

Supporting Information

Copper(II)-catalyzed oxidative [3 + 2] cycloaddition reactions of secondary amines with α -diazo compounds: facile and efficient synthesis of 1,2,3-triazoles

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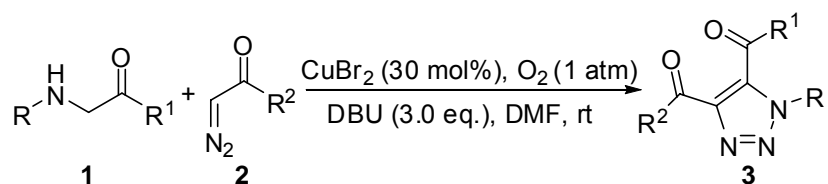
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I. General Information

All reactions were performed in dried glassware. Solvents (DMF) were commercial and were dried prior to use. The α -diazo compounds were prepared according to the previous method reported.¹ Unless otherwise noted, materials obtained from commercial suppliers were used without further purification. Chromatography was carried on flash silica gel (300-400 mesh). All reactions were monitored by TLC, which was performed on precoated aluminum sheets of silica gel 60 (F254). The ¹H NMR spectra were recorded at 500 MHz in CDCl₃ and the ¹³C NMR spectra were recorded at 125 MHz in CDCl₃ with TMS as internal standard. All coupling constants (J values) were reported in Hertz (Hz). High-resolution mass spectra (HRMS) were obtained using a Bruker microTOF II focus spectrometer (ESI). The compound **3a** was glued on a glass fiber. Data were collected at 293 K using graphite-monochromated Mo K α radiation ($\lambda = 0.71073\text{\AA}$) and IP technique in the range $2.19^\circ < \theta < 27.48^\circ$. Empirical absorption correction was applied. The structures were solved by the direct method and refined by the full-matrix least-squares method on F^2 using the SHELXS 97 crystallographic software package. Anisotropic thermal parameters were used to refine all non-hydrogen atoms. Hydrogen atoms were located from difference Fourier maps.

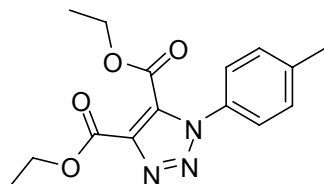
1. T. Toma, J. Shimokawa and T. Fukuyama, *Org. Lett.*, 2007, **9**, 3195.

II. General Procedure for the Preparation of 3 (3a as Example):



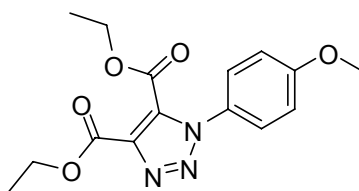
Glycine derivative **1a** (0.2 mmol, 38.6 mg), ethyl diazoacetate **2a** (1.0 mmol, 0.11 mL), DBU (0.6 mmol, 0.09 mL), CuBr₂ (0.06 mmol, 13.4 mg) and dry DMF (1.5 mL) were added to a 10 mL Schlenk tube equipped with a magnetic stir bar. Subsequently, the reaction mixture was stirred under an oxygen atmosphere (oxygen balloon) at room temperature for 10 h. After **1a** was consumed (monitored by TLC), the reaction mixture was poured into water (20.0 mL) and extracted with CH₂Cl₂ (3×10 mL). The combined organic extracts were dried over anhydrous MgSO₄, filtered and concentrated under reduced pressure to yield the corresponding crude product, which was purified by chromatography (silica gel, petroleum ether/ethyl acetate = 10/2, V/V) to give **3a** (51 mg, 84%) as a light yellow solid.

Diethyl 1-*p*-tolyl-1*H*-1,2,3-triazole-4,5-dicarboxylate (**3a**):



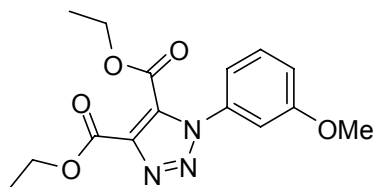
White solid; mp 53–55 °C; ¹H NMR (500 MHz, CDCl₃) δ: 1.27 (t, *J* = 7.0 Hz, 3H), 1.43 (t, *J* = 7.0 Hz, 3H), 2.45 (s, 3H), 4.37 (q, *J* = 7.0 Hz, 2H), 4.47 (q, *J* = 7.0 Hz, 2H), 7.33 (d, *J* = 7.5 Hz, 2H), 7.42 (d, *J* = 8.0 Hz, 2H); ¹³C NMR (125 MHz, CDCl₃) δ: 13.7, 14.2, 21.2, 61.8, 63.2, 124.1 (2C), 130.1 (2C), 132.7, 133.1, 138.7, 140.8, 159.1, 159.8; HRMS (ESI-TOF) Calcd for C₁₅H₁₈N₃O₄⁺ ([M + H]⁺): 304.1292. Found 304.1286.

Diethyl 1-(4-methoxyphenyl)-1*H*-1,2,3-triazole-4,5-dicarboxylate (**3b**):



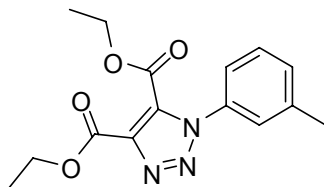
White solid; mp 43–45 °C; ^1H NMR (500 MHz, CDCl_3) δ : 1.27 (t, $J = 7.0$ Hz, 3H), 1.43 (t, $J = 7.0$ Hz, 3H), 3.88 (s, 3H), 4.36 (q, $J = 7.0$ Hz, 2H), 4.46 (q, $J = 7.0$ Hz, 2H), 7.02 (d, $J = 8.5$ Hz, 2H), 7.45 (d, $J = 8.5$ Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 13.8, 14.2, 55.6, 61.8, 63.2, 114.6 (2C), 125.9 (2C), 128.4, 132.8, 138.6, 159.1, 159.8, 161.0; HRMS (ESI-TOF) Calcd for $\text{C}_{15}\text{H}_{18}\text{N}_3\text{O}_5^+$ ($[\text{M} + \text{H}]^+$): 320.1241. Found 320.1242.

Diethyl 1-(3-methoxyphenyl)-1H-1,2,3-triazole-4,5-dicarboxylate (3c):



Yellow oil; ^1H NMR (500 MHz, CDCl_3) δ : 1.28 (t, $J = 7.0$ Hz, 3H), 1.43 (t, $J = 7.0$ Hz, 3H), 3.86 (s, 3H), 4.38 (q, $J = 7.0$ Hz, 2H), 4.47 (q, $J = 7.0$ Hz, 2H), 7.08 (s, 1H), 7.10 (d, $J = 8.5$ Hz, 2H), 7.43 (t, $J = 8.0$ Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ : 13.7, 14.1, 55.6, 61.8, 63.3, 109.9, 116.1, 116.3, 130.3, 132.7, 136.4, 138.6, 159.0, 159.6, 160.3; HRMS (ESI-TOF) Calcd for $\text{C}_{15}\text{H}_{18}\text{N}_3\text{O}_5^+$ ($[\text{M} + \text{H}]^+$): 320.1241. Found 320.1241..

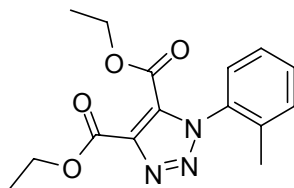
Diethyl 1-*m*-tolyl-1H-1,2,3-triazole-4,5-dicarboxylate (3d):



Yellow oil; ^1H NMR (500 MHz, CDCl_3) δ : 1.27 (t, $J = 7.0$ Hz, 3H), 1.43 (t, $J = 7.0$ Hz, 3H), 2.44 (s, 3H), 4.37 (q, $J = 7.0$ Hz, 2H), 4.47 (q, $J = 7.0$ Hz, 2H), 7.31 (d, $J = 7.5$ Hz, 1H), 7.35–7.37 (m, 2H), 7.41 (d, $J = 7.5$ Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ : 13.7, 14.2, 21.3, 61.8, 63.2, 121.2, 124.9, 129.3, 131.2, 132.7, 135.5, 138.7, 139.9, 159.1, 159.8; HRMS (ESI-TOF) Calcd for

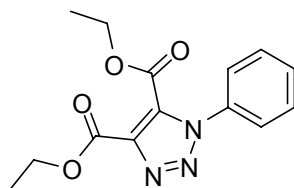
$C_{15}H_{18}N_3O_4^+$ ($[M + H]^+$): 304.1292. Found 304.1290.

Diethyl 1-*o*-tolyl-1*H*-1,2,3-triazole-4,5-dicarboxylate (3e):



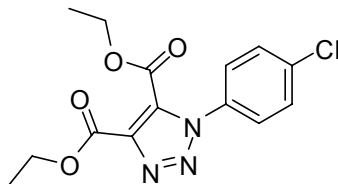
Yellow oil; 1H NMR (500 MHz, $CDCl_3$) δ : 1.14 (t, $J = 7.0$ Hz, 3H), 1.44 (t, $J = 7.0$ Hz, 3H), 2.11 (s, 3H), 4.26 (q, $J = 7.0$ Hz, 2H), 4.49 (q, $J = 7.0$ Hz, 2H), 7.27 (d, $J = 7.5$ Hz, 1H), 7.34 (t, $J = 7.5$ Hz, 1H), 7.39 (d, $J = 7.5$ Hz, 1H), 7.48 (t, $J = 7.5$ Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ : 13.6, 14.2, 17.2, 61.9, 62.9, 126.7, 126.8, 131.0, 131.2, 133.3, 134.7, 135.4, 138.7, 158.1, 159.9; HRMS (ESI-TOF) Calcd for $C_{15}H_{18}N_3O_4^+$ ($[M + H]^+$): 304.1292. Found 304.1286.

Diethyl 1-phenyl-1*H*-1,2,3-triazole-4,5-dicarboxylate (3f):



Yellow oil; 1H NMR (500 MHz, $CDCl_3$) δ : 1.26 (t, $J = 7.0$ Hz, 3H), 1.43 (t, $J = 7.0$ Hz, 3H), 4.37 (q, $J = 7.0$ Hz, 2H), 4.47 (q, $J = 7.0$ Hz, 2H), 7.54 (d, $J = 6.0$ Hz, 5H); ^{13}C NMR (125 MHz, $CDCl_3$) δ : 13.7, 14.2, 61.9, 63.3, 124.4 (2C), 129.6 (2C), 130.5, 132.7, 135.6, 138.8, 159.0, 159.8; HRMS (ESI-TOF) Calcd for $C_{14}H_{16}N_3O_4^+$ ($[M + H]^+$): 290.1135. Found 290.1130.

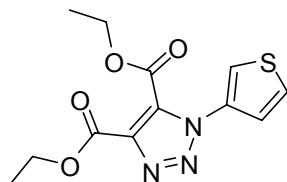
Diethyl 1-(4-chlorophenyl)-1*H*-1,2,3-triazole-4,5-dicarboxylate (3g):



The reaction was performed at 80 °C following the general procedure described above and **3g** was isolated as a white solid: mp 40–42 °C; 1H NMR (500 MHz, $CDCl_3$) δ : 1.30 (t, $J = 7.0$ Hz, 3H),

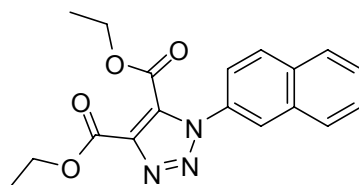
1.43 (t, $J = 7.0$ Hz, 3H), 4.38 (q, $J = 7.0$ Hz, 2H), 4.47 (q, $J = 7.0$ Hz, 2H), 7.52 (d, $J = 6.0$ Hz, 4H); ^{13}C NMR (125 MHz, CDCl_3) δ : 13.7, 14.1, 61.9, 63.4, 125.7, 129.8, 132.5, 134.0, 136.6, 139.1, 158.7, 159.6; HRMS (ESI-TOF) Calcd for $\text{C}_{14}\text{H}_{15}\text{ClN}_3\text{O}_4^+$ ($[\text{M} + \text{H}]^+$): 324.0746. Found 324.0726.

Diethyl 1-(thiophen-3-yl)-1*H*-1,2,3-triazole-4,5-dicarboxylate (3h):



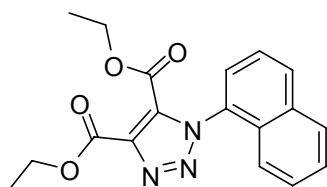
Red oil; ^1H NMR (500 MHz, CDCl_3) δ : 1.33 (t, $J = 7.0$ Hz, 3H), 1.42 (t, $J = 7.0$ Hz, 3H), 4.42 (q, $J = 7.0$ Hz, 2H), 4.46 (q, $J = 7.0$ Hz, 2H), 7.35 (dd, $J = 1.4$ Hz, 1H), 7.48 (dd, $J = 3.5$ Hz, 2H, 1H), 7.65 (dd, $J = 1.2$ Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ : 13.7, 14.1, 61.8, 63.4, 119.9, 123.1, 126.9, 132.4, 133.2, 138.4, 159.1, 159.6; Calcd for $\text{C}_{12}\text{H}_{14}\text{N}_3\text{O}_4\text{S}^+$ ($[\text{M} + \text{H}]^+$): 296.0700. Found 296.0688.

Diethyl 1-(naphthalen-2-yl)-1*H*-1,2,3-triazole-4,5-dicarboxylate (3i):



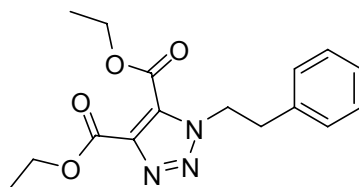
The reaction was performed at 60 °C following the general procedure described above and **3i** was isolated as a red oil. ^1H NMR (500 MHz, CDCl_3) δ : 1.25 (t, $J = 7.0$ Hz, 3H), 1.45 (t, $J = 7.0$ Hz, 3H), 4.36 (q, $J = 7.0$ Hz, 2H), 4.49 (q, $J = 7.0$ Hz, 2H), 7.61-7.63 (m, 3H), 7.94 (t, $J = 9.0$ Hz, 2H), 8.01 (d, $J = 8.5$ Hz, 1H), 8.05 (d, $J = 2.0$ Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ : 13.7, 14.2, 61.9, 63.3, 121.5, 123.4, 127.6, 127.8, 127.9, 128.4 (2C), 129.8, 132.7, 132.9, 133.5, 138.8, 159.1, 159.8; HRMS (ESI-TOF) Calcd for $\text{C}_{18}\text{H}_{18}\text{N}_3\text{O}_4^+$ ($[\text{M} + \text{H}]^+$): 340.1292. Found 340.1278.

Diethyl 1-(naphthalen-1-yl)-1*H*-1,2,3-triazole-4,5-dicarboxylate (3j):



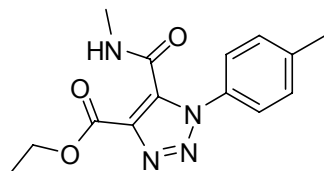
The reaction was performed at 60 °C following the general procedure described as above and **3j** was isolated as a red oil. ¹H NMR (500 MHz, CDCl₃) δ: 0.91 (t, *J* = 7.0 Hz, 3H), 1.46 (t, *J* = 7.0 Hz, 3H), 4.11 (q, *J* = 7.0 Hz, 2H), 4.52 (q, *J* = 7.0 Hz, 2H), 7.28 (d, *J* = 8.5 Hz, 1H), 7.52-7.61 (m, 4H), 7.97 (d, *J* = 7.5 Hz, 1H), 8.08 (d, *J* = 7.5 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 13.4, 14.2, 62.0, 62.8, 121.7, 124.7, 124.9, 127.2, 128.1, 128.2, 129.2, 131.4, 131.9, 133.9, 134.3, 138.8, 158.0, 159.9; HRMS (ESI-TOF) Calcd for C₁₈H₁₈N₃O₄⁺ ([M + H]⁺): 340.1292. Found 340.1282.

Diethyl 1-phenethyl-1H-1,2,3-triazole-4,5-dicarboxylate (3k):



Yellow oil; ¹H NMR (500 MHz, CDCl₃) δ: 1.36 (t, *J* = 7.0 Hz, 3H), 1.41 (t, *J* = 7.0 Hz, 3H), 3.20 (t, *J* = 7.5 Hz, 2H), 4.33 (q, *J* = 7.0 Hz, 2H), 4.42 (q, *J* = 7.0 Hz, 2H), 4.84 (t, *J* = 7.5 Hz, 2H), 7.11 (d, *J* = 7.0 Hz, 2H), 7.24-7.31 (m, 3H); ¹³C NMR (125 MHz, CDCl₃) δ: 13.8, 14.1, 36.7, 51.4, 61.7, 62.7, 127.1, 128.6, 128.7 (4C), 136.3 (2C), 158.4, 160.1; HRMS (ESI-TOF) Calcd for C₁₆H₂₀N₃O₄⁺ ([M + H]⁺): 318.1448. Found 318.1433.

