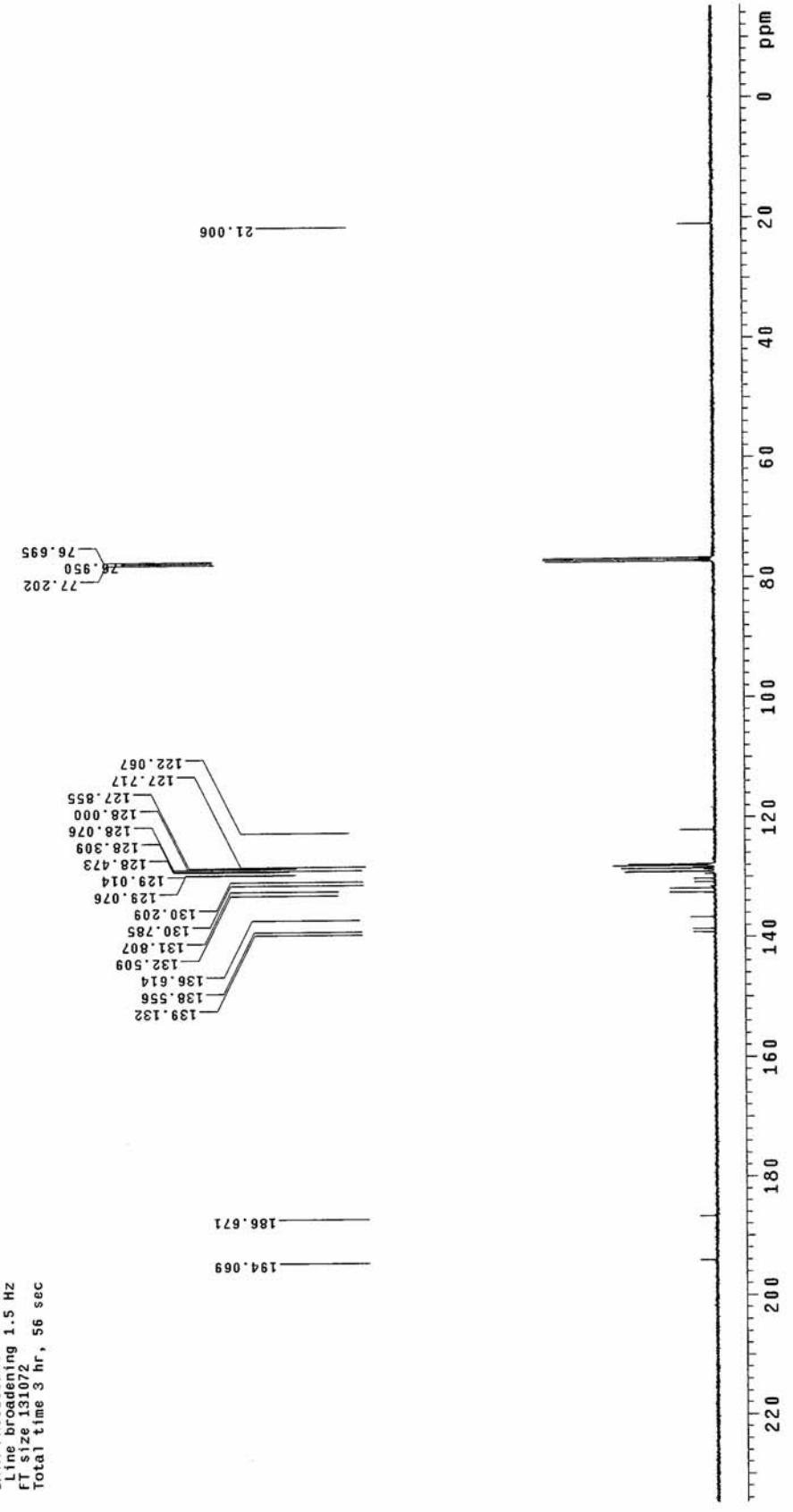


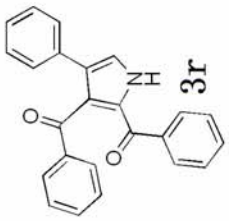
STANDARD CARBON PARAMETERS

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

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: q419 "NENU500"
INOVA-500 "NENU500"

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
448 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 3 hr, 56 sec





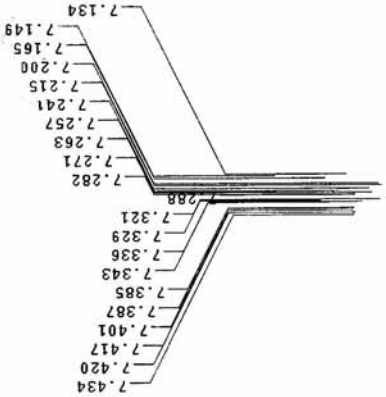
STANDARD PROTON PARAMETERS

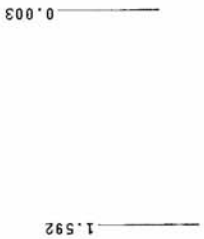
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:

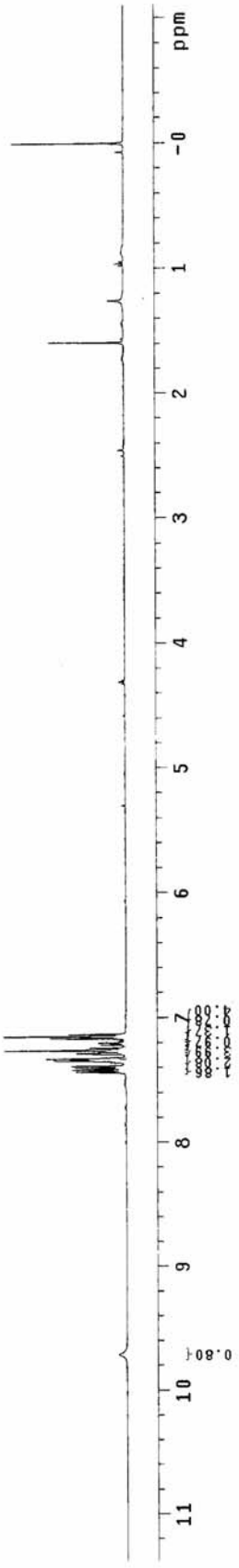
Pulse Sequence: s2pul

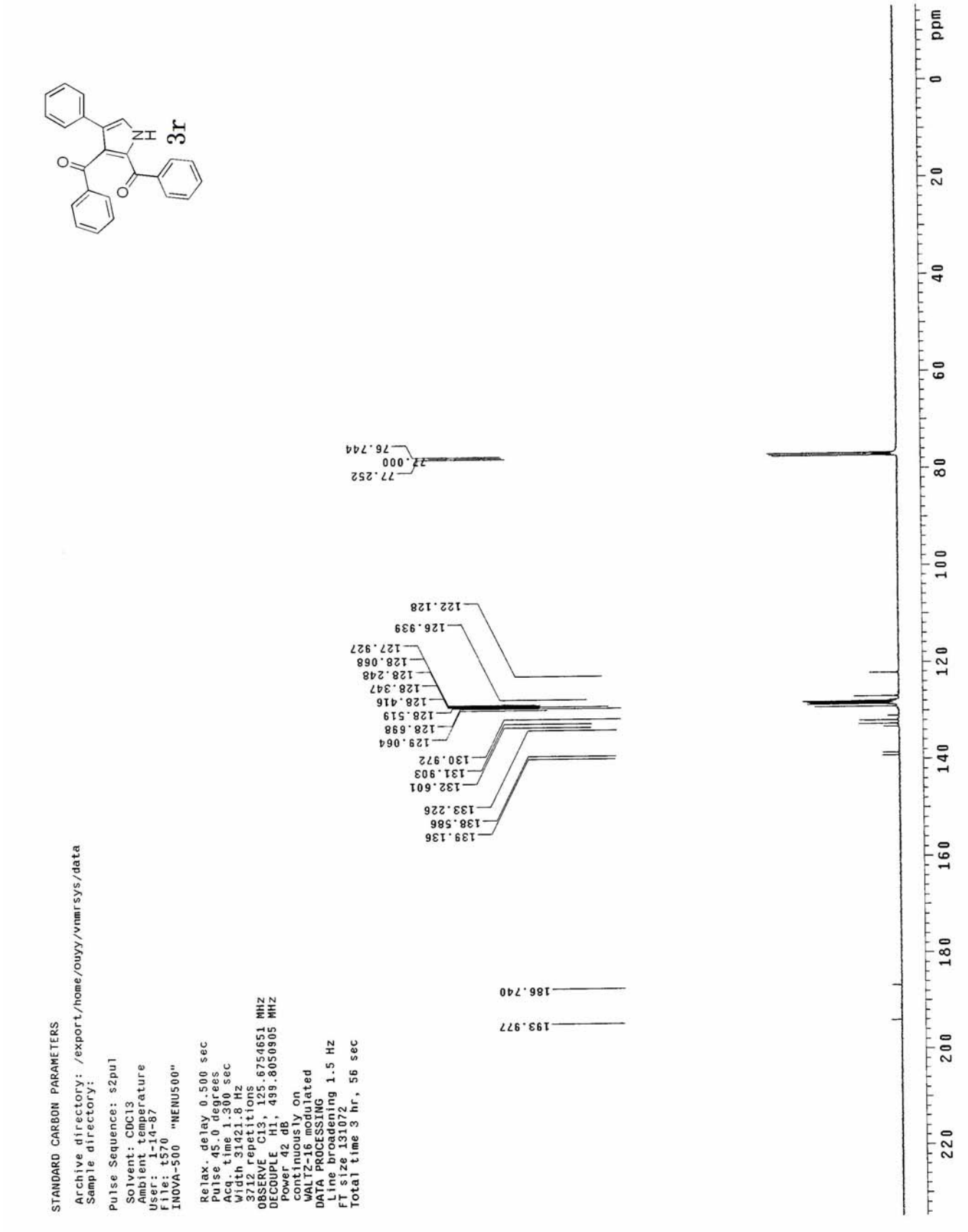
Solvent: CDCl3
Ambient temperature
File: r466
INOVA-500 "MENU500"

