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Supplementary Information

Ultrasound-Assisted Synthesis of 1-*N*-β-D-Glucopyranosyl-1*H*-1,2,3-triazole Benzoheterocycles and their Anti-Inflammatory Activities

Gilson B. da Silva,[a] Bruna M. Guimarães,[a] Shalom P. O. Assis,[b] Vera L. M. Lima[b] and Ronaldo N. de Oliveira*[*,a]

[a]Departamento de Ciências Moleculares, Universidade Federal Rural de Pernambuco, Rua Dom Manoel de Medeiros s/n, Dois Irmãos, 52171-900 Recife-PE, Brazil

[b]Departamento de Bioquímica, Laboratório de Química e Metabolismo de Lipídeos e Lipoproteínas, Universidade Federal Rural de Pernambuco, 50670-901 Recife-PE, Brazil

Synthesis of 2,3,4,6-tetra-*O*-acetyl-β-D-glucopyranosyl azide (**1**)

A 25 mL round flask was charged with D-glucose (5.0 g, 27.8 mmol), Ac_2O (25 mL) and 3.5 mol% I_2 (0.25 g, 1 mmol) in one portion. The reaction mixture was irradiated in the water bath of the ultrasonic cleaner at ambient temperature (30 °C) for 20 min. The resulting mixture was washed with 20% sodium thiosulfate ($Na_2S_2O_3$) and extracted with CH_2Cl_2 (3 × 20 mL). The combined organic layers were washed with saturated $NaHCO_3$ (30 mL). After drying over anhydrous sodium sulfate, the solvent was removed under vacuum. The peracetylated D-glucose was obtained as a colorless solid 95% (10.3 g) which was used without any purification. In the next step, HBr–AcOH (prepared by mixing 13 mL of 48% HBr with 24 mL of Ac_2O at 0 °C) was slowly added to a stirred solution of peracetylated D-glucose (5.0 g, 12.8 mmol) in 30 mL of CH_2Cl_2 at 0 °C. The reaction mixture was irradiated with ultrasound for 50 min at 25-30 °C. The crude material was washed successively with cold water and a cooled saturated aqueous solution of $NaHCO_3$. After drying the organic layers over Na_2SO_4, the solvent was removed under vacuum. The glucopyranosyl bromide was obtained as a colorless solid 70% (3.68 g) which was used in the next step without any purification. To a solution of the above bromide (2.0 g, 4.87 mmol) in acetone (20 mL), sodium azide (0.5 g, 7.69 mmol) and water (5 mL) were added. The reaction mixture was irradiated for 40 min at room temperature, and then extracted with CH_2Cl_2 (3 × 30 mL). After work-up, the solvent was removed and the crude mixture was crystallized from isopropanol to afford 2,3,4,6-tetra-*O*-acetyl-β-D-glucopyranosyl azide (**1**) in 92% yield (1.68 g); mp 110-112 °C (lit. 125-126 °C);[1] $[\alpha]_D^{26}$ −22 (*c*0.5, CH_2Cl_2) (lit. −29; *c* 2.0 in $CHCl_3$);[1] IR (KBr) ν_{max}/cm^{-1} 2118 (N_3), 1755 (C=O), 1372, 1240, 1058, 1038; ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.21 (dd, 1H, *J* 9.0, 9.6 Hz, H-3), 5.09 (dd, 1H, *J* 9.9, 9.6 Hz, H-4), 4.95 (dd, 1H, *J* 9.0, 9.0 Hz, H-2), 4.64 (d, 1H, *J* 9.0 Hz, H-1), 4.27 (dd, 1H, *J* 4.8, 12.6 Hz, H-6'), 4.16 (dd, 1H, *J* 2.4, 12.6 Hz, H-6), 3.79 (ddd, 1H, *J* 2.4, 4.8, 9.9 Hz, H-5), 2.09, 2.07, 2.02, 2.00 (4 × CH_3CO); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.6, 170.1, 169.3, 169.2, 87.9, 74.0, 72.6, 70.6, 67.8, 61.6, 20.7, 20.5.

Synthesis of propargylated *N*- and *S*-benzoheterocycles (**2c-g**)

1 mmol of K_2CO_3 and 1 mmol of the benzoheterocycle were suspended in anhydrous DMF (3 mL), and 1.5 equiv. propargyl bromide was added. The mixture was sonicated for a specific reaction time (10-60 min). Then, the mixture was extracted with 50% CH_2Cl_2/water (3 × 15 mL). The combined organic layers were dried over sodium sulfate anhydrous and concentrated under reduced pressure to afford the corresponding propargylated benzoheterocycles **2c**-**g**.

2-(2-Propyn-1-yl)-1*H*-isoindole-1,3-(2*H*)-dione (**2c**)

mp 141-143 °C (lit. 149 °C);[2] IR (KBr) ν_{max}/cm^{-1} 3293, 2965, 2115, 1769, 1715, 1469, 1429; ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.23 (t, 1H, *J* 2.4 Hz), 4.45 (d, 2H, *J* 2.4 Hz), 7.74 (m, 2H, phthalimide), 7.88 (m, 2H, phthalimide); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.0, 134.2, 131.9, 123.6, 77.1, 71.5, 26.9.

1-(2-Propyn-1-yl)-1*H*-benzimidazole (**2d**)

Oil (lit. 38-40 °C);[3] IR (KBr) ν_{max}/cm^{-1} 3176, 3099, 2113, 1500, 1458, 1288, 752; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.27 (s, 1H, N=CH), 7.85 (dd, 1H, *J* 1.6, 6.4 Hz), 7.52 (dd, 1H,

*e-mail: ronaldonoliveira@dcm.ufrpe.br

J 1.6, 6.4 Hz), 7.39-7.32 (m, 2H), 4.99 (d, 2H, *J* 2.8 Hz, CH_2-Het), 2.52 (t, 1H, *J* 2.8 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 142.5, 142.1, 134.5, 123.7, 123.1, 120.0, 109.9, 75.6, 75.2, 34.9.

2-(2-Propyn-1-ylthio)-benzothiazole (**2e**)

mp 40-42 °C (lit. 46 °C);[4] IR (KBr) ν_{max}/cm^{-1} 3272, 2965, 2121, 1453, 1425, 1390, 1000, 765; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.91 (ddd, 1H, *J* 0.6, 1.5, 8.1 Hz), 7.76 (ddd, 1H, *J* 0.6, 1.5, 8.1 Hz), 7.44 (dd, 1H, *J* 1.5, 7.5 Hz), 7.32 (ddd, 1H, *J* 1.5, 7.5, 7.5 Hz), 2.30 (t, 1H, *J* 2.4), 4.14 (d, 2H, *J* 2.4 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 164.6 (C=N), 153.0 (C_{Ar}–N), 135.4 (C_{Ar}–NH), 126.1, 124.5, 121.8, 121.1, 78.3 (C≡C), 72.3 (HC≡), 21.5 (CH_2).

2-(2-Propyn-1-ylthio)-1*H*-benzimidazole (**2f**)

mp 128-130 °C (lit. 153 °C);[5] IR (KBr) ν_{max}/cm^{-1} 3272, 2977, 2121, 1655, 1454, 1426, 1391; ^{1}H NMR (300 MHz, DMSO-d_6) δ 8.06 (dd, 1H, *J* 1.5, 8.1 Hz), 7.90 (dd, 1H, *J* 0.6, 8.1 Hz), 7.50 (ddd, 1H, *J* 1.5, 7.8, 7.8 Hz), 7.39 (ddd, 1H, *J* 1.5, 8.1, 8.1 Hz), 4.25 (d, 2H, *J* 2.4 Hz), 3.26 (t, 1H, *J* 2.4); ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.1 (C=N), 152.6 (C_{Ar}–N), 134.9 (C_{Ar}–NH), 126.5, 124.7, 122.0, 121.4, 79.4 (C≡C), 74.7 (HC≡), 21.1 (CH_2).

2-(2-Propyn-1-ylthio)-benzoxazole (**2g**)

mp 47-48 °C (lit. 51 °C);[6] IR (KBr) ν_{max}/cm^{-1} 3264, 2970, 2125, 1504, 1452, 1235, 1132, 738; ^{1}H NMR (300 MHz, DMSO-d_6) δ 7.71-7.65 (m, 2H), 7.39-7.31 (m, 2H), 4.22 (d, 2H, *J* 3.6 Hz), 3.32 (t, 1H, *J* 3.0 Hz); ^{13}C NMR (75 MHz, DMSO-d_6) δ 162.8 (C=N), 151.4 (C_{Ar}–O), 141.2 (C_{Ar}–N), 124.8, 124.6, 118.5 (β-C_{Ar}–N), 110.4 (β-C_{Ar}–N), 79.3 (C≡C), 74 .6 (HC≡), 20.3 (CH_2).