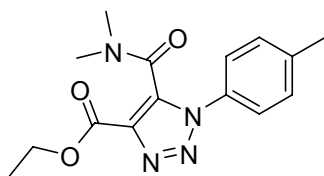
Ethyl 5-(methylcarbamoyl)-1-*p*-tolyl-1H-1,2,3-triazole-4-carboxylate (3l):



White solid; mp 103–105 °C; ¹H NMR (500 MHz, CDCl₃) δ: 1.47 (t, *J* = 7.0 Hz, 3H), 2.43 (s, 3H), 2.94 (d, *J* = 4.5 Hz, 3H), 4.50 (q, *J* = 7.0 Hz, 2H), 7.31 (t, *J* = 9.0 Hz, 4H), 9.22 (br s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 14.0, 21.2, 26.3, 62.7, 125.3 (2C), 129.5 (2C), 134.4, 134.5, 136.2,

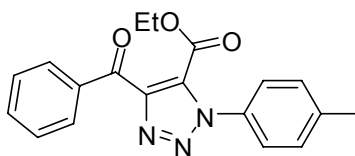
140.2, 156.6, 162.9; HRMS (ESI-TOF) Calcd for $C_{14}H_{17}N_4O_3^+$ ($[M + H]^+$): 289.1295. Found 289.1302.

Ethyl 5-(dimethylcarbamoyl)-1-*p*-tolyl-1*H*-1,2,3-triazole-4-carboxylate (3m):



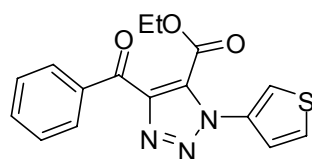
White solid; mp 177–179 °C; 1H NMR (500 MHz, $CDCl_3$) δ : 1.42 (t, $J = 7.0$ Hz, 3H), 2.43 (s, 3H), 2.78 (s, 3H), 3.09 (s, 3H), 4.43 (q, $J = 7.0$ Hz, 2H), 7.32 (d, $J = 8.0$ Hz, 2H), 7.50 (d, $J = 8.5$ Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ : 14.2, 21.2, 39.4, 37.6, 61.6, 123.2 (2C), 130.3 (2C), 133.1, 135.4, 136.9, 140.6, 159.5, 160.0; HRMS (ESI-TOF) Calcd for $C_{15}H_{19}N_4O_3^+$ ($[M + H]^+$): 303.1452. Found 303.1458.

Ethyl 4-benzoyl-1-*p*-tolyl-1*H*-1,2,3-triazole-5-carboxylate (3n):



Yellow solid; mp 93–95 °C; 1H NMR (500 MHz, $CDCl_3$) δ : 1.20 (t, $J = 7.0$ Hz, 3H), 2.46 (s, 3H), 4.31 (q, $J = 7.0$ Hz, 2H), 7.35 (d, $J = 8.0$ Hz, 2H), 7.47 (d, $J = 8.0$ Hz, 2H), 7.54 (t, $J = 7.5$ Hz, 2H), 7.65 (t, $J = 8.0$ Hz, 1H), 8.31 (d, $J = 8.0$ Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ : 13.6, 21.3, 63.0, 124.4 (2C), 128.5 (2C), 130.0 (2C), 130.5 (2C), 132.5, 133.2, 133.7, 136.1, 140.8, 146.5, 159.0, 185.5; HRMS (ESI-TOF) Calcd for $C_{19}H_{18}N_3O_3^+$ ($[M + H]^+$): 336.1343. Found 336.1348.

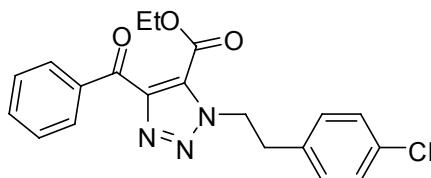
Ethyl 4-benzoyl-1-(thiophen-3-yl)-1*H*-1,2,3-triazole-5-carboxylate (3o):



Red oil; 1H NMR (500 MHz, $CDCl_3$) δ : 1.25 (t, $J = 7.0$ Hz, 3H), 4.37 (q, $J = 7.0$ Hz, 2H), 7.41 (dd,

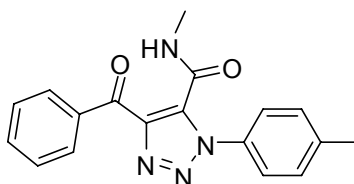
$J = 1.5, 3.5$ Hz, 1H), 7.48-7.55 (m, 3H), 7.64 (t, $J = 7.5$ Hz, 1H), 7.72 (d, $J = 2.0$ Hz, 1H), 8.31 (d, $J = 7.5$ Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 13.6, 63.2, 120.2, 123.4, 126.7, 128.5, 130.5 (2C), 132.3 (2C), 133.3, 133.7, 136.0, 146.3, 159.1, 185.2; HRMS (ESI-TOF) Calcd for $\text{C}_{16}\text{H}_{14}\text{N}_3\text{O}_3\text{S}^+$ ($[\text{M} + \text{H}]^+$): 328.0750. Found 328.0759.

Ethyl 4-benzoyl-1-(4-chlorophenethyl)-1H-1,2,3-triazole-5-carboxylate (3p):



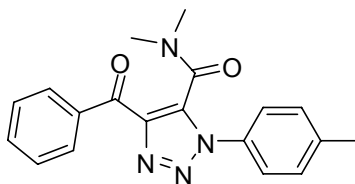
Yellow oil; ^1H NMR (500 MHz, CDCl_3) δ : 1.08 (t, $J = 7.0$ Hz, 3H), 3.25 (t, $J = 7.0$ Hz, 2H), 4.15 (q, $J = 7.0$ Hz, 2H), 4.94 (t, $J = 7.5$ Hz, 2H), 7.09 (d, $J = 8.0$ Hz, 2H), 7.27 (d, $J = 8.0$ Hz, 2H), 7.50 (t, $J = 7.5$ Hz, 2H), 7.62 (t, $J = 7.5$ Hz, 1H), 7.96 (d, $J = 7.5$ Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 13.3, 36.1, 51.1, 62.5, 128.5 (2C), 128.7 (2C), 128.9, 130.1 (2C), 130.3 (2C), 133.1, 133.8, 134.9, 136.5, 147.2, 158.2, 186.8; HRMS (ESI-TOF) Calcd for $\text{C}_{20}\text{H}_{19}\text{ClN}_3\text{O}_3^+$ ($[\text{M} + \text{H}]^+$): 384.1109. Found 384.1101.

4-Benzoyl-N-methyl-1-p-tolyl-1H-1,2,3-triazole-5-carboxamide (3q):



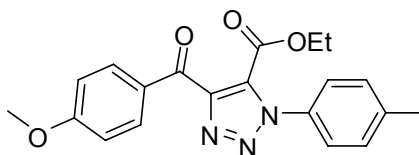
White solid; mp 174–176 °C; ^1H NMR (500 MHz, CDCl_3) δ : 2.45 (s, 3H), 2.96 (t, $J = 5.5$ Hz, 3H), 7.34 (d, $J = 8.5$ Hz, 2H), 7.39 (d, $J = 8.5$ Hz, 2H), 7.50-7.57 (m, 2H), 7.66-7.72 (m, 1H), 8.24 (d, $J = 7.5$ Hz, 2H), 9.34 (br s, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ : 21.3, 26.4, 119.7, 125.3 (2C), 128.4 (2C), 128.8, 129.6 (2C), 130.8, 131.3(2C), 134.1, 140.3, 143.1, 157.1, 189.4; HRMS (ESI-TOF) Calcd for $\text{C}_{18}\text{H}_{17}\text{N}_4\text{O}_2^+$ ($[\text{M} + \text{H}]^+$): 321.1346. Found 321.1332.

4-Benzoyl-N,N-dimethyl-1-p-tolyl-1H-1,2,3-triazole-5-carboxamide (3r):



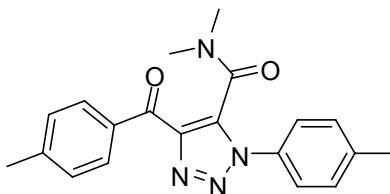
White solid; mp 171–173 °C; ^1H NMR (500 MHz, CDCl_3) δ : 2.44 (s, 3H), 2.81 (s, 3H), 3.13 (s, 3H), 7.34 (d, $J = 8.5$ Hz, 2H), 7.52–7.56 (m, 4H), 7.64 (t, $J = 7.0$ Hz, 1H), 8.48 (d, $J = 7.5$ Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 21.2, 34.9, 37.5, 123.2 (2C), 128.4 (2C), 130.3 (2C), 130.7 (2C), 133.2, 133.5, 136.1, 136.4, 140.6, 144.4, 160.2, 185.3; HRMS (ESI-TOF) Calcd for $\text{C}_{19}\text{H}_{19}\text{N}_4\text{O}_2^+$ ($[\text{M} + \text{H}]^+$): 335.1503. Found 335.1500.

Ethyl 4-(4-methoxybenzoyl)-1-p-tolyl-1H-1,2,3-triazole-5-carboxylate (3s):



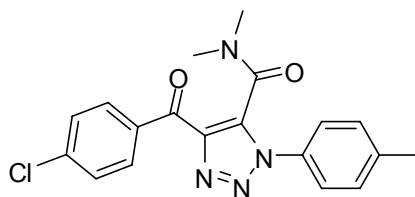
Yellow solid; mp 160–162 °C; ^1H NMR (500 MHz, CDCl_3) δ : 1.20 (t, $J = 7.5$ Hz, 3H), 2.46 (s, 3H), 3.90 (s, 3H), 4.32 (q, $J = 7.0$ Hz, 2H), 7.01 (dd, $J = 7, 2.0$ Hz, 2H), 7.35 (d, $J = 8.5$ Hz, 2H), 7.47 (d, $J = 8.5$ Hz, 2H), 8.35 (dd, $J = 7, 2.0$ Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 13.6, 21.3, 55.5, 63.0, 113.8 (2C), 124.4 (2C), 129.1, 130.0 (2C), 132.3, 133.0 (2C), 133.2, 140.7, 146.9, 159.2, 164.1, 183.8; HRMS (ESI-TOF) Calcd for $\text{C}_{20}\text{H}_{20}\text{N}_3\text{O}_4^+$ ($[\text{M} + \text{H}]^+$): 366.1448. Found 366.1454.

N,N-Dimethyl-4-(4-methylbenzoyl)-1-p-tolyl-1H-1,2,3-triazole-5-carboxamide (3t):



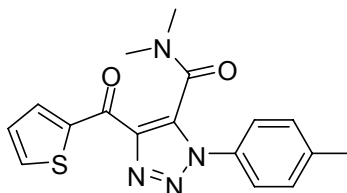
Yellow solid; mp 195–197 °C; ^1H NMR (500 MHz, CDCl_3) δ : 2.42–2.44 (d, 6H), 2.80 (s, 3H), 3.11 (s, 3H), 7.31–7.34 (m, 4H), 7.54 (d, $J = 8.5$ Hz, 2H), 8.41 (d, $J = 8.0$ Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 21.2, 21.7, 34.9, 37.5, 123.2 (2C), 129.1 (2C), 130.3 (2C), 130.8 (2C), 133.1, 133.5, 136.3, 140.5, 144.5, 144.5, 160.3, 184.7; HRMS (ESI-TOF) Calcd for $\text{C}_{20}\text{H}_{21}\text{N}_4\text{O}_2^+$ ($[\text{M} + \text{H}]^+$): 349.1659. Found 349.1661.

4-(4-Chlorobenzoyl)-*N,N*-dimethyl-1-*p*-tolyl-1*H*-1,2,3-triazole-5-carboxamide (3u):



Yellow solid; mp 172–174 °C; ^1H NMR (500 MHz, CDCl_3) δ : 2.45 (s, 3H), 2.80 (s, 3H), 3.13 (s, 3H), 7.34 (d, $J = 8$ Hz, 2H), 7.51–7.55 (m, 4H), 8.48 (d, $J = 8.5$ Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 21.2, 34.9, 37.5, 123.2 (2C), 128.8 (2C), 130.4 (2C), 132.1 (2C), 133.0, 134.3, 136.5, 140.1, 140.7, 144.1, 160.1, 183.8; HRMS (ESI-TOF) Calcd for $\text{C}_{19}\text{H}_{18}\text{ClN}_4\text{O}_2^+$ ($[\text{M} + \text{H}]^+$): 369.1113. Found 369.1120.

***N,N*-Dimethyl-4-(thiophene-2-carbonyl)-1-*p*-tolyl-1*H*-1,2,3-triazole-5-carboxamide (3v):**



Yellow solid; mp 192–194 °C; ^1H NMR (500 MHz, CDCl_3) δ : 2.44 (s, 3H), 2.82 (s, 3H), 3.13 (s, 3H), 7.25 (dd, $J = 5.0, 4.0$ Hz, 1H), 7.33 (d, $J = 8.5$ Hz, 2H), 7.54 (d, $J = 8.5$ Hz, 2H), 7.78 (dd, $J = 5.0, 1.0$ Hz, 1H), 8.78 (dd, $J = 3.5, 1.0$ Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ : 21.2, 34.9, 37.5, 123.2 (2C), 128.5, 130.3 (2C), 133.1, 135.4, 135.7, 136.5, 140.6, 142.1, 143.6, 159.9, 176.9; HRMS (ESI-TOF) Calcd for $\text{C}_{17}\text{H}_{17}\text{N}_4\text{O}_2\text{S}^+$ ($[\text{M} + \text{H}]^+$): 341.1067. Found 341.1066.