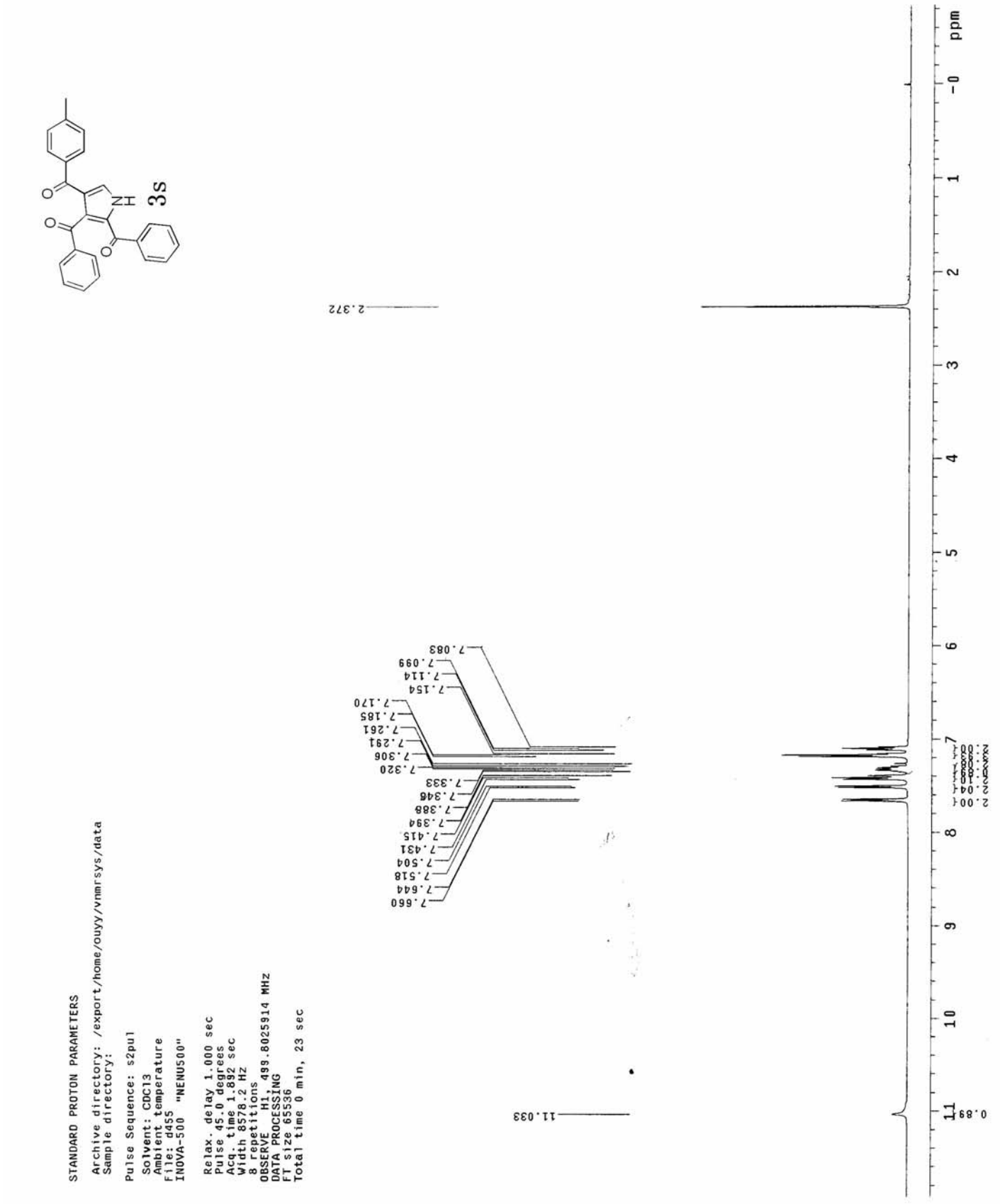
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8389.8 Hz
8 repetitions
OBSERVE H1, 499.8025906 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec

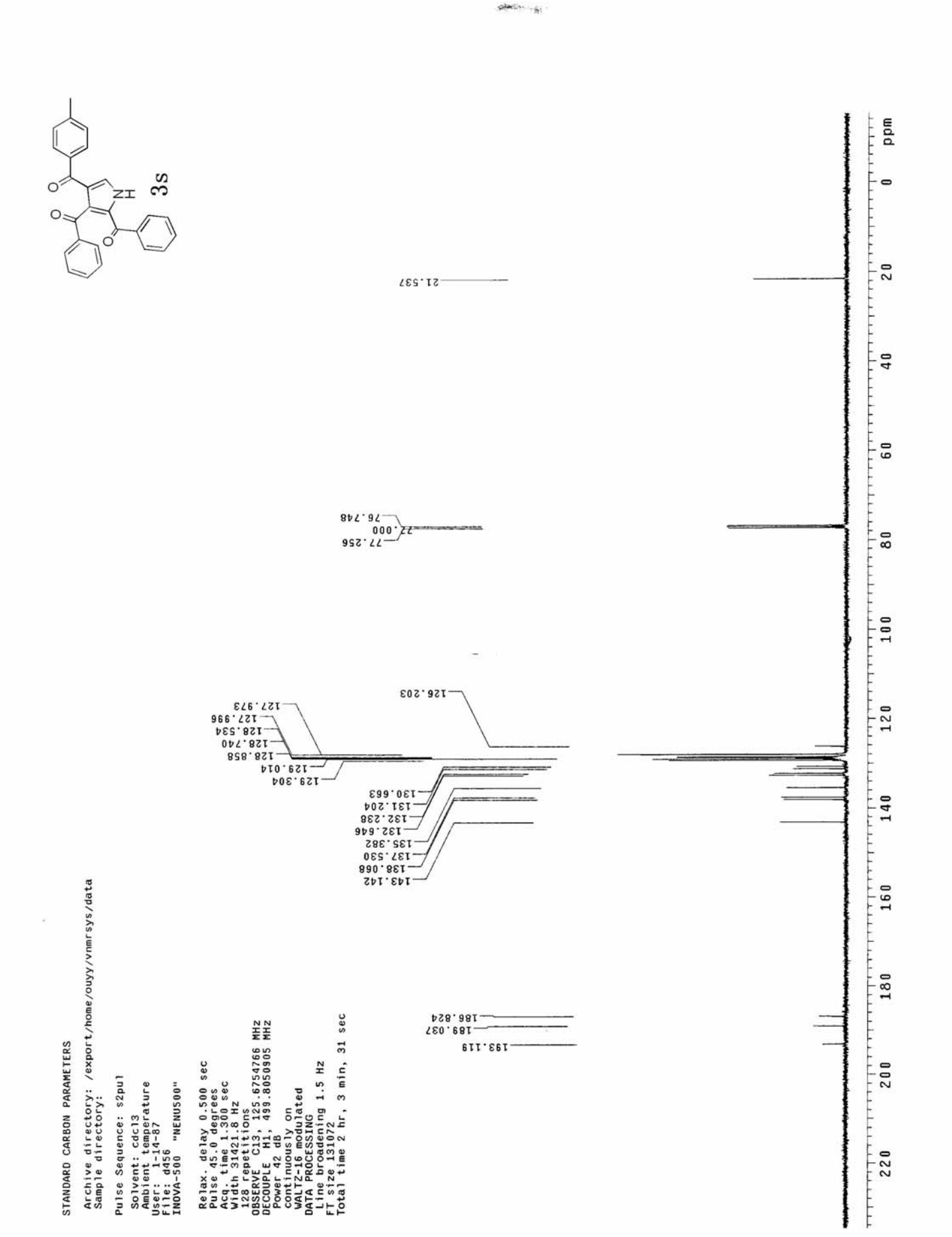


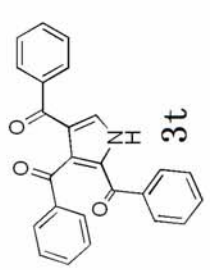





3r
STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: t570
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
3712 repetitions
OBSERVE C13, 125.6754651 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
193.977
186.740
139.136
138.586
133.226
132.601
131.903
130.972
129.064
128.698
128.519
128.416
128.347
128.248
128.068
127.927
126.939
122.128
77.252
77.000
76.744
ppm