Synthesis of *N*-glucosyl-1,2,3-triazoles (**3a**-**g**)

A solution of 10 mol% Et_3N (0.032 mmol, ca. 1 drop) and 10 mol% copper iodide (0.032 mmol) in CH_2Cl_2 (2 mL) were successively added to a mixture of the corresponding alkyne **2a**-**g** (0.32 mmol, 1.2 equiv.) and the azidosugar **1** (100 mg, 0.268 mmol, 1 equiv.) in CH_2Cl_2 (2 mL). The mixture was irradiated for 20-30 min at room temperature with ultrasound energy, under thin layer chromatographic (TLC) monitoring of the progress of the reaction. After dilution with cold water, the organic layer was separated and the aqueous phase was extracted with CH_2Cl_2 (3 × 15 mL), followed by washing the organic layer with saturated brine, then water. The combined organic layers were dried over anhydrous Na_2SO_4, filtered and the solvent evaporated under reduced pressure. The resulting mixture was chromatographed on silica gel using cyclohexane:EtOAc as eluent, which after work-up furnished compounds **3a**-**g** as colourless solids. The final product was crystallized from methylene chloride/cyclohexane.

1'-(2,3,4,6-Tetra-*O*-acetyl-β-D-glucopyranosyl)-4'-phenyl-1*H*-1',2',3'-triazole (**3a**)

Solid; mp 189-193 °C; $[\alpha]_D^{26}$ −19 (*c*1, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (s, 1H, $H_{triazole}$), 7.83 (d, 2H, *J* 7.2 Hz, $H_{arom.}$), 7.42 (dd, 2H, *J* 7.2, 8.0 Hz, H_{arom}), 7.34 (dd, 1H, *J* 7.2, 7.6 Hz, H_{arom}), 5.93 (d, 1H, *J* 9.2 Hz, H-1), 5.26-5.52 (3 × dd, 3H, *J* 9.2, 9.2, 9.6 Hz, H-2, H-3, H-4), 4.32 (dd, 1H, *J* 4.8, 12.8 Hz, H-6a), 4.16 (dd, 1H, *J* 2.0, 12.4 Hz, H-6b), 4.04 (ddd, 1H, *J* 2.0, 4.2, 10.0 Hz, H-5), 2.08 (s, 3H, CH_3CO), 2.07 (s, 3H, CH_3CO), 2.03 (s, 3H, CH_3CO), 1.87 (s, 3H, CH_3CO).

1'-(2,3,4,6-Tetra-*O*-acetyl-β-D-glucopyranosyl)-4'-propyl-1*H*-1',2',3'-triazole (**3b**)

Yellow solid; mp 153-155 °C; $[\alpha]_D^{26}$ −33 (*c*1, CH_2Cl_2); IR (KBr) ν_{max}/cm^{-1} 3483, 3074, 2960, 2872, 1739, 1558, 1368, 1217, 1039, 928; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 (s, 1H, $H_{triazole}$), 5.83 (d, 1H, *J* 8.8 Hz, H-1), 5.43-5.35 (m, 2H, H-2, H-3), 5.21 (dd, 1H, *J* 9.2, 9.6 Hz, H-4), 4.26 (dd, 1H, *J* 4.8, 12.8 Hz, H-6a), 4.10 (br d, 1H, *J* 12.8 Hz, H-6b), 3.99 (ddd, 1H, *J* 1.6, 4.4, 10.0 Hz, H-5), 2.65 (t, 2H, *J* 7.6 Hz, CH_2-Het), 2.03 (s, 3H, CH_3CO), 2.02 (s, 3H, CH_3CO), 1.81 (s, 3H, CH_3CO), 1.98 (s, 3H, CH_3CO), 1.65 (m, 2H, CH_2), 0.91 (t, 3H, *J* 7.2 Hz, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.4, 169.8, 169.3, 168.8, 148.8 (C-4'), 118.8 (C-5'), 85.5 (C-1), 74.9, 72.4, 70.1, 67.7, 61.5, 27.4 (CH_2-Het), 22.3 (CH_2), 20.5, 20.4, 20.4, 20.0, 13.5 (5 × CH_3); anal. calcd. for $C_{19}H_{27}N_3O_9$•(1/2 × H_2O) C, 50.56; H, 6.28; found: C, 50.91; H, 6.57. HRMS [($C_{19}H_{27}N_3O_9$) + H] calcd.: 442.1826; found: 442.1759.

1'-(2,3,4,6-Tetra-*O*-acetyl-β-D-glucopyranosyl)-4'-(methyl-1*H*-isoindole-1,3-(2*H*)-dione)-1*H*-1',2',3'-triazole (**3c**)

Solid; mp 131-134 °C; $[\alpha]_D^{26}$ −22 (*c*1, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (m, 2H, phthalimide), 7.82 (s, 1H, $H_{triazole}$), 7.72-7.70 (m, 2H, phthalimide), 5.83 (d, 1H, *J* 8.8 Hz, H-1), 5.42-5.35 (m, 2H, H-2, H-3), 5.20 (dd, 1H, *J* 9.6, 10.0 Hz, H-4), 5.00 (2 × d, 2H, *J* 15.2 Hz, CH_2-Het), 4.27 (dd, 1H, *J* 4.8, 12.8 Hz, H-6a), 4.11 (dd, 1H, *J* 2.0, 12.8 Hz, H-6b), 3.96 (ddd, 1H, *J* 2.4, 4.8, 10.0 Hz, H-5), 2.07 (s, 3H, CH_3CO), 2.05 (s, 3H, CH_3CO), 2.01 (s, 3H, CH_3CO), 1.83 (s, 3H, CH_3CO).

1'-(2,3,4,6-Tetra-*O*-acetyl-β-D-glucopyranosyl)-4'-(*N*-methyl-1*H*-benzimidazol-1-yl)-1*H*-1',2',3'-triazole (**3d**)

Solid; mp 182-184 °C; $[\alpha]_D^{26}$ −21 (*c*1, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (s, 1H, $H_{triazole}$), 7.64-

7.25 (m, 5H, H_{arom}), 5.81 (d, 1H, *J* 8.8 Hz, H-1), 5.48 (s, 2H, CH_2-Het), 5.40-5.29 (m, 2H, H-2, H-3), 5.17 (dd, 1H, (dd, 1H, *J* 9.6, 9.6 Hz, H-4), 4.25 (dd, 1H, *J* 4.6,12.6 Hz, H-6a), 4.09 (br d, 1H, *J* 12.6 Hz, H-6b), 3.96 (br dd, 1H, *J* 3.6, 10.0 Hz, H-5), 2.03 (s, 3H, CH_3CO), 2.02 (s, 3H, CH_3CO), 1.99 (s, 3H, CH_3CO), 1.78 (s, 3H, CH_3CO); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.4, 169.8, 169.2, 168.8, 161.4 (C=N), 143.4 (C-4' and C_{Ar}–N), 123.4, 122.3, 120.8 (C-5' and C_{Ar}–N), 109.7 (C_{Ar}–N), 85.8 (C-1), 75.2, 72.3, 70.3, 67.5, 61.4, 20.6, 20.6, 20.5, 20.5, 20.4 (CH_2-Het and 4 × CH_3CO); anal. calcd. for $C_{24}H_{27}N_5O_9$: C, 54.44; H, 5.14; found: C, 54.71; H, 5.47.

1'-(2,3,4,6-Tetra-*O*-acetyl-β-D-glucopyranosyl)-4'-(benzothiazol-2-ylsulfanyl)methyl-1*H*-1',2',3'-triazole (**3e**)

Solid; mp 152-153 °C; $[\alpha]_D^{26}$ −18 (*c*1, CH_2Cl_2); IR (KBr) ν_{max}/cm^{-1} 3467, 3111, 3069, 2954, 1759, 1429, 1369, 945; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87-7.84 (m, 2H, H_{arom}, $H_{triazole}$), 7.70 (d, 1H, *J* 8.0 Hz, H_{arom}), 7.37 (dd, 1H, *J* 7.6, 7.6 Hz, H_{arom}), 7.24 (dd, 1H, *J* 7.6, 8.0 Hz, H_{arom}), 5.82 (d, 1H, *J* 8.8 Hz, H-1), 5.38 (m, 2H, H-2, H-3), 5.21 (dd, 1H, *J* 9.6, 9.6 Hz, H-4), 4.64 (2xd, 2H, *J* 14.4 Hz, CH_2-Het), 4.21 (dd, 1H, *J* 4.8, 12.4 Hz, H-6a), 4.07 (br d, 1H, 12.4 Hz, H-6b), 3.96 (ddd, 1H, *J* 2.0, 4.8, 10.0 Hz, H-5), 2.00 (s, 3H, CH_3CO), 1.97 (s, 3H, CH_3CO), 1.95 (s, 3H, CH_3CO), 1.69 (s, 3H, CH_3CO); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.2, 169.5, 169.1, 168.5, 165.3 (C=N), 152.8 (C_{Ar}–N), 144.4 (C-4'), 135.2 (C_{Ar}-S), 125.9 (C-5'), 124.2, 121.4, 121.3, 120.9, 85.4 (C-1), 74.8, 72.5, 69.9, 67.5, 61.3, 27.4 (CH_2-Het), 20.4, 20.3, 20.3, 19.8; anal. calcd. for $C_{24}H_{26}N_4O_9S_2$: C, 49.82; H, 5.19; found: C, 50.20; H, 4.88.