III. Crystal ORTEP Drawing of Compound 3a:

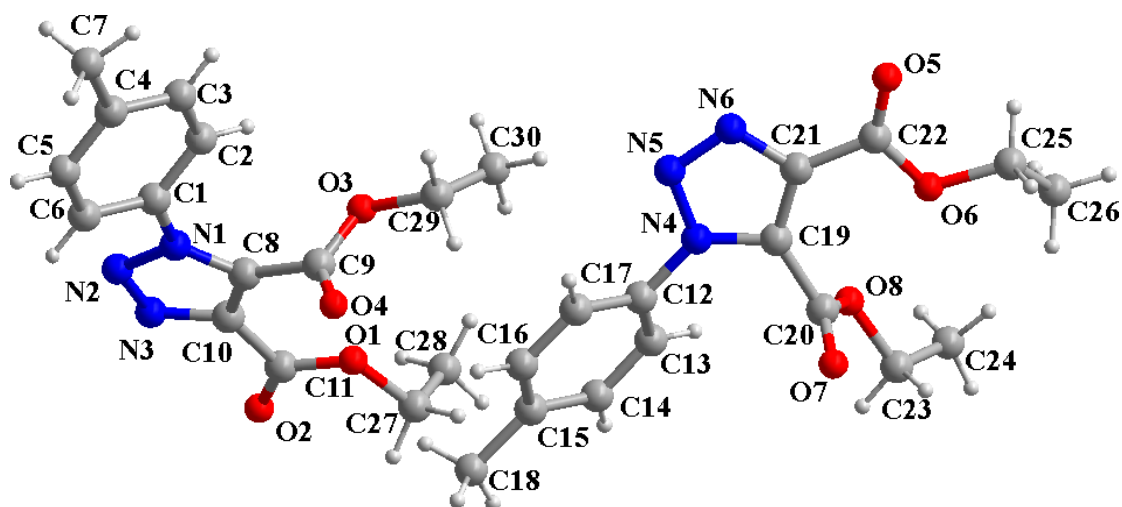


Figure 1 ORTEP drawing of 3a

IV. Copies of ^1H NMR and ^{13}C NMR spectra of compounds 3:

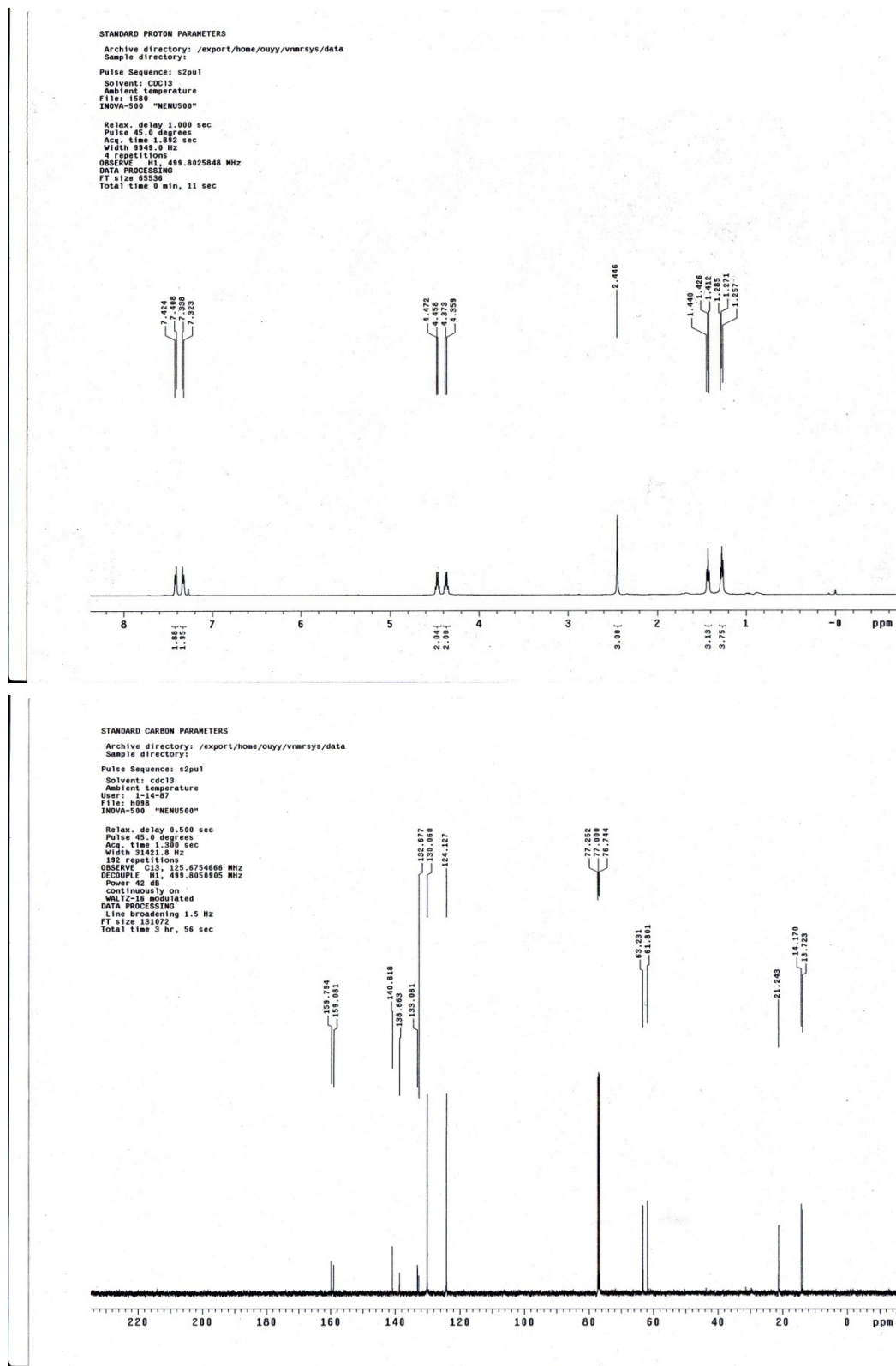
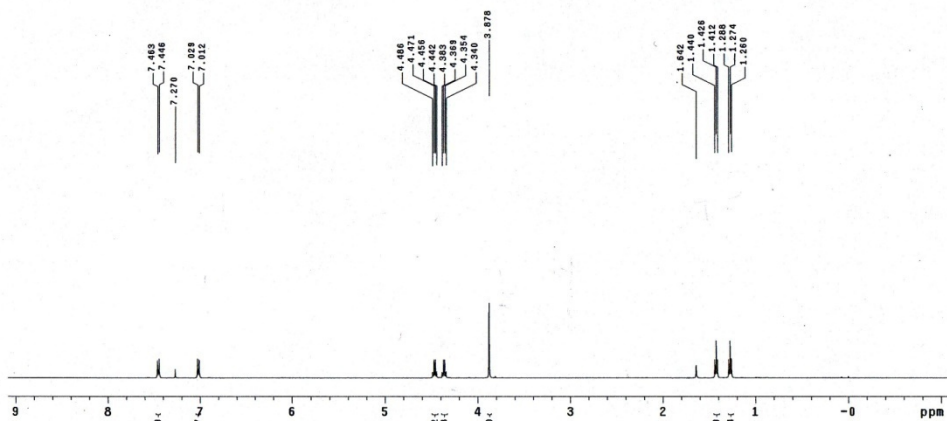


Figure 1. ^1H - (upper) and ^{13}C -NMR (lower) spectra of compound 3a.

STANDARD PROTON PARAMETERS
 Archive directory: /export/home/ouyy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 File: 1157
 INOVA-500 "NENUS00"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.092 sec
 Width 8969.0 Hz
 4 repetitions
 OBSERVE H1 499.8025863 MHz
 DATA PROCESSING
 FT size 85536
 Total time 0 min, 11 sec



STANDARD CARBON PARAMETERS
 Archive directory: /export/home/ouyy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pul
 Solvent: cdcl3
 Ambient temperature
 User: 1-14-87
 File: 1158
 INOVA-500 "NENUS00"
 Relax. delay 0.500 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 31421.8 Hz
 182 repetitions
 OBSERVE C13 125.6754846 MHz
 DECOUPLE H1 499.8050905 MHz
 Power 42 db
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.5 Hz
 FT size 131872
 Total time 30 min, 52 sec

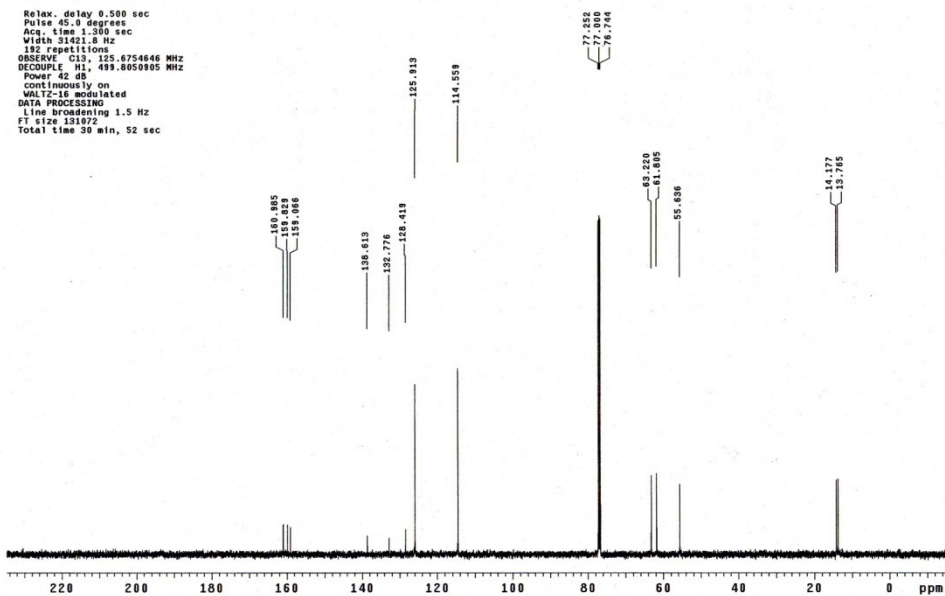


Figure 2. ^1H - (upper) and ^{13}C -NMR (lower) spectra of compound **3b**.

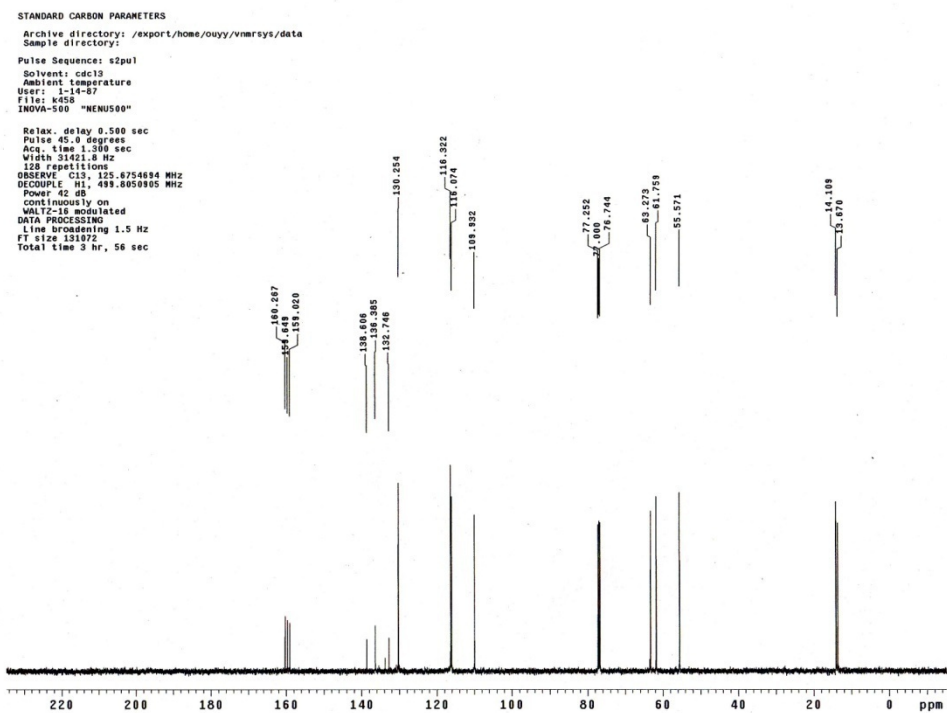
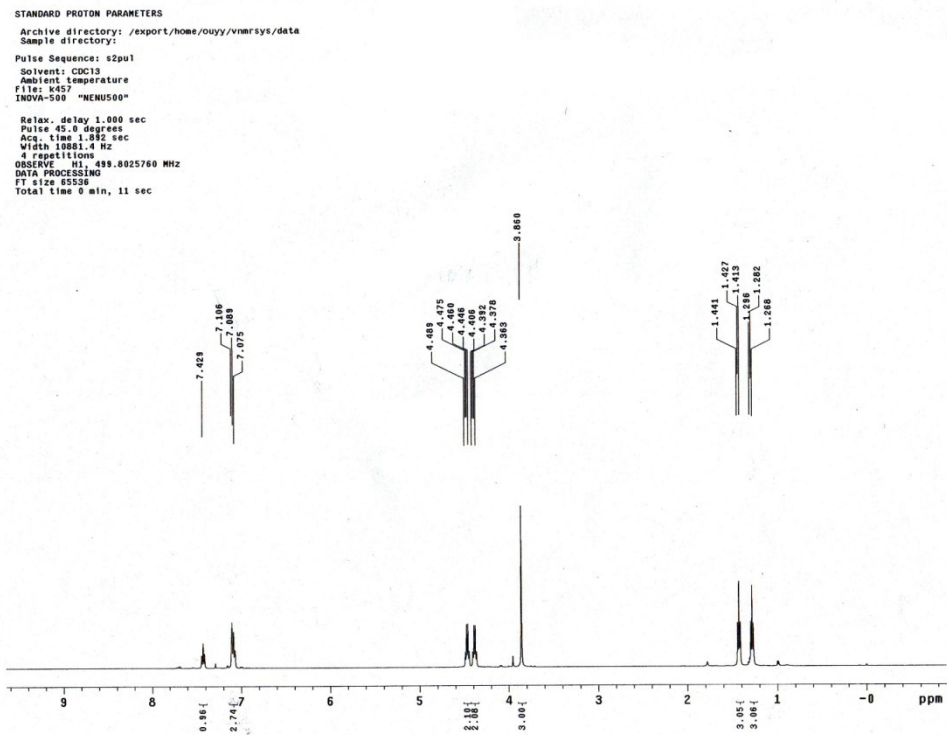
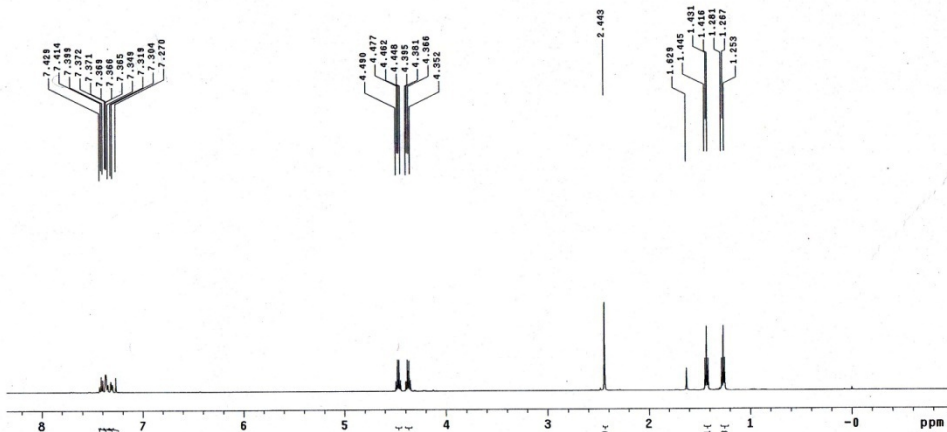


Figure 3. ^1H - (upper) and ^{13}C -NMR (lower) spectra of compound **3c**.

STANDARD PROTON PARAMETERS
 Archive directory: /export/home/ouyy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pu1
 Solvent: CDC13
 Ambient temperature
 File: 1161
 INOVA-500 "NENU500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.332 sec
 Width 9549.0 Hz
 OBSERVE H1, 499.8025869 MHz
 4 repetitions
 DATA PROCESSING
 FT size 65536
 Total time 0 min, 11 sec



STANDARD CARBON PARAMETERS
 Archive directory: /export/home/ouyy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pu1
 Solvent: cdc13
 Ambient temperature
 User: 1-14-87
 File: 1162
 INOVA-500 "NENU500"
 Relax. delay 0.500 sec
 Pulse 45.0 degrees
 Acq. time 1.330 sec
 Width 31421.8 Hz
 192 repetitions
 OBSERVE C13, 125.6754642 MHz
 DECOUPLE H1, 499.8050905 MHz
 Power 42 dB
 continuously on
 MULTIZ-16 modulated
 DATA PROCESSING
 Line broadening 1.5 Hz
 FT size 131072
 Total time 30 min, 52 sec

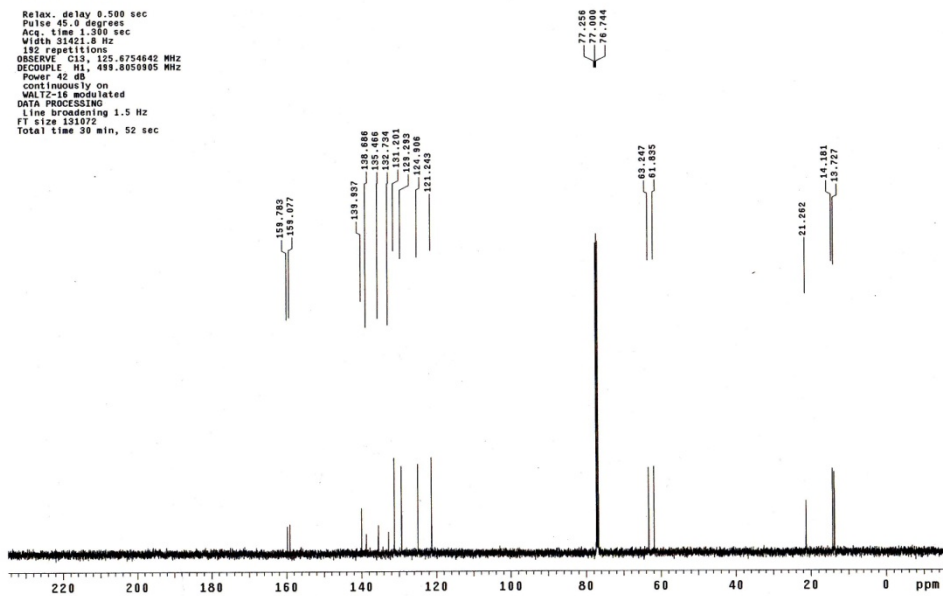


Figure 4. ^1H - (upper) and ^{13}C -NMR (lower) spectra of compound **3d**.