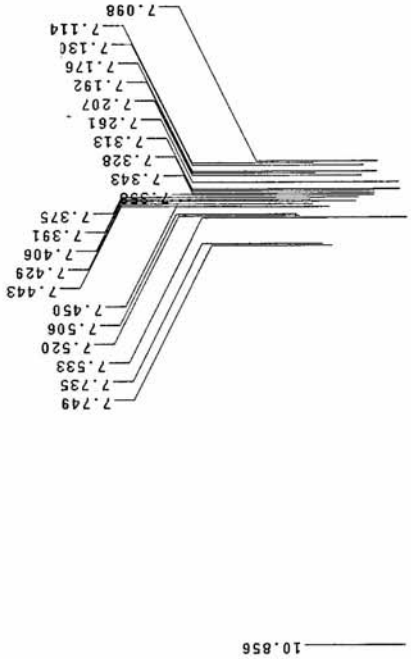
STANDARD PROTON PARAMETERS

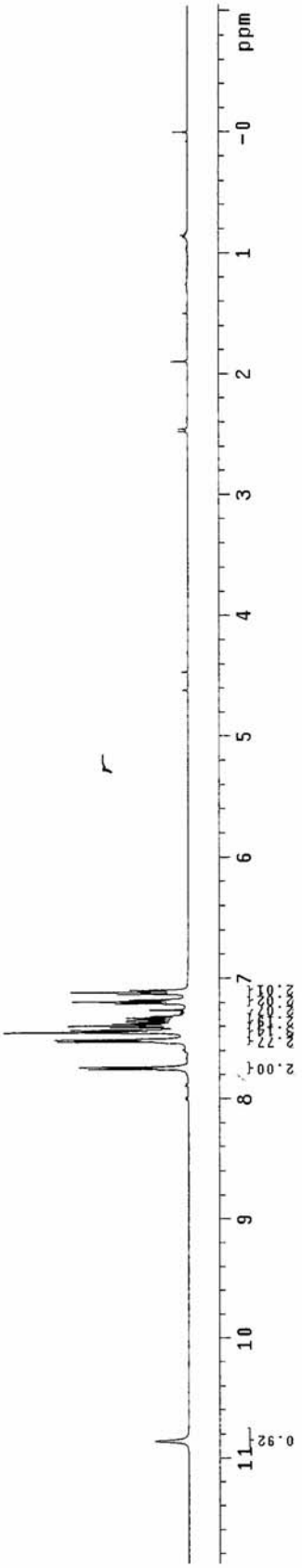
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:

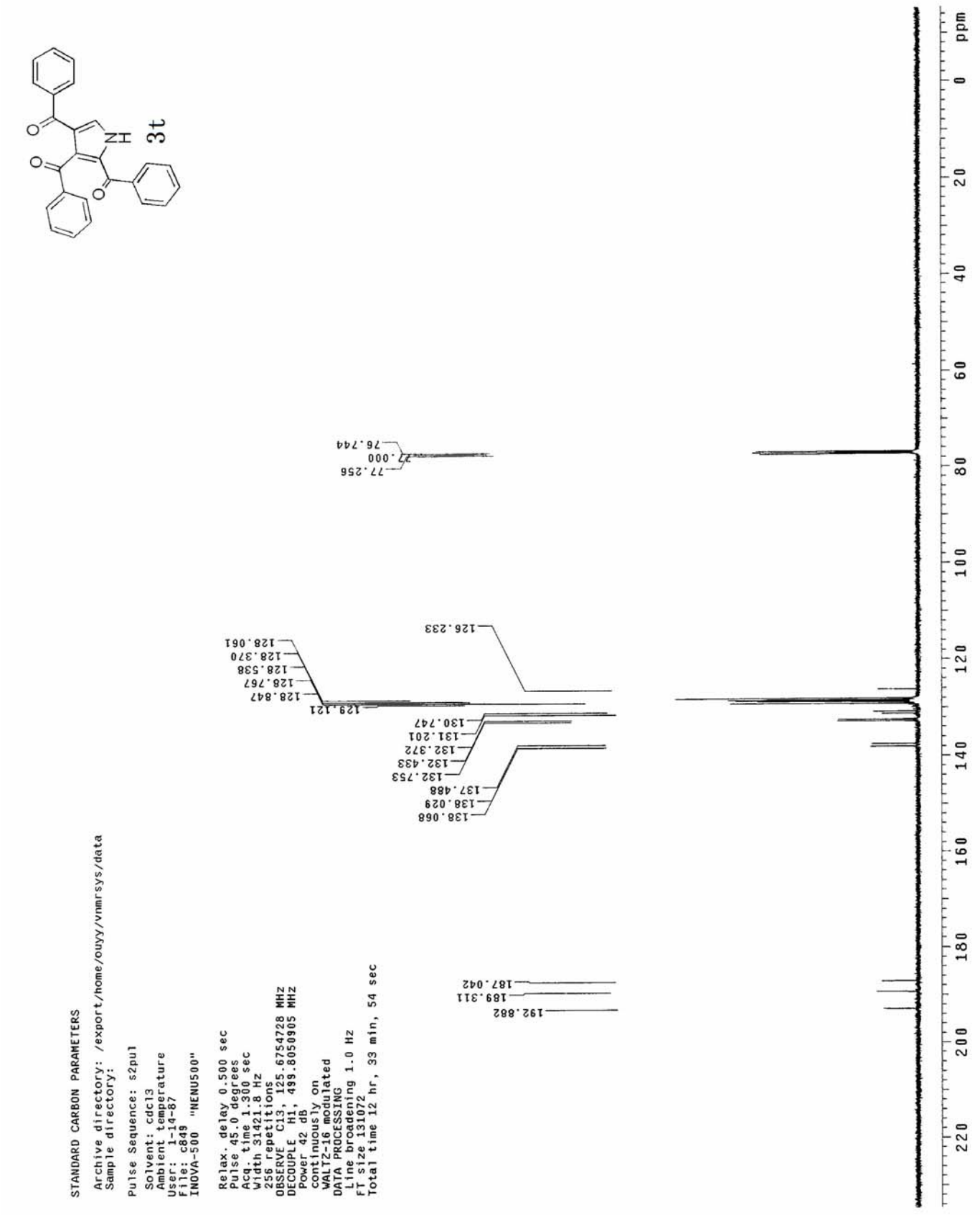
Pulse Sequence: s2pul

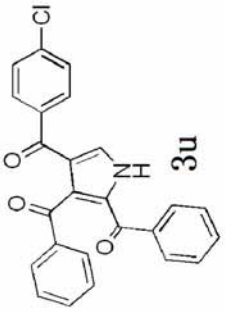
Solvent: CDCl3
Ambient temperature
File: c848 "NENU500"
INOVA-500 "NENU500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8578.2 Hz
8 repetitions
OBSERVE H1, 499.8025914 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec









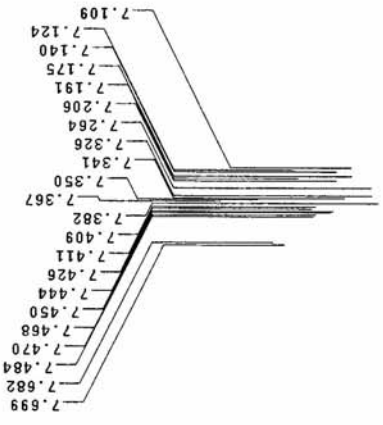
3u

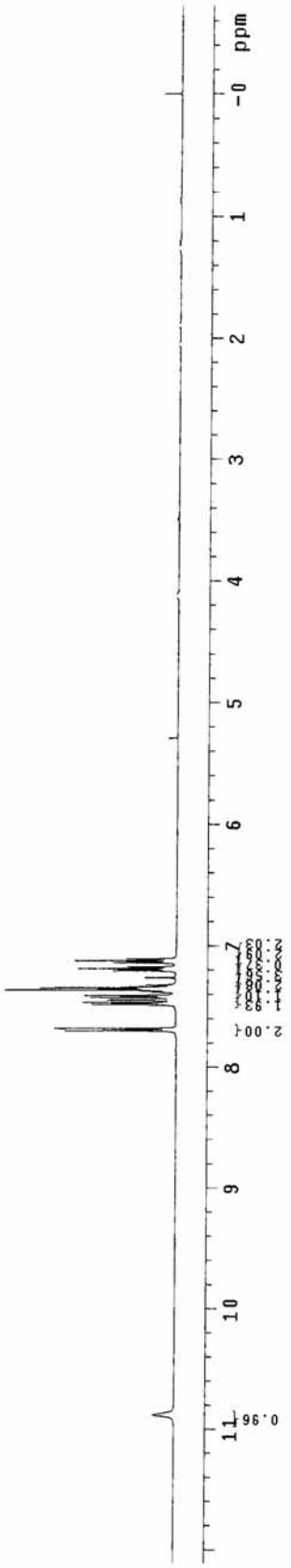
STANDARD PROTON PARAMETERS

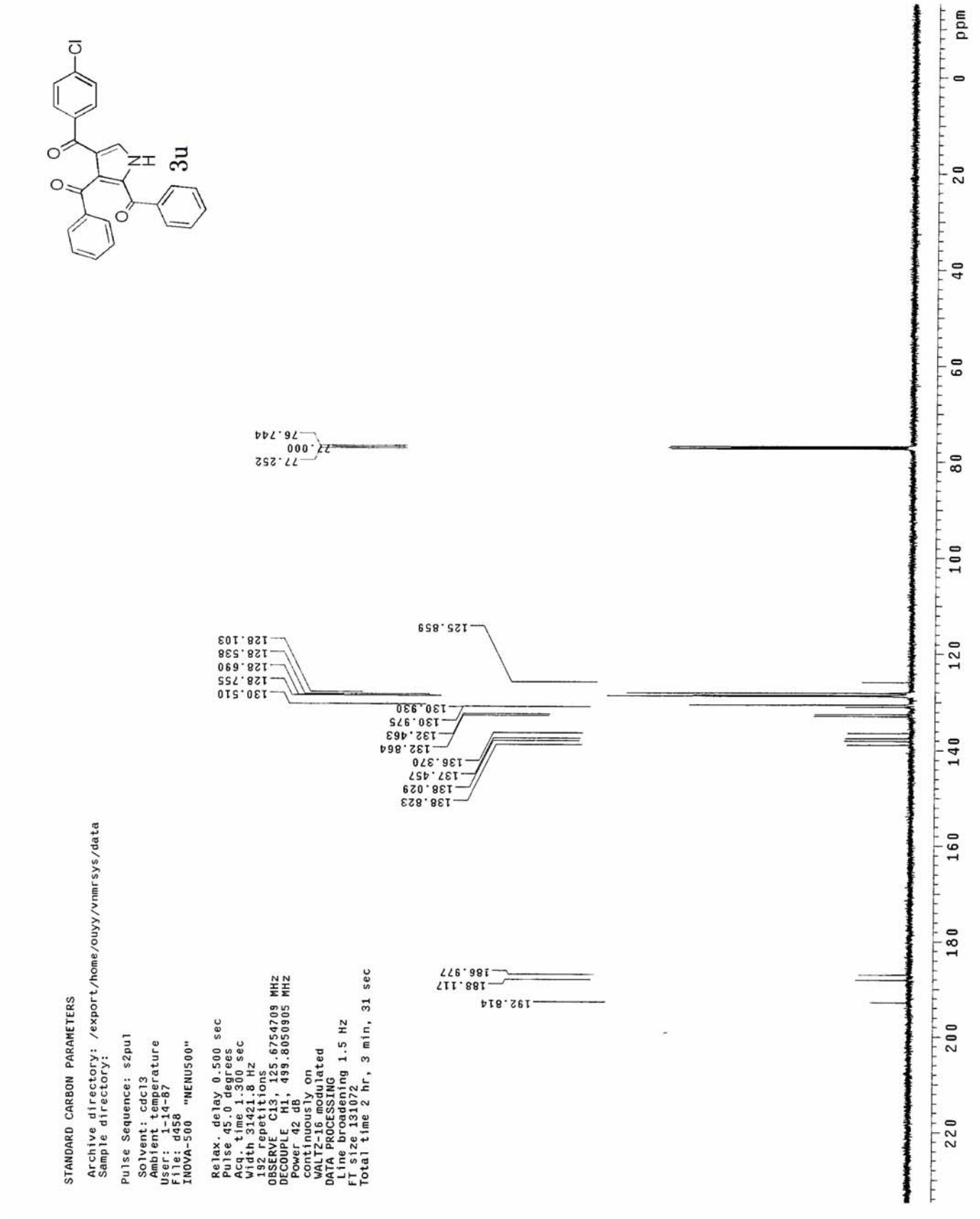
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:

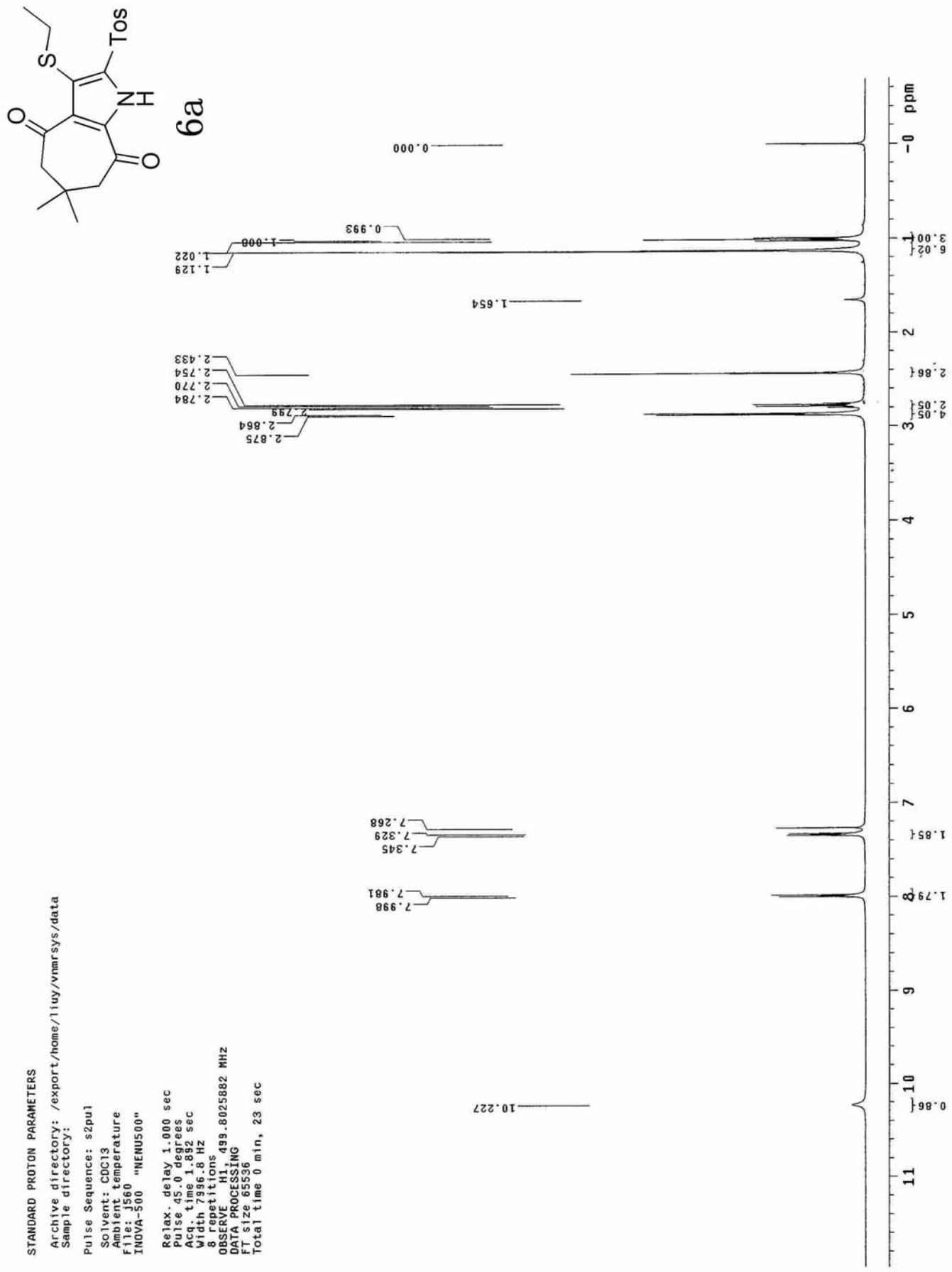
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: d457
INOVA-500 "NENU500"