1'-(2,3,4,6-Tetra-*O*-acetyl-β-D-glucopyranosyl)-4'-(benzimidazol-2-ylsulfanyl)methyl-1*H*-1',2',3'-triazole (**3f**)

Brown solid; mp 156-158 °C; $[\alpha]_D^{26}$ −21 (*c*1, CH_2Cl_2); IR (KBr) ν_{max}/cm^{-1} 3111, 3069, 2955, 2851, 1741, 1459, 1429, 1369, 1219, 1101, 1064, 946; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, 1H, *J* 8.0 Hz, H_{arom}), 7.85 (s, 1H, $H_{triazole}$), 7.74 (d, 1H, *J* 7.6 Hz, H_{arom}), 7.44-7.40 (dd, 1H, *J* 1.2, 8.0 Hz, H_{arom}), 7.32-7.28 (m, 1H, H_{arom}), 5.82 (d, 1H, *J* 8.8 Hz, H-1), 5.43-5.34 (m, 2H, H-2, H-3), 5.20 (dd, 1H, *J* 9.2, 9.6 Hz, H-4), 4.68 (2d, 2H, *J* 14.8 Hz, CH_2-Het), 4.25 (dd, 1H, *J* 4.8, 13.0 Hz, H-6a), 4.10 (dd, 1H, *J* 1.6, 13.0 Hz, H-6b), 3.97 (ddd, 1H, *J* 2.8, 5.0, 8.4 Hz, H-5), 2.04 (s, 3H, CH_3CO), 2.02 (s, 3H, CH_3CO), 2.00 (s, 3H, CH_3CO), 1.78 (br s, 1H, NH), 1.75 (s, 3H, CH_3CO); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.4, 169.8, 169.3, 168.7, 165.4 (C=N), 152.9 (C_{Ar}–N), 144.7 (C-4'), 135.4 (C_{Ar}–NH), 126.03 (C-5'), 124.4, 121.6, 121.3, 121.0, 85.6, 75.0, 72.6, 70.0, 67.6, 61.4, 27.5 (CH_2-Het), 21.5, 20.4, 20.0, 19.8; anal. calcd. for $C_{24}H_{27}N_5O_9S$: C, 51.33; H, 4.85; found: C, 51.63; H, 4.95.

1'-(2,3,4,6-Tetra-*O*-acetyl-β-D-glucopyranosyl)-4'-(benzoxazol-2-ylsulfanyl)methyl-1*H*-1',2',3'-triazole (**3g**)

Solid; mp 161-162 °C; $[\alpha]_D^{26}$ −26 (*c*1, CH_2Cl_2); IR (KBr) ν_{max}/cm^{-1} 3087, 2966, 1748, 1500, 1456, 1374, 1222, 1133, 1044, 943; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (s, 1H, $H_{triazole}$), 7.64 (d, 1H, *J* 7.6 Hz, H_{arom}), 7.40 (dd, 1H, *J* 0.8, 7.6 Hz, H_{arom}), 7.32-7.23 (m, 2H, H_{arom}), 5.83 (d, 1H, *J* 9.2 Hz, H-1), 5.55-5.35 (m, 2H, H-3, H-4), 5.21 (dd, 1H, *J* 9.2, 10.0 Hz, H-4), 4.67-4.57 (2xd, 2H, *J* 14.8 Hz, CH_2-Het), 4.25 (dd, 1H, *J* 4.8, 12.8 Hz, H-6a), 4.11 (dd, 1H, *J* 1.6, 12.8 Hz, H-6b), 3.97 (ddd, 1H, *J* 2.0, 4.8, 10.0 Hz, H-5), 2.05 (s, 3H, CH_3CO), 2.03 (s, 3H, CH_3CO), 2.00 (s, 3H, CH_3CO), 1.75 (s, 3H, CH_3CO); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.4, 169.9; 169.3, 168.7, 163.9 (C=N), 152.1 (C_{Ar}–O), 144.3 ($C_{4'}$), 141.8 (C_{Ar}–N), 124.3, 124.0, 121.3 (C-5'), 118.6, 1010 (C_{Ar}–O), 85.7 (C-1), 75.1, 72.6, 70.1, 67.6, 61.4, 26.6 (CH_2–Het), 20.6, 20.6, 20.5, 20.4; anal. calcd. for $C_{24}H_{26}N_4O_{10}S$: C, 51.24; H, 4.66; found: C, 51.04; H, 4.77.

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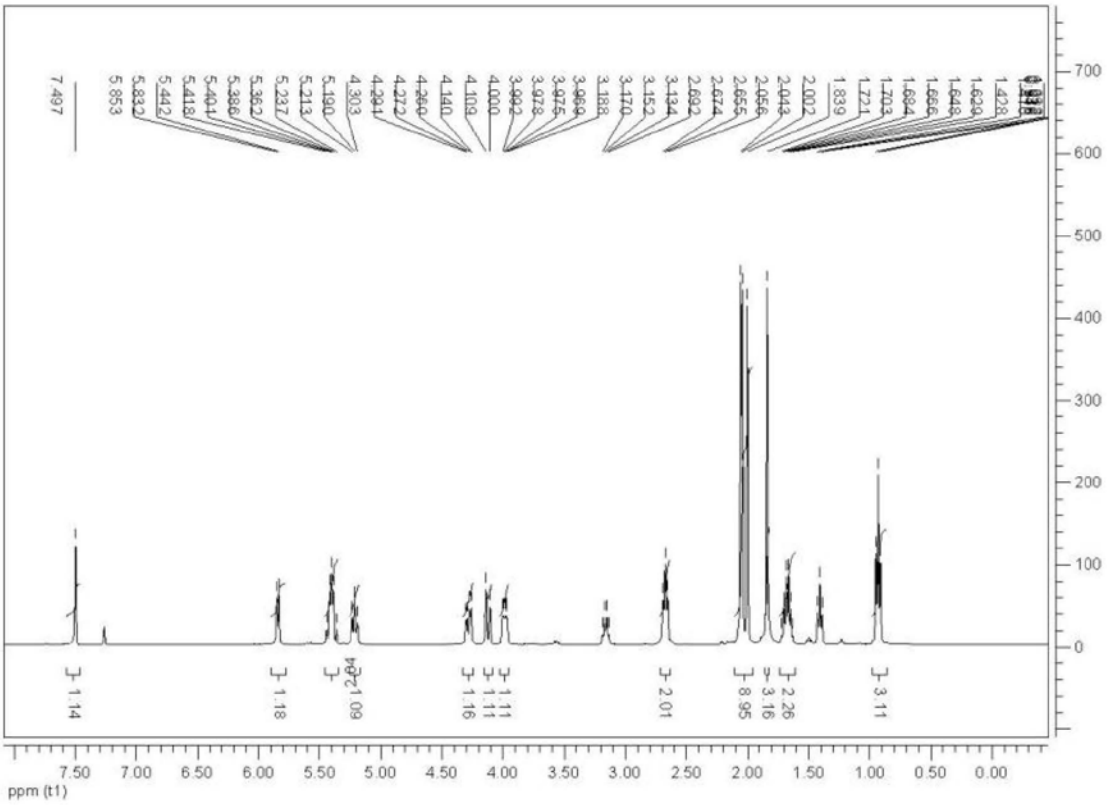

Figure S1. ^{1}H NMR (400 MHz) spectrum of **3b** in $CDCl_3$.