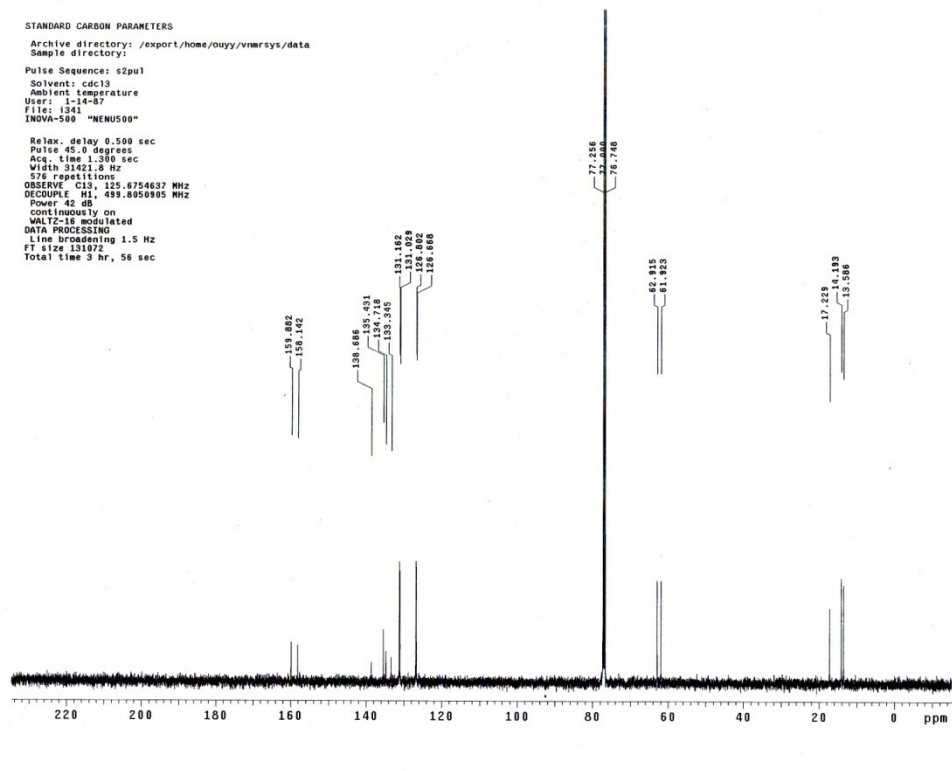
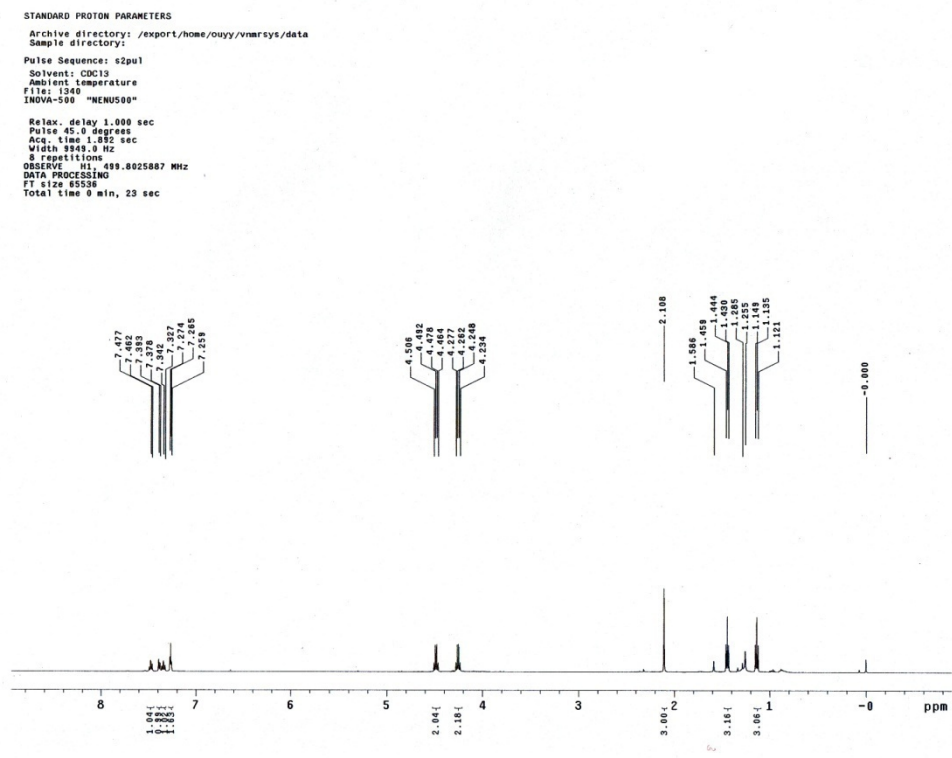
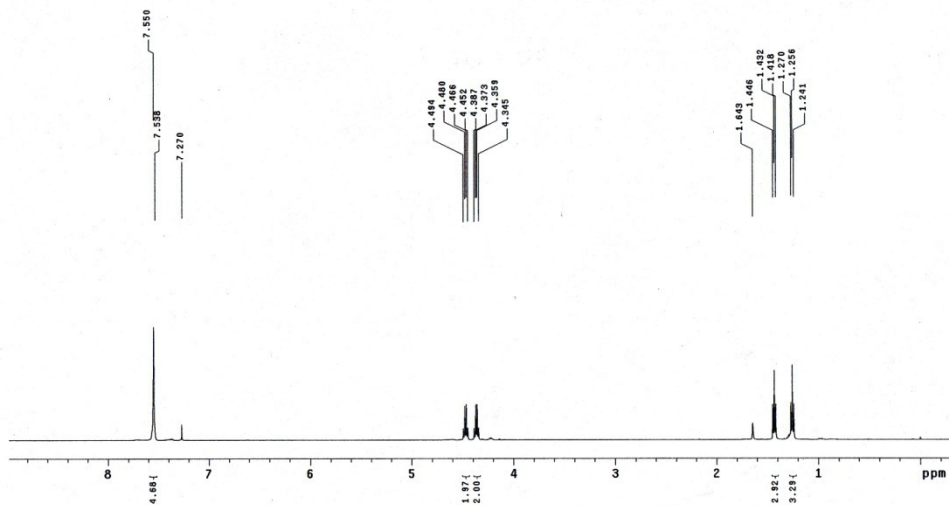


Figure 5. ¹H- (upper) and ¹³C-NMR (lower) spectra of compound 3e.

STANDARD PROTON PARAMETERS
 Archive directory: /export/home/ouyy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 File: 1159
 INOVA-500 "NENUS00"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.092 sec
 Width 8943.0 Hz
 OBSERVE H1, 499.8025863 MHz
 4 repetitions
 DATA PROCESSING
 FT size 85536
 Total time 0 min, 11 sec



STANDARD CARBON PARAMETERS
 Archive directory: /export/home/ouyy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pu1
 Solvent: cdcl3
 Ambient temperature
 User: 1-1-97
 File: 1160
 INOVA-500 "NENUS00"
 Relax. delay 8.500 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 31821.0 Hz
 256 repetitions
 OBSERVE C13, 125.6754646 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 30 min, 52 sec

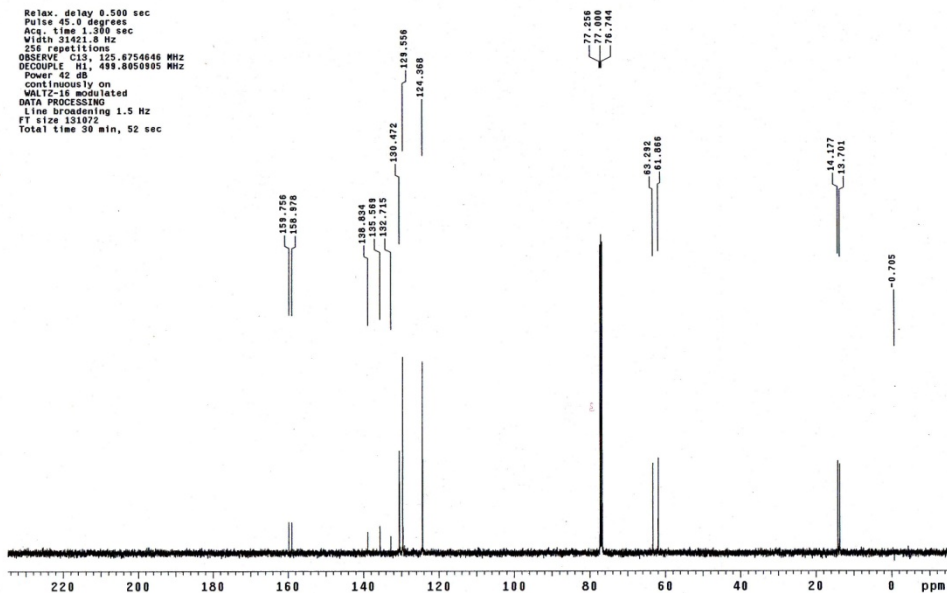
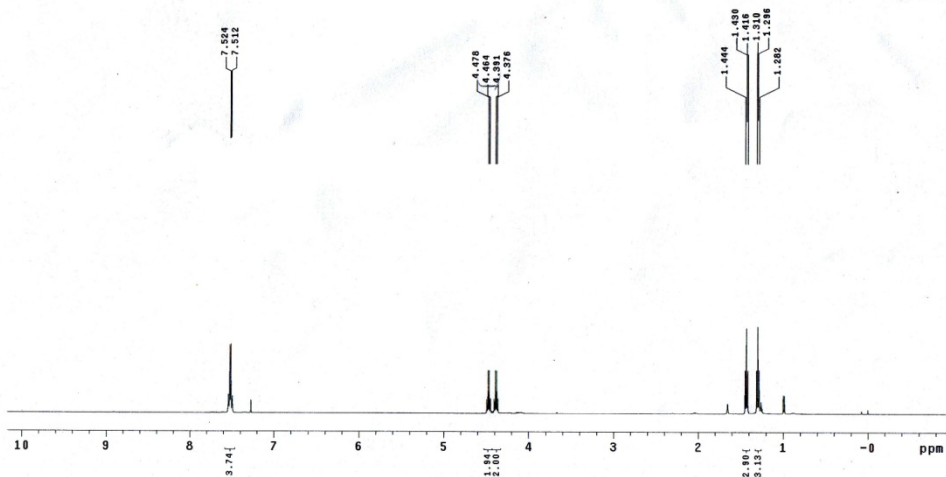


Figure 6. ^1H - (upper) and ^{13}C -NMR (lower) spectra of compound **3f**.

STANDARD PROTON PARAMETERS

Archive directory: /export/home/ouyy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient Temperature
 File: s409
 INOVA-500 "NENUS00"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.892 sec
 Width 10081.4 Hz
 4 repetitions
 OBSERVE H1, 499.8025050 MHz
 DATA PROCESSING
 FT size 65536
 Total time 0 min, 11 sec



STANDARD CARBON PARAMETERS

Archive directory: /export/home/ouyy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pul
 Solvent: cdcl3
 Ambient Temperature
 User: 1-14-87
 File: s411
 INOVA-500 "NENUS00"
 Relax. delay 0.500 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 31421.8 Hz
 128 repetitions
 OBSERVE C13, 125.6754690 MHz
 DECOUPLE H1, 499.8050905 MHz
 Power 42 dB
 continuously on
 MALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.5 Hz
 FT size 121072
 Total time 3 hr, 56 sec

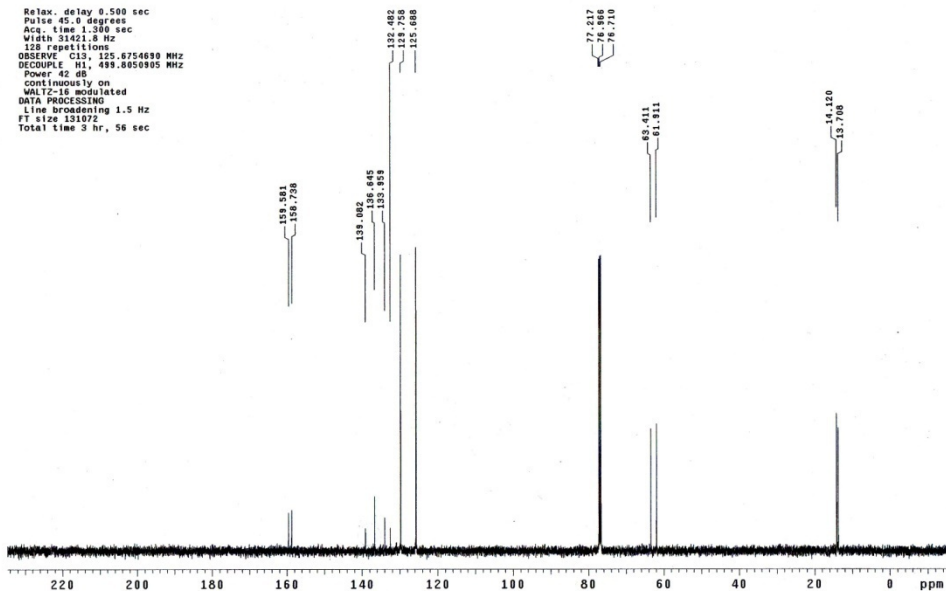


Figure 7. ¹H- (upper) and ¹³C-NMR (lower) spectra of compound 3g.

STANDARD PROTON PARAMETERS

Archive directory: /export/home/ouyy/vmr/sys/data
 Sample directory:

Pulse Sequence: s2pul

Solvent: CDC13

Ambient temperature

File: k917

INOVA-500 "NENUS00"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.882 sec

Width 10881.4 Hz

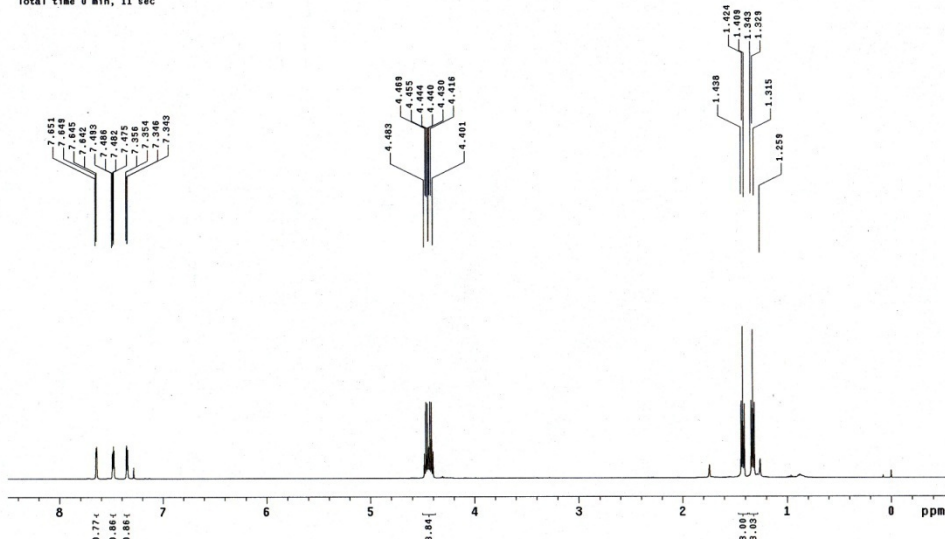
4 repetitions

OBSERVE H1, 499.8025786 MHz

DATA PROCESSING

FT size 65536

Total time 0 min, 11 sec



STANDARD CARBON PARAMETERS

Archive directory: /export/home/ouyy/vmr/sys/data
 Sample directory:

Pulse Sequence: s2pul

Solvent: cdcl3

Ambient temperature

User: 1-14-87

File: k918

INOVA-500 "NENUS00"

Relax. delay 0.500 sec

Pulse 45.0 degrees

Acq. time 1.380 sec

Width 31421.8 Hz

128 repetitions

OBSERVE C13, 125.6754675 MHz

DECOUPLE H1, 499.8025786 MHz

Power 42 dB

continuous by on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.5 Hz

FT size 131072

Total time 2 hr, 3 min, 31 sec

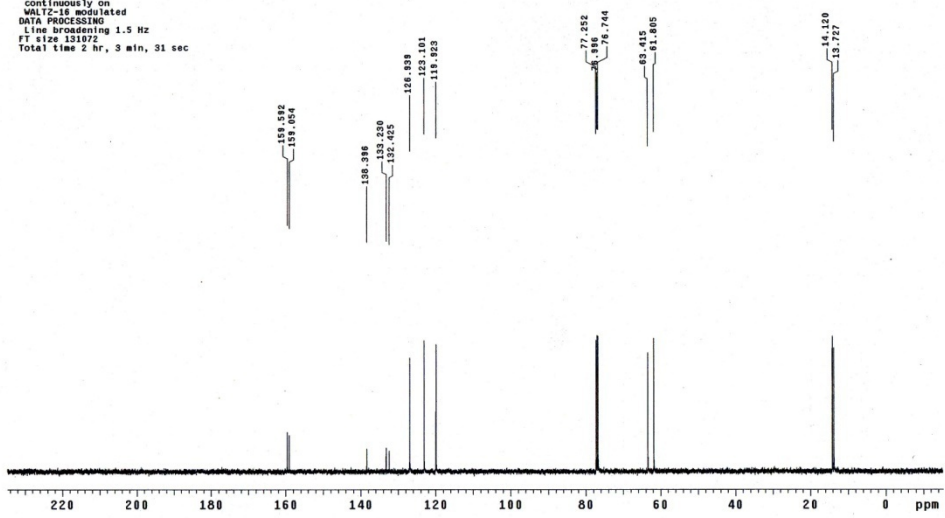
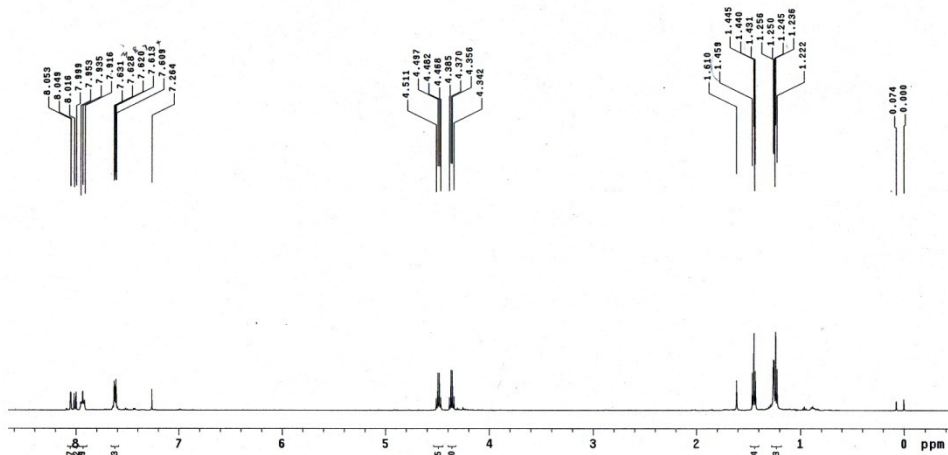


Figure 8. ¹H- (upper) and ¹³C-NMR (lower) spectra of compound **3h**.