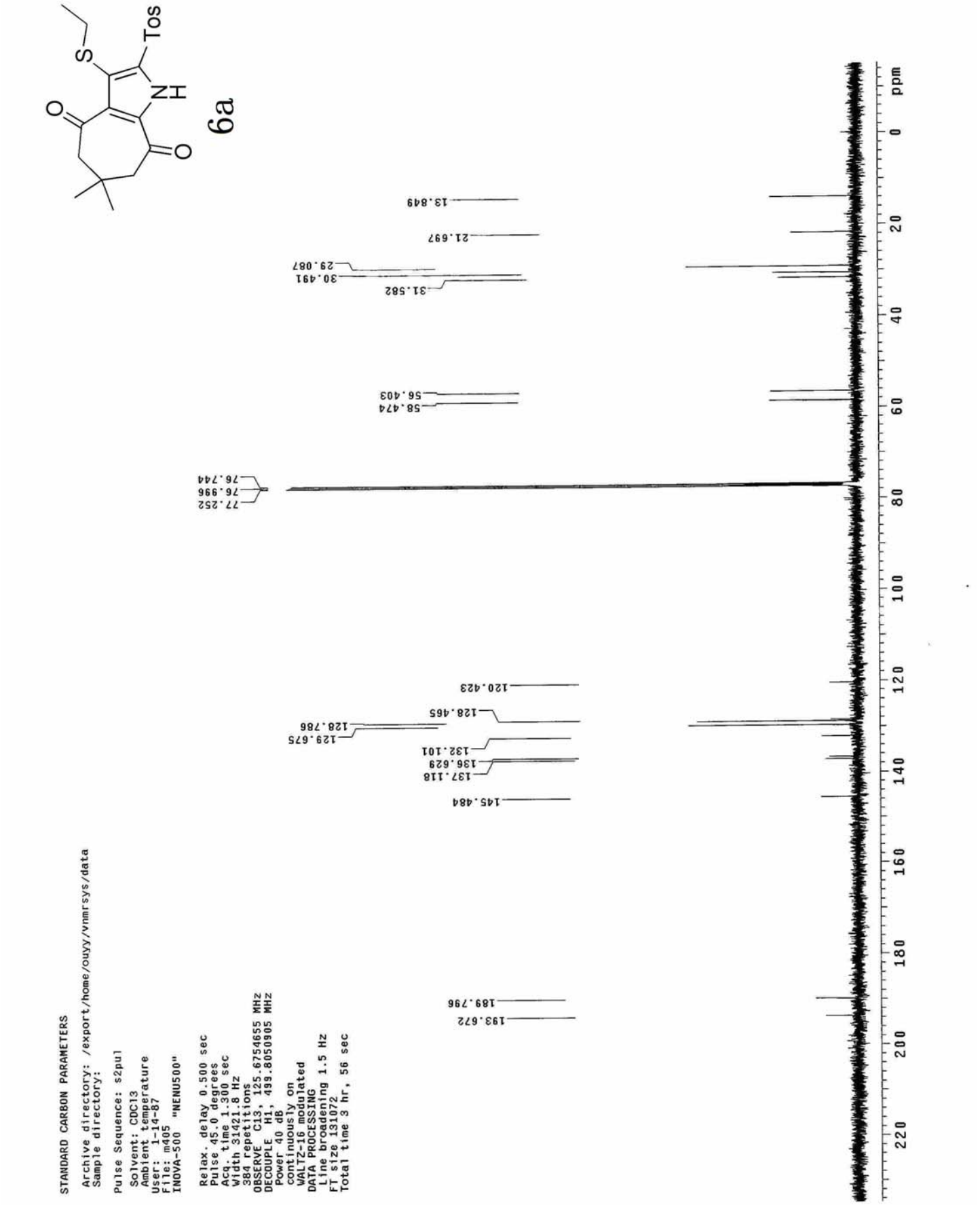
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8578.2 Hz
8 repetitions
OBSERVE H1, 499.8025901 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec

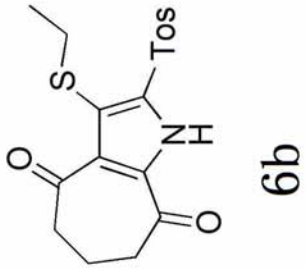




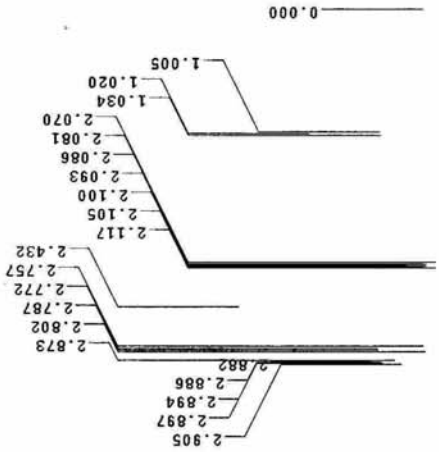


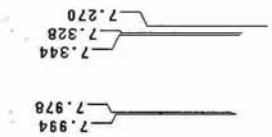


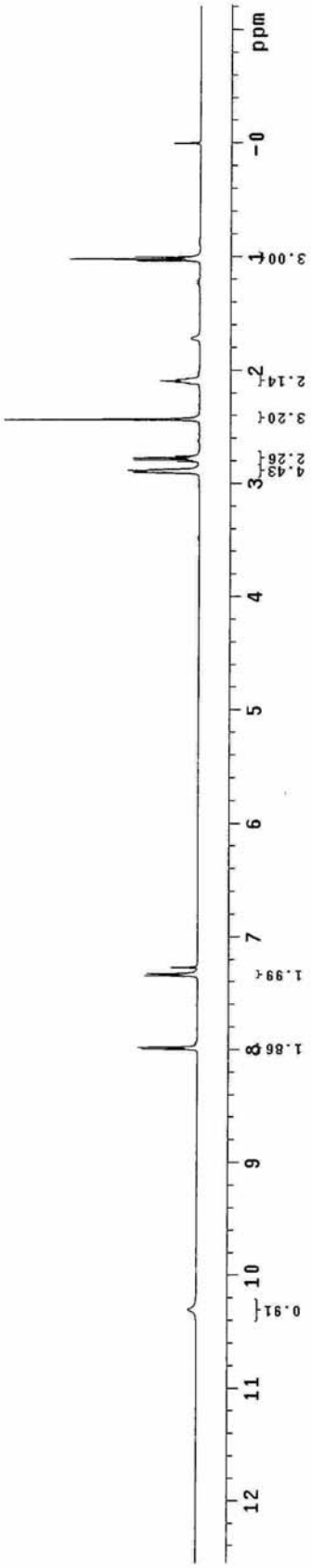

Tos
S
N
H
O
O
6a
13.849
21.697
29.087
30.491
31.582
56.403
58.474
76.744
76.996
77.252
120.423
128.465
128.786
129.675
132.101
136.629
137.118
145.484
189.796
193.672
ppm
STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: m405 "NENU500"
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
384 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 3 hr, 56 sec