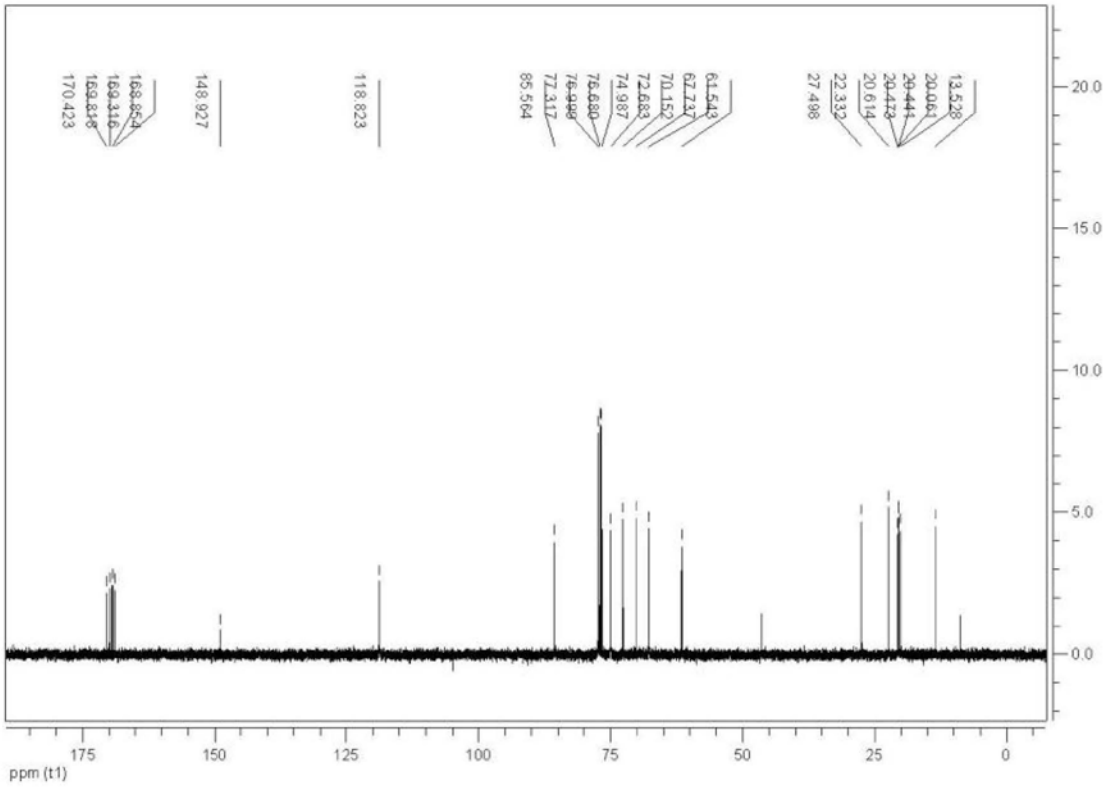

Figure S2. ^{13}C NMR (100 MHz) spectrum of **3b** in $CDCl_3$.

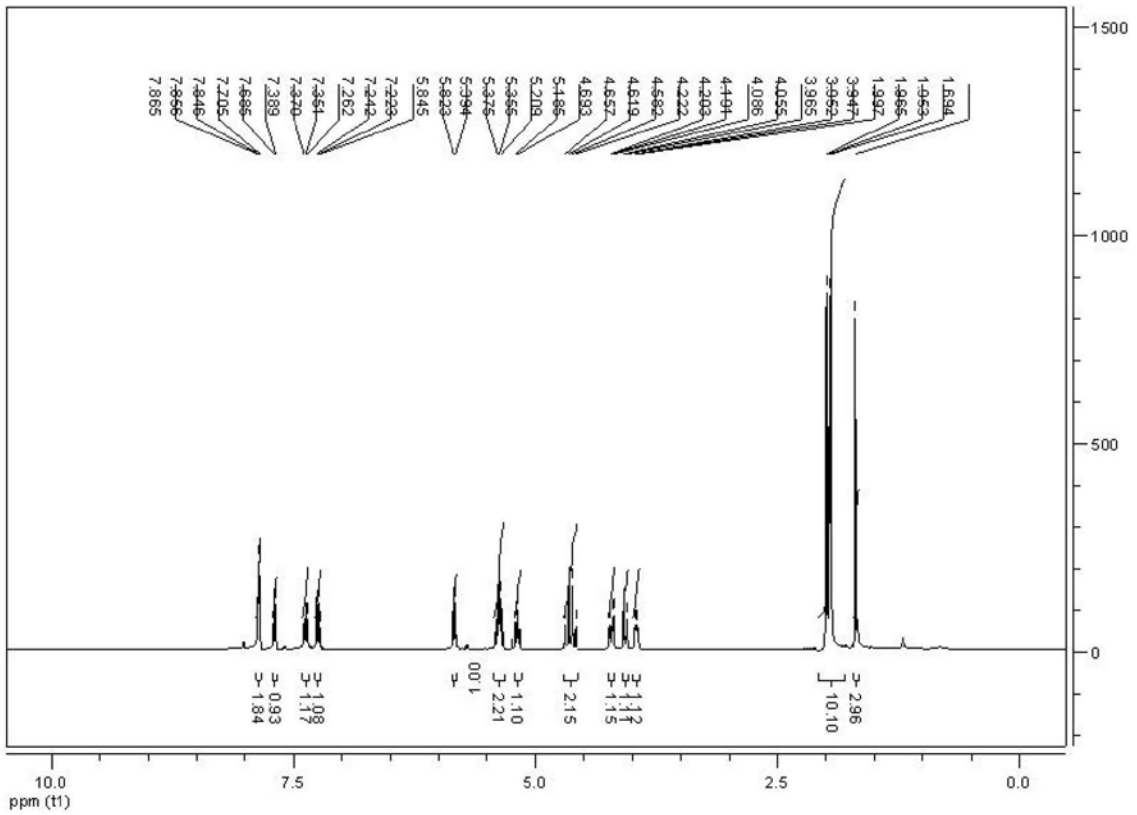

Figure S3. ^{1}H NMR (400 MHz) spectrum of **3e** in $CDCl_3$.

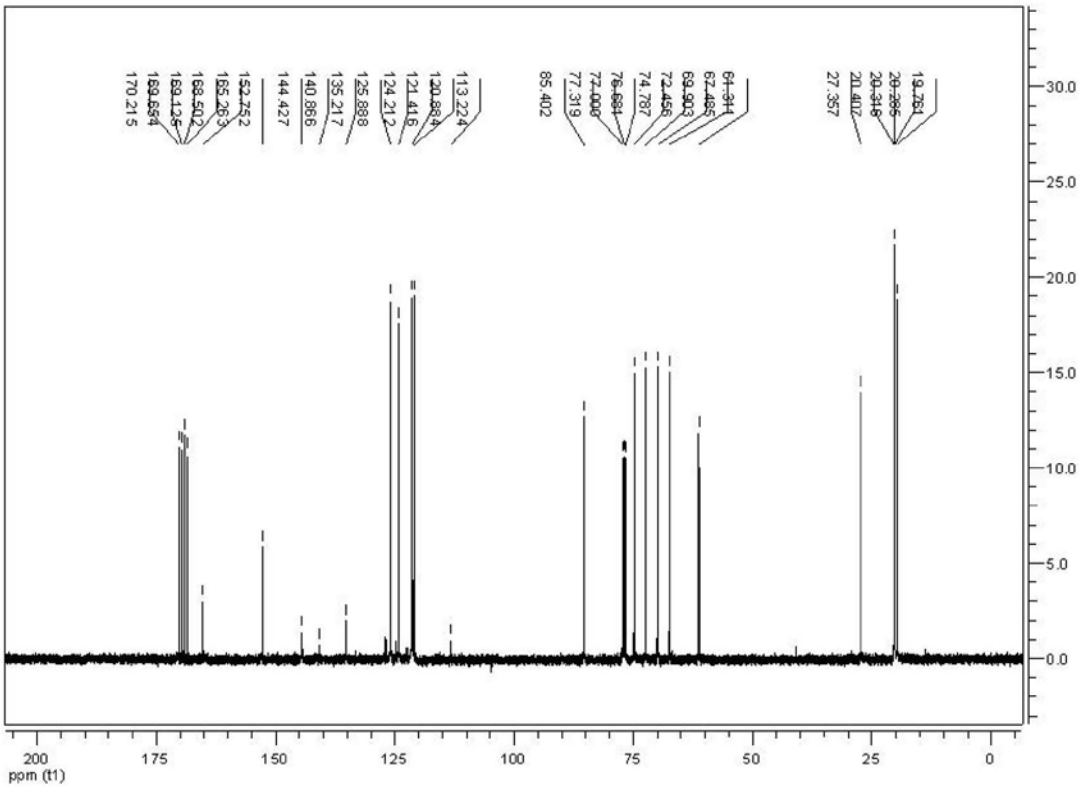

Figure S4. ^{13}C NMR (100 MHz) spectrum of **3e** in $CDCl_3$.