STANDARD PROTON PARAMETERS
 Archive directory: /export/home/ouyy/vmr/sys/data
 Sample directory:
 Pulse Sequence: e2pu1
 Solvent: CDCl3
 Ambient temperature
 File: j656
 INOVA-500 "NENU500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.332 sec
 Width 8948.0 Hz
 32 repetitions
 OBSERVE H1, 499.8025905 MHz
 DATA PROCESSING
 FT size 83536
 Total time 1 min, 32 sec



STANDARD CARBON PARAMETERS
 Archive directory: /export/home/ouyy/vmr/sys/data
 Sample directory:
 Pulse Sequence: e2pu1
 Solvent: cdcl3
 Ambient temperature
 User: 1-14-87
 File: j656
 INOVA-500 "NENU500"
 Relax. delay 0.500 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 31421.8 Hz
 152 repetitions
 OBSERVE C13, 125.6754680 MHz
 DECOUPLE H1, 499.8050905 MHz
 Power 42.08
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.5 Hz
 FT size 131072
 Total time 2 hr, 3 min, 31 sec

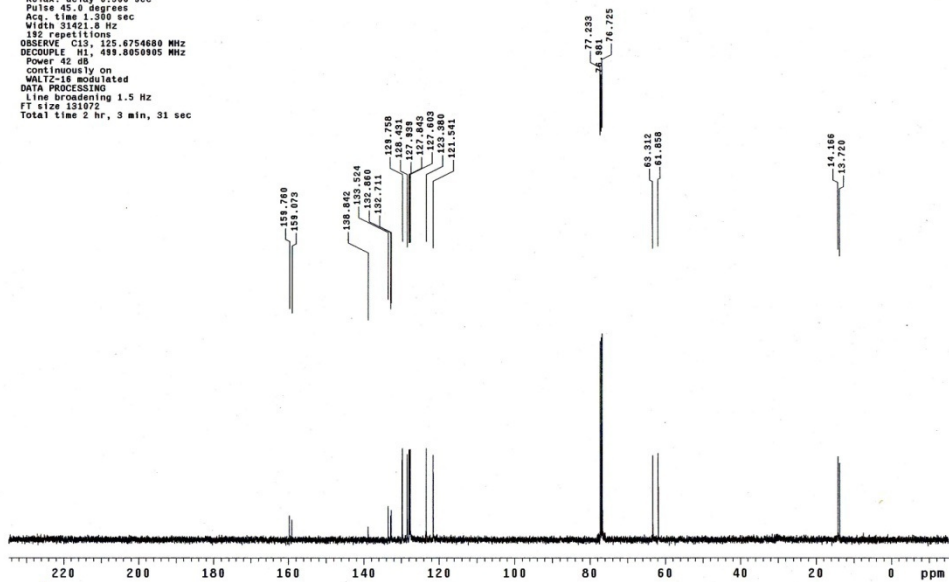


Figure 9. ^1H - (upper) and ^{13}C -NMR (lower) spectra of compound **3i**.

STANDARD PROTON PARAMETERS

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

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

File: 158

INOVA-500 "NENUS00"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.052 sec

Width 9999.0 Hz

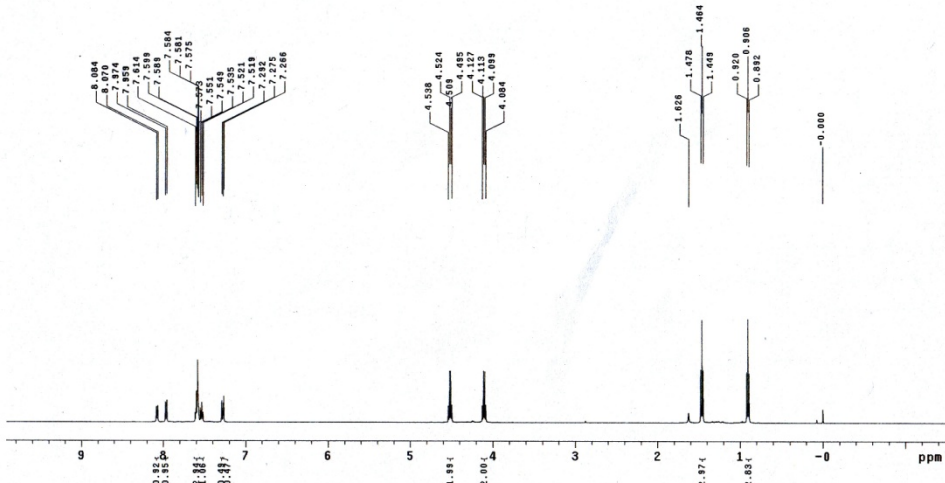
4 repetitions

OBSERVE H1 499.8025884 MHz

DATA PROCESSING

FT size 65536

Total time 0 min, 11 sec



STANDARD CARBON PARAMETERS

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

Pulse Sequence: s2pul

Solvent: cdcl3

Ambient temperature

User: l-14-87

File: 158

INOVA-500 "NENUS00"

Relax. delay 0.500 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 31421.8 Hz

320 repetitions

OBSERVE C13 125.6754651 MHz

DECOUPLE H1 499.8050905 MHz

Power 42 db

continuously on

MULTI-18 modulated

DATA PROCESSING

Line broadening 1.5 Hz

FT size 131972

Total time 3 hr, 56 sec

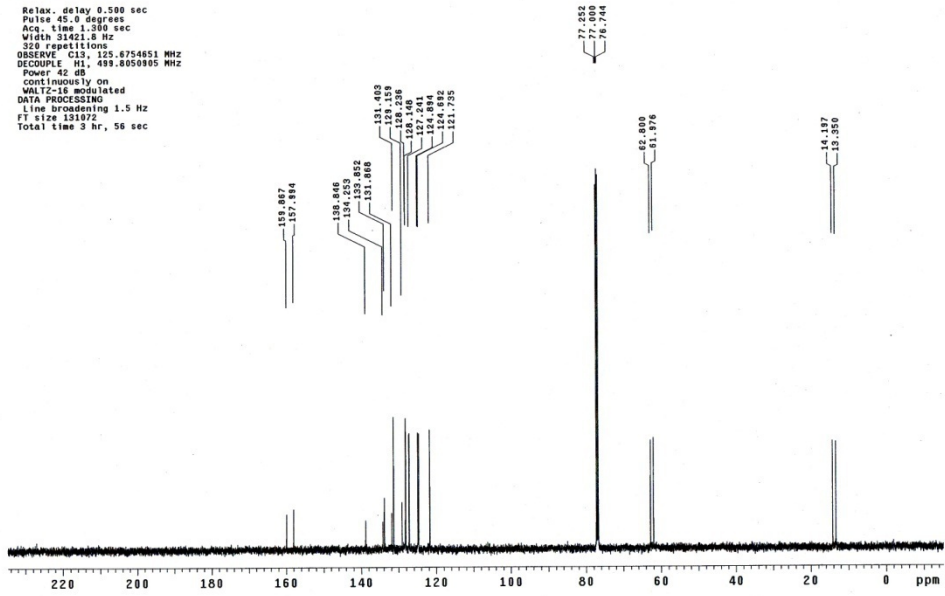
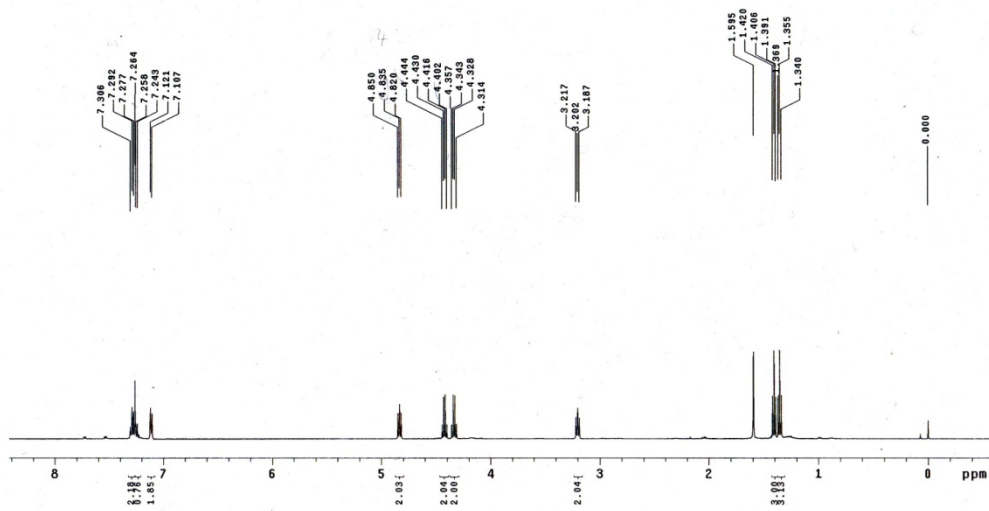


Figure 10. ¹H- (upper) and ¹³C-NMR (lower) spectra of compound 3j.

STANDARD PROTON PARAMETERS

Archive directory: /export/home/ouyy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 File: K184
 INOVA-500 "NENU500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.882 sec
 Width 10801.4 Hz
 4 repetitions
 OBSERVE H1, 499.8025899 MHz
 DATA PROCESSING
 FT size 8538
 Total time 0 min, 11 sec



STANDARD CARBON PARAMETERS

Archive directory: /export/home/ouyy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pul
 Solvent: cdcl3
 Ambient temperature
 User: 1-14-87
 File: K316
 INOVA-500 "NENU500"
 Relax. delay 0.500 sec
 Pulse 45.0 degrees
 Acq. time 1.380 sec
 Width 31421.8 Hz
 256 repetitions
 OBSERVE C13, 125.6754709 MHz
 DECOUPLE H1, 499.8050905 MHz
 Power 42 dB
 continuously on
 MLI2-18 modulated
 DATA PROCESSING
 Line broadening 1.5 Hz
 FT size 131972
 Total time 2 hr, 3 min, 31 sec

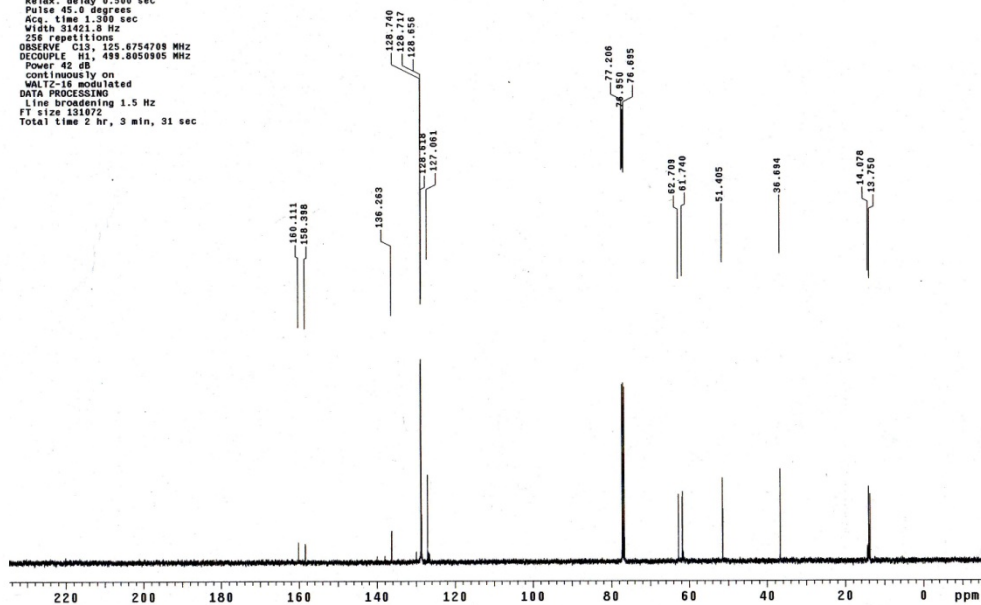


Figure 11. ¹H- (upper) and ¹³C-NMR (lower) spectra of compound **3k**.

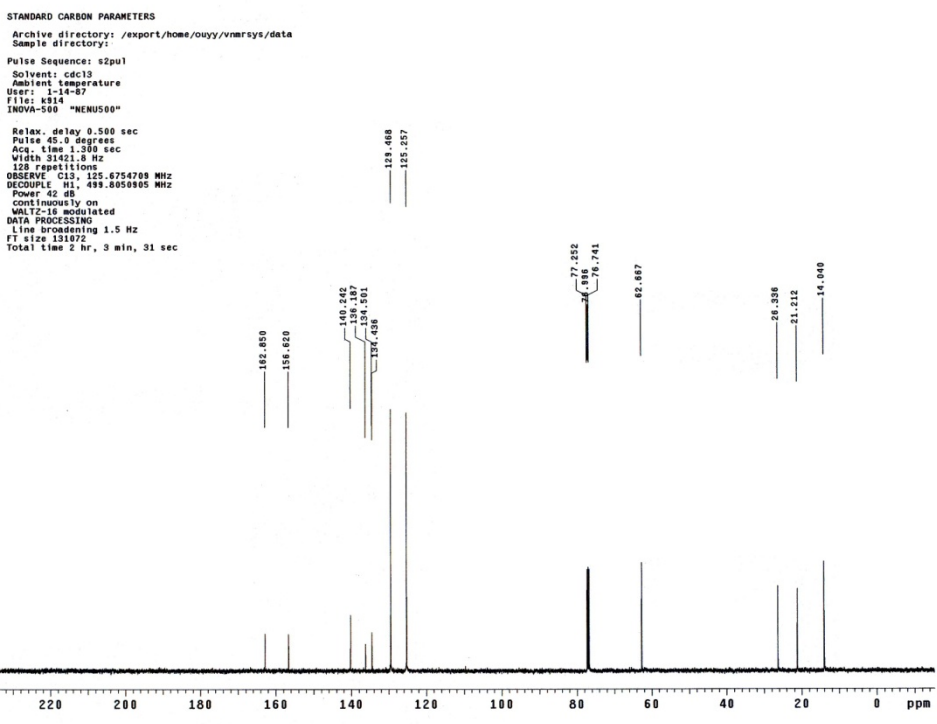
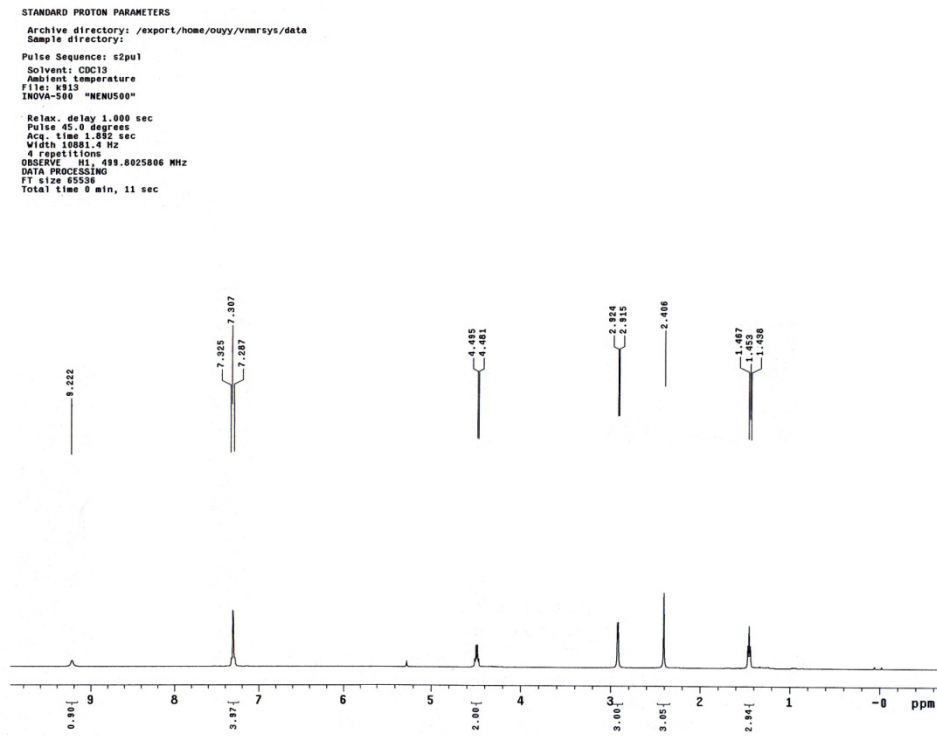


Figure 12. ¹H- (upper) and ¹³C-NMR (lower) spectra of compound 31.