6b





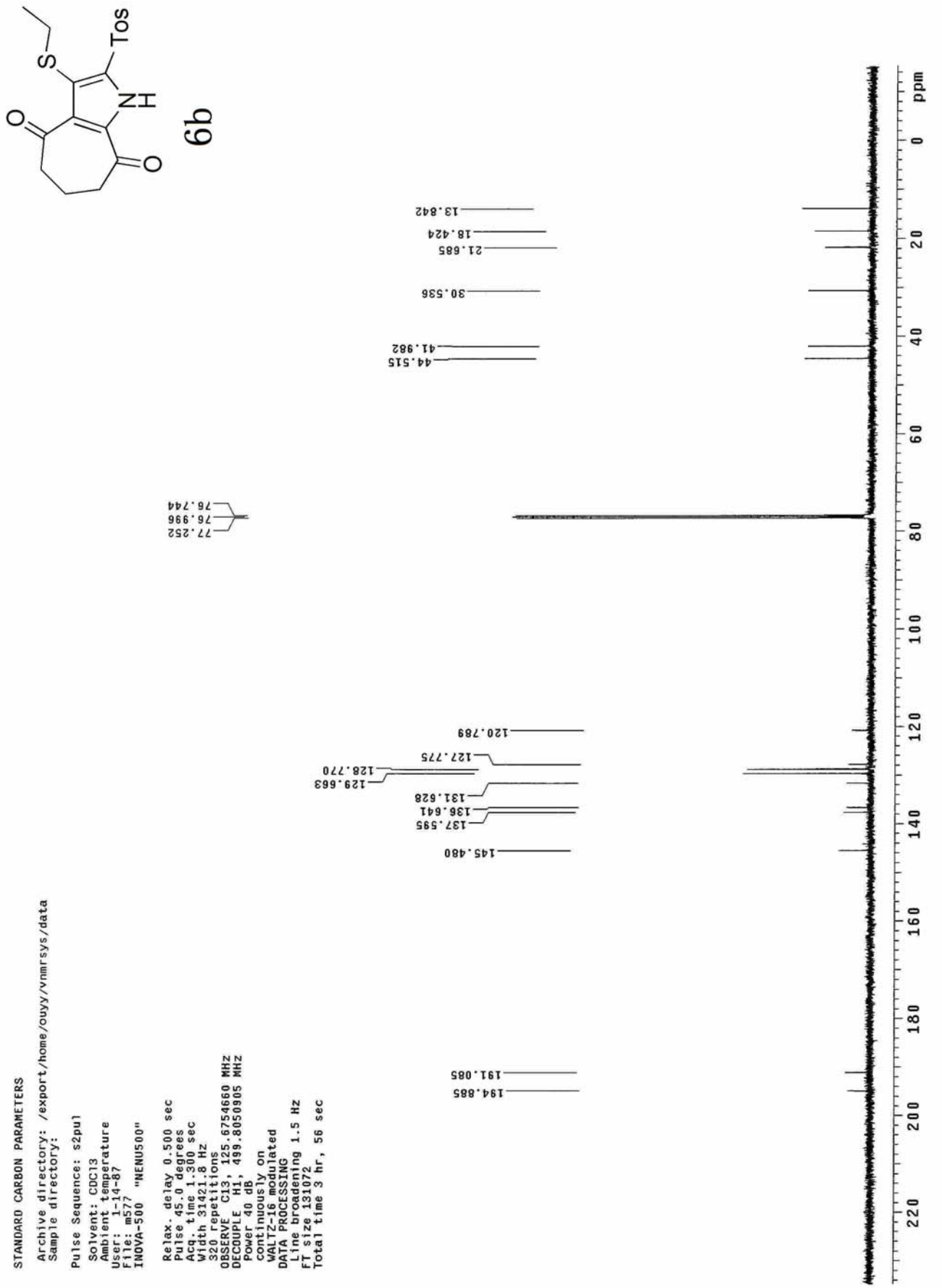


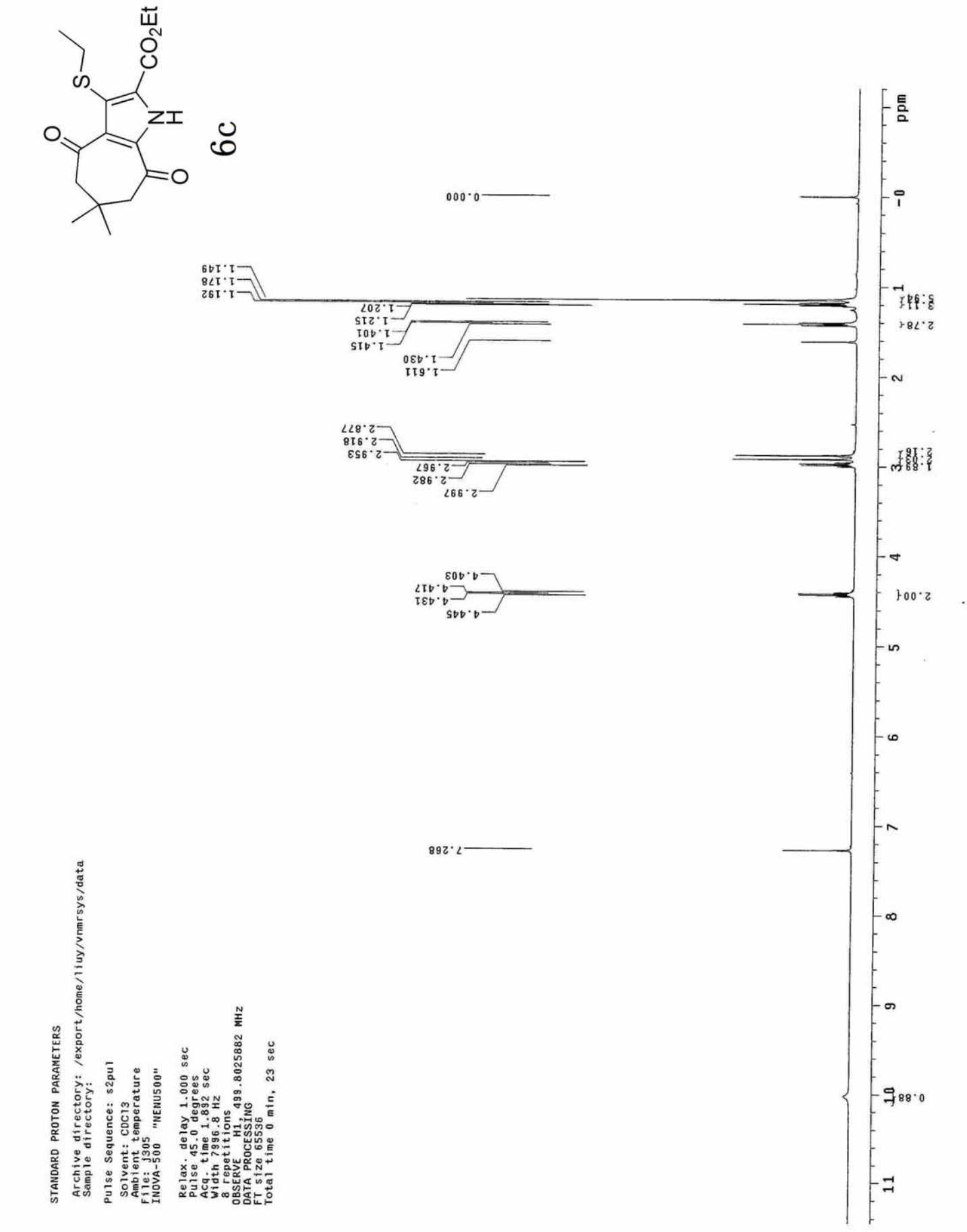
STANDARD PROTON PARAMETERS

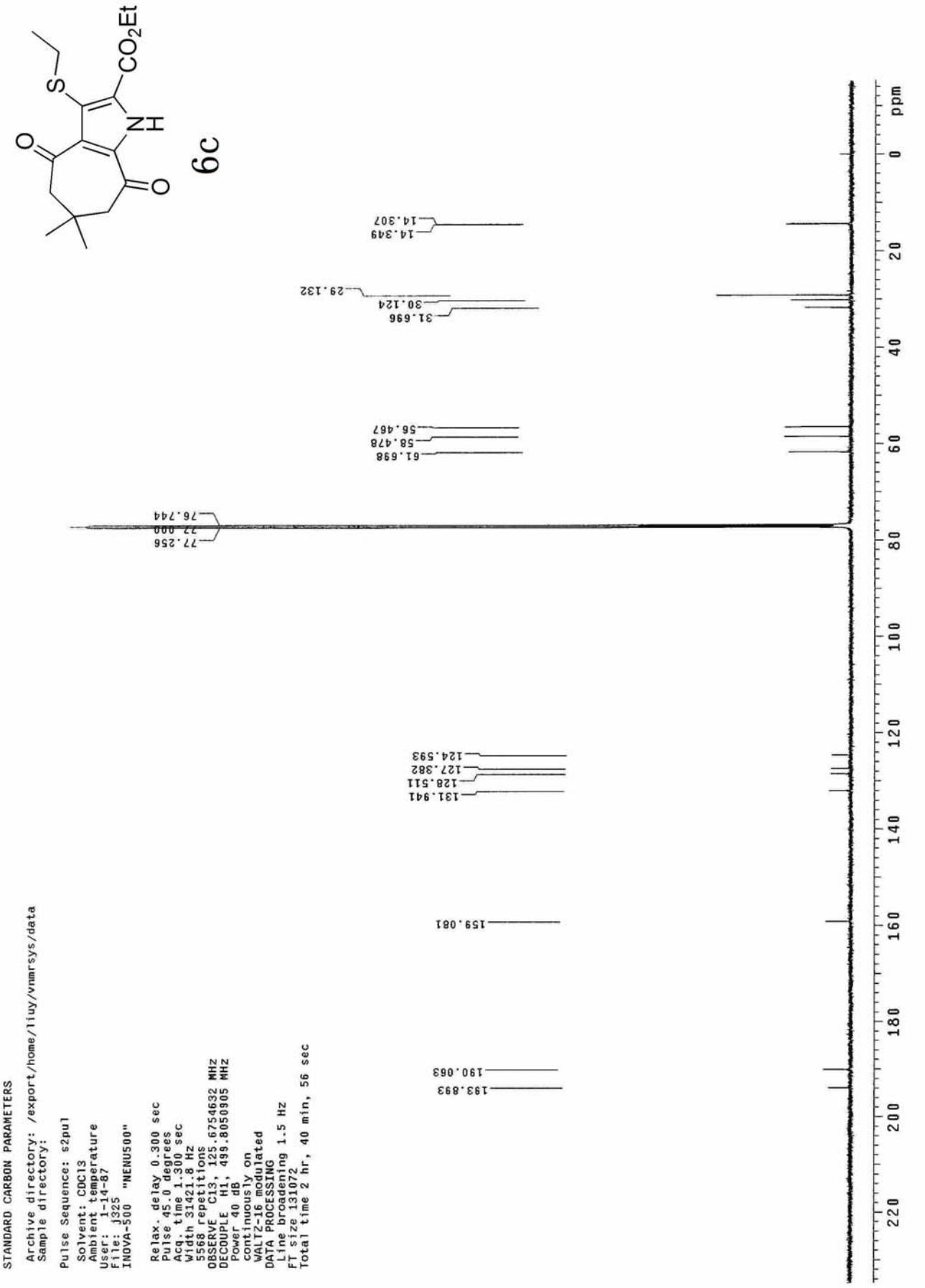
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:

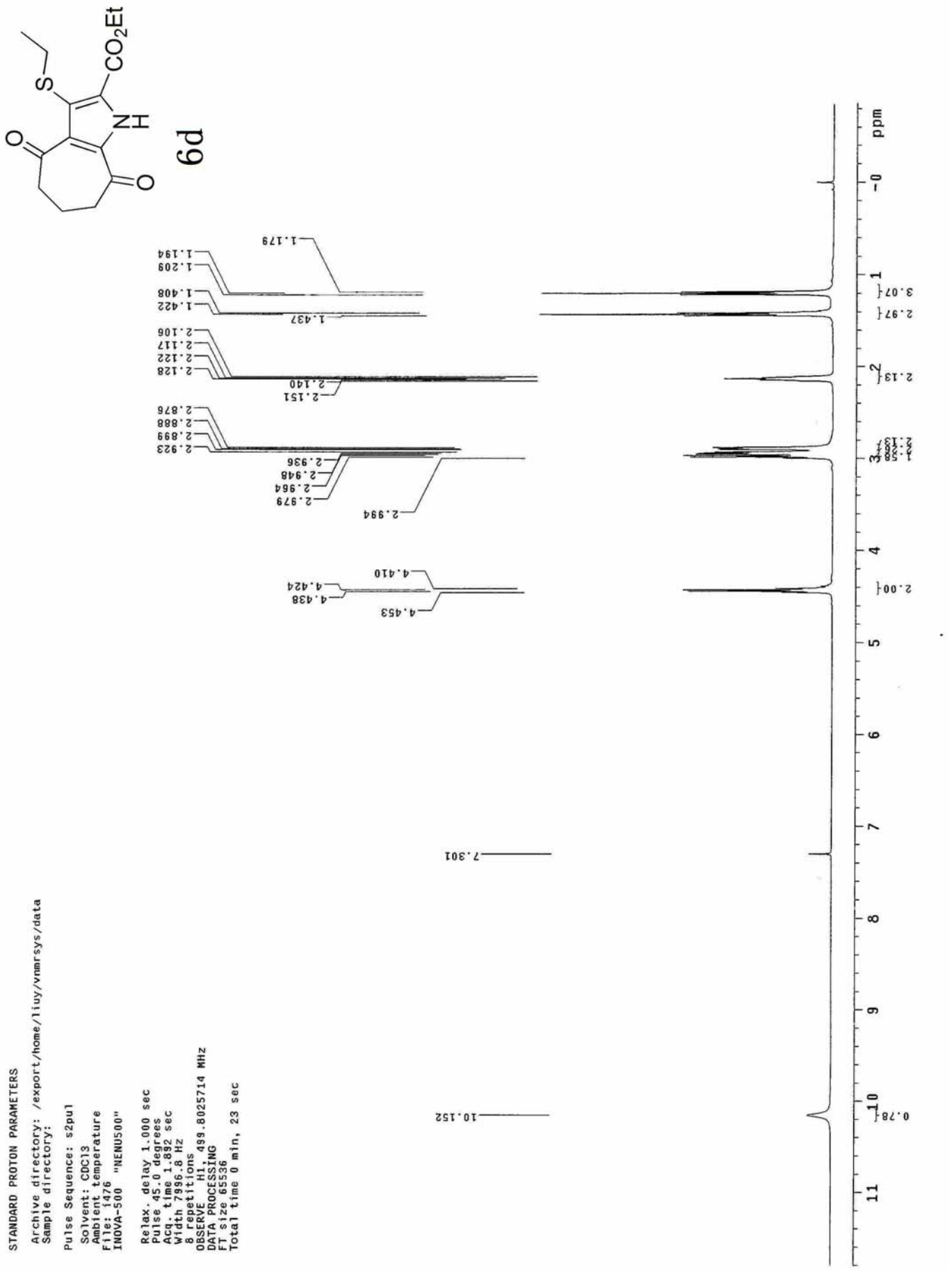
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: m578
INOVA-500 "NENU500"

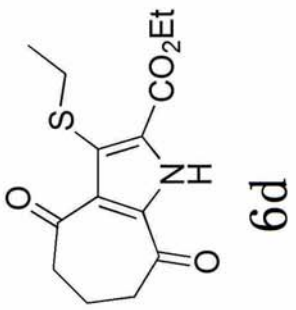
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9313.2 Hz
8 repetitions
OBSERVE H1, 499.8025870 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec




6c
CO2Et
S
N
H
O
O
STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: j305
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, 499.8025882 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
0.000
1.149
1.178
1.192
1.207
1.215
1.401
1.415
1.430
1.611
2.877
2.918
2.953
2.967
2.982
2.997
4.403
4.417
4.431
4.445
7.268
ppm

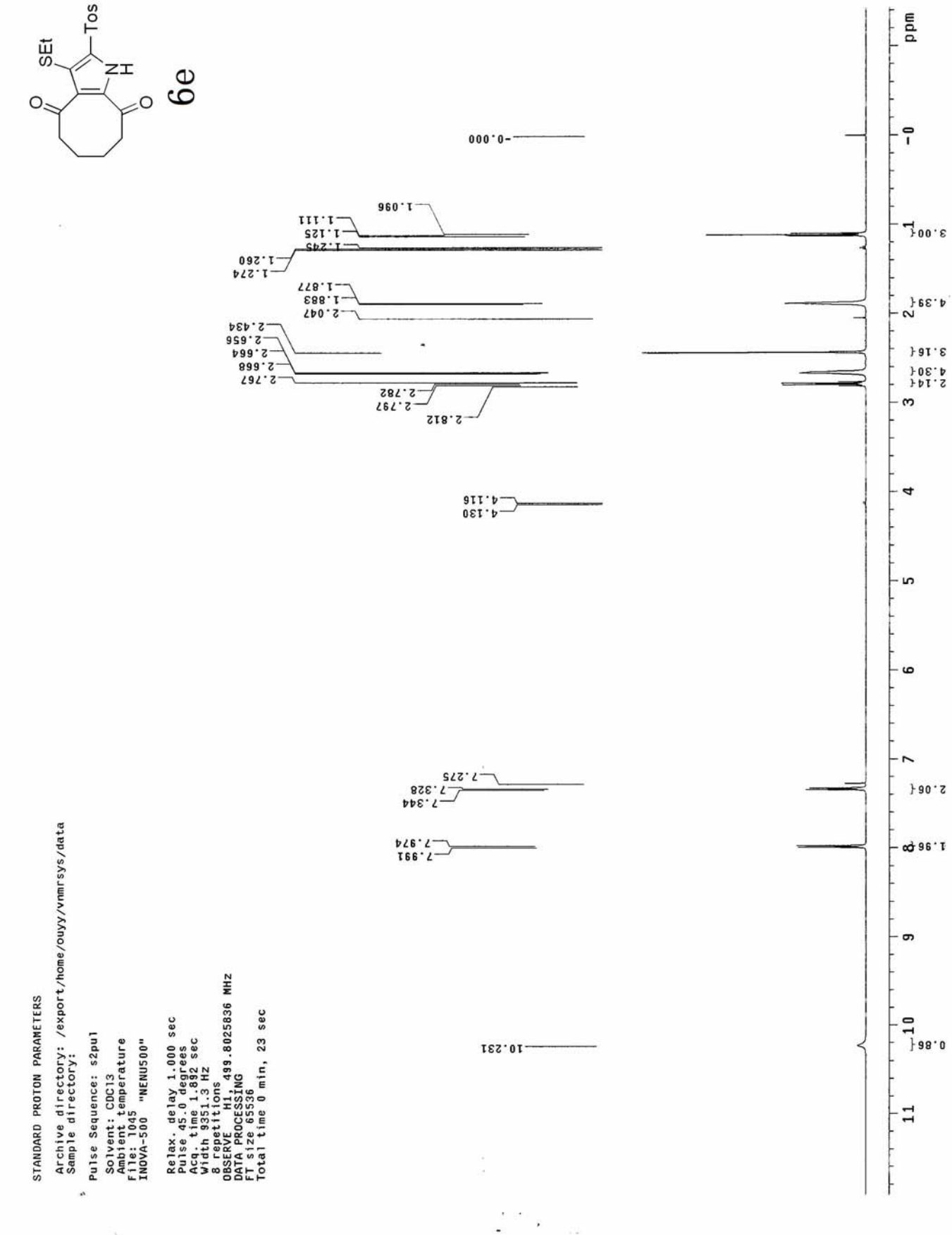

6c
CO2Et
S
N
H
O
O
14.307
14.349
29.132
30.124
31.696
56.467
58.478
61.698
76.744
77.000
77.256
124.593
127.382
128.511
131.941
159.081
190.063
193.893
ppm
0
20
40
60
80
100
120
140
160
180
200
220
STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: j325
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
5568 repetitions
OBSERVE C13, 125.6754632 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