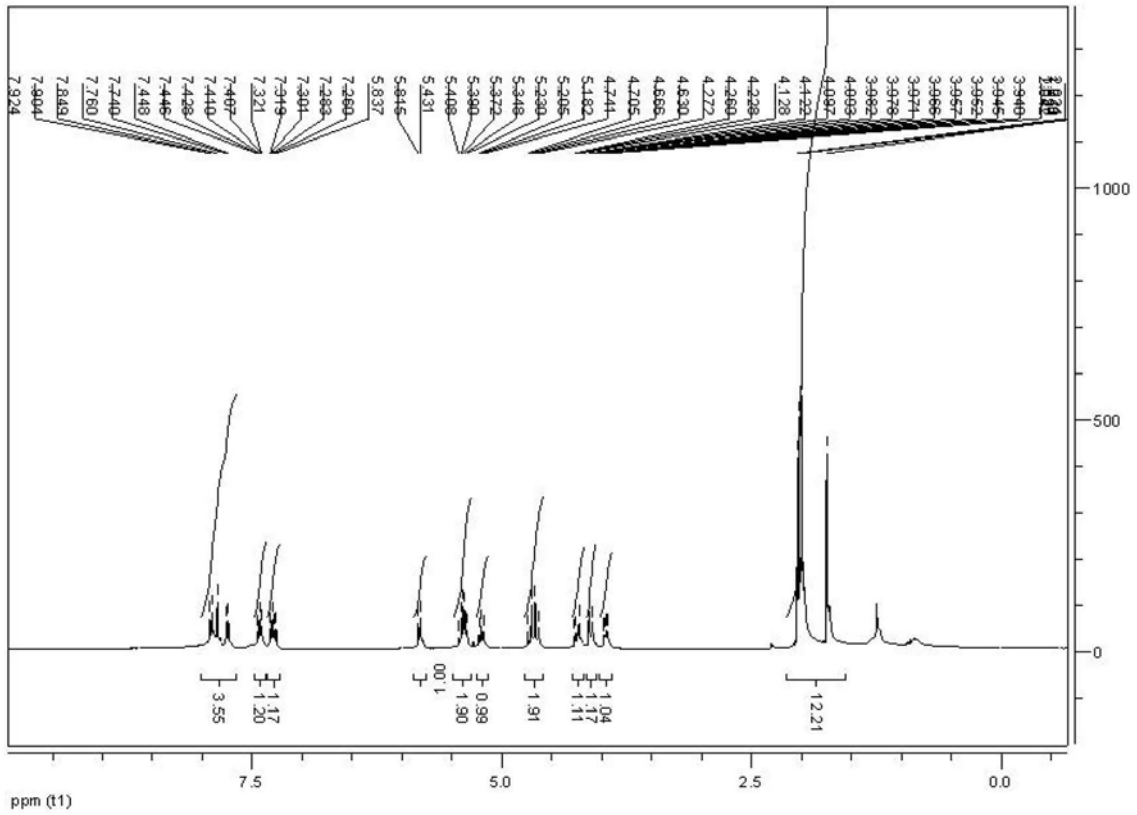

Figure S5. ^{1}H NMR (400 MHz) spectrum of **3f** in $CDCl_3$.

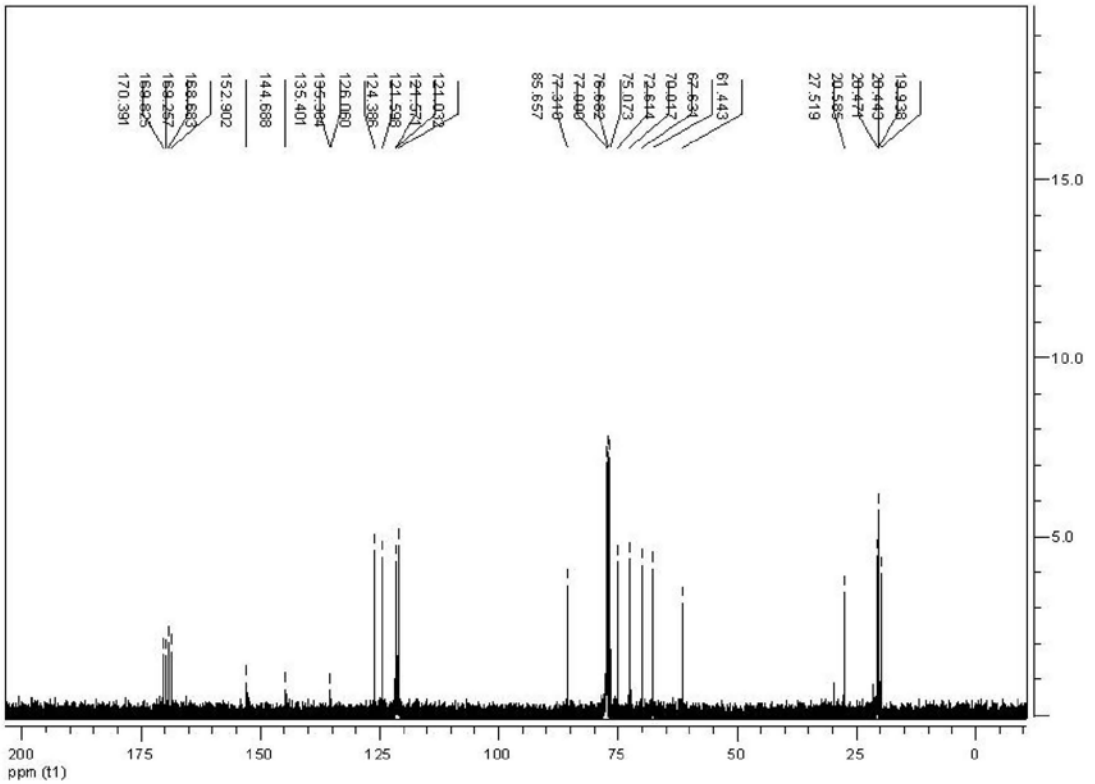

Figure S6. ^{13}C NMR (100 MHz) spectrum of **3f** in $CDCl_3$.

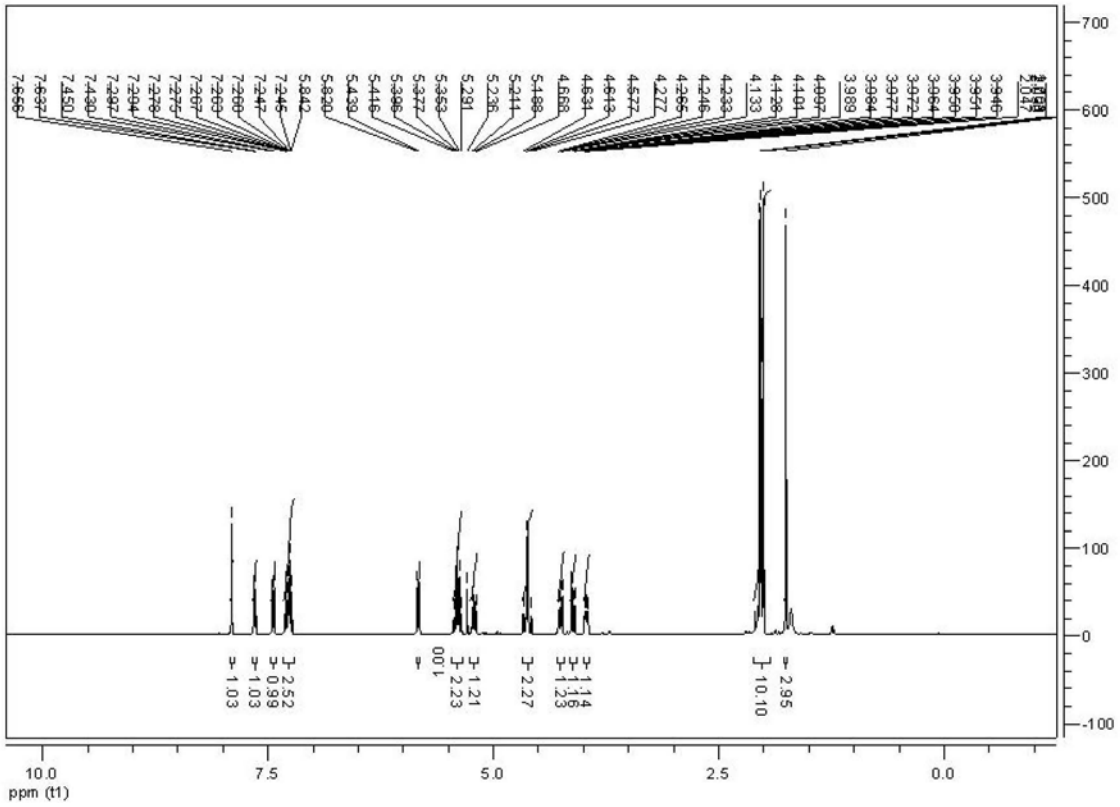

Figure S7. ^{1}H NMR (400 MHz) spectrum of **3g** in $CDCl_3$.