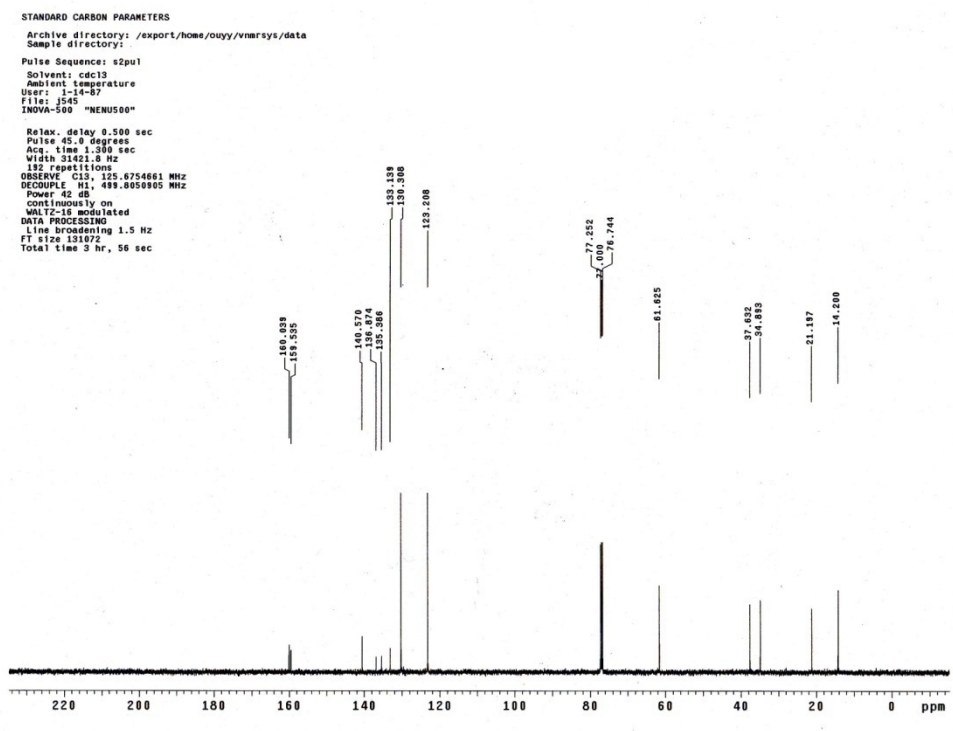
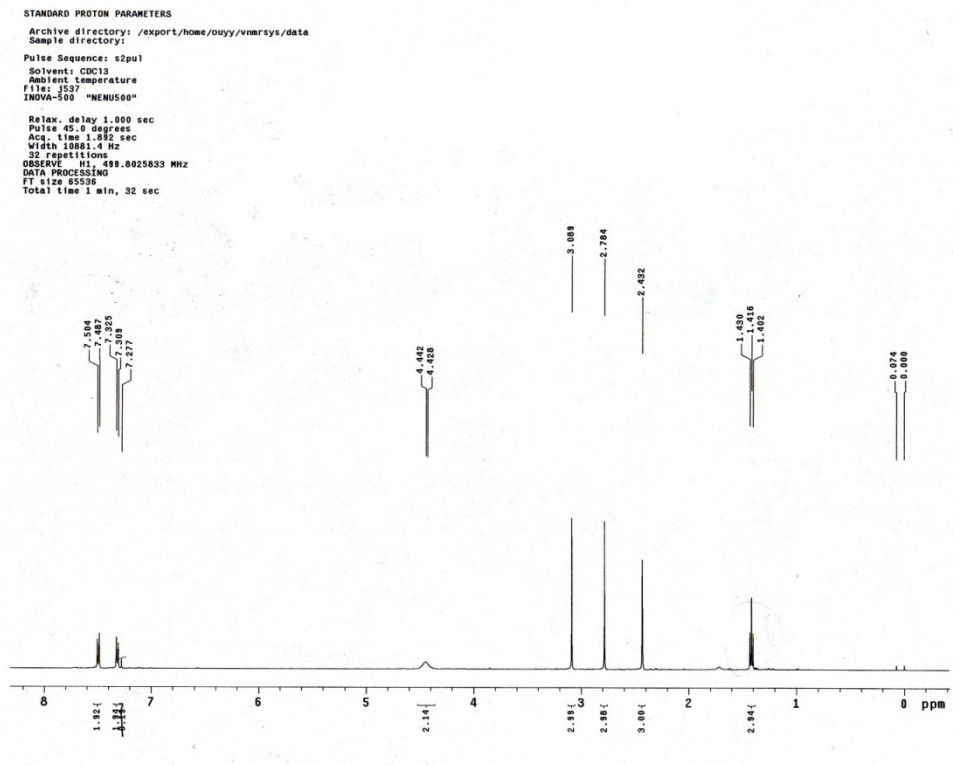
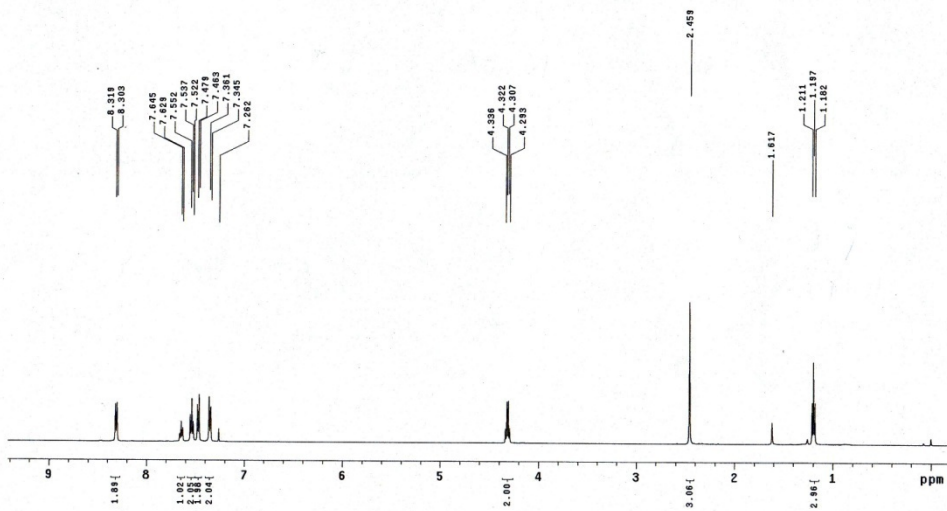


Figure 13. ¹H- (upper) and ¹³C-NMR (lower) spectra of compound 3m.

STANDARD PROTON PARAMETERS
 Archive directory: /export/home/ouyy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient Temperature
 File: 1043
 INOVA-500 "NENUS00"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.882 sec
 Width 10001.4 Hz
 4 repetitions
 OBSERVE H1, 499.8025899 MHz
 DATA PROCESSING
 FT size 65536
 Total time 0 min, 11 sec



STANDARD CARBON PARAMETERS
 Archive directory: /export/home/ouyy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pu1
 Solvent: cdcl3
 Ambient temperature
 User: 1-14-87
 File: 1050
 INOVA-500 "NENUS00"
 Relax. delay 0.500 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 31421.8 Hz
 128 repetitions
 OBSERVE C13, 125.6754656 MHz
 DECOUPLE H1, 499.8059365 MHz
 Power 42 dB
 Continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.5 Hz
 FT size 131072
 Total time 2 hr, 3 min, 31 sec

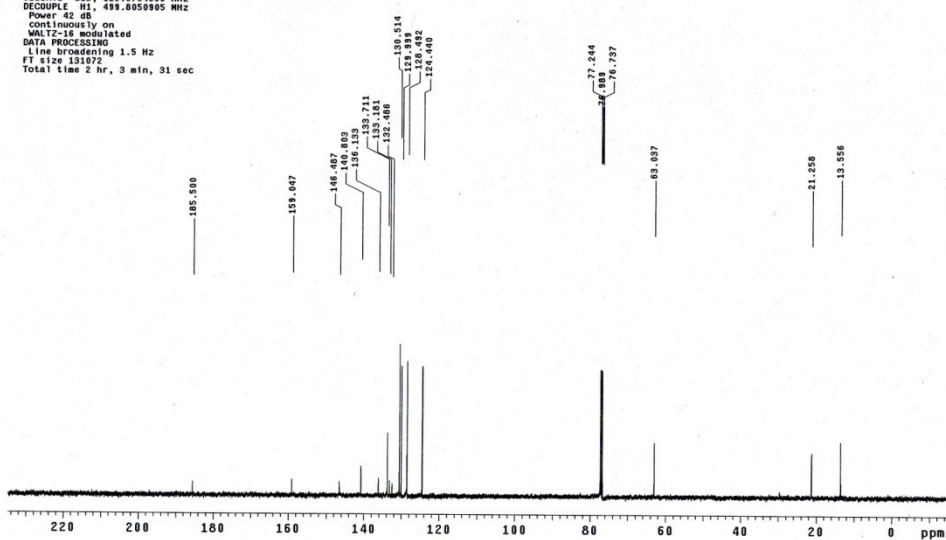
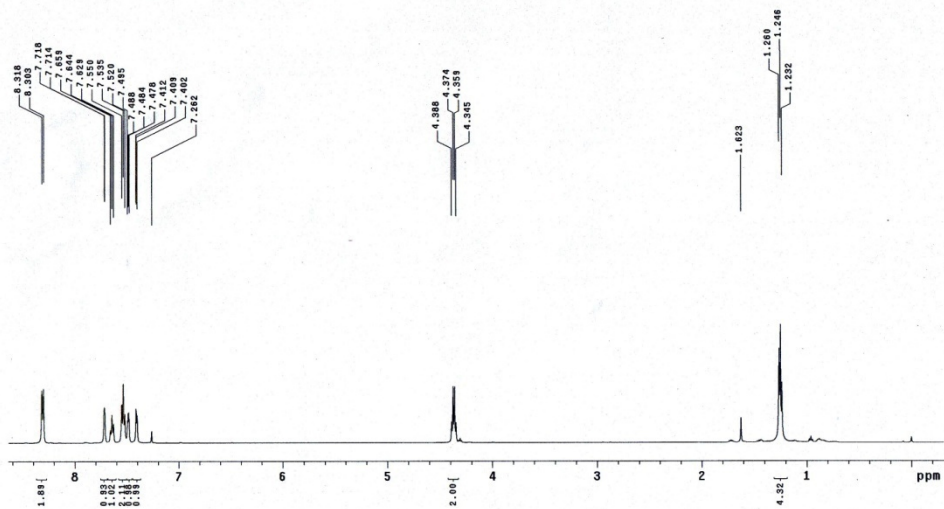


Figure 14. ^1H - (upper) and ^{13}C -NMR (lower) spectra of compound **3n**.

STANDARD PROTON PARAMETERS
 Archive directory: /export/home/ouyy/vmrsys/data
 Sample directory:
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 File: 1003
 INOVA-500 "NENU500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.892 sec
 Width 10881.6 Hz
 4 repetitions
 OBSERVE H1, 499.8025886 MHz
 DATA PROCESSING
 FI size 85536
 Total time 8 min, 11 sec



STANDARD CARBON PARAMETERS
 Archive directory: /export/home/ouyy/vmrsys/data
 Sample directory:
 Pulse Sequence: s2pu1
 Solvent: cdcl3
 Ambient temperature
 User: 1-14-87
 File: 1008
 INOVA-500 "NENU500"
 Relax. delay 0.500 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 21621.9 Hz
 128 repetitions
 OBSERVE C13, 125.6754656 MHz
 DECOUPLE H1, 499.8050965 MHz
 Power 42 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.5 Hz
 FI size 151072
 Total time 2 hr, 3 min, 31 sec

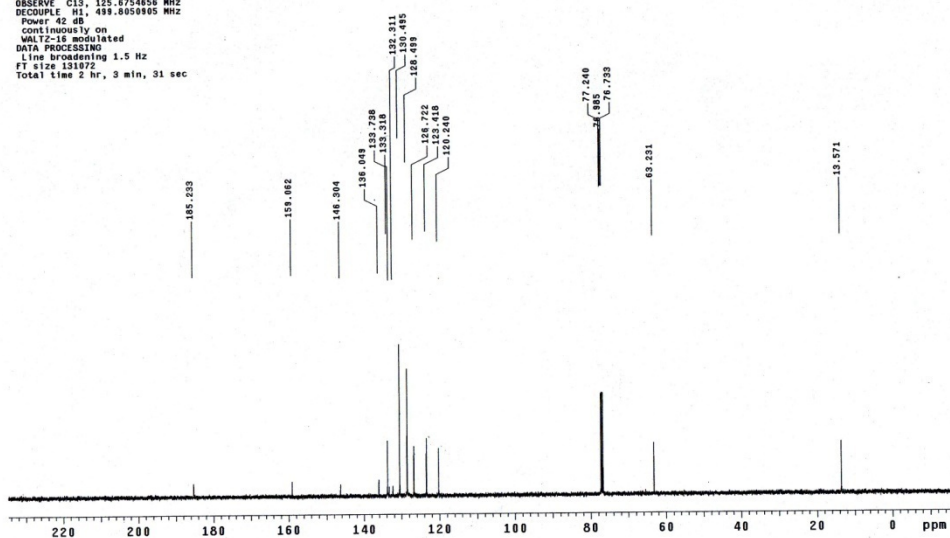
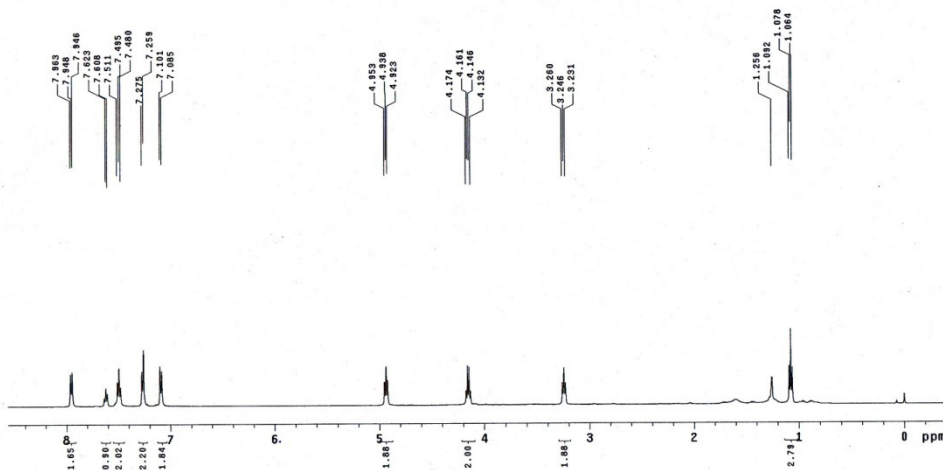


Figure 15. ^1H - (upper) and ^{13}C -NMR (lower) spectra of compound **30**.