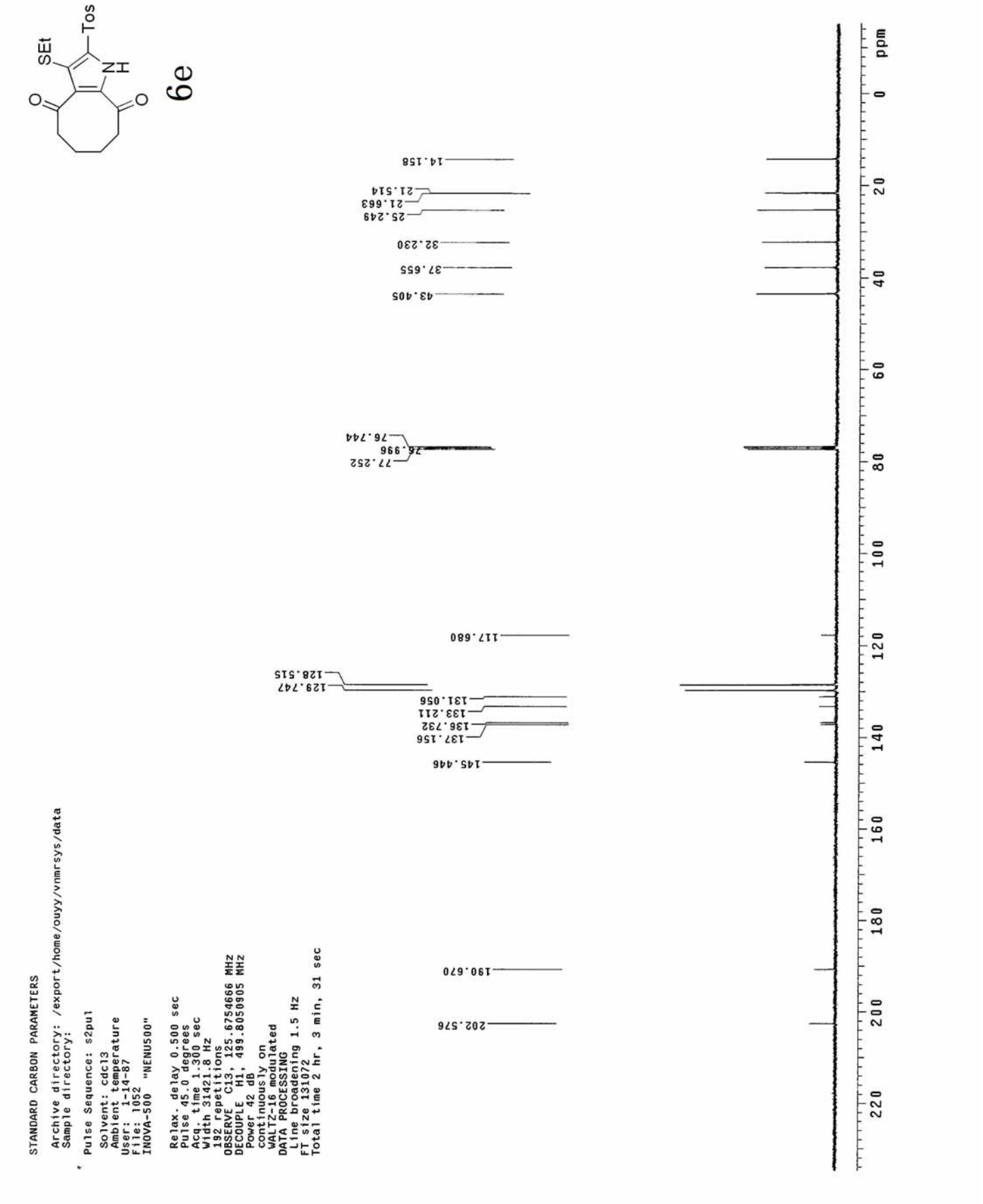


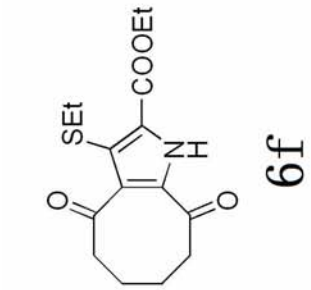

CO2Et
S
N
H
O
O

6d

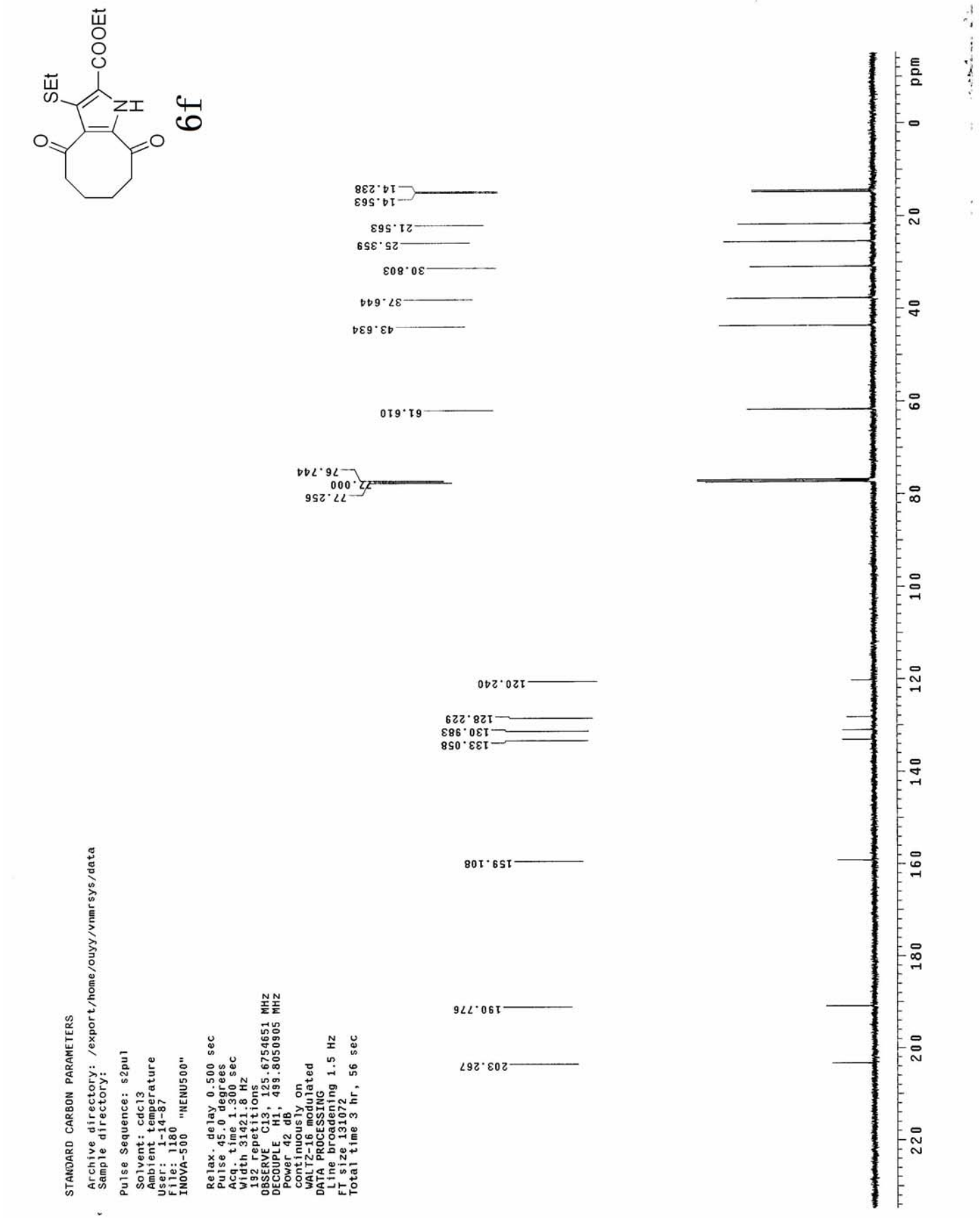
STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: i509 "NENU500"
INOVA-500
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
320 repetitions
OBSERVE C13, 125.6754733 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
14.208
14.246
18.565
30.063
41.821
44.305
61.629
76.744
77.000
77.256
124.723
127.756
127.824
131.391
158.993
191.398
195.259
220
200
180
160
140
120
100
80
60
40
20
0
ppm


SEt
Tos
NH
O
O
6e
0.000
1.096
1.111
1.125
1.245
1.260
1.274
1.877
1.883
2.047
2.434
2.656
2.664
2.668
2.767
2.782
2.797
2.812
4.116
4.130
7.275
7.328
7.344
7.974
7.991
10.231
ppm
STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 1045 "NENU500"
INOVA-500
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9351.3 Hz
8 repetitions
OBSERVE H1, 499.8025836 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec





6f


6f
SEt
COOEt
H
N
O
O
STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: l180 "NENU500"
INOVA-500
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
192 repetitions
OBSERVE C13, 125.6754651 MHz
DECOUPLE H1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
14.238
14.563
21.563
25.359
30.803
37.644
43.634
61.610
76.744
77.000
77.256
120.240
128.229
130.983
133.058
159.108
190.776
203.267
ppm