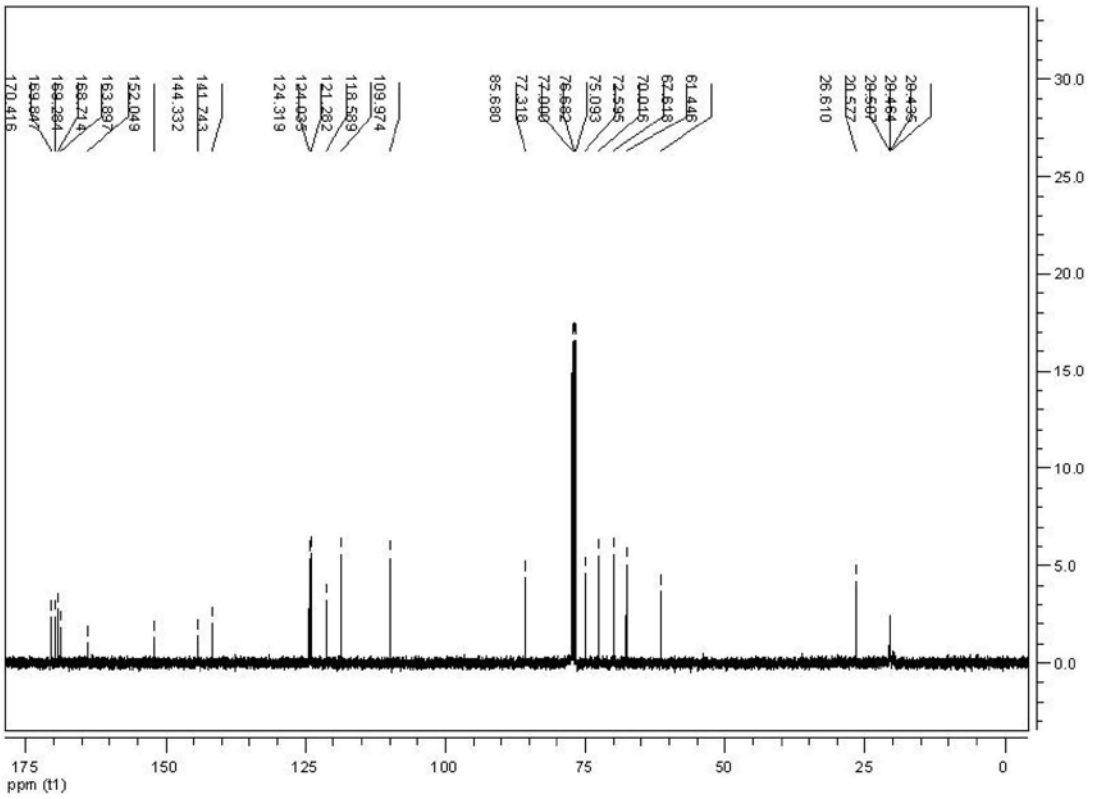

Figure S8. ^{13}C NMR (100 MHz) spectrum of **3g** in $CDCl_3$.

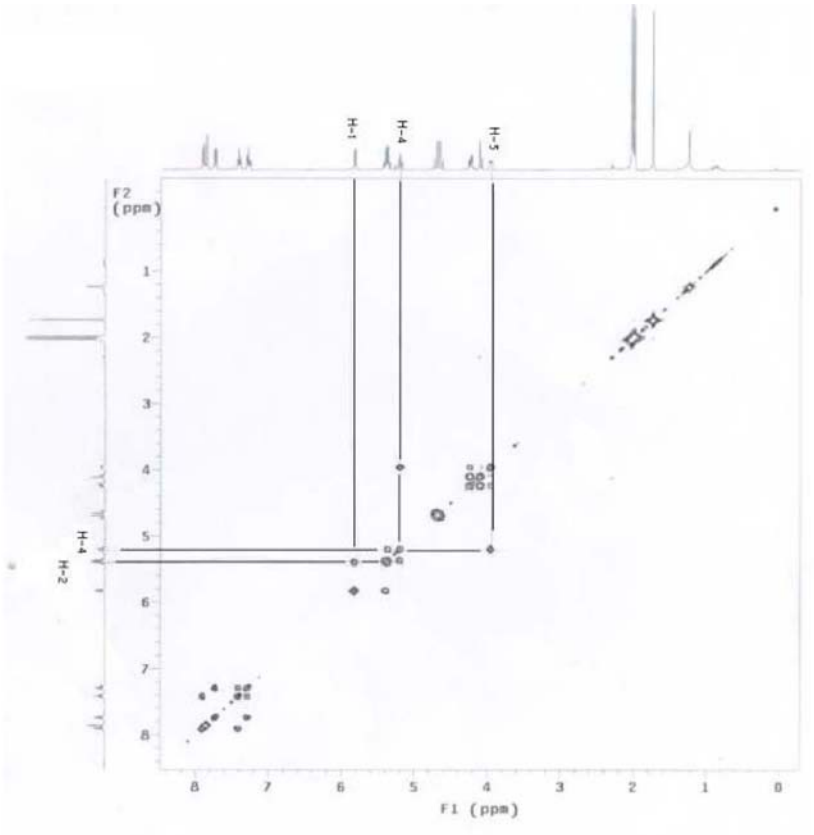

Figure S9. H,H-COSY (400 MHz) spectrum of **3e** in $CDCl_3$.

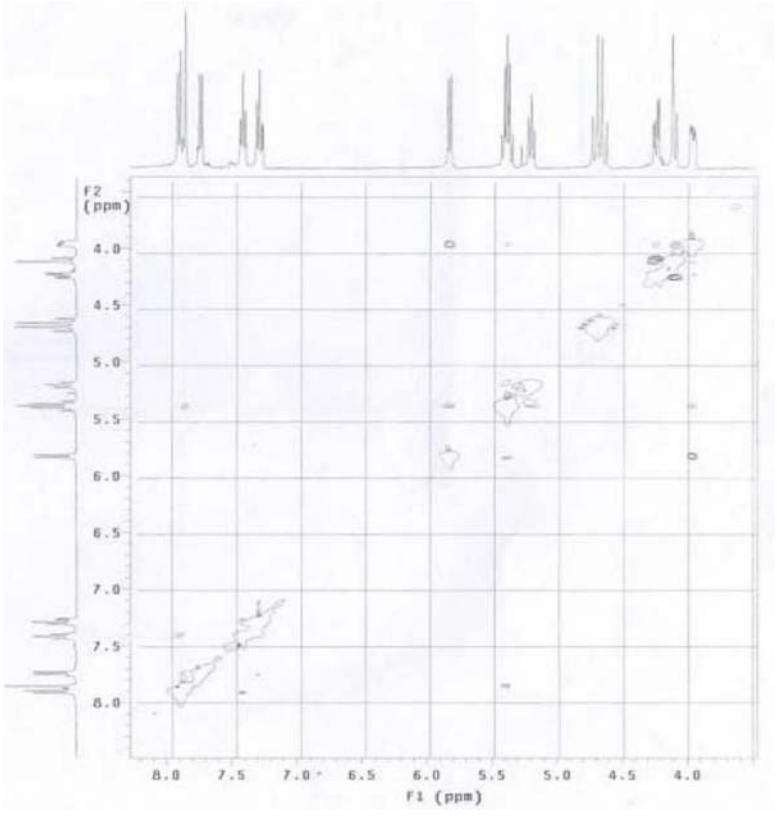

Figure S10. NOESY (400 MHz) spectrum of **3e** in $CDCl_3$.

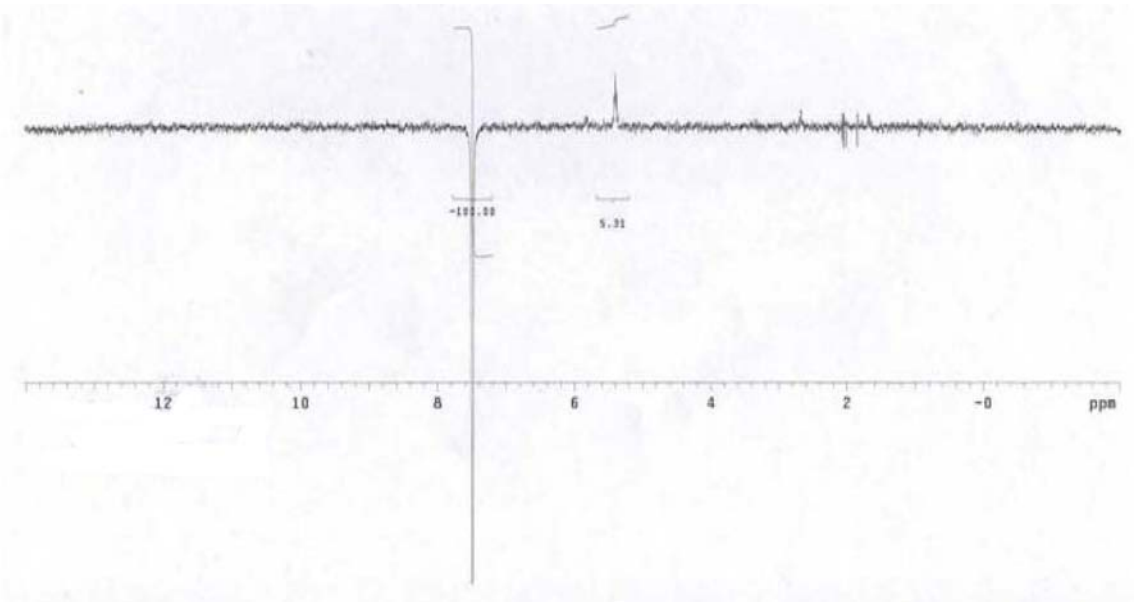

Figure S11. NOE DIFF (400 MHz) spectrum of **3b** in $CDCl_3$ (H_5, irradiated).