STANDARD PROTON PARAMETERS
 Archive directory: /export/home/ouyy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 File: 1001
 INOVA-500 "NENU500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.882 sec
 Width 10881.4 Hz
 4 repetitions
 OBSERVE H1, 499.8025906 MHz
 DATA PROCESSING
 FT size 65536
 Total time 0 min, 11 sec



STANDARD CARBON PARAMETERS
 Archive directory: /export/home/ouyy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pu1
 Solvent: cdcl3
 Ambient temperature
 User: l-14-87
 File: 1052
 INOVA-500 "NENU500"
 Relax. delay 0.500 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 21421.8 Hz
 192 repetitions
 OBSERVE C13, 125.6754656 MHz
 DECOUPLE H1, 499.8050905 MHz
 Power 42 dB
 continuously on
 MALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.5 Hz
 FT size 121072
 Total time 2 hr, 3 min, 31 sec

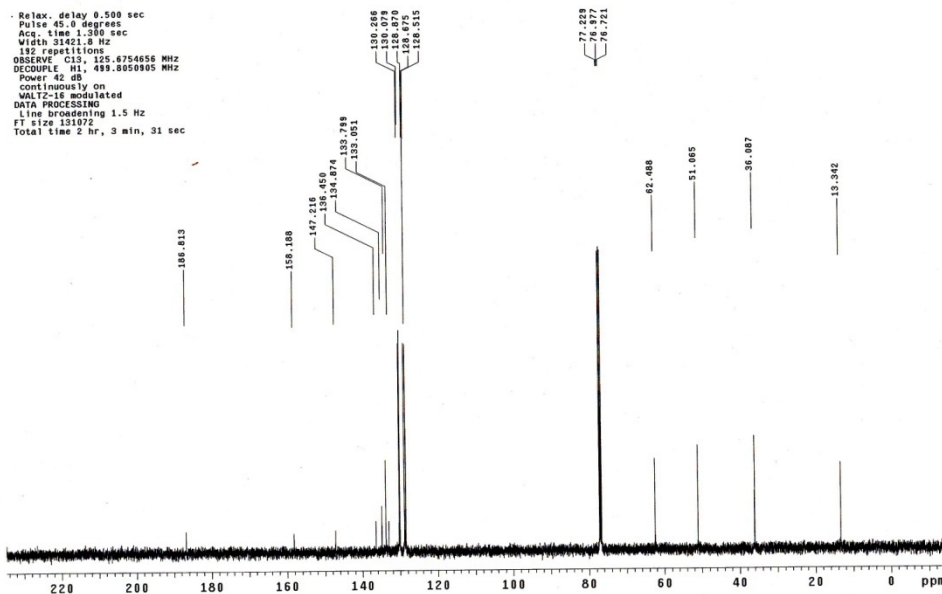
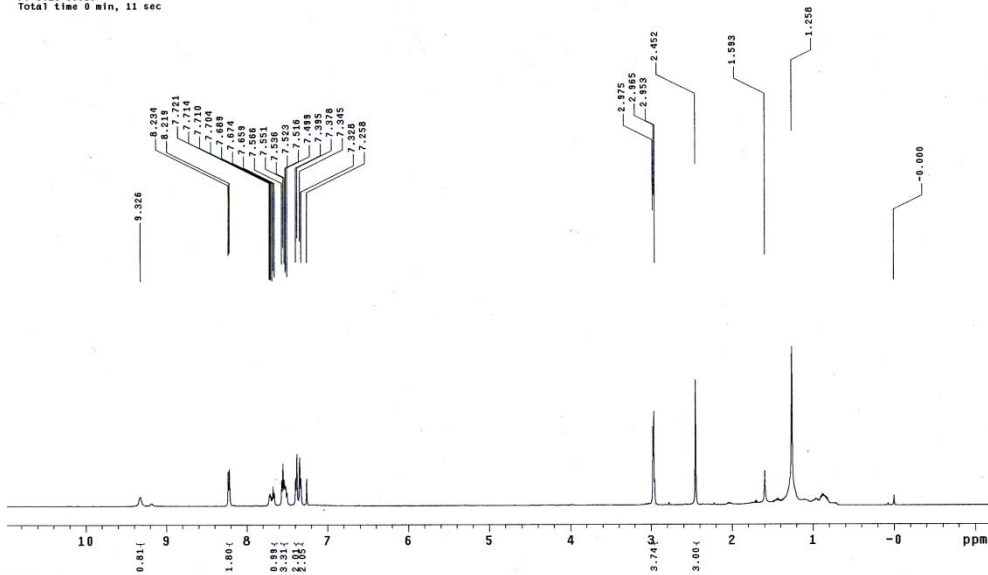


Figure 16. ¹H- (upper) and ¹³C-NMR (lower) spectra of compound **3p**.

STANDARD PROTON PARAMETERS

Archive directory: /export/home/ouyy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 File: 1062
 INOVA-500 "NENU500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.492 sec
 Width 10881.4 Hz
 4 repetitions
 OBSERVE H1, 499.8025919 MHz
 DATA PROCESSING
 FT size 65536
 Total time 0 min, 11 sec



STANDARD CARBON PARAMETERS

Archive directory: /export/home/ouyy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pu1
 Solvent: cdc13
 Ambient temperature
 User: 1-14-87
 File: 1063
 INOVA-500 "NENU500"
 Relax. delay 0.500 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 31421.8 Hz
 182 repetitions
 OBSERVE C13, 125.8754856 MHz
 DECOUPLE H1, 499.8050905 MHz
 Power 42 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.5 Hz
 FT size 131072
 Total time 2 hr, 3 min, 31 sec

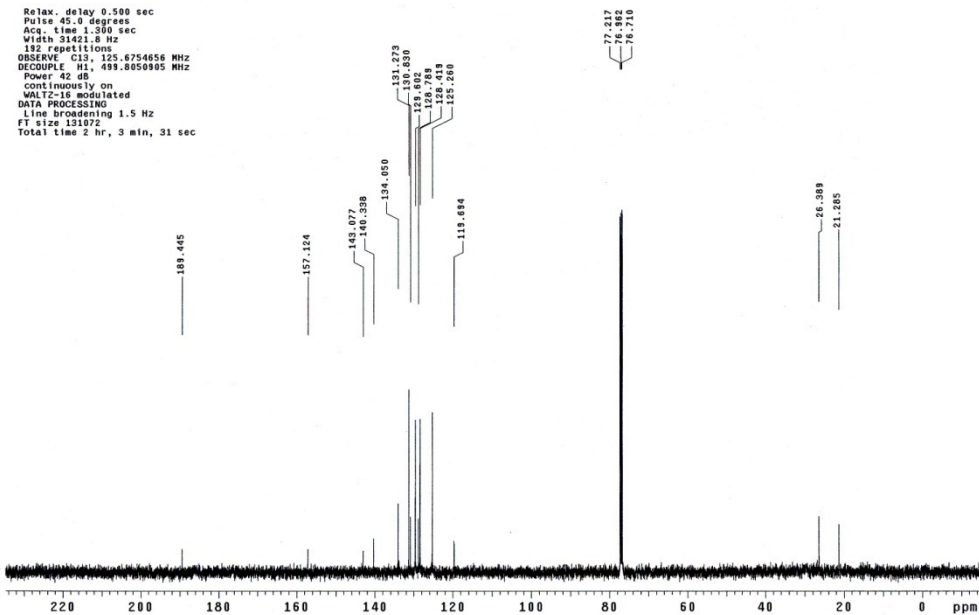


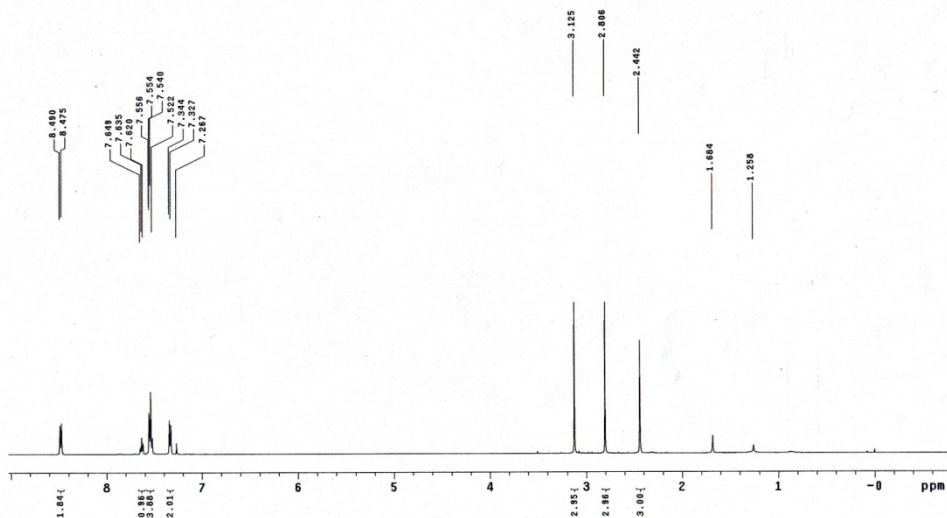
Figure 17. ^1H - (upper) and ^{13}C -NMR (lower) spectra of compound **3q**.

STANDARD PROTON PARAMETERS

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

Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient Temperature
File: k311
INOVA-500 "NENU500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.852 sec
Width 10881.4 Hz
4 repetitions
OBSERVE H1, 499.8025873 MHz
DATA PROCESSING
FT size 65536
Total time 9 min, 11 sec



STANDARD CARBON PARAMETERS

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

Pulse Sequence: s2pu1
Solvent: cdcl3
Ambient temperature
User: 1-14-87
File: k312
INOVA-500 "NENU500"

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
182 repetitions
OBSERVE C13, 125.6754858 MHz
DECUPLE R1, 499.8050905 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec

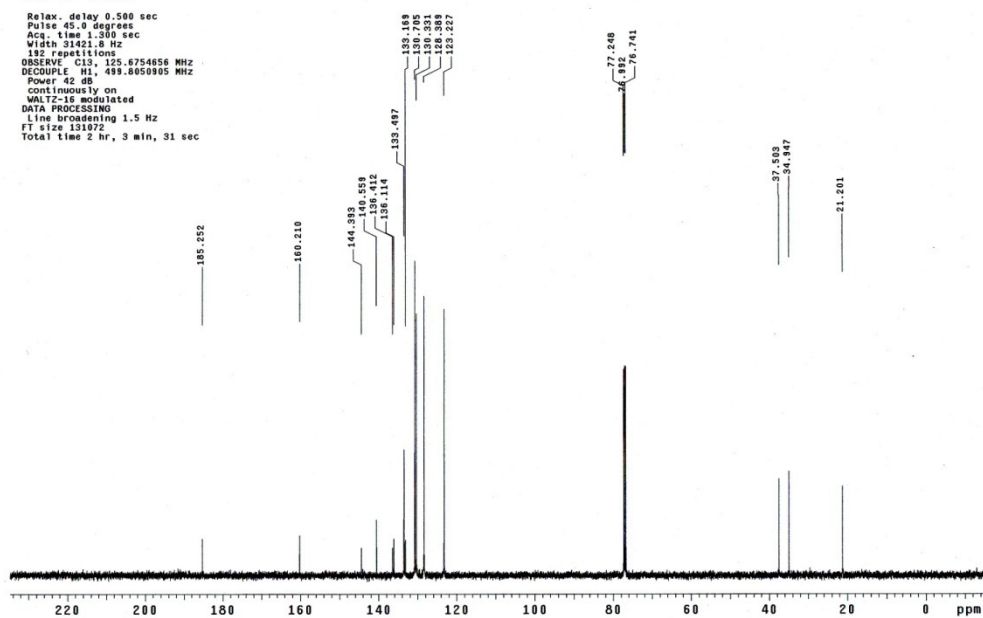


Figure 18. ^1H - (upper) and ^{13}C -NMR (lower) spectra of compound **3r**.

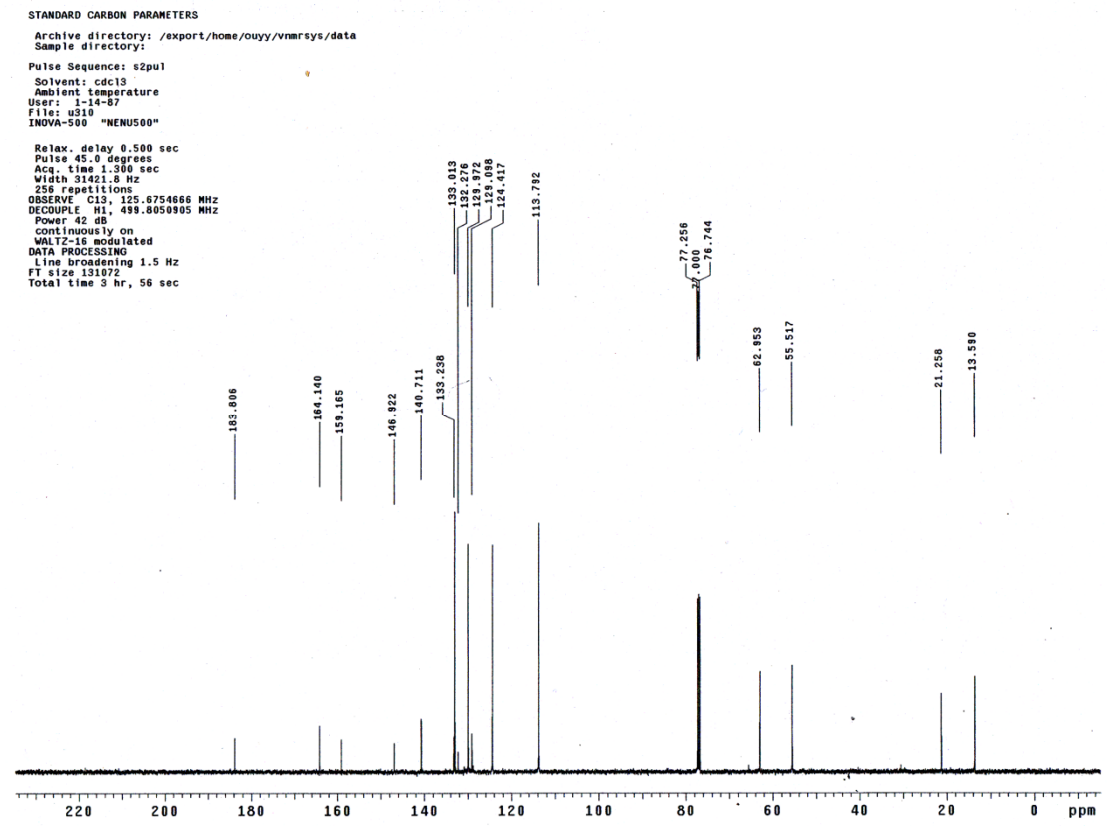
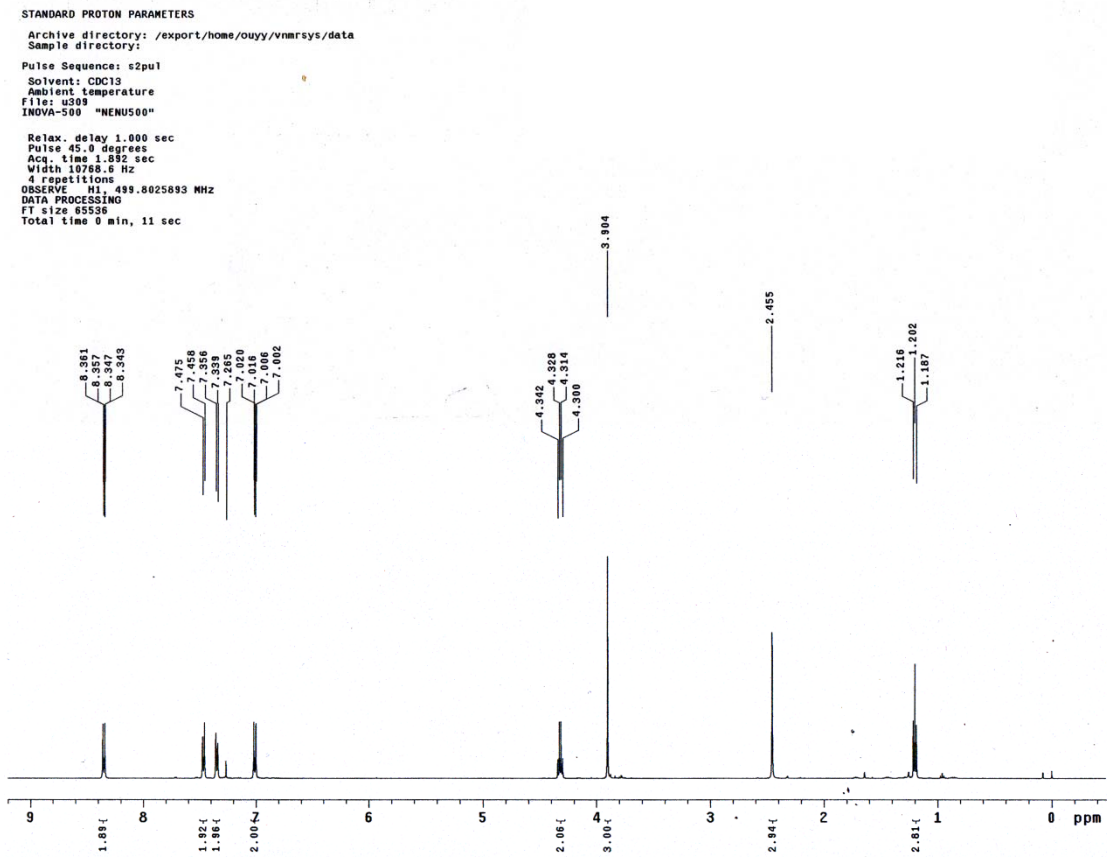
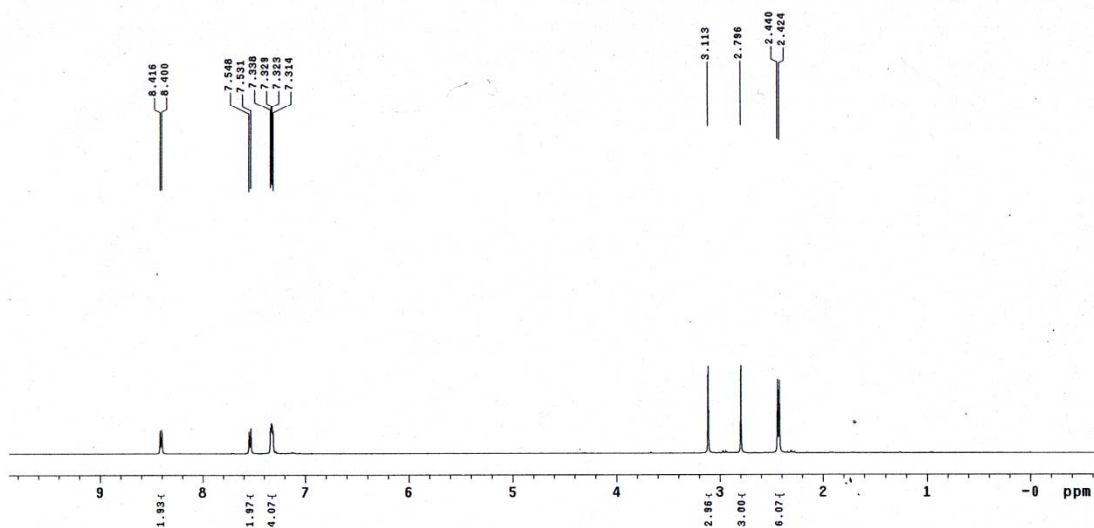


Figure 19. ¹H- (upper) and ¹³C-NMR (lower) spectra of compound 3s.