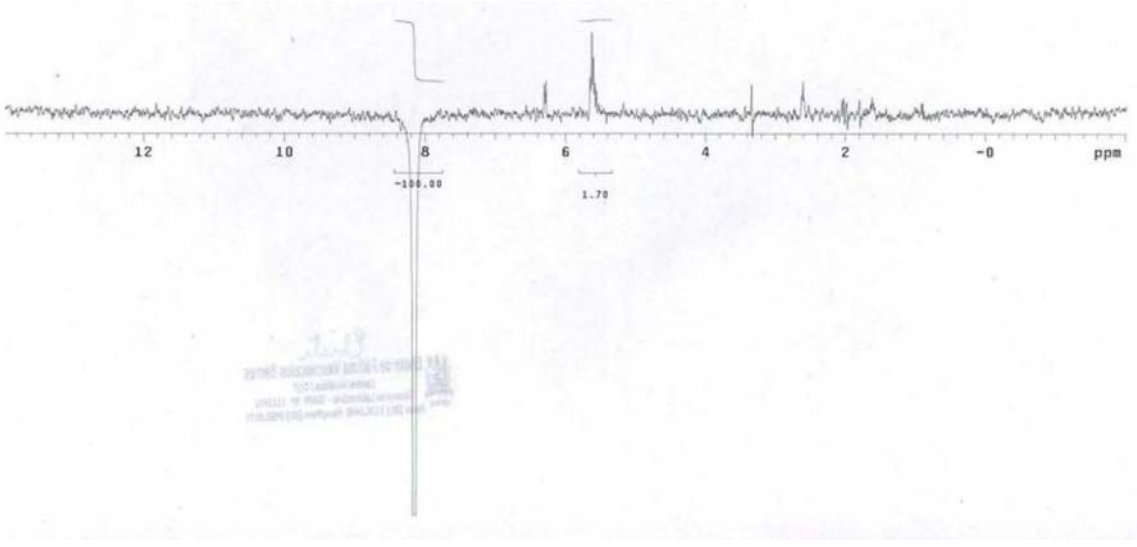

Figure S12. NOE DIFF (400 MHz) spectrum of **3b** in DMSO (H_5, irradiated).

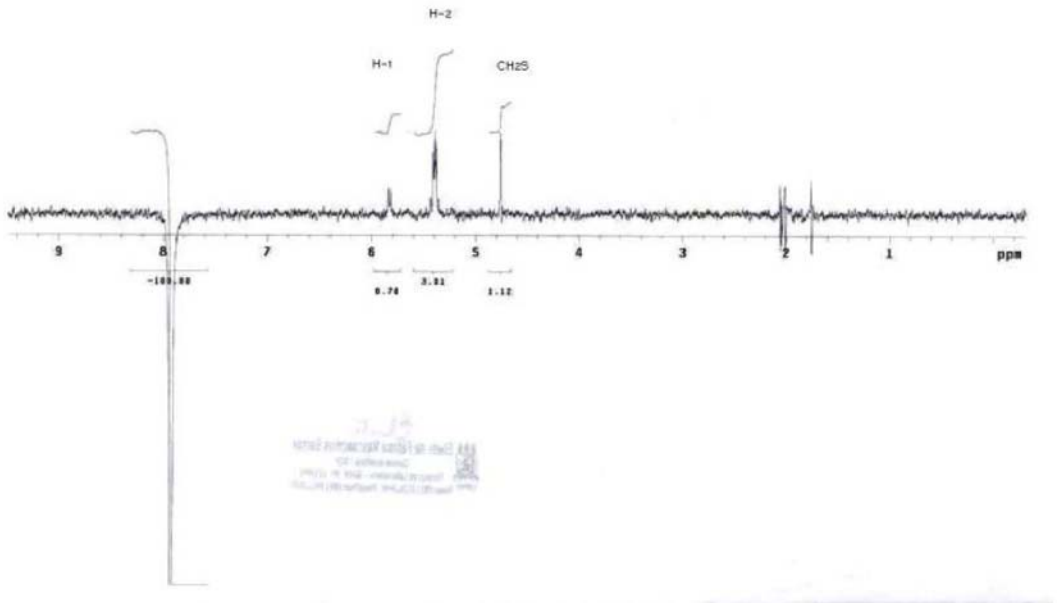

Figure S13. NOE DIFF (400 MHz) spectrum of **3e** in $CDCl_3$ (H_5, irradiated) .

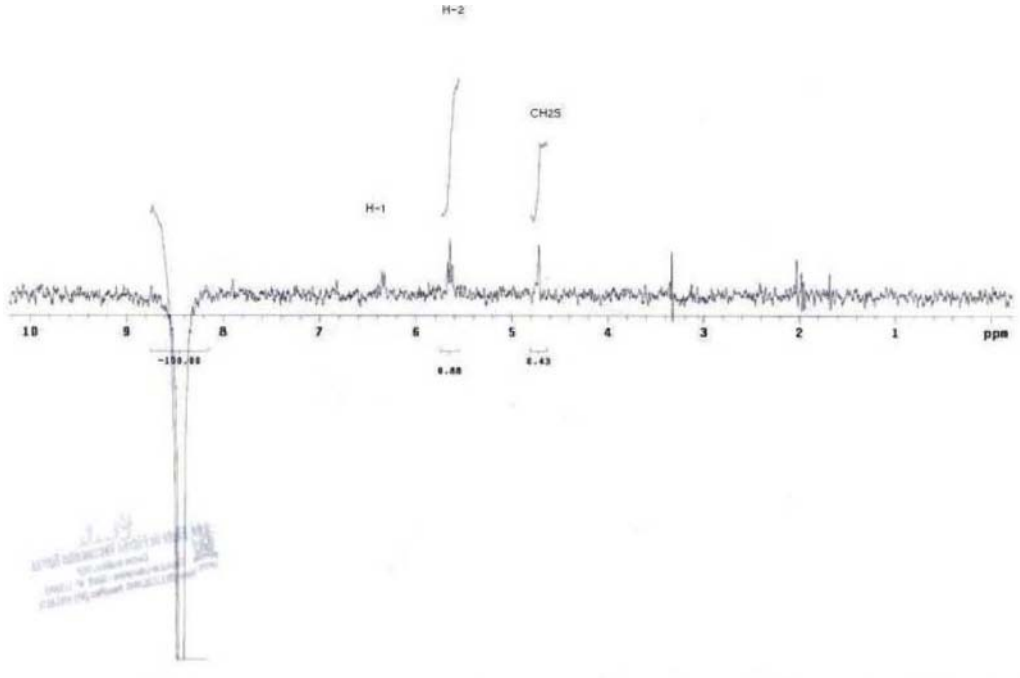

Figure S14. NOE DIFF (400 MHz) spectrum of **3e** in DMSO (H_5, irradiated).

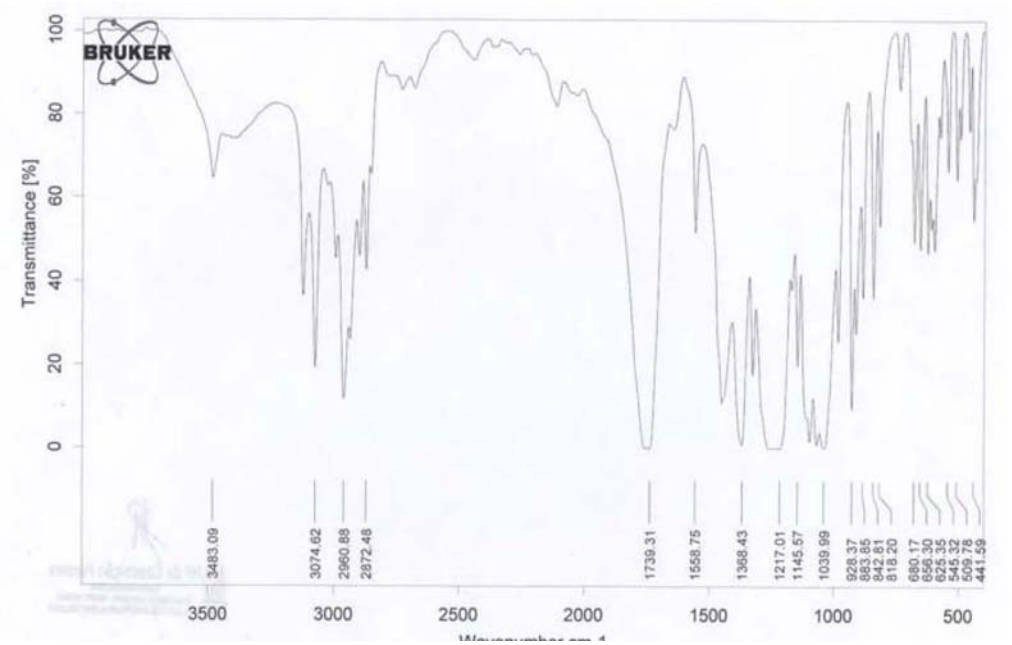

Figure S15. IR spectrum of **3b**.

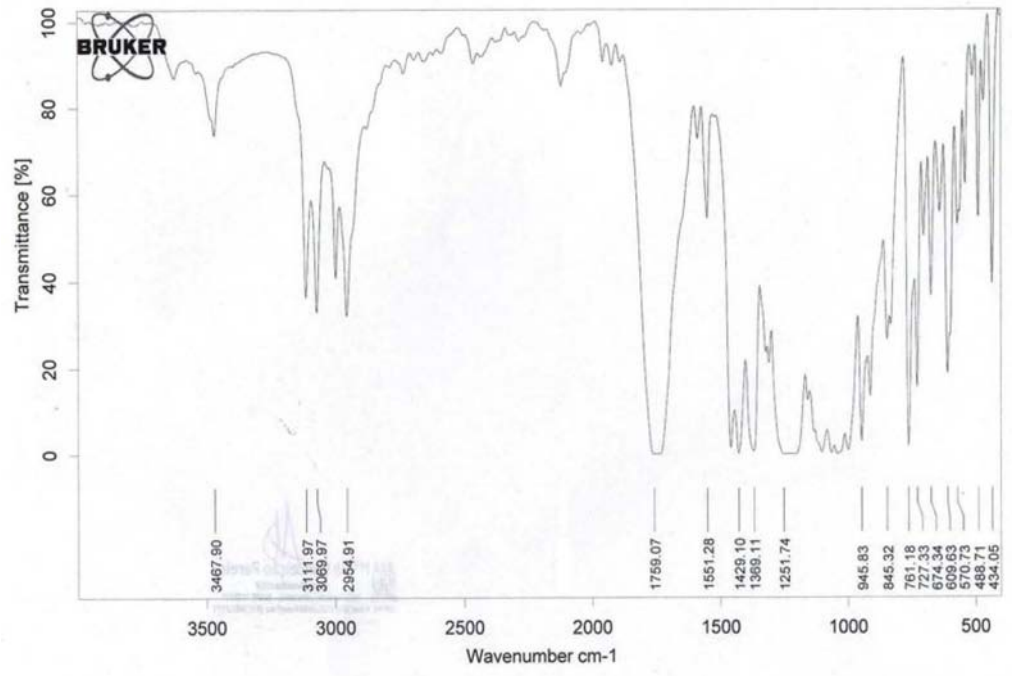

Figure S16. IR spectrum of **3e**.

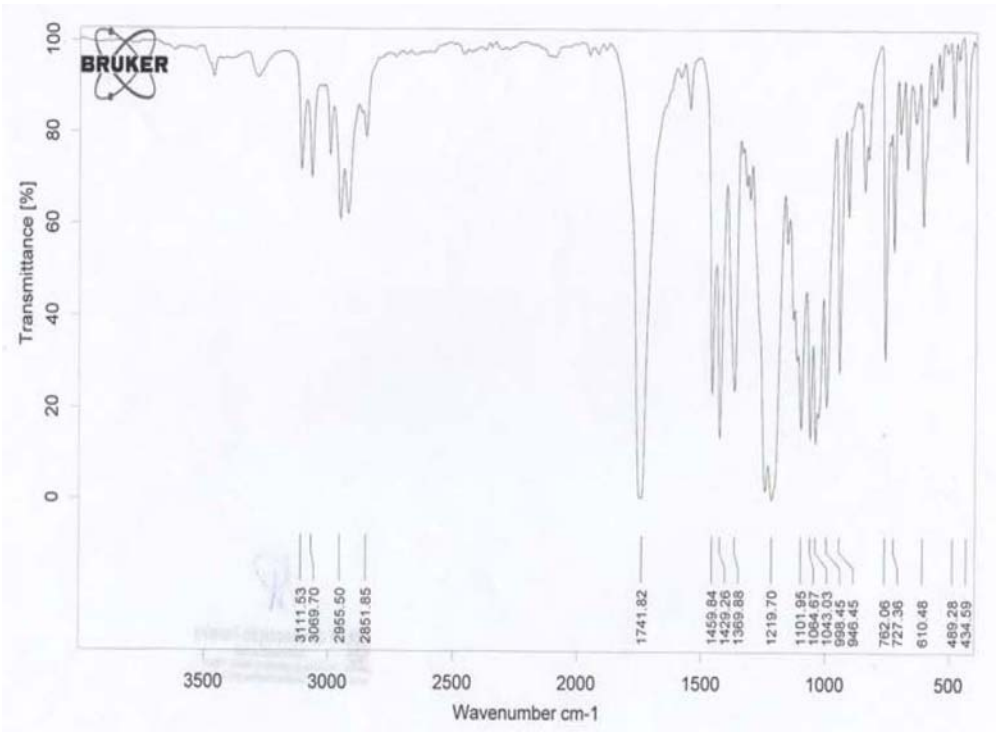

Figure S17. IR spectrum of **3f**.

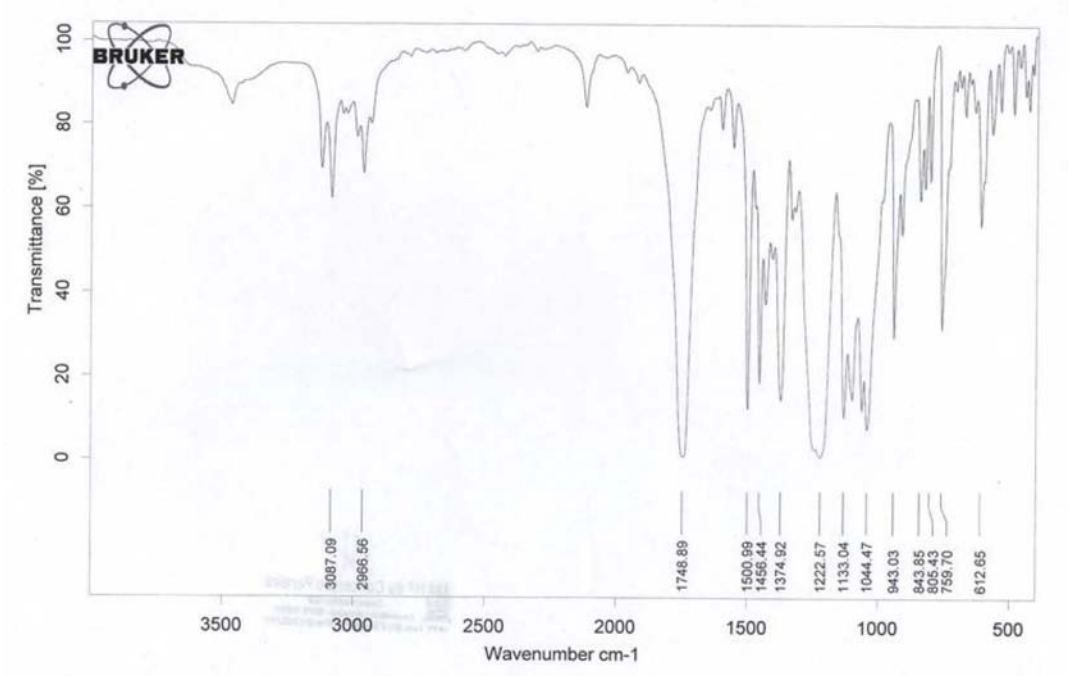

Figure S18. IR spectrum of **3g**.