STANDARD PROTON PARAMETERS

Archive directory: /export/home/ouyy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 File: u354
 INOVA-500 "NENU500"

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.882 sec
 Width 10768.6 Hz
 4 repetitions
 OBSERVE H1, 499.8025788 MHz
 DATA PROCESSING
 FT size 65536
 Total time 0 min, 11 sec



STANDARD CARBON PARAMETERS

Archive directory: /export/home/ouyy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pul
 Solvent: cdcl3
 Ambient temperature
 User: 1-14-87
 File: u355
 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, 125.6754684 MHz
 DECOUPLE H1, 499.8050805 MHz
 Power 42 dB
 continuous ly on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.5 Hz
 FT size 131072
 Total time 2 hr, 3 min, 31 sec

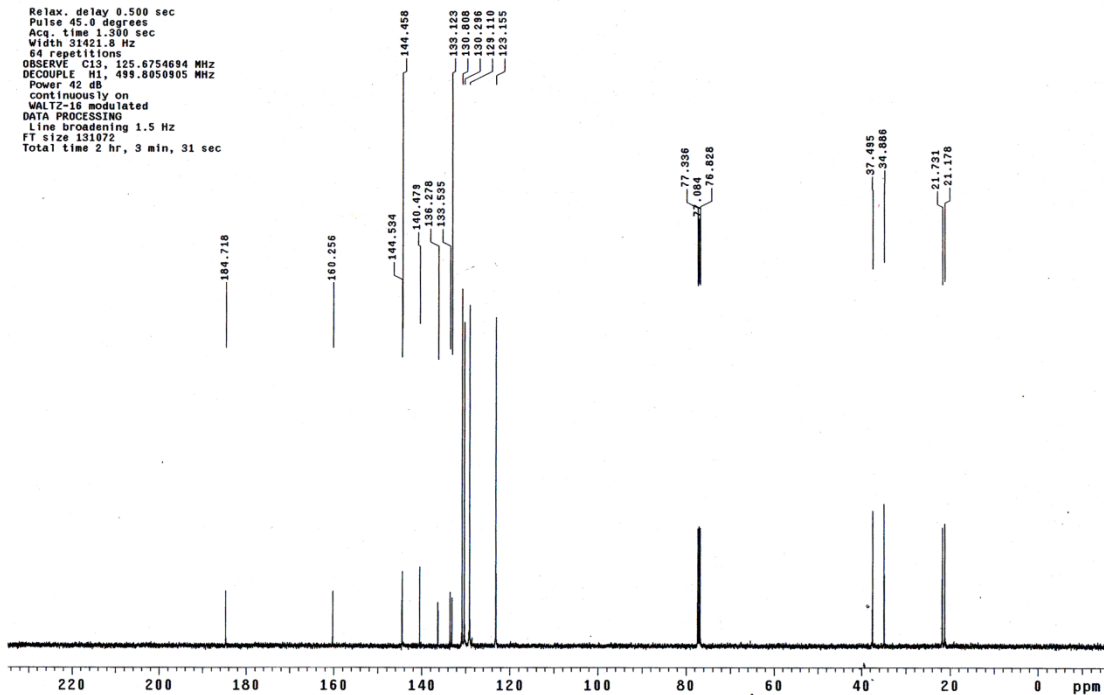


Figure 20. ^1H - (upper) and ^{13}C -NMR (lower) spectra of compound **3t**.

STANDARD PROTON PARAMETERS

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

Pulse Sequence: s2pul

Solvent: CDCl₃
Ambient temperature
File: u349
INOVA-500 "NENUS00"

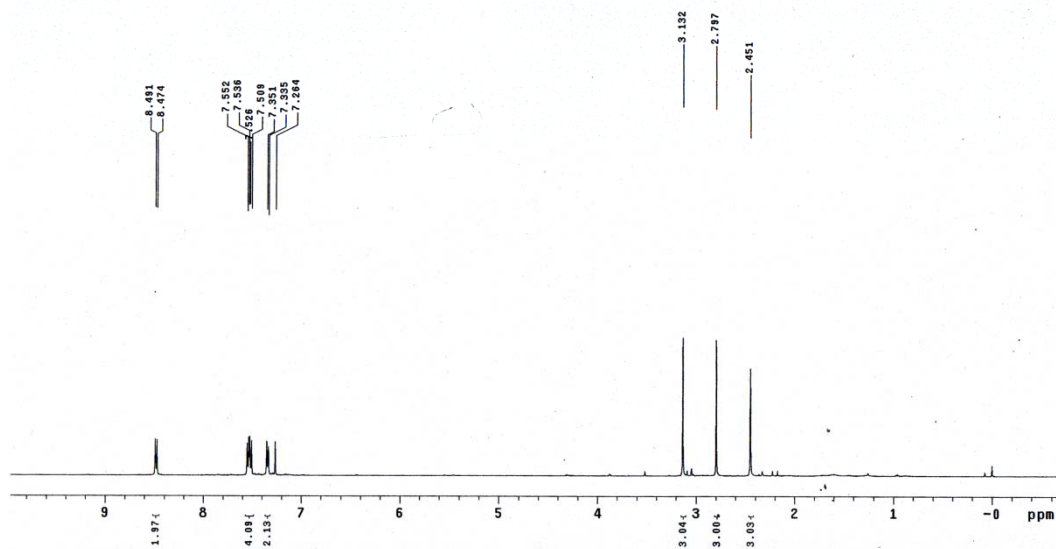
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.882 sec
Width 10768.6 Hz
4 repetitions

OBSERVE H1, 499.8025893 MHz

DATA PROCESSING

FT size 65536

Total time 0 min, 11 sec



STANDARD CARBON PARAMETERS

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

Pulse Sequence: s2pul

Solvent: cdcl₃
Ambient temperature
User: i-14-87
File: u357
INOVA-500 "NENUS00"

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz

182 repetitions

OBSERVE C13, 125.6754694 MHz

DECOUPLE H1, 499.8050905 MHz

Power 42 dB

Continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.5 Hz

FT size 131072

Total time 2 hr, 3 min, 31 sec

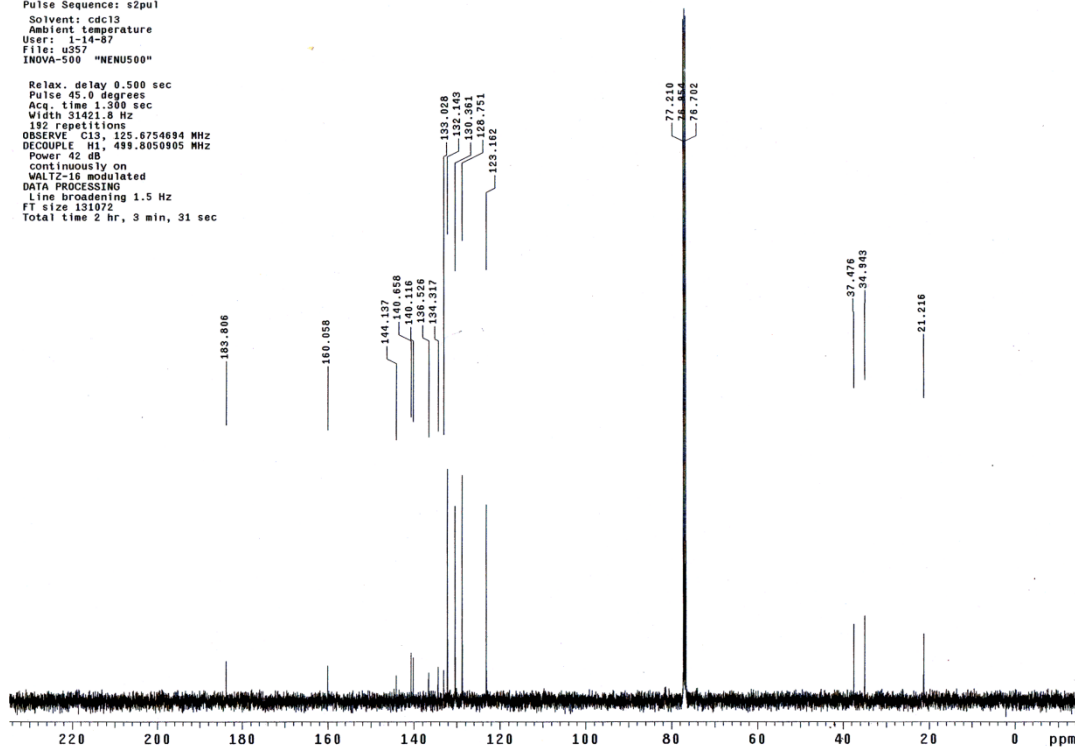


Figure 21. ¹H- (upper) and ¹³C-NMR (lower) spectra of compound **3u**.

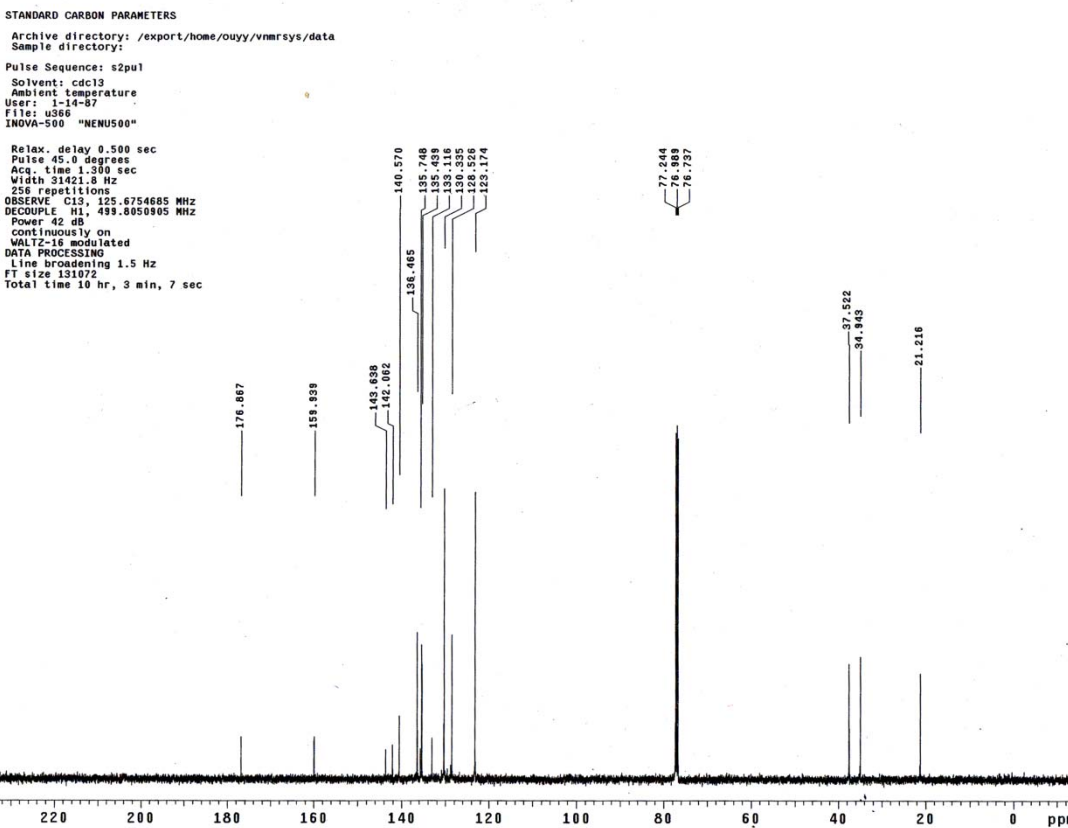
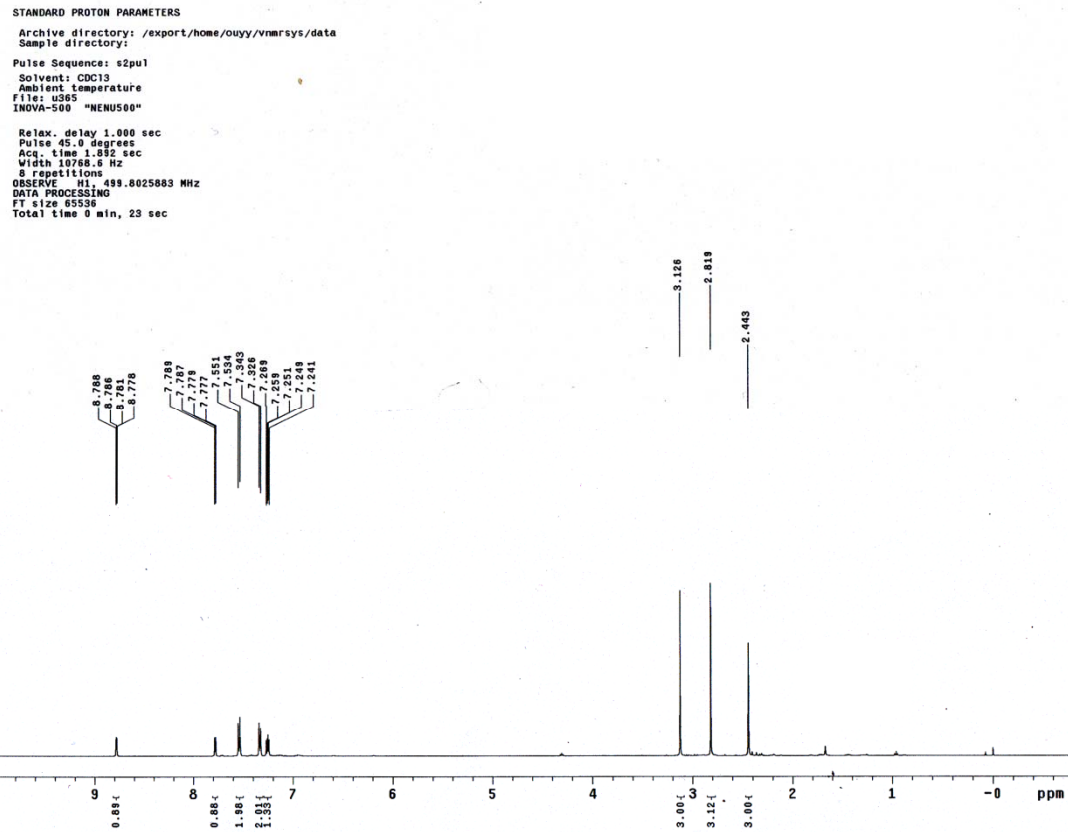


Figure 22. ^1H - (upper) and ^{13}C -NMR (lower) spectra of compound **3v**.